

ACETAMIPRID (246)

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EXPLANATION

Acetamiprid is a neonicotinoid insecticide with contact and stomach action against a range of *Hemiptera*, *Thysanoptera* and *Lepidoptera* plant pests, acting as an agonist of the nicotinic acetylcholine receptor in the insect central nervous system. It exhibits translaminar activity in plants and is authorised for use in North America, Europe and in a number of countries in Asia and the Pacific.

Acetamiprid was evaluated for the first time by the 2011 JMPR, where an ADI of 0–0.07 mg/kg bw/day and an ARfD of 0.1 mg/kg bw were established, and maximum residue levels were recommended for a range of plant and animal commodities. In 2012, JMPR reconsidered acute dietary risks from maximum residue levels recommended for leafy vegetables (except spinach) and spinach and then withdrew them. At the 2015 JMPR additional uses were evaluated.

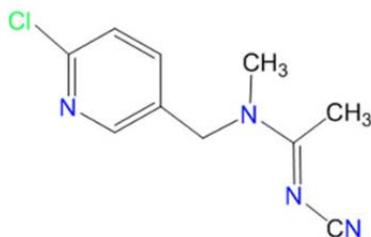
Acetamiprid was listed by the 48th session of the CCP (2016) for evaluation of additional MRLs by the 2017 JMPR. Iran submitted a use pattern and supervised residue trials on pistachio, which were evaluated by the present Meeting.

The residue definition for plant commodities, for compliance with MRLs and estimation of dietary exposure, is *acetamiprid*. The residue definition for animal commodities, for compliance with MRLs and estimation of dietary exposure, is the *sum of acetamiprid and desmethyl-acetamiprid, expressed as acetamiprid*.

The residue is not fat-soluble.

IDENTITY

ISO common name:	Acetamiprid
Chemical name	
IUPAC:	E)-N ¹ -[(6-chloro-3-pyridyl)methyl]-N ² -cyano-N ¹ -methyl acetamidine
CAS:	(E)-N-[(6-chloro-3-pyridinyl)methyl]-N'-cyano-N-methylethanimidamide
CAS Registry No:	135410-20-7
CIPAC No:	649
Structural formula:	



Molecular formula:	C ₁₀ H ₁₁ ClN ₄
Molecular weight:	222.68

RESIDUE ANALYSIS

Analytical methods used in study reports in plant commodities

In an open-literature study [Christia *et al*, 2015] the multi-residue QuEChERS method using liquid chromatography coupled with tandem mass spectrum detection (LC-MS/MS) has been validated at the LOQ of 0.01 mg/kg for determining acetamiprid residues in different matrices with dry, high water, acid, oily and high sugar content. The QuEChERS (EN 15662), which was evaluated by JMPR 2011, was used with small modifications for the determination of acetamiprid residues in pistachio in supervised residue trials conducted in Iran in 2015–2016.

After removal of the shells and separating kernels pistachio samples were fortified with acetamiprid solutions for the calibration samples and with the internal standard. The samples were mixed with 10 mL of water and 10 mL of acetonitrile. Next, the extraction salts based on protocol were added to the samples. Clean-up was carried out with MgSO₄, PSA and GCB salts then, the final extracts were analysed by High-Performance Liquid Chromatography system was coupled to a hybrid triple quadrupole linear ion trap mass spectrometer. The modification consisted of an additional step where the final supernatant was transferred to a new tube, dried under N₂ gas and reconstituted in the mobile phase before injection into the system. The above method was validated to determine and quantify acetamiprid in pistachio LC-MS/MS. Transition ions for acetamiprid was m/z = 223.16 → 126.30 for quantification and m/z = 223.16 → 90.20 for confirmation. The LOQ for the acetamiprid was 0.01 mg/kg. Validation data are summarised in Table 1. In each study, the linearity of the detector response was confirmed over the range 5–200 µg acetamiprid/L using six calibration solutions. In the studies, correlation coefficients (*R*²) were found to be at least 0.996. Relative standard deviation (RSD) was less than 12% showing that repeatability was also acceptable. At fortification levels of 0.01, 0.1 and 0.2 mg /kg, the mean recoveries (n=3) ranged within 95–98% (RSD < 11%) in sample matrix of pistachio.

Table 1 Validation data of QuEChERS (EN 15662) for acetamiprid in pistachio

commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery		RSD _r	control samples mg/kg (n)	calibration	reference, method
				mean	range				
pistachio	0.01	0.01	3	96	88-100	7	not reported	Linear R > 0.996 (n=6)	QuEChERS (EN 15662) with slight modifications
		0.1	3	98	94-100	8			
		0.2	3	95	91-98	3			

Stability of pesticide residues in stored analytical samples

No new data on stability of the residues in stored analytical pistachio samples were submitted.

The 2011 JMPR evaluated the stability of residues in stored analytical samples for a range of representative substrates covering those with a high water content (apple, cabbage, cucumber, grape, lettuce, tomato) a high starch content (potato), a high oil content (cotton seed) and a high acid content (orange) and their processed fractions, stored at ambient temperatures and at freezer temperatures.

In samples fortified with acetamiprid at levels of 0.5 mg/kg or 0.1 mg/kg and stored at either at room temperature for up to 7 days or frozen for up to 12 months (16 months for lettuce and 8 months for potatoes), residues were stable in all samples at the end of the storage periods, both at ambient temperature and under freezer conditions.

The (frozen) storage intervals between sampling and analysis were less than one month and therefore covered by the storage periods in these stability studies.

USE PATTERN

A copy and an English translation of acetamiprid label from Iran were made available to the Meeting. The use is summarised in Table 2.

Table 2 Registered pre-harvest uses of acetamiprid on pistachio

Crop	Country	Form	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number	
pistachio	Iran	SP 20%	high volume spraying ^a	0.05	0.005	3 (20-30)	not specified ^b

^a Application at immature fruit & fruiting stage

^b The submitted label indicates a PHI of 28-45 days, but at the time of this evaluation this was not yet studied and approved by the Iranian authorities.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Four independent trials were conducted on pistachios in Iran in 2015. Three foliar spray applications of a SP formulation were made at a rate of 0.05 kg ai/ha (0.15 kg ai/ha/season), using hydraulic handgun sprayer with re-treatment intervals of 29–60 days. Each of four field trial sites normally consisted of an untreated control plot and treated plots. Triplicate samples of at least 1 kg pistachio were harvested 28–30 days after last treatment from all segments of the tree. The fruits were separated from bunches. Samples were frozen within 4 hours of sampling. The maximum period of sample storage at -20 °C was less than 20 days. Pistachio samples were analysed within less than one month of sampling. Laboratory reports included method validation with procedural recoveries, RSD and calibration curves from spiking at residue levels similar to those occurring in samples from the supervised trials. No control data are recorded. Residue data are not corrected for percentage recovery. Residue data of pistachio from supervised trials are summarised in Table 3.

Table 3 Residues of a 20% SP formulation of acetamiprid in pistachio (nutmeat) after three foliar pre-harvest treatments

Location, year, (variety)	No (interval)	kg ai/ha	kg ai/hL	GS & last treatment day	DAT	residues, mg/kg ^a	reference
Zarand, Iran, 2015 (Fandoghi) [b]	3 (45, 31)	0.05	0.005	fruiting, 05 Aug	28	0.315/ 0.303/ 0.335 (0.318) ^b	Trial 001 summary - Zarand
Kerman, Iran, 2015 (Fandoghi) [b]	3 (45, 31)	0.05	0.005	fruiting, 05 Aug	28	0.496/ 0.467/ 0.510 (0.491) ^b	Trial 001 summary -
Qazvin, Iran, 2015 (Kale bozi)	3 (39, 29)	0.05	0.005	fruiting, 05 August	30	0.357/ 0.301/ 0.349 (0.336)	Trial 001 summary - Qazvin
Qhom, Iran, 2015 (Abbas Ali)	3 (60, 32)	0.05	0.005	fruiting, 07 August	30	0.215/ 0.170/ 0.224 (0.203)	Trial 001 summary - Qhom

^a Residues were measured in triplicate field samples. Mean is given in brackets.

^b Zarand is a city in Kerman province. Most of the pistachio orchards are in this region. Both experiments were performed there on the same day and the same variety. The locations were 120 km apart and can therefore be considered independent trials.

FATE OF RESIDUES IN STORAGE AND PROCESSING

No new data on storage and processing were submitted.

RESIDUES IN ANIMAL COMMODITIES

Pistachio (nutmeat) is not part of animal diet according to the OECD 2013 Feed Calculator. No new animal dietary burden calculation was carried out.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Data no longer required.

NATIONAL RESIDUE DEFINITION

No new data.

APPRAISAL

Acetamiprid is a neonicotinoid insecticide. The compound was first evaluated by the JMPR in 2011 (T,R) where an ADI of 0–0.07 mg/kg bw/day and an ARfD of 0.1 mg/kg bw were established. Maximum residue levels for a number of commodities were recommended by JMPR in 2011, 2012 and 2015. Acetamiprid was listed by the 48th Session of CCPR (2016) for evaluation of 2017 JMPR for additional uses.

The residue definition for acetamiprid in plant commodities for enforcement and dietary risk assessment is acetamiprid. The definition of the residue for animal commodities (for compliance with the MRL and estimation of dietary intake) is the sum of acetamiprid and N-desmethyl-acetamiprid, expressed as acetamiprid. The residue is not fat soluble

Residue data and GAP information were submitted to the present Meeting on pistachio nuts.

Methods of analysis

The analytical method QuEChERS (EN 15662) used in the supervised residue trials to determine parent compound acetamiprid had been previously evaluated by the JMPR (2011). The present Meeting received a summary of additional validation data for this method.

The multi-residue QuEChERS method using GC-MS and/or liquid chromatography coupled with tandem mass spectrum detection (LC-MS/MS) was validated at the LOQ of 0.01 mg/kg for determining acetamiprid residues in dry, high water, acid, oily and high sugar content matrices and in animal matrices. For pistachio nuts, the method was slightly modified. The modification was not detailed.

Stability of pesticide residues in stored analytical samples

At the 2011 JMPR evaluation, acetamiprid was shown to be stable for up to 12 months for a large range of commodities, including oil seeds. The (frozen) storage intervals between sampling and analysis of the submitted field trials with pistachio nuts were less than one month and therefore covered by the storage periods in these stability studies.

Results of supervised residue trials on crops

The Meeting received supervised residue trial data for foliar applications of acetamiprid on pistachio nuts.

Pistachio nuts

The critical GAP for acetamiprid on pistachio nuts in Iran is for 3 foliar applications at 0.05 kg ai/ha (interval of 20–30 days) and no specified PHI. The Meeting received four independent trials that were performed using an application rate of 3×0.05 kg ai/ha, RTI of 29–60 days and a PHI 28–30 days.

However, the supplied data did not match the GAP for pistachio nuts. Therefore, the Meeting could not estimate a maximum residue level, STMR or HR for acetamiprid in pistachio nuts.

REFERENCES

Code	Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
no code	Christia, C., Bizani E., Christophoridis C., Fytianos K.	2015	Pesticide residues in fruit samples: comparison of different QuEChERS methods using liquid chromatography–tandem mass spectrometry, Environ Sci Pollut Res, DOI 10.1007/s11356-015-4456-0
no code	JMPR/FAO	2011	Acetamiprid. Accessible at: http://www.fao.org/fileadmin/templates/agphome/documents/Pests_Pesticides/JMPR/Report11/Acetamiprid.pdf

REFERENCES REFERRED TO BUT NOT SYBMITTED AND NOT USED

Author	Year	Title, Institute & report number, Submitting manufacturer and report code, GLP/Non-GLP. Published/Unpublished
Simon-elso, N. et al.	2015	Systemic insecticides (neonicotinoids and fipronil): trends, uses, mode of action and metabolites, Environ Sci Pollut Res (2015) 22:5–34
DecourtyeA. Devillers J.	2010	Ecotoxicity of neonicotinoid insecticides to bees. Insect nicotinic acetylcholine receptors. Springer. pp. 85–95. Accessible at: http://link.springer.com/chapter/10.1007/978-1-4419-6445-8_8 . Accessed 10 Jul 2014
Tomizawa M.	2003	The neonicotinoid electronegative pharmacophore plays the crucial role in the high affinity and selectivity for the drosophila nicotinic receptor: an anomaly for the nicotinoid cation-pi interaction model. Biochemistry 42:7819–7827

