FLONICAMID (282)

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EXPLANATION

Flonicamid is the ISO approved common name for N-cyanomethyl-4-(trifluoromethyl)nicotinamide (IUPAC). Flonicamid (CAS No. 158062-67-0) is systemic pyridine carboxamide insecticide with selective activity against Hemipterous pests.

Flonicamid was first evaluated for residues and toxicological aspects by the 2015 JMPR. The 2015 JMPR established an ADI for flonicamid of 0–0.07 mg/kg bw and concluded that an ARfD was unnecessary.

The 2015 JMPR also recommended the following residue definition for flonicamid:

Definition of the residue for compliance with the MRL and dietary risk assessment in plant commodities: *Flonicamid*.

Definition of the residue for compliance with the MRL and dietary risk assessment in animal commodities: *Flonicamid and the metabolite TFNA-AM, expressed as parent.*

The residue is not fat-soluble.

Flonicamid was last evaluated in 2017 for additional maximum residue levels. At the Fiftieth Session of the CCPR (2017), flonicamid was listed for consideration of additional uses by the 2019 Extra JMPR. The Meeting received information on registered use patterns, analytical method information, storage stability data and supervised residue trials on citrus fruits with product labels from the USA.

RESIDUE ANALYSIS

Analytical methods

Flonicamid was first evaluated by the JMPR in 2015. Supervised field trials submitted to the current Meeting were analysed using method IB-2014-JLW-002-01-01 for citrus fruits. Additionally, methods H13-87 and 09604 were submitted used to investigated residues in stored analytical samples.

Table 1 Overview of analytical methods for flonicamid and its metabolites

Method	Matrix	Extraction	Clean-Up	Detection, LOQ
H13-87	High acid (citrus fruit)	methanol	Diatomaceous earth column Florisil SPE	GC-MS (peel/pulp) Flonicamid: m/z: 174, LOQ: 0.04/0.01 mg/kg TFNA: m/z: 174, LOQ: 0.04/0.01 mg/kg TFNG: m/z: 174, LOQ: 0.04/0.01 mg/kg
09604	High acid (strawberry)	acetonitrile:water (1:1, v/v)	none	LC-MS/MS Flonicamid: m/z: 230 → 203, LOQ: 0.0094 mg/kg TFNA: m/z: 192 → 148, LOQ: 0.0153 mg/kg TFNA-AM: m/z: 190 → 148, LOQ: 0.0134 mg/kg TFNG: m/z: 249 → 203, LOQ: 0.0104 mg/kg
IB-2014-JLW-002-01-01 PSM-16-06-02[a]	High acid (citrus fruit & pulp) Citrus oil	acetonitrile:water (1:1, v/v)	Partitioning with ethyl acetate	LC-MS/MS Flonicamid: m/z: 230 → 203, LOQ: 0.01 mg/kg TFNA: m/z: 192 → 148, LOQ: 0.01 mg/kg TFNA-AM: m/z: 191 → 148, LOQ: 0.01 mg/kg TFNG: m/z: 249 → 203, LOQ: 0.01 mg/kg

[a] Study PSM-16-06-02 used the same method IB-2014-JLW-002-01-01

Method H13-87 (Japan Food Research Laboratories, 2002, H13-87):

The method involves extraction with methanol, followed by clean-up on a diatomaceous earth column. Residues were eluted with ethyl acetate: acetic acid (99:1, v/v). The extract was cleaned-up with a florisil column. For the analysis of TFNA and TFNG, an additional derivatisation step was applied involving methylation of TFNA and TFNG using trimethylsilyldiazomethane after residue elution from the diatomaceous earth column and prior to clean-up on the florisil column. Final extracts were analysed by GC-MS.

LOQs for flonicamid, TFNA, TFNA-AM and TFNG were 0.04 and 0.01 mg/kg, in each of orange peel and pulp, respectively.

Table 2 Recovery data for method H13-87 in oranges

Matrix	Fortification level (mg/kg)	n	Recovery in % (mean)	RSD (%)	Analyte
Orange pulp	0.01	6	95-105 (100)	3.6	Flonicamid
	0.4	6	89-104 (99)	5.7	m/z 174
	0.01	6	94-112 (105)	8.0	TFNA
	0.4	6	79-86 (82)	2.9	m/z: 174
	0.01	6	84-96 (91)	4.4	TFNG
	0.4	6	85-101 (94)	6.0	m/z: 174
Orange peel	0.04	6	91-113 (102)	8.3	Flonicamid
	2.0	6	80-92 (87)	5.5	m/z 174
	0.04	6	80-112 (96)	14.2	TFNA
	2.0	6	90-103 (96)	5.2	m/z: 174
	0.04	6	82-108 (92)	10.7	TFNG
	2.0	6	84-97 (92)	5.0	m/z: 174

Method 09604 (Samoil, K. S., 2010, 09604):

Extraction with acetonitrile:water (1:1) followed by solvent removal and reconstitution in acetonitrile:water (7:3). Extracts were analysed in LC/MS/MS.

Table 3 Recovery data for method 09604 in strawberries

Matrix	Fortification level (mg/kg)	n	Recovery in % (mean)	RSD (%)	Analyte
Strawberry	0.02	7	90-100 (95)	5	Flonicamid m/z 230.023 → 203.125
	0.2	13	85-105 (99)	6	
	2.0	3	95-110 (100)	9	
	0.02	6	80-100 (88)	8	TFNA m/z : 192.008 → 148.060
	0.2	13	85-100 (93)	4	
	2.0	3	90-100 (93)	6	
	0.02	6	70-90 (79)	7	TFNA-AM m/z: 190.982 → 148.095
	0.2	13	75-95 (84)	5	
	2.0	3	80-90 (83)	6	
	0.02	6	70-80 (73)	5	TFNG m/z: 249.039 → 203.059
	0.2	13	70-90 (82)	6	
	2.0	3	80-90 (83)	6	

Method IB-2014-JLW-002-01-01 (Wiedmann, J. L. and McDonald, J. A., 2015, IB-2014-JLW-002-01-01):

For the analysis of residues in orange, orange pulp, orange juice, grapefruit and lemon, homogenised samples were extracted with acetonitrile:water (1:1, v/v) and the combined extracts were evaporated and reconstituted in HCl and water. The extract was partitioned three times with ethyl acetate. The ethyl acetate extract was dried and reconstituted in acetonitrile for LC/MS/MS analysis.

For analysis of orange pulp, homogenised samples were soaked in water, followed by extraction with acetonitrile. The extract aliquot was dried and reconstituted in water. For analysis of flonicamid and TFNA-AM, NaOH was added. For analysis of TFNA and TFNG, concentrated HCl was added. After addition of NaOH or HCl, the extract was partitioned 3 times with ethyl acetate. The ethyl acetate extract was dried and reconstituted in acetonitrile for LC/MS/MS analysis.

For analysis of residues in orange oil, samples were dissolved in hexane and partitioned with acetonitrile:water (50:50, v/v). The acetonitrile:water layer was removed and the oil was partitioned again. Collected acetonitrile:water extracts were diluted and further analysed by LC/MS/MS.

LOQs for flonicamid, TFNA, TFNA-AM and TFNG were 0.01 mg/kg, irrespective of orange, orange juice, orange pulp and orange oil.

Table 4 Recovery data for method IB-2014-JLW-002-01-01 in citrus fruits and processed products thereof

Matrix	Fortification level (mg/kg)	n	Recovery in % (mean)	RSD (%)	Analyte
Orange fruits	0.01	3	68-89 (82)	14.4	Flonicamid
	0.1	3	93-94 (94)	0.7	m/z: 230 → 203
	0.01	3	62-82 (75)	15.4	TFNA
	0.1	3	77-84 (80)	4.2	m/z: 192 → 148
	0.01	3	67-80 (75)	9.6	TFNA-AM
	0.1	3	77-83 (81)	4.0	m/z: 191 → 148
	0.01	3	81-90 (87)	5.6	TFNG
	0.1	3	89-91 (90)	1.3	249 → 203
Orange juice	0.01	3	86-95 (89)	6.0	Flonicamid
	0.1	3	91-96 (94)	2.9	m/z: 230 → 203
	0.01	3	97-115 (106)	8.5	TFNA
	0.1	3	91-102 (96)	5.9	m/z: 192 → 148
	0.01	3	71-80 (75)	6.2	TFNA-AM
	0.1	3	70-78 (75)	5.4	m/z: 191 → 148
	0.01	3	78-85 (83)	4.8	TFNG
	0.1	3	79-88 (83)	5.8	249 → 203
Orange pulp	0.01	3	83-96 (90)	7.3	Flonicamid
	0.1	3	83-87 (85)	2.1	m/z: 230 → 203
	2.0	3	80-83 (81)	1.8	
	0.01	3	77-113 (92)	20.7	TFNA
	0.1	3	78-85 (81)	4.8	m/z: 192 → 148
	0.01	3	79-82 (80)	2.0	TFNA-AM
	0.1	3	76-79 (78)	1.9	m/z: 191 → 148
	0.01	3	68-80 (73)	8.5	TFNG
	0.1	3	71-85 (76)	10.7	249 → 203
Orange oil	0.01	3	74-82 (79)	5.2	Flonicamid
	0.1	3	77-86 (82)	5.5	m/z: 230 → 203
	0.01	3	79-90 (84)	6.7	TFNA
	0.1	3	69-82 (76)	8.5	m/z: 192 → 148
	0.01	3	65-85 (75)	13.6	TFNA-AM
	0.1	3	71-80 (76)	6.1	m/z: 191 → 148
	0.01	3	68-91 (79)	15.0	TFNG
	0.1	3	78-93 (85)	8.6	249 → 203

Table 5 Recovery data of analytes in oranges for method used in PSM-16-06-02

Matrix	Fortification level (mg/kg)	n	Recovery in % (mean)	RSD (%)	Analyte
Orange fruits	0.01	3	80-103 (93)	12.4	Flonicamid
	0.1	3	82-85 (84)	2.0	m/z: 230 → 203
	0.01	3	81-87 (83)	4.1	TFNA
	0.1	3	86-91 (89)	2.6	m/z: 192 → 148
	0.01	3	81-93 (85)	8.0	TFNA-AM
	0.1	3	78-81 (79)	1.7	m/z: 191 → 148
	0.01	3	74-85 (79)	7.3	TFNG
	0.1	3	76-90 (82)	8.7	249 → 203

Stability of pesticide residues in stored analytical samples

The Meeting received two new studies investigating the storage stability of flonicamid and its metabolites in citrus fruits and strawberries.

In the first storage stability study (Japan Food Research Laboratories, 2002, H13-87), flonicamid, TFNA and TFNG in orange peel and orange pulp were investigated at -20 °C for at a period of 16 months (480-486 days).

In the second study (Samoil, K. S., 2010, 09604), flonicamid, TFNA-AM, TFNA and TFNG were investigated in strawberry for 460 days in storage at -20 °C.

In the following table, the recovered residues after storage are summarized.

Table 6 Storage stability of flonicamid in orange and strawberry matrices

Matrix	Storage in months (days)	Fortification level (mg/kg)	% remaining	Mean % remaining	Procedural recovery in % (n=1)
Flonicamid					
Orange pulp	14 (429) 16 (480)	1	89, 84 90, 84	86 87	Not reported
Orange peel	14 (435) 16 (486)	2	101, 93 96, 96	97 96	Not reported
Strawberry	15 (460)	0.2	95, 100, 100	98	100
TFNA					
Orange pulp	14 (429) 16 (480)	1	85,80 77, 77	82 77	Not reported
Orange peel	14 (435) 16 (486)	2	97, 92 94, 90	94 92	Not reported
Strawberry	15 (460)	0.2	85, 90, 90	88	90
TFNA-AM [a]					
Strawberry	15 (460)	0.2	80, 85, 85	83	80
TFNG					
Orange pulp	14 (429) 16 (480)	1	88, 86 92, 89	87 90	Not reported
Orange peel	14 (435) 16 (486)	2	96, 87 90, 89	92 90	Not reported
Strawberry	15 (460)	0.2	75, 80, 80	78	80

[a] No study of storage stability on TFNA-AM on orange pulp and peel

USE PATTERN

Flonicamid is intended for insecticidal use in citrus fruits by a foliar spray application in the USA.

Table 7 List of uses of flonicamid

Crops or crop groups	Country	Application detail					
		kg ai/ha	Growth stage at last treatment	Indoor/ Outdoor	No.	Interval in days	Pre harvest interval (PHI) in days
Citrus fruits	USA	0.1	At infestation	Outdoor	3	7	0

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as flonicamid equivalents. When residues were not detected they are shown as below the LOQ, e.g., < 0.01 mg/kg. Application rates, spray concentrations and mean residue results have generally been rounded to the even with two significant figures. HR and STMR values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. These results are underlined.

Laboratory reports included method validation including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for % recovery.

Flonicamid - supervised residue trials

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Lemons	Outdoor	Foliar	USA	8
Oranges	Outdoor	Foliar	USA	9
Grapefruits	Outdoor	Foliar	USA	10

Table 8 Residues of flonicamid following spray treatment on lemon trees

Location, year, trial reference, variety	Application					Matrix	DAT	Residues (mg/kg)	
	Spray Volume (L/ha)	Rate (kg ai/ha)	kg ai/hL	No.	RTI in days			Flonicamid	Mean
USA GAP: 3 × 0.1 kg ai/ha, PHI: 0 days									
Winter Garden, FL, USA 2014 IB-2014-JLW-002-19 Bearss	1100-1200	0.098-0.1	0.009	3	7-8	Lemon	0	0.22, 0.29	<u>0.25</u>
Newman, CA, USA 2014 IB-2014-JLW-002-20 Eureka	1200	0.095	0.008	3	7	Lemon	0	0.99, 0.43	<u>0.71</u>
Sanger, CA, USA 2015 IB-2014-JLW-002-21 Lisbon	1200	0.094-0.095	0.008	3	7	Lemon Lemon Lemon Lemon	0 3 7 10	0.17, 0.23 0.25, 0.20 0.13, 0.17 0.15, 0.16	0.20 <u>0.22</u> 0.15 0.15
Richgrove, CA, USA 2014 IB2014-JLW-002-22 Lisbon	1400	0.099-0.1	0.007	3	7	Lemon	0	0.12, 0.14	<u>0.13</u>
Yuma, AZ, USA 2014 IB-2014-JLW-002-23 Lisbon	1600	0.097-0.099	0.006	3	7	Lemon	0	0.10, 0.16	<u>0.13</u>

DAT: days after treatment

Table 9 Residues of flonicamid following spray treatment on orange trees

Location, year, trial reference, variety	Application					Matrix	DAT	Residues (mg/kg)	
	Spray Volume (L/ha)	Rate (kg ai/ha)	kg ai/hL	No.	RTI in days			Flonicamid	Mean
USA GAP: 3 × 0.1 kg ai/ha, PHI: 0 days									
Lake Wales, FL, USA 2014, IB-2014-JLW-002-01 Valencia	940	0.1	0.011	3	6-8	Orange	0	0.29, 0.18	0.24
Winter Garden, FL, USA I-2014-JLW-002-02 2014 Hamlin &	1200	0.1	0.009	3	7-8	Orange	0	0.13, 0.11	0.12
IB-2014-JLW-002-05 Easy Gold [1]	1200	0.1	0.008-0.009	3	7-8	Orange	0	0.23, 0.23	0.23
Umatilla, FL, USA 2014 IB-2014-JLW-002-03 Navel	1200	0.1	0.008-0.009	3	7	Orange Orange Orange Orange	0 3 7 10	0.049, 0.053 0.025, 0.028 0.018, 0.020 0.014, 0.017	0.051 0.027 0.019 0.016
De LeonSpring, FL, USA 2014 IB-2014-JLW-002-04 Navel	960-1000	0.1	0.01	3	7	Orange	0	0.11, 0.10	0.10
Winter Garden, FL, USA 2014 IB-2014-JLW-002-06 Hamlin	1700	0.1	0.006	3	7	Orange	0	0.15, 0.15	0.15
Oviedo, FL, USA 2014 IB-2014-JLW-002-07 Valencia	1400	0.1	0.007	3	7	Orange	0	0.11, 0.13	0.12
Bithlo, FL, USA 2014 IB-2014-JLW-002-08 Valencia	1400	0.099-0.1	0.007	3	7	Orange	0	0.12, 0.096	0.11
Raymondville, TX, USA 2014 IB-2014-JLW-002-09 Marrs	1000	0.1	0.01	3	7	Orange	0	0.066, 0.056	0.061
Porterville, CA, USA 2014 IB-2014-JLW-002-10 Valencia	1400	0.099	0.007	3	7-8	Orange	0	0.32, 0.12	0.22
Sanger, CA, USA 2015 IB-2014-JLW-002-11 Valencia	1400	0.094-0.095	0.007	3	6-8	Orange Orange Orange Orange	0 3 7 10	0.19, 0.25 0.19, 0.14 0.11, 0.092 0.066, 0.088	0.22 0.17 0.10 0.077
Orland, CA, USA 2014 IB-2014-JLW-002-12 Navel	940	0.1	0.011	3	7	Orange	0	0.059, 0.068	0.064
De Leon Spring, FL, USA 2016 Trial 01 PSM-16-06-02 Valencia	1000	0.099-0.1	0.01	3	6-8	Orange Orange Orange Orange	0 7 14 21	0.097, 0.078 0.075, 0.083 0.018, 0.019 <0.01, 0.015	0.088 0.079 0.018 0.012
Oak Hill, Florida, USA 2016 Trial 02 PSM-16-06-02 Valencia	1000	0.099-0.1	0.01	3	6-8	Orange Orange Orange Orange	0 7 14 21	0.081, 0.085 0.055, 0.086 0.017, 0.021 0.018, 0.017	0.083 0.070 0.019 0.018
Fresno, CA, USA 2016	940-950	0.1	0.011	3	7	Orange Orange	0 7	0.14, 0.21 0.075, 0.072	0.18 0.074

Location, year, trial reference, variety	Application					Matrix	DAT	Residues (mg/kg)	
	Spray Volume (L/ha)	Rate (kg ai/ha)	kg ai/hL	No.	RTI in days			Flonicamid	Mean
Trial 03 PSM-16-06-02 Navel						Orange Orange	14 21	0.072, 0.061 0.053, 0.040	0.066 0.046

DAT: days after treatment1: Same location, similar treatment dates, not considered independent

Table 10 Residues of flonicamid following spray treatment on grapefruit trees

Location, year, trial reference, variety	Application					Matrix	DAT	Residues (mg/kg)	
	Spray Volume (L/ha)	Rate (kg ai/ha)	kg ai/hL	No.	RTI in days			Flonicamid	Mean
USA GAP: 3 × 0.1 kg ai/ha, PHI: 0 d									
Lake Wales, FL, USA 2014 IB-2014-JLW-002-13 Flame	970	0.096-0.1	0.01	3	7-8	Grapefruit	0	0.13, 0.14	0.13
Umatilla, FL, USA 2014 IB-2014-JLW-002-14 Ray Red	1200	0.1	0.009	3	6-8	Grapefruit	0	0.031, 0.036	0.034
Oak Hill, Florida, USA 2014 IB-2014-JLW-002-15 Ray Red	1400	0.1	0.007	3	7	Grapefruit Grapefruit Grapefruit Grapefruit	0 3 7 10	0.061, 0.079 0.049, 0.046 0.016, 0.016 0.015, 0.011	0.070 0.048 0.016 0.013
Raymondville, TX, USA 2014 IB-2014-JLW-002-16 Rio Red	1000	0.1	0.01	3	7	Grapefruit	0	0.061, 0.053	0.057
Sanger, CA, USA 2014 IB-2014-JLW-002-17 Ruby Red	1400	0.093-0.094	0.007	3	7	Grapefruit	0	0.017, 0.021	0.019
Porterville, CA, USA 2014 IB-2014-JLW-002-18 Mellogold	1400	0.099-0.1	0.007	3	7	Grapefruit	0	0.077, 0.081	0.079

DAT: days after treatment

FATE OF RESIDUES DURING PROCESSING

Residues after processing

The fate of flonicamid during processing of raw agricultural commodity (RAC) was investigated in one supervised field trial on orange fruits. As a measure of the transfer of residues into processed products, a processing factor was used, which is defined as:

Processing factor = Residue in processed product (mg/kg) ÷ Residue in raw agricultural commodity (mg/kg)

If residues in the RAC were below the LOQ, no processing factor could be derived. In case of residues below the LOQ, but above the LOD in the processed product, the numeric value of the LOQ was used for the calculation. If residues in the processed product were below the LOD, the numeric value of the LOQ was used for the calculation but the PF was expressed as "less than" (e.g. < 0.5).

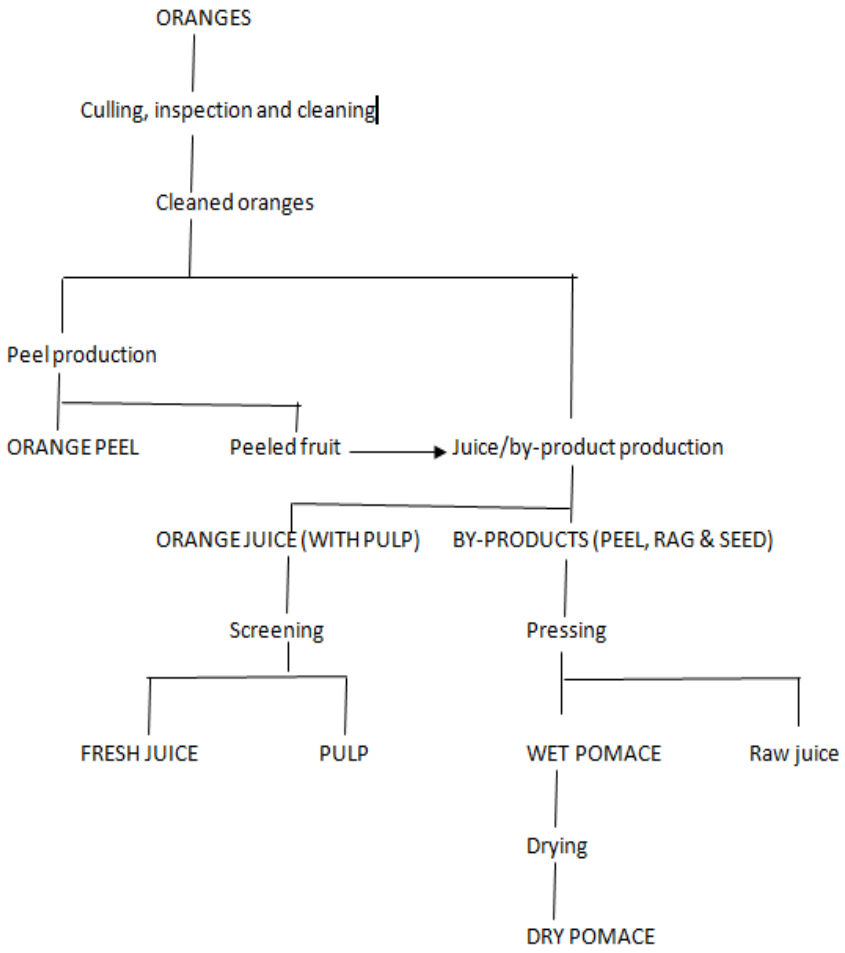
In the study IB-2014-JLW-002-01-01 (Wiedmann and McDonald 2015), only one supervised trial included a processing study. In the trial, orange trees were treated three times at minimum retreatment intervasl of 7 days, with each application of 0.5 kg ai/ha and harvested at 0 days. Orange fruits were processed into orange juice, dried orange pulp and orange oil.

To produce orange juice, whole cleaned orange were processed in a juicer machine to obtain juice and by-products (peel, rag and seed). Raw juice was sieved and pasteurised by heating.

To produce dried orange pulp (dry pomace), by-products from juice production were chopped and dewatered in hydraulic press. Solids were dried until moisture was <12%.

To produce peel oil, cleaned oranges were peeled with a modified abrasion peeler. During peeling, a spray of water was used to collect the peel oil being released. The liquid solution was sieved. The liquid was placed into a cooler and allow to separate into juice and an oil/water emulsion. Separation of peel oil from emulsion was achieved using centrifugation.

The flow chart of the processing study is shown in the following diagram.



In the following table the processing factors derived from the supervised field trial results are summarized:

Table 11 Processing factors for flonicamid in processed orange fruits based on one supervised field trial data

Trial, Location	Application	Matrix	Flonicamid in mg/kg	PF
IB-2014-JLW-002-10 Porterville, CA, USA	3 × 0.1 kg ai/ha, 7 d interval, 0 DALA	Orange (RAC) Orange, juice Orange, dried pulp Orange oil	0.544 <0.01 0.987 nd	- <0.02 1.81 <0.01

APPRAISAL

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The 2015 JMPR also recommended the following residue definition for flonicamid:

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The residue is not fat-soluble.

Flonicamid was last evaluated in 2017 for additional maximum residue levels. At the Fiftieth Session of the CCPR (2017), flonicamid was listed for consideration of additional uses by the 2019 Extra JMPR. The Meeting received information on registered use patterns, analytical method information, storage stability data and supervised residue trials on citrus fruits with product labels from the USA

Methods of analysis

The current Meeting received additional concurrent recovery information for the analysis of flonicamid in plant matrices.

Methods H13-87 and 09604 were used in the investigation of the storage stability in high acid matrices. In H13-87 method, methanol was used as extraction solvent. Residues were determined by GC-MS and individual LOQs of 0.01 mg/kg were validated for parent flonicamid and each of its metabolites TFNA, and TFNG in orange pulp. In orange peel, individual LOQs of 0.04 mg/kg were validated for parent flonicamid and each of its metabolites TFNA, and TFNG. The 09604 method involves extraction of the residue with acetonitrile:water (1:1). Determination was performed by LC-MS/MS. Based on concurrent recovery data, individual LOQs of 0.01 mg/kg were validated for parent flonicamid and each of its metabolites TFNA, TFNA-AM and TFNG.

Method IB-2014-JLW-002-01-01 was used for residue determination of field crop samples from the supervised trials. The method involves extraction of residues with acetonitrile:water (1:1; v/v). Determination was performed by LC-MS/MS and supported with concurrent recovery data suggesting individual LOQs of 0.01 mg/kg for parent flonicamid and each of its metabolites TFNA, TFNA-AM and TFNG.

Stability of residues in stored analytical samples

The Meeting received information on the storage stability of parent flonicamid and its metabolites TFNA, TFNA-AM and TFNG in high acidic matrices (citrus fruits and strawberries).

Flonicamid, TFNA and TFNG in orange peel and orange pulp were found to be stable in storage at -20 °C for at least 16 months (480–486 days). Flonicamid, TFNA, TFNA-AM and TFNG in strawberry were found to be stable in storage at -20 °C for at least 15 months (460 days).

Among all the samples from supervised trials in storage, the longest storage duration before analysis was 268 days. The Meeting concluded that all the residue results from supervised trials were analysed within acceptable storage intervals.

Results of supervised residue trials on crops

Flonicamid is registered for use on citrus fruits in the USA with a maximum GAP involving three foliar sprays of 0.1 kg ai/ha each (7 day interval), a maximum seasonal rate of 0.3 kg ai/ha and a PHI of 0 days. The Meeting received supervised trial data for applications of flonicamid on citrus fruits conducted in the USA.

Lemons and Limes

Corresponding supervised field trials conducted in the USA on lemons matching the GAP were submitted.

Residues of flonicamid in lemon fruits were (n=5): 0.13(2), 0.22 0.25 and 0.71 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg and a STMR value of 0.22 mg/kg for flonicamid in the subgroup lemons and limes.

Oranges, Sweet, Sour

Corresponding supervised field trials conducted in the USA on oranges matching the GAP were submitted.

Residues of flonicamid in orange fruits were (n=14): 0.051, 0.061, 0.064, 0.083, 0.088, 0.10, 0.11, 0.12, 0.15, 0.18, 0.22(2), 0.23 and 0.24 mg/kg.

The Meeting noted that the US GAP involves treatment of all citrus fruit and decided to use oranges as representative commodity for the subgroup of oranges, sweet, sour.

The Meeting estimated a maximum residue level of 0.4 mg/kg and a STMR value of 0.115 mg/kg for flonicamid in the subgroup oranges, sweet, sour.

Pummelos and Grapefruits

Corresponding supervised field trials conducted in the USA on grapefruits matching the GAP were submitted.

Residues of flonicamid in grapefruits were (n=6): 0.019, 0.034, 0.057, 0.070, 0.079 and 0.13 mg/kg.

The Meeting noted that the US GAP involves treatment of all citrus fruit and decided to use graperuits as representative commodity for the subgroup of pummelos and grapefruit.

The Meeting estimated a maximum residue level of 0.3 mg/kg and a STMR value of 0.0635 mg/kg for flonicamid in the subgroup pummelos and grapefruit.

The Meeting noted that data from mandarins were not available therefore the Meeting did not consider a recommendation for the citrus group.

Fate of residues during processing

The fate of flonicamid residues has been examined simulating commercial processing of orange fruits.

Estimated processing factors for the commodities considered at this Meeting are summarized below.

Raw commodity	Processed commodity	Flonicamid			
		Individual processing factors	Mean or best estimate processing factor	STMR or STMR-P (mg/kg)	Maximum residue level (mg/kg)
Citrus fruits	Lemon (RAC)			0.22	1.5
	Juice	0.02 (from orange)	0.02	0.0044	-
	Dried pulp	1.8 (from orange)	1.8	0.396	3
	Oil	0.01 (from orange)	0.01	0.0022	-

The Meeting estimated a maximum residue level of 3 mg/kg for citrus pulp, dry on the basis of the processing factor of 1.8 for orange pulp, dry and the maximum residue level for lemon of 1.5 mg/kg.

Residues in animal commodities

The Meeting recalculated the livestock dietary burden based on the uses considered by the current and previous Meeting on the basis of diets listed in the 2016 edition of FAO Manual Appendix IX (OECD Feedstuff Table). The addition of citrus pulp, dry does not add significantly to the maximum and mean dietary burdens of up to 27.7 ppm and 15.3 ppm calculated by the 2016 JMPR. The Meeting confirmed its previous recommendations for flonicamid in animal commodities.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue for compliance with the MRL and dietary risk assessment for plant commodities: *Flonicamid*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *Flonicamid and the metabolite TFNA-AM, expressed as parent*

The residue is not fat-soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FC 002	Lemons and limes, Subgroup of	1.5	-	0.22	-
FC 0004	Oranges, Sweet, Sour, Subgroup of	0.4	-	0.115	-
FC 005	Pummelos and Grapefruit, Subgroup of	0.3	-	0.0635	-
AB0001	Citrus pulp, dry	3 (dw)	-	Median: 0.396	-
JF 0001	Citrus juice			0.0044	
OR 0001	Citrus oil, edible			0.0022	

(dw) – dry weight

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for flonicamid is 0–0.07 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for flonicamid were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2019 Extra JMPR Report.

The IEDIs ranged from 1–10% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of flonicamid from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2015 JMPR decided that an ARfD for flonicamid was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of flonicamid from the uses considered is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institute, Report reference
H13-87	Anonymous	2002	Crop Residue Analysis Report. Japan Food Research Laboratories, Japan; report no. H13-87, GLP: no, Unpublished
09604	Samoil, K. S.	2010	Flonicamid: Magnitude of the residue on strawberry. IR-4, USA; report no. 09604, GLP: yes, Unpublished
IB-2014-JLW-002-01-01	Wiedmann, J. L. and McDonald, J. A.	2015	Magnitude of residues of Flonicamid on Citrus – USA in 2014. ISK Biosciences Corporation, USA; report no. IB-2014-JLW-002-01-01, GLP: yes, Unpublished
PSM-16-06-02	Schreier, T.	2017	Magnitude of The Residue of Beleaf 50 SG Insecticide in/on Orange, Precision Study Management LLC, USA; report no. PSM-16-06-02, GLP: yes, Unpublished