Evaluation Report

Prepared under Article 8 of Regulation (EC) No 396/2005

Supplemental residue data on indoxacarb requested in Commission Regulation (EU) No 668/2013 following review of indoxacarb according to Article 12 of Regulation (EU) 396/2005

Evaluating Member State: France

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TABLE OF CONTENTS

BACKGROUND

Indoxacarb was included into Annex I of Directive 91/414/EEC on 01 April 2006 and is listed as entry No. 119 in Regulation (EU) no. 540/2011. Following the active substance approval, EFSA initiated a review of existing indoxacarb MRLs according to Article 12 of Regulation (EU) 396/2005 with the Netherlands acting as Rapporteur Member State (RMS). The review considered previous reasoned opinions of EFSA on indoxacarb, the MRLs established by the Codex Alimnetarius Commission, additional information from the former RMS (the Netherlands) and comments from the Member States. The final review was published in the EFSA Journal 2011;9(8):2343, and the indoxacarb MRLs resulting from this review were published in Commission Regulation (EU) No. 668/2013.

The EFSA review identified certain tentative MRLs, which could not be finalised without submission of additional data. These data are to be submitted no later than 13th of July 2015 according to the provisions of Commission Regulation (EU) No. 668/2013. EFSA concluded that the following data would be needed to conclude on the tentative MRLs:

- 3 additional residue trials on broccoli supporting the northern outdoor GAP on broccoli and cauliflower
- 2 additional residue trials on broccoli supporting the southern outdoor GAP on broccoli and cauliflower
- 8 residue trials supporting the import tolerance on broccoli and cauliflower are required
- 4 residue trials supporting the southern outdoor GAP on lamb's lettuce, rocket, rucola, leaves and sprouts of brassica, chervil, celery leaves and parsley
- A hydrolysis study investigating the effect of sterilisation on the nature of residues, in particular for apples which are the main contributors to the chronic exposure
- Further information on the nature and occurrence of the metabolite F that was encountered in poultry
- Storage stability data for eggs

The EFSA review also cited data gaps that would not impact on the validity of the recommended MRLs, but might have an impact on National Authorizations:

- 2 residue trials supporting the southern outdoor GAP on tomatoes and aubergines
- 8 residue trials supporting the northern GAP in lettuce and escarole
- 8 residue trials supporting the Portuguese indoor GAP on escarole, rocket, rucola and leaves and sprouts of brassica are required

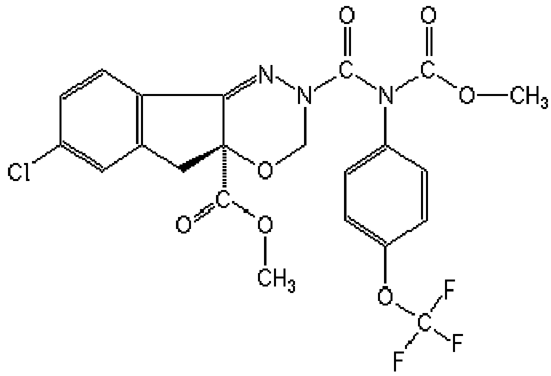
Furthermore, EFSA also cited minor deficiencies where additional data would be desired but not required:

- 1 additional residue trial complying with the northern GAP in head cabbage
- 4 trials complying with the southern GAP in maize forage

DATA GENERATED TO ADDRESS THESE OPEN POINTS ARE PRESENTED AND DISCUSSED IN THE RESIDUE SECTIONS BELOW.

THE ACTIVE SUBSTANCE AND ITS USE PATTERN

Indoxacarb is the ISO common name for methyl(S)-N-[7-chloro-2,3,4a,5-tetrahydro-4a-(methoxycarbonyl)indeno[1,2-e][1,3,4]oxadiazin-2-ylcarbonyl]-4-trifluoromethoxy)carbanilate (IUPAC).



Indoxacarb (C22H17ClF3N3O7)

Indoxacarb belongs to the chemical group of Oxadiazin insecticides and is a non-systemic substance. Indoxacarb as defined by ISO refers only to the S enantiomer while the technical material is normally a mixture of S and R enantiomers.

Indoxacarb is an insecticide, active as a larvicide by stomach and contact routes of entry into the insect. The importance of stomach versus contact action varies with the species and the crop situation. Indoxacarb is effective on all larvae stages of Lepidoptera and on some other groups (Orthoptera, some Hemiptera and Homoptera in some situations).

Indoxacarb was peer reviewed according to Commission Directive 91/414/EEC with The Netherlands being the designated Rapporteur Member State. The representative uses evaluated were outdoor applications on brassica vegetables (both in northern and southern Europe), indoor and outdoor applications (in southern Europe only) on tomatoes, pepper and cucurbits, edible and inedible peel, and outdoor application on lettuce in southern Europe at one to six applications at doses ranging from 25 g/ha to 37,5g/ha with a PHI ranging from one to three days. Following the peer review, which was not carried out by EFSA, a decision on inclusion of the active substance in Annex I to Directive 91/414/EEC was published by means of Commission Directive 2006/10/EC7, entering into force on 01 April 2006. According to Regulation (EU) No 540/20118, indoxacarb is deemed to have been approved under Regulation (EC) No 1107/20099 as well. This approval is restricted to uses as insecticide only. The renewal of approval of indoxacarb is ongoing with France being the designated Rapporteur Member State.

In European Community specific MRLs are set for indoxacarb. The residues are expressed in indoxacarb (sum of indoxacarb (S) and its R enantiomer) for monitoring and risk assessment in vegetable and animal commodities (EU Regulation N°668/2013 and documents SANCO/12375/2013, SANCO/11864/2013).

EU MRLs for indoxacarb in products of plant and animal origin have been set for the first time in 2007 by means of Directive 2007/27/EC10. After the entry into force of Regulation (EC) No 396/2005, these MRLs have been transferred to Annex II of Regulation (EC) No 396/2005. In Annex III of the Regulation temporary MRLs were established for crops that were not covered by previous Community MRL legislation.

Assessment

1. Methods of analysis

1.1. Methods for enforcement of residues in food of plant origin

Residue definition in plant: Indoxacarb (sum of isomers)

An analytical method (Čermák, J., 2013) and its ILV (Stanislowski, T., 2015) based on LC-MS/MS for the determination of indoxacarb residue in crops has been provided in the RAR of the active substance and validated with a LOQ of 0.01mg/kg in high water content, acidic, fatty and dry commodities. As data have been provided for two mass transitions, the method is considered as highly specific.

However a data gap was identified in the RAR concerning Extraction efficiency. Justification given by notifier for Extraction Efficiency, based on “similar” physical or chemical properties (e.g. density, dipole moment, dielectric constant, “polarity”) of the different solvents is not sufficient to be acceptable. Thus, extraction efficiency in different solvent systems used in monitoring studies (acetone/water for high water content, acidic and dry commodities and acetone/acetonitrile for high fat content crops) should be provided.

1.2. Methods for enforcement of residues in food of animal origin

Residue definition in animal tissues, milk, and eggs: parent (sum of isomers) and the metabolite IN JT333

(IN-JT333: methyl 7-chloro-2,5-dihydro-2-[[[4-(trifluoromethoxy)phenyl]amino]= carbonyl]indeno[1,2-e][1,3,4]oxadiazine-4a(3H)-carboxylate)

An analytical method (J.J Stry, 2004, report DuPont 12739 Rev1) for the determination of indoxacarb residue in animal products by LC-MS/MS has been provided and is validated with a LOQ of 0.01mg/kg for indoxacarb (sum of isomers) and metabolites IN-JU873, IN-JT333, IN-KB687, IN-KG433 and IN-KT319 in liver, muscle, fat, skin, whole eggs, egg white, egg yolks. For eggs, ion ratio data are acceptable for each analyte, so the method can be considered highly specific. For other matrix, the method is not highly specific since confirmatory data were not provided. Additionally, the Extraction efficiency was not presented.

No validation data were presented for milk matrix.

An ILV (P. Connolly, 2004, report DuPont 13651 Rev1) of method J.J Stry, 2004 (report DuPont 12739 Rev1) for the determination of indoxacarb residue in foodstuff of animal origin has been provided but is not validated. The number of samples per level used for accuracy/precision is not sufficient.

An analytical method (S. Richter, 2013) based on DFG S19 multiresidue method using LC-MS/MS for the determination of indoxacarb residue in foodstuff of animal origin (milk, eggs, liver and muscle) has been validated with a LOQ of 0.01mg/kg. Notifier should provide the extraction efficiency in different solvent systems used in monitoring studies. No validation data was presented for fat matrix. Additionally, no ILV was provided.

DFG multiresidue method S19 (Linkerhagner, M., Guinivan, R.A., 2001 And Class, T (2001), DuPont-2338, Revision No. 1 and DuPont-6224) was validated for indoxacarb (sum of isomers) and IN-JT333 in animal product (milk, eggs, meat, liver, fat, and kidney) in the previous DAR. However, according to actual guidance method is not considered as fully validate as linearity and specificity of main method were missing additionally, the test facilities are not reported and study reports were not presented. Its ILV (Class, T. 2000, DuPont-39006) have been provided for two columns of different polarity, the method can be regarded as highly specific.

1.3. Methods for risk assessment of residues in food of plant origin (data for studies on residues trials)

Analytical method used in the residue trials (Report DuPont-19901) is validated with LOQ of 0.2mg/kg for indoxacarb in eggs, liver, fat and muscle.

Analytical method used in the residue trials (DuPont 35819) is validated with LOQ of 0.01mg/kg for indoxacarb (sum of isomers) in leaf lettuce and lamb lettuce.

Analytical method used in the residue trials (reports DuPont 33516, DuPont 33518, 33517, 08 I CL DP P/A) is validated with LOQ of 0.01mg/kg for indoxacarb (sum of isomers) in apple, lettuce, green bean plant and pods.

Analytical method used in the residue trials (reports DuPont-35437, DuPont-39889, DuPont-35169 and DuPont-35170) is validated with LOQ of 0.01mg/kg for indoxacarb (sum of isomers) in broccoli, head cabbage, apple, unprocessed apple, peeled apple, canned apple with skin, apple juice, apple jam, apple sauce, wet pomace, tomato, tomato puree, tomato paste, tomato catsup, tomato juice.

Analytical method used in the residue trials (DuPont-35172) is validated with LOQ of 0.01mg/kg for indoxacarb (sum of isomers) in Forage maize.

Analytical method used in the residue trials (DuPont-6061 and DuPont-6062) is validated with LOQ of 0.01mg/kg for indoxacarb (sum of isomers) in broccoli, cauliflower and cabbage.

Analytical method used in the residue trials (DuPont-19688) is validated with LOQ of 0.01mg/kg for indoxacarb (sum of isomers) in broccoli and cauliflower.

2. Mammalian toxicology

Table 2-7. **Overview of the toxicological reference values**

	Source	**Year**	**Value (mg/kg bw/d)**	**Study relied upon**	**Safety factor**
Indoxacarb (mixture of S and R enantiomers)					
ADI	COM	2005	0.006 mg/kg bw/d	2 years, rat (racemic mixture of S and R enantiomers)	100
ARfD	COM	2005	0.125 mg/kg bw	Rat, acute neurotoxicity (mixture of S and R enantiomers (75%S/25%R))	100

3. Residues

3.1. Nature and magnitude of residues in plant

3.1.1. Primary crops

3.1.1.1. Nature of residues

Please refer to the art 12 EFSA reasoned opinion. EFSA did not require any additional data for plant metabolism studies in the MRL review.

3.1.1.2. Magnitude of residues

Storage stability

The EFSA review concluded that additional storage stability data for eggs were required (EFSA Journal 2011;9 (8):2343).

A storage stability study on poultry matrices has been conducted with DPX-MP062 and its metabolites.

The analyses of the control samples showed that no residues of any component were present. The residues of all components showed no significant decrease (>30% as compared to the fortification concentration) in the different matrices after storage deep frozen for at least 16 months.

According to the results, parent compound DPX-MP062 and its five metabolites (DPX-MP062, IN-KB687, IN-KG433, IN-KT319, IN-JU873 and IN-JT333) are stable at approximately -20°C for at least 16 months in hen matrices (whole egg, liver, fat, and muscle).

Residue trials

Results of supervised field trials are summarized in Table 3.1.2. More details concerning supervised field residue trials can be found in Appendix C.

(a) Broccoli and cauliflower

The EFSA MRL review identified the need for 3 additional residue trials on broccoli supporting the northern outdoor GAP on broccoli and cauliflower and 2 additional residue trials on broccoli supporting the southern outdoor GAP on broccoli and cauliflower.

Identification of critical GAPs

Crop	**Region**	**Outdoor/ Protected**	**Application**	**Number of applications (days interval)**	**Rate (g as/ha)**	**BBCH at last application / PHI**
Broccoli and cauliflower	NEU SEU	outdoor	Foliar spray	3 (10)	25.5	1

Five additional trials on broccoli and 2 additional trials on cauliflower supporting the northern outdoor GAP on broccoli and cauliflower (25.5 g a.s./ha, 3 applications at 10 day intervals with a 1 day PHI) using DPX-MP062 30WG and six additional trials on broccoli and 2 additional trials on cauliflower supporting the southern outdoor GAP on broccoli and cauliflower (25.5 g a.s./ha, 3 applications at 10 day intervals with a 1 day PHI using DPX-MP062 30WG) were conducted. Two trials conducted on broccoli with DPX-MP062-30WG and DPX-KN128-15EC in the northern of Europe according to the intended GAP are also available.

In addition, 1 northern trial and 2 southern trials conducted with a more critical GAP (37.5 g as/ha, 3 applications with a 1 day PHI) are also available. In order to take these trials into account, proportionality was applied.

For report DuPont-6061 (Hornshuh, M.J., Enriquez, M.A., Gakeme, B, 2002), analytical method is not fully validated. Chromatograms of control and fortified samples (cauliflower) at LOQ are required to check specificity.

As regards these new trials, residue levels of indoxacarb in broccoli ranged from 0.023 mg/kg to 0.089 mg/kg in northern trials and from <0.02 mg/kg to 0.15 mg/kg in the southern trials.

These residue data were pooled with those already assessed by EFSA in the framework of MRL review.

Then, sufficient residue data are available for broccoli and cauliflower and the calculations result in the proposal of **a MRL of 0.3 mg/kg.** This had no significant effect on the EFSA evaluation.

(b) Lettuce and other salad plants

Lettuce – northern GAP

Concerning lettuce, no trials were available to support the norther outdoor GAP (EFSA Journal 2011;9(8):2443). However, appropriate MRL and risk assessment values could be derived based on the US import tolerance GAP. During the Member States consultation, Belgium stated that the northern outdoor GAP on lettuce was largely covered by this practice and that further trials were therefore not required (Belgium, 2011). EFSA however does not agree with this statement because the US import tolerance on lettuce is identified in section 4 as a potential risk to consumers. **8 residue trials on lettuce compliant with the northern outdoor GAP are therefore still required.**

Identification of critical GAPs

Crop	**Region**	**Outdoor/ Protected**	**Application**	**Number of applications (days interval)**	**Rate (g as/ha)**	**BBCH at last application / PHI**
Lettuce	NEU	outdoor	Foliar treatment-spraying	2 (7d)	25.5	14 day

Eight additional trials on lettuce conducted in the northern Europe using DPX-KN128 30WG with 4 applications at 37.5 g as/ha and a 3 days PHI were submitted. One northern trial conducted using DPX-MP062 30WG and DPX-KN128 30WG with 6 applications at 37.5 g as/ha and a 1 to 14 days PHI is also available on lettuce. Since all the submitted trials were conducted according to a more critical GAP than the requested one, no trial was taken into account and no MRL was derived.

<u>Scarole – northern GAP</u>
See lettuce.

<u>Scarole, rocket, rucola, leaves and sprouts of brassica – indoor GAP</u>
The EFSA review also cited the need for 8 residue trials supporting the Portuguese indoor GAP on escarole, rocket, rucola and leaves and sprouts of brassica to fill data GAPs that might have an impact on National Authorizations.

Indoor residue trials to support the Portuguese indoor GAP on escarole, rocket, rucola and leaves and sprouts of brassica have not been conducted. There are no current plans to conduct these trials.

<u>Lamb's lettuce – southern GAP</u>
The southern outdoor GAP was not supported by GAP compliant residue trials (EFSA Journal 2011;9(8):2443). Although the southern lettuce data (compliant with a more critical GAP) can be extrapolated on a tentative basis, 8 residue trials supporting the southern outdoor GAP on this crop are still required. These trials can be performed on lettuce provided that open leaf varieties are used (EFSA Journal 2011;9(8):2443).

Identification of critical GAPs

Crop	**Region**	**Outdoor/ Protected**	**Application**	**Number of applications**	**Rate (g as/ha)**	**BBCH at last**

				(days interval)		application / PHI
Lamb's lettuce	SEU	outdoor	Foliar treatment-spraying	6 (10d)	37.5	3 days

Two additional residue trials conducted in southern Europe using DPX-MP062-30WG and DPX-KN128-15EC on lettuce (open leaf) with 6 applications at 37.5 g as/ha and with a 1 day PHI are available. Two other residue trials using DPX-MP062 30WG and DPX-KN128 30WG on open leaf lettuce with 6 applications at 37.5 g as/ha and with a 1 day PHI are also available.

Other available trials on lettuce were conducted with a less critical GAP (please refer to Appendix C).

A total of 4 residue trials conducted on open leaf lettuce are available and residue levels of indoxacarb ranged from 0.60 mg/kg to 1.7 mg/kg with a 1 day PHI (instead of a 3 days PHI).

Note: residue levels from residue trials conducted in northern Europe on lamb's lettuce with a lower application rate and for the same PHI were higher (residue levels ranged from 1.2 to 4.9 mg/kg). Therefore, extrapolation from open leaf lettuce to lamb's lettuce seems questionable (please refer to lamb's lettuce trials in Appendix C).

<u>Chervil, celery leaves and parsley – southern GAP</u>
In the framework of the MRL review, the southern outdoor GAP on chervil, celery leaves and parsley, was also not supported by GAP compliant residue trials and the southern lettuce data (compliant with a more critical GAP) were extrapolated on a tentative basis. Considering that in this case the indoor GAP was fully supported by data and leads to the same MRL, the MRL was not considered tentative. Nevertheless, 8 residue trials supporting the southern outdoor GAP on these crops have been required by EFSA for national authorisation (EFSA Journal 2011;9(8):2443).

No residue trials conducted according to the southern outdoor GAP were submitted (please refer to lettuce residue trials in Appendix C).

(c) Tomato

The EFSA review cited requirements for 2 residue trials supporting the southern outdoor GAP on tomatoes and aubergines.

Identification of critical GAPs

Crop	Region	Outdoor/ Protected	Application	Number of applications (days interval)	Rate (g as/ha)	BBCH at last application /

						PHI
tomato	SEU indoor	outdoor	Foliar spray	6 (10)	37.5	1

Three additional residue trials have been performed in the southern of Europe with indoxacarb (DPX-MP062 30WG) according to the intended GAP. Tomatoes were treated with 6 applications of 37.5 g a.s./ha and raw tomatoes for processing studies were sampled 1 day after the last application. Sampled tomatoes were frozen and shipped until processing phases. Analyses were performed following the DFG method S19 (DuPont report AMR 4271-96).

Analytical method was not validated for unprocessed tomato and canned tomato. Indeed, no specificity data have been provided for unprocessed tomato and canned tomato. Chromatograms of control and fortified unprocessed tomato and canned tomato at LOQ are required to check specificity.

As regards these new trials, residue levels of indoxacarb in tomato ranged from 0.04 mg/kg to 0.07 mg/kg.

These residue data were pooled with those already assessed by EFSA in the framework of MRL review and the calculations result in **a MRL of 0.4 mg/kg. However, the existing MRLs (0.5 mg/kg) based on import tolerance GAP is not affected.** This had no significant effect on the EFSA evaluation.

(a) Head cabbage

The EFSA review cited the need for one additional residue trial complying with the northern GAP in head cabbage.

Identification of critical GAPs

Crop	**Region**	**Outdoor/ Protected**	**Application**	**Number of applications (days interval)**	**Rate (g as/ha)**	**BBCH at last application / PHI**
Head cabbage	NEU / SEU	outdoor	Foliar spray	3 (10)	25	3

Four additional trial data have been generated for the NEU zone. These trials were conducted according to the intended GAP (25 g. as/ha, 3 applications with a 3 day PHI) using DPX-MP062 30WG.

Residue levels of indoxacarb in head cabbage ranged from <0.02 mg/kg to 0.14 mg/kg. These residue data were pooled with those already assessed by EFSA in the framework of MRL review and the calculations result in the proposal of **a MRL of 0.2 mg/kg.** This had no significant effect on the EFSA evaluation.

(a) Maize forage

According to EFSA, 4 trials complying with the southern GAP on maize forage would be desirable to ensure that the livestock dietary burden calculation also covers the southern outdoor use.

Identification of critical GAPs

Crop	**Region**	**Outdoor/ Protected**	**Application**	**Number of applications (days interval)**	**Rate (g as/ha)**	**BBCH at last application / PHI**
Maize forage	SEU	outdoor	Hydraulic ground directed boom	2 (20days)	38	BBCH 77

Five additional residue trials on maize forage using DPX-MP062 30WG with 2 applications at 37.5 g a.s./ha with the last application at BBCH 69 were conducted in field season 2012. The interval between applications was **10 days instead of 20 days required**. Allowing 2 weeks for the maize to develop from BBCH 69 (last treatment) to BBCH 86 (mid to late dough stage when maize is typically used as forage) the 14 day forage data were selected.

Residue levels of indoxacarb in maize forage ranged from 0.14 mg/kg to 0.77 mg/kg. These residue data were pooled with those already assessed by EFSA in the framework of MRL review and the calculations result in the proposal of **a STMR of 0.20 mg/kg and a HR of 0.77 mg/kg.**

Table 3.1.1-1: Overview of the available residues trials data

Commodity	Region [(a)]	Outdoor /Indoor	Individual trial results (mg/kg)		STMR (mg/kg) [(b)]	HR (mg/kg) [(c)]	MRL proposal (mg/kg)	Median CF [(d)]	Comments
			Enforcement	Risk assessment					
Enforcement residue **sum of indoxacarb and its R enantiomer**									
Broccoli and cauliflower	NEU	Outdoor	<0.02, **0.02[1], 0.023,** 0.03, **0.06, 0.061, 0.065,** 0.07, **0.076, 0.08[1], 0.088, 0.089,** 0.11, **0.12**, 0.13, 0.13	<0.02, **0.02[1], 0.023,** 0.03, **0.06, 0.061, 0.065,** 0.07, **0.076, 0.08[1], 0.088, 0.089,** 0.11, **0.12**, 0.13, 0.13	0.070	0.130	0.3 (unrounded : 0.224)	1	[1] Analytical method not validated
	SEU	Outdoor	2x<0.02, **<0.02, <0.02[1], 0.02[1], 0.025, 0.026, 0.040, 0.044,** 0.05, 0.07, **0.07**, 0.08, **0.12**, 0.13, 0.14, **0.15,** 0.16, 0.21	2x<0.02, **<0.02, <0.02[1], 0.02[1], 0.025, 0.026, 0.040, 0.044,** 0.05, 0.07, **0.07**, 0.08, **0.12**, 0.13, 0.14, **0.15,** 0.16, 0.21	0.050	0.210	**0.3 (unrounded : 0.309)**	1	[1] Analytical method not validated
Lettuce	NEU	outdoor	-	-	-	-	-	-	No trial available.
Lamb's lettuce	SEU	outdoor	0.16, 0.19, 0.25, 0.39, 2x0.52, 0.55, **0.60,** 0.86, 0.89, **1.4, 1.4,** 1.6, **1.7**	0.16, 0.19, 0.25, 0.39, 2x0.52, 0.55, **0.60,** 0.86, 0.89, **1.4, 1.4,** 1.6, **1.7**	0.6	1.7	*Tentative : 3*	-	Extrapolation from open leaf lettuce residue trials (PHI 1 day).
Tomato	SEU	Outdoor	0.03, **0.04[1],** 0.045, **0.047[1]**, 0.06, **0.07[1]**, 0.105, 0.155, 0.26	0.03, **0.04[1], 0.047[1]**, 0.05, 0.06, **0.07[1]**, 0.11, 0.16, 0.26	0.06	0.26	0.4 (unrounded: 0.392)	1	[1] Analytical method not validated
Head cabbage	NEU	outdoor	6x<0.02, **2x<0.02**, **0.029**, 0.09, **0.14**	6x<0.02, **2x<0.02**, **0.029**, 0.09, **0.14**	0.02	0.14	0.2 (unrounded: 0.197)	1	
	SEU	outdoor	4x<0.02, 0.02, 0.05, 0.07	4x<0.02, 0.02, 0.05, 0.07	0.02	0.07	0.2	1	
Maize forage	NEU	outdoor	0.04, 0.10, 0.16, 0.28	0.04, 0.10, 0.16, 0.28	0.13	0.28	0.70	1	
	SEU	outdoor	**0.14, 0.18, 0.32, 0.34, 0.77**	**0.14, 0.18, 0.32, 0.34, 0.77**	0.32	0.77	n.a.		

(a): NEU, SEU, EU or Import (country code). In the case of indoor uses there is no necessity to differentiate between NEU and SEU.
(b): Median value of the individual trial results according to the enforcement residue definition.
(c): Highest value of the individual trial results according to the enforcement residue definition.

(d): The median conversion factor for enforcement to risk assessment is obtained by calculating the median of the individual conversion factors for each residues trial.
(*): Indicates that the MRL is set at the limit of analytical quantification.
In bold: data from new residue trials not already assessed by EFSA

3.1.1.3. Effect of industrial processing and/or household preparation

<u>Nature of residue</u>

EFSA reviewed available high temperature hydrolysis information and concluded "The effect of processing on the nature of indoxacarb was investigated in the framework of the peer review. Studies were conducted simulating representative hydrolytic conditions for pasteurisation (20 minutes at 90°C, pH 4) and boiling/brewing/baking (60 minutes at 100°C, pH 5). No study simulating sterilisation (20 minutes at 120°C, pH 6) was carried out. The hydrolysis studies demonstrated that pasteurisation and baking/boiling conditions had little influence on the nature of the parent compound and were not resulting in a formation of toxicologically significant degradation products (the Netherlands, 2000). Thus, for processed commodities that are not subject to sterilisation, the same residue definition as for raw agricultural commodities (RAC) is applicable" (EFSA Journal 2011;9(8):2343). The EFSA review also stated "Considering however that the chronic exposure exceeds 10% of the ADI, a hydrolysis study for sterilisation is required for commodities that may be canned, in particular for apples which are the main contributors to the chronic exposure." A high temperature hydrolysis study has been conducted and is summarised in part C.4.2.

The results of the study confirm the stability of parent compound DPX-KN128 under pasteurisation and baking conditions since no metabolites > 10% were measured after high temperature conditions. However, the sterilisation study showed a decrease from 98.6% of the indanone and 100 % of the trifluoromethoxyphenyl-labelled parent compound to 46.5 % and 42.8% AR after 20 minutes at 120°C. In parallel, **metabolites IN-KT413, IN-MP819, IN-P0036 and IN-TMG00 were measured at a level ≥10% of the total dose.**

Table 3.1.1-2: Distribution and mass balance of radioactivity for pH 6 buffer test system [Indanone-^{14}C]and [4-Trifluoromethoxyphenyl-^{14}C]DPX-KN128

		Control 1-Indanone	Control 4-Trifluoro methoxyphenyl	120°C 1-Indanone	120°C 4-Trifluoro methoxyphenyl
DPX-KN128 (parent)	**% AR[a]**	**98.6**	**100.0**	**46.5**	**42.8**
IN-KT413	**% AR**	**0.5**	**0.0**	**28.7**	**2.5**
IN-MP819	**% AR**	**0.0**	**0.0**	**9.9**	**8.5**
IN-MK638	**% AR**	**N/A**	**0.0**	**N/A**	**9.9**
IN-P0036	**% AR**	**N/A**	**0.0**	**N/A**	**16.1**
IN-TMG00	**% AR**	**0.0**	**0.0**	**8.7**	**12.4**
Others[b]	**% AR**	**0.9**	**0.0**	**11.3**	**3.1**
Total % recovery[c]	**% AR**	**100.0**	**100.0**	**105.2**	**95.1**
Average % recovery	**% AR**	**100.0**		**100.1**	

nd = not detected

a AR = Applied Radioactivity or initial dose.

b Others = components not identified. Individual ^{14}C components detected by HPLC were less than 4.0% of the applied radioactivity

c Recovery of administered radioactivity as determined by LSC analyses of the dosing solution.

Magnitude of residue

EFSA evaluated indoxacarb processing studies and concluded “Studies investigating the magnitude of residues in some processed products are also available, but in most cases they only allowed EFSA to derive indicative processing factors. With regard to the risk assessment, further processing studies on the magnitude of residues are not required because they are not expected to affect the outcome of the risk assessment. However, if there would be the intention from risk managers to derive more processing factors for enforcement purposes, additional processing studies might be required.” (EFSA Journal 2011; 9(8):2343)). DuPont has conducted new processing studies for apples and tomatoes to derive additional processing factors (see § C.4.2).

Table 3.1.1-3: Overview of the available processing studies

Processed commodity	Number of values (new data)	Median TF [(a)] (new data)	Number of values (art.12)	Median TF [(a)] (art.12)	Mean TF (all data)	Median CF [(b)]
Enforcement residue **sum of indoxacarb and its R enantiomer**						
Juice to pasteurize (tomato)	3	1.45	1	1.1	1.28	-
Tomato puree	3	1.64				-
Tomato paste	3	2.75				-
Peeled tomatoes (canned tomatoes)	3	0.28				-
Ketchup	3	1.83	1	2.37	2.1	-
Tomato, sauce	-	-	1	0.30		
Peeled apples	3	0.18	-	-		-
Canned apples	3	0.48	-	-		-
Appel jam	3	0.03	-	-		-
Wet pomace	3	2.60	2	2.75	2.7	-
Filtered juice	3	0.03	2	0.30	0.16	-
Apple sauce	3	0.28	2	0.30	0.29	-

(a): The median transfer factor is obtained by calculating the median of the individual transfer factors of each processing study.
(b): The median conversion factor for enforcement to risk assessment is obtained by calculating the median of the individual conversion factors of each processing study.

3.1.2. Rotational crops

Please refer to the RAR. EFSA did not cite any required additional data for rotational crops studies in the MRL review.

3.2. Nature and magnitude of residues in livestock

3.2.1. Dietary burden

Table 3-4. Input values for the dietary burden calculation

Commodity	Median dietary burden	Maximum dietary burden

	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
sum of indoxacarb and its R enantiomer				
Apples pomace	0.58	Median x PF	0.58	Median x PF
Cabbage	0.44	Median residue	2.70	Highest residue
Kale	0.05	Median residue	0.13	Highest residue
Rape seed meal	0.01	Median residue	0.01	Median
Soya bean	0.03	Median residue	0.03	Median
Soya bean meal	0.03	Median residue	0.03	Median
Cotton seed	0.02	Median residue	0.02	Median
Cotton seed meal	0.02	Median residue	0.02	Median
Maize grain	0.01	Median residue	0.01	Median
Maize by product : milled by-products, hominy meal, gluten feed, gluten (meal)	0.01	Median residue	0.01	Median residue
Maize silage/forage	**0.22**	**Median residue**	**0.77**	**Highest residue**
Sugar beet leaves	0.41	Median residue	0.99	Highest residue

Table 3-5. **Results of the dietary burden calculation**

	Max dietary burden (mg/kg bw/d)	Median dietary burden (mg/kg bw/d)	Highest contributing commodity	Max dietary burden (mg/kg DM)	Trigger exceeded?
Beef cattle	0.093	0.0212	Cabbage, heads	3.90	**Yes**
Dairy cattle	0.147	0.030	Cabbage, heads	3.81	**Yes**
Sheep (ram/ewe)	0.067	0.019	Cabbage, heads	2.02	**Yes**
Sheep (Lamb)	0.086	0.024	Cabbage, heads	2.02	**Yes**
Breeding swine	0.042	0.007	Cabbage, heads	1.82	**Yes**
Finishing swine	0.001	0.001	Soybean meal	0.02	No
Poultry-broiler	0.001	0.001	Soybean meal	0.02	No
Poultry-layer	0.063	0.012	Cabbage, heads	0.93	**Yes**
Poultry turkey	0.001	0.001	Soybean meal	0.02	No

Calculated MRL for bovine, sheep, swine and poultry are lower than the existing CXL MRLs. Therefore, no new MRLs are proposed.

3.2.2. Nature of residues

The EFSA MRL review (EFSA Journal 2013;11(11):3458) cited the need for further information on the nature and occurrence of the metabolite F that was encountered in poultry. No other items related to metabolism were cited. In the 2011 EFSA Article 12 MRL review, EFSA commented about the lack of positive identification of Metabolite F in poultry and therefore could not finalize the definition of the residue for the risk assessment.

DuPont conducted a study, which is not yet complete and for which no information but the following summary was submitted.Hens were dosed for 5 consecutive days at an exaggerated dose (ca. 50/mg/kg dietary) of non-radiolabeled indoxacarb as DPX-JW062, the racemic mixture of DPX-KN128 and IN-KN127, to generate larger quantities of Metabolite F. Fat samples were extracted with acetonitrile, the extracts were concentrated and initially analysed by LC/MS. Upon confirmation of the presence of a metabolite with the same molecular weight and similar retention time as Metabolite F in the extract compared to that found in the original report (AMR 3187-94, previously submitted and evaluated at the EU level), the metabolite was isolated and purified. Attempts were made to deduce the structure by LC/MS/MS and NMR using the purified metabolite.

Spectral data was able to confirm some parts of the original proposed structure but not all components of the structure. NMR confirmed the presence of an impurity or impurities which prevented a definitive structure from being assigned even though the isolated metabolite was purified a number of times. Furthermore, it was found that the metabolite was not stable after 100 days under frozen conditions (-20°C). This mixture was re-purified resulting in a cleaner sample. However, the concentrations of the impurities were such that high resolution LC/MS and NMR could not conclusively assign a structure to the metabolite. Discussions are continuing on a resolution to resolve these issues and determine the best procedures to confirm the structure of Metabolite F.

3.2.3. Magnitude of residues

Please refer to the RAR. EFSA did not cite any required additional data for nature and magnitude of residues in livestock in the MRL review.

4. Consumer risk assessment

The consumer risk assessment regarding the parent compound indoxacarb was performed with the EFSA PRIMo-rev.2 (Pesticide Residue Intake Model).

Chronic exposure

A calculation of the long term dietary exposure to indoxacarb and its R enantiomer according to the European PRIMo model was performed based on an ADI of 0.006 mg/kg bw/d.

The calculation of TDMI was first realized with the in force MRL (Reg. (EU) 2015/845) and the new MRL proposed in the framework of this evaluation report (see table 3.1-4). The calculated TMDI exceed 100% of ADI for 18 diets (see Appendix B).

Table 3.1.1-4: Input values for the consumer risk assessment

Commodity	Chronic risk assessment		Acute risk assessment	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
sum of indoxacarb and its R enantiomer				
Flowering brassicace	0.3	Proposed MRL	0.21	Calculated HR
Lamb's lettuce	30	In force MRL	15*	EFSA-Q-2012-00027
Tomato, aubergine	0.5	In force MRL	0.3	EFSA-Q-2008-565 (IT)
Head cabbage	0.2	Proposed MRL	0.14	Calculated HR
Other commodities	In force MRL	Reg. (EU) 2015/845	-	-
sum of indoxacarb, its R enantiomer and IN-JT333				
Poultry meat	0.01x6	In force MRL x CF	-	-
Poultry fat	0.01x6	In force MRL x CF	-	-
Poultry liver	0.01x2	In force MRL x CF	-	-
Birds'eggs	0.01	In force MRL	-	-

Then to approach a more realistic assessment the calculation of the IEDI (International Estimated Daily Intake) was realized with STMRs values proposed in Table 3.1-5.

Table 3.1.1-5: Input refined values for the consumer risk assessment

Commodity	Chronic risk assessment		Acute risk assessment	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
sum of indoxacarb and its R enantiomer				
Flowering brassicace	0.07	Calculated STMR	0.21	Calculated HR
Lamb's lettuce*	8.6	STMR EFSA-Q-2012-00027	15	HR EFSA-Q-2012-00027

Commodity	Chronic risk assessment		Acute risk assessment	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Tomato, aubergine	0.11	STMR CXL **EFSA-Q-2008-565**	0.3	HR (IT) EFSA-Q-2008-565
Head cabbage	0.02	Calculated STMR	0.14	Calculated HR
Apple	0.21	STMR EFSA-Q-2008-565	-	-
Pear	0.21	STMR EFSA-Q-2008-565	-	-
Apricots	0.17	STMR CXL EFSA-Q-2008-565	-	-
Cherries	0.17	STMR CXL EFSA-Q-2008-565	-	-
Peaches	0.17	STMR CXL EFSA-Q-2008-565	-	-
Table and wine grapes	0.32	STMR EFSA-Q-2008-565	-	-
Strawberries	0.19	STMR EFSA-Q-2012-00027	-	-
blackberries	0.255	STMR EFSA-Q-2008-565	-	-
Raspberries	0.255	STMR EFSA-Q-2008-565	-	-
Blueberries, currants, gooseberries, rose hips, mulberries, azarole, elderberries and other small fruits	0.11	STMR EFSA-Q-2008-565	-	-
Cranberries	0.15	STMR CXL EFSA-Q-2008-565	-	-
Bananas	0.04	STMR EFSA-Q-2008-565	-	-
Radishes	0.02	STMR EFSA-Q-2008-565	-	-
Peppers	0.06	STMR EFSA-Q-2008-565	-	-
Brussels sprouts	0.02	STMR EFSA-Q-2008-565	-	-
Chinese cabbage	0.49	STMR EFSA-Q-2012-00027	-	-
Kale	0.05	STMR EFSA-Q-2008-565	-	-
Lettuce	0.52	STMR EFSA-Q-2008-565	-	-

Commodity	Chronic risk assessment		Acute risk assessment	
	Input value (mg/kg)	**Comment**	**Input value (mg/kg)**	**Comment**
Scarole, cress, land cress, red mustard and other lettuce and salad plants, purslane, beet leaves	0.27	STMR EFSA-Q-2008-565	-	-
Rocket, rucola, leaves and sprouts of Brassica, chervil, celery leaves, parsley	0.52	STMR EFSA-Q-2008-565	-	-
Spinach	0.78	STMR EFSA-Q-2008-565	-	-
Chives, sage, rosemary, thyme, basil, bay leaves, tarragon, other herbs	0.375	STMR EFSA-Q-2008-565	-	-
Beans (with pods)	0.11	STMR EFSA-Q-2013-00541	-	-
Cardoons, fennel, rhubarb	0.97	EFSA-Q-2012-00027	-	-
Celery	0.849	EFSA-Q-2008-565	-	-
Globe artichoke	0.035	EFSA-Q-2008-565	-	-
Beans (dry) peas (dry)	0.02	STMR CXL EFSA-Q-2008-565	-	-
Peanuts	0.01	STMR CXL EFSA-Q-2008-565	-	-
Rape seed	0.01	STMR EFSA-Q-2008-565	-	-
Soya bean	0.03	STMR EFSA-Q-2008-565	-	-
Cotton seed	0.01	STMR EFSA-Q-2013-00731	-	-
Maize grain	0.01	STMR EFSA-Q-2013-00731	-	-
Sugar beet (root)	0.026	STMR EFSA-Q-2008-565	-	-
Swine meat	0.01	STMR EFSA-Q-2008-565	-	-
Swine fat	0.38	STMR EFSA-Q-2008-565	-	-
Swine liver and kidneys	0.014	STMR EFSA-Q-2008-565	-	-
Ruminant meat	0.01	STMR EFSA-Q-2008-565	-	-
Ruminant fat	0.38	STMR EFSA-Q-2008-565	-	-
Ruminant liver and kidneys	0.14	STMR EFSA-Q-2008-565	-	-

Commodity	Chronic risk assessment		Acute risk assessment	
	Input value (mg/kg)	**Comment**	**Input value (mg/kg)**	**Comment**
milk	0.037	STMR EFSA-Q-2008-565	-	-
sum of indoxacarb, its R enantiomer and IN-JT333				
Poultry meat	0.06	STMRxCF EFSA-Q-2008-565	-	-
Poultry fat	0.06	STMRxCF EFSA-Q-2008-565	-	-
Poultry liver	0.02	STMRxCF EFSA-Q-2008-565	-	-
Birds'eggs	0.01	STMR CXL EFSA-Q-2008-565	-	-

* for lamb's lettuce value from EFSA-Q-2012-00027 was taken into account as a worst case.

The IEDI represents 91.4 % of the ADI. It can thus be concluded that there is an acceptable risk for the consumer (see Appendix B).

Acute exposure

A calculation of the short term dietary exposure to indoxacarb according to the European PRIMo model was performed based on an ARfD of 0.125 mg/kg/d.

The calculation of IESTI was realized with the HR or STMR proposed in table Table 3.1-5.

According to the PRIMo model, no exceedance of the ARfD was identified for any of the evaluated commodity (see Appendix B).

CONCLUSIONS AND RECOMMENDATIONS

Table 3.1-6: Overview of the proposed EC MRLs

Commodity	Existing EC MRL (mg/kg)	Proposed EC MRL (mg/kg)	Justification for the proposal
sum of indoxacarb and its R enantiomer			
Flowering Brassica	0.3	0.3	/
Lamb's lettuce	30	3	/
Tomatoes	0.5	0.5	/
Head cabbages	0.2	0.2	/

(*): Indicates that the MRL is set at the limit of analytical quantification.

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ADDITIONAL STUDIES RELIED UPON

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Author(s)	Year	Title/Testing Facility/Report No./GLP or GEP Status/Published or not	Submitter
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Aitken, A.	2014	Determination of the decline of residues of DPX-KN128 (indoxacarb) along with IN-KN127 in maize forage following applications of DPX-MP062 30WG - Southern Europe – 2012 Charles River Laboratories (UK) DuPont-35172 Published: No	DuPont
Clark, B.	2014	High temperature hydrolysis of ^{14}C-Indoxacarb (DPX-KN128) in Buffered Solutions at pH 4, 5 and 6 ABC Laboratories, Inc. DuPont-35316 Published: No	DuPont
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APPENDIX A – GOOD AGRICULTURAL PRACTICES (GAPS)

The critical EU GAPs for the use of indoxacarb are presented in the table below.

Crop and/ or situation (a)	Part of Europe	F G or I (b)	Pests or Group of pests controlled (c)	Formulation		Application				Application rate per treatment			PHI (days) (l)	Remarks: (m)
				Type (d-f)	Conc. of as (i)	Method Kind (f-h)	Growth stage & season (j)	number min - max (k)	interval between applications (min)	kg as/hL min - max	water L/ha min - max	g as/ha min - max		
Broccoli	NEU SEU	F	*M. brassicae, P. rapae, P. brassicae*	WG	30,0%	Foliar treatment - spraying	n.r.	1-3	10	n.r.	n.r.	25.5	1	
Cauliflower	NEU SEU	F	*M. brassicae, P. rapae, P. brassicae*	WG	30,0%	Foliar treatment - spraying	n.r.	1-3	10	n.r.	n.r.	25.5	1	
Lettuce	NEU	F	*n.r.*	WG	30,0%	Foliar treatment - spraying	n.r.	1-2	10	n.r.	n.r.	25.5	14	
Scarole	NEU	F	*n.r.*	WG	30,0%	Foliar treatment - spraying	n.r.	1-2	10	n.r.	n.r.	25.5	14	
Scarole, rocket, rucola, leaves and sprouts of brassica	-	I	n.r.	WG	30,0%	Foliar treatment - spraying	n.r.	3-4	10	n.r.	n.r.	37.5	14	
Lamb's lettuce	SEU	F	n.r.	WG	30,0%	Foliar treatment - spraying	n.r.	3-6	10	n.r.	n.r.	37.5	3	
Tomato and aubergine	SEU	F	*H. armigera, S. exigua, P. gamma*	WG	30,0%	Foliar treatment - spraying	n.r.	1-6	10	n.r.	n.r.	37.5	1	
Head cabbage	NEU SEU	F	*M. brassicae, P. rapae, P. brassicae*	WG	30,0%	Foliar treatment - spraying	n.r.	1-3	10	n.r.	n.r.	25.0	3	

Maize forage	SEU	F	n.r.	WG	30,0%	Foliar treatment - spraying	77	1-2	n.r.	n.r.	n.r.	38	n.a.	

APPENDIX B – PESTICIDE RESIDUES INTAKE MODEL (PRIMO)

Chronic risk assesment– not refined.

Chronic risk assessment

		TMDI (range) in % of ADI minimum - maximum			
		44	302		
		No of diets exceeding ADI:	18		

Highest calculated TMDI values in % of ADI	MS Diet	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
301,8	NL child	52,8	Apples	51,9	Swine: Meat	48,9	Milk and cream,
273,7	WHO Cluster diet B	71,4	Table and wine grapes	27,4	Bovine: Meat	25,8	Swine: Meat
273,1	DE child	100,6	Apples	42,3	Table and wine grapes	23,8	Milk and cream,
221,6	FR toddler	66,0	Milk and cream,	44,7	Bovine: Meat	23,6	Spinach
206,1	IE adult	38,8	Table and wine grapes	17,6	Stone fruit	13,6	Bovine: Meat
205,6	FR all population	136,9	Table and wine grapes	16,7	Bovine: Meat	9,8	Swine: Meat
190,3	ES child	47,2	Bovine: Meat	41,2	Swine: Meat	20,9	Lettuce
182,1	WHO regional European diet	42,1	Swine: Meat	35,2	Bovine: Meat	18,8	Lettuce
179,2	WHO cluster diet E	59,7	Table and wine grapes	23,5	Bovine: Meat	19,6	Swine: Meat
158,8	WHO Cluster diet F	38,6	Swine: Meat	29,9	Bovine: Meat	24,4	Table and wine grapes
146,1	ES adult	26,8	Lettuce	24,9	Bovine: Meat	23,8	Swine: Meat
142,9	NL general	30,9	Swine: Meat	28,6	Table and wine grapes	23,4	Bovine: Meat
136,0	FR infant	42,9	Milk and cream,	20,8	Apples	19,3	Bovine: Meat
129,2	UK Toddler	38,1	Sugar beet (root)	34,4	Milk and cream,	14,2	Apples
128,9	PT General population	92,2	Table and wine grapes	8,8	Apples	7,5	Tomatoes
128,1	UK Infant	64,5	Milk and cream,	16,8	Sugar beet (root)	13,0	Apples
126,6	WHO cluster diet D	19,9	Bovine: Meat	18,4	Table and wine grapes	9,2	Chinese cabbage
100,4	DK adult	49,0	Table and wine grapes	18,5	Bovine: Meat	8,9	Milk and cream,
94,8	DK child	21,1	Milk and cream,	19,4	Apples	13,6	Cucurbits - edible peel
89,8	SE general population 90th percentile	20,6	Milk and cream,	10,0	Chinese cabbage	8,8	Apples
86,2	LT adult	32,2	Swine: Meat	15,6	Apples	8,5	Bovine: Meat
79,3	IT adult	18,9	Lettuce	11,1	Stone fruit	9,7	Tomatoes
78,3	UK vegetarian	29,7	Table and wine grapes	7,0	Lettuce	6,3	Sugar beet (root)
77,1	UK Adult	37,8	Table and wine grapes	6,7	Sugar beet (root)	5,9	Lettuce
74,2	IT kids/toddler	14,5	Lettuce	11,9	Tomatoes	10,5	Stone fruit
51,8	PL general population	17,0	Apples	10,7	Table and wine grapes	7,4	Tomatoes
44,1	FI adult	10,8	Table and wine grapes	9,5	Milk and cream,	3,9	Lettuce

Chronic risk assessment - refined calculations

Chronic risk assessment - refined calculations						
		TMDI (range) in % of ADI minimum - maximum				
		13	91			
		No of diets exceeding ADI:	---			
Highest calculated TMDI values in % of ADI	MS Diet	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
91,4	IE adult	11,0	Tea (dried leaves and stalks,	11,0	Tea (dried leaves and stalks,	8,8	Other farm animals: Meat
91,2	DE child	42,2	Apples	8,8	Milk and milk products: Cattle	6,8	Table grapes
75,6	NL child	22,2	Apples	18,1	Milk and milk products: Cattle	4,8	Spinach
73,7	WHO Cluster diet B	9,6	Wine grapes	6,0	Watermelons	5,4	Tomatoes
47,3	FR infant	15,9	Milk and milk products: Cattle	8,8	Apples	5,8	Spinach
45,1	WHO cluster diet E	8,6	Wine grapes	3,0	Apples	2,8	Tea (dried leaves and stalks,
44,6	WHO cluster diet D	3,6	Watermelons	2,9	Milk and milk products: Cattle	2,9	Tea (dried leaves and stalks,
40,8	WHO regional European diet	3,3	Lettuce	3,2	Tea (dried leaves and stalks,	3,2	Tea (dried leaves and stalks,
39,2	FR toddler	9,2	Spinach	9,2	Apples	4,1	Courgettes
39,1	FR all population	21,3	Wine grapes	1,7	Melons	1,7	Apples
36,1	DK child	13,6	Cucumbers	8,1	Apples	2,4	Pears
35,6	ES child	7,7	Milk and milk products: Cattle	4,0	Apples	3,6	Lettuce
33,3	UK Toddler	9,9	Sugar beet (root)	6,0	Apples	2,3	Tea (dried leaves and stalks,
31,7	SE general population 90th percentile	7,6	Milk and milk products: Cattle	3,7	Apples	2,6	Cucumbers
29,0	UK Infant	5,5	Apples	4,8	Tea (dried leaves and stalks,	4,8	Tea (dried leaves and stalks,
28,7	WHO Cluster diet F	3,2	Wine grapes	2,6	Lettuce	2,4	Milk and milk products: Cattle
28,2	ES adult	4,6	Lettuce	3,0	Milk and milk products: Cattle	2,7	Apples
28,1	PT General population	13,3	Wine grapes	3,7	Apples	1,8	Potatoes
26,7	NL general	4,1	Apples	4,0	Milk and milk products: Cattle	3,4	Wine grapes
26,1	UK vegetarian	4,3	Wine grapes	4,1	Tea (dried leaves and stalks,	4,1	Tea (dried leaves and stalks,
25,1	IT adult	3,3	Lettuce	2,8	Apples	2,0	Tomatoes
24,5	UK Adult	5,8	Wine grapes	4,5	Tea (dried leaves and stalks,	4,5	Tea (dried leaves and stalks,
24,0	IT kids/toddler	3,1	Apples	2,5	Lettuce	2,5	Tomatoes
20,0	DK adult	7,4	Wine grapes	2,7	Apples	2,2	Cucumbers
18,8	LT adult	6,5	Apples	3,3	Cucumbers	2,4	Milk and milk products: Cattle
16,5	PL general population	7,2	Apples	1,7	Table grapes	1,5	Tomatoes
12,6	FI adult	2,2	Cucumbers	1,6	Wine grapes	1,4	Apples

Acute risk assessment

Acute risk assessment /children - refined calculations						Acute risk assessment / adults / general population - refined calculations					
The acute risk assessment is based on the ARfD.											
For each commodity the calculation is based on the highest reported MS consumption per kg bw and the corresponding unit weight from the MS with the critical consumption. If no data on the unit weight was available from that MS an average European unit weight was used for the IESTI calculation.											
In the **IESTI 1** calculation, the variability factors were 10, 7 or 5 (according to JMPR manual 2002), for lettuce a variability factor of 5 was used. In the **IESTI 2** calculations, the variability factors of 10 and 7 were replaced by 5. For lettuce the calculation was performed with a variabilty factor of 3.											
Threshold MRL is the calculated residue level which would leads to an exposure equivalent to 100 % of the ARfD.											
No of commodities for which ARfD/ADI is exceeded (IESTI 1):		**---**	**No of commodities for which ARfD/ADI is exceeded (IESTI 2):**		**---**	**No of commodities for which ARfD/ADI is exceeded (IESTI 1):**		**---**	**No of commodities for which ARfD/ADI is exceeded (IESTI 2):**		**---**
IESTI 1	***)**	****)**	**IESTI 2**	***)**	****)**	**IESTI 1**	***)**	****)**	**IESTI 2**	***)**	****)**
Highest % of ARfD/ADI	Commodities	pTMRL/ threshold MRL (mg/kg)	Highest % of ARfD/ADI	Commodities	pTMRL/ threshold MRL (mg/kg)	Highest % of ARfD/ADI	Commodities	pTMRL/ threshold MRL (mg/kg)	Highest % of ARfD/ADI	Commodities	pTMRL/ threshold MRL (mg/kg)
33,7	Lamb's lettuce	15 / -	33,7	Lamb's lettuce	15 / -	22,6	Lamb's lettuce	15 / -	22,6	Lamb's lettuce	15 / -
14,0	Tomatoes	0,3 / -	11,1	Cauliflower	0,21 / -	6,0	Aubergines (egg	0,3 / -	6,0	Aubergines (egg plants)	0,3 / -
11,1	Cauliflower	0,21 / -	10,1	Tomatoes	0,3 / -	5,3	Cauliflower	0,21 / -	5,3	Cauliflower	0,21 / -
9,8	Broccoli	0,21 / -	7,0	Broccoli	0,21 / -	3,7	Tomatoes	0,3 / -	3,6	Broccoli	0,21 / -
6,0	Aubergines (egg	0,3 / -	6,0	Aubergines (egg	0,3 / -	3,6	Broccoli	0,21 / -	2,9	Tomatoes	0,3 / -

APPENDIX C – DETAILED EVALUATION OF THE ADDITIONAL STUDIES RELIED UPON

C.1. Methods of analysis

C.1.1. Methods for enforcement of residues in food and feed of plant origin

C.1.1.1. Analytical method 1

C.1.1.1.1. Method validation

No analytical methods for enforcement are submitted (see RAR of indoxacarb).

C.1.2. Methods for enforcement of residues in food and feed of animal origin

No analytical methods for enforcement are submitted (see RAR of indoxacarb).

C.1.3. Methods for risk assessment of residues in food and feed of plant origin (data generation for supervised residue trials)

Reference:	Guinivan, R.A., Daussin, S. 2008, DuPont-19901 Recovery of DPX-MP062 and five metabolites from hen-derived matrices (whole eggs, muscle, fat and liver) after frozen storage
Test facility:	E. I. du Pont Nemours ~~and~~ Company, Wilmington, Delaware U.S.A.
GLP:	Yes
Acceptability of the method:	Yes

Principle of the method: Stability samples were analyzed for DPX-MP062 and its metabolites (IN-KB687, IN-KG433, IN-KT319, IN-JU873 and IN-JT333) using procedure described in the analytical method DFGS19 reported in the monitoring part and validated (DuPont 39006). The original method had an LOQ of 0.01 mg/kg.

Method was modified and modification involved changing the aliquots removed and the final volume of the extracts analyzed. The extraction procedure was no modified.

Principal of determination:

Samples of hen whole eggs, liver, fat, and muscle were fortified with DPX-MP062 and its 5 metabolites (IN KB687, IN-KG433, IN-KT319, IN-JU873 and IN-JT333), each at a level of 0.20 mg/kg were stored at approximately 20°C and then analyzed at 5 intervals over 16 months. Residues were extracted from whole eggs using acidified ethyl acetate, and from liver, muscle and fat using acidified acetonitrile. Following partitioning and purification steps, the residues were quantified by LC/MS/MS analysis (positive ion mode for DPX-MP062, IN-KG433, IN-KT319, IN-JU873 and IN-JT333 and negative for IN KB687).

ESI-LC-MS/MS conditions:

Analytes	Ions Monitored
DPX-MP062	528.0→ 293.2 ± 0.5 AMU **528.0→ 203.1 ± 0.5 AMU**
IN-KB687 (Negative Ion mode)	**234.0→ 202.0 ± 0.5 AMU** 234.0→ 85.3 ± 0.5 AMU
IN-KG433	516.0→ 221.1 ± 0.5 AMU **516.0→ 281.2 ± 0.5 AMU**
IN-KT319	516.0→ 221.1 ± 0.5 AMU **516.0→ 281.2 ± 0.5 AMU**
IN-JU873	**458.0→ 149.2 ± 0.5 AMU** 458.0→ 208.0 ± 0.5 AMU 458.0→ 255.2 ± 0.5 AMU
IN-JT333	470.0→ 150.2 ± 0.5 AMU **470.0→ 267.2 ± 0.5 AMU**

Results and discussion:

Specificity: Representative chromatograms for matrix standard, control and fortified samples of eggs and tissues were presented and show no interference >30% of the LOQ at the retention time of DPX-MP062 and metabolites.

Linearity: representative calibration (four-point) curves were generated each time an analysis set was run. Standard concentration ranged from 0.4 -2.0 ng/ml for indoxacarb and its metabolites, R2 were > 0.990.

Recovery

Prior analysis of the stability samples, a validation of the method was conducted in which egg, muscle, liver and fat. For each matrix, a control, three fortifications at 0.1 and three fortifications at 0.2 mg/kg were analysed.

		Percent Recovery (% Relative Standard Deviation)					
Analyte	Fortification Level (mg/kg)	IN-KB687	IN-KG433	IN-KT319	IN-JU873	DPX-MP062	IN-JT333
Whole Eggs	0.10	84 (9.1)	87 (6.9)	88 (9.0)	86 (17)	84 (6.9)	85 (2.4)
	0.20	89 (1.3)	88 (8.0)	85 (4.1)	84 (10)	75 (11)	84 (19)
Liver	0.10	89 (2.2)	94 (4.9)	97 (1.6)	92 (12)	94 (13)	78 (2.7)
	0.20	91(2.9)	97 (5.5)	96 (0.6)	98 (14)	95 (3.2)	91 (9.5)
Fat	0.10	91 (2.2)	94 (6.9)	97 (5.7)	91 (12)	108 (23)	94 (16)
	0.20	90 (1.3)	93 (3.4)	97 (3.2)	88 (9.5)	91 (16)	87 (10)
Muscle	0.10	88 (9.1)	94 (13)	92 (9.2)	100 (13)	91 (2.2)	93 (4.8)
	0.20	91 (2.8)	92 (6.0)	93 (3.9)	91 (12)	78 (4.9)	91 (5.4)

Limit of quantification: 0.2mg/kg in eggs, liver, fat and muscle.

Method used in the residue trials is based on analytical method DFG S19 reported in the monitoring part (DuPont-39006) and validated at LOQ of 0.01 mg/kg.Thus method was considered validated at a limit of quantification of 0.2mg/kg in eggs, liver, fat and muscle.

procedure described in the

Reference:	Determination of magnitude and decline of residues of DPX-KN128 (indoxacarb) along with IN-KN127in lettuce following applications of DPX-KN128 30 WG – Europe – initiated 2012, Spence CM, 2012, DuPont 35819
Test facility:	Charles River Laboratories, Tranent, Edinburgh, EH33 2NE, UK
GLP:	Yes
Acceptability of the method:	Acceptable

Principal of determination:

DPX-MP062 (DPX-KN128 (indoxacarb, S isomer) and IN-KN127 (R-isomer)) is extracted with acetone/water (module E1), partitioned with ethyl acetate/cyclohexane (1:1, v/v) and sodium chloride, purified with GPC and determined with LC-MS/MS (ESI positive mode, transition 528>218 (quantification), 528>249 m/z, 528>203m/z). DPX-KN128 and DPX-KN127 appear together as a single peak on chromatograms. MS spectra were not provided but are available in others studies.

Results

Specificity

Chromatograms have been provided for matrix matched calibration standards (leaf lettuce, lamb lettuce), control and fortified samples at LOQ (apple, peach, kiwi peel, kiwi pulp, leaf lettuce, lamb lettuce). No interference has been observed at the retention time of the analyte. Data have been provided for two transitions. Specificity is acceptable.

Linearity

Linearity has been performed with 6 or 7 calibration standards (in solvent and matrix matched (apple, peach, kiwi peel, kiwi pulp, leaf lettuce, lamb lettuce)) ranging from 0.25 to 50ng/mL. Regressions were linear with R^2>0.99. Data have been provided for the quantification transition (528>218m/z). Linearity is acceptable.

Accuracy/precision

Recovery results from the independent laboratory validation of indoxacarb residue using the analytical method.						
Matrix	*Fortification level (mg/kg)*	*No of samples per fortification level*	*recoveries obtained (%)*	*Mean recovery*	*RSD (%)*	*Comments*

Recovery results from the independent laboratory validation of indoxacarb residue using the analytical method.						
Matrix	*Fortification level (mg/kg)*	*No of samples per fortification level*	*recoveries obtained (%)*	*Mean recovery*	*RSD (%)*	*Comments*
Report 35819 Leaf lettuce Transition 528>218 m/z	0.01	14	94-94-99-75-91-99-96-75-79-75-75-70-71-80	84	13	acceptable
	0.1	14	107-106-98-92-106-104-107-66-70-71-99-94-82-86	92	16	
	1.0	5	89-101-102-107-89			
Report 35819 Lamb lettuce Transition 528>218 m/z	0.01	6	72-70-70-91-72-91	78	14	acceptable
	0.1	6	74-73-73-102-104-84	85	18	
	1.0	3	97-106-99	101	5	

LOQ

The limit of quantification is 0.01mg/kg for indoxacarb (as sum of isomers) in leaf lettuce and lamb lettuce.

Analytical method used in the residue trials is validated with LOQ of 0.01mg/kg for indoxacarb (sum of isomers) in leaf lettuce and lamb lettuce.

Reference:	Determination of magnitude of residues of DPX-KN128 (indoxacarb) together with IN-KN127 in appleq following applications of DPX-MP062-30WG and DPX-KN128-30WG (Europe – 2011), Lakaschus, S, Winkler, K, 2012, DuPont 33516
Test facility:	Eurofins Agroscience Services CHem GmbH, Grossmoorbogen 25, D-21079 Hamburg, Germany
GLP:	Yes
Acceptability of the method	acceptable

Reference:	Determination of magnitude of residues of DPX-KN128 (indoxacarb) together with IN-KN127 in lettuce following applications of DPX-MP062 30WG and DPX-KN128 30 WG – Europe, 2011, Lakaschus, S, Amann, S, 2012, report DuPont 33518
Test facility:	Eurofins Agroscience Services CHem GmbH, Grossmoorbogen 25, D-21079 Hamburg, Germany
GLP:	Yes
Acceptability of the method	Acceptable

Reference:	Determination of magnitude of residues of DPX-KN128 (indoxacarb) together with IN-KN127 in broccoli following applications of DPX-MP062 30WG and DPX-KN128 30WG - Europe, 2011 DuPont 33517
Test facility:	Eurofins Agroscience Services CHem GmbH, Grossmoorbogen 25, D-21079 Hamburg, Germany
GLP:	Yes
Acceptability of the method	Acceptable

Reference:	Determination of decline of residues of DPX-KN128 (indoxacarb) together with IN-KN127 in green beans (fresh legume vegetables) following four applications of STEWARD (300g/kg DPX-MP062) under protected conditions – Spain – season 2008, Promo Vert S.A, report 08 I CL DP P/A, Fernandez, E, 2010
Test facility (analytical part):	Eurofins Agroscience Services CHem GmbH, Grossmoorbogen 25, D-21079 Hamburg, Germany
GLP:	Yes
Acceptability of the method	Acceptable

Principle:

DPX-MP062 (DPX-KN128 (indoxacarb, S isomer) and IN-KN127 (R-isomer)) is extracted with acetone/water (module E1), partitioned with ethyl acetate/cyclohexane (1:1, v/v) and sodium chloride, purified with GPC) and determined with LC-MS/MS (ESI positive mode, transition 528>218 and 528>203 m/z). DPX-KN128 and DPX-KN127 appear together as a single peak on chromatograms. MS spectra were not provided but are available in others studies.

Specificity

Chromatograms have been provided for matrix matched calibration standards (apple, lettuce), control and fortified sample at 0.01mg/kg (apple, lettuce, green bean plant, green bean pods). Data have been provided for the two transitions. Interferences were below 30% of the LOQ. Specificity is acceptable.

Linearity

Linearity has been performed with 6 matrix matched (apple, lettuce, bean) calibration standards ranging from 0.25 to 50.0 ng/mL. Data have been provided for the transition 528>218 m/z. Regression was linear with R^2>0.99.

Accuracy/precision

Recovery results from the independent laboratory validation of indoxacarb residue using the analytical method.						
Matrix	*Fortification level (mg/kg)*	*No of samples per fortification level*	*recoveries obtained (%)*	*Mean recovery*	*RSD (%)*	*Comments*
Report 33516 Apple Transition 528>218 m/z	0.01 0.1 0.2	2 2 1	103-100-87 98-103 91	96.67 100.5 -	- -	Acceptable by compilation
Report 33518 Lettuce Transition 528>218m/z	0.01 0.1 0.64	3 2 1	70-74-92 75-67 91	78.67 71 -	- - -	Acceptable by compilation
Report 08 I CL DP P/A Green beans (plant, pods)	0.01 0.1 1.0 5.0	4 3 1 1	105-97-97-94 99-86-68 107 96	98.25 84.33 -	-	Acceptable by compilation
Report 33517 flower heads and stems	0.01 0.1 0.2	2 2 1	88-92-75 84-83 74	85 83.5	-	Acceptable by compilation

Data can be compiled to get sufficient samples at each fortified level.

LOQ

The limit of quantification is 0.01mg/kg for indoxacarb (as sum of isomers) in apple, lettuce, green bean plant and pods.

Analytical methods used in the residue trials have been validated with LOQ=0.01mg/kg in apple, lettuce, green bean plant and pods. Trials using this method can be taken into account.

Reference:	Aitken, A. 2014 Determination of the magnitude and decline of residues of DPX-KN128 (indoxacarb) along with IN-KN127 in broccoli following applications of DPX-MP062 30WG - Europe - 2012 Charles River Laboratories (UK) DuPont-35437
Test facility:	CETA, 296, 533 54 Rybitvi, Czech Republic

Reference:	Aitken, A. 2014. Determination of the decline of residues of DPX-KN128 (indixacarb) along with IN-KN127 in head cabbage following applications of DPX-MP062 30WG - northern Europe – 2012 (UK) DuPont-39889
Test facility:	CETA, 296, 533 54 Rybitvi, Czech Republic

Reference:	Aitken, A. 2014. Determination of the magnitude and decline of residues of DPX-KN128 (indoxacarb) along with IN-K127 in apples and apple processed fractions following applications of DPX-MP062 30WG - Europe - 2012 DuPont-35169
Test facility:	CETA, 296, 533 54 Rybitvi, Czech Republic
GLP:	yes

Reference:	Aitken, A. 2014 Determination of the decline of residues of DPX-KN128 (indoxacarb) along with IN-K127 in tomato and tomato processed fractions following applications of DPX-MP062 30WG - southern Europe - 2012 DuPont-35170 Charles River Laboratories (UK)
Test facility:	CETA, 296, 533 54 Rybitvi, Czech Republic

GLP: Yes

Principal of the method:

Extraction with acetone and detection/quantification was adapted to LC-MS/MS (non-chiral column, ESI+) from the gas chromatographic analysis. Mass transition monitored: MRM 528 ➔ 218 (quantification) and 528 ➔ 203. MS spectra have been provided in others studies.

Results and discussion:

Specificity:

Representative chromatograms for matrix standard, control and fortified samples were presented and show no interference (> 30% of the LOQ) at the retention time of indoxacarb.

Linearity:

Linearity has been performed with 7 matrix matched calibration standards (broccoli, head cabbage, apple, unprocessed whole apple, peeled apple, canned apple, apple juice, apple jam, apple sauce, wet pomace extract, tomato, tomato paste, tomato juice, tomato catsup, tomato puree) ranging from 0.2 to 20 ng/mL. Regressions were linear with R2>0.99. Data have been provided for the quantification transition. Linearity is acceptable.

Accuracy

Accuracy was performed with samples fortified at three levels. Mean recoveries were in acceptable range. Data have been provided for the quantification transition. Accuracy is acceptable.

Report	**matrix**	**Fortified levels**	**Recoveries (%)**	**Mean recovery (%)**	**RSD (%)**
DuPont-35437	broccoli	0.01 0.1	83/78/108/88/95/91/78/81 83/81/86/87/84/86/89/81	88 84	12 3.4
DuPont-39889	Head cabbage	0.01 0.1 1.0	75/76/74/77/85/100 82/98/73/70/86/102 89/101	81 85	12.3 15.2
DuPont-35169	apple	0.01 0.1	86/71/80/104/100/110 109/71/85/101/116/117	92 100	17 18
	Unprocessed whole apple	0.01 0.1	115/78/84/89/83 94/73/102/105/77	90 90	16 16
	Peeled apple	0.01 0.1	95/98/81 91/94/94	91 93	10 2
	Canned apple with skin	0.01 0.1	82/96/91/98 99/77/97/95	92 92	8 11
	Apple juice	0.01 0.1	85/79/70/83 84/79/83/93	79 85	8 7

	Apple jam	0.01 0.1	77/91/92/98 111/101/109/100	90 105	10 5
	Apple sauce	0.01 0.1	86/85/92/79 92/96/94/97	86 95	6 2
	Wet pomace	0.01 0.1	81/88/101/72 85/99/90/85	86 90	14 7
DuPont-35170	tomato	0.01 0.1 1.0	82/83/91/86/94/90 73/71/83/69/74/79 84/93/95	88 75 91	5.4 7.0 6.5
	Tomato puree	0.01 0.1	99/109/109/105 107/104/109/107	106 107	4.5 1.9
	Tomato paste	0.01 0.1	105/97/111/105 100/105/107/100	105 103	5.5 3.5
	Tomato catsup	0.01 0.1	107/71/98/107 99/100/106/99	96 101	17.8 3.3
	Unprocessed whole tomato	0.01 0.1	81/75/89/81/79/73 87/83/70/87/74/79	80 80	7.0 8.7
	Tomato juice	0.01 0.1	104/99/102/104 100/99/96/100	102 99	2.3 1.9
	Canned tomato	0.01 0.1	81/87/88/81 79/88/95/79	84 85	4.5 9.1

Precision

According to the previous table, RSD were in acceptable limits. Precision is acceptable.

LOQ

The limit of quantification is 0.01mg/kg in all samples.

Analytical method for the determination of indoxacarb residue in broccoli, head cabbage, apple, unprocessed apple, peeled and apple has been validated with LOQ of 0.01mg/kg. Trials using this method can be taken into account.

For unprocessed tomato and canned tomato, no specificity data have been provided and the fortification level number is insufficient to establish a LOQ.

Reference: Aitken, A. 2014 Determination of the decline of residues of DPX-KN128 (indoxacarb) along with DPX-KN127 in maize forage following applications of DPX-MP062 30WG - Southern Europe – 2012 DuPont-35172

Test facility: Alan Aitken HND Charles river Tranent Edinburgh UK

GLP: Yes

Principal of the method:

Extraction with acetone and detection/quantification was adapted to LC/MS/MS from the gas chromatographic analysis specified in the cited methods. Mass transition monitored: MRM 528 ➔ 218 (quantification) and 528 ➔ 203.

Results and discussion:

Specificity: Representative chromatograms for matrix standard, control and fortified samples of maize forage were presented and show no interference (> 30% of the LOQ) at the retention time of indoxacarb.

Linearity: has been performed with 7 matrix fortified solutions in the range 0.20 to 25 ng/ml. Regressions were linear and R^2 = 0.991.

Recovery: results in maize forage:

Fortification level mg/kg	Recovery %	N° of analysis	Mean %	RSD %
0.01	94, 87, 99, 102, 97, 92, 91, 89, 85	9	93.6	6.1
0.1	109, 98, 91, 98, 94, 96, 100, 95, 92	9	97	5.5
1.0	91, 90, 98, 93, 88	5	92	4.1

Method was validated with a determined Limit of Quantification (LOQ) in Forage maize was 0.010 mg/kg.

Reference: Hornshuh, M.J., Enriquez, M.A., Gakeme, B.2002. Decline of residues of DPX-KN128 (indoxacarb) together with IN-KN127 in cauliflower and broccoli (flowering brassica vegetables) following applications of DPX-MP062 30WG - Europe, season 2001
Battelle Europe-Centre de Recherche de Geneve
DuPont-6061

Test facility: Battelle, Geneva Research Centres Agrochemical Product

Development Geneva Switzerland

GLP: Yes

Reference: Hornshuh, M.J., Enriquez, M.A., Gakeme, B.2002. Decline of residues of DPX-KN128 (indoxacarb) together with IN-KN127 in cabbage (head brassica vegetables) following applications of DPX-MP062 30WG - northern Europe, season 2001
Battelle Europe-Centre de Recherche de Geneve DuPont-6062

Test facility: Battelle, Geneva Research Centres Agrochemical Product Development Geneva Switzerland

GLP: Yes

Principal of the method:
DPX-KN128/IN-KN127 residues were extracted from 5-grams of specimen with an ethyl acetate/distilled water solution, cleaned-up by solid phase extraction and determined by gas chromatography/mass selective detector.

Specificity:
Representative chromatograms for matrix standard control were presented and show no interference (> 30% of the LOQ) at the retention time of indoxacarb.

Linearity: has been performed with 7 matrix fortified solutions in the range 0.20 to 25 ng/ml. Regressions were linear and $R^2 = 0.996$
Recovery:

Reference	Crop fraction	Fortification level mg/kg	n	Mean recovery %	RSD %
DuPont-6061	Broccoli	0.02 0.2	7 5	98 83.2	13 10
	Cauliflower	0.02 0.2	8 4	84.9 85.8	12 6
DuPont-6061	Cabbage	0.02 0.2	5 5	95.4 101.1	11 4.6

Method was validated with a determined Limit of Quantification (LOQ) in broccoli, cauliflower and cabbage was 0.010 mg/kg.

Reference: Determination of magnitude of residues of DPX-KN128 (indoxacarb) together with IN-KN127 in lettuce and broccoli/cauliflower following applications of DPX-MP062-30WG and DPX-KN128-15EC - Europe 2006

	Charles River Laboratories (UK) DuPont-19688
Test facility:	E. I. du Pont de Nemous and Company, Delaware USA

GLP: Yes

Principal of the method: Specimens were analysed for residuesq of indoxacarb following method DFG S19 module E1 (extraction with acetone/water). The detection was performed with LC-MS/MS (ESI positive mode, transition 528>218 and 528>203 m/z).

Specificity:
Representative chromatograms for matrix standard control were presented and show no interference (> 30% of the LOQ) at the retention time of indoxacarb.

Linearity:
Has been performed with 6 matrix fortified solution (lettuce, Broccoli and cauliflower) in the range 0.40 to 50 ng/ml. Regressions were linear and $R^2 = 0.996$
Recovery:
Recovery data:

Matrix	Fortification level mg/kg	n	Mean recovery %
Broccoli	0.01 0.1 0.2	13 13 13	98 93 119
Cauliflower	0.01	10	95

Method was validated with a determined Limit of Quantification (LOQ) in broccoli and cauliflower was 0.010 mg/kg.

C.2. Mammalian toxicology

No new data submitted.

C.3. Residue data

C.3.1. Nature and magnitude of residues in primary crops

C.3.1.1. Nature of residues

See EFSA Journal 2011;9(8):2343.

C.3.1.2. Magnitude of residues

C.3.1.2.1. Broccoli and cauliflower

Report:	Lakaschus, S., Walker, K., 2012
Title:	Determination of magnitude of residues of DPX-KN128 (indoxacarb) together with IN-KN127 in broccoli following applications of DPX-MP062 30WG and DPX-KN128 30WG - Europe, 2011
Document No.:	DuPont-33517
Guidelines:	EC Commission Directive 91/414/EEC, SANCO/825/00 rev.8.1 (16/11/2010)
GLP:	Yes

Acceptability	Deviations
Yes	None Study dates June 2011 to October 2012

Table 3.1.1-1: Summary of global information on study 3

Comparative trials (between formulations, with and adjuvant/safener/synergist)	Yes DPX-MP062 30 WG and DPX-KN128 30 WG
Number of applications	3
Dose (g as/ha)	37.5
Mode of application	Spraying
PHI (days) and/or growth stage (BBCH)	1 d
Analytical method (Code +Type)	AMR 4271-96 (LC-MS/MS)
LoQ (mg/kg)	0.01

Table 3.1.1-2: Summary of the study 3 trials

N° Trial		1	2	3
North/South/Indoor		N	S	S
Decline (D)/Harvest (H) trial?		H	H	H
Formulation		WG	WG	WG
Equivalence between formulations		Y	Y	Y
Accordance with intended GAP		Y	Y	Y
Correct sampling		Y	Y	Y
Samples frozen within 24h		Y	Y	Y
Storage period (in days)	Sample	114	65	118
	Extract	1	1	1
Storage T° <-18°C		Y	Y	Y
Validated analytical method		Y	Y	Y
Negative controls		Y	Y	Y
Considered trial [1]		Y	Y	Y
Remarks				

[1] Trials conducted according to a more critical GAP (not in the range of 25% of the application dose rate)

Application of proportionality:

Actual application rate	Measured residue levels (mg/kg)	Intended application rate	Calculated residue levels (mg/kg)
37.7 g as/ha	0.18	25.5 g as/ha	0.12
37.6 g as/ha	0.18	25.5 g as/ha	0.12
38.4 g as/ha	0.11	25.5 g as/ha	0.07

Table 3.1.1-3: Summary of data from residue trials for study 1

GLP and trial details	Crop	Country	Application rate (g a.s./ha)	Crop growth stage	Commodity or matrix	PHI (days)	Residues found (mg/kg)	Recovery data
DuPont-33517; study to GLP, study carried out in 2011	Broccoli	North France Innenheim, Alsace	37.7 (126% of GAP) 37.4 (125% of GAP) 40.6 (135% of GAP) (DPX-MP062 30WG)	BBCH 41 BBCH 43 BBCH 48–49	Flower Heads and Stems	1	0.17 (DPX-KN128 plus IN-KN127)	Broccoli flower heads and stems mean recovery = 83%, RSD = 8.6% (n = 6 in 0.010 to 0.20 ppm fortification range)
			37.7 (126% of GAP) 37.3 (124% of GAP) 38.1 (127% of GAP) (DPX-KN128 30WG)	BBCH 41 BBCH 43 BBCH 48–49		1	0.18 (DPX-KN128)	
DuPont-33517; study to GLP, study carried out in 2011	Broccoli	South France Argelès-sur-Mer, Pyrénées - Orientales	38 (127% of GAP) 38 (127% of GAP) 37.1 (124% of GAP) (DPX-MP062 30WG)	BBCH 43 BBCH 43–45 BBCH 45–46	Flower Heads and Stems	1	0.18 (DPX-KN128 plus IN-KN127)	Broccoli flower heads and stems mean recovery = 83%, RSD = 8.6% (n = 6 in 0.010 to 0.20 ppm fortification range)
			38 (127% of GAP) 36.8 (123% of GAP) 37.9 (126% of GAP) (DPX-KN128 30WG)	BBCH 43 BBCH 43–45 BBCH 45–46		1	0.11 (DPX-KN128)	
DuPont-33517; study to GLP, study carried out in 2011	Broccoli	South France Merville, Midi Pyrénées	37.94 (126% of GAP) 37.27 (124% of GAP) 39.99 (133% of GAP) (DPX-MP062 30WG)	BBCH 19 BBCH 19–41 BBCH 19–49	Flower Heads and Stems	1	0.11 (DPX-KN128 plus IN-KN127)	Broccoli flower heads and stems mean recovery = 83%, RSD = 8.6% (n = 6 in 0.010 to 0.20 ppm fortification range)
			38.25 (128% of GAP) 34.77 (116% of GAP) 37.56 (125% of GAP) (DPX-KN128 30WG)	BBCH 19 BBCH 19–41 BBCH 19–49		1	0.10 (DPX-KN128)	

Report:	Aitken, A., 2014
Title:	Determination of the magnitude and decline of residues of DPX-KN128 (indoxacarb) along with IN-KN127 in broccoli following applications of DPX-MP062 30WG - Europe - 2012
Document No.:	DuPont-35437
Guidelines:	OECD 509, EC Commission Directive 91/414/EEC, SANCO/825/00 rev.8.1 (16/11/2010)
GLP:	Yes

Acceptability	Deviations
Yes	No deviations were thought to have impacted the outcome of the study. Study dates June 2012 to February 2014

Table 3.1.1-4: Summary of global information on study 1

Comparative trials (between formulations, with and adjuvant/safener/synergist)	No DPX-MP062
Number of applications	3
Dose (g as/ha)	25.5
Mode of application	Spraying
PHI (days) and/or growth stage (BBCH)	0, 1, 7, 14, 21 (decline trials), 1 (harvest trials)
Analytical method (Code +Type)	AMR 4271-96 (LC-MS/MS)
LoQ (mg/kg)	0.01

Table 3.1.1-5: Summary of the study 1 trials

N° Trial		1	2	3	4	5	6	7
North/South/Indoor		N	N	N	N	S	S	S
Decline (D)/Harvest (H) trial?		H	D	D	H	H	D	D
Formulation		WG	WG	WG	WG	WG	WG	WG
Equivalence between formulations		Y	Y	Y	Y	Y	Y	Y
Accordance with intended GAP		Y (1)	Y (1)	Y (1)	Y (1)	Y (1)	Y (1)	Y (1)
Correct sampling		Y	Y	Y	Y	Y	Y	Y
Samples frozen within 24h		Y	Y	Y	Y	Y	Y	Y
Storage period (in days)	Sample	299	242	274	246	225	240	250
	Extract	<1	1	<1	<1	<1	<1	<1
Storage T° <-18°C		Y	Y	Y	Y	Y	Y	Y
Validated analytical method		Y	Y	Y	Y	Y	Y	Y
Negative controls		Y	Y	Y	Y	Y	Y	N (2)
Considered trial		Y	Y	Y	Y	Y	Y	Y
Remarks								

(1) Application rate lower than the intended one (25.5 instead of 30 g as/ha) but within the -25% tolerance
(2) Control sample at 1 day PHI : 0.007 mg/kg; however, this level is considered as negligible as <LOQ

Table 3.1.1-6: Summary of data from residue trials for study 1

GLP and trial details	Crop	Country	Application rate (g a.s./ha)	Crop growth stage	Commodity or matrix	PHI (days)	Residues found (mg/kg)	Recovery data
DuPont-35437; study to GLP, study carried out in 2012	Field Broccoli	UK North Kelso, Borders Scotland	25.62 26.84 26.54	BBCH 43 BBCH 45 BBCH 49	Head and stem	1	**0.023**	Broccoli head and stem mean recovery = 86%, RSD = 8.6% (n = 16 in 0.01 to 0.10 ppm fortification range)
DuPont-35437; study to GLP, study carried out in 2012	Field Broccoli	UK South Holbeach St. Marks, Lincolnshire	24.40 25.93 24.40	BBCH 47–48 BBCH 47–48 BBCH 48	Head and stem	-0	0.040	
						+0	0.10	
						1	**0.089**	
						7	0.069	
						14	0.055	
						21	0.050	
DuPont-35437; study to GLP, study carried out in 2012	Field Broccoli	North France La Gorgue, Nord-Pas de Calais	25.32 24.40 25.01	BBCH 42–43 BBCH 44–45 BBCH 48–49	Head and stem	-0	0.018	
						+0	0.095	
						1	**0.065**	
						7	0.015	
						14	0.008	
						21	ND	
DuPont-35437; study to GLP, study carried out in 2012	Field Broccoli	Belgium Moorslede, West Vlaanderen	26.54 26.23 25.01	BBCH 42–44 BBCH 46 BBCH 48–49	Head and stem	1	**0.061**	
DuPont-35437; study to GLP, study carried out in 2012	Field Broccoli	North Spain Zaidin, Aragon	25.32 25.01 25.01	BBCH 41 BBCH 45 BBCH 49	Head and stem	1	**0.026**	
DuPont-35437; study to GLP, study carried out in 2012	Field Broccoli	North Spain Lleida, Catalunya	25.62 26.23 25.62	BBCH 41 BBCH 45 BBCH 49	Head and stem	-0	0.010	Broccoli head and stem mean recovery = 86%, RSD = 8.6% (n = 16 in 0.01 to 0.10 ppm fortification
						+0	0.041	
						1	**0.044**	
						7	0.019	
						14	0.012	
						21	0.012	

DuPont-35437; study to GLP, study carried out in 2012	Field Broccoli	Italy Roncoferraro, Lombardia	25.32 25.93 25.62	BBCH 42 BBCH 47 BBCH 49	Head and stem	-0	0.023	
						+0	0.028	
						1	0.022	
						7	**0.025**	
						14	0.016	
						21	0.013	

Report:	Old, J., Hansford, R.J., Ward, L., 2007
Title:	Determination of magnitude of residues of DPX-KN128 (indoxacarb) together with IN-KN127 in lettuce and broccoli/cauliflower following applications of DPX-MP062-30WG and DPX-KN128-15EC - Europe 2006
Document No.:	DuPont-19688
Guidelines:	Directive 91/414/EEC
GLP:	Yes

Acceptability	**Deviations**
Yes	Previous field history, crop husbandry records, or meteorological data were supplied by farmer co-operators and non-GLP facilities Study dates May 2006 to August 2007

Material and method:

The field program was conducted in 2006 at one location in Italy, at two locations in France (1 NEU and 1 SEU), and at one location in England. Six foliar applications of DPX-MP062 30WG or Explicit® EC were made at side-by-side bridging trials to lettuce at a target rate of 35.5 g DPX-KN128 a.s./ha each (equivalent to 0.125 kg/ha of DPX- P062 30WG or 0.250 kg/ha of Explicit® EC) when the crops were at BBCH growth stages 15–16, 19, 45, 47, 47–49, and 49. Three foliar applications of DPX-MP062 30WG or Explicit® EC were made to broccoli/cauliflower at a target rate of 25 g DPX-KN128 a.s./ha each (equivalent to 0.083 kg/ha of DPX-MP062 30WG or 0.167 kg/ha of Explicit® EC) when the crops were at BBCH growth stages 41–43, 43-45, and 47–49.

A total of four residue trials (two in northern Europe and two in southern Europe) were conducted over one growing season (2006). A summary of these lettuce and broccoli/cauliflower studies is given below. Specimens of lettuce, cauliflower, and broccoli were collected at sampling. A minimum of 12 heads per plot weighing >1.0 kg was collected in each case. One control specimen and one treated specimen at sampling per plot, for lettuce, cauliflower, and broccoli, were submitted for analysis.

Specimens were analysed for residues following procedures described in DuPont Report No. AMR 4271-96, “Testing of DFG Method S19 for the determination of residues of KN128 along with KN127 in crops which might be treated with DPX-MP062.” The detection was performed with LC/MS/MS as described in the report DUP-0602V, “Validation of multi-residue method DFG S19 (L 00.00-34) for the determination of residues of DPX-MP062 (DPX-KN128 (Indoxacarb) and IN-KN127) in grass.”

The experimentally determined Limit of Quantification (LOQ) was 0.010 mg/kg.

Results:

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Active ingredient : Indoxacarb (isomer S)
Crop / crop group : Lettuce and flowering cabbage

Content of a.i. (g/kg or g/l) : /
Formulation (e.g. WP) : EC or WG (side by side bridging)
Commercial product (name) : EXPLICIT EC or DPX-MP062 30WG (side by side bridging)
Applicant : Dupont Solutions SAS

Indoors / outdoors : Outdoor
Other a. s. in formulation (common name and content) : /
Residues calculated as : Sum of indoxacarb and its enantiomer R.

1	2	3	4			5	6	7	8	9	10
Report-No. Location incl. Postal code and date	Commodity/ Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest	Application rate per treatment			Dates of treatments or no. of treatments and last date	Growth stage at last treatment or date	Portion analysed	Residues (mg/kg)	PHI (days)	Remarks
			kg a.s./ha	Water hl/ha	kg a.s./hl						
	(a)	(b)				(c)		(a)		(d)	(e)
Flowering brassica: DPX-MP062 30WG											
DuPont-19688; 2006, Beaumont, Clacton On Sea, Essex, North	Cauliflower	1) 2) 3)	0.025			3	BBCH 43 BBCH 43 BBCH 47–49	Leaf	**0.076**	1	Mean recovery = 101%, RSD = 12 (n = 4 in 0.01-0.20 ppm fortification range)
DuPont-19688; 2006 France Stoner, Nord Pas de Calais, North	Broccoli	1) 2) 3)	0.025			3	BBCH 41–43 BBCH 45 BBCH 49	Leaf	**0.088**	1	

Flowering brassica: Explicit EC											
DuPont-19688; 2006, Beaumont, Clacton On Sea, Essex, North	Cauliflower	1) 2) 3)	0.025			3	BBCH 43 BBCH 43 BBCH 47– 49	Leaf	0.047	1	Mean recovery = 101%, RSD = 12 (n = 4 in 0.01-0.20 ppm fortification range)
DuPont-19688; 2006 France Stoner, Nord Pas de Calais, North	Broccoli	1) 2) 3)	0.025			3	BBCH 41– 43 BBCH 45 BBCH 49	Leaf	0.046	1	

Report:	Hornshuh, M.J., Enriquez, M.A., Gakeme, B., 2002
Title:	Decline of residues of DPX-KN128 (indoxacarb) together with IN-KN127 in cauliflower and broccoli (flowering brassica vegetables) following applications of DPX-MP062 30WG - Europe, season 2001
Document No.:	DuPont-6061
Guidelines:	Directive 91/414/EEC
GLP:	Yes

Acceptability	Deviations
Yes	None Study dates March 2001 to March 2002

Table 3.1.1-7: Summary of global information on study 2

Comparative trials (between formulations, with and adjuvant/safener/synergist)	No DPX-MP062
Number of applications	3
Dose (g as/ha)	25.5
Mode of application	Spraying
PHI (days) and/or growth stage (BBCH)	0, 1, 3d
Analytical method (Code +Type)	AMR 3493-95 (GC-MS)
LoQ (mg/kg)	0.02

Table 3.1.1-8: Summary of the study 2 trials

N° Trial		1	2	3	4	5	6	7	8
North/South/Indoor		S	S	S	N	S	S	N	N
Decline (D)/Harvest (H) trial?		D	D	D	D	D	D	D	D
Formulation		WG	WG	WG	WG	WG	WG	WG	WG
Equivalence between formulations		Y	Y	Y	Y	Y	Y	Y	Y
Accordance with intended GAP		Y							
Correct sampling		Y	Y	Y	Y	Y	Y	Y	Y
Samples frozen within 24h		Y	Y	Y	Y	Y	Y	Y	Y
Storage period (in days)	Sample	Less than 9 months							
	Extract	(1)							
Storage T° <-18°C		Y	Y	Y	Y	Y	Y	Y	Y
Validated analytical method		Y	N (2)	Y	N (2)	Y	N(2)	Y	N (2)
Negative controls		Y	Y	Y	Y	Y	Y	Y	Y
Considered trial		Y	Y	Y	Y	Y	Y	Y	Y
Remarks									

(1) Recovery data from fortifications run with each set of specimen analysed was within 70-110% ; therefore no data on the stability in extracts are necessary.

(2) For report DuPont-6061 (Hornshuh, M.J., Enriquez, M.A., Gakeme, B, 2002), chromatograms of control and fortified samples (cauliflower) at LOQ are required to check specificity.

Residues expressed as DPX-KN128 plus IN-KN127 in cauliflower and broccoli from supervised trials

Targeted GAP: Three foliar applications of DPX-MP062 30WG at 25.5 g a.s./ha each to cauliflower and broccoli as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-6061; study to GLP, study carried out in 2001	**Broccoli**	**Italy Cadriano di Granarolo, Bologna**	**25.6 (100% of GAP) 25.6 (100% of GAP) 25.6 100% of GAP)**	**BBCH 48–49 BBCH 48–49 BBCH 48–49**	**broccoli**	**-1h**	**0.03, 0.04**	**Broccoli mean recovery = 86.6%, RSD = 12 (n = 12 in 0.02-0.20 ppm fortification range) Cauliflower mean recovery = 85.2%, RSD = 10 (n = 12 in 0.02-0.20 ppm fortification range)**
						+2h	**0.19, 0.16**	
						1	**0.17, 0.12** (0.15)	
						3	**0.11, 0.09**	
DuPont-6061; study to GLP, study carried out in 2001	**Cauliflowe r**	**Italy S. Giorgio di Cesena, Forlì-Cesena**	**26.4 (104% of GAP) 26.4 (104% of GAP) 26.4 (104% of GAP)**	**BBCH 48–49 BBCH 48–49 BBCH 48–49**	**cauliflower**	**-1h**	**<0.02, <0.02**	
						+2h	**0.03, 0.04**	
						1	**0.02, 0.02 (0.02)**	
						3	**0.02, 0.02**	
DuPont-6061; study to GLP, study carried out in 2001	**Broccoli**	**Italy Borgo Tressanti, Foggia**	**25.6 (100% of GAP) 25.6 (100% of GAP) 25.6 (100% of GAP)**	**BBCH 48 BBCH 48 BBCH 48**	**broccoli**	**-1h**	**<0.02, <0.02**	
						+2h	**0.04, 0.04**	
						1	**0.04, 0.04** (0.04)	
						3	**0.03, 0.03**	
DuPont-6061; study to GLP, study carried out in 2001	Cauliflower	**France Berthenay, Indre et Loire**	**26.8 (105% of GAP) 26.8 (105% of GAP) 26.8 (105% of GAP)**	**BBCH 49 BBCH 49 BBCH 49**	**cauliflower**	**-1h**	**<0.02, <0.02**	
						+2h	**0.02, 0.02**	
						1	**0.02, 0.02 (0.02)**	
						3	**0.01, 0.01**	
DuPont-6061; study to GLP, study carried out in 2001	**Broccoli**	**France Serres-Castet, Pyrénées Atlantiques**	**26.6 (104% of GAP) 26.6 (104% of GAP) 26.6 (104% of GAP)**	**BBCH 55 BBCH 55 BBCH 55**	**broccoli**	**-1h**	**<0.02, <0.02**	
						+2h	**<0.02, <0.02**	
						1	**<0.02, <0.02** (<0.02)	
						3	**<0.02, <0.02**	

Residues expressed as DPX-KN128 plus IN-KN127 in cauliflower and broccoli from supervised trials

Targeted GAP: Three foliar applications of DPX-MP062 30WG at 25.5 g a.s./ha each to cauliflower and broccoli as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-6061; study to GLP, study carried out in 2001	Cauliflower	**France St Rémy de Provence, Bouches du Rhône**	**27.5 (108% of GAP) 27.5 (108% of GAP) 27.5 (108% of GAP)**	**BBCH 49 BBCH 49 BBCH 49**	**cauliflower**	**-1h**	**<0.02, <0.02**	
						+2h	**<0.02, <0.02**	
						1	**<0.02, <0.02 (<0.02)**	
						3	**<0.02, <0.02**	
DuPont-6061; study to GLP, study carried out in 2001	**Broccoli**	**France St Pierre des Corps, Indre et Loire**	**28.6 (112% of GAP) 28.6 (112% of GAP) 28.6 (112% of GAP)**	**BBCH 49 BBCH 49 BBCH 49**	**broccoli**	**-1h**	**0.02, 0.03**	
						+2h	**0.09, 0.12**	
						1	**0.06, 0.05** (0.06)	
						3	**0.04, 0.04**	
DuPont-6061; study to GLP, study carried out in 2001	Cauliflower	**Germany Motterwitz, Sachsen**	**28.0 (110% of GAP) 28.0 (110% of GAP) 28.0 (110% of GAP)**	**BBCH 49 BBCH 49 BBCH 49**	**cauliflower**	**-1h**	**0.03, 0.03**	
						+2h	**0.08, 0.08**	
						1	**0.07, 0.08(0.08)**	
						3	**0.06, 0.07**	

C.3.1.2.2. Lettuce

Report:	Old, J., Hansford, R.J., Ward, L., 2007
Title:	Determination of magnitude of residues of DPX-KN128 (indoxacarb) together with IN-KN127 in lettuce and broccoli/cauliflower following applications of DPX-MP062-30WG and DPX-KN128-15EC - Europe 2006
Document No:	DuPont-19688
Guidelines:	Directive 91/414/EEC (1991)
GLP	Yes

Acceptability	**Deviations**
Yes	Previous field history, crop husbandry records, or meteorological data were supplied by farmer co-operators and non-GLP facilities Study dates May 2006 to August 2007

<u>Material and method:</u>

The field program was conducted in 2006 at one location in Italy and one location in France (SEU). Six foliar applications of DPX-MP062 30WG or Explicit® EC were made at side-by-side bridging trials to lettuce at a target rate of 35.5 g DPX-KN128 a.s./ha each (equivalent to 0.125 kg/ha of DPX- P062 30WG or 0.250 kg/ha of Explicit® EC) when the crops were at BBCH growth stages 15–16, 19, 45, 47, 47–49, and 49.

A total of two residue trials were conducted over one growing season (2006). A summary of these lettuce studies is given below. Specimens of lettuce were collected at sampling. A minimum of 12 heads per plot weighing >1.0 kg was collected in each case. One control specimen and one treated specimen at sampling per plot, for lettuce were submitted for analysis.

Specimens were analysed for residues following procedures described in DuPont Report No. AMR 4271-96, “Testing of DFG Method S19 for the determination of residues of KN128 along with KN127 in crops which might be treated with DPX-MP062.” The detection was performed with LC/MS/MS as described in the report DUP-0602V, “Validation of multi-residue method DFG S19 (L 00.00-34) for the determination of residues of DPX-MP062 (DPX-KN128 (Indoxacarb) and IN-KN127) in grass.”

The experimentally determined Limit of Quantification (LOQ) was 0.010 mg/kg.

Table 3.1.1-9: Summary of data from residue trials for study 1

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Content of a.i. (g/kg or g/l) :
Formulation (e.g. WP) : EC or WG (side by side bridging)
Commercial product (name) : EXPLICIT EC or DPX-MP062 30WG (side by side bridging)
Applicant :

Active ingredient : Indoxacarb (isomer S)
Crop / crop group : Lettuce

Indoors / outdoors : Outdoor
Other a. s. in formulation
(common name and content) :
Dupont Solutions SAS : Sum of indoxacarb and its enantiomer R.

Residue trial summary for Lettuce and salad plants

Trial No./ Location/ Year	*Commodity/ Variety*	*Date of 1.Sowing or planting 2.Flowering 3. Harvest*	*Application rate per treatment*			*Dates of treatment or no. of treatments and last date*	*Growth stage at last treatment or date*	*Portion analyzed*	*Residues (mg/kg)*	*PHI (days)*	*Remarks*
			g a.s./ ha	*Water (l/ha)*	*g a.s./hl*				*DPX-KN128+ KN127*		
DuPont-19688;2006, Italy, Triginto di Mediglia, Lombardia,(SEU)	Lettuce / Lollo *Opeb leaf*	1.28 May 06 2 NR 3. 12 Jul 06	36.91 37.81 37.21 38.42 37.51 38.12	496 506 497 515 504 511	0.007 0.007 0.007 0.007 0.007 0.007	08 Jun 06 14 Jun 06 21 Jun 06 28 Jun 06 05 Jul 06 11 Jul 06	BBCH 16 BBCH 19 BBCH 45 BBCH 47 BBCH 49 BBCH 49	Heads	1.7	1	**WG**

Residue trial summary for Lettuce and salad plants

Trial No./ Location/ Year	*Commodity/ Variety*	*Date of 1.Sowing or planting 2.Flowering 3. Harvest*	*Application rate per treatment*			*Dates of treatment or no. of treatments and last date*	*Growth stage at last treatment or date*	*Portion analyzed*	*Residues (mg/kg)*	*PHI (days)*	*Remarks*
			g a.s./ ha	*Water (l/ha)*	*g a.s./hl*				*DPX-KN128+ KN127*		
DuPont-19688, Triginto di Mediglia, Lombardia 20060 Italy	Lettuce / Lollo *Open leaf*	1.28 May 06 2 NR 3. 12 Jul 06	36.77 37.37 38.58 38.43 36.92 37.98	489 498 513 511 491 505	0.008 0.008 0.008 0.008 0.008 0.008	08 Jun 06 14 Jun 06 21 Jun 06 28 Jun 06 05 Jul 06 11 Jul 06	BBCH 16 BBCH 19 BBCH 45 BBCH 47 BBCH 49 BBCH 49	Heads	1.1	1	**EC**
DuPont-19688, Lucernay, Rhone Alpes, 69480 France	Lettuce/ Feuille de Chêne *Open leaf*	1. 04 Sep 06 2. NR 3. 27 Oct 06	37.81 36.00 36.91 39.02 37.21 39.33	506 480 492 520 497 523	0.007 0.008 0.008 0.008 0.007 0.008	21 Sep 2006 28 Sep 2006 05 Oct 2006 13 Oct 2006 20 Oct 2006 26 Oct 2006	BBCH 15 BBCH 19 BBCH 45 BBCH 47 BBCH 47-49 BBCH 49	Heads	<u>1.4</u>	<u>1</u>	**WG**
DuPont-19688, Lucernay, Rhone Alpes, 69480 France	Lettuce/ Feuille de Chêne *Open leaf*	1. 04 Sep 06 2. NR 3. 27 Oct 06	38.13 38.73 36.92 35.86 36.31 35.86	509 516 493 479 485 478	0.007 0.008 0.007 0.007 0.007 0.008	21 Sep 2006 28 Sep 2006 05 Oct 2006 13 Oct 2006 20 Oct 2006 26 Oct 2006	BBCH 15 BBCH 19 BBCH 45 BBCH 47 BBCH 47-49 BBCH 49	Heads	0.43	1	**EC**

(a) According to BAYER codes
(b)Only if relevant

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, type or equipment used must be indicated

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4

(f) Minimum number of days after last application (Label pre-harvest interval, PHI, underline): DBLA = days before last application, DALA = days after last application

(g) Remarks may include: climatic conditions; reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

Residues calculated as DPX-KN128

Report:	Lakaschus, S., Amann, S.; 2012
Title:	Determination of Magnitude of Residues of DPX-KN128 (Indoxacarb) Together with IN-KN127 in Lettuce Following Applications of DPX-MP062 30WG and DPX-KN128 30WG - Europe, 2011
Document No:	DuPont-33518 Eurofins S11-00926
Guidelines:	European Communities Guidelines for the Generation of Data Concerning Residues, as Provided in Annex II, Part A, Section 6 and Annex III, Part A, Section 8 of EC Commission Directive 91/414/EEC. SANCO/825/00 rev.8.1 (16/11/2010) Guidance Document on Pesticide Residue Analytical Methods
GLP	Yes

Acceptability	Deviations
Yes	None

Table 3.1.1-10: Summary of global information on study 2

Comparative trials (between formulations, with and adjuvant/safener/synergist)	1/ DPX-MP062 30WG: Indoxacarb (DPX-KN128) 30g a.s./100g 2/ DPX-KN128 30WG: Indoxacarb (DPX-KN128) 30g a.s./100g
Number of applications	6 (RTI:7 days)
Dose (g as/ha)	1/ DPX-MP062 : 37,5 g a.s./ha 2/ DPX-KN128 : 37,5 g a.s./ha
Mode of application	Foliar Treatment
PHI (days) and/or growth stage (BBCH)	PHI : 1 day
Analytical method (Code +Type)	- Testing of DFG Method S19 for the g etermination of residue of KN128 along with KN127 in crops which might be treated with DPX-MP062. Dupont Report AMR 4271-96, 1997 - Confirmation of the Stability of DPX-KN128 (Indoxacarb) and IN-KN127 on Crops for which European Maximum Residue Limits are Proposed - Independent Laboratory Validation of the Analytical Residue Method AMR 4271-96 for the Determination of Residues of DPX-KN128 and IN-KN127 in Plant Material which might be Treated with DPX-MP062.
LoQ (mg/kg)	KN128 (indoxacarbe) 0,01 mg/kg (LOD: 0,003 mg/kg

Table 3.1.1-11: Summary of the study 2 trials

N° Trial		DuPont-33518 / 1	DuPont-33518 / 2	DuPont-33518 / 3	DuPont-33518 / 4
North/South/Indoor		N	S	S	S
Decline (D)/Harvest (H) trial?		H	H	H	H
Formulation		WG	WG	WG	WG
Equivalence between formulations		Y	Y	Y	Y
Accordance with intended GAP		Y(1)	Y	Y	Y
Correct sampling		Y	Y	Y	Y
Samples frozen within 24h		Y	Y	Y	Y
Storage period (in days)	Sample	< 8 months	< 7 months	< 7 months	< 4 months
	Extract	1-9 days	1-3 days	1-9 days	3 days
Storage T° <-18°C		Y	Y	Y	Y
Validated analytical method		Y	Y	Y	Y
Negative controls		Y	Y	Y	Y
Considered trial		Y	Y	Y	Y
Remarks					

PHI: Some deviations on the RTI 7+-1 can be 9 days in some trials.

Residue trial summary for Lettuce and salad plants												
Trial No./ Location/ Year	*Commodity/ Variety*	*Date of 1.Sowing or planting 2.Flowering 3. Harvest*	*Application rate per treatment*			*Dates of treatment or no. of treatments and last date*	*Growth stage at last treatment or date*	*Portion analyzed*	*Residues (mg/kg)*	*PHI (days)*	*Remarks*
			g a.s./ ha	*Water (l/ha)*	*g a.s./hl*				*DPX-KN128+KN127*		
					Northern Europe						
Sulniac 56250 - France (1)	Leaf lettuce/ Altadis	1. 11/05/2011 2. NR 3. 06/07/2011	35.6 36.4 35.6 39.7 24.0 34.7	-	-	31/05/2011 07/06/2011 16/06/2011 22/06/2011 28/06/2011 05/07/2011	BBCH 16 BBCH 19 BBCH 19 BBCH 41 BBCH 43 BBCH 49	Leaf lettuce	0.65	1 DALA/	**DPX-MP062 WG**
Sulniac 56250 - France (1)	Leaf lettuce/ Altadis	1. 11/05/2011 2. NR 3. 06/07/2011	38.7 36.1 34.5 40.3 19.3 39.5			31/05/2011 07/06/2011 16/06/2011 22/06/2011 28/06/2011 05/07/2011	BBCH 16 BBCH 19 BBCH 19 BBCH 41 BBCH 43 BBCH 49	Leaf lettuce	0.23	1 DALA	**DPX-KN128 WG**
					Southern Europe						
Elne 66200 - France (2)	Head lettuce/ Forlina (closed variety)	1. 26/06/2011 2. NR 3. 05/08/2011	38.0 37.5 39.3 39.0 37.5 36.3			01/07/2011 08/07/2011 15/07/2011 22/07/2011 29/07/2011 04/08/2011	BBCH 18 BBCH 19 BBCH 42 BBCH 42 BBCH 42 BBCH 49	Head lettuce	0.29	1 DALA	**DPX-MP062 WG**
Elne 66200 - France (2)	Head lettuce/ Forlina (closed variety)	1. 26/06/2011 2. NR 3. 05/08/2011	38.3 38.0 39.0 37.8 38.0 36.0			01/07/2011 08/07/2011 15/07/2011 22/07/2011 29/07/2011 04/08/2011	BBCH 18 BBCH 19 BBCH 42 BBCH 42 BBCH 42 BBCH 49	Head lettuce	0.15	1 DALA	**DPX-KN128 WG**
Elne 66200 - France (3)	Leaf lettuce/ Kitare (open leaf)	1. 26/06/2011 2. NR 3. 05/08/2011	38.7 38.7 38.2 39.0 39.3 38.0			01/07/2011 08/07/2011 15/07/2011 22/07/2011 29/07/2011 04/08/2011	BBCH 19 BBCH 41 BBCH 42 BBCH 42 BBCH 42 BBCH 49	Leaf lettuce	<u>0.60</u>	1 DALA	**DPX-MP062 WG**

Elne 66200 - France (3)	Leaf lettuce/ Kitare (open leaf)	1. 26/06/2011 2. NR 3. 05/08/2011	37.8 37.8 39.0 37.5 38.3 38.7			01/07/2011 08/07/2011 15/07/2011 22/07/2011 29/07/2011 04/08/2011	BBCH 19 BBCH 41 BBCH 42 BBCH 42 BBCH 42 BBCH 49	Leaf lettuce	0.42	1 DALA	**DPX-KN128 WG**
Nea Magnisia 57008 - Greece (4)	Leaf lettuce/ Manchester (open leaf)	1. 03/09/2011 2. NR 3. 25/10/2011	36.3 39.0 36.0 39.0 39.0 39.3			21/09/2011 28/09/2011 05/10/2011 12/10/2011 18/10/2011 24/10/2011	BBCH 37 BBCH 41 BBCH 43 BBCH 44 BBCH 47 BBCH 49	Leaf lettuce	<u>1.4</u>	1 DALA	**DPX-MP062 WG**
Nea Magnisia 57008 - Greece (4)	Leaf lettuce/ Manchester (open leaf)	1. 03/09/2011 2. NR 3. 25/10/2011	36.0 38.5 38.7 38.3 38.5 37.0			21/09/2011 28/09/2011 05/10/2011 12/10/2011 18/10/2011 24/10/2011	BBCH 37 BBCH 41 BBCH 43 BBCH 44 BBCH 47 BBCH 49	Leaf lettuce	0.93	1 DALA	**DPX-KN128 WG**

(a) According to BAYER codes
(b)Only if relevant

(c) High or low volume spraying, spreading, dusting etc., overall, broadcast, type or equipment used must be indicated

(e) BBCH Monograph, Growth Stages of Plants, 1997, Blackwell, ISBN 3-8263-3152-4

(f) Minimum number of days after last application (Label pre-harvest interval, PHI, underline): DBLA = days before last application, DALA = days after last application

(g) Remarks may include: climatic conditions; reference to analytical method; Information concerning the metabolites included, the method of storage, storage stability, analysis date

Residues calculated as DPX-KN128

Report:	Spence, C.M.; 2014
Title:	Determination of Magnitude and Decline of Residues of DPX-KN128 (Indoxacarb) along with IN-KN127 in Lettuce Following Applications of DPX-KN128 30WG – Europe – Initiated 2012
Document No:	DuPont-35819
Guidelines:	OECD Test Guideline 509, OECD Guideline for the Testing of Chemicals: Crop Field Trial, 07 September 2009. European Communities Guidelines for the Generation of Data Concerning Residues, as Provided in Annex II, Part A, Section 6 and Annex III, Part A, Section 8 of EC Commission Directive 91/414/EEC. SANCO/825/00 rev.8.1 (16/11/2010) Guidance Document on Pesticide Residue Analytical Methods
GLP	Yes

Acceptability	Deviations
Yes	None

Table 3.1.1-12: Summary of global information on study 3

Comparative trials (between formulations, with and adjuvant/safener/synergist)	1/ DPX-KN128-298 WG : indoxacarb 300 g/kg
Number of applications	4 (RTI:7 days)
Dose (g as/ha)	1/ DPX-KN128 : 37,5 g a.s./ha
Mode of application	Foliar Treatment
PHI (days) and/or growth stage (BBCH)	PHI : - 0,1,3,7,10,14 (Decline) - 1 and 3 (Harvest)
Analytical method (Code +Type)	- Testing of DFG Method S19 for the determination of residue of KN128 along with KN127 in crops which might be treated with DPX-MP062. Dupont Report AMR 4271-96, 1997 - Confirmation of the Stability of DPX-KN128 (Indoxacarb) and IN-KN127 on Crops for which European Maximum Residue Limits are Proposed - Independent Laboratory Validation of the Analytical Residue Method AMR 4271-96 for the Determination of Residues of DPX-KN128 and IN-KN127 in Plant Material which might be Treated with DPX-MP062.
LoQ (mg/kg)	KN128 (indoxacarbe) 0,01 mg/kg (LOD = 0,003mg/kg)

Table 3.1.1-13: Summary of the study 3 trials

N° Trial		DuPont-35819 / 1	DuPont-35819 / 2	DuPont-35819 / 3	DuPont-35819 / 4	DuPont-35819 / 5	DuPont-35819 / 6	DuPont-35819 / 7	DuPont-35819 / 8
North/South/Indoor		N	N	N	N	S	S	S	S
Decline (D)/Harvest (H) trial?		D	D	H	H	D	D	H	H
Formulation		WG	WG	WG	WG	WG	WG	WG	WG
Equivalence between formulations		Y	Y	Y	Y	Y	Y	Y	Y
Accordance with intended GAP		Y	Y	Y	Y	Y	Y	Y	Y
Correct sampling		Y	Y(2)	Y	Y	Y	Y	Y	Y
Samples frozen within 24h		Y	Y	Y	Y	Y	Y	Y	Y
Storage period (in days)	Sample	< 10 months	< 11 months	< 11 months	< 10 months	< 9 months	< 10 months	< 10 months	< 9 months
	Extract	< 1 day	1 day	< 1 day	< 1 day	< 1 day	1 day	< 1 day	< 1 day
Storage T° <-18°C		Y(1)	Y	Y	Y	Y	Y	Y(1)	Y
Validated analytical method		Y	Y	Y	Y	Y	Y	Y	Y
Negative controls		Y	Y	Y	Y	Y	Y	Y	Y
Considered trial		Y	Y	Y	Y	Y	Y	Y	Y
Remarks		Some deviations in the sprayer			Some deviations in the sprayer				

(1) Some temperature variations occurred during the storage up to -5°C but the trial is still considered as valid.
(2) Variation in the DALA duration during the sampling. At PHI 8/11 and 15 instead of PHI 7/10/14

N° Trial		DuPont-35819 / 9	DuPont-35819 / 10	DuPont-35819 / 11	DuPont-35819 / 12	DuPont-35819 / 13	DuPont-35819 / 14	DuPont-35819 / 15	DuPont-35819 / 16
North/South/Indoor		N	N	N	N	N	N	N	N
Decline (D)/Harvest (H) trial?		D	D	H	H	D	D	H	H
Formulation		WG	WG	WG	WG	WG	WG	WG	WG
Equivalence between formulations		Y	Y	Y	Y	Y	Y	Y	Y
Accordance with intended GAP		Y(1)	Y	Y	Y	Y	Y	Y	Y
Correct sampling		N(2)	Y	N(2)	Y	Y	Y	Y	N(4)
Samples frozen within 24h		Y	Y	Y	Y	Y	Y	Y	
Storage period (in days)	Sample	< 11 months	< 11 months	< 10 months	< 9 months	< 5 months	< 5 months	< 5 months	< 6 months
	Extract	< 1 day	< 1 day	1 day	1 day	2 days	1 day	< 1 day	< 1 day
Storage T° <-18°C		Y	Y	Y	Y(3)	Y	Y	Y	
Validated analytical method		Y	Y	Y	Y	Y	Y	Y	Y
Negative controls		Y	Y	Y(5)	Y	Y	Y	Y	Y
Considered trial		Y	Y	Y	Y	Y	Y	Y	Y
Remarks							No records for the past use on this site	No records for the past use on this site	

(1) RTI of 5 days instead of 7 (+-1)

(2) Some spare samples are missing due to a poor crop yield

(3) Some variations in the temperature during the shipment

(4) One sample is only up to 1kg instead of 2kg.

(5) Untreated sample analysed at a residue level of 0.005 mg/kg (LOD: 0.003 < LOQ = 0.01)

N° Trial		DuPont-35819 / 17	DuPont-35819 / 18	DuPont-35819 / 19
North/South/Indoor		S	S	S
Decline (D)/Harvest (H) trial?		D	D	H
Formulation		WG	WG	WG
Equivalence between formulations		Y	Y	Y
Accordance with intended GAP		Y	Y	Y
Correct sampling		Y	Y	Y
Samples frozen within 24h		Y	Y	Y
Storage period (in days)	Sample	< 7 months	< 6 months	< 5 months
	Extract	3 days	1 day	1 day
Storage T° <-18°C		Y(1)	Y	Y
Validated analytical method		Y	Y	Y
Negative controls		Y	Y	Y
Considered trial		Y	Y	Y
Remarks				

Frozen shipping temperature has been up to -2.5°C during sometimes instead of -18°C

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2012	Open leaf field lettuce	UK Gedney Marsh, Holbeach, Lincolnshire	37.25 (99% of GAP) 36.31 (97% of GAP) 36.31 (97% of GAP) 38.81 (103% of GAP)	BBCH 47 BBCH 47–48 BBCH 47–48 BBCH 49	Mature leaves	-0	0.18	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
						+0	0.51	
			38.50 (103% of GAP) 37.87 (101% of GAP) 36.62 (98% of GAP) 36.93 (98% of GAP)	BBCH 47 BBCH 46–48 BBCH 47–48 BBCH 48–49		1	0.59	
			36.62 (98% of GAP) 38.50 (103% of GAP) 38.19 (102% of GAP) 38.50 (103% of GAP)	BBCH 47 BBCH 43 BBCH 46 BBCH 48		3	0.32	
			40.69 (109% of GAP) 38.50 (103% of GAP) 37.25 (99% of GAP) 38.19 (102% of GAP)	BBCH 19 BBCH 47 BBCH 47–48 BBCH 47–48		7	0.24	
			39.75 (106% of GAP) 36.93 (98% of GAP) 37.87 (101% of GAP) 36.00 (96% of GAP)	BBCH 19 BBCH 47 BBCH 43 BBCH 46		10	0.12	
			36.93 (98% of GAP) 39.13 (104% of GAP) 37.56 (100% of GAP) 37.25 (99% of GAP)	BBCH 13–15 BBCH 19 BBCH 47 BBCH 47–48		14	0.071	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2012	Open leaf field lettuce	Germany Goch-Nierswalde, Kleve	36.56 (97% of GAP) 37.56 (100% of GAP) 38.06 (101% of GAP) 38.06 (101% of GAP)	BBCH 18 BBCH 41 BBCH 45 BBCH 49	Mature leaves	-0	0.14	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
						+0	0.84	
			39.06 (104% of GAP) 39.06 (104% of GAP) 37.56 (100% of GAP) 37.06 (99% of GAP)	BBCH 17 BBCH 37–39 BBCH 45 BBCH 47–49		1	0.45	
			39.06 (104% of GAP) 39.06 (104% of GAP) 39.06 (104% of GAP) 36.56 (97% of GAP)	BBCH 17 BBCH 33 BBCH 43 BBCH 47		3	0.24	
			39.06 (104% of GAP) 38.56 (103% of GAP) 38.56 (103% of GAP) 38.56 (103% of GAP)	BBCH 16 BBCH 18 BBCH 41 BBCH 45		8	0.16	
			38.56 (103% of GAP) 38.06 (101% of GAP) 38.06 (101% of GAP) 38.06 (101% of GAP)	BBCH 15 BBCH 17 BBCH 33 BBCH 43		11	0.067	
			38.06 (101% of GAP) 37.56 (100% of GAP) 38.06 (101% of GAP) 38.06 (101% of GAP)	BBCH 15 BBCH 16 BBCH 18 BBCH 41		15	0.044	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2012	Open leaf field lettuce	France Lorgies, Nord-Pas de Calais	36.62 (98% of GAP) 37.25 (99% of GAP) 39.44 (105% of GAP) 37.87 (101% of GAP)	BBCH 46 BBCH 47 BBCH 48 BBCH 48–49	Mature leaves	1	0.14	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
			38.81 (103% of GAP) 36.31 (97% of GAP) 35.68 (95% of GAP) 36.31 (97% of GAP)	BBCH 46 BBCH 47 BBCH 48 BBCH 48–49		3	0.17	
DuPont-35819; study to GLP, study carried out in 2012	Open leaf field lettuce	Czech Republic Český Těšín, Moravskoslezský kraj	36.23 (97% of GAP) 36.98 (99% of GAP) 37.30 (99% of GAP) 39.05 (104% of GAP)	BBCH 45 BBCH 46–47 BBCH 47–48 BBCH 48–49	Mature leaves	1	0.85	
			35.82 (96% of GAP) 37.08 (99% of GAP) 37.80 (101% of GAP) 36.12 (96% of GAP)	BBCH 45 BBCH 46 BBCH 47 BBCH 48		3	0.42	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2012	Open leaf field lettuce	Spain Benavent de Segria, Catalunya	37.87 (101% of GAP) 37.25 (99% of GAP) 38.50 (103% of GAP) 37.87 (101% of GAP)	BBCH 45 BBCH 47 BBCH 47 BBCH 49	Mature leaves	-0	0.17	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and
						+0	0.77	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

			38.81 (103% of GAP) 37.56 (100% of GAP) 37.87 (101% of GAP) 37.87 (101% of GAP)	BBCH 45 BBCH 47 BBCH 47 BBCH 49		1	0.53	6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
			37.87 (101% of GAP) 37.56 (100% of GAP) 37.87 (101% of GAP) 37.56 (100% of GAP)	BBCH 45 BBCH 47 BBCH 47 BBCH 49		3	0.44	
			37.56 (100% of GAP) 38.19 (102% of GAP) 37.87 (101% of GAP) 38.50 (103% of GAP)	BBCH 44 BBCH 45 BBCH 47 BBCH 47		7	0.14	
			37.87 (101% of GAP) 36.93 (98% of GAP) 38.81 (103% of GAP) 37.87 (101% of GAP)	BBCH 44 BBCH 45 BBCH 47 BBCH 47		10	0.14	
			38.50 (103% of GAP) 37.25 (99% of GAP) 37.87 (101% of GAP) 37.25 (99% of GAP)	BBCH 44 BBCH 44 BBCH 45 BBCH 47		14	0.084	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2012	Open leaf field lettuce	Greece Chalkidona, Thessaloniki	37.40 (100% of GAP) 37.60 (100% of GAP) 37.60 (100% of GAP) 37.56 (100% of GAP)	BBCH 32 BBCH 37 BBCH 41 BBCH 49	Mature leaves	-0	0.084	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
						+0	0.22	
			37.69 (101% of GAP) 37.64 (100% of GAP) 37.35 (100% of GAP) 37.56 (100% of GAP)	BBCH 32 BBCH 35/37 BBCH 41 BBCH 43/49		1	0.67	
			37.27 (99% of GAP) 37.52 (100% of GAP) 37.52 (100% of GAP) 37.52 (100% of GAP)	BBCH 19/32 BBCH 35 BBCH 41 BBCH 43/49		3	0.14	
			37.14 (99% of GAP) 37.52 (100% of GAP) 37.48 (100% of GAP) 37.52 (100% of GAP)	BBCH 19 BBCH 32 BBCH 37 BBCH 41		7	0.11	
			37.48 (100% of GAP) 37.48 (100% of GAP) 37.43 (100% of GAP) 37.64 (100% of GAP)	BBCH 19 BBCH 19/32 BBCH 35 BBCH 41		10	0.065	
			37.56 (100% of GAP) 37.39 (100% of GAP) 37.85 (101% of GAP) 37.60 (100% of GAP)	BBCH 16/17 BBCH 19 BBCH 32 BBCH 37		14	0.015	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2012	Open leaf field lettuce	France Lucenay, Rhone Alpes	37.25 (99% of GAP) 37.56 (100% of GAP) 38.50 (103% of GAP) 37.56 (100% of GAP)	BBCH 47 BBCH 47 BBCH 49 BBCH 49	Mature leaves	1	0.80	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
			38.50 (103% of GAP) 38.19 (102% of GAP) 37.25 (99% of GAP) 38.50 (103% of GAP)	BBCH 47 BBCH 47 BBCH 49 BBCH 49		3	0.65	
DuPont-35819; study to GLP, study carried out in 2012	Open leaf field lettuce	Spain Aguadulce, Andalucia	37.25 (99% of GAP) 39.13 (104% of GAP) 38.81 (103% of GAP) 36.93 (98% of GAP)	BBCH 33 BBCH 39 BBCH 39 BBCH 49	Mature leaves	1	0.29	
			38.19 (102% of GAP) 38.19 (102% of GAP) 38.50 (103% of GAP) 37.87 (101% of GAP)	BBCH 33 BBCH 35 BBCH 39 BBCH 49		3	0.23	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2012	Field Lambs lettuce	Austria Rohrau, Lower Austria	36.25 (97% of GAP) 37.17 (99% of GAP) 37.83 (101% of GAP) 39.20 (105% of GAP)	BBCH 16 BBCH 16 BBCH 16 BBCH 16–18	Mature leaves	-0	0.54	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
						+0	3.6	
			36.20 (97% of GAP) 38.15 (102% of GAP) 39.25 (105% of GAP) 38.88 (104% of GAP)	BBCH 16 BBCH 16 BBCH 16 BBCH 16		1	3.2	
			36.95 (99% of GAP) 36.25 (97% of GAP) 36.12 (96% of GAP) 37.38 (100% of GAP)	BBCH 14–16 BBCH 16 BBCH 18 BBCH 18–20		3	1.7	
			35.67 (95% of GAP) 36.87 (98% of GAP) 38.63 (103% of GAP) 37.28 (99% of GAP)	BBCH 14 BBCH 16 BBCH 18 BBCH 18		7	0.53	
			35.70 (95% of GAP) 37.75 (101% of GAP) 38.50 (103% of GAP) 36.60 (98% of GAP)	BBCH 12–14 BBCH 14–16 BBCH 16 BBCH 16		9	0.29	
			35.67 (95% of GAP) 37.70 (101% of GAP) 39.33 (105% of GAP) 39.33 (105% of GAP)	BBCH 12 BBCH 14 BBCH 16 BBCH 18		14	0.053	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2012	Field Lambs lettuce	Germany Goch-Nierswalde, Kleve	38.56 (103% of GAP) 38.06 (101% of GAP) 37.06 (99% of GAP) 39.06 (104% of GAP)	BBCH 18 BBCH 39 BBCH 45 BBCH 49	Mature leaves	-0 +0	0.67 2.7	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
			39.06 (104% of GAP) 38.56 (103% of GAP) 36.56 (97% of GAP) 38.56 (103% of GAP)	BBCH 17 BBCH 37 BBCH 45 BBCH 47–49		1	1.5	
			37.06 (99% of GAP) 38.06 (101% of GAP) 37.56 (100% of GAP) 38.06 (101% of GAP)	BBCH 17 BBCH 33 BBCH 41 BBCH 47		3	0.96	
			39.06 (104% of GAP) 37.56 (100% of GAP) 37.06 (99% of GAP) 38.56 (103% of GAP)	BBCH 16 BBCH 18 BBCH 39 BBCH 45		8	0.70	
			39.06 (104% of GAP) 38.56 (103% of GAP) 37.06 (99% of GAP) 38.56 (103% of GAP)	BBCH 15 BBCH 17 BBCH 33 BBCH 41		11	0.64	
			38.06 (101% of GAP) 38.06 (101% of GAP) 38.06 (101% of GAP) 38.06 (101% of GAP)	BBCH 15 BBCH 16 BBCH 18 BBCH 39		15	0.29	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2012	Field Lambs lettuce	Czech Republic Uherský Ostroh, Zlinsky kraj	36.98 (99% of GAP) 36.33 (97% of GAP) 36.00 (96% of GAP) 38.35 (102% of GAP)	BBCH 15 BBCH 16 BBCH 16–17 BBCH 16–18	Mature leaves	1	4.9	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
			36.68 (98% of GAP) 38.22 (102% of GAP) 36.72 (98% of GAP) 36.45 (97% of GAP)	BBCH 15 BBCH 16 BBCH 16–17 BBCH 16–18		3	3.6	
DuPont-35819; study to GLP, study carried out in 2012	Field Lambs lettuce	France Verlinghem, Nord-Pas de Calais	36.00 (96% of GAP) 38.50 (103% of GAP) 36.62 (98% of GAP) 36.31 (97% of GAP)	BBCH 47 BBCH 47–48 BBCH 48 BBCH 48–49	Mature leaves	1	1.2	
			35.68 (95% of GAP) 38.81 (103% of GAP) 37.87 (101% of GAP) 37.25 (99% of GAP)	BBCH 47 BBCH 47–48 BBCH 48 BBCH 48–49		3	1.1	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2012	Open leaf field lettuce	France Douai, Nord-Pas de Calais	39.44 (105% of GAP) 39.13 (104% of GAP) 39.13 (104% of GAP) 36.62 (98% of GAP)	BBCH 45 BBCH 46 BBCH 48 BBCH 49	Mature leaves	-0	0.034	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
						+0	0.31	
			36.31 (97% of GAP) 37.56 (100% of GAP) 35.68 (95% of GAP) 38.50 (103% of GAP)	BBCH 45 BBCH 46 BBCH 48 BBCH 48		1	0.29	
			35.06 (93% of GAP) 35.37 (94% of GAP) 36.31 (97% of GAP) 38.19 (102% of GAP)	BBCH 44 BBCH 46 BBCH 47 BBCH 48		3	0.18	
			35.68 (95% of GAP) 35.68 (95% of GAP) 35.68 (95% of GAP) 37.56 (100% of GAP)	BBCH 41–42 BBCH 45 BBCH 46 BBCH 48		8	0.055	
			36.00 (96% of GAP) 36.93 (98% of GAP) 36.00 (96% of GAP) 36.62 (98% of GAP)	BBCH 19 BBCH 44 BBCH 46 BBCH 47		10	0.045	
			38.19 (102% of GAP) 36.93 (98% of GAP) 38.81 (103% of GAP) 36.31 (97% of GAP)	BBCH 16–18 BBCH 41–42 BBCH 45 BBCH 46		15	0.013	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

<table>
<tr><th colspan="9">GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide</th></tr>
<tr><th>GLP and trial details</th><th>Crop</th><th>Country</th><th>Application rate
(g a.s./ha)</th><th>Crop growth stage</th><th>Commodity or matrix</th><th>PHI
(days)</th><th>Residues found (mg/kg)</th><th>Recovery data</th></tr>
<tr><td rowspan="7">DuPont-35819;
study to GLP,
study carried out in 2013</td><td rowspan="7">Open leaf field lettuce</td><td rowspan="7">Belgium
Wytschate,
Hainaut</td><td rowspan="2">36.62 (98% of GAP)
37.87 (101% of GAP)
39.44 (105% of GAP)
38.50 (103% of GAP)</td><td rowspan="2">BBCH 16–17
BBCH 44
BBCH 48
BBCH 48–49</td><td rowspan="7">Mature leaves</td><td>-0</td><td>0.14</td><td rowspan="7">Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)</td></tr>
<tr><td>+0</td><td>0.19</td></tr>
<tr><td>36.31 (97% of GAP)
36.93 (98% of GAP)
37.56 (100% of GAP)
36.62 (98% of GAP)</td><td>BBCH 16–17
BBCH 44
BBCH 48
BBCH 48–49</td><td>1</td><td>0.27</td></tr>
<tr><td>37.87 (101% of GAP)
36.62 (98% of GAP)
37.87 (101% of GAP)
38.19 (102% of GAP)</td><td>BBCH 16–17
BBCH 43
BBCH 44
BBCH 48</td><td>3</td><td>0.19</td></tr>
<tr><td>37.56 (100% of GAP)
36.93 (98% of GAP)
36.93 (98% of GAP)
36.62 (98% of GAP)</td><td>BBCH 16
BBCH 16–17
BBCH 44
BBCH 48</td><td>7</td><td>0.072</td></tr>
<tr><td>36.31 (97% of GAP)
38.19 (102% of GAP)
36.62 (98% of GAP)
38.81 (103% of GAP)</td><td>BBCH 15
BBCH 16–17
BBCH 43
BBCH 44</td><td>10</td><td>0.067</td></tr>
<tr><td>37.25 (99% of GAP)
38.19 (102% of GAP)
37.25 (99% of GAP)
36.00 (96% of GAP)</td><td>BBCH 14
BBCH 16
BBCH 16–17
BBCH 44</td><td>14</td><td>0.043</td></tr>
</table>

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2013	Open leaf field lettuce	UK Freuchie, Fife	37.87 (101% of GAP) 39.44 (105% of GAP) 37.87 (101% of GAP) 37.25 (99% of GAP)	BBCH 37 BBCH 39 BBCH 41 BBCH 47	Mature leaves	1	0.28	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
			37.25 (99% of GAP) 37.87 (101% of GAP) 37.87 (101% of GAP) 36.31 (97% of GAP)	BBCH 37 BBCH 39 BBCH 41 BBCH 45		3	0.19	
DuPont-35819; study to GLP, study carried out in 2013	Open leaf field lettuce	Poland Urbanowice, Gościęcin, Opeln	39.0 (104% of GAP) 38.7 (103% of GAP) 39.3 (105% of GAP) 36.9 (98% of GAP)	BBCH 41 BBCH 42 BBCH 43 BBCH 44	Mature leaves	1	0.18	
			39.0 (104% of GAP) 38.7 (103% of GAP) 39.3 (105% of GAP) 36.6 (98% of GAP)	BBCH 41 BBCH 42 BBCH 43 BBCH 44		3	0.11	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2013	Open leaf field lettuce	Spain Utrera, Andalucia	36.93 (98% of GAP) 38.19 (102% of GAP) 37.87 (101% of GAP) 38.19 (102% of GAP)	BBCH 34 BBCH 37 BBCH 45 BBCH 49	Mature leaves	-0	0.016	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
						+0	0.10	
			37.56 (100% of GAP) 36.93 (98% of GAP) 38.19 (102% of GAP) 37.25 (99% of GAP)	BBCH 34 BBCH 37 BBCH 45 BBCH 49		1	0.060	
			37.25 (99% of GAP) 38.50 (103% of GAP) 36.93 (98% of GAP) 38.19 (102% of GAP)	BBCH 33 BBCH 34 BBCH 39 BBCH 47		3	0.028	
			36.93 (98% of GAP) 37.87 (101% of GAP) 37.25 (99% of GAP) 36.62 (98% of GAP)	BBCH 19 BBCH 34 BBCH 37 BBCH 45		7	0.017	
			37.25 (99% of GAP) 36.93 (98% of GAP) 38.19 (102% of GAP) 37.56 (100% of GAP)	BBCH 16 BBCH 33 BBCH 34 BBCH 39		10	0.003	
			38.19 (102% of GAP) 37.25 (99% of GAP) 38.19 (102% of GAP) 37.25 (99% of GAP)	BBCH 15 BBCH 19 BBCH 34 BBCH 37		14	ND	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2013	Open leaf field lettuce	Italy Torrevecchia Pia, Lombardia	37.56 (100% of GAP) 39.13 (104% of GAP) 38.50 (103% of GAP) 37.56 (100% of GAP)	BBCH 17 BBCH 41 BBCH 47 BBCH 49	Mature leaves	-0	0.051	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
						+0	0.71	
			36.93 (98% of GAP) 38.50 (103% of GAP) 37.25 (99% of GAP) 38.50 (103% of GAP)	BBCH 17 BBCH 41 BBCH 47 BBCH 49		1	0.35	
			36.62 (98% of GAP) 37.87 (101% of GAP) 36.62 (98% of GAP) 37.87 (101% of GAP)	BBCH 15 BBCH 19 BBCH 42 BBCH 47		3	0.16	
			37.87 (101% of GAP) 38.19 (102% of GAP) 37.87 (101% of GAP) 35.68 (95% of GAP)	BBCH 14 BBCH 17 BBCH 41 BBCH 47		7	0.079	
			36.31 (97% of GAP) 37.56 (100% of GAP) 38.81 (103% of GAP) 37.25 (99% of GAP)	BBCH 14 BBCH 15 BBCH 19 BBCH 42		10	0.032	
			36.62 (98% of GAP) 36.93 (98% of GAP) 36.93 (98% of GAP) 38.81 (103% of GAP)	BBCH 13 BBCH 14 BBCH 17 BBCH 41		14	0.011	

Residues expressed as DPX-KN128 plus IN-KN127 in field lettuce from supervised trials (continued)

GAP is general for all countries: Four foliar applications of DPX-KN128 30WG at 37.5 g a.s./ha each to lettuce as a post-emergence insecticide								
GLP and trial details	**Crop**	**Country**	**Application rate (g a.s./ha)**	**Crop growth stage**	**Commodity or matrix**	**PHI (days)**	**Residues found (mg/kg)**	**Recovery data**
DuPont-35819; study to GLP, study carried out in 2013	Open leaf field lettuce	Greece Nea Magnisia, Thessaloniki	37.86 (101% of GAP) 37.79 (101% of GAP) 37.49 (100% of GAP) 37.59 (100% of GAP)	BBCH 14/17 BBCH 19/33 BBCH 33/35 BBCH 39	Mature leaves	1	0.36	Lettuce leaves mean recovery = 82%, 90%, and 99%, RSD = 13%, 16%, and 6.9% (n = 20, 20, and 8 in 0.01, 0.10, and 1.0 ppm, respectively)
			37.63 (100% of GAP) 37.69 (101% of GAP) 37.49 (100% of GAP) 37.53 (100% of GAP)	BBCH 14/17 BBCH 19/33 BBCH 35/39 BBCH 49		3	0.28	

C.3.1.2.3. Tomato

Report:	**Spence, C., 2014b**
Title:	**Determination of the decline of residues of DPX-KN128 (indoxacarb) along with IN-K127 in tomato and tomato processed fractions following applications of DPX-MP062 30WG - southern Europe - 2012**
Document No.:	**DuPont-35170**
Guidelines:	**OECD 508, OECD 509, EC Commission Directive 91/414/EEC, SANCO/825/00 rev.8.1 (16/11/2010)**
GLP:	**Yes**

During 2012, a total of three residue trials have been performed with indoxacarb on tomatoes. In these trials, tomatoes were treated with 6 applications of 37.5 g a.s./ha and raw tomatoes for processing studies were sampled 1 day after the last application. Sampled tomatoes were frozen and shipped until processing phases. Analyses were performed following the DFG method S19 (DuPont report AMR 4271-96).

Recovery studies carried out with the samples proved the residue was stable at least for 12 to 18 months.

Table 3.1.1-14: Summary of global information on study 3

Comparative trials (between formulations, with and adjuvant/safener/synergist)	DPX-MP062 300 g/kg WG DPX-KN128: 75 % IN-KN127: 25%
Number of applications	6
Dose (g as/ha)	37.5
Mode of application	Foliar spray
PHI (days) and/or growth stage (BBCH)	PHI 1
Analytical method (Code +Type)	DFG Method S 19
LoQ (mg/kg)	

Table 3.1.1-15: Summary of the study 3 trials

N° Trial		1	2	3
North/South/Indoor		SEU	SEU	SEU
Decline (D)/Harvest (H) trial?		D	D	D
Formulation		WG	WG	WG
Equivalence between formulations		Y	Y	Y
Accordance with intended GAP		Y	Y	Y
Correct sampling		Y	Y	Y
Samples frozen within 24h		Y	Y	Y
Storage period (in days)	Sample	15 months	15 months	15 months
	Extract	-	-	-
Storage T° <-18°C		Y	Y	Y
Validated analytical method *				

Negative controls	Y	Y	Y (except at 7 DALA specimen and tomato puree, tomato paste and tomato catsup processed commodities)
Considered trial	Y	Y	Y
Remarks			

* Validated excepted for unprocessed tomato and canned tomato.

Table 3.1.1-16: Summary of data from residue trials for study 3

Targeted GAP: Six foliar applications of DPX-MP062 30WG at 37.5 g a.s./ha each to fruiting tomato as a post-emergence insecticide							
GLP and trial details	Crop	Country	Application rate (g a.s./ha)	Crop growth stage	Commodity or matrix	PHI (days)	Residues found (mg/kg)
DuPont-35170; study to GLP, study carried out in 2012	**Field Tomato**	**Spain El Trobal, Andalucia**	**37.21 (99% of GAP) 37.82 (101% of GAP) 37.52 (100% of GAP) 37.52 (100% of GAP) 37.52 (100% of GAP) 37.21 (99% of GAP)**	**BBCH 64 BBCH 64 BBCH 66 BBCH 71 BBCH 78 BBCH 89**	**Mature fruit**	**-1**	**0.038**
						+0	**0.20**
						7	0.070
						14	**0.050**
						21	**0.067**
						28	**0.038**
					Unprocessed tomato	**1**	**0.042, 0.040, 0.044 (mean : 0.042)**
					Tomato juice		**0.072**
					Tomato puree		**0.069**
					Tomato paste		**0.13**
					Canned tomato		**0.013**
					Tomato catsup		**0.086**

Targeted GAP: Six foliar applications of DPX-MP062 30WG at 37.5 g a.s./ha each to fruiting tomato as a post-emergence insecticide							
GLP and trial details	Crop	Country	Application rate (g a.s./ha)	Crop growth stage	Commodity or matrix	PHI (days)	Residues found (mg/kg)
DuPont-35170; study to GLP, study carried out in 2012	**Field Tomato**	**South France Vaunaveys la Rochette, Rhone Alpes**	**36.60 (98% of GAP) 36.91 (98% of GAP) 37.52 (100% of GAP) 38.43 (102% of GAP) 37.21 (99% of GAP) 38.43 (102% of GAP)**	**BBCH 71 BBCH 71–73 BBCH 79 BBCH 81 BBCH 85 BBCH 85–87**	**Mature fruit**	**-1**	**0.006**
						+0	**0.025**
						7	**0.010**
						14	**0.008**
						21	**0.009**
						28	**0.007**
					Unprocessed tomato	**1**	**0.035, 0.038, 0.048** (mean: 0.040)
					Tomato juice		**0.058**
					Tomato puree		**0.059**
					Tomato paste		**0.11**
					Canned tomato		**0.011**
					Tomato catsup		**0.073**

Targeted GAP: Six foliar applications of DPX-MP062 30WG at 37.5 g a.s./ha each to fruiting tomato as a post-emergence insecticide							
GLP and trial details	Crop	Country	Application rate (g a.s./ha)	Crop growth stage	Commodity or matrix	PHI (days)	Residues found (mg/kg)
DuPont-35170; study to GLP, study carried out in 2012	**Field Tomato**	**Italy Rovereto Landi, Emilia Romagna**	**37.52 (100% of GAP) 37.21 (99% of GAP) 37.82 (101% of GAP) 37.82 (101% of GAP) 37.21 (99% of GAP) 38.74 (103% of GAP)**	**BBCH 68 BBCH 71 BBCH 72 BBCH 74 BBCH 85 BBCH 89**	**Mature fruit**	**-1**	**0.056**
						+0	**0.095**
						7	<u>0.047</u>
						14	**0.020**
						21	**0.041**
						28	**0.058**
					Unprocessed tomato	**1**	**0.027, 0.038, 0.032 (mean: 0.032)**
					Tomato juice		**0.033**
					Tomato puree		**0.065**
					Tomato paste		**0.061**
					Canned tomato		**0.007**
					Tomato catsup		**0.041**

C.3.1.2.4. Head cabbage

Report:	Lakaschus, S., Walker, K., 2012
Title:	Determination of magnitude of residues of DPX-KN128 (indoxacarb) together with IN-KN127 in broccoli following applications of DPX-MP062 30WG and DPX-KN128 30WG - Europe, 2011
Document No.:	DuPont-33517
Guidelines:	EC Commission Directive 91/414/EEC, SANCO/825/00 rev.8.1 (16/11/2010)
GLP:	Yes

Acceptability	**Deviations**
Yes	None Study dates June 2011 to October 2012

Table 3.1.1-17: Summary of global information on study 3

Comparative trials (between formulations, with and adjuvant/safener/synergist)	Yes DPX-MP062 30 WG and DPX-KN128 30 WG
Number of applications	3
Dose (g as/ha)	37.5
Mode of application	Spraying
PHI (days) and/or growth stage (BBCH)	1 d
Analytical method (Code +Type)	AMR 4271-96 (LC-MS/MS)
LoQ (mg/kg)	0.01

Table 3.1.1-18: Summary of the study 3 trials

N° Trial		1	2	3
North/South/Indoor		N	S	S
Decline (D)/Harvest (H) trial?		H	H	H
Formulation		WG	WG	WG
Equivalence between formulations		Y	Y	Y
Accordance with intended GAP		Y	Y	Y
Correct sampling		Y	Y	Y
Samples frozen within 24h		Y	Y	Y
Storage period (in days)	Sample	114	65	118
	Extract	1	1	1
Storage T° <-18°C		Y	Y	Y
Validated analytical method		Y	Y	Y
Negative controls		Y	Y	Y
Considered trial [1]		N	N	N
Remarks				

[1] Trials conducted according to a more critical GAP (not in the range of 25% of the application dose rate) the proportionality was applied:

Actual dose rate applied	measured residue levels	Intended dose rate	Calculated residue

			levels
37.7 g as/ha	0.18 mg/kg	25.5 g as/ha	**0.12 mg/kg**
37.7 g as/ha	0.18 mg/kg	25.5 g as/ha	**0.12 mg/kg**
38.4 g as/ha	0.11 mg/kg	25.5 g as/ha	**0.07 mg/kg**

Table 3.1.1-19: Summary of data from residue trials for study 3

GLP and trial details	Crop	Country	Application rate (g a.s./ha)	Crop growth stage	Commodity or matrix	PHI (days)	Residues found (mg/kg)	Recovery data
DuPont-33517; study to GLP, study carried out in 2011	Broccoli	North France Innenheim, Alsace	37.7 (126% of GAP) 37.4 (125% of GAP) 40.6 (135% of GAP) (DPX-MP062 30WG)	BBCH 41 BBCH 43 BBCH 48–49	Flower Heads and Stems	1	0.17 (DPX-KN128 plus IN-KN127)	Broccoli flower heads and stems mean recovery = 83%, RSD = 8.6% (n = 6 in 0.010 to 0.20 ppm fortification range)
			37.7 (126% of GAP) 37.3 (124% of GAP) 38.1 (127% of GAP) (DPX-KN128 30WG)	BBCH 41 BBCH 43 BBCH 48–49		1	0.18 (DPX-KN128)	
DuPont-33517; study to GLP, study carried out in 2011	Broccoli	South France Argelès-sur-Mer, Pyrénées - Orientales	38 (127% of GAP) 38 (127% of GAP) 37.1 (124% of GAP) (DPX-MP062 30WG)	BBCH 43 BBCH 43–45 BBCH 45–46	Flower Heads and Stems	1	0.18 (DPX-KN128 plus IN-KN127)	Broccoli flower heads and stems mean recovery = 83%, RSD = 8.6% (n = 6 in 0.010 to 0.20 ppm fortification range)
			38 (127% of GAP) 36.8 (123% of GAP) 37.9 (126% of GAP) (DPX-KN128 30WG)	BBCH 43 BBCH 43–45 BBCH 45–46		1	0.11 (DPX-KN128)	
DuPont-33517; study to GLP, study carried out in 2011	Broccoli	South France Merville, Midi Pyrénées	37.94 (126% of GAP) 37.27 (124% of GAP) 39.99 (133% of GAP) (DPX-MP062 30WG)	BBCH 19 BBCH 19–41 BBCH 19–49	Flower Heads and Stems	1	0.11 (DPX-KN128 plus IN-KN127)	Broccoli flower heads and stems mean recovery = 83%, RSD = 8.6% (n = 6 in 0.010 to 0.20 ppm fortification range)
			38.25 (128% of GAP) 34.77 (116% of GAP) 37.56 (125% of GAP) (DPX-KN128 30WG)	BBCH 19 BBCH 19–41 BBCH 19–49		1	0.10 (DPX-KN128)	

Report:	**Aitken, A., 2014**
Title:	**Determination of the decline of residues of DPX-KN128 (indixacarb) along with IN-KN127 in head cabbage following applications of DPX-MP062 30WG - northern Europe - 2012**
Document No.:	**DuPont-39889**
Guidelines:	**EC Commission Directive 91/414/EEC, OECD 509, SANCO/825/00 rev.8.1 (16/11/2010)**
GLP:	**Yes**

Acceptability	Deviations
Yes	No deviations were thought to have impacted the outcome of the study. Study dates June 2012 to February 2014

Table IIIA 3.1.1-20: Summary of global information on study 1

Comparative trials (between formulations, with and adjuvant/safener/synergist)	No DPX-MP062 30 WG
Number of applications	3
Dose (g as/ha)	25.5
Mode of application	Spraying
PHI (days) and/or growth stage (BBCH)	-0, 0, 1, 3, 7, 14, 28
Analytical method (Code +Type)	AMR 4271-96 (LC-MS/MS)
LoQ (mg/kg)	0.01

Table 3.1.1-21: Summary of the study 1 trials

N° Trial		1	2
North/South/Indoor		N	N
Decline (D)/Harvest (H) trial?		D	D
Formulation		WG	WG
Equivalence between formulations		Y	Y
Accordance with intended GAP		Y	Y
Correct sampling		Y	Y
Samples frozen within 24h		Y	Y
Storage period (in days)	Sample	<7 months	
	Extract	(1)	
Storage T° <-18°C		Y	Y
Validated analytical method		Y	Y
Negative controls		Y	Y
Considered trial		Y	Y
Remarks			

(1) Recovery data for fortification run concurrently with the treated samples are within 70-110%; therefore no extract stability data are necessary.

Table 3.1.1-22: Summary of data from residue trials for study 1

GLP and trial details	Crop	Country	Application rate (g a.s./ha)	Crop growth stage	Commodity or matrix	PHI (days)	Residues found (mg/kg)	Recovery data
DuPont-35171; study to GLP, study carried out in 2012	Field Head cabbage	UK South Beaumont, Essex	26.23 25.93 25.32	BBCH 45 BBCH 46 BBCH 48	Mature head cabbage	-0	0.019	Mean recovery = 85%, RSD = 13 (n = 14 in 0.01–1.0 ppm fortification range) Calculated for this summary from recovery data in the report.
						+0	0.035	
						1	0.005	
						3	0.030	
						7	0.053, 0.061[a]	
						14	0.056	
						28	**0.14**[b]	
DuPont-35171; study to GLP, study carried out in 2012	Field Head cabbage	UK North East Fortune, East Lothian	25.01 26.23 26.54	BBCH 43 BBCH 47 BBCH 49	Mature head cabbage	-0	ND	
						+0	0.004	
						1	0.007	
						3	**0.029**	
						7	0.006	
						14	ND	
						28	ND	

[a] Reanalysis results (the original measured value was 0.420 mg/kg; this is considered an outlier and will not be used for calculations).
[b] Average value (original measured value was 0.129 mg/kg; the results of the reanalysis were 0.140 and 0.140 mg/kg).

Report:	Hornshuh, M.J., Enriquez, M.A., Majdi, R., Matni, H., 2002
Title:	Decline of residues of DPX-KN128 (indoxacarb) together with IN-KN127 in cabbage (head brassica vegetables) following applications of DPX-MP062 30WG - northern Europe, season 2001
Document No.:	DuPont-6062
Guidelines:	Directive 91/414/EEC
GLP:	Yes

Acceptability	Deviations
Yes	No deviations were thought to have impacted the outcome of the study. Study dates March 2001 to December 2001

Table IIIA 3.1.1-23: Summary of global information on study 2

Comparative trials (between formulations, with and adjuvant/safener/synergist)	No DPX-MP062 30 WG
Number of applications	3
Dose (g as/ha)	25.5 to 28
Mode of application	Spraying
PHI (days) and/or growth stage (BBCH)	0, 1, 3
Analytical method (Code +Type)	AMR 3493-95 (GC-MS)
LoQ (mg/kg)	0.01

Table IIIA 3.1.1-24: Summary of the study 2 trials

N° Trial		1	2
North/South/Indoor		N	N
Decline (D)/Harvest (H) trial?		D	D
Formulation		WG	WG
Equivalence between formulations		Y	Y
Accordance with intended GAP		Y	Y
Correct sampling		Y	Y
Samples frozen within 24h		Y	Y
Storage period (in days)	Sample	<4 months	
	Extract	(1)	
Storage T° <-18°C		Y	Y
Validated analytical method		Y	Y
Negative controls		Y	Y
Considered trial		Y	Y
Remarks			

(1) Recovery data for fortification run concurrently with the treated samples are within 70-110%; therefore no extract stability data are necessary.

Table 3.1.1-25: Summary of data from residue trials for study 2

Residues expressed as DPX-KN128 plus IN-KN127 in head cabbage from supervised trials

Targeted GAP: Three foliar applications of DPX-MP062 30WG at 25.5 g a.s./ha each to head cabbage as a post-emergence insecticide								
GLP and trial details	Crop	Country	Application rate (g a.s./ha)	Crop growth stage	Commodity or matrix	PHI (days)	Residues found (mg/kg)	Recovery data
DuPont-6062; study to GLP, study carried out in 2001	**Savoy cabbage/ Wirosa**	**France St. Genouph, Indre et Loire,**	**27.2 (107% of GAP) 27.2 (107% of GAP) 27.2 (107% of GAP)**	**BBCH 49**	**Mature head cabbage**	**-1h**	**<0.02, <0.02**	**Mean recovery = 101%, RSD = 5% (n = 4 in 0.020–0.20 ppm fortification range)**
						+2h	**<0.02, <0.02**	
						1	**<0.02, <0.02**	
						3	**<0.02, <0.02**	
DuPont-6062; study to GLP, study carried out in 2001	**Cabbage/ Castello**	**Germany Motterwitz, Sachsen**	**26.7 (105% of GAP) 26.7 (105% of GAP) 26.7 (105% of GAP)**	**BBCH 49**	**Mature head cabbage**	**-1h**	**<0.02, <0.02**	
						+2h	**<0.02, 0.02**	
						1	**<0.02, <0.02**	
						3	**<0.02, <0.02**	

C.3.1.2.5. Maize forage

Report:	Aitken A., 2014
Title:	Determination of the Decline of Residues of DPX-KN128 (Indoxacarb) along with IN-KN127 in Maize Forage Following Applications of DPX-MP062 30WG - Southern Europe - 2012
Document No:	DuPont-35172
Guidelines:	OECD Principles of Good Laboratory Practices ENV/MC/CHEM(98)17,OECD, Paris, 1998 Application of the GLP Principles to field studies ENV/JM/MONO(99)22
GLP	Yes

Acceptability	**Deviations**
Yes	No

Table IIIA 3.1.1-26: Summary of global information on study 1

Comparative trials (between formulations, with and adjuvant/safener/synergist)	DPX-MP062-613 30 WG (300 g a.s./kg)
Number of applications	2
Dose (g as/ha)	37.5 g a.s./ha
Mode of application	Foliar spray
PHI (days) and/or growth stage (BBCH)	-0, 0, 7, 14, 21, 28 DALA
Analytical method (Code +Type)	AMR 4271-96 “Testing of DFG Method S 19 for the determination of residues of KN128 along with KN127 in crops which might be treated with DPX-MP062”
LoQ (mg/kg)	0.01 mg/kg

Table IIIA 3.1.1-27: Summary of the study 1 trials

N° Trial	35172/01	35172/02	35172/03	35172/04	35172/05
North/South/Indoor	S	S	S	S	S
Decline (D)/Harvest (H) trial?	D	D	D	D	D
Formulation	WG	WG	WG	WG	WG
Equivalence between formulations	Y	Y	Y	Y	Y
Accordance with intended GAP	Y[(1)]	Y[(1)]	Y[(1)]	Y[(1)]	Y[(1)]
Correct sampling	Y	Y	Y	Y	Y
Samples frozen within 24h	Y	Y	Y	Y	Y

Storage period (in days)	Sample	<12 months	<13 months (375 d)	<12 months	<13 months (385 d)	<12 months
	Extract	≤1	≤1	≤1	≤1	≤1
Storage T° <-18°C		Y	Y	Y	Y	Y
Validated analytical method						
Negative controls		Y	Y	Y	Y	Y
Considered trial		Y	Y	Y	Y	Y
Remarks						

(1) 10 days interval between the two applications instead of 20 indicated.

Summary of data from residue trials for study 1

RESIDUES DATA SUMMARY FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)
Notifier: E. I. du Pont de Nemours and Company
address Wilmington, Delaware 19898, U.S.A.
1

Active ingredient : indoxacarb
Crop / crop group : Maize forage

Content of a.i. (g/kg or g/l) : 300 g a.i./kg
Formulation (e.g. WP) : WG
Commercial product (name) : STEWARD
Applicant : Foliar application using a boom sprayer

Indoors / outdoors : Outdoor
Other a. s. in formulation (common name and content) :
Residues calculated as : DPX-KN128/IN-KN127

1	2	3	4			5	6	7	8	9	10
Report-No. Location incl. Postal code and date	Commodity / Variety (a)	Date of 1)Sowing or planting 2)Flowering 3) Harvest (b)	Application rate per treatment			Dates of treatments or no. of treatments and last date (c)	Growth stage at last treatment or date	Portion analysed (a)	Residues (mg/kg)	PHI (days) (d)	Remarks (e)
			kg a.s./ha	Water hl/ha	**g** a.s./hl						
DuPont-35172 Termens, Catalunya, 25670, Spain	Maize / PR 33Y-74	1. 20 Apr 12 2. NR 3. 21 Jul 12 28 Jul 12 04 Aug 12 11 Aug 12 18 Aug 12	38.43 38.74	511 517	7.52 7.49	A1 = 11 Jul 12 A2 = 21 Jul 12	61 69	Maize forage	0.37 0.61 0.82 0.32 0.080 0.13	-0 +0 7 14 21 28	

DuPont-35172 Tocina, Andalucia, 41340, Spain	Maize / MAS 58	1. 20 Feb 12 2. NR 3. 22 Jun 12 29 Jun 12 06 Jul 12 13 Jul 12 20 Jul 12	37.52 37.82	500 503	7.50 7.52	A1= 12 Jun 12 A2= 22 Jun 12	65 69	Maize forage	0.16 1.2 0.86 <u>0.77</u> 0.16 0.17	-0 +0 7 14 21 28	
DuPont-35172 Cervesina, 27050, Lombardia, Italy	Maize / Grizly	1. 27 Mar 12 2. NR 3. 23 Jul 12 30 Jul 12 06 Aug 12 13 Aug 12 20 Aug 12	38.13 38.74	510 517	7.48 7.49	A1 = 13 Jul 12 A2 = 23 Jul 12	67 69	Maize forage	0.19 1.9 0.30 <u>0.14</u> 0.081 0.057	-0 +0 7 14 21 28	
DuPont-35172 Graffignana 26813, Lombardia, Italy	Maize / Antiss	1. 25 Mar 12 2. NR 3. 23 Jul 12 30 Jul 12 06 Aug 12 13 Aug 12 20 Aug 12	37.82 39.04	502 521	7.49 7.49	A1 = 13 Jul 12 A2 = 23 Jul 12	67 69	Maize Forage	0.20 0.71 0.27 0.30 0.31 <u>0.34</u>	-0 +0 7 14 21 28	
DuPont-35172 Sandrans, Rhône Alpes, 01990, South France	Maize / Octet	1. 07 May 12 2. NR 3. 06 Aug 12 13 Aug 12 20 Aug 12 27 Aug 12 03 Sep 12	36.30 36.91	486 492	7.47 7.50	A1 = 27 Jul 12 A2 = 06 Aug 12	65 69	Maize Forage	0.099 0.84 0.36 <u>0.18</u> 0.12 0.087	-0 +0 7 14 21 28	

Remarks:
(a) According to CODEX Classification / Guide
(b) Only if relevant
(c) Year must be indicated
(d) Days after last application (Label pre-harvest interval, PHI, underline)
(e) Remarks may include: Climatic conditions; Reference to analytical method and information which metabolites are included

C.3.2. Nature and magnitude of residues in processed commodities

C.3.2.1. Nature of residues

C.3.2.1.1. High temperature hydrolysis study

Report:	**Clark, B., 2014**
Title:	**High Temperature Hydrolysis of ^{14}C-Indoxacarb (DPX-KN128) in Buffered Solutions at pH 4, 5 and 6**
Document No.:	**DuPont-35316**
Guidelines:	**OECD 507 (2007)**
GLP:	**Yes**

Study design

The purpose of this study was to investigate the nature of ^{14}C-indoxacarb residues under the temperature and conditions designed to simulate typical processing events: pasteurization at 90°C for 20 minutes at pH 4; baking, brewing, or boiling at 100°C for 60 minutes at pH 5; sterilization at 120°C for 20 minutes at pH 6.

Individual test systems were prepared in vials containing [phenyl (U)-^{14}C] [TF] and [indanone-1-^{14}C] [IN] DPX-KN128. DPX-KN128 is also known as indoxacarb. Each test vessel was filled, leaving a small headspace to allow for expansion during heat treatment, with approximately 20 mL of pH 4, pH 5, or pH 6 buffer containing approximately 0.04 – 0.09 g as/mL of solution. The hydrolysis test vessels were capped and heat treated at 90°C, 100°C, or 120°C. Control samples were incubated at ambient temperature alongside the heat treated vessels.
Samples were taken immediately after the addition of test substance to the buffered solutions and after exposure to the processing conditions, following a period of time for the samples to return to room temperature (approximately 20°C). Samples were analysed for total radioactivity and for DPX-KN128 and hydrolysis product(s).

Results

<u>Pasteurisation</u>
Approximately 95.3% of the indanone and 92.4% of the trifluoromethoxyphenyl-labelled parent compound were present at initiation increasing to 108.5% and 114.8% AR after 20 minutes at 90°C. Although some minor peaks appeared, no single peak exceeded a level > 10% of the total dose. Results are presented in table below.

Sample ID	Indoxacarb (%AR)	IN-KT413 (%AR)	IN-MK638 (%AR)	Sum Unassigned Peaks (%AR) [a]
Before high temperature:				
D-pH4-IN-A-T0	95.3%	0.0%	N/A	4.7%
D-pH4-TF-A-T0	92.4%	0.0%	0.5%	7.1%
After high temperature:				
D-pH4-IN-R1	108.5%	0.0%	N/A	6.5%
D-pH4-TF-R1	114.8%	1.7%	0.0%	8.0%

[a] Sum Unassigned consists of multiple components none of which exceeded 3.6%.

Note: Values are not rounded during spreadsheet calculations.

Table 3.1.1-7: Distribution and mass balance of radioactivity for pH 4 buffer test system [Indanone-^{14}C]and [4-Trifluoromethoxyphenyl-^{14}C]DPX-KN128

		Control 1-Indanone	Control 4-Trifluoro methoxyphenyl	90°C 1-Indanone	90°C 4-Trifluoro methoxyphenyl
DPX-KN128 (parent)	**% AR[a]**	**95.3**	**92.4**	**108.5**	**114.8**
IN-KT413	**% AR**	**0.0**	**0.0**	**0.0**	**1.7**
IN-MK638	**% AR**	**N/A**	**0.5**	**N/A**	**0.0**
Others[b]	**% AR**	**4.7**	**7.1**	**6.5**	**8.0**
Total % recovery[c]	**% AR**	**100.0**	**100.0**	**115.0**	**124.5**
Average % recovery	**% AR**	**100.0**		**119.7**	

nd = not detected

[a] AR = Applied Radioactivity or initial dose.

[b] Others = components not identified. Individual ^{14}C components detected by HPLC were less than 3.6% of the applied radioactivity.

[c] Recovery of administered radioactivity as determined by LSC analyses of the dosing solution.

Baking/boiling

Approximately 98.2% of the indanone and 99.1% of the trifluoromethoxyphenyl-labelled parent compound were present at initiation decreasing to 86.0 % and 78.7% AR after 60 minutes at 100°C. Although some minor peaks appeared, no single peak exceeded a level > 10% of the total dose.

Sample ID	Indoxacarb (%AR)	IN-KT413 (%AR)	IN-MK638 (%AR)	Sum Unassigned Peaks (%AR) [a]
Before high temperature:				
D-pH5-IN-A-T0	98.2%	0.0%	N/A	1.8%
D-pH5-TF-A-T0	99.1%	0.0%	0.0%	0.9%
After high temperature:				
D-pH5-IN-R1	86.0%	0.9%	N/A	3.6%
D-pH5-TF-R1	78.7%	0.0%	0.6%	2.9%

[a] Sum Unassigned consists of multiple components none of which exceeded 2.2%.

Note: Values are not rounded during spreadsheet calculations.

Table 3.1.1-8: Distribution and mass balance of radioactivity for pH 5 buffer test system [Indanone-^{14}C]and [4-Trifluoromethoxyphenyl-^{14}C]DPX-KN128

		Control 1-Indanone	Control 4-Trifluoro methoxyphenyl	100°C 1-Indanone	100°C 4-Trifluoro methoxyphenyl
DPX-KN128 (parent)	**% AR[a]**	**98.2**	**99.1**	**86.0**	**78.7**
IN-KT413	**% AR**	**0.0**	**0.0**	**0.9**	**0.0**
IN-MK638	**% AR**	**N/A**	**0.0**	**N/A**	**0.6**
Others[b]	**% AR**	**1.8**	**0.9**	**3.6**	**2.9**
Bottle Extraction	**% AR**	**N/A**	**N/A**	**9.9**	**5.8**
Total % recovery[c]	**% AR**	**100.0**	**100.0**	**100.4**	**87.9**
Average % recovery	**% AR**	**100.0**		**94.1**	

nd = not detected.

a AR = Applied Radioactivity or initial dose.

b Others = components not identified. Individual ^{14}C components detected by HPLC were less than 2.2% of the applied radioactivity.

c Recovery of administered radioactivity as determined by LSC analyses of the dosing solution.

<u>Sterilization</u>

Approximately 98.6% of the indanone and 100 % of the trifluoromethoxyphenyl-labelled parent compound were present at initiation decreasing to 46.5 % and 42.8% AR after 20 minutes at 120°C. **Metabolites IN-KT413, IN-MP819, IN-P0036 and IN-TMG00 were measured at a level ≥10% of the total dose.**

Results are presented in table below.

Sample ID	Indoxacarb (%AR)	IN-KT413 (%AR)	IN-MP819 (%AR)	IN-MK638 (%AR)	IN-P0036 (%AR)	IN-TMG00 (%AR)	Sum Unassigned Peaks (%AR) [a]
Before high temperature:							
D3-pH6-IN-A-T0	98.6%	0.5%	0.0%	N/A	N/A	0.0%	0.9%
D3-pH6-TF-A-T0	100.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
After high temperature:							
D3-pH6-IN-R1	46.5%	28.7%	9.9%	N/A	N/A	8.7%	11.3%
D3-pH6-TF-R1	42.8%	2.5%	8.5%	9.6%	16.1%	12.4%	3.1%

[a] Sum Unassigned consists of multiple components none of which exceeded 4.0%.

Note: Values are not rounded during spreadsheet calculations.

Table 3.1.1-9: Distribution and mass balance of radioactivity for pH 6 buffer test system [Indanone-^{14}C]and [4-Trifluoromethoxyphenyl-^{14}C]DPX-KN128

		Control 1-Indanone	Control 4-Trifluoro methoxyphenyl	120°C 1-Indanone	120°C 4-Trifluoro methoxyphenyl
DPX-KN128 (parent)	**% AR[a]**	**98.6**	**100.0**	**46.5**	**42.8**
IN-KT413	**% AR**	**0.5**	**0.0**	**28.7**	**2.5**
IN-MP819	**% AR**	**0.0**	**0.0**	**9.9**	**8.5**
IN-MK638	**% AR**	**N/A**	**0.0**	**N/A**	**9.9**
IN-P0036	**% AR**	**N/A**	**0.0**	**N/A**	**16.1**
IN-TMG00	**% AR**	**0.0**	**0.0**	**8.7**	**12.4**
Others[b]	**% AR**	**0.9**	**0.0**	**11.3**	**3.1**
Total % recovery[c]	**% AR**	**100.0**	**100.0**	**105.2**	**95.1**
Average % recovery	**% AR**	**100.0**		**100.1**	

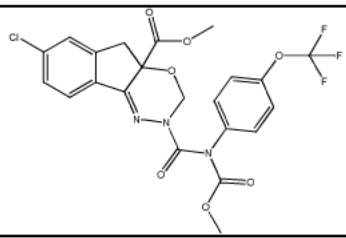
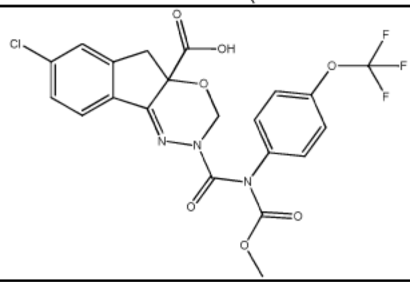
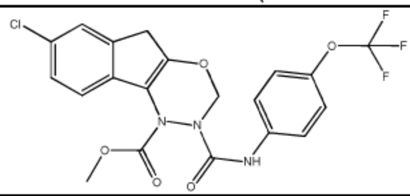
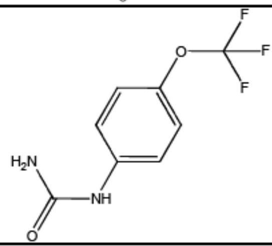
nd = not detected

[a] AR = Applied Radioactivity or initial dose.

[b] Others = components not identified. Individual ^{14}C components detected by HPLC were less than 4.0% of the applied radioactivity

[c] Recovery of administered radioactivity as determined by LSC analyses of the dosing solution.

Table 3.1.1-10: Identification of compounds from high temperature hydrolysis study

Common name/code ID No.	HPLC System #1 Retention Time (min)	CAS Chemical name	Chemical structure
DPX-KN128 (parent)	**30.0**	**Methyl (4aS)-7-chloro-2,5-dihydro-2-[[(methoxycarbonyl)[4-(trifluoromethoxy)phenyl]amino]carbonyl]indeno[1,2-e][1,3,4]oxadiazine-4a(3H)-carboxylate**	
IN-KT413	**14.5**	**Not available**	
IN-MP819	**31.0**	**Not available**	
IN-MK638	**16.5**	**Not available**	

(continued)

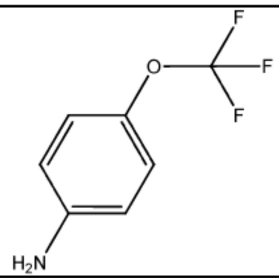
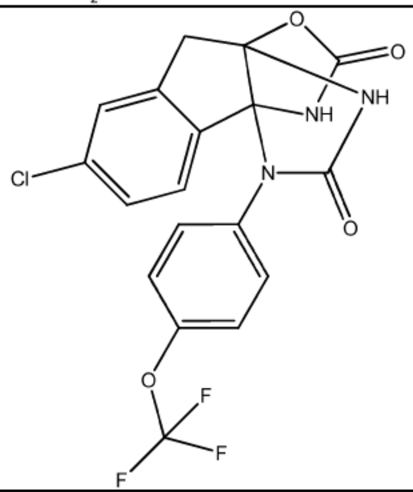
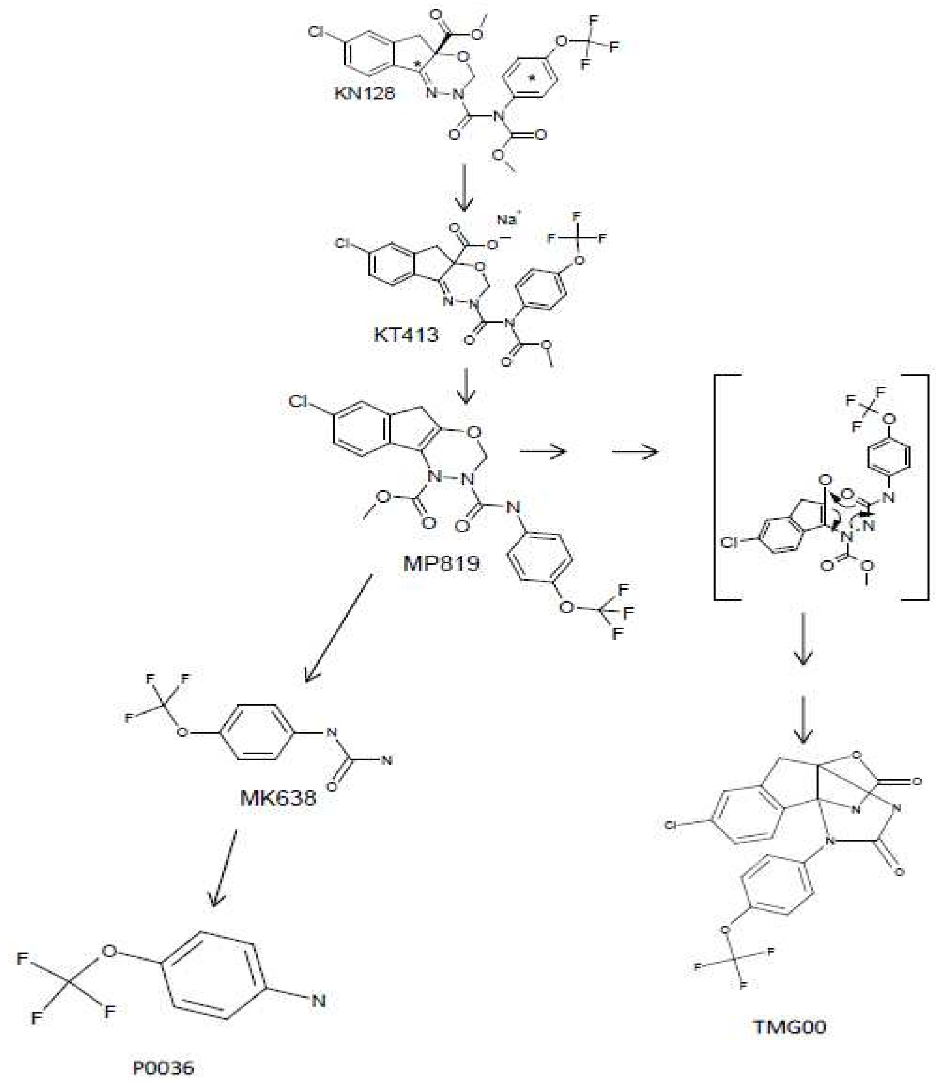
Common name/code ID No.	HPLC System #1 Retention Time (min)	CAS Chemical name	Chemical structure
IN-P0036	**19.0**	**Not available**	
IN-TMG00	**22.0**	**Not available**	

FIGURE 17 PROPOSED HYDROLYSIS PATHWAY FOR INDOXACARB (DPX-KN128) IN STERILE PH 6 BUFFER



C.3.2.2. Magnitude of residues

<u>Study 1 - Tomato</u>

Report:	KIIIA1 8.5.3/02, Spence C., 2014
Title:	Determination of the decline of residues of DPX-KN128 (indoxacarb) along with IN-K127 in tomato and tomato processed fractions following applications of DPX-MP062 30WG - southern Europe - 2012
Document No:	DuPont-35170
Guidelines:	OECD Test Guideline 508, OECD Guideline for the Testing of Chemicals: Magnitude of the Pesticide Residues in Processed Commodities, 03 October 2008 OECD Test Guideline 509, OECD Guideline for the Testing of Chemicals:

	Crop Field Trial, 07 September 2009 European Communities Guidelines for the Generation of Data Concerning Residues, as Provided in Annex II, Part A, Section 6 and Annex III, Part A, Section 8 of EC Commission Directive 91/414/EEC. SANCO/825/00 rev.8.1 (16/11/2010) Guidance Document on Pesticide Residue Analytical Methods
GLP	Yes

Acceptability	Deviations
Yes	None

Materials and method

During 2012, a total of three residue trials have been performed with indoxacarb on tomatoes. In these trials, tomatoes were treated with 6 applications of 37.5 g a.s./ha and raw tomatoes for processing studies were sampled 1 day after the last application. Sampled tomatoes were frozen and shipped until processing phases. Analyses were performed following the DFG method S19 (DuPont report AMR 4271-96).

Recovery studies carried out with the samples proved the residue was stable at least for 12 to 18 months.

Juice processing

Unwashed tomatoes were crushed and sieved. Wet pomace was weight and discard. Controlling the Brix degree, salt and citric acid were added to correct the pH. Juice was then pasteurized during one minute at 85°C. After cooling, juice was transferred into glass bottles and frozen.

Puree processing

Unwashed tomatoes were crushed and put in a double jacketed saucepan to be reduced. At a Brix degree of 12-13% reduction was stopped. Reduced tomatoes were put into an automatic siever to remove peels and seeds. Salt and citric acid were added to correct the pH. Puree was then transferred into glass bottles, and sterilized at 115-120°C for 10 minutes. After cooling, bottles were deep-frozen.

Paste processing

Unwashed tomatoes were crushed and put in a double jacketed saucepan to be concentrated. At a Brix degree of 24-26% concentration was stopped. Concentrated tomatoes were put into an automatic siever to remove peels and seeds. pH was corrected by adding salt and citric acid. Tomato paste was then transferred into glass bottles and sterilized at 115-120°C for 10 minutes. After cooling, bottles were deep-frozen.

Canned tomatoes

Unwashed tomatoes were blanched in boiling water then plunged into cold water to crack the peels. Blanched tomatoes were peeled. Peeled tomatoes were used to fill the cans and cans were sterilized at 115-120°C for 10 minutes. After cooling, cans were deep-frozen.

Ketchup processing

Unwashed tomatoes were crushed and put in a double jacketed saucepan to be reduced. At a Brix degree of 14-15% reduction was stopped. Reduced tomatoes were put into an automatic siever to remove peels and seeds. Ketchup was then prepared with tomato puree, brown sugar, cider vinegar and salt. Ketchup was put in glass bottles and sterilized at 115-120°C for 10 minutes. Bottles were deep-frozen.

Results

In this table, each line corresponds to one trial. There were 3 trials used for these processing studies

Table 3.1.1-11: Median transfer factor in tomato processed commodities

	Weight (kg)	indoxacarb residue level (mg/kg)	indoxacarb amount (g)	Percentage of residue level	Indoxacarb Transfer Factor	Median Transfer Factor
TOMATO JUICE						
Whole Fruits	10,09	0,042	0,42			
	10,01	0,04	0,40			
	10,49	0,032	0,34			
Juice to pasteurize	6,31	0,072	0,45	107%	**1,71**	**1,45**
	5,11	0,058	0,30	74%	**1,45**	
	4,65	0,033	0,15	46%	**1,03**	
TOMATO PUREE						
Whole Fruits	4,02	0,042	0,17			
	4,07	0,04	0,16			
	4,07	0,032	0,13			
Puree	1,03	0,069	0,07	42%	**1,64**	**1,64**
	1,15	0,059	0,07	42%	**1,48**	
	0,78	0,065	0,05	39%	**2,03**	
TOMATO PASTE						
Whole Fruits	6,05	0,042	0,25			
	6,08	0,04	0,24			
	6,04	0,032	0,19			
Paste	0,34	0,13	0,04	17%	**3,10**	**2,75**
	0,39	0,11	0,04	18%	**2,75**	
	0,56	0,061	0,03	18%	**1,91**	
CANNED TOMATOES						
Whole Fruits	3,06	0,042	0,13			
	3,07	0,04	0,12			
	3,05	0,032	0,10			
Peeled tomatoes	2,75	0,013	0,04	28%	**0,31**	**0,28**
	2,8	0,011	0,03	25%	**0,28**	
	2,58	0,007	0,02	19%	**0,22**	
TOMATO KETCHUP						
Whole	4,02	0,042	0,17			

Fruits	4,01	0,04	0,16			
	4,07	0,032	0,13			
Ketchup	1,03	0,086	0,09	52%	**2,05**	**1,83**
	0,76	0,073	0,06	35%	**1,83**	
	0,58	0,041	0,02	18%	**1,28**	

Except canned tomatoes, every processing phase induces an increase of the residue level in the processed commodity compared to the raw tomatoes. Thus, ketchup, paste, puree and juice processing are concentrating indoxacarb residue level. Only the canning process is results in a decrease of indoxacarb residue level compared to the raw tomatoes.

These results are not complying with the monograph data which show residue level decreasing in the tomato juice processing. However, monograph studies were performed in 1995 and data could be obsolete today.

Study 2 - Apple

Report:	Spence C., 2014
Title:	Determination of magnitude and decline of residues of DPX-KN128 (indoxacarb) along with IN-KN127 in apples and Apple processed fractions following applications of DPX-MP062 30WG – Europe –2012
Document No:	DuPont-35169
Guidelines:	OECD Test Guideline 508, OECD Guideline for the Testing of Chemicals: Magnitude of the Pesticide Residues in Processed Commodities, 03 October 2008 OECD Test Guideline 509, OECD Guideline for the Testing of Chemicals: Crop Field Trial, 07 September 2009 European Communities Guidelines for the Generation of Data Concerning Residues, as Provided in Annex II, Part A, Section 6 and Annex III, Part A, Section 8 of EC Commission Directive 91/414/EEC. SANCO/825/00 rev.8.1 (16/11/2010) Guidance Document on Pesticide Residue Analytical Methods
GLP	Yes

Materials and method

During 2012 a total of three residue trials were performed both in Northern and Southern Europe (UK and France) and allowed the author to study processing on apples.

Apples were treated with 4 applications of indoxacarb at a rate of 75 g a.s./ha. Samples of whole apples unwashed were taken 7 days after the last application and sampled fruits were shipped to prepare the processing phase. Analyses were performed following the DFG method S19 (DuPont report AMR 4271-96). Recovery studies carried out with the samples proved the residue was stable at least for 12 to 18 months.

Apple peeling
Unwashed apples were peeled with a knife. The skins were weighted and discarded.

Canned apples with skin

Unwashed apples were blanched directly in boiling water for 2 minutes (Apples from trial n°3 were peeled before this phase so the result is not considered). Blanched apples were cored and cut. Syrup of sugar and pH 3.5 were added. Two canned were then pasteurized at 90°C for one minute before being frozen (-18°C).

Apple jam
Unwashed apples were blanched in boiling water for 1 minute blanched apples were cored and peeled with a knife before being crushed. After the calculation of the Brix degree, white sugar was added. The mix was concentrated by heating until 62% Brix degree. Apple jam were then placed in glass bottles, and sterilized at 115/120°C during 10 minutes.

Apple juice
Unwashed apples were crushed in an electric crusher and pressed with a water press. Pomace remaining was placed in a plastic bag and frozen for analysis. Pectolytic enzymes were added to the juice fraction for depectinisation. The juice was left to settle until deposit had formed in the bottom of the tank. After racking the clear juice, it has been filtered over trimming plates. The juice was then pasteurized by heating at 85°C for 1 minute.

Apple sauce processing
Unwashed apples were blanched into boiling water for 2 minutes and were then crushed in an electric crusher. Crushed fruits were sieved to separate puree from peel and pips. Sugar was added and the puree was reduced by heating to obtain a 24% Brix degree. Apple sauce was transferred into a glass jar and was pasteurized at 115/120°C for ten minutes, before being frozen to analysis.

Results
In this table, each line corresponds to one trial. There were 3 trials used for these processing studies except for the canned apple processing where the processing plan was not respected in the third trial. Thus, the canned apple with skin part show only two lines corresponding to trials 1 and 2.

Table 3.1.1-12: Median transfer factor in apple processed commodities

	Weight (kg)	Indoxacarb residue level (mg/kg)	Indoxacarb amount (g)	Percentage of residue level	Indoxacarb Transfer Factor	Median Transfer Factor
APPLES PEELING						
Whole Fruits	2,01	0,1	0,20			
	3,64	0,12	0,44			
	3,55	0,065	0,23			
Peeled Fruits	1,69	0,008	0,01	7%	**0,08**	**0.18**
	3,2	0,021	0,07	15%	**0,18**	
	3,07	0,028	0,09	37%	**0,43**	
CANNED APPLE WITH SKIN						
Whole Fruits	2	0,1	0,20			
	2,63	0,12	0,32			
Canned apples	0,75	0,057	0,04	10%	**0,48**	**0.48**
	0,752	0,032	0,02	10%	**0,49**	

APPLE JAM						
Whole Fruits	2	0,1	0,20			
	3,51	0,12	0,42			
	3,65	0,065	0,24			
Obtained jam	1,49	0,003	0,00	2%	**0,03**	**0.03**
	2,83	0,003	0,01	2%	**0,03**	
	2,82	0,005	0,01	6%	**0,08**	
APPLE JUICE						
Whole Fruits	4,88	0,1	0,49			
	8,09	0,12	0,97			
	8,01	0,065	0,52			
Wet pomace	1,35	0,26	0,35	72%	**2,60**	**2.60**
	2,07	0,26	0,54	55%	**2,17**	
	2,08	0,18	0,37	72%	**2,77**	
Filtered juice	2,62	0,003	0,01	4%	**0,03**	**0.03**
	4,8	0,003	0,01	5%	**0,03**	
	4,61	0,005	0,02	54%	**0,09**	
APPLE SAUCE						
Whole Fruits	2	0,1	0,20			
	3,52	0,12	0,42			
	3,6	0,065	0,23			
Reduced apples	0,835	0,047	0,04	20%	**0,47**	**0.28**
	1,45	0,02	0,03	7%	**0,17**	
	1,61	0,018	0,03	12%	**0,28**	

Conclusion

On the basis of these available processing studies indoxacarb residue level decrease in most of the apple process. Apple juice and apple jam indoxacarb residue level decrease the most during the process with a TF of 0.03 compared to the raw fruit residue level. Peeling process decreases indoxacarb residue level from the raw fruit with a TF of 0.18. Canning and Sauce processing decrease indoxacarb residue level with, respectively, TF of 0.48 and 0.28.

Among processing phases, only wet pomace is increasing indoxacarb residue level compared to the raw fruit with a TF of 2.60.

C.3.3. Storage stability

C.3.3.1. Storage stability of residues in plant products

A storage stability study on poultry matrices has been conducted with DPX-MP062 and its metabolites and is summarised below.

Report:	CA, 6.1/01, Guinivan, R.A., Daussin, S., 2008
Title:	Recovery of DPX-MP062 and five metabolites from hen-derived matrices (whole eggs, muscle, fat and liver) after frozen storage.
Document No:	DuPont-19901
Guidelines:	OPPTS 860.1380
GLP	Yes

Materials and method

Samples of hen whole eggs, liver, fat, and muscle were fortified with DPX-MP062 and its 5 metabolites (IN-KB687, IN-KG433, IN-KT319, IN-JU873 and IN-JT333), each at a level of 0.20 mg/kg were stored at approximately -20°C and then analyzed at 5 intervals over 16 months. Residues were extracted from whole eggs using acidified ethyl acetate, and from liver, muscle and fat using acidified acetonitrile. Following partitioning and purification steps, the residues were quantified by LC/MS/MS analysis.

To prepare samples for fortification, the hen muscle, fat and liver samples were cut into small pieces and wheighted (10g) into 250 mL plastic centrifuge bottles. Egg samples were removed from the shells and and homogenized by rigorous mixing. Samples were stored at approximately -20°C until fortification.

For each stability time point, one control, two freshly fortified and two aged fortified samples of each matrix were analysed. Samples were removed from storage and analysed after approximate intervals of 0, 1, 3, 7, 11 and 16 months.

Results

The analyses of the control samples showed that no residues of any component were present.

The residues of all components showed no significant decrease (>30% as compared to the fortification concentration) in the different matrices after storage deep frozen for at least 16 months.

Table 3.1.1-13: Stability of DPX-MP062, IN-KB687, IN-KG433, IN-KT319, IN-JU873 and IN-JT333 residues in hen, whole egg, liver, fat, and muscle following storage at -20 ± 10°C

Commodity/Matrix	Approx. storage interval (Months)	Residue recovered in mg/kg			
		Stored (-20°C) forts[a]	Fresh forts[a]	Recovery of stored forts as a % of 0-day[c]	Recovery of fresh forts as a % of 0-day[d]

DPX-MP062							
Whole Egg	0	0.19	0.19	0.21	0.19	**97**	**103**
	1	0.18	0.17	0.16	0.19	**90**	**90**
	3	0.21	0.21	0.21	0.22	**108**	**110**
	7	0.17	0.17	0.16	0.17	**87**	**85**
	11	0.20	0.22	0.23	0.25	**108**	**123**
	16	0.23	0.18	0.23	0.20	**105**	**110**
Liver	0	0.20	0.19	0.19	0.18	**102**	**97**
	1	0.24	0.22	0.21	0.20	**121**	**108**
	3	0.19	0.20	0.21	0.22	**103**	**113**
	7	0.17	0.19	0.17	0.17	**95**	**89**
	11	0.24	0.22	0.24	0.24	**121**	**126**
	16	0.26	0.22	0.26	0.20	**126**	**121**
Fat	0	0.20	0.17	0.19	0.19	**99**	**101**
	1	0.20	0.23	0.16	0.16	**115**	**85**
	3	0.20	0.17	0.19	0.20	**99**	**104**
	7	0.17	0.18	0.17	0.15	**93**	**85**
	11	0.19	0.17	0.16	0.18	**96**	**91**
	16	0.20	0.20	0.17	0.16	**107**	**88**
Muscle	0	0.18	0.18	0.20	0.19	**96**	**104**
	1	0.23	0.23	0.19	0.17	**123**	**96**
	3	0.21	0.19	0.20	0.20	**107**	**107**
	7	0.16	0.15	0.15	0.15	**83**	**80**
	11	0.16	0.15	0.14	0.15	**83**	**77**
	16	0.23	0.21	0.25	0.19	**117**	**117**

Commodity/Matrix	**Approx. storage interval (Months)**	**Residue recovered in mg/kg**					
		Stored (-20°C) forts[a]		**Fresh forts[a]**		**Recovery of stored forts as a % of 0-day[c]**	**Recovery of fresh forts as a % of 0-day[d]**
IN-KB687							
Whole Egg	0	0.18	0.17	0.18	0.18	**99**	**101**
	1	0.18	0.17	0.17	0.17	**99**	**96**
	3	0.19	0.19	0.19	0.19	**107**	**107**
	7	0.17	0.17	0.16	0.18	**96**	**96**
	11	0.18	0.19	0.18	0.17	**104**	**99**
	16	0.19	0.21	0.20	0.20	**113**	**113**
Liver	0	0.18	0.18	0.19	0.20	**96**	**104**
	1	0.19	0.19	0.20	0.19	**101**	**104**
	3	0.19	0.19	0.20	0.20	**101**	**107**
	7	0.18	0.18	0.17	0.16	**96**	**88**
	11	0.20	0.20	0.21	0.18	**107**	**104**
	16	0.19	0.20	0.20	0.21	**104**	**109**
Fat	0	0.18	0.16	0.17	0.19	**97**	**103**
	1	0.18	0.19	0.18	0.17	**106**	**100**
	3	0.18	0.17	0.16	0.18	**100**	**97**
	7	0.18	0.16	0.17	0.16	**97**	**94**
	11	0.16	0.17	0.15	0.17	**94**	**91**
	16	0.17	0.19	0.17	0.17	**103**	**97**
Muscle	0	0.20	0.19	0.18	0.21	**100**	**100**
	1	0.18	0.19	0.20	0.19	**95**	**100**
	3	0.19	0.19	0.20	0.19	**97**	**100**
	7	0.20	0.18	0.17	0.16	**97**	**84**
	11	0.18	0.20	0.16	0.18	**97**	**87**
	16	0.19	0.21	0.22	0.20	**103**	**108**

Commodity/Matrix	Approx. storage interval (Months)	Residue recovered in mg/kg					
		Stored (-20°C) forts[a]		Fresh forts[a]		Recovery of stored forts as a % of 0-day[c]	Recovery of fresh forts as a % of 0-day[d]
IN-KG433							
Whole Egg	0	0.19	0.19	0.20	0.21	96	104
	1	0.20	0.19	0.19	0.19	99	96
	3	0.20	0.20	0.20	0.21	101	104
	7	0.18	0.18	0.19	0.19	91	96
	11	0.19	0.19	0.18	0.18	96	91
	16	0.23	0.20	0.22	0.18	109	101
Liver	0	0.21	0.20	0.18	0.20	**104**	**96**
	1	0.21	0.21	0.20	0.20	**106**	**101**
	3	0.21	0.20	0.22	0.22	**104**	**111**
	7	0.17	0.17	0.18	0.17	**86**	**89**
	11	0.20	0.19	0.22	0.21	**99**	**109**
	16	0.19	0.19	0.21	0.21	**96**	**106**
Fat	0	0.20	0.18	0.19	0.20	**99**	**101**
	1	0.19	0.21	0.19	0.18	**104**	**96**
	3	0.18	0.18	0.19	0.20	**94**	**101**
	7	0.17	0.17	0.18	0.18	**88**	**94**
	11	0.18	0.18	0.18	0.19	**94**	**96**
	16	0.23	0.23	0.21	0.22	**119**	**112**
Muscle	0	0.19	0.18	0.20	0.21	**95**	**105**
	1	0.21	0.21	0.20	0.19	**108**	**100**
	3	0.19	0.19	0.21	0.20	**97**	**105**
	7	0.15	0.16	0.17	0.17	**79**	**87**
	11	0.17	0.17	0.17	0.18	**87**	**90**
	16	0.17	0.19	0.21	0.20	**92**	**105**

Commodity/Matrix	Approx. storage interval (Months)	Residue recovered in mg/kg			
		Stored (-20°C) forts[a]	Fresh forts[a]	Recovery of stored forts as a % of 0-day[c]	Recovery of fresh forts as a % of 0-day[d]
IN-KT319					

Whole Egg	0	0.19	0.18	0.19	0.19	**99**	**101**
	1	0.20	0.18	0.19	0.19	**101**	**101**
	3	0.16	0.17	0.19	0.21	**88**	**107**
	7	0.19	0.18	0.18	0.20	**99**	**101**
	11	0.17	0.18	0.17	0.17	**93**	**91**
	16	0.20	0.19	0.19	0.20	**104**	**104**
Liver	0	0.20	0.19	0.20	0.20	**99**	**101**
	1	0.18	0.19	0.20	0.19	**94**	**99**
	3	0.19	0.18	0.20	0.20	**94**	**101**
	7	0.17	0.20	0.21	0.20	**94**	**104**
	11	0.20	0.19	0.21	0.19	**99**	**101**
	16	0.19	0.19	0.24	0.22	**96**	**116**
Fat	0	0.21	0.18	0.19	0.21	**99**	**101**
	1	0.20	0.19	0.18	0.17	**99**	**89**
	3	0.19	0.18	0.18	0.18	**94**	**91**
	7	0.20	0.19	0.18	0.19	**99**	**94**
	11	0.20	0.19	0.17	0.16	**99**	**83**
	16	0.23	0.23	0.21	0.22	**116**	**109**
Muscle	0	0.19	0.19	0.20	0.20	**97**	**103**
	1	0.20	0.19	0.19	0.18	**100**	**95**
	3	0.19	0.19	0.20	0.20	**97**	**103**
	7	0.15	0.16	0.16	0.16	**79**	**82**
	11	0.18	0.18	0.17	0.17	**92**	**87**
	16	0.20	0.20	0.22	0.22	**103**	**113**

Commodity/Matrix	**Approx. storage interval (Months)**	**Residue recovered in mg/kg**					
		Stored (-20°C) forts[a]		**Fresh forts[a]**		**Recovery of stored forts as a % of 0-day[c]**	**Recovery of fresh forts as a % of 0-day[d]**
IN-JU873							
Whole Egg	0	0.18	0.19	0.21	0.20	**95**	**105**
	1	0.20	0.16	0.17	0.20	**92**	**95**
	3	0.20	0.21	0.22	0.23	**105**	**115**
	7	0.18	0.17	0.19	0.17	**90**	**92**
	11	0.19	0.20	0.19	0.19	**100**	**97**
	16	0.23	0.20	0.22	0.16	**110**	**97**
Liver	0	0.21	0.21	0.19	0.20	**104**	**96**
	1	0.25	0.23	0.19	0.21	**119**	**99**
	3	0.20	0.20	0.22	0.24	**98**	**114**
	7	0.18	0.16	0.18	0.16	**84**	**84**
	11	0.21	0.20	0.22	0.21	**101**	**106**
	16	0.20	0.17	0.24	0.21	**91**	**111**
Fat	0	0.20	0.18	0.18	0.20	**100**	**100**
	1	0.21	0.22	0.18	0.16	**113**	**89**
	3	0.20	0.19	0.20	0.20	**103**	**105**
	7	0.20	0.17	0.19	0.17	**97**	**95**
	11	0.18	0.18	0.16	0.18	**95**	**89**
	16	0.21	0.21	0.17	0.19	**111**	**94**
Muscle	0	0.17	0.19	0.21	0.21	**92**	**108**
	1	0.24	0.24	0.19	0.18	**123**	**95**
	3	0.20	0.21	0.20	0.21	**105**	**105**
	7	0.16	0.16	0.17	0.17	**82**	**87**
	11	0.16	0.16	0.15	0.17	**82**	**82**
	16	0.17	0.19	0.22	0.18	**92**	**103**

Commodity/Matrix	Approx. storage interval (Months)	Residue recovered in mg/kg					
		Stored (-20°C) forts[a]		Fresh forts[a]		Recovery of stored forts as a % of 0-day[c]	Recovery of fresh forts as a % of 0-day[d]
IN-JT333							
Whole Egg	0	0.18	0.17	0.18	0.17	**100**	**100**
	1	0.22	0.21	0.22	0.22	**123**	**126**
	3	0.20	0.18	0.20	0.20	**109**	**114**
	7	0.16	0.15	0.16	0.16	**89**	**91**
	11	0.20	0.20	0.17	0.18	**114**	**100**
	16	0.26	0.20	0.28	0.19	**131**	**134**
Liver	0	0.17	0.20	0.18	0.17	**103**	**97**
	1	0.22	0.21	0.19	0.20	**119**	**108**
	3	0.21	0.22	0.19	0.23	**119**	**117**
	7	0.17	0.18	0.15	0.18	**97**	**92**
	11	0.22	0.22	0.21	0.19	**122**	**111**
	16	0.20	0.19	0.22	0.19	**108**	**114**
Fat	0	0.18	0.16	0.18	0.17	**99**	**101**
	1	0.19	0.22	0.18	0.20	**119**	**110**
	3	0.20	0.17	0.18	0.17	**107**	**101**
	7	0.18	0.16	0.17	0.16	**99**	**96**
	11	0.16	0.17	0.12	0.16	**96**	**81**
	16	0.19	0.19	0.17	0.18	**110**	**101**
Muscle	0	0.16	0.17	0.18	0.17	**97**	**103**
	1	0.18	0.22	0.18	0.14	**118**	**94**
	3	0.20	0.22	0.18	0.22	**123**	**118**
	7	0.15	0.14	0.15	0.14	**85**	**85**
	11	0.15	0.16	0.17	0.11	**91**	**82**
	16	0.18	0.15	0.20	0.17	**97**	**109**

[a] On Day-0 all fortifications are fresh. All fortifications are at 0.2 mg/kg (ppm)
[b] Average stored recovery / average fresh recovery
[c] Average of the 2 stored fortifications per matrix per interval / Average of the 4 fortifications done on day-0 per matrix with the result rounded to the nearest whole percent.
[d] Average of the 2 fresh fortifications per matrix per interval / Average of the 4 fortifications done on day-0 per matrix rounded to the nearest whole percent.
NOTE: All values from report were rounded to two significant figures before placement in this table. Calculations reported were calculated from values in this table.
Additional Note: Crossed out values were changes from original table due to transcription errors in the original table.

Conclusion

The data indicate that the residues of DPX-MP062 and its five metabolites are stable at approximately -20°C for at least 16 months in hen matrices (whole egg, liver, fat, and muscle).