REGISTRATION REPORT

# Part B

# Section 7

# Metabolism and Residues

Detailed summary of the risk assessment

Product code: ADM.4651.H.1.A (former A18032E)

Product name: NIKITA

Chemical active substances:

Dicamba, 312.5 g/kg

Mesotrione, 150 g/kg

Nicosulfuron, 100 g/kg

Central Zone

Zonal Rapporteur Member State: Poland

CORE ASSESSMENT

Sponsor: ADAMA

Applicant: ADAMA

Submission date: June 2020

MS Finalisation date: March 2022 (initial Core Assessment)

June 2022 (final Core Assessment)

# Version history

| When | What |
|---|---|
| June 2020 | Applicant initial dRR |
| January 2021 | Consumer risk assessment has been updated in the dRR by Applicant. |
| March 2022 | Initial assessment by the zRMS<br>The report in the dRR format has been prepared by the Applicant, therefore all comments, additional evaluations and conclusions of the zRMS are presented in grey commenting boxes. Minor changes are introduced directly in the text and highlighted in grey. Not agreed or not relevant information are ~~struck through and shaded for transparency~~. |
| June 2022 | Final report (Core Assessment updated following the commenting period).<br>Additional information/assessments included by the zRMS in the report in response to comments recieved from the cMS and the Applicant are highlighted in yellow. Information no longer relevant ~~is struck through and shaded~~. |

ADAMA use the code ADM.4651.H.1.A for the formulation but for consistency the former Syngenta code A18032E is used throughout the dRR

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# 7 Metabolism and residue data (KCA section 6)

## 7.1 Summary and zRMS Conclusion

### 7.1.1 Critical GAP(s) and overall conclusion

**Selection of critical uses and justification**
The critical GAPs with respect to consumer intake and risk assessment for A18032E are presented in Table 7.1-1. They have been selected from the individual GAPs in the central zone for maize. A list of all intended uses within the central zone is given in Part B, Section 0.

**Overall conclusion**
The data available are considered sufficient for risk assessment. An exceedance of the current MRL of 0.5 mg/kg for dicamba, 0.01* mg/kg for mesotrione and nicosulfuron as laid down in Reg. (EU) 396/2005 is not expected.
The chronic and the short-term intakes of dicamba, mesotrione and nicosulfuron residues are unlikely to present a public health concern.
As far as consumer health protection is concerned, zRMS Poland agrees with the authorization of the intended use(s).

According to available data, no specific mitigation measures should apply.

**Data gaps**
Noticed data gaps are: None.

**Table 7.1-1: Acceptability of critical GAPs (and respective fall-back GAPs, if applicable)**

GAP , date: March 2022

| | | | |
|---|---|---|---|
| PPP (product name/code): | NIKITA/A18032E | Formulation type: | WG [a, b] |
| Active substance 1: | Mesotrione | Conc. of as 1: | 150 g/kg [c] |
| Active substance 2: | Dicamba | Conc. of as 2: | 312.5 g/kg [c] |
| Active substance 3: | Nicosulfuron | Conc. of as 3: | 100 g/kg [c] |
| Safener: | None | Conc. of safener: | Not applicable |
| Synergist: | None | Conc. of synergist: | Not applicable |
| Applicant: | ADAMA | Professional use: | ☒ |
| Zone(s): | Central | Non professional use: | ☐ |
| Verified by MS: | No | | |
| Field of use: | Herbicide | | |

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 11 | 11 | 12 | 13 | 14 | 15 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Use-No.** [e] | **Member state(s)** | **Crop and/ or situation (crop destination / purpose of crop)** | **F, Fn, Fpn G, Gn, Gpn or I** | **Pests or Group of pests controlled** (additionally: developmental stages of the pest or pest group) | **Application** | | | | | | **Application rate** | | | **PHI** (days) | **Remarks:** e.g. g safener/synergist per ha | **Conclusion** |
| | | | | | Method / Kind | Timing / Growth stage of crop & season | Max. number a) per use b) per crop/ season | Min. interval between applications (days) | kg or L A18032E/ ha a) max. rate per appl. b) max. total rate per crop/season | g or kg as/ha Mesotrione a) max. rate per appl. b) max. total rate per crop/season | g or kg as/ha Dicamba a) max. rate per appl. b) max. total rate per crop/season | g or kg as/ha Nicosulfuron a) max. rate per appl. b) max. total rate per crop/season | Water L/ha min / max | | | |
| **Zonal uses (field or outdoor uses, certain types of protected crops)** | | | | | | | | | | | | | | | | |
| 1 | Poland | Maize (ZEAMX) | F | Annual/perennial grass and broadleaved weeds | Foliar, spraying, overall | - / BBCH 12-14 Spring | a) 1 b) 1 | n.a. | a) 0.4 b) 0.4 | a) 60 b) 60 | a) 125 b) 125 | a) 40 b) 40 | 80-400 | n.a. | Tank-mixed adjuvant needed (e.g. Adigor with 1.0 - 1.5 L/ha, STYK (alternative and exclusive ADAMA name: INSERT) with 0.2 L/ha, Olejan with 1.5 L/ha) | A |
| 2 | Poland | Maize (ZEAMX) | F | Annual/perennial grass and broadleaved weeds | Foliar, spraying, overall | - / BBCH 12-14 Spring | a) 1 b) 1 | n.a. | a) 0.4 b) 0.4 | a) 60 b) 60 | a) 125 b) 125 | a) 40 b) 40 | 80-400 | n.a. | Application in tank mix with 0.8 L/ha, Efica 960 EC | A |
| **Interzonal uses (use as seed treatment, in greenhouses (or other closed places of plant production), as post-harvest treatment or for treatment of empty storage rooms)** | | | | | | | | | | | | | | | | |
| None | | | | | | | | | | | | | | | | |

| **Minor uses according to Article 51 (zonal uses)** |
|---|
| None |
| **Minor uses according to Article 51 (interzonal uses)** |
| None |

F: professional field use, Fn: non-professional field use, Fpn: professional and non-professional field use, G: professional greenhouse use, Gn: non-professional greenhouse use, Gpn: professional and non-professional greenhouse use, I: indoor application
N/A: not applicable

Explanation for Column 15 “Conclusion”

| | |
|---|---|
| A | Exposure acceptable without risk mitigation measures, safe use |
| R | Further refinement and/or risk mitigation measures required |
| N | Exposure not acceptable, no safe use |

## 7.1.2 Summary of the evaluation

The preparation A18032E is composed of dicamba, mesotrione and nicosulfuron.

**Table 7.1-2: Toxicological reference values for the dietary risk assessment of mesotrione**

| Reference value | Source | Year | Value | Study relied upon | Safety factor |
|---|---|---|---|---|---|
| Dicamba – Dicamba and 5-OH-dicamba, free and conjugated | | | | | |
| ADI | EFSA | 2011 | 0.3 mg/kg bw/d | Rat, 2-generation study | 100 |
| ARfD | EFSA | 2011 | 0.3 mg/kg bw/d | Rabbit, teratology study | 100 |
| Mesotrione - Mesotrione | | | | | |
| ADI | EFSA | 2016 | 0.01 mg/kg bw/d | Mouse multi-generation study | 200 |
| ARfD | EFSA | 2016 | 0.02 mg/kg bw/d | Mouse multi-generation study | 100 |
| Nicosulfuron – Nicosulfuron | | | | | |
| ADI | EFSA | 2007 | 2 mg/kg bw/day | Chronic rat supported by subchronic dog | 100 |
| ARfD | EFSA | 2007 | Not Necessary | | |

### 7.1.2.1 Summary for dicamba

**Table 7.1-3: Summary for dicamba**

| Use-No.* | Crop | Plant metabolism covered? | Sufficient residue trials? | PHI sufficiently supported? | Sample storage covered by stability data? | MRL compliance | Chronic risk for consumers identified? | Acute risk for consumers identified? |
|---|---|---|---|---|---|---|---|---|
| 1, 2 | Maize | Yes | Yes (8 trials) | Yes | Yes | Yes | No | No |

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0 should be given in column 1

As residues of dicamba do not exceed the trigger values defined in Reg (EU) No 283/2013, there is no need to investigate the effect of industrial and/or household processing.

Residues in succeeding crops have been sufficiently investigated taking into account the specific circumstances of the cGAP uses being considered here. It is very unlikely that residues will be present in succeeding crops.

The requested use and the new mode of calculation modify the theoretical maximum daily intake for animals, but regarding available feeding data, there is no risk for animal MRL to be exceeded.

The proposed use of dicamba in the product NIKITA (A18032E) do not represent unacceptable acute and chronic risks for the consumer.

### 7.1.2.2 Summary for mesotrione

**Table 7.1-4: Summary for mesotrione**

| Use-No.* | Crop | Plant metabolism covered? | Sufficient residue trials? | PHI sufficiently supported? | Sample storage covered by stability data? | MRL compliance | Chronic risk for consumers identified? | Acute risk for consumers identified? |
|---|---|---|---|---|---|---|---|---|
| 1, 2 | Maize | Yes | Yes/No (14 trials) | Yes | Yes | Yes | No | No |

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0 should be given in column 1

As residues of mesotrione do not exceed the trigger values defined in Reg (EU) No 283/2013, there is no need to investigate the effect of industrial and/or household processing.

Residues in succeeding crops have been sufficiently investigated taking into account the specific circumstances of the cGAP uses being considered here. It is very unlikely that residues will be present in succeeding crops.

Considering dietary burden and based on the intended uses, no significant modification of the intake was calculated for livestock. Further investigation of residues as well as the modification of MRLs in commodities of animal origin is therefore not necessary.

The proposed use of mesotrione in the product NIKITA (A18032E) do not represent unacceptable acute and chronic risks for the consumer.

### 7.1.2.3 Summary for nicosulfuron

**Table 7.1-5: Summary for nicosulfuron**

| Use-No.* | Crop | Plant metabolism covered? | Sufficient residue trials? | PHI sufficiently supported? | Sample storage covered by stability data? | MRL compliance | Chronic risk for consumers identified? | Acute risk for consumers identified? |
|---|---|---|---|---|---|---|---|---|
| 1, 2 | Maize | Yes | Yes/No (18N, 4 new trials) | Yes | Yes | Yes | No | No |

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0 should be given in column 1

Neither trials have been submitted with the tank-mixed oil-based adjuvant intended. However residues levels of nicosulfuron are expected to be below the LOQ at the intended GAP and the TMDI represents only 0.1% of the ADI. It could be expected that the use of an adjuvant will not lead to an exceedance of the MRL and will not lead to an unacceptable chronic risk for the consumer.

As residues of nicosulfuron do not exceed the trigger values defined in Reg (EU) No 283/2013, there is no need to investigate the effect of industrial and/or household processing.

Residues in succeeding crops have been sufficiently investigated. It is very unlikely that residues will be present in succeeding crops.

Considering dietary burden and based on the intended uses, no significant modification of the intake was calculated for livestock. Further investigation of residues as well as the modification of MRLs in commodities of animal origin is therefore not necessary.

### 7.1.2.4 Summary for A18032E

**Table 7.1-6: Information on A18032E (KCA 6.8)**

| Crop | PHI for A18032E proposed by applicant | PHI/ Withholding period* sufficiently supported for | | | PHI for A18032E proposed by zRMS | zRMS Comments (if different PHI proposed) |
|---|---|---|---|---|---|---|
| | | Dicamba | Mesotrione | Nicosulfuron | | |
| Maize | F | Yes | Yes | Yes | F | - |

NR: not relevant
* Purpose of withholding period to be specified
** F: PHI is defined by the application stage at last treatment (time elapsing between last treatment and harvest of the crop).

**Table 7.1-7: Waiting periods before planting succeeding crops**

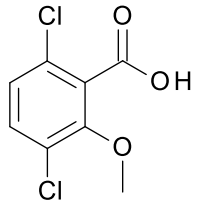
| Waiting period before planting succeeding crops | | | | Overall waiting period proposed by zRMS for A18032E |
|---|---|---|---|---|
| Crop group | Led by dicamba | Led by mesotrione | Led by nicosulfuron | |
| All crops | NR | NR | NR | NR |

NR: not relevant

**Assessment**

## 7.2 Dicamba

General data on dicamba are summarized in the table below (last updated 2016/11/18).

| | |
|---|---|
| Active substance (ISO Common Name) | Dicamba |
| IUPAC | 3,6-dichloro-2-methoxybenzoic acid |
| Chemical structure |  |
| Molecular formula | $C_8H_6Cl_2O_3$ |
| Molar mass | 221.0 g/mol |
| Chemical group | Chlorophenoxy herbicide |
| Mode of action (if available) | Auxin-like growth regulation |
| Systemic | Yes |
| Company (ies) | Syngenta |
| Rapporteur Member State (RMS) | Denmark |
| Approval status | Approved<br>01/01/2009<br>Commission Directive 2008/69/EC.<br>Expiration of approval<br>31/12/2022 |
| Restriction | Restricted to uses as a herbicide |
| Review Report | SANCO/829/08 – final rev. 2<br>12/07/2016<br>[Finalised14/03/2008] |
| Current MRL regulation | Regulation (EU) 2015/845 |
| Peer review of MRLs according to Article 12 of Reg No 396/2005 EC performed | EFSA-Q-2009-00102<br>Pesticides MRL Review (Reg.396/2005 - Art. 12) – ongoing |
| EFSA Journal : Conclusion on the peer review | Yes (EFSA, 2011) |
| EFSA Journal: conclusion on article 12 | No |
| Current MRL applications on intended uses | No MRL applications are pending |

### 7.2.1 Stability of Residues (KCA 6.1)

#### 7.2.1.1 Stability of residues during storage of samples

**Available data**

*Reference: Denmark, 2007; Denmark 2010*

The potential for degradation of residues during storage has been previously assessed in the framework of the peer review for dicamba. Storage stability of dicamba and 5-OH-dicamba was demonstrated for the following periods in the commodities listed in the table below when frozen (approximately -18°C).

*Reference: EFSA, 2011:*

"Dicamba and 5-OH-dicamba residues stable in maize (forage, fodder and grain) when stored at -17ºC for up to 36 months. In animal matrices, dicamba residues stable at -12ºC for at least 18 months, in beef

tissues and milk"
No new data submitted in the framework of this application.

**Table 7.2-1: Summary of stability data achieved at ≤ - 18°C (unless stated otherwise)**

| Commodity category | Commodity | Acceptable maximum storage period | Report Reference | Source |
|---|---|---|---|---|
| **EU reviewed data** | | | | |
| **Plant products** | | | | |
| High water content | Maize, forage | 36 months | Jimenez, 1995 Report No: 127 | Denmark, 2007 |
| | Maize, silage | 36 months | | |
| | Maize, fodder | 36 months | | |
| High starch content | Maize, grain | 36 months | | |
| **Animal Products** | | | | |
| Meat | Ruminant | 18 months [(a)] | Formanski, 1996 Report No: 151 | Denmark, 2007 |
| Fat | Ruminant | 18 months [(a)] | | |
| Liver | Ruminant | 18 months [(a)] | | |
| Kidney | Ruminant | 18 months[(a)] | | |
| Milk | Ruminant | 18 months [(a)] | | |

(a) Animal commodities were stored at -12°C

**Table 7.2-2: Summary of stability data for 5-OH-dicamba**

| Commodity category | Commodity | Acceptable maximum storage period | Report Reference | Source |
|---|---|---|---|---|
| **EU reviewed data** | | | | |
| **Plant products** | | | | |
| High water content | Maize, forage | 36 months | Jimenez, 1995 Report No: 127 | Denmark, 2007 |
| | Maize, silage | 24 months [(a)] | | |
| | Maize, fodder | 36 months | | |
| High starch content | Maize, grain | 36 months | | |

(a) The stability of 5-OH-dicamba in silage was tested for up to 24 months

## Conclusion on stability of residues during storage

The storage stability of dicamba has been investigated in different groups, including high water content commodities (maize forage, silage & fodder), high starch commodities (maize grain) and animal products. Sufficient stability has been demonstrated to support the residue data presented in the submission.
Dicamba and 5-OH-dicamba are stable for at least 36 months in plant matrices. Dicamba is stable for at least 18 months in animal products.

**zRMS comments:**

In the EFSA Journal 2011;9(1):1965 it is stated that dicamba and 5-OH-dicamba are considered to be stable in maize matrices: forage, fodder and grain (in crop commodities with high water and high starch content) for up to 36 months in, when stored frozen at c.a. -17°C. In animal matrices, dicamba residues stable at -12 °C for at least 18 months, in beef tissues and milk.
The studies on the magnitude of residues are valid with regard to storage stability.
No additional data are required.

### 7.2.1.2 Stability of residues in sample extracts (KCA 6.1)

**Available data**

In method GRM022.07A, final extracts of maize kernel, maize forage, oilseed seed, lentils and orange fortified at the LOQ, were retained in vials and stored at a temperature of approximately between 0 and 9°C. They were suitable for dicamba and 5-OH-dicamba residue analysis, for storage periods of 8 - 15 days, with mean recovery rates of 70-110% and RSDs <20%.
In method GRM022.05A, the stability of dicamba residues in final extracts stored at 4ºC (between 0 and 9ºC) was assessed in eggs. Samples were re-analysed after a 12 day interval. Results determined from this matrix at the 12 day interval were similar to those from the original analysis (the mean recovery rate was in the range 70-110%, RSD <20%).

**Conclusion on stability of residues in sample extracts**

Tests on extract stability demonstrate the stability of residues of dicamba in sample extracts of plant and animal matrices and fully support the residue data presented in the submission.

**zRMS comments:**

Information submitted by Applicant is sufficient and accepted.
No further data are required.

## 7.2.2 Nature of residues in plants, livestock and processed commodities

### 7.2.2.1 Nature of residue in primary crops (KCA 6.2.1)

**Available data**

*Reference: Denmark, 2007; Denmark 2010*

The metabolism of dicamba was investigated for foliar application on wheat and for droplet applications to leaves on sugar cane, soya bean and cotton using $C^{14}$ phenyl-U labelled-dicamba.
No new data submitted in the framework of this application.

**Table 7.2-3: Summary of plant metabolism studies**

| Crop Group | Crop | Label position | Application and sampling details | | | | Report Reference | Source |
|---|---|---|---|---|---|---|---|---|
| | | | Method, F or G[(a)] | Rate | No | Sampling (DAT) | | |
| **EU reviewed data** | | | | | | | | |
| **Pulses and oilseeds** | Soybean | phenyl-(U)-$^{14}$C | Local leaf application, F | 5.17 µg/plant (ca. 1.7 g a.s./ha[b]) | 1 (early growth stage) | Leaves: 0, 7, 14 | Butz & Atallah, 1982 Report No: 39 | Denmark, 2007 |
| | | | | 5.17 µg/plant (ca. 1.7 g a.s./ha[b]) | 1 (late growth stage) | Leaves: 6 | | |
| | Cotton | phenyl-(U)-$^{14}$C | Local leaf application, F | 60 µg/plant (ca. 5.9 g a.s./ha[b]) | 1 (green boll stage) | Whole plant: 0, 7, 14 | Butz, 1982 & 1984 Report Nos: 44 & 70 | Denmark, 2007 |
| | Cotton | phenyl-(U)-$^{14}$C | F | 2240g or 4480 g as/ha | 1 (late green boll stage) | Boll (carpel, lint, seed) Leaf, Seed, Root | Butz, 1988 Report No: 9 | Denmark, 2007 |
| **Cereals** | Spring wheat | phenyl-(U)-$^{14}$C | Foliar spray, F | 144 g a.s./ha | 1 (BBCH 29) | Forage: 18 Grain, straw: 85 | Völlmin, 1999 Report No: | Denmark, 2007 |

| Crop Group | Crop | Label position | Application and sampling details | | | | Report Reference | Source |
|---|---|---|---|---|---|---|---|---|
| | | | Method, F or G[(a)] | Rate | No | Sampling (DAT) | | |
| | | | | | | | 97SV01 | |
| | Sugar-cane | phenyl-(U)-$^{14}$C | Local leaf application, G | 3.06 mg/plant (ca. 1120 g a.s./ha[b]) | 1 (8-9 leaves) | Whole plant: 0, 1, 2, 5, 12, 21, 28 | Butz & Attallah, 1981 & 1981a Report Nos: 24 & 13 | Denmark, 2007 |

(a): Outdoor/field application (F) or glasshouse/protected/indoor application (G)
(b): Rates per ha should be considered as rough estimates since derived from local application on leaves using micro-syringe.

**Summary of plant metabolism studies reported in the EU**

*Reference: EFSA, 2011*

"In sugar cane, soya and cotton, where the characterization of the residues was investigated shortly after the application (6 to 28 days), dicamba remains the major component of the residues, accounting for 22 - 29% of the TRR in sugar cane leaves, 44 - 94% of the TRR in soya beans, and 72% of the TRR in cotton seed. Other identified metabolites were observed in low proportions (< 2% TRR), except 5-OH-dicamba, which represented 47% and 20% of the TRR in sugar cane leaves, 12 and 28 days after application, respectively. In wheat, dicamba seems to be more extensively metabolised, accounting for 10% of the TRR in immature plants (forage), and 2% and 16% of the TRR respectively in straw and grain at harvest. 5-OH-dicamba is detected as the major metabolite in wheat forage (65% TRR), but it represents less than 4% TRR in grain and straw at harvest. Both the parent compound and 5-OH-dicamba were observed in free and conjugated form.

Considering the different structures identified, the following metabolic pathway in plants was proposed. The metabolism of dicamba proceeds first by hydroxylation to form 5-OH-dicamba, or by demethylation to the DCSA metabolite, both compounds being further degraded to DCGA. Based on these studies, it was proposed to define the residue for monitoring as dicamba and its salts (free and conjugates). For risk assessment, the PRAPeR TC 50 discussed whether 5-OH-dicamba should be included additionally in the residue definition, since it was not observed at significant levels in the edible parts used for human consumption. Finally, and considering the conclusion of the PRAPeR meeting on mammalian toxicology (PRAPeR 83) stating that 5-OH-dicamba is not of higher toxicity than the parent compound, and having regard to the important levels at which this metabolite was observed in the residue trials conducted on pasture, it was agreed to include this metabolite in the residue definition for risk assessment."

**Conclusion on metabolism in primary crops**

The metabolism of dicamba in plants following foliar application is sufficiently addressed to support the proposed uses of the A18032E.

**zRMS comments:**

Information submitted by Applicant is sufficient and accepted.
Metabolism in plants was investigated in cereals (wheat, sugar cane) and in the pulse/oilseed plant group (soya, cotton), using $^{14}$C-dicamba labelled on the phenyl moiety applied by foliar spraying (wheat), or by droplet applications by means of a micro-syringe to a limited number of leaves (sugar cane, soya, cotton).
The metabolic pathway in plant is sufficiently addressed and no additional metabolism studies are necessary to support the intended uses for ADM.4651.H.1.A WG (A18032E) / Nikita.

**Definition:**

According to the EFSA Journal 2011;9(1):1965:
The residue definition for enforcement is proposed as dicamba and its salts and conjugated dicamba expressed as dicamba.
The residue definition for risk assessment is proposed as dicamba + 5-OH-dicamba, free and conjugated.
It is noted that the current enforcement residue definition established in Regulation (EC) No 396/2005 comprises only the parent compound dicamba.
No further data are required to support the proposed uses.

### 7.2.2.2 Nature of residue in rotational crops (KCA 6.6.1)

**Available data**

*Reference: Denmark, 2007*

The metabolism of dicamba in rotational crops was investigated in leafy crops (mustard and collard greens), root and tuber crops (turnips and carrots), pulses (soybean) and cereals (wheat and barley), using $C^{14}$ phenyl-U labelled-dicamba.

"Wheat, turnips and mustard were grown in soil treated with $^{14}$C-dicamba, equivalent to 0.56 kg as/ha, which corresponds to about 1.5 and 0.75 times the intended use for maize and pasture, respectively. No dicamba, DCSA or 5-OH-dicamba was found in amounts of >0.01 mg/kg 32, 131 and 365 DAT, respectively.

Barley, carrots and collard greens were planted as rotational crops to maize treated with $^{14}$C-dicamba, equivalent to 0.840 kg a.s./ha and corresponding to about twice the rate according to the intended use for maize and pasture. TRR was <0.04 mg/kg 120 DAT. TRR 30 DAT was high (1.022 mg/kg in carrot roots and 0.272 mg/kg in barley grain). Since no residues of dicamba, DCSA and 5-OH-dicamba was found in the study, where wheat, turnips and mustard were used as rotational crops it is not expected either that the residues found in barley, carrots and collard greens 30 DAT will be due to dicamba, DCSA or 5-OH-dicamba. They could be due to incorporation of $^{14}CO_2$ or other breakdown products into plant constituents such as lignin or cellulose.

There will therefore be no restriction of planting or sowing succeeding and rotational crops."

A new metabolism study has been submitted by the applicant in the framework of this application. This study is summarised in the table below. The detailed assessment of the study is presented in Appendix 2. These data are currently under evaluation for the renewal of approval of the active substance, dicamba (Commission Implementing Regulation (EU) No. 844/2012 of 18 September 2012).

In preparation for this renewal of the registration of dicamba under Commission Implementing Regulation (EU) 844/2012, an internal technical review assessment of the existing data package was conducted and it was found that the original studies were not compliant with current guidance under Commission Regulation (EU) No 283/2013. This was with respect to bare soil application and planting intervals with particular lack of information around a bare soil application at the highest application rate and the lack of 30 day interval data.

Comments made by EFSA at the last review indicated residues from confined rotational crops were adequately characterised. The applicant however believes a more detailed characterisation and quantification of the large unassigned radioactive residue component(s) was warranted. The applicant also acknowledged that insufficient radiovalidation of the residue analytical method for crops currently existed and consequently has taken the opportunity to use commodity samples generated from a new study to provide these data.

A new study has therefore been carried out to update the current residue knowledge base and provide where possible additional radiovalidation for the residue analytical method. The new study was not therefore previously reviewed in the original Annex I submission.

**Table 7.2-4: Summary of metabolism studies in rotational crops**

| Crop group | Crop | Label position | Application and sampling details | | | | Report reference | Source |
|---|---|---|---|---|---|---|---|---|
| | | | Method, F or G[(a)] | Rate (g a.s./ha) | Sowing intervals (DAT) | Harvest Intervals (DAT) | | |
| **EU reviewed data** | | | | | | | | |
| **Leafy vegetables** | Mustard | phenyl-(U)-$^{14}$C | Soil application, F | 560 | 32, 131, 369 | Maturity (tops) | Moore, 1989 Report No: 16 | Denmark, 2007 |
| | Collard greens | phenyl-(U)-$^{14}$C | Soil application, F | 840 | 30, 120 | Maturity (tops) | Pierotti, 1995 Report No: 22 | |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **Root and tuber vegetables** | Turnip | phenyl-(U)-$^{14}$C | Soil application, F | 560 | 32, 131, 369 | Maturity (tops and roots) | Moore, P.A., 1989 Report No: 16 | |
| | Carrot | phenyl-(U)-$^{14}$C | Soil application, F | 840 | 30, 120 | Maturity (roots) | Pierotti, 1995 Report No: 22 | |
| **Pulses and oilseeds** | Soybean | phenyl-(U)-$^{14}$C | Soil application, F | 840 | 120 | Immature (forage) Maturity (grain, straw, chaff) | Pierotti, 1995 Report No: 22 | |
| **Cereals** | Wheat | phenyl-(U)-$^{14}$C | Soil application, F | 560 | 32, 131, 369 | Immature, forage: 32, 396 Maturity, grain, straw, chaff: 131 | Moore, P.A., 1989 Report No: 16 | |
| | Barley | phenyl-(U)-$^{14}$C | Soil application, F | 840 | 30, 120, 365 | Immature (forage) Maturity (seed, hay) | Pierotti, 1995 Report No: 22 | |
| **New data** | | | | | | | | |
| **Leafy vegetables** | Spinach | phenyl-(U)-$^{14}$C | Soil application, F | 488 | 30, 111, 285 | Immature (tops) Maturity (tops) | Swales, 2016. Report no. 3200368 | Syngenta |
| **Root and tuber vegetables** | Radish | phenyl-(U)-$^{14}$C | Soil application, F | 488 | 30, 111, 285 | Maturity (leaves, roots) | | |
| **Cereals** | Wheat | phenyl-(U)-$^{14}$C | Soil application, F | 488 | 30, 111, 285 | Immature (forage, hay) Maturity (grain, straw) | | |

(a): Outdoor/field application (F) or glasshouse/protected/indoor application (G)

**Summary of metabolism studies in rotational crops reported in the EU**

*Reference: Denmark, 2007*

*"No residue >0.01 mg/kg of dicamba, DCSA or 5-OH-dicamba were seen 32, 131 and 369 DAT, respectively in mustards tops, turnips tops and roots, wheat forage, wheat straw, wheat grain and wheat chaff in a confined rotation crop study."*

**Summary of new metabolism study in rotational crops not yet evaluated in the EU**

The data from the new rotational crop study confirms the high total radioactive residues (TRRs) at 30 DAT (0.027-0.886 mg/kg) and progressive and marked decline in subsequent 111 DAT (0.017–0.090 mg/kg) and 285 DAT (0.001–0.016 mg/kg) rotational intervals that were observed in the earlier studies. Additionally, a more detailed characterisation and quantification of identified residue components in all rotational intervals and a better understanding of the nature of previously unassigned residues (including naturally incorporated residues) have also been achieved as summarised below.

Parent was the principal residue identified in all 30 DAT commodities (≤67% TRR; ≤0.204 mg/kg) except that of wheat straw and grain (≤1.9% TRR; 0.002 mg/kg). NOA405873 was the principal identified metabolite in wheat forage, hay and straw (≤56.3% TRR; ≤0.342 mg/kg) but was present at much lower levels in all other commodities (≤5.5% TRR; ≤0.005 mg/kg). Residue levels of the other two

identified residues NOA414746 (≤6.5% TRR; ≤0.052 mg/kg) and R740230 (≤1.9% TRR; ≤0.009% TRR) were generally much lower than that of parent and NOA405873.
By the time of 111 DAT rotational crop harvests, residues of parent and all identified metabolites had declined to ≤0.007 mg/kg (except for NOA405873 in wheat hay, 0.017 mg/kg).
By the time of 285 DAT rotational crop harvests, residues of parent and all the above identified metabolites were not detectable (except for NOA414746 in wheat hay, 0.001 mg/kg).
The decline in parent and identified metabolite residues in successive rotational intervals was accompanied by an increase in the proportion of the total radioactive residue associated with naturally incorporated radioactivity (≤ 41.7% TRR identified as $^{14}$C-glucose).
Analysis of wheat forage and hay samples using the prescribed residue analytical method (GRM022.07A) demonstrated efficient extraction of radioactive residues and efficient hydrolysis of conjugated residues when compared to that of the metabolism method used in this new confined rotational crop study.
These studies demonstrate that the metabolic pathway in rotational crops is identical to that previously elucidated in the primary metabolism studies. The key biotransformations of this metabolic pathway are summarised.

**Conclusion on metabolism in rotational crops**
A specific residue definition for rotational crops is not deemed necessary due to the very low residue levels expected.

**zRMS comments:**

Information submitted by Applicant is sufficient and accepted.
New studies for metabolism in rotational crops have been submitted by the applicant in the framework of this application. This data is currently under evaluation for the renewal of approval of the active substance, dicamba. New study demonstrates that the metabolic pathway in rotational crops is identical to that previously elucidated in the primary metabolism studies.
zRMS agrees, the metabolic pathway in rotational plant is sufficiently addressed and no additional data are necessary to support the intended uses for ADM.4651.H.1.A WG (A18032E) / Nikita.

### 7.2.2.3 Nature of residues in processed commodities (KCA 6.5.1)

**Available data**
*Reference: Denmark, 2007*
The effect of processing on the nature of dicamba was investigated in the framework of the peer review. Studies were conducted simulating representative hydrolytic conditions for pasteurisation (20 minutes at 90°C, pH 4), boiling/brewing/baking (60 minutes at 100°C, pH 5) and sterilisation (20 minutes at 120°C, pH 6). The results are summarised in the table below.
One new hydrolysis study has been submitted by the applicant in the framework of this application. This data is currently under evaluation for the renewal of approval of the active substance, dicamba (Commission Implementing Regulation (EU) No. 844/2012 of 18 September 2012).
The effect of processing on the nature of 5-OH-dicamba was investigated. A study was conducted simulating representative hydrolytic conditions for pasteurisation (20 minutes at 90°C, pH 4), boiling/brewing/baking (60 minutes at 100°C, pH 5) and sterilisation (20 minutes at 120°C, pH 6). The results are summarised in the table below and described in detail in Appendix A 2.1.2.

**Table 7.2-5: Nature of the residues in processed commodities**

| Conditions | Identified compound(s) (%) | Report reference | Source |
|---|---|---|---|
| **EU reviewed data** | | | |
| Pasteurisation (20 minutes, 90°C, pH 4) | Dicamba (100.7%) | Grout, 2003 RJ3333B | Denmark, 2007 |
| Baking, boiling, brewing (60 minutes, 100°C, pH 5) | Dicamba (105.1%) | | |
| Sterilisation (20 minutes, 120°C, pH 6) | Dicamba (106.6%) | | |

| Conditions | Identified compound(s) (%) | Report reference | Source |
|---|---|---|---|
| **New data** | | | |
| Pasteurisation (20 minutes, 90°C, pH 4) | 5-OH-Dicamba (96.4%) | Yeomans, 2015 3200933 | Syngenta |
| Baking, boiling, brewing (60 minutes, 100°C, pH 5) | 5-OH-Dicamba (97.4%) | | |
| Sterilisation (20 minutes, 120°C, pH 6) | 5-OH-Dicamba (96.5%) | | |

**Conclusion on nature of residues in processed commodities**

From the studies on dicamba and 5-OH-dicamba, it was concluded that processing by pasteurisation, baking/brewing/boiling and sterilisation is not expected to have a significant impact on the composition of residues in matrices of plant origin. No breakdown or reaction products were formed during hydrolysis of dicamba or 5-OH-dicamba under representative processing conditions. The relevant residue for enforcement and risk assessment in processed commodities is therefore expected to be the same as for primary crops.

**zRMS comments:**

Information given by the Applicant is sufficient.
Dicamba and 5-OH-dicamba are hydrolytically stable under conditions representative of pasteurisation, baking, brewing and boiling and sterilisation.
For details, please refer to the evaluation presented in Appendix 2, point A 2.1.2.1.3.
No further data are required to support the proposed use.

### 7.2.2.4 Conclusion on the nature of residues in commodities of plant origin (KCA 6.7.1)

**Table 7.2-6: Summary of the nature of residues in commodities of plant origin**

| **Endpoints** | |
|---|---|
| Plant groups covered | Pulses and oilseeds (Soybean, Cotton)<br>Cereals (Spring wheat, Sugar-cane) |
| Rotational crops covered | EFSA 2011:<br>Leafy crops (mustard, collard), root vegetables (turnip, carrot), pulses (soybean) and cereals (wheat and barley)<br><br>New data<br>Leafy crops (spinach), root vegetables (radish) and cereals (wheat) |
| Metabolism in rotational crops similar to metabolism in primary crops? | EFSA 2011:<br>No parent, 5-OH-dicamba and DCSA identified in the rotational crops. DCSA identified in soil.<br><br>[comment does not account for new data in AIR3] |
| Processed commodities | *EFSA 2011:* Not provided and not required.<br>*Denmark 2007:* Dicamba: Hydrolytically stable under representative conditions<br>*New data:* 5-OH-Dicamba: Hydrolytically stable under representative conditions |
| Residue pattern in processed commodities similar to pattern in raw commodities? | *EFSA 2011:* Not applicable<br>Denmark 2007: Yes |
| Plant residue definition for monitoring | Dicamba and its salts and conjugated dicamba expressed as dicamba (EFSA, 2011)<br>Dicamba (Regulation (EU) 2015/845) |
| Plant residue definition for risk assessment | Dicamba and 5-OH-dicamba, free and conjugated (EFSA, 2011) |
| Conversion factor from enforcement to RA | None (EFSA, 2011) |

## 7.2.2.5 Nature of residues in livestock (KCA 6.2.2-6.2.5)

### Available data

*Reference: Denmark, 2007*

The metabolism of dicamba was investigated in lactating cow, goats and laying poultry using $C^{14}$ phenyl-U labelled-dicamba. These studies are summarised in the table below.

No new data submitted in the framework of this application.

**Table 7.2-7: Summary of animal metabolism studies**

| Group | Species | Label position | No of animal | Application details | | Sample details | | Report reference | Reference |
|---|---|---|---|---|---|---|---|---|---|
| | | | | Rate (mg/kg bw/d) | Duration (days) | Commodity | Time of sampling | | |
| **EU reviewed data** | | | | | | | | | |
| **Lactating ruminants** | Cow | phenyl-(U)-$^{14}$C | 1 | 2.2 mg/kg bw/day | 5 | Milk | Twice daily | Oehler, 1980 J. Agric. Food Chem. Vol. 28, No. 4, 1980 | Denmark, 2007 |
| | | | | | | Urine | Twice daily | | |
| | | | | | | Faeces | Daily | | |
| | | | | | | Tissues | After sacrifice | | |
| | Goat | phenyl-(U)-$^{14}$C | 2 | Goat A: 0.42 mg/kg bw/day 10 mg/kg feed<br>Goat B: 43 mg/kg bw/day 1000 mg/kg feed[(a)] | 4 | Milk | Twice daily | Guirguis, 1994 Report No: 28 | |
| | | | | | | Urine & faeces | Twice daily | | |
| | | | | | | Tissues | After sacrifice | | |
| **Laying poultry** | Hen | phenyl-(U)-$^{14}$C | 12 (3 groups of 4) | Group A: 1 mg/kg bw Group B: 100 mg/kg bw Group B: 1 mg/kg bw (IV) [(b)] | 1 | Eggs | Protocol within the report infers that eggs will be collected and radioassayed (but not an objective of the study at the time). Egg residue levels were not reported | Yu & Atallah, 1983 Report No: 65 | |
| | | | | | | Blood | Taken from 2 animals per group at 0.5h, 1h, 2h, 4h, 6h and 7h. No further blood then taken but animals maintained until sacrifice at 24h when | | |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | tissues taken (see "Tissues" below). The remaining 2 animals per group maintained until sacrifice at 96h when tissues taken (see "Tissues" below). | | |
| | | | | | | Excreta | Taken from 4 animals per group at 7h and 24h (2 animals per group then sacrificed) and then from remaining 2 animals per group at 48h, 72h, and 96h (2 animals per group sacrificed). | | |
| | | | | | | Tissues | After sacrifice (24h and 96h after dosing; 2 animals per group) Taken after sacrifice at 24h (2 animals per group) and 96h (2 animals per group) | | |
| | Hen | phenyl-(U)-$^{14}$C | 8 (2 groups:) | Group A (5 hens): 0.6mg/kg bw/day Group B (3 hens): 30 mg/kg bw/day | 4 | Eggs | Daily | Nietschmann & Yu, 1994 Report No: 25 | |
| | | | | | | Excreta | Daily | | |
| | | | | | | Tissues | After sacrifice | | |

(a): Goat A was used for metabolite characterisation and Goat B was used to generate metabolites for instrumental analysis. In this study only results from Goat A are presented.
(b): Groups A and B: oral dosing (intubation); Group C: intravenous injection.

**Summary of animal metabolism studies reported in the EU**

*Reference: Denmark, 2007*

"The metabolism of [Phenyl- (U) - $^{14}$C]-dicamba has been investigated in cow, goat, hen and rat.
In summary dicamba was, after oral administration to lactating cow, lactating goat, laying hens and rats, quickly adsorbed and eliminated in excreta (mainly via urine). A very low transfer of residues into tissues, milk and eggs was observed. Therefore residue levels in tissues were low. Unchanged dicamba was the major compound observed in animal excreta, tissues and hen eggs representing between 63 – 102 % of the TRR. DCSA represented 8-21 % of the TRR in the cow (highest in liver) and 1-12 % (0.0001 –

0.0057 mg/kg) of the TRR in the goat (highest in liver). The metabolites 2,5-dichlorophenol (DCP) and glucuronide-conjugate of DCSA were detected in cow urine in amounts of 2-4 % and 2-3 % of the TRR, respectively. The metabolite 2-amino-3,6-dichloro-phenol (2A36DCP) was found in hen liver (36 % of TRR, 0.001 mg/kg).
Based on the work performed with cow, goat, hen and rat the metabolism of 14C-dicamba appears to follow the same pathways comprising the steps detailed below (see figure B.7.2.1)
• O-demethylation of dicamba to DCSA (NOA 414746)
• Conjugation of DCSA (NOA 414746) with glucuronic acid
• Decarboxylation of DCSA (NOA 414746) to 2,5-dichlorophenol (DCP)
• Decarboxylation of DCSA (NOA 414746) followed by substitution by an amino group to form 2-amino-3,6-dichlorophenol (2A36DCP)
• Hydroxylation of dicamba to 5-OH dicamba (NOA 405873)."

*Reference: EFSA, 2011*
"Metabolism in animals was considered in cow, goat and poultry, using $^{14}$C-dicamba. The transfer in fat, milk and eggs was limited, the highest TRRs being observed in kidney and liver. Dicamba (free and conjugated) was by far the major compound identified in all animal matrices, accounting for more than 50% of the TRR. In addition, DCSA was also observed in ruminants, but only in kidney and liver, up to 21% of the TRR. 5-OH-dicamba was not detected in animal matrices, except in urine and excreta, but at insignificant levels and proportions (<0.01% TRR). Having regard to the high levels of 5-OH-dicamba in grass, and consequently its significant intake by ruminants (c.a. 1.5 mg/kg bw/day), the PRAPeR TC 50 meeting of experts discussed whether a specific metabolism study using this metabolite needs to be required. The experts were of the opinion that a similar pathway to the parent is expected for 5-OH-dicamba, this metabolite being probably more extensively excreted than the parent compound since it is more polar. This assertion is supported by the results of the cow feeding study conducted with 5-OH-dicamba, where this metabolite was almost not detected in any matrices, except in kidney, at the 5N dose rate. It was therefore concluded that a specific ruminant metabolism study should not be required for 5-OH-dicamba. Finally, dicamba and its salts (free and conjugates) was proposed to define the residue for monitoring, and MRLs were derived for ruminant products from the feeding study conducted with the parent dicamba. For risk assessment, considering the lower ADI of 0.01 mg/kg bw/day proposed for the metabolite DCSA when compared to the parent compound (see section 2), the PRAPeR TC 50 meeting of experts proposed to define the residue as parent dicamba and DCSA (free and conjugates). However, after the meeting, the consumer risk assessment conducted for DCSA, taking into account the expected residue levels in kidney and liver, showed highest intakes below 0.2 % of the ADI. Having regard to the very low contribution of the DCSA metabolite to the consumer exposure and considering that the animal intake will not be increased if additional uses are envisaged, EFSA is of the opinion that the residue definition for risk assessment should be limited to the parent dicamba only.
No concern for the consumers was identified, the highest TMDI being less than 1% of the ADI for all the diets included in the EFSA PRIMo model, and the highest IESTI only 2% of the ARfD (milk).

**Conclusion on metabolism in livestock**
The metabolism of dicamba in livestock is sufficiently addressed to support the proposed uses of the A18032E.

**zRMS comments:**

Information given by the Applicant is sufficient.
In EFSA Journal 2011;9(1):1965 it is stated that metabolism studies in lactating goat, lactating cow and hen using $^{14}$C-dicamba and feeding studies on cow using dicamba and 5-OH-dicamba were assessed.
It should be noted that the animal metabolism studies have shown dicamba (free and conjugated) to be the major component of the residues accounting for more than 50% TRR in all animal matrices. However, DCSA was also identified as a significant metabolite in ruminant kidney and liver, up to 21% TRR. The experts were of the opinion that a similar pathway to the parent is expected for 5-OH-dicamba. Dicamba and its salts (free and conjugates) was proposed to define the residue for monitoring.
Nonetheless, the conclusion of the peer review was not to include DCSA in the animal residue definition since low residues of DCSA are expected to be present in ruminant liver and kidney, leading to insignificant contribution to the consumer intakes (<0.2% ADI). The residue definition for risk assessment was therefore proposed as

dicamba alone.
No additional data are required.

### 7.2.2.6 Conclusion on the nature of residues in commodities of animal origin (KCA 6.7.1)

**Table 7.2-8: Summary on the nature of residues in commodities of animal origin**

| **Endpoints** | |
|---|---|
| Animals covered | Cow, goat and hens |
| Time needed to reach a plateau concentration | Milk: *c.a.* 20 days<br><br>Updated in AIR3 MCA S6 to 3.5 days |
| Animal residue definition for monitoring | Dicamba and its salts and conjugated dicamba expressed as dicamba (EFSA, 2011)<br>Dicamba (Regulation (EU) 2015/845) |
| Animal residue definition for risk assessment | Dicamba (free and conjugated) (EFSA, 2011) |
| Conversion factor | Not applicable (EFSA, 2011) |
| Metabolism in rat and ruminant similar | Yes |
| Fat soluble residue | No |

## 7.2.3 Magnitude of residues in plants (KCA 6.3)

### 7.2.3.1 Summary of European data and new data supporting the intended uses

New studies on the magnitude of residue have been submitted by the applicant in the framework of this application. These studies are summarized in the Table below. The detailed assessment of these studies is presented in Appendix 2.
For the sake of clarity, summaries to residue trials that are relevant for this submission and that have previously been EU reviewed are also presented in Appendix 2.

**Table 7.2-9: Summary of EU reported and new data supporting the intended uses of A18032E and conformity to existing MRL**

| Commodity | Source | Residue zone (N-EU, S-EU, EU, outside EU) | Evaluation<br>GAP<br>Residue levels (mg/kg)<br>E = according to enforcement residue definition<br>RA = according to risk assessment residue definition | STMR (mg/kg) | HR (mg/kg) | Unrounded OECD calculator MRL (mg/kg) | Current EU MRL (mg/kg) * | MRL compliance |
|---|---|---|---|---|---|---|---|---|
| **Maize grain** | Denmark, 2007[a] | N-EU | GAP[b]: 1 x 0.360 kg as/ha, BBCH 16, PHI N/A, outdoor<br>E: 4x <0.01<br>R: 4x <0.02 | <0.01 | <0.01 | 0.01* | 0.5 | Yes |
| | | S-EU | GAP[b]: 1 x 0.360 kg as/ha, BBCH 16, PHI N/A, outdoor<br>E: <0.01, 0.01<br>R: <0.02, 0.02 | <0.01 | 0.01 | 0.015 | | Yes |
| | New trials | N-EU | Trials GAP: 1 x 0.188 kg as/ha, BBCH 12-19, PHI NA, outdoor<br>E: 4x <0.01<br>R: 4x <0.02 | <0.01 | <0.01 | 0.01* | | Yes |
| | | | Trials GAP: 1 x ~~288 kg as/ha~~ 0.288 kg as/ha, BBCH 12-19, PHI NA, outdoor<br>E: 4x <0.01<br>R: 4x <0.02 | <0.01 | <0.01 | 0.01* | | Yes |
| | | S-EU | Trials GAP: 1 x 0.188 kg as/ha, BBCH 12-19, PHI NA, outdoor<br>E: 6x <0.01<br>RA: 6x <0.02 | <0.01 | <0.01 | 0.01* | | Yes |
| | | | Trials GAP: 1 x ~~240-288 kg as/ha~~ 0.24-0.288 kg as/ha, BBCH 12-19, PHI NA, outdoor<br>E: 8x <0.01<br>R: 8x <0.02 | <0.01 | <0.01 | 0.01* | | Yes |
| | Overall supporting | N-EU | E: 12x <0.01<br>R: 12x <0.02 | <0.01 | <0.01 | 0.01* | | Yes |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | data | S-EU | E: 15x <0.01, 0.01<br>R: 15x <0.02, 0.02 | <0.01 | 0.01 | 0.015 | | Yes |

Source of EU MRL: Regulation. (EU) No 2015/845
N/A – not applicable
(a) Only data are considered that fully comply with current data requirements.
(b) New AIR3 critical GAP is 288 g a.s./ha (BBCH 12-19). Trials were conducted at 280-360 g a.s./ha, which is within the 25% tolerance limit of the new cGAP.
(c) Residue definition for enforcement is not relevant for animal feed items since no MRL is set. Residue definition for risk assessment: combined residues of dicamba and 5-OH-dicamba free and conjugated

### 7.2.3.2 Conclusion on the magnitude of residues in plants

Maize is a major crop in all regions of Europe (**SANCO 7525/VI/95 rev.10**); sufficient trials are available to support the proposed use.
According to the available data, the intended uses on maize are considered acceptable, for outdoor uses.
The data submitted show that no exceedance of the MRL will occur. The uses are considered acceptable.

**zRMS comments:**

Maize is the major crop in northern Europe (EU guideline Document SANCO 7525/VI/95 - rev.10.3 of 13 June 2017 and SANTE/2019/12752). A minimum of eight independent trials representative of the proposed growing area for outdoor are required.

**Table 7.2: Summary of EU critical GAP and intended GAP in maize for dicamba (DAR, 2007).**

| Type of GAP | Method | Number of applications | Application rate per treatment (kg as/ha) | Interval between application (days) | Growth stage at last application | PHI (days) |
|---|---|---|---|---|---|---|
| cGAP EU (DAR, 2007) – maize | overall spray | 1 | 0.36 | - | BBCH 16 | Since the period between treatment and harvest is more than 100 days, no PHI is applicable. |
| cGAP AIR 3 (a) | Foliar spray | 1 | 0.288 | - | BBCH 19 | N/A |
| Intended GAP-maize | Foliar, spraying, overall | 1 | 0.125 | - | BBCH 12 – 14 | - |

N/A – not applicable
(a) This data is currently under evaluation for the renewal of approval of the active substance, dicamba (Commission Implementing Regulation (EU) No. 844/2012 of 18 September 2012)

The critical EU GAP for maize is most critical than proposed GAP.
The trials conducted on maize (1 x 0.36 kg a.s./ha, post-emergence until BBCH 16, since the period between treatment and harvest is more than 100 days, no PHI is applicable, outdoor) have been evaluated at the EU level (EFSA Journal 2011;9(1):1965). No PHI is proposed in the critical EU GAP since the application is made early post-emergence and the maize grain is harvested at maturity. The samples were analysed for dicamba and 5-OH-dicamba, using an analytical method including a hydrolysis step in order to take into account the conjugates.
No residues were detected in maize grains, with all values being below the LOQs of 0.01 and 0.05 mg/kg mg/kg for both compounds.

EFSA Journal 2011;9(1):1965:

**Summary of residues data according to the representative uses on raw agricultural commodities and feedingstuffs (Annex IIA, point 6.3, Annex IIIA, point 8.2)**

| Crop | Northern or Southern Region, field or glasshouse | Trials results relevant to the representative uses (a) | Recommendation/comments | MRL estimated from trials according to representative use | HR (c) | STMR (b) |
|---|---|---|---|---|---|---|
| Maize | Northern | Dicamba: 6x <0.01, 3x <0.05<br>5-OH-dicamba: 6x <0.01, 3x <0.05 | Note: dicamba and 5-OH-dicamba residue levels are given in their respective order. | 0.01* | 0.05 | 0.01 |
| | Southern | Dicamba: 4x <0.01<br>5-OH-dicamba: 4x <0.01 | | | | |
| Pasture | Northern | Dicamba: 1.84, 2.63, 2.87, 3.40, 5.12, 8.39, 8.41, 9.48, 13.80<br>5-OH-dicamba: 1.51, 0.15, 3.36, 1.40, 6.97, 1.28, 2.35, 5.45, 7.90 | Majority of trials from northern EU conducted with a single application (underlined values), but considered acceptable since similar levels as for 2 applications. Moreover, when 2 treatments, low levels were observed prior to the second application. | n/a | 13.80 | 5.12 |
| | Southern | Dicamba: 0.78, 1.50, 2.00, 2.51, 2.62, 4.67, 6.14, 8.70, 9.05<br>5-OH-dicamba: 0.08, 1.70; 0.86, 4.24, 0.03, 0.08, 0.06, 0.05, 1.01 | | | 9.05 | 2.62 |

(a) Numbers of trials in which particular residue levels were reported *e.g.* 3 x <0.01, 1 x 0.01, 6 x 0.02, 1 x 0.04, 1 x 0.08, 2 x 0.1, 2 x 0.15, 1 x 0.17
(b) Supervised Trials Median Residue *i.e.* the median residue level estimated on the basis of supervised trials relating to the critical GAP
(c) Highest residue

These residue data are supported by the storage stability study showing dicamba and 5-OH-dicamba residues to be

stable for up to 36 months in maize matrices (forage, fodder and grain), when stored frozen at c.a. -17°C.

The Applicant provided four reports covered a total of 8 field trials in Northern Europe conducted on maize.
1. Andrews, G., 2016, Syngenta Report No. TK0258007-REG
Four decline residue field trials on maize were conducted in Northern France, Germany and Poland during 2015.
One application, at BBCH 16-18, of A19658H was made at 288 g ai/ha for dicamba. Maize samples: whole plant, remaining plant and whole cobs were collected.
Results:
Residues of dicamba and 5-OH-dicamba in whole cob samples taken at 75-79 BBCH and 89 BBCH were below the limit of quantification (<0.01 mg/kg).

Residues of dicamba in remaining plant samples taken at 75-79 BBCH were between <0.01 and 0.04 mg/kg and at 89 BBCH were between 0.01 and 0.04 mg/kg.
Residues of NOA405873 in remaining plant samples taken at 75-79 BBCH were between 0.03 and 1.4 mg/kg and at 89 BBCH were between <0.01 and 0.96 mg/kg.

Residues of dicamba in whole plant samples taken at 39 BBCH were between <0.01 and 0.09 mg/kg, at 63 BBCH were between <0.01 and 0.03 mg/kg and at 83-85 BBCH were between <0.01 and 0.04 mg/kg.
Residues of NOA405873 in whole plant samples taken at 39 BBCH were between 0.11 and 1.6 mg/kg, at 63 BBCH were between 0.05 and 1.1 mg/kg and at 83-85 BBCH were between <0.01 and 0.97 mg/kg.

Residues of dicamba in untreated samples were below the limit of quantification (<0.01 mg/kg). This is with the exception of remaining plant at BBCH 89 where residues range from <0.01 to 0.04 mg/kg.
Residues of NOA405873 in untreated samples were below the limit of quantification (<0.01 mg/kg). This is with the exception of remaining plant at BBCH 75-79 and BBCH 89 where residues range from <0.01 to 0.04 mg/kg and <0.01 to 0.01 mg/kg, respectively.

2. Mound, L., Gardinal, P., Kwiatkowski, A., 2006, Syngenta Report No. 04-7016
One residue field trial on maize was conducted in Northern Eurpoe, Switzerland during 2004.
To treated plot P1 and P2, one application, at BBCH 17-18, of A14031B was made at 187.5 g dicamba/ha and at 18.75 g prosulfuron/ha. Additionally adjuvants Trend 90 at 0.2 L/ha (plot 1) and Etolag 149 at 1.0 L/ha (plot 2) were added. Maize samples (whole plant, remaining plant and whole cobs) were collected
Results:
No residues of dicamba (<LOQ) were found in any maize commodity, either controls or samples.
No residues of 5-hydroxy dicamba (<LOQ) were found in grain, but low residues were found in whole plant and remainder samples from plot 2 (Etolag 149), at 0.03-0.06 mg/kg and 0.01 mg/kg, respectively. No residues of 5-hydroxy dicamba were found in any controls samples.

3. Simon, P., 2006, Syngenta Report No.gmz043004
One residue field trial on maize was conducted in Saxonia.
The test product was applied once as a foliar treatment (at BBCH 17-18), on one plot in tank mixture with Etolag 149 and in another plot with Trend 90. The total nominal amount of A14031B was 0.375 kg product/ha (corresponding to 18.75 g prosulfuron/ha and 187.5 g dicamba/ha).
No residues of dicamba at or below 0.01 mg/kg were detected in any of the treated and untreated (control) specimens.
Residues of 5-hydroxy dicamba in whole plant specimens at 37 days after treatment were at 0.09 mg/kg (tankmix with Trend 90) or 0.03 mg/kg (tankmix with Etolag 149) and were reduced to 0.03 mg/kg or 0.01 mg/kg at grain harvest stage. No residues of 5-OH-dicamba were detected in or on the other treated maize (cobs or kernel) samples.
No residues of 5-hydroxy dicamba were found in any controls samples.

4. Oppilliart, S., 2009, Syngenta Report No. T001033-07-REG
Two decline residue field trial on maize was conducted in Northern Europe, in France, during 2007.
Dicamba and prosulfuron were applied to corn by one broadcast foliar application of a 55 WG formulation (A14031E) at commercial rates at BBCH 18. On adjuvant Trend 90 was applied at a rate of 0.2 L/ha.
Corn whole plant, immature whole cob, whole cob and remaining plant were collected.
Residues of dicamba in treated corn whole plant samples ranged from below the limit of quantification (0.01 mg/kg) to 0.05 mg/kg at BBCH 39 and from below the limit of quantification to 0.01 mg/kg at BBCH 83-85. Except for one treated remaining cob sample (0.02 mg/kg at BBCH 89), no residues of dicamba were found in kernel, remaining cob and remaining plant treated corn samples at or above the limit of quantification (0.01 mg/kg).
Residues of 5-hydroxy dicamba in treated corn whole plant samples ranged from 0.01 to 0.11 mg/kg at BBCH 39 and declined below the limit of quantification (0.01 mg/kg) at BBCH 83-85. No residues of 5-hydroxy dicamba

were found in treated kernel samples at BBCH 89. Residues of 5-hydroxy dicamba in remaining cob and remaining plant treated corn samples ranged from below the limit of quantification (0.01 mg/kg) to 0.01mg/kg.
No residues of dicamba or 5-hydroxy dicamba were detected at or above the limit of quantification (0.01 mg/kg) in any of the untreated corn samples.

Summary:
E: 8x <0.01 mg/kg,
R: 8x <0.02 mg/kg.
Residues of dicamba and 5-OH-dicamba in maize grain were below the limit of quantification (<0.01 mg/kg).
Available results show that the in force MRL on maize/corn of 0.5 mg/kg (Reg. (EU) 2015/845) will not be exceeded. The current EU MRL for dicamba are sufficient to support the proposed use.

Applicant proposes to add tank-mixed adjuvant (Adigor, STYK, Olejan or Efica 960 EC) to product ADM.4651.H.1.A (Nikita). Some trials have been submitted with the tank-mixed adjuvant (Etolag 149 and Trend 90). Residues levels of dicamba in these studies were below the LOQ, so the addition of an adjuvant should not affect the residue level in this appliacation. It could be expected that the use of an adjuvant will not lead to an exceedance of the MRL and will not lead to an unacceptable chronic risk for the consumer. Hence, at the intended GAP, the use of an adjuvant is considered to be supported for dicamba.
No further data are required to support the proposed uses.

## 7.2.4 Magnitude of residues in livestock

### 7.2.4.1 Dietary burden calculation

The use of A18032E on maize may result in residues of dicamba in animal feed items, therefore the possible transfer of residues in animal commodities from the proposed uses should be considered.
This submission is intended for product renewal, and all uses that are relevant to the zone have been considered.
As clarified in Regulation (EU) No. 1136/2014 (Regulation (EU) No 283/2013 as regards the transitional measures applying to procedures concerning plant protection products) and the associated guidance document SANCO/11509/2013 – rev. 5, May 2015) the old (AS) data requirements are applicable to ASs whose approval submission was made prior to 1 January 2014 until they are re-approved, and the animal intake triggering the submission of animal studies remains 0.1 mg/kg DM for the active substances falling under Reg. (EU) No 544/2011. Dicamba meets these criteria and as such the EFSA calculation method that described in "Lundehn Appendix G" (EC Document 7031/VI/95 rev.4, July 1996) is presented below.
In order to satisfy the requirements of certain member states, calculation of the animal dietary intake is also presented using the OECD calculation method described in the OECD Guidance Document on Residues in Livestock (ENV/JM/MONO(2013)8).

**Table 7.2-10: Input values for the dietary burden calculation (considering the uses authorised within the zone and the uses under consideration) (EFSA PROFile calculator)**

| Feed Commodity | Median dietary burden | | Maximum dietary burden | |
|---|---|---|---|---|
| | Input value (mg/kg) | Comment | Input value (mg/kg) | Comment |
| **Risk assessment residue definition:** Combined residues of dicamba and 5-OH-dicamba free and conjugated | | | | |
| Barley, Straw (Cereal straws group) | 0.25 | Residue data in CA 6.3.6 | 1.78 | Residue data in CA 6.3.6 |
| Corn, Field, Forage/Silage (Green forages group) | 0.31 | Residue data in CA 6.3.2 | 0.62 | Residue data in CA 6.3.2 |
| Grass, fresh (Green forages group) | 6.48 | Residue data in IIA 6.3.9[(a)] | 25.6 | Residue data in IIA 6.3.9[(a)] |
| Grass, hay (Green forages group) | 22.8 | Residue data in IIA 6.3.9[(a)] OECD increase in %DM | 90.3 | Residue data in IIA 6.3.9[(a)] OECD increase in %DM |

| Feed Commodity | Median dietary burden | | Maximum dietary burden | |
|---|---|---|---|---|
| | Input value (mg/kg) | Comment | Input value (mg/kg) | Comment |
| | | 88% DM in hay / 25% DM in fresh forage<br>[Median value x %DM increase 6.48 x 88/25 = 22.8] | | 88% DM in hay / 25% DM in fresh forage<br>[Max value x %DM increase 25.6 x 88/25 = 90.1] |
| Grass, silage (Green forages group) | 10.4 | Residue data in IIA 6.3.9[(a)]<br>OECD increase in %DM<br>40% DM in silage / 25% DM in fresh forage<br>[Median value x %DM increase 6.48 x 40/25 = 10.4] | 41.0 | Residue data in IIA 6.3.9[(a)]<br>OECD increase in %DM<br>40% DM in silage / 25% DM in fresh forage<br>[Max value x %DM increase 25.6 x 40/25 = 41.0] |
| Oat, Straw (Cereals straw group) | 0.25 | Residue data in CA 6.3.6 | 1.78 | Residue data in CA 6.3.6 |
| Rye, Straw (Cereals straw group) | 0.25 | Residue data in CA 6.3.6 | 1.78 | Residue data in CA 6.3.6 |
| Wheat, Straw (Cereals straw group) | 0.25 | Residue data in CA 6.3.6 | 1.78 | Residue data in CA 6.3.6 |
| Barley, Grain (Cereal grains group) | 0.05 | Residue data in CA 6.3.6 | N/A[(b)] | |
| Corn, Field, Grain (Cereal grains group) | 0.02 | Residue data in CA 6.3.2 | N/A[(b)] | |
| Oat, Grain (Cereal grains group) | 0.05 | Residue data in CA 6.3.6 | N/A[(b)] | |
| Rye, Grain (Cereal grains group) | 0.05 | Residue data in CA 6.3.6 | N/A[(b)] | |
| Wheat, Grain (Cereal grains group) | 0.05 | Residue data in CA 6.3.6 | N/A[(b)] | |
| Wheat, bran (Cereal grains group) | 0.41 | Residue data in CA 6.3.6<br>Mean processing factor of 8.0 in CA 6.5.3 used in calculation<br>[Median value x PF malt 0.051 x 8.0 = 0.41 | N/A[(b)] | |
| Rye, bran (Cereal grains group) | 0.41 | Residue data in CA 6.3.6<br>Mean processing factor of 8.0 in CA 6.5.3 used in calculation<br>[Median value x PF malt 0.051 x 8.0 = 0.41 | N/A[(b)] | |

(a) MII Section 4 Core MRL dossier, 25 August 2011.
(b) Only median relevant for grain and processed fractions

**Table 7.2-11: Input values for the dietary burden calculation (considering the uses authorised within the zone and the uses under consideration) (OECD calculator)**

| Feed Commodity | Median dietary burden | | Maximum dietary burden | |
|---|---|---|---|---|
| | Input value (mg/kg) | Comment | Input value (mg/kg) | Comment |
| **Risk assessment residue definition:** Combined residues of dicamba and 5-OH-dicamba free and conjugated | | | | |
| Barley, Straw (Forages group) | 0.25 | Residue data in CA 6.3.6 | 1.78 | Residue data in CA 6.3.6 |
| Corn, Field, Forage/Silage | 0.31 | Residue data in CA 6.3.2 | 0.62 | Residue data in CA 6.3.2 |

| Feed Commodity | Median dietary burden | | Maximum dietary burden | |
|---|---|---|---|---|
| | Input value (mg/kg) | Comment | Input value (mg/kg) | Comment |
| (Forages group) | | | | |
| Corn, Field, Stover (Forages group) | 0.03 | Residue data in CA 6.3.2 | 0.53 | Residue data in CA 6.3.2 |
| Grass, Forage (fresh) (Forages group) | 6.48 | Residue data in IIA 6.3.9[(a)] | 25.6 | Residue data in IIA 6.3.9[(a)] |
| Grass, Hay (Forages group) | 22.8 | Residue data in IIA 6.3.9[(a)] OECD increase in %DM 88% DM in hay / 25% DM in fresh forage [Median value x %DM increase 6.48 x 88/25 = 22.8] | 90.3 | Residue data in IIA 6.3.9[(a)] OECD increase in %DM 88% DM in hay / 25% DM in fresh forage [Max value x %DM increase 25.6 x 88/25 = 90.1] |
| Grass, Silage (Forages group) | 10.4 | Residue data in IIA 6.3.9[(a)] OECD increase in %DM 40% DM in silage / 25% DM in fresh forage [Median value x %DM increase 6.48 x 40/25 = 10.4] | 41.0 | Residue data in IIA 6.3.9[(a)] OECD increase in %DM 40% DM in silage / 25% DM in fresh forage [Max value x %DM increase 25.6 x 40/25 = 41.0] |
| Oat, Straw (Forages group) | 0.25 | Residue data in CA 6.3.6 | 1.78 | Residue data in CA 6.3.6 |
| Rye, Straw (Forages group) | 0.25 | Residue data in CA 6.3.6 | 1.78 | Residue data in CA 6.3.6 |
| Sorghum, Forage (Forages group) | 0.32 | Residue data in CA 6.3.7 | 0.60 | Residue data in CA 6.3.7 |
| Sorghum, Stover (Forages group) | 0.355 | Residue data in CA 6.3.7 | 0.80 | Residue data in CA 6.3.7 |
| Sorghum, Silage (Forages group) | 0.32 | Residue data in CA 6.3.7 Forage data used as surrogate | 0.60 | Residue data in CA 6.3.7 Forage data used as surrogate |
| Triticale, Straw (Forages group) | 0.25 | Residue data in CA 6.3.6 | 1.78 | Residue data in CA 6.3.6 |
| Wheat, Straw (Forages group) | 0.25 | Residue data in CA 6.3.6 | 1.78 | Residue data in CA 6.3.6 |
| Barley, Grain (Grain/Seeds group) | 0.05 | Residue data in CA 6.3.6 | N/A[(b)] | |
| Corn, Field, Grain (Grain/Seeds group) | 0.02 | Residue data in CA 6.3.2 | N/A[(b)] | |
| Oat, Grain (Grain/Seeds group) | 0.05 | Residue data in CA 6.3.6 | N/A[(b)] | |
| Rye, Grain (Grain/Seeds group) | 0.05 | Residue data in CA 6.3.6 | N/A[(b)] | |
| Sorghum, Grain (Grain/Seeds group) | 0.05 | Residue data in CA 6.3.7 | N/A[(b)] | |
| Triticale, Grain (Grain/Seeds group) | 0.05 | Residue data in CA 6.3.6 | N/A[(b)] | |
| Wheat, Grain (Grain/Seeds group) | 0.05 | Residue data in CA 6.3.6 | N/A[(b)] | |
| Brewer's grain (wheat or barley), Dried (By-products group) | 0.05 | Residue data in CA 6.3.6 Mean processing factor of 0.9 for barley malt in CA | N/A[(b)] | |

| Feed Commodity | Median dietary burden | | Maximum dietary burden | |
|---|---|---|---|---|
| | Input value (mg/kg) | Comment | Input value (mg/kg) | Comment |
| | | 6.5.3 used in calculation [Median value x PF malt 0.051 x 0.9 = 0.05] | | |
| Corn, Field, Milled Byprods. (By-products group) | 0.16 | Residue data in CA 6.3.2 OECD theoretical maximum processing of 8.0 used [Median value x PF 0.02 x 8 = 0.16] | N/A(b) | |
| Corn, Field, Hominy Meal (By-products group) | 0.05 | Residue data in CA 6.3.2 OECD theoretical maximum processing of 2.5 used [Median value x PF 0.02 x 2.5 = 0.05] | N/A(b) | |
| Corn, Field, Gluten Feed (By-products group) | 0.05 | Residue data in CA 6.3.2 OECD theoretical maximum processing of 2.5 used [Median value x PF 0.02 x 2.5 = 0.05] | N/A(b) | |
| Corn, Field, Gluten Meal (By-products group) | 0.15 | Residue data in CA 6.3.2 OECD theoretical maximum processing of 2.5 used [Median value x PF 0.02 x 7.5 = 0.15] | N/A(b) | |
| Distiller's grain (wheat or barley), Dried (By-products group) | 0.05 | Residue data in CA 6.3.6 Mean processing factor of 0.9 for barley malt in CA 6.5.3 used in calculation [Median value x PF malt 0.051 x 0.9 = 0.05] | N/A(b) | |
| Wheat, Gluten Meal (By-products group) | 0.41 | Residue data in CA 6.3.6 Mean processing factor of 8.0 in CA 6.5.3 used in calculation [Median value x PF malt 0.051 x 8.0 = 0.41 | N/A(b) | |
| Wheat, Milled Byprods. (By-products group) | 0.41 | Residue data in CA 6.3.6 Mean processing factor of 8.0 in CA 6.5.3 used in calculation [Median value x PF malt 0.051 x 8.0 = 0.41 | N/A(b) | |

(c) MII Section 4 Core MRL dossier, 25 August 2011.
(b) Only median relevant for grain and processed fractions

Dicamba currently falls under old data requirements, therefore the only categories considered are dairy and beef cattle, laying poultry and pig. The results of the dietary burden calculations are reported in the following tables. The calculated dietary burdens for all livestock were found to exceed the trigger value of 0.1 mg/kg DM in feed of dairy and meat ruminants and pigs, using both, the EFSA PROFile calculator and the OECD dietary burden calculator. The trigger is not exceeded for poultry when using the EFSA PROFile calculator, but is exceeded according to the OECD dietary burden calculation. Further investigation of residues is therefore required.

**Table 7.2-12: Results of the dietary burden calculation (EFSA PROFile calculator)**

| Animal species | Median dietary burden (mg/kg bw/d) | Maximum dietary burden (mg/kg bw/d) | Highest contributing commodity | Max dietary burden (mg/kg DM) | Trigger exceeded (Y/N) |
|---|---|---|---|---|---|
| Risk assessment residue definition: Combined residues of dicamba and 5-OH-dicamba free and conjugated | | | | | |
| Dairy ruminants* | 1.891 | 7.455 | Grass silage | 205 | Y |
| Meat ruminants* | 2.229 | 8.786 | Grass silage | 205 | Y |
| Finishing swine* | 0.316 | 1.23 | Grass silage | 30.75 | Y |
| Laying hens* | 0.0044 | 0.0044 | Wheat bran | 0.069 | N |

* These categories correspond to those (formerly) assessed at EU level.

**Table 7.2-13: Results of the dietary burden calculation (OECD calculator)**

| Animal species | Median dietary burden (mg/kg bw/d) | Maximum dietary burden (mg/kg bw/d) | Highest contributing commodity | Max dietary burden (mg/kg DM) | Trigger exceeded (Y/N) |
|---|---|---|---|---|---|
| Risk assessment residue definition: Combined residues of dicamba and 5-OH-dicamba free and conjugated | | | | | |
| Dairy ruminants* | 0.606 | 2.375 | 1. Grass, Forage (fresh) (Forages group) | 61.75 | Y |
| Meat ruminants* | 0.315 | 1.235 | 1. Grass, Hay (Forages group) | 51.45 | Y |
| Finishing swine* | 0.008 | 0.008 | 1. Wheat, Milled By-products (By-products group) | 0.26 | Y |
| Laying hens* | 0.016 | 0.024 | 1. Wheat, Straw (Forages group) | 0.35 | Y |

* These categories correspond to those (formerly) assessed at EU level.

**zRMS comments:**

Information presented by Applicant are sufficient.
The median and maximum dietary burdens were calculated for different groups of livestock using the EFSA PROFile calculator and EFSA Animal model 2017.
The results of the dietary burden calculation (OECD calculator, 2017) are presented in the Table 7.2-13 and below:

# Animal burden calculation

According to: "OECD Guidance Document, Series on testing and assessment No 64 and Series on pesticides No 32" and "OECD Guidance Document on Residues in livestock, Series on Pesticides No 73"

| Maximum Intake | Cattle | | | | | | Sheep | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Beef | 500 kg / 12 kg | | Dairy | 650 kg / 25 kg | | Ram/Ewe | 75 kg / 2,5 kg | | Lamb | 40 kg / 1,7 kg | |
| (mg/kg bw/d) | 1,233 | mg/kg bw/d | % | 2,371 | mg/kg bw/d | % | 3,244 | mg/kg bw/d | % | 2,189 | mg/kg bw/d | % |
| Contributor 1 | Grass | forage (fresh | 50 | Grass | silage | 60 | Grass | forage (fresh | 95 | Grass | forage (fresh | 50 |
| Contributor 2 | Wheat gluten | meal | 15 | Wheat gluten | meal | 20 | Wheat gluten | meal | 5 | Wheat gluten | meal | 30 |
| Contributor 3 | Sorghum | grain | 35 | Sorghum | grain | 20 | | | 0 | Sorghum | grain | 20 |
| Contributor 4 | | | | | | | | | | | | |
| Median intake | 0,3151 | mg/kg bw/d | | 0,6063 | mg/kg bw/d | | 0,8225 | mg/kg bw/d | | 0,5640 | mg/kg bw/d | |

| Maximum Intake | Swine | | | | | |
|---|---|---|---|---|---|---|
| | Breeding | 260 kg / 6 kg | | Finishing | 100 kg / 3 kg | |
| (mg/kg bw/d) | 0,478 | mg/kg bw/d | % | 0,008 | mg/kg bw/d | % |
| Contributor 1 | Grass | silage | 20 | Wheat | milled bypdts | 50 |
| Contributor 2 | Wheat | milled bypdts | 50 | Sorghum | grain | 50 |
| Contributor 3 | Sorghum | grain | 30 | | | |
| Contributor 4 | | | | | | |
| Median intake | 0,125 | mg/kg bw/d | | 0,008 | mg/kg bw/d | |

Intakes >0.004 mg/kg bw/d are highlighted

| Maximum Intake | Poultry | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Broiler | 1,7 kg / 0,12 kg | | Layer | 1,9 kg / 0,13 kg | | Turkey | 7 kg / 0,5 kg | |
| (mg/kg bw/d) | 0,010 | mg/kg bw/d | % | 0,024 | mg/kg bw/d | % | 0,010 | mg/kg bw/d | % |
| Contributor 1 | Corn, field | milled bypdts | 60 | Wheat | straw | 10 | Wheat gluten | meal | 10 |
| Contributor 2 | Sorghum | grain | 40 | Wheat gluten | meal | 10 | Rye | grain | 60 |
| Contributor 3 | | | | Barley | grain | 80 | | | |
| Contributor 4 | | | | | | | | | |
| Median intake | 0,010 | mg/kg bw | | 0,016 | mg/kg bw | | 0,010 | mg/kg bw | |

**Intakes expressed on the dry mater basis** (mg/kg DM)

| mg/kg DM | Cattle | | Sheep | | Swine | |
|---|---|---|---|---|---|---|
| | Beef | Dairy | Ram/Ewe | Lamb | Breeding | Finishing |
| Maximum | 51,37 | 61,65 | 97,3 | 51,51 | 20,72 | 0,26 |
| Median | 13,13 | 15,76 | 24,67 | 13,27 | 5,43 | 0,26 |

| | Poultry | | |
|---|---|---|---|
| | Broiler | Layer | Turkey |
| Maximum | 0,14 | 0,35 | 0,13 |
| Median | 0,14 | 0,24 | 0,13 |

**Intake >0.1 mg/kg DM in red characters**

New data requirements — Regulation (EU) No 283/2013)

Column to be deleted if not relevant

| Relevant groups | Dietary burden expressed in | | | | Most critical diet (a) | Most critical commodity (b) | | Trigger exceeded (Yes/No) | Previous assessment |
|---|---|---|---|---|---|---|---|---|---|
| | mg/kg bw per day | | mg/kg DM | | | | | 0.004 | Max burden |
| | Median | Maximum | Median | Maximum | | | | mg/kg bw | mg/kg bw |
| Cattle (all diets) | 0,606 | 2,371 | 15,76 | 61,65 | Dairy cattle | Grass | silage | Yes | |
| Cattle (dairy only) | 0,606 | 2,371 | 15,76 | 61,65 | Dairy cattle | Grass | silage | Yes | |
| Sheep (all diets) | 0,822 | 3,244 | 24,67 | 97,33 | Ram/Ewe | Grass | forage (fresh) | Yes | |
| Sheep (ewe only) | 0,822 | 3,244 | 24,67 | 97,33 | Ram/Ewe | Grass | forage (fresh) | Yes | |
| Swine (all diets) | 0,125 | 0,478 | 5,43 | 20,72 | Swine (breeding) | Grass | silage | Yes | |
| Poultry (all diets) | 0,016 | 0,024 | 0,24 | 0,35 | Poultry layer | Wheat | straw | Yes | |
| Poultry (layer only) | 0,016 | 0,024 | 0,24 | 0,35 | Poultry layer | Wheat | straw | Yes | |

(a): When several diets are relevant (e.g. cattle, sheep and poultry "all diets"), the most critical diet is identified from the maximum dietary burdens expressed as "mg/kg bw per day"
(b): The most critical commodity is the major contributor identified from the maximum dietary burden expressed as "mg/kg bw per day".

The calculated dietary burdens for dicamba for all groups of livestock were found to exceed the trigger value of 0.1 mg/kg DM (or 0.004 mg/kg bw/d, respectively). Further investigation of residues is therefore required in all commodities of animal origin.

### 7.2.4.2 Livestock feeding studies (KCA 6.4.1-6.4.3)

**Available data**
No new data are submitted in the framework of this application.

Reference*: Denmark, 2007*
Livestock feeding studies for dicamba have been conducted in laying hens and lactating goats, and a further feeding study for 5-OH-dicamba has been conducted in lactating goats. No pig feeding study was conducted the dietary exposure of pigs is lower than that calculated for ruminants (lambs). Data from ruminants can be used to address the potential for residues in pigs. The studies were EU reviewed and are considered to be acceptable; the results are summarised in the tables below.

**Table 7.2-14: Overview of the values derived from livestock feeding studies (EFSA PROFile calculator)**

| Commodity | Dietary burden | | Results of the livestock feeding study | | | | | | Median residue (mg/kg)[b] | Highest residue (mg/kg)[c] | Calculated MRL (mg/kg) | CF for RA[d] |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Med. (mg/kg bw/d) | Max. (mg/kg bw/d) | Dose Level (mg/kg bw/d)(a) | No | Result for enforcement | | Result for RA | | | | | |
| | | | | | Mean (mg/kg) | Max. (mg/kg) | Mean (mg/kg) | Max. (mg/kg) | | | | |
| **EU reviewed data (Denmark, 2007 – Ruminant: Report No. 379 and 324/00; Poultry: Report No. 107-203 and 74)** | | | | | | | | | | | | |
| **Dicamba and its salts and conjugated dicamba expressed as dicamba** | | | | | | | | | | | | |
| Pig meat | Finishing: 0.316 | Finishing: 1.23 | 0.93 | 3 | <0.01[e] | <0.01[e] | <0.01[e] | <0.01[e] | 0.01[f] | 0.011 | 0.015 | -- |
| | | | 2.78 | 3 | 0.012[e] | 0.014[e] | 0.012[e] | 0.014[e] | | | | |
| | | | 9.3 | 3 | 0.030[e] | 0.037[e] | 0.030[e] | 0.037[e] | | | | |
| Pig fat | Finishing: 0.316 | Finishing: 1.23 | 0.93 | 3 | 0.023[e] | 0.046[e] | 0.023[e] | 0.046[e] | 0.008[f] | 0.061 | 0.06 | -- |
| | | | 2.78 | 3 | 0.025[e] | 0.034[e] | 0.025[e] | 0.034[e] | | | | |
| | | | 9.3 | 3 | 0.047[e] | 0.059[e] | 0.047[e] | 0.059[e] | | | | |
| Pig liver | Finishing: 0.316 | Finishing: 1.23 | 0.93 | 3 | 0.026[e] | 0.029[e] | 0.026[e] | 0.029[e] | 0.009[f] | 0.038 | 0.040 | -- |
| | | | 2.78 | 3 | 0.066[e] | 0.070[e] | 0.066[e] | 0.070[e] | | | | |
| | | | 9.3 | 3 | 0.207[e] | 0.207[e] | 0.207[e] | 0.207[e] | | | | |
| Pig kidney | Finishing: 0.316 | Finishing: 1.23 | 0.93 | 3 | 0.154[e] | 0.174[e] | 0.154[e] | 0.174[e] | 0.052[f] | 0.230 | 0.300 | -- |
| | | | 2.78 | 3 | 0.282[e] | 0.288[e] | 0.282[e] | 0.288[e] | | | | |
| | | | 9.3 | 3 | 0.646[e] | 0.885[e] | 0.646[e] | 0.885[e] | | | | |
| Ruminant meat | Dairy: 2.229 | Dairy: 8.786 | 0.93 | 3 | <0.01[e] | <0.01[e] | <0.01[e] | <0.01[e] | 0.012 | 0.032 | 0.040 | -- |
| | | | 2.78 | 3 | 0.012[e] | 0.014[e] | 0.012[e] | 0.014[e] | | | | |
| | | | 9.3 | 3 | 0.030[e] | 0.037[e] | 0.030[e] | 0.037[e] | | | | |
| Ruminant fat | Dairy: 2.229 | Dairy: 8.786 | 0.93 | 3 | 0.023[e] | 0.046[e] | 0.023[e] | 0.046[e] | 0.025 | 0.057 | 0.060 | -- |
| | | | 2.78 | 3 | 0.025[e] | 0.034[e] | 0.025[e] | 0.034[e] | | | | |
| | | | 9.3 | 3 | 0.047[e] | 0.059[e] | 0.047[e] | 0.059[e] | | | | |
| Ruminant liver | Dairy: 2.229 | Dairy: 8.786 | 0.93 | 3 | 0.026[e] | 0.029[e] | 0.026[e] | 0.029[e] | 0.054 | 0.196 | 0.200 | -- |
| | | | 2.78 | 3 | 0.066[e] | 0.070[e] | 0.066[e] | 0.070[e] | | | | |

| Commodity | Dietary burden | | Results of the livestock feeding study | | | | | | Median residue (mg/kg)[b] | Highest residue (mg/kg)[c] | Calculated MRL (mg/kg) | CF for RA[d] |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Med. (mg/kg bw/d) | Max. (mg/kg bw/d) | Dose Level (mg/kg bw/d)(a) | No | Result for enforcement | | Result for RA | | | | | |
| | | | | | Mean (mg/kg) | Max. (mg/kg) | Mean (mg/kg) | Max. (mg/kg) | | | | |
| **EU reviewed data (Denmark, 2007 – Ruminant: Report No. 379 and 324/00; Poultry: Report No. 107-203 and 74)** | | | | | | | | | | | | |
| **Dicamba and its salts and conjugated dicamba expressed as dicamba** | | | | | | | | | | | | |
| | | | 9.3 | 3 | 0.207[e] | 0.207[e] | 0.207[e] | 0.207[e] | | | | |
| Ruminant kidney | Dairy: 2.229 | Dairy: 8.786 | 0.93 | 3 | 0.154[e] | 0.174[e] | 0.154[e] | 0.174[e] | 0.244 | 0.838 | 0.900 | -- |
| | | | 2.78 | 3 | 0.282[e] | 0.288[e] | 0.282[e] | 0.288[e] | | | | |
| | | | 9.3 | 3 | 0.646[e] | 0.885[e] | 0.646[e] | 0.885[e] | | | | |
| Milk | Dairy: 1.891 | Dairy: 7.455 | 0.93 | 3 | 0.02[e] | 0.029[e] | 0.02[e] | 0.029[e] | 0.030 | 0.142 | 0.150 | -- |
| | | | 2.78 | 3 | 0.035[e] | 0.055[e] | 0.035[e] | 0.055[e] | | | | |
| | | | 9.3 | 3 | 0.177[e] | 0.294[e] | 0.177[e] | 0.294[e] | | | | |
| Poultry meat | Laying: 0.0044 | Laying: 0.0044 | 0.15 | 10 | n.r. | n.r. | n.r. | n.r. | <0.01[f] | <0.01[f] | <0.01 | -- |
| | | | 0.46 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | | | | |
| | | | 1.5 | 10 | 0.01 | 0.013 | 0.01 | 0.013 | | | | |
| Poultry fat | Laying: 0.0044 | Laying: 0.0044 | 0.15 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01[f] | <0.01[f] | <0.01* | -- |
| | | | 0.46 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | | | | |
| | | | 1.5 | 10 | 0.01 | 0.025 | 0.01 | 0.025 | | | | |
| Poultry liver+kidney | Laying: 0.0044 | Laying: 0.0044 | 0.15 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01[f] | <0.01[f] | <0.01* | -- |
| | | | 0.46 | 10 | 0.015 | 0.023 | 0.015 | 0.023 | | | | |
| | | | 1.5 | 10 | 0.031 | 0.053 | 0.031 | 0.053 | | | | |
| Eggs | Laying: 0.0044 | Laying: 0.0044 | 0.15 | 10 | n.a. | n.a. | n.a. | n.a. | <0.01[f] | <0.01[f] | <0.01* | -- |
| | | | 0.46 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | | | | |
| | | | 1.5 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | | | | |

n.r.: Not reported
(*): Indicates that the MRL is set at the limit of analytical quantification.
(a): Ruminant: based on a 570 kg animal consuming 13.2 kg feed DM/day.
(b): Median residue value according to the enforcement residue definition, derived by interpolation/extrapolation from the feeding study for the median dietary burden (FAO, 2009).

(c): Highest residue value (tissues, eggs) or mean residue value (milk) according to the enforcement residue definition, derived by interpolation/extrapolation of the maximum dietary burden between the relevant feeding groups of the study (FAO, 2009).
(d): The median conversion factor for enforcement to risk assessment.
(e): Residues were determined as dicamba and DCSA together.
(f): Estimated intakes below 30% of lowest feeding level. No values derived by linear regression.

**Table 7.2-15: Overview of the values derived from livestock feeding studies (OECD calculator)**

| Commodity | Dietary burden | | Results of the livestock feeding study | | | | | | Median residue (mg/kg)[b] | Highest residue (mg/kg)[c] | Calculated MRL (mg/kg) | CF for RA[d] |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Med. (mg/kg bw/d) | Max. (mg/kg bw/d) | Dose Level (mg/kg bw/d)(a) | No | Result for enforcement | | Result for RA | | | | | |
| | | | | | Mean (mg/kg) | Max. (mg/kg) | Mean (mg/kg) | Max. (mg/kg) | | | | |
| **EU reviewed data (Denmark, 2007 – Ruminant: Report No. 379 and 324/00; Poultry: Report No. 107-203 and 74)** | | | | | | | | | | | | |
| **Dicamba and its salts and conjugated dicamba expressed as dicamba** | | | | | | | | | | | | |
| Pig meat | Breeding: 0.125 | Breeding: 0.479 | 0.93 | 3 | <0.01[e] | <0.01[e] | <0.01[e] | <0.01[e] | 0.01[f] | 0.002 | <0.01* | -- |
| | | | 2.78 | 3 | 0.012[e] | 0.014[e] | 0.012[e] | 0.014[e] | | | | |
| | | | 9.3 | 3 | 0.030[e] | 0.037[e] | 0.030[e] | 0.037[e] | | | | |
| Pig fat | Breeding: 0.125 | Breeding: 0.479 | 0.93 | 3 | 0.023[e] | 0.046[e] | 0.023[e] | 0.046[e] | 0.003[f] | 0.024 | 0.03 | -- |
| | | | 2.78 | 3 | 0.025[e] | 0.034[e] | 0.025[e] | 0.034[e] | | | | |
| | | | 9.3 | 3 | 0.047[e] | 0.059[e] | 0.047[e] | 0.059[e] | | | | |
| Pig liver | Breeding: 0.125 | Breeding: 0.479 | 0.93 | 3 | 0.026[e] | 0.029[e] | 0.026[e] | 0.029[e] | 0.004[f] | 0.015 | 0.015 | -- |
| | | | 2.78 | 3 | 0.066[e] | 0.070[e] | 0.066[e] | 0.070[e] | | | | |
| | | | 9.3 | 3 | 0.207[e] | 0.207[e] | 0.207[e] | 0.207[e] | | | | |
| Pig kidney | Breeding: 0.125 | Breeding: 0.479 | 0.93 | 3 | 0.154[e] | 0.174[e] | 0.154[e] | 0.174[e] | 0.021[f] | 0.090 | 0.090 | -- |
| | | | 2.78 | 3 | 0.282[e] | 0.288[e] | 0.282[e] | 0.288[e] | | | | |
| | | | 9.3 | 3 | 0.646[e] | 0.885[e] | 0.646[e] | 0.885[e] | | | | |
| Ruminant meat | Dairy: 0.6064 | Dairy: 2.375 | 0.93 | 3 | <0.01[e] | <0.01[e] | <0.01[e] | <0.01[e] | 0.010 | 0.032 | 0.04 | -- |
| | | | 2.78 | 3 | 0.012[e] | 0.014[e] | 0.012[e] | 0.014[e] | | | | |
| | | | 9.3 | 3 | 0.030[e] | 0.037[e] | 0.030[e] | 0.037[e] | | | | |
| Ruminant fat | Dairy: 0.6064 | Dairy: 2.375 | 0.93 | 3 | 0.023[e] | 0.046[e] | 0.023[e] | 0.046[e] | 0.015 | 0.037 | 0.04 | -- |
| | | | 2.78 | 3 | 0.025[e] | 0.034[e] | 0.025[e] | 0.034[e] | | | | |

| Commodity | Dietary burden | | Results of the livestock feeding study | | | | | | Median residue (mg/kg)[b] | Highest residue (mg/kg)[c] | Calculated MRL (mg/kg) | CF for RA[d] |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Med. (mg/kg bw/d) | Max. (mg/kg bw/d) | Dose Level (mg/kg bw/d)(a) | No | Result for enforcement | | Result for RA | | | | | |
| | | | | | Mean (mg/kg) | Max. (mg/kg) | Mean (mg/kg) | Max. (mg/kg) | | | | |
| **EU reviewed data (Denmark, 2007 – Ruminant: Report No. 379 and 324/00; Poultry: Report No. 107-203 and 74)** | | | | | | | | | | | | |
| **Dicamba and its salts and conjugated dicamba expressed as dicamba** | | | | | | | | | | | | |
| | | | 9.3 | 3 | 0.047[e] | 0.059[e] | 0.047[e] | 0.059[e] | | | | |
| Ruminant liver | Dairy: 0.6064 | Dairy: 2.375 | 0.93 | 3 | 0.026[e] | 0.029[e] | 0.026[e] | 0.029[e] | 0.017 | 0.061 | 0.07 | -- |
| | | | 2.78 | 3 | 0.066[e] | 0.070[e] | 0.066[e] | 0.070[e] | | | | |
| | | | 9.3 | 3 | 0.207[e] | 0.207[e] | 0.207[e] | 0.207[e] | | | | |
| Ruminant kidney | Dairy: 0.6064 | Dairy: 2.375 | 0.93 | 3 | 0.154[e] | 0.174[e] | 0.154[e] | 0.174[e] | 0.100 | 0.279 | 0.30 | -- |
| | | | 2.78 | 3 | 0.282[e] | 0.288[e] | 0.282[e] | 0.288[e] | | | | |
| | | | 9.3 | 3 | 0.646[e] | 0.885[e] | 0.646[e] | 0.885[e] | | | | |
| Milk | Dairy: 0.6064 | Dairy: 2.375 | 0.93 | 3 | 0.02[e] | 0.029[e] | 0.02[e] | 0.029[e] | 0.013 | 0.039 | 0.04 | -- |
| | | | 2.78 | 3 | 0.035[e] | 0.055[e] | 0.035[e] | 0.055[e] | | | | |
| | | | 9.3 | 3 | 0.177[e] | 0.294[e] | 0.177[e] | 0.294[e] | | | | |
| Poultry meat | Laying: 0.016 | Laying: 0.024 | 0.15 | 10 | n.r. | n.r. | n.r. | n.r. | <0.01 | <0.01 | <0.01 | -- |
| | | | 0.46 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | | | | |
| | | | 1.5 | 10 | 0.01 | 0.013 | 0.01 | 0.013 | | | | |
| Poultry fat | Laying: 0.016 | Laying: 0.024 | 0.15 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01* | -- |
| | | | 0.46 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | | | | |
| | | | 1.5 | 10 | 0.01 | 0.025 | 0.01 | 0.025 | | | | |
| Poultry liver+kidney | Laying: 0.016 | Laying: 0.024 | 0.15 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01* | -- |
| | | | 0.46 | 10 | 0.015 | 0.023 | 0.015 | 0.023 | | | | |
| | | | 1.5 | 10 | 0.031 | 0.053 | 0.031 | 0.053 | | | | |
| Eggs | Laying: 0.016 | Laying: 0.024 | 0.15 | 10 | n.a. | n.a. | n.a. | n.a. | <0.01 | <0.01[f] | <0.01* | -- |
| | | | 0.46 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | | | | |

| Commodity | Dietary burden | | Results of the livestock feeding study | | | | | | Median residue (mg/kg)[b] | Highest residue (mg/kg)[c] | Calculated MRL (mg/kg) | CF for RA[d] |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Med. (mg/kg bw/d) | Max. (mg/kg bw/d) | Dose Level (mg/kg bw/d)(a) | No | Result for enforcement | | Result for RA | | | | | |
| | | | | | Mean (mg/kg) | Max. (mg/kg) | Mean (mg/kg) | Max. (mg/kg) | | | | |
| **EU reviewed data (Denmark, 2007 – Ruminant: Report No. 379 and 324/00; Poultry: Report No. 107-203 and 74)** | | | | | | | | | | | | |
| **Dicamba and its salts and conjugated dicamba expressed as dicamba** | | | | | | | | | | | | |
| | | | 1.5 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | | | | |

n.r.: Not reported
(*): Indicates that the MRL is set at the limit of analytical quantification.
(a): Ruminant: based on a 570 kg animal consuming 13.2 kg feed DM/day.
(b): Median residue value according to the enforcement residue definition, derived by interpolation/extrapolation from the feeding study for the median dietary burden (FAO, 2009).
(c): Highest residue value (tissues, eggs) or mean residue value (milk) according to the enforcement residue definition, derived by interpolation/extrapolation of the maximum dietary burden between the relevant feeding groups of the study (FAO, 2009).
(d): The median conversion factor for enforcement to risk assessment.
(e): Residues were determined as dicamba and DCSA together.
(f): Estimated intakes below 30% of lowest feeding level. No values derived by linear regression.

**Conclusion on feeding studies**

The requested uses modify the theoretical maximum daily intake for animals, but regarding available feeding data, there is no risk for animal MRL to be exceeded.
EU MRLs for animal commodities as specified in Reg. (EU) 2015/845 all exceed the calculated MRLs.

**Evaluator comment:**

Information presented by Applicant are sufficient.
Additionally, Table 7.2-16 with calculations of expected residues at dietary burden calculated with Animal model 2017 has been added below.

The requested use and the new mode of calculation modify the theoretical maximum daily intake for animals, but regarding available feeding data, there is no risk for animal MRL to be exceeded.
No additional data are required.

**Table 7.2-16: Overview of the values derived from livestock feeding studies (OECD calculator)**

| Commodity | Dietary burden | | Results of the livestock feeding study | | | | | | Median residue (mg/kg)[b] | Highest residue (mg/kg)[c] | Calculated MRL (mg/kg) | CF for RA[d] |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Med. (mg/kg bw/d) | Max. (mg/kg bw/d) | Dose Level (mg/kg bw/d)(a) | No | Result for enforcement | | Result for RA | | | | | |
| | | | | | Mean (mg/kg) | Max. (mg/kg) | Mean (mg/kg) | Max. (mg/kg) | | | | |
| **EU reviewed data (Denmark, 2007 – Ruminant: Report No. 379 and 324/00; Poultry: Report No. 107-203 and 74)** | | | | | | | | | | | | |
| **Dicamba and its salts and conjugated dicamba expressed as dicamba** | | | | | | | | | | | | |
| Pig meat | Breeding: 0.125 | Breeding: 0.478 | 0.93 | 3 | <0.01[e] | <0.01[e] | <0.01[e] | <0.01[e] | <0.01[f] | <0.01 | 0.01* | -- |
| | | | 2.78 | 3 | 0.012[e] | 0.014[e] | 0.012[e] | 0.014[e] | | | | |
| | | | 9.3 | 3 | 0.030[e] | 0.037[e] | 0.030[e] | 0.037[e] | | | | |
| Pig fat | Breeding: 0.125 | Breeding: 0.478 | 0.93 | 3 | 0.023[e] | 0.046[e] | 0.023[e] | 0.046[e] | <0.01[f] | 0.024 | 0.03 | -- |
| | | | 2.78 | 3 | 0.025[e] | 0.034[e] | 0.025[e] | 0.034[e] | | | | |
| | | | 9.3 | 3 | 0.047[e] | 0.059[e] | 0.047[e] | 0.059[e] | | | | |
| Pig liver | Breeding: 0.125 | Breeding: 0.478 | 0.93 | 3 | 0.026[e] | 0.029[e] | 0.026[e] | 0.029[e] | <0.01[f] | 0.015 | 0.015 | -- |
| | | | 2.78 | 3 | 0.066[e] | 0.070[e] | 0.066[e] | 0.070[e] | | | | |
| | | | 9.3 | 3 | 0.207[e] | 0.207[e] | 0.207[e] | 0.207[e] | | | | |
| Pig kidney | Breeding: 0.125 | Breeding: 0.478 | 0.93 | 3 | 0.154[e] | 0.174[e] | 0.154[e] | 0.174[e] | 0.021[f] | 0.089 | 0.09 | -- |
| | | | 2.78 | 3 | 0.282[e] | 0.288[e] | 0.282[e] | 0.288[e] | | | | |
| | | | 9.3 | 3 | 0.646[e] | 0.885[e] | 0.646[e] | 0.885[e] | | | | |
| Ruminant meat | Dairy: 0.6063 | Dairy: 2.371 | 0.93 | 3 | <0.01[e] | <0.01[e] | <0.01[e] | <0.01[e] | 0.010 | 0.014 | 0.015 | -- |
| | | | 2.78 | 3 | 0.012[e] | 0.014[e] | 0.012[e] | 0.014[e] | | | | |
| | | | 9.3 | 3 | 0.030[e] | 0.037[e] | 0.030[e] | 0.037[e] | | | | |
| Ruminant fat | Dairy: 0.6063 | Dairy: 2.371 | 0.93 | 3 | 0.023[e] | 0.046[e] | 0.023[e] | 0.046[e] | 0.018 | 0.037 | 0.04 | -- |
| | | | 2.78 | 3 | 0.025[e] | 0.034[e] | 0.025[e] | 0.034[e] | | | | |
| | | | 9.3 | 3 | 0.047[e] | 0.059[e] | 0.047[e] | 0.059[e] | | | | |
| Ruminant liver | Dairy: 0.6063 | Dairy: 2.371 | 0.93 | 3 | 0.026[e] | 0.029[e] | 0.026[e] | 0.029[e] | 0.017 | 0.060 | 0.06 | -- |
| | | | 2.78 | 3 | 0.066[e] | 0.070[e] | 0.066[e] | 0.070[e] | | | | |

| Commodity | Dietary burden | | Results of the livestock feeding study | | | | | | Median residue (mg/kg)[b] | Highest residue (mg/kg)[c] | Calculated MRL (mg/kg) | CF for RA[d] |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Med. (mg/kg bw/d) | Max. (mg/kg bw/d) | Dose Level (mg/kg bw/d)(a) | No | Result for enforcement | | Result for RA | | | | | |
| | | | | | Mean (mg/kg) | Max. (mg/kg) | Mean (mg/kg) | Max. (mg/kg) | | | | |
| **EU reviewed data (Denmark, 2007 – Ruminant: Report No. 379 and 324/00; Poultry: Report No. 107-203 and 74)** | | | | | | | | | | | | |
| **Dicamba and its salts and conjugated dicamba expressed as dicamba** | | | | | | | | | | | | |
| | | | 9.3 | 3 | 0.207[e] | 0.207[e] | 0.207[e] | 0.207[e] | | | | |
| Ruminant kidney | Dairy: 0.6063 | Dairy: 2.371 | 0.93 | 3 | 0.154[e] | 0.174[e] | 0.154[e] | 0.174[e] | 0.100 | 0.279 | 0.3 | -- |
| | | | 2.78 | 3 | 0.282[e] | 0.288[e] | 0.282[e] | 0.288[e] | | | | |
| | | | 9.3 | 3 | 0.646[e] | 0.885[e] | 0.646[e] | 0.885[e] | | | | |
| Milk | Dairy: 0.6063 | Dairy: 2.371 | 0.93 | 3 | 0.02[e] | 0.029[e] | 0.02[e] | 0.029[e] | 0.013 | 0.032 | 0.04 | -- |
| | | | 2.78 | 3 | 0.035[e] | 0.055[e] | 0.035[e] | 0.055[e] | | | | |
| | | | 9.3 | 3 | 0.177[e] | 0.294[e] | 0.177[e] | 0.294[e] | | | | |
| Poultry meat | Laying: 0.016 | Laying: 0.024 | 0.15 | 10 | n.r. | n.r. | n.r. | n.r. | <0.01 | <0.01 | <0.01* | -- |
| | | | 0.46 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | | | | |
| | | | 1.5 | 10 | 0.01 | 0.013 | 0.01 | 0.013 | | | | |
| Poultry fat | Laying: 0.016 | Laying: 0.024 | 0.15 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01* | -- |
| | | | 0.46 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | | | | |
| | | | 1.5 | 10 | 0.01 | 0.025 | 0.01 | 0.025 | | | | |
| Poultry liver+kidney | Laying: 0.016 | Laying: 0.024 | 0.15 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01* | -- |
| | | | 0.46 | 10 | 0.015 | 0.023 | 0.015 | 0.023 | | | | |
| | | | 1.5 | 10 | 0.031 | 0.053 | 0.031 | 0.053 | | | | |
| Eggs | Laying: 0.016 | Laying: 0.024 | 0.15 | 10 | n.a. | n.a. | n.a. | n.a. | <0.01 | <0.01[f] | <0.01* | -- |
| | | | 0.46 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | | | | |
| | | | 1.5 | 10 | <0.01 | <0.01 | <0.01 | <0.01 | | | | |

n.r.: Not reported
(*): Indicates that the MRL is set at the limit of analytical quantification.
(a): Ruminant: based on a 570 kg animal consuming 13.2 kg feed DM/day.
(b): Median residue value according to the enforcement residue definition, derived by interpolation/extrapolation from the feeding study for the median dietary burden (FAO, 2009).

(c): Highest residue value (tissues, eggs) or mean residue value (milk) according to the enforcement residue definition, derived by interpolation/extrapolation of the maximum dietary burden between the relevant feeding groups of the study (FAO, 2009).
(d): The median conversion factor for enforcement to risk assessment.
(e): Residues were determined as dicamba and DCSA together.
(f): Estimated intakes below 30% of lowest feeding level. No values derived by linear regression.

## 7.2.5 Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation) (KCA 6.5.2-6.5.3)

### 7.2.5.1 Available data for all crops under consideration

Processing studies have been conducted in barley, oats and wheat; these have not been previously submitted for evaluation under Directive 91/414/EEC. This data is currently under evaluation for the renewal of approval of the active substance, dicamba (Commission Implementing Regulation (EU) No. 844/2012 of 18 September 2012).
Since processing studies are not triggered for maize, which is the only crop relevant for this submission, details of these data are not presented.
No new data were submitted in the framework of this application.

### 7.2.5.2 Conclusion on processing studies

Processing studies are not required to support the proposed uses of dicamba in this submission.

**zRMS comments:**

Information given by the Applicant is sufficient.
Processing studies are required if the level of residue in the plant or plant product to be processed exceeds >0.1 mg/kg (OECD Test Guideline 508: Magnitude of the pesticide residues in processed commodities). Total residues of dicamba and 5-OH-dicamba not exceeded 0.1 mg/kg in the RAC for maize in supervised residue trials for representative use, so processing studies are not required to support the proposed use of dicamba in this submission.
No additional data are required.

## 7.2.6 Magnitude of residues in representative succeeding crops

The crops under consideration can be grown in rotation.

### 7.2.6.1 Field rotational crop studies (KCA 6.6.2)

**Available data**

New studies for residues in succeeding crops have been submitted by the applicant in the framework of this application. This data is currently under evaluation for the renewal of approval of the active substance, dicamba (Commission Implementing Regulation (EU) No. 844/2012 of 18 September 2012).
Four field trials were conducted between 2014 and 2016 to investigate the magnitude of the residues of dicamba and its metabolite 5-OH-dicamba in succeeding/rotated crops. Two trials were conducted in northern Europe and two trials were conducted in southern Europe. A summary of the trials is presented in the table below. The detailed results are presented in Appendix (see A 2.1.6).

**Table 7.2-17: Summary of studies on the magnitude of residues in field rotational crops**

| Primary crop | Rate (kg a.s./ha) (GS at application or PHI) | Residue levels in succeeding crops | | | |
|---|---|---|---|---|---|
| | | Succeeding crop group | Succeeding crop | Sowing intervals (DAT) | Reference / Remarks |
| **EU reviewed data** | | | | | |
| **New data** | | | | | |
| None/bare soil | 480 | Leafy vegetables | Spinach | 31, 61, 294<br>or<br>29, 60, 270 | Andrews, 2016a<br>Report no. TK0223573-REG |
| None/bare soil | 480 | Root and tuber vegetables | Carrot | 31, 61, 294<br>or<br>29, 60, 270 | |

| Primary crop | Rate (kg a.s./ha) (GS at application or PHI) | Residue levels in succeeding crops | | | |
|---|---|---|---|---|---|
| | | Succeeding crop group | Succeeding crop | Sowing intervals (DAT) | Reference / Remarks |
| None/bare soil | 480 | Cereals | Spring barley | 31, 61, 294<br>or<br>29, 60, 270 | |
| None/bare soil | 480 | Leafy vegetables | Spinach | 31, 61, 294<br>or<br>29, 60, 270 | Andrews & Austin, 2016<br>Report no. NC14032 |
| None/bare soil | 480 | Root and tuber vegetables | Carrot | 31, 61, 294<br>or<br>29, 60, 270 | |
| None/bare soil | 480 | Cereals | Spring barley | 31, 61, 294<br>or<br>29, 60, 270 | |

**Summary of new studies on the magnitude of residues in rotational crops not yet evaluated in the EU**
*Reference: Andrews, 2016a, Andrews & Austin, 2016*
Four limited rotational field trials were conducted to investigate the magnitude of residues in rotational crops. Residues of parent dicamba and 5-OH-dicamba were observed in barley commodities (whole plant, straw, grain) and carrot (tops and leaves). These were predominantly seen at the 30DAT plantback interval and declined over time across the later plantback intervals. This magnitude and distribution of residues is consistent with those seen in the confined rotational studies.
The observed residue levels for cereal commodities were significantly lower than those seen in the magnitude of residue trials for cereals and therefore have no impact on the dietary burden calculations. [The dietary exposure determined in the dietary burden calculations used these magnitude of residue trial input values thus providing a worst case scenario]. Residue levels of parent dicamba were lower in wheat and barley grain samples than the established MRLs for these crop commodities. Therefore any uptake of parent dicamba into cereals as rotated crops is not anticipated to impact on established MRLs where application is made at the maximum seasonal rate of 480g a.s./ha.
Very low residues of parent dicamba were observed in one trial only in carrot top and leaves at the nominal 30 and 60 day plant back intervals and no detectable residues were observed at the final 270 day plantback timing. When taking account of these root crop leaf residues based on the low levels found in this single trial (0.02 mg/kg), the contribution to the animal dietary burden is negligible. Therefore these findings do not present any concern.
The proposed definition of the residue in succeeding crops is therefore considered consistent with the definition of the residue for risk assessment in primary crops.

**Conclusion on rotational crops studies**
Considering the overdosing factor of the above study and the fact that dicamba was applied to a bare soil (interception of dicamba by the plants is expected in practice), it can be concluded that dicamba residue levels in rotational commodities are not expected to exceed 0.01 mg/kg, provided that dicamba is applied in compliance with the GAPs supported for this submission.

**zRMS comments:**

Information given by the Applicant is sufficient.
Four rotational field trials were conducted in northern and southern Europe to investigate the magnitude of residues in rotational crops. This data is currently under evaluation for the renewal of approval of the active substance, dicamba.
Since these trials are overdosed compared to the intended use pattern, residues of dicamba and 5-OH- dicamba is not expected in edible parts of crops above the LOQ of 0.01 mg/kg. Restriction on rotational crops are therefore not necessary.
For details, please refer to the evaluation presented in Appendix 2, point A 2.1.6.

Crops under evaluation are expected to be grown in rotation.
No waiting periods beyond normal agricultural practice are proposed for succeeding crops to be planted.

## 7.2.7 Other / special studies (KCA 6.10, 6.10.1)

The available data for the active substance sufficiently address aspects of the residue situation that might arise from the use of A18032E. Therefore, other special studies are not needed.

**zRMS comments:**

Information given by the Applicant is sufficient.
No additional data are requred.

## 7.2.8 Estimation of exposure through diet and other means (KCA 6.9)

Toxicological reference values relevant for dietary risk assessment are reported in the summary of the evaluation (see Table 7.1-2).

### 7.2.8.1 Input values for the consumer risk assessment

**Table 7.2-18: Input values for the consumer risk assessment**

| Commodity | Chronic risk assessment | | Acute risk assessment | |
|---|---|---|---|---|
| | Input value (mg/kg) | Comment | Input value (mg/kg) | Comment |
| Risk assessment residue definition:<br>Sum of dicamba, 5-OH-dicamba, free and conjugated, expressed as dicamba | | | | |
| Intended/relevant uses | | | | |
| Maize / corn | 0.5 | EU MRL* | 0.5 | EU MRL* |
| Further uses | | | | |
| Other commodities of plant or animal origin | variable | EU MRL* | Acute risk assessment was undertaken only for the crops under consideration. | |

* Regulation (EU) 2015/845 of 27 May 2015

### 7.2.8.2 Conclusion on consumer risk assessment

Extensive calculation sheets are presented below (see 0).

**Table 7.2-19: Consumer risk assessment**

| | |
|---|---|
| ADI | 0.3 mg/kg bw/d |
| TMDI (% ADI) according to EFSA PRIMo rev. 3.1 | 19% (based on GEMS/Food G11 diet) |
| IEDI (% ADI) according to EFSA PRIMo rev. 3.1 | Not relevant. TMDI < 100%. |
| ARfD | 0.3 mg/kg bw |
| IESTI (% ARfD) according to EFSA PRIMo rev. 3.1* | Unprocessed commodities:<br>Maize / corn: 1% (based on UK infant diet)<br><br>Processed commodities:<br>Maize / oil: 4% (based on NL toddler diet)<br>Maize / processed (not specified): 0.4% (based on NL toddler diet) |
| NTMDI (% ADI) ** | Not relevant. |
| NEDI (% ADI)** | Not relevant. |

| | |
|---|---|
| NESTI (% ARfD) ** | Not relevant. |

* include raw and processed commodities if both values are required for PRIMo
** if national model is available

Chronic and acute exposure calculations were performed using revision 3.1 of the EFSA Pesticide Residues Intake Model (PRIMo rev. 3.1) provided on the internet homepage of EFSA (https://www.efsa.europa.eu/). This exposure assessment model contains the relevant European food consumption data for different subgroups of the EU population. The model was developed to calculate simultaneously the short-term (acute) and long-term (chronic) dietary exposure to pesticide residue in food according to internationally agreed methodologies. The exposure is compared to the toxicological reference values (i.e. the ADI and the ARfD).

- The potential chronic dietary exposure was compared to the ADI of 0.3 mg/kg bw/d and TMDI values were achieved. Input values for all commodities were derived from existing EU MRL (Reg. (EU) 2015/845), representing a worst case scenario. The highest chronic exposure was calculated for GEMS/Food G11 diet, representing 19% of the ADI. For this diet the highest contributors were soyabeans (12% of ADI), wheat (2% of ADI) and barley (2% of ADI). Since TMDI values are below 100%, there is no need to perform higher tier/refined chronic exposure calculation.
- The potential acute dietary exposure was compared to the ARfD of 0.3 mg/kg bw and IESTI values were achieved. Input values were put from existing EU MRL (Reg. (EU) 2015/845) representing a worst case scenario, only for intended/relevant uses. With regard to the acute exposure there is no exceedance of the ARfD for children and adults in unprocessed and processed commodities. The maximum IESTI calculated for unprocessed commodity: maize / corn is 1% of ARfD (UK infant diet) and for processed commodity: maize / oil: 4% of ARfD (NL toddler diet), maize / processed (not specified): 0.4 % of ARfD (NL toddler diet). Since IESTI values are below 100%, there is no need to perform higher tier/refined acute exposure calculation.

The proposed uses of dicamba in the formulation NIKITA (A18032E) does not represent unacceptable chronic and acute risks for the consumer.

**zRMS comment:**

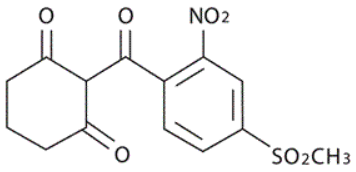
Information given by the Applicant is sufficient.
The calculation of the TMDI using EFSA model (PRIM0 ver. 3.1) and MRLs according to Regulation (EU) 2015/845 of 27 May 2015 led to a utilisation of the ADI of 19% for GEMS/Food G11 diet with the highest value. For this diet, the highest contributor is soyabeans with 12% of the ADI. The intended uses will not result in a consumer chronic exposure exceeding the ADI.

An acute consumer risk assessment was performed with MRL for mazie only. The highest International Estimated Short-Term Intake (IESTI) is at 1% and 0.4% of the ARfD for the consumption of mazie/corn by children and by adults respectively.
The proposed use of dicamba in the product NIKITA (A18032E) do not represent unacceptable acute and chronic risks for the consumer.
No further data are required to support the proposed use.

## 7.3 Mesotrione

General data on mesotrione are summarized in the table below (last updated 2016/12/01)

**Table 7.3-1: General information on mesotrione**

| | |
|---|---|
| Active substance (ISO Common Name) | Mesotrione |
| IUPAC | 2-(4-mesyl-2-nitrobenzoyl) cyclohexane -1,3-dione |
| Chemical structure |  |
| Molecular formula | $C_{14}H_{13}NO_7S$ |
| Molar mass | 339.3 g/mol |
| Chemical group | Triketone herbicide |
| Mode of action (if available) | Inhibition of HPPD (p-Hydroxyphenylpyruvate dioxygenase) |
| Systemic | Yes |
| Company (ies) | Syngenta |
| Rapporteur Member State (RMS) | United Kingdom |
| Approval status | Approved<br>01/10/2003<br>Commission Directive 2003/68/EC.<br>Renewal of the approval of mesotrione<br>01/06/2017<br>Commission Implementing Regulation (EU) 2017/725 of 24 April 2017 |
| Restriction | Restricted to uses as a herbicide |
| Review Report | SANCO/1416/2001 – Final 14/04/2003<br>SANTE/11654/2016, 23 March 2017 |
| Current MRL regulation | ~~Regulation (EU) 2016/53~~<br>Commission Regulation (EU) 2017/626 of 31 March 2017 |
| Peer review of MRLs according to Article 12 of Reg No 396/2005 EC performed | Yes (EFSA, 2015) |
| EFSA Journal : Conclusion on the peer review | Yes (EFSA, 2016) |
| Current MRL applications on intended uses | None |

### 7.3.1 Stability of Residues (KCA 6.1)

#### 7.3.1.1 Stability of residues during storage of samples

**Available data**

No new data submitted in the framework of this application.

**Table 7.3-2: Summary of stability data achieved at ≤ - 18°C (unless stated otherwise)**

| Commodity category | Commodity | Acceptable maximum storage period | Report Reference | Source |
|---|---|---|---|---|
| **EU reviewed data** | | | | |
| **Plant products** | | | | |
| High water content | Maize, forage | 31 months | Wiebe, 1997<br>Report No: RR 97-042B | UK, 2015, 2015a |
| | Maize, fodder | 42 months | | |

| Commodity category | Commodity | Acceptable maximum storage period | Report Reference | Source |
|---|---|---|---|---|
| | Radish, root | 44 months | INT<br>Wiebe & Peyton, 1999<br>Report No. RR 97-042B<br>FIN | |
| High starch content | Maize, grain | 42 months | | |
| High protein content | Soybean, seed | 40 months | | |
| **Animal Products** | | | | |
| -- | -- | -- | Not required | UK, 2015, 2015a |

**Conclusion on stability of residues during storage**
The potential for degradation of residues during storage has been previously assessed in the framework of the peer review for mesotrione. Storage stability of mesotrione for high water, starch and protein commodities when frozen (approximately -18°C) was demonstrated as listed in the table above.
Sufficient stability has been demonstrated to support the residue data presented in the submission.

**zRMS comments:**

In the RAR (2015) it is stated that mesotrione and metabolite MNBA are considered to be stable in corn grain and fodder for at least 42 months and in corn forage for 31 months and 42 months respectively under freezer storage at -18°C ± 5°C.
According to the EFSA Journal 2016;14(3):4419:

**Stability of residues (Regulation (EU) N° 283/2013, Annex Part A, point 6.1)**

| Plant products (Category) | Commodity | T (°C) | Stability (Months) | |
|---|---|---|---|---|
| | | | Mesotrione | MNBA |
| High water content | Maize forage | -18°C±5°C | 31 | 42 |
| High oil content | | | | |
| High protein content | | | | |
| High starch content | Maize grain | -18°C±5°C | 42 | 42 |
| High acid content | | | | |

The studies on the magnitude of residues are valid with regard to storage stability.
No additional data are required.

### 7.3.1.2 Stability of residues in sample extracts (KCA 6.1)

**Conclusion on stability of residues in sample extracts**
Procedural recoveries obtained during residue analysis demonstrate the stability of residues of mesotrione in sample extracts and fully support the residue data presented in the submission.

## 7.3.2 Nature of residues in plants, livestock and processed commodities

### 7.3.2.1 Nature of residue in primary crops (KCA 6.2.1)

**Available data**
With relevance to the nature of residues in crops, new data have been submitted in the framework of this application to address further the toxicological profile of metabolite AMBA[1]. A full assessment of the new study is given in the Appendix 2 to Section B6.4.2 (KCA 5.8.1) .

[1] 2-amino-4-methylsulfonyl benzoic acid

**Table 7.3-3: Summary of plant metabolism studies**

| Crop Group | Crop | Label position | Application and sampling details | | | | Report Reference | Source |
|---|---|---|---|---|---|---|---|---|
| | | | Method, F or G[(a)] | Rate | No | Sampling (DAT) | | |
| **EU reviewed data** | | | | | | | | |
| **Pulses and oilseeds** | Peanut | phenyl-(U)-$^{14}$C | Pre-emergence, F | 305 g a.s./ha<br><br>796 g a.s./ha | 1 (1d after planting) | Foliage: 90<br>Hay: 153<br>Hulls, nutmeat: 169 | Brown, 2003. Report No: 1286-01 | UK, 2015, 2015a |
| | | cyclohexane-(U)-$^{14}$C | Pre-emergence, F | 327 g a.s./ha<br><br>836 g a.s./ha | 1 (1d after planting) | Foliage: 90<br>Hay, hulls, nutmeat: 154 | Brumback, 2003 Report No: 1287-01 | |
| | HT soybean | phenyl-(U)-$^{14}$C | Pre-emergence, G | 217.7 g a.s./ha | 1 (1d after planting) | Forage: 28<br>Hay: 42<br>Seed: 123 | Dohn & Chu, 2012 Report No. NC 27419 | UK, 2015, 2015a |
| | | | Pre-/post-emergence, G | 345.5 g a.s./ha (217.7 g a.s./ha pre- plus 127.8 g a.s./ha post-emerge.) | 2 (1 d, 34 d after planting) | Forage: 28/--<br>Hay: 42/9<br>Seed: 123/90 | | |
| | | | Post-emergence, G | 224.2 g a.s./ha | 1 (12 d after planting) | Forage: 22<br>Hay: 40<br>Seed: 110 | | |
| | | cyclohexane-(U)-$^{14}$C | Pre-emergence, G | 225.8 g a.s./ha | 1 (1d after planting) | Forage: 28<br>Hay: 42<br>Seed: 123 | | |
| | | | Pre-/post-emergence, G | 356 g a.s./ha (225.8 g a.s./ha pre-, plus 130.2 g a.s./ha post-emerge.) | 2 (1 d, 34 d after planting) | Forage: 28/--<br>Hay: 42/9<br>Seed: 123/90 | | |
| | | | Post-emergence, G | 229.6 g a.s./ha | 1 (12 d after planting) | Forage: 22<br>Hay: 40<br>Seed: 118 | | |
| **Cereals/grass crops** | Maize | phenyl-(U)-$^{14}$C | Pre-emergence, F | 280 g a.s./ha | 1 (at planting) | Forage: 27<br>Grain, fodder: 153 | Tarr & van Neste, 1997 Report No: RR 96-007B | UK, 2015, 2015a |
| | | | Post-emergence, F | 164 g a.s./ha | 1 (28 d after planting) | Forage: 28<br>Grain, fodder: 125 | Wei & Dohn, 1997 Report No: RR 96-026B | |
| | | cyclohexane-(U)-$^{14}$C | Pre-emergence, F | 307 g a.s./ha | 1 (at planting) | Forage: 27<br>Grain, fodder: 153 | Tarr & van Neste, 1997 Report No: RR 96-007B | |
| | | | Post- | 161 g | 1 (28 d | Forage: 28 | Wei & | |

| Crop Group | Crop | Label position | Application and sampling details | | | | Report Reference | Source |
|---|---|---|---|---|---|---|---|---|
| | | | Method, F or G[(a)] | Rate | No | Sampling (DAT) | | |
| | | | emergence, F | a.s./ha | after planting) | Grain, fodder: 153 | Dohn, 1997 Report No: RR 96-026B | |

**Summary of plant metabolism studies reported in the EU**

*Reference:* EFSA, 2016

"Plant metabolism was studied in maize (pre- and post-emergence), peanuts (pre-emergence) and genetically modified soya bean (pre-, post-emergence and combined pre-/post-emergence) with mesotrione labelled on cyclohexane-2-$^{14}$C and phenyl-U-$^{14}$C.

The metabolic pattern of mesotrione was found to be quantitatively different in conventional crops (maize, peanut) compared to genetically modified soya bean. In maize and peanuts, parent mesotrione was hardly recovered (3% TRR in maize forage only) whilst the most pertinent metabolites identified in the feed items were MNBA[1] (up to 20% TRR in maize forage leaves) and AMBA[2], free and conjugated (13% and 28% TRR respectively in maize forage leaves and fodder; 15% TRR in peanut meat). Further identification of metabolites was not conducted in maize grain due to the very low recovered total residues (0.014 mg/kg).

In genetically modified herbicide tolerant soya bean, parent mesotrione was less extensively metabolised compared to conventional crops and occurred in forage at up to 18% TRR and in soya bean seed (10% TRR). The predominant compounds were identified as 4/5-hydroxy mesotrione (forage 19% TRR; hay 25% TRR; seed 8% TRR) and MNBA (forage 25% TRR; hay 20% TRR; seed 5% TRR). AMBA compound was never detected.

The unextracted radioactivity was further characterized as polar compounds (soya bean), lipids (peanut meat) and carbohydrates (maize) incorporated into the natural constituents of the plant. The metabolism of mesotrione in maize, peanuts and soya bean proceeds by oxidation of the parent molecule to 4/5-hydroxy mesotrione and to MNBA with subsequent reduction to AMBA and its conjugates observed in conventional maize and peanuts only. The metabolism of mesotrione in rotational crops was found to be similar to the primary crops.

Since the absolute concentration of all metabolites was below 0.01 mg/kg in the seeds, the residue definition for enforcement and risk assessment was set as mesotrione only for food commodities.

For feed commodities, the potential inclusion of the predominant metabolites MNBA and AMBA (free and conjugated) besides mesotrione in the residue definition for risk assessment was envisaged. MNBA was characterized as non-genotoxic and of lower toxicity compared to the parent compound and was never detected in the GAP-compliant residue trials on maize (<0.01 mg/kg).

In contrast, a genotoxic potential in vivo could not be excluded for AMBA and repeated dose toxicity profile needed to be addressed (see data gap in Mammalian toxicity). For risk assessment in feed commodities and pending on the toxicological profile of AMBA conjugates, the residue definition is provisionally proposed as mesotrione and AMBA (including its conjugates).

If it can be demonstrated that the conjugates of AMBA are not genotoxic and of no toxicological relevance, additional residue trials on maize where AMBA is analysed for are not needed and only mesotrione has to be included in the residue definition. These residue definitions are valid for conventional crops (cereals, pulses and oilseeds) only."

**New data:**

A new study was conducted to address the data gap regarding the toxicological profile of AMBA[3].

AMBA is of low toxicity by the oral route. Although repeat dose studies have not been conducted with AMBA, data available shows that MNBA is almost quantitatively reduced to AMBA in the gastro intestinal

[1] 2-nitro-4-methylsulfonyl benzoic acid

[2] 2-amino-4-methylsulfonyl benzoic acid

[3] See Section B6.4.2 (KCA 5.8.1) Mammalian Toxicology: Dunton, J., 2016. AMBA – Oral (Gavage) Rat Micronucleus Test. Sequani Ltd. Sequani Report No. BFI0493, issue date: 29 July 2016. Unpublished. Syngenta File No. R044276_10010. Please refer to the actual Annex point in B6 document.

tract within 24 hours and it is therefore possible to use data from studies with MNBA to conclude that AMBA would be of low toxicity by the oral route when administered for up to 90 days.
AMBA is negative in the Ames study but was weakly positive in the IVC (In Vivo Cytogenetics) assay. Data on MNBA suggests that AMBA would not be genotoxic in vivo and it is therefore possible to conclude that AMBA would not present a genotoxic hazard. Further, a recently conducted rat micronucleus assay on AMBA confirms that it is not clastogenic in vivo. There was no evidence of clastogenicity or aneugenicity following oral (gavage) administration of AMBA up to the OECD 474 limit dose of 2000 mg/kg/day in male rats. AMBA is considered to be neither clastogenic nor aneugenic in the rat bone marrow micronucleus assay and is of overall low toxicological concern.
It can therefore be concluded that the residue definition for mesotrione in feed commodities of conventional crops (cereals, pulses and oilseeds) does not need to include AMBA and its conjugates.

**Conclusion on metabolism in primary crops**
Based on the available metabolism data on plants and the new toxicological data on AMBA, it can be concluded that the residue definition for risk assessment in food and feed commodities should include mesotrione parent compound only.

**zRMS comments:**

Information submitted by Applicant is sufficient and accepted.
EFSA (2016) is of the opinion that the metabolism of mesotrione was sufficiently addressed in cereals (for soil and foliar application) and oilseeds (for soil application). Plant metabolism was studied in maize (pre- and post-emergence), peanuts (pre-emergence) and genetically modified soya bean (pre-, post-emergence and combined pre-/post-emergence) with mesotrione labelled on cyclohexane-2-$^{14}$C and phenyl-U-$^{14}$C.
The metabolism of mesotrione in plants following pre- and post-emergence applications is sufficiently addressed to support the proposed use of the product A18032E (Nikita).

**Definition:**
According to the EFSA Journal 2016;14(3):4419:
"*Since the absolute concentration of all metabolites was below 0.01 mg/kg in the seeds, the residue definition for enforcement and risk assessment was set as mesotrione only for food commodities. For feed commodities, the potential inclusion of the predominant metabolites MNBA and AMBA (free and conjugated) besides mesotrione in the residue definition for risk assessment was envisaged. MNBA was characterized as non genotoxic and of lower toxicity compared to the parent compound and was never detected in the GAP-compliant residue trials on maize (<0.01 mg/kg). In contrast, a genotoxic potential in vivo could not be excluded for AMBA and repeated dose toxicity profile needs to be addressed (see data gap in section 2). For risk assessment in feed commodities and pending on the toxicological profile of AMBA conjugates, the residue definition is provisionally proposed as mesotrione and AMBA (including its conjugates). If it can be demonstrated that the conjugates of AMBA are not genotoxic and of no toxicological relevance, additional residue trials on maize where AMBA is analysed for are not needed and only mesotrione has to be included in the residue definition. These residue definitions are valid for conventional crops (cereals, pulses and oilseeds) only. For future uses on genetically modified crops and considering the significant proportions of 4/5-hydroxy mesotrione recovered in soya bean forage and hay, this compound may have to be included in the residue definition for risk assessment pending on its toxicological relevance."*

The residue definition for enforcement is proposed as mesotrione only in cereal grains and pulses and oilseeds.
The residue definition for risk assessment is proposed as:
- for food commodities: mesotrione only in cereal grains and pulses and oilseeds.
- for feed commodities: mesotrione and AMBA (including its conjugates) (cereals, pulses and oilseeds only – conventional crops) – provisional.

In EFSA Journal 2016;14(3):4419 it is stated that *"The consumer dietary risk assessment could not be finalised with regard to products of animal origin as the genotoxic potential of AMBA in vivo could not be ruled out due to positive results obtained in an in vitro cytogenetic assay"*(Issues that could not be finalised).
Applicant presented a new study conducted to address the data gap regarding the toxicological profile of AMBA. AMBA is negative in the Ames study but was weakly positive in the IVC (In Vivo Cytogenetics) assay. Data on MNBA suggests that AMBA would not be genotoxic in vivo and it is therefore possible to conclude that AMBA would not present a genotoxic hazard. AMBA is considered to be neither clastogenic nor aneugenic in the rat bone marrow micronucleus assay and is of overall low toxicological concern.
According to the EFSA Supporting publication 2018:EN-1527 - *"Outcome of the consultation on confirmatory data*

*used in risk assessment for mesotrione":* ***It was agreed that the metabolite AMBA is unlikely to be genotoxic, however it is proposed to further discuss its toxicological profile in an experts' consultation since the metabolite is relevant to consumer exposure.***

The metabolism of mesotrione in rotational crops was found to be similar to the primary crops.
No further data are required to support the proposed uses at the moment.

### 7.3.2.2 Nature of residue in rotational crops (KCA 6.6.1)

**Available data**
No new data submitted in the framework of this application.

**Table 7.3-4: Summary of metabolism studies in rotational crops**

| Crop group | Crop | Label position | Application and sampling details | | | | Report reference | Source |
|---|---|---|---|---|---|---|---|---|
| | | | Method, F or G[(a)] | Rate (g a.s./ha) | Sowing intervals (DAT) | Harvest Intervals (DAT) | | |
| **EU reviewed data** | | | | | | | | |
| **Leafy vegetables** | Endive | phenyl-(U)-$^{14}$C | Soil application, G | 164 | 120, 300[(b)] | Maturity: 198 | Gorder *et al.*, 1997. Report No: RR 96-084B | UK, 2015, 2015a |
| | | cyclohexane-(U)-$^{14}$C | Soil application, G | 164 | 120, 300[(b)] | Maturity: 198 | Spillner *et al.*, 1997 Report No: RR 95-042B | |
| **Root and tuber vegetables** | Radish | phenyl-(U)-$^{14}$C | Soil application, G | 164 | 120, 300[(b)] | Maturity (tops and roots): 176 | Gorder *et al.*, 1997. Report No: RR 96-084B | |
| | | cyclohexane-(U)-$^{14}$C | Soil application, G | 164 | 120, 300[(b)] | Maturity (tops and roots): 176 | Spillner *et al.*, 1997 Report No: RR 95-042B | |
| **Cereals** | Wheat | phenyl-(U)-$^{14}$C | Soil application, G | 164 | 120, 300[(b)] | Forage: 142 Hay: 177 Grain, straw: 254 | Gorder *et al.*, 1997. Report No: RR 96-084B | |
| | | cyclohexane-(U)-$^{14}$C | Soil application, G | 164 | 120, 300[(b)] | Forage: 142 Hay: 177 Grain, straw: 254 | Spillner *et al.*, 1997 Report No: RR 95-042B | |

(a) Outdoor/field application (F) or glasshouse/protected/indoor application (G)
(b) Crops sown 300 DAT were not harvested due to the low level of radioactive residues in the 120 DAT crops.

**Summary of plant metabolism studies on rotational crops reported in the EU**
*Reference:* United Kingdom, 2015, 2015a
One confined rotational crop study investigating the nature of residues following different plant-back

intervals is available.
The metabolism and distribution of mesotrione[1] was investigated in the rotational crops wheat, endive and radish planted 120 and 300 days following soil application of [$^{14}$C] mesotrione to soil in pots at ca 1.2N. A replanting interval of 30 days was not investigated but is not of concern since replanting after this interval would not be anticipated for this crop.
TRR in the plants grown in the soil treated with [$^{14}$C]-cyclohexane labelled mesotrione were <0.001-0.002 mg/kg. TRR in the crops grown in soil treated with [$^{14}$C]-phenyl labelled mesotrione were 0.004 mg/kg in both radish roots and tops, 0.012 mg/kg in endive and 0.033, 0.018, 0.031 and 0.006 mg/kg in wheat forage, hay, straw and grain respectively. The 300 DAT crops were not harvested due to the low levels of radioactivity in the 120 DAT crops.
MNBA, AMBA sulphate and AMBA conjugate were present in all extracts of wheat forage, hay and straw, the only significant component was MNBA at 0.011 mg/kg in wheat forage (33% TRR). Mesotrione was not detected.
All of the plant metabolites have also been determined in mammalian metabolism studies.

**Conclusion on metabolism in rotational crops**
The metabolism of mesotrione is similar in rotational crops to that observed in primary crops (EFSA, 2016).

**zRMS comments:**

The metabolism of mesotrione in rotational crops was found to be similar to the primary crops.
Data presented by Applicant in point 7.3.2.2 have been accepted and are sufficient to support the proposed use.
No further data are required to support the proposed uses.

### 7.3.2.3 Nature of residues in processed commodities (KCA 6.5.1)

**Available data**
No new data submitted in the framework of this application.

**Conclusion on nature of residues in processed commodities**
All samples at harvest contained no residues of mesotrione or metabolite MNBA, with a limit of quantification of 0.01 mg/kg. In addition, consumer intakes accounted for less than 1% of the ADI. Therefore it is considered that processing studies are not required (EFSA, 2016).

**zRMS comments:**

Information given by the Applicant is sufficient. Hydrolysis studies addressing the nature of the residues in processed commodities are not triggered (mesotrione residue levels in maize grain <0.01 mg/kg).
No further data are required to support the proposed uses.

### 7.3.2.4 Conclusion on the nature of residues in commodities of plant origin (KCA 6.7.1)

**Table 7.3-5: Summary of the nature of residues in commodities of plant origin**

| **Endpoints** | |
|---|---|
| Plant groups covered | Pulses and oilseeds (Peanut, HT soybean)<br>Cereals (Maize) |
| Rotational crops covered | Leafy crops (endive), root vegetables (radish) and cereals (wheat) |
| Metabolism in rotational crops similar to metabolism in primary crops? | Yes |
| Processed commodities | Not required |
| Residue pattern in processed commodities similar to pattern | Not applicable |

[1] Referred to as ZA 1296 in the original report

| | |
|---|---|
| in raw commodities? | |
| Plant residue definition for monitoring | Mesotrione (parent only) (Cereals and pulses/oilseeds only) |
| Plant residue definition for risk assessment | Food commodities: Mesotrione (parent only) (cereals and pulses/oilseeds only)<br>Feed commodities: Mesotrione (cereals, pulses and oilseeds only – conventional crops). |
| Conversion factor from enforcement to RA | Not applicable (EFSA, 2016) |

### 7.3.2.5 Nature of residues in livestock (KCA 6.2.2-6.2.5)

**Available data**

With relevance to the nature of residues in animals, new data have been submitted in the framework of this application to address further the toxicological profile of metabolite AMBA. A full assessment of the new study is given in the Appendix 2 to Section B6.4.2 (KCA 5.8.1).

**Table 7.3-6: Summary of animal metabolism studies**

| Group | Species | Label position | No of animal | Application details | | Sample details | | Report reference | Reference |
|---|---|---|---|---|---|---|---|---|---|
| | | | | Rate (mg/kg bw/d) | Duration (days) | Commodity | Time of sampling | | |
| **EU reviewed data** | | | | | | | | | |
| **Lactating ruminants**[a] | Cow | phenyl-(U)-$^{14}$C AMBA | 1 | 0.389 mg/kg bw/day AMBA | 7 | Milk | Twice daily | Hand, 1997 Report No. RJ2309B | UK, 2015, 2015a |
| | | | | | | Liver | 23h after sacrifice | | |
| | | | | | | Kidney | 23h after sacrifice | | |
| | | | | | | Subcutaneous fat | 23h after sacrifice | | |
| | | | | | | Perirenal fat | 23h after sacrifice | | |
| **Laying poultry**[a] | -- | -- | -- | -- | -- | -- | -- | None | |
| **Fish**[b] | -- | -- | -- | -- | -- | -- | -- | None | |

(a) "Since animal intakes are less than 0.004 mg/kg bw/day the need for metabolism studies is not triggered" (UK, 2015, 2015a).
(b) "No guideline is available for possible design of fish metabolism studies or for estimation of dietary burden for farmed fish diet. However, from the uses of mesotrione and the magnitude of residues (all <0.01 mg/kg) it can be expected that there is no potential for residues in commercial fish diet." (UK, 2015, 2015a)

**Summary of animal metabolism studies reported in the EU**

*Reference:* EFSA, 2016

"The livestock dietary burden was tentatively estimated using the highest magnitude of AMBA conjugates residues in maize forage, fodder from the metabolism study and the total residues in maize grain. In this case, livestock metabolism studies are not triggered. A ruminant metabolism study was however conducted with phenyl-U-$^{14}$C AMBA. The total residues were below 0.01 mg/kg in all matrices except in kidney (0.053 mg/kg) and fat (0.018 mg/kg) with AMBA being the predominant compound that accounted for 79% TRR and 62% TRR, respectively. At the estimated dietary burden, the transfer of AMBA residues in all matrices was shown to be negligible and residue definitions for animal commodities are provisionally not required for the representative use. This assessment has however to be reconsidered pending the outcome of AMBA toxicity. Furthermore, the setting of residue definitions for products of animal origin will also have to be assessed with regard to the authorized European uses for mesotrione (maize forage, grass) (EFSA, 2015), and in the case animals are fed with genetically modified soya bean seed (meal) where mesotrione can be

found at significant proportions."
"A fish metabolism study is also not requested."

**New data:**
A new study assessing the toxicological profile of AMBA[1] has shown that AMBA does not present a genotoxic hazard. Further, a recently conducted rat micronucleus assay on AMBA confirms that it is not clastogenic *in vivo*. There was no evidence of clastogenicity or aneugenicity following oral (gavage) administration of AMBA up to the OECD 474 limit dose of 2000 mg/kg/day in male rats. AMBA is considered to be neither clastogenic nor aneugenic in the rat bone marrow micronucleus assay.
It is concluded that with regard to AMBA toxicity, a residue definition for animal commodities is not required.

**Conclusion on metabolism in livestock**
A residue definition for animal commodities is not considered necessary.

**zRMS comments:**

A ruminant, lactating Friesian cow, study using radio-labelled AMBA ((phenyl-U-$^{14}$C AMBA) was assessed for the original approval of mesotrione. As no significant intakes are expected for animals no further data are required.
EFSA stated in its peer review (2016) that the transfer of AMBA residues in all matrices was shown to be negligible at the estimated dietary burden, and that residue definitions for animal commodities are provisionally not required for the representative use on maize.
In EFSA Supporting publication 2018:EN-1527 it is stated that ***the metabolite AMBA is unlikely to be genotoxic, however it is proposed to further discuss its toxicological profile in an experts' consultation since the metabolite is relevant to consumer exposure.***
Data presented by Applicant in point 7.3.2.5 have been accepted and are sufficient to support the proposed use.

## 7.3.2.6 Conclusion on the nature of residues in commodities of animal origin (KCA 6.7.1)

**Table 7.3-7: Summary on the nature of residues in commodities of animal origin**

| | **Endpoints** |
|---|---|
| Animals covered | Not applicable (cow for [phenyl-U-$^{14}$C]- AMBA) |
| Time needed to reach a plateau concentration | Not applicable (milk: 5 days for [phenyl-U-$^{14}$C]-AMBA) |
| Animal residue definition for monitoring | Not applicable |
| Animal residue definition for risk assessment | Not applicable |
| Conversion factor | Not applicable |
| Metabolism in rat and ruminant similar | Not applicable |
| Fat soluble residue | Not applicable |

[1] See Section B6.4.2 (KCA 5.8.1) Mammalian Toxicology: Dunton, J., 2016. AMBA – Oral (Gavage) Rat Micronucleus Test. Sequani Ltd. Sequani Report No. BFI0493, issue date: 29 July 2016. Unpublished. Syngenta File No. R044276_10010. Please refer to the actual Annex point in B6 document.

## 7.3.3 Magnitude of residues in plants (KCA 6.3)

### 7.3.3.1 Summary of European data and new data supporting the intended uses

No new data are submitted in the framework of this application. For the sake of clarity, summaries to all residue trials that are relevant for this submission are presented in Appendix 2.

**Table 7.3-8: Summary of EU reported data supporting the intended uses of A18032E and conformity to existing MRL**

| Commodity | Source | Residue zone (N-EU, S-EU, EU, outside EU) | Evaluation<br>GAP<br>Residue levels (mg/kg)<br>E = according to enforcement residue definition<br>RA = according to risk assessment residue definition | STMR (mg/kg) | HR (mg/kg) | Unrounded OECD calculator MRL (mg/kg) | Current EU MRL (mg/kg)* | MRL compliance |
|---|---|---|---|---|---|---|---|---|
| **Maize grain** | UK, 2015, 2015a | N-EU | GAP: 1x 0.15 kg as/ha, BBCH 12-18, PHI N/A[b]<br>14x <0.01[a] | <0.01 | <0.01 | 0.01* | 0.01* | Yes |
| | | S-EU | GAP: 1x 0.15 kg as/ha, BBCH 12-18, PHI N/A[b]<br>10x <0.01[a] | <0.01 | <0.01 | 0.01* | | Yes |

* Source of EU MRL: ~~Regulation. (EU) No 2016/53~~ Regulation (EU) 2017/626

N/A – not applicable

(a) Definition of residue for enforcement and risk assessment are the same: mesotrione

(b) PHI is determined by crop maturity

(c) Definition of residue for enforcement is not relevant for feed items since no MRL is set.

### 7.3.3.2 Conclusion on the magnitude of residues in plants

Maize is a major crop in central Europe (**SANCO 7525/VI/95 rev.10.1**); sufficient trials are available to support the proposed uses. The trials evaluated at the EU level in the original DAR (United Kingdom, 2015, 2015a) correspond to the proposed uses and demonstrate the zero residue situation.
The data submitted show that no detectable residues are expected in treated crops and no exceedance of the MRL will occur. The proposed uses are considered acceptable.

**zRMS comments:**

Maize is the major crop in northern Europe (EU guideline Document SANCO 7525/VI/95 - rev.10.3 of 13 June 2017 and SANTE/2019/12752). A minimum of eight independent trials representative of the proposed growing area for outdoor are required.

**Table 7.1: Summary of EU critical GAP and intended GAP in maize for mesotrione (EFSA, 2016).**

| Type of GAP | Method | Number of applications | Application rate per treatment (kg as/ha) | Interval between application (days) | Growth stage at last application | PHI (days) |
|---|---|---|---|---|---|---|
| cGAP EU (EFSA, 2016) – maize | Foliar spray | 1 | 0.12 – 0.15 | - | BBCH 12 – 18 | - |
| Intended GAP- maize | Foliar, spraying, overall | 1 | 0.060 | - | BBCH 12 – 14 | - |

The critical EU GAP for maize is most critical than proposed GAP.
The trials conducted on maize (1 x 0.15 kg a.s./ha, BBCH 12-18, PHI not specified, outdoor) have been evaluated at the EU level in the original RAR (United Kingdom, 2015). No PHI is proposed in the critical EU GAP since the application is made pre- or early post-emergence and the maize grain is harvested at maturity.

**Residues of mesotrione in all grain, silage and forage specimens were below the limit of quantification (<0.01 mg/kg).**
Available results show that the in force MRL on maize/corn of 0.01* mg/kg (Reg. (EU) 2017/626) will not be exceeded. Therefore the uses of A18032E (Nikita) can be considered as supported on maize in Central Europe.
The current EU MRLs for mesotrione are sufficient to support the proposed uses.

Applicant proposes to add tank-mixed adjuvant (Adigor, STYK, Olejan or Efica 960 EC) to product ADM.4651.H.1.A (Nikita). Nevertheless, neither trials have been submitted with the tank-mixed adjuvant (Adigor, STYK, Olejan or Efica 960 EC). Residues levels of mesotrione are expected to be below the LOQ at the intended GAP and the TMDI represents only 12% of the ADI. In our opinion, the addition of an adjuvant should not affect the residue level in this appliacation. It could be expected that the use of an adjuvant will not lead to an exceedance of the MRL and will not lead to an unacceptable chronic risk for the consumer. Hence, at the intended GAP, the use of an adjuvant is considered to be supported for mesotrione.
No further data are required to support the proposed uses.

(*) Indicates lower limit of analytical determination

## 7.3.4 Magnitude of residues in livestock

### 7.3.4.1 Dietary burden calculation

The use of A18032E may result in residues of mesotrione in animal feed items, therefore the possible transfer of residues in animal commodities from the proposed uses should be considered. Livestock intake calculations and feeding studies undertaken are provided below in Table 7.3-9.
A review of all existing MRLs has been conducted by EFSA in the framework of Article 12 of Regulation (EC) No 396/2005. This submission is intended for product renewal, therefore all GAPs previously registered in Europe are re-assessed and consequently no reference is made to the Article 12.

**Table 7.3-9: Input values for the dietary burden calculation (considering the uses evaluated in Art. 12 procedure and the uses under consideration)**

| Feed Commodity | Median dietary burden | | Maximum dietary burden | |
|---|---|---|---|---|
| | Input value (mg/kg) | Comment | Input value (mg/kg) | Comment |
| Risk assessment residue definition: mesotrione | | | | |
| Corn, Field, Forage/Silage (Forages group) | 0.01 | STMR from maize trials presented = LOQ | 0.01 | HR from maize trials presented = LOQ |
| Corn, Field, Stover (Forages group) | 0.01 | STMR from maize trials presented = LOQ | 0.01 | HR from maize trials presented = LOQ |
| Corn, Field, Grain (Grain/Seeds group) | 0.01 | STMR from maize trials presented = LOQ | N/A[b] | |
| Corn, Field, Milled Byprods. (By-products group) | 0.01 | LoQ for maize trials and no PF as <LOQ[a] | N/A[b] | |
| Corn, Field, Hominy Meal (By-products group) | 0.01 | LoQ for maize trials and no PF as <LOQ[a] | N/A[b] | |
| Corn, Field, Gluten Feed (By-products group) | 0.01 | LoQ for maize trials and no PF as <LOQ[a] | N/A[b] | |
| Corn, Field, Gluten Meal (By-products group) | 0.01 | LoQ for maize trials and no PF as <LOQ[a] | N/A[b] | |

(a) No default processing factor was applied because terbuthyalzine is applied early in the growing season and residues are expected to be below the LOQ. Concentration of residues in these commodities is therefore not expected.
(b) Only median relevant for grain and processed fractions

**Table 7.3-10: Results of the dietary burden calculation**

| Animal species | Median dietary burden (mg/kg bw/d) | Maximum dietary burden (mg/kg bw/d) | Highest contributing commodity | Max dietary burden (mg/kg DM) | Trigger exceeded (Y/N) |
|---|---|---|---|---|---|
| Risk assessment mesotrione | | | | | |
| Beef cattle* | 0.001 | 0.001 | Corn, field, forage/silage | 0.03 | N |
| Dairy cattle* | 0.001 | 0.001 | Corn, field, forage/silage | 0.02 | N |
| Ram/ewe | 0.0004 | 0.0004 | Corn, field, gluten feed | 0.01 | N |
| Lamb | 0.0005 | 0.0005 | Corn, field, gluten feed | 0.01 | N |
| Breeding swine | 0.0003 | 0.0003 | Corn, field, forage/silage | 0.01 | N |
| Finishing swine* | 0.0003 | 0.0003 | Corn, field, milled by-products | 0.01 | N |
| Broiler poultry | 0.001 | 0.001 | Corn, field, milled by-products | 0.01 | N |
| Layer poultry* | 0.001 | 0.001 | Corn, field, forage/silage | 0.01 | N |
| Turkey | 0.001 | 0.001 | Corn, field, milled by-products | 0.01 | N |

* These categories correspond to those (formerly) assessed at EU level.

### 7.3.4.2 Livestock feeding studies (KCA 6.4.1-6.4.3)

Livestock animals are not exposed to residues *via* feed above the trigger value established in Reg. (EC) No 1107/2009. Feeding studies are therefore not required.
Considering the low levels of residues expected in livestock feed commodities from the crops and associated GAP supported in this submission along with extrapolation of residue results from the livestock nature or residue studies in goats and hens, residues of mesotrione in edible tissues, milk or eggs are not expected to be quantifiable above 0.01 mg/kg.
No new data were submitted in the framework of this application.

**zRMS comments:**

Data presented by Applicant in point 7.3.4 have been accepted and are sufficient to support the proposed uses.

The calculated dietary burdens for mesotrione were found to be below the trigger value of 0.004 mg/kg bw per day for all animal species. Further investigation of residues in animal commodities is therefore not required. However, a ruminant metabolism study was conducted with phenyl-U-$^{14}$C AMBA. The metabolism of mesotrione in livestock is sufficiently addressed to support the proposed uses of the product A12739A / Callisto. The total residues were below 0.01 mg/kg in all matrices except in kidney (0.053 mg/kg) and fat (0.018 mg/kg) with AMBA being the predominant compound that accounted for 79% TRR and 62% TRR, respectively.

In the EFSA Journal 2016;14(3):4419 it is stated that at the estimated dietary burden, the transfer of AMBA residues in all matrices was shown to be negligible and residue definitions for animal commodities are provisionally not required for the representative uses. In EFSA Supporting publication 2018:EN-1527 it is stated that ***the metabolite AMBA is unlikely to be genotoxic, however it is proposed to further discuss its toxicological profile in an experts' consultation since the metabolite is relevant to consumer exposure (see above).***
No additional data are required at the moment.

### 7.3.5 Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation) (KCA 6.5.2-6.5.3)

As quantifiable residues of mesotrione are not expected in the treated crops and the TMDI is <10% (see also 7.2.8), there is no need to investigate the effect of industrial and/or household processing.

### 7.3.5.1 Available data for all crops under consideration

No new data were submitted in the framework of this application.

### 7.3.5.2 Conclusion on processing studies

Processing studies are not required to support the proposed uses of mesotrione in this submission.

**zRMS comments:**

As quantifiable residues of mesotrione are not expected in the treated crops and the chronic exposure does not exceed 10% of the ADI, there is no need to investigate the effect of industrial and/or household processing.
No additional data are required.

### 7.3.6 Magnitude of residues in representative succeeding crops

The crops under consideration can be grown in rotation.
Considering available data dealing with nature of residues (see 7.3.2.2) no study dealing with magnitude of residues in succeeding crops is needed.

### 7.3.6.1 Field rotational crop studies (KCA 6.6.2)

**Available data**

No new data were submitted in the framework of this application.

**Table 7.3-11: Summary of available studies in field rotational crops**

| Primary crop | Rate (kg a.s./ha) (GS at application or PHI) | Residue levels in succeeding crops | | | |
|---|---|---|---|---|---|
| | | Succeeding crop group | Succeeding crop | Sowing intervals (DAT) | Reference / Remarks |
| **EU reviewed data** | | | | | |
| None/bare soil | 340 | Oilseeds/pulses | Soybean | 30 | Barnes & Wiebe, 1997<br>Report: RR 97-044B<br><br>UK, 2015, 2015a |
| | | | | 29 | |
| Maize | 340 + 220 | Leafy | Endive | 74 | |
| | | | | 98 | |
| None/bare soil | 340 | Root and tuber vegetables | Radish | 30 | |
| | | | | 29 | |
| Maize | 340 + 220[(a)] | | | 85 | |
| | | | | 98 | |
| None/bare soil | 340[(a)] | Cereals | Millet | 30 | |
| | | | | 29 | |
| None/bare soil | 340[(a)] | | Sorghum | 30 | |
| | | | | 29 | |
| Maize | 340 + 220[(a)] | | Wheat | 100 | |
| | | | | 98 | |

(a) 0.34 kg as/ha incorporated into soil before the maize crop was planted, and the 0.22 kg a.s./ha applied post-emergent to the maize. The maize crop was removed prior to the planting of the succession crops.

**Conclusion on rotational crops studies**

Field rotational crop studies are not triggered considering the very low TRRs in rotational crops after a bare soil application at ca. 1N rate (see Table 7.3-4) and considering also the low to moderate persistence of mesotrione, MNBA and AMBA.

US rotational crop field trials were conducted on pulses/oilseeds (soya bean), leafy vegetables (endive), root vegetables (radish) and cereals (small grains (wheat)) after bare soil application at 0.34 kg a.s./ha or after bare soil application (0.34 kg a.s./ha ) followed by a post-emergence application (0.22 kg a.s./ha). Residues of mesotrione and MNBA were < 0.01 mg/kg in all crop parts (EFSA, 2016).

**zRMS comments:**

In the EFSA Journal 2015;13(1):3976 it is stated that rotational crop field trials conducted in North America were evaluated in the framework of the peer review (United Kingdom, 1999). Mesotrione was applied either on bare soil, incorporated prior to maize planting at a dose of 0.34 kg a.s./ha (1 N) or on bare soil with incorporation followed by a post-emergence application on maize at doses of 0.34 and 0.22 kg a.s./ha, respectively. The magnitude of residues was investigated on several succeeding crops sown at different plant back intervals following application of the active substance. At harvest, levels of mesotrione and metabolite MNBA in all mature plant parts were below the LOQ of 0.01 mg/kg. These studies were considered as sufficient by EFSA to demonstrate the absence of residues in rotational crops, provided that mesotrione is applied in compliance with the GAPs.

The crops under consideration can be grown in rotation.

No waiting periods beyond normal agricultural practice are proposed for succeeding crops to be planted.

No additional data are required.

### 7.3.7 Other / special studies (KCA6.10, 6.10.1)

The available data for the active substance sufficiently address aspects of the residue situation that might arise from the use of A18032E. Therefore, other special studies are not needed.
*Reference: United Kingdom, 2015, 2015a*
"There is currently no EU agreed guidance for residue levels in pollen and bee products. Therefore these data are not currently required.
The data requirement objective of these studies is to determine the residue in pollen and bee products for human consumption resulting from residues taken up by honeybees from crops at blossom.
Mesotrione is not known to have any toxic effects to bees and maize is not a flowering crop frequented by bees therefore data from field studies conducted on bees are not available. Furthermore, for the supported use of mesotrione on maize, applied at early growth stages (typically BBCH 02 to 18), there is no likelihood of mesotrione exposure to honey bees and residues of mesotrione in pure blossom honey or other bee products will not occur from this use."
*Reference: EFSA, 2016*
"A data gap was also identified for the determination of the residues in pollen and bee products for human consumption. It is noted that RMS disagreed with the setting of this data gap."

**zRMS comments:**

Information given by the Applicant is sufficient.
No further data are required to support the proposed uses.

### 7.3.8 Estimation of exposure through diet and other means (KCA 6.9)

Toxicological reference values relevant for dietary risk assessment are reported in the summary of the evaluation (see Table 7.1-2).

#### 7.3.8.1 Input values for the consumer risk assessment

**Table 7.3-12: Input values for the consumer risk assessment**

| Commodity | Chronic risk assessment | | Acute risk assessment | |
|---|---|---|---|---|
| | Input value (mg/kg) | Comment | Input value (mg/kg) | Comment |
| Risk assessment residue definition:<br>Food commodities: Mesotrione (cereals and pulses/oilseeds only).<br>Feed commodities: Mesotrione and AMBA (including its conjugates) (Cereals, pulses and oilseeds only – Conventional crops) – Provisional. | | | | |
| Intended/relevant uses | | | | |
| Maize / corn | 0.01 | EU MRL* | 0.01 | EU MRL* |
| Further uses | | | | |
| Other commodities of plant or animal origin | variable | EU MRL* | Acute risk assessment was undertaken only for the crops under consideration. | |

* Regulation (EU) 2017/626 of 31 March 2017

#### 7.3.8.2 Conclusion on consumer risk assessment

Extensive calculation sheets are presented in 0.

**Table 7.3-13: Consumer risk assessment**

| | |
|---|---|
| ADI | 0.01 mg/kg bw/d |
| TMDI (% ADI) according to EFSA PRIMo rev. 3.1 | 12% (based on NL toddler diet) |

| | |
|---|---|
| IEDI (% ADI) according to EFSA PRIMo rev. 3.1 | Not relevant. TMDI < 100%. |
| ARfD | 0.02 mg/kg bw/d |
| IESTI (% ARfD) according to EFSA PRIMo rev. 3.1* | Unprocessed commodities:<br>Maize / corn: 0.3% (based on UK infant diet)<br><br>Processed commodities:<br>Maize / oil: 1% (based on NL toddler diet)<br>Maize / processed (not specified): 0.1% (based on NL toddler diet) |
| NTMDI (% ADI) ** | Not relevant. |
| NEDI (% ADI)** | Not relevant. |
| NESTI (% ARfD) ** | Not relevant. |

* include raw and processed commodities if both values are required for PRIMo
** if national model is available

Chronic and acute exposure calculations were performed using revision 3.1 of the EFSA Pesticide Residues Intake Model (PRIMo rev. 3.1) provided on the internet homepage of EFSA (https://www.efsa.europa.eu/). This exposure assessment model contains the relevant European food consumption data for different subgroups of the EU population. The model was developed to calculate simultaneously the short-term (acute) and long-term (chronic) dietary exposure to pesticide residue in food according to internationally agreed methodologies. The exposure is compared to the toxicological reference values (i.e. the ADI and the ARfD).

- The potential chronic dietary exposure was compared to the ADI of 0.01 mg/kg bw/d and TMDI values were achieved. Input values for all commodities were derived from existing EU MRL (Reg. (EU) 2017/626), representing a worst case scenario. The highest chronic exposure was calculated for NL toddler diet, representing 12% of the ADI. For this diet the highest contributors were milk: cattle (6% of ADI), apples (1% of ADI) and maize / corn (0.7% of ADI). Since TMDI values are below 100%, there is no need to perform higher tier/refined chronic exposure calculation.
- The potential acute dietary exposure was compared to the ARfD of 0.02 mg/kg bw/d and IESTI values were achieved. Input values were put from existing EU MRL (Reg. (EU) 2017/626) representing a worst case scenario, only for intended/relevant uses. With regard to the acute exposure there is no exceedance of the ARfD for children and adults in unprocessed and processed commodities. The maximum IESTI calculated for unprocessed commodity: maize / corn was 0.3% of ARfD (UK infant diet) and for processed commodity: maize / oil: 1% of ARfD (NL toddler diet), maize / processed (not specified): 0.1% (NL toddler diet). Since IESTI values are below 100%, there is no need to perform higher tier/refined acute exposure calculation.

The proposed uses of mesotrione in the formulation NIKITA (A18032E) does not represent unacceptable chronic and acute risks for the consumer.

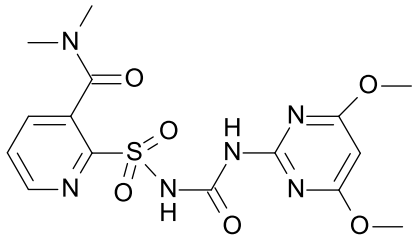
**zRMS comment:**

Information given by the Applicant is sufficient.
The calculation of the TMDI using EFSA model (PRIM0 ver. 3.1) and MRLs according to Regulation (EU) 2017/626 of 31 March 2017 led to a utilisation of the ADI of 12% for the NL toddler diet being the population group with the highest value. For this diet, the highest contributor is milk:Cattle with 6% of the ADI. The intended uses will not result in a consumer chronic exposure exceeding the ADI.
An acute consumer risk assessment was performed with MRL for mazie only. The highest International Estimated Short-Term Intake (IESTI) is at 0.3% and 0.1% of the ARfD for the consumption of mazie/corn by children and by adults respectively.
The proposed use of mesotrione in the product NIKITA (A18032E) do not represent unacceptable acute and chronic risks for the consumer.
No further data are required to support the proposed use.

## 7.4 Nicosulfuron

General data on nicosulfuron are summarized in the table below (last updated 2017/11/21).

**Table 7.4-1: General information on nicosulfuron**

| | |
|---|---|
| Active substance (ISO Common Name) | Nicosulfuron |
| IUPAC | 2-[(4,6-dimethoxypyrimidin-2-ylcarbamoyl)sulfamoyl]-*N*,*N*-dimethylnicotinamide<br>or<br>1-(4,6-dimethoxypyrimidin-2-yl)-3-(3-dimethylcarbamoyl-2-pyridylsulfonyl)urea |
| Chemical structure |  |
| Molecular formula | $C_{15}H_{18}N_6O_6S$ |
| Molar mass | 410.4 g/mol |
| Chemical group | Sulfonylurea herbicide |
| Mode of action (if available) | Acetolactate synthase (ALS) inhibition |
| Systemic | Yes |
| Company (ies) | ISK Biosciences Europe S.A. |
| Rapporteur Member State (RMS) | Current RMS: Latvia<br>Original RMS: UK |
| Approval status | Approved<br>01/01/2009<br>Commission Directive 2008/40/EC. |
| Restriction | Restricted to uses as a herbicide |
| Review Report | SANCO/3780/07 – Rev 1<br>22/01/2008 |
| Current MRL regulation | Regulation (EC) No 617/2014 |
| Peer review of MRLs according to Article 12 of Reg No 396/2005 EC performed | Yes (EFSA, 2012) |
| EFSA Journal : Conclusion on the peer review | Yes (EFSA, 2007) |
| Current MRL applications on intended uses | None |

### 7.4.1 Stability of Residues (KCA 6.1)

#### 7.4.1.1 Stability of residues during storage of samples

**Available data**

New data submitted in the framework of this application, which have previously been submitted as part of the AIR process for nicosulfuron.

**Table 7.4-2: Summary of stability data achieved at ≤ - 18°C (unless stated otherwise)**

| Commodity category | Commodity | Acceptable maximum storage period | Report Reference | Source |
|---|---|---|---|---|
| **EU reviewed data** | | | | |

| Commodity category | Commodity | Acceptable maximum storage period | Report Reference | Source |
|---|---|---|---|---|
| **Plant products** | | | | |
| High Water Content | Maize, whole plant | 9 months[(a)] | Schulz & Ullrich-Mitzel, 1995<br>Report No: 4 | United Kingdom, 2006 |
| High Starch Content | Maize, grain | 9 months[(a)] | | |
| **New data** | | | | |
| **Plant products** | | | | |
| High Water Content | Maize, forage | 12 months | Peatman, 2007<br>Report No: 0676/032-D2149 (NIS-42) | Nicosulfuron Task Force (AIR) |
| High Starch Content | Maize, grain,<br>Maize, stover<br>Maize, sweetcorn | 12 months | | |

(a): Storage stability was tested for a period of 30 months; the RMS concluded that the results demonstrated that nicosulfuron residues are stable (recovery >70%) for 9 months when stored at -20°C in the dark.

**For corn treated with Conclusion on stability of residues during storage**

The storage stability of nicosulfuron has been investigated in high water and high starch commodities. Sufficient stability has been demonstrated to support the residue data presented in the submission.

**zRMS comments:**

Storage stability studies for nicosulfuron was evaluated and accepted at EU level. Storage stability of nicosulfuron was demonstrated for a period of 9 months at -20°C in dry/high starch commodities (maize grain) and in high water content commodities (maize whole plant).
New study for storage stability studies for nicosulfuron have been submitted by the applicant in the framework of this application. Stability of nicosulfuron residues in sweet corn, dried kernels, stover and forage has been demonstrated for storage intervals of up to twelve months under frozen (nominally -20°C) conditions.

A residue definition for animal products has not been proposed, since the uses of nicosulfuron will not lead to significant residues in any edible animal tissue or milk. Thus, no stability data in commodities of animal origin are required.

The studies on the magnitude of residues are valid with regard to storage stability.
No additional data are required.

### 7.4.1.2 Stability of residues in sample extracts (KCA 6.1)

**Available data**

During validation of method GRM074.01A (Andrews, 2016, see KCP 5.1.2), the stability of sample extracts was tested. Extracts fortified with nicosulfuron at the LOQ level was checked after a storage period of 8 to 9 days in a refrigerator at 3-8 ºC against freshly prepared calibration standards. The results proved that the nicosulfuron residues in the stored fortified samples were stable.

**Conclusion on stability of residues in sample extracts**

Procedural recoveries obtained during residue analysis demonstrate the stability of residues of nicosulfuron in sample extracts and fully support the residue data presented in the submission.

**zRMS comments:**

Information submitted by Applicant is sufficient and accepted.
No further data are required.

## 7.4.2 Nature of residues in plants, livestock and processed commodities

### 7.4.2.1 Nature of residue in primary crops (KCA 6.2.1)

**Available data**

New data are submitted in the framework of this application, which have previously been submitted as part of the AIR process for nicosulfuron.

**Table 7.4-3: Summary of plant metabolism studies**

| Crop Group | Crop | Label position | Application and sampling details | | | | Report Reference | Source |
|---|---|---|---|---|---|---|---|---|
| | | | Method, F or G[(a)] | Rate (g a.s./ha) | No | Sampling (DAT) | | |
| **EU reviewed data** | | | | | | | | |
| **Cereals** | Maize | 5-$^{14}$C-pyrimidinyl | Foliar, G | 60, 300 | 1 | Silage: 0, 14, 30, 60 Harvest: 102 | Schanné, 1991 Report No: 274173 | United Kingdom, 2006 |
| | | 2-$^{14}$C-pyridyl | Foliar, G | 60, 300 | 1 | Silage: 0, 14, 30, 60 Harvest: 102 | Mamouni, 1995 Report No: 272158 | United Kingdom, 2006 |
| **New data** | | | | | | | | |
| **Cereals** | Maize | 5-$^{14}$C-pyrimidinyl | Foliar, G | 70 | 1 | Silage: 0, 7, 14, 30, 49 Harvest: 113 | Scott, 1989 Report No. AMR 861-87 | Nicosulfuron Task Force (AIR) |
| | | 2-$^{14}$C-pyridyl | Foliar, G | 70 | 1 | Silage: 0, 7, 14, 30, 49 Harvest: 113 | | |

(a): Outdoor/field application (F) or glasshouse/protected/indoor application (G)

**Summary of plant metabolism studies reported in the EU**

*Reference: EFSA, 2012*

"In maize, 102 days after application, the highest TRR was identified in straw (0.082-0.13 mg eq/kg), whereas in grain the TRR was low (0.001-0.003 mg eq/kg) for pyridyl and pyrimidinyl labels respectively. In both studies, nicosulfuron was the predominant residue for all harvest times, accounting for 29% (0.03 mg eq/kg) and 54% TRR (0.06 mg eq/kg) 102 days after treatment for pyridyl and pyrimidinyl labels respectively [… *in the silage samples*].

Immediately after application, in the pyrimidinyl study, a considerable metabolisation had already occurred, metabolites ranging from 4% TRR (0.11 mg/kg) for HMUD[1] to 27.7% TRR (0.79 mg/kg) for M2 [*AUSN*[2]]. At day 60, the metabolite profile had changed considerably. Among the four metabolites identified (up to 12.6% TRR; 0.007 mg/kg for M1 [*unknown metabolite*]), two were not present initially (DMPU[3] 5.9% TRR and ADMP[4] 5.5% TRR). At the 102 day harvest point, the residue profile was very similar to the 60 day harvest; however some slight increases in parent levels were noted which is deemed a result of a decrease in water content (54% TRR, 0.055 mg/kg). In the pyridyl labelled study at day 0, six metabolite fractions were characterised and three were identified as AUSN (20.4% TRR; 0.32 mg eq/kg), HMUD (3.6% TRR; 0.056 mg eq/kg) and ASDM[5] (17.3% TRR; 0.27 mg eq/kg). AUSN and ASDM were not identified in the pyrimidinyl study since cleavage of the ring structures has occurred. At day 60, the main identified metabolites were AUSN at 13.5 % TRR (0.008 mg eq/kg) and ASDM at 16.7% TRR (0.01 mg eq/kg.). At the 102 day interval,

[1] HMUD = 2-{[(4-hydroxy-6-methoxypyrimidin-2-yl)carbamoyl]sulfamoyl}-*N*,*N*-dimethylpyridine-3-carboxamide
[2] AUSN = 2-[(carbamimidoylcarbamoyl)sulfamoyl]-N,N-dimethylpyridine-3-carboxamide
[3] DMPU = (4,6-dimethoxypyrimidin-2-yl) urea
[4] ADMP = 4,6-dimethoxypyrimidin-2-amine
[5] ASDM = *N*,*N*-dimethyl-2-sulfamoylpyridine-3-carboxamide

the M1 metabolite fraction had increased from 0.1% TRR to 29% TRR. In spite of further work [~~*was*~~] undertaken to clarify how metabolite M1 was formed, the reason [*for its formation*] was still unclear. However, M1 was shown to be a fraction of metabolites (partially conjugates of parent and ASDM) rather than one single metabolite and residues were generally low. Of the metabolites identified, none of them are considered to be toxicologically significant as they are found in the rat metabolism or are conjugates of rat metabolites."

**New data**

For corn treated with [pyridine-2-$^{14}$C] nicosulfuron, the concentration of parent in each RAC was less than 0.010 mg/kg. ASDM (referred to as pyridine sulphonamide in the report) was the major metabolite in the RACs comprising 0.021 mg/kg (metabolite equivalents). Individual metabolite concentrations in the 30-day and 49-day samples were each ≤0.005 mg/kg (nicosulfuron equivalents). Total radioactive residues in grain and cob were 0.002 and 0.004 mg/kg, nicosulfuron equivalents, respectively, and were therefore not analysed further.

For corn treated with [pyrimidine-2-$^{14}$C] nicosulfuron, the concentration of nicosulfuron was <0.01 mg/kg in all RACs except fodder (Day 113, mature sample) which contained 0.021 mg/kg. Total radioactive residues in grain were 0.003 mg/kg parent equivalents. Polar metabolites were the only other radiochemical peak comprising 0.012 mg/kg (nicosulfuron equivalents).

For samples harvested before 30 days after application, the metabolites 5-GDUD and 5-HDUD (referred to as 5'-O-Glucoside and 5'-hydroxy DPX-V9360 in the report), were identified. However these metabolites were not present in any raw agricultural commodity.

Nicosulfuron was metabolised primarily by two pathways: (1) hydrolysis of the sulfonylurea bridge resulting in formation ASDM and ADMP, which undergoes rapid metabolism as it was not a significant metabolite in any extract; and (2) hydroxylation of the 5'-position of the pyrimidine ring of intact sulfonylurea followed by glucosidation.

**Conclusion on metabolism in primary crops**

The metabolism of nicosulfuron in plants following foliar application is sufficiently addressed to support the proposed uses of the product A18032E.

**zRMS comments:**

Information submitted by Applicant is sufficient and accepted.

Metabolism of nicosulfuron was investigated for foliar application on maize, using 5-$^{14}$C-pyrimidinyl and 2-$^{14}$C-pyridyl labelled nicosulfuron (United Kingdom, 2005). Based on these studies, the relevant residue for enforcement and risk assessment in cereals is defined as nicosulfuron only.

New data are submitted in the framework of this application, which have previously been submitted as part of the AIR process for nicosulfuron. More details are presented in Appendix 2.

In our opinion the evaluation of metabolism study of nicosulfuron in plants and the setting residue definition should be carried out at the EU level and the active substance level, not at the level of plant protection product registration in the Central Zone.

The metabolic pathway in plant is sufficiently addressed and no additional metabolism studies are necessary to support the intended uses for ADM.4651.H.1.A WG (A18032E) / Nikita.

**Definition:**

According to the EFSA Journal 2012;10(12):3048:

Plant residue definition for monitoring and risk assessment: nicosulfuron.

It is noted that the current enforcement residue definition established in Regulation (EC) No 396/2005 comprises only the parent compound nicosulfuron.

No further data are required to support the proposed use.

### 7.4.2.2 Nature of residue in rotational crops (KCA 6.6.1)

**Available data**

No studies on residues in rotational crops were submitted for Annex I inclusion of nicosulfuron. Based on the soil $DT_{50}$ of nicosulfuron and the phytotoxic effects of nicosulfuron which limits the re-planting period, it was concluded that no significant residues would be expected in rotational crops and no further data would be necessary in the EFSA conclusion for nicosulfuron (2007). However in the EFSA MRL review of nicosulfuron (2012) further investigation of residues in rotational crops was considered relevant as maize and sweet corn may be grown in rotation.

Under field conditions $DT_{90}$ values for nicosulfuron ranged from between 23.75 to 204.6 days and the soil metabolites ASDM, ADMP and AUSN have $DT_{90}$ values higher than 365 days. Thus soil residues may be available crop uptake by succeeding crops. However the phytotoxic effect of nicosulfuron leads to limitations in the re-planting period.

A new crop rotation study in succeeding crops, not yet reviewed under Uniform Principles, is available. This study has been submitted as part of the AIR process for Nicosulfuron. The studies are summarised in detail in Appendix 2.

**Table 7.4-4: Summary of metabolism studies in rotational crops**

| Crop group | Crop | Label position | Application and sampling details | | | | Report reference | Source |
|---|---|---|---|---|---|---|---|---|
| | | | Method, F or G[(a)] | Rate (g a.s./ha) | Sowing intervals (DAT) | Harvest Intervals (DAT) | | |
| **New data** | | | | | | | | |
| **Leafy vegetables** | Lettuce | 5-$^{14}$C-pyrimidinyl<br>2-$^{14}$C-pyridyl | Soil application, G | 70 | 120, 305 | Maturity (tops) | Dietrickh, 1989 Report no. AMR 874-87<br><br>Scott, 2004 Report No. AMR 874-87, Supplement No. 1 | Nicosulfuron Task Force (AIR) |
| **Root and tuber vegetables** | Radish | 5-$^{14}$C-pyrimidinyl<br>2-$^{14}$C-pyridyl | Soil application, G | 70 | 305 | Maturity (leaves, roots) | | |
| **Cereals** | Wheat | 5-$^{14}$C-pyrimidinyl<br>2-$^{14}$C-pyridyl | Soil application, G | 70 | 120, 305 | Maturity (grain, straw) | | |
| **Pulses** | Soybean | 5-$^{14}$C-pyrimidinyl<br>2-$^{14}$C-pyridyl | Soil application, G | 70 | 30, 120, 305 | Maturity (grain) | | |

(a): Outdoor/field application (F) or glasshouse/protected/indoor application (G)

**Summary of new metabolism study in rotational crops not yet evaluated in the EU**

Data from the confined greenhouse study indicated that nicosulfuron does not accumulate in rotational crops grown in soil treated with Nicosulfuron 75WG applied at a rate of 70 g a.s./ha. Nicosulfuron concentrations were very low and decreased with soil aging time. Concentrations in wheat grain and straw/chaff from a 120-day soil aging period were 0.002-0.007 mg/kg and were ≤0.002 mg/kg in all other crop fractions.

ASDM, a major soil degradate of nicosulfuron, was found in most extracts of plants grown in soil treated with [pyridine-2-$^{14}$C] nicosulfuron. Soybean forage/hay from the 30-day and 120-day soil aging periods contained 0.080 and 0.071 mg/kg. Mature wheat straw contained 0.056 and 0.038 mg/kg from plants grown in soil aged 120-days and 10-months, respectively. In all other crops, the concentration of ASDM was ≤0.023 mg/kg. ASDM has no biological activity and EFSA have concluded the metabolite to be non-toxicologically relevant. It is not included in the crop residue definition for nicosulfuron.

Other compounds were identified at low concentrations in plant extracts. MU-467 was detected in soybean

forage/hay from 30-day and 120-day aging periods at a concentration of 0.024 and 0.020 mg/kg, respectively (mean values) but declined to 0.001-0.002 mg/kg from the 10-month aging period. An alanine conjugate of ASDM and a malonyl-alanine conjugate of ASDM were also identified in soybeans. Only the malonyl-alanine conjugate's concentration from the 30-day aged soil was >0.05 mg/kg (0.06 mg/kg). The concentrations of both conjugates from the 120-day soil aging periods were <0.04 mg/kg and reached 0.01 mg/kg in soybeans grown in soil aged 10 months.

**Conclusion on metabolism in rotational crops**
A specific residue definition for rotational crops is not deemed necessary due to the very low residue levels expected.

**zRMS comments:**

Information submitted by Applicant is sufficient and accepted.
In EFSA Journal 2012;10(12):3048 1 it is stated that *"Maize and sweet corn may be grown in rotation and according to the soil degradation from field studies evaluated in the framework of the peer review, $DT_{90}$ values of nicosulfuron range between 30 and 210 days, and relevant soil metabolites ASDM, AUSN and UCSN have $DT_{90}$s higher than 365 days exceeding the trigger value of 100 days (EFSA, 2007b). According to the European guidelines on rotational crops (EC, 1997b), further investigation of residues in rotational crops is relevant.*
*In spite of high persistence however, lysimeter studies where crops of wheat and rye were grown following maize treated at the proposed application rate of 60 g/ha, indicated low uptake of parent and metabolites by the following cereal plants (TRR <0.01 mg/kg). Moreover, it was shown that the phytotoxic effect of nicosulfuron and its soil metabolites on dicotyledone plants leads to a self-limitation in the re-planting period. After a plant back interval of 27 to 30 days and with the exception of maize, marked phytotoxic effects were observed in the following crops (cereals, sugar beet, maize, oilseed rape and clover) while residues of nicosulfuron and its soil metabolites were found to be below the LOQ (0.01 mg/kg). Thus the following crops could not be grown until the following spring at which time residues in soil of nicosulfuron and relevant metabolites have decreased to <0.001 mg/kg. Furthermore, ASDM and AUSN are also found in maize but are non toxicological relevant. As UCSN metabolite has a similar toxicological profile as AUSN, it is considered non relevant as well (EFSA, 2007b).*

*Consequently, these studies are considered sufficient by EFSA to demonstrate the absence of residues in rotational crops, provided that nicosulfuron is applied in compliance with the GAPs reported in Appendix A."*

New data are submitted in the framework of this application, which have previously been submitted as part of the AIR process for nicosulfuron. More details are presented in Appendix 2.
In our opinion the evaluation of metabolism study of nicosulfuron in rotational crops and the setting residue definition should be carried out at the EU level and the active substance level, not at the level of plant protection product registration in the Central Zone.

No further data are required to support the proposed use for ADM.4651.H.1.A WG (A18032E) / Nikita.

### 7.4.2.3 Nature of residues in processed commodities (KCA 6.5.1)

No data on processing are required as residues are below the LOQ.
*Reference*: *EFSA, 2012*
"As quantifiable residues of nicosulfuron are not expected in the treated crops and the chronic exposure does not exceed 10% of the ADI, there is no need to investigate the effect of industrial and/or household processing."

**zRMS comments:**

Information given by the Applicant is sufficient.
No data are required to support the proposed use.

### 7.4.2.4 Conclusion on the nature of residues in commodities of plant origin (KCA 6.7.1)

**Table 7.4-5: Summary of the nature of residues in commodities of plant origin**

| **Endpoints** | |
|---|---|
| Plant groups covered | Cereals (Maize) |
| Rotational crops covered | Lettuce, radish, wheat, soybean |
| Metabolism in rotational crops similar to metabolism in primary crops? | Yes |
| Processed commodities | No data required |
| Residue pattern in processed commodities similar to pattern in raw commodities? | Not applicable |
| Plant residue definition for monitoring | Nicosulfuron (Regulation (EC) No 617/2014) |
| Plant residue definition for risk assessment | Nicosulfuron (EFSA 2007) |
| Conversion factor from enforcement to RA | None (EFSA, 2007) |

### 7.4.2.5 Nature of residues in livestock (KCA 6.2.2-6.2.5)

**Available data**

New data are submitted in the framework of this application, which have previously been submitted as part of the AIR process for nicosulfuron.

**Table 7.4-6: Summary of animal metabolism studies**

| **Group** | **Species** | **Label position** | **No of animal** | **Application details** | | **Sample details** | | **Report reference** | **Reference** |
|---|---|---|---|---|---|---|---|---|---|
| | | | | **Rate (mg/kg bw/d)** | **Duration (days)** | **Commodity** | **Time of sampling** | | |
| **EU reviewed data** | | | | | | | | | |
| **Lactating ruminants** | Goat | $^{14}$C-pyridyl | 1 | 8.3 | 3 | Milk | Twice daily | Burri, 1995a, Report No: 358323 | United Kingdom, 2006 |
| | | | | | | Urine & faeces | Daily | | |
| | | | | | | Tissues | After sacrifice | | |
| | | $^{14}$C-pyrimi-dinyl | 1 | 8.6 | 3 | Milk | Twice daily | Burri, 1995b, Report No: 358312 | |
| | | | | | | Urine & faeces | Daily | | |
| | | | | | | Tissues | After sacrifice | | |
| | | $^{14}$C-pyrimi-dinyl | 1 | 0.007 | 3 | Milk | Twice daily | Burri, 1995c, Report No: 367356 | |
| | | | | | | Urine & faeces | Daily | | |
| | | | | | | Tissues | After sacrifice | | |
| **New data** | | | | | | | | | |
| **Lactating ruminants** | Goat | $^{14}$C-pyridyl | 1 | 1.84 | 3 | Milk | Twice daily | Lin & Scott, 1989 Report No: AMR 947-87 | Nicosulfuron Task Force (AIR) |
| | | | | | | Urine & faeces | Daily | | |
| | | | | | | Tissues | After sacrifice | | |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | $^{14}$C-pyrimi-dinyl | 1 | 1.44 | 3 | Milk | Twice daily | | |
| | | | | | | Urine & faeces | Daily | | |
| | | | | | | Tissues | After sacrifice | | |

**Summary of animal metabolism studies reported in the EU**

*Reference: EFSA, 2012*

"Lactating goats were dosed with nicosulfuron ranging from 0.007 to 8.6 mg/kg bw per d, corresponding to approximately 2 to 2700 times the exposure of meat ruminant. Studies demonstrate that the majority of radioactivity was rapidly excreted (from 69 to 91% AR). In the highest dosed studies, parent nicosulfuron was the most abundant component found in tissues for both radiolabels with up to 66% TRR (0.62 mg eq/kg) in kidney. Two major metabolites were detected in tissues: ASDM with up to 18% TRR (0.11 mg eq/kg) in kidney and ADMP with up to 16% TRR (0.11 mg eq/kg) in liver. In addition the pyridine ring specific compounds SNA and ODPD were found in significant levels in liver for the pyridine labelled study (24 and 13% TRR; 0.09 and 0.05 mg eq/kg respectively). For milk, nicosulfuron and ADMP were found in equal amounts in the pyridil labelled study (30% TRR; 0.05 mg eq/kg). In the pyrimidinyl labelled study, Mi 12 was the most abundant compound found at levels twice those of nicosulfuron. In the lowest dose level study (2N), no significant residues were detected in tissues and milk (<0.001 mg/kg). ASDM and ADMP are toxicological irrelevant metabolites whereas no information on the toxicity of metabolites SNA, ODPD and Mi12 was available. The toxicity of these metabolites found at significant levels is not known but any requirement for further investigation of these compounds is currently not triggered. It is therefore not possible to propose residue definition in animal products at this time (United Kingdom, 2007)".

**Summary of new animal metabolism studies**

Approximately 49.1 and 80.2 % of the dose from the goat dosed with pyrimidine label and pyridine label was recovered, with the remainder assumed to be still present in the bladder and digestive tract at sacrifice. Excretion of the test item proceeded mainly via faeces (31.8 % and 64.2% of dose, respectively). In urine, excreted radioactivity accounted for 17.0% and 15.3% of the dose. Small amounts 0.14% and 0.69% of the dose were detected in the bile. A negligible amount was excreted in milk (0.07% dose in the pyrimidine label and below detection limit in pyridine label) and 0.03-0.04% of dose was detected in tissues.

Nicosulfuron residues did not accumulate in meat and milk. Total radioactive residues (TRR) in liver, kidney, fat and muscle were each less than 0.100 mg/kg nicosulfuron equivalents. TRR in milk from the goat dosed with pyridine-labelled nicosulfuron were not quantifiable (<twice background) for all samples. TRR in milk samples from the goat dosed with pyrimidine-labelled nicosulfuron were 0.008 to 0.032 mg/kg nicosulfuron equivalents and represented 0.07% of the total administered dose. Characterisation of residues in milk samples was not conducted due to the low concentration of $^{14}$C-residues. Residues in urine, faeces, bile and liver were primarily nicosulfuron. Residue concentrations in kidney, muscle and fat were too low to allow further characterisation.

Metabolites identified in urine from the goat dosed with pyridine-labelled nicosulfuron included ASDM, MU-467, DDTP, MU-466 and MU-2275. The major urinary metabolite from the goat dosed with pyrimidine-labelled nicosulfuron was a polar conjugate of AHDP.

It was concluded that nicosulfuron and its metabolites were neither accumulated nor retained in the lactating goat after repeated oral administration at a highly exaggerated dose.

**Conclusion on metabolism in livestock**

The metabolism of nicosulfuron in livestock is sufficiently addressed to support the proposed uses of the product A18032E.

**zRMS comments:**

Information submitted by Applicant is sufficient and accepted. zRMS agrees, the metabolic pathway is sufficiently addressed.

In EFSA Journal 2012;10(12):3048 1 it is stated that *"*

*"In spite of animal intakes of nicosulfuron not being significant, the nature of nicosulfuron residues in commodities of animal origin was investigated in the framework of Directive 91/414/EEC (United Kingdom, 2005). Reported metabolism*

*studies include three studies in lactating goats using $^{14}$C-pyridil and $^{14}$C-pyrimidinyl labelled nicosulfuron.*
*(…)no significant exposure of livestock to nicosulfuron residues is expected. Some data is available for future reference, however at this time it is not possible to propose residue definitions in animal products. However, based on the authorised uses on maize grain and forage, exposure of livestock is not expected to be significant and MRLs for products of animal origin are currently not required."*

It should be noted that based on the authorised uses, residues in animal products are not expected to be significant (animal dietary intakes are < 0.004 mg/kg bw <) and MRLs for products of animal origin are currently not required.

New data are submitted in the framework of this application, which have previously been submitted as part of the AIR process for nicosulfuron. More details are presented in Appendix 2.
In our opinion the evaluation of metabolism study of nicosulfuron in livestock and the setting residue definition should be carried out at the EU level and the active substance level, not at the level of plant protection product registration in the Central Zone.

No further data are required to support the proposed use for ADM.4651.H.1.A WG (A18032E) / Nikita.

### 7.4.2.6 Conclusion on the nature of residues in commodities of animal origin (KCA 6.7.1)

**Table 7.4-7: Summary on the nature of residues in commodities of animal origin**

| **Endpoints** | |
|---|---|
| Animals covered | Ruminants |
| Time needed to reach a plateau concentration | Unable to assess due to low total radioactive residues |
| Animal residue definition for monitoring | Unable to propose, however intakes are not significant (<0.1 mg/kg diet). (EFSA, 2007) |
| Animal residue definition for risk assessment | Unable to propose, however intakes are not significant (<0.1 mg/kg diet). (EFSA, 2007, EFSA 2012) |
| Conversion factor | None (EFSA, 2007, EFSA 2012) |
| Metabolism in rat and ruminant similar | Yes |
| Fat soluble residue | No |

## 7.4.3 Magnitude of residues in plants

### 7.4.3.1 Summary of European data and new data supporting the intended uses

New studies on the magnitude of residue have been submitted by the applicant in the framework of this application. These studies are summarized in the Table below. The detailed assessment of these studies is presented in Appendix 2.

**Table 7.4-8: Summary of EU reported and new data supporting the intended uses of A18032E and conformity to existing MRL**

| Commodity | Source | Residue zone (N-EU, S-EU, EU, outside EU) | Evaluation<br>GAP<br>Residue levels (mg/kg)[a] | STMR (mg/kg) | HR (mg/kg) | Unrounded OECD calculator MRL (mg/kg) | Curent EU MRL (mg/kg)* | MRL compliance |
|---|---|---|---|---|---|---|---|---|
| **Maize grain** | EFSA, 2012 | NEU | GAP: 1x 0.06 kg as/ha, BBCH 12-20, PHI N/A[b]<br>18x <0.01 | <0.01 | <0.01 | 0.01* | 0.01* (tentative)[c] | Yes |
| | New trials | | Trials GAP: 1x 0.065 kg as/ha, BBCH 16-18, PHI N/A[b]<br>4x <0.01 | <0.01 | <0.01 | 0.01* | | Yes |
| | EFSA, 2012 | SEU | GAP: 1x 0.06 kg as/ha, BBCH 12-20, PHI N/A[b]<br>15x <0.01 | <0.01 | <0.01 | 0.01* | 0.01* (tentative)[c] | Yes |
| | New trials | | Trials GAP: 1x 0.065 kg as/ha, BBCH 16-18, PHI N/A[b]<br>4x <0.01 | <0.01 | <0.01 | 0.01* | | Yes |
| | Overall supporting data | NEU | 22x <0.01 | <0.01 | <0.01 | 0.01* | 0.01* (tentative)[c] | Yes |
| | | SEU | 19x <0.01 | <0.01 | <0.01 | 0.01* | 0.01* (tentative)[c] | Yes |

* Source of EU MRL: Regulation. (EU) No 617/2014
N/A – not applicable
(a) Definition of residue for enforcement and risk assessment are the same: nicosulfuron
(b) PHI is determined by crop maturity
(c) Due to the lack of confirmatory analytical data for enforcement in dry commodities, only a tentative MRL was adopted
(d) Residue definition for enforcement is not relevant for animal feed items since no MRL is set.

### 7.4.3.2 Conclusion on the magnitude of residues in plants

Maize is a major crop in all regions of Europe (**SANCO 7525/VI/95 rev.10**); sufficient trials are available to support the proposed use.
According to the available data, the intended uses on maize are considered acceptable, for outdoor uses.
The data submitted show that no detectable residues are expected in treated crops and no exceedance of the MRL will occur. The proposed uses are considered acceptable.

**zRMS comments:**

Maize is the major crop in northern Europe (EU guideline Document SANCO 7525/VI/95 - rev.10.3 of 13 June 2017 and SANTE/2019/12752). A minimum of eight independent trials representative of the proposed growing area for outdoor are required.

**Table 7.4: Summary of EU critical GAP and intended GAP in maize for niclosulfuron (EFSA, 2012).**

| Type of GAP | Method | Number of applications | Application rate per treatment (kg as/ha) | Interval between application (days) | Growth stage at last application | PHI (days) |
|---|---|---|---|---|---|---|
| cGAP EU (EFSA, 2012) – maize | Foliar tratment - spraying | 1 | 0.06 | - | BBCH 12-20 | N/A |
| Intended GAP-maize | Foliar, spraying, overall | 1 | 0.04 | - | BBCH 12 – 14 | - |

N/A – not applicable

The active substance nicosulfuron is authorised for foliar outdoor applications on maize (grain and forage) in northern and southern Europe and on sweet corn in northern Europe. The critical EU GAP for maize is most critical than proposed GAP.
The trials conducted on maize (18 trials N-EU, 1 x 0.06 kg a.s./ha, at BBCH 12-20, no PHI is applicable, outdoor) have been evaluated at the EU level (EFSA Journal 2012;10(12):3048). No PHI is proposed in the critical EU GAP since the application is made early post-emergence and the maize grain is harvested at maturity.
No residues were detected in maize grains, with all values being below the LOQs of 0.01.
In EFSA Journal 2012;10(12):3048 it is stated that "Consequently, the available residues data are considered sufficient to derive MRL proposals as well as risk assessment values for maize and sweet corn (see also Table 3-2). Considering the lack of analytical data for enforcement, only tentative MRLs are derived. Tentative MRLs were also derived for maize forage in view of the future need to set MRLs in feed items."

The Applicant provided one report covered a total of 4 field trials in Northern Europe conducted on maize.
1. Andrews, G., 2016, Syngenta Report No. TK0258007-REG
Four decline residue field trials on maize were conducted in Northern France, Germany and Poland during 2015.
One application, at BBCH 16-18, of A19658H was made at 65.45 g ai/ha for nicosulfuron. Maize samples: whole plant, remaining plant and whole cobs were collected.
Results:
Residues of nicosulfuron in whole cob samples taken at 75-79 BBCH and 89 BBCH were below the limit of quantification (<0.01 mg/kg).
Residues of nicosulfuron in remaining plant samples taken at 75-79 BBCH and 89 BBCH were below the limit of quantification (<0.01 mg/kg).
Residues of nicosulfuron in whole plant samples taken at 39 BBCH were between <0.01 and 0.01 mg/kg and at 63 BBCH and 83-85 BBCH were below the limit of quantification (<0.01 mg/kg).
Residues of nicosulfuron in untreated samples were below the limit of quantification (<0.01 mg/kg).

Summary:
E: 4x <0.01 mg/kg,
R: 4x <0.01 mg/kg.
Residues of nicosulfuron in maize grain were below the limit of quantification (<0.01 mg/kg).
Available results show that the in force MRL on maize/corn of 0.01* mg/kg (Reg. (EU) No 617/2014) will not be exceeded. The current EU MRL for nicosulfuron is sufficient to support the proposed use.

Applicant proposes to add tank-mixed adjuvant (Adigor, STYK, Olejan or Efica 960 EC) to product ADM.4651.H.1.A (Nikita). Although, no trials have been submitted with the tank-mixed adjuvant, in our opinion, it could be expected that the use of an adjuvant will not lead to an exceedance of the MRL and will not lead to an unacceptable chronic risk for the consumer.
No further data are required to support the proposed uses.

*the limit of quantification

## 7.4.4 Magnitude of residues in livestock

### 7.4.4.1 Dietary burden calculation

The use of A18032E may result in residues of nicosulfuron in animal feed items, therefore the possible transfer of residues in animal commodities from the proposed uses should be considered.
As clarified in Regulation (EU) No. 1136/2014 (Regulation (EU) No 283/2013 as regards the transitional measures applying to procedures concerning plant protection products) and the associated guidance document SANCO/11509/2013 – rev. 5, May 2015) the old (AS) data requirements are applicable to active substances whose approval submission was made prior to 1 January 2014 until they are re-approved, and the animal intake triggering the submission of animal studies remains 0.1 mg/kg DM for the active substances falling under Reg. (EU) No 544/2011. Nicosulfuron meets these criteria and as such the EFSA calculation method that described in "Lundehn Appendix G" (EC Document 7031/VI/95 rev.4, July 1996) is presented below.
In order to satisfy the requirements of certain member states calculation of the animal dietary intake is also presented using the OECD calculation method described in the OECD Guidance Document on Residues in Livestock (ENV/JM/MONO(2013)8).
All livestock intake calculations undertaken are provided below.

**Table 7.4-9: Input values for the dietary burden calculation (considering the uses authorized in the country of the zRMS) (EFSA PROFile calculator)**

| Feed Commodity | Median dietary burden | | Maximum dietary burden | |
|---|---|---|---|---|
| | Input value (mg/kg) | Comment | Input value (mg/kg) | Comment |
| Risk assessment residue definition: nicosulfuron | | | | |
| Maize silage (Green Forages group) | 0.01 | Median residue (EFSA, 2012) | 0.015 | Highest residue (EFSA, 2012) |
| Maize grain (Cereal grains group) | 0.01 | Median residue (EFSA, 2012) | 0.01 | Median residue (EFSA, 2012) |

(a) No default processing factor was applied because nicosulfuron is applied early in the growing season and residues are expected to be below the LOQ. Concentration of residues in these commodities is therefore not expected.

**Table 7.4-10: Input values for the dietary burden calculation (considering the uses authorized in the country of the zRMS) (OECD calculator)**

| Feed Commodity | Median dietary burden | | Maximum dietary burden | |
|---|---|---|---|---|
| | Input value (mg/kg) | Comment | Input value (mg/kg) | Comment |
| Risk assessment residue definition: nicosulfuron | | | | |
| Corn, Field, Forage/Silage (Forages group) | 0.01 | Median residue (EFSA, 2012) | 0.015 | Highest residue (EFSA, 2012) |
| Corn, Field, Stover (Forages group) | 0.01 | Median residue (EFSA, 2012) | 0.015 | Highest residue (EFSA, 2012) |

| Feed Commodity | Median dietary burden | | Maximum dietary burden | |
|---|---|---|---|---|
| | Input value (mg/kg) | Comment | Input value (mg/kg) | Comment |
| Corn, Field, Grain (Grain/Seeds group) | 0.01 | Median residue (EFSA, 2012) | 0.01 | Median residue (EFSA, 2012) |
| Corn, Field, Milled Byprods. (By-products group) | 0.01 | Median residue (EFSA, 2012) and no PF as <LOQ[(a)] | 0.01 | Median residue (EFSA, 2012) and no PF as <LOQ[(a)] |
| Corn, Field, Hominy Meal (By-products group) | 0.01 | Median residue (EFSA, 2012) and no PF as <LOQ[(a)] | 0.01 | Median residue (EFSA, 2012) and no PF as <LOQ[(a)] |
| Corn, Field, Gluten Feed (By-products group) | 0.01 | Median residue (EFSA, 2012) and no PF as <LOQ[(a)] | 0.01 | Median residue (EFSA, 2012) and no PF as <LOQ[(a)] |
| Corn, Field, Gluten Meal (By-products group) | 0.01 | Median residue (EFSA, 2012) and no PF as <LOQ[(a)] | 0.01 | Median residue (EFSA, 2012) and no PF as <LOQ[(a)] |
| Sugarcane molasses (By-products group) | 0.01 | Median residue maize forage (EFSA, 2012) and no PF as <LOQ[(a)] | 0.01 | Median residue maize forage (EFSA, 2012) and no PF as <LOQ[(a)] |

(a) No default processing factor was applied because nicosulfuron is applied early in the growing season and residues are expected to be below the LOQ. Concentration of residues in these commodities is therefore not expected.

Nicosulfuron currently falls under old data requirements, therefore the only categories considered are dairy and beef cattle, laying poultry and pig. The results of the calculations are reported in the following table. Since the calculated dietary burdens for all groups of livestock were found to be below the trigger value of 0.1 mg/kg DM, further investigation of residues in commodities of animal origin is not necessary.

**Table 7.4-11: Results of the dietary burden calculation (EFSA PROFile calculator)**

| Animal species | Median dietary burden (mg/kg bw/d) | Maximum dietary burden (mg/kg bw/d) | Highest contributing commodity | Max dietary burden (mg/kg DM) | Trigger exceeded (Y/N) |
|---|---|---|---|---|---|
| Risk assessment residue definition: Nicosulfuron | | | | | |
| Beef cattle* | 0.0021 | 0.0032 | Corn, field, forage/silage | 0.075 | N |
| Dairy cattle* | 0.0018 | 0.0027 | Corn, field, forage/silage | 0.075 | N |
| Finishing swine* | 0.0005 | 0.0006 | Corn, field, milled byproducts | 0.016 | N |
| Layer poultry* | 0.0005 | 0.0005 | Corn, field, forage/silage | 0.0008 | N |

* These categories correspond to those (formerly) assessed at EU level.

**Table 7.4-12: Results of the dietary burden calculation (OECD calculator)**

| Animal species | Median dietary burden (mg/kg bw/d) | Maximum dietary burden (mg/kg bw/d) | Highest contributing commodity | Max dietary burden (mg/kg DM) | Trigger exceeded (Y/N) |
|---|---|---|---|---|---|
| Risk assessment residue definition: Nicosulfuron | | | | | |
| Beef cattle* | 0.0006 | 0.0006 | Corn, field, forage/silage | 0.03 | N |
| Dairy cattle* | 0.0009 | 0.0009 | Corn, field, forage/silage | 0.02 | N |
| Finishing swine* | 0.0003 | 0.0003 | Corn, field, milled byproducts | 0.01 | N |

| Animal species | Median dietary burden (mg/kg bw/d) | Maximum dietary burden (mg/kg bw/d) | Highest contributing commodity | Max dietary burden (mg/kg DM) | Trigger exceeded (Y/N) |
|---|---|---|---|---|---|
| Layer poultry* | 0.0009 | 0.0009 | Corn, field, forage/silage | 0.01 | N |

* These categories correspond to those (formerly) assessed at EU level.

### 7.4.4.2 Livestock feeding studies (KCA 6.4.1-6.4.3)

According to the metabolism studies (see 7.2.2.5), it is concluded that, after exposure to the maximum dietary burden (about 2-2700 times lower than the dose level of the metabolism studies), residue levels in livestock commodities are expected to remain below the enforcement LOQ of 0.01 mg/kg in milk and tissues. Hence, no livestock feeding study is needed.
No new data were submitted in the framework of this application.

*Reference: EFSA, 2012*
"No significant exposure of livestock to nicosulfuron residues is expected. Some data is available for future reference, however at this time it is not possible to propose residue definitions in animal products. However, based on the authorised uses on maize grain and forage, exposure of livestock is not expected to be significant and MRLs for products of animal origin are currently not required."

**zRMS comments:**

Information presented by Applicant are sufficient.
The median and maximum dietary burdens were calculated for different groups of livestock using the EFSA PROFile calculator and OECD calculator.
This submission should follow the new data requirements, the calculation of the dietary burden should be calculated with the Animal model 2017 based on the OECD guidance 73. Hence, zRMS calculation, including the same input values as the applicant, is presented below with results for all animals groups.
Results of the dietary burden calculation

| Relevant groups | Dietary burden expressed in | | | | Most critical diet (a) | Most critical commodity (b) | | Trigger exceeded (Yes/No) |
|---|---|---|---|---|---|---|---|---|
| | mg/kg bw per day | | mg/kg DM | | | | | 0.004 |
| | Median | Maximum | Median | Maximum | | | | mg/kg bw |
| Cattle (all diets) | 0,001 | 0,002 | 0,04 | 0,05 | Dairy cattle | Corn, field | gluten feed | No |
| Cattle (dairy only) | 0,001 | 0,002 | 0,03 | 0,04 | Dairy cattle | Corn, field | gluten feed | No |
| Sheep (all diets) | 0,001 | 0,001 | 0,02 | 0,02 | Lamb | Corn, field | gluten feed | No |
| Sheep (ewe only) | 0,001 | 0,001 | 0,02 | 0,02 | Ram/Ewe | Corn, field | gluten feed | No |
| Swine (all diets) | 0,001 | 0,001 | 0,02 | 0,03 | Swine (breeding) | Corn, field | gluten feed | No |
| Poultry (all diets) | 0,002 | 0,002 | 0,02 | 0,03 | Poultry layer | Corn, field | hominy meal | No |
| Poultry (layer only) | 0,002 | 0,002 | 0,02 | 0,03 | Poultry layer | Corn, field | hominy meal | No |

(a): When several diets are relevant (e.g. cattle, sheep and poultry "all diets"), the most critical diet is identified from the maximum dietary burdens expressed a
(b): The most critical commodity is the major contributor identified from the maximum dietary burden expressed as "mg/kg bw per day".

The calculated dietary burdens for nicosulfuron for all groups of livestock were found to be below the trigger value of 0.004 mg/kg bw per day for all animal species. Further investigation of residues in animal commodities is therefore not required.

### 7.4.5 Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation) (KCA 6.5.2-6.5.3)

*Reference: EFSA, 2012*
"As quantifiable residues of nicosulfuron are not expected in the treated crops and the chronic exposure does not exceed 10% of the ADI, there is no need to investigate the effect of industrial and/or household processing."

**zRMS comments:**

Information given by the Applicant is sufficient.
Processing studies are required if the level of residue in the plant or plant product to be processed exceeds >0.1 mg/kg (OECD Test Guideline 508: Magnitude of the pesticide residues in processed commodities). Total residues of nicosulfuron not exceeded 0.1 mg/kg in the RAC for maize in supervised residue trials for representative use, so processing studies are not required to support the proposed use of nicosulfuron in this submission.
No further data are required to support the proposed use.

## 7.4.6 Magnitude of residues in representative succeeding crops

Based on the results of the confined rotational crop study presented (see 7.4.2.2), no residues of nicosulfuron are to be expected in rotational crops. Residues of the metabolite ASDM were found in crops grown in soil treated with [pyridine-2-$^{14}$C] nicosulfuron at concentrations > 0.01 mg/kg. However ASDM is not a residue of concern and therefore a field rotational study is not required.
*Reference: EFSA, 2012*
"Occurrence of nicosulfuron residues in rotational crops was already investigated during the peer review of nicosulfuron. It was concluded that significant residues in rotational crops are not expected. These conclusions also apply to the GAPs reported in the framework of this review.

**zRMS comments:**

Information given by the Applicant is sufficient.
According to the EFSA Journal 2012;10(12):3048:*"Maize and sweet corn may be grown in rotation and according to the soil degradation from field studies evaluated in the framework of the peer review, $DT_{90}$ values of nicosulfuron range between 30 and 210 days, and relevant soil metabolites ASDM, AUSN and UCSN have $DT_{90}$s higher than 365 days exceeding the trigger value of 100 days (EFSA, 2007b). According to the European guidelines on rotational crops (EC, 1997b), further investigation of residues in rotational crops is relevant.*
*In spite of high persistence however, lysimeter studies where crops of wheat and rye were grown following maize treated at the proposed application rate of 60 g/ha, indicated low uptake of parent and metabolites by the following cereal plants (TRR <0.01 mg/kg). Moreover, it was shown that the phytotoxic effect of nicosulfuron and its soil metabolites on dicotyledone plants leads to a self-limitation in the re-planting period.* ***After a plant back interval of 27 to 30 days and with the exception of maize, marked phytotoxic effects were observed in the following crops (cereals, sugar beet, maize, oilseed rape and clover) while residues of nicosulfuron and its soil metabolites were found to be below the LOQ (0.01 mg/kg).*** *Thus* ***the following crops could not be grown until the following spring at which time residues in soil of nicosulfuron and relevant metabolites have decreased to <0.001 mg/kg****. Furthermore, ASDM and AUSN are also found in maize but are non toxicological relevant. As UCSN metabolite has a similar toxicological profile as AUSN, it is considered non relevant as well (EFSA, 2007b).*
*Consequently, these studies are considered sufficient by EFSA to demonstrate the absence of residues in rotational crops, provided that nicosulfuron is applied in compliance with the GAPs reported in Appendix A."*

The crops under consideration can be grown in rotation.
No waiting periods beyond normal agricultural practice are proposed for succeeding crops to be planted.
No additional data are required.

## 7.4.7 Other / special studies (KCA6.10, 6.10.1)

The available data for the active substance sufficiently address aspects of the residue situation that might arise from the use of A18032E. Therefore, other special studies are not needed.

## 7.4.8 Estimation of exposure through diet and other means (KCA 6.9)

Toxicological reference values relevant for dietary risk assessment are reported in the summary of the evaluation (see 7.1.2).
As ARfD was not deemed necessary, acute risk assessment is not relevant.

### 7.4.8.1 Input values for the consumer risk assessment

**Table 7.4-13: Input values for the consumer risk assessment**

| Commodity | Chronic risk assessment | | Acute risk assessment | |
|---|---|---|---|---|
| | Input value (mg/kg) | Comment | Input value (mg/kg) | Comment |
| Risk assessment residue definition: nicosulfuron | | | | |
| Intended/relevant uses | | | | |
| Maize / corn | 0.01 | EU MRL* | Not relevant. ARfD was not deemed necessary. | |
| Further uses | | | | |
| Other commodities of plant or animal origin | variable | EU MRL* | Not relevant. ARfD was not deemed necessary. | |

* Regulation (EU) 617/2014 of 3 June 2014

As ARfD was not deemed necessary, acute risk assessment is not relevant.

### 7.4.8.2 Conclusion on consumer risk assessment

Extensive calculation sheets are presented in 0.

**Table 7.4-14: Consumer risk assessment**

| | |
|---|---|
| ADI | 2 mg/kg bw/d |
| TMDI (% ADI) according to EFSA PRIMo rev. 3.1 | 0.1% (based on NL toddler diet) |
| IEDI (% ADI) according to EFSA PRIMo rev. 3.1 | Not relevant. TMDI < 100%. |
| ARfD | Not relevant. ARfD was not deemed necessary. |
| IESTI (% ARfD) according to EFSA PRIMo rev. 3.* | Not relevant. ARfD was not deemed necessary. |
| NTMDI (% ADI) ** | Not relevant. |
| NEDI (% ADI)** | Not relevant. |
| NESTI (% ARfD) ** | Not relevant. |

* include raw and processed commodities if both values are required for PRIMo
** if national model is available

Chronic exposure calculations were performed using revision 3.1 of the EFSA Pesticide Residues Intake Model (PRIMo rev. 3.1) provided on the internet homepage of EFSA (https://www.efsa.europa.eu/). This exposure assessment model contains the relevant European food consumption data for different subgroups of the EU population. The model was developed to calculate simultaneously the short-term (acute) and long-term (chronic) dietary exposure to pesticide residue in food according to internationally agreed methodologies. The exposure is compared to the toxicological reference values (i.e. the ADI and the ARfD). As ARfD for nicosulfuron was not deemed necessary (EFSA *Scientific Report* (2007) 120, 1-91), acute risk assessment is not relevant.
The potential chronic dietary exposure was compared to the ADI of 2 mg/kg bw/d and TMDI values were achieved. Input values for all commodities were derived from existing EU MRL (Reg. (EU) 617/2014), representing a worst case scenario. The highest chronic exposure was calculated for NL toddler diet, representing 0.1% of the ADI. For this diet the highest contributors were milk: cattle (0.1% of ADI), apples (0.0% of ADI) and maize / corn (0.0% of ADI). Since TMDI values are below 100%, there is no need to perform higher tier/refined chronic exposure calculation.

The proposed uses of nicosulfuron in the formulation NIKITA (A18032E) does not represent unacceptable chronic risks for the consumer.

**zRMS comment:**

Information given by the Applicant is sufficient.

The calculation of the TMDI using EFSA model (PRIM0 ver. 3.1) and MRLs according to Regulation (EU) 617/2014 of 3 June 2014 led to a utilisation of the ADI of 0.1% for the NL toddler diet being the population group with the highest value. For this diet, the highest contributor is milk:Cattle with 0.1% of the ADI. The intended uses will not result in a consumer chronic exposure exceeding the ADI.
An acute consumer risk assessment was not deemed necessary.
The proposed use of mesotrione in the product NIKITA (A18032E) do not represent unacceptable chronic risk for the consumer.
No further data are required to support the proposed use.

## 7.5 Combined exposure and risk assessment

The product is a mixture of three active substances and for at least two of them an acute reference dose has been allocated. Therefore, combined acute exposure can be considered.

### 7.5.1 Acute consumer risk assessment from combined exposure

In a first step, dose-addition of residues of the individual active substances is assumed by making use of the Hazard Index (HI) concept. The Hazard Quotient (HQ) is calculated for all active substances in the PPP that are acutely toxic by performing deterministic IESTI/NESTI calculations with the calculation models EFSA PRIMo (rev.3.1) and appropriate national models, if required, and dividing the individual exposure levels by the respective ARfD. Addition of the individual HQs irrespective of any considerations on phenomenological effects or mode(s)/mechanisms of action results in the HI. The results of the HQ/HI calculations are summarized in the following table.

**Table 7.5-1: Acute consumer risk assessment from combined exposure**

| Crop | Active Ingredient | HQ (based on IESTI according to EFSA PRIMo rev. 3.1) |
|---|---|---|
| Maize / corn | dicamba | 0.01 |
| | mesotrione | 0.003 |
| | nicosulfuron | - |
| | **Cumulative risk Maize / corn (HI)** | **0.013** |

The Hazard Index is <1. Thus combined exposure to all active substances in NIKITA (A18032E) is not expected to present a consumer risk. No further refinement of the assessment is required.

### 7.5.2 Chronic consumer risk assessment from combined exposure

The uses under consideration provide only a minor contribution to the overall chronic exposure of consumers to pesticide residues. The issue requires a more universal consideration and possibly the generic usage of monitoring data. A harmonised approach is not yet available, and currently no specific consideration is warranted in the scope of this evaluation.

**zRMS comments:**

From a scientific point of view it is regarded necessary to take into account potential combination effects. However, the evaluation of cumulative or synergistic effects as requested by Art. 4 (3b) of Regulation (EC) No. 1107/2009 should only be performed when harmonised “scientific methods accepted by the Authority to assess such effects are available.”
Currently, no EU-harmonized guidance is available on the risk assessment of combined exposure to multiple active substances; this approach is not mandatory at EU level.

## 7.6 References

**Dicamba**

Denmark, 2007. Draft assessment report on the active substance dicamba prepared by the rapporteur Member State Denmark in the framework of Council Directive 91/414/EEC, February 2007.

Denmark, 2010. Final addendum to the draft assessment report on the active substance dicamba prepared by the rapporteur Member State Denmark in the framework of Council Directive 91/414/EEC, compiled by EFSA, November 2010.

EC (European Commission), 2008. Review report for the active substance dicamba. Finalised in the Standing Committee on the Food Chain and Animal Health at its meeting on 14 March 2008 in view of the inclusion of dicamba in Annex I of Council Directive 91/414/EEC. SANCO/829/2008- final rev.2, 07 March 2008. Updated in the meeting of the Standing Committee on 27 September 2011; final revision on 12 July 2016

EFSA (European Food Safety Authority), 2011. Conclusion on the peer review of the pesticide risk assessment of the active substance dicamba. EFSA Journal 2011;9(1):1965, [52 pp.] doi:10.2903/j.efsa.2011.1965.

EFSA (European Food Safety Authority), 2013a. Reasoned opinion on the modification of the existing MRL(s) for dicamba in genetically modified soybean. EFSA Journal 2013;11(10):3440, [38 pp.] doi:10.2903/j.efsa.2013.3440.

EFSA (European Food Safety Authority), 2013b. Reasoned opinion on the modification of the existing MRL(s) for dicamba in herbs and herbal infusions (leaves and flowers). EFSA Journal 2013;11(11):3470, [25 pp.] doi:10.2903/j.efsa.2013.3470.

FAO (Food and Agriculture Organisation of the United Nations), 2011a. Dicamba. In: Pesticide residues in food – 2010. Evaluations. Part I. Residues. FAO Plant Production and Protection Paper 206.

FAO (Food and Agriculture Organisation of the United Nations), 2011b. Dicamba. In: Pesticide residues in food – 2011. Evaluations. Part I. Residues. FAO Plant Production and Protection Paper 212.

FAO (Food and Agriculture Organisation of the United Nations), 2011c. Dicamba. In: Pesticide residues in food – 2010. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 200.

FAO (Food and Agriculture Organisation of the United Nations), 2011d. Dicamba. In: Pesticide residues in food – 2011. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 211.

**Mesotrione**

EC European Commission, 2003b. Review report for the active substance mesotrione. Finalised in the Standing Committee on the Food Chain and Animal Health at its meeting on 11 April 2003 in view of the inclusion of mesotrione in Annex I of Directive 91/414/EEC. Mesotrione SANCO/1416/2001 - Final, 14 April 2003.

EFSA (European Food Safety Authority), 2015. Reasoned opinion on the review of the existing maximum residue levels (MRLs) for mesotrione according to Article 12 of Regulation (EC) No 396/2005. EFSA Journal 2015;13(1):3976, 36 pp. doi:10.2903/j.efsa.2015.3976

EFSA (European Food Safety Authority), 2016. Peer review report to the conclusion regarding the peer review of the pesticide risk assessment of the active substance mesotrione. EFSA Journal 2016;14(3):4419, [103 pp.] doi:10.2903/j.efsa.2016.4419.

FAO (Food and Agriculture Organisation of the United Nations), 2015. Mesotrione. In: Pesticide residues in food – 2014. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Core Assessment Group on Pesticide Residues. FAO Plant Production and Protection Paper 221.

United Kingdom, 2015. Renewal assessment report (RAR) on the active substance mesotrione prepared by the rapporteur Member State, the United Kingdom, in the framework of Commission Implementing Regulation (EU) No 844/2012, February 2015.

United Kingdom, 2015a. Revised renewal assessment report (RAR) on mesotrione, compiled by EFSA, December 2015.

**Nicosulfuron**

EC (European Commission), 2008. Review report for the active substance nicosulfuron. Finalised in the Standing Committee on the Food Chain and Animal Health at its meeting on 22 January 2008 in view of the inclusion of nicosulfuron in Annex I of Council Directive 91/414/EEC. SANCO/3780/07-rev.1, 22 January 2008.
EFSA (European Food Safety Authority), 2007. Conclusion regarding the peer review of the pesticide risk assessment of the active substance nicosulfuron. EFSA Scientific Report (2007) 120, 1-91.
EFSA (European Food Safety Authority), 2012. Reasoned opinion on the review of the existing maximum residue levels (MRLs) for nicosulfuron according to Article 12 of Regulation (EC) No 396/2005. EFSA Journal 2012;10(12):3048, [27 pp.] doi:10.2903/j.efsa.2012.3048.
United Kingdom, 2006. Draft assessment report on the active substance nicosulfuron prepared by the rapporteur Member State United Kingdom in the framework of Council Directive 91/414/EEC, June 2006.
United Kingdom, 2007. Final addendum to the draft assessment report on the active substance nicosulfuron prepared by the rapporteur Member State United Kingdom in the framework of Council Directive 91/414/EEC, compiled by EFSA, July 2007.

# Appendix 1 Lists of data considered in support of the evaluation

**List of data submitted by the applicant and relied on**

**Dicamba**

| Data point | Author(s) | Year | Title<br>Company Report No.<br>Source (where different from company)<br>GLP or GEP status<br>Published or not | Vertebrate study Y/N | Owner |
|---|---|---|---|---|---|
| KCA1 6.3.1 / 02 | Andrews G. | 2016 | Nicosulfuron and Dicamba - Residue Validation and Study on Maize in Northern France, Germany and Poland in 2015<br>Syngenta<br>Battelle UK Ltd, Chelmsford, Essex, UK, TK0258007-REG<br>GLP<br>not published<br>Syngenta File No A19658H_10060 | N | SYN (ADAMA has LoA) |
| KCA1 6.3.1 / 03 | Mound L., Gardinal P., Kwiatkowski A. | 2006 | Prosulfuron (CGA152005) and Dicamba (SAN837): Residue Study in or on Maize In Switzerland<br>Syngenta Crop Protection AG, Basel, Switzerland<br>Syngenta - Jealott's Hill International, Bracknell, Berkshire, United Kingdom, 04-7016<br>GLP<br>not published<br>Syngenta File No SAN837/6543 | N | SYN (ADAMA has LoA) |
| KCA1 6.3.1 / 04 | Simon P. | 2006 | Prosulfuron and Dicamba: Residue study in or on maize in Germany 2004 (Test product: A14031B)<br>Syngenta Crop Protection AG, Basel, Switzerland<br>Syngenta Agro GmbH, Maintal, Germany, gmz043004<br>GLP<br>not published<br>Syngenta File No SAN837/6545 | N | SYN (ADAMA has LoA) |
| KCA1 6.3.1 / 05 | Oppilliart S | 2009 | Dicamba (CGA57706) and Prosulfuron (CGA152005) - Residue Study on Corn in France (North) in 2007<br>Syngenta - Jealott's Hill, Bracknell, United Kingdom<br>Eurofins - ADME Bioanalyses, Vergeze, France, T001033-07-REG<br>GLP<br>not published<br>Syngenta File No A14031E_10809 | N | SYN (ADAMA has LoA) |
| KCA1 6.5.1 / 01 | Yeomans P. | 2015 | NOA405873 - Simulated Processing Practices: Aqueous Hydrolysis of [14C] 5-Hydroxy-Dicamba at 90, 100 and 120 - C<br>Syngenta, BASF Corporation, Research Triangle Park, NC, USA<br>Smithers Viscient (ESG) Ltd, Harrogate, UK, 3200933 | N | SYN (ADAMA has LoA) |

| Data point | Author(s) | Year | Title<br>Company Report No.<br>Source (where different from company)<br>GLP or GEP status<br>Published or not | Vertebrate study Y/N | Owner |
|---|---|---|---|---|---|
| | | | GLP<br>not published<br>Syngenta File No NOA405873_10000 | | |
| KCA1 6.6.1 / 01 | Swales S. | 2016 | SAN837 - Uptake and Metabolism of [14C]-SAN837 in Confined Rotational Crops<br>Syngenta, BASF Corporation, Research Triangle Park, NC, USA<br>Smithers Viscient (ESG) Ltd, Harrogate, UK, AgroChemex Ltd, Manningtree, United Kingdom, 3200368<br>GLP<br>not published<br>Syngenta File No SAN837_11645 | N | SYN (ADAMA has LoA) |
| KCA1 6.6.2 / 01 | Andrews G. | 2016a | Dicamba- Residue Study on Rotational Crops in Southern France and Spain in 2014<br>Syngenta<br>Battelle UK Ltd, Chelmsford, Essex, UK, TK0223573-REG<br>GLP<br>not published<br>Syngenta File No A7254B_10456 | N | SYN (ADAMA has LoA) |
| KCA1 6.6.2 / 02 | Austin R., Andrews G. | 2016 | Dicamba - Residue Study on Rotational Crops in the United Kingdom and Northern France in 2014<br>Syngenta<br>Battelle UK Ltd, Chelmsford, Essex, UK, NC14032<br>GLP<br>not published<br>Syngenta File No A7254B_10460 | N | SYN (ADAMA has LoA) |

**Nicosulfuron**

| Data point | Author(s) | Year | Title<br>Company Report No.<br>Source (where different from company)<br>GLP or GEP status<br>Published or not | Vertebrate study Y/N | Owner |
|---|---|---|---|---|---|
| KCA3 6.1 (report available from data owner) | Peatman M. H. | 2007 | Nicosulfuron: Storage Stability of Residues in Corn Matrices Stored Deep Frozen<br>Report No. 0676/032-D2149 (NIS-42)<br>GLP, unpublished | N | Cheminova A/S (ADAMA has access (3 LOA) ) |

| Data point | Author(s) | Year | **Title**<br>**Company Report No.**<br>**Source (where different from company)**<br>**GLP or GEP status**<br>**Published or not** | Vertebrate study Y/N | Owner |
|---|---|---|---|---|---|
| KCA3 6.2.1 (report available from data owner) | Scott M.T. | 1989 | Plant metabolism study of $^{14}$C-DPX-V9360 in corn.<br>Report No. AMR 861-87<br>GLP, unpublished | N | E.I. du Pont de Nemours and Company (Study no longer has data protection) |
| KCA3 6.6.1 (report available from data owner) | Dietrich R.F. | 1989 | Confined accumulation study of 14C-DPX-V9360 on rotational crops<br>Report No. AMR 874-87<br>Non-GLP, unpublished | N | E.I. du Pont de Nemours and Company (Study no longer has data protection) |
| KCA3 6.6.1 (report available from data owner) | Scott M.T. | 2004 | Confined accumulation study of 14C-DPX-V9360 on rotational crops.<br>Report No. AMR 874-87, Supplement No. 1<br>Non-GLP, unpublished | | E.I. du Pont de Nemours and Company (Study no longer has data protection) |
| KCA3 6.2.3 (report available from data owner) | Lin P.T., Scott M.T., | 1989. | Metabolism of [pyrimidine-2-14C]DPX-V9360 and [pyridine 2 14C]DPX-V9360 in lactating goats<br>Syngenta Report No. AMR 947-87<br>GLP, unpublished | N | E.I. du Pont de Nemours and Company (Study no longer has data protection) |
| KCA3 6.3.1 / 01 | Andrews G. | 2016 | Nicosulfuron and Dicamba - Residue Validation and Study on Maize in Northern France, Germany and Poland in 2015<br>Syngenta<br>Battelle UK Ltd, Chelmsford, Essex, UK, TK0258007-REG<br>GLP<br>not published<br>Syngenta File No A19658H_10060 | N | SYN (ADAMA has LoA) |

**List of data submitted or referred to by the applicant and relied on, but already evaluated at EU peer review**

**Dicamba**

| Data Point | Author(s) | Year | Title<br>Company Report No.<br>Source (where different from company)<br>GLP or GEP status<br>Published or not | Vertebrate study Y/N | Owner |
|---|---|---|---|---|---|
| KCA1 6.3.1 / 01 | Hertl P. | 1995 | Determination of Residues of DICAMBA in CORN (Zea mais) after Application of BANVEL 4S or SAN 845 H 70 WG under Field Conditions in the FEDERAL REPUBLIC of GERMANY, 1993.<br>Novartis Crop Protection AG, Basel, Switzerland<br>Sandoz Agro Ltd., Huningue, France, R10280<br>GLP<br>not published<br>Syngenta File No SAN837/5434 | N | SYN (ADAMA has LoA) |

**Mesotrione**

| Data point | Author(s) | Year | Title<br>Company Report No.<br>Source (where different from company)<br>GLP or GEP status<br>Published or not | Vertebrate study Y/N | Owner |
|---|---|---|---|---|---|
| KCA2 6.3.1 / 01 | Barnes J. | 1997 | ZA1296: Residue Levels in Maize from Trials Carried out in Germany During 1995 (WRC-96-114)<br>Zeneca Agrochemicals, Jealott's Hill, United Kingdom<br>, RR 96-078B<br>GLP<br>not published<br>Syngenta File No ZA1296/0409 | N | SYN (ADAMA has LoA) |
| KCA2 6.3.1 / 02 | Heillaut C | 2009 | Glyphosate (ASF71), Mesotrione (ZA1296) and S-Metolachlor (CGA77102) - Residue Study on GA21 (MON----21-9) Corn in France (North) and Czech Republic in 2007<br>Syngenta<br>ADME - Bioanalyses, Vergeze, France, T011085-06<br>GLP<br>not published<br>Syngenta File No A15189G_10009 | N | SYN (ADAMA has LoA) |
| KCA2 6.3.1 / 03 | Heillaut C | 2009a | Glyphosate, Mesotrione and S-Metolachlor - Residue Study on GA21 (MON-00021-9) Corn in Denmark and Sweden in 2008<br>Syngenta<br>ADME - Bioanalyses, Vergeze, France, T009533-07-REG<br>GLP<br>not published | N | SYN (ADAMA has LoA) |

| Data point | Author(s) | Year | Title<br>Company Report No.<br>Source (where different from company)<br>GLP or GEP status<br>Published or not | Vertebrate study Y/N | Owner |
|---|---|---|---|---|---|
| | | | Syngenta File No A15189G_10014 | | |
| KCA2 6.3.1 / 04 | Meyer M | 2011 | Mesotrione - Residue Study on Field Corn in Germany and the United Kingdom in 2009<br>Syngenta - Jealott's Hill, Bracknell, United Kingdom<br>SGS INSTITUT FRESENIUS GmbH, Im Maisel 14, D-65232 Taunusstein, Germany, T000920-09-REG<br>GLP<br>not published<br>Syngenta File No A14203B_10105 | N | SYN (ADAMA has LoA) |
| KCA2 6.3.1 / 05 | Klimmek S., Gizler A. | 2008 | Mesotrione and Nicosulfuron: Residue Study on Maize in Northern France in 2007<br>Syngenta - Jealott's Hill, Bracknell, United Kingdom<br>Eurofins - Dr Specht & Partner, Hamburg, Germany, T011368-06-REG<br>GLP<br>not published<br>Syngenta File No A14351BX_10613 | N | SYN (ADAMA has LoA) |
| KCA2 6.3.1 / 06 | Schulz H | 2010 | Mesotrione and Nicosulfuron - Residue Study on Maize in France (North) in 2008<br>Syngenta - Jealott's Hill, Bracknell, United Kingdom<br>SGS INSTITUT FRESENIUS GmbH, Im Maisel 14, D-65232 Taunusstein, Germany, T009530-07-REG<br>GLP<br>not published<br>Syngenta File No ZA1296_10049 | N | SYN (ADAMA has LoA) |

**List of data submitted by the applicant and not relied on**

| Data point | Author(s) | Year | Title<br>Company Report No.<br>Source (where different from company)<br>GLP or GEP status<br>Published or not | Vertebrate study Y/N | Owner |
|---|---|---|---|---|---|
| - | - | - | - | - | - |

**List of data relied on and not submitted by the applicant but necessary for evaluation**

| Data point | Author(s) | Year | Title<br>Company Report No.<br>Source (where different from company)<br>GLP or GEP status<br>Published or not | Vertebrate study Y/N | Owner |
|---|---|---|---|---|---|
| - | - | - | - | - | - |

# Appendix 2 Detailed evaluation of the additional studies relied upon

## A 2.1 Dicamba

### A 2.1.1 Stability of residues

No new data are submitted in the framework of this application.

### A 2.1.2 Nature of residues in plants, livestock and processed commodities

**Nature of residue in plants**

#### A 2.1.2.1.1 Nature of residue in primary crops

No new data are submitted in the framework of this application.

#### A 2.1.2.1.2 Nature of residue in rotational crops

#### A 2.1.2.1.2.1 Study TK0103764

| | |
|---|---|
| Comments of zRMS: | Syngenta has conducted a new study in confined rotational crops and this confirms the high total radioactive residues (TRRs) at 30 DAT (0.027-0.886 mg/kg) and progressive and marked decline in subsequent 111 DAT (0.017–0.097 mg/kg) and 285 DAT (0.001–0.016 mg/kg) rotational intervals that were observed in the earlier studies.<br>This data is currently under evaluation for the renewal of approval of the active substance, dicamba.<br>The study demonstrates that the metabolic pathway in rotational crops is identical to that previously elucidated in the primary metabolism studies.<br>The study was conducted to comply with current data requirements and study guidelines (OECD Guideline for the Testing of Chemicals, 502, Metabolism in Rotational Crops, 2007).<br>The study is acceptable. |

| | |
|---|---|
| Reference: | KCA1 6.6.1/01 |
| Report | Swales S, 2016. SAN837 – Uptake and Metabolism of [$^{14}$C-SAN837 in Confined Rotational Crops. Report Number 3200368. Smithers Viscient (ESG), 108 Woodfield Drive, Harrogate, North Yorkshire, HG1 4LS, UK.<br>Study Dates: July 2013 – May 2016.<br>Syngenta Report No TK0103764<br>Syngenta File No. SAN837_11645)<br>GLP, unpublished |
| Guideline(s): | OECD Guideline for the Testing of Chemicals, 502, Metabolism in Rotational Crops. (January 2007)<br>EPA Residue Chemistry Test Guidelines OPPTS 860.1850, Confined Accumulation in Rotational Crops (August 1996)<br>Commission of the EC, Working document 7524/VI/95 rev. 2 (July 1997)<br>Japanese MAFF Guidelines on the Application for Agricultural Chemicals Registration Nohsan No 8147 (November 2000) |
| Deviations: | No |
| GLP: | Yes |

Acceptability: Yes

**Executive Summary**

This study was designed to provide information on the uptake and metabolism of dicamba (SAN837) in rotational crops following a single application to soil, to qualitatively and quantitatively determine the nature of the likely residue. A single radiolabelled form was used in the study, [phenyl-U-$^{14}$C]-SAN837.

A single spray application onto the surface of bare sandy loam soil in nine containers was performed at a nominal application rate of 480 g dicamba acid equivalents/ha employing a SL480 formulation (A7254B; dimethylammonium salt) at a spray volume of 407 L/ha. The actual application rate achieved was 488 g dicamba acid equivalents/ha (102% of the target weight). The containers of soil were kept outside for a period of 20 days after application before being transferred to a greenhouse for the remainder of the biological phase of the study. At intervals of 30, 111 and 285 days after application the three representative rotational crops (spinach, radish and spring wheat) were sown into the treated soil. The biological phase of the study was performed at Agrochemex Limited, Aldhams Farm, Lawford, Manningtree, Essex CO11 2NF, UK.

For each rotational crop interval the samples summarised in the table below were harvested and separated into representative food and feed items as appropriate.

| **Commodity** | **Raw Agricultural Commodity (RAC)** | **Growth Stage (BBCH)** |
|---|---|---|
| Spinach | Immature | 42 – 43 |
| | Mature | 49 |
| Radish | Mature Roots | 49 |
| | Mature Leaves | 49 |
| Wheat | Forage | 23-30 |
| | Hay | 45–47 |
| | Grain | 89 |
| | Straw | 89 |

Following hay harvest, the raw crop was air dried in the glasshouse until classified a hay (4-5 days).

At final harvest, wheat at growth stage BBCH 89 was threshed by hand to separate grain from chaff. Chaff was included in the straw samples.

The crop samples were frozen and transported by courier in dry ice to Smithers Viscient (ESG). Each commodity was homogenised in dry ice and the radioactive residue (RR) determined by combustion analysis/LSC.

In all instances combusted radioactive residues in commodities were greatest from the 30 DAT rotational interval (0.027-0.886 mg/kg) but decreased progressively and markedly in successive 111 DAT (0.017-0.097 mg/kg) and 285 DAT (0.001-0.016 mg/kg) rotational intervals (see Table A 1).

Radioactive residues in the 30 DAT rotational commodities were highest in wheat hay (0.886 mg/kg), forage (0.533 mg/kg) and straw (0.472 mg/kg) and radish leaf (0.279 mg/kg). Lower residues were observed in wheat grain (0.090 mg/kg), immature spinach (0.070 mg/kg), radish root (0.028 mg/kg) and mature spinach (0.027 mg/kg).

The overall lower residues in 111 DAT commodities were highest in wheat hay (0.097 mg/kg) and straw (0.086 mg/kg) with the remaining commodity residues ranging from 0.017 mg/kg (radish root) to 0.040 mg/kg (radish leaf).

By the 285 DAT harvest, only residues in wheat hay (0.016 mg/kg), straw (0.014 mg/kg) and grain (0.011 mg/kg) were greater than 0.01 mg/kg. Other commodity residues ranged from 0.001 mg/kg (wheat forage) to 0.006 mg/kg (immature spinach; with mature spinach, radish leaf and root all with 0.002 mg/kg).

With the exception of the 285 day radish (leaves and roots), spinach (immature and mature) and wheat (forage) all commodities therefore contained radioactive residues >0.01 mg/kg and consequently required further analysis.

Sub-samples of homogenates from all crop commodities of the 30 DAT rotational interval were

sequentially extracted with solvents of increasing polarity (mixtures of acetonitrile:water) and finally acetone (crop debris drying solvent), subsequently referred to as the *solvent extraction method*. Radioactive residues present in extracts were fractionated and characterised.

Sub-samples of commodity homogentates from the 111 DAT rotational interval with radioactive residues >0.01 mg/kg (with the exception of 111 DAT wheat straw which was analysed as indicated for 30 DAT commodities) were similarly extracted using the solvent extraction method but, due to the larger proportions of i) polar extractable residue observed in these solvent extracts and ii) non-extractable residue remaining in the crop debris, additional sub-samples of crop homogenates were taken and extracted in separate analyses with hydrochloric acid at elevated temperature (100ºC) and under progressively more forcing extract conditions (1M HCl and where appropriate 6M HCl; subsequently referred to as the *acid extraction method*). Radioactive residues present in acid extracts were fractionated and characterised.

By 285 DAT the limited number of crop commodities with residues >0.01 mg/kg were extracted either using both solvent and acid extraction methods in separate analyses (wheat hay) or by the acid extraction method only (wheat straw and grain).

Extractability of residues by the solvent extraction method (see Table A 2) was generally efficient for 30 DAT rotational commodities with 74.6-94.0% TRR extractable in all instances except wheat grain (28.4% TRR).

With the exception of radish roots (91.4.% TRR) the efficiency of solvent extraction was generally lower in 111 DAT rotational commodities (33.3-77.3% TRR in all other commodities except that of grain which was even lower; 16.9% TRR). Extractability of the corresponding 111 DAT commodities using the acid extraction method (see Table A 3) was generally higher (45.8-91.7% TRR) than that of the solvent method and was much more efficient for the grain commodity (86.6% TRR).

By the 285 DAT rotational interval harvest, only wheat hay, straw and grain required extraction analysis and of these three commodities only wheat hay was deemed appropriate for solvent extraction analysis (yielding 53.1% TRR) as only very low solvent extractabilities were demonstrated by straw (33.3% TRR) and grain (16.9% TRR) in 111 DAT commodities. Extractability by the acid extraction method was only moderate for wheat hay and straw (35.5-49.6% TRR) but was relatively efficient for wheat grain (76.4% TRR).

Selected solvent extract fractions from all the 30 DAT commodities analyses and the 111 DAT wheat straw commodity were taken forward for full nature of residue analysis. This involved concentration and, where appropriate, hydrolysis with hydrochloric acid (1M; 100ºC) and/or liquid/liquid partition procedures in order to produce suitable fractions for chromatographic analysis. Where appropriate, further characterisation of selected non-extractable fractions were also subject to hydrolysis with hydrochloric acid (1M and 6M; 100ºC) and the resulting fractions were, where appropriate, taken forward chromatographic analysis. Results from these analyses allowed the levels of identified parent and metabolites, quantified separately as the free (unconjugated) and conjugated forms of the residue, to be determined.

Analysis of remaining 111 DAT commodities was conducted in two separate stages. Initially, selected extracts derived from the solvent extract method were subject to limited analysis only (i.e. concentration and chromatographic analysis) in order to determine the levels of parent and metabolite residues present in the free (unconjugated) forms only. Then, extract fractions derived from the acid extract method were taken forward for full nature of residue analysis. This involved concentration and, where appropriate, liquid/liquid partition procedures in order to produce suitable fractions for chromatographic analysis. Results from these acid extraction analyses allowed the levels of identified parent and metabolites (quantified as their collective free plus conjugated form of the residue) to be determined.

Using residue results from both the solvent extraction (free residues only) and acid extraction analyses (free plus conjugated residue) allowed levels of identified parent and metabolites, present as the conjugated form of the residue, to be derived.

Analysis of 285 DAT wheat hay was undertaken using the approach taken for the bulk of the 111 DAT commodity analyses (separate solvent extract method and acid extract method analyses). Analysis of 285 DAT wheat straw and grain was by via acid extraction method only, because it was anticipated that negligible residue would be extracted using the solvent extraction method, based on

the very limited solvent extractability evident in the corresponding 111 DAT wheat straw and grain analysis.
Radioactivity present in the resulting principal residue containing fractions were subject to thin layer chromatography (TLC: 2D-TLC and/or 1D-TLC) with radiodetection for quantification and identification of individual radiocomponents (by comparison with authentic reference standards of parent and its putative metabolites). Confirmation of the identity of metabolites present in selected fractions was undertaken by high performance liquid chromatography (HPLC) with radiodetection.
Specific chromatographic analyses were also undertaken on selected analytical fractions aimed at characterising the presence of natural incorporated radioactivity ($^{14}$C-glucose evident following acid extract/hydrolysis procedures).
Identified residues following the above analyses are summarised below and are also presented in Table A 4.
Parent residues (SAN837) were detected in all 30 DAT and 111DAT analysed commodities but were not detectable in 285 DAT commodities.
Parent residues were greatest in 30 DAT commodities and was the predominant identified residue in 30 DAT radish and spinach commodities (45.2 – 67.0% TRR; 0.014 – 0.204 mg/kg). Parent also accounted for a large proportion of the 30 DAT wheat forage (20.7% TRR; 0.107 mg/kg) and hay (13.8% TRR; 0.109 mg/kg) total radioactive residue but was much less evident in wheat straw (0.4% TRR; 0.002 mg/kg) and grain (1.9% TRR; 0.002 mg/kg).
The principal identified metabolite in 30 DAT wheat commodities was NOA405873 and was greatest in forage, hay and straw (43.2 – 56.3% TRR; 0.260 – 0.342 mg/kg) but was present at much lower residues in grain (5.5% TRR; 0.005 mg/kg). Residues of this metabolite were even lower in 30 DAT radish leaves and immature spinach (≤2.0% TRR; ≤0.004 mg/kg) and were not detected in radish root and mature spinach. Residues of the other two metabolites identified in 30 DAT commodities (NOA414746: ≤6.5% TRR; ≤0.052 mg/kg and R740230: ≤1.9% TRR; ≤0.009 mg/kg) were generally either present at much lower levels than NOA405873 or were not detected at all.
By the time of 111 DAT rotational harvests, levels of parent and the metabolites identified above had either decreased markedly or were not detectable. Residues of parent were always ≤ 0.007 mg/kg and were not detected at all in wheat grain. Residues of NOA405873 were only detected in wheat hay (15.8% TRR; 0.017 mg/kg), wheat straw (5.7% TRR; 0.004 mg/kg) and mature spinach (2.3% TRR; 0.001 mg/kg). Residues of NOA414746 were either ≤0.003 mg/kg (immature and mature spinach, wheat hay and straw) or not detected at all and residues of R740230 were not detected in any of the analysed commodities at this rotational interval.
By the time of 285 DAT rotational harvests parent was not detectable in any of the commodities analysed and the only identified metabolite detected was NOA414746 which was present in wheat hay only (4.0% TRR; 0.001 mg/kg).
Where identified in all the above analyses, parent, NOA405873 and NOA414746 were found in the free and/or conjugated forms of the residue. R740230 was found exclusively in the conjugated form of the metabolite. Formal determination of the type of conjugation was not undertaken in this study, however conjugate characterisation of parent and these metabolites was undertaken in a previously undertaken $^{14}$C-dicamba wheat metabolism study (Report Number 97SV01 previously EU reviewed – see CA 6.2.1) where they were characterised to be β-glucoside conjugates. It is therefore also likely that conjugates present in this crop rotation study were β-glucoside conjugates.
The decline in parent and identified metabolite residues in successive rotational intervals was accompanied by an increase in the proportion of the total radioactive residue associated with polar and/or bound endogenous radioactive components characterised by the release of $^{14}$C-glucose following acid hydrolysis (wheat grain: ≤41.7% TRR; radish root ≤31.0% TRR; wheat hay: ≤17.8% TRR; wheat straw: ≤16.1% TRR; wheat forage: ≤14.0% TRR; immature spinach: ≤7.9% TRR). These results are consistent with parent (SAN837) and/or its identified metabolites being extensively and completely metabolised to small $^{14}$C-containing fragments (either in the crop and/or in the soil) which are then assimilated within the natural biosynthetic pool (including glucose) of endogenous crop components, a process known as natural incorporation. Precedent for the rapid mineralisation of $^{14}$C-dicamba to $^{14}CO_2$ in soil (Report Number 99AG08 previously EU Reviewed - see CA 7.1.1.1) and the presence of naturally incorporated residues in the grain and straw samples of a $^{14}$C-dicamba wheat

metabolism study (Report Number 97SV01 previously EU reviewed – see CA 6.2.1) exist.
The levels of $^{14}$C-glucose detected in commodity analyses within this crop rotation study probably represent only a relatively small proportion of the total levels of natural incorporation that exist in reality because i) the hydrolysis methods used to produce $^{14}$C-glucose were not exhaustive and ii) the analyses did not take into account the contribution to natural incorporation from other significant endogenous sources such as protein (via incorporated amino acids) and lignin. These additional contributions may potentially account for a significant proportion of the remaining unassigned extractable and un-extractable residue.
The results show that:

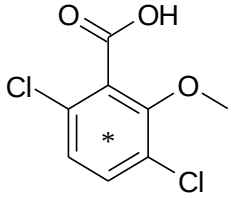
- Radioactive residues observed in 30 DAT rotational commodities declined rapidly and progressively in subsequent 111 DAT and 285 DAT commodities.
- In 30 DAT rotational crops :
    - Parent AI was the principal residue identified in radish and spinach commodities (≤67% TRR; ≤0.204 mg/kg) and was a major residue in wheat forage and hay (≤20.7% TRR; ≤0.109 mg/kg) but was only a minor residue in wheat straw and grain (≤1.9% TRR; 0.002 mg/kg).
    - NOA405873 was the principal identified metabolite in wheat, the residue levels of which were greatest in forage, hay and straw (≤56.3% TRR; ≤0.342 mg/kg) but were much lower in all other commodities (≤5.5% TRR; ≤0.005 mg/kg).
    - Residue levels of the other two identified metabolites NOA414746 (≤6.5% TRR; ≤0.052 mg/kg) and R740230 (≤1.9% TRR; ≤0.009% TRR) were generally much lower than NOA405873.
- By the time of 111 DAT rotational harvests, residues of parent and all identified metabolites had declined to ≤0.007 mg/kg (except for NOA405873 in wheat hay, 0.017 mg/kg).
- By the time of 285 DAT rotational harvests, residues of parent and all metabolites identified above were not detectable (except for NOA414746 in wheat hay, 0.001 mg/kg).
- The decline in parent and identified metabolite residues in successive rotational intervals was accompanied by an increase in the proportion of the total radioactive residue associated with naturally incorporated radioactivity (≤ 41.7% TRR identified as $^{14}$C glucose).

The observed metabolites of dicamba (SAN837) were generated via:

- hydroxylation of the aromatic carbon para to the methoxy functionality of dicamba to produce NOA405873
- demethylation of the methoxy functionality of dicamba to produce NOA414746
- either, demethylation of the methoxy functionality of NOA405873, and/or hydroxylation of the aromatic carbon para to the existing hydroxy functionality of NOA414746 to produce, in both instances, the dihydroxylated metabolite R740230
- glucose conjugation of both dicamba and the above metabolites

**Materials and Methods**

**Test Materials**

| | |
|---|---|
| Structure/Label | [Phenyl-U-$^{14}$C]-SAN837 |
| | <br>(* = $^{14}$C position) |
| CAS Number | 1918-00-9 |
| Batch Number | CDC-XXXVI-7-1 |
| Specific Activity | 2.738 MBq/mg (74 µCi/mg) |
| Radiochemical Purity | 99.1% (as stated on certificate of analysis) |
| Appearance | White powder |

**Test System**
The crops selected to represent the three different groupings (leafy, cereal and root) required in this confined rotational crop study were spinach (*Spinacia oleracia*; variety *F1 Mississippi*), spring wheat (*Triticum aestivum*; variety *Tybalt*) and radish (*Raphanus sativus*; variety *F1 Apache*) respectively.

**Test Soil**
A sandy loam soil was used in this study.

**Study Design**
**Experimental Conditions**

| | |
|---|---|
| Nominal application rate | 480 g acid equivalents/ha |
| Number of applications | 1 |
| Target seasonal application rate | 480 g acid equivalents/ha |
| Achieved seasonal application rate | 488 g acid equivalents/ha |
| Formulation type | Soluble liquid (SL) |
| Formulation code | (A7254B; dimethylammonium salt) |
| Formulation concentration over applications | 3.279 MBq/mL |
| Spray rate | 407 L/ha |
| Method of application | A lance and trigger sprayer with an even flat fan nozzle and $CO_2$ propellant |
| Plant back intervals | 30, 111 and 285 days after application |
| Environmental conditions | Treated containers of soil maintained outside for 20 days then moved into glasshouse with artificial lighting for remainder of biological phase of study |

**Test Samples**
For each plant-back interval (i.e. 30, 111 and 285 days after application) the following crop samples were taken:

- Immature spinach (BBCH 42-43)
- Mature spinach (BBCH 49)
- Mature radish roots and leaves (BBCH 49)
- Wheat forage (BBCH 23-30)
- Wheat hay (BBCH 45-47) and air dried in greenhouse until deemed classified as hay (4-5 days)
- Wheat straw and grain (BBCH 89)

**Sample Preparation**
Suitable sub-samples of all crop commodities (excluding grain) were homogenised using a blender in the presence of dry ice. Grain and chaff were separated and the chaff was added to the corresponding straw sample. Grain was homogenized using a mill to produce a fine powder. Following homogenisation, the radioactive residue in each sample was quantified by combustion analysis of replicate aliquots followed by LSC. These values are summarized in Table A 1.

Following processing, the homogenised samples were returned to a freezer and stored at ≤-10ºC (nominally -20º) until required for analysis.

**Extraction and Fractionation of Residues**
Crop commodities containing radioactive residues > 0.01 mg/kg were sequentially extracted as detailed below.
Sub-samples of homogentates from all crop commodities of the 30 DAT rotational interval were sequentially extracted with solvents of increasing polarity (mixtures of acetonitrile:water) and finally acetone (crop debris drying solvent), referred to as the *solvent extraction method*.
Sub-samples of commodity homogentates from the 111 DAT rotational interval with radioactive residues >0.01 mg/kg (with the exception of 111 DAT wheat straw which was analysed as indicated

for 30 DAT commodities) were similarly extracted using the solvent extraction method but, due to the larger proportions of i) polar extractable residue observed in these solvent extracts and ii) non-extractable residue remaining in the crop debris, additional sub-samples of crop homogenates were taken and extracted in separate analyses with hydrochloric acid at elevated temperature (100ºC) and under progressively more forcing extract conditions (1M and 6M HCl; referred to as the *acid extraction method*). Radioactive residues present in acid extracts were fractionated and characterised.
By 285 DAT the limited number of crop commodities with residues >0.01 mg/kg were extracted either using both solvent and acid extraction methods in separate analyses (wheat hay) or by the acid extraction method only (wheat straw and grain).

The overall levels of extractability in commodities are summarised in Table A 2 for the solvent extractability method and Table A 3 for the acid extractability method.
Selected solvent extract fractions from all the 30 DAT commodities analyses and the 111 DAT wheat straw commodity were taken forward for full nature of residue analysis. This involved concentration and, where appropriate, hydrolysis with hydrochloric acid (1M; 100ºC) and/or liquid/liquid partition procedures in order to produce suitable fractions for chromatographic analysis. Where appropriate, further characterisation of selected non-extractable fractions were also subject to hydrolysis with hydrochloric acid (1M and 6M; 100ºC) and the resulting fractions were, where appropriate, taken forward chromatographic analysis. Results from these analyses allowed the levels of identified parent and metabolites, quantified separately as the free (unconjugated) and conjugated forms of the residue, to be determined.
Analysis of remaining 111 DAT commodities was conducted in two separate stages. Initially, selected extracts derived from the solvent extract method were subject to limited analysis only (i.e. concentration and chromatographic analysis) in order to determine the levels of parent and metabolite residues present in the free (unconjugated) forms only. Then, extract fractions derived from the acid extract method were taken forward for full nature of residue analysis. This involved concentration and, where appropriate, liquid/liquid partition procedures in order to produce suitable fractions for chromatographic analysis. Results from these acid extraction analyses allowed the levels of identified parent and metabolites (quantified as their collective free plus conjugated form of the residue) to be determined. Using residue results from both the solvent extraction (free only residues) and acid extraction analyses (free plus conjugated residue) allowed levels of identified parent and metabolites, present as the conjugated forms of the residue, to be derived.
Analysis of 285 DAT wheat hay was undertaken using the approach taken for the bulk of the 111 DAT commodity analyses (separate solvent extract method and acid extract method analyses). Analysis of 285 DAT wheat straw and grain was by via acid extraction method only, because it was anticipated that negligible residue would be extracted using the solvent extraction method, based on the very limited solvent extractability evident in the corresponding 111 DAT wheat straw and grain analysis.
Radioactivity present in the resulting principal residue containing fractions were subject to thin layer chromatography (TLC: 2D-TLC and/or 1D-TLC) with radio-detection for quantification and identification of individual radio-components (by comparison with authentic reference standards of parent and its putative metabolites). Confirmation of the identity of metabolites present in selected fractions was undertaken by high performance liquid chromatography (HPLC) with radio-detection.
Specific chromatographic analyses were also undertaken on selected analytical fractions aimed at characterising the presence of natural incorporated radioactivity ($^{14}$C-glucose evident following acid extract/hydrolysis procedures).
Identified residues following the above analyses are summarised in Table A 4 and more detailed overall summaries of analyses are given in Table A 5 to Table A 23.

**Results and Discussion**
**Total Radioactive Residues and Extractability**

**Table A 1: Summary of Radioactive Residues Determined by Combustion Analysis in Rotational Crop Samples Grown in Soil Treated with [$^{14}$C]-SAN837**

| Crop Type | Commodity | Radioactive residues (mg/kg) | | |
|---|---|---|---|---|
| | | 30 DAT | 111 DAT | 285 DAT |
| Radish | Leaf | 0.279 | 0.040 | 0.002 |
| | Root | 0.028 | 0.017 | 0.002 |
| Spinach | Immature | 0.070 | 0.037 | 0.006 |
| | Mature | 0.027 | 0.019 | 0.002 |
| Wheat | Forage | 0.533 | 0.021 | 0.001 |
| | Hay | 0.886 | 0.097 | 0.016 |
| | Straw | 0.472 | 0.086 | 0.014 |
| | Grain | 0.090 | 0.036 | 0.011 |

**Table A 2: Summary of Total Radioactive Residues and Extractability (Solvent Extraction Method) in Rotational Crop Samples Grown in Soil Treated with [$^{14}$C]-SAN837**

| Crop | Commodity | Rotational Interval | Extracted[1] | | Debris[2] | | Total radioactive residue (TRR)[3] |
|---|---|---|---|---|---|---|---|
| | | | % TRR | mg/kg | % TRR | mg/kg | |
| Radish | Leaves | 30 DAT | 94.0 | 0.287 | 6.0 | 0.018 | 0.305 |
| | | 111 DAT[4] | 77.3 | 0.024 | 22.6 | 0.007 | 0.031 |
| | | 285 DAT | Not analysed as combusted residue <0.01 mg/kg | | | | |
| | Roots | 30 DAT | 82.8 | 0.022 | 17.1 | 0.005 | 0.027 |
| | | 111 DAT[4] | 91.4 | 0.011 | 8.6 | 0.001 | 0.012 |
| | | 285 DAT | Not analysed as combusted residue <0.01 mg/kg | | | | |
| Spinach | Immature | 30 DAT | 81.6 | 0.063 | 18.4 | 0.014 | 0.077 |
| | | 111 DAT[4] | 76.5 | 0.029 | 23.5 | 0.009 | 0.038 |
| | | 285 DAT | Not analysed as combusted residue <0.01 mg/kg | | | | |
| | Mature | 30 DAT | 80.6 | 0.025 | 19.4 | 0.006 | 0.031 |
| | | 111 DAT[4] | 72.5 | 0.017 | 27.6 | 0.006 | 0.023 |
| | | 285 DAT | Not analysed as combusted residue <0.01 mg/kg | | | | |
| Wheat | Forage | 30 DAT | 91.5 | 0.473 | 8.6 | 0.044 | 0.517 |
| | | 111 DAT[4] | 65.5 | 0.012 | 34.5 | 0.006 | 0.018 |
| | | 285 DAT | Not analysed as combusted residue <0.01 mg/kg | | | | |
| | Hay | 30 DAT | 78.2 | 0.619 | 21.7 | 0.172 | 0.791 |
| | | 111 DAT[4] | 63.2 | 0.049 | 36.8 | 0.028 | 0.077 |
| | | 285 DAT[4] | 53.1 | 0.012 | 46.8 | 0.011 | 0.023 |
| | Straw | 30 DAT | 74.6 | 0.354 | 25.4 | 0.121 | 0.475 |
| | | 111 DAT[4] | 33.3 | 0.026 | 66.7 | 0.053 | 0.079 |
| | | 285 DAT[5] | Not analysed for reason given in superscript | | | | |
| | Grain | 30 DAT | 28.4 | 0.025 | 71.7 | 0.064 | 0.089 |
| | | 111 DAT[4] | 16.9 | 0.007 | 83.1 | 0.034 | 0.041 |
| | | 285 DAT[5] | Not analysed for reason given in superscript | | | | |

NA – Value not applicable for the reason given in the superscript.

[1] Where values are present, the commodity was extracted sequentially with i) acetonitrile:water 4:1 v/v, ii) acetonitrile:water 1:1 v/v and acetone (drying solvent) and the resulting extracted residue given here.

[2] Residue remaining in crop debris following solvent extraction.

[3] TRR (mg/kg) calculated from total radioactivity (sum of extracted and debris radioactivity) and specific activity.

[4] Analysis of extracted residues indicated that they contained high proportions of polar and/or un-extractable radioactivity and consequently, additional subsamples of crop commodities were also extracted with hydrochloric acid under elevated temperature conditions (see next table).

[5] Samples not extracted with solvents as % TRR extracted by this method in the previous rotational interval was small. As a result, these samples were extracted using the acid extraction method (see next table).

**Table A 3: Summary of Total Radioactive Residues and Extractability (Acid Extraction Method) in Rotational Crop Samples Grown in Soil Treated with [$^{14}$C]-SAN837**

| Crop | Commodity | Rotational Interval | Extracted[1] | | Debris[2] | | Total radioactive residue (TRR)[3] |
|---|---|---|---|---|---|---|---|
| | | | % TRR | mg/kg | % TRR | mg/kg | |
| Radish | Leaves | 111 DAT | 91.7 | 0.023 | 8.2 | 0.002 | 0.025 |
| | Roots | 111 DAT | 80.7 | 0.011 | 19.3 | 0.003 | 0.014 |
| Spinach | Immature | 111 DAT | 81.2 | 0.028 | 18.8 | 0.006 | 0.034 |
| | Mature | 111 DAT | 79.1 | 0.017 | 20.9 | 0.005 | 0.022 |
| Wheat | Forage | 111 DAT | 68.1 | 0.014 | 31.9 | 0.007 | 0.021 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | Hay | 111 DAT | 61.9 | 0.065 | 38.2 | 0.040 | 0.105 |
| | | 285 DAT | 35.5 | 0.005 | 64.5 | 0.010 | 0.015 |
| | Straw | 111 DAT | 45.8 | 0.019 | 54.2 | 0.023 | 0.042 |
| | | 285 DAT | 49.6 | 0.007 | 50.4 | 0.008 | 0.015 |
| | Grain | 111 DAT | 86.6 | 0.030 | 13.4 | 0.005 | 0.035 |
| | | 285 DAT | 76.4 | 0.008 | 23.6 | 0.003 | 0.011 |

[1] The commodity was extracted sequentially with i) 1M HCl at 100°C for 1 hour followed by water at 100°C for 1 hour (all samples), followed by:
ii) water at ambient temperature (111 DAT wheat hay only), followed by:
iii) 6M HCl at 100°C for 1 hour (all except 285 DAT wheat grain, 111 DAT mature spinach and 111 DAT radish root)

[2] Residue remaining in crop debris following acid extraction.

[3] TRR (mg/kg) calculated from total radioactivity (sum of extracted and debris radioactivity) and specific activity.

**Characterisation and Identification of Residues**

The extracted radioactivity was fractionated and analysed as summarised in the previous section.
The identified components for each commodity are summarised in Table A 4.
Residue tables summarising whole analyses are given in Table A 5 to Table A 23.
In all these tables the identified residue values without parentheses represent the sum of both the free and conjugated forms of the component. Values within parentheses are the residue values for the conjugated form only.

**Table A 4: Summary of Identified Residues in Rotational Crop Samples Grown in Soil Treated with [$^{14}$C]-SAN837**

| Crop | Commodity | Rotational Interval (DAT) | Identified Residue | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | SAN837 | | NOA405873 | | NOA414746 | | R740230 | | 14C-Glucose | |
| | | | % TRR | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR | mg/kg | % TRR | mg/kg |
| Radish | Leaf | 30 | 67.0 (9.7) | 0.204 (0.029) | 1.4 (1.4) | 0.004 (0.004) | 4.1 (4.1) | 0.013 (0.013) | ND (ND) | ND (ND) | ND | ND |
| | | 111 | 32.0 (10.6) | 0.007 (0.003) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | 8.5 | 0.002 |
| | | 285 | Commodity not analysed (<0.01 mg/kg) | | | | | | | | | |
| | Root | 30 | 58.9 (ND) | 0.016 (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND | ND |
| | | 111 | 4.3 (ND) | 0.001 (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | 31.0 | 0.004 |
| | | 285 | Commodity not analysed (<0.01 mg/kg) | | | | | | | | | |
| Spinach | Immature | 30 | 57.9 (2.7) | 0.045 (0.002) | 2.0 (2.0) | 0.002 (0.002) | 1.0 (1.0) | 0.001 (0.001) | ND (ND) | ND (ND) | ND | ND |
| | | 111 | 19.7 (5.6) | 0.007 (0.002) | ND (ND) | ND (ND) | 3.2 (3.2) | 0.001 (0.001) | ND (ND) | ND (ND) | 7.9 | 0.003 |
| | | 285 | Commodity not analysed (<0.01 mg/kg) | | | | | | | | | |
| | Mature | 30 | 45.2 (ND) | 0.014 (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND | ND |
| | | 111 | 23.9 (14.4) | 0.005 (0.003) | 2.3 (2.3) | 0.001 (0.001) | 3.8 (3.8) | 0.001 (0.001) | ND (ND) | ND (ND) | NQ | NQ |
| | | 285 | Commodity not analysed (<0.01 mg/kg) | | | | | | | | | |
| Wheat | Forage | 30 | 20.7 (ND) | 0.107 (ND) | 50.4 (8.2) | 0.260 (0.042) | 6.4 (5.9) | 0.033 (0.033) | 1.6 (1.6) | 0.008 (0.008) | ND | ND |
| | | 111 | ND (ND) | ND (ND) | 1.1 (ND) | <0.001 (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | 14.0 | 0.003 |
| | | 285 | Commodity not analysed (<0.01 mg/kg) | | | | | | | | | |
| | Hay | 30 | 13.8 (2.3) | 0.109 (0.018) | 43.2 (20.8) | 0.342 (0.164) | 6.5 (6.5) | 0.052 (0.052) | 0.7 (0.7) | 0.006 (0.006) | 4.2 | 0.033 |
| | | 111 | 0.6 (<0.1) | 0.001 (<0.001) | 15.8 (9.2) | 0.017 (0.010) | 2.4 (2.4) | 0.003 (0.003) | ND (ND) | ND (ND) | 17.8 | 0.018 |
| | | 285 | ND (ND) | ND (ND) | ND (ND) | ND (ND) | 4.0 (4.0) | 0.001 (0.001) | ND (ND) | ND (ND) | NQ | NQ |
| | Straw | 30 | 0.4 (ND) | 0.002 (ND) | 56.3 (42.8) | 0.268 (0.204) | 3.0 (3.0) | 0.014 (0.014) | 1.9 (1.9) | 0.009 (0.009) | 3.9 | 0.018 |
| | | 111 | 7.4 (6.4) | 0.006 (0.005) | 5.7 (1.7) | 0.004 (0.001) | 0.9 (ND) | 0.001 (ND) | ND (ND) | ND (ND) | 9.2 | 0.018 |
| | | 285 | NA (NA) | NA (NA) | NA (NA) | NA (NA) | NA (NA) | NA (NA) | NA (NA) | NA (NA) | NA | NA |
| | Grain | 30 | 1.9 (0.1) | 0.002 (<0.001) | 5.5 (3.8) | 0.005 (0.003) | 0.9 (0.9) | 0.001 (0.001) | ND (ND) | ND (ND) | 41.7 | 0.037 |
| | | 111 | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | ND (ND) | 14.5 | 0.005 |
| | | 285 | NA (NA) | NA (NA) | NA (NA) | NA (NA) | NA (NA) | NA (NA) | NA (NA) | NA (NA) | NA | NA |

NA – Analytical fractions were produced but due to the diminishingly small residues present no chromatographic analysis was conducted
ND – Not detected
NQ – $^{14}$C-glucose not quantifiable due to the very low residues present but deemed to be present due to its detection either in the previous rotational interval or in a related (wheat) or maturity (spinach) commodities

**Table A 5: Summary of Identification and Characterisation of Radioactive Residues in 30 DAT Radish Leaf Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation (mg/kg)[1] | 0.305 | |
| | TRR from direct quantification (mg/kg)[2] | 0.279 | |
| | Percentage of TRR chromatographed (%)[3] | 74.9 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | SAN837 (parent) | 67.0 (9.7) | 0.204 (0.029) |
| | NOA405873 | 1.4 (1.4) | 0.004 (0.004) |

| | | | |
|---|---|---|---|
| | NOA414746 | 4.1<br>(4.1) | 0.013<br>(0.013) |
| | Unassigned[4] | 1.0 | 0.003 |
| | Baseline[5] | 1.4 | 0.004 |
| | Other fractions[6] | 2.8 | 0.009 |
| | Unextracted[7] | 6.0 | 0.018 |
| | Losses[8] | 16.3 | 0.050 |
| | Total | 100.0 | 0.305 |

**ID Accountability (%TRR) = 72.5**

[1] The total radioactive residue determined from the sum of radioactivity present in extract and debris fractions.
[2] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[3] The total radioactive residue that was chromatographed.
[4] Discrete unassigned radio-components that chromatographed away from the origin in 2D-TLC comprising at least two components, no individual one of which accounted for >0.6% TRR (>0.002 mg/kg).
[5] Polar radio-component(s) chromatographing on the origin in 2D-TLC.
[6] The total residue attributable to two other unanalysed fractions, the largest of which accounted for 2.5% TRR (0.008 mg/kg).
[7] Radioactivity remaining in sample debris following solvent extraction.
[8] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only.

**Table A 6: Summary of Identification and Characterisation of Radioactive Residues in 111 DAT Radish Leaf Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation (solvent), mg/kg[1] | 0.031 | |
| | TRR from summation (acid) mg/kg[2] | 0.024 | |
| | TRR from direct quantification (mg/kg)[3] | 0.040 | |
| | Percentage of TRR chromatographed (%)[4] | 75.0 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | SAN837 (parent) | 32.0<br>(10.6) | 0.007<br>(0.003) |
| | Unassigned[5] | 7.1 | 0.002 |
| | Baseline (including $^{14}$C-glucose)[6] | 35.7 | 0.008 |
| | Remainder[7] | 0.2 | <0.001 |
| | Unextracted[8] | 8.2 | 0.002 |
| | Losses[9] | 16.8 | 0.005 |
| | Total | 100.0 | 0.024 |
| | ID Accountability (%TRR) = 32.0 + 8.5 ($^{14}$C-glucose) = | 40.5 | |

[1] The total radioactive residue determined from the sum of radioactivity present in solvent extract and debris fractions
[2] The total radioactive residue determined from the sum of radioactivity present in acid extracts and debris fractions.
[3] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[4] The total radioactive residue that was chromatographed.
[5] Unassigned radio-components that chromatographed away from the origin in 2D-TLC comprising at least three components, no individual one of which accounted for >4.0% TRR (>0.001 mg/kg).
[6] Polar unassigned radio-component(s) derived from three fractions chromatographing on the origin in 2D-TLC but shown in stronger 1D-TLC to comprise multiple components including:
- $^{14}$C-glucose (8.5% TRR; 0.002 mg/kg).
- radio-component(s) persisting on the origin of the TLC plate (26.5% TRR; 0.006 mg/kg)
[7] Low level diffuse radioactive components of wide ranging polarity in 2D-TLC.
[8] Radioactivity remaining in sample debris following acid extraction.
[9] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only.

**Table A 7: Summary of Identification and Characterisation of Radioactive Residues in 30 DAT Radish Root Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation (mg/kg)[1] | 0.027 | |
| | TRR from direct quantification (mg/kg)[2] | 0.028 | |
| | Percentage of TRR chromatographed (%)[3] | 63.3 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | SAN837 (parent) | 58.9 | 0.016 |
| | Unassigned[4] | 4.4 | 0.001 |
| | Baseline[5] | ND | ND |
| | Other fractions[6] | 14.9 | 0.004 |
| | Unextracted[7] | 17.1 | 0.005 |
| | Losses[8] | 4.7 | 0.001 |
| | Total | 100.0 | 0.027 |

ID Accountability (%TRR) = 58.9

ND = not detected
[1] The total radioactive residue determined from the sum of radioactivity present in extract and debris fractions.
[2] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[3] The total radioactive residue that was chromatographed.
[4] Discrete unassigned radio-components that chromatographed away from the origin in 2D-TLC comprising at least three components, no individual one of which accounted for >2.1% TRR (>0.001 mg/kg).
[5] Polar radio-component(s) chromatographing on the origin in 2D-TLC.
[6] The total residue attributable to three other unanalysed fractions, the largest of which accounted for 13.6% TRR (0.004 mg/kg).
[7] Radioactivity remaining in sample debris following solvent extraction. Following acid hydrolysis (1M HCl; 100°C; 1 hour) of this residue a further 8.9% TRR (0.002 mg/kg) was released into the resulting acid hydrolysate (neither acid extracted or un-extracted residue analysed further).
[8] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified residue values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only. For radish root parent was only found in the free form, hence there are no values in parentheses.

**Table A 8: Summary of Identification and Characterisation of Radioactive Residues in 111 DAT Radish Root Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation (solvent), mg/kg[1] | 0.012 | |
| | TRR from summation (acid), mg/kg[2] | 0.014 | |
| | TRR from direct quantification (mg/kg)[3] | 0.017 | |
| | Percentage of TRR chromatographed (%)[4] | 47.3 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | Unassigned[5] | 10.0 | 0.001 |
| | Baseline (includes $^{14}$C-glucose)[6] | 36.4 | 0.005 |
| | Remainder[7] | 0.9 | <0.001 |
| | Other fraction (includes SAN837)[8] | 6.7 | 0.001 |
| | Unextracted[9] | 19.3 | 0.003 |
| | Losses[10] | 26.7 | 0.004 |
| | Total | 100.0 | 0.014 |
| | ID Accountability (%TRR) = 4.3 + 31.0 ($^{14}$C-glucose) = | 35.3 | |

[1] The total radioactive residue determined from the sum of radioactivity present in solvent extract and debris fractions
[2] The total radioactive residue determined from the sum of radioactivity present in acid extract and debris fractions.
[3] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[4] The total radioactive residue that was chromatographed.
[5] An unassigned radio-component that chromatographed away from the origin in 2D-TLC (10.0% TRR; (0.001 mg/kg).
[6] Polar radio-component(s), chromatographing on the origin in 2D-TLC but shown in stronger 1D-TLC to comprise at least three components the largest or which was characterised as $^{14}$C-glucose (31.0% TRR; 0.004 mg/kg)
[7] Low level diffuse radioactive components of wide ranging polarity in 2D-TLC.
[8] Residue contained in a single organic partition phase which was not analysed by TLC due to very low levels of radioactivity but is likely to comprise predominantly SAN837 (4.3% TRR; 0.001 mg/kg) detected in related fraction of the solvent extracted analysis.
[9] Radioactivity remaining in sample debris following acid extraction. Not characterised further due to the small residue present.
[10] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified residue values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only. For radish root parent was only found in the free form, hence there are no values in parentheses.

**Table A 9: Summary of Identification and Characterisation of Radioactive Residues in 30 DAT Immature Spinach Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation (mg/kg)[1] | 0.077 | |
| | TRR from direct quantification (mg/kg)[2] | 0.070 | |
| | Percentage of TRR chromatographed (%)[3] | 66.3 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | SAN837 (parent) | 57.9<br>(2.7) | 0.045<br>(0.002) |
| | NOA405873 | 2.0<br>(2.0) | 0.002<br>(0.002) |
| | NOA414746 | 1.0<br>(1.0) | 0.001<br>(0.001) |
| | Unassigned[4] | 4.4 | 0.003 |
| | Baseline[5] | 3.4 | 0.003 |
| | Other fractions[6] | 3.4 | 0.003 |
| | Unextracted[7] | 18.4 | 0.014 |
| | Losses[8] | 9.5 | 0.006 |
| | Total | 100.0 | 0.077 |

ID Accountability (%TRR) = 60.9

[1] The total radioactive residue determined from the sum of radioactivity present in extract and debris fractions.
[2] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[3] The total radioactive residue that was chromatographed.
[4] Discrete unassigned radio-components that chromatographed away from the origin in 2D-TLC comprising at least six components, no individual one of which accounted for >1.4% TRR (0.001 mg/kg).
[5] Polar radio-component(s) chromatographing on the origin in 2D-TLC.
[6] The total residue attributable to two other unanalysed fractions, the largest of which accounted for 2.4% TRR (0.002 mg/kg).
[7] Radioactivity remaining in sample debris following solvent extraction. Following acid hydrolysis (1M HCl; 100°C; 1 hour) of this residue a further 5% TRR (0.004 mg/kg) was released into the resulting acid hydrolysate (neither acid extracted or un-extracted residue analysed further).
[8] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified residue values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only.

**Table A 10: Summary of Identification and Characterisation of Radioactive Residues in 111 DAT Immature Spinach Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation (solvent), mg/kg[1] | 0.038 | |
| | TRR from summation (acid) mg/kg[2] | 0.033 | |
| | TRR from direct quantification (mg/kg)[3] | 0.037 | |
| | Percentage of TRR chromatographed (%)[4] | 65.6 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | SAN837 (parent) | 19.7<br>(5.6) | 0.007<br>(0.002) |
| | NOA414746 | 3.2<br>(3.2) | 0.001<br>(0.001) |
| | Unassigned[5] | 7.4 | 0.003 |
| | Baseline (including $^{14}$C-glucose)[6] | 34.3 | 0.011 |
| | Remainder[7] | 0.8 | <0.001 |
| | Unextracted[8] | 18.8 | 0.006 |
| | Losses[9] | 15.6 | 0.005 |
| | Total | 100.0 | 0.033 |
| | ID Accountability (%TRR) = 22.9 + 7.9 ($^{14}$C-glucose) = | 30.8 | |

[1] The total radioactive residue determined from the sum of radioactivity present in solvent extract and debris fractions
[2] The total radioactive residue determined from the sum of radioactivity present in acid extract and debris fractions.
[3] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[4] The total radioactive residue that was chromatographed.
[5] Unassigned radiocomponents that chromatographed away from the origin in 2D-TLC comprising at least three components, no individual one of which accounted for > 2.5% TRR (>0.001 mg/kg).
[6] Polar radiocomponents from 3 fractions, chromatographing on the origin in 2D-TLC but shown in stronger 1D-TLC to comprise at least two components, $^{14}$C-glucose (7.9% TRR; 0.003 mg/kg) and a polar unassigned radiocomponent persisting on the origin in 1D-TLC (25.9% TRR; 0.008 mg/kg).
[7] Low level diffuse radioactive components of wide ranging polarity in 2D-TLC.
[8] Radioactivity remaining in sample debris following acid extraction. Not characterised further due to the small residue present.
[9] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radiocomponent reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified residue values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only.

**Table A 11: Summary of Identification and Characterisation of Radioactive Residues in 30 DAT Mature Spinach Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation (mg/kg)[1] | 0.031 | |
| | TRR from direct quantification (mg/kg)[2] | 0.027 | |
| | Percentage of TRR chromatographed (%)[3] | 45.2 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | SAN837 (parent) | 45.2 | 0.014 |
| | Unassigned[4] | ND | ND |
| | Baseline[5] | ND | ND |
| | Other fractions[6] | 12.1 | 0.004 |
| | Unextracted[7] | 19.4 | 0.006 |
| | Losses[8] | 23.3 | 0.007 |
| | Total | 100.0 | 0.031 |
| | ID Accountability (%TRR) = | 45.2 | |

ND = not detected
[1] The total radioactive residue determined from the sum of radioactivity present in extract and debris fractions.
[2] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[3] The total radioactive residue that was chromatographed.
[4] Discrete unassigned radio-components that chromatographed away from the origin in 2D-TLC (none detected).
[5] Polar radio-component(s) chromatographing on the origin in 2D-TLC (none detected).
[6] The total residue attributable to three other unanalysed fractions, the largest of which accounted for 8.2% TRR (0.003 mg/kg).
[7] Radioactivity remaining in sample debris following solvent extraction.
[8] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified residue values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only. For radish root parent was only found in the free form, hence there are no values in parentheses.

**Table A 12: Summary of Identification and Characterisation of Radioactive Residues in 111 DAT Mature Spinach Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation (solvent), mg/kg[1] | 0.023 | |
| | TRR from summation (acid) mg/kg[2] | 0.022 | |
| | TRR from direct quantification (mg/kg)[3] | 0.019 | |
| | Percentage of TRR chromatographed (%)[4] | 52.4 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | SAN837 (parent) | 23.9<br>(14.4) | 0.005<br>(0.003) |
| | NOA405873 | 2.3<br>(2.3) | 0.001<br>(0.001) |
| | NOA414746 | 3.8<br>(3.8) | 0.001<br>(0.001) |
| | Baseline[5] | 22.0 | 0.005 |
| | Remainder[6] | 0.4 | <0.001 |
| | Other fractions[7] | 17.8 | 0.004 |
| | Unextracted[8] | 20.9 | 0.005 |
| | Losses[9] | 8.9 | 0.001 |
| | Total | 100.0 | 0.022 |
| | ID Accountability (%TRR) = | 30.0 | |

[1] The total radioactive residue determined from the sum of radioactivity present in solvent extract and debris fractions
[2] The total radioactive residue determined from the sum of radioactivity present in acid extract and debris fractions.
[3] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[4] The total radioactive residue that was chromatographed.
[5] Polar unassigned radio-component(s) chromatographing on the origin in 2D-TLC. In the 111 DAT immature spinach analysis this radioactivity was characterised to be at least two components by chromatographing in the stronger 1D-TLC, one of which was $^{14}$C-glucose and the other, polar material persisting on the origin of the TLC plate. The same chromatographic analysis in the 111 DAT mature spinach showed the nature of this material to be similar, although accurate quantification of the relative amounts of $^{14}$C-glucose and origin material was not possible due to the very small amounts of radioactivity involved and high amounts of endogenous co-extractives present.
[6] Low level diffuse radioactive components of wide ranging polarity in 2D-TLC.
[7] Residue contained in a single aqueous partition phase which was not analysed by chromatography (very low levels of radioactivity). The corresponding fraction in the immature spinach showed the nature of this fraction to comprise at least two components including $^{14}$C-glucose.
[8] Radioactivity remaining in sample debris following acid extraction. Not characterised further due to small residue present.
[9] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only.

**Table A 13: Summary of Identification and Characterisation of Radioactive Residues in 30 DAT Wheat Forage Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation (mg/kg)[1] | 0.517 | |
| | TRR from direct quantification (mg/kg)[2] | 0.533 | |
| | Percentage of TRR chromatographed (%)[3] | 87.1 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | SAN837 (parent) | 20.7 | 0.107 |
| | NOA405873 | 50.4<br>(8.2) | 0.260<br>(0.042) |
| | NOA414746 | 6.4<br>(5.9) | 0.033<br>(0.030) |
| | R470230 | 1.6<br>(1.6) | 0.008<br>(0.008) |
| | Baseline[4] | 8.0 | 0.041 |
| | Other fractions[5] | 1.0 | 0.005 |
| | Unextracted[6] | 8.6 | 0.044 |
| | Losses[7] | 3.3 | 0.019 |
| | Total | 100.0 | 0.517 |
| | ID Accountability (%TRR) = | 79.1 | |

ND = not detected
[1] The total radioactive residue determined from the sum of radioactivity present in extract and debris fractions.
[2] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[3] The total radioactive residue that was chromatographed.
[4] Polar radiocomponent(s) chromatographing on the origin in 2D-TLC.
[5] The total residue attributable to one other unanalysed extract fraction.
[6] Radioactivity remaining in sample debris following extraction with solvent.
[7] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radiocomponent reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified residue values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only.

**Table A 14: Summary of Identification and Characterisation of Radioactive Residues in 111 DAT Wheat Forage Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation, solvent (mg/kg)[1] | 0.018 | |
| | TRR from summation, acid (mg/kg)[2] | 0.021 | |
| | TRR from direct quantification (mg/kg)[3] | 0.021 | |
| | Percentage of TRR chromatographed (%)[4] | 63.5 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | NOA405873 | 1.1 | <0.001 |
| | Unassigned[5] | 2.9 | <0.001 |
| | Baseline (including $^{14}$C-glucose)[6] | 58.4 | 0.013 |
| | Remainder[7] | 1.1 | <0.001 |
| | Unextracted[8] | 31.9 | 0.007 |
| | Losses[9] | 4.6 | 0.001 |
| | Total | 100 | 0.021 |
| | ID Accountability (%TRR) = 1.1 + 14.0 ($^{14}$C-glucose) = | 15.1 | |

[1] The total radioactive residue determined from the sum of radioactivity present in solvent extract and debris fractions.
[2] The total radioactive residue determined from the sum of radioactivity present in acid extract and debris fractions.
[3] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[4] The total radioactive residue that was chromatographed.
[5] Unassigned radio-components derived from three separate fractions that chromatographed away from the origin in 2D-TLC comprising at least one component, 2.9% TRR (<0.001 mg/kg).
[6] Polar radio-component(s) chromatographing on the origin in 2D-TLC but shown in stronger 1D-TLC to comprise multiple components, the largest individual ones of which were characterised to be:
- $^{14}$C-glucose (14.0% TRR; 0.003 mg/kg);
- at least three unassigned radio-components chromatographing away from the origin, the largest of which was 10.6% TRR (0.002 mg/kg);
- polar material persisting on the origin of the TLC plate (7.0% TRR; 0.002 mg/kg)
[7] Low level diffuse radioactive components of wide ranging polarity in 2D-TLC SS5.
[8] Radioactivity remaining in sample debris following acid extraction and likely to comprise predominantly naturally incorporated residues based on precedent characterisation in 30 DAT hay and straw.
[9] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified residue values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only. For wheat forage, parent was only found in the free form, hence there are no values in parentheses.

**Table A 15: Summary of Identification and Characterisation of Radioactive Residues in 30 DAT Wheat Hay Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation (mg/kg)[1] | 0.792 | |
| | TRR from direct quantification (mg/kg)[2] | 0.886 | |
| | Percentage of TRR chromatographed (%)[3] | 70.0 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | SAN837 (parent) | 13.8<br>(2.3) | 0.109<br>(0.018) |
| | NOA405873 | 43.2<br>(20.8) | 0.342<br>(0.164) |
| | NOA414746 | 6.5<br>(6.5) | 0.052<br>(0.052) |
| | R470230 | 0.7<br>(0.7) | 0.006<br>(0.006) |
| | Unassigned[4] | 2.3 | 0.018 |
| | Baseline[5] | 3.5 | 0.028 |
| | Other fractions[6] | NA | NA |
| | Unextracted (includes $^{14}$C-glucose)[7] | 21.7 | 0.172 |
| | Losses[8] | 9.0 | 0.071 |
| | Total | 100.0 | 0.792 |
| | ID Accountability (%TRR) = 64.2 + 4.2 ($^{14}$C-glucose) = | 68.4 | |

NA = Not Applicable
[1] The total radioactive residue determined from the sum of radioactivity present in extract and debris fractions.
[2] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[3] The total radioactive residue that was chromatographed.
[4] Discrete unassigned radio-components that chromatographed away from the origin in 2D-TLC comprising at least three components, no individual one of which accounted for >1.1% TRR (>0.009 mg/kg).
[5] Polar radio-component(s) chromatographing on the origin in 2D-TLC.
[6] The total residue attributable to other unanalysed fraction(s); not applicable as all fractions were analysed.
[7] Radioactivity remaining in sample debris following solvent extraction. Characterised further following progressively harsher hydrochloric acid hydrolyses to include naturally incorporated radioactivity ($^{14}$C-glucose: 4.2% TRR; 0.033 mg/kg) and a mixture of unassigned radio-components (no single one of which >1.1% TRR; 0.009 mg/kg) and un-extractable residue (6.6% TRR; 0.052 mg/kg).
[8] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified residue values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only.

**Table A 16: Summary of Identification and Characterisation of Radioactive Residues in 111 DAT Wheat Hay Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation, solvent (mg/kg)[1] | 0.076 | |
| | TRR from summation, acid (mg/kg)[2] | 0.105 | |
| | TRR from direct quantification (mg/kg)[3] | 0.097 | |
| | Percentage of TRR chromatographed (%)[4] | 52.7 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | SAN837 (parent) | 0.6<br>(<0.1) | 0.001<br>(<0.001) |
| | NOA405873 | 15.8<br>(9.2) | 0.017<br>(0.010) |
| | NOA414746 | 2.4<br>(2.4) | 0.003<br>(0.003) |
| | Unassigned[5] | 6.9 | 0.006 |
| | Baseline (including $^{14}$C-glucose)[6] | 24.9 | 0.026 |
| | Remainder[7] | 2.1 | 0.002 |
| | Other fractions[8] | 5.0 | 0.005 |
| | Unextracted[9] | 38.2 | 0.040 |
| | Losses[10] | 4.1 | 0.005 |
| | Total | 100.0 | 0.105 |
| | ID Accountability (%TRR) = 18.8 + 17.8 ($^{14}$C-glucose) = | 36.6 | |

[1] The total radioactive residue determined from the sum of radioactivity present in solvent extract and debris fractions.
[2] The total radioactive residue determined from the sum of radioactivity present in acid extract and debris fractions.
[3] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[4] The total radioactive residue that was chromatographed.
[5] Unassigned radio-components derived from 3 separate fractions that chromatographed away from the origin in 2D-TLC comprising at least 4 components, the largest individual one of which accounting for 3.1% TRR (0.003 mg/kg).
[6] Polar radio-components derived from three separate fractions that chromatographed on the origin in 2D-TLC but shown in stronger 1D-TLC to comprise multiple components, the largest individual ones of which were characterised to be:
- $^{14}$C-glucose (17.8% TRR; 0.018 mg/kg);
- polar unassigned radioactivity persisting on the origin of the TLC (7.0% TRR; 0.007 mg/kg)
[7] Low level diffuse radioactive components of wide ranging polarity in 2D-TLC.
[8] Residue contained in a single aqueous extract which was not analysed by chromatography due to high levels of endogenous co-extractives and relatively low levels of radioactivity.
[9] Radioactivity remaining in sample debris following acid extraction and likely to comprise predominantly naturally incorporated residues based on precedent characterisation.
[10] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified residue values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only.

**Table A 17: Summary of Identification and Characterisation of Radioactive Residues in 285 DAT Wheat Hay Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation, solvent (mg/kg)[1] | 0.023 | |
| | TRR from summation, acid (mg/kg)[2] | 0.015 | |
| | TRR from direct quantification (mg/kg)[3] | 0.016 | |
| | Percentage of TRR chromatographed (%)[4] | 27.3 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | NOA414746 | 4.0<br>(4.0) | 0.001<br>(0.001) |
| | Unassigned[5] | 15.5 | 0.002 |
| | Baseline[6] | 7.5 | 0.001 |
| | Remainder[7] | 0.3 | <0.001 |
| | Other fractions[8] | 12.3 | 0.002 |
| | Unextracted[9] | 64.5 | 0.010 |
| | Losses[10] | -4.1 | -0.001 |
| | Total | 100 | 0.015 |

| | |
|---|---|
| ID Accountability (%TRR) | 4.0 |

[1] The total radioactive residue determined from the sum of radioactivity present in solvent extract and debris fractions.
[2] The total radioactive residue determined from the sum of radioactivity present in acid extract and debris fractions.
[3] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[4] The total radioactive residue that was chromatographed.
[5] Unassigned radio-components derived from 1 fraction that chromatographed away from the origin in 2D-TLC comprising at least 2 components, the largest individual one of which accounted for 8.7% TRR (0.001 mg/kg).
[6] Polar radio-components chromatographing on the origin in 2D-TLC.
[7] Low level diffuse radioactive components of wide ranging polarity in 2D-TLC.
[8] Residue contained in two fractions (the largest 8.6% TRR; 0.001 mg/kg) which were not analysed by chromatography due to high levels of endogenous co-extractives and relatively low levels of radioactivity.
[9] Radioactivity remaining in sample debris following acid extraction. Not analysed further as considered likely to comprise predominantly naturally incorporated radioactivity as characterised in precedent rotational interval wheat extractable and debris analyses.
[10] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified residue values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only.

**Table A 18: Summary of Identification and Characterisation of Radioactive Residues in 30 DAT Wheat Straw Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation (mg/kg)[1] | 0.476 | |
| | TRR from direct quantification (mg/kg)[2] | 0.472 | |
| | Percentage of TRR chromatographed (%)[3] | 75.1 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | SAN837 (parent) | 0.4 | 0.002 |
| | NOA405873 | 56.3<br>(42.8) | 0.268<br>(0.204) |
| | NOA414746 | 3.0<br>(3.0) | 0.014<br>(0.014) |
| | R470230 | 1.9<br>(1.9) | 0.009<br>(0.009) |
| | Unassigned[4] | 5.9 | 0.028 |
| | Baseline[5] | 7.6 | 0.036 |
| | Unextracted (including $^{14}$C-glucose)[6] | 25.4 | 0.121 |
| | Losses[7] | 1.4 | 0.007 |
| | Total | 100.0 | 0.476 |
| | ID Accountability (%TRR) = 61.6 + 3.9 ($^{14}$C-glucose) = | 65.5 | |

[1] The total radioactive residue determined from the sum of radioactivity present in extract and debris fractions.
[2] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[3] The total radioactive residue that was chromatographed.
[4] Discrete unassigned radio-components that chromatographed away from the origin in 2D-TLC comprising at least two components, no individual one of which accounted for >3.7% TRR (>0.018 mg/kg).
[5] Polar radio-component(s) chromatographing on the origin in 2D-TLC.
[6] Radioactivity remaining in sample debris following solvent extraction. Characterised further following progressively harsher hydrochloric acid hydrolyses to contain naturally incorporated radioactivity ($^{14}$C-glucose: 3.9% TRR; 0.018 mg/kg), and a mixture of unassigned radio-components (no single one of which >1.3% TRR; 0.006 mg/kg) and un-extractable residue (15.8% TRR; 0.075 mg/kg).
[7] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified residue values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only.

**Table A 19: Summary of Identification and Characterisation of Radioactive Residues in 111 DAT Wheat Straw Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation (solvent), mg/kg[1] | 0.080 | |
| | TRR from direct quantification (mg/kg)[2] | 0.086 | |
| | Percentage of TRR chromatographed (%)[3] | 17.0 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | SAN837 (parent) | 1.0 | 0.001 |
| | NOA405873 | 5.7<br>(1.7) | 0.004<br>(0.001) |
| | NOA414746 | 0.9 | 0.001 |
| | Unassigned[4] | 4.0 | 0.003 |
| | Baseline (predominantly $^{14}$C-glucose)[5] | 5.9 | 0.005 |
| | Remainder[6] | 0.1 | <0.001 |
| | Other fractions[7] | 6.3 | 0.005 |
| | Un-extracted (including SAN837; $^{14}$C-glucose)[8] | 66.7 | 0.053 |
| | Losses[9] | 9.4 | 0.008 |
| | Total | 100.0 | 0.080 |

ID Accountability (%TRR) = 7.0 + 9.2 ($^{14}$C-glucose) = 16.2

NA = not applicable
[1] The total radioactive residue determined from the sum of radioactivity present in solvent extract and debris fractions.
[2] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[3] The total radioactive residue that was chromatographed.
[4] Unassigned radio-components that chromatographed away from the origin in 2D-TLC comprising at least two components, the largest individual one of which accounting for 3.0% TRR (0.002 mg/kg).
[5] Polar radio-components that chromatographing on the origin in 2D-TLC SS5 but shown in stronger 1D-TLC to comprise predominantly $^{14}$C-glucose (5.9 % TRR; 0.005 mg/kg)
[6] Low level diffuse radioactive components of wide ranging polarity in 2D-TLC.
[7] Residue present in two separate un-chromatographed extracts the largest of which was 4.8% TRR (0.004 mg/kg).
[8] - Radioactivity remaining in straw debris following solvent extraction and further characterised by successive acid hydrolysis procedures to contain $^{14}$C-glucose (3.3% TRR; 0.003 mg/kg), SAN837 (6.4% TRR; 0.005 mg/kg), multiple other unassigned polar radio-components (no single one of which >6.0% TRR; 0.005 mg/kg), a precipitate fraction (11.6% TRR; 0.009 mg/kg) and un-extractable residue persisting in straw debris (23.1% TRR; 0.018 mg/kg).
[9] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified residue values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only.

**Table A 20: Summary of Identification and Characterisation of Radioactive Residues in 285 DAT Wheat Straw Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | |
|---|---|---|
| TRR from summation, acid (mg/kg)[1] | 0.015 | |
| TRR from direct quantification (mg/kg)[2] | 0.014 | |
| Percentage of TRR chromatographed (%)[3] | 0.0 | |
| Component | %TRR | mg/kg |
| Organic B[4] | 3.6 | 0.001 |
| Aqueous A[4] | 16.1 | 0.002 |
| 6M HCl[5] | 11.8 | 0.002 |
| Unextracted[6] | 50.4 | 0.008 |
| Losses[7] | 18.1 | 0.002 |
| Total | 100 | 0.015 |

[1] – The total radioactive residue determined from the sum of radioactivity present in solvent extract and debris fractions.
[2] - The radioactive residue determined by direct quantification by combustion analysis/LSC.
[3] - The total radioactive residue that was chromatographed.
[4] - Aqueous and organic partition fractions of acidic extracted radioactivity. Not chromatographed due to the very small residue and high endogenous co-extractive content. Likely to comprise significant proportions of naturally incorporated residue based on the nature of previous rotational interval wheat analyses.
[5] - A single 6M HCl extract. Not chromatographed due to the very small residue and high endogenous co-extractive content. Likely to comprise significant proportions of naturally incorporated residue based on the nature of previous rotational interval wheat analyses.
[6] - Radioactivity remaining in sample debris following acid extraction. Not analysed further as deemed likely to comprise predominantly naturally incorporated radioactivity as characterised from previous rotational interval wheat debris analyses.
[7] - The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

**Table A 21: Summary of Identification and Characterisation of Radioactive Residues in 30 DAT Wheat Grain Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation (mg/kg)[1] | 0.089 | |
| | TRR from direct quantification (mg/kg)[2] | 0.090 | |
| | Percentage of TRR chromatographed (%)[3] | 8.4 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | SAN837 (parent) | 1.9<br>(0.1) | 0.002<br>(<0.001) |
| | NOA405873 | 5.5<br>(3.8) | 0.005<br>(0.003) |
| | NOA414746 | 0.9<br>(0.9) | 0.001<br>(0.001) |
| | Remainder[4] | 0.1 | <0.001 |
| | Other fractions[5] | 5.5 | 0.005 |
| | Un-extracted (includes $^{14}$C-glucose)[6] | 71.7 | 0.064 |
| | Losses[7] | 14.4 | 0.012 |
| | Total | 100.0 | 0.089 |
| | ID Accountability (%TRR) = 8.3 + 41.7 ($^{14}$C-glucose) = | 50.0 | |

NA = not applicable
[1] The total radioactive residue determined from the sum of radioactivity present in extract and debris fractions.
[2] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[3] The total radioactive residue that was chromatographed.
[4] Low level diffuse radioactive components of wide ranging polarity in 2D-TLC.
[5] The residue attributable to an un-chromatographed post acid hydrolysis aqueous partition phase, likely to contain $^{14}$C-glucose derived from starch hydrolysis based on characterisation of debris residue.
[6] Radioactivity remaining in sample debris following solvent extraction. Characterised further, following progressively harsher hydrochloric acid hydrolyses, to be predominantly naturally incorporated radioactivity ($^{14}$C-glucose: 41.7% TRR; 0.037 mg/kg) and three unassigned fractions (not chromatographed due to the low associated residue and high endogenous content; the largest of which was 6.5% TRR (0.006 mg/kg).
[7] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

The values in the table are the levels of radioactive residue for each radio-component reported and expressed as %TRR and mg dicamba equivalents per kilogram crop sample. The identified residue values without parentheses are the sum of both free and conjugated forms of the residue. The identified residue values within parentheses indicate the proportion of the residues found in the conjugated form only.

**Table A 22: Summary of Identification and Characterisation of Radioactive Residues in 111 DAT Wheat Grain Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | | |
|---|---|---|---|
| | TRR from summation, solvent (mg/kg)[1] | 0.041 | |
| | TRR from summation, acid (mg/kg)[2] | 0.035 | |
| | TRR from direct quantification (mg/kg)[3] | 0.036 | |
| | Percentage of TRR chromatographed (%)[4] | 80.2 | |
| Origin of Components | Component | %TRR | mg/kg |
| Chromatographed | Unassigned[5] | 4.2 | 0.001 |
| | Baseline (including $^{14}$C-glucose)[6] | 75.6 | 0.026 |
| | Remainder[7] | 0.4 | <0.001 |
| | Unextracted[8] | 13.4 | 0.005 |
| | Losses[9] | 6.4 | 0.003 |
| | Total | 100.0 | 0.035 |

ID Accountability (%TRR) = 14.5 ($^{14}$C-glucose) = 14.5

[1] The total radioactive residue determined from the sum of radioactivity present in solvent extract and debris fractions.
[2] The total radioactive residue determined from the sum of radioactivity present in acid extract and debris fractions.
[3] The radioactive residue determined by direct quantification by combustion analysis/LSC.
[4] The total radioactive residue that was chromatographed.
[5] An unassigned radio-component derived from one fraction that chromatographed away from the origin in 2D-TLC.
[6] Polar radio-components chromatographing on the origin in 2D-TLC but shown in stronger 1D-TLC to comprise multiple components (including $^{14}$C-glucose: 14.5% TRR; 0.005 mg/kg) and others likely to be associated to naturally incorporated endogenous components based on the characterisation evident from the corresponding 30 DAT wheat grain analysis.
[7] Many low level diffuse radioactive components of wide ranging polarity in 2D-TLC.
[8] Radioactivity remaining in sample debris following acid extraction and likely to comprise predominantly naturally incorporated residues based on precedent characterisation.
[9] The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

**Table A 23: Summary of Identification and Characterisation of Radioactive Residues in 285 DAT Wheat Grain Grown in Soil Treated with [$^{14}$C]-SAN837**

| | | |
|---|---|---|
| TRR from summation, acid (mg/kg)[1] | 0.011 | |
| TRR from direct quantification (mg/kg)[2] | 0.011 | |
| Percentage of TRR chromatographed (%)[3] | 0.0 | |
| Component | %TRR | mg/kg |
| Organic B[4] | 30.7 | 0.003 |
| Aqueous A[4] | 37.7 | 0.004 |
| Unextracted[5] | 23.6 | 0.003 |
| Losses[6] | 8.0 | 0.001 |
| Total | 100.0 | 0.011 |

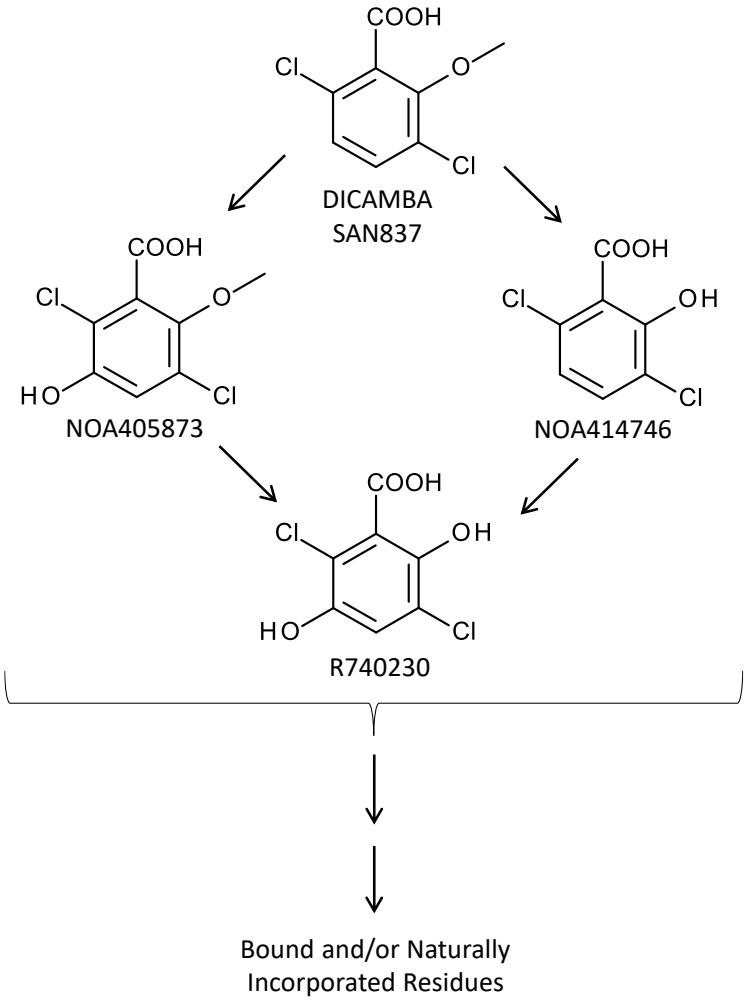
[1] – The total radioactive residue determined from the sum of radioactivity present in solvent extract and debris fractions.
[2] - The radioactive residue determined by direct quantification by combustion analysis/LSC.
[3] - The total radioactive residue that was chromatographed.
[4] - Aqueous and organic partition fractions of acidic extracted radioactivity. Not chromatographed due to the small residue and high endogenous co-extractive content. Likely to comprise significant proportions of naturally incorporated residue based on the nature of previous rotational interval wheat analyses.
[5] - Radioactivity remaining in sample debris following acid extraction. Not analysed further as deemed likely to comprise predominantly naturally incorporated radioactivity as characterised from previous rotational interval wheat debris analyses.
[6] - The net cumulative incremental losses (+ve) or gains (-ve) during analysis. Calculated as 100% TRR - (sum of all components % TRR).

**Storage Stability**
Chromatographic profiles of all initial crop extracts were obtained within 6 months.
Comparison of the initial and final radio-component profiles obtained from the selected samples showed that no significant change in the profiles had occurred during the interim period of storage.

**Radio-validation of the Residue Analytical Method**
Samples of forage and hay from the 30 day rotational interval were extracted as per the prescribed residue analytical method (GRM022.07A – see M-CA Section 4).
Extractability of the radioactive residue from forage and hay was always ≥ 78.2% TRR whether using the metabolism method or the prescribed residue extraction method. For both commodities, the relative efficiency of the residue extraction method was found to be high (Forage: 114.6%; Hay: 104.1%) when compared to the metabolism extraction procedure. These data are presented in Table A 24.
Furthermore when radioactivity extracted by the residue method was analysed by 2D-TLC the levels of parent (SAN837) and metabolites NOA40587 and NOA414746 present were found to be in close agreement in all instances with the corresponding levels determined by the metabolism methodology (relative residue levels 93.8 – 110.1%; these data are presented in Table A 25.

**Proposed Metabolic Pathway**
A metabolic pathway for dicamba (SAN837) in rotational crops is given in Figure A 1.

**Figure A 1: Proposed Metabolic Pathway of Dicamba (SAN837) in Rotational Crops**




SAN837, NOA405873 (5-OH-dicamba) and NOA414746 (DCSA) found in both the free and β-glucoside conjugated forms. R740230 found in the β-glucoside conjugated form only.

**Table A 24: Comparison of Solvent Extractabilities for Metabolism and Residue Analytical Methods**

| Commodity | Components | Metabolism Method | | Residue Method | | Relative efficiency of extraction[1] |
|---|---|---|---|---|---|---|
| | | %TRR | mg/kg | %TRR | mg/kg | |
| Forage | Extract | 91.5 | 0.473 | 104.9 | 0.559 | 114.6 |
| Hay | Extract | 78.2 | 0.619 | 81.4 | 0.721 | 104.1 |

[1] – (Residue method %TRR/Metabolism method %TRR) x 100

**Table A 25: Comparison of Parent (SAN837) and Metabolite (NOA405873 and NOA414746) Residue Levels in Forage and Hay Following Analysis Using Metabolism and Residue Analytical Methods**

| Commodity | Components | Metabolism Method | | Residue Method | | Relative residue levels detected[1] |
|---|---|---|---|---|---|---|
| | | %TRR | mg/kg | %TRR | mg/kg | |
| Forage | SAN837 | 20.7 | 0.107 | 21.7 | 0.116 | 104.8 |
| | NOA405873 | 50.4 | 0.260 | 55.5 | 0.296 | 110.1 |
| | NOA414746 | 6.4 | 0.033 | 6.0 | 0.032 | 93.8 |
| Hay | SAN837 | 13.8 | 0.109 | 14.5 | 0.128 | 105.1 |
| | NOA405873 | 43.2 | 0.342 | 45.5 | 0.403 | 105.3 |
| | NOA414746 | 6.5 | 0.052 | 6.3 | 0.056 | 96.9 |

[1] – (Residue method %TRR/Metabolism method %TRR) x 100

## Conclusions

Following a single treatment of [phenyl-U-$^{14}$C]-SAN837 to bare soil at a rate of 480 g acid equivalents/ha, radish, spinach and wheat were sown in the soil after periods of 30, 111 and 285 days (the rotational intervals) after treatment (DAT). Samples of immature and mature spinach, radish root and leaves and wheat forage, hay, straw and grain were subsequently taken for analysis.

The radioactive residue in all crop commodities was initially determined by combustion analysis. In all crop commodities, radioactive residues were highest at the 30 DAT rotational interval (0.027-0.886 mg/kg) but decreased progressively and markedly in subsequent rotational intervals (111 DAT: 0.017–0.090 mg/kg; 285 DAT: 0.001–0.017 mg/kg).

Crop commodities containing radioactive residues < 0.01 mg/kg by combustion analysis were not analysed further (285 DAT radish leaves (0.002 mg/kg), radish roots (0.002 mg/kg), immature spinach (0.006 mg/kg), mature spinach (0.002 mg/kg) and wheat forage (0.001 mg/kg).

All other commodities were taken forward for extraction with organic solvents (30 DAT samples), and/or extracted with mineral acid under elevated temperature conditions (111 DAT and 285 DAT samples). Principal residue fractions derived from subsequent analysis of extracted radioactivity were analysed by chromatography to determine the nature of constituent residues.

The results show that:

- Radioactive residues observed in 30 DAT rotational commodities declined rapidly and progressively in subsequent 111 DAT and 285 DAT commodities.
- In 30 DAT rotational crops :
    - Parent was the principal residue identified in radish and spinach commodities (≤67% TRR; ≤0.204 mg/kg) and was a major residue in wheat forage and hay (≤20.7% TRR; ≤0.109 mg/kg) but was only a minor residue in wheat straw and grain (≤1.9% TRR; 0.002 mg/kg).
    - NOA405873 was the principal identified metabolite in wheat, the residue levels of which were greatest in forage, hay and straw (≤56.3% TRR; ≤0.342 mg/kg) but were much lower in all other commodities (≤5.5% TRR; ≤0.005 mg/kg).
    - Residue levels of the other two identified residues NOA414746 (≤6.5% TRR; ≤0.052 mg/kg) and R740230 (≤1.9% TRR; ≤0.009% TRR) were generally much lower than NOA405873.
- By the time of 111 DAT rotational crop harvests, residues of parent and all identified metabolites

had declined to ≤0.007 mg/kg (except for NOA405873 in wheat hay, 0.017 mg/kg).
- By the time of 285 DAT rotational crop harvests, residues of parent and all metabolites identified above were not detectable (except for NOA414746 in wheat hay, 0.001 mg/kg).
- The decline in parent and identified metabolite residues in successive rotational intervals was accompanied by an increase in the proportion of the total radioactive residue associated with naturally incorporated radioactivity (≤ 41.7% TRR identified as $^{14}$C-glucose).

The observed metabolites of dicamba (SAN837) were generated via:
- hydroxylation of the aromatic carbon para to the methoxy functionality of dicamba to produce NOA405873
- demethylation of the methoxy functionality of dicamba to produce NOA414746
- either, demethylation of the methoxy functionality of NOA405873, and/or hydroxylation of the aromatic carbon para to the existing hydroxy functionality of NOA414746 to produce, in both instances, the dihydroxylated metabolite R740230
- glucose conjugation of both dicamba and the above metabolites

## A 2.1.2.1.3 Nature of residues in processed commodities

## A 2.1.2.1.3.1 Study TK0207450

| | |
|---|---|
| Comments of zRMS: | The effect of processing on the nature of 5-OH-dicamba was investigated. A study was conducted simulating representative hydrolytic conditions for pasteurisation (20 minutes at 90°C, pH 4), boiling/brewing/baking (60 minutes at 100°C, pH 5) and sterilisation (20 minutes at 120°C, pH 6).<br>Total recoveries ranged from 95.8 and 97.7 % in control samples and from 96.0 to 97.5 % in hydrolysate samples indicating no degradation of dicamba.<br>5-OH-dicamba was hydrolytically stable under conditions representative of pasteurisation, baking, brewing, boiling and sterilisation.<br>No breakdown or reaction products were formed during hydrolysis of dicamba or 5-OH-dicamba under representative processing conditions. The relevant residue for enforcement and risk assessment in processed commodities is therefore expected to be the same as for primary crops.<br>The study is accepted. |

| | |
|---|---|
| Reference: | KCA1 6.5.1/01 |
| Report | Yeomans P, 2015. NOA405873 – Simulated Processing Practices: Aqueous Hydrolysis of [14C] 5- Hydroxy-Dicamba at 90, 100 and 120°C. Report Number: 3200933. Smithers Viscient (ESG) Ltd, UK.<br>Syngenta Report No. TK0207450)<br>Syngenta File No. NOA405873_10000<br>GLP, unpublished |
| Guideline(s): | OECD Guidelines for the Testing of Chemicals, Nature of the Pesticide Residues in Processed Commodities - High Temperature Hydrolysis, Document 507 (16th October 07).<br>Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market.<br>Commission Regulation (EU) No 283/2013 of 1 March 2013 setting out the data requirements for active substances, in accordance with Regulation (EC) No 1107/2009 of the European Parliament and of the Council concerning the placing of plant protection products on the market. |
| Deviations: | No |
| GLP: | Yes |

Acceptability: Yes

**Executive Summary**

This study was conducted to investigate the hydrolytic stability of [phenyl-U-$^{14}$C] 5-OH-dicamba (referred to as [14C] NOA405873 in the report) under conditions representative of food processing. These conditions are summarised below.

| Temperature (°C) | Time (min) | pH | Process Represented |
|---|---|---|---|
| 90 | 20 | 4 | Pasteurisation |
| 100 | 60 | 5 | Baking, Brewing, Boiling |
| 120 | 20 | 6 | Sterilisation |

Individual aqueous solutions of [phenyl-U-$^{14}$C] 5-OH-dicamba (nominal concentration 1 mg/L) were prepared in sterile citrate buffers at pH 4, 5 and 6. Duplicate samples for each buffer solution were incubated as detailed in the table above. A further set of duplicate samples for each buffer were prepared as above but not subjected to heating. These control vessels were incubated at room temperature.

The actual concentrations of [phenyl-U-$^{14}$C] 5-OH-dicamba, based upon the applied radioactivity and the volume (4 mL), ranged from 0.99 to 1.00 mg/L.

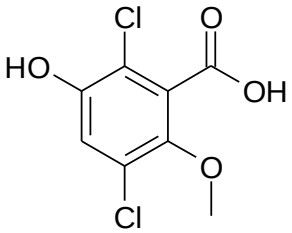
Recovery for test and control samples was >90% total applied radioactivity (TAR), indicating that there were no significant losses of radioactivity during the experimental procedures.

Aliquots of all test and control samples were analysed by TLC and indicated that after elevated temperature hydrolysis no significant degradation had taken place. The majority of the recovered radioactivity was present as 5-OH-dicamba (96.0 – 97.6% chromatographed radioactivity). Other unidentified degradation products were present at low levels accounting for ≤1.9% of the chromatographed radioactivity.

It is concluded that 5-OH-dicamba was hydrolytically stable under conditions representative of pasteurisation, baking/brewing/boiling and sterilisation.

**Materials and Methods**

**Test Materials**

| | |
|---|---|
| Structure/Label | [Phenyl-U-$^{14}$C]5-OH-dicamba |
| | <br>(* = $^{14}$C position) |
| CAS Number | 7600-50-2 |
| Batch Number | RDR-XXI-69 |
| Specific Activity | 2.153 MBq/mg (58.2 μCi/mg) |
| Radiochemical Purity | 98.3% |

**Test Facilities**

The study was performed at Smithers Viscient (ESG) Ltd, 108 Woodfield Drive, Harrogate, North Yorkshire, HG1 4LG, UK.

**Study Design and Methods**

**Hydrolysis Phase**

A nominal weight of 18 μg of test item was added to three vessels in acetonitrile which was removed under a stream of nitrogen. Autoclave sterilised citrate buffers at pH 4, 5 and 6 (18 mL) were added to the vessels and the pH checked. An assessment of the solubility and adsorption to incubation vessels was carried out in a trial experiment.

Aliquots of each treatment buffer solution were transferred quantitatively to duplicate pre-weighed incubation vessels. The pH 4 solutions were heated to 90°C for 20 minutes, the pH 5 solutions were heated to 100°C for 60 minutes and the pH 6 solutions were heated to 120°C for 20 minutes in a water bath containing polyethylene glycol. Duplicate control samples from each pH group were incubated at room temperature for the test duration. All samples were wrapped in aluminium foil to exclude any light from the samples and therefore remove the likelihood of the test item degrading as a result of photolysis. Incubated test and control samples were transferred to individual pre-weighed storage vessels and the incubation vessel rinses and pH probe rinses added.

**Analytical Phase**

Triplicate aliquots of the test and control samples were removed from each pH group for quantification by LSC to determine the total radioactive recovery.

Aliquots of each duplicate test and control samples were analysed by normal-phase thin layer chromatography (TLC) in order to determine the amount of [phenyl-U-$^{14}$C] 5-OH-dicamba present. Reverse-phase high performance liquid chromatography (HPLC) was used to confirm the identity of [phenyl-U-$^{14}$C] 5-OH-dicamba in representative samples.

**Results and Discussion**

**Radioactive Recoveries of Applied Radioactivity**

Assessment of the solubility of [phenyl-U-$^{14}$C] 5-OH-dicamba in trial experiments confirmed solubility at 1 mg/L in pH 4, pH 5 and pH 6 buffer solutions. Assessment of the adsorption of [phenyl-U-$^{14}$C] 5-OH-dicamba to test glassware in a trial experiment showed 98.4 to 103.1% of applied radioactivity in the buffer solution was recovered.

The post-hydrolysis quantification results based on the actual amount of radioactivity applied to the solutions shows recoveries ranging from 92.4 to 97.8% TAR for the test samples and 95.8 to 97.7% TAR for the control samples.

These results indicated that there was no significant adsorption to glassware or losses of radioactivity during the experimental procedures.

Details of recoveries for test and control samples are summarised in

Table A 26.

**Table A 26: Summary of radioactive recoveries in test and control samples**

| **Sample** | **Radioactivity recovery (% TAR)** | | | | | |
|---|---|---|---|---|---|---|
| | **pH 4, 90°C, 20 min** | | **pH 5, 100°C, 60 min** | | **pH 6, 120°C, 20 min** | |
| Treated incubate | 96.5 | 96.8 | 97.6 | 97.8 | 92.4 | 94.3 |
| | **pH 4, unheated, 20 min** | | **pH 5, unheated, 60 min** | | **pH 6, unheated, 20 min** | |
| Treated incubate control | 96.0 | 97.7 | 96.7 | 96.9 | 96.9 | 95.8 |

TAR: Total Applied Radioactive

**Characterisation and Identification of Residues**

Aliquots of all test and control samples were analysed by TLC (chosen as the primary quantitative method). It was demonstrated that the majority of the recovered radioactivity was [phenyl-U-$^{14}$C]5-OH-dicamba (96.0-97.6% of the chromatographed radioactivity).Between 0.0 and 0.9% of the chromatographed radioactivity remained at the origin. Other unknown degradation products were present at low levels accounting for ≤1.9% of the chromatographed radioactivity. 5-OH-dicamba was further confirmed by HPLC and LC-MS.

The levels of [phenyl-U-$^{14}$C] 5-OH-dicamba in the test samples are summarised in

Table A 27.

**Table A 27: Summary of levels of [phenyl-U-$^{14}$C] 5-OH-dicamba in test and control samples**

| **Sample** | **5-OH-dicamba (% TAR)** | | | | | |
|---|---|---|---|---|---|---|
| | **pH 4, 90°C, 20 min** | | **pH 5, 100°C, 60 min** | | **pH 6, 120°C, 20 min** | |
| Treated incubate | 96.2 | 96.6 | 97.4 | 97.5 | 97.1 | 96.0 |
| | **pH 4, unheated, 20 min** | | **pH 5, unheated, 60 min** | | **pH 6, unheated, 20 min** | |
| Treated incubate control | 96.3 | 97.5 | 96.9 | 96.6 | 96.4 | 97.6 |

TAR: Total Applied Radioactive

**Storage Stability of Residues**
All test and control samples were analysed by HPLC on the day following incubation and by TLC 23 days later. No degradation of [phenyl-U-$^{14}$C] 5-OH-dicamba was detected in any sample. Storage stability information was therefore not required.

**Conclusion**
5-OH-dicamba was hydrolytically stable under conditions representative of pasteurisation, baking, brewing, boiling and sterilisation.
No breakdown or reaction products were formed during hydrolysis of 5-OH-dicamba under representative processing conditions. The relevant residue for enforcement and risk assessment in processed commodities is therefore expected to be the same as for primary crops.

## A 2.1.2.2 Nature of residues in livestock

No new data are submitted in the framework of this application.

## A 2.1.3 Magnitude of residues in plants

### A 2.1.3.1 Maize

**Table A 28: Comparison of intended and critical EU GAPs**

| Type of GAP | Number of applications | Application rate per treatment (precise unit) | Interval between application | Growth stage at last application | PHI (days) |
|---|---|---|---|---|---|
| cGAP EU (DAR, Denmark, 2007) | 1 | 0.36 kg a.s./ha | N/A | BBCH 16 | Since the period between treatment and harvest is more than 100 days, no PHI is applicable. |
| cGAP AIR 3 [(a)] | 1 | 0.288 kg a.s./ha | N/A | BBCH 19 | N/A[(b)] |
| Intended cGAP; Use number ~~3,6,7~~ 1, 2 * | 1 | ~~0.1875~~ 125 kg a.s./ha | N/A | BBCH 12 – ~~19~~ 14 | N/A[(b)] |

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0
N/A – not applicable
(a) This data is currently under evaluation for the renewal of approval of the active substance, dicamba (Commission Implementing Regulation (EU) No. 844/2012 of 18 September 2012)
(b) PHI is determined by crop maturity

#### A 2.1.3.1.1 Report No R10280 – EU reviewed

| | |
|---|---|
| Comments of zRMS: | This study has been reviewed at the EU level. |

| | |
|---|---|
| Reference: | KCA1 6.3.1/01 |
| Report | Hertl, P., 1995. Determination of residues of DICAMBA in Corn (Zea mais) after application of BANVEL 4S or SAN 845 H 70 WG under field conditions in the Federal Republic of Germany, 1993.<br>Syngenta Report No. R10280<br>Syngenta File No. SAN837/5434<br>GLP, unpublished |
| Guideline(s): | No<br>No guidelines stated |
| Deviations: | Yes<br>Start of trials was delayed from 18th week to 22nd week (1993).<br>Missing information on test substance was not supplied.<br>At sampling date 30DAA, 12 stalk samples were cut into 4 sections. Each section should have contained parts of 3 plants and the remainder refused, instead sections of all 12 plants were retained.<br>None of the deviations had any influence on the study results. |
| GLP: | Yes |
| Acceptability: | Yes |

## Table A 29: Summary R10280 trials

| | | | |
|---|---|---|---|
| **Field Trials, Crop Residue (Summary) :** Determination of residues of DICAMBA in Corn (Zea mais) after application of BANVEL 4S or SAN 845 H 70 WG under field conditions in the Federal Republic of Germany, 1993. | | | |
| **Active Substance (common name):** | Dicamba | **Commercial Product (name):** | $Commercial.Name$ |
| **Crop/Crop Group:** | Maize | **Producer of commercial product:** | Syngenta AG |
| **Responsible body for reporting (name, address):** | Syngenta AG, Basel, Switzerland | **Indoor/Glasshouse/Outdoor:** | Field |
| **Country:** | GERMANY | **Other active substance in the formulation (common name and content):** | None |
| **Content of active substance (g/kg or g/L):** | 480 g AI/L | **Residues calculated as:** | $Residue.Units$ |
| **Formulation (e.g. WP):** | A7254B SL | | |
| **Analytical Method:**<br>Dicamba (Cob, Grain, Stover (fodder), Whole plant) P-14.063; 0.01 mg/kg<br>Dicamba-5-OH (Cob, Grain, Stover (fodder), Whole plant) P-14.063; 0.01 mg/kg | | | |
| **Recovery data:**<br>Dicamba Cob Mean = 100% RSD = 7% (n = 8 in 0.0101 - 0.101 mg/kg spiking range)<br>Dicamba Grain Mean = 96% RSD = 14% (n = 6 in 0.01 - 0.1 mg/kg spiking range)<br>Dicamba Stover (fodder) Mean = 106% RSD = 33% (n = 12 in 0.0101 - 0.101 mg/kg spiking range)<br>Dicamba Whole plant Mean = 109% RSD = 10% (n = 15 in 0.101 - 30.2 mg/kg spiking range)<br>NOA405873 Cob Mean = 87% RSD = 18% (n = 8 in 0.0102 - 0.102 mg/kg spiking range)<br>NOA405873 Grain Mean = 91% RSD = 7% (n = 6 in 0.0102 - 0.102 mg/kg spiking range)<br>NOA405873 Stover (fodder) Mean = 110% RSD = 13% (n = 12 in 0.0102 - 0.102 mg/kg spiking range)<br>NOA405873 Whole plant Mean = 103% RSD = 13% (n = 15 in 0.102 - 1.22 mg/kg spiking range) | | | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| R10280<br>C-07-35-95-2<br>GERMANY<br>(Europe North) | Maize (Bonny) | 1.04 May 1993<br>2 –<br>3 - | Foliar | | 400 L/ha | 360.00 g ai/ha<br><br>(-) A7254B 480 SL | 05 Jun 1993 | 7 leaves | Whole plant | 17.00 | 0.538 | 17.538 | 0 | 05 Jun 1993 | Field A7254B<br><br>SP (max days): Dicamba/Grain: 125 Dicamba/Whole plant: 348 |
| | | | | | | | | | Whole plant | 13.00 | 0.481 | 13.481 | 0 | 05 Jun 1993 | |
| | | | | | | | | | Whole plant | 10.90 | 0.193 | 11.093 | 0 | 05 Jun 1993 | |
| | | | | | | | | | Whole plant | 10.60 | 0.178 | 10.778 | 0 | 05 Jun 1993 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Whole plant | 0.023 | 0.198 | 0.221 | 30 | 05 Jul 1993 | Dicamba/Cob: 215<br>Dicamba/Stover (fodder): 237<br>Dicamba-5-OH/Stover (fodder): 237<br>Dicamba-5-OH/Grain: 125<br>Dicamba-5-OH/Whole plant: 348<br>Dicamba-5-OH/Cob: 215 |
| | | | | | | | | | Whole plant | 0.015 | 0.17 | 0.185 | 30 | 05 Jul 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 60 | 04 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 60 | 04 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | < 0.01 | 0.027 | 0.037 | 60 | 04 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | < 0.01 | 0.023 | 0.033 | 60 | 04 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 90 | 03 Sep 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 90 | 03 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | < 0.01 | 0.041 | 0.051 | 90 | 03 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | < 0.01 | 0.048 | 0.058 | 90 | 03 Sep 1993 | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 140 | 23 Oct 1993 | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 140 | 23 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | < 0.01 | 0.01 | 0.02 | 140 | 23 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | 0.017 | < 0.01 | 0.027 | 140 | 23 Oct 1993 | |
| | Maize (Bonny) | 1.04 May 1993 | Foliar | | 400 L/ha | 360.00 g ai/ha | 05 Jun 1993 | 7 leaves | Whole plant | 15.60 | 0.774 | 16.374 | 0 | 05 Jun 1993 | Field A9781A |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | 2 –<br>3 - | | | | (-)<br><br>A9781A 700 WG | | | Whole plant | 16.00 | 0.358 | 16.358 | 0 | 05 Jun 1993 | SP (max days): Dicamba/Stover (fodder): 239 Dicamba/Grain: 125 Dicamba/Cob: 216 Dicamba/Whole plant: 319 Dicamba-5-OH/Stover (fodder): 239 Dicamba-5-OH/Grain: 125 Dicamba-5-OH/Whole plant: 319 Dicamba-5-OH/Cob: 216 |
| | | | | | | | | | Whole plant | 0.034 | 0.209 | 0.243 | 30 | 05 Jul 1993 | |
| | | | | | | | | | Whole plant | 0.026 | 0.185 | 0.211 | 30 | 05 Jul 1993 | |
| | | | | | | | | | Cob | 0.018 | < 0.01 | 0.028 | 60 | 04 Aug 1993 | |
| | | | | | | | | | Cob | 0.012 | < 0.01 | 0.022 | 60 | 04 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | < 0.01 | 0.025 | 0.035 | 60 | 04 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | < 0.01 | 0.018 | 0.028 | 60 | 04 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 90 | 03 Sep 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 90 | 03 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | < 0.01 | 0.039 | 0.049 | 90 | 03 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | < 0.01 | 0.017 | 0.027 | 90 | 03 Sep 1993 | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 140 | 23 Oct 1993 | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 140 | 23 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | < 0.01 | < 0.01 | < 0.02 | 140 | 23 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | 0.012 | < 0.01 | 0.022<br>,,, | 140 | 23 Oct 1993 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| R10280<br>C-07-49-95-2<br>GERMANY (Europe North) | Maize (Apache) | 1.27 Apr 1993<br>2 –<br>3 - | Foliar | | 200 L/ha | 310.00 g ai/ha<br><br>(-)<br><br>A7254B 480 SL | 07 Jun 1993 | 7-8 leaves | Grain | < 0.01 | < 0.01 | < 0.02 | 151 | 05 Nov 1963 | Field A7254B<br><br>SP (max days): Dicamba/Grain: 11073 Dicamba/Whole plant: 346 Dicamba/Cob: 216 Dicamba/Stover (fodder): 214 Dicamba-5-OH/Stover (fodder): 214 Dicamba-5-OH/Grain: 11073 Dicamba-5-OH/Whole plant: 346 Dicamba-5-OH/Cob: 216 |
| | | | | | | | | | Whole plant | 21.20 | 0.098 | 21.298 | 0 | 07 Jun 1993 | |
| | | | | | | | | | Whole plant | 18.10 | 0.064 | 18.164 | 0 | 07 Jun 1993 | |
| | | | | | | | | | Whole plant | 0.073 | 0.303 | 0.376 | 30 | 07 Jul 1993 | |
| | | | | | | | | | Whole plant | 0.051 | 0.283 | 0.334 | 30 | 07 Jul 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 60 | 06 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 60 | 06 Aug 1993 | |
| | | | | | | | | | Whole plant | 0.011 | 0.048 | 0.059 | 60 | 06 Aug 1993 | |
| | | | | | | | | | Whole plant | < 0.01 | 0.02 | 0.03 | 60 | 06 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | 0.01 | 0.022 | 0.032 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | 0.023 | 0.068 | 0.091 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 151 | 05 Nov 1993 | |
| | | | | | | | | | Stover (fodder) | 0.026 | 0.02 | 0.046 | 151 | 05 Nov 1993 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Stover (fodder) | 0.033 | < 0.01 | 0.043 | 151 | 05 Nov 1993 | |
| | Maize (Apache) | 1.27 Apr 1993<br>2 –<br>3 - | Foliar | | 200 L/ha | 318.00 g ai/ha<br><br>(-)<br><br>A9781A 700 WG | 07 Jun 1993 | 7-8 leaves | Whole plant | < 0.01 | < 0.01 | < 0.02 | 0 | 07 Jun 1993 | Field A9781A<br><br>SP (max days): Dicamba/Grain: 115 Dicamba/Whole plant: 345 Dicamba/Cob: 227 Dicamba/Stover (fodder): 267 Dicamba-5-OH/Stover (fodder): 267 Dicamba-5-OH/Grain: 115 Dicamba-5-OH/Whole plant: 345 Dicamba-5-OH/Cob: 227 |
| | | | | | | | | | Whole plant | 32.80 | 0.056 | 32.856 | 0 | 07 Jun 1993 | |
| | | | | | | | | | Whole plant | 19.00 | 0.061 | 19.061 | 0 | 07 Jun 1993 | |
| | | | | | | | | | Whole plant | 0.055 | 0.289 | 0.344 | 30 | 07 Jul 1993 | |
| | | | | | | | | | Whole plant | 0.055 | 0.129 | 0.184 | 30 | 07 Jul 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 60 | 06 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 60 | 06 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | 0.017 | 0.071 | 0.088 | 60 | 06 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | 0.015 | 0.036 | 0.051 | 60 | 06 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | 0.013 | 0.027 | 0.04 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | 0.029 | 0.143 | 0.172 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | 0.023 | 0.135 | 0.158 | 90 | 05 Sep 1993 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 151 | 05 Nov 1993 | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 151 | 05 Nov 1993 | |
| | | | | | | | | | Stover (fodder) | 0.026 | 0.012 | 0.038 | 151 | 05 Nov 1993 | |
| | | | | | | | | | Stover (fodder) | 0.039 | 0.026 | 0.065 | 151 | 05 Nov 1993 | |
| R10280<br>C-07-71-95-2<br>GERMANY<br>(Europe North) | Maize (Mixture) | 1.21 Apr 1993<br>2 –<br>3 - | Foliar | | 300 L/ha | 361.00 g ai/ha<br><br>(-) A7254B 480 SL | 05 Jun 1993 | 6-7 leaves | Whole plant | 10.80 | 0.083 | 10.883 | 0 | 05 Jun 1993 | Field A7254B<br><br>SP (max days): Dicamba/Grain: 140 Dicamba/Whole plant: -1513 Dicamba/Cob: 227 Dicamba/Stover (fodder): 286 Dicamba-5-OH/Stover (fodder): 286 Dicamba-5-OH/Grain: 140 Dicamba-5-OH/Whole plant: -1513 Dicamba-5-OH/Cob: 227 |
| | | | | | | | | | Whole plant | 15.40 | 0.106 | 15.506 | 0 | 05 Jun 1993 | |
| | | | | | | | | | Whole plant | 0.051 | 0.23 | 0.281 | 30 | 05 Jul 1993 | |
| | | | | | | | | | Whole plant | 0.036 | 0.287 | 0.323 | 30 | 05 Jul 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 63 | 07 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 63 | 07 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | 0.024 | 0.084 | 0.108 | 63 | 07 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | 0.032 | 0.147 | 0.179 | 63 | 07 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | 0.052 | 0.529 | 0.581 | 63 | 07 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | 0.054 | 0.474 | 0.528 | 63 | 07 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 94 | 07 Sep 1993 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 94 | 07 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | 0.046 | 0.323 | 0.369 | 94 | 07 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | 0.043 | 0.181 | 0.224 | 94 | 07 Sep 1993 | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 130 | 13 Oct 1993 | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 130 | 13 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | 0.014 | 0.023 | 0.037 | 130 | 13 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | 0.024 | 0.033 | 0.057 | 130 | 13 Oct 1993 | |
| | Maize (Mixture) | 1.21 Apr 1993<br>2 –<br>3 - | Foliar | | 300 L/ha | 318 g ai/ha<br><br>(-) A9781A 700 WG | 05 Jun 1993 | 6-7 leaves | Whole plant | 8.88 | 0.137 | 9.017 | 0 | 05 Jun 1993 | Field A9781A<br><br>SP (max days): Dicamba/Stover (fodder): 262 Dicamba/Grain: 140 Dicamba/Cob: 228 Dicamba/Whole plant: 353 Dicamba-5-OH/Stover (fodder): 262 Dicamba-5- |
| | | | | | | | | | Whole plant | 10.50 | 0.222 | 10.722 | 0 | 05 Jun 1993 | |
| | | | | | | | | | Whole plant | 18.60 | 0.145 | 18.745 | 0 | 05 Jun 1993 | |
| | | | | | | | | | Whole plant | 19.90 | 0.184 | 20.084 | 0 | 05 Jun 1993 | |
| | | | | | | | | | Whole plant | 0.047 | 0.299 | 0.346 | 30 | 05 Jul 1993 | |
| | | | | | | | | | Whole plant | 0.051 | 0.366 | 0.417 | 30 | 05 Jul 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 63 | 07 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 63 | 07 Aug 1993 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Stover (fodder) | 0.021 | 0.14 | 0.161 | 63 | 07 Aug 1993 | OH/Grain: 140 Dicamba-5-OH/Whole plant: 353 Dicamba-5-OH/Cob: 228 |
| | | | | | | | | | Stover (fodder) | 0.048 | 0.146 | 0.194 | 63 | 07 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 94 | 07 Sep 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 94 | 07 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | 0.03 | 0.259 | 0.289 | 94 | 07 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | 0.022 | 0.229 | 0.251 | 94 | 07 Sep 1993 | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 130 | 13 Oct 1993 | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 130 | 13 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | 0.066 | 0.01 | 0.076 | 130 | 13 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | 0.018 | < 0.01 | 0.028 | 130 | 13 Oct 1993 | |
| R10280 C-07-88-95-2 GERMANY (Europe North) | Maize (Figaro) | 1.18 Apr 1993 2 – 3 - | Foliar | | 300 L/ha | 360.00 g ai/ha<br><br>(-)<br><br>A7254B 480 SL | 07 Jun 1993 | 6-7 leaves | Whole plant | 17.40 | 0.243 | 17.643 | 0 | 07 Jun 1993 | Field A7254B<br><br>SP (max days): Dicamba/Stover (fodder): 277 Dicamba/Grain: 139 Dicamba/Cob: 244 |
| | | | | | | | | | Whole plant | 18.30 | 0.248 | 18.548 | 0 | 07 Jun 1993 | |
| | | | | | | | | | Whole plant | 0.058 | 0.133 | 0.191 | 31 | 08 Jul 1993 | |
| | | | | | | | | | Whole plant | 0.039 | 0.132 | 0.171 | 31 | 08 Jul 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 61 | 07 Aug 1993 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 61 | 07 Aug 1993 | Dicamba/Whole plant: 337<br>Dicamba-5-OH/Stover (fodder): 277<br>Dicamba-5-OH/Grain: 139<br>Dicamba-5-OH/Whole plant: 337<br>Dicamba-5-OH/Cob: 244 |
| | | | | | | | | | Stover (fodder) | 0.016 | 0.124 | 0.14 | 61 | 07 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | 0.02 | 0.082 | 0.102 | 61 | 07 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | 0.024 | 0.171 | 0.195 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | 0.033 | 0.113 | 0.146 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 131 | 16 Oct 1993 | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 131 | 16 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | 0.099 | 0.051 | 0.15 | 131 | 16 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | 0.104 | 0.039 | 0.143 | 131 | 16 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | 0.476 | 0.049 | 0.525 | 131 | 16 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | 0.431 | 0.072 | 0.503 | 131 | 16 Oct 1993 | |
| | Maize (Figaro) | 1.18 Apr 1993<br>2 –<br>3 - | Foliar | | 300 L/ha | 360.00 g ai/ha<br><br>(-) | 07 Jun 1993 | 6-7 leaves | Whole plant | 16.70 | 0.303 | 17.003 | 0 | 07 Jun 1993 | Field A9781A<br><br>SP (max days): |
| | | | | | | | | | Whole plant | 11.40 | 0.193 | 11.593 | 0 | 07 Jun 1993 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | A9781A 700 WG | | | Whole plant | 7.40 | 0.127 | 7.527 | 0 | 07 Jun 1993 | Dicamba/Stover (fodder): 296 Dicamba/Grain: 139 Dicamba/Whole plant: 357 Dicamba/Cob: 230 Dicamba-5-OH/Stover (fodder): 296 Dicamba-5-OH/Grain: 139 Dicamba-5-OH/Whole plant: 357 Dicamba-5-OH/Cob: 230 |
| | | | | | | | | | Whole plant | 7.65 | 0.153 | 7.803 | 0 | 07 Jun 1993 | |
| | | | | | | | | | Whole plant | 0.062 | 0.451 | 0.513 | 31 | 08 Jul 1993 | |
| | | | | | | | | | Whole plant | 0.102 | 0.515 | 0.617 | 31 | 08 Jul 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 61 | 07 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 61 | 07 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | 0.02 | 0.053 | 0.073 | 61 | 07 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | 0.011 | 0.041 | 0.052 | 61 | 07 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | 0.16 | 0.882 | 1.042 | 61 | 07 Aug 1993 | |
| | | | | | | | | | Stover (fodder) | 0.054 | 0.448 | 0.502 | 61 | 07 Aug 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | 0.04 | 0.296 | 0.336 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Stover (fodder) | 0.078 | 0.554 | 0.632 | 90 | 05 Sep 1993 | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 131 | 16 Oct 1993 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Grain | < 0.01 | < 0.01 | < 0.02 | 131 | 16 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | < 0.01 | 0.016 | < 0.02 | 131 | 16 Oct 1993 | |
| | | | | | | | | | Stover (fodder) | 0.022 | 0.038 | 0.06 | 131 | 16 Oct 1993 | |

(a) According to Codex (or other e.g. EU) classification

(b) Only if relevant

(c) Year must be indicated

(d) Minimum number of days after last application (Label pre-harvest interval, PHI, underline)

(e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included.

(*) Indicates sample taken prior to application

(#) Indicates corrected Residue values

(^) PHI calculated using cut date

(+) Indicates calculated Residue value

(DBA) Days Before Application

SP (max days): Maximum storage period

## A 2.1.3.1.2 Study TK0258007-REG (NC15022)

| | |
|---|---|
| Comments of zRMS: | Four decline residue field trials on maize were successfully conducted in Northern France, Germany and Poland during 2015. Determination of dicamba and its metabolite, NOA405873 and nicosulfuron on maize was undertaken to evaluate the magnitude of residues.<br><br>Dicamba and nicosulfuron were applied as A19658H, an oil dispersion (OD) formulation containing dicamba at 220 g per litre and nicosulfuron at 50 g per litre. To treated plot P2, one application, at BBCH 16-18, of A19658H was made at 288 g ai/ha for dicamba at 65.45 g ai/ha for nicosulfuron. Following the application, maize samples (whole plant, remaining plant and whole cobs) were collected at set intervals with untreated maize samples (whole plant, remaining plant and whole cobs) collected first. Samples were analysed for both dicamba and its metabolite, NOA405873 and nicosulfuron. The analytical method, GRM074.01A was successfully validated for the determination of nicosulfuron in maize kernels (dry commodity) and maize whole plant (high water commodity) with an LOQ of 0.01 mg/kg. The validation of the analytical method was conducted according to EU Guidance Document SANCO/3029/99 rev. 4 11/07/2000.<br><br>Results:<br><u>Dicamba</u><br>Residues of dicamba in whole cob samples taken at 75-79 BBCH and 89 BBCH were below the limit of quantification (<0.01 mg/kg).<br>Residues of dicamba in remaining plant samples taken at 75-79 BBCH were between <0.01 and 0.04 mg/kg and at 89 BBCH were between 0.01 and 0.04 mg/kg.<br>Residues of dicamba in whole plant samples taken at 39 BBCH were between <0.01 and 0.09 mg/kg, at 63 BBCH were between <0.01 and 0.03 mg/kg and at 83-85 BBCH were between <0.01 and 0.04 mg/kg.<br>Residues of dicamba in untreated samples were below the limit of quantification (<0.01 mg/kg). This is with the exception of remaining plant at BBCH 89 where residues range from <0.01 to 0.04 mg/kg.<br><br><u>5-OH-dicamba (NOA405873)</u><br>Residues of NOA405873 in whole cob samples taken at 75-79 BBCH and 89 BBCH were below the limit of quantification (<0.01 mg/kg).<br>Residues of NOA405873 in remaining plant samples taken at 75-79 BBCH were between 0.03 and 1.4 mg/kg and at 89 BBCH were between <0.01 and 0.96 mg/kg.<br>Residues of NOA405873 in whole plant samples taken at 39 BBCH were between 0.11 and 1.6 mg/kg, at 63 BBCH were between 0.05 and 1.1 mg/kg and at 83-85 BBCH were between <0.01 and 0.97 mg/kg.<br>Residues of NOA405873 in untreated samples were below the limit of quantification (<0.01 mg/kg). This is with the exception of remaining plant at BBCH 75-79 and BBCH 89 where residues range from <0.01 to 0.04 mg/kg and <0.01 to 0.01 mg/kg, respectively.<br><br>The study is acceptable. |

| | |
|---|---|
| Reference: | KCA 6.3.1/02 |
| Report | Nicosulfuron and Dicamba- Residue Validation and Study on Maize in Northern France, Germany and Poland in 2015.<br>Andrews, G., 2016, Syngenta Report No. TK0258007-REG<br>Syngenta File No. A19658H_10060, |
| Guideline(s): | Yes<br>Commission of the European Communities, General Recommendations for the Design, Preparation and Realization of Residue Trials; 7029/VI/95 (rev. 5, working document). |

| | |
|---|---|
| | Guidelines and Criteria for the Preparation and Presentation of Complete Dossiers and of Summary Dossiers for the Inclusion of Active Substances in Annex I of Directive 91/414/EEC (Article 5.3 and 8.2), 1996. |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

## Table A 30: Summary TK0258007-REG trials

| | | | |
|---|---|---|---|
| **Field Trials, Crop Residue (Summary) :** NICOSULFURON AND DICAMBA- RESIDUE VALIDATION AND STUDY ON MAIZE IN NORTHERN FRANCE, GERMANY AND POLAND IN 2015 | | | |
| **Active Substance (common name):** | Dicamba | **Commercial Product (name):** | $Commercial.Name$ |
| **Crop/Crop Group:** | Maize | **Producer of commercial product:** | Syngenta AG |
| **Responsible body for reporting (name, address):** | Syngenta AG, Basel, Switzerland | **Indoor/Glasshouse/Outdoor:** | Field |
| **Country:** | FRANCE, POLAND, GERMANY | **Other active substance in the formulation (common name and content):** | Nicosulfuron (5 % w/v) |
| **Content of active substance (g/kg or g/L):** | 22 % w/v | **Residues calculated as:** | $Residue.Units$ |
| **Formulation (e.g. WP):** | A19658H | | |
| **Analytical Method:**<br>Dicamba (Cob without Kernel, Kernel, Kernels plus cob, Remaining Plant, Whole plant) GRM022.07A; 0.01 mg/kg<br>Dicamba-5-OH (Cob without Kernel, Kernel, Kernels plus cob, Remaining Plant, Whole plant) GRM022.07A; 0.01 mg/kg | | | |
| **Recovery data:**<br>Dicamba Cob without Kernel Mean = 78% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Dicamba Kernel Mean = 73% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Dicamba Remaining Plant Mean = 78% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Dicamba Whole plant Mean = 80% RSD = 8% (n = 3 in 0.01 - 0.1 mg/kg spiking range)<br>NOA405873 Cob without Kernel Mean = 85% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>NOA405873 Kernel Mean = 78% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>NOA405873 Remaining Plant Mean = 85% RSD = 8% (n = 4 in 0.01 - 2 mg/kg spiking range)<br>NOA405873 Whole plant Mean = 98% RSD = 13% (n = 4 in 0.01 - 1.98 mg/kg spiking range) | | | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| TK0258007-REG NC15022-01 FRANCE (Europe North) (-) | Maize (KROISSAN) | 1.23 Mar 2015 2 – 3 - | Foliar | | 226.78 L/ha | 288.52977 g ai/ha (-) | 13 Jun 2015 | BBCH 17 - 18 | Whole plant | 0.09 | 1.60 | 1.69 | 20 | 03 Jul 2015 | Field A19658H SP (max days): Dicamba/Cob |
| | | | | | | | | | Whole plant | 0.03 | 1.14 | 1.17 | 34 | 17 Jul 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | < 0.01 | < 0.02 | 55 | 07 Aug 2015 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 55 | 07 Aug 2015 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | < 0.01 + | < 0.02 + | 55 | 07 Aug 2015 | without Kernel: 230 Dicamba/Kernels plus cob: 230 Dicamba/Kernel: 230 Dicamba/Rema |
| | | | | | | | | | Remaining Plant | 0.04 | 1.41 | 1.45 | 55 | 07 Aug 2015 | |
| | | | | | | | | | Whole plant | 0.04 | 0.97 | 1.01 | 86 | 07 Sep 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | < 0.01 | < 0.02 | 97 | 18 Sep 2015 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 97 | 18 Sep 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | < 0.01 + | < 0.02 + | 97 | 18 Sep 2015 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Remaining Plant | 0.04 | 0.96 | 1.00 | 97 | 18 Sep 2015 | ining Plant: 230 Dicamba/Whole plant: 265 Dicamba-5-OH/Whole plant: 265 Dicamba-5-OH/Kernels plus cob: 230 Dicamba-5-OH/Remaining Plant: 230 Dicamba-5-OH/Kernel: 230 Dicamba-5-OH/Cob without Kernel: 230 |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| TK0258007-REG NC15022-02 FRANCE (Europe North) (-) | Maize (AXXYS) | 1.14 Apr 2015 2 – 3 - | Foliar | | 305.15 L/ha | 291.69705 g ai/ha<br><br>(-) | 03 Jun 2015 | BBCH 17 | Whole plant | < 0.01 | 0.14 | 0.15 | 28 | 01 Jul 2015 | Field A19658H<br><br>SP (max days): Dicamba/Cob without Kernel: 239 Dicamba/Kernels plus cob: 239 Dicamba/Kernel: 239 Dicamba/Rema |
| | | | | | | | | | Whole plant | < 0.01 | 0.06 | 0.07 | 34 | 07 Jul 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | < 0.01 | < 0.02 | 56 | 29 Jul 2015 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 56 | 29 Jul 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | < 0.01 + | < 0.02 + | 56 | 29 Jul 2015 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 0.03 | 0.04 | 56 | 29 Jul 2015 | |
| | | | | | | | | | Whole plant | < 0.01 | 0.01 | 0.02 | 83 | 25 Aug 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | < 0.01 | < 0.02 | 114 | 25 Sep 2015 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 114 | 25 Sep 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | < 0.01 + | < 0.02 + | 114 | 25 Sep 2015 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Remaining Plant | 0.02 | < 0.01 | 0.03 | 114 | 25 Sep 2015 | ining Plant: 239 Dicamba/Whole plant: 267 Dicamba-5-OH/Remaining Plant: 239 Dicamba-5-OH/Kernels plus cob: 239 Dicamba-5-OH/Whole plant: 267 Dicamba-5-OH/Kernel: 239 Dicamba-5-OH/Cob without Kernel: 239 |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| TK0258007-REG NC15022-03 GERMANY (Europe North) | Maize (Amaretto) | 1.04 May 2015<br>2 –<br>3 - | Foliar | | 200 L/ha | 286.10767 g ai/ha<br><br>(-) | 02 Jun 2015 | BBCH 16 | Whole plant | 0.03 | 0.46 | 0.49 | 31 | 03 Jul 2015 | Field A19658H<br><br>SP (max days): Dicamba/Kernels plus cob: 237 Dicamba/Cob without Kernel: 237 Dicamba/Kernel: 237 Dicamba/Rema |
| | | | | | | | | | Whole plant | 0.01 | 0.38 | 0.39 | 44 | 16 Jul 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | < 0.01 | < 0.02 | 59 | 31 Jul 2015 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 59 | 31 Jul 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | < 0.01 + | < 0.02 + | 59 | 31 Jul 2015 | |
| | | | | | | | | | Remaining Plant | 0.02 | 0.42 | 0.44 | 59 | 31 Jul 2015 | |
| | | | | | | | | | Whole plant | < 0.01 | 0.28 | 0.29 | 73 | 14 Aug 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | < 0.01 | < 0.02 | 86 | 27 Aug 2015 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 86 | 27 Aug 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | < 0.01 + | < 0.02 + | 86 | 27 Aug 2015 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Remaining Plant | 0.01 | 0.16 | 0.17 | 86 | 27 Aug 2015 | ining Plant: 237 Dicamba/Whole plant: 265 Dicamba-5-OH/Whole plant: 265 Dicamba-5-OH/Kernels plus cob: 237 Dicamba-5-OH/Remaining Plant: 237 Dicamba-5-OH/Kernel: 237 Dicamba-5-OH/Cob without Kernel: 237 |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| TK0258007-REG NC15022-05 POLAND (Europe North) | Maize (NK Cooler) | 1.21 Apr 2015<br>2 –<br>3 - | Foliar | | 296.67 L/ha | 283.54335 g ai/ha<br><br>(-) | 17 Jun 2015 | BBCH 18 | Whole plant | < 0.01 | 0.11 | 0.12 | 19 | 06 Jul 2015 | Field A19658H<br><br>SP (max days): Dicamba/Kernels plus cob: 220 Dicamba/Cob without Kernel: 220 Dicamba/Kernel: 220 Dicamba/Rema |
| | | | | | | | | | Whole plant | < 0.01 | 0.05 | 0.06 | 35 | 22 Jul 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | < 0.01 | < 0.02 | 61 | 17 Aug 2015 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 61 | 17 Aug 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | < 0.01 + | < 0.02 + | 61 | 17 Aug 2015 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 0.05 | 0.06 | 61 | 17 Aug 2015 | |
| | | | | | | | | | Whole plant | < 0.01 | < 0.01 | < 0.02 | 89 | 14 Sep 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | < 0.01 | < 0.02 | 111 | 06 Oct 2015 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 111 | 06 Oct 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | < 0.01 + | < 0.02 + | 111 | 06 Oct 2015 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Remaining Plant | 0.04 | 0.02 | 0.06 | 111 | 06 Oct 2015 | ining Plant: 220 Dicamba/Whole plant: 262 Dicamba-5-OH/Whole plant: 262 Dicamba-5-OH/Kernels plus cob: 220 Dicamba-5-OH/Remaining Plant: 220 Dicamba-5-OH/Kernel: 220 Dicamba-5-OH/Cob without Kernel: 220 |

(a) According to Codex (or other e.g. EU) classification

(b) Only if relevant

(c) Year must be indicated

(d) Minimum number of days after last application (Label pre-harvest interval, PHI, underline)

(e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included.

(*) Indicates sample taken prior to application

(#) Indicates corrected Residue values

(^) PHI calculated using cut date

(+) Indicates calculated Residue value

(DBA) Days Before Application

SP (max days): Maximum storage period

## A 2.1.3.1.3 Study 04-7016

| | |
|---|---|
| Comments of zRMS: | One residue field trial on maize was conducted in Northern Eurpoe, Switzerland during 2004. Determination of dicamba and its metabolite, NOA405873 and prosulfuron on maize was undertaken to evaluate the magnitude of residues.<br>Dicamba and prosulfuron were applied as A14031B (55WG) formulation containing dicamba at 500 g per kg and prosulfuron at 50 g per kg.<br><br>To treated plot P1 and P2, one application, at BBCH 17-18, of A14031B was made at 187.5 g dicamba/ha and at 18.75 g prosulfuron/ha. Additionally adjuvants Trend 90 at 0.2 L/ha (plot 1) and Etolag 149 at 1.0 L/ha (plot 2) were added.<br>Following the application, maize samples (whole plant, remaining plant and whole cobs) were collected at set intervals with untreated maize samples (whole plant, grain (kernels) and remaining plant) collected first. Samples were analysed for both dicamba and its metabolite, NOA405873 and prosulfuron.<br><br>Results:<br>Dicamba<br>No residues of dicamba (<LOQ) were found in any maize commodity, either controls or samples.<br>5-hydroxy dicamba<br>No residues of 5-hydroxy dicamba (<LOQ) were found in grain, but low residues were found in whole plant and remainder samples from plot 2 (Etolag 149), at 0.03-0.06 mg/kg and 0.01 mg/kg, respectively. No residues of 5-hydroxy dicamba were found in any controls samples.<br><br>Method REM 193.05 for dicamba and 5-hydroxy dicamba was used in all analyses.<br>Determination: LC-MS/MS.<br>LOQ=0.01 mg/kg.<br><br>The study is acceptable. |

| | |
|---|---|
| Reference: | KCA 6.3.1/03 |
| Report | Mound, L., Gardinal, P., Kwiatkowski, A., 2006. Prosulfuron (CGA152005) and Dicamba (SAN837): Residue Study in or on Maize In Switzerland.<br>Syngenta Report No. 04-7016<br>Syngenta File No. SAN837/6543<br>GLP, unpublished |
| Guideline(s): | Yes<br>FAO Guidelines on Producing Pesticide Residues Data from Supervised Trials (Rome, 1990).<br>Commission of the European Communities, General Recommendations for the Design, Preparation and Realization of Residue Trials; 7029/VI/95 (rev. 5, working document)<br>Guidelines and Criteria for the Preparation and Presentation of Complete Dossiers and of Summary Dossiers for the Inclusion of Active Substances in Annex I of Directive 91/414/EEC (Article 5.3 and 8.2), 1996 |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

## Table A 31: Summary 04-7016 trials

| | | | |
|---|---|---|---|
| **Field Trials, Crop Residue (Summary) :** Prosulfuron (CGA152005) and Dicamba (SAN837) - Residue Study in or on Maize In Switzerland | | | |
| **Active Substance (common name):** | Dicamba | **Commercial Product (name):** | $Commercial.Name$ |
| **Crop/Crop Group:** | Maize | **Producer of commercial product:** | Syngenta AG |
| **Responsible body for reporting (name, address):** | Syngenta AG, Basel, Switzerland | **Indoor/Glasshouse/Outdoor:** | Field |
| **Country:** | SWITZERLAND | **Other active substance in the formulation (common name and content):** | Prosulfuron (50 g AI/kg) |
| **Content of active substance (g/kg or g/L):** | 500 g AI/kg | **Residues calculated as:** | $Residue.Units$ |
| **Formulation (e.g. WP):** | A14031B WG | | |
| **Analytical Method:**<br>Dicamba (Cob + Remainder Of Plant, Thresh, Whole plant) REM 193.05; 0.01 mg/kg<br>Dicamba-5-OH (Cob + Remainder Of Plant, Thresh, Whole plant) REM 193.05; 0.01 mg/kg | | | |
| **Recovery data:**<br>Dicamba Cob Mean = 83% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Dicamba Grain Mean = 89% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Dicamba Whole plant Mean = 87% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>NOA405873 Cob Mean = 99% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>NOA405873 Grain Mean = 110% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>NOA405873 Whole plant Mean = 95% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range) | | | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| 04-7016<br>CH-HR-04-0198<br>SWITZERLAND<br>(Europe North) | Maize (Kuxxar) | 1.30 Apr 2004<br>2 –<br>3 - | Foliar | | 310 L/ha | 193.80 g ai/ha<br><br>(-, 0.2 L/ha) | 09 Jun 2004 | BBCH 17 - 18 | Whole plant | < 0.01 | < 0.01 | < 0.02 | 42 | 21 Jul 2004 | Field A14031B<br><br>SP (max days): Dicamb |
| | | | | | | | | | Whole plant | < 0.01 | < 0.01 | < 0.02 | 77 | 25 Aug 2004 | |
| | | | | | | | | | Cob + Remainder Of Plant | < 0.01 | < 0.01 | < 0.02 | 113 | 30 Sep 2004 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | *Kernel* | < 0.01 | < 0.01 | < 0.02 | 113 | 30 Sep 2004 | a/: 281 Dicamba/Cob + Remainder Of Plant: 281 Dicamba/Whole plant: 352 Dicamba-5-OH/: 281 Dicamba-5-OH/Cob + Remainder Of Plant: 281 Dicamba-5-OH/Whole plant: 352 |
| | Maize (Kuxxar) | 1.30 Apr 2004<br>2 –<br>3 - | Foliar | | 311.10 L/ha | 194.40 g ai/ha<br><br>(-, 1 L/ha) | 09 Jun 2004 | BBCH 17 - 18 | Whole plant | < 0.01 | 0.06 | 0.07 | 42 | 21 Jul 2004 | Field A14031 B |
| | | | | | | | | | Whole plant | < 0.01 | 0.03 | 0.04 | 77 | 25 Aug 2004 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Cob + Remainder Of Plant | < 0.01 | 0.01 | 0.02 | 113 | 30 Sep 2004 | SP (max days): Dicamba/: 281 Dicamba/Cob + Remainder Of Plant: 281 Dicamba/Whole plant: 352 Dicamba-5-OH/: 281 Dicamba-5-OH/Cob + Remainder Of Plant: 281 Dicamba-5-OH/Whole plant: 352 |
| | | | | | | | | | *Kernel* | < 0.01 | < 0.01 | < 0.02 | 113 | 30 Sep 2004 | |

(a) According to Codex (or other e.g. EU) classification

(b) Only if relevant

(c) Year must be indicated

(d) Minimum number of days after last application (Label pre-harvest interval, PHI, underline)

(e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included.

(*) Indicates sample taken prior to application

(#) Indicates corrected Residue values

(^) PHI calculated using cut date

(+) Indicates calculated Residue value

(DBA) Days Before Application

SP (max days): Maximum storage period

## A 2.1.3.1.4 Study gmz043004

| | |
|---|---|
| Comments of zRMS: | One residue field trial on maize was conducted in Saxonia. Determination of dicamba and its metabolite, NOA405873 (5-OH-dicamba) and prosulfuron on maize was undertaken to evaluate the magnitude of residues in maize fodder, silage and kernels.<br>Dicamba and prosulfuron were applied as A14031B (55 WG) formulation containing dicamba at 500 g per kg and prosulfuron at 50 g per kg.<br>The test product was applied once as a foliar treatment (at BBCH 17-18), on one plot in tank mixture with Etolag 149 and in another plot with Trend 90. The total nominal amount of A14031B was 0.375 kg product/ha (corresponding to 18.75 g prosulfuron/ha and 187.5 g dicamba/ha).<br><br>Results:<br>Dicamba<br>No residues of dicamba at or below 0.01 mg/kg were detected in any of the treated and untreated (control) specimens.<br>5-hydroxy dicamba<br>Residues of 5-hydroxy dicamba in whole plant specimens at 37 days after treatment were at 0.09 mg/kg (tankmix with Trend 90) or 0.03 mg/kg (tankmix with Etolag 149) and were reduced to 0.03 mg/kg or 0.01 mg/kg at grain harvest stage. No residues of 5-OH-dicamba were detected in or on the other treated maize (cobs or kernel) samples.<br>No residues of 5-hydroxy dicamba were found in any controls samples.<br><br>Method REM 193.05 for dicamba and 5-hydroxy dicamba was used in all analyses.<br>Determination: GC-MSD.<br>LOQ=0.01 mg/kg.<br>Mean recovery: 92% and 93% with RSD of 6.8% and 11.2% for dicamba and 5-OH-dicamba respectively.<br><br>The study is acceptable. |

| | |
|---|---|
| Reference: | KCA 6.3.1/04 |
| Report | Simon, P., 2006. Prosulfuron and Dicamba: Residue Study in or on Maize in Germany 2004 (Test Product: A14031B).<br>Syngenta Report No.gmz043004<br>Syngenta File No. SAN837/6545<br>GLP, unpublished |
| Guideline(s): | Yes<br>EC (European Commission), Guidelines for the generation of data concerning residues as provided in Annex II part A, section 6 and Annex III, part A, section 8 of Directive 91/414/EEC concerning the placing of plant protection products on the market, EU 1999: 1607/VI/97 (rev. 2) |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

## Table A 32: Summary gmz043004 trials

| | | | |
|---|---|---|---|
| **Field Trials, Crop Residue (Summary) :** Prosulfuron and Dicamba - Residue study in or on maize in Germany 2004 | | | |
| **Active Substance (common name):** | Dicamba | **Commercial Product (name):** | $Commercial.Name$ |
| **Crop/Crop Group:** | Maize | **Producer of commercial product:** | Syngenta AG |
| **Responsible body for reporting (name, address):** | Syngenta AG, Basel, Switzerland | **Indoor/Glasshouse/Outdoor:** | Field |
| **Country:** | GERMANY | **Other active substance in the formulation (common name and content):** | Prosulfuron (50 g AI/kg) |
| **Content of active substance (g/kg or g/L):** | 500 g AI/kg | **Residues calculated as:** | $Residue.Units$ |
| **Formulation (e.g. WP):** | A14031B WG | | |
| **Analytical Method:**<br>Dicamba (Cob, Kernel, Remainder Of Plant, Whole plant) REM 193.05; 0.01 mg/kg<br>Dicamba-5-OH (Cob, Kernel, Remainder Of Plant, Whole plant) REM 193.05; 0.01 mg/kg | | | |
| **Recovery data:**<br>Dicamba Cob Mean = 91% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Dicamba Kernel Mean = 87% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Dicamba Remainder Of Plant Mean = 101% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Dicamba Whole plant Mean = 91% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>NOA405873 Cob Mean = 90% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>NOA405873 Kernel Mean = 84% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>NOA405873 Remainder Of Plant Mean = 101% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>NOA405873 Whole plant Mean = 96% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range) | | | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| gmz043004<br>DE-HR-04-4320<br>GERMANY<br>(Europe North) | Maize<br>(Benicia) | 1.15 Apr 2004<br>2 –<br>3 - | Foliar | | 300 L/ha | 187.50 g ai/ha<br><br>(-, 0.2 L/ha, Trend 90) | 20 Jun 2004 | BBCH 17 - 18 | Whole plant | < 0.01 | 0.09 | 0.10 | 37 | 27 Jul 2004 | Field<br>A14031B<br><br>SP (max days): Dicamb |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 66 | 25 Aug 2004 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 66 | 25 Aug 2004 | |
| | | | | | | | | | Whole plant | < 0.01 | 0.09 | 0.10 | 66 | 25 Aug 2004 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | 0.02 | 102 | 30 Sep 2004 | a/Kernel: 329 Dicamba/Cob: |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | 0.02 | 102 | 30 Sep 2004 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 0.03 | 0.04 | 102 | 30 Sep 2004 | 329 Dicamba/Whole plant: 358 Dicamba/Remainder Of Plant: 293 Dicamba-5-OH/Remainder Of Plant: 293 Dicamba-5-OH/Whole plant: 358 Dicamba-5-OH/Kernel: 329 Dicamba-5-OH/Cob: 329 |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | Maize (Benicia) | 1.15 Apr 2004<br>2 –<br>3 - | Foliar | | 300 L/ha | 187.50 g ai/ha<br><br>(-, 1 L/ha, Etolag 149) | 20 Jun 2004 | BBCH 17 - 18 | Whole plant | < 0.01 | 0.03 | 0.04 | 37 | 27 Jul 2004 | Field A14031 B<br><br>SP (max days): Dicamba/Kernel: 329 Dicamba/Cob: |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 66 | 25 Aug 2004 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 66 | 25 Aug 2004 | |
| | | | | | | | | | Whole plant | < 0.01 | 0.05 | 0.06 | 66 | 25 Aug 2004 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 102 | 30 Sep 2004 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 102 | 30 Sep 2004 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | Dicamba-5-OH (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 0.01 | 0.02 | 102 | 30 Sep 2004 | 329 Dicamba/Whole plant: 358 Dicamba/Remainder Of Plant: 293 Dicamba-5-OH/Remainder Of Plant: 293 Dicamba-5-OH/Whole plant: 358 Dicamba-5-OH/Kernel: 329 Dicamba-5-OH/Cob: 329 |

(a) According to Codex (or other e.g. EU) classification

(b) Only if relevant

(c) Year must be indicated

(d) Minimum number of days after last application (Label pre-harvest interval, PHI, underline)

(e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included.

(*) Indicates sample taken prior to application

(#) Indicates corrected Residue values

(^) PHI calculated using cut date

(+) Indicates calculated Residue value

(DBA) Days Before Application

SP (max days): Maximum storage period

## A 2.1.3.1.5 Study T001033-07-REG

| | |
|---|---|
| Comments of zRMS: | Two decline residue field trial on maize was conducted in Northern Europe, in France, during 2007. Dicamba and prosulfuron were applied to corn by one broadcast foliar application of a 55 WG formulation (A14031E) at commercial rates at BBCH 18. On adjuvant Trend 90 was applied at a rate of 0.2 L/ha.<br>Corn whole plant, immature whole cob, whole cob and remaining plant were collected at intervals and normal commercial harvest for the determination of dicamba, 5-OH-dicamba and prosulfuron.<br><br>Results:<br><u>Dicamba</u><br>Residues of dicamba in treated corn whole plant samples ranged from below the limit of quantification (0.01 mg/kg) to 0.05 mg/kg at BBCH 39 and from below the limit of quantification to 0.01 mg/kg at BBCH 83-85. Except for one treated remaining cob sample (0.02 mg/kg at BBCH 89), no residues of dicamba were found in kernel, remaining cob and remaining plant treated corn samples at or above the limit of quantification (0.01 mg/kg).<br><br><u>5-hydroxy dicamba</u><br>Residues of 5-hydroxy dicamba in treated corn whole plant samples ranged from 0.01 to 0.11 mg/kg at BBCH 39 and declined below the limit of quantification (0.01 mg/kg) at BBCH 83-85. No residues of 5-hydroxy dicamba were found in treated kernel samples at BBCH 89. Residues of 5-hydroxy dicamba in remaining cob and remaining plant treated corn samples ranged from below the limit of quantification (0.01 mg/kg) to 0.01mg/kg.<br><br>No residues of dicamba or 5-hydroxy dicamba were detected at or above the limit of quantification (0.01 mg/kg) in any of the untreated corn samples.<br><br>Method REM 193.01 for dicamba and 5-hydroxy dicamba was used in all analyses.<br>Determination: GC-MS.<br>LOQ=0.01 mg/kg.<br><br>The study is acceptable. |

| | |
|---|---|
| Reference: | KCA1 6.3.1/05 |
| Report | Oppilliart, S., 2009. Dicamba (CGA57706) and Prosulfuron (CGA152005) – Residue study on Corn in France (North) in 2007.<br>Syngenta Report No. T001033-07-REG<br>Syngenta File No. A14031E_10809<br>GLP, unpublished |
| Guideline(s): | Yes<br>FAO Guidelines on Producing Pesticide Residues Data from Supervised Trials (Rome, 1990).<br>European Commission, General Recommendations for the Design, Preparation and Realization of Residue Trials; 7029/VI/95 (rev. 5, working document) |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

## Table A 33: Summary T001033-07-REG trials

| | | | |
|---|---|---|---|
| **Field Trials, Crop Residue (Summary) :** Dicamba (CGA57706) and Prosulfuron (CGA152005) - Residue study on Corn in France (north) in 2007 | | | |
| **Active Substance (common name):** | Dicamba | **Commercial Product (name):** | $Commercial.Name$ |
| **Crop/Crop Group:** | Corn | **Producer of commercial product:** | Syngenta AG |
| **Responsible body for reporting (name, address):** | Syngenta AG, Basel, Switzerland | **Indoor/Glasshouse/Outdoor:** | Field |
| **Country:** | FRANCE | **Other active substance in the formulation (common name and content):** | Prosulfuron (50 g AI/kg) |
| **Content of active substance (g/kg or g/L):** | 500 g AI/kg | **Residues calculated as:** | $Residue.Units$ |
| **Formulation (e.g. WP):** | A14031E WG | | |
| **Analytical Method:**<br>Dicamba (Cob, Cob + Kernel, Kernel, Remaining Plant, Whole plant) REM 193.01; 0.01 mg/kg<br>5-hydroxy dicamba (Cob, Cob + Kernel, Kernel, Remaining Plant, Whole plant) REM 193.01; 0.01 mg/kg | | | |
| **Recovery data:**<br>5-hydroxy dicamba Cob Mean = 83% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>5-hydroxy dicamba Kernel Mean = 80% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>5-hydroxy dicamba Remaining Plant Mean = 87% RSD = 8% (n = 4 in 0.01 - 0.1 mg/kg spiking range)<br>Dicamba Cob Mean = 82% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Dicamba Kernel Mean = 82% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Dicamba Remaining Plant Mean = 83% RSD = 3% (n = 4 in 0.01 - 0.1 mg/kg spiking range) | | | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | 5-hydroxy dicamba (mg/kg) | Analyte Sum (mg/kg) | | | |
| T001033-07-REG<br>FR-HR-07-0102<br>FRANCE (Europe North)<br>(-) | Corn (Anjou 219) | 1.21 Apr 2007<br>2 –<br>3 - | Foliar | | 213 L/ha | 199.60 g ai/ha<br><br>(-, - -) | 08 Jun 2007 | BBCH 18 | Whole plant | < 0.01 | 0.01 | 0.02 | 40 | 18 Jul 2007 | Field A14031E<br><br>SP (max days): Dicamba/Kernel: 280 |
| | | | | | | | | | Whole plant | < 0.01 | < 0.01 | < 0.02 | 45 | 23 Jul 2007 | |
| | | | | | | | | | Whole plant | < 0.01 | 0.01 | 0.02 | 56 | 03 Aug 2007 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 76 | 23 Aug 2007 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | < 0.01 + | < 0.02 + | 76 | 23 Aug 2007 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | 5-hydroxy dicamba (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 76 | 23 Aug 2007 | Dicamba/Cob + Kernel: 280<br>Dicamba/Whole plant: 316<br>Dicamba/Remaining Plant: 280<br>Dicamba/Cob: 280<br>5-hydroxy dicamb |
| | | | | | | | | | Remaining Plant | < 0.01 | 0.01 | 0.02 | 76 | 23 Aug 2007 | |
| | | | | | | | | | Cob | < 0.01 | 0.01 | 0.02 | 94 | 10 Sep 2007 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 0.01 + | 0.02 | 94 | 10 Sep 2007 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 94 | 10 Sep 2007 | |
| | | | | | | | | | Remaining Plant | < 0.01 | < 0.01 | < 0.02 | 94 | 10 Sep 2007 | |
| | | | | | | | | | Whole plant | < 0.01 | < 0.01 | < 0.02 | 115 | 01 Oct 2007 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 136 | 22 Oct 2007 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | < 0.01 + | < 0.02 + | 136 | 22 Oct 2007 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 136 | 22 Oct 2007 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | | | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Dicamba (mg/kg) | 5-hydroxy dicamba (mg/kg) | Analyte Sum (mg/kg) | | | |
| | | | | | | | | | Remaining Plant | < 0.01 | 0.01 | 0.02 | 136 | 22 Oct 2007 | a/Remaining Plant: 280 5-hydroxy dicamba/Cob + Kernel: 280 5-hydroxy dicamba/Kernel: 280 5-hydroxy dicamba/Whole plant: 316 5-hydroxy dicamba/Cob: 280 |

| | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| T001033-07-REG FR-HR-07-0103 FRANCE (Europe North) (-) | Corn (Matrixxo) | 1.25 Apr 2007 2 – 3 - | Foliar | | 200 L/ha | 187.80 g ai/ha (-, - -) | 07 Jun 2007 | BBCH 18 | Whole plant | 0.05 | 0.11 | 0.16 | 18 | 25 Jun 2007 | Field A14031E SP (max days): Dicamba/Kernel: 282 Dicamba/Cob + Kernel: 282 Dicamba/Remaining Plant: 282 Dicamba/Cob: 282 Dicamba/Whole plant: 339 5-hydroxy dicamb |
| | | | | | | | | | Whole plant | < 0.01 | 0.01 | 0.02 | 60 | 06 Aug 2007 | |
| | | | | | | | | | Whole plant | < 0.01 | 0.01 | 0.02 | 67 | 13 Aug 2007 | |
| | | | | | | | | | Cob | < 0.01 | 0.01 | 0.02 | 75 | 21 Aug 2007 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 0.01 + | 0.02 | 75 | 21 Aug 2007 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 75 | 21 Aug 2007 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 0.02 | 0.03 | 75 | 21 Aug 2007 | |
| | | | | | | | | | Whole plant | 0.01 | < 0.01 | 0.02 | 88 | 03 Sep 2007 | |
| | | | | | | | | | Cob | < 0.01 | < 0.01 | < 0.02 | 90 | 05 Sep 2007 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | < 0.01 + | < 0.02 + | 90 | 05 Sep 2007 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | 0.02 | 90 | 05 Sep 2007 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 0.01 | 0.02 | 90 | 05 Sep 2007 | |
| | | | | | | | | | Cob | 0.02 | 0.01 | 0.03 | 123 | 08 Oct 2007 | |
| | | | | | | | | | Cob + Kernel | 0.01 + | < 0.01 + | 0.02 | 123 | 08 Oct 2007 | |
| | | | | | | | | | Kernel | < 0.01 | < 0.01 | < 0.02 | 123 | 08 Oct 2007 | |

| | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | Remaining Plant | < 0.01 | 0.01 | 0.02 | 123 | 08 Oct 2007 | a/Remaining Plant: 2825-hydroxy dicamba/Whole plant: 3395-hydroxy dicamba/Kernel: 2825-hydroxy dicamba/Cob + Kernel: 2825-hydroxy dicamba/Cob: 282 |

(a) According to Codex (or other e.g. EU) classification

(b) Only if relevant

(c) Year must be indicated

(d) Minimum number of days after last application (Label pre-harvest interval, PHI, underline)

(e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included.

(*) Indicates sample taken prior to application

(#) Indicates corrected Residue values

(^) PHI calculated using cut date

(+) Indicates calculated Residue value

(DBA) Days Before Application

SP (max days): Maximum storage period

## A 2.1.4 Magnitude of residues in livestock

### A 2.1.4.1 Livestock feeding studies

No new data are submitted in the framework of this application.

## A 2.1.5 Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation)

### A 2.1.5.1 Distribution of the residue in peel/pulp

No applicable.

### A 2.1.5.2 Processing studies on a core set of representative processes

No new data are submitted in the framework of this application.

## A 2.1.6 Magnitude of residues in representative succeeding crops

### Study TK0223573-REG (Southern Europe)

| | |
|---|---|
| Comments of zRMS: | Two rotational crop trials were conducted to investigate the magnitude of residues in rotational crops in Southern France and Spain in 2014-2016 using formulation A7254B, a soluble concentrate containing nominally 480 g dicamba/L, applied to bare soil at a target rate of 480 g a.s./ha. This application rate corresponds to 1.7 the maximum seasonal application rate.<br>Residues of parent dicamba and 5-OH-dicamba were observed in barley commodities (whole plant, straw, grain) and carrot (tops and leaves). These were predominantly seen at the 30DAT plantback interval and declined over time across the later plantback intervals. This magnitude and distribution of residues is consistent with those seen in the confined rotational studies.<br>The observed residue levels for cereal commodities were significantly lower than those seen in the magnitude of residue trials for cereals. Residue levels of parent dicamba were lower in wheat and barley grain samples than the established MRLs for these crop commodities.<br>Very low residues of parent dicamba were observed in one trial only in carrot top and leaves at the nominal 30 and 60 day plant back intervals (0.02 mg/kg) and no detectable residues were observed at the final 270 day plantback timing.<br>Since these trials are overdosed compared to the intended use pattern residues of dicamba and 5-OH- dicamba is not expected in edible parts of crops above the LOQ of 0.01 mg/kg.<br><br>The study was conducted to comply with current data requirements and study guidelines (SANCO 7524/VI/95 rev.2, 22/7/1997).<br>The study is acceptable. |

| | |
|---|---|
| Reference: | KCA1 6.6.2/01 |
| Report | Andrews G, 2016a. Dicamba - Residue Study on Rotational Crops in Southern France and Spain in 2014. Battelle UK Ltd, Chelmsford, UK.<br>Study Dates: June 2014 – March 2016.<br>Syngenta Report No. TK0223573-REG.<br>Syngenta File No. A7254B_10456<br>GLP, unpublished |
| Guideline(s): | Commission of the European Communities, Crop Rotation Studies; |

7524/VI/95 (rev. 2).
Commission of the European Communities, General Recommendations for the Design, Preparation and Realisation of Residue Trials; 7029/VI/95 (rev. 5, working document).

Deviations: No

GLP: Yes

Acceptability: Yes

**Executive Summary**

Two field crop rotation trials were carried out in Southern France and Spain (southern Europe) between 2014 and 2016 to determine residues of dicamba and metabolite 5-OH-dicamba in crops grown in an area previously treated with dicamba. On the basis of the radio-labelled confined rotational crop study, crop commodities were analysed for dicamba and 5-OH-dicamba.

One application of dicamba as a 480 g/L SL formulation (A7254B) was made to bare soil plots at a target rate of 480 g a.s./ha. Applications were made nominally at 30 days before sowing (plot 2), 60 days (plot 3) before sowing and 270 days before sowing (plot 4).

The representative crops used were spring barley, carrot and spinach. Each crop was sown into each plot. They were maintained according to normal agricultural practices.

Due to poor crop development in the French trial (NC14031-01) carrot samples were not obtained.

Carrot roots and tops were sampled at normal commercial harvest (BBCH 79). Immature spinach samples were taken at BBCH 41 – 43 and mature spinach at normal commercial harvest (BBCH 49). Barley whole plant samples were taken at BBCH 47 – 49, grain and straw samples were taken at normal commercial harvest (BBCH 89). The samples were analysed for dicamba and 5-OH-dicamba using analytical method GRM022.07A. The method has been validated using data from these studies. The method will be fully validated and the method reissued by end August 2016.

The residues of dicamba and 5-OH-dicamba in rotational crops (cereal, leafy vegetable and root crops) are summarised in Table A 36 and Table A 37.

**Materials and Methods**

**Test Materials**

The test formulation was a 480 g a.i./L SL formulation of dicamba product code A7254B.

**Test Plants**

The rotational crops shown in Table A 34 were used.

**Table A 34: Rotational Crops used**

| Representative Crop Type | Crop | Plant Back Interval (Days) | Variety |
|---|---|---|---|
| **Trial NC14031-01** | | | |
| Leafy Vegetable | Spinach (*Spinacia oleracea*) (EPPO code SPQOL) | 31, 61 and 294 | Samos F1 |
| Root Vegetable | Carrot (*Daucus carota*) (EPPO code DAUCS) | 31, 61 and 294 | Chambor F1 |
| Cereal | Spring barley (*Hordeum vulgaris*) (EPPO code HORVX) | 31, 61 and 294 | KWS Irina |
| **Trial NC14031-02** | | | |
| Leafy Vegetable | Spinach (*Spinacia oleracea*) (EPPO code SPQOL) | 29, 60 and 270 | Matador |
| Root Vegetable | Carrot (*Daucus carota*) (EPPO code DAUCS) | 29, 60 and 270 | Nantese |
| Cereal | Spring barley (*Hordeum vulgaris*) (EPPO code HORVX) | 29, 60 and 270 | Mane |

**Trial sites**

The trial sites for the two trials were:

Trial NC14031-01 – Marsillargues, France

Trial NC14031-02 – Alcacer, Spain

**Test soils**
The test soils used in the two trials were:
Trial NC14031-01 – Silty clay loam, pH 8.5, soil OM 2.79%
Trial NC14031-02 – Loam sand, pH 8.7, soil OM 1.01%

**Study Design and Methods**
**Experimental conditions**
In each trial, plots were cultivated prior to making a single application of dicamba to a bare soil at a nominal rate of 480 g a.s./ha. Three rotated crops were used (spinach, carrot and barley), representative of the leafy crop, root crop and cereal crop groups (***SANCO 7524/VI/95 rev.2, 22/7/1997***). They were each planted back into the treated plots at nominal intervals of 30, 60 and 270 days.
In trial NC14031-01 spinach ($2.98x10^5$ seeds/ha), carrots ($5.56x10^5$ seeds/ha) and spring barley (180 kg seed/ha) were planted into the treated soil in side-by-side sub-plots at 31, 60 or 294 days after application.
In trial NC14031-02 spinach ($1.5x10^5$ seeds/ha), carrots ($2.0x10^5$ seeds/ha) and spring barley (270 kg seed/ha) were planted into the treated soil in side-by-side sub-plots at 29, 60 or 270 days after the application.

**Table A 35: Application and Planting Dates**

| Date | Activity | Plant-back interval (days after application) |
|---|---|---|
| **Trial NC14031-01** | | |
| 20 June 2014 | Moldboard plow P4 | - |
| 20 June 2014 | Rotovator P4 | - |
| 23 June 2014 | Application of A7254B to P4 (529.6 g a.s./ha) | 294 |
| 12 February 2015 | Moldboard plow P3 | - |
| 12 February 2015 | Application of A7254B to P3 (464.6 g a.s./ha) | 60 |
| 11 March 2015 | Moldboard plow P2 | - |
| 11 March 2015 | Rotovator P2 | - |
| 13 March 2015 | Application of A7254B to P2 (520.8 g a.s./ha) | 31 |
| 13 April 2015 | Rotovator all plots | - |
| 13 April 2015 | Sowing of Spinach, carrot and barley | - |
| **Trial NC14031-02** | | |
| 20 June 2014 | Rotovator P4 | - |
| 01 July 2014 | Application of A7254B to P4 (495.8 g a.s./ha) | 270 |
| 14 January 2014 | Rotovator P3 | - |
| 27 January 2015 | Application of A7254B to P3 (502.7 g a.s./ha) | 60 |
| 16 February 2015 | Rotovator P2 | - |
| 27 February 2015 | Application of A7254B to P2 (509.5 g a.s./ha) | 29 |
| 16 March 2015 | Rotovator plot P4 | - |
| 28 March 2015 | Sowing of spinach, carrot and barley | - |

**Sampling**
Immature spinach was sampled at BBCH 41 – 43 and immature spring barley (whole plant) at BBCH 47 – 49. Mature samples of spinach were taken at BBCH 49, of spring barley (grain and straw) at BBCH 89 and of carrot (roots and tops with leaves) at BBCH 79. Due to poor crop development, carrot samples were not obtained in Trial NC14031-01. Specimens were shipped frozen to the test facility for residue analysis and stored frozen at <-18°C for period of less than 10 months from sampling to analysis. On the basis of storage stability data for representative crops summarised in Point CA 6.1 no decline in residues of dicamba and 5-OH-dicamba in the crop matrices is anticipated over this storage period.

**Analysis**
Spinach, barley (whole plant and straw) and carrot samples were prepared in a food cutter with solid $CO_2$. Grain was ground in a cutting mill prior to analysis.
Dicamba and 5-OH-dicamba residues in crop samples were analysed by draft method GRM022.07A with a limit of quantification (LOQ) of 0.01 mg/kg for each analyte.

## Results and Discussion

The results of the analysis of crop samples are presented in Table A 36 and Table A 37.

**Table A 36: Analysis of Rotational Crops for Residues of Dicamba and 5-OH-dicamba (Trial NC14031-01)**

| Plant back interval (DAA) | Sampling interval (DAA) | Timing and/or Growth Stage (BBCH) | Crop Part** | Residue found (mg/kg)* | |
|---|---|---|---|---|---|
| | | | | Dicamba | 5-OH-dicamba |
| 31 | 84 | 39 | Barley whole plant | <0.01 | <0.01 |
| | 130 | 89 | Barley Grain | <0.01 | <0.01 |
| | 130 | 89 | Barley Straw | <0.01 | <0.01 |
| 60 | 113 | 39 | Barley whole plant | <0.01 | <0.01 |
| | 159 | 89 | Barley Grain | <0.01 | <0.01 |
| | 159 | 89 | Barley Straw | 0.01 | <0.01 |
| 294 | 347 | 39 | Barley whole plant | <0.01 | <0.01 |
| | 393 | 89 | Barley Grain | <0.01 | <0.01 |
| | 393 | 89 | Barley Straw | 0.01 | <0.01 |
| 31 | 76 | 41 – 43 | Immature spinach | <0.01 | <0.01 |
| | 89 | 49 | Spinach | <0.01 | <0.01 |
| 60 | 105 | 41 – 43 | Immature spinach | <0.01 | <0.01 |
| | 118 | 49 | Spinach | <0.01 | <0.01 |
| 294 | 339 | 41 – 43 | Immature spinach | <0.01 | <0.01 |
| | 352 | 49 | Spinach | <0.01 | <0.01 |

DAA – days after application
*No correction of results for either control residues or recovery values has been performed.
**Due to poor crop development, the carrot samples were not obtained.

**Table A 37: Analysis of Rotational Crops for Residues of Dicamba and 5-OH-dicamba (Trial NC14031-02)**

| Plant back interval (DAA) | Sampling interval (DAA) | Timing and/or Growth Stage (BBCH) | Crop Part | Residue found (mg/kg)* | |
|---|---|---|---|---|---|
| | | | | Dicamba | 5-OH-dicamba |
| 29 | 79 | 39 | Barley whole plant | 0.07 | 0.08 |
| | 138 | 89 | Barley Grain | 0.01 | <0.01 |
| | 138 | 89 | Barley Straw | 0.15 | 0.08 |
| 60 | 110 | 39 | Barley whole plant | <0.01 | <0.01 |
| | 169 | 89 | Barley Grain | <0.01 | <0.01 |
| | 169 | 89 | Barley Straw | 0.07 | <0.01 |
| 270 | 320 | 39 | Barley whole plant | <0.01 | <0.01 |
| | 379 | 89 | Barley Grain | <0.01 | <0.01 |
| | 379 | 89 | Barley Straw | 0.05 | <0.01 |
| 29 | 96 | 41 – 43 | Immature spinach | <0.01 | <0.01 |
| | 107 | 49 | Spinach | <0.01 | <0.01 |
| 60 | 127 | 41 – 43 | Immature spinach | <0.01 | <0.01 |
| | 138 | 49 | Spinach | <0.01 | <0.01 |
| 270 | 337 | 41 – 43 | Immature spinach | <0.01 | <0.01 |
| | 348 | 49 | Spinach | <0.01 | <0.01 |
| 29 | 140 | 79 | Carrot roots | <0.01 | <0.01 |
| | 140 | 79 | Carrot tops + leaves | 0.01 | <0.01 |
| 60 | 171 | 79 | Carrot roots | <0.01 | <0.01 |
| | 171 | 79 | Carrot tops + leaves | 0.02 | <0.01 |
| 270 | 381 | 79 | Carrot roots | <0.01 | <0.01 |
| | 381 | 79 | Carrot tops + leaves | <0.01 | <0.01 |

DAA – days after applicatio
*No correction of results for either control residues or recovery values has been performed.

(Andrews G, 2016a)

## A 2.1.6.1.1 Study NC14032 (Northern/Central Europe)

| | |
|---|---|
| Comments of zRMS: | Two field crop rotational trials (spinach, carrots and spring wheat or spring barley) were conducted to investigate the magnitude of residues in rotational crops in Northern France and the United Kingdom (northern Europe) between 2014 and 2016 using formulation A7254B, a soluble concentrate containing nominally 480 g dicamba/L, applied to bare soil at a target rate of 480 g a.s./ha.<br>In crops planted 30, 60 and 270 days after application, dicamba and 5-OH-dicamba residues in spinach immature leaves, spinach mature leaves, carrot roots, carrot tops + leaves, spring wheat forage, spring wheat grain, spring wheat straw, spring barley forage, spring barley grain and spring barley straw were <0.01 mg/kg (limit of quantification; LOQ).<br>No residues of dicamba and 5-OH-dicamba were found at or above the limit of quantification (LOQ) in any of the untreated samples.<br><br>The study was conducted to comply with current data requirements and study guidelines (7029/VI/95 and 7524/VI/95).<br>The study is acceptable. |

Reference: KCA1 6.6.2/02

Report: Andrews G, Austin R, 2016. Dicamba - Residue Study on Rotational Crops in the United Kingdom and Northern France in 2014. Battelle UK Ltd, Chelmsford, UK. Study Dates: June 2014 – April 2016.
Report No. NC14032.
Syngenta File No. A7254B_10460
GLP, unpublished

| | |
|---|---|
| Guideline(s): | Commission of the European Communities, Crop Rotation Studies; 7524/VI/95 (rev. 2).<br>Commission of the European Communities, General Recommendations for the Design, Preparation and Realisation of Residue Trials; 7029/VI/95 (rev. 5, working document). |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

**Executive Summary**

Two field crop rotation trials were carried out in Northern France and the United Kingdom (northern Europe) between 2014 and 2016 to determine any residues of dicamba and metabolite 5-OH-dicamba in crops grown in an area previously treated with dicamba. On the basis of the radio-labelled confined rotational crop study, crop commodities were analysed for dicamba and 5-OH-dicamba.

One application of dicamba as a 480 g/L SL formulation (A7254B) was made to bare soil plots at a target rate of 480 g a.s./ha. Applications were made nominally at 30 days before sowing (plot 2), 60 days (plot 3) before sowing and 270 days before sowing (plot4).

The representative crops used were spring barley, carrot and spinach. Each crop was sown into each plot. They were maintained according to normal agricultural practices.

Due to poor crop development in the French trial (NC14032-02), the spinach samples were not obtained.

Carrot roots and tops were sampled at normal commercial harvest (BBCH 79). Immature spinach samples were taken at BBCH 41 – 43 and mature spinach at normal commercial harvest (BBCH 49). Barley whole plane samples were taken at BBCH 47 – 49, grain and straw samples were taken at normal commercial harvest (BBCH 89). The samples were analysed for dicamba and 5-OH-dicamba using analytical method GRM022.07A. The method has been validated using data from these studies. The method will be fully validated and the method reissued by end August 2016.

**Materials**

**Test Materials**

The test formulation was a 480 g a.i./L SL formulation of dicamba product code A7254B.

**Test Plants**

The rotational crops shown in

Table A 38 were used.

**Table A 38: Rotational Crops used**

| Representative Crop Type | Crop | Plant Back Interval (Days) | Variety |
|---|---|---|---|
| **Trial NC14032-01** | | | |
| Leafy Vegetable | Spinach (*Spinacia oleracea*) (EPPO code SPQOL) | 31, 62 and 272 | Renegade F1 |
| Root Vegetable | Carrot (*Daucus carota*) (EPPO code DAUCS) | 31, 62 and 272 | Mokum |
| Cereal | Spring wheat (*Triticum aestivum*) (EPPO code TRZAS) | 31, 62 and 272 | KWS Willow |
| **Trial NC14032-02** | | | |
| Leafy Vegetable | Spinach (*Spinacia oleracea*) (EPPO code SPQOL) | 29, 60 and 270 | Lanzarote |
| Root Vegetable | Carrot (*Daucus carota*) (EPPO code DAUCS) | 29, 60 and 270 | Karotan RZ |
| Cereal | Spring barley (*Hordeum vulgaris*) (EPPO code HORVX) | 29, 60 and 270 | Sabastian |

**Trial sites**

The trial sites were:

Trial NC14032-01 – Bucknell, UK

Trial NC14032-02 – Mennessis, France

**Test soils**
The test soils used were:
Trial NC14032-01 – clay, pH 8.1 and soil OM 5.7%;
Trial NC14032-02 – sandy loam, pH 6.3 and soil OM 2.09%.

**Study Design and Methods**
**Experimental conditions**
In each trial, plots were cultivated prior to making a single application of dicamba to a bare soil at a nominal rate of 480 g ai/ha. Three rotated crops were used (spinach, carrot and barley), representative of the leafy crop, root crop and cereal crop groups (***SANCO 7524/VI/95 rev.2, 22/7/1997***). They were each planted back into the treated plots at nominal intervals of 30, 60 and 270 or 272 days.
In trial NC14032-01 spinach ($1.35x10^6$ seeds/ha), carrots ($1.6x10^5$ seeds/ha) and spring wheat (224 kg seed/ha) were planted into the treated soil in side-by-side sub-plots at 31, 62 or 272 days after application.
In trial NC14032-02 spinach ($8.0x10^5$ seeds/ha), carrots ($8.0x10^5$ seeds/ha) and spring barley ($3.3x10^6$ seed/ha) were planted into the treated soil in side-by-side sub-plots at 29, 60 or 270 days after the application.

**Table A 39: Application and Planting Dates**

| Date | Activity | Plant-back interval (days after application) |
|---|---|---|
| **Trial NC14032-01** | | |
| 09 June 2014 | Power harrow | - |
| 01 July 2014 | Application of A7254B to P4 (462.1 g ai/ha) | 272 |
| 27 January 2015 | Application of A7254B to P3 (480.7 g ai/ha) | 62 |
| 27 February 2015 | Application of A7254B to P2 (482.2 g ai/ha) | 31 |
| 20 March 2015 | Cultivator | - |
| 30 March 2015 | Sowing of Spinach, carrot and wheat | - |
| **Trial NC14032-02** | | |
| 19 June 2014 | Power harrow | - |
| 03 July 2014 | Application of A7254B to P4 (492.4 g ai/ha) | 270 |
| 08 February 2015 | Application of A7254B to P3 (492.4 g ai/ha) | 60 |
| 10 March 2015 | Application of A7254B to P2 (504.6 g ai/ha) | 29 |
| 01 April 2015 | Alternative harrow | - |
| 09 April 2015 | Power harrow | - |
| 09 April 2015 | Sowing of spinach, carrot and barley | - |

**Sampling**
Immature spinach was sampled at BBCH 41 – 43 and immature spring barley or wheat (whole plant) at BBCH 39. Mature samples of spinach were taken at BBCH 49, of spring wheat or barley (grain and straw) at BBCH 89 and of carrot (roots and tops with leaves) at BBCH 49. Due to poor crop development, spinach samples were not obtained in Trial NC14032-02. Specimens were shipped frozen to the test facility for residue analysis and stored frozen at < -18°C for period of less than 10 months from sampling to analysis. On the basis of storage stability data for representative crops summarised in Point CA 6.1 no decline in residues of dicamba and 5-OH-dicamba in the crop matrices is anticipated over this storage period.

**Analysis**
Spinach, wheat or barley (whole plant and straw) and carrot samples were prepared in a food cutter with solid $CO_2$. Grain was ground in a cutting mill prior to analysis.
Dicamba and 5-OH-dicamba residues in crop samples were analysed by draft method GRM022.07A with a limit of quantification (LOQ) of 0.01 mg/kg for each analyte.

**Results and Discussion**
The results of the analysis of crop samples are presented in

Table A 40 and

Table A 41.

**Table A 40: Analysis of Rotational Crops for Residues of Dicamba and 5-OH-dicamba (Trial NC14032-01)**

| Plant back interval (DAA) | Sampling interval (DAA) | Timing and/or Growth Stage (BBCH) | Crop Part | Residue found (mg/kg)* | |
|---|---|---|---|---|---|
| | | | | Dicamba | 5-OH-dicamba |
| 31 | 112 | 39 | Wheat whole plant | <0.01 | <0.01 |
| | 195 | 89 | Wheat Grain | <0.01 | <0.01 |
| | 195 | 89 | Wheat Straw | <0.01 | <0.01 |
| 62 | 143 | 39 | Wheat whole plant | <0.01 | <0.01 |
| | 226 | 89 | Wheat Grain | <0.01 | <0.01 |
| | 226 | 89 | Wheat Straw | <0.01 | <0.01 |
| 272 | 353 | 39 | Wheat whole plant | <0.01 | <0.01 |
| | 436 | 89 | Wheat Grain | <0.01 | <0.01 |
| | 436 | 89 | Wheat Straw | <0.01 | <0.01 |
| 31 | 116 | 41 – 43 | Immature spinach | <0.01 | <0.01 |
| | 123 | 49 | Spinach | <0.01 | <0.01 |
| 62 | 147 | 41 – 43 | Immature spinach | <0.01 | <0.01 |
| | 154 | 49 | Spinach | <0.01 | <0.01 |
| 272 | 357 | 41 – 43 | Immature spinach | <0.01 | <0.01 |
| | 364 | 49 | Spinach | <0.01 | <0.01 |
| 31 | 179 | 49 | Carrot roots | <0.01 | <0.01 |
| | 179 | 49 | Carrot tops + leaves | <0.01 | <0.01 |
| 62 | 210 | 49 | Carrot roots | <0.01 | <0.01 |
| | 210 | 49 | Carrot tops + leaves | <0.01 | <0.01 |
| 272 | 420 | 49 | Carrot roots | <0.01 | <0.01 |
| | 420 | 49 | Carrot tops + leaves | <0.01 | <0.01 |

DAA – days after application
*No correction of results for either control residues or recovery values has been performed.

**Table A 41: Analysis of Rotational Crops for Residues of Dicamba and 5-OH-dicamba (Trial NC14032-02)**

| Plant back interval (DAA) | Sampling interval (DAA) | Timing and/or Growth Stage (BBCH) | Crop Part | Residue found (mg/kg)* | |
|---|---|---|---|---|---|
| | | | | Dicamba | 5-OH-dicamba |
| 29 | 113 | 39 | Wheat whole plant | <0.01 | <0.01 |
| | 154 | 89 | Wheat Grain | <0.01 | <0.01 |
| | 154 | 89 | Wheat Straw | <0.01 | <0.01 |
| 60 | 143 | 39 | Wheat whole plant | <0.01 | <0.01 |
| | 184 | 89 | Wheat Grain | <0.01 | <0.01 |
| | 184 | 89 | Wheat Straw | <0.01 | <0.01 |
| 270 | 363 | 39 | Wheat whole plant | <0.01 | <0.01 |
| | 404 | 89 | Wheat Grain | <0.01 | <0.01 |
| | 404 | 89 | Wheat Straw | <0.01 | <0.01 |
| 29 | 224 | 49 | Carrot roots | <0.01 | <0.01 |
| | 224 | 49 | Carrot tops + leaves | <0.01 | <0.01 |
| 60 | 254 | 49 | Carrot roots | <0.01 | <0.01 |
| | 254 | 49 | Carrot tops + leaves | <0.01 | <0.01 |
| 270 | 474 | 49 | Carrot roots | <0.01 | <0.01 |
| | 474 | 49 | Carrot tops + leaves | <0.01 | <0.01 |

DAA – days after application
*No correction of results for either control residues or recovery values has been performed.
Due to poor crop development, no spinach samples were obtained.

(Andrews G, Austin R, 2016)

## A 2.1.7 Other/Special Studies

No new data are submitted in the framework of this application.

## A 2.2 Mesotrione

### A 2.2.1 Stability of residues

#### A 2.2.1.1 Stability of residues during storage of samples

No new data are submitted in the framework of this application.

### A 2.2.2 Nature of residues in plants, livestock and processed commodities

No new data are submitted in the framework of this application.

### A 2.2.3 Magnitude of residues in plants

No new data are submitted in the framework of this application. Summaries of the relevant residue studies are presented below for the sake of clarity.

#### A 2.2.3.1 Maize

**Table A 42: Comparison of intended and critical EU GAPs**

| Type of GAP | Number of applications | Application rate per treatment (precise unit) | Interval between application | Growth stage at last application | PHI (days) |
|---|---|---|---|---|---|
| cGAP EU (UK, 2015, 2015a) | 1 | 0.150 kg a.s./ha | N/A | BBCH 12-18 | N/A[(a)] |
| cGAP EU (Art. 12, EFSA, 2016) | 1 | 0.150 kg a.s./ha | N/A | BBCH 12-19 | N/A[(a)] |
| Intended cGAP; use number 3,6,7* | 1 | 0.090 kg a.s./ha | N/A | BBCH 12-19 | N/A[(a)] |

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0
N/A – not applicable
(a) PHI is determined by crop maturity

##### A 2.2.3.1.1 Study RR 96-078B – EU reviewed

| | |
|---|---|
| Comments of zRMS: | This study has been reviewed at the EU level. |

| | |
|---|---|
| Reference: | KCA2 6.3.1/01 |
| Report | Barnes J P, 1997. ZA1296 - Residue Levels in Maize from Trials Carried out in Germany During 1995<br>Syngenta Report No. RR 96-078B<br>Syngenta File No. ZA1296/0409<br>GLP, unpublished |
| Guideline(s): | Yes<br>Commission of the European Communities, General Recommendations for the Design, Preparation and Realization of Residue Trials; 7029/VI/95 |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

## Table A 43: Summary of RR 96-078B trials

| | | | |
|---|---|---|---|
| **Field Trials, Crop Residue (Summary) :** ZA1296 - Residue Levels in Maize from Trials Carried out in Germany During 1995 | | | |
| **Active Substance (common name):** | Mesotrione | **Commercial Product (name):** | $Commercial.Name$ |
| **Crop/Crop Group:** | Maize | **Producer of commercial product:** | Syngenta AG |
| **Responsible body for reporting (name, address):** | Syngenta AG, Basel, Switzerland | **Indoor/Glasshouse/Outdoor:** | Field |
| **Country:** | GERMANY | **Other active substance in the formulation (common name and content):** | None |
| **Content of active substance (g/kg or g/L):** | 100 g a.s./L | **Residues calculated as:** | Mesotrione |
| **Formulation (e.g. WP):** | A12739 SC | | |
| **Analytical Method:**<br>AMBA (Cob + Grain, Cob + Grain + Husk, Forage, Grain, Silage, Whole plant) TMR0643B; 0.01 mg/kg<br>For analysis, the residues of MNBA and Mesotrione were both reduced/converted to AMBA (Conversion Factor = 0.153) and the method analysed for AMBA | | | |
| **Recovery data:** | | | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| RR 96-078B RS-9512-B1 (94-GR-95-854) GERMANY (Europe North) | Maize (Diamant) | 1.29 Apr 1995<br>2 –<br>3 - | Foliar | 100.00 g a.s./L | 300 L/ha | 150.00 g a.s./ha<br>(-) | 17 Jun 1995 | BBCH 17 | Whole plant | 0.08 | 0 | 17 Jun 1995 | Field<br>SP (max days):<br>AMBA/Silage: 434<br>AMBA/Cob + Grain + Husk: 424<br>AMBA/Grain: 424<br>AMBA/Forage: 470<br>AMBA/Whole plant: 502<br>AMBA/Cob + Grain: 424 |
| | | | | | | | | | Whole plant | < 0.01 | 13 | 30 Jun 1995 | |
| | | | | | | | | | Forage | < 0.01 | 32 | 19 Jul 1995 | |
| | | | | | | | | | Silage | < 0.01 | 68 | 24 Aug 1995 | |
| | | | | | | | | | Cob + Grain | < 0.01 | 78 | 03 Sep 1995 | |
| | | | | | | | | | Cob + Grain + Husk | < 0.01 | 78 | 03 Sep 1995 | |
| | | | | | | | | | Grain | < 0.01 | 78 | 03 Sep 1995 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| RR 96-078B RS-9512-G1 (94-GR-95-855) GERMANY (Europe North) | Maize (General) | 1.26 Apr 1995<br>2 –<br>3 - | Foliar | 100.00 g a.s./L | 300 L/ha | 150.00 g a.s./ha<br><br>(-) | 19 Jun 1995 | BBCH 17 | Whole plant | 10.31 | 0 | 19 Jun 1995 | Field<br><br>SP (max days):<br>AMBA/Silage: 427<br>AMBA/Grain: 386<br>AMBA/Whole plant: 500<br>AMBA/Cob + Grain + Husk: 386<br>AMBA/Forage: 468<br>AMBA/Cob + Grain: 386 |
| | | | | | | | | | Whole plant | < 0.01 | 14 | 03 Jul 1995 | |
| | | | | | | | | | Forage | < 0.01 | 32 | 21 Jul 1995 | |
| | | | | | | | | | Silage | < 0.01 | 73 | 31 Aug 1995 | |
| | | | | | | | | | Cob + Grain + Husk | < 0.01 | 114* | 11 Oct 1995 | |
| | | | | | | | | | Cob + Grain | < 0.01 | 114 | 11 Oct 1995 | |
| | | | | | | | | | Grain | < 0.01 | 114 | 11 Oct 1995 | |
| RR 96-078B RS-9512-G2 (94-GR-95-856) GERMANY (Europe North) | Maize (Graf) | 1.02 May 1995<br>2 –<br>3 - | Foliar | 100.00 g a.s./L | 300 L/ha | 150.00 g a.s./ha<br><br>(-) | 19 Jun 1995 | BBCH 16 - 17 | Whole plant | 11.57, 11.54 | 0 | 19 Jun 1995 | Field<br><br>SP (max days):<br>AMBA/Silage: 422<br>AMBA/Cob + Grain + Husk: 386<br>AMBA/Grain: 386<br>AMBA/Whole plant: 500<br>AMBA/Forage: 468<br>AMBA/Cob + Grain: 386 |
| | | | | | | | | | Whole plant | < 0.01 | 14 | 03 Jul 1995 | |
| | | | | | | | | | Forage | < 0.01 | 32 | 21 Jul 1995 | |
| | | | | | | | | | Silage | < 0.01 | 78 | 05 Sep 1995 | |
| | | | | | | | | | Cob + Grain | < 0.01 | 114 | 11 Oct 1995 | |
| | | | | | | | | | Cob + Grain + Husk | < 0.01 | 114 | 11 Oct 1995 | |
| | | | | | | | | | Grain | < 0.01 | 114 | 11 Oct 1995 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| RR 96-078B<br>RS-9512-K1<br>(94-GR-95-857)<br>GERMANY (Europe North) | Maize (Anjou 207) | 1.02 May 1995<br>2 –<br>3 - | Foliar | 100.00 g a.s./L | 300 L/ha | 150.00 g a.s./ha<br><br>(-) | 20 Jun 1995 | BBCH 16 - 17 | Whole plant | 5.81, 6.14 | 0 | 20 Jun 1995 | Field<br><br>SP (max days): AMBA/Silage: 429 AMBA/Cob + Grain + Husk: 387 AMBA/Grain: 387 AMBA/Whole plant: 499 AMBA/Forage: 469 AMBA/Cob + Grain: 387 |
| | | | | | | | | | Whole plant | < 0.01 | 15 | 05 Jul 1995 | |
| | | | | | | | | | Forage | < 0.01 | 30 | 20 Jul 1995 | |
| | | | | | | | | | Silage | < 0.01 | 70 | 29 Aug 1995 | |
| | | | | | | | | | Cob + Grain | < 0.01 | 112 | 10 Oct 1995 | |
| | | | | | | | | | Cob + Grain + Husk | < 0.01 | 112 | 10 Oct 1995 | |
| | | | | | | | | | Grain | < 0.01 | 112 | 10 Oct 1995 | |

(a) According to Codex (or other e.g. EU) classification

(b) Only if relevant

(c) Year must be indicated

(d) Minimum number of days after last application (Label pre-harvest interval, PHI, underline)

(e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included.

(*) Indicates sample taken prior to application

(#) Indicates corrected Residue values

(^) PHI calculated using cut date

(+) Indicates calculated Residue value

(DBA) Days Before Application

SP (max days): Maximum storage period

### A 2.2.3.1.2 Study T011085-06-REG– EU reviewed

| | |
|---|---|
| Comments of zRMS: | This study has been reviewed at the EU level. |

| | |
|---|---|
| Reference: | KCA2 6.3.1/02 |
| Report | Heillaut C., 2009. Glyphosate (ASF71), Mesotrione (ZA1296) and S-Metolachlor (CGA77102) - Residue study on GA21 (MON-ØØØ21-9) Corn in France (north) and Czech Republic in 2007<br>Syngenta Report No. T011085-06-REG<br>Syngenta File No. A15189G_10009<br>GLP, unpublished |
| Guideline(s): | Yes<br>FAO Guidelines on Producing Pesticide Residues Data from Supervised Trials (Rome, 1990).<br>Commission of the European Communities, General Recommendations for the Design, Preparation and Realization of Residue Trials; 7035/VI/95 (rev. 5, working document). |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

## Table A 44: Summary of T011085-06-REG trials

| | | | |
|---|---|---|---|
| **Field Trials, Crop Residue (Summary) :** Glyphosate (ASF71), Mesotrione (ZA1296) and S-Metolachlor (CGA77102) - Residue study on GA21 (MON-ØØØ21-9) Corn in France (north) and Czech Republic in 2007 | | | |
| **Active Substance (common name):** | Mesotrione | **Commercial Product (name):** | $Commercial.Name$ |
| **Crop/Crop Group:** | Corn | **Producer of commercial product:** | Syngenta AG |
| **Responsible body for reporting (name, address):** | Syngenta AG, Basel, Switzerland | **Indoor/Glasshouse/Outdoor:** | Field |
| **Country:** | CZECH REPUBLIC, FRANCE | **Other active substance in the formulation (common name and content):** | S-Metolachlor (250 g a.s./L), Glyphosate (360 g a.s./L) |
| **Content of active substance (g/kg or g/L):** | 25 g a.s./L | **Residues calculated as:** | Mesotrione |
| **Formulation (e.g. WP):** | A15189G SC | | |
| **Analytical Method:**<br>Mesotrione (Cob, Cob + Kernel, Kernel, Remaining Plant, Whole plant) RAM 366/01; 0.01 mg/kg | | | |
| **Recovery data:**<br>Mesotrione Cob Mean = 92% RSD = 3% (n = 4 in 0.01 - 0.1 mg/kg spiking range)<br>Mesotrione Kernel Mean = 89% RSD = 4% (n = 4 in 0.01 - 0.1 mg/kg spiking range)<br>Mesotrione Remaining Plant Mean = 88% RSD = 7% (n = 4 in 0.01 - 0.1 mg/kg spiking range)<br>Mesotrione Whole plant Mean = 98% RSD = 8% (n = 4 in 0.01 - 0.1 mg/kg spiking range) | | | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity / Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| T011085-06-REG<br>CZ-HR-07-0064<br>CZECH REPUBLIC (Europe North) | Corn (GA21 (MON-00021-9)) | 1.27 Apr 2007<br>2 –<br>3 - | Foliar | 25.00 g a.s./L | 306 L/ha | 156.00 g a.s./ha<br><br>(-) | 30 May 2007 | BBCH 15 - 16 | Whole plant | < 0.01 | 65 | 03 Aug 2007 | Field A15189G<br><br>SP (max days): Mesotrione/Cob + Kernel: 424 Mesotrione/Whole plant: 457 Mesotrione/Remaining Plant: 424 Mesotrione/Kernel: 424 Mesotrione/Cob: 424 |
| | | | | | | | | | Cob | < 0.01 | 98 | 05 Sep 2007 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 98 | 05 Sep 2007 | |
| | | | | | | | | | Kernel | < 0.01 | 98 | 05 Sep 2007 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 98 | 05 Sep 2007 | |
| | | | | | | | | | Whole plant | < 0.01 | 104 | 11 Sep 2007 | |
| | | | | | | | | | Cob | < 0.01 | 134 | 11 Oct 2007 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 134 | 11 Oct 2007 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity / Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| | | | | | | | | | Kernel | < 0.01 | 134 | 11 Oct 2007 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 134 | 11 Oct 2007 | |
| T011085-06-REG<br>FR-HR-07-0063<br>FRANCE (Europe North)<br>(-) | Corn (GA21 (MON-00021-9)) | 1.03 May 2007<br>2 –<br>3 - | Foliar | 25.00 g a.s./L | 308 L/ha | 158.00 g a.s./ha<br><br>(-) | 07 Jun 2007 | BBCH 15 | Whole plant | < 0.01 | 61 | 07 Aug 2007 | Field A15189G<br><br>SP (max days): Mesotrione/Remaining Plant: 422 Mesotrione/Whole plant: 453 Mesotrione/Kernel: 422 Mesotrione/Cob + Kernel: 422 Mesotrione/Cob: 422 |
| | | | | | | | | | Cob | < 0.01 | 92 | 07 Sep 2007 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 92 | 07 Sep 2007 | |
| | | | | | | | | | Kernel | < 0.01 | 92 | 07 Sep 2007 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 92 | 07 Sep 2007 | |
| | | | | | | | | | Whole plant | < 0.01 | 109 | 24 Sep 2007 | |
| | | | | | | | | | Cob | < 0.01 | 137 | 22 Oct 2007 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 137 | 22 Oct 2007 | |
| | | | | | | | | | Kernel | < 0.01 | 137 | 22 Oct 2007 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 137 | 22 Oct 2007 | |

(a) According to Codex (or other e.g. EU) classification

(b) Only if relevant

(c) Year must be indicated

(d) Minimum number of days after last application (Label pre-harvest interval, PHI, underline)

(e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included.

(*) Indicates sample taken prior to application

(#) Indicates corrected Residue values

(^) PHI calculated using cut date

(+) Indicates calculated Residue value

(DBA) Days Before Application

SP (max days): Maximum storage period

### A 2.2.3.1.3 Study 009533-07-REG– EU reviewed

| | |
|---|---|
| Comments of zRMS: | This study has been reviewed at the EU level. |

| | |
|---|---|
| Reference: | KCA2 6.3.1/03 |
| Report | Heillaut C., 2009a. Glyphosate, Mesotrione and S-Metolachlor - Residue study on GA21 (MON-00021-9) corn in Denmark and Sweden in 2008<br>Syngenta Report No. T009533-07-REG<br>Syngenta File No. A15189G_10014<br>GLP, unpublished |
| Guideline(s): | Yes<br>FAO Guidelines on Producing Pesticide Residues Data from Supervised Trials (Rome, 1990).<br>Commission of the European Communities, General Recommendations for the Design, Preparation and Realization of Residue Trials; 7029/VI/95 (rev. 5, working document).<br>Guidelines and Criteria for the Preparation and Presentation of Complete Dossiers and of Summary Dossiers for the Inclusion of Active Substances in Annex I of Directive 91/414/EEC (Article 5.3 and 8.2), 1996.<br>EC (European Commission), Guidelines for the generation of data concerning residues as provided in Annex II part A, section 6 and Annex III, part A, section 8 of Directive 91/414/EEC concerning the placing of plant protection products on the market, EU 1999: 1607/VI/97 (rev. 2)<br>Commission of the European Communities. Guidance Document on Residue Analytical Methods (SANCO/825/00 rev. 7, 17/03/2004).<br>Commission of the European Communities. Guidance Document on Residue Analytical Methods (SANCO/3029/99 rev. 4, 11/07/2000).<br>The national requirements are based on the OECD Principles of Good Laboratory Practice, which are accepted by regulatory authorities throughout the European Community, the United States of America (FDA and EPA) and Japan (MHW, MAFF and METI) on the basis of intergovernmental agreements. |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

## Table A 45: Summary of T009533-07-REG trials

| | | | |
|---|---|---|---|
| **Field Trials, Crop Residue (Summary) :** Glyphosate, Mesotrione and S-Metolachlor - Residue study on GA21 (MON-00021-9) corn in Denmark and Sweden in 2008 | | | |
| **Active Substance (common name):** | Mesotrione | **Commercial Product (name):** | $Commercial.Name$ |
| **Crop/Crop Group:** | Corn | **Producer of commercial product:** | Syngenta AG |
| **Responsible body for reporting (name, address):** | Syngenta AG, Basel, Switzerland | **Indoor/Glasshouse/Outdoor:** | Field |
| **Country:** | SWEDEN, DENMARK | **Other active substance in the formulation (common name and content):** | S-Metolachlor (250 g a.s./L), Glyphosate (360 g a.s./L) |
| **Content of active substance (g/kg or g/L):** | 25 g a.s./L | **Residues calculated as:** | Mesotrione |
| **Formulation (e.g. WP):** | A15189G SC | | |
| **Analytical Method:**<br>Mesotrione (Cob, Cob + Kernel, Kernel, Remaining Plant, Whole plant) RAM 366/01; 0.01 mg/kg | | | |
| **Recovery data:**<br>Mesotrione Cob Mean = 100% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Mesotrione Kernel Mean = 91% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Mesotrione Remaining Plant Mean = 89% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Mesotrione Whole plant Mean = 102% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range) | | | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| T009533-07-REG<br>DK-HR-08-0007<br>DENMARK (Europe North) | Corn (GA21 (MON-00021-9)) | 1.21 May 2008<br>2 –<br>3 - | Foliar | 25.00 g a.s./L | 224 L/ha | 167.70 g a.s./ha<br>(-) | 09 Jun 2008 | BBCH 15 | Whole plant | < 0.01 | 63 | 11 Aug 2008 | Field<br>A15189G<br>SP (max days):<br>Mesotrione/Cob + Kernel: 228<br>Mesotrione/Whole plant: 257<br>Mesotrione/Remaining Plant: 228<br>Mesotrione/Kernel: 228<br>Mesotrione/Cob: 228 |
| | | | | | | | | | Cob | < 0.01 | 92 | 09 Sep 2008 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 92 | 09 Sep 2008 | |
| | | | | | | | | | Kernel | < 0.01 | 92 | 09 Sep 2008 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 92 | 09 Sep 2008 | |
| | | | | | | | | | Cob | < 0.01 | 128 | 15 Oct 2008 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 128 | 15 Oct 2008 | |
| | | | | | | | | | Kern3el | < 0.01 | 128 | 15 Oct 2008 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| | | | | | | | | | Remaining Plant | < 0.01 | 128 | 15 Oct 2008 | |
| | Corn (GA21 (MON-00021-9)) | 1.21 May 2008<br>2 –<br>3 - | Foliar | 25.00 g a.s./L | 196 L/ha | 147 g a.s./ha<br><br>(NIS, 0.49 L/ha)<br>(AMS, 2.94 L/ha) | 09 Jun 2008 | BBCH 15 | Whole plant | < 0.01 | 63 | 11 Aug 2008 | |
| | | | | | | | | | Cob | < 0.01 | 92 | 09 Sep 2008 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 92 | 09 Sep 2008 | |
| | | | | | | | | | Kernel | < 0.01 | 92 | 09 Sep 2008 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 92 | 09 Sep 2008 | |
| | | | | | | | | | Cob | < 0.01 | 128 | 15 Oct 2008 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 128 | 15 Oct 2008 | |
| | | | | | | | | | Kernel | < 0.01 | 128 | 15 Oct 2008 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 128 | 15 Oct 2008 | |
| T009533-07-REG SE-HR-08-0003 SWEDEN (Europe North) | Corn (GA21 (MON-00021-9)) | 1.16 May 2008<br>2 –<br>3 - | Foliar | 25.00 g a.s./L | 198 L/ha | 148.50 g a.s./ha<br><br>(-) | 15 Jun 2008 | BBCH 16 - 17 | Whole plant | < 0.01 | 65 | 19 Aug 2008 | Field A15189G<br><br>SP (max days): Mesotrione/Remaining Plant: 220 Mesotrione/Whole plant: 249 Mesotrione/Kernel: 220 Mesotrione/Cob + Kernel: 220 Mesotrione/Cob: 220 |
| | | | | | | | | | Cob | < 0.01 | 94 | 17 Sep 2008 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 94 | 17 Sep 2008 | |
| | | | | | | | | | Kernel | < 0.01 | 94 | 17 Sep 2008 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 94 | 17 Sep 2008 | |
| | | | | | | | | | Cob | < 0.01 | 120 | 13 Oct 2008 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 120 | 13 Oct 2008 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| | | | | | | | | | Kernel | <u>< 0.01</u> | 120 | 13 Oct 2008 | |
| | | | | | | | | | Remaining Plant | <u>< 0.01</u> | 120 | 13 Oct 2008 | |
| | Corn (GA21 (MON-00021-9)) | 1.16 May 2008<br>2 –<br>3 - | Foliar | 25.00 g a.s./L | 207 L/ha | 155.30 g a.s./ha<br><br>(NIS, 0.52 L/ha)<br>(AMS, 3.11 L/ha) | 15 Jun 2008 | BBCH 16 - 17 | Whole plant | < 0.01 | 65 | 19 Aug 2008 | |
| | | | | | | | | | Cob | < 0.01 | 94 | 17 Sep 2008 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 94 | 17 Sep 2008 | |
| | | | | | | | | | Kernel | < 0.01 | 94 | 17 Sep 2008 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 94 | 17 Sep 2008 | |
| | | | | | | | | | Cob | < 0.01 | 120 | 13 Oct 2008 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 120 | 13 Oct 2008 | |
| | | | | | | | | | Kernel | < 0.01 | 120 | 13 Oct 2008 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 120 | 13 Oct 2008 | |

(a) According to Codex (or other e.g. EU) classification

(b) Only if relevant

(c) Year must be indicated

(d) Minimum number of days after last application (Label pre-harvest interval, PHI, underline)

(e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included.

### A 2.2.3.1.4 Study T00920-09-REG– EU reviewed

| | |
|---|---|
| Comments of zRMS: | This study has been reviewed at the EU level. |

| | |
|---|---|
| Reference: | KCA2 6.3.1/04 |
| Report | Meyer M., 2011. Mesotrione - Residue study on Field Corn in Germany and the United Kingdom in 2009<br>Syngenta Report No. T000920-09-REG<br>Syngenta File No. A14203B_10105<br>GLP, unpublished |
| Guideline(s): | Yes<br>FAO Guidelines on Producing Pesticide Residues Data from Supervised Trials (Rome, 1990).<br>Commission of the European Communities, General Recommendations for the Design, Preparation and Realization of Residue Trials; 7029/VI/95 (rev. 5, working document).<br>The national requirements are based on the OECD Principles of Good Laboratory Practice, which are accepted by regulatory authorities throughout the European Community, the United States of America (FDA and EPA) and Japan (MHW, MAFF and METI) on the basis of intergovernmental agreements. |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

## Table A 46: Summary of T00920-09-REG trials

| | | | |
|---|---|---|---|
| **Field Trials, Crop Residue (Summary) :** Mesotrione - Residue study on Field Corn in Germany and the United Kingdom in 2009 | | | |
| **Active Substance (common name):** | Mesotrione | **Commercial Product (name):** | $Commercial.Name$ |
| **Crop/Crop Group:** | Field Corn | **Producer of commercial product:** | Syngenta AG |
| **Responsible body for reporting (name, address):** | Syngenta AG, Basel, Switzerland | **Indoor/Glasshouse/Outdoor:** | Field |
| **Country:** | GERMANY, UNITED KINGDOM | **Other active substance in the formulation (common name and content):** | None |
| **Content of active substance (g/kg or g/L):** | 500 g AI/kg | **Residues calculated as:** | Mesotrione |
| **Formulation (e.g. WP):** | A14203B WG | | |
| **Analytical Method:**<br>Mesotrione (Cob, Cob + Kernel, Kernel, Remaining Plant, Whole plant) RAM 366/01, modified; 0.01 mg/kg | | | |
| **Recovery data:**<br>Mesotrione Cob Mean = 92% RSD = 17% (n = 4 in 0.01 - 0.1 mg/kg spiking range)<br>Mesotrione Kernel Mean = 98% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Mesotrione Remaining Plant Mean = 96% RSD = 10% (n = 4 in 0.01 - 0.1 mg/kg spiking range) | | | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| T000920-09-REG<br>09-DE-059<br>GERMANY<br>(Europe North) | Field Corn (Nescio) | 1.01 May 2009<br>2 –<br>3 - | Foliar | 500.00 g AI/kg | 284 L/ha | 143.00 g a.s./ha<br>(-) | 05 Jun 2009 | BBCH 16 - 18 | Whole plant | < 0.01 | 34 | 09 Jul 2009 | Field<br>SP (max days):<br>Mesotrione/Cob + Kernel: 377<br>Mesotrione/Whole plant: 390<br>Mesotrione/Remaining Plant: 377<br>Mesotrione/Kernel: 342<br>Mesotrione/Cob: 342 |
| | | | | | | | | | Cob + Kernel | < 0.01 | 47 | 22 Jul 2009 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 47 | 22 Jul 2009 | |
| | | | | | | | | | Whole plant | < 0.01 | 47 | 22 Jul 2009 | |
| | | | | | | | | | Whole plant | < 0.01 | 66 | 10 Aug 2009 | |
| | | | | | | | | | Cob | < 0.01 | 82 | 26 Aug 2009 | |
| | | | | | | | | | Kernel | < 0.01 | 82 | 26 Aug 2009 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 82 | 26 Aug 2009 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 82 | 26 Aug 2009 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| | | | | | | | | | Cob | < 0.01 | 91 | 04 Sep 2009 | |
| | | | | | | | | | Kernel | < 0.01 | 91 | 04 Sep 2009 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 91 | 04 Sep 2009 | |
| | | | | | | | | | Whole plant | < 0.01 | 110 | 23 Sep 2009 | |
| | | | | | | | | | Cob | < 0.01 | 143 | 26 Oct 2009 | |
| | | | | | | | | | Kernel | < 0.01 | 143 | 26 Oct 2009 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 143 | 26 Oct 2009 | |
| T000920-09-REG 09-UK-060 UNITED KINGDOM (Europe North) | Field Corn (Ohio) | 1.18 Apr 2009 2 – 3 - | Foliar | 500.00 g AI/kg | 196 L/ha | 147.00 g a.s./ha<br><br>(-) | 19 Jun 2009 | BBCH 16 - 17 | Whole plant | < 0.01 | 20 | 09 Jul 2009 | Field<br><br>SP (max days): Mesotrione/Remaining Plant: 369 Mesotrione/Whole plant: 390 Mesotrione/Kernel: 312 Mesotrione/Cob + Kernel: 369 Mesotrione/Cob: 312 |
| | | | | | | | | | Whole plant | < 0.01 | 35 | 24 Jul 2009 | |
| | | | | | | | | | Cob + Kernel | < 0.01 | 41 | 30 Jul 2009 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 41 | 30 Jul 2009 | |
| | | | | | | | | | Cob + Kernel | < 0.01 | 53 | 11 Aug 2009 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 53 | 11 Aug 2009 | |
| | | | | | | | | | Whole plant | < 0.01 | 60 | 18 Aug 2009 | |
| | | | | | | | | | Whole plant | < 0.01 | 87 | 14 Sep 2009 | |
| | | | | | | | | | Cob | < 0.01 | 98 | 25 Sep 2009 | |
| | | | | | | | | | Kernel | < 0.01 | 98 | 25 Sep 2009 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| | | | | | | | | | Remaining Plant | < 0.01 | 98 | 25 Sep 2009 | |
| | | | | | | | | | Cob | < 0.01 | 112 | 09 Oct 2009 | |
| | | | | | | | | | Kernel | < 0.01 | 112 | 09 Oct 2009 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 112 | 09 Oct 2009 | |

(a) According to Codex (or other e.g. EU) classification

(b) Only if relevant

(c) Year must be indicated

(d) Minimum number of days after last application (Label pre-harvest interval, PHI, underline)

(e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included.

(*) Indicates sample taken prior to application

(#) Indicates corrected Residue values

(^) PHI calculated using cut date

(+) Indicates calculated Residue value

(DBA) Days Before Application

SP (max days): Maximum storage period

### A 2.2.3.1.5 Study T011368-06-REG– EU reviewed

| | |
|---|---|
| Comments of zRMS: | This study has been reviewed at the EU level. |

| | |
|---|---|
| Reference: | KCA2 6.3.1/05 |
| Report | Klimmek S., 2008. Mesotrione and Nicosulfuron - Residue study on maize in northern France in 2007<br>Syngenta Report No. T011368-06-REG<br>Syngenta File No. A14351BX_10205,<br>GLP, unpublished |
| Guideline(s): | Yes<br>FAO Guidelines on Producing Pesticide Residues Data from Supervised Trials (Rome, 1990).<br>Commission of the European Communities, General Recommendations for the Design, Preparation and Realization of Residue Trials; 7029NII95 (rev. 5, working document).<br>Guidelines and Criteria for the Preparation and Presentation of Complete Dossiers and of Summary Dossiers for the Inclusion of Active Substances in Annex I of Directive 91/414/EEC (Article 5.3 and 8.2), 1996. |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

## Table A 47: Summary of T011368-06-REG trials

| | | | |
|---|---|---|---|
| **Field Trials, Crop Residue (Summary) :** Mesotrione and Nicosulfuron - Residue study on maize in northern France in 2007 | | | |
| **Active Substance (common name):** | Mesotrione | **Commercial Product (name):** | $Commercial.Name$ |
| **Crop/Crop Group:** | Maize | **Producer of commercial product:** | Syngenta AG |
| **Responsible body for reporting (name, address):** | Syngenta AG, Basel, Switzerland | **Indoor/Glasshouse/Outdoor:** | Field |
| **Country:** | FRANCE | **Other active substance in the formulation (common name and content):** | Nicosulfuron (30 g a.s./L) |
| **Content of active substance (g/kg or g/L):** | 75 g a.s./L | **Residues calculated as:** | Mesotrione |
| **Formulation (e.g. WP):** | A14351BX OD | | |
| **Analytical Method:**<br>Mesotrione (Cob, Kernel, Remainder Of Plant, Whole plant) RAM 366/01; 0.01 mg/kg | | | |
| **Recovery data:**<br>Mesotrione Cob Mean = 88% RSD = 14% (n = 3 in 0.01 - 0.1 mg/kg spiking range)<br>Mesotrione Kernel Mean = 82% RSD = 19% (n = 3 in 0.01 - 0.1 mg/kg spiking range)<br>Mesotrione Whole plant Mean = 90% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range) | | | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| T011368-06-REG<br>FR-HR-07-0121<br>FRANCE (Europe North)<br>(-) | Maize (Anjou 248) | 1<br>2 –<br>3 - | Foliar | 75.00 g a.s./L | 250 L/ha | 149.06 g a.s./ha<br>(-) | 03 Jun 2007 | BBCH 18 | Whole plant | < 0.01 | 24 | 27 Jun 2007 | Field A14351BX<br>SP (max days): Mesotrione/Remainder Of Plant: 276 Mesotrione/Whole plant: 327 Mesotrione/Kernel: 276 |
| | | | | | | | | | Whole plant | < 0.01 | 38 | 11 Jul 2007 | |
| | | | | | | | | | Whole plant | < 0.01 | 44 | 17 Jul 2007 | |
| | | | | | | | | | Whole plant | < 0.01 | 60 | 02 Aug 2007 | |
| | | | | | | | | | Cob | < 0.01 | 75 | 17 Aug 2007 | |
| | | | | | | | | | Kernel | < 0.01 | 75 | 17 Aug 2007 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 75 | 17 Aug 2007 | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 75 | 17 Aug 2007 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| | | | | | | | | | Cob | < 0.01 | 94 | 05 Sep 2007 | Mesotrione/Cob: 276 |
| | | | | | | | | | Kernel | < 0.01 | 94 | 05 Sep 2007 | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 94 | 05 Sep 2007 | |
| | | | | | | | | | Whole plant | < 0.01 | 99 | 10 Sep 2007 | |
| | | | | | | | | | Cob | < 0.01 | 120 | 01 Oct 2007 | |
| | | | | | | | | | Kernel | < 0.01 | 120 | 01 Oct 2007 | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 120 | 01 Oct 2007 | |
| T011368-06-REG<br>FR-HR-07-0122<br>FRANCE (Europe North)<br>(-) | Maize (Marcello) | 1<br>2 –<br>3 - | Foliar | 75.00 g a.s./L | 300 L/ha | 148.00 g a.s./ha<br><br>(-) | 14 Jun 2007 | BBCH 18 | Whole plant | < 0.01 | 34 | 18 Jul 2007 | Field A14351BX<br><br>SP (max days): Mesotrione/Remainder Of Plant: 270 Mesotrione/Whole plant: 306 Mesotrione/Kernel: 270 Mesotrione/Cob: 270 |
| | | | | | | | | | Whole plant | < 0.01 | 40 | 24 Jul 2007 | |
| | | | | | | | | | Whole plant | < 0.01 | 43 | 27 Jul 2007 | |
| | | | | | | | | | Whole plant | < 0.01 | 60 | 13 Aug 2007 | |
| | | | | | | | | | Cob | < 0.01 | 70 | 23 Aug 2007 | |
| | | | | | | | | | Kernel | < 0.01 | 70 | 23 Aug 2007 | |
| | | | | | | | | | Cob+Kernel | < 0.01 + | 70 | 23 Aug 2007 | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 70 | 23 Aug 2007 | |
| | | | | | | | | | Cob | < 0.01 | 90 | 12 Sep 2007 | |
| | | | | | | | | | Kernel | < 0.01 | 90 | 12 Sep 2007 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione (mg/kg) | | | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 90 | 12 Sep 2007 | |
| | | | | | | | | | Whole plant | < 0.01 | 106 | 28 Sep 2007 | |
| | | | | | | | | | Cob | < 0.01 | 130 | 22 Oct 2007 | |
| | | | | | | | | | Kernel | < 0.01 | 130 | 22 Oct 2007 | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 130 | 22 Oct 2007 | |

(a) According to Codex (or other e.g. EU) classification

(b) Only if relevant

(c) Year must be indicated

(d) Minimum number of days after last application (Label pre-harvest interval, PHI, underline)

(e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included.

(*) Indicates sample taken prior to application

(#) Indicates corrected Residue values

(^) PHI calculated using cut date

(+) Indicates calculated Residue value

(DBA) Days Before Application

SP (max days): Maximum storage period

### A 2.2.3.1.6 Study T009530-07-REG– EU reviewed

| | |
|---|---|
| Comments of zRMS: | This study has been reviewed at the EU level. |

| | |
|---|---|
| Reference: | KCA2 6.3.1/06 |
| Report | Schulz H., 2010. Mesotrione and Nicosulfuron - Residue study on maize in France (north) in 2008<br>Syngenta Report No. T009530-07-REG<br>Syngenta File No. ZA1296_10049,<br>GLP, unpublished |
| Guideline(s): | FAO Guidelines on Producing Pesticide Residues Data from Supervised Trials (Rome, 1990).<br>Commission of the European Communities, General Recommendations for the Design, Preparation and Realization of Residue Trials; 7029/VI/95 (rev. 5, working document).<br>Guidelines and Criteria for the Preparation and Presentation of Complete Dossiers and of Summary Dossiers for the Inclusion of Active Substances in Annex I of Directive 91/414/EEC (Article 5.3 and 8.2), 1996.<br>The national requirements are based on the OECD Principles of Good Laboratory Practice, which are accepted by regulatory authorities throughout the European Community, the United States of America (FDA and EPA) and Japan (MHW, MAFF and METI) on the basis of intergovernmental agreements. |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

## Table A 48: Summary of T009530-07-REG trials

| | | | |
|---|---|---|---|
| **Field Trials, Crop Residue (Summary) :** Mesotrione and Nicosulfuron - Residue study on maize in France (north) in 2008 | | | |
| **Active Substance (common name):** | Mesotrione | **Commercial Product (name):** | $Commercial.Name$ |
| **Crop/Crop Group:** | Maize | **Producer of commercial product:** | Syngenta AG |
| **Responsible body for reporting (name, address):** | Syngenta AG, Basel, Switzerland | **Indoor/Glasshouse/Outdoor:** | Field |
| **Country:** | FRANCE | **Other active substance in the formulation (common name and content):** | Nicosulfuron (30 g a.s./L) |
| **Content of active substance (g/kg or g/L):** | 75 g a.s./L | **Residues calculated as:** | Mesotrione |
| **Formulation (e.g. WP):** | A14351BX OD | | |
| **Analytical Method:**<br>Mesotrione (Cob, Cob + Kernel, Kernel, Leaf Sheath, Remainder Of Plant, Whole plant) RAM 366/01, modified; 0.01 n/k | | | |
| **Recovery data:**<br>Mesotrione Cob Mean = 93% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Mesotrione Kernel Mean = 107% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Mesotrione Leaf Sheath Mean = 95% RSD = N/A (n = 2 in 0.01 - 0.1 mg/kg spiking range)<br>Mesotrione Remainder Of Plant Mean = 87% RSD = 9% (n = 4 in 0.01 - 0.1 mg/kg spiking range) | | | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione | | | |
| T009530-07-REG<br>PDA-08-4166-FR01<br>FRANCE (Europe North)<br>(-) | Maize (PR37 F73) | 1.06 May 2008<br>2 –<br>3 - | Foliar | 75.00 g a.s./L | 351 L/ha | 150.38 g a.s./ha<br>(-) | 18 Jun 2008 | BBCH 18 | Whole plant | < 0.01 | 30 | 18 Jul 2008 | Field A14351BX<br>SP (max days): Mesotrione/Cob + Kernel: 481 Mesotrione/Remainder Of Plant: 481 Mesotrione/Whole |
| | | | | | | | | | Whole plant | < 0.01 | 33 | 21 Jul 2008 | |
| | | | | | | | | | Cob + Kernel | < 0.01 | 42 | 30 Jul 2008 | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 42 | 30 Jul 2008 | |
| | | | | | | | | | Cob | < 0.01 | 63 | 20 Aug 2008 | |
| | | | | | | | | | Kernel | < 0.01 | 63 | 20 Aug 2008 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 63 | 20 Aug 2008 | |
| | | | | | | | | | Leaf Sheath | < 0.01 | 63 | 20 Aug 2008 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione | | | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 63 | 20 Aug 2008 | plant: 493 Mesotrione/Leaf Sheath: 460 Mesotrione/Kernel: 460 Mesotrione/Cob: 460 |
| | | | | | | | | | Whole plant | < 0.01 | 63 | 20 Aug 2008 | |
| | | | | | | | | | Whole plant | < 0.01 | 76 | 02 Sep 2008 | |
| | | | | | | | | | Cob | < 0.01 | 92 | 18 Sep 2008 | |
| | | | | | | | | | Kernel | < 0.01 | 92 | 18 Sep 2008 | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 92 | 18 Sep 2008 | |
| | | | | | | | | | Cob | < 0.01 | 124 | 20 Oct 2008 | |
| | | | | | | | | | Kernel | < 0.01 | 124 | 20 Oct 2008 | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 124 | 20 Oct 2008 | |
| T009530-07-REG PDA-08-4166-FR02 FRANCE (Europe North) (-) | Maize (Kornadi) | 1.23 Apr 2008 2 – 3 - | Foliar | 75.00 g a.s./L | 156 L/ha | 156.08 g a.s./ha<br><br>(-) | 16 Jun 2008 | BBCH 18 | Whole plant | < 0.01 | 25 | 11 Jul 2008 | Field A14351BX<br><br>SP (max days): Mesotrione/Cob + Kernel: 483 Mesotrione/Remainder Of Plant: 483 Mesotrione/Whole |
| | | | | | | | | | Whole plant | < 0.01 | 36 | 22 Jul 2008 | |
| | | | | | | | | | Cob + Kernel | < 0.01 | 42 | 28 Jul 2008 | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 42 | 28 Jul 2008 | |
| | | | | | | | | | Cob | < 0.01 | 58 | 13 Aug 2008 | |
| | | | | | | | | | Kernel | < 0.01 | 58 | 13 Aug 2008 | |
| | | | | | | | | | Cob + Kernel | < 0.01 + | 58 | 13 Aug 2008 | |
| | | | | | | | | | Leaf Sheath | < 0.01 | 58 | 13 Aug 2008 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Mesotrione | | | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 58 | 13 Aug 2008 | plant: 500 Mesotrione/Leaf Sheath: 467 Mesotrione/Kernel: 467 Mesotrione/Cob: 467 |
| | | | | | | | | | Whole plant | < 0.01 | 64 | 19 Aug 2008 | |
| | | | | | | | | | Whole plant | < 0.01 | 77 | 01 Sep 2008 | |
| | | | | | | | | | Cob | < 0.01 | 91 | 15 Sep 2008 | |
| | | | | | | | | | Kernel | < 0.01 | 91 | 15 Sep 2008 | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 91 | 15 Sep 2008 | |
| | | | | | | | | | Cob | < 0.01 | 130 | 24 Oct 2008 | |
| | | | | | | | | | Kernel | < 0.01 | 130 | 24 Oct 2008 | |
| | | | | | | | | | Remainder Of Plant | < 0.01 | 130 | 24 Oct 2008 | |

(a) According to Codex (or other e.g. EU) classification

(b) Only if relevant

(c) Year must be indicated

(d) Minimum number of days after last application (Label pre-harvest interval, PHI, underline)

(e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included.

(*) Indicates sample taken prior to application

(#) Indicates corrected Residue values

(^) PHI calculated using cut date

(+) Indicates calculated Residue value

(DBA) Days Before Application

## A 2.2.4 Magnitude of residues in livestock

No new data are submitted in the framework of this application.

## A 2.2.5 Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation)

No new data are submitted in the framework of this application.

## A 2.2.6 Magnitude of residues in representative succeeding crops

No new data are submitted in the framework of this application.

## A 2.2.7 Other/Special Studies

No new data are submitted in the framework of this application.

## A 2.3 Nicosulfuron

## A 2.3.1 Stability of residues

## A 2.3.1.1 Stability of residues during storage of samples

## A 2.3.1.1.1 Study 0676/032-D2149 (NIS-42)

| | |
|---|---|
| Comments of zRMS: | Stability of nicosulfuron residues in sweet corn, dried kernels, stover and forage has been demonstrated for storage intervals of up to twelve months under frozen (nominally -20°C) conditions.<br>The study is acceptable. |

| | |
|---|---|
| Reference: | KCA3 6.1 (report available from data owner) |
| Report | Peatman M.H., 2007. Nicosulfuron: Storage Stability of Residues in Corn Matrices Stored Deep Frozen<br>Report No. 0676/032-D2149 (NIS-42)<br>GLP, unpublished |
| Guideline(s): | Yes<br>OECD Guidelines for the Testing of Chemicals – Stability of Pesticide Residues in Stored Commodities, No. 506, OECD, 10/2007<br>EPA Residue Chemistry Test Guidelines OPPTS 860.1380. |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

**Executive Summary**

A storage stability test was performed on sweet corn, dried kernels, stover and forage when stored in a deep freezing compartment (at approximately -20°C) in the dark. For this purpose, control samples of sweet corn, dried kernels, stover and forage were fortified at 0.5 mg/kg with nicosulfuron. Treated samples were analysed in triplicate after different intervals of storage (0, 3, 6 and 12 months) using LC-MS/MS. At each storage intervals, one control and two procedural samples were also analysed to verify performance of the analytical procedure. The results of this stability test showed stability of the active ingredient up to 12 months in sweet corn, dried kernels, stover and forage.

**Materials and Methods**

**Materials:**

| | |
|---|---|
| Test Materials: | Nicosulfuron |
| Description: | Crystalline solid |
| Batch No.: | 40315 |
| Purity: | 98.0% |
| CAS No.: | 111991-09-4 |
| Manufacturer: | Dr. Ehrenstorfer |
| Spiking levels: | 0.5 mg/kg |
| Test commodity Crop: | Maize |
| Origin: | Field trials in Poland |
| Crop parts: | untreated sweet corn, dried kernels, stover and forage |
| Sample size: | 10g |

**Study Design:**

The study was conducted during the period December 2005 to December 2066 by Covance Laboratory Limited, United Kingdom.

**Test Procedure:**

Separate untreated control aliquots of sweet corn, stover, dried kernels and forage (10g) were fortified with nicosulfuron at concentrations of 0.5 mg/kg. All samples were stored in a deep freezing compartment (at approximately -20°C) in the dark.

Treated samples were analysed in triplicate after different intervals of storage (0, 3, 6 and 12 months) using LC-MS/MS. At each storage intervals, one control and two procedural recovery samples (at 0.5 mg/kg) were also analysed to verify performance of the analytical procedure. Control samples were maintained under the same freezer conditions as fortified samples.

**Description of analytical procedures:**

The samples were analysed, using an appropriate analytical method for each matrix.

Residues of nicosulfuron were extracted by macerating each sample with 20mM pH 6 phosphate buffer, followed by centrifuging and filtration. For forage and stover an aliquot was taken, filtered and diluted with methanol:5mM ammonium acetate (1:9, v:v) ready for determination. For sweet corn and dried kernels an aliquot was taken for clean-up with an Env+ SPE cartridge. The elution mixture was evaporated under nitrogen and reconstituted in methanol:5mM ammonium acetate (1:9, v:v) ready for determination of nicosulfuron by LC-MS/MS.

**Results and Discussion**

The average percent recovery for nicosulfuron after 12 months of storage in deep freezing condition, was 74% in sweet corn, 70% in dried kernels, 78% in stover and 81% in forage. No residues of nicosulfuron were found above the limits of quantification (0.04 mg/kg) in the control samples. All batch mean procedural recoveries for nicosulfuron in sweet corn, dried kernels, stover and forage were within the acceptable range of 70-120%.

A summary of the results for nicosulfuron is given in the table below.

**Table A 49: Mean storage stability of nicosulfuron in maize samples, values give recovery in % - uncorrected**

| Matrix | Time of storage in months | | | |
|---|---|---|---|---|
| | **0** | **3** | **6** | **12** |
| Sweet corn | 84.4 | 81.5 | 78.0 | 74.2 |
| Dried kernels | 81.5 | 80.8 | 82.1 | 70.0 |
| Stover | 80.0 | 89.9 | 95.7 | 78.0 |
| Forage | 92.3 | 93.0 | 91.7 | 81.2 |

**Conclusion**
The results of this stability test showed stability of the active ingredient up to 12 months in sweet corn, dried kernels, stover and forage.

## A 2.3.2 Nature of residues in plants, livestock and processed commodities

## A 2.3.2.1 Nature of residue in plants

## A 2.3.2.1.1 Nature of residue in primary crops

## A 2.3.2.1.1.1 Study AMR 861-87

| | |
|---|---|
| Comments of zRMS: | This study has previously been submitted as part of the AIR process for nicosulfuron.<br>Nicosulfuron was metabolised primarily by two pathways:<br>(1) hydrolysis of the sulfonylurea bridge resulting in formation ASDM and ADMP, which undergoes rapid metabolism as it was not a significant metabolite in any extract; and<br>(2) hydroxylation of the 5'-position of the pyrimidine ring of intact sulfonylurea followed by glucosidation.<br>The study is acceptable. |

| | |
|---|---|
| Reference: | KCA3 6.2.1 (report available from data owner) |
| Report | Scott M.T., 1989. Plant metabolism study of $^{14}$C-DPX-V9360 in corn<br>Report No. AMR 861-87<br>GLP, unpublished |
| Guideline(s): | Yes<br>U.S. EPA 171-4a (1982) |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

**Executive Summary**
The metabolism of nicosulfuron was investigated in corn (Cargill 937). The corn seeds were planted in pots (~37.5 cm in diameter) containing a silty loam soil from a typical corn-growing region in the United States (Rochelle, IL.). The pots were maintained in a greenhouse at DuPont's Experimental Station Site, (Wilmington, DE, USA). Thirteen days after the seeds were planted, when the corn was in the 4-5 leaf stage, six of the pots were treated with a simulated formulation solution of [pyridine-2-$^{14}$C] nicosulfuron and another six pots were treated with a simulated formulation solution of [pyrimidine-2-$^{14}$C] nicosulfuron. All pots were treated at an application rate of 70 g a.s./ha. Representative plant samples were taken immediately after the application solution had dried and at 7, 14, 30, 49 (silage) and 113 days (maturity). At maturity the samples were separated into the raw agricultural commodities (RACs) grain, cob, and stalk (stover) for analysis. At each sampling time, total radioactive residues and the distribution of residues were determined.
For corn treated with [pyridine-2-$^{14}$C] nicosulfuron, the concentration of parent in each RAC was less than 0.010 mg/kg. ASDM (referred to as pyridine sulphonamide in the report) was the major metabolite in the RACs comprising 0.021 mg/kg (metabolite equivalents). Individual metabolite concentrations in the 30-day and 49-day samples were each ≤0.005 mg/kg (nicosulfuron equivalents). Total radioactive residues in grain and cob were 0.002 and 0.004 mg/kg, nicosulfuron equivalents, respectively, and were therefore not analysed further.
For corn treated with [pyrimidine-2-$^{14}$C] nicosulfuron, the concentration of nicosulfuron was <0.01 mg/kg in all RACs except fodder (Day 113, mature sample) which contained 0.021 mg/kg. Total radioactive residues in grain were 0.003 mg/kg parent equivalents. Polar metabolites were the only

other radiochemical peak comprising 0.012 mg/kg (nicosulfuron equivalents).
For samples harvested before 30 days after application, the metabolites 5-GDUD and 5-HDUD (referred to as 5'-O-Glucoside and 5'-hydroxy DPX-V9360 in the report), were identified. However these metabolites were not present in any raw agricultural commodity.
Nicosulfuron was metabolised primarily by two pathways: (1) hydrolysis of the sulfonylurea bridge resulting in formation ASDM and ADMP, which undergoes rapid metabolism as it was not a significant metabolite in any extract; and (2) hydroxylation of the 5'-position of the pyrimidine ring of intact sulfonylurea followed by glucosidation.

**Materials and Methods**
**Materials:**

1. Test material: Nicosulfuron radiolabelled technical
   Lot/Batch #: See radiolabelled Lot/Batch #
   Purity: See radiolabelled purity
   Description: Tan solid
   CAS#: 111991-09-4

2 Radiochemical test material: $^{14}$C Nicosulfuron technical
a. Lot/Batch #: [Pyridine-2-$^{14}$C] V9360 HOTC 282
   Purity: 99.0%
   Specific activity: 62.9 μCi/mg (1.40 x $10^5$ dpm/μg)
   Stability of test compound: Shown to be stable under the conditions of the test

b. Lot/Batch #: [Pyrimidine-2-$^{14}$C] V9360 HOTC 281
   Purity: 98.8%
   Specific activity : 62.2 μCi/mg (1.38 x $10^5$ dpm/μg)
   Stability of test compound: Shown to be stable under the conditions of the test

3. Soil: Loam/Silt Loam

| Soil series | Soil type | pH | OM (%) | Sand (%) | Silt (%) | Clay (%) | CEC meg/100 g |
|---|---|---|---|---|---|---|---|
| Rochelle, IL, USA | Loam/Silt Loam | 6.9, 7.1 | 5.6, 6.1 | 27.2, 29.2 | 48.0, 52.0 | 18.8, 24.8 | 26.4, 26.7 |

**Study design**

The study was conducted from July 20, 1987 (application of treatment solution) to January 11, 1989 (final report signed), the end date of the experimental work is not given in the report. Work was conducted at DuPont's Experimental Station in Wilmington, Delaware, USA.

1. Experimental conditions
   The metabolism in corn was studied using pots (37.47 cm inner diameter) filled with a loam/silt loam soil obtained from a typical corn-growing region (Rochelle, Illinois, USA). These pots were maintained in a greenhouse. A single application of a simulated aqueous suspension was applied post emergence to corn at the 4-5 leaf stage using a Pre-Val® sprayer.
2. Sampling
   Corn samples were taken immediately after the application solution had dried (Day 0), at 7, 14, 30, 49 (silage stage), and 113 days (maturity, November 11, 1987) after application. Mature corn samples were separated into grain, cob and stalk. All samples were stored frozen (-20°C.).

**Results and Discussion**
**A. Total Radioactive Residues (TRRS)**
The total radioactive residues (TRRs) in corn after application of $^{14}$C-nicosulfuron at 70 g a.s./ha were determined by combustion of samples followed by liquid scintillation counting (LSC). The results are shown below.

**Table A 50: Total radioactive residues, TRR (mg/kg, nicosulfuron equivalents and % TRR), in corn following post-emergent application of $^{14}$C-nicosulfuron at 70 g a.s./ha**

| Days after application | Corn sample | [Pyridine-2-$^{14}$C]nicosulfuron | | [Pyrimidine-2-$^{14}$C]nicosulfuron | |
|---|---|---|---|---|---|
| | | mg/kg | % TRR | mg/kg | % TRR |
| 0 | Whole Plant | 4.33 | 100 | 3.29 | 100 |
| 7 | Whole Plant | 1.31 | 100 | 0.890 | 100 |
| 14 | Whole Plant | 0.212 | 100 | 0.257 | 100 |
| 30 | Whole Plant | 0.014 | 100 | 0.006 | 100 |
| 49 | Silage | 0.005 | 100 | 0.003 | 100 |
| 113 (Maturity) | Grain | 0.002 | 1.3 | 0.003 | 0.9 |
| 113 | Cobs | 0.004 | 0.4 | 0.004 | 0.3 |
| 113 | Fodder[a] | 0.065 | 100 | 0.058 | 100 |
| 113 | Stover[b] | 0.096 | ND[c] | 0.075 | ND |

a Combustion of grain, cob and stalk
b Combustion of stalk
c Not determined

## B. Extraction and Characterisation 0f Residues

Whole plant samples from Days 0, 7, 14 and 30 were initially rinsed with acetone:water (20:80) to remove surface radioactivity. The acetone was removed by rotary evaporation and the aqueous solution was stored frozen until analysis. Rinsed plant samples were allowed to dry and were stored frozen until further analysis. Silage and mature corn were too large and cumbersome to rinse effectively.

Mature maize was separated into grain, cobs and stalk (stover) prior to lyophilisation. TRR was determined by combustion/LSC. Those maize fractions having TRR of >0.01 mg/kg were extracted using acetone:0.1 M ammonium carbonate (9:1) with a 1:100 plant:solvent ratio. The plant-solvent mixture was placed in a sonic bath maintained at 40-50°C for 30 minutes. After filtration, the plant material was extracted twice more following the initial procedure. All three extracts were combined, analysed for TRR by LSC and the acetone was removed under reduced pressure. Aliquots of the remaining aqueous solution were counted for TRR by LSC. The aqueous solutions were stored frozen (-20°C) until HPLC analysis. The extracted solids were air-dried and stored frozen until combustion to determine un-extracted $^{14}$C-residues. Extraction results are shown in the table below.

**Table A 51: Extraction of $^{14}$C-nicosulfuron residues from maize**

| Days after application | maize sample | [Pyridine-2-$^{14}$C] nicosulfuron | | [Pyrimidine-2-$^{14}$C] nicosulfuron | |
|---|---|---|---|---|---|
| | | mg/kg | % TRR[b] | mg/kg | % TRR[b] |
| 0 | Whole plant[a] | 4.12 | 93.9 | 3.25 | 96.4 |
| 7 | Whole plant[a] | 1.26 | 96.3 | 0.925 | 95.2 |
| 14 | Whole plant[a] | 0.195 | 91.9 | 0.175 | 91.2 |
| 30 | Whole plant[a] | 0.012 | 92.7 | 0.004 | 73.3 |
| 49 | Silage | 0.005 | 109.6 | 0.002 | 51.7 |
| 113 | Stover | 0.114 | 118.2 | 0.062 | 83.4 |

a Sum of 14C in rinse plus 14C in extract.
b TRR was based on combustion results

Note: Mature grain and cob were not extracted due to low concentrations of TRR (<0.005 mg/kg, nicosulfuron equivalents).

1. Extraction and characterisation of residues in grain and cob

TRR in mature grain and cob were each <0.005 mg/kg, nicosulfuron equivalents, and therefore they were not extracted.

2. Extraction and characterisation of residues in whole plants

Extraction (and rinsing) of residues from whole plants resulted in >90% recovery of TRR except for the Day 30 sample from [pyrimidine-2-$^{14}$C] nicosulfuron-treated plants (73.3% TRR). [Extraction for the Day 49 silage sample from [pyrimidine-2-$^{14}$C] nicosulfuron-treated plants was 51.7%.] For all samples, 95% to 125% of the initial radioactivity, as determined by combustion, was recovered. Sample recoveries >100% indicated variation in sample homogeneity.

3. Storage stability of residues

Application solutions and extracts were analysed as a function of time. Analysis of the [pyridine-2-$^{14}$C] nicosulfuron treatment solution soon after application and again after 4 months of freezer storage showed no significant differences. The pyrimidine-labelled application solution was re-analysed after two months of freezer storage and no significant differences were found.

The extract from the Day 30 sample from the pyridine-labelled nicosulfuron treated corn was re-analysed after four months. The concentrations of residues were the same as in the original analysis within experimental error.

The results indicated that nicosulfuron was stable in the application and in extracts for at least 4 months.

4. Identity of residues

Residues were analysed for individual metabolites from rinse solutions and extracts by HPLC retention time matching and comparison of TLC $R_f$ values with known synthetic standards. The results for samples treated with [pyridine-2-$^{14}$C] nicosulfuron from corn samples from [pyrimidine-2-$^{14}$C] nicosulfuron application are shown in the tables below.

**Table A 52: Concentration of residues in corn samples after application of [pyridine-2-$^{14}$C]nicosulfuron at 70 g a.s./ha**

| | | Nicosulfuron | | ASDM | | 5-HDUD | | 5-GDUD[a] | | Compound C | | Compound A | | Polar | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| DAA[b] | Sample | mg/kg[c] | %[d] | mg/kg[e] | % | mg/kg | % | mg/kg | % | mg/kg | % | mg/kg | % | mg/kg | % |
| 0 | Whole Plant | 3.17 | 72.9 | 0.166 | 4.2 | 0.027 | 0.6 | 0.014 | 0.3 | 0.162 | 3.7 | 0.311 | 7.2 | ND[f] | - |
| 7 | Whole Plant | 0.667 | 51.0 | 0.307 | 23.9 | 0.011 | 0.8 | 0.035 | 2.6 | 0.027 | 2.1 | 0.047 | 3.6 | ND | |
| 14 | Whole Plant | 0.074 | 34.8 | 0.063 | 29.6 | 0.001 | 1.0 | 0.005 | 2.5 | 0.006 | 2.7 | 0.008 | 3.9 | ND | |
| 30 | Whole Plant | 0.003 | 20.0 | 0.004 | 32.8 | ND | | ND | | ND | | ND | | 0.002 | 18.5 |
| 49 | Whole Plant (Silage) | 0.0005 | 10.1 | 0.002 | 31.6 | ND | | ND | | ND | | ND | | ND | |
| 113 | Stalk (Stover) | 0.014 | 14.2 | 0.057 | 58.9 | ND | | ND | | ND | | ND | | ND | |
| 113 | Whole Plant (Fodder) | 0.008 | 9.6 | 0.021 | 39.9 | ND | | ND | | ND | | ND | | ND | |

a Glucoside of 5'-HDUD
b Days after application
c Total Radioactive Residue, TRR, from both rinse (whole plant from Days 0-30) and extract
d Percent of TRR, both rinse (whole plant from Days 0-30) and extract
e TRR in ASDM equivalents
f Not detected

**Table A 53: Concentration of residues in corn samples after application of [pyrimidine-2-$^{14}$C]nicosulfuron at 70 g a.s./ha**

| DAA [b] | Sample | Nicosulfuron | | 5-HDUD | | 5-GDUD[a] | | DMPU | | Compound C | | Compound B | | Compound A | | Polar residue A<br>Polar residue B | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | mg/kg[c] | %[d] | mg/kg | % | mg/kg | % | mg/kg | % | mg/kg | % | mg/kg | % | mg/kg | % | mg/kg | % |
| 0 | Whole plant | 2.361 | 71.5 | 0.279 | 9.1 | 0.015 | 0.4 | NDe | | 0.128 | 3.7 | ND | - | 0.183 | 5.3 | 0.024f | 0.7 |
| 7 | Whole plant | 0.559 | 65.2 | 0.008 | 0.8 | 0.037 | 3.9 | 0.020 | 2.1 | 0.019 | 1.9 | 0.009 | 1.9 | 0.034 | 3.5 | 0.044f | 4.5 |
| 14 | Whole plant | 0.093 | 48.6 | 0.001 | 0.6 | 0.010 | 5.2 | ND | | 0.007 | 3.5 | 0.004 | 1.9 | 0.011 | 5.6 | 0.019f | 9.9 |
| 30 | Whole plant | 0.001 | 32.9 | <0.001 | <0.1 | <0.001 | 1.0 | ND | | ND | | ND | | ND | | 0.001f<br><0.001g | 21.0<br>5.5 |
| 49 | Whole plant (Silage) | <0.001 | 5.0 | <0.001 | <0.1 | <0.001 | <0.1 | ND | | ND | | ND | | <0.001 | 1.9 | <0.001f | 27.1 |
| 113 | Stalk (Stover) | 0.028 | 37.4 | ND | | ND | | ND | | ND | | ND | | ND | | 0.016f | 21.1 |
| 113 | Whole plant (Fodder) | 0.022 | 18.4 | ND | | ND | | ND | | ND | | ND | | ND | | 0.012f | 13.2 |

a Glucoside of 5'-HDUD
b Days after application
c Total Radioactive Residue, TRR, from both rinse (whole plant from Days 0-30) and extract
d Percent of TRR, both rinse (whole plant from Days 0-30) and extract
e Not detected
f Polar Residue A
g Polar Residue B

5. Identity of residues

Concentrations of residues in mature grain were too low to allow identification of residues. Consequently, a metabolic pathway is proposed based on identification of metabolites found in immature samples. For corn treated with pyridine-labelled nicosulfuron, the major residues in immature foliage were nicosulfuron and pyridine sulphonamide. Though other residues were detected, they were not observed in samples taken after 14 days. The major residue found in immature foliage samples after application of pyrimidine-labelled nicosulfuron was nicosulfuron (parent). Again, though other residues were present in samples taken from 0 through 14 days, they were not observed in subsequent samples (<0.01 mg/kg nicosulfuron equivalents). The analytes of concern are proposed to be parent compound and pyridine sulphonamide.

6. Identification of metabolites

A number of metabolites were identified in immature foliage samples by comparison with synthetic standards using HPLC and TLC. Parent was the major residue found in foliage from corn treated with either radiolabelled form of nicosulfuron. ASDM, which is not herbicidally active, was the major metabolite observed in samples from pyridine-labelled-treated maize. The metabolite 5-HDUD and its glucoside (5-GDUD) were also identified in plants that were treated with either radiolabelled form of nicosulfuron. DPMU was observed only in Day 7 samples. These metabolites were not present in samples taken after 14 days.

7. Proposed degradation pathway

Concentrations of residues in mature grain were too low to allow identification of residues. Consequently, a metabolic pathway is proposed based on identification of metabolites found in immature samples. Metabolism proceeds primarily by hydrolysis of the sulphonyl urea bridge resulting in the formation of ASDM and ADMP; however, ADMP was not detected presumably because it was rapidly metabolised to more polar compounds. Another metabolic pathway was hydroxylation of nicosulfuron to form 5-HDUD which was then conjugated to form 5-GDUD.

**Conclusion**

Maize was treated at the 4-5 leaf stage with formulated [pyridine-2-$^{14}$C] nicosulfuron or [pyrimidine-2-$^{14}$C] nicosulfuron at 70 g a.s./ha. Mature raw agricultural commodities (RACs), grain, stover and fodder, each contained less than 0.010 mg/kg nicosulfuron.

For maize treated with pyridine-labelled nicosulfuron, ASDM was the major metabolite in RACs (0.038 mg/kg nicosulfuron equivalents). ASDM, a cleavage product formed from hydrolysis of the sulfonylurea bridge, was also a major metabolite found in the rat. In grain, the concentration of total radioactive residues (TRR) was 0.002 mg/kg (nicosulfuron equivalents). This low concentration precluded further analysis of grain residues.

For maize treated with pyrimidine-labelled nicosulfuron, the concentration of nicosulfuron in all RACs was less than 0.010 mg/kg parent equivalents except for fodder (0.028 mg/kg). Mature grain contained 0.003 mg/kg total radioactive residues and was not analysed further.

Metabolism of nicosulfuron in maize proceeded by cleavage of the sulfonylurea bridge leading to formation of ASDM and presumably ADMP. ADMP was not found in any sample and is assumed to undergo rapid subsequent metabolism. Hydroxylation of nicosulfuron in the 5′-position of the pyrimidine ring was another metabolic pathway which led to formation of 5-HDUD and its glucoside (5-GDUD). The 5′-hydroxy metabolite and its glucoside were only found in samples through Day 14 and were not found in any RACs.

## A 2.3.2.1.2 Nature of residue in rotational crops

### A 2.3.2.1.2.1 Study AMR 874-87

| | |
|---|---|
| Comments of zRMS: | The studies have been submitted as part of the AIR process for Nicosulfuron.<br>The studies have not been conducted in accordance with the GLP requirements.<br>Applicant explanation: *GLP was not applicable to this study at the time it was conducted; however the study was conducted in the spirit of compliance with GLP requirements*<br><br>The original study (DuPont Document No. AMR-874-87) described the uptake of radiolabeled DPX-V9360 and its soil degradates by soybeans, lettuce, wheat, and radishes grown in soil 120 days and 10 months after 70 g ai/ha treatment. In addition, uptake in soybeans grown in soil aged 30 days post-treatment was also examined.<br>Low concentrations of radiolabeled degradates detected in soybeans grown in soil previously treated with 70 g DPX-V9360/ha precluded their isolation and identification. Larger quantities of metabolites were generated by growing soybeans in soil treated with 2 kg $^{14}$C-Pyridine Sulfonamide/ha. Pyridine Sulfonamide (IN-V9367) is a major soil degradate of DPX-V9360 and was shown to be the precursor of radiolabeled residues in soybeans as described in the original report. The high treatment rate is possible because of the lack of phytotoxicity of Pyridine Sulfonamide.<br>The supplement (Scott M.T., 2004) describes the isolation and characterization of radiolabeled components designated Compounds A, B, and C from soybean seed in the original report.<br><br>Data indicated that nicosulfuron does not accumulate in rotational crops grown in soil treated with Nicosulfuron 75WG applied at a rate of 70 g a.s./ha. Nicosulfuron concentrations were very low and decreased with soil aging time.<br>ASDM, a major soil degradate of nicosulfuron, was found in most extracts of plants grown in soil treated with [pyridine-2-$^{14}$C] nicosulfuron. ASDM has no biological activity and EFSA have concluded the metabolite to be non-toxicologically relevant. It is not included in the crop residue definition for nicosulfuron.<br>Other compounds were identified at low concentrations in plant extracts.<br>The studies are acceptable. |

| | |
|---|---|
| Reference: | KCA3 6.6.1 (report available from data owner) |
| Report | Dietrich R.F., 1989. Confined accumulation study of $^{14}$C-DPX-V9360 on rotational crops.<br>Report No. AMR 874-87 |
| Guideline(s): | U.S. EPA 165-1 |
| Deviations: | No |
| GLP: | No, GLP was not applicable to this study at the time it was conducted; however the study was conducted in the spirit of compliance with GLP requirements. |
| Acceptability: | Yes |

| | |
|---|---|
| Reference: | KCA3 6.6.1 (report available from data owner) |
| Report | Scott M.T., 2004. Confined accumulation study of $^{14}$C-DPX-V9360 on rotational crops.<br>Report No. AMR 874-87, Supplement No. 1 |
| Guideline(s): | U.S. EPA 165-1 |
| Deviations: | No |
| GLP: | No, GLP was not applicable to this study at the time it was conducted; however the study was conducted in the spirit of compliance with GLP requirements. |
| Acceptability: | Yes |

**Executive Summary**

A confined rotational crop study was conducted to determine residues in crops grown in soil previously treated with nicosulfuron, either in the normal rotation or as a result of the failure of a maize crop following treatment.

[Pyrimidine-2-$^{14}$C]nicosulfuron and [pyridine-2-$^{14}$C]nicosulfuron were applied as a formulated solution to pots containing Sassafras loamy sand soil at a rate equivalent to 70 g a.s./ha, higher than the proposed maximum use rate in Europe. Soils were aged in the greenhouse for 30 days, 120 days or 10 months. After the 30-day soil aging period, soybeans were planted. After 120 days, soybeans, lettuce and wheat were planted and after 10 months, those three crops and radishes were planted. Crops were grown to maturity, harvested and analysed for total radioactive residues (TRRs). Individual crop fractions were extracted and analysed for $^{14}$C-nicosulfuron and its residues.

In general, crops grown in soil treated with pyridine-labelled nicosulfuron had higher concentrations of total radioactive residues than crops grown in soil where pyrimidine-labelled nicosulfuron was applied. TRRs in each crop decreased with increased soil aging periods.

The concentration of nicosulfuron was low, <0.002 mg/kg, in all crop fractions except for wheat from the 120-day soil aging period. Wheat straw/chaff contained 0.004–0.007 mg/kg nicosulfuron from that aging period and grain contained 0.005 mg/kg nicosulfuron. Nicosulfuron was not detected (<0.001 mg/kg) in these same wheat fractions after the 10-month soil aging period.

The major radiolabelled metabolite identified in crop fractions was ASDM (referred to as pyridine sulphonamide in the report). Its concentration was highest (0.080 mg/kg) in soybean forage from 30-day aged soil; its concentration in soybean forage decreased to 0.071 and 0.021 mg/kg from 120-day and 10-month aged soils, respectively. In soybean seed, ASDM concentrations decreased from 0.023 to 0.015 to 0.013 mg/kg from 30-day, 120-day and 10-month aged soil, respectively. In wheat straw, ASDM concentrations decreased from 0.056 mg/kg from 120-day aged soil to 0.038 mg/kg from 10-month aged soil. Mature lettuce and radish greens contained 0.019 and 0.010 mg/kg ASDM, respectively. All other crop fractions contained <0.008 mg/kg ASDM.

The next most abundant $^{14}$C-metabolite in crop fractions was MU-467 (referred to as N-desmethyl DPX-V9360 in the report). Soybean forage from 30-day aged soil contained 0.022–0.025 mg/kg MU-467. Concentrations decreased to 0.001 to 0.002 mg/kg MU-467 in soybean forage from the 10-month soil-aging period.

Three other chromatographically resolved residues were detected in soybean seed and have been shown to be the result of further metabolism of ASDM. The concentrations of these degradates (Compounds A, B and C) were reported as 0.017, 0.020 and 0.034 mg/kg (as ASDM equivalents) from 30-day aged soil and decreased to 0.006, 0.007 and 0.008 mg/kg in seed from the 10-month aged soil. These residues did not have the same HPLC retention time as any known reference standard. Additional work was undertaken to further characterise these unknown metabolites by treating soybean with formulated $^{14}$C-ASDM, followed by extraction and isolation of the metabolites. Additional HPLC analysis indicated that Compound A was a mixture of at least three radiolabelled metabolites each of which was <0.010 mg/kg ASDM equivalents. Compound B was identified as a serine conjugate of ASDM by mass spectrometry, proton NMR and synthesis of an authentic reference standard. Its concentration was recalculated (in the report addendum) as based on molecular weight of the conjugate and decreased from 0.030 mg/kg in soybeans from the 30-day soil-aging period to 0.01 mg/kg in the 10-month aging study. Compound C was identified as the N-malonyl conjugate of Compound B using mass spectrometry, proton NMR and synthesis of an authentic reference standard to confirm structure. Its concentration (based on molecular weight of the conjugate in the report addendum) decreased from 0.06 mg/kg in the 30-day aging study to <0.01 mg/kg for the 10-month study.

TRRs in soil from the 70 g a.s./ha applications were determined at treatment, planting and at harvest. Immediately after treatment, soil contained 0.031 and 0.035 mg/kg TRR from pyrimidine-labelled nicosulfuron and pyridine-labelled nicosulfuron, respectively. At 30 days after application, these concentrations decreased to 0.015 and 0.009 mg/kg, respectively. Very little change in TRR occurred throughout the remainder of the study.

The concentration of nicosulfuron in soils immediately after application was 0.029 and 0.033 mg/kg

from the pyrimidine-labelled nicosulfuron and pyridine-labelled nicosulfuron treated soils, respectively. These concentrations decreased to 0.010 and 0.005 mg/kg, respectively, 30 days after application. The major extractable soil radiolabelled degradate was ASDM. Its highest concentration was 0.004 mg/kg 227 days after application. Other unidentified extractable soil degradates, which did not co-chromatograph with any known reference standard, were each ≤0.003 mg/kg (parent equivalents) for soils treated with either radiolabelled form of nicosulfuron. Un-extractable/bound residues ranged from <0.0005 mg/kg immediately after treatment to a high of 0.007 mg/kg (parent equivalents) from pyridine-labelled nicosulfuron treated soil 168 days after application.
In summary, the accumulation potential of nicosulfuron and its metabolites/degradates was studied in rotational crops following application of [pyrimidine-2-$^{14}$C] nicosulfuron or [pyridine-2-$^{14}$C] nicosulfuron. Crops were planted after soil aging periods of 30-days, 120 days and 10-months. There was some uptake of residues by crops; however concentrations of these residues were low. The concentration of nicosulfuron was <0.010 mg/kg in all crop fractions from all three soil-aging periods. The major identified metabolite was ASDM, which is herbicidally inactive. Its concentration was <0.050 mg/kg in all crop fractions from all three aging periods except for soybean forage (0.080 and 0.071mg/kg, from the 30-day and 120-day aging periods, respectively) and in wheat straw from the 120-day soil-aging period (0.056 mg/kg). All other residues had concentrations of <0.050 mg/kg and were generally <0.030 mg/kg.

**Materials and Methods**

**A. Materials**

| | | |
|---|---|---|
| 1. | Test material: | Nicosulfuron technical and ASDM (referenced as IN-V9367 in the report) technical metabolite |
| | Lot/Batch #: | Not given |
| | Purity: | Not given |
| | Description: | Tan solid |
| | CAS#: | V9360: 111991-09-4, V9367: 112006-75-4 |
| 2 | Radiochemical test material: | $^{14}$C Nicosulfuron technical |
| a. | Lot/Batch #: | [Pyridine-2-$^{14}$C] V9360 HOTC 282 |
| | Purity: | 99% |
| | Specific activity: | 62.9 μCi/mg (1.40 x $10^5$ dpm/μg) |
| | Stability of test compound: | Shown to be stable under the conditions of the test |
| b. | Lot/Batch #: | [Pyrimidine-2-$^{14}$C] V9360 HOTC 281 Lot No.: 2385-050 |
| | Purity: | 99% |
| | Specific activity: | 62.9 μCi/mg (1.40 x $10^5$ dpm/μg) |
| | Stability of test compound: | Shown to be stable under the conditions of the test |
| | Radiochemical test material | $^{14}$C IN-V9367 |
| c. | Lot/Batch #: | [Pyridine-2-$^{14}$C]V9367 Lot No.: 2385-113 |
| | Purity: | 99% |
| | Specific activity: | 62.9 μCi/mg (1.38 x $10^5$ dpm/μg) ) assuming 100% conversion to ASDM (referred to as pyridine sulphonamide in the report) |
| 3. | Soil: | Sassafras loamy sand soil contained in pots each 37.47 cm |

| Soil series | Soil type | pH* | OM (%) | Sand (%) | Silt (%) | Clay (%) | CEC (meg/100 g) |
|---|---|---|---|---|---|---|---|
| Sassafras (Penns Grove, NJ, USA) | Loamy sand | 6.4 | 0.9 | 80.8 | 12.8 | 6.4 | 3.4 |

**B. Study design and methods**

The study was initiated in July, 1987. The date of the end of the experimental phase is not given in the report. The study was conducted by the DuPont Company, Wilmington, Delaware, USA.

**Original study**

Protocol Signed: 20 July 1987

Application Date: 10 August 1987
Report Date: 23 January 1989
The report supplement was issued in 2004 to provide more details of the additional work to generate and then characterise and identify three unknown soybean metabolites briefly mentioned in the original report. The experimental work described in the report supplement was run concurrently with the confined crop rotation study.

**Report supplement**
Start (Preparation of Application Solution): 22 March 1988
Soybean Sowing Date: 10 June 1988
Harvest Date: 29 August 1988
Experimental Completion Date: 18 January 1989
Report Supplement Date: 7 October 2004

1. Experimental conditions for the original study
Soybeans were chosen as the rotational crop for the 30-day aging as a replacement crop in case of failure of the primary crop-corn. Radishes were grown only in soils aged 10 months because of the relatively high sensitivity of root crops to nicosulfuron. Soybeans, radishes, wheat and lettuce were considered representative rotational crops of oil-producing, root, cereal and leafy crop groups for corn. Thirty-eight pots, each 37.47 cm in diameter, were filled with Sassafras loamy sand to a depth of about 30 cm. The pots were kept in a greenhouse located at DuPont's Experimental Station facility (Wilmington, DE, USA) whose temperature ranged from 15-40°C. The pots were watered regularly during the aging periods to keep the soil moist. Agrochemical treatments were made to soil/crops as needed and did not have any effect on this study.
Stock solutions of [pyrimidine-2-$^{14}$C]nicosulfuron or [pyridine-2-$^{14}$C]nicosulfuron (ca. 25 mg each) in 2.0 mL of 0.1 M ammonium carbonate were prepared. Ammonium carbonate was used to facilitate solubility and stability. The stock solutions were each diluted with water containing the inert ingredients used to make Nicosulfuron 75WG and a surfactant at a concentration to mimic actual field application. Fifty millilitres of either diluted stock solution were applied to bare soil in each pot on August 10, 1987. This volume delivered 0.77 mg per pots which corresponded to a rate of 70 g a.s./ha. Two additional pots, one for each radiolabelled form of nicosulfuron, were treated with solutions identical to those described but containing ten times the concentration of nicosulfuron. These pots were used to facilitate extraction and analyses of soil samples.
Crops were sown in soil treated at 70 g a.s./ha 30 days, 120 days and 10 months after application. Just prior to each planting, the soils were lightly cultivated. Any weeds were pulled and left on the soil in the pot from which they were pulled. Only soybeans were planted after the 30-day soil aging period. Soybeans were chosen to simulate what could be planted after failure of corn, the primary crop. Soybeans, wheat and lettuce were grown in soil aged 120 days while those three crops and radishes were grown in soil aged 10 months. Radishes were chosen because they are sensitive to nicosulfuron. These crops are representative of oil-producing, leafy, cereal and root crop groups.

2. Experimental conditions for work described in the supplement
For the additional work to characterise the three unknown soybean metabolites, [pyridine-2-$^{14}$C]-ASDM (referred to as pyridine sulphonamide in the report), was generated by hydrolysing 5 mL of a 1 mg/mL solution of [pyridine-2-$^{14}$C]nicosulfuron [synthesised by Perkin Elmer (formerly New England Nuclear) Lot No. 2385-113, 99.0% radiochemical purity, Specific Activity 62.9 μCi/mg, CAS 111991-09-4] in 0.05 M sodium acetate buffer, pH 5, in a bath maintained at 90°C for 2.5 hours. An aliquot of the solution was counted for total $^{14}$C by liquid scintillation counting, LSC. Another aliquot was analysed by HPLC to determine the percent of $^{14}$C-nicosulfuron converted to ASDM.
One millilitre of the hydrolysis solution containing $^{14}$C-ASDM (equivalent to 0.559 mg of $^{14}$C-ASDM assuming a 100% conversion of $^{14}$C-nicosulfuron) was added to a vessel containing 44.3 mg of non-radiolabelled ASDM, notebook reference E49646-33), 0.25 mL of X-77® surfactant (Ortho) and 100 mL of HPLC-grade water. This solution contained 0.44 mg/mL pyridine sulphonamide. Aliquots were removed for determining total radioactivity and radiochemical purity.

The application solution (50 mL) was pipetted evenly over the soil surface of each pot. The soil in each pot was tilled after the solution had dried. On June 10, 1988, soybean seeds were planted at a depth of 1.5 inches in each pot after the soil was tilled. After planting, about 2 litres of water was added to each pot.

3. Sampling

Original study

Soil was sampled to a depth of about 30 cm using a soil sampling tube of about 1.9 cm after the application soil had dried and at each planting and harvest. Samples were pooled, air-dried, weighed and homogenised.

Crops were thinned as needed to maintain healthy growth in each pot. These samples were labelled, weighed and frozen. These samples were analysed as additional samples if needed. At maturity, all of the remaining plants were harvested. The plants were immediately separated into RACs and weighed. The following table shows when each crop was harvested for each soil-aging period.

| **Soil aging period** | **Number of days after application when crop was harvested (days after planting)** | | | |
|---|---|---|---|---|
| | **Soybeans** | **Lettuce** | **Wheat** | **Radishes** |
| 30 days | 134 (104) | Not grown | Not grown | Not grown |
| 120 days | 227(107) | 168 (48) | 241 (120) | Not grown |
| 10 months (305 days) | 385 (80) | 357 (52) | 408 (103) | 347 (42) |

Wheat grain yields for 120 d plant back wheat were almost a factor of 10 lower than those achieved for the 10 month plant back period.

Supplemental work

Samples were harvested August 29, 1988 corresponding to 80 days after planting.

4. Sample storage for original and supplemental work

All soil and plant samples were stored frozen (-20°C.) until used.

5. Determination of total radioactive residues for original and supplemental work

Aliquots of air-dried, homogenised (Waring Blendor®) soil were combusted and the resultant solution was analysed by LSC to determine total radioactive residues (TRR). TRR values were calculated as mg/kg nicosulfuron equivalents on a dry-weight basis.

Each crop fraction was weighed, lyophilised, re-weighed and homogenised. Triplicate aliquots (0.1-0.3 g each) were combusted and analysed by LSC to determine TRR. Results were reported as mg/kg nicosulfuron equivalents.

6. Extraction of soil

Fifty grams of homogenised, air-dried soil from each high-dose study was weighed into a 250-mL plastic centrifuge bottle. The soil was extracted three times with 100-mL portions of acetone:0.1 M ammonium carbonate (9:1, v/v) by agitation in an ultrasonic bath maintained at 50°C for 15 minutes. After centrifugation, the supernatants were decanted. After the third extraction, the soil was extracted with 100 mL of 0.1 M ammonium carbonate for 1 hour at 50°C in the ultrasonic bath followed by washing with acetone. The extracts and washes were combined and TRR was determined by LSC. The extracts were concentrated in vacuo at *ca.* 40°C.

7. Extraction of plant samples

Five grams of lyophilised plant material were weighed into a 250-mL plastic centrifuge bottle, soaked with 100 mL of acetone: 0.1 M ammonium carbonate (1:1, v/v) and agitated in an ultrasonic bath maintained at 40°C for 15 minutes. After centrifugation, the supernatant was decanted and the plant tissue was extracted twice more. The supernatants were pooled after the third extraction. The pooled supernatant was concentrated by rotary evaporation at about 40°C. The concentrate was analysed for TRR by LSC.

Supplemental work

Extraction procedures were the same as those in the original report. Essentially, about 5 g of freeze-dried plant material were soaked with 100 mL of acetone: 0.1 M ammonium carbonate (1:1, v/v) and then agitated in an ultrasonic bath maintained at 40°C for 15 minutes. The mixture was then centrifuged and the supernatant was decanted. The plant tissue was then extracted twice more using the same procedure. The supernatants were combined and concentrated by rotary evaporation at about 40°C. The extract was analysed for total radioactivity by LSC. The pH of the concentrate was then adjusted to 4.0 and applied to a 20-g C-18 BondElut® (Varian, Walnut Creek, CA) which had been conditioned with acetonitrile (ACN) followed by HPLC-grade water. The $^{14}$C-residues were eluted with ACN:$H_20$ (5:95, v/v). The $^{14}$C-residues were then isolated by HPLC.

8. Determination of unextracted $^{14}$C-residues in soil and plant tissue

Extracted soil and plant tissue were air-dried and aliquots were combusted to determine unextracted radioactive residues by LSC. TRRs for plant tissues were reported on a fresh-weigh basis in mg/kg nicosulfuron equivalents and TRR values for soil were reported on a dry-weigh basis.

**Results and Discussion**

**A. Total radioactive residues and composition of residues in soil**

Total radioactive residues in soil and the composition of radiolabelled residues in soils are reported below (see). Nicosulfuron residues decreased from 0.029 and 0.033 mg/kg for the pyrimidine-treated and pyridine-treated soils immediately after application to 0.010 and 0.015 mg/kg, respectively, after 30 days of aging in the greenhouse.

The major extractable soil degradate was ASDM. Its maximum soil concentration reached 0.004 mg/kg, 227 days after application. Other unidentified soil degradates were present at concentrations of ≤0.003 mg/kg nicosulfuron equivalents for either radiolabelled form of nicosulfuron. Unextractable or bound residues had concentrations ranging from <0.0005 mg/kg parent equivalents immediately after application to 0.007 mg/kg in soil treated with pyrimidine-labelled nicosulfuron 168 days after application. While ASDM was detected in soil, ADMP (referred to as pyrimidine amine in the report), the other product of hydrolysis of the sulphonylurea bridge, was not detected. This may have been due to its or its degradates tight binding to the soil even though extraction included extraction with 0.1M ammonium carbonate for 1 hour at 50°C. in an ultrasonic bath.

**B. Total radioactive residues and composition of residues in crop samples**

1. Soybeans

Soybean samples were taken to represent hay/forage use as flowering to early pod formation as well as mature seeds. TRR in soybean forage/hay samples decreased from 0.221 mg/kg to 0.191mg/kg to 0.075 mg/kg from pyridine-labelled treated soil aged 30 days, 120 days and 10-months, respectively. Concentrations of TRR in seed were similar: 0.226, 0.157 and 0.069 mg/kg for the same aging periods. For soybeans grown in soil treated with pyrimidine-labelled nicosulfuron, forage/hay concentrations were much lower (0.060, 0.056 and 0.014 mg/kg, respectively) as well as for seed samples (0.043, 0.033 and 0.012 mg/kg, respectively) for the same samples.

The major $^{14}$C-residue found in soybean forage/hay was ASDM. Its concentration decreased from 0.080 mg/kg to 0.071 mg/kg to 0.021 mg/kg in plants grown after 30-day, 120-day and 10-month soil aging period, respectively. ASDM was also the major residue found in seed but at lower concentrations: 0.023, 0.015 and 0.013 mg/kg in plants grown after 30-day, 120-day and 10-month soil aging period, respectively.

In soybeans grown in soil treated with [pyrimidine-2-$^{14}$C]nicosulfuron, the only residue greater than 0.01 mg/kg was MU-467. Its concentration declined from 0.022 mg/kg to 0.019 mg/kg to 0.001 mg/kg in forage hay samples from the 30-day, 120-day and 10-month soil aging periods, respectively. Concentrations of MU-467 from soybeans grown in pyridine-labelled treated soil were 0.001-0.002 mg/kg.

In soybeans treated with [pyridine-2-$^{14}$C]-ASDM in the supplemental work, radiolabelled constituents designated as Compound A in the original report were shown to be a mixture of at least three radiolabelled components when subjected to further chromatographic analyses.

Fractions corresponding to compound A were collected and combined from chromatographic analyses of the extract of mature soybeans from the 30-day soil aging study with [pyridine-2-$^{14}$C]nicosulfuron. This sample had been shown to contain the highest levels of Compound A in the original study.
When the combined fractions were reanalysed under different conditions, Compound A was resolved into three radiolabelled components. The three radiolabelled components individually accounted for approximately 21%, 24%, and 55% of the original 0.017 ppm Compound A, or 0.004, 0.004, and 0.009 ppm, expressed as ASDM equivalents, based on the origin of these components from ASDM as reported originally. Total levels of Compound A from the 120-day soil aging study and 10-month aging study were 0.015 ppm and 0.006 ppm, respectively, and would be expected to be reduced similarly into multiple components.
Compound B was isolated from plant material from the $^{14}$C-ASDM soybean uptake study (supplemental work) described in the original report. The metabolite was identified as an amino acid conjugate of ASDM, where the beta carbon atom of alanine is attached to the sulphonamide nitrogen. Compound B was likely to have formed from conjugation of ASDM with the hydroxyl group of serine followed by loss of water. The metabolite was characterised by proton nuclear magnetic resonance spectroscopy (NMR), mass spectrometry, and the structure confirmed by comparison with an authentic synthetic reference standard.
Similarly, Compound C was isolated from samples generated in the $^{14}$C-ASDM uptake study. Compound C was identified as an N-malonyl conjugate of Compound B, with an amide linkage to the alpha amino group. As with Compound B, Compound C was characterised by NMR, mass spectrometry, and the structure confirmed by comparison with an authentic reference standard.
Compounds B and C isolated from the $^{14}$C-ASDM soybean uptake study were shown to be chromatographically identical to Compounds B and C in the actual $^{14}$C-nicosulfuron confined rotational crop samples. Concentrations of Compound B dropped from 0.03 ppm in soybean seed from plants grown in soil aged 30 days after treatment to 0.01 ppm in the 10-month soil aging study. Compound C decreased from 0.06 ppm to 0.01 ppm in the same samples. These values (based on the molecular weight of the conjugates) are slightly different than those in the original report which were expressed as ASDM equivalents based on their origin from that compound.

2. Lettuce

TRR concentrations in lettuce for samples grown in soil treated with pyridine-labelled nicosulfuron decreased from 0.05 mg/kg for the 120-day soil aging period to 0.011 mg/kg for the 10-month soil aging period. For the pyrimidine-labelled study, TRR values were lower ranging from 0.015 mg/kg for the 120-day aging period to 0.003 mg/kg for the 10-month aging period. The only significant (>0.01 mg/kg) residue was ASDM. Its concentration was 0.019 mg/kg for the 120-day aging period and 0.004 mg/kg for the 10-month soil aging period.

3. Wheat

TRR values in wheat were also higher when grown in pyridine-labelled treated soil than in pyrimidine-labelled treated soil. TRR values declined in samples from the 10-month soil aging period compared to the 120-day soil aging period. No individual residue was >0.005 mg/kg in grain for either aging period or either radiolabel. The major residue found in forage/hay samples was ASDM; its concentration was 0.008 mg/kg in the 10-month soil aging period.
TRR values were highest in mature straw/chaff due to drying down as the sample reaches maturity. ASDM was the major residue found in plants grown in [pyridine-2-$^{14}$C]nicosulfuron treated soil. Its concentration was 0.056 mg/kg for the 120-day soil aging period and 0.038 mg/kg for the 10-month soil aging period. Although concentrations of other unidentified residues were ≤0.057 and ≤0.049 mg/kg for the 120-day and 10-month aging periods, respectively, their concentrations were most likely to be much less than those values because these residues were probably lower molecular weight degradates/metabolites. In straw from soil treated with pyrimidine-labelled nicosulfuron, unidentified polar degradates decreased from 0.032 mg/kg for the 120-day aging period to 0.007 mg/kg for the 10-month aging period.

4. Radishes

TRR values for radishes only exceeded 0.01 mg/kg for plants grown in soil treated with pyridine-labelled nicosulfuron (radish greens, 0.027 mg/kg). ASDM was present in this sample at 0.010 mg/kg. For radish roots grown in the same soil, TRR was 0.006 mg/kg; for radishes grown in soil

treated with pyrimidine-labelled nicosulfuron, TRR was 0.004 and 0.003 for greens and roots, respectively.

**Table A 54: Total radioactive residues (TRR) in soil**

| Event | Days after treatment | TRR in mg/kg (nicosulfuron equivalents) | |
|---|---|---|---|
| | | [Pyrimidine-2-$^{14}$C] nicosulfuron treated soil | [Pyridine-2-$^{14}$C] nicosulfuron treated soil |
| Soil treatment | 0 | 0.031 | 0.035 |
| Planting (30-Day aging) | 30 | 0.015 | 0.009 |
| Planting (120-Day aging) | 120 | 0.008 | 0.013 |
| Soybean harvest (30-Day aging) | 134 | 0.010 | 0.014 |
| Lettuce harvest (30-Day aging) | 168 | 0.014 | 0.021 |
| Soybean harvest (30-Day aging) | 227 | 0.015 | 0.020 |
| Wheat harvest (30-Day aging) | 241 | 0.011 | 0.014 |
| Planting (10-Month aging) | 305 | 0.010 | 0.016 |
| Radish harvest (10-Month aging) | 347 | 0.012 | 0.015 |
| Lettuce harvest (10-Month aging) | 357 | 0.009 | 0.021 |
| Soybean harvest (10-Month aging) | 385 | 0.009 | 0.013 |
| Wheat harvest (10-Month Aging) | 408 | 0.011 | 0.017 |

**Table A 55: Composition of radiolabelled residues in [pyrimidine-2-$^{14}$C]nicosulfuron treated soil**

| Event | Days after treatment | TRR (mg/kg) | Nicosulfuron (mg/kg) | Bound (mg/kg)[a] | Others[a] |
|---|---|---|---|---|---|
| Soil treatment | 0 | 0.031 | 0.029 | <0.005 | <0.005 |
| Planting (30-Day Aging) | 30 | 0.015 | 0.010 | 0.001 | <0.001 |
| Planting (120-Day Aging) | 120 | 0.008 | 0.001 | 0.003 | <0.001 |
| Soybean harvest (30-Day aging) | 134 | 0.010 | 0.004 | 0.003 | <0.001 |
| Lettuce harvest (30-Day aging) | 168 | 0.014 | 0.002 | 0.006 | <0.002 |
| Soybean harvest (30-Day aging) | 227 | 0.015 | 0.003 | 0.006 | <0.003 |
| Wheat harvest (30-Day aging) | 241 | 0.011 | 0.003 | 0.003 | <0.002 |
| Planting (10-Month aging) | 305 | 0.010 | 0.001 | 0.005 | <0.001 |
| Radish harvest (10-Month aging) | 347 | 0.012 | 0.001 | 0.005 | <0.002 |
| Lettuce harvest (10-Month aging) | 357 | 0.009 | 0.001 | 0.004 | <0.001 |
| Soybean harvest (10-Month aging) | 385 | 0.009 | 0.001 | 0.003 | <0.002 |
| Wheat harvest (10-Month aging) | 408 | 0.011 | 0.001 | 0.004 | <0.002 |

a Concentrations expressed as mg/kg nicosulfuron equivalents

**Table A 56: Composition of radiolabelled residues in [pyridine-2-$^{14}$C]nicosulfuron treated soil**

| Event | DAT | TRR (mg/kg) [a] | Nicosulfuron (mg/kg) | ASDM (mg/kg) [b] | Bound (mg/kg) [a] | Others (mg/kg) [a] |
|---|---|---|---|---|---|---|
| Soil treatment | 0 | 0.035 | 0.033 | 0.001 | <0.0005 | <0.0005 |
| Planting (30-day aging) | 30 | 0.009 | 0.005 | 0.001 | 0.001 | <0.001 |
| Planting (120-day aging) | 120 | 0.013 | 0.002 | 0.001 | 0.005 | <0.002 |
| Soybean harvest (30-day aging) | 134 | 0.014 | 0.003 | 0.002 | 0.003 | <0.002 |
| Lettuce harvest (30-day aging) | 168 | 0.021 | 0.004 | 0.002 | 0.007 | <0.003 |
| Soybean harvest (30-day aging) | 227 | 0.020 | 0.003 | 0.004 | 0.004 | <0.002 |
| Wheat harvest (30-day aging) | 241 | 0.014 | 0.001 | 0.004 | 0.002 | <0.002 |
| Planting (10-month aging) | 305 | 0.016 | 0.001 | 0.004 | 0.003 | <0.002 |
| Radish harvest (10-month aging) | 347 | 0.015 | 0.001 | 0.003 | 0.004 | <0.002 |
| Lettuce harvest (10-month aging) | 357 | 0.021 | 0.001 | 0.004 | 0.006 | <0.002 |
| Soybean harvest (10-month aging) | 385 | 0.013 | <0.0005 | 0.003 | 0.003 | <0.002 |
| Wheat harvest (10-month aging) | 408 | 0.017 | 0.001 | 0.004 | 0.004 | <0.002 |

a Concentrations expressed as mg/kg nicosulfuron equivalents
b Concentrations expressed as mg/kg ASDM equivalents
= mg/kg nicosulfuron equivalents x (molecular weight of ASDM, 224)
(molecular weight of nicosulfuron, 410)

**Table A 57: Total radioactive residues in crops grown in soil treated with [pyridine-2-$^{14}$C]nicosulfuron**

| | **Days after application** | | | **Days after planting** | | | **Total radioactive residues, mg/kg[a]** | | |
|---|---|---|---|---|---|---|---|---|---|
| **Crop or crop fraction** | **30-day aging** | **120-day aging** | **10-month aging** | **30-day aging** | **120-day aging** | **10-month aging** | **30-day aging** | **120-day aging** | **10-month aging** |
| Soybean forage/hay | 63 | 149 | 330 | 33 | 29 | 25 | 0.221 | 0.191 | 0.075 |
| Soybean seed | 134 | 227 | 385 | 104 | 107 | 80 | 0.226 | 0.157 | 0.069 |
| Lettuce | [b] | 168 | 357 | [b] | 48 | 52 | --- | 0.050 | 0.011 |
| Wheat forage/hay | [b] | 158 | 339 | [b] | 38 | 34 | --- | 0.112 | 0.043 |
| Wheat straw/chaff | | 240 | 408 | | 120 | 103 | --- | 0.246 | 0.185 |
| Wheat grain | | 240 | 408 | | 120 | 103 | --- | 0.025 | 0.014 |
| Radish greens | [b] | [b] | 347 | [b] | | 42 | --- | --- | 0.027 |
| Radish roots | | | 347 | | | 42 | --- | --- | 0.006 |

a Concentrations expressed as mg/kg nicosulfuron equivalents on a fresh weight basis
b Crop not grown for that soil aging period

**Table A 58: Total radioactive residues in crops grown in soil treated with [pyrimidine-2-$^{14}$C]nicosulfuron**

| | **Days after treatment** | | | **Days after seeding** | | | **Total radioactive residues, mg/kg[a]** | | |
|---|---|---|---|---|---|---|---|---|---|
| **Crop or crop fraction** | **30-day aging** | **120-day aging** | **10-month aging** | **30-day aging** | **120-day aging** | **10-month aging** | **30-day aging** | **120-day aging** | **10-month aging** |
| Soybean forage/hay | 63 | 149 | 330 | 33 | 29 | 25 | 0.060 | 0.056 | 0.014 |
| Soybean seed | 134 | 227 | 385 | 104 | 107 | 80 | 0.043 | 0.033 | 0.012 |
| Lettuce | [b] | 168 | 357 | [b] | 48 | 52 | --- | 0.015 | 0.003 |
| Wheat forage/hay | [b] | 158 | 339 | [b] | 38 | 34 | --- | 0.033 | 0.009 |
| Wheat straw/chaff | | 240 | 408 | | 120 | 103 | --- | 0.099 | 0.056 |
| Wheat grain | | 240 | 408 | | 120 | 103 | --- | 0.020 | 0.006 |
| Radish greens | [b] | [b] | 347 | [b] | | 42 | --- | --- | 0.004 |
| Radish roots | | | 347 | | | 42 | --- | --- | 0.003 |

| Crop or crop fraction | Days after treatment | | | Days after seeding | | | Total radioactive residues, mg/kg[a] | | |
|---|---|---|---|---|---|---|---|---|---|
| | 30-day aging | 120-day aging | 10-month aging | 30-day aging | 120-day aging | 10-month aging | 30-day aging | 120-day aging | 10-month aging |

a Concentrations expressed as mg/kg nicosulfuron equivalents on a fresh weight basis.
b Crop not grown for that soil aging period

**Table A 59: Concentration of radiolabelled residues in soybeans expressed in mg/kg as nicosulfuron equivalents (unless otherwise noted)**

| **[Pyridine-2-$^{14}$C]nicosulfuron treated soil** | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| **Crop fraction** | **Soil aging period (days)** | **Nicosulfuron** | **ASDM [a]** | **MU-467 [b]** | **Cmpd. A [c]** | **Alanine conjugate [d, e]** | **Malonyl conjugate [f, g]** | **Polars [h]** | **Others [i]** | **Bound [j]** |
| Forage-Hay | 30 | 0.002 | 0.080 | 0.025 | ND | ND | ND | 0.013 | <0.007 | 0.007 |
| | 120 | 0.001 | 0.071 | 0.022 | ND | ND | ND | 0.019 | <0.006 | 0.007 |
| | 10-month | ND | 0.021 | 0.002 | ND | ND | ND | 0.002 | <0.012 | 0.003 |
| Seed | 30 | ND | 0.023 | ND | 0.017 | 0.03 | 0.06 | 0.006 | 0.006 | 0.015 |
| | 120 | ND | 0.015 | ND | 0.015 | 0.02 | 0.04 | 0.009 | 0.009 | 0.010 |
| | 10-month | ND | 0.013 | ND | 0.006 | 0.01 | 0.01 | 0.003 | 0.003 | 0.003 |
| [Pyrimidine-2-$^{14}$C]nicosulfuron treated soil | | | | | | | | | | |
| Forage-Hay | 30 | 0.002 | ND | 0.022 | ND | ND | ND | 0.005 | <0.007 | 0.005 |
| | 120 | 0.002 | ND | 0.019 | ND | ND | ND | 0.002 | <0.007 | 0.004 |
| | 10-month | ND | ND | 0.001 | ND | ND | ND | 0.001 | <0.003 | 0.002 |
| Seed | 30 | ND | ND | 0.003 | ND | ND | ND | 0.009 | <0.006 | 0.012 |
| | 120 | ND | ND | 0.002 | ND | ND | ND | 0.009 | <0.003 | 0.011 |
| | 10-month | ND | ND | ND | ND | ND | ND | 0.003 | <0.001 | 0.005 |

a Expressed as mg/kg of ASDM
b Expressed as mg/kg of MU-467
c Compound A in original study. A mixture of 3 components, expressed as mg/kg in ASDM equivalents based on origin from that compound, shown in pyridine uptake study. Each component was <0.01 mg/kg ASDM equivalents.
d Compound B in original work. Identified as an alanine conjugate (or serine conjugate with loss of water) of ASDM in supplemental work.
e Concentrations are based on its molecular weight and not ASDM equivalents as in original report.
f Compound C in original work. Identified as a malonyl alanine conjugate of ASDM in supplemental work.
g Concentrations are based on its molecular weight and not ASDM equivalents as in original report.
h Radiolabelled degradates with HPLC retention times <6 minutes, expressed as nicosulfuron equivalents
i Mixture components, each of which is present at concentrations lower than the stated value, expressed as nicosulfuron equivalents
j Unextracted radiolabelled residue determined from combustion analysis, expressed as nicosulfuron equivalents

**Table A 60: Concentration of radiolabelled residues in lettuce, expressed in mg/kg as nicosulfuron equivalents (unless otherwise noted)**

| **[Pyridine-2-$^{14}$C]nicosulfuron treated soil** | | | | | |
|---|---|---|---|---|---|
| **Crop Fraction** | **Soil Aging Period (Days)** | **Nicosulfuron** | **ASDM [a]** | **Others[b]** | **Bound[c]** |
| Mature Lettuce Leaves | 120 | 0.002 | 0.019 | <0.005 | 0.001 |
| | 10-month | ND[d] | 0.004 | <0.001 | <0.0005 |
| **[Pyrimidine-2-$^{14}$C]nicosulfuron treated soil** | | | | | |
| Mature Lettuce Leaves | 120 | 0.002 | ND | <0.004 | 0.001 |
| | 10-month | --- [e] | --- | --- | --- |

a Expressed as mg/kg of ASDM
b Mixture components, each of which is present at concentrations lower than the stated value, expressed as nicosulfuron equivalents
c Unextracted radiolabelled residue determined from combustion analysis, expressed as nicosulfuron equivalents
d Not detected
e Not analysed due to low concentration (<0.01 mg/kg nicosulfuron equivalents) based on combustion analysis

**Table A 61: Concentration of radiolabelled residues in wheat, expressed in mg/kg as nicosulfuron equivalents (unless otherwise noted)**

| **[Pyridine-2-$^{14}$C]nicosulfuron treated soil** | | | | | | |
|---|---|---|---|---|---|---|
| **Crop fraction** | **Soil aging period (Days)** | **Nicosulfuron** | **ASDM [a]** | **Polars[b]** | **Others[c]** | **Bound[d]** |
| Forage/Hay | 120 | NE[e] | NE | NE | NE | NE |
| | 10-month | 0.001 | 0.008 | 0.001 | <0.010 | 0.002 |
| Straw/Chaff | 120 | 0.004 | 0.056 | 0.005 | <0.057 | 0.021 |
| | 10-month | ND[f] | 0.038 | 0.003 | <0.049 | <0.0005 |
| Grain | 120 | 0.005 | 0.003 | 0.004 | <0.001 | 0.003 |
| | 10-month | 0.002 | 0.003 | 0.001 | <0.005 | <0.0005 |
| **[Pyrimidine-2-$^{14}$C]nicosulfuron treated soil** | | | | | | |
| Forage/Hay | 120 | NE | NE | NE | NE | NE |
| | 10-month | NA[g] | NA | NA | NA | NA |
| Straw/Chaff | 120 | 0.007 | ND | 0.032 | <0.016 | 0.020 |
| | 10-month | ND | ND | 0.007 | <0.009 | 0.003 |
| Grain | 120 | 0.005 | ND | 0.004 | <0.001 | 0.004 |
| | 10-month | NA | NA | NA | NA | NA |

a Expressed as mg/kg of ASDM
b Radiolabelled degradates with HPLC retention times <6 minutes, expressed as nicosulfuron equivalents
c Mixture components, each of which is present at concentrations lower than the stated value, expressed as nicosulfuron equivalents
d Unextracted radiolabelled residue determined from combustion analysis, expressed as nicosulfuron equivalents
e Not extracted due to insufficient amount of sample
f Not detected
g Not analysed due to low concentration (<0.01 mg/kg nicosulfuron equivalents) based on combustion analysis

**Table A 62: Concentration of radiolabelled residues in lettuce, expressed in mg/kg as nicosulfuron equivalents (unless otherwise noted)**

| **[Pyridine-2-$^{14}$C] nicosulfuron treated soil** | | | | | |
|---|---|---|---|---|---|
| **Crop fraction** | **Soil aging period (months)** | **Nicosulfuron** | **ASDM [a]** | **Polars[b]** | **Bound[c]** |
| Greens | 10 | 0.001 | 0.010 | <0.0005 | 0.001 |
| Roots | 10 | NA[d] | NA | NA | NA |
| **[Pyrimidine-2-$^{14}$C] nicosulfuron treated soil** | | | | | |
| Greens | 10 | NA | NA | NA | NA |
| Roots | 10 | NA | NA | NA | NA |

a Expressed as mg/kg of ASDM
b Radiolabelled degradates with HPLC retention times <6 minutes, expressed as nicosulfuron equivalents
c Unextracted radiolabelled residue determined from combustion analysis, expressed as nicosulfuron equivalents
d Not analysed due to low concentration (<0.01 mg/kg nicosulfuron equivalents) based on combustion analysis

## Conclusions

Data from the confined greenhouse study indicated that nicosulfuron does not accumulate in rotational

crops grown in soil treated with Nicosulfuron 75WG applied at a rate of 70 g a.s./ha. Nicosulfuron concentrations were very low and decreased with soil aging time. Concentrations in wheat grain and straw/chaff from a 120-day soil aging period were 0.002-0.007 mg/kg and were ≤0.002 mg/kg in all other crop fractions.
ASDM, a major soil degradate of nicosulfuron, was found in most extracts of plants grown in soil treated with [pyridine-2-$^{14}$C] nicosulfuron. Soybean forage/hay from the 30-day and 120-day soil aging periods contained 0.080 and 0.071 mg/kg. Mature wheat straw contained 0.056 and 0.038 mg/kg from plants grown in soil aged 120-days and 10-months, respectively. In all other crops, the concentration of ASDM was ≤0.023 mg/kg. ASDM has no biological activity and EFSA have concluded the metabolite to be non-toxicologically relevant. It is not included in the crop residue definition for nicosulfuron.
Other compounds were identified at low concentrations in plant extracts. MU-467 was detected in soybean forage/hay from 30-day and 120-day aging periods at a concentration of 0.024 and 0.020 mg/kg, respectively (mean values) but declined to 0.001-0.002 mg/kg from the 10-month aging period. An alanine conjugate of ASDM and a malonyl-alanine conjugate of ASDM were also identified in soybeans. Only the malonyl-alanine conjugate's concentration from the 30-day aged soil was >0.05 mg/kg (0.06 mg/kg). The concentrations of both conjugates from the 120-day soil aging periods were <0.04 mg/kg and reached 0.01 mg/kg in soybeans grown in soil aged 10 months.

## A 2.3.2.2 Nature of residues in livestock

### A 2.3.2.2.1 Study AMR 947-87

| | |
|---|---|
| Comments of zRMS: | The studies have been submitted as part of the AIR process for Nicosulfuron.<br>The studies have not been conducted in accordance with the GLP requirements.<br><br>The metabolism of nicosulfuron in goats proceeds primarily by three pathways:<br>(1) hydrolysis of the sulphonylurea bridge;<br>(2) N-demethylation and loss of sulphur dioxide to form DDTP and<br>(3) oxidation and conjugation at the 5-position of the pyrimidine ring.<br>It should be noted that the dose level represents > 1000 times the calculated maximum dietary burden of nicosulfuron residues in animal feed. Data indicated that nicosulfuron and its metabolites were neither accumulated nor retained in the lactating goat after repeated oral administration. Nicosulfuron residues were rapidly metabolised and excreted with little transfer of residues to milk or edible tissues.<br><br>The study is acceptable. |

| | |
|---|---|
| Reference: | KCA3 6.2.3 (report available from data owner) |
| Report | Lin P.T., Scott M.T., 1989. Metabolism of [pyrimidine-2-$^{14}$C]DPX-V9360 and [pyridine-2-$^{14}$C]DPX-V9360 in lactating goats<br>Syngenta Report No. AMR 947-87<br>Syngenta File No.<br>GLP, unpublished |
| Guideline(s): | Yes<br>U.S. EPA 171-4a (1982) |
| Deviations: | No |
| GLP: | ~~Yes~~ No, this study was conducted in the spirit of compliance with GLP regulations. |
| Acceptability: | Yes |

**Executive Summary**

A goat metabolism study was performed with [pyrimidine-2-$^{14}$C]- and [pyridine-2-$^{14}$C]- labelled nicosulfuron. Goats were dosed orally twice daily for 3 days with each radiolabel (one goat per radiolabel) at a dose level of 1.44 and 1.84 mg/kg body weight per day, respectively. The dose level represents > 1000 times the calculated maximum dietary burden of nicosulfuron residues in animal feed.

The concentration profiles of radioactivity in milk as well as the excretion patterns via bile, urine and faeces were followed. The goat was sacrificed 22 hours after the last dose and kidney, liver, muscle and fat were collected. Residual radioactivity and metabolite patterns in these organs were determined.

Approximately 49.1 and 80.2 % of the dose from the goat dosed with pyrimidine label and pyridine label was recovered, with the remainder assumed to be still present in the bladder and digestive tract at sacrifice. Excretion of the test item proceeded mainly via faeces (31.8 % and 64.2% of dose, respectively). In urine, excreted radioactivity accounted for 17.0% and 15.3% of the dose. Small amounts 0.14% and 0.69% of the dose were detected in the bile. A negligible amount was excreted in milk (0.07% dose in the pyrimidine label and below detection limit in pyridine label) and 0.03-0.04% of dose was detected in tissues.

Total radioactive residues detected in animal matrices are summarised below.

| **Tissue** | **Pyrimidine Label (µg/g parent equivs)** | **Pyridine Label (µg/g parent equivs)** |
|---|---|---|
| Milk | 0.032 (3 d) | Not detected |
| Liver | 0.053 | 0.098 |
| Kidney | 0.047 | 0.067 |
| Muscle | 0.001-0.010 | 0.010-0.017 |
| Fat | 0.005-0.018 | 0.010-0.027 |

Nicosulfuron residues did not accumulate in meat and milk. Total radioactive residues (TRR) in liver, kidney, fat and muscle were each less than 0.100 mg/kg nicosulfuron equivalents. TRR in milk from the goat dosed with pyridine-labelled nicosulfuron were not quantifiable (<twice background) for all samples. TRR in milk samples from the goat dosed with pyrimidine-labelled nicosulfuron were 0.008 to 0.032 mg/kg nicosulfuron equivalents and represented 0.07% of the total administered dose. Characterisation of residues in milk samples was not conducted due to the low concentration of $^{14}$C-residues. Residues in urine, faeces, bile and liver were primarily nicosulfuron. Residue concentrations in kidney, muscle and fat were too low to allow further characterisation.

Metabolites identified in urine from the goat dosed with pyridine-labelled nicosulfuron included ASDM, MU-467, DDTP, MU-466 and MU-2275. The major urinary metabolite from the goat dosed with pyrimidine-labelled nicosulfuron was a polar conjugate of AHDP.

It was concluded that nicosulfuron and its metabolites were neither accumulated nor retained in the lactating goat after repeated oral administration at a highly exaggerated dose.

**Materials and Methods**

**A. Materials**

| | | |
|---|---|---|
| 1. | Test material: | Nicosulfuron technical |
| | Lot/Batch #: | Not given |
| | Purity: | Not given |
| | Description: | Tan solid |
| | CAS#: | 111991-09-4 |
| 2 | Radiochemical test material: | $^{14}$C Nicosulfuron technical |
| a. | Lot/Batch #: | [Pyridine-2-$^{14}$C] Nicosulfuron Lot # 2385-050 |
| | Purity: | >95% |
| | Specific activity: | 62.9 µCi/mg (1.40 x $10^5$ dpm/µg) |
| | Stability of test compound: | Shown to be stable under the conditions of the test |

| | | |
|---|---|---|
| b. | Lot/Batch #: | [Pyrimidine-2-$^{14}$C] Nicosulfuron Lot # 2406-007 |
| | Purity: | >95% |
| | Specific activity: | 62.2 μCi/mg (1.38 x $10^5$ dpm/μg) |
| | Stability of test compound: | Shown to be stable under the conditions of the test |
| c. | Lot/Batch #: | [Pyridine-2-$^{13}$C] Nicosulfuron Lot #s 2424-030 and 2425-035 |
| | Purity: | 99% |
| | Specific activity: | Not given |
| | Stability of test compound: | Shown to be stable under the conditions of the test |
| d. | Lot/Batch #: | [Pyrimidine-2-$^{13}$C] Nicosulfuron HOTC 281 |
| | Purity: | 97% |
| | Specific activity: | Not given |
| | Stability of test compound: | Shown to be stable under the conditions of the test |

| | | |
|---|---|---|
| 3. | Final test substance: | Above batches combined with non-radiolabelled nicosulfuron |
| | Specific activity: | [Pyridine-2-$^{14}$C] Nicosulfuron: 1.35 μCi/mg<br>[Pyrimidine-2-$^{14}$C] Nicosulfuron: 1.04 μCi/mg |
| 4. | Test animals: | |
| | Species: | Goat (Togenberg breed) |
| | Gender: | Female |
| | Age: | Unknown |
| | Weight at dosing: | 42.7 kg (Goat 1, dosed with [Pyrimidine-2-$^{14}$C] nicosulfuron) and 33.6 kg (Goat 2, dosed with [Pyridine-2-$^{14}$C] nicosulfuron) |
| | Number of animals: | One per each different radiolabel; no control |
| | Acclimation period: | 9 days (Goat 1) and 11 days (Goat 2). Each goat had a bile-duct cannula inserted and were allowed to recuperate for 4 days before dosing |
| | Diet: | Commercial feed supplemented with alfalfa hay (type not reported) |
| | Water: | Tap water ad libitum |
| | Housing: | Individual metabolism cages |
| | Husbandry: | Husbandry conditions were in accordance with the USPHS-NIH publication *Guide to the Care and Use of Laboratory Animals* |
| 5. | Environmental conditions | |
| | Temperature: | Ambient |
| | Humidity: | Ambient |
| | Air change: | Unknown |
| | Photoperiod: | Alternating 12-hour light and dark cycles |

## B. Study design and methods

| | | |
|---|---|---|
| 1. | Dosing regime | |
| | Oral: Amount of Dose: | 1.84 mg/kg bw/d [Pyridine-2-14C] nicosulfuron<br>1.44 mg/kg bw/d [Pyrimidine-2-14C] nicosulfuron |
| | Food consumption: | 0.58–0.87 kg/day |
| | Vehicle: | Gelatine capsule |
| | Timing: | Twice daily (after morning and evening milkings) |
| | Duration: | Three days |
| 2. | Sample collection | |
| | Milk and Bile collection: | Twice daily |
| | Urine and faeces collection: | Once daily |
| | Interval from last dose to sacrifice: | 22 hours |

Tissues harvested and analysed: Fat, muscle, skin, kidneys, intestinal contents, rumen contents

3. Storage stability of samples

The concentration of total radioactive residues in milk, kidney, fat and muscle were low and were not analysed further. Thus, the storage stability of these samples was not studied.

All tissue and milk specimens were kept frozen at –20°C, from time of sacrifice (Oct. 20, 1987 for pyridine-label and Dec. 2, 1987 for pyrimidine-label) until analysis (Nov. 10, 1987 and Jan. 22, 1988, for pyridine and pyrimidine-labels, respectively). All analyses were conducted within a 1-6 month time frame.

**Results and Discussion**

**A. Total radioactive residues (TRRs)**

Total radioactive residues (TRRs) are presented in the table below. Most of the dose was eliminated in excreta, approximately 79.5% of the does for the pyridine-label and 48.8% for the pyrimidine-label was found in faeces and urine.

Concentrations of TRRs in milk were only above background for the goat dosed with pyrimidine-labelled nicosulfuron and these represented 0.07% of the total administrated dose. All tissues had TRRs of approximately 0.07 mg/kg nicosulfuron equivalents or less except for liver from the pyridine-labelled dosed goat (0.098 mg/kg nicosulfuron equivalents). TRR in bile, which was collected to facilitate identification of metabolites, represented 0.14% and 0.69% of the total administered dose for the pyrimidine-labelled and pyridine-labelled dosed goats, respectively. The overall $^{14}$C-recoveries of the administered dose were ca. 71% for the goat dosed with [pyridine-2-$^{14}$C]nicosulfuron and about 50% for the goat dosed with pyrimidine-labelled nicosulfuron.

**Table A 63: Summary of Total Radioactive Residues (TRR) in lactating goat after repeated administration of [$^{14}$C]nicosulfuron at a rate of 1.44 and 1.84 mg/kg bw/day (expressed as mg/kg nicosulfuron equivalents and % of dose)**

| | [Pyrimidine-2-$^{14}$C]nicosulfuron | | [Pyridine-2-$^{14}$C]nicosulfuron | |
|---|---|---|---|---|
| **Matrix** | **mg/kg** | **% of dose** | **mg/kg** | **% of dose** |
| Milk | | | | |
| Milk, Day 1[a] | 0.014 | 0.01 | ND[e] | -- |
| Milk, Day 2 | 0.027 | 0.03 | ND[e] | -- |
| Milk, Day 3 | 0.029 | 0.03 | ND[e] | -- |
| Total | | 0.07 | | ND |
| Muscle, liver, kidney, fat | | | | |
| Muscle[b] | 0.001-0.010 | [c] | 0.013-0.017 | [c] |
| Liver | 0.053 | 0.03 | 0.098 | 0.04 |
| Kidney | 0.047 | <0.01 | 0.067 | <0.01 |
| Fat[b] | 0.005-0.018 | [c] | 0.010-0.027 | [c] |
| Bile | | | | |
| Bile, Day 1[a] | 0.413 | 0.02 | 2.407 | 0.15 |
| Bile, Day 2 | 1.123 | 0.03 | 3.094 | 0.32 |
| Bile, Day 3 | 0.869 | 0.09 | 2.431 | 0.22 |
| Total | | 0.14 | | 0.69 |
| Urine | | | | |
| Urine, Day 1 | N/A | 2.9 | N/A | 4.0 |
| Urine, Day 2 | N/A | 6.8 | N/A | 5.2 |
| Urine Day 3 | N/A | 7.3 | N/A | 6.1 |
| Total | | 17.0 | | 15.3 |
| Faeces | | | | |
| Faeces, Day 0 | N/A | 0.04 | N/A | 0.05 |

| | [Pyrimidine-2-$^{14}$C]nicosulfuron | | [Pyridine-2-$^{14}$C]nicosulfuron | |
|---|---|---|---|---|
| **Matrix** | **mg/kg** | **% of dose** | **mg/kg** | **% of dose** |
| Faeces, Day 1 | N/A | 1.9 | N/A | 12.5 |
| Faeces, Day 2 | N/A | 14.3 | N/A | 24.3 |
| Faeces, Day 3 | N/A | 15.6 | N/A | 24.3 |
| Total | | 31.8 | | 64.2 |

a Average of morning and evening samples
b Range for flank, loin and leg muscle samples
c Total tissues were not taken and therefore data cannot be generated
d Range for back, omental, peripheral and renal fat samples
e ND=not detected

## B. Extraction and hydrolysis of residues

Total $^{14}$C-concentrations in milk, kidney, fat and muscle were extremely low and were not extracted for that reason. Liver samples were extracted with acetonitrile:water (9:1) in a 2:1 solvent:sample ratio using a wrist-action shaker for 30 minutes. After extraction, the mixtures were centrifuged at 9000 rpm for 20 minutes. The supernatants were decanted and aliquots were counted for total $^{14}$C by LSC. This extraction procedure was repeated twice more and the supernatants were combined. The combined supernatant was concentrated using a rotary evaporator followed by a stream of nitrogen.

Faeces were extracted following the same procedure used for liver except that a Waring® blender was used and the solvent-to-sample ratio was 5:1. Only the Day 3 faeces samples were extracted because these contained the highest concentrations of TRR. The concentration of unextracted $^{14}$C-residues was determined by combustion/LSC of the dried, extracted faecal samples.

Urine samples were extracted using XAD-2® resin washed with methanol in a Soxhlet apparatus for ten hours. The washed resin was transferred to a glass column and after conditioning with methanol-water and finally water, 100 mL of raw Day 3 urine was loaded onto the column. The eluent was collected after loading. The column was then eluted with water (150 mL) and then methanol (100 mL). Aliquots were counted to determine TRRs in each fraction. The eluent fractions were concentrated using a stream of nitrogen (N-Evap®, Organomaton, Berlin, MA, USA).

One urine fraction was also subjected to enzymatic hydrolysis using β-glucuronidase B1, α-glutamyltranspeptidase and sulfatase. The water fraction contained only 3.1% of the urinary $^{14}$C and was not analysed further.

## C. Characterisation and identification of residues

The methanolic fraction from urine from the goat dosed with [pyridine-2-$^{14}$C]nicosulfuron subjected to the XAD2® procedure described above contained five metabolites and parent. The 5 compounds were isolated by a combination of TLC and HPLC procedures. The isolated residues were identified by packed capillary LC/MS using a Finnigan MAT8230 magnetic mass spectrometer. Nicosulfuron was identified by comparing its HPLC retention time to that of a synthetic standard. The metabolites were identified as MU-467, DDTP, ASDM, MU-466 and MU-2275 (referred to as N-desmethyl nicosulfuron, nicosulfuron cyclised ipso compound, pyridine sulphonamide, N-desmethyl pyridine sulphonamide and pyridine sulphonamide carboxamide in the report. Identification of all metabolites, except for DDTP, was confirmed by matching spectra with known reference standards. For urine from the goat dosed with [pyrimidine-2-$^{14}$C]nicosulfuron, the methanolic fraction produced by the XAD2® contained one metabolite initially identified as AHDP (called 5-hydroxy pyrimidine amine in the report) by LC/MS. Comparison of the metabolite's HPLC retention time with a synthetic standard indicated that it could be a conjugate of AHDP. Incubation with β-glucuronidase B1, α-glutamyltranspeptidase and sulfatase and subsequent HPLC analysis indicated that the metabolite was not one of these conjugates.

Analysis of liver, bile and faeces indicated that nicosulfuron was the major residue in these samples comprising 80%, 84.3% and 91.4% of the injected radioactivity, respectively, or 0.015%, 1.3% and 22.2% of the total administered dose. The remaining $^{14}$C in liver consisted of a number of peaks each <0.01 mg/kg (nicosulfuron equivalents).

The distribution of nicosulfuron and its metabolites in urine is summarised below.

**Table A 64: Concentration of nicosulfuron and its metabolites in goat urine after dosing at a rate of 1.44 and 1.84 mg/kg bw/day**

| Residue | Day 2 urine | | Day 3 urine | | | |
|---|---|---|---|---|---|---|
| | [Pyridine-2-$^{14}$C] nicosulfuron | | [Pyridine-2-$^{14}$C] nicosulfuron | | [Pyrimidine-2-$^{14}$C] nicosulfuron | |
| | mg/kg | % dose | mg/kg | % dose | mg/kg | % dose |
| Nicosulfuron | 16.31 | 3.69 | 7.3 | 3.52 | 7.23 | 5.0 |
| MU-467 | 0.441 | 0.1 | 0.386 | 0.18 | NA[a] | -- |
| DDTP | 0.456 | 0.1 | 0.114 | 0.06 | NA | -- |
| ASDM | 0.444 | 0.1 | 0.785 | 0.39 | NA | -- |
| MU-466 | 0.833 | 0.18 | 0.188 | 0.09 | NA | -- |
| MU-2275 | 4.383 | 0.96 | 0.078 | 0.04 | NA | -- |
| AHDP conjugate | NA | -- | NA | -- | 1.50 | 1.1 |

a Not Applicable

Metabolism of nicosulfuron in the lactating goat proceeds primarily by three mechanisms as shown in Figure 6.2-2:
(1) hydrolysis of the sulphonylurea bridge to form ASDM and ADMP, which both undergo further metabolism;
(2) N-demethylation and loss of sulphur dioxide to form the cyclised ipso compound DDTP; and
(3) oxidation and conjugation at the 5-position of the pyrimidine ring to form AHDP and its conjugate.

**Conclusion**
Following daily dosing at 1.44 and 1.84 mg/kg body weight per day for [pyrimidine-2-$^{14}$C]- and [pyridine-2-$^{14}$C]- labelled nicosulfuron, it was concluded that nicosulfuron and its metabolites were neither accumulated nor retained in the lactating goat after repeated oral administration. The dose level represents > 1000 times the calculated maximum dietary burden of nicosulfuron residues in animal feed.
Minimal transfer of residues into milk and edible tissues were observed at this highly exaggerated dose. Total radioactive residues in muscle, kidney and fat were each less than 0.07 mg/kg and were not characterised due to their low concentration. Total $^{14}$C-residues in milk from the goat dosed with [pyridine-2-$^{14}$C]nicosulfuron were less than twice background levels and were thus not quantifiable. Total radioactive residues in milk from the goat dosed with [pyrimidine-2-$^{14}$C]nicosulfuron were between 0.008 and 0.032 mg/kg and represented 0.07% of the total administered dose. Characterisation of these residues was not possible due to their low concentrations.
Residues in liver, urine, faeces and bile were primarily unmetabolised nicosulfuron. Urinary metabolites identified from the goat dosed with [pyridine-2-$^{14}$C]nicosulfuron were ASDM, MU-467, DDTP, MU-466 and MU-2275. The major metabolite identified from the goat dosed with [pyrimidine-2-$^{14}$C]nicosulfuron was a polar conjugate of AHDP. This conjugate was not a glucuronide, sulphate or glutamate based on incubation of urine with β-glucuronidase, α-glutamyltranspeptidase and sulfatase, respectively.
The metabolism of nicosulfuron in goats proceeds primarily by three pathways: (1) hydrolysis of the sulphonylurea bridge; (2) N-demethylation and loss of sulphur dioxide to form DDTP and (3) oxidation and conjugation at the 5-position of the pyrimidine ring.
In conclusion, if goats were exposed to nicosulfuron residues through the diet, they are rapidly metabolised and excreted with little transfer of residues to milk or edible tissues.

## A 2.3.3 Magnitude of residues in plants

### A 2.3.3.1 Maize

**Table A 65: Comparison of intended and critical EU GAPs**

| Type of GAP | Number of applications | Application rate per treatment (precise unit) | Interval between application | Growth stage at last application | PHI (days) |
|---|---|---|---|---|---|
| cGAP EU (UK, 2006) | 1 | 0.060 kg a.s./ha | N/A | BBCH 18 | N/A[(a)] |
| cGAP EU (Art. 12, EFSA, 2016) | 1 | 0.060 kg a.s./ha | N/A | BBCH 12-20 | N/A[(a)] |
| Intended cGAP; use number 6* | 1 | 0.060 kg a.s./ha | N/A | BBCH 12-19 | N/A[(a)] |

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0
N/A – not applicable
(a) PHI is determined by crop maturity

## A 2.3.3.1.1 Study TK0258007-REG

| | |
|---|---|
| Comments of zRMS: | Four decline residue field trials on maize were successfully conducted in Northern France, Germany and Poland during 2015. Determination of dicamba and its metabolite, NOA405873 and nicosulfuron on maize was undertaken to evaluate the magnitude of residues. A method validation was performed on method GRM074.01A for the determination of nicosulfuron on maize kernels and whole plant as part of this study.<br>Dicamba and nicosulfuron were applied as A19658H, an oil dispersion (OD) formulation containing dicamba at 220 g per litre and nicosulfuron at 50 g per litre. To treated plot P2, one application, at BBCH 16-18, of A19658H was made at 288 g ai/ha for dicamba at 65.45 g ai/ha for nicosulfuron. Following the application, maize samples (whole plant, remaining plant and whole cobs) were collected at set intervals with untreated maize samples (whole plant, remaining plant and whole cobs) collected first. Samples were analysed for both dicamba and its metabolite, NOA405873 and nicosulfuron. The analytical method, GRM074.01A was successfully validated for the determination of nicosulfuron in maize kernels (dry commodity) and maize whole plant (high water commodity) with an LOQ of 0.01 mg/kg. The validation of the analytical method was conducted according to EU Guidance Document SANCO/3029/99 rev. 4 11/07/2000.<br><br>Results:<br><u>Nicosulfuron</u><br>Residues of nicosulfuron in whole cob samples taken at 75-79 BBCH and 89 BBCH were below the limit of quantification (<0.01 mg/kg).<br>Residues of nicosulfuron in remaining plant samples taken at 75-79 BBCH and 89 BBCH were below the limit of quantification (<0.01 mg/kg).<br>Residues of nicosulfuron in whole plant samples taken at 39 BBCH were between <0.01 and 0.01 mg/kg and at 63 BBCH and 83-85 BBCH were below the limit of quantification (<0.01 mg/kg).<br>Residues of nicosulfuron in untreated samples were below the limit of quantification (<0.01 mg/kg).<br><br>The study is acceptable. |

| | |
|---|---|
| Reference: | KCA3 6.3.1/01 |
| Report | Nicosulfuron and Dicamba- Residue Validation and Study on Maize in Northern France, Germany and Poland in 2015.<br>Andrews, G., 2016, Syngenta Report No. TK0258007-REG<br>Syngenta File No. A19658H_10060, |
| Guideline(s): | Yes<br>Commission of the European Communities, General Recommendations |

| | |
|---|---|
| | for the Design, Preparation and Realization of Residue Trials; 7029/VI/95 (rev. 5, working document).<br>Guidelines and Criteria for the Preparation and Presentation of Complete Dossiers and of Summary Dossiers for the Inclusion of Active Substances in Annex I of Directive 91/414/EEC (Article 5.3 and 8.2), 1996. |
| Deviations: | No |
| GLP: | Yes |
| Acceptability: | Yes |

**Table A 66: Summary of TK0258007-REG trials**

| | | | |
|---|---|---|---|
| **Field Trials, Crop Residue (Summary) :**NICOSULFURON AND DICAMBA- RESIDUE VALIDATION AND STUDY ON MAIZE IN NORTHERN FRANCE, GERMANY AND POLAND IN 2015 | | | |
| **Active Substance (common name):** | Nicosulfuron | **Commercial Product (name):** | $Commercial.Name$ |
| **Crop/Crop Group:** | Maize | **Producer of commercial product:** | Syngenta AG |
| **Responsible body for reporting (name, address):** | Syngenta AG, Basel, Switzerland | **Indoor/Glasshouse/Outdoor:** | Field |
| **Country:** | FRANCE, POLAND, GERMANY | **Other active substance in the formulation (common name and content):** | Dicamba (220 g/L |
| **Content of active substance (g/kg or g/L):** | 50 g/L | **Residues calculated as:** | $Residue.Units$ |
| **Formulation (e.g. WP):** | A19658H OD | | |
| **Analytical Method:**<br>Nicosulfuron (Cob without Kernel, Kernel, Kernels plus cob, Remaining Plant, Whole plant) GRM074.01A; 0.01 mg/kg | | | |
| **Recovery data:**<br>Nicosulfuron Cob without Kernel Mean = 103% RSD = N/A (n = 2 in 0.01 - 0.101 mg/kg spiking range)<br>Nicosulfuron Kernel Mean = 79% RSD = N/A (n = 2 in 0.01 - 0.101 mg/kg spiking range)<br>Nicosulfuron Remaining Plant Mean = 103% RSD = 19% (n = 4 in 0.01 - 0.101 mg/kg spiking range)<br>Nicosulfuron Whole plant Mean = 110% RSD = 24% (n = 4 in 0.01 - 0.101 mg/kg spiking range) | | | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Nicosulfuron (mg/kg) | | | |
| TK0258007-REG NC15022-01 FRANCE (Europe North) (-) | Maize (KROISSAN ) | 1.23 Mar 2015<br>2 –<br>3 - | Foliar | | 226.78 L/ha | 65.57495 g ai/ha<br>(-) | 13 Jun 2015 | BBCH 17 - 18 | Whole plant | 0.01 | 20 | 03 Jul 2015 | Field A19658H<br>SP (max days): Nicosulfuron/Kernel: 230 Nicosulfuron/Cob without Kernel: 230 Nicosulfuron/Rema |
| | | | | | | | | | Whole plant | < 0.01 | 34 | 17 Jul 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | 55 | 07 Aug 2015 | |
| | | | | | | | | | Kernel | < 0.01 | 55 | 07 Aug 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | 55 | 07 Aug 2015 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 55 | 07 Aug 2015 | |
| | | | | | | | | | Whole plant | < 0.01 | 86 | 07 Sep 2015 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Nicosulfuron (mg/kg) | | | |
| | | | | | | | | | Cob without Kernel | < 0.01 | 97 | 18 Sep 2015 | ining Plant: 230 Nicosulfuron/Kernels plus cob: 230 Nicosulfuron/Whole plant: 265 |
| | | | | | | | | | Kernel | < 0.01 | 97 | 18 Sep 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | 97 | 18 Sep 2015 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 97 | 18 Sep 2015 | |
| TK0258007-REG NC15022-02 FRANCE (Europe North) (-) | Maize (AXXYS) | 1.14 Apr 2015 2 – 3 - | Foliar | | 305.15 L/ha | 66.29478 g ai/ha<br><br>(-) | 03 Jun 2015 | BBCH 17 | Whole plant | < 0.01 | 28 | 01 Jul 2015 | Field A19658H<br><br>SP (max days): Nicosulfuron/Kernel: 239 Nicosulfuron/Cob without Kernel: 239 Nicosulfuron/Remaining Plant: 239 Nicosulfuron/Kernels plus cob: 239 Nicosulfuron/Whole plant: 267 |
| | | | | | | | | | Whole plant | < 0.01 | 34 | 07 Jul 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | 56 | 29 Jul 2015 | |
| | | | | | | | | | Kernel | < 0.01 | 56 | 29 Jul 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | 56 | 29 Jul 2015 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 56 | 29 Jul 2015 | |
| | | | | | | | | | Whole plant | < 0.01 | 83 | 25 Aug 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | 114 | 25 Sep 2015 | |
| | | | | | | | | | Kernel | < 0.01 | 114 | 25 Sep 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | 114 | 25 Sep 2015 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 114 | 25 Sep 2015 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Nicosulfuron (mg/kg) | | | |
| TK0258007-REG NC15022-03 GERMANY (Europe North) | Maize (Amaretto) | 1.04 May 2015<br>2 –<br>3 - | Foliar | | 200 L/ha | 65.02447 g ai/ha<br><br>(-) | 02 Jun 2015 | BBCH 16 | Whole plant | < 0.01 | 31 | 03 Jul 2015 | Field A19658H<br><br>SP (max days): Nicosulfuron/Kernel: 237 Nicosulfuron/Cob without Kernel: 237 Nicosulfuron/Remaining Plant: 237 Nicosulfuron/Kernels plus cob: 237 Nicosulfuron/Whole plant: 265 |
| | | | | | | | | | Whole plant | < 0.01 | 44 | 16 Jul 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | 59 | 31 Jul 2015 | |
| | | | | | | | | | Kernel | < 0.01 | 59 | 31 Jul 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | 59 | 31 Jul 2015 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 59 | 31 Jul 2015 | |
| | | | | | | | | | Whole plant | < 0.01 | 73 | 14 Aug 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | 86 | 27 Aug 2015 | |
| | | | | | | | | | Kernel | < 0.01 | 86 | 27 Aug 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | 86 | 27 Aug 2015 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 86 | 27 Aug 2015 | |
| TK0258007-REG NC15022-05 POLAND (Europe North) | Maize (NK Cooler) | 1.21 Apr 2015<br>2 –<br>3 - | Foliar | | 296.67 L/ha | 64.44167 g ai/ha<br><br>(-) | 17 Jun 2015 | BBCH 18 | Whole plant | < 0.01 | 19 | 06 Jul 2015 | Field A19658H<br><br>SP (max days): Nicosulfuron/Kernel: 220 Nicosulfuron/Cob without |
| | | | | | | | | | Whole plant | < 0.01 | 35 | 22 Jul 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | 61 | 17 Aug 2015 | |
| | | | | | | | | | Kernel | < 0.01 | 61 | 17 Aug 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | 61 | 17 Aug 2015 | |

| (1) Report No. Trial No. Location (Region) (Postcode) | (2) Commodity/ Variety (a) | (3) Date of 1. Sowing or Planting 2. Flowering 3. Harvest (b) | (4) Method of Treatment | (5) Application rate per treatment | | | (6) Date of treatment(s) or no of treatment(s) and last date<br><br>Application Interval (days) (c) | (7) Growth Stage at Treatment | (8) Portion Analyzed | (9) Residue found (Uncorrected) | (10) PHI (d) | (11) Sample Date (Cut Date) (d) | (12) Trial Details (e) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Conc'n | Water | Rate (Additive Type, Rate) | | | | Nicosulfuron (mg/kg) | | | |
| | | | | | | | | | Remaining Plant | < 0.01 | 61 | 17 Aug 2015 | Kernel: 220 Nicosulfuron/Remaining Plant: 220 Nicosulfuron/Kernels plus cob: 220 Nicosulfuron/Whole plant: 262 |
| | | | | | | | | | Whole plant | < 0.01 | 89 | 14 Sep 2015 | |
| | | | | | | | | | Cob without Kernel | < 0.01 | 111 | 06 Oct 2015 | |
| | | | | | | | | | Kernel | < 0.01 | 111 | 06 Oct 2015 | |
| | | | | | | | | | Kernels plus cob | < 0.01 + | 111 | 06 Oct 2015 | |
| | | | | | | | | | Remaining Plant | < 0.01 | 111 | 06 Oct 2015 | |

(a) According to Codex (or other e.g. EU) classification

(b) Only if relevant

(c) Year must be indicated

(d) Minimum number of days after last application (Label pre-harvest interval, PHI, underline)

(e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included.

(*) Indicates sample taken prior to application

(#) Indicates corrected Residue values

(^) PHI calculated using cut date

(+) Indicates calculated Residue value

(DBA) Days Before Application

SP (max days): Maximum storage period

### A 2.3.4 Magnitude of residues in livestock

No new data submitted in the framework of this application.

### A 2.3.5 Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation)

No new data submitted in the framework of this application.

### A 2.3.6 Magnitude of residues in representative succeeding crops

No new data submitted in the framework of this application.

### A 2.3.7 Other/Special Studies

No new data are submitted in the framework of this application.

# Appendix 3 Pesticide Residue Intake Model (PRIMo)

## A 3.1 TMDI calculations - dicamba

### A 3.1.1 Dicamba

European Food Safety Authority

EFSA PRIMo revision 3.1; 2021/01/06

**dicamba**

LOQs (mg/kg) range from: 0,05 to: 0,05

**Toxicological reference values**

| | | | |
|---|---|---|---|
| ADI (mg/kg bw/day): | 0,3 | ARfD (mg/kg bw): | 0,3 |
| Source of ADI: | EFSA | Source of ARfD: | EFSA |
| Year of evaluation: | 2011 | Year of evaluation: | 2011 |

Input values

Details - chronic risk assessment | Supplementary results - chronic risk assessment

Details - acute risk assessment/children | Details - acute risk assessment/adults

Comments:

**Normal mode**

**Chronic risk assessment: JMPR methodology (IEDI/TMDI)**

No of diets exceeding the ADI : ---

| TMDI/NEDI/IEDI calculation (based on average food consumption) | Calculated exposure (% of ADI) | MS Diet | Expsoure (µg/kg bw per day) | Highest contributor to MS diet (in % of ADI) | Commodity / group of commodities | 2nd contributor to MS diet (in % of ADI) | Commodity / group of commodities | 3rd contributor to MS diet (in % of ADI) | Commodity / group of commodities | Exposure resulting from MRLs set at the LOQ (in % of ADI) | Exposure resulting from commodities not under assessment (in % of ADI) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | 19% | GEMS/Food G11 | 57,54 | 12% | Soyabeans | 2% | Wheat | 2% | Barley | 0,3% | 0,0% |
| | 17% | GEMS/Food G10 | 51,27 | 11% | Soyabeans | 3% | Wheat | 1% | Barley | 0,3% | 0,1% |
| | 16% | NL toddler | 48,50 | 10% | Milk: Cattle | 3% | Wheat | 1% | Maize/corn | 0,6% | 1% |
| | 14% | GEMS/Food G08 | 40,80 | 7% | Soyabeans | 3% | Wheat | 2% | Barley | 0,3% | 0,1% |
| | 13% | GEMS/Food G15 | 38,94 | 6% | Soyabeans | 3% | Wheat | 2% | Barley | 0,3% | 0,1% |
| | 13% | GEMS/Food G07 | 37,67 | 6% | Soyabeans | 3% | Wheat | 1% | Barley | 0,3% | 0,1% |
| | 11% | GEMS/Food G06 | 32,96 | 5% | Wheat | 4% | Soyabeans | 0,6% | Sugar canes | 0,3% | 0,2% |
| | 9% | UK infant | 27,10 | 6% | Milk: Cattle | 2% | Wheat | 0,2% | Bovine: Muscle/meat | 0,2% | 0,2% |
| | 9% | NL child | 25,55 | 4% | Milk: Cattle | 3% | Wheat | 0,7% | Soyabeans | 0,5% | 0,0% |
| | 8% | FR child 3 15 yr | 23,60 | 4% | Milk: Cattle | 3% | Wheat | 0,2% | Bovine: Muscle/meat | 0,3% | 0,1% |
| | 8% | FR toddler 2 3 yr | 23,48 | 5% | Milk: Cattle | 2% | Wheat | 0,2% | Bovine: Muscle/meat | 0,3% | 0,0% |
| | 8% | DE child | 23,31 | 3% | Milk: Cattle | 3% | Wheat | 0,4% | Apples | 0,4% | 0,0% |
| | 7% | UK toddler | 20,13 | 3% | Milk: Cattle | 3% | Wheat | 0,2% | Bovine: Muscle/meat | 0,3% | 0,0% |
| | 7% | DK child | 19,84 | 3% | Wheat | 2% | Milk: Cattle | 0,9% | Rye | 0,2% | 0,0% |
| | 6% | RO general | 17,67 | 3% | Wheat | 2% | Milk: Cattle | 0,2% | Maize/corn | 0,3% | 0,2% |
| | 6% | ES child | 17,20 | 3% | Wheat | 2% | Milk: Cattle | 0,2% | Bovine: Muscle/meat | 0,2% | 0,0% |
| | 5% | DE general | 16,28 | 2% | Milk: Cattle | 1% | Wheat | 1% | Barley | 0,2% | 0,0% |
| | 5% | SE general | 16,26 | 2% | Wheat | 2% | Milk: Cattle | 0,7% | Bovine: Muscle/meat | 0,3% | |
| | 5% | DE women 14-50 yr | 14,74 | 2% | Milk: Cattle | 1% | Wheat | 0,4% | Barley | 0,3% | 0,0% |
| | 5% | IT toddler | 14,52 | 4% | Wheat | 0,2% | Other cereals | 0,0% | Apples | 0,1% | 0,0% |
| | 4% | NL general | 12,92 | 1% | Milk: Cattle | 1% | Wheat | 0,7% | Barley | 0,2% | 0,0% |
| | 4% | PT general | 12,48 | 3% | Wheat | 1% | Soyabeans | 0,1% | Potatoes | 0,2% | 0,1% |
| | 4% | ES adult | 11,92 | 2% | Wheat | 1% | Barley | 0,8% | Milk: Cattle | 0,2% | 0,0% |
| | 4% | FR infant | 10,98 | 3% | Milk: Cattle | 0,5% | Wheat | 0,1% | Bovine: Muscle/meat | 0,2% | 0,0% |
| | 3% | IE adult | 9,72 | 2% | Wheat | 0,7% | Milk: Cattle | 0,2% | Asparagus | 0,4% | 0,0% |
| | 3% | IT adult | 9,18 | 3% | Wheat | 0,1% | Other cereals | 0,0% | Apples | 0,1% | 0,0% |
| | 3% | FR adult | 8,09 | 1% | Wheat | 0,7% | Milk: Cattle | 0,1% | Bovine: Muscle/meat | 0,2% | 0,0% |
| | 2% | UK vegetarian | 6,55 | 1% | Wheat | 0,5% | Milk: Cattle | 0,0% | Barley | 0,1% | 0,0% |
| | 2% | DK adult | 6,05 | 0,9% | Milk: Cattle | 0,7% | Wheat | 0,1% | Bovine: Muscle/meat | 0,1% | |
| | 2% | UK adult | 5,93 | 1% | Wheat | 0,5% | Milk: Cattle | 0,1% | Bovine: Muscle/meat | 0,1% | 0,0% |
| | 2% | LT adult | 5,92 | 0,7% | Wheat | 0,7% | Milk: Cattle | 0,2% | Rye | 0,1% | 0,0% |
| | 1% | FI 3 yr | 4,44 | 0,8% | Wheat | 0,2% | Barley | 0,1% | Rye | 0,2% | 0,0% |
| | 1% | IE child | 4,43 | 0,8% | Wheat | 0,6% | Milk: Cattle | 0,0% | Rice | 0,0% | 0,0% |
| | 1% | FI 6 yr | 3,57 | 0,6% | Wheat | 0,1% | Barley | 0,1% | Rye | 0,2% | 0,0% |
| | 0,7% | FI adult | 2,07 | 0,2% | Wheat | 0,1% | Rye | 0,1% | Coffee beans | 0,2% | 0,0% |
| | 0,2% | PL general | 0,64 | 0,1% | Apples | 0,1% | Potatoes | 0,0% | Tomatoes | 0,1% | 0,0% |

**Conclusion:**
The estimated long-term dietary intake (TMDI/NEDI/IEDI) was below the ADI.
The long-term intake of residues of dicamba is unlikely to present a public health concern.
DISCLAIMER: Dietary data from the UK were included in PRIMO when the UK was a member of the European Union.

# A 3.1.2 Mesotrione

European Food Safety Authority
EFSA PRIMo revision 3.1; 2021/01/06

**mesotrione**

| | | | |
|---|---|---|---|
| LOQs (mg/kg) range from: | 0,01 | to: | 0,05 |

**Toxicological reference values**

| | | | |
|---|---|---|---|
| ADI (mg/kg bw/day): | 0,01 | ARfD (mg/kg bw): | 0,02 |
| Source of ADI: | EFSA | Source of ARfD: | EFSA |
| Year of evaluation: | 2016 | Year of evaluation: | 2016 |

Input values
Details - chronic risk assessment
Supplementary results - chronic risk assessment
Details - acute risk assessment/children
Details - acute risk assessment/adults

Comments:

**Normal mode**

**Chronic risk assessment: JMPR methodology (IEDI/TMDI)**

No of diets exceeding the ADI : ---

| | Calculated exposure (% of ADI) | MS Diet | Expsoure (µg/kg bw per day) | Highest contributor to MS diet (in % of ADI) | Commodity / group of commodities | 2nd contributor to MS diet (in % of ADI) | Commodity / group of commodities | 3rd contributor to MS diet (in % of ADI) | Commodity / group of commodities | Exposure resulting from MRLs set at the LOQ (in % of ADI) | Exposure resulting from commodities not under assessment (in % of ADI) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| TMDI/NEDI/IEDI calculation (based on average food consumption) | 12% | NL toddler | 1,24 | 6% | Milk: Cattle | 1% | Apples | 0,7% | Maize/corn | 12% | 0,7% |
| | 7% | NL child | 0,67 | 2% | Milk: Cattle | 0,8% | Sugar beet roots | 0,6% | Apples | 7% | 0,0% |
| | 6% | DE child | 0,64 | 2% | Milk: Cattle | 1% | Apples | 0,4% | Wheat | 6% | 0,0% |
| | 6% | UK infant | 0,61 | 4% | Milk: Cattle | 0,3% | Potatoes | 0,3% | Wheat | 6% | 0,1% |
| | 6% | FR toddler 2 3 yr | 0,56 | 3% | Milk: Cattle | 0,3% | Apples | 0,3% | Wheat | 6% | 0,0% |
| | 6% | FR child 3 15 yr | 0,55 | 2% | Milk: Cattle | 0,5% | Wheat | 0,4% | Sugar beet roots | 6% | 0,0% |
| | 5% | GEMS/Food G11 | 0,49 | 1% | Soyabeans | 0,8% | Milk: Cattle | 0,4% | Potatoes | 4% | 0,0% |
| | 4% | UK toddler | 0,45 | 2% | Milk: Cattle | 0,4% | Wheat | 0,3% | Potatoes | 4% | 0,0% |
| | 4% | GEMS/Food G10 | 0,43 | 1,0% | Soyabeans | 0,5% | Milk: Cattle | 0,4% | Wheat | 3% | 0,1% |
| | 4% | GEMS/Food G07 | 0,42 | 0,6% | Milk: Cattle | 0,5% | Soyabeans | 0,4% | Wheat | 4% | 0,0% |
| | 4% | GEMS/Food G08 | 0,42 | 0,6% | Soyabeans | 0,6% | Milk: Cattle | 0,4% | Wheat | 4% | 0,0% |
| | 4% | GEMS/Food G15 | 0,42 | 0,7% | Milk: Cattle | 0,5% | Soyabeans | 0,5% | Wheat | 4% | 0,1% |
| | 4% | DK child | 0,41 | 1% | Milk: Cattle | 0,6% | Rye | 0,4% | Wheat | 4% | 0,0% |
| | 4% | GEMS/Food G06 | 0,41 | 0,7% | Wheat | 0,4% | Soyabeans | 0,4% | Tomatoes | 4% | 0,1% |
| | 4% | RO general | 0,38 | 1% | Milk: Cattle | 0,5% | Wheat | 0,4% | Potatoes | 4% | 0,1% |
| | 4% | ES child | 0,38 | 1% | Milk: Cattle | 0,4% | Wheat | 0,3% | Cocoa beans | 4% | 0,0% |
| | 4% | SE general | 0,37 | 1% | Milk: Cattle | 0,4% | Bovine: Muscle/meat | 0,4% | Potatoes | 4% | |
| | 4% | DE women 14-50 yr | 0,37 | 1% | Milk: Cattle | 0,5% | Sugar beet roots | 0,3% | Apples | 4% | 0,0% |
| | 4% | DE general | 0,36 | 1% | Milk: Cattle | 0,4% | Sugar beet roots | 0,2% | Apples | 4% | 0,0% |
| | 4% | FI adult | 0,35 | 3% | Coffee beans | 0,1% | Potatoes | 0,1% | Rye | 4% | 0,0% |
| | 3% | IE adult | 0,33 | 0,4% | Milk: Cattle | 0,4% | Sweet potatoes | 0,2% | Wheat | 3% | 0,0% |
| | 3% | NL general | 0,30 | 0,8% | Milk: Cattle | 0,3% | Sugar beet roots | 0,2% | Potatoes | 3% | 0,0% |
| | 3% | FR infant | 0,29 | 2% | Milk: Cattle | 0,2% | Potatoes | 0,2% | Apples | 3% | 0,0% |
| | 2% | FR adult | 0,22 | 0,4% | Milk: Cattle | 0,2% | Wine grapes | 0,2% | Wheat | 2% | 0,0% |
| | 2% | PT general | 0,21 | 0,5% | Potatoes | 0,4% | Wheat | 0,2% | Wine grapes | 2% | 0,0% |
| | 2% | ES adult | 0,21 | 0,5% | Milk: Cattle | 0,2% | Wheat | 0,1% | Oranges | 2% | 0,0% |
| | 2% | FI 3 yr | 0,18 | 0,5% | Potatoes | 0,1% | Bananas | 0,1% | Wheat | 2% | 0,0% |
| | 2% | IT toddler | 0,16 | 0,7% | Wheat | 0,2% | Other cereals | 0,1% | Tomatoes | 2% | 0,0% |
| | 2% | DK adult | 0,16 | 0,5% | Milk: Cattle | 0,1% | Potatoes | 0,1% | Wheat | 2% | |
| | 2% | LT adult | 0,16 | 0,4% | Milk: Cattle | 0,3% | Potatoes | 0,2% | Apples | 2% | 0,0% |
| | 1% | UK vegetarian | 0,15 | 0,3% | Milk: Cattle | 0,2% | Wheat | 0,1% | Potatoes | 1% | 0,0% |
| | 1% | FI 6 yr | 0,14 | 0,4% | Potatoes | 0,1% | Cocoa beans | 0,1% | Wheat | 1% | 0,0% |
| | 1% | UK adult | 0,14 | 0,3% | Milk: Cattle | 0,2% | Wheat | 0,1% | Potatoes | 1% | 0,0% |
| | 1% | IT adult | 0,12 | 0,4% | Wheat | 0,1% | Tomatoes | 0,1% | Apples | 1% | 0,0% |
| | 1,0% | PL general | 0,10 | 0,3% | Potatoes | 0,2% | Apples | 0,1% | Tomatoes | 1,0% | 0,0% |
| | 0,8% | IE child | 0,08 | 0,4% | Milk: Cattle | 0,1% | Wheat | 0,1% | Potatoes | 0,8% | 0,0% |

**Conclusion:**
The estimated long-term dietary intake (TMDI/NEDI/IEDI) was below the ADI.
The long-term intake of residues of mesotrione is unlikely to present a public health concern.
DISCLAIMER: Dietary data from the UK were included in PRIMO when the UK was a member of the European Union.

# A 3.1.3 Nicosulfuron

European Food Safety Authority

EFSA PRIMo revision 3.1; 2021/01/06

## nicosulfuron

LOQs (mg/kg) range from: **0,01** to: **0,05**

**Toxicological reference values**

| | | | |
|---|---|---|---|
| ADI (mg/kg bw/day): | **2** | ARfD (mg/kg bw): | **not necessary** |
| Source of ADI: | **EFSA** | Source of ARfD: | **EFSA** |
| Year of evaluation: | **2007** | Year of evaluation: | **2007** |

Input values

Details - chronic risk assessment

Supplementary results - chronic risk assessment

Details - acute risk assessment/children

Details - acute risk assessment/adults

Comments:

**Normal mode**

**Chronic risk assessment: JMPR methodology (IEDI/TMDI)**

No of diets exceeding the ADI : ---

| TMDI/NEDI/IEDI calculation (based on average food consumption) | Calculated exposure (% of ADI) | MS Diet | Exposure (µg/kg bw per day) | Highest contributor to MS diet (in % of ADI) | Commodity / group of commodities | 2nd contributor to MS diet (in % of ADI) | Commodity / group of commodities | 3rd contributor to MS diet (in % of ADI) | Commodity / group of commodities | Exposure resulting from MRLs set at the LOQ (in % of ADI) | Exposure resulting from commodities not under assessment (in % of ADI) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0,1% | NL toddler | 1,91 | 0,1% | Milk: Cattle | 0,0% | Apples | 0,0% | Maize/corn | 0,1% | |
| | 0,1% | UK infant | 1,04 | 0,0% | Milk: Cattle | 0,0% | Potatoes | 0,0% | Eggs: Chicken | 0,1% | |
| | 0,0% | NL child | 0,97 | 0,0% | Milk: Cattle | 0,0% | Sugar beet roots | 0,0% | Apples | 0,0% | |
| | 0,0% | FR toddler 2 3 yr | 0,90 | 0,0% | Milk: Cattle | 0,0% | Apples | 0,0% | Wheat | 0,0% | |
| | 0,0% | DE child | 0,87 | 0,0% | Milk: Cattle | 0,0% | Apples | 0,0% | Wheat | 0,0% | |
| | 0,0% | FR child 3 15 yr | 0,85 | 0,0% | Milk: Cattle | 0,0% | Wheat | 0,0% | Sugar beet roots | 0,0% | |
| | 0,0% | UK toddler | 0,69 | 0,0% | Milk: Cattle | 0,0% | Wheat | 0,0% | Potatoes | 0,0% | |
| | 0,0% | DK child | 0,59 | 0,0% | Milk: Cattle | 0,0% | Rye | 0,0% | Swine: Muscle/meat | 0,0% | |
| | 0,0% | GEMS/Food G11 | 0,58 | 0,0% | Milk: Cattle | 0,0% | Soyabeans | 0,0% | Potatoes | 0,0% | |
| | 0,0% | ES child | 0,56 | 0,0% | Milk: Cattle | 0,0% | Wheat | 0,0% | Bovine: Muscle/meat | 0,0% | |
| | 0,0% | SE general | 0,55 | 0,0% | Milk: Cattle | 0,0% | Bovine: Muscle/meat | 0,0% | Potatoes | 0,0% | |
| | 0,0% | GEMS/Food G07 | 0,53 | 0,0% | Milk: Cattle | 0,0% | Wheat | 0,0% | Potatoes | 0,0% | |
| | 0,0% | RO general | 0,53 | 0,0% | Milk: Cattle | 0,0% | Wheat | 0,0% | Potatoes | 0,0% | |
| | 0,0% | GEMS/Food G15 | 0,52 | 0,0% | Milk: Cattle | 0,0% | Wheat | 0,0% | Potatoes | 0,0% | |
| | 0,0% | DE women 14-50 yr | 0,52 | 0,0% | Milk: Cattle | 0,0% | Sugar beet roots | 0,0% | Apples | 0,0% | |
| | 0,0% | GEMS/Food G08 | 0,52 | 0,0% | Milk: Cattle | 0,0% | Wheat | 0,0% | Soyabeans | 0,0% | |
| | 0,0% | DE general | 0,51 | 0,0% | Milk: Cattle | 0,0% | Sugar beet roots | 0,0% | Apples | 0,0% | |
| | 0,0% | GEMS/Food G10 | 0,51 | 0,0% | Milk: Cattle | 0,0% | Soyabeans | 0,0% | Wheat | 0,0% | |
| | 0,0% | FR infant | 0,47 | 0,0% | Milk: Cattle | 0,0% | Potatoes | 0,0% | Apples | 0,0% | |
| | 0,0% | GEMS/Food G06 | 0,45 | 0,0% | Wheat | 0,0% | Milk: Cattle | 0,0% | Tomatoes | 0,0% | |
| | 0,0% | NL general | 0,43 | 0,0% | Milk: Cattle | 0,0% | Sugar beet roots | 0,0% | Potatoes | 0,0% | |
| | 0,0% | IE adult | 0,42 | 0,0% | Milk: Cattle | 0,0% | Sweet potatoes | 0,0% | Wheat | 0,0% | |
| | 0,0% | FI adult | 0,35 | 0,0% | Coffee beans | 0,0% | Potatoes | 0,0% | Rye | 0,0% | |
| | 0,0% | FR adult | 0,30 | 0,0% | Milk: Cattle | 0,0% | Wine grapes | 0,0% | Wheat | 0,0% | |
| | 0,0% | ES adult | 0,29 | 0,0% | Milk: Cattle | 0,0% | Wheat | 0,0% | Bovine: Muscle/meat | 0,0% | |
| | 0,0% | DK adult | 0,24 | 0,0% | Milk: Cattle | 0,0% | Swine: Muscle/meat | 0,0% | Potatoes | 0,0% | |
| | 0,0% | LT adult | 0,22 | 0,0% | Milk: Cattle | 0,0% | Potatoes | 0,0% | Swine: Muscle/meat | 0,0% | |
| | 0,0% | PT general | 0,22 | 0,0% | Potatoes | 0,0% | Wheat | 0,0% | Wine grapes | 0,0% | |
| | 0,0% | UK vegetarian | 0,18 | 0,0% | Milk: Cattle | 0,0% | Wheat | 0,0% | Potatoes | 0,0% | |
| | 0,0% | UK adult | 0,18 | 0,0% | Milk: Cattle | 0,0% | Wheat | 0,0% | Potatoes | 0,0% | |
| | 0,0% | FI 3 yr | 0,18 | 0,0% | Potatoes | 0,0% | Bananas | 0,0% | Wheat | 0,0% | |
| | 0,0% | IT toddler | 0,17 | 0,0% | Wheat | 0,0% | Other cereals | 0,0% | Tomatoes | 0,0% | |
| | 0,0% | FI 6 yr | 0,14 | 0,0% | Potatoes | 0,0% | Cocoa beans | 0,0% | Wheat | 0,0% | |
| | 0,0% | IE child | 0,12 | 0,0% | Milk: Cattle | 0,0% | Wheat | 0,0% | Potatoes | 0,0% | |
| | 0,0% | IT adult | 0,12 | 0,0% | Wheat | 0,0% | Tomatoes | 0,0% | Apples | 0,0% | |
| | 0,0% | PL general | 0,10 | 0,0% | Potatoes | 0,0% | Apples | 0,0% | Tomatoes | 0,0% | |

**Conclusion:**
The estimated long-term dietary intake (TMDI/NEDI/IEDI) was below the ADI.
The long-term intake of residues of nicosulfuron is unlikely to present a public health concern.
DISCLAIMER: Dietary data from the UK were included in PRIMO when the UK was a member of the European Union.

## A 3.2 IEDI calculations

### A 3.2.1 Dicamba

Not required. TMDI < 100%.

### A 3.2.2 Mesotrione

Not required. TMDI < 100%.

### A 3.2.3 Nicosulfuron

Not required. TMDI < 100%.

# A 3.3 IESTI calculations - - unprocessed and processed commodities

## A 3.3.1 Dicamba

| Acute risk assessment /children | Acute risk assessment / adults / general population | Acute risk assessment /children | Acute risk assessment / adults / general population |
|---|---|---|---|
| Details - acute risk assessment /children | Details - acute risk assessment/adults | Hide IESTI new calculations | Show IESTI new calculations |

The acute risk assessment is based on the ARfD. DISCLAIMER: Dietary data from the UK were included in PRIMO when the UK was a member of the European

The calculation is based on the large portion of the most critical consumer group.

**IESTI new calculations:**
The calculation is performed with the MRL and the peeling/processing factor (PF), taking into account the residue in the edible portion and/or the conversion factor for the residue definition (CF). For case 2a, 2b and 3 calculations a variability factor of 3 is used. Since this methodology is not based on internationally agreed principles, the results are considered as indicative only.
**Since this methodology is not based on internationally agreed principles, the results are considered as indicative only.**

**Show results of IESTI calculation only for crops with GAPs under assessment**

**Unprocessed commodities**

| **Results for children** | **Results for adults** | **IESTI new** **Results for children** | **IESTI new** **Results for adults** |
|---|---|---|---|
| No. of commodities for which ARfD/ADI is exceeded (IESTI): --- | No. of commodities for which ARfD/ADI is exceeded (IESTI): --- | No. of commodities for which ARfD/ADI is exceeded (IESTI new): --- | No. of commodities for which ARfD/ADI is exceeded (IESTI new): --- |

**IESTI**

| Highest % of ARfD/ADI | Commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) |
|---|---|---|---|
| 1% | Maize/corn | 0,5 / 0,5 | 3,4 |

**IESTI**

| Highest % of ARfD/ADI | Commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) |
|---|---|---|---|
| 0,4% | Maize/corn | 0,5 / 0,5 | 1,1 |

IESTI new

| Highest % of ARfD/ADI | Commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) |
|---|---|---|---|
| 1% | Maize/corn | 0,5 / 0,5 | 3,4 |

IESTI new

| Highest % of ARfD/ADI | Commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) |
|---|---|---|---|
| 0,4% | Maize/corn | 0,5 / 0,5 | 1,1 |

| **Total number of commodities exceeding the ARfD/ADI in children and adult diets (IESTI calculation)** | | **Total number of commodities found exceeding the ARfD/ADI in children and adult diets (IESTI new calculation)** | |
|---|---|---|---|

**Processed commodities**

| **Results for children** | **Results for adults** | **Results for children** | **Results for adults** |
|---|---|---|---|
| No of processed commodities for which ARfD/ADI is exceeded (IESTI): --- | No of processed commodities for which ARfD/ADI is exceeded (IESTI): --- | No of processed commodities for which ARfD/ADI is exceeded (IESTI new): --- | No of processed commodities for which ARfD/ADI is exceeded (IESTI new): --- |

**IESTI**

| Highest % of ARfD/ADI | Processed commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) |
|---|---|---|---|
| 4% | Maize / oil | 0,5 / 12,5 | 12 |
| 0,4% | Maize / processed (not spe | 0,5 / 0,5 | 1,1 |

**IESTI**

| Highest % of ARfD/ADI | Processed commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) |
|---|---|---|---|
| 2% | Maize / oil | 0,5 / 12,5 | 6,3 |

**IESTI new**

| Highest % of ARfD/ADI | Processed commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) |
|---|---|---|---|
| 4% | Maize / oil | 0,5 / 12,5 | 12 |
| 0,4% | Maize / processed (not | 0,5 / 0,5 | 1,1 |

**IESTI new**

| Highest % of ARfD/ADI | Processed commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) |
|---|---|---|---|
| 2% | Maize / oil | 0,5 / 12,5 | 6,3 |

Expand/collapse list

**Conclusion:**
No exceedance of the toxicological reference value was identified for any unprocessed commodity.
A short term intake of residues of dicamba is unlikely to present a public health risk.
For processed commodities, no exceedance of the ARfD/ADI was identified.

## A 3.3.2 Mesotrione

| Acute risk assessment /children | Acute risk assessment / adults / general population | Acute risk assessment /children | Acute risk assessment / adults / general population |
|---|---|---|---|
| Details - acute risk assessment /children | Details - acute risk assessment/adults | Hide IESTI new calculations | Show IESTI new calculations |

The acute risk assessment is based on the ARfD. DISCLAIMER: Dietary data from the UK were included in PRIMO when the UK was a member of the Eu

The calculation is based on the large portion of the most critical consumer group.

**IESTI new calculations:**
The calculation is performed with the MRL and the peeling/processing factor (PF), taking into account the residue in the edible portion and/or the conversion factor for the residue definition (CF). For case 2a, 2b and 3 calculations a variability factor of 3 is used. Since this methodology is not based on internationally agreed principles, the results are considered as indicative only.
**Since this methodology is not based on internationally agreed principles, the results are considered as indicative only.**

**Show results of IESTI calculation only for crops with GAPs under assessment**

**Unprocessed commodities**

| **Results for children** No. of commodities for which ARfD/ADI is exceeded (IESTI): --- | | | | **Results for adults** No. of commodities for which ARfD/ADI is exceeded (IESTI): --- | | | | **IESTI new Results for children** No. of commodities for which ARfD/ADI is exceeded (IESTI new): --- | | | | **IESTI new Results for adults** No. of commodities for which ARfD/ADI is exceeded (IESTI new): --- | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **IESTI** | | | | **IESTI** | | | | IESTI new | | | | IESTI new | | | |
| Highest % of ARfD/ADI | Commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) | Highest % of ARfD/ADI | Commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) | Highest % of ARfD/ADI | Commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) | Highest % of ARfD/ADI | Commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) |
| 0,3% | Maize/corn | 0,01 / 0,01 | 0,07 | 0,1% | Maize/corn | 0,01 / 0,01 | 0,02 | 0,3% | Maize/corn | 0,01 / 0,01 | 0,07 | 0,1% | Maize/corn | 0,01 / 0,01 | 0,02 |
| **Total number of commodities exceeding the ARfD/ADI in children and adult diets (IESTI calculation)** | | | | | | | | **Total number of commodities found exceeding the ARfD/ADI in children and adult diets (IESTI new calculation)** | | | | | | | |

**Processed commodities**

| **Results for children** No of processed commodities for which ARfD/ADI is exceeded (IESTI): --- | | | | **Results for adults** No of processed commodities for which ARfD/ADI is exceeded (IESTI): --- | | | | **Results for children** No of processed commodities for which ARfD/ADI is exceeded (IESTI new): --- | | | | **Results for adults** No of processed commodities for which ARfD/ADI is exceeded (IESTI new): --- | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **IESTI** | | | | **IESTI** | | | | **IESTI new** | | | | **IESTI new** | | | |
| Highest % of ARfD/ADI | Processed commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) | Highest % of ARfD/ADI | Processed commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) | Highest % of ARfD/ADI | Processed commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) | Highest % of ARfD/ADI | Processed commodities | MRL / input for RA (mg/kg) | Exposure (µg/kg bw) |
| 1% | Maize / oil | 0,01 / 0,25 | 0,23 | 0,6% | Maize / oil | 0,01 / 0,25 | 0,13 | 1% | Maize / oil | 0,01 / 0,25 | 0,23 | 0,6% | Maize / oil | 0,01 / 0,25 | 0,13 |
| 0,1% | Maize / processed (not spe | 0,01 / 0,01 | 0,02 | | | | | 0,1% | Maize / processed (not | 0,01 / 0,01 | 0,02 | | | | |
| Expand/collapse list | | | | | | | | | | | | | | | |

**Conclusion:**
No exceedance of the toxicological reference value was identified for any unprocessed commodity.
A short term intake of residues of mesotrione is unlikely to present a public health risk.
For processed commodities, no exceedance of the ARfD/ADI was identified.

## A 3.3.3 Nicosulfuron

Not relevant. ARfD was not deemed necessary.

# Appendix 4 Additional information provided by the applicant

No additional data provided.