REGISTRATION REPORT

Part B

Section 7

Metabolism and Residues

Detailed summary of the risk assessment

Product code: ARY-0469-04

Product name: ASAHI MAX

1.8%Chemical active substance(s):

Sodium 5-nitroguaiacolate, 3 g/L
Sodium o-nitrophenolate, 6 g/L

Sodium p-nitrophenolate, 9 g/L

Central Zone

Zonal Rapporteur Member State: POLAND

CORE ASSESSMENT

Applicant: Asahi Chemical Europe s.r.o

Submission date: June 2022, update February 2023

MS Finalisation date: March 2023 (initial Core Assessment)

June 2023 (final Core Assessment)

Version history

When	What
June 2022	Initial version of dRR for submission to zRMS
February 2023	Dossier updated on request of zRMS.
March 2023	Initial zRMS assessment. 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 2023	Final report (Core Assessment updated following the commenting period) No additional information or assessments after the commenting period.

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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 the preparation ARY-0469-04 are presented in Table 7.1-1. They have been selected from the individual GAPs in the Central zone for oilseed rape, winter wheat and sugar beet. A list of all intended uses within the Central zone is given in Part B, Section 0.

In Poland is registered a formulation Asahi SL, that is 3 times less concentrated. There were selected crops, which use is already registered for Asahi SL.

Overall conclusion
The data available are considered sufficient for risk assessment. An exceedance of the current MRL of 0.03 mg/kg for oilseed rape, winter wheat and sugar beet for Sodium 5-nitroguaiacolate, Sodium o-nitrophenolate and Sodium p-nitrophenolate as laid down in Reg. (EU) 396/2005 is not expected.
The chronic and the short-term intakes of Sodium 5-nitroguaiacolate, Sodium o-nitrophenolate and Sodium p-nitrophenolate residues are unlikely to present a public health concern.
As far as consumer health protection is concerned, zRMS 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:
- Available stability data are not cover the storage time for whole plant of cereals and oilseeds.

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

1	2	3	4	5	6	7		8				9			10	11	
GAP number (see part B.0)*	**Crop and/ or situation ****	**Zone**	**Product name**	**F, Fn, Fpn G, Gn, Gpn or I*****	**Pests or Group of pests controlled**	**Formulation**		**Application**				**Application rate per treatment**			**PHI (days)**	**Rate / ha**	**Conclusion**
						Type	**Conc. of as**	**method kind**	**growth stage & season**	**number min max**	**interval between appl. (min)**	**g as/hL min max**	**water L/ha min max**	**g as/ha min max**			
1	Winter oilseed rape, *mustard, spring rape, turnip rape, camelina, garden radish, poppy, linseed, hemp sunflower, borage.*	C	ASAHI MAX	F	Plant growth regulator, number of pods per plant, number of seeds per plant, higher lignification of pods	SL	Na 5NG: 3.0 Na oNP: 6.0 Na pNP: 9.0	Spray	BBCH 29-69 (spring)	2	7	Na 5NG: 0.12-0.3 Na oNP: 0.24-0.6 Na pNP: 0.36-0.9	200-500	Na 5NG: 0.6 Na oNP: 1.2 Na pNP: 1.8	28	0.2	A
2	Winter wheat, *spring rye, spelt, emmer wheat, small spelt, durum wheat.*	C	ASAHI MAX	F	Plant growth regulator, number of tillers and ears, portion above the sieves, germination energy	SL	Na 5NG: 3.0 Na oNP: 6.0 Na pNP: 9.0	Spray	BBCH 21-49 (spring)	1	-	Na 5NG: 0.2 Na oNP: 0.4 Na pNP: 0.6	200-300	Na 5NG: 0.6 Na oNP: 1.2 Na pNP: 1.8	28	0.2	A
3	Sugar beet *fodder beet, red beet, swede, turnip.*	C	ASAHI MAX	F	Plant growth regulator, effect on higher yield of sugar, lower content of unwanted Sodium	SL	Na 5NG: 3.0 Na oNP: 6.0 Na pNP: 9.0	Spray	BBCH 12-49 (spring-summer)	2	7	Na 5NG: 0.12-0.3 Na oNP: 0.24-0.6 Na pNP: 0.36-0.9	200-500	Na 5NG: 0.6 Na oNP: 1.2 Na pNP: 1.8	15	0.2	A

Crops marked ~~in yellow~~ in *italic* is registered as a minor crops on the base of art 51 (extrapolation from main crop).

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

** Use also code numbers according to Annex I of Regulation (EU) No 396/2005

*** 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

Explanation for Column 11 “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 ASAHI MAX is composed of Sodium 5-nitroguaiacolate, Sodium o-nitrophenolate and Sodium p-nitrophenolate.

Table 7.1-2: Toxicological reference values for the dietary risk assessment of Sodium 5-nitroguaiacolate, Sodium o-nitrophenolate and Sodium p-nitrophenolate

Reference value	Source	Year	Value	Study relied upon	Safety factor
Sodium 5-nitroguaiacolate, Sodium o-nitrophenolate and Sodium p-nitrophenolate					
ADI	EFSA	2009	0.003 mg.kg bw/day	1-year dog	100
ARfD	EFSA	2009	0.045 mg.kg bw	Developmental rabbit	300

7.1.2.1 Summary for sodium 5-nitroguaiacolate, sodium *o*-nitrophenolate and sodium *p*-nitrophenolate

Table 7.1-3: Summary for sodium 5-nitroguaiacolate, sodium *o*-nitrophenolate and sodium *p*-nitrophenolate

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	Oilseed rape	Yes	Yes (7 trials, all <0.01 mg/kg)	Yes	Yes for seed	Yes	No	No
2	Winter wheat	Yes	Yes (6 trials all <0.01 mg/kg)	Yes	Yes for grain and straw	Yes		No
3	Sugar beet	Yes	Yes (6 trials all <0.01 mg/kg)	Yes	Yes for root and leaves and tops	Yes		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 sodium nitrocompounds 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.

An acute risk has been identified for crop. The use of product code on crop is therefore not acceptable.

7.1.2.2 Summary for ARY-0469-04 / ASAHI MAX

Table 7.1-4: Information on ARY-0469-04 / ASAHI MAX (KCA 6.8)

Crop	PHI for product code proposed by applicant	PHI/ Withholding period* sufficiently supported for	PHI for product code proposed by zRMS	zRMS Comments (if different PHI proposed)
		Sodium 5-nitroguaiacolate, Sodium o-nitrophenolate and Sodium p-nitrophenolate		
oilseed rape	28 days	yes	28 days	-
winter wheat	28 days	yes	28 days	-
sugar beet	15 days	yes	15 days	-

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 7.1-5: Waiting periods before planting succeeding crops

Waiting period before planting succeeding crops		Overall waiting period proposed by zRMS for ASAHI MAX
Crop group	Sodium 5-nitroguaiacolate, Sodium o-nitrophenolate and Sodium p-nitrophenolate	
All crops	NR	NR

NR: not relevant

Necessary Waiting Periods or Other Precautions to Avoid Phytotoxic Effects on Succeeding Crops

Minimum waiting periods or other precautions between last application and sowing or planting succeeding crops: Not applicable
Limitations on choice of succeeding crops: Not applicable

Background

Sodium 5-nitroguaiacolate, sodium *o*-nitrophenolate and sodium *p*-nitrophenolate, which are the active substances of Atonik, were included in Annex I to Council Directive 91/414/EEC on 1 November 2009 by means of Commission Directive 2009/11/EC,[1] and have been deemed to be approved under Regulation (EC) No 1107/2009[2], in accordance with Commission Implementing Regulation (EU) No 540/2011[3] as amended by Commission Implementing Regulation (EU) No 541/2011[4]. The conclusions of the EFSA peer review of the pesticide risk assessment of the compounds of Atonik under Directive 91/414/EEC are available[5], based on representative uses on oilseed rape, tomato and sugar beet.

In compliance with Article 12(1) of the Regulation (EC) No 396/2005[6], EFSA initiated the collection of data for the sodium nitrocompounds and provided in 2015 a reasoned opinion on the review of the existing MRLs for sodium 5-nitroguaiacolate, sodium *o*-nitrophenolate and sodium *p*-nitrophenolate[7]. Based on the assessment of the available data, MRL proposals were derived and a consumer risk assessment was carried out. As there is no risk for consumers, the MRLs for those active substances were set at the existing level or the level identified by EFSA. Some information on analytical methods was identified as missing. The requirement for a validated analytical method for monitoring in high acid content commodities, dry commodities and hops (dried) was confirmed in the footnote to the MRL in the Annexes of the Regulation (EC) 2016/1785[8]. This information was submitted and evaluated by The Netherlands as Evaluating Member State (EMS). The data gap provided for the confirmatory data following the Article 12 MRL review was considered satisfactory addressed, the new information provided does not require a revision of the existing MRLs; the risk assessment performed for the three active substances sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate in the framework of the MRL review remains valid (EFSA, 2020a[9]).

[1] Commission Directive 2009/11/EC of 18 February 2009 amending Council Directive 91/414/EEC to include bensulfuron, sodium 5-nitroguaiacolate, sodium *o*-nitrophenolate, sodium *p*-nitrophenolate and tebufenpyrad as active substances. OJ L 48, 19.2.2009, p. 5-12.

[2] 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 and repealing Council Directives 79/117/EEC and 91/414/EEC. OJ L 309, 24.11.2009, p. 1-50.

[3] Commission Implementing Regulation (EU) No 540/2011 of 25 May 2011 implementing Regulation (EC) No 1107/2009 of the European Parliament and of the Council as regards the list of approved active substances. OJ L 153, 11.6.2011, p.1-186.

[4] Commission Implementing Regulation (EU) No 541/2011 of 1 June 2011 amending Implementing Regulation (EU) No 540/2011 implementing Regulation (EC) No 1107/2009 of the European Parliament and of the Council as regards the list of approved active substances. OJ L 153, 11.6.2011, p.187-188.

[5] EFSA Scientific Report (2008) 191, 1-130 Conclusion on the peer review of sodium 5-nitroguaiacolate, sodium *o*-nitrophenolate and sodium *p*-nitrophenolate

[6] Regulation (EC) No 396/2005 of the European Parliament and of the Council of 23 February 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin and amending Council Directive 91/414/EEC. OJ L 70, 16.3.2005, p. 1–16.

[7] EFSA (European Food Safety Authority), 2015. Reasoned opinion on the review of the existing maximum residue levels for sodium 5-nitroguaiacolate, sodium *o*-nitrophenolate and sodium *p*-nitrophenolate according to Article 12 of Regulation (EC) No 396/2005. EFSA Journal 2015;13(12):4356, 39 pp. doi:10.2903/j.efsa.2015.4356

[8] Commission regulation (EU) 2016/1785 of 7 October 2016 amending Annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for cymoxanil, phosphane and phosphide salts and sodium 5-nitroguaiacolate, sodium *o*-nitrophenolate and sodium *p*-nitrophenolate in or on certain products

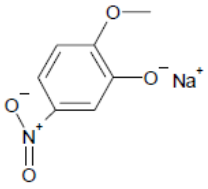
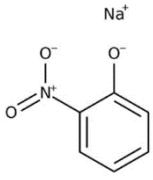
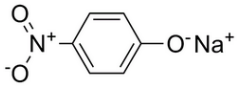
[9] EFSA (European Food Safety Authority), 2020. Reasoned opinion on the evaluation of confirmatory data following the Article 12 MRL review for sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate (sodium nitrocompounds). EFSA Journal 2020;18(4):6060, 14 pp.

Assessment

7.2 Sodium 5-nitroguaiacolate, Sodium o-nitrophenolate, Sodium p-nitrophenolate

General data on Sodium 5-nitroguaiacolate, Sodium o-nitrophenolate and Sodium p-nitrophenolate are summarized in the table below (last updated 2020/05/16)

Table 7.2-1: General information on Sodium 5-nitroguaiacolate, Sodium *o*-nitrophenolate and Sodium *p*-nitrophenolate

Approved	Date of (01/11/2009) and reference to decision (Commission Directive 2009/11/EC of 18 February 2009 amending Council Directive 91/414/EEC) - http://data.europa.eu/eli/dir/2009/11/oj	
Restriction	see Approval Directive / Regulation	
Review Report	SANCO/210/08 – rev. 2, 2 December 2008 SANCO/210/08 – rev. 2, 17 May 2013	
Current MRL regulation	~~Reg. (EU) 2016/1785~~- Reg. (EU) 2021/1098	
Peer review of MRLs according to Article 12 of Reg No 396/2005 EC performed	Yes https://www.efsa.europa.eu/en/efsajournal/pub/4356	
EFSA Journal : Conclusion on the peer review	Yes http://www.efsa.europa.eu/en/efsajournal/pub/rn-191	
EFSA Journal: conclusion on article 12	Yes https://doi.org/10.2903/j.efsa.2020.6060	
Current MRL applications on intended uses	-	
Chemical group	Sodium nitrocompounds	
Mode of action (if available)	Plant growth regulator	
Systemic	Yes	
Company	Asahi Chemical Europe s.r.o.	
Rapporteur Member State (RMS)	The Netherlands	
Active substance (ISO Common Name)		
Sodium 5-nitroguaiacolate (Na 5NG)	Sodium o-nitrophenolate (Na *o*-NP)	Sodium p-nitrophenolate (Na *p*-NP)
IUPAC		
Sodium 2-methoxy-5-nitrophenolate	Sodium 2-nitrophenolate	Sodium 4-nitrophenolate
Chemical structure		
		
Molecular formula		
$C_7H_5NNaO_4$	$C_6H_4NNaO_3$	$C_6H_4NNaO_3$
Molar mass		
191.12 g/mol	161.09 g/mol	161.09 g/mol

7.2.1 Stability of Residues (KCA 6.1)

7.2.1.1 Stability of residues during storage of samples

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

New residue study in nectar, including a storage stability testing has been submitted by the applicant in the framework of this application. Results are summarized in the Table below. The detailed assessment of this study is presented in Appendix 2.

Table 7.2-2: Summary of stability data achieved at ≤ - 18°C (unless stated otherwise)

Matrix	Characteristics of the matrix	Acceptable Maximum Storage duration	Reference	Comment
New data				
Plant products				
Nectar	high water content	7.7 months (232 days)	Kugel, D. 2020, S19-03993	Study submitted for AIR, not yet evaluated on EU level

Conclusion on stability of residues during storage
Sufficient data has been provided to cover the storage period of nectar samples for Na 5-NG, Na *o*-NP and Na *p*-NP in the framework of this application. Indeed, for Na 5-NG, Na *o*-NP and Na *p*-NP, storage stability was shown in nectar for a period of at least 7.7 months (232 days), respectively, at temperatures below -18°C.

7.2.1.2 Stability of residues in sample extracts (KCA 6.1)

No new data submitted in the framework of this application.

All final sample extracts were analysed within 24 hours after extraction. Therefore, a study is not necessary to demonstrate the sample extracts stability. However, storage stability of Na 5 NG, Na o-NP and Na p-NP in nectar extracts was tested within the residue study in nectar. A detailed de-scription regarding the investigated storage stability in extracts is presented in Appendix 2.

Procedural recoveries were performed on the day of analysis and spiked stored sample extracts were analysed after 7 days of storage at temperatures of 1 to 10°C.
All extracts analysed for Na 5-NG, Na o-NP and Na p-NP, stored for 7 days, showed recoveries within 70 – 110%.
Conclusion on stability of residues in sample extracts
Procedural recoveries were analysed concurrently with the stored extract samples showing recoveries between 70 and 110% and thus, are demonstrating the accuracy of the method on the day of analysis. Furthermore, the storage stability of Na 5 NG, Na o NP and Na p NP in nectar extracts stored at 1 to 10°C has been investigated showing stability of Na 5 NG, Na o NP and Na p NP for 7 days in nectar extracts.

zRMS comments:
Studies on the storage stability of sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate in crop under frozen conditions were not assessed in the framework of Directive 91/414/EEC.
In the framework of the MRL review, the RMS has reported four storage stability studies respectively performed on cucumbers, oilseed rape, maize grain and strawberries hereby covering the four main plant matrices (Greece, 2015b). According to these studies, the three sodium nitrocompounds are stable for a period of 9 months in high oil content and dry commodities. In high water content and high acid content commodities, sodium 5-nitroguaiacolate and sodium p-nitrophenolate were also demonstrated stable for a period of 9 months but a significant degradation of sodium o-nitrophenolate was observed after 3 months of storage.
Hence, it is concluded that the sodium nitrocompounds are stable for a period of 9 months in high oil content and dry commodities and for a period of 3 months in high water content and high acid content commodities.

According to the EFSA Journal 2015;13(12):4356:
Stability of residues in plants:

Plant products (available studies)	Category	Commodity	T (°C)	Stability (Months/years)
	High water content	Cucumbers	Not reported	≤3 month(a)
	High oil content	Oilseed rape	Not reported	9 months
	Dry	Maize grain	Not reported	9 months

	High acid content	Strawberries	Not reported	≤3 months(a)
	(a): critical storage period was observed for sodium o-nitrophenolate which showed significant degradation after 3 months while sodium 5-nitroguaiacolate and sodium p-nitrophenolate were stable for a longer period (9-10 months).			

New residue study in nectar, including a storage stability testing has been submitted by the Applicant in the framework of this application. Storage stability was shown in nectar for a period of at least 7.7 months at temperatures below 18°C. For more details, please refer to Appendix 2.

Sufficient stability data are available to support the residue data presented in the present dossier for:
- oilseed rape seed only (for whole plant – not);
- cereals grain and straw (for whole plant – not);
- sugar beet roots and leaves with tops.

Stability data are not cover the storage time for whole plant of cereals and oilseeds.

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)

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					Reference
			Method, F or G (a)	Rate (kg a.s./ha)	No	Sampling (DAT)	Remarks	
EU data								
Fruits and fruiting vegetable	Tomato	^{14}C Atonik	foliar treatment, F	~~6~~ 60 g as/ha	3	6		Greece, 2009 EFSA, 2015
Leafy vegetables								
Root and tuber vegetables	Sugar beet	^{14}C Atonik	foliar treatment, F	~~6~~ 60 g as/ha	3	90		Greece, 2009 EFSA, 2015
Pulses and oilseeds	Oilseed rape	^{14}C Atonik	foliar treatment, F	~~6~~ 60 g as/ha	2	60		Greece, 2009 EFSA, 2015
Cereals								

Summary of plant metabolism studies reported in the EU
On the basis of these metabolism studies, the residue definition for monitoring and risk assessment for all plant commodities was proposed as the sum of sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate, expressed as sodium 5-nitroguaiacolate in the conclusion of the peer review (EFSA, 2015). The current residue definition set in Regulation (EC) No 396/2005 is identical to the residue definition for enforcement derived in the peer review.

Conclusion on metabolism in primary crops
The proposed use on oilseed rape, winter wheat and sugar beet are covered by the previously evaluated metabolism studies as the metabolism in three diverse crop groups has been assessed and was shown to be similar. The residue definition of 'sum of sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate, expressed as sodium 5-nitroguaiacolate' (EFSA 2008/2015) is therefore considered applicable to oilseed rape, winter wheat and sugar beet. The residue definition for enforcement is also considered in Regulation (EC) No ~~2016/1785~~ Reg. (EU) 2021/1098.

zRMS comments:
Information given by the Applicant is acceptable and sufficient.

In EFSA Journal 2015;13(12):4356 it is stated that:
The metabolism of the three sodium nitrocompounds considered in this review (sodium 5-nitroguaiacolate, sodium o-nitrophenolate, sodium p-nitrophenolate) was investigated in fruit crops, root crops and pulses and oilseeds. Metabolism studies have been performed using foliar application of a mixture of the three active substances (in concentrations representative of the authorised formulations). The tested application rates were exaggerated by a factor of 10 compared to the authorised GAPs but the total radioactivity was very low in sugar beet roots, tomatoes and rape seeds (0.03-0.05 mg eq/kg, expressed as the sum of the three sodium nitrocompounds). Further characterisation demonstrated that the radioactivity was made of several constituents, none of them exceeding 0.013 mg eq/kg. Among these constituents, only 5-nitroguaiacol and p-nitrophenol were identified. Other significant metabolites were not identified. Therefore, at the authorised application rates, significant residues are not expected in edible parts of the investigated crops.
In sugar beet leaves however, the total radioactive residue (TRR) was significantly higher, accounting for 0.40 mg eq/kg. The two main important fractions, named metabolite 6 and 7 during the peer review (EFSA, 2008) and respectively accounting for 0.11 and 0.06 mg eq/kg, were not identified. Even considering the overdosing factor of the application rate, these compounds may still exceed 0.01 mg/kg in sugar beet leaves when active substances are applied in accordance with GAP. During the peer review, the experts already concluded that further information on the structure of these two unknown fractions should be requested. As a GAP on sugar beet (roots and tops) is authorised within Europe, this data requirement is still applicable. Meanwhile, the nature of residue in sugar beet leaves remains not elucidated.

The residue definitions:
As similar results were observed in three different crop groups (fruits, roots and pulses/oilseeds), a general residue definition can be proposed for food commodities of plant origin.
EFSA proposed a common residue definition for monitoring and risk assessment being **the sum of sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate, expressed as sodium 5-nitroguaicolate**. Due to uncertainties observed in sugar beet leaves where two significant metabolites remain unidentified, the proposed residue definition is tentative for sugar beet leaves.

No further data are required.

7.2.2.2 Nature of residue in rotational crops (KCA 6.6.1)

Consideration of the nature and magnitude of residues in rotational crops was included in previous evaluations:
EFSA, 2008
No rotational crop studies have been provided, the highest DT_{50} being 2.2 days for Na p-NP.

EFSA, 2015
Several crops under consideration may be grown in rotation. According to the soil degradation studies which were evaluated in the framework of the peer review for the three sodium nitrocompounds, the maximum DT_{90} value is 7.5 days (sodium p-nitrophenolate), which is far below the trigger value of 100 days (EFSA, 2008). According to the European guidelines on rotational crops (European Commission, 1997b), further investigation of residues in rotational crops is not required and relevant residues in rotational crops are not expected.
No studies are therefore considered required on rotational crops.

zRMS comments:
Information given by the Applicant is acceptable and sufficient.
As DT_{90} values of sodium nitrocompounds are all expected to be well below the trigger value of 100 days, investigation of residues in rotational crops was not required.
No further data are required.

7.2.2.3 Nature of residues in processed commodities (KCA 6.5.1)

The level of residue in oilseed rape, winter wheat and sugar beet is below the trigger of 0.03 mg/kg, and contribution of the commodity under consideration to the theoretical maximum daily intake (TMDI) is <

10% of the ADI (refer to Appendix 3) and therefore studies on the magnitude of residues in processed commodities are not required.

zRMS comments:
Information given by the Applicant is acceptable and sufficient.
The effect on the nature of sodium nitrocompounds has not been investigated in the framework of the EU pesticides peer review.
As residues of sodium nitrocompounds exceeding 0.1 mg/kg are not expected in the treated crops, there is no need to investigate the effect of industrial and/or household processing.
No further data are required.

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

Table 7.2-4: Summary of the nature of residues in commodities of plant origin

Endpoints	
Plant groups covered	Fruits and fruiting vegetables (tomato) Root and tuber vegetables (sugar beet) Pulses and oilseeds (oilseed rape)
Rotational crops covered	Not required - the maximum DT_{90} value is 7.5 days
Metabolism in rotational crops similar to metabolism in primary crops?	-
Processed commodities	Not required
Residue pattern in processed commodities similar to pattern in raw commodities?	-
Plant residue definition for monitoring	Sum of sodium 5-nitroguaiacolate + sodium *o*-nitrophenolate + sodium *p*-nitrophenolate (Reg. (EU) 2021/1098) Proposed by EFSA (2009) after the meeting, peer reviewed for high water and high oil, not peer reviewed for high acid, dry commodities and hops
Plant residue definition for risk assessment	Sum of sodium 5-nitroguaiacolate + sodium *o*-nitrophenolate + sodium *p*-nitrophenolate Proposed by EFSA (2009) after the meeting, peer reviewed for high water and high oil, not peer reviewed for high acid, dry commodities and hops
Conversion factor from enforcement to RA	Not required

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

No data on the nature of residues in livestock are required. The following conclusions have been reached in previous evaluations:
EFSA, 2009
Since significant residues are not expected to result from the intended uses of the active substances in livestock feed, no metabolism or feeding studies were provided and no MRLs were proposed for products of animal origin.

EFSA, 2015
Although the trigger value is exceeded in ruminants and pigs, a significant intake is not expected because calculations are overly conservative in this case (use of the combined LOQ of 0.03 mg/kg while no residues are expected in root crops, apples, cereals and oilseeds). Consequently, EFSA concluded that MRLs for nitrocompounds in animal commodities were not required. However, it is highlighted that this conclusion does not take into consideration the possible intake of sugar beet leaves for which uncertainties on the nature and magnitude of residues were identified. Therefore, Member States will need to pay attention to this issue when authorising a good agricultural practice (GAP) on sugar beet.*

zRMS comments:
Information given by the Applicant is acceptable and sufficient.
No further data are required.

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

Updated enforcement analytical methods for the determination of sodium 5-nitroguaiacolate, sodium *o*-nitrophenolate and sodium *p*-nitrophenolate applied to dry crops, high acid, high water, high oil and difficult matrices as the Article 12 confirmatory data had been submitted, the data gap provided for the confirmatory data following the Article 12 MRL review was considered satisfactory addressed, the new information provided does not require a revision of the existing MRLs; the risk assessment performed for the three active substances sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate in the framework of the MRL review remains valid (EFSA, 2020a). Other residue trials, related risk assessment input values and MRL proposals were assessed in the framework of the review of existing MRLs for sodium nitrocompounds (EFSA, 2015, EFSA 2020b).

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 7.2.3-1 below. The detailed assessment of these studies is presented in Appendix 2.

Oilseed rape in North Zone
2 at-harvest trials were conducted in 2005 in Germany, 1 at-harvest trial was conducted in 2008 in Poland, 2 at-harvest trials and 1 decline trial were conducted in 2018 in Poland and Germany and 1 decline trials was conducted in 2019 in United Kingdom. Two applications of Atonik (1,0 g/L Na-5NG, 2,0 g/L Na-oNP and 3,0 g/L Na-pNP) were applied at 1,0 L product/ha (a total of 6 g a.i./ha), diluted with water immediately prior to application to a spray volume 200-300 L/ha. Specimens of crop from the untreated and treated plots were sampled 27 respective 37 days in 2005, 30 days in 2008, 28 days in 2018 and 2019 after the final application at normal commercial harvest
In trials conducted in 2006 the specimens were analysed within 63 days of sampling, in trial conducted in 2008 the specimens were analysed within 233 days of sampling, and in trials conducted in 2018 the specimens were analysed within 107 (seed) respective 244 (whole plant) days of sampling, in trial conducted in 2019 the specimens were analysed within 120 (seed) respective 151 (whole plant) days of sampling. The application rate used in the trials is corresponding to the proposed intended use (1,0 L/ha of product). The data from all 7 trials are considered acceptable as all residues were <LOQ (0.01 mg/kg).

zRMS comments:
Oilseed rape
Oilseed rape **is** the major crop in northern Europe (Technical Guidelines SANTE/2019/12752). A minimum of eight independent trials representative of the proposed growing area for outdoor are required.

The Applicant provided four new studies covered a total of 7 field trials in Northern Europe conducted on oilseed rape according to the intended GAP uses of sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate in the product ARY-0469-04 / Asahi Max following two foliar applications of Atonic.
The trials were performed within the GAP: 2×0.6 g / ha Na-5NG, 1.2 g/ ha Na-oNP 1.8 g/ ha Na-pNP, interval of applications of 7-30 days, PHI of 27-37 days) and therefore can be used to support the registration of ARY-0469-04 / Asahi Max.
The residues of Na 5-NG, Na o-NP and Na p-NP in oilseed rape seeds at harvest were <0.01 mg/kg.

According to the SANTE/2019/12752 if the residue levels in plants or plant products are lower than the limit of quantification (LOQ), the number of independent trials may be reduced. The number of trials shall not be below the minimum of four per zone for major crops. So there are sufficient residue trials to support the intended use of Na 5-NG, Na o-NP and Na p-NP on oilseed rape.

According to the SANTE/2019/12752 the residue trials on any representatives of the group Oilseeds, except peanuts/groundnuts (0401020) may be extrapolated to Whole group Oilseeds (0401000), except peanuts/groundnuts (0401020) when application is done before or after the forming of the edible part in oilseeds.

Available results show that the in force MRL on Oilseeds (0401000) of 0.03* mg/kg (Reg. (EU) 2021/1098) will not be exceeded.
Therefore, sufficient residue trials are available to support the intended GAP uses on oilseeds (winter oilseed rape, mustard, spring rape, turnip rape, camelina, garden radish, poppy, linseed, hemp sunflower, borage).

Maximum storage period was 4 months for seeds and 9 months for whole plant.
According to the OECD 506 oilseed rape seed belongs to high oil commodity and whole plant belongs to high water commodity.
The sodium nitrocompounds are stable for a period of 9 months in high oil content and for a period of 3 months in high water content commodities. So stability data are not cover the storage time for whole plant.

No further data are required to support the proposed uses on oilseeds seeds.

Winter wheat in North Zone

2 at-harvest trials (spring wheat) were conducted in 2005 in Germany, 1 at-harvest (Poland) and 2 decline trials (German, United Kingdom) were conducted in 2018 and 1 at-harvest trial was conducted in 2019 in United Kingdom.
Two applications of Atonik (1,0 g/L Na-5NG, 2,0 g/L Na-oNP and 3,0 g/L Na-pNP) were applied at 0,6 L product/ha (a total of 6 g a.i./ha), diluted with water immediately prior to application to a spray volume 300 L/ha. In the 2005 trials the specimens of crop from the untreated and treated plots were sampled at 55-68 days after the final application at normal commercial harvest, in 2018 and 2019 trials the specimens of crop from the untreated and treated plots were sampled at 28 days after the final application at normal commercial harvest.
In trials conducted in 2005 the specimens were analysed within 132-145 days of sampling (both grain and straw), in trials conducted in 2018 the specimens were analysed within 267 days (whole plant) respective 241 (straw) and 122 days (grain) of sampling, in trial conducted in 2019 the specimens were analysed within 44 (seed) respective 45 (straw) days of sampling. The application rate used in the trials is corresponding to the proposed intended use (0,6 L/ha of product).
In the 2005 trials the LOQ in grain was set to 0,01 mg/kg, for straw was set to 0,02 mg/kg. All residues were <LOQ (0.01 mg/kg), therefore the trials are considered acceptable.
In the 2018 and 19 trials the LOQ for Na-5NG, Na-oNP and Na-pNP in grain was set to 0,01 mg/kg, in whole plant and straw the LOQ for Na-5NG, Na-oNP to 0,01 mg/kg and for Na-pNP to 0,1 mg/kg. No residues above 30% of the LOQ were detected in the control (untreated) residue samples, except in straw sample S18-05052-02-007A, where residues of p-nitrophenol (expressed as sodium salt) were found at the LOD of 0.03 mg/kg. No residues of Na-5NG, Na-oNP and Na-pNP above the set LOQ were detected in gran, whole plant or straw of treated crops.
The data from all 6 trials are considered acceptable as all residues were <LOQ (0.01 mg/kg) for Na-5NG, Na-oNP and Na-pNP in grain, simultaneously residues were < LOQ 0,01 mg/kg for Na-5NG, Na-oNP and < LOQ 0,1 mg/kg for Na-pNP in whole plant and straw.

zRMS comments:
Cereals
Wheat is the major crop in northern Europe (Technical Guidelines SANTE/2019/12752). A minimum of eight independent trials representative of the proposed growing area for outdoor are required.

The Applicant provided three new studies covered a total of 6 field trials in Northern Europe conducted on wheat and barley according to the intended GAP uses of sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate in the product ARY-0469-04 / Asahi Max following two foliar applications of Atonic.
The trials were performed within the GAP: 2×0.6 g / ha Na-5NG, 1.2 g/ ha Na-oNP 1.8 g/ ha Na-pNP, interval of applications of 10-28 days, PHI of 27-37 days) and therefore can be used to support the registration of ARY-0469-04 / Asahi Max. Samples of wheat were taken 0, 6-8, 14-15, 21-22 and 28 (NCH) days after the final application.

The residues of Na 5-NG, Na o-NP and Na p-NP in wheat and barley grain at harvest were <0.01 mg/kg.

According to the SANTE/2019/12752 if the residue levels in plants or plant products are lower than the limit of quantification (LOQ), the number of independent trials may be reduced. The number of trials shall not be below the minimum of four per zone for major crops. So there are sufficient residue trials to support the intended use of Na 5-NG, Na o-NP and Na p-NP on cereals.

According to the SANTE/2019/12752 the residue trials on wheat may be extrapolated to rye, when application is done before or after the forming of the edible part in cereals.

Available results show that the in force MRL on cereals of 0.03* mg/kg (Reg. (EU) 2021/1098) will not be exceeded.
Therefore, sufficient residue trials are available to support the intended GAP uses on cereals (winter wheat, spring rye, spelt, emmer wheat, small spelt, durum wheat).

Maximum storage period was 5 months for grain, 8 months in straw and ~9 months for whole plant.
According to the OECD 506 wheat grain and straw belongs to dry commodity and whole plant belongs to high water commodity.
The sodium nitrocompounds are stable for a period of 9 months in dry matrices and for a period of 3 months in high water content commodities. So stability data are not cover the storage time for whole plant.

No further data are required to support the proposed uses on grain and straw of cereals.

Sugar beet in North Zone

2 at-harvest trials were conducted in 2005 in Germany, 1 at-harvest trial was conducted in 2013 in France and 2 decline trials and 1 at-harvest trial were conducted in 2019 in Hungary and Germany and France.
Four applications of Atonik (1,0 g/L Na-5NG, 2,0 g/L Na-oNP and 3,0 g/L Na-pNP) were applied at 1,0 L product/ha (a total of 6 g a.i./ha), diluted with water immediately prior to application to a spray volume 300- L/ha in 2005 and 2013. Three applications of Atonik (1,0 g/L Na-5NG, 2,0 g/L Na-oNP and 3,0 g/L Na-pNP) were applied at 1,0 L product/ha (a total of 6 g a.i./ha), diluted with water immediately prior to application to a spray volume 200-300 L/ha in 2019. Specimens of crop from the untreated and treated plots were sampled 13-15 days after the final application at normal commercial harvest.
In trials conducted in 2005 the specimens were analysed within 55 respective 76 days of sampling, in trials conducted in 2013 and 2019 the specimens were analysed within 30 days of sampling. The application rate used in the trials is higher than the proposed intended use (0,6 L/ha of product).
The data from all 6 trials are considered acceptable as all residues were <LOQ (0.01 mg/kg).

zRMS comments:
Sugar beet
Sugar beet is the major crop in northern Europe (Technical Guidelines SANTE/2019/12752). A minimum of eight independent trials representative of the proposed growing area for outdoor are required.

The Applicant provided three new studies covered a total of 6 field trials in Northern Europe conducted on sugar beet. The trials were performed within the GAP: 3 or 4 ×1 g/ha Na-5NG, 2 g/ha Na-oNP, 3 g/ha Na-pNP, interval of applications of 6-8 days, PHI of 15 days. This GAP is more critical than intended GAP for ARY-0469-04 / Asahi Max. In our opinion it can be used to support the registration of ARY-0469-04 / Asahi Max. Samples (roots, leaves with tops) were taken at harvest, at PHI 15 days.

The residues of Na 5-NG, Na o-NP and Na p-NP in in roots and leaves with tops at harvest were <0.01 mg/kg.

According to the SANTE/2019/12752 if the residue levels in plants or plant products are lower than the limit of quantification (LOQ), the number of independent trials may be reduced. The number of trials shall not be below the minimum of four per zone for major crops. So there are sufficient residue trials to support the intended use of Na 5-NG, Na o-NP and Na p-NP on sugar beet.

According to the SANTE/2019/12752 the residue trials on sugar beet may be extrapolated to beetroots (0213010), celeriacs/turnip rooted celeries (0213030), horseradishes (0213040), swedes/rutabagas (0213100), turnips (0213110) and chicory roots (0900030) when application is done before or after the forming of the edible part in sugar beet.

Available results show that the in force MRL on sugar beet and other root and tuber vegetables except sugar beets of 0.03* mg/kg (Reg. (EU) 2021/1098) will not be exceeded.
Therefore, sufficient residue trials are available to support the intended GAP uses sugar beet, red beet, swede, turnip and fodder beet.

The stability data are cover the storage time for sugar beet roots and tops.
No further data are required to support the proposed uses.

Table 7.2.3-1: Summary of EU reported and new data supporting the intended uses of ASAHI MAX and conformity to existing MRL

Commodity (trial GAP)	**Source**	**Region / Indoor** (a)	**E = according to enforcement residue definition** **RA = according to risk assessment residue definition**	**STMR (mg/kg)**	**HR (mg/kg)**	**Current EU MRL (mg/kg) ***	**MRL compliance**
Residue definition for enforcement and risk assessment: **sodium 5-nitroguaiacolate, sodium *ortho*-nitrophenolate and sodium *para*-nitrophenolate**							
Oilseed rape (GAP: 2 x 6 g as/ha, PHI 28d, outdoor)	EFSA, 2015	3x N-EU	E: 5-NG: 3 x <0.01 *o*-NP: 3 x <0.01 *p*-NP: 3 x <0.01 Sum: 3 x <0.03	NEU: 0.03	NEU: 0.03	0.03*	Yes
Oilseed rape (GAP: 2 x 6 g as/ha, PHI 28d, outdoor)	New trial	4x N-EU	E: 5-NG: 4 x <0.01 *o*-NP: 4 x <0.01 *p*-NP: 4 x <0.01 Sum: 4 x <0.03	NEU: 0.03	NEU: 0.03	0.03*	Yes
Winter wheat (GAP: 2 x 6 g as/ha, PHI 55d, outdoor)	EFSA, 2015	2x N-EU	E: 5-NG: 2 x <0.01 *o*-NP: 2 x <0.01 *p*-NP: 2 x <0.01 Sum: 2 x <0.03	NEU: 0.03	NEU: 0.03	0.03*	Yes
Winter wheat (GAP: 2 x 3,6 g as/ha, PHI 28d, outdoor)	New trials	4x N-EU	E: 5-NG: 4 x <0.01 *o*-NP: 4 x <0.01 *p*-NP: 3 x <0.01 Sum: 4 x <0.03	NEU: 0.03	NEU: 0.03	0.03*	Yes
Sugar beet (GAP: 4 x 6 g as/ha, PHI 15d, outdoor)	EFSA, 2015	2x N-EU	E: 5-NG: 3 x <0.01 *o*-NP: 3 x <0.01 *p*-NP: 3 x <0.01 Sum: 3 x <0.03	NEU: 0.03	NEU: 0.03	0.03*	Yes
Sugar beet (GAP: 4 x 6 g as/ha, PHI 15d, outdoor)	New trials	4x N-EU	E: 5-NG: 4 x <0.01 *o*-NP: 4 x <0.01 *p*-NP: 4 x <0.01 Sum: 4 x <0.03	NEU: 0.03	NEU: 0.03	0.03*	Yes

* Source of EU MRL: Reg. (EU) 2021/1098

(a): **NEU** or **SEU** for **outdoor** trials in northern or southern Europe (**N+SEU** if both zones), **Indoor** for glasshouse/protected trials, **Country** or **Country/indoor** if non-EU location.

Note: '*' denotes MRL value is equal to the analytical Limit of Quantification (LOQ).

7.2.4 Magnitude of residues in livestock

7.2.4.1 Dietary burden calculation

According to Commission Regulation (EU) No. 283/2013 animal metabolism studies are required, when a pesticide is to be used in crops whose parts or products are fed to animals and where the intake is expected to exceed 0.004 mg/kg bw/d.
The potential total feed residues of the sum of Na 5-NG, Na *o*-NP and Na *p*-NP, expressed as Na 5-NG were calculated for cattle, sheep and swine and poultry based on the residue levels determined in residue trials taking into account the composition of the feed for the different livestock diets.
The dietary burden was calculated according to the feedstuff tables listed in the OECD Guidance ENV/JM/MONO(2009)31 and detailed in the OECD guidance ENV/JM/MONO(2013)8. The animal dietary burden was calculated using the EFSA Excel calculator (Animal model 2017). In accordance to the most recent evaluation of relevant data for Article 12 MRL review (EFSA Journal 2015;13(12):4356), conversion factors were not considered for summation of the LOQ for all three compounds. The combined LOQ of 0.03 mg/kg for Na 5-NG, Na *o*-NP and Na *p*-NP is taken into account for sugar beet, winter wheat and oilseed rape related matrices.
As the residue levels for all sodium nitrocomponents are below the LOQ (< 0.01 mg/kg) the default processing factors (PF) for the by-products are replaced with 1 in accordance to the Animal model 2017.
The input data for the mean and maximum animal diet burden calculation is presented in Table 7.2.4-1, animal dietary burden overview is presented in Table 7.2.4-3, and results of calculated dietary burden is presented in Table 7.2.4-3.

Table 7.2.4-1: Input values for the dietary burden calculation

	Median dietary burden		**Maximum dietary burden**	
	Input values [mg/kg]	**Comment**	**Input values [mg/kg]**	**Comment**
Risk assessment residue definition: sum of Sodium 5-Nitroguaiacolate, Sodium *o*-Nitrophenolate and Sodium *p*-Nitrophenolate, expressed as Sodium 5-Nitroguaiacolate				
Forages				
Beet, sugar -tops	0.03*	STMR	0.03*	HR
Cereal grains / crops seeds				
Wheat grain	0.03*	STMR	0.03*	HR
By-products				
Beet, sugar – dried pulp	0.03*	STMR (0.03) x PF (1)	0.03*	STMR (0.03) x PF (1)
Beet, sugar – ensiled pulp	0.03*	STMR (0.03) x PF (1)	0.03*	STMR (0.03) x PF (1)
Beet, sugar – molasses	0.03*	STMR (0.03) x PF (1)	0.03*	STMR (0.03) x PF (1)
Rape seed - meal	0.03*	STMR (0.03) x PF (1)	0.03*	STMR (0.03) x PF (1)
Rape meal	0.03*	STMR (0.03) x PF (1)	0.03*	STMR (0.03) x PF (1)
Brewer's grain	0.03*	STMR (0.03) x PF (1)	0.03*	STMR (0.03) x PF (1)
Distillers grain	0.03*	STMR (0.03) x PF (1)	0.03*	STMR (0.03) x PF (1)
Wheat gluten	0.03*	STMR (0.03) x PF (1)	0.03*	STMR (0.03) x PF (1)
Wheat milled by-pds.	0.03*	STMR (0.03) x PF (1)	0.03*	STMR (0.03) x PF (1)

* Indicates that the input value is proposed at the limit of quantification.
Default processing factors (PF) are set to 1 as residues in RAC samples are <LOQ.

Table 7.2.4-2: Overview of the dietary burden calculation

Animal burden calculation							Asahi Max					
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)	0,002	mg/kg bw/d	%	0,005	mg/kg bw/d	%	0,002	mg/kg bw/d	%	0,003	mg/kg bw/d	%
Contributor 1	Beet, sugar	ensiled pulp	25	Beet, sugar	ensiled pulp	40	Beet, sugar	tops	20	Beet, sugar	tops	20
Contributor 2	Beet, sugar	tops	20	Beet, sugar	tops	30	Wheat gluten	meal	30	Wheat gluten	meal	30
Contributor 3	Wheat	grain	40	Wheat	grain	30	Wheat	grain	40	Wheat	grain	50
Contributor 4												
Median intake	0,0021	mg/kg bw/d		0,0050	mg/kg bw/d		0,0021	mg/kg bw/d		0,0028	mg/kg bw/d	

Maximum Intake	Swine					
	Breeding	260 kg 6 kg		Finishing	100 kg 3 kg	
(mg/kg bw/d)	0,001	mg/kg bw/d	%	0,001	mg/kg bw/d	%
Contributor 1	Beet, sugar	tops	10	Wheat	milled bypdt	50
Contributor 2	Wheat	milled bypdt	50	Wheat	grain	50
Contributor 3	Wheat	grain	40			
Contributor 4						
Median intake	0,001	mg/kg bw/d		0,001	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)	0,002	mg/kg bw/d	%	0,003	mg/kg bw/d	%	0,002	mg/kg bw/d	%
Contributor 1	Wheat gluten	meal	10	Beet, sugar	tops	5	Wheat gluten	meal	10
Contributor 2	Wheat	grain	70	Wheat gluten	meal	10	Wheat	grain	50
Contributor 3				Wheat	grain	70			
Contributor 4									
Median intake	0,002	mg/kg bw		0,003	mg/kg bw		0,002	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	0,09	**0,13**	0,1	0,07	0,04	0,03
Median	0,09	0,13	0,06	0,07	0,04	0,03

	Poultry		
	Broiler	Layer	Turkey
Maximum	0,03	0,04	0,02
Median	0,03	0,04	0,02

Intake >0.1 mg/kg DM in red characters

Table 7.2.4-3: 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 mg/kg bw
	Median	Maximum	Median	Maximum				
Cattle (all diets)	0.005	0.005	0.13	0.13	Dairy cattle	Beet, sugar	ensiled pulp	Yes
Cattle (dairy only)	0.005	0.005	0.13	0.13	Dairy cattle	Beet, sugar	ensiled pulp	Yes
Sheep (all diets)	0.003	0.003	0.07	0.07	Lamb	Beet, sugar	tops	No
Sheep (ewe only)	0.002	0.002	0.06	0.06	Ram/Ewe	Beet, sugar	tops	No
Swine (all diets)	0.001	0.001	0.04	0.04	Swine (finishing)	Wheat	milled bypdts	No
Poultry (all diets)	0.003	0.003	0.04	0.04	Poultry layer	Beet, sugar	tops	No
Poultry (layer only)	0.003	0.003	0.04	0.04	Poultry layer	Beet, sugar	tops	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 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 dietary burden for cattle is slightly above the trigger value and driven by sugar beet ensiled pulp, sugar beet tops and wheat grain for which the input values were the combined LOQ of 0.03 mg/kg. As a no residue situation for sugar beet and wheat grain can be expected based on the 10X overdosed metabolism studies as well as the residue levels are < LOQ for Na 5-NG, Na *o*-NP and Na *p*-NP in the residue trials, the input value of 0.03 mg/kg is expected to be overly conservative.

As already concluded in the 91/414/EEC review and within the EFSA Article 12 MRL review (EFSA Journal 2015;13(12):4356) confirmed, metabolism studies in animal livestock are not required. Furthermore, a residue definition in animal products is not needed and the setting of MRLs in commodities of animal origin is not necessary.

zRMS comments:
The dietary burdens calculated for cattle were found to exceed the trigger value of 0.1 mg/kg DM. Therefore, behaviour of residues should normally be assessed in this group of livestock.
However, the calculated dietary burdens were close to the trigger value of 0.1 mg/kg DM and may have been overestimated because of the use of the combined LOQ of 0.03* mg/kg as an input value for crops where a no-residue situation was demonstrated. Consequently, EFSA concluded that MRLs for nitrocompounds in animal commodities were not required.
However, it is highlighted that this conclusion does not take into consideration the possible intake of sugar beet leaves for which uncertainties on the nature and magnitude of residues were identified. Therefore, Member States will need to pay attention to this issue when authorising a GAP on sugar beet.

No further data are required.

7.2.4.2 Livestock feeding studies (KCA 6.4.1-6.4.3)

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

7.2.4.3 Poultry

Based on the animal diet burden calculation presented above (please refer to KCA 7.2.4.1) it is evident, that the maximum diet burden expected for poultry is below the trigger value of 0.004 mg/kg bw/d for a livestock metabolism study (see Table 7.2.4-2 and 7.2.4-3). Thus, metabolism studies in poultry are not needed.

zRMS comments:
Information given by the Applicant is acceptable and sufficient.
No further data are required.

7.2.4.4 Lactating ruminants (goat or cow)

Although the trigger value of 0.004 mg/kg bw/d is exceeded for cattle, a significant intake is not expected because calculations are overly conservative as they are based on the combined LOQ for Na 5-NG, Na o-NP and Na p-NP (please refer to KCA 7.2.4.1). Thus, metabolism studies in ruminants are not needed.

zRMS comments:
Information given by the Applicant is acceptable and sufficient.
Please refer to point 7.2.4.1.
No further data are required.

7.2.4.5 Pigs

Based on the animal diet burden calculation presented above (please refer to KCA 7.2.4.1) it is evident, that the maximum diet burden expected for pigs is below the trigger value of 0.004 mg/kg bw/d for a livestock metabolism study (see Table 7.2.4-2 and 7.2.4-3). Thus, metabolism studies in pigs are not needed.

zRMS comments:

Information given by the Applicant is acceptable and sufficient.
No further data are required.

7.2.4.6 Fish

As given in the fish working document (SANCO/11187/2013, rev. 3), the accumulation of compounds of relatively low lipophilicity (log Pow < 3) via the diet is known to be negligible and thus, fish metabolism studies are only required for active substances where the log Pow is greater than or equal to three.
As the log Pow is < 3 for all three active components Na 5-NG, Na o-NP and Na p-NP, fish metabolism studies are not needed.

zRMS comments:
Information given by the Applicant is acceptable and sufficient.
No further data are required.

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

No study required.

7.2.5.1 Available data for all crops under consideration

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

7.2.5.2 Conclusion on processing studies

No study required.

zRMS comments:
As residues of sodium nitrocompounds exceeding 0.1 mg/kg are not expected in the treated crops, there is no need to conduct processing studies.
No further data are required.

7.2.6 Magnitude of residues in representative succeeding crops

Not studies required, as the maximum DT_{90} value is 7.5 days. Please refer to 7.2.2.2.

7.2.6.1 Field rotational crop studies (KCA 6.6.2)

No new data submitted in the framework of this application.

zRMS comments:
Information given by the Applicant is acceptable and sufficient.
As DT_{90} values of sodium nitrocompounds are all expected to be well below the trigger value of 100 days, investigation of residues in rotational crops was not required.
No further data are required.

7.2.7 Other / special studies (KCA6.10, 6.10.1)

~~No new data submitted in the framework of this application.~~ New residue study in nectar has been submitted by the applicant in the framework of this application.

Effect on the residue level in pollen and bee products
Oilseed rape is classified as melliferous crops, according to the technical guideline SANTE/11956/2016 rev. 9. As for oilseed rape an application during flowering cannot be excluded, a guideline compliant study

investigating the magnitude of residues of Na 5-NG, Na *o*-NP and Na *p*-NP in nectar is summarised below. The detailed assessment of this study is presented in Appendix 2.

Table 7.2.7-1 Residues of Sodium 5-Nitroguaiacolate (Na 5-NG) in nectar

Matrix	Residue levels [mg/kg]
Nectar	< 0.01, 0.0107, 0.0141, 0.0175

Limit of quantification (LOQ) sodium 5-nitroguaiacolate: 0.01 mg/kg in nectar

Table 7.2.7-2 Residues of Sodium o-Nitrophenolate (Na *o*-NP) in nectar

Matrix	Residue levels [mg/kg]
Nectar	< 0.01 (4)

Limit of quantification (LOQ) sodium *o*-nitrophenolate: 0.01 mg/kg in nectar

Table 7.2.7-3 Residues of Sodium p-Nitrophenolate (Na *p*-NP) in nectar

Matrix	Residue levels [mg/kg]
Nectar	0.0603, 0.0700, 0.0720, 0.110

Limit of quantification (LOQ) sodium *p*-nitrophenolate: 0.01 mg/kg in nectar

Residues in untreated control samples were below the limit of quantification (LOQ = 0.01 mg/kg).

This new residue study in nectar, including a storage stability testing, supports the intended uses of ASAHI MAX and the conformity to existing MRL on honey. A transfer factor of 1 from aerial parts (nectar) to honey is considered for the assessment:

Table 7.2.7-4: Summary of new data supporting the intended uses of ARY-0469-01 and conformity to existing MRL on honey

Commodity	Zone	Evaluation GAP Residue levels (mg/kg)	STMR[(a)] [mg/kg]	HR[(a)] [mg/kg]	Calculated MRL[(a)] [mg/kg]
Nectar → extrapolated to Honey	NEU/SEU	Trials GAP: 4 x (Na 5-NG: 1.0 g as/ha; Na o-NP; 2.0 g as/ha; Na p-NP: 3.0 g as/ha), BBCH 64-67, PHI 0d NEU: 0.092; 0.138 SEU: 0.081; 0.0941	0.093[#]	0.14	0.14[##]

[(a)] STMR and HR for Sodium 5-Nitroguaiacolate, Sodium *o*-Nitrophenolate and Sodium *p*-Nitrophenolate based on plant residue definition for risk assessment: sum of Sodium 5-Nitroguaiacolate, Sodium *o*-Nitrophenolate and Sodium *p*-Nitrophenolate, expressed as Sodium 5-Nitroguaiacolate. In accordance to the Art 12 evaluation (EFSA Journal 2015;13(12):4356), no conversion factor was used.

[#]The calculation of the STMR is presented on Appendix 1.

[##]According to SANTE/11956/2016 rev. 9, calculated MRL is based on the highest residue level (HR) in nectar and the hypothesis of a transfer factor of 1 from aerial parts (nectar) to honey. The calculation of the HR is based on plant residue definition for risk assessment: sum of Sodium 5-Nitroguaiacolate, Sodium *o*-Nitrophenolate and Sodium *p*-Nitrophenolate, expressed as Sodium 5-Nitroguaiacolate. For the calculation of Sodium *o*-Nitrophenolate and Sodium *p*-Nitrophenolate to Sodium 5-Nitroguaiacolate, no molecular weight factor was used as usual in EU assessments (EFSA Scientific Report (2008) 191, 1-130 and EFSA Journal 2015;13(12):4356).

CONCLUSION

Residues of Na 5-NG in nectar are between <0.01 mg/kg (LOQ) and 0.0175 mg/kg, when ATONIK is applied four times at a nominal rate of 1.0 L product/ha (nominal rate of components: Na 5-NG: 1.0 g as/ha; Na *o*-NP; 2.0 g as/ha Na *p*-NP: 3.0 g as/ha).

Residues of Na *o*-NP in nectar are below the limit of quantification (LOQ = 0.01 mg/kg), when ATONIK is applied four times at a nominal rate of 1.0 L product/ha (nominal rate of components: Na 5-NG: 1.0 g as/ha; Na *o*-NP: 2.0 g as/ha; Na *p*-NP: 3.0 g as/ha).

Residues of Na *p*-NP in nectar are between 0.060 mg/kg and 0.11 mg/kg, when ATONIK is applied four times at a nominal rate of 1.0 L product/ha (nominal rate of components: Na 5-NG: 1.0 g as /ha Na *o*-NP: 2.0 g as/ha Na *p*-NP: 3.0 g as/ha).

For honey, the calculated MRL based on these residues data for the sum of Sodium 5-Nitroguaiacolate, Sodium o-Nitrophenolate and Sodium p-Nitrophenolate, expressed as Sodium 5-Nitroguaiacolate as

calculated above is below the existing MRL of 0.15* mg/kg (Commission Regulation (EU) No. 2021/1098).

zRMS comments:
According to the SANTE/11956/2016 rev. 9, 14 September 2018, the residues are expected in honey after pesticide application when a substance is applied during the flowering stage (BBCH 60-69) of a crop which is foraged by bees and when a substance with systemic properties is applied prior to the flowering stage (before BBCH 60), including treatment of seeds, of a crop which is foraged by bees.
At the request of the zRMS, the Applicant submitted a new nectar residue study in the framework of this application. Four nectar residue trials were conducted with *Phacelia tanacetifolia* between 2019 and 2020 in Germany (2 trials), Spain (1 trial) and Southern France (1 trial). ATONIK was applied four times to the treated plot at a nominal rate of 1.0 L product/ha (nominal rate of components: Na 5-NG: 1.0 g as/ha; Na o-NP: 2.0 g as/ha; Na p-Na: 3.0 g as/ha). Four applications were conducted at BBCH 57-60, 62-63, 63-65 and 64-67. Samples were collected once after application 4 by hand.

Results:
Residues of Sodium 5-Nitroguaiacolate and Sodium p-Nitrophenolate were detected in the treated nectar samples at levels of 0.0175 and 0.110 mg/kg in trial -01, 0.00880 (<LOQ) and 0.0720 mg/kg in trial -02, 0.0107 and 0.0603 mg/kg in trial -04 and 0.0141 and 0.0700 mg/kg in trial -05. No residues of Sodium o-Nitrophenolate were detected at or above the limit of quantification (0.01 mg/kg) in the nectar samples of any of the trials.

HR of 0.14 mg/kg for Sodium 5-Nitroguaiacolate, Sodium *o*-Nitrophenolate and Sodium *p*-Nitrophenolate based on plant residue definition for risk assessment: sum of Sodium 5-Nitroguaiacolate, Sodium *o*-Nitrophenolate and Sodium *p*-Nitrophenolate, expressed as Sodium 5-Nitroguaiacolate is below the existing MRL of 0.15* mg/kg (Commission Regulation (EU) No. 2021/1098).
Based on the study, MRL of 0.14 mg/kg in honey for the sum of Sodium 5-Nitroguaiacolate, Sodium o-Nitrophenolate and Sodium p-Nitrophenolate, expressed as Sodium 5-Nitroguaiacolate was calculated.
The study is acceptable.
No further data are required.

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

The consumer risk assessment was performed under the Article 12 MRL review with revision 3.1 of the EFSA Pesticide Residues Intake Model (PRIMo).
The chronic consumer risk assessment has been updated taking into account the residue inputs used in the Article 12 MRL review, including the contribution from olives (CR EU 2021/1098 of 2 July 2021) and oranges (recently approved use in Greece).

7.2.8.1 Input values for the consumer risk assessment

Table 7.2-5: Overview of the toxicological reference values used for PRIMo

	Source	Year	Value	Study relied upon	Safety factor
Na 5-NG, Na *o*-NP and Na *p*-NP					
ADI	Review Report (SANCO/210/08 – rev. 2)	2013	0.003 mg/kg bw/day	1-year oral dog	100
ARfD	Review Report (SANCO/210/08 – rev. 2)	2013	0.045 mg/kg bw	Dev. tox. rabbit	300

Table 7.2-6: Input values for the consumer risk assessment

Commodity	Chronic risk assessment		Acute risk assessment	
	Input (mg/kg)	Comment	Input (mg/kg)	Comment
Oranges	0.03*	STMR (EFSA 2015)	-	-
Apples	0.03*	STMR (EFSA 2015)	-	-
Cherries	0.03*	STMR (EFSA 2015)	-	-

Commodity	Chronic risk assessment		Acute risk assessment	
	Input (mg/kg)	Comment	Input (mg/kg)	Comment
Table grapes	0.03*	STMR (EFSA 2015)	-	-
Wine grapes	0.03*	STMR (EFSA 2015)	-	-
Strawberries	0.03*	STMR (EFSA 2015)	-	-
Raspberries	0.03*	STMR (EFSA 2015)	-	-
Currants (red, black, white)	0.03*	STMR (EFSA 2015)	-	-
Table olives	0.12*	STMR (EFSA 2020)	-	-
Potatoes	0.03*	STMR (EFSA 2015)	-	-
Carrots	0.03*	STMR (EFSA 2015)	-	-
Onions	0.03*	STMR (EFSA 2015)	-	-
Tomatoes	0.03*	STMR (EFSA 2015)	-	-
Peppers	0.03*	STMR (EFSA 2015)	-	-
Aubergines (egg plant)	0.03*	STMR (EFSA 2015)	-	-
Cucumbers	0.03*	STMR (EFSA 2015)	-	-
Courgettes	0.03*	STMR (EFSA 2015)	-	-
Melon	0.03*	STMR (EFSA 2015)	-	-
Water melon	0.03*	STMR (EFSA 2015)	-	-
Poppy seed	0.03*	STMR (EFSA 2015)	-	-
Sunflower seed	0.03*	STMR (EFSA 2015)	-	-
Rape seed	0.03*	STMR (EFSA 2015)	-	-
Olives for oil production	0.12*	STMR (EFSA 2020)	-	-
Maize grain	0.03*	STMR (EFSA 2015)	-	-
Rice grain	0.03*	STMR (EFSA 2015)	-	-
Wheat grain	0.03*	STMR (EFSA 2015)	-	-
Hops (dried)	0.3*	STMR (EFSA 2015)	-	-
Sugar beet (root)	0.03*	STMR (EFSA 2015)	-	-
Products of animal origin				
-				

7.2.8.2 Conclusion on consumer risk assessment

Extensive calculation sheets are presented in 0.

Table 7.2-7: Consumer risk assessment

TMDI (% ADI) according to EFSA PRIMo 3.1	- % - oilseed rape – no record 7.0% - wheat – GEMS/Food G06 8.0% - sugar beet – NL child
IEDI (% ADI) according to EFSA PRIMo 3.1	41% - NL toddler
IESTI (% ARfD) according to EFSA PRIMo*	oilseed rape unprocessed: 0.09% for children oilseed rape processed: 0.0% for children wheat unprocessed: 1% for children wheat processed: 0.8% for children

	sugar beet unprocessed: - % for children sugar beet processed: 7.0% for children
NTMDI (% ADI) **	-
NEDI (% ADI)**	-
NESTI (% ARfD) **	-

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

A highest estimated chronic intake has been shown for NL toddler and represents 41% of the ADI.
Acute consumer risk has been assessed as well. The input values used in the exposure calculations are summarised in Tables 7.2-5 and 7.2-6. No exceedance of the ARfD was identified for the proposed commodities. The highest contribution for children is potato and melon for unprocessed (10% of ARfD) and Sugar beet (root) / sugar (10% of ARfD) for processed commodities.
It is concluded that the long-term and short-term intake of residues resulting from the proposed uses of ~~Atonik~~ Asahi Max on ~~orange and strawberry~~ oilseed rape, cereals and sugar beet are unlikely to present a risk to consumer health.

zRMS comments:
Information given by the Applicant is acceptable and sufficient.
The proposed uses of ARY-0469-04 / ASAHI MAX do not represent unacceptable acute and chronic risks for the consumer.

7.3 References

Commission Directive 7029/VI/95 rev.5 of 22nd July 1997. Appendix B, General recommendation for the design, preparation and realization of residue trials.

Commission Directive 2009/11/EC of 18 February 2009 amending Council Directive 91/414/EEC to include bensulfuron, sodium 5-nitroguaiacolate, sodium o-nitrophenolate, sodium p-nitrophenolate and tebufenpyrad as active substances. OJ L 48, 19.2.2009, p. 5-12.

Commission Implementing Regulation (EU) No 540/2011 of 25 May 2011 implementing Regulation (EC) No 1107/2009 of the European Parliament and of the Council as regards the list of approved active substances. OJ L 153, 11.6.2011, p.1-186.

Commission Implementing Regulation (EU) No 541/2011 of 1 June 2011 amending Implementing Regulation (EU) No 540/2011 implementing Regulation (EC) No 1107/2009 of the European Parliament and of the Council as regards the list of approved active substances. OJ L 153, 11.6.2011, p.187-188.

Commission Regulation (EU) 2016/1785 of 7 October 2016 amending Annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for cymoxanil, phosphane and phosphide salts and sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate in or on certain products.

Council Directive 91/414/EEC of 15 July 1991 concerning the placing of plant protection products on the market. OJ L 230, 19.8.1991, p. 1-32. Repealed by Regulation (EC) No 1107/2009.

CTGB, 2019. Evaluation Report Prepared under Article 8 of Regulation (EC) No 396/2005; Sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate; MRL application on the setting of an MRL in melons, and evaluation of confirmatory data following review according to Article 12 of Regulation (EC) No 396/2005, July 2019

EFSA Scientific Report (2008) 191, 1-130 Conclusion on the peer review of sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate

EFSA (European Food Safety Authority), 2015. Reasoned opinion on the review of the existing maximum residue levels for sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate according to Article 12 of Regulation (EC) No 396/2005. EFSA Journal 2015;13(12):4356, 39 pp. doi:10.2903/j.efsa.2015.4356

EFSA (European Food Safety Authority), 2020 a. Reasoned opinion on the evaluation of confirmatory data following the Article 12 MRL review for sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate (sodium nitrocompounds). EFSA Journal 2020;18(4):6060, 14 pp.

EFSA (European Food Safety Authority), 2020 b. Reasoned opinion on the evaluation of confirmatory data following the Article 12 MRL review for sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate (sodium nitrocompounds). EFSA Journal 2020;18(4):6060, 14 pp.

Greece, 2008. Final addendum to the Draft Assessment Report (DAR) - public version -Initial risk assessment provided by the rapporteur Member State Greece for the existing active substance sodium nitrocompounds, September 2008

Greece, 2011. Evaluation report prepared under Article 12.1 of Regulation (EC) No 396/2005. Review of Existing MRLs for sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate in food of plant and animal origin.

Greece, 2017. Sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate; Confirmatory data following review according to Article 12 of Regulation (EC) No 396/2005, June 2017.

Greece, 2019. Evaluation Report Prepared under Article 8 of Regulation (EC) No 396/2005; Sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate; MRL application on the setting of an MRL in olive, September 2019

Regulation (EC) No 396/2005 of the European Parliament and of the Council of 23 February 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin and amending Council Directive 91/414/EEC. OJ L 70, 16.3.2005, p. 1–16.

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 and repealing Council Directives 79/117/EEC and 91/414/EEC. OJ L 309, 24.11.2009, p. 1-50.

SANCO/210/08 – rev. 2, 17 May 2013. Review report for the active substances sodium 5-nitroguaiacolate, sodium o-nitrophenolate, sodium p-nitrophenolate, finalised in the Standing Committee on the Food

Chain and Animal Health at its meeting on 17 May 2013 in view of the inclusion of sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate in Annex I of Directive 91/414/EEC

SANCO 7525/VI/95 - rev. 10.3, 13 June 2017. Guidance document, Guidelines on comparability, extrapolation, group tolerances and data requirements for setting MRLs.

Appendix 1 Lists of data considered in support of the evaluation

Proposed Good Agricultural Practices (GAPs)

Crop and/ or situation *	Zone	Product code	F, Fn, Fpn G, Gn, Gpn or I**	Pests or Group of pests controlled	Formulation		Application				Application rate per treatment			PHI (days)	Conclusion
					Type	Conc. of as	method kind	growth stage & season	number min max	interval between applications (min)	kg as/hL min max	water L/ha min max	kg as/ha min max		
Winter oilseed ape, *Mustard, Oilseed rape spring), Poppy, unflower, Gold-f-pleasure*	C	ASAHI MAX	F	Plant growth regulator, number of pods per plant, number of seeds per plant, higher lignification of pods	SL	Na 5NG: 3.0 Na oNP: 6.0 Na pNP: 9.0	Spray	BBCH 29-69 (spring)			Na 5NG: 0.12-0.3 Na oNP: 0.24-0.6 Na pNP: 0.36-0.9	200-500	Na 5NG: 0.6 Na oNP: 1.2 Na pNP: 1.8	28	A
Winter wheat, *pring rye, Spelt, mmer wheat, mall spelt, urum wheat*	C	ASAHI MAX	F	Plant growth regulator, number of tillers and ears, portion above the sieves, germination energy	SL	Na 5NG: 3.0 Na oNP: 6.0 Na pNP: 9.0	Spray	BBCH 21-49 (spring)			Na 5NG: 0.2 Na oNP: 0.4 Na pNP: 0.6	00	Na 5NG: 0.6 Na oNP: 1.2 Na pNP: 1.8	28	A
Sugar beet *Fodder beet, Red beet, Mangold /chard, Swede, Turnip*	C	ASAHI MAX	F	Plant growth regulator, effect on higher yield of sugar, lower content of unwanted Sodium	SL	Na 5NG: 3.0 Na oNP: 6.0 Na pNP: 9.0	Spray	BBCH 12-49 (spring-summer)	3	7	Na 5NG: 0.12-0.3 Na oNP: 0.24-0.6 Na pNP: 0.36-0.9	200-500	Na 5NG: 0.6 Na oNP: 1.2 Na pNP: 1.8	15	A

Crops marked ~~in yellow~~ in *italic* is registered on the base of art 51.

* Use also code numbers according to Annex I of Regulation (EU) No 396/2005

** 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

*** Number of application timings is higher than number of possible applications

List of data submitted by the applicant and relied on

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCA 6.3.1-1	Diehl, m.	2006	Determination of residues of sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate in oil seed rape (rac seeds) following two treatments with atonik in northern europe, 2005 Company report no a06028 Rcc ltd Glp Unpublished	N	Asahi Chemical Europe s.r.o
KCA 6.3.1-2	Oxspring, s.	2010	Residues of sodium 5-nitroguaiacolate, sodium ortho-nitrophenolate and sodium para-nitrophenolate after two applications of atonik in oilseed rape at 1 site in northern europe 2008 Company report no s08-01067 Eurofins Glp Unpublished	N	Asahi Chemical Europe s.r.o
KCA 6.3.1-3	White, t.	2019	Atonik - study to generate specimens of oilseed rape following two applications of atonik. Three trials in northern europe during 2018 (final report amendment 1) Company report no s18-05054 Eurofins Glp Unpublished	N	Asahi Chemical Europe s.r.o
KCA 6.3.1-4	Guserle, r.	2019	Analysis of residues of sodium 5-nitroguaiacolate, sodium ortho-nitrophenolate and sodium para-nitrophenolate in field samples originating from a field study performed under eas study code s18-05054 with two applications of atonik in oilseed rape at three trials in norhtern europe during 2018 Company report no s18-05054 / laboratory report: p 4928 g Eurofins Glp Unpublished	N	Asahi Chemical Europe s.r.o
KCA 6.3.1-5	White, t.	2020	Atonik - study to generate samples of oilseed rape following two applications of atonik. One trial in northern europe during 2019 Company report no s19-00203 Eurofins Glp Unpublished	N	Asahi Chemical Europe s.r.o
KCA 6.3.1-6	Guserle, r.	2020	Analysis of residues of sodium 5-nitroguaiacolate, sodium ortho-nitrophenolate and sodium para-nitrophenolate in field samples originating from a field study performed under eas study code s19-00203 with two applications of atonik in oilseed rape at one trial in northern europe during 2019	N	Asahi Chemical Europe s.r.o

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
			Company report no s19-00203/ laboratory report: p 5295 g Eurofins Glp Unpublished		
KCA 6.3.1-7	Diehl, m.	2006	Determination of residues of sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitropehnolate in cereals (rac straw and grain) following two treatments with atonik in northern europe 2005 Company report no a05995 Rcc ltd Glp Unpublished	N	Asahi Chemical Europe s.r.o
KCA 6.3.1-8	White, t.	2018	Atonik – study to generate specimens of winter wheat following two applications of atonik. Three trials in northern europe during 2018 Company report no s18-05052 Eurofins Glp Unpublished	N	Asahi Chemical Europe s.r.o
KCA 6.3.1-9	Guserle, r.	2019	Analysis of residues of sodium 5-nitroguaiacolate, sodium ortho-nitrophenolate and sodium para-nitrophenolate in field samples originating from a field study performed under eas study code s18-05052 with two applications of atonik in winter wheat at 3 trials in northern europe during 2018 Company report no s18-05052/ laboratory report: p 4930 g Eurofins Glp Unpublished	N	Asahi Chemical Europe s.r.o
KCA 6.3.1-10	White, t.	2020	Atonik – study to generate specimens of winter wheat following two applications of atonik. One trial in northern europe during 2019 Company report no s19-00202 Eurofins Glp Unpublished	N	Asahi Chemical Europe s.r.o
KCA 6.3.1-11	Guserle, r.	2020	Analysis of residues of sodium 5-nitroguaiacolate, sodium ortho-nitrophenolate and sodium para-nitrophenolate in field samples originating from a field study performed under eas study code s19-00202 with two applications of atonik in winter wheat at 1 trial in northern europe during 2020 Company report no s19-00202/ laboratory report: p 5296 g Eurofins Glp	N	Asahi Chemical Europe s.r.o

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
			Unpublished		
KCA 6.3.1-12	Diehl, m.	2006	Determination of residues of sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate in sugar beet (rac roots and leaves) following four treatments with atonik in northern europe, 2005 Company report no a05973 Rcc ltd Glp Unpublished	N	Asahi Chemical Europe s.r.o
KCA 6.3.1-13	Oxspring, s.	2014	Atonik - determination of residues of sodium 5-nitroguaiacolate, sodium ortho-nitrophenolate and sodium para-nitrophenolate after four applications of atonik in sugar beet at 1 site in northern europe 2013 Company report no s12-04698 Eurofins Glp Unpublished	N	Asahi Chemical Europe s.r.o
KCA 6.3.1-14	White, t.	2020	Atonik - determination of residues of sodium 5-nitroguaiacolate, sodium o-nitrophenolate and sodium p-nitrophenolate in sugar beet following three applications of atonik under field conditions - three trials in northern europe during 2019 Company report no s19-04275 Eurofins Glp Unpublished	N	Asahi Chemical Europe s.r.o
KCA 6.10, 6.10.1	Kugel, D.	2020	Determination of Residues of 5-Nitroguaiacol, *o*-Nitrophenol and *p*-Nitrophenol in Nectar after four Applications of ATONIK containing Sodium 5-Nitroguaiacolate, Sodium *o*-Nitrophenolate and Sodium *p*-Nitrophenolate in *Phacelia tanacetifolia* at 4 Sites in Central and Southern Europe in 2019. Report No.: S19-03993 (634-96002) Eurofins Agroscience Services Ecotox GmbH, Niefern-Öschelbronn, Germany GLP, unpublished	N	Asahi Chemical Europe s.r.o.

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

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCA 6.2.1-1	Diehl M	2004	14C-ATONIK: Plant metabolism in sugar beet.	N	Asahi Chemical

Data point	Author(s)	Year	**Title** **Company Report No.** **Source (where different from company)** **GLP or GEP status** **Published or not**	**Vertebrate study Y/N**	**Owner**
			RCC Ltd., Report No: 815433 GLP Unpublished		Europe s.r.o.
KCA 6.2.1-2	Diehl M	2004	14C-ATONIK: Plant metabolism in tomato. RCC Ltd., Report No: 815444 GLP Unpublished	N	Asahi Chemical Europe s.r.o.
KCA 6.2.1-3	Diehl M	2004	14C-ATONIK: Plant metabolism in rape. RCC Ltd., Report No: 815455 GLP Unpublished	N	Asahi Chemical Europe s.r.o.

List of data submitted by the applicant and not relied on

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

Appendix 2 Detailed evaluation of the additional studies relied upon

A 2.1 Sodium 5-nitroguaiacolate, Sodium o-nitrophenolate and Sodium p-nitrophenolate

A 2.1.1 Stability of residues

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

A 2.1.1.1 Stability of residues during storage of samples

A 2.1.1.1.1 Storage stability of residues in plant products

A 2.1.1.1.1.1 Study 1

A residue study in nectar, including a storage stability testing is summarised below:

Comments of zRMS:	The stability was demonstrated for 5-Nitroguaiacol, o-Nitrophenol and p-Nitrophenol expressed as Sodium 5-Nitroguaiacolate, Sodium o-Nitrophenolate and Sodium p-Nitrophenolate in nectar upon storage at ≤ -18 °C in the dark for at least 232 days. The average amount of analyte recovered relative to the initial recovery at day 0 was ≥70% at the testing intervals (113 and 232 days), which can be seed as criterion for sufficient storage stability. The study is acceptable.

Data point addressed:	**KCA 6.10, 6.10.1**
Author(s) (year):	Kugel, D. (2020)
Title:	Determination of Residues of 5-Nitroguaiacol, *o*-Nitrophenol and *p*-Nitrophenol in Nectar after four Applications of ATONIK containing Sodium 5-Nitroguaiacolate, Sodium *o*-Nitrophenolate and Sodium *p*-Nitrophenolate in *Phacelia tanacetifolia* at 4 Sites in Central and Southern Europe in 2019
Laboratory report / project Number (Doc. No.):	S19-03993 (634-96002)
Testing facility:	Eurofins Agroscience Services Ecotox GmbH, Niefern-Öschelbronn, Germany
Published:	No
Test guideline used:	SANTE/11956/2016 rev. 9, OECD No. 509 (2009), SANCO/825/00, rev. 8.1 (2010), SANCO/3029/99, rev. 4 (2000), OECD Testing and assessment No. 72 and Series on Pesticides No. 39, ENV/JM/MONO(2007)17
Deviations:	None
Previous evaluation:	No, not previously provided
GLP:	Yes; certified by the Landesanstalt für Umwelt Baden-Württemberg, Germany
Acceptability/Reliability:	Yes

Executive Summary

The freezer storage stability study investigates the stability of Sodium 5-Nitroguaiacolate, Sodium *o*-Nitrophenolate and Sodium *p*-Nitrophenolate in nectar.

Homogenised samples were fortified with Na 5-NG, Na *o*-NP and Na *p*-NP at a nominal rate of 0.128 mg/kg (12.8x LOQ) and were stored deep frozen (below -18°C). Samples were analysed after 0, 113 and 232 days of storage. Untreated control and freshly fortified samples were prepared and analysed together with the stored fortified samples.

At day 0, three storage samples per matrix spiked at 12.8x LOQ (0.128 mg/kg), corresponding also to procedural recovery samples at day 0, were analysed directly together with one control sample. After 0, 113 and 232 days of deep-frozen storage one untreated control sample and three stored fortified samples were analysed together with two freshly fortified samples acting as procedural recoveries.

For Na 5-NG storage stability was shown in nectar for a period of at least 7.7 months (232 days), respectively, at temperatures below -18°C.

For Na *o*-NP storage stability it will be shown in nectar for a period of at least 7.7 months (232 days), respectively, at temperatures below -18°C.
For Na *p*-NP storage stability it will be shown in nectar for a period of at least 7.7 months (232 days), respectively, at temperatures below -18°C.

I. MATERIALS AND METHODS:
A. MATERIALS
1. Test material/Reference item

Table A2-1 Test material/Reference item

Report number:	S19-03993
Test material:	5-Nitroguaiacol
CAS #:	636-93-1
Lot/Batch #:	5NG2527C9
Content of a.s. (actual):	100 %
Stability of test compound (Expiry date)	27 March 2022

Report number:	S19-03993
Test material:	*o*-Nitrophenol
CAS #:	88-75-5
Lot/Batch #:	ONP2527C9
Content of a.s. (actual):	100 %
Stability of test compound (Expiry date)	27 March 2022

Report number:	S19-03993
Test material:	*p*-Nitrophenol
CAS #:	100-02-7
Lot/Batch #:	PNP2527C9
Content of a.s. (actual):	100 %
Stability of test compound (Expiry date)	27 March 2022

Table A2-2 Test commodity

Crop:	Nectar
Processed:	homogenised
Sampling	20 different units (flowers) from 12 different locations
Sample size:	0.2 g – 0.66 g

B. STUDY DESIGN AND METHODS
The study was conducted between 17 May 2019 and 05 November 2020 by Eurofins Agroscience Services Ecotox GmbH, Niefern-Öschelbronn, Germany.

1. Test procedure
The freezer storage stability of Na 5-NG, Na *o*-NP and Na *p*-NP was determined in nectar. Homogenised samples were fortified with Na 5-NG, Na *o*-NP and Na *p*-NP at a nominal rate of 0.128 mg/kg (12.8x LOQ), and were stored frozen (below -18°C). Samples were analysed after 0, 113 and 232 days of frozen storage (below -18°C). Untreated control samples and freshly fortified samples were prepared and analysed together with the stored fortified samples.
At day 0, three storage samples spiked at 12.8x LOQ (0.128 mg/kg), corresponding also to procedural recovery samples at day 0, were analysed directly together with one control sample. After 0, 113 and 232 days of deep-frozen storage one untreated control sample and three stored fortified samples were analysed together with two freshly fortified samples acting as procedural recoveries.

2. Description of analytical procedures
The analytical method was successfully validated in nectar within the current study. The method validation is described in more detail in dRR section B5.

Residues of Na 5-NG, Na *o*-NP and Na *p*-NP were extracted from homogenised nectar with water/acetonitrile (8:2) and centrifuged. Separation was carried out by liquid chromatography, followed by triplequadrupole mass spectrometric detection (MS/MS). The quantification was performed with the free phenol reference item all expressed as the sodium salt.
The limit of quantification (LOQ) in nectar was 0.01 mg/kg for Na 5-NG, Na *o*-NP and Na *p*-NP, respectively. The limit of detection (LOD) for Na 5-NG, Na *o*-NP and Na *p*-NP was set at 0.003 mg/kg.

II. RESULTS AND DISCUSSION

The spiked stored samples were analysed after 0,113 and 232 days.
Procedural recoveries were analysed concurrently with the stored samples showing recoveries between 70 and 110% and thus, are demonstrating the accuracy of the method on the day of analysis.
Nectar extracts were analysed on the day of extraction. The stability of Na 5-NG, Na *o*-NP and Na *p*-NP in the specimen extracts was proven for the longest storage period by procedural recoveries, which were stored under the same conditions together with the sample extracts. In addition, Na 5-NG, Na *o*-NP and Na *p*-NP was shown to be stable when stored at 1 to 10 °C for 7 days. This time period was not exceeded in the current study.
The results are summarised in the following tables:

Table A2-3 Storage stability of Sodium 5-Nitroguaiacolate (Na 5-NG) in homogenised nectar stored at < -18°C

Nominal storage period [months] (days*)	Nominal spiking level [mg/kg]	Recovery of stored sample					Nominal spiking level [mg/kg]	Recovery of freshly spiked sample			
		[mg/kg] of nominal fortification level		[%] of nominal fortification level		[%] of initial		[mg/kg]		[%]	
		Single	Mean**	Single	Mean	Mean		Single	Mean**	Single	Mean
Nectar											
0	0.128	0.123 0.123 0.119	0.122	96 96 93	95	100	0.128	0.123 0.123 0.119	0.122	96 96 93	95
3.7 months (113 days)	0.128	0.126 0.128 0.130	0.128	98 110 102	100	105	0.0128 0.128	0.0106 0.125	--	83 98	--
7.7 month (232 days)	0.128	0.137 0.131 0.129	0.132	107 102 101	103	108	0.0128 0.128	0.0130 0.146	--	102 114	--

* Storage time: fortification until extraction
**calculated by notifier

Table A2-4 Storage stability of Sodium *o*-Nitrophenolate (Na *o*-NP) in homogenised nectar stored at < -18°C

Nominal storage period [months] (days*)	Nominal spiking level [mg/kg]	Recovery of stored sample					Nominal spiking level [mg/kg]	Recovery of freshly spiked sample			
		[mg/kg] of nominal fortification level		[%] of nominal fortification level		[%] of initial		[mg/kg]		[%]	
		Single	Mean**	Single	Mean	Mean		Single	Mean**	Single	Mean
Nectar											
0	0.128	0.111 0.108 0.097	0.105	87 84 76	82	100	0.128	0.111 0.108 0.097	0.105	87 84 76	82
3.7 months (113 days)	0.128	0.109 0.111 0.110	0.110	85 87 86	86	105	0.0128 0.128	0.0115 0.116	--	90 91	--
7.7 month (232 days)	0.128	0.110 0.114 0.118	0.114	86 89 92	89	109	0.0128 0.128	0.0140 0.139	--	109 109	--

* Storage time: fortification until extraction
**calculated by notifier

Table A2-5 Storage stability of Sodium *p*-Nitrophenolate (Na *p*-NP) in homogenised nectar stored at < -18°C

Nominal storage period [months] (days*)	Nominal spiking level [mg/kg]	Recovery of stored sample					Nominal spiking level [mg/kg]	Recovery of freshly spiked sample			
		[mg/kg] of nominal fortification level		[%] of nominal fortification level		[%] of initial		[mg/kg]		[%]	
		Single	Mean**	Single	Mean	Mean		Single	Mean**	Single	Mean
Nectar											
0	0.128	0.128 0.128 0.125	0.127	100 100 98	99	100	0.128	0.128 0.128 0.125	0.127	100 100 98	99
3.7 months (113 days)	0.128	0.123 0.124 0.129	0.125	96 97 101	98	99	0.0128 0.128	0.0121 0.121	--	95 95	--
7.7 month (232 days)	0.128	0.125 0.135 0.136	0.132	98 105 106	103	104	0.0128 0.128	0.0141 0.138	--	110 108	--

* Storage time: fortification until extraction
**calculated by notifier

It was shown that Na 5-NG residues are stable in nectar for at least 7.7 months (232 days) when the commodities are stored homogenised under frozen conditions (below -18°C).
It was shown that Na *o*-NP residues are stable in nectar for at least 7.7 months (232 days) when the commodities are stored homogenised under frozen conditions (below -18°C).
It was shown that Na *p*-NP residues are stable in nectar for at least 7.7 months (232 days) when the commodities are stored homogenised under frozen conditions (below -18°C).

In addition, storage stability of Na p-NP in nectar extracts was tested within the residue study in nectar. A detailed description regarding the investigated storage stability in extracts is provided in the following.

Executive Summary
The storage stability of Na 5-NG, Na *o*-NP and Na *p*-NP in nectar samples stored at 1 to 10°C has been investigated showing stability of Na 5-NG, Na *o*-NP and Na *p*-NP for 7 days in nectar samples.

I. MATERIALS AND METHODS
A. MATERIALS
1. Test material/Reference item

Table A2-6 Test material/Reference item

Report number:	S19-03993
Test material:	5-Nitroguaiacol
CAS #:	636-93-1
Lot/Batch #:	5NG2527C9
Content of a.s. (actual):	100 %
Stability of test compound (Expiry date)	27 March 2022

Report number:	S19-03993
Test material:	*o*-Nitrophenol
CAS #:	88-75-5
Lot/Batch #:	ONP2527C9
Content of a.s. (actual):	100 %
Stability of test compound (Expiry date)	27 March 2022

Report number:	S19-03993
Test material:	*p*-Nitrophenol
CAS #:	100-02-7
Lot/Batch #:	PNP2527C9
Content of a.s. (actual):	100 %
Stability of test compound (Expiry date)	27 March 2022

Table A2-7 Test commodity

Crop:	Nectar
Processed:	homogenised
Sampling	20 different units (flowers) from 12 different locations
Sample size:	0.2 g – 0.66 g

B. STUDY DESIGN AND METHODS
The study was conducted between 17 May 2019 and 05 November 2020 by Eurofins Agroscience Services Ecotox GmbH, Niefern-Öschelbronn, Germany.

1. Test procedure
The extract stability of Na 5-NG, Na *o*-NP and Na *p*-NP was determined in nectar. Homogenised samples were fortified with Na 5-NG, Na *o*-NP and Na *p*-NP at a nominal rate of 0.1 mg/kg (10x LOQ). Samples were extracted and analysed and then stored for 7 days at 1 to 10°C. The stored samples were re-analysed together with freshly prepared calibration solutions.

2. Description of analytical procedures

The analytical method was successfully validated in nectar within the current study. The method validation is described in more detail in dRR section B5.

Residues of Na 5-NG, Na *o*-NP and Na *p*-NP were extracted from homogenised nectar with water/acetonitrile (8:2) and centrifuged. Separation was carried out by liquid chromatography, followed by triplequadrupole mass spectrometric detection (MS/MS). The quantification was performed with the free phenol reference item all expressed as the sodium salt.

The limit of quantification (LOQ) in nectar was 0.01 mg/kg for Na 5-NG, Na *o*-NP and Na *p*-NP, respectively. The limit of detection (LOD) for Na 5-NG, Na *o*-NP and Na *p*-NP was set at 0.003 mg/kg.

II. RESULTS AND DISCUSSION

The spiked stored samples were analysed after 7 days of storage at temperatures of 1 to 10°C.

Extracts analysed for Na 5-NG, Na *o*-NP and Na *p*-NP, were stored for 7 days, showing recoveries within 70 – 110 %.

The results are summarised in the following table:

Table A2-8 Storage stability of Na 5-NG, Na *o*-NP and Na *p*-NP in nectar extracts stored at 1 to 10°C.

Matrix	Storage period [days]	Nominal spiking [mg/kg] **	Recoveries				Difference (in %) of recoveries after storage to recoveries before storage in % *
			Peak area 1st injection	Recovery 1st injection	Peak area 2st injection	Recovery 2st injection	
Na 5-NG							
Nectar	7	0.1	3342301	88	2957697	85	3
			3532527	93	3102004	89	
			3143799	83	3180776	92	
			3426141	91	3212609	93	
			3103543	82	3220566	93	
Na *o*-NP							
Nectar	7	0.1	286575	82	208596	70	0
			233131	67	255237	86	
			334613	96	264908	90	
			306315	88	272058	92	
			321129	92	261741	88	
Na *p*-NP							
Nectar	7	0.1	4320976	90	4168085	89	-2
			4362599	91	4067521	87	
			4275144	89	4241386	91	
			4566971	95	4387022	94	
			4472665	93	4273067	91	

* Difference = (100 x MeanRecoveries, stored / MeanRecoveries, fresh) – 100; calculated from rounded values
**expressed as sodium salts

The mean recoveries of the re-analysed nectar extracts were in the range of 70 – 110 % and within ±20 % of the original result. Therefore, the nectar extracts are considered to be stable when stored at 1°C to 10 °C for 7 days.

III. CONCLUSION

It was shown that Na 5-NG residues are stable in nectar extracts for at least 7 days, when the extracts are stored at 1 to 10°C.

It was shown that Na *o*-NP residues are stable in nectar extracts for at least 7 days, when the extracts are stored at 1 to 10°C.

It was shown that Na *o*-NP residues are stable in nectar extracts for at least 7 days, when the extracts are stored at 1 to 10°C.

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

No new data submitted in the framework of this application.

A 2.1.3 Magnitude of residues in plants

A 2.1.3.1 Oilseed rape

Table A 1: 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)
Critical GAP NEU	2x	0.6 g / ha Na-5NG 1.2 g/ ha Na-*o*NP 1.8 g/ ha Na-*p*NP	7 days	BBCH ~~29-69~~ 30-60	28
Proposed GAP NEU	2x	0.6 g / ha Na-5NG 1.2 g/ ha Na-oNP 1.8 g/ ha Na-pNP	7 days	BBCH 29-69	28

A 2.1.3.1.1 Study A06028

Comments of zRMS:	Two residue trials on oilseed rape were conducted in northern Europe to determine residue of Na 5-NG, Na o-NP and Na p-NP. Oilseed rape was treated twice at application rate of 1.0 L product/ha (corresponding to 1 g/ha sodium 5-nitroguaiacolate +2 g/ha sodium o-nitrophenolate + 3 g/ha sodium p-nitrophenolate) with 30 days interval between applications. Seeds were collected at commercial harvest (27 or 37 days after last application). All procedures for specimen preparation and analysis for Sodium 5-Nitroguaiacolate, Sodium o-Nitrophenolate and Sodium p-Nitrophenolate followed as validated under RCC Study No. 850917 ("Development and Validation of a Residue Analytical Method for Sodium 5-Nitroguaiacolate, Sodium o-Nitrophenolate and Sodium p-Nitrophenolate (as Active Ingredients in Atonik Formulated Product) in Tomato (Fruits), Sugar Beet (Roots and Tops with Leaves) and Oil Seed Rape"; Dr. Alexander Krainz; September 16, 2004. The limit of quantification (LOQ) was 0.01 mg/kg for seed for Na 5-NG, Na o-NP and Na p-NP. In analytical report for oilseed rape, only one recovery determination at LOQ has been presented for Na 5-NG, Na o-NP and Na p-NP respectively. This is not fully with the requirement of SANTE/2020/12830, Rev.1, 24. February 2021. Five recovery determinations at LOQ (0.01 mg/kg and five recovery determinations at 10x LOQ (0.1 mg/kg) should be performed for this purpose. At least one reagent blank and two control samples should be analysed. For procedural recoveries: all residue samples should be measured in one analytical set per matrix, which included five fortifications at LOQ and five fortifications at 10x LOQ. Maximum storage period – 63 days. <u>Results:</u> The residues of Na 5-NG, Na o-NP and Na p-NP in oilseed rape seeds at harvest were <0.01 mg/kg. The study is acceptable.

Data point addressed:	KCA 6.3.1-1
Author(s) (year):	Diehl, M. (2006)
Title:	DETERMINATION OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM O-NITROPHENOLATE AND SODIUM P-NITROPHENOLATE IN OIL SEED RAPE (RAC SEEDS) FOLLOWING TWO TREATMENTS WITH ATONIK IN NORTHERN EUROPE, 2005
Laboratory report / project Number (Doc. No.):	A06028
Testing facility:	RCC Ltd, Environmental Chemistry & Pharmanalytics Division, Itingen, Switzerland

Published:	No
Test guideline used:	96/68/EC (1996), 7029/VI/95 rev. 5 (1997), in accordance to OECD 509 (2009)
Deviations:	None
Previous evaluation:	No; not previously submitted
GLP:	Yes; certified by Swiss Federal Office of Public Health, Bern
Acceptability/Reliability:	Yes

Materials and methods

Description:	**ATONIK**		
Components:	Na 5-NG	Na *o*-NP	Na *p*-NP
CAS #:	67233-85-6	824-39-5	824-78-2
Lot/Batch #:	2005-AC-50628		
Content of a.s. (actual):	0.12 %	0.22 %	0.32 %
Stability of test compound (Expiry date):	May 2008		

The residue study on oilseed rape has been performed at two field sites in Germany. This residue study provides data relevant to conditions in the Northern European region. The experimental setup includes two at-harvest trials.

Atonik was applied twice to the treated oilseed rape plots at a nominal rate of 1.0 L product/ha.
Sodium 5-Nitroguaiacolate was applied in the range of 1.11 and 1.23 g/ha for the first application and in the range of 1.23 and 1.29 g/ha for the second application.
Sodium o-Nitrophenolate was applied in the range of 2.05 and 2.26 g/ha for the first application and in the range of 2.26 and 2.37 g/ha for the second application.
Sodium p-Nitrophenolate was applied in the range of 2.97 and 3.28 g/ha for the first application and in the range of 3.28 and 3.45 g/ha for the second application.
All trials were carried out in the Germany. Samples (seeds) were taken at harvest, at PHI 27 and 37, respectively.
The field phase was performed by GAB Biotechnology GmbH, Niefern-Öschelbronn, Germany, whereas the analytical work associated with the studies was performed by RCC Ltd., Itingen, Switzerland.

Crop specimens were analyzed for residues of Na 5-NG, Na *o*-NP and Na *p*-NP using an HPLC method described below.
Principle of the method:
Na 5-NG was extracted from homogenised seeds with methanol. Extracts were cleaned up by solid phase extraction. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).
Na o-NP was extracted from homogenised seeds with methanol. Extracts were cleaned up by solid phase extraction. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).
Na p-NP was extracted from homogenised seeds with methanol. Extracts were cleaned up by solid phase extraction. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).
The limit of quantification (LOQ) for Na 5-NG, Na o-NP and Na p-NP was 0.01 mg/kg in seeds. The limit of detection (LOD) for Na 5-NG, Na o-NP and Na p-NP was set at 0.005 mg/kg in seeds.
For Na 5-NG, the maximum sampling to analysis interval at -18°C was 63 days in oilseeds. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na 5-NG in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na o-NP, the maximum sampling to analysis interval at -18°C was 63 days in oilseeds. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na o-NP in the samples extract. All recoveries were within the range between

70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na p-NP, the maximum sampling to analysis interval at -18°C was 63 days in oilseeds. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na p-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.

Results and discussions
Residues in untreated control samples were not detectable.
Residues of Na 5-NG, Na o-NP, Na p-NP in oilseed rape seeds at harvest (PHI 28 days) are below the limit of quantification (LOQ = 0.01 mg/kg), when Atonik is applied twice at a nominal rate of 0.6 g/ha Na 5-NG, 1.2 g/ha Na o-NP and 1.8 g/ha Na p-NP with a PHI of 28 days in Northern Europe.

A 2.1.3.1.2 Study S08-01067

Comments of zRMS:	One residue trial on oilseed rape was conducted in northern Europe to determine residue of Na 5-NG, Na o-NP and Na p-NP. Oilseed rape was treated twice at application rate of 1.0 L product/ha (corresponding to 1 g/ha sodium 5-nitroguaiacolate +2 g/ha sodium o-nitrophenolate + 3 g/ha sodium p-nitrophenolate) with 13 days interval between applications. Specimens of crop from the untreated and treated plot were taken by hand 30 days after the final application. The method was validated in another study conducted in 2009 on sunflower specimens: ARYST/TOUR/08.01 (Eurofins Agroscience study number S08-01147) by the realisation of three recovery experiments conducted at the limit of quantification and one control sample. The mean recovery was between 70% and 110% with a relative standard deviation lower than 20%. The specificity of the method had been demonstrated, interferences due to the substrate were less than 30% of the limit of quantification. The limit of quantification (LOQ) was 0.01 mg/kg for seed for Na 5-NG, Na o-NP and Na p-NP. In analytical report for oilseed rape, only one recovery determination at LOQ has been presented for Na 5-NG, Na o-NP and Na p-NP respectively. This is not fully with the requirement of SANTE/2020/12830, Rev.1, 24. February 2021. Five recovery determinations at LOQ (0.01 mg/kg and five recovery determinations at 10x LOQ (0.1 mg/kg) should be performed for this purpose. At least one reagent blank and two control samples should be analysed. For procedural recoveries: all residue samples should be measured in one analytical set per matrix, which included five fortifications at LOQ and five fortifications at 10x LOQ. Maximum storage period – 233 days. Results: No residues above the limit of determination were found in any of the treated or untreated specimens. The residues of Na 5-NG, Na o-NP and Na p-NP in oilseed rape seeds at harvest were <0.01 mg/kg. The study is acceptable.

Data point addressed:	KCA 6.3.1-2
Author(s) (year):	Oxspring, S. (2010)
Title:	DETERMINATION OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM ORTHO-NITROPHENOLATE AND SODIUM PARA-NITROPHENOLATE AFTER TWO APPLICATIONS OF ATONIK IN OILSEED RAPE AT 1 SITE IN NORTHERN EUROPE 2008
Laboratory report / project Number (Doc. No.):	S08-01067
Testing facility:	Eurofins Agroscience Services Ltd., Melbourne, United Kingdom

Published:	No
Test guideline used:	96/68/EC, in accordance to OECD 509 (2009)
Deviations:	None
Previous evaluation:	No, not previously submitted
GLP:	Yes; certified by the Department of Health of the Government of the United Kingdom and Groupe Interministeriel des Produits Chimiques, Paris
Acceptability/Reliability:	Yes

Materials and methods

Description:	**ATONIK**		
Components:	Na 5-NG	Na *o*-NP	Na *p*-NP
CAS #:	67233-85-6	824-39-5	824-78-2
Lot/Batch #:	037C8		
Content of a.s. (actual):	0.12 %	0.22 %	0.33 %
Stability of test compound (Expiry date):	13 March 2011		

The residue study on oilseed rape has been performed at one field site in Poland. This residue study provides data relevant to conditions in the Northern European region. The experimental setup includes one at-harvest trials.

Atonik was applied twice to the treated oilseed rape plot at a nominal rate of 1.0 L product/ha.
Sodium 5-Nitroguaiacolate was applied with an application rate of 0.97 g/ha for the first application and 1.09 g/ha for the second application for Sodium 5-Nitroguaiacolate.
Sodium o-Nitrophenolate was applied with an application rate of 1.93 g/ha for the first application and 2.17 g/ha for the second application.
Sodium p-Nitrophenolate was applied with an application rate of 2.90 g/ha for the first application and 3.26 g/ha for the second application.
The trial was carried out in Poland. Samples (seeds) were taken at harvest (PHI 30 days). The residue study was thus conducted in accordance with the cGAP.
The field phase was performed by Eurofins Agroscience Services Ltd., Melbourne, United Kingdom, whereas the analytical work associated with the study was performed by GIRPA, 49070 Beaucouzé, France.

Crop specimens were analyzed for residues of Na 5-NG, Na *o*-NP and Na *p*-NP using an HPLC method described below.
Principle of the method:
Na 5-NG was extracted by maceration with methanol. Extracts were purified by a clean-up cartridge. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).

Na o-NP was extracted by maceration with methanol. Extracts were purified by a clean-up cartridge. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).

Na p-NP was extracted by maceration with methanol. Extracts were purified by a clean-up cartridge. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).

The limit of quantification (LOQ) for Na 5-NG, Na o-NP and Na p-NP was 0.01 mg/kg in seeds.
For Na 5-NG, the maximum sampling to analysis interval at -18°C was 233 days in oilseeds. The maximum extraction to quantification interval was 1 day for Na 5-NG. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na 5-NG in the samples extract. All recoveries were within the range between 70 – 110%. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na o-NP, the maximum sampling to analysis interval at -18°C was 233 days in oilseeds. The maximum extraction to quantification interval was 1 day for Na o-NP. Procedural recoveries were handled and stored

in the same way and for the same time periods as the analytical samples thereby proving stability of Na o-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na p-NP, the maximum sampling to analysis interval at -18°C was 233 days in oilseeds. The maximum extraction to quantification interval was 1 days for Na p-NP. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na p-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.

Results and discussions
Residues in untreated control samples were below the limit of quantification (LOQ = 0.01 mg/kg).
Residues of Na 5-NG, Na o-NP, Na p-NP in oilseed rape seeds at harvest (PHI 28 days) are below the limit of quantification (LOQ = 0.01 mg/kg), when Atonik is applied twice at a nominal rate of 0.6 g/ha Na 5-NG, 1.2 g/ha Na o-NP and 1.8 g/ha Na p-NP with a PHI of 28 days in Northern Europe.

A 2.1.3.1.3 Study S18-05054

Comments of zRMS:	Three residue trials on oilseed rape were conducted in northern Europe to determine residue of Na 5-NG, Na o-NP and Na p-NP. Oilseed rape was treated twice at application rate of 1.0 L product/ha (corresponding to 1 g/ha sodium 5-nitroguaiacolate +2 g/ha sodium o-nitrophenolate + 3 g/ha sodium p-nitrophenolate) with 7 days interval between applications. For the decline trial, samples were taken at day 0, 7, 14, 20 and 28 after the last application. For the at-harvest trials, samples were taken at day 28 after the last application. Analytical Method Sample extraction and determination of residues were performed based on the method used during EAS Study Code S12-04715 after adaption and validation oilseed rape (OSR) whole plant and seed. Quantification was performed by use of LC-MS/MS detection. The limit of quantification (LOQ) of the analytical method was 0.01 mg/kg per analyte (expressed as sodium salt) for seed matrix. For determination of the analytes in OSR whole plant the LOQ was set to 0.01 mg/kg (for Na 5-NG and Na o-NP) and 0.05 mg/kg (for Na p-NP). The mean recovery was between 70% and 110% with a relative standard deviation lower than 20%. The method is fully validated with the requirement of SANTE/2020/12830, Rev.1, 24. February 2021 in this report for oilseed rape. Maximum storage period was 107 days (3 months) for seeds and 244 days (9 months) for whole plant. According to the OECD 506 oilseed rape seed belongs to high oil commodity and whole plant belongs to high water commodity. The sodium nitrocompounds are stable for a period of 9 months in high oil content and for a period of 3 months in high water content commodities. So stability data are not cover the storage time for whole plant. Results: The residues of Na 5-NG, Na o-NP and Na p-NP in oilseed rape seeds at harvest were <0.01 mg/kg. The study is acceptable.

Data point addressed:	KCA 6. 3.1-3
Author(s) (year):	White, T. (2019)
Title:	ATONIK - STUDY TO GENERATE SPECIMENS OF OILSEED RAPE FOLLOWING TWO APPLICATIONS OF ATONIK. THREE TRIALS IN NORTHERN EUROPE DURING 2018 (FINAL REPORT AMENDMENT 1)
Laboratory report / project Number (Doc. No.):	S18-05054

Testing facility:	Eurofins Agroscience Services Ltd., Melbourne, United Kingdom
Published:	No
Test guideline used:	OECD No. 509, 7029/VI/95 rev. 5 (1997)
Deviations:	None
Previous evaluation:	No. not previously submitted
GLP:	Yes; certified by the Department of Health of the Government of the United Kingdom
Acceptability/Reliability:	Yes

Analytical part:

Data point addressed:	KCA 6. 3.1-4
Author(s) (year):	Guserle, R. (2019)
Title:	ANALYSIS OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM ORTHO-NITROPHENOLATE AND SODIUM PARA-NITROPHENOLATE IN FIELD SAMPLES ORIGINATING FROM A FIELD STUDY PERFORMED UNDER EAS STUDY CODE S18-05054 WITH TWO APPLICATIONS OF ATONIK IN OILSEED RAPE AT THREE TRIALS IN NORHTERN EUROPE DURING 2018
Laboratory report / project Number (Doc. No.):	P 4928 G S18-05054
Testing facility:	EAG Laboratories GmbH, Ulm, Germany
Published:	No
Test guideline used:	SANCO/3029/00 rev. 4 (2000)
Deviations:	None
Previous evaluation:	No. not previously submitted
GLP:	Yes; certified by LUBW Landesanstalt für Umwelt, Messungen und Naturschutz Baden-Württemberg, Karlsruhe
Acceptability/Reliability:	Yes

Materials and methods

Description:	**ATONIK**		
Components:	Na 5-NG	Na *o*-NP	Na *p*-NP
CAS #:	67233-85-6	824-39-5	824-78-2
Lot/Batch #:	CG7-101		
Content of a.s. (actual):	0.095 %	0.21 %	0.31 %
Stability of test compound (Expiry date):	31 May 2020		

The residue study on oilseed rape has been performed during 2018 at one field site in Poland and on two sites in Germany. This residue study provides data relevant to conditions in the Northern European region. The experimental setup includes two at-harvest trial and one decline curve trial.

Atonik was applied twice to the treated oilseed rape plots at a nominal rate of 1.0 L product/ha.
Sodium 5-Nitroguaiacolate was applied with an application rate of 1 g/ha for the first and second application.
Sodium o-Nitrophenolate was applied with an application rate of 2 g/ha for the first and second application.
Sodium p-Nitrophenolate was applied with an application rate of 3 g/ha for the first and second application.
The trial was carried out in Poland and Germany. For the decline trial, samples were taken at day 0, 7, 14, 20 and 28 after the last application. For the at-harvest trials, samples were taken at day 28 after the last application. The residue study was thus conducted in accordance with the cGAP.
The field phase was performed by Eurofins Agroscience Services Ltd., Melbourne, United Kingdom, whereas the analytical work associated with the study was performed by EAG Laboratories, 89081 Ulm, Germany.

Crop specimens were analyzed for residues of Na 5-NG, Na *o*-NP and Na *p*-NP using an HPLC method described in the analytical part of the study.
Principle of the method:

Na 5-NG was extracted twice with acetonitrile. Extracts were combined, diluted with water (1x1; v/v) and filtered. Separation was carried out on LC, followed by triplequadrupole mass spectrometric detection (MS/MS).
Na o-NP was extracted twice with acetonitrile. Extracts were combined, diluted with water (1x1; v/v) and filtered. Separation was carried out on LC, followed by triplequadrupole mass spectrometric detection (MS/MS).
Na p-NP was extracted twice with acetonitrile. Extracts were combined, diluted with water (1x1; v/v) and filtered. Separation was carried out on LC, followed by triplequadrupole mass spectrometric detection (MS/MS).
The limit of quantification (LOQ) for Na 5-NG and Na o-NP was 0.01 mg/kg in whole plant and seeds. For Na p-NP the LOQ was 0.01 mg/kg in seeds and 0.05 mg/kg in whole plant. The limit of detection (LOD) for Na 5-NG and Na o-NP was set at 0.003 mg/kg in whole plant and seeds. For Na p-NP the LOD was set at 0.003 mg/kg in seeds and 0.015 mg/kg in whole plants.
For Na 5-NG, the maximum sampling to analysis interval at -18°C was 107 days for seeds and 244 days for whole plant. The maximum extraction to quantification interval for Na 5-NG was 1 day for seeds and for whole plant. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na 5-NG in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na o-NP, the maximum sampling to analysis interval at -18°C was 107 days for seeds and 244 days for whole plant. The maximum extraction to quantification interval for Na o-NP was 1 day for seeds and for whole plant. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na o-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na p-NP, the maximum sampling to analysis interval at -18°C was 107 days for seeds and 244 days for whole plant. The maximum extraction to quantification interval for Na p-NP was 1 day for seeds and for whole plant. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na p-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.

Results and discussions
Residues in untreated control samples were below the limit of quantification (LOQ = 0.01 mg/kg).
Residues of Na 5-NG, Na o-NP, Na p-NP in oilseed rape seeds at harvest (PHI 28 days) are below the limit of quantification (LOQ = 0.01 mg/kg), when Atonik is applied twice at a nominal rate of 0.6 g/ha Na 5-NG, 1.2 g/ha Na o-NP and 1.8 g/ha Na p-NP with a PHI of 28 days in Northern Europe.

A 2.1.3.1.4 Study S19-00203

Comments of zRMS:	One residue trial on oilseed rape was conducted in northern Europe to determine residue of Na 5-NG, Na o-NP and Na p-NP. Oilseed rape was treated twice at application rate of 1.0 L product/ha (corresponding to 1 g/ha sodium 5-nitroguaiacolate + 2 g/ha sodium o-nitrophenolate + 3 g/ha sodium p-nitrophenolate) with 7 days interval between applications. Samples of oilseed rape from the treated plots were taken by hand 0, 7, 14, 22 and 30 days (at normal commercial harvest: NCH) after the final application. <u>Analytical Method</u> Sample extraction and determination of residues were performed based on the method used in EAS Study S12-04715 after adaption and concurrent validation for oilseed rape (OSR) whole plant and seed EAG Study P 4928 G). Quantification was performed by use of LC-MS/MS detection. The method is fully validated with the requirement of SANTE/2020/12830, Rev.1, 24. February 2021 in this report for oilseed rape. All residue samples were measured in one analytical set per matrix, which included in total a number of at least three fortifications at LOQ and three fortifications at 10xLOQ for procedural recoveries.

	The limit of quantification (LOQ) of the analytical method was 0.01 mg/kg per analyte (expressed as sodium salt) for seed matrix. For determination of the analytes in OSR whole plant the LOQ was set to 0.01 mg/kg (for Na 5-NG and Na o-NP) and 0.05 mg/kg (for Na p-NP). The mean recovery was between 70% and 110% with a relative standard deviation lower than 20%. Maximum storage period was 120 days (4 months) for seeds and 151 days (5 months) for whole plant. According to the OECD 506 oilseed rape seed belongs to high oil commodity and whole plant belongs to high water commodity. The sodium nitrocompounds are stable for a period of 9 months in high oil content and for a period of 3 months in high water content commodities. So stability data are not cover the storage time for whole plant. Results: The residues of Na 5-NG, Na o-NP and Na p-NP in oilseed rape seeds at harvest were <0.01 mg/kg. The study is acceptable.

Data point addressed:	KCA 6. 3.1-5
Author(s) (year):	White, T. (2020)
Title:	ATONIK - STUDY TO GENERATE SAMPLES OF OILSEED RAPE FOLLOWING TWO APPLICATIONS OF ATONIK. ONE TRIAL IN NORTHERN EUROPE DURING 2019
Laboratory report / project Number (Doc. No.):	S19-00203
Testing facility:	Eurofins Agroscience Services Ltd., Melbourne, United Kingdom
Published:	No
Test guideline used:	OECD No. 509, 7025/VI/95 rev. 10.3 (2017)
Deviations:	None
Previous evaluation:	No, not previously submitted
GLP:	Yes; certified by the Department of Health of the Government of the United Kingdom
Acceptability/Reliability:	Yes

Analytical part:

Data point addressed:	KCA 6. 3.1-6
Author(s) (year):	Guserle, R. (2020)
Title:	ANALYSIS OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM ORTHO-NITROPHENOLATE AND SODIUM PARA-NITROPHENOLATE IN FIELD SAMPLES ORIGINATING FROM A FIELD STUDY PERFORMED UNDER EAS STUDY CODE S19-00203 WITH TWO APPLICATIONS OF ATONIK IN OILSEED RAPE AT ONE TRIAL IN NORTHERN EUROPE DURING 2019
Laboratory report / project Number (Doc. No.):	P 5295 G S19-00203
Testing facility:	EAG Laboratories GmbH, Ulm, Germany
Published:	No
Test guideline used:	SANCO/3029/99 rev. 4, SANCO/825/00 rev. 8.1, OECD Testing and assessment No. 72 and Series on Pesticides No. 39 , ENV/JM/MONO(2007)17
Deviations:	None
Previous evaluation:	
GLP:	Yes; certified by LUBW Landesanstalt für Umwelt Baden-Württemberg, Karlsruhe
Acceptability/Reliability:	Yes

Materials and methods

Description:	**ATONIK**		
Components:	Na-5-NG	Na *o*-NP	Na *p*-NP

CAS #:	67233-85-6	824-39-5	824-78-2
Lot/Batch #:	CG7-101		
Content of a.s. (actual):	0.095 % (w/w)	0.21 % (w/w)	0.31 % (w/w)
Stability of test compound (Expiry date):	May 2020		

The residue study on oilseed rape has been performed during 2019 at one field site in the United Kingdom. This residue study provides data relevant to conditions in the Northern European region. The experimental setup includes one decline curve trial.

Atonik was applied twice to the treated oilseed rape plot at a nominal rate of 1.0 L product/ha.
Sodium 5-Nitroguaiacolate was applied with an application rate of 1 g/ha for the first and second application.
Sodium o-Nitrophenolate was applied with an application rate of 2 g/ha for the first and second application.
Sodium p-Nitrophenolate was applied with an application rate of 3 g/ha for the first and second application.
The trial was carried out in The United Kingdom. Samples of whole plant were taken at day 0, 7, 14 and 22 and samples of seeds at day 30 after the last application. The residue study was thus conducted in accordance with the cGAP.
The field phase was performed by Eurofins Agroscience Services Ltd., Melbourne, United Kingdom. The analytical phase was conducted in the EAG Laboratories GmbH in Ulm, Germany.

Crop specimens were analyzed for residues of Na 5-NG, Na *o*-NP and Na *p*-NP using an HPLC method described in the analytical part of the study.
Principle of the method:
Na 5-NG was extracted twice with acetonitrile. Extracts were combined, diluted with water (1x1; v/v) and filtered. Separation was carried out on LC, followed by triplequadrupole mass spectrometric detection (MS/MS).
Na o-NP was extracted twice with acetonitrile. Extracts were combined, diluted with water (1x1; v/v) and filtered. Separation was carried out on LC, followed by triplequadrupole mass spectrometric detection (MS/MS).
Na p-NP was extracted twice with acetonitrile. Extracts were combined, diluted with water (1x1; v/v) and filtered. Separation was carried out on LC, followed by triplequadrupole mass spectrometric detection (MS/MS).
In OSR seeds, the limit of quantification (LOQ) for Na 5-NG, Na o-NP and Na p-NP was 0.01 mg/kg in seeds. The limit of detection (LOD) was set at 0.003 mg/kg per analyte.
In OSR whole plant, the LOQ of the method was 0.01 mg/kg for Na 5-NG and Na o-NP and 0.05 mg/kg for Na p-NP. The LOD was set at 0.003 mg/kg for Na 5-NG and Na o-NP and 0.015 mg/kg for Na p-NP.
For Na 5-NG, the maximum sampling to analysis interval at -18°C was 151 days for whole plant and 120 days for seeds. The maximum extraction to quantification interval for Na 5-NG was < 1 day for whole plant and seeds. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na 5-NG in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na o-NP, the maximum sampling to analysis interval at -18°C was 151 days for whole plant and 120 days for seeds. The maximum extraction to quantification interval for Na o-NP was < 1 day for for whole plant and seeds. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na o-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na p-NP, the maximum sampling to analysis interval at -18°C was 151 days for whole plant and 120 days for seeds. The maximum extraction to quantification interval for Na p-NP was < 1 day for whole plant and seeds. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na p-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.

Results and discussions
Residues in untreated control samples were below the limit of quantification (LOQ = 0.01 mg/kg).
Residues of Na 5-NG, Na o-NP, Na p-NP in oilseed rape seeds at harvest (PHI 28 days) are below the limit of quantification (LOQ = 0.01 mg/kg), when Atonik is applied twice at a nominal rate of 0.6 g/ha Na 5-NG, 1.2 g/ha Na o-NP and 1.8 g/ha Na p-NP with a PHI of 28 days in Northern Europe.

Overall review about the results of residue studies in oilseed rape seeds in Northern Europe:
Residue data on 7 trials were performed according to a GAP with a higher application rate. Residues of Na 5-NG, Na o-NP, Na p-NP in oilseed rape seeds at harvest (PHI 28 days) are below the limit of quantification (LOQ = 0.01 mg/kg), when Atonik is applied twice at a nominal rate of 0.6 g/ha Na 5-NG, 1.2 g/ha Na o-NP and 1.8 g/ha Na p-NP with a PHI of 28 days in Northern Europe. No further data are needed.

Details of the trials and the analytical results can be found in the Tier 1 summary forms provided below.

Table A 2: Residue trials on oilseed rape (NEU)

Table A 2-1 Supervised residue trials in oilseed rape – Northern Europe

Reference: DETERMINATION OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM *O*-NITROPHENOLATE AND SODIUM *P*-NITROPHENOLATE IN OIL SEED RAPE (RAC SEEDS) FOLLOWING TWO TREATMENTS WITH ATONIK IN NORTHERN EUROPE, 2005; Diehl, M., 2006, report number: A06028, KCA 6.3.1-1

GLP:	Yes	**Sample storage conditions:**	Max. 63 days (all analytes) below -18°C (sampling till analysis)
Crop/crop group:	Oilseed rape / Oilseeds	**Analytical method:**	HPLC-MS/MS, validated
Indoor/Glasshouse/Outdoor:	Outdoor	**Limit of Quantification (mg/kg):**	0.01 mg/kg (all analytes)
Formulation *(e.g.* WP):	SL	**Limit of detection (mg/kg):**	0.005 mg/kg (all analytes)
Content of active components (nominal)	Sodium 5-Nitroguaiacolate: 0.12 % Sodium *o*-Nitrophenolate: 0.22 % Sodium *p*-Nitrophenolate: 0.32 %	**Residues calculated as:**	Sodium 5-Nitroguaiacolate (Na 5-NG) Sodium *o*-Nitrophenolate (Na *o*-NP) Sodium *p*-Nitrophenolate (Na *p*-NP)

1	2	3	4	5			6	7	8	9			10	11
Trial No./ Location/ Year	**Commodity/ Variety**	**Date of 1.Sowing or planting 2.Flowering 3. Harvest**	**Method of treatment**	**Application rate per treatment Sodium 5-Nitroguaiacolate Sodium *o*-Nitrophenolate Sodium *p*-Nitrophenolate**			**Dates of treatment or no. of treatments and last date**	**Growth stage at last treatment or date**	**Portion analyzed**	**Residues [mg/kg]**			**PHI [days]**	**Remarks**
	(a)	**(b)**	**(c)**	**[g a.s./hL]**	**Water [L/ha]**	**[g a.s./ha]**	**(d)**	**(e)**	**(a)**	**Na 5-NG**	**Na *o*-NP**	**Na *p*-NP**	**(f)**	**(g)**
A/GE/G/05/172 74909 Meckesheim, Baden-Württemberg Germany 2005	Oilseed rape / Express	1. 04/09/2004 2. 16/04 – 20/05 2005 3. 15/07/2005	Broadcast foliar application with a boom sprayer	0.40 /0.73 /1.07 0.40 /0.74 /1.07	278 322	1.11 /2.05 /2.97 1.29 /2.37 /3.45	2 18 June 2005	78	Seeds	< 0.01	< 0.01	< 0.01	27	No residues >LOQ were found in any of the untreated samples
A/GE/G/05/173 75248 Ölbronn-Dürrn, Baden-Württemberg Germany 2005	Oilseed rape / Pioneer PR 46 D01	1. 01/09/2004 2. 09 – 20/05/2005 3. 27/07/2005	Broadcast foliar application with a boom sprayer	0.40 /0.74 /1.07 0.40 /0.74 /1.07	307 307	1.23 /2.26 /3.28 1.23 /2.26 /3.28	2 20 June 2005	79 - 80	Seeds	< 0.01	< 0.01	< 0.01	37	No residues >LOQ were found in any of the untreated samples

(a) According to EPPO code (formerly BAYER code)
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting, etc., overall, broadcast, type of equipment used must be indicated
(d) Year must be indicate;
(e) BBCH Monograph, Growth stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4
(f) Minimum number of days after last application (DALA) (label pre-harvest interval, PHI, underline)
(g) Remarks may include: Climatic conditions; Reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date
Limit of quantification (LOQ) = 0.01 mg/kg for all analytes

Table A 2-2 **Supervised residue trials in oilseed rape – Northern Europe**

Reference: DETERMINATION OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM *ORTHO*-NITROPHENOLATE AND SODIUM *PARA*-NITROPHENOLATE AFTER TWO APPLICATIONS OF ATONIK IN OILSEED RAPE AT 1 SITE IN NORTHERN EUROPE 2008 Oxspring, S., 2010, report number: S08-01067, KCA 6.3.1-2

GLP:	Yes	**Sample storage conditions:**	Max. 233 days (all analytes) below -18°C (sampling till extraction) Max. 1 day (all analytes) below -18°C (extraction till analysis)
Crop/crop group:	Oilseed rape / Oilseeds	**Analytical method:**	HPLC-MS/MS, validated
Indoor/Glasshouse/Outdoor:	Outdoor	**Limit of Quantification (mg/kg):**	0.01 mg/kg (all analytes)
Formulation *(e.g.* WP):	SL	**Limit of detection (mg/kg):**	30% of the LOQ (all analytes)
Content of active components (nominal)	Sodium 5-Nitroguaiacolate: 0.12 % Sodium *o*-Nitrophenolate: 0.22 % Sodium *p*-Nitrophenolate: 0.33 %	**Residues calculated as:**	Sodium 5-Nitroguaiacolate (Na 5-NG) Sodium *o*-Nitrophenolate (Na *o*-NP) Sodium *p*-Nitrophenolate (Na *p*-NP)

1	2	3	4	5			6	7	8	9			10	11
Trial No./ Location/ Year	**Commodity/ Variety**	**Date of 1.Sowing or planting 2.Flowering 3. Harvest**	**Method of treatment**	**Application rate per treatment Sodium 5-Nitroguaiacolate Sodium *o*-Nitrophenolate Sodium *p*-Nitrophenolate**			**Dates of treatment or no. of treatments and last date**	**Growth stage at last treatment or date**	**Portion analyzed**	**Residues [mg/kg]**			**PHI [days]**	**Remarks**
	(a)	**(b)**	**(c)**	**[g a.s./hL]**	**Water [L/ha]**	**[g a.s./ha]**	**(d)**	**(e)**	**(a)**	**Na 5-NG**	**Na *o*-NP**	**Na *p*-NP**	**(f)**	**(g)**
S08-01067-01 Weilkopolska, 64560, Poland 2008	Oilseed rape / Californium	1. 27/08/2007 2. 27/04 – 16/05/2008 3. 19/07/2008	Foliar application with a boom sprayer	0.40 /0.73 /1.07 0.40 /0.74 /1.07	193 217	0.97 /1.93 /2.90 1.09 /2.17 /3.26	2 19 June 2008	75	Seeds	< 0.01	< 0.01	< 0.01	30	No residues >LOQ were found in any of the untreated samples

(a) According to EPPO code (formerly BAYER code)
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting, etc., overall, broadcast, type of equipment used must be indicated
(d) Year must be indicated
(e) BBCH Monograph, Growth stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4
(f) Minimum number of days after last application (DALA) (label pre-harvest interval, PHI, underline)
(g) Remarks may include: Climatic conditions; Reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date
Limit of quantification (LOQ) = 0.01 mg/kg for all analytes

Table A 2-3 **Supervised residue trials in oilseed rape – Northern Europe**

Reference: ATONIK - STUDY TO GENERATE SPECIMENS OF OILSEED RAPE FOLLOWING TWO APPLICATIONS OF ATONIK. THREE TRIALS IN NORTHERN EUROPE DURING 2018 (FINAL REPORT AMENDMENT 1)
White, T., 2019, report number: S18-05054, KCA 6.3.1-3 – Field part

ANALYSIS OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM ORTHO-NITROPHENOLATE AND SODIUM PARA-NITROPHENOLATE IN FIELD SAMPLES ORIGINATING FROM A FIELD STUDY PERFORMED UNDER EAS STUDY CODE S18-05054 WITH TWO APPLICATIONS OF ATONIK IN OILSEED RAPE AT THREE TRIALS IN NORHTERN EUROPE DURING 2018
Guserle, R., 2019, report number: P 4928 G, S18-05054, KCA 6.3.1-4 – Analytical part

GLP:	Yes	**Sample storage conditions:**	Max. 244 days: whole plant (all analytes) Max. 107 days: seeds (all analytes) below -18°C (sampling till extraction) Max. 1 day : whole plant and seeds (all analytes) below -18°C (extraction till analysis)
Crop/crop group:	Oilseed rape / Oilseeds	**Analytical method:**	LC-MS/MS, validated
Indoor/Glasshouse/Outdoor:	Outdoor	**Limit of Quantification (mg/kg):**	Seeds (all analytes): 0.01 mg/kg Whole plant (Na 5-NG, Na *o*-NP): 0.01 mg/kg Whole plant (Na *p*-NP): 0.05 mg/kg
Formulation *(e.g.* WP):	SL	**Limit of detection (mg/kg):**	Seeds (all analytes): 0.003 mg/kg Whole plant (Na 5-NG, Na *o*-NP): 0.003 mg/kg Whole plant (Na *p*-NP): 0.015 mg/kg)
Content of active components (nominal)	Sodium 5-Nitroguaiacolate: 0.12 % Sodium *o*-Nitrophenolate: 0.22 % Sodium *p*-Nitrophenolate: 0.33 %	**Residues calculated as:**	Sodium 5-Nitroguaiacolate (Na 5-NG) Sodium *o*-Nitrophenolate (Na *o*-NP) Sodium *p*-Nitrophenolate (Na *p*-NP)

1	2	3	4	5			6	7	8	9			10	11
Trial No./ Location/ Year	**Commodity/ Variety**	**Date of 1.Sowing or planting 2.Flowering 3. Harvest**	**Method of treatment**	**Application rate per treatment Sodium 5-Nitroguaiacolate Sodium *o*-Nitrophenolate Sodium *p*-Nitrophenolate**			**Dates of treatment or no. of treatments and last date**	**Growth stage at last treatment or date**	**Portion analyzed**	**Residues [mg/kg]**			**PHI [days]**	**Remarks**
	(a)	**(b)**	**(c)**	**[g a.s./hL]**	**Water [L/ha]**	**[g a.s./ha]**	**(d)**	**(e)**	**(a)**	**Na 5-NG**	**Na *o*-NP**	**Na *p*-NP**	**(f)**	**(g)**
S18-05054-02 Böblingen, Baden Württemberg, Germany 2018	Oilseed rape / Müller 24	1. 23/08/2018 2. 13/04 - 02/05/2018 3. 27/07/2018	Foliar application with a boom sprayer	0.24 /0.67/1.01	297	1 /2 /3	2 29 June 2018	85	Whole plant	< 0.01	< 0.01	< 0.05	0	No residues >LOQ were found in any of the untreated samples
										< 0.01	< 0.01	< 0.05	7	
										< 0.01	< 0.01	< 0.05	14	
				0.34 /0.68/1.02	295	1 /2 /3				< 0.01	< 0.01	< 0.05	20	
									Seeds	< 0.01	< 0.01	< 0.01	28	

S18-05054-03 21698, Ohrensen, Lower Saxony, Germany 2018	Oilseed rape / Alvaro KWS	1. 05/09/2017 2. Not applicable 3. 25/07/2018	Foliar application with a boom sprayer	0.35 /0.71/1.06 0.35 /0.69/1.04	283 288	1 /2 /3 1 /2 /3	2 27 June 2018	87	Seeds	< 0.01	< 0.01	< 0.01	28	No residues >LOQ were found in any of the untreated samples
S18-05054-04 64-520, Gaj Maly, Wielkopolskie, Poland 2018	Oilseed rape / California	1. 24/08/2017 2. 20/04 - 12/05/2018 3. 25/07/2018	Foliar application with a boom sprayer	0.32 /0.64/0.96 0.34 /0.67/1.01	314 298	1. /2 /3 1 /2 /3	2 27 June 2018	83	Seeds	< 0.01	< 0.01	< 0.01	28	No residues >LOQ were found in any of the untreated samples

(a) According to EPPO code (formerly BAYER code)
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting, etc., overall, broadcast, type of equipment used must be indicated
(d) Year must be indicate;
(e) BBCH Monograph, Growth stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4
(f) Minimum number of days after last application (DALA) (label pre-harvest interval, PHI, underline)
(g) Remarks may include: Climatic conditions; Reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

Limit of quantification (LOQ for seeds) = 0.01 mg/kg (all analytes)
Limit of quantification (LOQ for whole plant) = 0.01 mg/kg for Sodium 5-Nitroguaiacolate and Sodium *o*-Nitrophenolate and 0.05 mg/kg for Sodium *p*-Nitrophenolate)

Table A 2-4 **Supervised residue trials in oilseed rape – Northern Europe**

Reference: ATONIK – STUDY TO GENERATE SAMPLES OF OILSEED RAPE FOLLOWING TWO APPLICATIONS OF ATONIK. ONE TRIAL IN NORTHERN EUROPE DURING 2019
White, T., 2020, report number: S19-00203, KCA 6.3.1-5 – Field part
ANALYSIS OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM *ORTHO*-NITROPHENOLATE AND SODIUM *PARA*-NITROPHENOLATE IN FIELD SAMPLES ORIGINATING FROM A FIELD STUDY PERFORMED UNDER EAS STUDY CODE S19-00203 WITH TWO APPLICATIONS OF ATONIK IN OILSEED RAPE AT ONE TRIAL IN NORTHERN EUROPE DURING 2019
Guserle, R., 2019, report number: P 5295 G, S19-00203, KCA 6.3.1-6 – Analytical part

GLP: Yes

Crop/crop group: Oilseed rape / Oilseeds

Indoor/Glasshouse/Outdoor: Outdoor

Formulation *(e.g.* WP): SL

Content of active components (nominal)
Sodium 5-Nitroguaiacolate: 0.12 %
Sodium *o*-Nitrophenolate: 0.22 %
Sodium *p*-Nitrophenolate: 0.33 %

Sample storage conditions:
Max. 151 days: whole plant (all analytes)
Max. 120 days: seeds (all analytes) below -18°C (sampling till extraction)
< 1 day : whole plant and seeds (all analytes) below -18°C (extraction till analysis)

Analytical method: LC-MS/MS, validated

Limit of Quantification (mg/kg):
Seeds (all analytes): 0.01 mg/kg
Whole plant (Na 5-NG, Na *o*-NP): 0.01 mg/kg
Whole plant (Na *p*-NP): 0.05 mg/kg

Limit of detection (mg/kg):
Seeds (all analytes): 0.003 mg/kg
Whole plant (Na 5-NG, Na *o*-NP): 0.003 mg/kg
Whole plant (Na *p*-NP): 0.015 mg/kg

Residues calculated as:
Sodium 5-Nitroguaiacolate (Na 5-NG)
Sodium *o*-Nitrophenolate (Na *o*-NP)
Sodium *p*-Nitrophenolate (Na *p*-NP)

1	2	3	4	5			6	7	8	9			10	11
Trial No./ Location/ Year	**Commodity/ Variety**	**Date of 1.Sowing or planting 2.Flowering 3. Harvest**	**Method of treatment**	**Application rate per treatment Sodium 5-Nitroguaiacolate Sodium *o*-Nitrophenolate Sodium *p*-Nitrophenolate**			**Dates of treatment or no. of treatments and last date**	**Growth stage at last treatment or date**	**Portion analyzed**	**Residues [mg/kg]**			**PHI [days]**	**Remarks**
	(a)	**(b)**	**(c)**	**[g a.s./hL]**	**Water [L/ha]**	**[g a.s./ha]**	**(d)**	**(e)**	**(a)**	**Na 5-NG**	**Na *o*-NP**	**Na *p*-NP**	**(f)**	**(g)**
S19-00203 -01 WN8 9TH, Bickerstaffe, Lancashire, United Kingdom 2019	Oilseed rape / Graton	1. 15/09/2018 2. not recorded 3. 17/07/2019	Foliar application with a boom sprayer	0.24 /0.48/0.72 0.26 /0.51 /0.77	418 391	1 /2 /3 1 /2 /3	2 17June 2019	79	Whole plant	< 0.01	< 0.01	< 0.05	0	No residues >LOQ were found in any of the untreated samples
										< 0.01	< 0.01	< 0.05	7	
										< 0.01	< 0.01	< 0.05	14	
										< 0.01	< 0.01	< 0.05	22	
									Seeds	< 0.01	< 0.01	< 0.01	30	

(a) According to EPPO code (formerly BAYER code)
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting, etc., overall, broadcast, type of equipment used must be indicated
(d) Year must be indicated
(e) BBCH Monograph, Growth stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4
(f) Minimum number of days after last application (DALA) (label pre-harvest interval, PHI, underline)
(g) Remarks may include: Climatic conditions; Reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

Limit of quantification (LOQ for seeds) = 0.01 mg/kg (all analytes)
Limit of quantification (LOQ for whole plant) = 0.01 mg/kg for Sodium 5-Nitroguaiacolate and Sodium *o*-Nitrophenolate and 0.05 mg/kg for Sodium *p*-nitrophenolate

A 2.1.3.2 Wheat

Table A 3: 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)
Intended GAP NEU	1x	0,6 g / ha Na-5NG 1,2 g/ ha Na-oNP 1,8 g/ ha Na-pNP	-	BBCH 21-49	28
Critical GAP* NEU: Poland	2x	0,6 g / ha Na-5NG 1,2 g/ ha Na-oNP 1,8 g/ ha Na-pNP	20-50 days	BBCH 21-49	54

*Please see Appendix 1

A 2.1.3.2.1 A05995

Comments of zRMS:	Three residue trials on cereals were conducted in northern Europe to determine the residues of Na 5-NG, Na o-NP and Na p-NP, but only two of the three were independent trials. Cereals were treated twice at application rate of 0.6 L product/ha (corresponding to 0.6 g/ha sodium 5-nitroguaiacolate + 1.2 g/ha sodium o-nitrophenolate + 1.8 g/ha sodium p-nitrophenolate) with 20-28 days interval between applications. Straw and grain were collected at commercial harvest (55 to 68 days after last application). All procedures for specimen preparation and analysis for Sodium 5-Nitroguaiacolate, Sodium o-Nitrophenolate and Sodium p-Nitrophenolate followed as validated under RCC Study No. A07356 ("Development and Validation of a Residue Analytical Method for Sodium 5-Nitroguaiacolate, Sodium o-Nitrophenolate and Sodium p-Nitrophenolate (as Active Ingredients in Atonik Formulated Product) in wheat grain and straw. The limit of quantification (LOQ) in grain was 0.01 mg/kg for Na 5-NG, Na o-NP and Na p-NP. In straw, the LOQ was 0.02 mg/kg for each compound. In analytical report for cereals, only one recovery determination at LOQ and one recovery at 10xLOQ has been presented for Na 5-NG, Na o-NP and Na p-NP respectively. This is not fully with the requirement of SANTE/2020/12830, Rev.1, 24. February 2021. Maximum storage period – 157 days (5 months). <u>Results:</u> The residues of Na 5-NG, Na o-NP and Na p-NP at harvest were <0.01 mg/kg in grain and <0.02 mg/kg in straw. The study is acceptable.

Data point addressed:	KCA 6.3.1-7
Author(s) (year):	Diehl, M. (2006)
Title:	DETERMINATION OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM O-NITROPHENOLATE AND SODIUM P-NITROPEHNOLATE IN CEREALS (RAC STRAW AND GRAIN) FOLLOWING TWO TREATMENTS WITH ATONIK IN NORTHERN EUROPE 2005
Laboratory report / project Number (Doc. No.):	A05995
Testing facility:	RCC Ltd, Environmental Chemistry & Pharmanalytics Division, Itingen, Switzerland
Published:	No
Test guideline used:	96/68/EC (1996), 7029/VI/95 rev. 5 (1997), in accordance to OECD 509 (2009)
Deviations:	None
Previous evaluation:	Yes; evaluated and accepted in dRR STEP 2 in Poland, Czech Republic, Slovakia, Hungary
GLP:	Yes; certified by Swiss Federal Office of Public Health, Bern

Acceptability/Reliability:	Yes

Materials and methods

Description:	**ATONIK**		
Components:	Na 5-NG	Na *o*-NP	Na *p*-NP
CAS #:	67233-85-6	824-39-5	824-78-2
Lot/Batch #:	2005-AC-50628		
Content of a.s. (actual):	0.12 %	0.22 %	0.32 %
Stability of test compound (Expiry date):	May 2008		

The residue study on spring barley (1x) and spring wheat (2x) has been performed at two field sites in Germany. This residue study provides data relevant to conditions in the Northern European region. The experimental setup includes two at-harvest trials.

Atonik was applied twice to the treated spring barley and wheat plots at a nominal rate of 0,6 L product/ha at BBCH 27-30 (1st application) and BBCH 49-51 (2nd application) at interval of 20 days.
Sodium 5-Nitroguaiacolate was applied in the range of 0.71 and 0.79 g/ha for the first application and in the range of 0.71 and 0.78 g/ha for the second application.
Sodium o-Nitrophenolate was applied in the range of 1.31 and 1.46 g/ha for the first application and in the range of 1.31 and 1.43 g/ha for the second application.
Sodium p-Nitrophenolate was applied in the range of 1.91 and 2.12 g/ha for the first application and in the range of 1.91 and 2.08 g/ha for the second application.
All trials were carried out in the Germany. Samples (grain and straw) were taken at harvest, at PHI 65 for barley and PHI 55 and 68 for wheat.
The field phase was performed by GAB Biotechnology GmbH, Stade, Germany, whereas the analytical work associated with the studies was performed by RCC Ltd., Itingen, Switzerland.

Crop specimens were analyzed for residues of Na 5-NG, Na *o*-NP and Na *p*-NP using an HPLC method described below.
Principle of the method:
Na 5-NG was extracted from homogenised grain and straw with methanol. Extracts were cleaned up by solid phase extraction. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).
Na o-NP was extracted from homogenised grain and straw with methanol. Extracts were cleaned up by solid phase extraction. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).
Na p-NP was extracted from homogenised grain and straw with methanol. Extracts were cleaned up by solid phase extraction. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).
The limit of quantification (LOQ) for Na 5-NG, Na o-NP and Na p-NP was 0.01 mg/kg in grain and 0.02 mg/kg in straw. The limit of detection (LOD) for Na 5-NG, Na o-NP and Na p-NP was set at 0.005 mg/kg in grain and 0.01 mg/kg in straw.
For Na 5-NG, the maximum sampling to analysis interval at -18°C was 157 days in both grain and straw. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na 5-NG in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na o-NP, the maximum sampling to analysis interval at -18°C was 157 days in both grain and straw. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na o-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na p-NP, the maximum sampling to analysis interval at -18°C was 157 days in both grain and straw. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na p-NP in the samples extract. All recoveries were within

the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.

Results and discussions
Residues in untreated control samples were not detectable.
Residues of Na 5-NG, Na o-NP, Na p-NP in spring barley grain and straw at harvest (PHI 65 days) are below the limit of quantification (LOQ = 0.01 mg/kg for grain and LOQ = 0.02 mg/kg for straw), in spring wheat grain and straw at harvest (PHI 68 respective 55 days) are below the limit of quantification (LOQ = 0.01 mg/kg for grain and LOQ = 0.02 mg/kg for straw), when Atonik is applied twice at a nominal rate of 0.6 g/ha Na 5-NG, 1.2 g/ha Na o-NP and 1.8 g/ha Na p-NP with a PHI of 55 days in Northern Europe.

A 2.1.3.2.2 S18-05052

Comments of zRMS:	Three residue trials on cereals were conducted in northern Europe to determine the residues of Na 5-NG, Na o-NP and Na p-NP. Cereals were treated twice at application rate of 0.6 L product/ha (corresponding to 0.6 g/ha sodium 5-nitroguaiacolate + 1.2 g/ha sodium o-nitrophenolate + 1.8 g/ha sodium p-nitrophenolate) with 10-11 days interval between applications. Samples of wheat were taken 0, 6-8, 14-15, 21-22 and 28 (NCH) days after the final application. Analytical Method Sample extraction and determination of residues were performed based on the method used in EAS Study S12-04715 after adaption and prospective validation for winter wheat whole plant, grain and straw. Quantification was performed by use of LC-MS/MS detection. The method is fully validated with the requirement of SANTE/2020/12830, Rev.1, 24. February 2021 in this report for cereals (validated with 1 reagent blank, 2 control blanks, 5 fortifications with 1xLOQ and 5 fortifications with 10xLOQ for grain, whole plant and straw matrices, respectively). For determination of the analytes in winter wheat whole plant and straw the limit of quantification (LOQ) was set to 0.01 mg/kg (for sodium 5-nitroguaiacolate and sodium ortho-nitrophenolate) and 0.10 mg/kg (for sodium para-nitrophenolate). The mean recovery was between 70% and 110% with a relative standard deviation lower than 20%. Maximum storage period – 122 days in grain and 267 days in whole plant and 241 days in straw. According to the OECD 506 wheat grain and straw belongs to dry commodity and whole plant belongs to high water commodity. The sodium nitrocompounds are stable for a period of 9 months in dry commodity and for a period of 3 months in high water content commodities. So stability data are not cover the storage time for whole plant. Results: Residues of Na 5-NG, Na o-NP, Na p-NP in wheat whole plant, grain and straw at harvest (PHI 28 days) are below the limit of quantification (LOQ) of 0.01 mg/kg in grain for Na 5-NG, Na o-NP and Na p-NP as well as below LOQ of 0.01 mg/kg for Na 5-NG, Na o-NP and 0.1 mg/kg for Na p-NP in wheat whole plant and straw. The study is acceptable.

Data point addressed:	KCA 6. 3.1-8
Author(s) (year):	White, T. (2018)
Title:	ATONIK – STUDY TO GENERATE SPECIMENS OF WINTER WHEAT FOLLOWING TWO APPLICATIONS OF ATONIK. THREE TRIALS IN NORTHERN EUROPE DURING 2018
Laboratory report / project Number (Doc. No.):	S18-05052
Testing facility:	Eurofins Agroscience Services Ltd., Melbourne, United Kingdom
Published:	No

Test guideline used:	OECD No. 509, 7025/VI/95 rev. 5
Deviations:	None
Previous evaluation:	No, not previously submitted
GLP:	Yes; certified by the Department of Health of the Government of the United Kingdom
Acceptability/Reliability:	Yes

Analytical part:

Data point addressed:	KCA 6. 3.1-9
Author(s) (year):	Guserle, R. (2019)
Title:	ANALYSIS OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM ORTHO-NITROPHENOLATE AND SODIUM PARA-NITROPHENOLATE IN FIELD SAMPLES ORIGINATING FROM A FIELD STUDY PERFORMED UNDER EAS STUDY CODE S18-05052 WITH TWO APPLICATIONS OF ATONIK IN WINTER WHEAT AT 3 TRIALS IN NORTHERN EUROPE DURING 2018
Laboratory report / project Number (Doc. No.):	P 4930 G S18-05052
Testing facility:	EAG Laboratories GmbH, Ulm, Germany
Published:	No
Test guideline used:	SANCO/3029/99 rev. 4, OECD Principles on Good Laboratory Practice and Compliance Monitoring ENV/MC/CHEM(98)17
Deviations:	None
Previous evaluation:	No, not previously submitted
GLP:	Yes; certified by LUBW Landesanstalt für Umwelt, Messungen und Naturschutz Baden-Württemberg, Karlsruhe
Acceptability/Reliability:	Yes

Materials and methods

Description:	**ATONIK**		
Components:	Na 5-NG	Na *o*-NP	Na *p*-NP
CAS #:	67233-85-6	824-39-5	824-78-2
Lot/Batch #:	CG7-101		
Content of a.s. (actual):	0.11 %	0.19 %	0.29 %
Stability of test compound (Expiry date):	May 2020		

The residue study on winter wheat has been performed at three field sites in Germany, Poland and UK. This residue study provides data relevant to conditions in the Northern European region. The experimental setup includes two decline and one at-harvest trials.

Atonik was applied twice to the treated winter wheat plots at a nominal rate of 0,6 L product/ha at BBCH 73-75 (1st application) and BBCH 83-85 (2nd application) at interval of 10 days.
Sodium 5-Nitroguaiacolate was applied in the range of 0.57 and 0.63 g/ha for the first application and in the range of 0.58 and 0.62 g/ha for the second application.
Sodium o-Nitrophenolate was applied in the range of 1.13 and 1.27 g/ha for the first application and in the range of 1.17 and 1.23 g/ha for the second application.
Sodium p-Nitrophenolate was applied in the range of 1.7 and 1.9 g/ha for the first application and in the range of 1.75 and 1.85 g/ha for the second application.
Trials were carried out in the Germany, Poland and UK. Samples of wheat were taken 0, 6-8, 14-15, 21-22 and 28 (NCH) days after the final application.
The field phase was performed by Eurofins Agroscience Services, based in Stade - Germany, Kazmierz - Poland and Lathom - UK, whereas the analytical work associated with the studies was performed by EAG Laboratories GmbH, Ulm, Germany.

Crop specimens were analyzed for residues of Na 5-NG, Na *o*-NP and Na *p*-NP using an HPLC method described below.
Principle of the method:

Na 5-NG was extracted from homogenised grain and straw with methanol. Extracts were cleaned up by solid phase extraction. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).
Na o-NP was extracted from homogenised grain and straw with methanol. Extracts were cleaned up by solid phase extraction. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).
Na p-NP was extracted from homogenised grain and straw with methanol. Extracts were cleaned up by solid phase extraction. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).
The limit of quantification (LOQ) for Na 5-NG, Na o-NP and Na p-NP was 0.01 mg/kg in grain, the LOQ in wheat whole plant and straw was 0.01 mg/kg for Na 5-NG, Na o-NP and 0.1 mg/kg for Na p-NP.
The limit of detection (LOD) for Na 5-NG, Na o-NP and Na p-NP was set at 0.003 mg/kg in grain. The limit of detection (LOD) in wheat whole plant and straw was set at 0.003 mg/kg for Na 5-NG, Na o-NP and 0.03 mg/k for Na p-NP.
For Na 5-NG, the maximum sampling to analysis interval at -18°C was 122 days in grain and 267 days in whole plant and 241 days in straw. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na 5-NG in the samples extract. All recoveries were within the range between 70 – 120 %, mean recoveries in the range 70-110% with relative standard deviation(s) below 20% for all matrices and analytes. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na o-NP, the maximum sampling to analysis interval at -18°C was 122 days in grain and 267 days in whole plant and 241 days in straw. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na o-NP in the samples extract. All recoveries were within the range between 70 – 120 %, mean recoveries in the range 70-110% with relative standard deviation(s) below 20% for all matrices and analytes. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na p-NP, the maximum sampling to analysis interval at -18°C was 122 days in grain and 267 days in whole plant and 241 days in straw. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na p-NP in the samples extract. All recoveries were within the range between 70 – 120 %, mean recoveries in the range 70-110% with relative standard deviation(s) below 20% for all matrices and analytes. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.

Results and discussions
No residues above the 30% of the LOQ were detected in untreated control samples, except in straw sample S18-05052-02-007A, where, residues of p-nitrophenol (expressed as sodium salt) were found at the LOD of 0.03 mg/kg. This sample was not used for procedural recovery fortifications.
Residues of Na 5-NG, Na o-NP, Na p-NP in wheat whole plant, grain and straw at harvest (PHI 28 days) are below the limit of quantification (LOQ) of 0.01 mg/kg in grain for Na 5-NG, Na o-NP and Na p-NP as well as below LOQ of 0.01 mg/kg for Na 5-NG, Na o-NP and 0.1 mg/kg for Na p-NP in wheat whole plant and straw, when Atonik is applied twice at a nominal rate of 0.6 g/ha Na 5-NG, 1.2 g/ha Na o-NP and 1.8 g/ha Na p-NP with a PHI of 28 days in Northern Europe.

A 2.1.3.2.3 S19-00202

Comments of zRMS:	One residue trial on wheat was conducted in northern Europe to determine the residues of Na 5-NG, Na o-NP and Na p-NP. Cereals were treated twice at application rate of 0.6 L product/ha (corresponding to 0.6 g/ha sodium 5-nitroguaiacolate + 1.2 g/ha sodium o-nitrophenolate + 1.8 g/ha sodium p-nitrophenolate) with 10 days interval between applications. Samples of wheat were taken 28 (NCH) days after the final application. <u>Analytical Method</u> The analytical method followed the method used in EAS Study S12-04715 with adaptions of volumes of the extraction solvent. Quantification was performed by use of LC-MS/MS detection.

	The method is fully validated with the requirement of SANTE/2020/12830, Rev.1, 24. February 2021 in this report for wheat (validated with 1 reagent blank, 2 control blanks, 5 fortifications with 1xLOQ and 5 fortifications with 10xLOQ for grain, whole plant and straw matrices, respectively). The limit of quantification (LOQ) of the analytical method was 0.01 mg/kg per analyte (expressed as sodium salt) for grain matrix. For determination of the analytes in straw the limit of quantification (LOQ) was 0.01 mg/kg (for sodium 5-nitroguaiacolate and sodium ortho-nitrophenolate) and 0.10 mg/kg (for sodium para-nitrophenolate). The mean recovery was between 70% and 110% with a relative standard deviation lower than 20%. Maximum storage period – 44 days in grain and 45 days in straw. <u>Results:</u> Residues of Na 5-NG, Na o-NP, Na p-NP in wheat grain and straw at harvest (PHI 28 days) are below the limit of quantification (LOQ) of 0.01 mg/kg in grain for Na 5-NG, Na o-NP and Na p-NP as well as below LOQ of 0.01 mg/kg for Na 5-NG, Na o-NP and 0.1 mg/kg for Na p-NP in wheat straw. The study is acceptable.

Data point addressed:	KCA 6. 3.1-10
Author(s) (year):	White, T. (2020)
Title:	ATONIK – STUDY TO GENERATE SPECIMENS OF WINTER WHEAT FOLLOWING TWO APPLICATIONS OF ATONIK. ONE TRIAL IN NORTHERN EUROPE DURING 2019
Laboratory report / project Number (Doc. No.):	S19-00202
Testing facility:	Eurofins Agroscience Services Ltd., Melbourne, United Kingdom
Published:	No
Test guideline used:	OECD (2009): Series on Testing and Assessment No. 64 and Series on Pesticides No. 32; OECD Test Guideline 509: Crop field trials; OECD (2016) Guidance Document ENV/JM/MONO(2011)50/REV1, Second Edition, on Crop Field Trials (Series on Testing and Assessment No. 164 and Series on Pesticides No. 66); SANCO 7525/VI/95, Rev. 10.3, 13/06/17
Deviations:	None
Previous evaluation:	No, not previously submitted
GLP:	Yes; certified by the Department of Health of the Government of the United Kingdom
Acceptability/Reliability:	Yes

Analytical part:

Data point addressed:	KCA 6. 3.1-11
Author(s) (year):	Guserle, R. (2020)
Title:	ANALYSIS OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM ORTHO-NITROPHENOLATE AND SODIUM PARA-NITROPHENOLATE IN FIELD SAMPLES ORIGINATING FROM A FIELD STUDY PERFORMED UNDER EAS STUDY CODE S19-00202 WITH TWO APPLICATIONS OF ATONIK IN WINTER WHEAT AT 1 TRIAL IN NORTHERN EUROPE DURING 2020
Laboratory report / project Number (Doc. No.):	P 5296 G S19-00202
Testing facility:	EAG Laboratories GmbH, Ulm, Germany
Published:	No
Test guideline used:	SANCO/3029/99 rev. 4, OECD Principles on Good Laboratory Practice and Compliance Monitoring ENV/MC/CHEM(98)17
Deviations:	None
Previous evaluation:	No, not previously submitted
GLP:	Yes; certified by LUBW Landesanstalt für Umwelt, Messungen und Naturschutz Baden-Württemberg, Karlsruhe
Acceptability/Reliability:	Yes

Materials and methods

Description:	**ATONIK**		
Components:	Na 5-NG	Na *o*-NP	Na *p*-NP
CAS #:	67233-85-6	824-39-5	824-78-2
Lot/Batch #:	CG7-101		
Content of a.s. (actual):	0.11 %	0.19 %	0.29 %
Stability of test compound (Expiry date):	May 2020		

The residue study on winter wheat has been performed at one field site in UK. This residue study provides data relevant to conditions in the Northern European region. The experimental setup includes one at-harvest trial.

Atonik was applied twice to the treated winter wheat plots at a nominal rate of 0,6 L product/ha at BBCH 75 (1st application) and BBCH 77 (2nd application) at interval of 10 days.
Sodium 5-Nitroguaiacolate was applied at rate of 0.65 g/ha for the first application and at rate of 0.63 and 0.62 g/ha for the second application.
Sodium o-Nitrophenolate was applied at rate of 1.3 g/ha for the first application and at rate of 1.27 and 1.23 g/ha for the second application.
Sodium p-Nitrophenolate was applied at rate of 1.95 g/ha for the first application and at rate of 1.9 g/ha for the second application.
Trial weas carried out in the UK. Samples of wheat were taken 28 (NCH) days after the final application.
The field phase was performed by Eurofins Agroscience Services, based in Lathom - UK, whereas the analytical work associated with the study was performed by EAG Laboratories GmbH, Ulm, Germany.

Crop specimens were analyzed for residues of Na 5-NG, Na *o*-NP and Na *p*-NP using an HPLC method described below.
Principle of the method:
Na 5-NG was extracted from homogenised grain and straw with methanol. Extracts were cleaned up by solid phase extraction. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).
Na o-NP was extracted from homogenised grain and straw with methanol. Extracts were cleaned up by solid phase extraction. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).
Na p-NP was extracted from homogenised grain and straw with methanol. Extracts were cleaned up by solid phase extraction. Separation was carried out on HPLC with column switching, followed by triplequadrupole mass spectrometric detection (MS/MS).
The limit of quantification (LOQ) for Na 5-NG, Na o-NP and Na p-NP was 0.01 mg/kg in grain, the LOQ in wheat whole plant and straw was 0.01 mg/kg for Na 5-NG, Na o-NP and 0.1 mg/kg for Na p-NP.
The limit of detection (LOD) for Na 5-NG, Na o-NP and Na p-NP was set at 0.003 mg/kg in grain. The limit of detection (LOD) in wheat whole plant and straw was set at 0.003 mg/kg for Na 5-NG, Na o-NP and 0.03 mg/k for Na p-NP.
For Na 5-NG, the maximum sampling to analysis interval at -18°C was ~~122~~ 44 days in grain and ~~267 days in whole plant and 241~~ 45 days in straw. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na 5-NG in the samples extract. All recoveries were within the range between 70 – 120 %, mean recoveries in the range 70-110% with relative standard deviation(s) below 20% for all matrices and analytes. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na o-NP, the maximum sampling to analysis interval at -18°C was ~~122~~ 44 days in grain and ~~267 days in whole plant and 241~~ 45 days in straw. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na o-NP in the samples extract. All recoveries were within the range between 70 – 120 %, mean recoveries in the range 70-110% with relative standard deviation(s) below 20% for all matrices and analytes. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na p-NP, the maximum sampling to analysis interval at -18°C was ~~122~~ 44 days in grain and ~~267 days in whole plant and 241~~ 45 days in straw. Procedural recoveries were handled and stored in the same way

and for the same time periods as the analytical samples thereby proving stability of Na p-NP in the samples extract. All recoveries were within the range between 70 – 120 %, mean recoveries in the range 70-110% with relative standard deviation(s) below 20% for all matrices and analytes. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.

Results and discussions
No residues above the 30% of the LOQ were detected in untreated control samples, except in straw sample S18-05052-02-007A, where, residues of p-nitrophenol (expressed as sodium salt) were found at the LOD of 0.03 mg/kg. This sample was not used for procedural recovery fortifications.
Residues of Na 5-NG, Na o-NP, Na p-NP in wheat ~~whole plant,~~ grain and straw at harvest (PHI 28 days) are below the limit of quantification (LOQ) of 0.01 mg/kg in grain for Na 5-NG, Na o-NP and Na p-NP as well as below LOQ of 0.01 mg/kg for Na 5-NG, Na o-NP and 0.1 mg/kg for Na p-NP in wheat ~~whole plant and~~ straw, when Atonik is applied twice at a nominal rate of 0.6 g/ha Na 5-NG, 1.2 g/ha Na o-NP and 1.8 g/ha Na p-NP with a PHI of 28 days in Northern Europe.

Details of the trials and the analytical results can be found in the Tier 1 summary forms provided below.

Table A 4: Residue trials on wheat (NEU)

Table A 4-1 Supervised residue trials in spring barley and spring wheat – Northern Europe

Reference: DETERMINATION OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM O-NITROPHENOLATE AND SODIUM P-NITROPEHNOLATE IN CEREALS (RAC STRAW AND GRAIN) FOLLOWING TWO TREATMENTS WITH ATONIK IN NORTHERN EUROPE 2005
Diehl, M., 2006, report number: A05995, KCA 6.3.1-7

GLP: Yes

Sample storage conditions: Max. 157 days: grain (all analytes)
Max. 157 days: straw (all analytes) below -18°C (sampling till extraction)

Crop/crop group: Spring barley and wheat / cereals

Analytical method: LC-MS/MS, validated

Indoor/Glasshouse/Outdoor: Outdoor

Limit of Quantification (mg/kg): Grain (all analytes): 0.01 mg/kg
Straw (Na 5-NG, Na *o*-NP): 0.02 mg/kg

Formulation *(e.g.* WP): SL

Limit of detection (mg/kg): Grain (all analytes): 0.005 mg/kg
Straw (all analytes): 0.01 mg/kg

Content of active components (nominal)
Sodium 5-Nitroguaiacolate: 0.12 %
Sodium *o*-Nitrophenolate: 0.22 %
Sodium *p*-Nitrophenolate: 0.33 %

Residues calculated as:
Sodium 5-Nitroguaiacolate (Na 5-NG)
Sodium *o*-Nitrophenolate (Na *o*-NP)
Sodium *p*-Nitrophenolate (Na *p*-NP)

1	2	3	4	5			6	7	8	9			10	11
Trial No./ Location/ Year	**Commodity/ Variety**	**Date of 1.Sowing or planting 2.Flowering 3. Harvest**	**Method of treatment**	**Application rate per treatment Sodium 5-Nitroguaiacolate Sodium *o*-Nitrophenolate Sodium *p*-Nitrophenolate**			**Dates of treatment or no. of treatments and last date**	**Growth stage at last treatment or date**	**Portion analyzed**	**Residues [mg/kg]**			**PHI [days]**	**Remarks**
	(a)	**(b)**	**(c)**	**[g a.s./hL]**	**Water [L/ha]**	**[g a.s./ha]**	**(d)**	**(e)**	**(a)**	**Na 5-NG**	**Na *o*-NP**	**Na *p*-NP**	**(f)**	**(g)**
A/GE/G/05/168 27449, Mulsum, Lower Saxony, Germany 2005	Spring barley / Orthega	1. 03/30/2005 2. Not applicable 3. 25/07/2018	Foliar application with a boom sprayer	0,24 / 0,44 / 0,64	317	0,76 / 1,40 / 2,04	2 6 June 2005	49	Grain	< 0.01	< 0.01	< 0.01	65	No residues >LOQ were found in any of the untreated samples
				0,24 / 0,44 / 0,64	323	0,78 / 1,43 / 2,08			Straw	< 0.02	< 0.02	< 0.02		
A/GE/G/05/169 27478, Altenbruch, Lower Saxony, Germany 2005	Spring wheat / Amaretto	1. 04/03/2005 2. Not applicable 3. 25/07/2018	Foliar application with a boom sprayer	0,24 / 0,44 / 0,64	297	0,71 / 1,31 / 1,91	2 27 May 2005	51	Grain	< 0.01	< 0.01	< 0.01	68	
				0,24 / 0,44 / 0,64	310	0,74 / 1,37 / 1,99			Straw	< 0.02	< 0.02	< 0.02		
A/GE/G/05/170 27478, Altenbruch, Lower Saxony, Germany 2005	Spring wheat / Monsun	1. 04/20/2005 2. Not applicable 3. 25/07/2018	Foliar application with a boom sprayer	0,24 / 0,44 / 0,64	330	0,79 / 1,46 / 2,12	2 17 June 2005	49	Grain	< 0.01	< 0.01	< 0.01	55	
				0,24 / 0,44 / 0,64	297	0,71 / 1,31 / 1,91			Straw	< 0.02	< 0.02	< 0.02		

(a) According to EPPO code (formerly BAYER code)
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting, etc., overall, broadcast, type of equipment used must be indicated
(d) Year must be indicated
(e) BBCH Monograph, Growth stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4
(f) Minimum number of days after last application (DALA) (label pre-harvest interval, PHI, underline)
(g) Remarks may include: Climatic conditions; Reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

Limit of quantification (LOQ for grain) = 0.01 mg/kg (all analytes)
Limit of quantification (LOQ for whole plant and straw) = 0.01 mg/kg for Sodium 5-Nitroguaiacolate and Sodium *o*-Nitrophenolate and 0.05 mg/kg for Sodium *p*-nitrophenolate

Table A 4-2 **Supervised residue trials in winter wheat – Northern Europe**

Reference: ATONIK – STUDY TO GENERATE SPECIMENS OF WINTER WHEAT FOLLOWING TWO APPLICATIONS OF ATONIK. THREE TRIALS IN NORTHERN EUROPE DURING 2018
White, T., 2018, report number: S18-05052, KCA 6.3.1-8 – Field part

ANALYSIS OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM ORTHO-NITROPHENOLATE AND SODIUM PARA-NITROPHENOLATE IN FIELD SAMPLES ORIGINATING FROM A FIELD STUDY PERFORMED UNDER EAS STUDY CODE S18-05052 WITH TWO APPLICATIONS OF ATONIK IN WINTER WHEAT AT 3 TRIALS IN NORTHERN EUROPE DURING 2018
Guserle, R., 2018, report number: S18-05052/P 4930 G, KCA 6.3.1-9 – Analytical part

GLP:	Yes	**Sample storage conditions:**	Max. 267 days: whole plant (all analytes) Max. 122 days: grain (all analytes) below -18°C (sampling till extraction) Max. 241 day: straw (all analytes) below -18°C (extraction till analysis)
Crop/crop group:	Winter wheat / cereals	**Analytical method:**	LC-MS/MS, validated
Indoor/Glasshouse/Outdoor:	Outdoor	**Limit of Quantification (mg/kg):**	Seeds (all analytes): 0.01 mg/kg Whole plant and straw (Na 5-NG, Na *o*-NP): 0.01 mg/kg, (Na *p*-NP): 0.05 mg/kg
Formulation *(e.g.* WP):	SL	**Limit of detection (mg/kg):**	Seeds (all analytes): 0.003 mg/kg Whole plant and straw (Na 5-NG, Na *o*-NP): 0.003 mg/kg, (Na *p*-NP): 0.03 mg/kg
Content of active components (nominal)	Sodium 5-Nitroguaiacolate: 0.11 % Sodium *o*-Nitrophenolate: 0.19 % Sodium *p*-Nitrophenolate: 0.29 %	**Residues calculated as:**	Sodium 5-Nitroguaiacolate (Na 5-NG) Sodium *o*-Nitrophenolate (Na *o*-NP) Sodium *p*-Nitrophenolate (Na *p*-NP)

1	2	3	4	5			6	7	8	9			10	11
Trial No./ Location/ Year	**Commodity/ Variety**	**Date of 1.Sowing or planting 2.Flowering 3. Harvest**	**Method of treatment**	**Application rate per treatment Sodium 5-Nitroguaiacolate Sodium *o*-Nitrophenolate Sodium *p*-Nitrophenolate**			**Dates of treatment or no. of treatments and last date**	**Growth stage at last treatment or date**	**Portion analyzed**	**Residues [mg/kg]**			**PHI [days]**	**Remarks**
	(a)	**(b)**	**(c)**	**[g a.s./hL]**	**Water [L/ha]**	**[g a.s./ha]**	**(d)**	**(e)**	**(a)**	**Na 5-NG**	**Na *o*-NP**	**Na *p*-NP**	**(f)**	**(g)**
S18-05052-01 L39 8SJ, Halsall, Lancashire, UK 2018	Winter wheat / JB Diego	1. 01/09/2017 2. Not applicable 3. 18/08/2018	Foliar application with a boom sprayer	0.20 /0,40/0,60 0.20 /0,40/0,60	317 308	0,63 /1,27/1,9 0,62 /1,23/1,85	2 21 July 2018	85	Whole plant	< 0.01	< 0.01	< 0.1	0	No residues >LOQ were found in any of the untreated samples
										< 0.01	< 0.01	< 0.1	6	
										< 0.01	< 0.01	< 0.1	14	
										< 0.01	< 0.01	< 0.1	21	
									Straw	< 0.01	< 0.01	< 0.1	28	
									Grain	< 0.01	< 0.01	< 0.01	28	

S18-05052-02 21706, Assel, Lower Saxony, German 2018	Winter wheat / Akteur	1. 15/10/2017 2. Not applicable 3. 18/08/2018	Foliar application with a boom sprayer	0.20 /0,40/0,60 0.20 /0,39/0,58	317 308	0,63 /1,27/1,9 0,6 /1,2/1,8	2 8 July 2018	83	Whole plant	< 0.01	< 0.01	< 0.1	0	No residues >LOQ were found in any of the untreated samples
										< 0.01	< 0.01	< 0.1	8	
										< 0.01	< 0.01	< 0.1	15	
										< 0.01	< 0.01	< 0.1	22	
									Straw	< 0.01	< 0.01	< 0.1	28	
									Grain	< 0.01	< 0.01	< 0.01	28	
S18-05054-04 64-606, Gorka, Wielkopolskie, Poland 2018	Winter wheat / Mulan	1. 22/09/2017 2. 6-14 June 2018 3. 22/08/2018	Foliar application with a boom sprayer	0.20 /0,40/0,60 0.20 /0,40/0,61	284 289	0,57 /1,13/1,7 0,58 /1,17/1,75	2 6 July 2018	83	Straw	< 0.01	< 0.01	< 0.1	28	
									Grain	< 0.01	< 0.01	< 0.01	28	

(a) According to EPPO code (formerly BAYER code)
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting, etc., overall, broadcast, type of equipment used must be indicated
(d) Year must be indicated
(e) BBCH Monograph, Growth stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4
(f) Minimum number of days after last application (DALA) (label pre-harvest interval, PHI, underline)
(g) Remarks may include: Climatic conditions; Reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

Limit of quantification (LOQ for grain) = 0.01 mg/kg (all analytes)
Limit of quantification (LOQ for whole plant and straw) = 0.01 mg/kg for Sodium 5-Nitroguaiacolate and Sodium *o*-Nitrophenolate and 0.05 mg/kg for Sodium *p*-nitrophenolate

Table A 4-3 **Supervised residue trials in winter wheat – Northern Europe**

Reference: ATONIK – STUDY TO GENERATE SPECIMENS OF WINTER WHEAT FOLLOWING TWO APPLICATIONS OF ATONIK. ONE TRIAL IN NORTHERN EUROPE DURING 2019
White, T., 2020, report number: S19-00202, KCA 6.3.1-10– Field part

ANALYSIS OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM ORTHO-NITROPHENOLATE AND SODIUM PARA-NITROPHENOLATE IN FIELD SAMPLES ORIGINATING FROM A FIELD STUDY PERFORMED UNDER EAS STUDY CODE S19-00202WITH TWO APPLICATIONS OF ATONIK IN WINTER WHEAT AT 1 TRIAL IN NORTHERN EUROPE DURING 2019
Guserle, R., 2020, report number: S19-00202/P 5296 G, KCA 6.3.1-11– Analytical part

GLP:	Yes	**Sample storage conditions:**	Max. 44 days: grain (all analytes) below -18°C (sampling till extraction) Max. 45 day: straw (all analytes) below -18°C (extraction till analysis)
Crop/crop group:	Winter wheat / cereals	**Analytical method:**	LC-MS/MS, validated
Indoor/Glasshouse/Outdoor:	Outdoor	**Limit of Quantification (mg/kg):**	Seeds (all analytes): 0.01 mg/kg Whole plant and straw (Na 5-NG, Na *o*-NP): 0.01 mg/kg, (Na *p*-NP): 0.05 mg/kg
Formulation *(e.g.* WP):	SL	**Limit of detection (mg/kg):**	Seeds (all analytes): 0.003 mg/kg Whole plant and straw (Na 5-NG, Na *o*-NP): 0.003 mg/kg, (Na *p*-NP): 0.03 mg/kg
Content of active components (nominal)	Sodium 5-Nitroguaiacolate: 0.095 % Sodium *o*-Nitrophenolate: 0.21 % Sodium *p*-Nitrophenolate: 0.31 %	**Residues calculated as:**	Sodium 5-Nitroguaiacolate (Na 5-NG) Sodium *o*-Nitrophenolate (Na *o*-NP) Sodium *p*-Nitrophenolate (Na *p*-NP)

1	2	3	4	5			6	7	8	9			10	11
Trial No./ Location/ Year	**Commodity/ Variety**	**Date of 1.Sowing or planting 2.Flowering 3. Harvest**	**Method of treatment**	**Application rate per treatment Sodium 5-Nitroguaiacolate Sodium *o*-Nitrophenolate Sodium *p*-Nitrophenolate**			**Dates of treatment or no. of treatments and last date**	**Growth stage at last treatment or date**	**Portion analyzed**	**Residues [mg/kg]**			**PHI [days]**	**Remarks**
	(a)	**(b)**	**(c)**	**[g a.s./hL]**	**Water [L/ha]**	**[g a.s./ha]**	**(d)**	**(e)**	**(a)**	**Na 5-NG**	**Na *o*-NP**	**Na *p*-NP**	**(f)**	**(g)**
S19-00202-01 Halsall, Lancashire, UK 2018	Winter wheat / JB Diego	1. 4/10/2018 2. Not applicable 3. 17/08/2019	Foliar application with a boom sprayer	0.20 /0,40/0,60	325	0.65 /1,3/1,95	2 20 July 2019	77	Straw	< 0.01	< 0.01	< 0.1	28	No residues >LOQ were found in any of the untreated samples
				0.20 /0,40/0,61	313	0.63 /1,27/1.9			Grain	< 0.01	< 0.01	< 0.01	28	

(a) According to EPPO code (formerly BAYER code)
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting, etc., overall, broadcast, type of equipment used must be indicated
(d) Year must be indicated
(e) BBCH Monograph, Growth stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4
(f) Minimum number of days after last application (DALA) (label pre-harvest interval, PHI, underline)
(g) Remarks may include: Climatic conditions; Reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

Limit of quantification (LOQ for grain) = 0.01 mg/kg (all analytes)
Limit of quantification (LOQ for whole plant and straw) = 0.01 mg/kg for Sodium 5-Nitroguaiacolate and Sodium *o*-Nitrophenolate and 0.05 mg/kg for Sodium *p*-nitrophenolate

A 2.1.3.3 Sugar beet

Table A 5: 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)
Proposed GAP NEU: Poland	2x	0,6 g / ha Na-5NG 1,2 g/ ha Na-oNP 1,8 g/ ha Na-pNP	7 days	BBCH 12-49	15
Critical GAP NEU: Poland	4x	~~0,6~~ 1 g / ha Na-5NG ~~1,2~~ 2 g/ ha Na-oNP ~~1,8~~ 3 g/ ha Na-pNP	7 days	BBCH 12-49	15

A 2.1.3.3.1 A05973

Comments of zRMS:	Two residue trials on sugar beet were conducted in Germany (2 locations, distance between the trials was 75 km) to determine the residues of Na 5-NG, Na o-NP and Na p-NP, but only two of the three were independent trials. Sugar beets were treated four times at application rate of 1 L product/ha (corresponding to 1 g/ha sodium 5-nitroguaiacolate + 2 g/ha sodium o-nitrophenolate + 3 g/ha sodium p-nitrophenolate) with 6-8 days interval between applications. Samples (roots, leaves with tops) were taken at harvest, at PHI 15 days. All procedures for specimen preparation and analysis for Sodium 5-Nitroguaiacolate, Sodium o-Nitrophenolate and Sodium p-Nitrophenolate followed as validated under RCC Study No. 850917 ("Development and Validation of a Residue Analytical Method for Sodium 5-Nitroguaiacolate, Sodium o-Nitrophenolate and Sodium p-Nitrophenolate (as Active Ingredients in Atonik Formulated Product) in Tomato (Fruits), Sugar Beet (Roots and Tops with Leaves) and Oil Seed Rape"; dr Alexander Krainz, 2004.. To demonstrate the validity of the method used, untreated specimens were fortified with different amounts of Na 5-NG, Na o-NP and Na p-NP. The limit of quantification (LOQ) in roots and leaves with tops was 0.01 mg/kg for Na 5-NG, Na o-NP and Na p-NP. In analytical report for sugar beet, only one recovery determination at LOQ and one recovery at 10xLOQ has been presented for Na 5-NG, Na o-NP and Na p-NP respectively. This is not fully with the requirement of SANTE/2020/12830, Rev.1, 24. February 2021. Maximum storage period – 76 days. <u>Results:</u> The residues of Na 5-NG, Na o-NP and Na p-NP at harvest were <0.01 mg/kg in roots and leaves with tops. The study is acceptable.

Data point addressed:	KCA 6.3.1-12
Author(s) (year):	Diehl, M. (2006)
Title:	DETERMINATION OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM O-NITROPHENOLATE AND SODIUM P-NITROPHENOLATE IN SUGAR BEET (RAC ROOTS AND LEAVES) FOLLOWING FOUR TREATMENTS WITH ATONIK IN NORTHERN EUROPE, 2005
Laboratory report / project Number (Doc. No.):	A05973
Testing facility:	RCC Ltd, Environmental Chemistry & Pharmanalytics Division, Itingen, Switzerland
Published:	No
Test guideline used:	96/68/EC (1996), 7029/VI/95 rev. 5 (1997), in accordance to OECD 509 (2009)

Deviations:	None
Previous evaluation:	No; not previously submitted
GLP:	Yes; certified by Swiss Federal Office of Public Health, Bern
Acceptability/Reliability:	Yes

Materials and methods

Description:	**ATONIK**		
Components:	Na 5-NG	Na *o*-NP	Na *p*-NP
CAS #:	67233-85-6	824-39-5	824-78-2
Lot/Batch #:	2005-AC-50628		
Content of a.s. (actual):	0.12 %	0.22 %	0.32 %
Stability of test compound (Expiry date):	May 2008		

The residue study on sugar beet has been performed at two field sites in Germany. This residue study provides data relevant to conditions in the Northern European region. The experimental setup includes two at-harvest trials.

Atonik was applied four times to the treated sugar beet plots with an interval of 6-8 days at a nominal rate of 1.0 L product/ha.
Sodium 5-Nitroguaiacolate was applied in the range of 1.11-1.29 g/ha for first application, at rate of 1.21 g/ha for second, in the range of 1.19-1.27 g/ha for the third and in the range of 1.2-1.25 g/ha for the fourth application.
Sodium o-Nitrophenolate was applied in the range of 2.31-2.38 g/ha for first application, in the range of 2.23-2,24 g/ha for second, in the range of 2.18-2.34 g/ha for the third and in the range of 2.21-2.31 g/ha for the fourth application.
Sodium p-Nitrophenolate was applied in the range of 3.35-3.46 g/ha for first application, in the range of 3.24-3,25 g/ha for second, in the range of 3.17-3.39 g/ha for the third and in the range of 3.21-3.36 g/ha for the fourth application.
All trials were carried out in the Germany. Samples (roots, leaves with tops) were taken at harvest, at PHI 15.
The field phase was performed by GAB Biotechnology GmbH, Stade, Germany, whereas the analytical work associated with the studies was performed by RCC Ltd., Itingen, Switzerland.

Crop specimens were analyzed for residues of Na 5-NG, Na *o*-NP and Na *p*-NP using an HPLC method described below.
Principle of the method:
Na 5-NG was extracted from homogenised roots with methanol and from leaves with tops with methanol/water. Extracts were filtered and diluted with water (1:3). Separation was carried out on HPLC with column switching, technique followed by triple stage quadrupole mass spectrometric detection (MS/MS).
Na o-NP was extracted from homogenised roots with methanol and from leaves with tops with methanol/water. Extracts were filtered thorugh a syringe filter into an HPLC vial. Separation was carried out on HPLC with column switching, technique followed by triple stage quadrupole mass spectrometric detection (MS/MS).
Na p-NP was extracted from homogenised roots with methanol and from leaves with tops with methanol/water. Extracts were filtered and diluted with water (1:3). Separation was carried out on HPLC with column switching, technique followed by triple stage quadrupole mass spectrometric detection (MS/MS).
The limit of quantification (LOQ) for Na 5-NG, Na o-NP and Na p-NP was 0.01 mg/kg in roots and leaves with tops. The limit of detection (LOD) for Na 5-NG, Na o-NP and Na p-NP was set at 0.005 mg/kg in roots and leaves with tops.
For Na 5-NG, the maximum sampling to analysis interval at -18°C was 76 days in sugar beet roots and leaves with tops. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na 5-NG in the samples extract. All recoveries

were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na o-NP, the maximum sampling to analysis interval at -18°C was 76 days in sugar beet roots and leaves with tops. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na o-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na p-NP, the maximum sampling to analysis interval at -18°C was 76 days in sugar beet roots and leaves with tops. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na p-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
Results and discussions
Residues in untreated control samples were not detectable.
Residues of Na 5-NG, Na o-NP, Na p-NP in sugar beet roots and tops with leaves at harvest (PHI 15 days) are below the limit of quantification (LOQ = 0.01 mg/kg), when Atonik is applied four times at a nominal rate of 1,0 g/ha Na 5-NG, 2,0 g/ha Na o-NP and 3,0 g/ha Na p-NP with a PHI of 15 days in Northern Europe.

A 2.1.3.3.2 S12-04698

Comments of zRMS:	One residue trial on sugar beet was conducted in northern Europe to determine the residues of Na 5-NG, Na o-NP and Na p-NP. Sugar beets were treated four times at application rate of 1 L product/ha (corresponding to 1 g/ha sodium 5-nitroguaiacolate + 2 g/ha sodium o-nitrophenolate + 3 g/ha sodium p-nitrophenolate) with 6-7 days interval between applications. Samples (roots, leaves with tops) were taken at harvest, at PHI 13 days. <u>Analytical Method</u> The analytical method was fully validated on cucumber during a study performed at GIRPA in 2013, ARSTA-VAL-13.01. A reduced validation was performed on sugar beet (roots and tops) during another analytical study performed at GIRPA in 2013, referenced B13-A1-NOP-01 (study number S12-04679), for each specimen, by 6 spiked samples, 3 recovery experiments fortified at the LOQ and 3 recovery at 10x LOQ and 1 control sample. Quantification was performed by use of LC-MS/MS detection. The daily sample sets were validated with the determination of at least one recovery experiment per sample set. The limit of quantification (LOQ) of the analytical method was 0.01 mg/kg per analyte (expressed as sodium salt) for sugar beet root and tops. The mean recovery was between 70% and 110% with a relative standard deviation lower than 20%. Maximum storage period – 21 days in sugar beet roots and tops. <u>Results:</u> Residues of Na 5-NG, Na o-NP, Na p-NP in sugar beet root and tops at harvest (PHI 13 days) are below the limit of quantification (LOQ) of 0.01 mg/kg for Na 5-NG, Na o-NP and Na p-NP. The study is acceptable.

Data point addressed:	KCA 6.3.1-13
Author(s) (year):	Oxspring, S. (2014)
Title:	ATONIK - DETERMINATION OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM ORTHO-NITROPHENOLATE AND SODIUM PARA-NITROPHENOLATE AFTER FOUR APPLICATIONS OF ATONIK IN SUGAR BEET AT 1 SITE IN NORTHERN EUROPE 2013

Laboratory report / project Number (Doc. No.):	S12-04698
Testing facility:	Eurofins Agroscience Services Ltd., Melbourne, United Kingdom
Published:	No
Test guideline used:	1607/VI/97 (1999), SANCO/3029/99 rev. 4, 7029/VI/95 (rev. 5), in accordance to OECD 509 (2009)
Deviations:	None
Previous evaluation:	No; not previously submitted
GLP:	Yes; certified by the Department of Health of the Government of the United Kingdom and Groupe Interministeriel des Produits Chimiques, Paris
Acceptability/Reliability:	Yes

Materials and methods

Description:	**ATONIK**		
Components:	Na 5-NG	Na *o*-NP	Na *p*-NP
CAS #:	67233-85-6	824-39-5	824-78-2
Lot/Batch #:	2439/ATO		
Content of a.s. (actual):	1 g/L	2 g/L	3 g/L
Stability of test compound (Expiry date):	13 Dec 2014		

The residue study on sugar beet has been performed during 2013 at one field site in Northern France. This residue study provides data relevant to conditions in the Northern European region. The experimental setup includes one at-harvest trial.

Atonik was applied four times to the treated sugar beet plots with an interval of 6-7 days at a nominal rate of 1.0 L product/ha.
Sodium 5-Nitroguaiacolate was applied at rate of 1.1 g/ha for first, at rate of 1.0 g/ha for second, third and fourth application.
Sodium o-Nitrophenolate was applied at rate of 2.1 g/ha for first, at rate of 2.0 g/ha for second, third and fourth application.
Sodium p-Nitrophenolate was applied at rate of 3.2 g/ha for first, applied at rate of 3.0 g/ha for second, applied at rate of 2,9 g/ha for the third and applied at rate of 3.1 g/ha for the fourth application.
Trial was carried out in France. Samples (roots, leaves with tops) were taken at harvest, at PHI 13.
The field phase was performed by EAS France SAS, Saint Pierre, whereas the analytical work associated with the studies was performed by EAS Ltd., Melbourne UK.

Crop specimens were analyzed for residues of Na 5-NG, Na *o*-NP and Na *p*-NP using an HPLC method described below.
Principle of the method:
Na 5-NG was extracted from homogenised roots and leaves with tops samples by agitation with acidified acetonitrile (first step) and a second time with acetonitrile. The quantification was done by liquid chromatography with tandem mass spectrometry detection (LC-MS/MS), monitoring two quantification transitions.
Na o-NP was extracted from homogenised roots and leaves with tops samples by agitation with acidified acetonitrile (first step) and a second time with acetonitrile. The quantification was done by liquid chromatography with tandem mass spectrometry detection (LC-MS/MS), monitoring two quantification transitions.
Na p-NP was extracted from homogenised roots and leaves with tops samples by agitation with acidified acetonitrile (first step) and a second time with acetonitrile. The quantification was done by liquid chromatography with tandem mass spectrometry detection (LC-MS/MS), monitoring two quantification transitions.
The limit of quantification (LOQ) for Na 5-NG, Na o-NP and Na p-NP was 0.01 mg/kg in roots and leaves with tops. The limit of detection (LOD) for Na 5-NG, Na o-NP and Na p-NP was set at 30% of the LOQ in roots and leaves with tops.

For Na 5-NG, the maximum sampling to analysis interval at -18°C was 21 days in sugar beet roots and leaves with tops. The maximum extraction to quantification interval for Na 5-NG was 1 day for sugar beet roots and leaves with tops. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na 5-NG in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na o-NP, the maximum sampling to analysis interval at -18°C was 21 days in sugar beet roots and leaves with tops. The maximum extraction to quantification interval for Na o-NP was 1 day for sugar beet roots and leaves with tops. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na o-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na p-NP, the maximum sampling to analysis interval at -18°C was 21 days in sugar beet roots and leaves with tops. The maximum extraction to quantification interval for Na p-NP was 1 day for sugar beet roots and leaves with tops. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na p-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.

Results and discussions
Residues in untreated control samples were not detectable.
Residues of Na 5-NG, Na o-NP, Na p-NP in sugar beet roots and tops with leaves at harvest (PHI 15 days) are below the limit of quantification (LOQ = 0.01 mg/kg), when Atonik is applied four times at a nominal rate of 1,0 g/ha Na 5-NG, 2,0 g/ha Na o-NP and 3,0 g/ha Na p-NP with a PHI of 15 days in Northern Europe.

A 2.1.3.3.3 S19-04275

Comments of zRMS:	Three residue trials on sugar beet was conducted in northern Europe to determine the residues of Na 5-NG, Na o-NP and Na p-NP. Sugar beets were treated three times at application rate of 1 L product/ha (corresponding to 1 g/ha sodium 5-nitroguaiacolate + 2 g/ha sodium o-nitrophenolate + 3 g/ha sodium p-nitrophenolate) with 7-9 days interval between applications. Samples (roots, leaves with tops) were taken directly before the final application (0 DBA3) as well as after 1, 3, 7, 10 and 15 (i.e. normal commercial harvest: NCH) days after the final application. <u>Analytical Method</u> Sugar beet samples were analysed for residues of 5-nitroguaiacol, ortho-nitrophenol and para-nitrophenol (all expressed as sodium salts) based on the analytical method that was previously validated for the determination of 5-NG, o-NP and p-NP (expressed as the respective sodium salts) in sugar beet samples as described in Eurofins Agroscience Services Study S12-04698. Procedural recoveries were performed during the present study to demonstrate acclicability of the method used and to show extract stability of the analytes when recoveries are within the acceptable range of 70-120%. The method is fully validated with the requirement of SANTE/2020/12830, Rev.1, 24. February 2021 in this report for sugar beet (5 fortifications with 1xLOQ and 5 fortifications with 10xLOQ for Na 5-NG, Na o-NP and Na p-NP for root and leaves with tops of sugar beet)). Quantification was performed by use of LC-MS/MS detection. The limit of quantitation for 5-nitroguaiacol, ortho-nitrophenol and para-nitrophenol (all expressed as sodium salts) in sugar beet is set at 0.01 mg/kg. The mean recovery was between 70% and 110% with a relative standard deviation below 20%. Maximum storage period – 30 days in sugar beet roots and tops. <u>Results:</u>

	Residues of Na 5-NG, Na o-NP, Na p-NP in sugar beet root and tops at harvest (PHI 15 days) are below the limit of quantification (LOQ) of 0.01 mg/kg for Na 5-NG, Na o-NP and Na p-NP. The study is acceptable.

Data point addressed:	KCA 6.3.1-14
Author(s) (year):	White, T. (2020)
Title:	ATONIK - DETERMINATION OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM O-NITROPHENOLATE AND SODIUM P-NITROPHENOLATE IN SUGAR BEET FOLLOWING THREE APPLICATIONS OF ATONIK UNDER FIELD CONDITIONS - THREE TRIALS IN NORTHERN EUROPE DURING 2019
Laboratory report / project Number (Doc. No.):	S19-04275
Testing facility:	Eurofins Agroscience Services Ltd., Melbourne, United Kingdom
Published:	No
Test guideline used:	OECD No. 509, ENV/JM/MONO(2009)31, ENV/JM/MONO(2011)50, SANCO/3029/99 rev. 4, SANCO/825/00 rev. 8.1, ENV/JM/MONO(2007)17
Deviations:	None
Previous evaluation:	No, not previously submitted
GLP:	Yes; certified by the Department of Health of the Government of the United Kingdom
Acceptability/Reliability:	Yes

Materials and methods

Description:	**ATONIK**		
Components:	Na 5-NG	Na *o*-NP	Na *p*-NP
CAS #:	67233-85-6	824-39-5	824-78-2
Lot/Batch #:	CG7-101		
Content of a.s. (actual):	0.095 (% w/w)	0.095 (% w/w)	0.095 (% w/w)
Stability of test compound (Expiry date):	May 2020		

The residue study on sugar beet has been performed during 2019 at three field sites in Hungary, Germany and Northern France. This residue study provides data relevant to conditions in the Northern European region. The experimental setup includes two decline trials and one at-harvest trial.

Atonik was applied three times to the treated sugar beet plots with an interval of 7-9 days at a nominal rate of 1.0 L product/ha.
Sodium 5-Nitroguaiacolate was applied at rate of 1.0 g/ha.
Sodium o-Nitrophenolate was applied at rate of 2.0 g/ha.
Sodium p-Nitrophenolate was applied at rate of 3.0 g/ha.
The trials were carried out in Hungary, Germany and Northern France. For trials S19-04275-01 and 02 samples (roots and leaves with tops) were taken directly after application as well as after 1, 3, 7 and 10 days and at harvest (PHI 15 days). For trial S19-04275-03 samples were taken at harvest (PHI 15 d).
The field phase was performed by Eurofins Agroscience Services Ltd., Melbourne, United Kingdom, whereas the analytical work associated with the study was performed by EAG Laboratories GmbH, Ulm, Germany.

Crop specimens were analyzed for residues of Na 5-NG, Na *o*-NP and Na *p*-NP using an HPLC method described below.
Principle of the method:
Na 5-NG was extracted from homogenised roots and leaves with tops samples by agitation with acidified acetonitrile (first step) and a second time with acetonitrile. The quantification was done by liquid chromatography with tandem mass spectrometry detection (LC-MS/MS), monitoring two quantification transitions.

Na o-NP was extracted from homogenised roots and leaves with tops samples by agitation with acidified acetonitrile (first step) and a second time with acetonitrile. The quantification was done by liquid chromatography with tandem mass spectrometry detection (LC-MS/MS), monitoring two quantification transitions.
Na p-NP was extracted from homogenised roots and leaves with tops samples by agitation with acidified acetonitrile (first step) and a second time with acetonitrile. The quantification was done by liquid chromatography with tandem mass spectrometry detection (LC-MS/MS), monitoring two quantification transitions.
The limit of quantification (LOQ) for Na 5-NG, Na o-NP and Na p-NP was 0.01 mg/kg in roots and leaves with tops. The limit of detection (LOD) for Na 5-NG, Na o-NP and Na p-NP was set at 30% of the LOQ in roots and leaves with tops.

For Na 5-NG, the maximum sampling to analysis interval at -18°C was 30 days in sugar beet roots and leaves with tops. The maximum extraction to quantification interval for Na 5-NG was 1 day for sugar beet roots and leaves with tops. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na 5-NG in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na o-NP, the maximum sampling to analysis interval at -18°C was 30 days in sugar beet roots and leaves with tops. The maximum extraction to quantification interval for Na o-NP was 1 day for sugar beet roots and leaves with tops. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na o-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na p-NP, the maximum sampling to analysis interval at -18°C was 30 days in sugar beet roots and leaves with tops. The maximum extraction to quantification interval for Na p-NP was 1 day for sugar beet roots and leaves with tops. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na p-NP in the samples extract. All recoveries were within the range between 70 – 110 %. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.

Results and discussions

Residues in untreated control samples were not detectable.
Residues of Na 5-NG, Na o-NP, Na p-NP in sugar beet roots and tops with leaves at harvest (PHI 15 days) are below the limit of quantification (LOQ = 0.01 mg/kg), when Atonik is applied three times at a nominal rate of 1,0 g/ha Na 5-NG, 2,0 g/ha Na o-NP and 3,0 g/ha Na p-NP with a PHI of 15 days in Northern Europe.

Details of the trials and the analytical results can be found in the Tier 1 summary forms provided below.

Table A 6: Residue trials on sugar beet (NEU)

Table A 6-1 Supervised residue trials in sugar beet – Northern Europe

Reference: DETERMINATION OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM O-NITROPHENOLATE AND SODIUM P-NITROPHENOLATE IN SUGAR BEET (RAC ROOTS AND LEAVES) FOLLOWING FOUR TREATMENTS WITH ATONIK IN NORTHERN EUROPE, 2005
Diehl, M., 2006, report number: A05973, KCA 6.3.1-12

GLP: Yes
Crop/crop group: Sugar beet / Sugar plants
Indoor/Glasshouse/Outdoor: Outdoor
Formulation *(e.g.* WP): SL
Content of active components (nominal): Sodium 5-Nitroguaiacolate: 0.12%; Sodium *o*-Nitrophenolate: 0.22%; Sodium *p*-nitrophenolate: 0.32%
Sample storage conditions: Max. 76 days (all analytes) below -18°C (sampling till analysis)
Analytical method: HPLC-MS/MS, validated
Limit of Quantification (mg/kg): 0.01 mg/kg (all analytes and matrices)
Limit of detection (mg/kg): 0.005 mg/kg (all analytes and matrices)
Residues calculated as: Sodium 5-Nitroguaiacolate (Na 5-NG); Sodium *o*-Nitrophenolate (Na *o*-NP); Sodium *p*-Nitrophenolate (Na *p*-NP)

1	2	3	4	5			6	7	8	9			10	11
Trial No./ Location/ Year	**Commodity/ Variety**	**Date of 1.Sowing 2.Flowering 3. Harvest**	**Method of treatment**	**Application rate per treatment Sodium 5-Nitroguaiacolate Sodium *o*-Nitrophenolate Sodium *p*-Nitrophenolate**			**Dates of treatment or no. of treatments and last date**	**Growth stage at last treatment or date**	**Portion analyzed**	**Residues [mg/kg]**			**PHI [days]**	**Remarks**
	(a)	**(b)**	**(c)**	**[g a.s./hL]**	**Water [L/ha]**	**[g a.s./ha]**	**(d)**	**(e)**	**(a)**	**Na 5-NG**	**Na *o*-NP**	**Na *p*-NP**	**(f)**	**(g)**
A/GE/G/05/177 71120 Grafenau, Baden-Württemberg Germany 2005	Sugar beet / Mars	1. 12/04/2005 2. n.r 3. 12/10/2005	Broadcast foliar application with a boom sprayer	0.40 /0.74 /1.07 0.40 /0.74 /1.07 0.40 /0.73 /1.07 0.40 /0.74 /1.07	313 303 297 300	1.25 /2.31 /3.35 1.21 /2.23 /3.24 1.19 /2.18 /3.17 1.20 /2.21 /3.21	4 27 Sep 2005	39-49	Roots	< 0.01	< 0.01	< 0.01	15	No residues >LOQ were found in any of the untreated samples
									Leaves with tops	< 0.01	< 0.01	< 0.01	15	
A/GE/G/05/178 69124 Heidelberg, Baden-Württemberg Germany 2005	Sugar beet / Tatjana	1. 24/03/2005 2. n.r 3. 21/09/2005	Broadcast foliar application with a boom sprayer	0.40 /0.74 /1.07 0.40 /0.74 /1.07 0.40 /0.74 /1.07 0.40 /0.74 /1.07	323 303 317 313	1.29 /2.38/ 3.46 1.21 /2.24 /3.25 1.27 /2.34 /3.39 1.25 /2.31 /3.36	4 06 Sep 2005	49	Roots	< 0.01	< 0.01	< 0.01	15	
									Leaves with tops	< 0.01	< 0.01	< 0.01	15	

(a) According to EPPO code (formerly BAYER code)
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting, etc., overall, broadcast, type of equipment used must be indicated
(d) Year must be indicated
(e) BBCH Monograph, Growth stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4
(f) Minimum number of days after last application (DALA) (label pre-harvest interval, PHI, underline)
(g) Remarks may include: Climatic conditions; Reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

Limit of quantification (LOQ) = 0.01 mg/kg for all analytes
n.r. = not recorded

Table A 6-2 **Supervised residue trials in sugar beet – Northern Europe**

Reference: ATONIK - DETERMINATION OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM ORTHO-NITROPHENOLATE AND SODIUM PARA-NITROPHENOLATE AFTER FOUR APPLICATIONS OF ATONIK IN SUGAR BEET AT 1 SITE IN NORTHERN EUROPE 2013
Oxspring, S., 2014, report number: S12-04698, KCA 6.3.1-13

GLP: Yes

Sample storage conditions: Max. 21 days (all analytes) below -18°C (sampling till analysis)
Max. 1 day (all analytes) below -18°C (extraction till analysis)

Crop/crop group: Sugar beet / Sugar plants

Analytical method: HPLC-MS/MS, validated

Indoor/Glasshouse/Outdoor: Outdoor

Limit of Quantification (mg/kg): 0.01 mg/kg (all analytes and matrices)

Formulation *(e.g.* WP): SL

Limit of detection (mg/kg): 30% of the LOQ (all analytes and matrices)

Content of active components (nominal)
Sodium 5-Nitroguaiacolate: 1.0 g/L
Sodium o-Nitrophenolate: 2.0 g/L
Sodium p-Nitrophenolate: 3.0 g/L

Residues calculated as:
Sodium 5-Nitroguaiacolate (Na 5-NG)
Sodium o-Nitrophenolate (Na o-NP)
Sodium p-Nitrophenolate (Na p-NP)

1	2	3	4	5			6	7	8	9			10	11
Trial No./ Location/ Year	**Commodity/ Variety**	**Date of 1.Sowing 2.Flowering 3. Harvest**	**Method of treatment**	**Application rate per treatment Sodium 5-Nitroguaiacolate Sodium *o*-Nitrophenolate Sodium *p*-Nitrophenolate**			**Dates of treatment or no. of treatments and last date**	**Growth stage at last treatment or date**	**Portion analyzed**	**Residues [mg/kg]**			**PHI [days]**	**Remarks**
	(a)	**(b)**	**(c)**	**[g a.s./hL]**	**Water [L/ha]**	**[g a.s./ha]**	**(d)**	**(e)**	**(a)**	**Na 5-NG**	**Na *o*-NP**	**Na *p*-NP**	**(f)**	**(g)**
S12-04698-01 67140 Epfig Northern France 2013	Sugar beet / Iceberg	1. 01/04/2013 2. n.r 3. 26/08/2013	Foliar application with a boom sprayer	0.34 /0.66 /1.0 0.33 /0.66 /1.0 0.34 /0.68 /0.99 0.33 /0.68 /1.0	320 300 293 307	1.1 /2.1/ 3.2 1.0 /2.0 /3.0 1.0 /2.0 /2.9 1.0 /2.0 /3.1	4 13 Aug 2013	BBCH 39	Roots	< 0.01	< 0.01	< 0.01	13	No residues >LOQ were found in any of the untreated samples
									Leaves with tops	< 0.01	< 0.01	< 0.01	13	

(a) According to EPPO code (formerly BAYER code)
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting, etc., overall, broadcast, type of equipment used must be indicated
(d) Year must be indicated
Limit of quantification (LOQ) = 0.01 mg/kg for all analytes

(e) BBCH Monograph, Growth stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4
(f) Minimum number of days after last application (DALA) (label pre-harvest interval, PHI, underline)
(g) Remarks may include: Climatic conditions; Reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

n.r. = not recorded

Table A 4-2 **Supervised residue trials in sugar beet – Northern Europe**

Reference: ATONIK - DETERMINATION OF RESIDUES OF SODIUM 5-NITROGUAIACOLATE, SODIUM ORTHO-NITROPHENOLATE AND SODIUM PARA-NITROPHENOLATE IN SUGAR BEET FOLLOWING THREE APPLICATIONS OF ATONIK UNDER FIELD CONDITIONS – THREE TRIALS IN NORTHERN EUREOPE DURING 2019
White, T., 2020, report number: S19-04275, KCA 6.3.1-14

GLP: Yes

Sample storage conditions: Max. 30 days (all analytes) below -18°C (sampling till analysis)
Max. 1 day (all analytes) below -18°C (extraction till analysis)

Crop/crop group: Winter wheat / cereals

Analytical method: LC-MS/MS, validated

Indoor/Glasshouse/Outdoor: Outdoor

Limit of Quantification (mg/kg): 0.01 mg/kg (all analytes and matrices)

Formulation *(e.g.* WP): SL

Limit of detection (mg/kg): 30% of the LOQ (all analytes and matrices)

Content of active components (nominal)
Sodium 5-Nitroguaiacolate: 0.095 %
Sodium *o*-Nitrophenolate: 0.21 %
Sodium *p*-Nitrophenolate: 0.31 %

Residues calculated as:
Sodium 5-Nitroguaiacolate (Na 5-NG)
Sodium *o*-Nitrophenolate (Na *o*-NP)
Sodium *p*-Nitrophenolate (Na *p*-NP)

1	2	3	4	5			6	7	8	9			10	11
Trial No./ Location/ Year	**Commodity/ Variety**	**Date of 1.Sowing or planting 2.Flowering 3. Harvest**	**Method of treatment**	**Application rate per treatment Sodium 5-Nitroguaiacolate Sodium *o*-Nitrophenolate Sodium *p*-Nitrophenolate**			**Dates of treatment or no. of treatments and last date**	**Growth stage at last treatment or date**	**Portion analyzed**	**Residues [mg/kg]**			**PHI [days]**	**Remarks**
	(a)	**(b)**	**(c)**	**[g a.s./hL]**	**Water [L/ha]**	**[g a.s./ha]**	**(d)**	**(e)**	**(a)**	**Na 5-NG**	**Na *o*-NP**	**Na *p*-NP**	**(f)**	**(g)**
S19-04275-01 H-7159, Kisdorog, Tolna, Hungary 2019	Sugar beet/ Antek	1. 24/04/2019 2. n.r. 3. 24/10/2019	Foliar application with a boom sprayer	0.31 /0.63 /0.94 0.33 /0.66 /0.99 0.34 /0.68 /1.01	320 304 295	1.0 /2.0 /3.0 1.0 /2.0 /3.0 1.0 /2.0 /3.0	25 Sep 2019 02 Oct 2019 09 Oct 2019	BBCH 45	Roots	< 0.01	< 0.01	< 0.01	0	No residues >LOQ were found in any of the untreated samples
									Leaves with tops	< 0.01	< 0.01	< 0.01		
									Roots	< 0.01	< 0.01	< 0.01	1	
									Leaves with tops	< 0.01	< 0.01	< 0.01		
									Roots	< 0.01	< 0.01	< 0.01	3	
									Leaves with tops	< 0.01	< 0.01	< 0.01		
									Roots	< 0.01	< 0.01	< 0.01	7	
									Leaves with tops	< 0.01	< 0.01	< 0.01		
									Roots	< 0.01	< 0.01	< 0.01	10	
									Leaves with tops	< 0.01	< 0.01	< 0.01		
									Roots	< 0.01	< 0.01	< 0.01	15	
									Leaves with tops	< 0.01	< 0.01	< 0.01		
S19-04275-02	Sugar beet/		Foliar				24 Sep 2019	BBCH 49	Roots	< 0.01	< 0.01	< 0.01	0	No residues

21739, Dollern, Niedersachsen, Germany 2019	Danicia	1. 07/04/2019 2. n.r. 3. 25/10/2019	application with a boom sprayer	0.34 /0.68 /1.02 0.33 /0.65 /0.98 0.34 /0.68 /1.02	294 307 294	1.0 /2.0 /3.0 1.0 /2.0 /3.0 1.0 /2.0 /3.0	03 Oct 2019 10 Oct 2019		Leaves with tops	< 0.01	< 0.01	< 0.01		>LOQ were found in any of the untreated samples
									Roots	< 0.01	< 0.01	< 0.01	1	
									Leaves with tops	< 0.01	< 0.01	< 0.01		
									Roots	< 0.01	< 0.01	< 0.01	3	
									Leaves with tops	< 0.01	< 0.01	< 0.01		
									Roots	< 0.01	< 0.01	< 0.01	7	
									Leaves with tops	< 0.01	< 0.01	< 0.01		
									Roots	< 0.01	< 0.01	< 0.01	10	
									Leaves with tops	< 0.01	< 0.01	< 0.01		
									Roots	< 0.01	< 0.01	< 0.01	15	
									Leaves with tops	< 0.01	< 0.01	< 0.01		
S19-04275-03 67 230, Benfeld, Bas Rhin / Alsace, Northern France 2019	Sugar beet/ Rainette	1. 25/03/2019 2. n.r. 3. 19/09/2019	Foliar application with a boom sprayer	0.47 /0.94 /1.42 0.47 /0.93 /1.40 0.48 /0.97 /1.45	212 214 207	1.0 /2.0 /3.0 1.0 /2.0 /3.0 1.0 /2.0 /3.0	21 Aug 2019 28 Aug 2019 04 Sep 2019	BBCH 49	Roots	< 0.01	< 0.01	< 0.01	15	No residues >LOQ were found in any of the untreated samples
									Leaves with tops	< 0.01	< 0.01	< 0.01		

(a) According to EPPO code (formerly BAYER code)
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting, etc., overall, broadcast, type of equipment used must be indicated
(d) Year must be indicated
Limit of quantification (LOQ) = 0.01 mg/kg for all analytes

(e) BBCH Monograph, Growth stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4
(f) Minimum number of days after last application (DALA) (label pre-harvest interval, PHI, underline)
(g) Remarks may include: Climatic conditions; Reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

n.r. = not recorded

A 2.1.4 Magnitude of residues in livestock

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

A 2.1.5.2 Processing studies on a core set of representative processes

No new data submitted in the framework of this application.

A 2.1.6 Magnitude of residues in representative succeeding crops

No new data submitted in the framework of this application.

A 2.1.7 Other/Special Studies

~~No new data submitted in the framework of this application.~~
A residue study in nectar is summarised below:

Comments of zRMS:	Four residue trials located in Germany, Spain and France were conducted in 2019 to determine residues of Sodium 5-Nitroguaiacolate, Sodium o-Nitrophenolate and Sodium p-Nitrophenolate in nectar samples after four applications of Atonik at a nominal rate of 1.0 L product/ha in *Phacelia tanacetifolia* under semi-field conditions. Applications were conducted at BBCH 57-60, 62-63, 63-65 and BBCH 64-67. Nectar from flowers was used for the determination of residues of 5-Nitroguaiacol, o-Nitrophenol and p-Nitrophenol expressed as Sodium 5-Nitroguaiacolate, Sodium o-Nitrophenolate and Sodium p-Nitrophenolate. Samples were collected once after application 4 by hand. The analytical method has been validated in accordance with SANTE/2020/12830, Rev.1, 24. February 2021. The limit of quantification (LOQ) of the analytical method for the determination of Sodium 5-Nitroguaiacolate, Sodium o-Nitrophenolate or Sodium p-Nitrophenolate is 0.01 mg/kg. Maximum storage period – 232 days. The stability was demonstrated for 5-Nitroguaiacol, o-Nitrophenol and p-Nitrophenol expressed as Sodium 5- Nitroguaiacolate, Sodium o-Nitrophenolate and Sodium p-Nitrophenolate in nectar upon storage at ≤ -18 °C in the dark for at least 232 days. <u>Results:</u> Residues of Sodium 5-Nitroguaiacolate and Sodium p-Nitrophenolate were detected in the treated nectar samples at levels of 0.0175 and 0.110 mg/kg in trial -01, 0.00880 (<LOQ) and 0.0720 mg/kg in trial -02, 0.0107 and 0.0603 mg/kg in trial -04 and 0.0141 and 0.0700 mg/kg in trial -05. No residues of Sodium o-Nitrophenolate were detected at or above the limit of quantification (0.01 mg/kg) in the nectar samples of any of the trials. The study is acceptable.

Data point addressed:	**KCA 6.10, 6.10.1**
Author(s) (year):	Kugel, D. (2020)
Title:	Determination of Residues of 5-Nitroguaiacol, *o*-Nitrophenol and *p*-Nitrophenol in Nectar after four Applications of ATONIK containing Sodium 5-Nitroguaiacolate, Sodium *o*-Nitrophenolate and Sodium *p*-Nitrophenolate in *Phacelia tanacetifolia* at 4 Sites in Central and Southern Europe in 2019

Laboratory report / project Number (Doc. No.):	S19-03993 (634-96002)
Testing facility:	Eurofins Agroscience Services Ecotox GmbH, Niefern-Öschelbronn, Germany
Published:	No
Test guideline used:	SANTE/11956/2016 rev. 9, OECD No. 509 (2009), SANCO/825/00, rev. 8.1 (2010), SANCO/3029/99, rev. 4 (2000), OECD Testing and assessment No. 72 and Series on Pesticides No. 39, ENV/JM/MONO(2007)17
Deviations:	None
Previous evaluation:	No, not previously provided
GLP:	Yes; certified by the Landesanstalt für Umwelt Baden-Württemberg, Germany
Acceptability/Reliability:	Yes

As for oilseed rape ~~and apple~~ the application during flowering is proposed, a guideline compliant investigation for the magnitude of residues of Na 5-NG, Na *o*-NP and Na *p*-NP in nectar is summarised below.

I. MATERIALS AND METHODS:

A. MATERIALS

Test material

Description:	**ATONIK**		
Components:	Na 5-NG	Na *o*-NP	Na *p*-NP
CAS #:	67233-85-6	824-39-5	824-78-2
Lot/Batch #:	CG7-101		
Content of a.s. (actual):	0.095 %	0.21 %	0.31 %
Stability of test compound (Expiry date):	31 May 2020		

B. STUDY DESIGN AND METHODS

The nectar residue study on *Phacelia tanacetifolia* has been performed between 2019 and 2020 at two field sites in Germany, one in Spain and one in Southern France. This residue study provides data relevant to conditions in the Northern and Southern European region.

Test system:

Crop:	*Phacelia tanacetifolia* in full bloom
Trial size (treated area):	Germany: 400 m^2; Spain: 200 m^2; France: 200 m^2

1. Test procedure

Four nectar residue trials were conducted with *Phacelia tanacetifolia* between 2019 and 2020 in Germany (2 trials), Spain (1 trial) and Southern France (1 trial). ATONIK was applied four times to the treated plot at a nominal rate of 1.0 L product/ha (nominal rate of components: Na 5-NG: 1.0 g as/ha; Na *o*-NP: 2.0 g as/ha; Na *p*-Na: 3.0 g as/ha).

Three applications were conducted at BBCH 57-60, 62-63 and 63-65. The fourth application was conducted on the day of sampling, in the morning until noon at BBCH 64-67.

Sodium 5-Nitroguaiacolate was applied in the range of 0.95 and 1.02 g as/ha.

Sodium *o*-Nitrophenolate was applied in the range of 1.90 and 2.03 g as/ha.

Sodium *p*-Nitrophenolate was applied in the range of 2.85 and 3.05 g as/ha.

Samples from flowers were collected once after the last application by hand.

The study was performed by Eurofins Agroscience Services Ecotox GmbH, Niefern-Öschelbronn, Germany.

2. Description of analytical procedures

The analytical method was successfully validated within the current study. The method validation is described in more detail in dRR section B5.

Principle of the method:

Na 5-NG was extracted from homogenised nectar with water/acetonitrile (8:2) and centrifuged. Separation was carried out by LC, followed by triplequadrupole mass spectrometric detection (MS/MS).

Na *o*-NP was extracted from homogenised nectar with water/acetonitrile (8:2 and centrifuged. Separation was carried out by LC, followed by triplequadrupole mass spectrometric detection (MS/MS).
Na *p*-NP was extracted from homogenised nectar with water/acetonitrile (8:2) and centrifuged. Separation was carried out by LC, followed by triplequadrupole mass spectrometric detection (MS/MS).
The limit of quantification (LOQ) for Na 5-NG, Na *o*-NP and Na *p*-NP was 0.01 mg/kg in nectar. The limit of detection (LOD) for Na 5-NG, Na *o*-NP and Na *p*-NP was set at 0.003 mg/kg.

For Na 5-NG, the maximum sampling to analysis interval at -18°C was 232 days for nectar. The maximum extraction to quantification interval for Na 5-NG was < 1 day. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na 5-NG in the samples extract. All recoveries were within the range between 70 – 110 %. In addition, the stability of the sample extracts was shown for a period of 7 days within the current study at temperatures between 1 to 10°C. This storage period was not exceeded within the current study. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na *o*-NP, the maximum sampling to analysis interval at -18°C was 232 days for nectar. The maximum extraction to quantification interval for Na *o*-NP was < 1 day. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na *o*-NP in the samples extract. All recoveries were within the range between 70 – 110 %. In addition, the stability of the sample extracts was shown for a period of 7 days within the current study at temperatures between 1 to 10°C. This storage period was not exceeded within the current study. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.
For Na *p*-NP, the maximum sampling to analysis interval at -18°C was 232 days for nectar. The maximum extraction to quantification interval for Na *p*-NP was < 1 day. Procedural recoveries were handled and stored in the same way and for the same time periods as the analytical samples thereby proving stability of Na *p*-NP in the samples extract. All recoveries were within the range between 70 – 110 %. In addition, the stability of the sample extracts was shown for a period of 7 days within the current study at temperatures between 1 to 10°C. This storage period was not exceeded within the current study. Thus, the sample extracts were stable for the storage periods between extraction and analysis in this study.

II. RESULTS AND DISCUSSION

Results of the residue study in nectar are summarised below:

Table A2-9 Residues of Sodium 5-Nitroguaiacolate (Na 5-NG) in nectar

Matrix	Residue levels [mg/kg]
Nectar	< 0.01, 0.0107, 0.0141, 0.0175

Limit of quantification (LOQ) sodium 5-nitroguaiacolate: 0.01 mg/kg in nectar

Table A2-10 Residues of Sodium *o*-Nitrophenolate (Na *o*-NP) in nectar

Matrix	Residue levels [mg/kg]
Nectar	< 0.01 (4)

Limit of quantification (LOQ) sodium *o*-nitrophenolate: 0.01 mg/kg in nectar

Table A2-11 Residues of Sodium *p*-Nitrophenolate (Na *p*-NP) in nectar

Matrix	Residue levels [mg/kg]
Nectar	0.0603, 0.0700, 0.0720, 0.110

Limit of quantification (LOQ) sodium *p*-nitrophenolate: 0.01 mg/kg in nectar

Residues in untreated control samples were below the limit of quantification (LOQ = 0.01 mg/kg).

III. CONCLUSION

Residues of Na 5-NG in nectar are below 0.0175 mg/kg, when ATONIK is applied four times at a nominal rate of 1.0 L product/ha (nominal rate of components: Na 5-NG: 1.0 g as/ha; Na *o*-NP; 2.0 g as/ha Na *p*-NP: 3.0 g as/ha).
Residues of Na *o*-NP in nectar are below the limit of quantification (LOQ = 0.01 mg/kg), when ATONIK is applied four times at a nominal rate of 1.0 L product/ha (nominal rate of components: Na 5-NG: 1.0 g as/ha; Na *o*-NP: 2.0 g as/ha; Na *p*-NP: 3.0 g as/ha).

Residues of Na *p*-NP in nectar are below 0.11 mg/kg, when ATONIK is applied four times at a nominal rate of 1.0 L product/ha (nominal rate of components: Na 5-NG: 1.0 g as/ha Na *o*-NP: 2.0 g as/ha Na *p*-NP: 3.0 g as/ha)

Table A2-12: Summary of the study S19-03993 trials

Trial No. / Location / Year	Commodity/ Variety	Date of 1) Sowing or Planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatment or number and last date	Growth Stage at last treatment	Portion analysed	Residues (mg/Kg)			PHI (days)	Remarks:
			g as/ha[a]	Water (L/ha)	**g as/hL**				Na 5-NG	Na oNP	Na p-NP		
Trial S19-03993-01 Tiefenbach, Baden-Württemberg (DE), 76684 (EU Northern Zone)/ 2019	*Phacelia tanacetifolia*/ Stala	**1.** 24-Jun-2019 **2.** N/A **3.** 09-Jul-2019	**1.** 0.99 + 1.98 + 2.97 **2.** 1.00 + 1.99 + 2.99 **3.** 0.99 + 1.99 + 2.98 **4.** 1.01 + 2.02 + 3.02	**1.** 395.44 **2.** 398.32 **3.** 397.88 **4.** 403.00	-	**1.** 24-Jun-2019 **2.** 01-Jul-2019 **3.** 05-Jul-2019 **4.** 09-Jul-2019	BBCH.65	Control Nectar	nd 0.0175	nd nd	nd 0.110	N/A	LOQ for Na 5-NG, Na o-NP and Na p-NP = 0.01 mg/kg LOD for Na 5-NG, Na o-NP and Na p-NP = 0.003 mg/kg Maximum Storage: 232 days Procedural recoveries (70 – 110 %)
Trial S19-03993-02 Stutensee, Baden-Württemberg (DE), 76297 (EU Northern Zone)/ 2019	*Phacelia tanacetifolia*/ Stala	**1.** 15-May-2019 **2.** N/A **3.** 15-Jul-2019	**1.** 1.00 + 1.99 + 2.99 **2.** 1.00 + 2.00 + 3.00 **3.** 1.01 + 2.01 + 3.01 **4.** 1.00 + 2.00 + 3.00	**1.** 398.16 **2.** 399.80 **3.** 401.00 **4.** 400.60	-	**1.** 25-Jun-2019 **2.** 02-Jul-2019 **3.** 08-Jul-2019 **4.** 15-Jul-2019	BBCH.67	Control Nectar	nd 0.00880 (<LOQ)	nd nd	nd 0.0720	N/A	
Trial S19-03993-04 Ayora, Valencia (ES), 46620 (EU Southern Zone)/ 2019	*Phacelia tanacetifolia*/ Stala	**1.** 04-Mar-2019 **2.** N/A **3.** 07-Jun-2019	**1.** 0.97 + 1.94 + 2.91 **2.** 1.00 + 2.00 + 3.00 **3.** 0.97 + 1.94 + 2.90 **4.** 0.95 + 1.90 + 2.85	**1.** 388.10 **2.** 399.40 **3.** 387.20 **4.** 380.60	-	**1.** 22-May-2019 **2.** 29-May -2019 **3.** 03-Jun-2019 **4.** 07-Jun-2019	BBCH.64 - 66	Control Nectar	nd 0.0107	nd nd	nd 0.0603	N/A	
Trial S19-03993-05 Monheurt, Lot et Garonne (FR), 47160 (EU Southern Zone)/ 2019	*Phacelia tanacetifolia*/ Stala	**1.** 1st week of Nov 2019 **2.** N/A **3.** 28-Apr-2020	**1.** 0.99 + 1.98 + 2.97 **2.** 1.02 + 2.03 + 3.05 **3.** 1.01 + 2.02 + 3.02 **4.** 0.99 + 1.98 + 2.97	**1.** 395.84 **2.** 406.44 **3.** 403.00 **4.** 395.60	-	**1.** 09-Apr-2020 **2.** 16-Apr -2020 **3.** 22-Apr -2020 **4.** 28-Apr -2020	BBCH.66	Control Nectar	nd 0.0141	nd nd	nd 0.0700	N/A	

[a] Na 5-NG, Na o-NP and Na p-NP

Appendix 3 Pesticide Residue Intake Model (PRIMo V. 3.1)

A 3.1 TMDI calculations

A 3.2 IEDI calculations

efsa
European Food Safety Authority
EFSA PRIMo revision 3.1; 2019/03/19

Na-5NG, Na o-NP, Na p-NP

LOQs (mg/kg) range from: to:

Input values | Details - | Supplementary results

Toxicological reference values

ADI (mg/kg bw/day):	0,003	ARfD (mg/kg bw):	0,045
Source of ADI:	SANCO/210/08 – rev. 2	Source of ARfD:	SANCO/210/08 – rev.
Year of evaluation:	2013	Year of evaluation:	2013

Details - | Details - acute risk

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)
41%	NL toddler	1,23	11%	Apples	7%	Maize/corn	5%	Sugar beet roots		
33%	DE child	1,00	12%	Apples	4%	Wheat	4%	Oranges		
29%	NL child	0,86	8%	Sugar beet roots	6%	Apples	4%	Wheat		
27%	GEMS/Food G06	0,81	7%	Wheat	4%	Tomatoes	2%	Potatoes		
22%	RO general	0,65	5%	Wheat	4%	Potatoes	2%	Tomatoes		
20%	FR child 3 15 yr	0,61	5%	Wheat	4%	Sugar beet roots	3%	Oranges		
20%	GEMS/Food G08	0,59	4%	Wheat	4%	Potatoes	3%	Olives for oil production		
19%	PT general	0,57	5%	Potatoes	4%	Wheat	2%	Wine grapes		
18%	GEMS/Food G15	0,55	5%	Wheat	4%	Potatoes	1%	Tomatoes		
18%	GEMS/Food G07	0,53	4%	Wheat	4%	Potatoes	1%	Wine grapes		
17%	GEMS/Food G10	0,52	4%	Wheat	3%	Potatoes	2%	Olives for oil production		
17%	UK toddler	0,52	4%	Wheat	3%	Potatoes	3%	Sugar beet roots		
17%	DE women 14-50 yr	0,51	5%	Sugar beet roots	3%	Apples	2%	Wheat		
17%	ES child	0,50	4%	Wheat	3%	Olives for oil production	2%	Oranges		
16%	DE general	0,47	4%	Sugar beet roots	2%	Apples	2%	Wheat		
16%	FR toddler 2 3 yr	0,47	3%	Apples	3%	Wheat	3%	Sugar beet roots		
15%	GEMS/Food G11	0,46	4%	Potatoes	4%	Wheat	2%	Apples		
15%	UK infant	0,44	3%	Potatoes	3%	Wheat	2%	Apples		
15%	DK child	0,44	4%	Wheat	2%	Potatoes	2%	Apples		
13%	SE general	0,39	4%	Potatoes	3%	Wheat	1%	Apples		
13%	NL general	0,39	3%	Sugar beet roots	2%	Potatoes	2%	Wheat		
13%	IT toddler	0,38	7%	Wheat	1%	Tomatoes	0,9%	Potatoes		
12%	FI 3 yr	0,35	5%	Potatoes	1%	Wheat	1%	Cucumbers		
12%	IE adult	0,35	2%	Wheat	2%	Potatoes	1%	Wine grapes		
11%	ES adult	0,32	2%	Wheat	2%	Olives for oil production	1%	Oranges		
10%	FR adult	0,29	2%	Wine grapes	2%	Wheat	0,8%	Sugar beet roots		
9%	FI 6 yr	0,28	4%	Potatoes	1,0%	Wheat	0,7%	Cucumbers		
9%	IT adult	0,27	4%	Wheat	1%	Tomatoes	0,8%	Apples		
8%	PL general	0,25	3%	Potatoes	2%	Apples	0,9%	Cherries (sweet)		
8%	UK vegetarian	0,25	2%	Wheat	1%	Potatoes	0,9%	Oranges		
8%	FR infant	0,24	2%	Potatoes	2%	Apples	1%	Sugar beet roots		
8%	LT adult	0,24	3%	Potatoes	2%	Apples	1%	Wheat		
7%	UK adult	0,22	2%	Wheat	1%	Potatoes	1%	Wine grapes		
6%	DK adult	0,19	1%	Potatoes	1%	Wheat	1,0%	Apples		
5%	FI adult	0,14	1%	Potatoes	0,6%	Apples	0,6%	Tomatoes		
3%	IE child	0,09	1%	Wheat	0,6%	Potatoes	0,3%	Apples		

Conclusion:
The estimated long-term dietary intake (TMDI/NEDI/IEDI) was below the ADI.
The long-term intake of residues of Na-5NG, Na o-NP, Na p-NP is unlikely to present a public health concern.

A 3.3 IESTI calculations - Raw commodities

Show results for all crops

Results for children

No. of commodities for which ARfD/ADI is exceeded (IESTI): ---

IESTI

Highest % of ARfD/ADI	Commodities	MRL / input for RA (mg/kg)	Exposure (µg/kg bw)
10%	Potatoes	0,03 / 0,03	4,6
10%	Melons	0,03 / 0,03	4,6
9%	Oranges	0,03 / 0,03	4,0
8%	Cherries (sweet)	0,3 / 0,3	3,7
8%	Cherries (sweet)	0,3 / 0,3	3,7
7%	Apples	0,03 / 0,03	3,2
5%	Table grapes	0,03 / 0,03	2,2
4%	Cucumbers	0,03 / 0,03	2,0
4%	Carrots	0,03 / 0,03	1,9
4%	Sweet peppers/bell	0,03 / 0,03	1,8
4%	Tomatoes	0,03 / 0,03	1,7
3%	Courgettes	0,03 / 0,03	1,4
2%	Aubergines/egg	0,03 / 0,03	0,75
2%	Onions	0,03 / 0,03	0,68
1%	Strawberries	0,03 / 0,03	0,49
1,0%	Wheat	0,03 / 0,03	0,43
0,9%	Table olives	0,12 / 0,12	0,40
0,8%	Rice	0,03 / 0,03	0,38
0,6%	Wine grapes	0,03 / 0,03	0,28
0,6%	Raspberries (red and	0,03 / 0,03	0,28
0,5%	Currants (red, black	0,03 / 0,03	0,24
0,4%	Maize/corn	0,03 / 0,03	0,20
0,3%	Olives for oil	0,12 / 0,12	0,15
0,2%	Sunflower seeds	0,03 / 0,03	0,10
0,09%	Rapeseeds/canola	0,03 / 0,03	0,04
0,03%	HOPS (dried)	0,3 / 0,3	0,01

Results for adults

No. of commodities for which ARfD/ADI is exceeded (IESTI): ---

IESTI

Highest % of ARfD/ADI	Commodities	MRL / input for RA	Exposure (µg/kg bw)
7%	Cherries (sweet)	0,3 / 0,3	3,0
3%	Watermelons	0,03 / 0,03	1,2
3%	Melons	0,03 / 0,03	1,2
2%	Table grapes	0,03 / 0,03	1,0
2%	Oranges	0,03 / 0,03	0,92
2%	Potatoes	0,03 / 0,03	0,90
2%	Apples	0,03 / 0,03	0,84
2%	Cucumbers	0,03 / 0,03	0,83
2%	Aubergines/egg	0,03 / 0,03	0,81
2%	Wine grapes	0,03 / 0,03	0,71
2%	Courgettes	0,03 / 0,03	0,70
1%	Carrots	0,03 / 0,03	0,59
1%	Sweet peppers/bell	0,03 / 0,03	0,49
1%	Tomatoes	0,03 / 0,03	0,48
1,0%	Onions	0,03 / 0,03	0,45
0,6%	Strawberries	0,03 / 0,03	0,28
0,6%	Rice	0,03 / 0,03	0,26
0,6%	Wheat	0,03 / 0,03	0,25
0,4%	Currants (red, black	0,03 / 0,03	0,20
0,4%	Raspberries (red and	0,03 / 0,03	0,16
0,3%	Table olives	0,12 / 0,12	0,12
0,2%	Olives for oil	0,12 / 0,12	0,09
0,1%	Maize/corn	0,03 / 0,03	0,07
0,1%	HOPS (dried)	0,3 / 0,3	0,05
0,07%	Sunflower seeds	0,03 / 0,03	0,03
0,05%	Poppy seeds	0,03 / 0,03	0,02
0,04%	Rapeseeds/canola	0,03 / 0,03	0,02

Total number of commodities exceeding the ARfD/ADI in children and adult diets (IESTI calculation)

A 3.4 IESTI calculations - Processed commodities

Processed commodities

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):			---
IESTI				IESTI			
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)
7%	Sugar beets (root) / su	0,03 / 0,36	3,3	3%	Sugar beets (root) /	0,03 / 0,36	1,3
6%	Potatoes / fried	0,03 / 0,03	2,8	2%	Apples / juice	0,03 / 0,03	1,00
4%	Potatoes / dried (flake	0,03 / 0,14	1,8	2%	Courgettes / boiled	0,03 / 0,03	0,69
4%	Apples / juice	0,03 / 0,03	1,6	1%	Wine grapes / juice	0,03 / 0,03	0,62
4%	Oranges / juice	0,03 / 0,03	1,6	1%	Oranges / juice	0,03 / 0,03	0,45
3%	Wine grapes / juice	0,03 / 0,03	1,3	0,9%	Currants (red, black	0,03 / 0,03	0,38
2%	Carrots / juice	0,03 / 0,03	1,1	0,8%	Maize / oil	0,03 / 0,75	0,38
2%	Courgettes / boiled	0,03 / 0,03	1,1	0,6%	Wine grapes / wine	0,03 / 0,03	0,28
2%	Currants (red, black a	0,03 / 0,03	0,86	0,6%	Onions / boiled	0,03 / 0,03	0,28
2%	Maize / oil	0,03 / 0,75	0,70	0,6%	Potatoes / chips	0,03 / 0,03	0,25
1%	Tomatoes / juice	0,03 / 0,03	0,57	0,5%	Tomatoes /	0,03 / 0,03	0,25
0,8%	Wheat / milling (flour	0,03 / 0,03	0,36	0,5%	Carrots / canned	0,03 / 0,03	0,24
0,8%	Raspberries / juice	0,03 / 0,03	0,35	0,4%	Table grapes / raisins	0,03 / 0,14	0,17
0,6%	Tomatoes / sauce/pur	0,03 / 0,03	0,29	0,4%	Potatoes / dried	0,03 / 0,14	0,17
0,5%	Olives for oil producti	0,12 / 0,24	0,22	0,3%	Table olives /	0,12 / 0,12	0,15
0,4%	Rice / milling (polishi	0,03 / 0,01	0,18	0,3%	Wheat / bread/pizza	0,03 / 0,03	0,13
0,4%	Wheat / milling (whol	0,03 / 0,03	0,17	0,3%	Rice / milling	0,03 / 0,01	0,12
0,3%	Table olives / canned	0,12 / 0,12	0,13	0,3%	Wheat / pasta	0,03 / 0,03	0,11
0,2%	Sunflower seeds / oils	0,03 / 0,06	0,07	0,2%	Wheat / bread	0,03 / 0,03	0,10
0,1%	Maize / processed (no	0,03 / 0,03	0,06	0,10%	Hops / beer	0,3 / 0	0,04
0,0%	Rapeseeds / oils	0,03 / 0,06	0,02	#ČÍSLO!	#ČÍSLO!	#ČÍSLO!	#ČÍSLO!
Expand/collapse list							

Conclusion:

No exceedance of the toxicological reference value was identified for any unprocessed commodity.

A short term intake of residues of Na-5NG, Na o-NP, Na p-NP is unlikely to present a public health risk.

For processed commodities, no exceedance of the ARfD/ADI was identified.