

FINAL REGISTRATION REPORT

Part B

Section 7

Metabolism and Residues

Detailed summary of the risk assessment

Product code: AKO 600 SC

Product name(s): Tanger 600 SC, Libretto 600 SC

Chemical active substance:

Aclonifen, 600 g/L

Central Zone

Zonal Rapporteur Member State: Poland

Co-Rapporteur Member State: Hungary, Slovakia, Germany,
Romania

CORE ASSESSMENT

(authorization)

Applicant: Innvigo Sp. z o.o.

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When	What
June 2025	zRMS evaluation
October 2025	Following commenting period
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7 Metabolism and residue data (KCA section 6)

According to Regulation 1107/2009 data protection for data at European level lasts for 10 years. Due to the fact that the aclonifen annex I was approved on 1/08/2009, data protection has expired 1/08/2019. Therefore, Innvigo can use the European data for aclonifen for the registration of the product AKO 600 SC [Libretto 600 SC, Tanger 600 SC]

7.1 Summary and zRMS Conclusion

This document reviews the metabolism and residue data relevant for the product AKO 600 SC containing the active substance aclonifen. A full risk assessment according to Uniform Principles is provided which demonstrates that the product is safe for consumers.

Stability

Residues of aclonifen were stable at $\leq 18^{\circ}\text{C}$ for 12 and 24 months in high water/dry and high protein/oil content commodities respectively. The storage stability data includes the required use of aclonifen in AKO 600 SC.

Metabolism in plants

The metabolism of aclonifen is comparable in different investigated commodities. Hence, a residue definition for monitoring and risk assessment is proposed as aclonifen.

The metabolism in rotational crops is similar to metabolism in primary crops.

Metabolism in animals

The metabolism in ruminants and rats is similar. The definition of the residues for monitoring and risk assessment in commodities of ani-mal origin is proposed as parent aclonifen only.

Nature of residues in processed commodities

As residues of aclonifen exceeding 0.1 mg/kg are not expected in the treated crops and the chronic exposure does not exceed 10 % of the ADI, there is no need to investigate the effect of industrial and/or household processing.

Magnitude of residues in plants

The evaluated representative use is as a pre-emergence herbicide on sunflower. Sufficient number of residue trials was reviewed. Aclonifen was applied to soil prior to emergence of the sunflowers, with application rates ranging from 2.1 to 4.8 kg a.s./ha. For Northern Europe, 9 field trials were found to match the critical GAP. No residues above the LOQ for aclonifen have been found in sunflower seed at harvest. In the required GAP, the dose is 1.8 kg a.s./ha which is less critical compared to already evaluated GAP. The proposed use of aclonifen in sunflower is acceptable. According to SANTE/2019/12752 rev.02 guidelines, it is possible to extrapolate residue data from any representative of the oilseed group, with the exception of peanuts/peanuts groundnuts (0401020), to the entire oilseed group (0401000), except peanuts/groundnuts (0401020) and caraway (0820030) when the product is used before forming of the edible part. Thus, the application in rapeseed, flax, poppy, mustard, soybeans, pumpkin seeds and hemp is acceptable. The use in tobacco, forest plants, ornamentals, basket willow, purple willow, energetic willow and Christmas trees was not evaluated as such crops are not predicted for consumption.

The Applicant presented new studies for residues of aclonifen in potatoes. The residues in potato were <0.001 mg/kg which is well below of the current MRL of 0.02 mg/kg.

Livestock feeding

The calculated dietary burdens were found to be below the trigger value of 0.004 mg/kg bw hence no livestock feeding studies are required.

Rotational crops

EFSA Review of the existing MRLs for aclonifen (2015): “The rotational crop metabolism study showed that in the majority of crops no significant residues occurred. Nevertheless, since significant residues were found in carrots grown in rotation, during the peer review a data gap was set for a rotational crop residue study in root and tuber vegetables (EFSA, 2008). Two additional studies investigating the magnitude of residues in turnips planted 30 and 60 days after application of 2.4 kg a.s./ha to bare soil were submitted and evaluated in the framework of this review (Germany, 2011). According to the results of both studies, no residues are expected in root and tuber vegetables grown in rotation with crops treated with aclonifen (residues were below the LOQ of 0.01 mg/kg in all samples of leaves and roots analysed).”

Magnitude of residues in processed commodities

As residues are less than 0.1 mg/kg and theoretical maximum daily intake (TMDI) is <10% of the ADI for any EU consumer group diet, therefore magnitude of residue in processed commodities is not required.

EFSA conclusion (2008): “Eleven supervised field trials analysing residues in sunflower seed raw and processed to oil and press cake are available. However, since residues in raw and processed samples were always <LOQ, processing factors could not be derived (EFSA, 2008). The studies are considered as additional information only and additional studies are not required.

Other studies

Sunflower, rapeseed, flaxseed, poppy seeds, mustard seeds, soya beans and pumpkin seeds are considered a melliferous crop according to the technical guidelines (SAN-TE/11956/2016 rev. 9). Therefore, the evaluation of residues in honey is in principle necessary. However, available residue data for sunflower (9 NEU and 11 SEU, DAR, 2006), conducted with the exaggerated pre-emergence application rates ranged from 2100 to 4800 g a.s./ha, indicated residues of aclonifen in sunflower seeds always below the LOQ at mature harvest. Thus, there is no risk of residues above LOQ following intended uses (pre-emergence application of 1.8 kg/ha). It is therefore considered that an application well before flowering at a dose below that already assessed in the DAR will not result in the residues of aclonifen above MRL of 0.05 mg/kg in honey when the product is used according to the recommended GAP. The intended maximum seasonal rate in this submission is 84 g a.s./ha, much lower than presented in studies evaluated at EU level for honey (2 x 100 g/ha). Therefore, no risk of exceeding the MRL for honey is expected.

Consumer risk assessment

The proposed uses of aclonifen in the product AKO 600 SC do not represent unacceptable risks for the consumers. The calculation of the TMDI led to an utilisation of the ADI of 2% based on NL toddler being the population group with the highest value. For this diet, the highest contributor is milk with 0.9% of the ADI. As no ARfD was established for aclonifen no acute risk assessment was performed.

For potato AKO 600 SC is used in tank mix with BOA PRO 480 EC containing clomazone as an active substance. For clomazone, the Applicant refers to the residue data uses in potatoes evaluated by IOR-PIB and approved in Poland (RR, 21.01.2013). Sufficient residue trials are available to support the GAP uses in potatoes in NEU. In total, 13 residue trials were conducted involving pre-emergence or early post-emergence application at or above the required GAP rate, i.e. 96 g a.s./ha.

In all potato trials performed in NEU, the residues level in potato tubers was <0.01 mg/kg.

Dietary risk assessment for the active substance was recalculated by zRMS using EFSA Primo 3.1. The TMDI estimation is always below 1 % of the ADI. As no acute reference has been set for clomazone, there is no need to evaluate the acute risk for this active substance.

It can be concluded that the use of clomazone does not lead to unacceptable risk for consumer when applied according to the recommendations.

7.1.1 Critical GAP(s) and overall conclusion

Selection of critical uses and justification

Overall conclusion

Data gaps

Noticed data gaps are: none

- ~~data gap 1~~
- ~~data gap 2~~
- ~~data gap 3~~

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

GAP ver. 1.4 date: 22.05.2024

PPP (product name/code): AKO 600 SC / Tanger 600 SC, Libretto 600 SC
Active substance 1: Aclonifen
Safener:
Synergist:
Applicant: Innvigo sp. z o.o.
Zone(s): Central Zone
Verified by MS:
Field of use: Herbicide

Formulation type: SC
Conc. of as 1: 600 g/L
Conc. of as 2: (c)
Conc. of as: (c)
Conc. of safener: (c)
Conc. of synergist: (c)
Professional use:
Non professional use:

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Use- No. (e)	Member state(s)	Crop and/ or situation (crop destination / purpose of crop)	F, Fn, Fpn G, Gn, Gpn or I	Pests or Group of pests controlled (additionally: devel- opmental stages of the pest or pest group)	Application				Application rate			PHI (days)	Remarks: e.g. g safen- er/synergist per ha (f)	Conclusion
					Method / Kind	Timing / Growth stage of crop & season	Max. number a) per use b) per crop/ season	Min. interval between appli- cations (days)	kg or L product / ha a) max. rate per appl. b) max. total rate per crop/season	g or kg as/ha a) max. rate per appl. b) max. total rate per crop/season	Water L/ha min / max			Groundwater
Zonal uses (field or outdoor uses, certain types of protected crops)														
1	PL, DE	Potato <i>Solanum tuberosum</i> SOLTU (0211000)	F	Mono and dicots weeds	Spray	BBCH 01-09	a) 1 b) 1	n/a	a) 1.95 - 3.0 L/ha b) 1.95 - 3.0 L/ha	a) 1.17 - 1.8 kg as/ha b) 1.17 - 1.8 kg as/ha	200- 400	40		A

2	PL	Potato <i>Solanum tuberosum</i> SOLTU (0211000)	F	Mono and dicots weeds	Spray	BBCH 01-08	a) 1 b) 1	n/a	a) 0.8- 2.0 L/ha AKO 600 SC + 0.2 L/ha Boa Pro 480 EC (clomazon 480 g/L) b) 0.8-2.0 L/ha AKO 600 SC + 0.2 L/ha Boa Pro 480 EC (clomazon 480 g/L)	a) 0.48 - 1.2 + 0.096 kg as/ha b) 0.48 - 1.2 + 0.096 kg as/ha	200- 400	40		
3	HU, RO, SK	Sunflower <i>Helianthus annuus</i> HELAN (0401050)	F	Mono and dicots weeds	Spray	BBCH 00-08	a) 1 b) 1	n/a	a) 1.95 - 3.0 L/ha b) 1.95 - 3.0 L/ha	a) 1.17 - 1.8 kg as/ha b) 1.17 - 1.8 kg as/ha	200- 300	n/a		A
Interzonal uses (use as seed treatment, in greenhouses (or other closed places of plant production), as post-harvest treatment or for treatment of empty storage rooms)														
4														
5														
Minor uses according to Article 51 (zonal uses)														
6	PL	Sunflower <i>Helianthus annuus</i> HELAN (0401050)	F	Mono and dicots weeds	Spray	BBCH 00-08	a) 1 b) 1	n/a	a) 1.95 - 3.0 L/ha b) 1.95 - 3.0 L/ha	a) 1.17 - 1.8 kg as/ha b) 1.17 - 1.8 kg as/ha	200- 300	n/a		A
7	PL, DE, SK	Spring rapeseed BRNSN (0401060)	F	Mono and dicots weeds	Spray	BBCH 00-08	a) 1 b) 1	n/a	a) 1.95 - 3.0 L/ha b) 1.95 - 3.0 L/ha	a) 1.17 - 1.8 kg as/ha b) 1.17 - 1.8 kg as/ha	200- 300	n/a		A
8	PL, HU, RO, SK	Flax LIUUT (0401010)	F	Mono and dicots weeds	Spray	BBCH 00-08	a) 1 b) 1	n/a	a) 1.95 - 3.0 L/ha b) 1.95 - 3.0 L/ha	a) 1.17 - 1.8 kg as/ha b) 1.17 - 1.8 kg as/ha	200- 300	n/a		A
9	PL, DE, HU, RO, SK	Opium poppy <i>Papaver somniferum</i> PAPSO (0401030)	F	Mono and dicots weeds	Spray	BBCH 00-08	a) 1 b) 1	n/a	a) 1.95 - 3.0 L/ha b) 1.95 - 3.0 L/ha	a) 1.17 - 1.8 kg as/ha b) 1.17 - 1.8 kg as/ha	200- 300	n/a		A
10	PL, RO, SK	Mustard 3MUSC (0401080)	F	Mono and dicots weeds	Spray	BBCH 00-08	a) 1 b) 1	n/a	a) 1.95 - 3.0 L/ha b) 1.95 - 3.0 L/ha	a) 1.17 - 1.8 kg as/ha b) 1.17 - 1.8 kg as/ha	200- 300	n/a		A

[illegible]

22														
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2 – AKO 600 SC + BOA PRO 480 EC

* 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 AKO 600 SC is composed of aclonifen.

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

Reference value	Source	Year	Value	Study relied upon	Safety factor
Aclonifen - Parent compound (if applicable)					
ADI	EFSA Scientific Report (2008) 149, 1-80	2008	0.07 mg/kg bw/day	2-year, rat supported by multigeneration study in rat and carcinogenicity study in mouse	100
ARfD	EFSA Scientific Report (2008) 149, 1-80	2008	Not allocated, not necessary - experts concluded not to allocate an ARfD since the data available did not show an acute toxicological alert		

7.1.2.1 Summary for aclonifen

Table 7.1-3: Summary for aclonifen

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	Potato	Yes	Yes	Yes	Yes	Yes	No	No
2, 5-12	Sunflower Spring rape-seeds, Flax, breadseed poppy, mustard, soyabeans, pumpkin, hemp	Yes	Yes	Yes	Yes	Yes	No	No
14-20	Tobacco, forest nurseries, ornamental shrubs, basket willow, purple willow, forest tree, christmas tree, energetic willow	Yes	Yes	Yes	Yes	Yes	No	No

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

It has been shown that aclonifen residues are stable when stored at or below -18°C. Stability was tested for at least 24 months in sunflower seeds, peas and tomatoes and for at least 12 months in corn grain, fodder corn and potatoes. In the case of aclonifen metabolism studies, it has been proven to be comparable in all products tested. Aclonifen undergoes monohydroxylation on the phenyl ring and/or reduction of the nitro group. As a secondary route, the diphenyl ether bond is broken, resulting in the formation of several derivatives. The proposed residue definition is aclonifen. Moreover, based on the studies, the magnitude of residues was not found to exceed the LOQ value for aclonifen, therefore its concentration is

considered as less than 0.1 mg/kg.

7.1.2.2 Summary for AKO 600 SC

Table 7.1-4: Information on AKO 600 SC (KCA 6.8)

Crop	PHI for AKO 600 SC proposed by applicant	PHI/ Withholding period* sufficiently supported for			PHI for product code proposed by zRMS	zRMS Comments (if different PHI proposed)
		Aclonifen				
Potato	NR	NR			NR	-
Sunflower Spring rapeseeds, Flax, breadseed poppy, mustard, soyabeans, pumpkin, hemp	NR	NR			NR	-
Tobacco, forest nurseries, ornamental shrubs, basket willow, purple willow, forest tree, christmas tree, energetic willow	NR	NR			NR	-

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

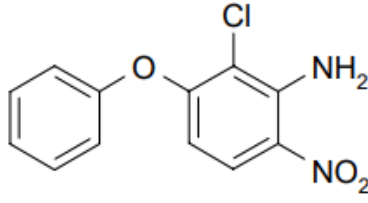
Waiting period before planting succeeding crops				Overall waiting period proposed by zRMS for AKO 600 SC
Crop group	Led by aclonifen			
Leafy vegetables	NR	NR	NR	No proposal or comment
Root vegetables	NR	NR	NR	No proposal or comment
...	NR	NR	NR	No proposal or comment

NR: not relevant

7.2 Aclonifen

General data on aclonifen are summarized in the table below (last updated 19/08/2016)

Table 7.2-1: General information on aclonifen

Active substance (ISO Common Name)	Aclonifen
IUPAC	2-chloro-6-nitro-3-phenoxyaniline
Chemical structure	
Molecular formula	C ₁₂ H ₉ ClN ₂ O ₃
Molar mass	264.7 g/mol
Mode of action (if available)	Blocking chlorophyll production in weeds
Systemic	Yes
Company (ies)	Bayer CropScience SA (BAY4)
Rapporteur Member State (RMS)	RMS for ongoing/upcoming approval/renewal - Netherlands Co-RMS for ongoing/upcoming approval/renewal - Norway
Approval status	Approved
Restriction	Aclonifen SANCO/161/08 – rev. 2 28 September 20121 - the specification of the technical material as commercially manufactured must be confirmed and supported by appropriate analytical data. The test material used in the toxicity dossiers should be compared and verified against this specification of the technical material; - the protection of the operators safety. Authorised conditions of use must prescribe the application of adequate personal protective equipment and risk mitigation measures to reduce the exposure; - the residues in rotational crops and evaluate the dietary exposure of consumers; - the protection of birds, mammals, aquatic organisms and non target plants. In relation to these identified risks, risk mitigation measures, such as buffer zones, should be applied where appropriate.
Review Report	SANCO/161/08 – rev. 2 28 September 2012
Current MRL regulation	Reg. (EU) 2021/1531
Peer review of MRLs according to Article 12 of Reg No 396/2005 EC performed	Yes - EFSA Journal 2020;18(5):6102
EFSA Journal : Conclusion on the peer review	EFSA Scientific Report (2008) 149, 1-80

EFSA Journal: conclusion on article 12	Yes - EFSA Journal 2020;18(5):6102
Current MRL applications on intended uses	n/a

* Notifier in the EU process to whom the a.s. belong(s)

** If yes: EFSA, YYYY - see list of references

7.2.1 Stability of Residues (KCA 6.1)

7.2.1.1 Stability of residues during storage of samples

Available data

No new data submitted in the framework of this application.

Table 7.2-2: Summary of stability data achieved at $\leq -18^{\circ}\text{C}$ (unless stated otherwise)

Matrix	Characteristics of the matrix	Acceptable Maximum Storage duration	Reference
Data relied on in EU			
Plant products			
Potato	High water content	12 months	Wasser, C. (2000, RIP2005-72) - Storage stability of aclonifen in corn grains, corn plants and potatoes samples at less than -18°C
Maize grains	Dry commodities	12 months	Wasser, C. (2000, RIP2005-72) - Storage stability of aclonifen in corn grains, corn plants and potatoes samples at less than -18°C
Pea	High protein content	24 months	Uceda, L and Kieken, J.-L. (2004, RIP2005-73) Aclonifen: : Storage stability at around -20°C in sunflower seed, pea and tomato
Sunflower seed	High oil content	24 months	Uceda, L and Kieken, J.-L. (2004, RIP2005-73) Aclonifen: : Storage stability at around -20°C in sunflower seed, pea and tomato

Conclusion on stability of residues during storage

Aclonifen residues were stable at -18°C or below for at least 24 months in sunflower seed, pea and tomato and for at least 12 months in maize grain, maize forage and potato. All four main categories of plant matrices for which storage stability data may be required have been addressed.

zRMS comment: No new studies have been submitted in the framework of this application. The Applicant referred to the studies evaluated in the renewal process of the substance. Residues of aclonifen were stable at $\leq -18^{\circ}\text{C}$ for 12 and 24 months in high water/dry and high protein/oil content commodities respectively. The storage stability data includes the required use of aclonifen in AKO 600 SC.

7.2.1.2 Stability of residues in sample extracts (KCA 6.1)

Not relevant - analysis time were less than 24 hours between extraction and analysis or it was shown that study extracts are stable.

7.2.1.3 Nature of residue in primary crops (KCA 6.2.1)

Available data

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								
Root and tuber vegetables	Potatoes	[¹⁴ C]-aniline radiolabelled aclonifen	F	Soil application: 2.5 Foliar application: 1.5	-	Soil application: 93 days after planting and treatment Foliar application: 100 days after planting and 42 days after treatment	-	Schlüter, H. and Pistel, F. (1992, RIP94-00251) Final report - Metabolism of aclonifen in potatoes
Pulses and oilseeds	Peas	[¹⁴ C]-aniline radiolabelled aclonifen	F	Pre-emergence treatment: 2.97 Post-emergence treatment: 0.396	-	Pre-emergency days - 0, 70, 78, 108 Post-emergency days – 0, 42, 57, 93	-	Newby, S.E. (1996, RIP2005-75) [14C]-Aclonifen: Metabolism in Peas
Cereals	Wheat	[¹⁴ C]-aniline radiolabelled aclonifen	F	Pre-emergence treatment: 3.248 Post-emergence treatment: 0.303	-	Pre-emergency days – 0, 54, 76, 152 Post-emergency days – 0, 22, 41, 138	-	Newby, S.E. (1996, RIP2005-76) Final Report - Aclonifen: Metabolism in Wheat

Conclusion on metabolism in primary crops

The metabolism, distribution and expression of residues in plants has been investigated in peas, wheat and potatoes following either pre-emergence or post-emergence application of [¹⁴C-aniline]-labelled aclonifen. Peas are representative for the crop group pulses and oilseeds thus covering the intended use in sunflowers. Metabolism studies were conducted at rates in excess of the intended application rate of 2.4 kg as/ha. Underground edible plant parts that were in contact with the soil exhibited comparably high TRRs while all aerial portions of plants that represented human-edible commodities had low overall residue levels. The majority of the extractable residue in virtually all plant parts was aclonifen. The total residue level in wheat grain and peas was very low and the residue was not characterised further. In wheat straw the amount of aclonifen was low and accompanied by several metabolites in similarly low individual amounts. The metabolism of aclonifen is comparable in all investigated agricultural commodities. Aclonifen undergoes mono-hydroxylation on the phenyl ring (RPA 407074) and/or reduction of the nitro group (RPA 407291). As a minor pathway, cleavage of the diphenylether bond occurs, resulting in the formation of RPA 508285 and RPA 407288.

The proposed residue definition for plants is aclonifen.

zRMS comment: The Applicant referred to the studies provided during review process of the substance. According to the results of the available metabolism studies, the metabolism of aclonifen is comparable in all investigated commodities. Hence, a residue definition for monitoring and risk assessment is proposed

as Aclonifen.

7.2.1.4 Nature of residue in rotational crops (KCA 6.6.1)

Available data

No new data submitted in the framework of this application.

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

Crop group	Crop	Label position	Application and sampling details					Reference
			Method, F or G *	Rate (kg a.s./ha)	Sowing intervals (DAT)	Rotation intervals (DAT)	Remarks	
EU data								
Cereals	Barley	[¹⁴ C-aniline]-radiolabelled aclonifen	F	3.72	0	29, 120, 365	-	McEwen, A. (2003, RIP2005-99) [Aniline-U- ¹⁴ C]-aclonifen: A Confined Crop Rotation Study
Leafy vegetables	Spinach	[¹⁴ C-aniline]-radiolabelled aclonifen	F	3.72	0	29, 120, 365	-	McEwen, A. (2003, RIP2005-99) [Aniline-U- ¹⁴ C]-aclonifen: A Confined Crop Rotation Study
Root and tuber vegetables	Carrot	[¹⁴ C-aniline]-radiolabelled aclonifen	F	3.72	0	29, 120, 365	-	McEwen, A. (2003, RIP2005-99) [Aniline-U- ¹⁴ C]-aclonifen: A Confined Crop Rotation Study

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

Summary of plant metabolism studies reported in the EU

Uptake of radioactive residues into rotational crops was observed for all investigated crops (root crop, leafy crop, cereal crop) but was not pronounced for leafy and cereal crops. The radioactive residue became less available for plant uptake with time. TRRs in carrot foliage, spinach, immature barley, barley grain and barley chaff remained below 0.1 mg/kg during all three rotation intervals (29, 120 and 365 days). TRR in carrot root reached a maximum of 0.491 mg as-eq/kg after the first rotation interval, decreasing to 0.200 mg as-eq/kg after the second and to 0.035 mg as-eq/kg after the third rotation interval. Residues in barley straw amounted to 0.245 mg as-eq/kg after the first rotation interval, decreasing to 0.040 and 0.077 mg as-eq/kg after the second and the third interval, respectively.

Conclusion on metabolism in rotational crops

The extractable residue in carrot root consisted exclusively of aclonifen which was also the only major component in carrot foliage and the only identifiable compound in spinach. Barley grain contained one major polar component which could not be identified. Taking into account the low absolute amount of this metabolite (0.004 mg/kg), identification is not required. Barley straw comprised aclonifen and RPA 508285 in minor amounts. In none of the samples further compounds occurred in individual amounts > 10 %. The unidentified compounds predominantly were polar in nature. It can be concluded from the study that the only significant residue to be considered in rotational crops is the parent - aclonifen.

zRMS comment: The Applicant referred to the studies provided during review process of the substance. Based on the results of the metabolism studies, it is concluded that metabolism in rotational crops is similar to metabolism in primary crops.

7.2.1.5 Nature of residues in processed commodities (KCA 6.5.1)

Available data

No new data submitted in the framework of this application.

Conclusion on nature of residues in processed commodities

According to w COMMISSION REGULATION (EU) No 283/2013, studies on the nature of residues in processing shall be provided where residues in products of plant or animal origin subject to processing may occur at a level of or higher than 0,01 mg/kg (based on the residue definition for risk assessment for the raw commodity). Since level of residues in plants presented in the GAP and its processed products is < LOQ, studies on the nature of residues in processed commodities are not required.

zRMS comment: As residues of aclonifen exceeding 0.1 mg/kg are not expected in the treated crops and the chronic exposure does not exceed 10 % of the ADI, there is no need to investigate the effect of industrial and/or household processing.

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

Table 7.2-5: Summary of the nature of residues in commodities of plant origin

Endpoints	
Plant groups covered	Root and tuber vegetables (Potatoes), Cereals (Wheat), Pulses and oilseeds
Rotational crops covered	Cereals, Leafy vegetables, Root and tuber vegetables
Metabolism in rotational crops similar to metabolism in primary crops?	Yes
Processed commodities	a.s. is stable
Residue pattern in processed commodities similar to pattern in raw commodities?	n/a
Plant residue definition for monitoring	Aclonifen, EFSA Scientific Report (2008) 149, 1-80, Regulation (EU) 2021/1531
Plant residue definition for risk assessment	Aclonifen, EFSA Scientific Report (2008) 149, 1-80
Conversion factor from enforcement to RA	not applicable

* If residue pattern in processed commodities is not similar to that in raw commodities

~~** A more recent proposal by EFSA may be provided as additional information (EFSA RO XXXX).~~
~~*** If no EFSA proposal is available, a proposal should be made by the applicant/zRMS.~~

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

Available data

No new data submitted in the framework of this application.

Table 7.2-6: Summary of animal metabolism studies

Group	Species	Label position	No of animal	Application details		Sample details		Reference
				Rate (mg/kg bw/d)	Duration (days)	Commodity	Time of sampling	
EU data								
Lactating ruminants	Goat	[¹⁴ C-aniline]-labelled aclonifen	3	a) 0.036 b) 0.34 c) 2.76	7	Milk	twice daily	DAR – Aclonifen – Annex B.7: Residue data - Thornley, K. and Lappin, G. (1998, RIP2005-77

Summary of plant metabolism studies reported in the EU

The metabolism of aclonifen has been investigated in lactating goats following oral administration of three different doses of [¹⁴C-aniline]-labelled aclonifen (Thornley, K. and Lappin, G. (1998, RIP2005-77)). Taking into account the theoretical residue level in livestock feed, which has been calculated for the representative GAP of aclonifen, only the results from the low dose group – though still overdosed - were relevant for risk assessment. The majority of the administered radioactivity (74 - 77 %) was excreted via urine and faeces. Residues in milk were low throughout the dosing period (< 0.01 mg/kg in the low dose group) and mainly comprised aclonifen and a couple of polar metabolites. A plateau concentration was reached by day 4. Residues in meat and fat were below the LOQ. Residues in kidney were also clearly below 0.01 mg/kg in the low dose group. Main metabolite in kidney in the higher dose groups was RPA 407074- methylimidazole together with one polar metabolite fraction, while aclonifen did not occur in kidney. Liver contained levels of radioactivity above the LOQ (0.026 mg/kg) in the low dose group. One third of the residue was attributed to aclonifen while the rest was distributed among at least 3 polar metabolites. Neither aclonifen nor one of the metabolites individually exceeded 0.01 mg/kg in liver. The metabolism of aclonifen in the goat involves a combination of hydroxylation on the phenyl ring, N-acetylation of the amine and reduction of the nitro group. The dehydration of metabolites where the amine was N-acetylated and the nitro group had been reduced led to the formation of methylimidazole derivatives. No cleavage of the diphenyl ether bond was observed. The general metabolic pathways in ruminants and rats were considered comparable.

A poultry metabolism study is not required since poultry feed is unlikely to exceed 0.1 mg/kg feed.

Aclonifen appears to undergo biotransformation via hydroxylation, reduction of the nitro group and N-acetylation both in the rat and in the goat. The hydroxylated aclonifen metabolite RPA 407074 occurred in both species. The direct precursors of RPA 407074-methylimidazole and aclonifen-methylimidazole have also been detected in both species, though the methylimidazole derivatives themselves did not occur in the rat. The general metabolic pathways observed in the rat and in the goat are considered comparable. A pig metabolism study therefore is not required.

Conclusion on metabolism in livestock

According to residues in animal feed are unlikely to exceed 0.1 mg/kg. From the goat metabolism study it was further concluded that no measurable residues of aclonifen or metabolites (i.e. no residues > 0.01 mg/kg) will be transferred to animal commodities at the lowest dose level of the study. This lowest dose was still distinctly higher than the maximum calculated dietary intake for ruminants. As a consequence, livestock feeding studies were not required.

zRMS comment: The Applicant referred to the studies provided during review process of the substance. Based on the results of the livestock metabolism studies, it is concluded that metabolism in ruminants and rats is similar. The definition of the residues for monitoring and risk assessment in commodities of animal origin is proposed as parent aclonifen only.

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

Table 7.2-7: Summary on the nature of residues in commodities of animal origin

	Endpoints
Animals covered	Lactating goats
Time needed to reach a plateau concentration	4 days in milk
Animal residue definition for monitoring	Aclonifen EFSA Scientific Report (2008) 149, 1-80
Animal residue definition for risk assessment	Aclonifen EFSA Scientific Report (2008) 149, 1-80
Conversion factor	Not applicable (EFSA Journal 2015;13(11):4323).
Metabolism in rat and ruminant similar	Yes
Fat soluble residue	Yes EFSA Scientific Report (2008) 149, 1-80, (EFSA Journal 2015;13(11):4323).

* A more recent proposal by EFSA may be provided as additional information (EFSA RO XXXX)

** If no EFSA proposal is available, a proposal should be made by the applicant/zRMS.

*** If metabolism in rat and ruminant are not similar

7.2.2 Magnitude of residues in plants (KCA 6.3)

7.2.2.1 Summary of European data and new data supporting the intended uses

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

Table 7.2-8: Summary of EU reported and new data supporting the intended uses of AKO 600 SC and conformity to existing MRL

Commodity	Source	Residue zone (N-EU, S-EU, EU, outside EU)	Evaluation GAP Residue levels (mg/kg) E = according to enforcement residue definition RA = according to risk assessment residue definition	STMR (mg/kg)	HR (mg/kg)	Unrounded OECD calculator MRL (mg/kg)	Current EU MRL (mg/kg) *	MRL compliance
Sunflower	EFSA Scientific Report (2008) 149, 1-80	N-EU	GAP on which MRL/EU a.s. assessment is based: 1 x 1.4 4.2 2.1-4.8 kg as/ha, pre-emergency, PHI: 132 – 154, outdoor 3 x < 0.01 g/kg, 6 x < 0.02 mg/kg	N/A				
	Overall supporting data for cGAP	N-EU	3 x < 0.01 mg/kg, 6 x < 0.02 mg/kg	0.02	0.02	-	0.02 [†]	Yes
Potato tubers	Wańczyk K., 2024, 23SGS30, DPL/89/2023; Wańczyk K., 2024, 23SGS31, DPL/90/2023; Wańczyk K., 2024, 23SGS32, DPL/91/2023; Wańczyk K., 2024, 23SGS32, DPL/92/2023	N-EU	Trials: 1 x 1.8 kg as/ha, BBCH 00 PHI: 41- 94 d. outdoor 4x < 0.001 mg/kg	N/A				
	Overall supporting	N-EU	4 x < 0.001 mg/kg	0.001	0.001	-	0.02 [†]	Yes

	data for cGAP							
Sunflower	According to SANTE/2019/12752 rev.02 magnitude of residue studies performed in sunflower can be extrapolated on the following crops: - Spring rapeseeds, - Flax, - Breadseed poppy, - Mustard, - Soyabeans, - Pumpkin, - Hemp,							

* Source of EU MRL: Reg. (EU) 2021/1531

According to COMMISSION REGULATION (EU) No 283/2013 point “Magnitude of residue trials in plants” studies shall always be performed where the plant protection product is to be applied to plants or plant products that are used as food or feed. For uses 18-25 from GAP table plant protection product is not relevant since those crops are not designed for eating or animal feeding.

According to information from SANTE/2019/12752 **rev.02**:

- Residue data set for Sunflower (EU data level - EFSA Scientific Report (2008) 149, 1-80), can be extrapolated for uses of: spring rapeseeds, flax, breadseed poppy, mustard, soyabeans, pumpkin and hemp.
- As mentioned above extrapolation for tobacco, forest nurseries, ornamental shrubs, basket willow, purple willow, forest tree, Christmas tree, energetic willow is not required due to inedible parts of those plants.

7.2.2.2 Conclusion on the magnitude of residues in plants

According to the available data, the intended uses on all crops are considered acceptable, for outdoor uses.

The data submitted show that no exceedance of the MRL will occur.

All intended uses are considered acceptable.

RMS comment: The evaluated representative use is as a pre-emergence herbicide on sunflower. The Applicant referred to the Conclusion on the peer review of aclonifen (EFSA, 2008). Aclonifen was applied to soil prior to emergence of the sunflowers, with application rates ranging from 2.1 to 4.8 kg as/ha. For Northern Europe, 9 field trials were found to match the critical GAP. No PHI was indicated. No residues above the LOQ for aclonifen have been found in sunflower seed at harvest. In the required GAP, the dose is 1.8 kg a. s./ha which is less critical compared to already evaluated GAP. Additionally, data from Review of the existing MRLs for aclonifen (EFSA Journal 2015;13(11):4323), indicate residue levels observed in the trials conducted with more critical dose as: 9x<0.01 mg/kg and 10x<0.02 mg/kg. The proposed use of aclonifen in sunflower is acceptable. According to SANTE/2019/12752 **rev.02** guidelines, it is possible to extrapolate residue data from any representative of the oilseed group, with the exception of peanuts/~~peanuts~~ groundnuts (0401020), to the entire oilseed group (0401000), **except peanuts/groundnuts (0401020) and caraway (0820030)** when the product is used before forming of the edible part. Thus the application in rapeseed, flax, poppy, mustard, soybeans, pumpkin seeds and hemp is acceptable. The use in tobacco, forest plants, ornamentals, basket willow, purple willow, energetic willow and Christmas trees was not evaluated as such crops are not predicted for consumption. The Applicant presented new studies for residues of aclonifen in potatoes. The residues in potato were <0.001 mg/kg, therefore the reduced number of trials is accepted. The proposed use of aclonifen in potato is supported.

7.2.3 Magnitude of residues in livestock

7.2.3.1 Dietary burden calculation

Table 7.2-9: Input values for the dietary burden calculation (considering the uses authorized in the country of the zRMS)

Feed Commodity	Median dietary burden		Maximum dietary burden	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Risk assessment residue definition – Aclonifen				
Potato, culls	0.001	See point 7.2.2.1	0.001	See point 7.2.2.1
Potato, process waste				
Potato, dried pulp				
Sunflower, meal	0.02	See point 7.2.2.1	0.02	See point 7.2.2.1
Rape, meal	0.02	See point 7.2.2.1	0.02	See point 7.2.2.1
Canola (Rape seed)				
Flaxseed/Linseed, meal	0.02	See point 7.2.2.1	0.02	See point 7.2.2.1
Soyabean, seed	0.02	See point 7.2.2.1	0.02	See point 7.2.2.1
Soyabean, meal				
Soyabean, hulls				

Animal burden calculation												Aclonifen		
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			Dairy			Ram/Ewe			Lamb				
(mg/kg bw/d)	0.002	mg/kg bw/d	%	0.002	mg/kg bw/d	%	0.002	mg/kg bw/d	%	0.003	mg/kg bw/d	%		
Contributor 1	Potato	process was	40	Potato	process was	30	Potato	process was	40	Soybean	hulls	20		
Contributor 2	Soybean	seed	10	Soybean	seed	10	Soybean	seed	10	Soybean	seed	20		
Contributor 3	Potato	culls	30	Potato	culls	30	Potato	culls	30	Potato	culls	20		
Contributor 4														
Median intake	0.0017	mg/kg bw/d		0.0021	mg/kg bw/d		0.0023	mg/kg bw/d		0.0027	mg/kg bw/d			
Maximum Intake	Swine						Intakes >0.004 mg/kg bw/d are highlighted							
	Breeding			Finishing										
(mg/kg bw/d)	0.001	mg/kg bw/d	%	0.001	mg/kg bw/d	%								
Contributor 1	Potato	process was	20	Soybean	hulls	10								
Contributor 2	Soybean	seed	10	Soybean	seed	20								
Contributor 3	Potato	culls	50	Potato	culls	50								
Contributor 4														
Median intake	0.001	mg/kg bw/d		0.001	mg/kg bw/d									
Maximum Intake	Poultry													
	Broiler			Layer			Turkey							
(mg/kg bw/d)	0.001	mg/kg bw/d	%	0.001	mg/kg bw/d	%	0.001	mg/kg bw/d	%					
Contributor 1	Soybean	hulls	5	Soybean	hulls	5	Soybean	meal	45					
Contributor 2	Soybean	seed	20	Soybean	seed	15	Soybean	seed	15					
Contributor 3	Potato	culls	10	Potato	culls	10	Potato	culls	20					
Contributor 4														
Median intake	0.001	mg/kg bw		0.001	mg/kg bw		0.001	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	0.07	0.05		0.1	0.06		0.04	0.04						
Median	0.07	0.05		0.07	0.06		0.04	0.04						
	Poultry													
	Broiler	Layer		Turkey										
Maximum	0.02	0.02		0.02										
Median	0.02	0.02		0.02										

New data requirements		☐ Regulation (EU) No 283/2013)						
Relevant groups	Dietary burden expressed in				Most critical diet (a)	Most critical commodity (b)		Trigger exceeded (Yes/No)
	mg/kg bw per day		mg/kg DM					0.004
	Median	Maximum	Median	Maximum				mg/kg bw
Cattle (all diets)	0,002	0,002	0,07	0,07	Dairy cattle	Potato	process waste	No
Cattle (dairy only)	0,002	0,002	0,05	0,05	Dairy cattle	Potato	process waste	No
Sheep (all diets)	0,003	0,003	0,07	0,07	Lamb	Soybean	hulls	No
Sheep (ewe only)	0,002	0,002	0,07	0,07	Ram/Ewe	Potato	process waste	No
Swine (all diets)	0,001	0,001	0,04	0,04	Swine (finishing)	Soybean	hulls	No
Poultry (all diets)	0,001	0,001	0,02	0,02	Poultry broiler	Soybean	hulls	No
Poultry (layer only)	0,001	0,001	0,02	0,02	Poultry layer	Soybean	hulls	No

zRMS comment: The calculated dietary burdens were found to be below the trigger value of 0.004 mg/kg bw hence no livestock feeding studies are required.

7.2.3.2 Livestock feeding studies (KCA 6.4.1-6.4.3)

Available data

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

From the goat metabolism study it was concluded that no measurable residues of aclonifen or metabolites (i.e. no residues > 0.01 mg/kg) will be transferred to animal commodities at the lowest dose level of the study. This lowest dose was still distinctly higher than the calculated maximum dietary intake for ruminants. Residues in animal feed are unlikely to exceed 0.1 mg/kg. As a consequence, livestock feeding studies were not required.

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

The studies of magnitude of the residues in processed commodities, according to COMMISSION REGULATION (EU) No 283/2013, are not necessary since the level of residues in the plants and processed plant products is below 0.1 mg/kg.

Sunflower seed samples from supervised field trials were processed into oil and press cake. The processing trials only cover pressing under ambient temperature conditions and filtration. These procedures do not involve heating at high temperatures or treatment with bases or acids and, therefore, are not likely to modify significantly the nature of aclonifen residues. As anticipated from the residue level in the unprocessed sunflower seed (< 0.02 mg/kg), no measurable residues of aclonifen were found in sunflower oil or in press/extraction cake either. Since no fortified samples have been used and residues in raw and processed samples were always < LOQ, no processing factors could be derived. The studies are not required just considered as additional information only.

zRMS comment: As residues are less than 0.1 mg/kg and theoretical maximum daily intake (TMDI) is <10% of the ADI for any EU consumer group diet, therefore magnitude of residue in processed commodities is not required.

EFSA conclusion (2008):” *Eleven supervised field trials analysing residues in sunflower seed raw and processed to oil and press cake are available. However, since residues in raw and processed samples were always <LOQ, processing factors could not be derived (EFSA, 2008). The studies are considered as additional information only and additional studies are not required.*

7.2.4.1 Available data for all crops under consideration

7.2.4.2 Conclusion on processing studies

Not relevant

7.2.5 Magnitude of residues in representative succeeding crops

The crops under consideration can be grown in rotation.

Data dealing with magnitude of residues in succeeding crops are have been submitted and are summa-

rized hereafter.

7.2.5.1 Field rotational crop studies (KCA 6.6.2)

Available data

No new data submitted in the framework of this application.

Table 7.2-10: Summary of available studies in field rotational crops

Primary crop	Rate (kg a.s./ha) (GS at applica- tion or PHI)	Residue levels in succeeding crops			
		Succeeding crop group	Succeeding crop	Sowing intervals (DAT)	Reference / Remarks
EU data					
Bare soil	3.72 kg as/ha	Leafy vegetables	Spinach	29 120 365	McEwen, A. (2003, RIP2005-99) Aniline-U-14C]-aclonifen: A Confined Crop Rotation Study
	3.72 kg as/ha	Root and tuber vegetables	Carrot	29 120 365	McEwen, A. (2003, RIP2005-99) Aniline-U-14C]-aclonifen: A Confined Crop Rotation Study
	3.72 kg as/ha	Cereal	Barley	29 120 365	McEwen, A. (2003, RIP2005-99) Aniline-U-14C]-aclonifen: A Confined Crop Rotation Study

Conclusion on rotational crops studies

A confined rotational crop study with rotation intervals of one, six and twelve months was conducted with spinach, carrot and barley using [¹⁴C-aniline]-labelled aclonifen. Uptake of radioactive residues into leafy and cereal crops was only marginal, but was more pronounced into root crops. TRRs in carrot foliage, spinach, immature barley, barley grain and barley chaff remained below 0.1 mg/kg. TRR in carrot root reached a maximum of 0.491 mg as eq/kg after the first rotation interval, decreasing to 0.200 mg as-eq/kg after the second and 0.035 mg as-eq/kg after the third rotation interval. Residues in barley straw amounted to 0.245 mg as-eq/kg after the first rotation interval, decreasing to 0.040 and 0.077 mg as-eq/kg after the second and the third interval, respectively. The extractable residue in carrot root consisted exclusively of aclonifen which was also the only major component in carrot foliage and the only identifiable compound in spinach. Barley grain contained one major polar component in a low absolute amount (0.004 mg/kg). Barley straw comprised aclonifen and RPA 508285 in minor amounts. The unidentified compounds predominantly were polar in nature. It can be concluded from the study that the only significant residue to be considered in rotational crops is the parent aclonifen which is consistent with the findings from the pre-emergence metabolism studies. The transfer of aclonifen into human-edible commodities which are derived from leafy crops and cereals is anticipated to be negligible. The occurrence of aclonifen residues > 0.02 mg/kg in human-edible commodities derived from root crops, however, can't be excluded.

EFSA Review of the existing MRLs for aclonifen (2015): “The rotational crop metabolism study showed that in the majority of crops no significant residues occurred. Nevertheless, since significant residues were found in carrots grown in rotation, during the peer review a data gap was set for a rotational crop residue study in root and tuber vegetables (EFSA, 2008). Two additional studies investigating the magnitude of residues in turnips planted 30 and 60 days after application of 2.4 kg a.s./ha to bare soil

were submitted and evaluated in the framework of this review (Germany, 2011). According to the results of both studies, no residues are expected in root and tuber vegetables grown in rotation with crops treated with aclonifen (residues were below the LOQ of 0.01 mg/kg in all samples of leaves and roots analysed)."

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

The available data for the active substance sufficiently address aspects of the residue situation that might arise from the use of AKO 600 SC. Therefore, other special studies are not needed.

Based on the Technical Guidelines for Determining the Magnitude of Pesticide Residues in Honey and Setting Maximum Residue Levels in Honey (SANTE/11956/2016 rev. 9, 14 September 2018), the presence of pesticide residues in honey is expected, particularly when GAP (Good Agricultural Practices) involves the use of pesticides on melliferous crops. Residues are likely if the pesticide is applied during the flowering stage (BBCH 60-69) of a melliferous crop that is foraged by bees.

Data from both, EU level studies and our own ecotoxicological data package have shown that aclonifen poses no unacceptable risk to honey bees and other pollinators (see Part B9). Furthermore AKO 600 SC formulation, as proposed in the GAP is intended to be applied on crops during spring season within the BBCH 00-09 range, therefore the major part of the application period for this plant protection product does not overlap with the flowering period of the crop and during which time bees are not actively foraging. Additionally, since the intended use of aclonifen does not coincide with the flowering periods of non-target plants, residues originating from the nectar of non-target plant flowers are also irrelevant. Potential risk of transporting residues directly to pollen and nectar in case of application on melliferous crops can also be considered as negligible.

According to the residue data from supervised field trials presented in DAR – Aclonifen – Volume B7, the residue level in sunflowers for all studies is below the LOQ. These results were obtained for all applications taken into the study - the range of which, in terms of active substance, was: 1.4 kg a.s./ha - 4.2 kg a.s./ha. Despite that the difference between the highest doses rate used in the studies compared to the doses proposed in the GAP by the applicant is exaggerated (maximum dose proposed in the GAP by the applicant is 1.8 kg as/ha) the residue level in plants is still below the LOQ. It should be concluded that using the plant protection product AKO 600 SC, in accordance with the proposed GAP the residue level in plants will also be < LOQ. Taking into account the conclusions and results of magnitude of residues in plants it should be assumed that magnitude of residues in honey should be considered insignificant and not exceeding the EU MRL.

Similar argumentation has been used in renewal process of aminopyralid on EU level.

Given this data, the applicant has decided to not conduct new studies on residues in honey and consider magnitude of residues in honey not relevant and not exceeding maximum residue level in in honey"

zRMS comment: Sunflower, rapeseed, flaxseed, poppy seeds, mustard seeds, soya beans and pumpkin seeds are considered a melliferous crop according to the technical guidelines (SANTE/11956/2016 rev. 9). Therefore, the evaluation of residues in honey is in principle necessary. However, available residue data for sunflower (9 NEU and 11 SEU, DAR, 2006), conducted with the exaggerated pre- emergence application rates ranged from 2100 to 4800 g as/ha, indicated residues of aclonifen in sunflower seeds always below the LOQ at mature harvest. Thus, there is no risk of residues above LOQ following intended uses (pre-emergence application of 1.8 kg/ha). It is therefore considered that an application well before flowering at a dose below that already assessed in the DAR will not result in the residues of aclonifen above MRL of 0.05 mg/kg in honey when the product is used according to the recommended GAP.

7.2.6.1 Input values for the consumer risk assessment

Table 7.2-11: Input values for the consumer risk assessment

Commodity	Chronic risk assessment	
	Input value (mg/kg)	Comment
Risk assessment residue definition – Aclonifen		
Tier I		
Intended uses		
Potato	0.02*	Reg. (EU) 2021/1531
Sunflower	0.02*	Reg. (EU) 2021/1531
Spring rapeseed	0.01*	Reg. (EU) 2021/1531
Flax	0.01*	Reg. (EU) 2021/1531
Breadseed poppy	0.01*	Reg. (EU) 2021/1531
Mustard	0.01*	Reg. (EU) 2021/1531
Soyabeans	0.01*	Reg. (EU) 2021/1531
Pumpkin seeds	0.01*	Reg. (EU) 2021/1531
Hemp	0.01*	Reg. (EU) 2021/1531
Further uses		
All other commodities	-	Not relevant

7.2.6.2 Conclusion on consumer risk assessment

Extensive calculation sheets are presented in Appendix 3.

Table 7.2-12: Consumer risk assessment

Aclonifen	
ADI	0.07 mg/kg bd
TMDI (% ADI) according to EFSA PRIMo rev. 3.1	2% (based on NL toddler) for milk: cattle
IEDI (% ADI) according to EFSA PRIMo rev. 3.1	0.1% (based on GEMS/FOOD G11) for soyabeans
ARfD	Not relevant
IESTI (% ARfD) according to EFSA PRIMo rev. 3.1	Not required

* include raw and processed commodities if both values are required for PRIMo

** if national model is available

The proposed uses of aclonifen in the formulation AKO 600 SC do not represent unacceptable chronic risks for the consumer.

zRMS comment: Input values used to perform the consumer risk assessment are the EU MRLs set in Reg. (EU) 2021/1531 and are considered as the worst case. Extensive calculation sheets are presented in Appendix 3.

The calculation of the TMDI led to an utilisation of the ADI of 2% % based on NL toddler being the population group with the highest value. For this diet, the highest contributor is milk with 0.9% of the ADI.

As no ARfD was established for aclonifen no acute risk assessment was performed. The proposed uses of aclonifen in the product AKO 600 SC do not represent unacceptable risks for the consumers.

7.3 Clomazone

The assessment of residues in clomazone is not the subject of this document. However, it's required for combined exposure assessment of a mixture: AKO 600 SC and BOA PRO 480 EC. Therefore, for details of residue assessment of Clomazone please refer to core dossier of BOA PRO 480 EC.

zRMS comment: For potato, AKO 600 SC is used in tank mix with BOA PRO 480 EC containing clomazone as an active substance. For clomazone, the Applicant refers to the residue data uses in potatoes evaluated by IOR-PIB and approved in Poland (RR, 21.01.2013).

Sufficient residue trials are available to support the GAP uses in potatoes in NEU. In total, 13 residue trials were conducted involving pre-emergence or early post-emergence application at or above the required GAP rate, i.e. 96 g a.s./ha.

In all potato trials performed in NEU, the residues level in potato tubers was <0.01 mg/kg.

Dietary risk assessment for the active substance was re-calculated by zRMS using EFSA Primo 3.1. The TMDI estimation is always below 1 % of the ADI. As no acute reference has been set for clomazone, there is no need to evaluate the acute risk for this active substance.

It can be concluded that the use of clomazone does not lead to unacceptable risk for consumer when applied according to the recommendations.

 European Food Safety Authority EFSA PRIMO revision 3.1; 2019/03/19	Clomazone				Input values						
	LOQs (mg/kg) range from: to:				Details - chronic risk assessment						
	Toxicological reference values				Supplementary results - chronic risk assessment						
	ADI (mg/kg bw/day): 0.133 ARfD (mg/kg bw): not necessary				Details - acute risk assessment/children						
Source of ADI: Year of evaluation:				Source of ARfD: Year of evaluation:				Details - acute risk assessment/adults			
Comments:											
Normal mode											
Chronic risk assessment: JMPR methodology (IED/TMDI)											
No of diets exceeding the ADI: ---											
TMDI/IED (IED) calculation (based on average food consumption)	Calculated exposure (% of ADI)	MS Diet	Exposure (µg/kg bw per day)	Highest contributor to MS diet (in % of ADI)	Commodity / group of commodities	2nd contributor to MS diet (in % of ADI)	Commodity / group of commodities	3rd contributor to MS diet (in % of ADI)	Commodity / group of commodities	MRLs set at the LOQ (in % of ADI)	commodities not under assessment (in % of ADI)
	0.2%	NL toddler	0.27	0.1%	Malacorn	0.0%	Potatoes	0.0%	Wheat		
	0.2%	GEMS/Food G06	0.26	0.1%	Wheat	0.0%	Tomatoes	0.0%	Soybeans		
	0.2%	GEMS/Food G10	0.24	0.0%	Soybeans	0.0%	Wheat	0.0%	Potatoes		
	0.2%	GEMS/Food G11	0.22	0.1%	Soybeans	0.0%	Potatoes	0.0%	Wheat		
	0.2%	GEMS/Food G08	0.22	0.0%	Wheat	0.0%	Soybeans	0.0%	Potatoes		
	0.2%	GEMS/Food G15	0.22	0.0%	Wheat	0.0%	Potatoes	0.0%	Soybeans		
	0.2%	GEMS/Food G07	0.21	0.0%	Wheat	0.0%	Potatoes	0.0%	Soybeans		
	0.1%	DK child	0.18	0.0%	Rye	0.0%	Wheat	0.0%	Potatoes		
	0.1%	RO general	0.18	0.0%	Wheat	0.0%	Potatoes	0.0%	Tomatoes		
	0.1%	IE adult	0.17	0.0%	Sweet potatoes	0.0%	Wheat	0.0%	Potatoes		
	0.1%	PT general	0.15	0.0%	Potatoes	0.0%	Wheat	0.0%	Tomatoes		
	0.1%	DE child	0.15	0.0%	Wheat	0.0%	Potatoes	0.0%	Carrots		
	0.1%	SE general	0.14	0.0%	Potatoes	0.0%	Wheat	0.0%	Carrots		
	0.1%	NL child	0.14	0.0%	Wheat	0.0%	Potatoes	0.0%	Rapeseeds/canola seeds		
	0.1%	FR child 3-15 yr	0.13	0.0%	Wheat	0.0%	Potatoes	0.0%	Tomatoes		
	0.1%	IT toddler	0.13	0.0%	Wheat	0.0%	Other cereals	0.0%	Tomatoes		
	0.1%	FI 3 yr	0.12	0.0%	Potatoes	0.0%	Wheat	0.0%	Cucumbers		
	0.1%	UK infant	0.12	0.0%	Potatoes	0.0%	Potatoes	0.0%	Carrots		
	0.1%	UK toddler	0.11	0.0%	Wheat	0.0%	Potatoes	0.0%	Beans		
	0.1%	FR toddler 2-3 yr	0.11	0.0%	Wheat	0.0%	Potatoes	0.0%	Beans (with pods)		
	0.1%	ES child	0.11	0.0%	Wheat	0.0%	Potatoes	0.0%	Tomatoes		
	0.1%	FI 6 yr	0.10	0.0%	Potatoes	0.0%	Wheat	0.0%	Cucumbers		
	0.1%	IT adult	0.10	0.0%	Wheat	0.0%	Tomatoes	0.0%	Other cereals		
	0.1%	NL general	0.09	0.0%	Potatoes	0.0%	Wheat	0.0%	Rapeseeds/canola seeds		
	0.1%	LT adult	0.08	0.0%	Potatoes	0.0%	Rye	0.0%	Wheat		
	0.1%	ES adult	0.07	0.0%	Wheat	0.0%	Potatoes	0.0%	Tomatoes		
	0.1%	DE general	0.07	0.0%	Wheat	0.0%	Potatoes	0.0%	Tomatoes		
	0.1%	DE women 14-50 yr	0.07	0.0%	Wheat	0.0%	Potatoes	0.0%	Tomatoes		
	0.1%	FR infant	0.07	0.0%	Potatoes	0.0%	Carrots	0.0%	Wheat		
	0.1%	UK vegetarian	0.07	0.0%	Wheat	0.0%	Potatoes	0.0%	Tomatoes		
	0.0%	FR adult	0.07	0.0%	Wheat	0.0%	Potatoes	0.0%	Tomatoes		
	0.0%	PL general	0.06	0.0%	Potatoes	0.0%	Tomatoes	0.0%	Head cabbages		
	0.0%	UK adult	0.05	0.0%	Wheat	0.0%	Potatoes	0.0%	Tomatoes		
	0.0%	DK adult	0.05	0.0%	Potatoes	0.0%	Wheat	0.0%	Rye		
0.0%	FI adult	0.05	0.0%	Potatoes	0.0%	Rye	0.0%	Tomatoes			
0.0%	IE child	0.03	0.0%	Wheat	0.0%	Potatoes	0.0%	Rice			
Conclusion: The estimated long-term dietary intake (TMDI/IED/IEDI) was below the ADI. The long-term intake of residues of Clomazone is unlikely to present a public health concern.											

7.4 Combined exposure and risk assessment

From a scientific point of view it is regarded necessary to take into account potential combination effects. However, the evaluation of cumulative or synergistic effects as requested by Art. 4 (3b) of Regulation (EC) No. 1107/2009 should only be performed when harmonised “scientific methods accepted by the Authority to assess such effects are available.”

Currently, no EU-harmonized guidance is available on the risk assessment of combined exposure to mul-

multiple active substances; this approach is not mandatory at EU level.

The product might be used as a tank-mixture as a combination with second active substances. Since for both active substances an acute reference dose has not been allocated, combined exposure cannot be considered.

7.4.1 Acute consumer risk assessment from combined exposure

Not relevant

7.4.2 Chronic consumer risk assessment from combined exposure

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

However following the approach of calculations of the acute consumer risk assessment for mixtures, the Hazard Quotients (HQ) have been determined for each active substance. Based on Hazard Indexes (HI) calculations by performing TMDI/IEDI calculations with the model EFSA PRIMO (rev. 3.1), chronic consumer risk assessment for the mixture of AKO 600 SC and BOE PRO 480 EC has been determined.

Table 0.2-1: Chronic consumer risk assessment from combined exposure

	Active Ingredient	HQ (based on IEDI/TMDI according to EFSA PRIMo)
	Aclonifen	0.02
	Clomazone	0.01
	Cumulative risk (HI)	0.03

The Hazard Index is < 1 . Thus combined exposure to all active substances in the AKO 600 SC and BOA PRO 480 EC tank mix is not expected to present a consumer risk. No further refinement of the assessment is required.

zRMS comment: The uses under consideration provide only a minor contribution to the overall chronic exposure of consumers to pesticide residues. A harmonised approach is not yet available, and currently no specific consideration is warranted in the scope of this evaluation.

7.5 References

EFSA Scientific Report (2008) 149, 1-80, Conclusion on the peer review of aclonifen

DAR Aclonifen - August 2006

Appendix 1 Lists of data considered in support of the evaluation

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	Wańczyk, K.	2024	Magnitude of the Aclonifen residues in potato (Raw Agricultural Commodity) after one application of AKO 600 SC – one harvest study trial in Poland – 2023 SGS POLSKA Sp. z o.o. Jerozolimskie Business Park Al. Jerozolimskie 146A 02-305 Warszawa LABORATORIUM SGS POLSKA ul. Cieszyńska 52 A 43-200 Pszczyna Poland STUDY NUMBER: 23SGS30 Analytical phase code: DPL/89/2023 GLP, unpublished	N	Chemrol
KCA 6.3	Wańczyk, K.	2024	Magnitude of the Aclonifen residues in potato (Raw Agricultural Commodity) after one application of AKO 600 SC – one harvest study trial in Germany – 2023 SGS POLSKA Sp. z o.o. Jerozolimskie Business Park Al. Jerozolimskie 146A 02-305 Warszawa LABORATORIUM SGS POLSKA ul. Cieszyńska 52 A 43-200 Pszczyna Poland STUDY NUMBER: 23SGS31 ANALYTICAL PHASE CODE: DPL/90/2023 GLP, unpublished	N	Chemrol
KCA 6.3	Wańczyk, K.	2024	Magnitude of the Aclonifen residues in potato (Raw Agricultural Commodity) after one application of AKO 600 SC – one decline curve study trial in Poland – 2023 SGS POLSKA Sp. z o.o. Jerozolimskie Business Park Al. Jerozolimskie 146A 02-305 Warszawa LABORATORIUM SGS POLSKA ul. Cieszyńska 52 A 43-200 Pszczyna Poland STUDY NUMBER: 23SGS32 ANALYTICAL PHASE CODE: DPL/91/2023 GLP, unpublished	N	Chemrol
KCA 6.3	Wańczyk, K.	2024	Magnitude of the Aclonifen residues in potato (Raw Agricultural Commodity) after one application of AKO 600 SC – one decline curve study trial in Hungary – 2023 SGS POLSKA Sp. z o.o. Jerozolimskie Business Park Al. Jerozolimskie 146A 02-305 Warszawa	N	Chemrol

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
			LABORATORIUM SGS POLSKA ul. Cieszyńska 52 A 43-200 Pszczyna Poland STUDY NUMBER: 23SGS33 ANALYTICAL PHASE CODE: DPL/92/2023 GLP, unpublished		
KCA 6.3	Głowiak K.	2024	Validation of an analytical method for the determination of residues of aclonifen in potato Study code: VAL/24/2023; Głowiak K GLP: Yes Unpublished	N	Chemiról

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 6.1	Wasser, C.	2000	Storage stability of aclonifen in corn grains, corn plants and potatoes samples at less than -18 °C - FINAL REPORT. R008582 GLP, unpublished RIP2005-72	N	BAY4
KCA 6.1	Uceda, L., and Kieken, J.L.	2004	Aclonifen: Storage stability at around -20 degrees Celsius in sunflower seed, pea and tomato. C041932 GLP, unpublished RIP2005-73	N	BAY4
KCA 6.2.1	Newby, S.E.	1996	Aclonifen: Metabolism in wheat. R007086	N	BAY4

			GLP, unpublished RIP2005-76		
KCA 6.2.1	Newby, S.E.	1996	[¹⁴ C]-ACLONIFEN: Metabolism in Peas. R007087 GLP, unpublished RIP2005-75	N	BAY4
KCA 6.2.1	Schlüter, H. and Pistel, F.	1992	Metabolism of Aclonifen in Potatoes. SHELL AGRAR DOC. NO. 127AX-641-004 ! R007395 not GLP, unpublished RIP94-00251	N	BAY4
KCA 6.5.1	Muller, M.-A	1995	Aclonifen - Flurtamone - Formulation EXP30985A (SC) Trials conducted in France 1994 - Residues in sunflowers (seed). C028887 ! R&D/CRLD/AN/bd/9515133 not GLP, unpublished RIP2005-92	N	BAY4
KCA 6.5.1	Muller, M.-A.	1993	Aclonifen - Flurtamone - Formulation EXP30985A (SC) Trials conducted in France 1993 - Residues in sunflowers (seed). C028886 ! R&D/CRLD/AN/fb/9317293 not GLP, unpublished RIP2005-90	N	BAY4
KCA 6.5.1	Muller, M.-A.	1994	Aclonifen - Oxadiazon - Formulation EXP30825A (EC) Trials conducted in France 1992 - Residues in sunflowers (seed). C028888 ! R&D/CRLD/AN/bd/9415393 GLP, unpublished RIP2005-88	N	BAY4
KCA 6.5.1	Maestracci, M.	1997	Aclonifen and Oxadiargyl Formulation EXP31443A (SC) Trials Italy 1996-1997 Residues in sunflower (seed, oil and pressed cake). R003246 GLP, unpublished RIP2005-97	N	BAY4
KCA 6.5.1	Maestracci, M.	1998	Aclonifen - Oxadiargyl - Formulation EXP31443A (SC) Trials Italy 1996-1997 Residues in sunflower (seed, oil and pressed cake). R003470 GLP, unpublished RIP2005-98	N	BAY4

KCA 6.5.1	Muller, M.-A.	1994	Aclonifen - Flurtamone - Formulation EXP30985A (SC) Trials in France 1993 Residues in sunflowers (oil and oil cake). C035828 ! R&D/CRLD/AN/cb/9416504 not GLP, unpublished RIP2005-95	N	BAY4
KCA 6.6.1	McEwen, A.	2003	[Aniline-U-14C]-aclonifen: A confined Crop Rotation Study. C031669 GLP, unpublished RIP2005-99.	N	BAY4
KCA 6.2.2 KCA 6.2.5 KCA 6.7.1	[REDACTED]	1998	[¹⁴ C]-aclonifen: Absorption, distribution, metabolism and excretion following repeated oral administration to the lactating goat. [REDACTED] GLP, unpublished [REDACTED]	Y	BAY4

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 Aclonifen

A 2.1.1 Stability of residues

No new studies were submitted.

A 2.1.1.1 Stability of residues during storage of samples

No new studies were submitted.

A 2.1.1.1.1 Storage stability of residues in plant products

No new studies were submitted.

A 2.1.1.1.2 Storage stability of residues in animal products

No new studies were submitted.

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

No new studies were submitted.

A 2.1.2.1 Nature of residue in plants

No new studies were submitted.

A 2.1.2.1.1 Nature of residue in primary crops

No new studies were submitted.

A 2.1.2.1.2 Nature of residue in rotational crops

No new studies were submitted.

A 2.1.2.1.3 Nature of residues in processed commodities

No new studies were submitted.

A 2.1.2.2 Nature of residues in livestock

No new studies were submitted.

A 2.1.3 Magnitude of residues in plants

Potato Intended GAP					
Crop	Number of applications	Application rate per treatment (precise unit)	Interval between application	Growth stage at last application	PHI (days)
Potato	1	1800 g a.s/ha	N/A	BBCH: 01-09	40

Study 1.

Comments of zRMS:	Study performed according to GLP requirements, relevant guidelines and in line with the proposed GAP. The study is accepted. The objective of the study was to determine the residue level of aclonifen in potato in one harvest study following one application of the formulated product AKO 600 SC in Poland. No residues above LOD for potato were found. The intended use is accepted.
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Reference:	KCA 6.3
Report	Magnitude of the Aclonifen residues in potato (Raw Agricultural Commodity) after one application of AKO 600 SC – one harvest study trial in Poland - 2023; Study number: 23SGS30; May 2024, Wańczyk K.
Guideline(s):	Regulations (EU) 283/2013 and 284/2013 implementing Regulation (EC) 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 Commission Working Document 7029/VI/95 Rev. 5, General Recommendations for the Design, Preparation and Realization of Residue Trials, July 22, 1997 OECD Guideline for the testing of chemicals on Crop Field Trial (TG 509 published 14 June 2021) SANTE/2020/12830 Rev.2, 14 February 2023
Deviations:	No
GLP:	Yes
Acceptability:	Yes
Duplication (if vertebrate study)	No

Field Phase

One single harvest study (HS) was established in Poland. Trial consisted of one untreated plot U and one treated plot T.

Environmental conditions did not alter the normal growth, development and maturity of the crop at the trial site to such a degree as to have negative impact on the integrity and validity of this study.

One typical for herbicide (pre-emergence) application of AKO 600 SC was performed with boom sprayer on the treated plot at the target dose rate of 3,0 L/ha, equivalent to 1800 g aclonifen. Spray volume was 200-400 L/ha according to Good Agricultural Practice. The reported dose rate of test item was:

- A1: 2,940 L/ha (293,7 L/ha water), at BBCH stage 00

Remaining spray mixture after application was measured and the volume applied to the treated plot was calculated to verify delivery rate. Deviation to the target rate was -2,0 %.
RAC specimens for analyses were collected:

- S1 (potato tubbers)- BBCH 49/CH

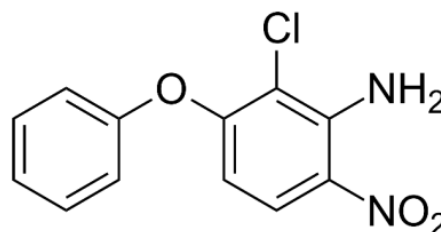
Plot number	Specimen ID		Sampling occasion*	Specimen type	Minimum sample size	Storage Condition
U	23SGS30-01	1	CH/BBCH 49	Potato tubbers	12 units, at least 2 kg	frozen
U	23SGS30-01	1R				
T	23SGS30-01	2	CH/BBCH 49	Potato tubbers	12 units, at least 2 kg	frozen
T	23SGS30-01	2R				

Materials and methods

Reference items

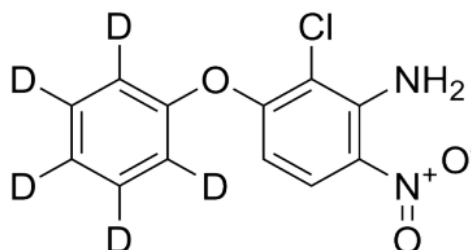
a) Aclonifen

Common Name: Aclonifen
IUPAC Name: 2-chloro-6-nitro-3-phenoxyaniline
CAS –No: 74070-46-5
Molecular formula: C₁₂H₉ClN₂O₃
Chemical structure:
Molecular weight: 264.66 g/mol
Purity: 98.03% (g/g)
Storage: 20°C ± 4°C
Lot: G1418464
Expiry date: 10.01.2028



b) Aclonifen D5

Common Name: Aclonifen D5 (phenyl D5)
IUPAC Name: 2-chloro-6-nitro-3-phenoxyaniline-d5
CAS –No: 2469279-06-7
Molecular formula: C₁₂D₅H₄ClN₂O₃
Chemical structure:
Molecular weight: 269.70 g/mol
Purity: 96.9% (g/g)
Storage: 20°C ± 4°C
Lot: 1347322
Expiry date: 22.02.2027



Analytical method

ANALYTICAL PROCEDURE DPL-67 Determination of residues of aclonifen in potato using QuEChERS method and LC-MS/MS tandem mass spectrometry, based on:

EN 15662:2018 Foods of plant origin. Multimethod for the determination of pesticide residues using GC and LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE. Modular QuEChERS-method

The field specimens arrived at the Test Site in good conditions, frozen and were stored in a freezer at $\leq -18^{\circ}\text{C}$ before analysis. After removal from the freezer the samples were homogenized at Test Site, using a knife grinder. The homogenized samples were divided into few portions: one portion was used as test sample in this study (DPL/89/2023), other portions were prepared as archival samples and the rest of the homogenized material was kept for use as a reference matrix, e.g. for method validation studies or freezer storage stability studies. The homogenized specimens were further stored at $\leq -18^{\circ}\text{C}$ until beginning of analysis

10 g of the homogenized sample was weighed into a 50 mL centrifuge tube. 10 mL of acetonitrile was added together with 100 μL of internal standard solution (1.4) and the mixture was shaken vigorously by hand for one minute. After addition of buffering salts (4 g anhydrous magnesium sulfate, 1 g sodium chloride, 1 g trisodium citrate dehydrate, 0.5 g disodium hydrogencitrate sesquihydrate), the mixture was shaken again intensively for 1 min, then centrifuged at 4700 rpm for 5 min for phase separation and finally subjected to a freezing process at $\leq -18^{\circ}\text{C}$ for 1.5 h. After that, the extract (organic phase) was filtered through a membrane filter and the final extract was directly employed for LC-MS/MS analysis. Quantification was performed using an internal standard, which was added to the extract after the initial addition of acetonitrile.

Results and discussions

The extracts were analyzed using liquid chromatography coupled with mass spectrometry, by single extraction and single injection to the detection system. Final extracts were employed for LC-MS/MS analysis directly after completion of the extraction procedure (on the same day). Data acquisition was carried out in the MRM mode. The analysis was performed using internal standard addition. For each analyte, one mass transition was evaluated and used for quantification. Representative chromatograms are shown in this report. A second (and third) mass transition was monitored for confirmation of peak identity but was not used for quantification.

The detector signals corresponding to aclonifen and internal standards peaks were integrated separately, there was used manually integration. The concentration of active substance was calculated based on the corresponding calibration curves.

The internal standard method was used for the calculation. The calculation process was as follows:

Residue concentrations of aclonifen detected in analyzed field samples (Study No.: 23SGS30, Trial No.: 23SGS30-01 – Harvest Study)

No	Timing	Sponsor's sample identification	Type of commodity	Sample number given by the laboratory	Result [mg/kg]
1	CH/BBCH 49	23SGS30-01 1	potato (tubers)	DPL/89/2023/01U	< LOD
2		23SGS30-01 2	potato (tubers)	DPL/89/2023/02T	< LOD

CH – Commercial Harvest Residues are not corrected for procedural recoveries; Calculation based on unrounded values, LOD = 0.001 mg/kg, LOQ = 0.010 mg/kg

Conclusion

This study was fully performed as anticipated, in accordance with the study plan and the amendment issued. The collected specimens were suitable for the purpose of the study and the residue values can therefore be considered as representative of the crop and of the application timing(s) and rate(s).

The results acquired during validation of the analytical method (accuracy and repeatability) were in the range of 70 – 120% and RSD ≤ 20% for average recovery.

The limit of quantification of the method was established at 0.010 mg/kg for potato.

There were no interfering signals at retention time of analyzed compound in examined control matrix. The analytical method for determining the residues of aclonifen in potato meets the criteria of SAN-TE/2020/12830, Rev.2 guidelines in terms of precision, accuracy and uncertainty.

TEST VALIDITY CRITERIA

The method for determination of aclonifen in potato (tubers) was validated at Test Site SGS Polska Sp. z o.o., ul. Cieszyńska 52A, Pszczyna, according to SANTE/2020/12830 Rev. 2, 14 February 2023. Validation criteria and results were summarized in study VAL/24/2023 entitled: “Validation of an analytical method for the determination of residues of aclonifen in potato”.

Study 2.

Comments of zRMS:	Study performed according to GLP requirements, relevant guidelines and in line with the proposed GAP. The study is accepted. The objective of the study was to determine the residue level of aclonifen in potato in one harvest study following one application of the formulated product AKO 600 SC in Germany. No residues above LOD for potato were found. The intended use is accepted.
-------------------	---

Reference:

KCA 6.3

Report

Magnitude of the Aclonifen residues in potato (Raw Agricultural Commodity) after one application of AKO 600 SC – one harvest study trial in Germany – 2023; Study code: 23SGS31; May 2024, Wańczyk K.

Guideline(s):

Regulations (EU) 283/2013 and 284/2013 implementing Regulation (EC) 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
Commission Working Document 7029/VI/95 Rev. 5, General Recommendations for the Design, Preparation and Realization of Residue Trials, July 22, 1997

OECD Guideline for the testing of chemicals on Crop Field Trial (TG 509
published 14 June 2021)
SANTE/2020/12830 Rev.2, 14 February 2023

Deviations: No
GLP: Yes
Acceptability: Yes
Duplication (if vertebrate study) No

Field phase

One single harvest study (HS) was established in Germany. Trial consisted of one untreated plot U and one treated plot T.

Environmental conditions did not alter the normal growth, development and maturity of the crop at the trial site to such a degree as to have negative impact on the integrity and validity of this study.

One typical for herbicide (pre-emergence) application of AKO 600 SC was performed with boom sprayer on the treated plot at the target dose rate of 3,0 L/ha, equivalent to 1800 g aclonifen. Spray volume was 200-400 L/ha according to Good Agricultural Practice. The reported dose rate of test item was:

- A1: 3,000 L/ha (300,00 L/ha water), at BBCH stage 02

Remaining spray mixture after application was measured and the volume applied to the treated plot was calculated to verify delivery rate. Deviation to the target rate was +0,0 %. RAC specimens for analyses were collected:

- S1 (potato tubbers)- BBCH 49/CH

Plot number	Specimen ID		Sampling occasion*	Specimen type	Minimum sample size	Storage Condition
U	23SGS31-01	1	CH/BBCH 49	Potato tubbers	12 units, at least 2 kg	frozen
U	23SGS31-01	1R				
T	23SGS31-01	2	CH/BBCH 49	Potato tubbers	12 units, at least 2 kg	frozen
T	23SGS31-01	2R				

Materials and methods

Reference items

a) Aclonifen

Common Name: Aclonifen

IUPAC Name: 2-chloro-6-nitro-3-phenoxyaniline

CAS –No: 74070-46-5

Molecular formula: C₁₂H₉ClN₂O₃

Chemical structure:

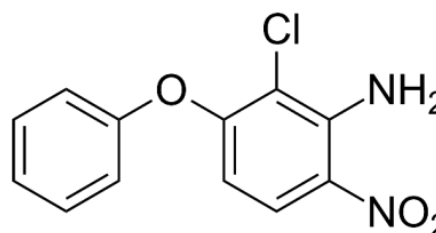
Molecular weight: 264.66 g/mol

Purity: 98.03% (g/g)

Storage: 20°C ± 4°C

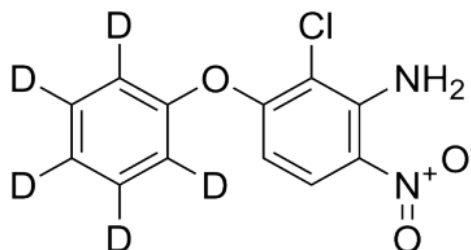
Lot: G1418464

Expiry date: 10.01.2028



b) Aclonifen D5

Common Name: Aclonifen D5 (phenyl D5)
IUPAC Name: 2-chloro-6-nitro-3-phenoxyaniline-d5
CAS –No: 2469279-06-7
Molecular formula: C₁₂D₅H₄ClN₂O₃
Chemical structure:
Molecular weight: 269.70 g/mo
Purity: 96.9% (g/g)
Storage: 20°C ± 4°C
Lot: 1347322
Expiry date: 22.02.2027



Analytical method

ANALYTICAL PROCEDURE DPL-67 Determination of residues of aclonifen in potato using QuEChERS method and LC-MS/MS tandem mass spectrometry, based on:

EN 15662:2018 Foods of plant origin. Multimethod for the determination of pesticide residues using GC and LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE. Modular QuEChERS-method

The field specimens arrived at the Test Site in good conditions, frozen and were stored in a freezer at ≤ -18°C before analysis. After removal from the freezer the samples were homogenized at Test Site, using a knife grinder. The homogenized samples were divided into few portions: one portion was used as test sample in this study (DPL/90/2023), other portions were prepared as archival samples and the rest of the homogenized material was kept for use as a reference matrix, e.g. for method validation studies or freezer storage stability studies. The homogenized specimens were further stored at ≤ -18°C until beginning of analysis

10 g of the homogenized sample was weighed into a 50 mL centrifuge tube. 10 mL of acetonitrile was added together with 100 µL of internal standard solution (1.4) and the mixture was shaken vigorously by hand for one minute. After addition of buffering salts (4 g anhydrous magnesium sulfate, 1 g sodium chloride, 1 g trisodium citrate dehydrate, 0.5 g disodium hydrogencitrate sesquihydrate), the mixture was shaken again intensively for 1 min, then centrifuged at 4700 rpm for 5 min for phase separation and finally subjected to a freezing process at ≤ -18 °C for 1.5 h. After that, the extract (organic phase) was filtered through a membrane filter and the final extract was directly employed for LC-MS/MS analysis. Quantification was performed using an internal standard, which was added to the extract after the initial addition of acetonitrile.

Results and discussions

The extracts were analyzed using liquid chromatography coupled with mass spectrometry, by single extraction and single injection to the detection system. Final extracts were employed for LC-MS/MS analysis directly after completion of the extraction procedure (on the same day). Data acquisition was carried out in the MRM mode. The analysis was performed using internal standard addition. For each analyte, one mass transition was evaluated and used for quantification. Representative chromatograms are shown in this report. A second (and third) mass transition was monitored for confirmation of peak identity but was not used for quantification.

Compound name	POLARIZATION (+)	
	m/z (quantifier)	m/z (qualifier)
Aclonifen	265.00 → 182.00	265.00 → 218.00 265.00 → 194.00
Aclonifen D5	270.00 → 187.00	270.00 → 223.00 270.00 → 253.00

The detector signals corresponding to aclonifen and internal standards peaks were integrated separately, there was used manually integration. The concentration of active substance was calculated based on the corresponding calibration curves.

The following residues concentration was determined in the field samples

Residue concentrations of aclonifen detected in analyzed field samples (Study No.: 23SGS31, Trial No.: 23SGS31-01 – Harvest Study)

No	Timing	Sponsor's sample identification	Type of commodity	Sample number given by the laboratory	Result [mg/kg]
1	CH/BBCH 49	23SGS31-01 1	potato (tubers)	DPL/90/2023/01U	< LOD
2		23SGS31-01 2	potato (tubers)	DPL/90/2023/02T	< LOD

CH – Commercial Harvest Residues are not corrected for procedural recoveries; Calculation based on unrounded values, LOD = 0.001 mg/kg, LOQ = 0.010 mg/kg

Conclusion

This study was fully performed as anticipated, in accordance with the study plan and the amendment issued. The collected specimens were suitable for the purpose of the study and the residue values can therefore be considered as representative of the crop and of the application timing(s) and rate(s).

The results acquired during validation of the analytical method (accuracy and repeatability) were in the range of 70 – 120% and RSD ≤ 20% for average recovery.

The limit of quantification of the method was established at 0.010 mg/kg for potato.

There were no interfering signals at retention time of analyzed compound in examined control matrix. The analytical method for determining the residues of aclonifen in potato meets the criteria of SAN-TE/2020/12830, Rev.2 guidelines in terms of precision, accuracy and uncertainty

TEST VALIDITY CRITERIA

The method for determination of aclonifen in potato (tubers) was validated at Test Site SGS Polska Sp. z o.o., ul. Cieszyńska 52A, Pszczyna, according to SANTE/2020/12830 Rev. 2, 14 February 2023. Validation criteria and results were summarized in study VAL/24/2023 entitled: “Validation of an analytical method for the determination of residues of aclonifen in potato”.

Study 3.

Comments of zRMS:	Study performed according to GLP requirements, relevant guidelines and in line with the proposed GAP. The study is accepted.
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	The objective of the study was to determine the residue level of aclonifen in potato in one decline study following one application of the formulated product AKO 600 SC in Poland. No residues above LOD for potato were found. The intended use is accepted.
--	---

Reference: KCA 6.3

Report Magnitude of the Aclonifen residues in potato (Raw Agricultural Commodity) after one application of AKO 600 SC – one decline curve study trial in Poland – 2023; Study code: 23SGS32; May 2024, Wańczyk K.

Guideline(s): Regulations (EU) 283/2013 and 284/2013 implementing Regulation (EC) 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
Commission Working Document 7029/VI/95 Rev. 5, General Recommendations for the Design, Preparation and Realization of Residue Trials, July 22, 1997
OECD Guideline for the testing of chemicals on Crop Field Trial (TG 509 published 14 June 2021)
SANTE/2020/12830 Rev.2, 14 February 2023

Deviations: No

GLP: Yes

Acceptability: Yes

Duplication No
(if vertebrate study)

Field phase

One single decline curve study (DCS) was established in Poland. Trial consisted of one untreated plot U and one treated plot T.

Environmental conditions did not alter the normal growth, development and maturity of the crop at the trial site to such a degree as to have negative impact on the integrity and validity of this study.

One typical for herbicide (pre-emergence) application of AKO 600 SC was performed with boom sprayer on the treated plot at the target dose rate of 3,0 L/ha, equivalent to 1800 g aclonifen. Spray volume was 200-400 L/ha according to Good Agricultural Practice. The reported dose rate of test item was:

- A1: 2,970 L/ha (296,7 L/ha water), at BBCH stage 00 Remaining spray mixture after application was measured and the volume applied to the treated plot was calculated to verify delivery rate. Deviation to the target rate was -1,0 %. RAC specimens for analyses were collected:

- S1 (potato tubbers)- BBCH 41
- S2 (potato tubbers)- BBCH 43
- S3 (potato tubbers)- BBCH 45
- S4 (potato tubbers)- BBCH 47
- S5 (potato tubbers)- BBCH 49/CH

Plot number	Specimen ID		Sampling occasion	Specimen type	Minimum sample size	Storage Condition
U	23SGS32-01	1	BBCH 41	Potato Tubers	12 units, at least 2 kg	Frozen
U	23SGS32-01	1R				
T	23SGS32-01	2		Potato Tubers	12 units, at least 2 kg	Frozen
T	23SGS32-01	2R				
T	23SGS32-01	3	BBCH 43	Potato Tubers	12 units, at least 2 kg	Frozen
T	23SGS32-01	3R				
U	23SGS32-01	4	BBCH 45	Potato Tubers	12 units, at least 2 kg	Frozen
U	23SGS32-01	4R				
T	23SGS32-01	5		Potato Tubers	12 units, at least 2 kg	Frozen
T	23SGS32-01	5R				
T	23SGS32-01	6	BBCH 47	Potato Tubers	12 units, at least 2 kg	frozen
T	23SGS32-01	6R				
U	23SGS32-01	7	CH/BBCH 49	Potato Tubers	12 units, at least 2 kg	Frozen
U	23SGS32-01	7R				
T	23SGS32-01	8	CH/BBCH 49	Potato Tubers	12 units, at least 2 kg	Frozen
T	23SGS32-01	8R				

Materials and methods

Reference items

a) Aclonifen

Common Name: Aclonifen

IUPAC Name: 2-chloro-6-nitro-3-phenoxyaniline

CAS –No: 74070-46-5

Molecular formula: C₁₂H₉ClN₂O₃

Chemical structure:

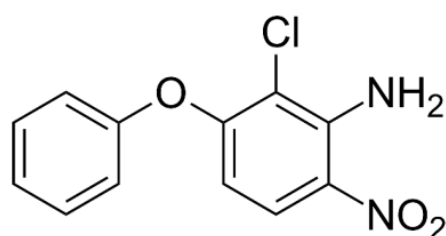
Molecular weight: 264.66 g/mol

Purity: 98.03% (g/g)

Storage: 20°C ± 4°C

Lot: G1418464

Expiry date: 10.01.2028



b) Aclonifen D5

Common Name: Aclonifen D5 (phenyl D5)

IUPAC Name: 2-chloro-6-nitro-3-phenoxyaniline-d5

CAS –No: 2469279-06-7

Molecular formula: C₁₂D₅H₄ClN₂O₃

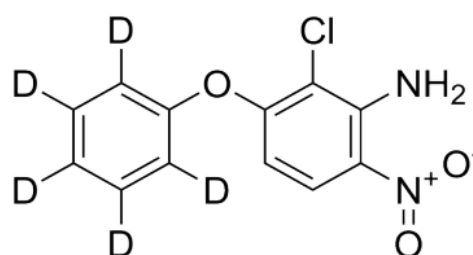
Chemical structure:

Molecular weight: 269.70 g/mol

Purity: 96.9% (g/g)

Storage: 20°C ± 4°C

Lot: 1347322



Expiry date: 22.02.2027

Analytical method

ANALYTICAL PROCEDURE DPL-67 Determination of residues of aclonifen in potato using QuEChERS method and LC-MS/MS tandem mass spectrometry, based on:

EN 15662:2018 Foods of plant origin. Multimethod for the determination of pesticide residues using GC and LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE. Modular QuEChERS-method

The field specimens arrived at the Test Site in good conditions, frozen and were stored in a freezer at $\leq -18^{\circ}\text{C}$ before analysis. After removal from the freezer the samples were homogenized at Test Site, using a knife grinder. The homogenized samples were divided into few portions: one portion was used as test sample in this study (DPL/91/2023), other portions were prepared as archival samples and the rest of the homogenized material was kept for use as a reference matrix, e.g. for method validation studies or freezer storage stability studies. The homogenized specimens were further stored at $\leq -18^{\circ}\text{C}$ until beginning of analysis.

10 g of the homogenized sample was weighed into a 50 mL centrifuge tube. 10 mL of acetonitrile was added together with 100 μL of internal standard solution (1.4) and the mixture was shaken vigorously by hand for one minute. After addition of buffering salts (4 g anhydrous magnesium sulfate, 1 g sodium chloride, 1 g trisodium citrate dehydrate, 0.5 g disodium hydrogencitrate sesquihydrate), the mixture was shaken again intensively for 1 min, then centrifuged at 4700 rpm for 5 min for phase separation and finally subjected to a freezing process at $\leq -18^{\circ}\text{C}$ for 1.5 h. After that, the extract (organic phase) was filtered through a membrane filter and the final extract was directly employed for LC-MS/MS analysis. Quantification was performed using an internal standard, which was added to the extract after the initial addition of acetonitrile.

Results and discussions

The extracts were analyzed using liquid chromatography coupled with mass spectrometry, by single extraction and single injection to the detection system. Final extracts were employed for LC-MS/MS analysis directly after completion of the extraction procedure (on the same day). Data acquisition was carried out in the MRM mode. The analysis was performed using internal standard addition.

For each analyte, one mass transition was evaluated and used for quantification. Representative chromatograms are shown in this report. A second (and third) mass transition was monitored for confirmation of peak identity but was not used for quantification.

Compound name	POLARIZATION (+)	
	m/z (quantifier)	m/z (qualifier)
Aclonifen	265.00 $\rightarrow$ 182.00	265.00 $\rightarrow$ 218.00 265.00 $\rightarrow$ 194.00
Aclonifen D5	270.00 $\rightarrow$ 187.00	270.00 $\rightarrow$ 223.00 270.00 $\rightarrow$ 253.00

The detector signals corresponding to aclonifen and internal standards peaks were integrated separately, there was used manually integration. The concentration of active substance was calculated based on the corresponding calibration curves.

The following residues concentration was determined in the field samples analyzed.

No	Timing	Sponsor's sample identification	Type of commodity	Sample number given by the laboratory	Result [mg/kg]
1	BBCH 41	23SGS32-01 1	potato tubers	DPL/91/2023/01U	< LOD
2		23SGS32-01 2	potato tubers	DPL/91/2023/02T	< LOD
3	BBCH 43	23SGS32-01 3	potato tubers	DPL/91/2023/03T	< LOD
4	BBCH 45	23SGS32-01 4	potato tubers	DPL/91/2023/04U	< LOD
5		23SGS32-01 5	potato tubers	DPL/91/2023/05T	< LOD
6	BBCH 47	23SGS32-01 6	potato tubers	DPL/91/2023/06T	< LOD
7	CH/BBCH 49	23SGS32-01 7	potato tubers	DPL/91/2023/07U	< LOD
8		23SGS32-01 8	potato tubers	DPL/91/2023/08T	< LOD

CH – Commercial Harvest Residues are not corrected for procedural recoveries; Calculation based on unrounded values, LOD = 0.001 mg/kg, LOQ = 0.010 mg/kg

Conclusion

This study was fully performed as anticipated, in accordance with the study plan and the amendment issued. The collected specimens were suitable for the purpose of the study and the residue values can therefore be considered as representative of the crop and of the application timing(s) and rate(s).

The results acquired during validation of the analytical method (accuracy and repeatability) were in the range of 70 – 120% and $RSD \leq 20\%$ for average recovery.

The limit of quantification of the method was established at 0.010 mg/kg for potato.

There were no interfering signals at retention time of analyzed compound in examined control matrix.

The analytical method for determining the residues of aclonifen in potato meets the criteria of SAN-TE/2020/12830, Rev.2 guidelines in terms of precision, accuracy and uncertainty

TEST VALIDITY CRITERIA

The method for determination of aclonifen in potato (tubers) was validated at Test Site SGS Polska Sp. z o.o., ul. Cieszyńska 52A, Pszczyna, according to SANTE/2020/12830 Rev. 2, 14 February 2023. Validation criteria and results were summarized in study VAL/24/2023 entitled: “Validation of an analytical method for the determination of residues of aclonifen in potato”.

Study 4.

Comments of zRMS:	Study performed according to GLP requirements, relevant guidelines and in line with the proposed GAP. The study is accepted. The objective of the study was to determine the residue level of aclonifen in potato in one decline study following one application of the formulated product AKO 600 SC in Hungary. No residues above LOD for potato were found. The intended use is accepted.
-------------------	---

Reference: KCA 6.3

Report Magnitude of the Aclonifen residues in potato (Raw Agricultural Commodity) after one application of AKO 600 SC – one decline curve study trial in Hungary – 2023; Study code: 23SGS33; May 2024, Wańczyk K.

Guideline(s): Regulations (EU) 283/2013 and 284/2013 implementing Regulation (EC) 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
Commission Working Document 7029/VI/95 Rev. 5, General Recommendations for the Design, Preparation and Realization of Residue Trials, July 22, 1997
OECD Guideline for the testing of chemicals on Crop Field Trial (TG 509 published 14 June 2021)
SANTE/2020/12830 Rev.2, 14 February 2023

Deviations: No

GLP: Yes

Acceptability: Yes

Duplication (if vertebrate study) No

Field phase

One single decline curve study (DCS) was established in Hungary. Trial consisted of one untreated plot U and one treated plot T.

Environmental conditions did not alter the normal growth, development and maturity of the crop at the trial site to such a degree as to have negative impact on the integrity and validity of this study.

One typical for herbicide (pre-emergence) application of AKO 600 SC was performed with boom sprayer on the treated plot at the target dose rate of 3,0 L/ha, equivalent to 1800 g aclonifen. Spray volume was 200-400 L/ha according to Good Agricultural Practice. The reported dose rate of test item was:

- A1: 2,921 L/ha (295,0 L/ha water), at BBCH stage 08

Remaining spray mixture after application was measured and the volume applied to the treated plot was calculated to verify delivery rate. Deviation to the target rate was -2,6 %. RAC specimens for analyses were collected:

- S1 (potato tubbers)- BBCH 41
- S2 (potato tubbers)- BBCH 43
- S3 (potato tubbers)- BBCH 45
- S4 (potato tubbers)- BBCH 47

- S5 (potato tubbers)- BBCH 49/CH

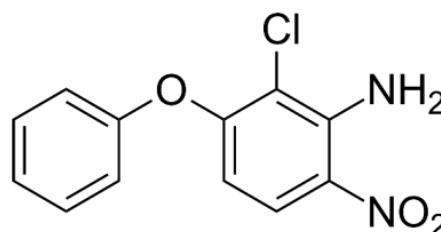
Plot number	Specimen ID		Sampling occasion	Specimen type	Minimum sample size	Storage Condition
U	23SGS33-01	1	BBCH 41	Potato Tubers	12 units, at least 2 kg	Frozen
U	23SGS33-01	1R				
T	23SGS33-01	2		Potato Tubers	12 units, at least 2 kg	Frozen
T	23SGS33-01	2R				
T	23SGS33-01	3	BBCH 43	Potato Tubers	12 units, at least 2 kg	Frozen
T	23SGS33-01	3R				
U	23SGS33-01	4	BBCH 45	Potato Tubers	12 units, at least 2 kg	Frozen
U	23SGS33-01	4R				
T	23SGS33-01	5		Potato Tubers	12 units, at least 2 kg	Frozen
T	23SGS33-01	5R				
T	23SGS33-01	6	BBCH 47	Potato Tubers	12 units, at least 2 kg	frozen
T	23SGS33-01	6R				
U	23SGS33-01	7	CH/BBCH 49	Potato Tubers	12 units, at least 2 kg	Frozen
U	23SGS33-01	7R				
T	23SGS33-01	8	CH/BBCH 49	Potato Tubers	12 units, at least 2 kg	Frozen
T	23SGS33-01	8R				

Materials and methods

Reference items

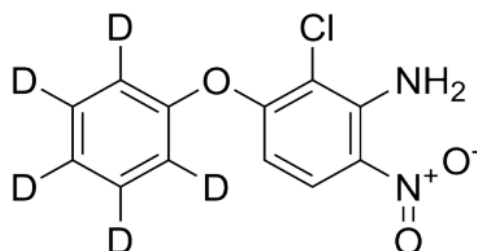
a) Aclonifen

Common Name: Aclonifen
IUPAC Name: 2-chloro-6-nitro-3-phenoxyaniline
CAS –No: 74070-46-5
Molecular formula: C₁₂H₉ClN₂O₃
Chemical structure:
Molecular weight: 264.66 g/mol
Purity: 98.03% (g/g)
Storage: 20°C ± 4°C
Lot: G1418464
Expiry date: 10.01.2028



b) Aclonifen D5

Common Name: Aclonifen D5 (phenyl D5)
IUPAC Name: 2-chloro-6-nitro-3-phenoxyaniline-d5
CAS –No: 2469279-06-7
Molecular formula: C₁₂D₅H₄ClN₂O₃
Chemical structure:



Molecular weight: 269.70 g/mo

Purity: 96.9% (g/g)

Storage: 20°C ± 4°C

Lot: 1347322

Expiry date: 22.02.2027

Analytical method

ANALYTICAL PROCEDURE DPL-67 Determination of residues of aclonifen in potato using QuEChERS method and LC-MS/MS tandem mass spectrometry, based on:
EN 15662:2018 Foods of plant origin. Multimethod for the determination of pesticide residues using GC and LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE. Modular QuEChERS-method

The field specimens arrived at the Test Site in good conditions, frozen and were stored in a freezer at $\leq -18^{\circ}\text{C}$ before analysis. After removal from the freezer the samples were homogenized at Test Site, using a knife grinder. The homogenized samples were divided into few portions: one portion was used as test sample in this study (DPL/92/2023), other portions were prepared as archival samples and the rest of the homogenized material was kept for use as a reference matrix, e.g. for method validation studies or freezer storage stability studies. The homogenized specimens were further stored at $\leq -18^{\circ}\text{C}$ until beginning of analysis.

10 g of the homogenized sample was weighed into a 50 mL centrifuge tube. 10 mL of acetonitrile was added together with 100 μL of internal standard solution (1.4) and the mixture was shaken vigorously by hand for one minute. After addition of buffering salts (4 g anhydrous magnesium sulfate, 1 g sodium chloride, 1 g trisodium citrate dehydrate, 0.5 g disodium hydrogencitrate sesquihydrate), the mixture was shaken again intensively for 1 min, then centrifuged at 4700 rpm for 5 min for phase separation and finally subjected to a freezing process at $\leq -18^{\circ}\text{C}$ for 1.5 h. After that, the extract (organic phase) was filtered through a membrane filter and the final extract was directly employed for LC-MS/MS analysis. Quantification was performed using an internal standard, which was added to the extract after the initial addition of acetonitrile

Results and discussions

The extracts were analyzed using liquid chromatography coupled with mass spectrometry, by single extraction and single injection to the detection system. Final extracts were employed for LC-MS/MS analysis directly after completion of the extraction procedure (on the same day). Data acquisition was carried out in the MRM mode. The analysis was performed using internal standard addition.

For each analyte, one mass transition was evaluated and used for quantification. Representative chromatograms are shown in this report. A second (and third) mass transition was monitored for confirmation of peak identity but was not used for quantification.

Compound name	POLARIZATION (+)	
	m/z (quantifier)	m/z (qualifier)
Aclonifen	265.00 $\rightarrow$ 182.00	265.00 $\rightarrow$ 218.00 265.00 $\rightarrow$ 194.00
Aclonifen D5	270.00 $\rightarrow$ 187.00	270.00 $\rightarrow$ 223.00 270.00 $\rightarrow$ 253.00

The detector signals corresponding to aclonifen and internal standards peaks were integrated separately, there was used manually integration. The concentration of active substance was calculated based on the corresponding calibration curves

The following residues concentration was determined in the field samples analyzed

No	Timing	Sponsor's sample identification	Type of commodity	Sample number given by the laboratory	Result [mg/kg]
1	BBCH 41	23SGS33-01 1	potato tubers	DPL/92/2023/01U	< LOD
2		23SGS33-01 2	potato tubers	DPL/92/2023/02T	< LOD
3	BBCH 43	23SGS33-01 3	potato tubers	DPL/92/2023/03T	< LOD
4	BBCH 45	23SGS33-01 4	potato tubers	DPL/92/2023/04U	< LOD
5		23SGS33-01 5	potato tubers	DPL/92/2023/05T	< LOD
6	BBCH 47	23SGS33-01 6	potato tubers	DPL/92/2023/06T	< LOD
7	CH/BBCH 49	23SGS33-01 7	potato tubers	DPL/92/2023/07U	< LOD
8		23SGS33-01 8	potato tubers	DPL/92/2023/08T	< LOD

CH – Commercial Harvest Residues are not corrected for procedural recoveries; Calculation based on unrounded values, LOD = 0.001 mg/kg, LOQ = 0.010 mg/kg

Conclusion

This study was fully performed as anticipated, in accordance with the study plan and the amendment issued. The collected specimens were suitable for the purpose of the study and the residue values can therefore be considered as representative of the crop and of the application timing(s) and rate(s).

The results acquired during validation of the analytical method (accuracy and repeatability) were in the range of 70 – 120% and $RSD \leq 20\%$ for average recovery.

The limit of quantification of the method was established at 0.010 mg/kg for potato.

There were no interfering signals at retention time of analyzed compound in examined control matrix.

The analytical method for determining the residues of aclonifen in potato meets the criteria of SAN-TE/2020/12830, Rev.2 guidelines in terms of precision, accuracy and uncertainty.

TEST VALIDITY CRITERIA

The method for determination of aclonifen in potato (tubers) was validated at Test Site SGS Polska Sp. z o.o., ul. Cieszyńska 52A, Pszczyna, according to SANTE/2020/12830 Rev. 2, 14 February 2023. Validation criteria and results were summarized in study VAL/24/2023 entitled: “Validation of an analytical method for the determination of residues of aclonifen in potato”.

Summary of studies nr 1, 2, 3 and 4

	Report No.	Study No.	Commodity / Variety	Date of: 1. Sowing 2. Flowering 3. Harvest	Method of treatment	Actual application rate			Date of treatment	BBCH at treatment	Commodity analysed	Residues (1)	DALA or CH (2)	Remarks
						L f.p./ha	Water (L/ha)	g a.i. (/ha)				Aclonifen		
Study 1	23SGS30	Poland Kolonia Bodzanowska (Kujawsko-Pomorskie) Harvest Study 23SGS30-01	Potato Red Sonia	1. Sowing – 30.04.2023 2. Flowering – 20.06.2023 – 10.07.2023 3. Harvest – 29.09.2023	Soil application	2.940	293.7	1764	12.05.2023	00	Potato tubers Potato tubers	<LOD (U) <LOD (T)	140 (CH) 140 (CH)	Analytical Method: LC-MS/MS
Study 2	23SGS31	Germany Bahlingen (Oberrhein) Harvest Study 23SGS31-01	Potato Dita	1. Sowing – 19.04.2023 2. Flowering – 12.06.2023 – 26.06.2023 3. Harvest – 21.08.2023 – 09.09.2023	Soil application	3.000	300.00	1800	03.05.2023	02	Potato tubers Potato tubers	<LOD (U) <LOD (T)	112 (CH) 112 (CH)	Analytical Method: LC-MS/MS
Study 3	23SGS32	Poland Cerekwica (Kujawsko-Pomorskie) Decline Curve Study 23SGS32-01	Potato Red Sonia	1. Sowing – 16.04.2023 2. Flowering – 15.06.2023 – 08.07.2023 3. Harvest – 28.09.2023	Soil application	2.970	296.7	1782	16.05.2023	00	Potato tubers Potato tubers Potato tubers Potato tubers Potato tubers	<LOD (U,T) <LOD (T) <LOD (U,T) <LOD (T) <LOD (U,T)	94 106 115 126 135	Analytical Method: LC-MS/MS
Study 4	23SGS33	Hungary (Szabolcs-Szatmár-Bereg County) Decline Curve Study 23SGS33-01	Potato Delila	1. Sowing – 04.05.2023 2. Flowering – 20.06.2023 – 10.07.2023 3. Harvest – 28.08.2023 – 29.08.2023	Soil application	2.921	295.0	1797	20.05.2023	08	Potato tubers Potato tubers Potato tubers Potato tubers Potato tubers	<LOD (U,T) <LOD (T) <LOD (U,T) <LOD (T) <LOD (U,T)	41 48 62 74 100	Analytical Method: LC-MS/MS

(2) DALA – Days After Last Application; CH – Commercial Harvest; (1) Residues are not corrected for procedural recoveries; Calculation based on unrounded values,

A 2.1.4 Magnitude of residues in livestock

No new studies were submitted.

A 2.1.4.1 Livestock feeding studies

No new studies were submitted.

A 2.1.5 Magnitude of residues in processed commodities (Industrial Processing and/or Household Preparation)

No new studies were submitted.

A 2.1.6 Magnitude of residues in representative succeeding crops

No new studies were submitted.

A 2.1.7 Other/Special Studies

No new studies were submitted.

Appendix 3 Pesticide Residue Intake Model (PRIMo)

A 3.1 TMDI calculations

 European Food Safety Authority EFSA PRIMo revision 3.1; 2019/03/19		Aclonifen (F)				Input values					
		LOOs (mg/kg) range from: _____ to: _____				Details - chronic risk assessment					
		Toxicological reference values				Supplementary results - chronic risk assessment					
		ADI (mg/kg bw/day): 0.07		ARD (mg/kg bw): calculation with ADI (no ARD was inserted)		Details - acute risk assessment/children		Details - acute risk assessment/adults			
Source of ADI: EFSA		Source of ARD: -									
Year of evaluation: 2008		Year of evaluation: -									
Comments:											
Normal mode											
Chronic risk assessment: JMPR methodology (IEDI/TMDI)											
No of diets exceeding the ADI : _____											
TMDI/IEDI calculation (based on average food consumption)	Calculated exposure (% of ADI)	MS Diet	Exposure (µg/kg bw per day)	Highest contributor to MS diet (in % of ADI)	Commodity / group of commodities	2nd contributor to MS diet (in % of ADI)	Commodity / group of commodities	3rd contributor to MS diet (in % of ADI)	Commodity / group of commodities	Exposure resulting from MRLs set at the LOQ (in % of ADI)	commodities not under assessment (in % of ADI)
	2%	NL toddler	1.44	0.9%	Milk: Cattle	0.2%	Apples	0.1%	Potatoes		0.1%
	1%	DE child	0.85	0.3%	Milk: Cattle	0.2%	Apples	0.1%	Carrots		0.1%
	1%	UK infant	0.78	0.6%	Milk: Cattle	0.2%	Carrots	0.1%	Potatoes		0.1%
	1%	NL child	0.76	0.3%	Milk: Cattle	0.1%	Sugar beet roots	0.1%	Potatoes		0.1%
	1%	FR toddler 2-3 yr	0.73	0.4%	Milk: Cattle	0.1%	Beans (with pods)	0.1%	Carrots		0.1%
	1.0%	FR child 3-15 yr	0.69	0.3%	Milk: Cattle	0.1%	Wheat	0.1%	Carrots		0.1%
	0.9%	GEMS/Food G11	0.62	0.1%	Celeriacs/turnip rooted celeriacs	0.1%	Potatoes	0.1%	Milk: Cattle		0.2%
	0.8%	UK toddler	0.58	0.3%	Milk: Cattle	0.1%	Potatoes	0.1%	Beans		0.1%
	0.8%	GEMS/Food G15	0.55	0.1%	Potatoes	0.1%	Milk: Cattle	0.1%	Wheat		0.1%
	0.8%	DK child	0.55	0.2%	Milk: Cattle	0.2%	Carrots	0.1%	Rye		0.1%
	0.8%	GEMS/Food G07	0.54	0.1%	Potatoes	0.1%	Milk: Cattle	0.1%	Wheat		0.2%
	0.8%	IE adult	0.53	0.1%	Potatoes	0.1%	Milk: Cattle	0.1%	Celeriacs/turnip rooted celeriacs		0.1%
	0.7%	SE general	0.52	0.2%	Milk: Cattle	0.1%	Potatoes	0.1%	Carrots		0.1%
	0.7%	GEMS/Food G08	0.52	0.1%	Potatoes	0.1%	Milk: Cattle	0.1%	Wheat		0.2%
	0.7%	GEMS/Food G08	0.49	0.1%	Wheat	0.1%	Potatoes	0.1%	Tomatoes		0.1%
	0.7%	GEMS/Food G10	0.49	0.1%	Potatoes	0.1%	Milk: Cattle	0.1%	Wheat		0.1%
	0.7%	RO general	0.48	0.2%	Milk: Cattle	0.1%	Potatoes	0.1%	Wheat		0.1%
	0.7%	ES child	0.48	0.2%	Milk: Cattle	0.1%	Wheat	0.1%	Potatoes		0.1%
	0.6%	FR infant	0.44	0.2%	Milk: Cattle	0.1%	Carrots	0.1%	Beans (with pods)		0.1%
	0.6%	DE women 14-50 yr	0.44	0.2%	Milk: Cattle	0.1%	Sugar beet roots	0.0%	Apples		0.0%
	0.6%	DE general	0.43	0.2%	Milk: Cattle	0.1%	Sugar beet roots	0.0%	Potatoes		0.0%
	0.6%	FI adult	0.39	0.4%	Coffee beans	0.0%	Carrots	0.0%	Potatoes		0.0%
0.5%	NL general	0.37	0.1%	Milk: Cattle	0.1%	Potatoes	0.1%	Sugar beet roots		0.1%	
0.5%	PT general	0.34	0.2%	Potatoes	0.1%	Carrots	0.1%	Wheat		0.2%	
0.4%	FI 3 yr	0.30	0.1%	Potatoes	0.1%	Carrots	0.0%	Bananas		0.1%	
0.4%	FR adult	0.29	0.1%	Milk: Cattle	0.0%	Wine grapes	0.0%	Wheat		0.0%	
0.4%	ES adult	0.26	0.1%	Milk: Cattle	0.0%	Wheat	0.0%	Beans (with pods)		0.0%	
0.3%	FI 6 yr	0.24	0.1%	Potatoes	0.1%	Carrots	0.0%	Cocoa beans		0.1%	
0.3%	DK adult	0.22	0.1%	Milk: Cattle	0.1%	Carrots	0.0%	Potatoes		0.0%	
0.3%	IT toddler	0.22	0.1%	Wheat	0.0%	Potatoes	0.0%	Other cereals		0.0%	
0.3%	UK vegetarian	0.21	0.0%	Milk: Cattle	0.0%	Beans	0.0%	Potatoes		0.0%	
0.3%	LT adult	0.21	0.1%	Potatoes	0.1%	Milk: Cattle	0.0%	Apples		0.1%	
0.3%	UK adult	0.19	0.0%	Milk: Cattle	0.0%	Potatoes	0.0%	Beans		0.0%	
0.3%	PL general	0.18	0.1%	Potatoes	0.0%	Carrots	0.0%	Apples		0.1%	
0.3%	IT adult	0.18	0.1%	Wheat	0.0%	Beans (with pods)	0.0%	Potatoes		0.0%	
0.1%	IE child	0.10	0.1%	Milk: Cattle	0.0%	Carrots	0.0%	Potatoes		0.0%	
Conclusion: The estimated long-term dietary intake (TMDI/IEDI) was below the ADI. The long-term intake of residues of Aclonifen (F) is unlikely to present a public health concern.											

A 3.2 IEDI calculations



Input values	
Details - chronic risk assessment	Supplementary results - chronic risk assessment
Details - acute risk assessment/children	Details - acute risk assessment/adults

Comments:

Chronic risk assessment: JMPR methodology (IEDI/TMDI)

Conclusion:
The estimated long-term dietary intake (TMDI/NEDI) was below the ADL.
The long-term intake of residues of Aclonifen (F) is unlikely to present a public health concern.

A 3.3 IESTI calculations - Raw commodities

Not relevant – no ARfD was determined.

A 3.4 IESTI calculations - Processed commodities

Not relevant – no ARfD was determined.