PROPYLENE OXIDE (250)

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EXPLANATION

Propylene oxide is used in agriculture as an insecticidal fumigant and sterilant, to control bacteria contamination, mould contamination, insect infestations, and microbial spoilage of food products as well as to control insects in non-food products. Propylene oxide is also a commercially important industrial chemical finding application as an intermediate for a wide array of products. It was first evaluated by JMPR in 2011 and an ADI of 0–0.04 mg/kg bw and an ARfD of 0.04 mg/kg bw established. Residue definitions established by the 2011 JMPR are:

Definition of the residue (for compliance with MRL): propylene oxide.

Definition of the residue (for estimation of dietary intake): propylene oxide, propylene chlorohydrin and propylene bromohydrin. Propylene chlorohydrin and propylene bromohydrin to be considered separately from propylene oxide.

The residue is not considered fat soluble.

Propylene oxide was scheduled at the 48th session of the CCPR for the evaluation of additional MRLs in 2017 JMPR.

Residue studies were submitted by the manufacturers to support tree nuts.

METHODS OF RESIDUE ANALYSIS

Descriptions of analytical methods together with validation data for residues of PPO, PCH-1, PCH-2, PBH-1 and PBH-2[3] in tree nuts were made available to the meeting.

Residues of PPO, PCH, and PBH were measured based on chromatographic and spectrometric agreement against standards. Gas chromatographic retention time and/or mass spectrometry were used for chemical verification.

Walse *et al.,* (2013) developed a method for PPO, PCH, and PBH residues based on modification to the method of King *et al.,* (1981), Heuser and Scudamore (1968), and Hoffman (2004). In the method a 0.5 L air-tight blending vessel is filled with 15 g of shelled walnuts or 25 g shelled almonds. Chilled water (pH 7) is then added to the blending vessel before introduction of methyl tert-butyl ether (MTBE) and the vessel sealed. If laboratory fortified blanks are prepared, such as for the residue calibration studies, injections of PPO, PCH or PBH stocks are made through the septa covering the sampling port. Samples are macerated and the homogenate decanted and centrifuged in capped cetrifugation tubes. Aliquots of MTBE supernatant are withdrawn and transferred to GC-vials pre-charged with MTBE containing methyl bromide standard and clamp-sealed with teflon lined caps. Samples are analysed using GC-EIMS analysis for PPO and PCH and using GC-ECD for PBH. In addition, for PBH separate 9 mL MTBE aliquots of supernatant for each sample were withdrawn and transferred to a volumetrically-graduated glass vial pre-charged with 1.0 mL MTBE containing methyl bromide internal standard. The contents were concentrated to 1.0 mL with a gentle N_2 stream, prior to transfer to GC-vials that were sealed as described above. In other modifications for PBH analysis, an aliquot (10 mL) of the MTBE extract is transferred volumetrically to a graduated glass vial and concentrated to approximately 0.95 mL (Jimenez *et al.,* 2015) or 0.5 mL (Walse and Jimenez 2016) with a gentle stream of N_2 prior to addition of the methyl bromide standard.

GC-EIMS: qualitative verification: methyl bromide, m/z (% rel. inten.) 93.9 (49), 94.9 (1), 95.9 (50); PPO, m/z 58.0 (97), 59.0 (3); PCH-1, m/z 45 (71) 79.0 (21), 81.0 (5), 93.0 (2), 95.0 (<1);

[3] PCH-1 and PCH-2 are isomers of PCH, 1-chloropropan-2-ol (PCH-1) and 2-chloropropan-1-ol (PCH-2) while PBH-1 and PBH-2 are isomers of PBH, 1-bromopropan-2-ol (PBH-1) and 2-bromopropan-1-ol (PBH-2).

PCH-2, m/z 58.1 (46), 62.1 (22), 63.1 (18), 65.1 (14), 93.0 (<1), 95.0 (<1); PBH-1, m/z 45 (79), 80.1 (<1), 82.1 (<1), 122.8 (10), 124.8 (8); PBH-2, m/z 59.0 (60), 106.9 (9), 108.9 (9),122.8 (11), 124.8 (11). Respective GC-EIMS LOQs for PPO, PCH-1 and PCH-2, in non-concentrated MTBE extracts based on the lowest fortification level for which satisfactory recoveries were obtained (Table 1) are 1, 2 and 2 mg/kg for PPO, PCH-1 and PCH-2 respectively in almonds and walnuts.

GC-ECD: In 10-fold concentrated extracts the GC-ECD LOQs for PBH-1 and PBH-2 were 0.08 mg/kg for both almonds and walnuts (Table 1). With a 20-fold concentration of the MTBE extract, LOQs for PBH-1 and PBH-2 in concentrated MTBE extracts were 0.04 mg/kg for almonds and walnuts (Table 2). PPO is not amenable to analysis with GC-ECD detection.

GC-NCIMS spectra were acquired for qualitative verification: PCH-1, m/z (% rel. inten.) 58.0 (18), 63.0 (7), 65.0 (7), 78.8 (42), 80.8 (17), 93.0 [M-H]- (5), 95.0 (4); PCH-2, m/z 58.0 (40), 63.0 (18), 65.0 (18), 79.0(12), 81.0 (5), 93.0 [M-H]- (4), 95.0 (3); PBH-1, m/z 59.0 (18), 107.0 (19), 109.0 (12), 123.0 (19), 125.0 (12), 137.0 (11) [M-H]-,139.0 (9); PBH-2, m/z 59.0 (23), 107.0 (19), 109.0 (13), 123.0 (18), 125.0 (9), 137.0 (10) [M-H]-,139.0 (8).

Table 1 Summary of recoveries for almonds and walnuts using GC-EIMS (Walse *et al.,* 2013)

Fortification (mg/kg)	%Recovery (mean % ± RSD, n=5)					
	Almond			Walnut		
	PPO	PCH-1	PCH-2	PPO	PCH-1	PCH-2
1	94 ± 2.3	BDL	BDL	90 ± 6.5	BDL	BDL
2	96 ± 3.5	90 ± 3.9	90 ± 3.8	102 ± 4.6	95±6.5	90±8.9
5	100 ± 4.5	94 ± 5.1	95 ± 5.6	106 ± 6.5	91±9.8	96±3.6
10	96 ± 4.7	94 ± 4.2	92 ± 5.2	100 ± 4.9	96 ± 5.3	90 ± 4.6
25	96 ± 3.5	96 ± 6.5	97 ± 6.1	97 ±6.8	90±6.1	99±5.4
50	98 ± 4.8	90 ± 5.4	99 ± 7.1	95 ± 2.2	96±7.2	907±6.7
100	92 ± 5.1	96 ± 3.3	93 ± 3.9	96 ± 5.6	100 ± 5.6	96 ± 7.2
10-fold concentration of MTBE extract (10 → 1 mL)						
		PBH-1	PBH-2		PBH-1	PBH-2
5		81 ± 7.2	101 ± 7.7		85 ± 6.3	95 ± 2.1
10		102±3.6	98±3.6		93 ± 5.1	101 ± 9.2
25		105±5.2	95±5.4		90±7.2	90±9.7
50		100± 7.1	97 ± 4.8		99±5.8	93±4.1
100		96±3.8	99 ±5.3		98 ± 4.6	92 ± 3.9

BDL = below the detection limit

Table 2 Summary of recoveries for PBH-1 and PBH-2 in almonds and walnuts (mg/kg) using GC-ECD (Walse *et al.,* 2013)

Fortification (mg/kg)	%Recovery (mean ± RSD, n=5)			
	Almond			
	PCH-1	PCH-2	PBH-1	PBH-2
MTBE extract (1 mL)				
1			85 ± 9.3	96 ± 6.5
2	86±7.8	91±8.8	88±6.5	95±7.2
5	81±9.9	88±6.9	101±7.0	90±9.4
10	95 ± 7.1	90 ± 11	95 ± 7.4	85 ± 10
25	100±4.5	100±8.7	86±5.8	84±5.4
50	92±8.1	102±6.9	97±4.7	94±6.4
100	90±8.2	89±7.1	100±3.4	95 ± 7.1
10-fold concentration of MTBE extract (10 → 1 mL)				
0.08			102.1 ± 9.6	102 ± 9.1
0.1			105.4±10	95±4.6
1			95.8±7.2	97±7.2
10			96.3±6.3	100±8.8
100			104.5±8.6	102±7.6
	Walnut			
	PCH-1	PCH-2	PBH-1	PBH-2
MTBE extract (1 mL)				

Fortification (mg/kg)	%Recovery (mean ± RSD, n=5)			
	Almond			
	PCH-1	PCH-2	PBH-1	PBH-2
1			101 ± 9.6	99 ± 10
2	95±8.5	93±5.6	100±8.8	95±7.5
5	103±9.6	86±4.5	96±7.4	95±6.4
10	101 ± 6.5	86 ± 5.9	93± 6.5	86 ± 8.7
25	95±5.7	91±8.6	86±5.1	92±6.5
50	96±8.9	91±5.2	90±4.6	87±5.5
100	85±10	95±4.6	92±6.5	90 ± 8.4
10-fold concentration of MTBE extract (10 → 1 mL)				
0.08			104 ± 6.9	102± 6.7
0.1			97±10	96±9.4
1			89±6.2	90±6.0
10			88±7.0	88±5.7
100			96±7.4	95±7.3

Table 3 Summary of recoveries for PBH-1 and PBH-2 in almonds and walnuts (mg/kg) using GC-ECD (Walse and Jimenez 2016)

Fortification (mg/kg)	%Recovery (mean ± RSD, n=5)			
	Almond		Walnut	
	PBH-1	PBH-2	PBH-1	PBH-2
MTBE extract (1 mL)				
1	91.2 ± 5.6	79.8 ± 4.2	92.5 ± 4.1	104 ± 9.6
	95.8±9.8	102.5±7.8	110.5±10	105±7.6
	100.5±8.7	96.8±6.4	101.3±7.8	96±4.7
10	106.7 ± 8.1	92.4 ± 5.3	104.4 ± 5.7	96 ± 5.7
	95.1±7.5	94.5±5.1	89.7±6.5	92±4.7
	89.6±4.4	100.8±6.8	101±6.5	86±9.8
100	101.0 ± 7.5	107.5 ± 5.2	89.2 ± 3.9	95 ± 4.9
20-fold concentration of MTBE extract (10 → 0.5 mL)				
0.04	103±7.3	105±8.7	96±7.4	94±4.9
0.1	96±5.5	86±5.7	92±6.5	88±5.2
1	82 ± 4.5	83 ± 8.1	100 ± 7.9	114 ± 7.1
10	110 ± 5.2	86 ± 6.4	102 ± 7.4	89 ± 4.2
100	91 ± 2.5	95 ± 4.5	85± 2.8	106± 3.9

Stock solutions (100 and 1000 mg/L) of PPO, PCH and PBH in MTBE were found to be stable for > 200 days at -5 °C (Walse and Jimenez, 2016).

Stability of Residues in Stored Analytical Samples

In the supervised residue trials samples were extracted on the day of collection with sample collection to extraction intervals of < 4 hours, extraction to preparation for analysis < 25 min and preparation to completion of analysis < 12 hours.

USE PATTERNS

Propylene oxide is used as fumigant and sterilant to control bacterial and mould contamination, insect infestations, and microbial spoilage of food products (tree nuts, spices, dried fruits and cocoa beans and powder), either as an end-use product alone or in mixtures with carbon dioxide. As it is a liquid at room temperature, fumigations are carried out at elevated temperatures but less than 52 °C and exposure times of up to 48 hours. Additionally PPO is flammable at concentrations in air of between 1.7 and 37% and is therefore used under conditions of reduced pressure. To enable fumigations at atmospheric pressure a non-flammable mix with carbon dioxide is used (8% PPO with 92% CO_2).

Table 4 Registered uses of propylene oxide on tree nut** commodities in the US.

Commodity	Rate (g ai /L chamber)	Temp [a] (°C)	Flush [b]	Degas and post fumigation interval
Fumigation	2.0 for < 6 hours (=2000 mg ai/L or 2000 g ai/m^3)	<52	4×	28 days @ 25 °C or 35 °C prior to shipment or until residues PPO <300 mg/kg

[a] temperature for fumigation treatment

[b] number of volumes of fumigation chamber that must be replaced with air or inert gas

** US EPA Crop group 14: tree nuts: (Almond, Beech nut, Brazil nut, Butternut, Cashew, chestnut, Chinquapin, Filbert (hazelnut), Hickory nut, macadamia nut (bush nut), pecan and Walnut, black and English (Persian)

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised trials for PPO on the following commodities:

Commodity	Table No
Almonds – pasteurisation trials	6
Walnuts – pasteurisation trials	7
Almonds – commercial scale trials	8
Walnuts (shelled) – commercial scale trials	9
Walnuts (in-shell) – commercial scale trials	10

Where duplicate samples from an unreplicated fumigation were taken at each sampling time and were analysed separately, the mean of the two analytical results was taken as the best estimate of the residues in the product and the means are recorded in the tables. When residues were not detected they are shown as below the LOQ (e.g., < 2 mg/kg). Residues and fumigation rates have generally been rounded to two significant figures. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are underlined.

Conditions of the supervised residue trials were generally well reported in detailed fumigation reports. The post-fumigation interval (PFI) is assumed to start at the end of the fumigation period and includes the period when the fumigation chamber is flushed with air.

Tree nuts

Storage facilities and fumigation chambers used for PPO fumigations are constructed to industry standards and are provided by a limited number of manufacturers and do not have the same level of variability that would be expected to be seen in field trials. The operating conditions are tightly controlled.

Two series of trials were conducted on tree nuts, the first in 2012/13 that utilised a treatment rate suitable for pasteurisation of nuts and the second in 2016 with rates close to the maximum approved in the USA. Each trial was conducted on a new lot of nuts loaded into the chamber and treated independently. The storage facilities used for the trials were located close to laboratories. Following sampling, the samples were immediately transported to the laboratory for analysis which occurred within 12 hours of sampling.

Pastuerisation trials conducted in 2012/2013 (Walse *et al.,* 2013).

For almonds, three commercial 94 m^3 vacuum-chambers were used for almond fumigation. Chambers were heated to *ca.* 38 °C and were each loaded to an estimated percentage of 41% capacity ($100 \times V_{commodity}/V_{chamber}$) with 24 wooden bins or 24 triwall bins that were "double-stacked" and placed in two rows. Each bin was lined with a polyethylene bag that was filled with *ca.* 900 kg of shelled

almonds which were preheated, or "conditioned", to *ca.* 32 °C. The sealed chamber was heated to 48.9 ± 2.2 °C. Pressure in the chamber was reduced to *ca.* 100 mm Hg. The vaporizer for fumigant delivery was heated to a temperature of 60 to 71 °C and propylene oxide was added, to achieve the requisite pasteurization dose of 500 g/m^3 afterwhich the pessure in the chamber was increased to *ca.* 600 mm Hg marking the beginning of the 4 hour exposure period. After 4-h the pressure in the chamber was reduced and then aeration valves were opened to atmosphere; four consecutive vacuum-aeration cycles were conducted. At the end of the aeration cycles the chamber was opened and the bins were removed. For the initial (day 0) post-fumigation residue measurement, a sample of almonds was collected from the top and bottom of the rear-most, middle, and front-most situated bins (middle bins were not sampled in two trials), and immediately transferred to a cooler filled with dry-ice. Palletized bins were transferred to an incubator at *ca.* 32 °C to off-gas PPO for 3 days and then the nuts removed to a storage facility maintained at 15 °C and 65% relative humidity (RH).

For walnut, two commercial vacuum-chambers, each with a volume of *ca.* 33 m^3, were used for the fumigations. Chambers were heated to *ca.* 41 °C and then each loaded sequentially (back to front) to 30% capacity with 7 pallets each comprising 40-50 cardboard boxes. Each box was commercially packed, having a plastic poly-liner bag filled with 11.4 kg of in-shell Hartley variety walnuts (the liner was folded over the top of the walnuts). Walnuts were "conditioned" as described to *ca.* 30 °C. The chamber was heated to a treatment temperature of 43.3 °C. Pressure in the chambers was reduced to *ca.* 100 mm Hg. The fumigant vaporizer was heated to a temperature of 60 to 71 °C and PPO added to achieve a dose of 830 g/m^3. Pressure in the chamber was increased to *ca.* 600 mm Hg marking the beginning of the 4-h exposure period and after this, four consecutive vacuum-aeration cycles were conducted. For the initial (day 0) post-fumigation residue measurement, a box of walnuts from the top as well as the bottom of the two rear-most and two front-most situated pallets were sampled with samples immediately transferred to a cooler filled with dry-ice. The liner was refolded, the sampled-boxes were then resealed as well as incorporated back into the respective pallet, and the pallets were transferred to an incubator at *ca.* 38 °C to offgas PPO for six days when PPO levels were < 300 mg/kg based on Eagle Gas Detection Unit calibrated for PPO. Sampled-boxes were removed from the respective pallets to a storage facility maintained at 15 °C and 65% RH.

Acceptable concurrent recovery data were obtained for samples fortified at 2 or 10 mg/kg for PPO, PCH-1 and PCH-2 and using GC-EIMS and at 0.08 and 1 mg/kg for PBH-1 and PBH-2 using GC-ECD with 10-fold MTBE concentration.

The laboratory sample for analysis was a 25 g subsample for almonds and a 15 g subsample of nutmeat for walnuts. The laboratory sample sizes do not correspond to the generally accepted prescribed sample size of 1 kg for tree nuts, see the "*FAO manual on the submission and evaluation of pesticide residues data for the estimation of maximum residue levels in food and feed*, 3rd edition" (2016), p 168 and also note "*Guidelines on Good Laboratory Practice in Pesticide Residue Analysis*", CAC/GL 40-1993. However, at each sampling time eight to twelve individual subsamples, each of 15 or 25 g were analysed and the overall mean represents the analysis of 120 to 300 g of nutmeat. The overall mean values are considered to adequately represent the residue in the lot sampled.

PCH-2 levels in all samples from pastuerisation treatments were below the GC-ECD LOQ. PBH-1 and PBH-2 residue levels in all samples from pastuerisation treatments were below the GC-ECD LOQs as well as the GC-ECD LODs when MTBE extracts were concentrated 10-fold prior to analysis.

In addition, the same calibration curve was used to convert instrument responses to residue values, even though the analysis dates spanned a period of 28 days. As instrument response changes with time the practice is not acceptable and the residue values reported require additional clarification.

The half-lives for PPO loss for walnuts and almonds were *ca.* 19 and 22 days, respectively.

Levels of PPO residues on a subset of fumigated nuts analysed with GC-FID detection via a modified method of Gunther (1965) were approximately < 50% of the levels measured using the solvent extraction-based methodology using GC-EIMS and GC-ECD. In the GC-FID method 50 g of nuts are added to a 1 L air-tight glass blending vessel, water added and the sample distilled until the

required volume is collected and an aliquot analysed with GC-FID. The difference in results is likely due to the hydrolysis of PPO during homogenization and/or distillation of nut samples.

Table 5 Residues of PPO and PCH in commercially treated almonds following pasteurisation (Walse *et al.,* 2013)

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	sample (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
1	500×4h	48.9±2.2	0	15.0±2.2	1	t	0	0	782	5.1
									813	6.4
						b			1201	7.6
									1189	8.2
					3	t			997	10
									891	4.2
						b			1415	6.7
									1418	5.6
					6	t			829	5.4
									922	6.2
						b			1205	5.6
									1265	5.4
					mean				*1077*	*6.4*
			3		1	t	0	3	161	34
									147	35
						b			213	39
									281	55
					3	t			140	31
									148	32
						b			232	43
									245	56
					6	t			150	28
									166	35
						b			217	44
									238	43
					mean				*195*	*40*
					1	t	4	7	70	26
									91	34
						b			140	52
									139	41
					3	t			79	27
									63	28
						b			173	49
									156	39
					6	t			78	32
									87	29
						b			136	33
									120	37
					mean				*111*	*36*
					1	t	7	10	82	32
									94	39
						b			148	50
									162	49
					3	t			99	28
									61	26
						b			150	48
									136	37
					6	t			88	35
									87	31
						b			131	34
									137	46
					mean				*114*	*38*
					1	t	12	15	80	37
									82	37
						b			157	52

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	sample (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
									127	53
					3	t			67	45
									74	53
						b			162	36
									151	40
					6	t			76	37
									85	37
						b			116	44
									123	44
					mean				*108*	*43*
					1	t	27	30	69	44
									68	49
						b			139	83
									132	59
					3	t			78	45
									81	52
						b			92	41
									107	53
					6	t			47	43
									58	45
						b			64	46
									77	50
					mean				*84*	*51*
					1	t	42	45	50	35
									53	41
						b			102	61
									112	52
					3	t			57	37
									61	44
						b			64	33
									70	44
					6	t			23	35
									34	36
						b			49	39
									50	46
					mean				*60*	*42*
2	500×4h	48.9 ± 2.2	0	15.0 ± 2.2	1	t	0	0	803	5.4
									896	5.3
						b			1230	4.8
									1208	5.1
					3	t			752	6.6
									770	5.2
						b			1150	6.7
									1216	5.6
					6	t			856	5.6
									898	5.7
						b			1503	5.7
									1492	7.1
					mean				*1064*	*5.7*
			3		1	t	0	3	200	33
									220	32
						b			254	37
									351	49
					3	t			244	30
									199	26
						b			356	38
									232	35
					6	t			215	33
									232	34
						b			292	38
									256	39

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	sample (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
					mean				*254*	*35*
					1	t	4	7	134	37
									124	28
						b			248	44
									215	44
					3	t			130	41
									124	27
						b			200	42
									116	30
					6	t			124	35
									120	34
						b			171	39
									192	47
					mean				*158*	*37*
					1	t	7	10	117	34
									144	33
						b			308	56
									250	53
					3	t			134	41
									132	28
						b			214	42
									122	31
					6	t			134	39
									127	38
						b			179	41
									209	50
					mean				*172*	*41*
					1	t	12	15	98	31
									90	23
						b			173	50
									216	49
					3	t			124	31
									100	40
						b			150	30
									143	40
					6	t			89	22
									115	29
						b			145	34
									127	32
					mean				*131*	*34*
					1	t	27	30	52	41
									46	47
						b			90	53
									91	68
					3	t			60	43
									52	47
						b			74	46
									87	55
					6	t			68	48
									81	40
						b			72	55
									92	44
					mean				*72*	*49*
					1	t	42	45	43	36
									36	41
						b			66	49
									71	56
					3	t			34	37
									44	44
						b			61	41
									58	46

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	sample (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
					6	t			50	43
									58	38
						b			60	54
									60	38
					mean				*53*	*44*
3	500×4h	48.9 ± 2.2	0	15.0 ± 2.2	1	t	0	0	782	4.3
									1005	5.1
						b			1258	6.1
									1277	6.5
					6	t			849	4.6
									972	6.0
						b			1438	5.7
									1323	5.4
					mean				*1113*	*5.5*
			3		1	t	0	3	158	22
									154	24
						b			244	29
									284	35
					6	t			149	18
									173	23
						b			230	30
									267	30
					mean				*207*	*26*
					1	t	4	7	72	18
									99	24
						b			149	37
									154	30
					6	t			84	23
									94	22
						b			136	23
									112	24
					mean				*112*	*25*
					1	t	7	10	68	20
									82	22
						b			160	36
									150	31
					6	t			80	24
									83	21
						b			140	24
									120	27
					mean				*110*	*26*
					1	t	12	15	75	22
									66	22
						b			149	33
									109	31
					6	t			76	24
									90	27
						b			133	32
									120	28
					mean				*102*	*27*
					1	t	27	30	61	34
									62	27
						b			114	51
									110	41
					6	t			48	24
									46	32
						b			52	33
									65	28
					mean				*70*	*34*
					1	t	42	45	53	24
									52	28

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	sample (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
						b			112	44
									114	35
					6	t			25	25
									35	25
						b			50	30
									43	31
					mean				*61*	*30*
4	500×4h	48.9 ± 2.2	0	15.0 ± 2.2	1	t	0	0	1099	14
									1105	15
						b			1485	15
									1534	16
					6	t			947	15
									1068	15
						b			1504	15
									1518	15
					mean				*1282*	*15*
			3		1	t	0	3	168	27
									150	27
						b			256	30
									324	35
					6	t			155	24
									190	27
						b			250	32
									291	31
					mean				*223*	*29*
					1	t	4	7	61	24
									113	26
						b			129	34
									58	30
					6	t			149	27
									131	25
						b			232	26
									224	28
					mean				*137*	*28*
					1	t	7	10	40	25
									70	28
						b			134	33
									136	31
					6	t			60	27
									56	26
						b			125	27
									91	28
					mean				*89*	*28*
					1	t	12	15	59	27
									30	27
						b			135	34
									117	34
					6	t			56	27
									49	27
						b			129	33
									111	31
					mean				*86*	*30*
					1	t	27	30	23	33
									24	30
						b			94	45
									82	38
					6	t			11	26
									1.7	32
						b			17	33
									23	29
					mean				*34*	*33*

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	sample (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
					1	t	42	45	17	34
									35	30
						b			92	45
									89	37
					6	t			7.8	26
									2.1	33
						b			27	33
									41	31
					mean				*39*	*33*
5	500×4h	48.9 ± 2.2	0	15.0 ± 2.2	1	t	0	0	1069	15
									999	15
						b			1575	16
									1616	16
					3	t			1346	17
									1341	14
						b			1942	16
									1787	15
					6	t			1094	15
									1231	15
						b			1761	15
									1732	14
					mean				1458	15
			3		1	t	0	3	158	27
									140	27
						b			232	29
									285	32
					3	t			147	26
									156	27
						b			257	30
									281	36
					6	t			126	23
									151	26
						b			217	30
									265	30
					mean				*201*	*29*
					1	t	4	7	29	23
									60	26
						b			124	34
									136	30
					3	t			30	23
									31	24
						b			168	32
									156	29
					6	t			55	26
									66	26
						b			120	27
									100	28
					mean				*90*	*27*
					1	t	7	10	26	24
									50	27
						b			127	32
									137	31
					3	t			46	22
									25	24
						b			169	33
									145	29
					6	t			62	27
									56	25
						b			124	27
									89	28
					mean				*88*	*27*

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	sample (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
					1	t	12	15	28	26
									42	27
						b			137	33
									100	32
					3	t			43	32
									26	33
						b			141	26
									160	30
					6	t			32	27
									56	27
						b			104	31
									110	30
					mean				*82*	*30*
					1	t	27	30	23	27
									14	30
						b			96	39
									90	33
					3	t			37	30
									38	32
						b			50	27
									71	32
					6	t			4.7	27
									12	29
						b			21	29
									32	30
					mean				*41*	*30*
					1	t	42	45	9.5	27
									12	30
						b			94	39
									76	32
					3	t			14	29
									20	32
						b			22	27
									48	33
					6	t			10	27
									15	27
						b			<1	30
									1.0	30
					mean				*27*	*30*

Table 6 Residues of PPO and PCH in commercially treated in-shell walnuts following pasteurisation (Walse *et al.,* 2013)

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	placement (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
1	830×4 h	43.3 ± 2.4	0	15.0 ± 2.2	1	t	0	0	1958	7.3
									1791	5.6
						b			3050	5.9
									3232	6.2
					3	t			1621	5.8
									1765	5.3
						b			2867	7.7
									3700	6.5
					6	t			1786	9.1
									3097	6.3
						b			1874	6.4
									2870	6.7
					7	t			1472	6.1

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	placement (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
									1951	6.0
						b			2960	8.2
									2749	8.8
					mean				*2422*	*6.7*
			6		1	t	0	6	169	31
									132	29
						b			290	35
									329	42
					3	t			130	28
									148	29
						b			294	36
									308	45
					6	t			144	23
									187	29
						b			268	36
									331	37
					7	t			154	22
									144	23
						b			256	36
									251	31
					mean				*221*	*32*
					1	t	4	10	112	27
									137	31
						b			255	47
									253	40
					3	t			134	24
									106	27
						b			292	46
									270	37
					6	t			139	33
									153	30
						b			224	31
									204	34
					7	t			136	28
									134	23
						b			169	32
									231	32
					mean				*185*	*33*
					1	t	9	15	106	32
									115	29
						b			256	43
									209	44
					3	t			123	47
									102	46
						b			266	33
									260	37
					6	t			120	35
									121	32
						b			192	40
									207	37
					7	t			121	32
									92	32
						b			142	38
									130	41
					mean				*160*	*38*
					1	t	24	30	101	40
									128	55
						b			227	69
									199	46
					3	t			115	42
									118	46

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	placement (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
						b			158	38
									187	50
					6	t			54	41
									85	43
						b			122	46
									108	43
					7	t			54	38
									55	48
						b			130	54
									114	52
					mean				*122*	*47*
					1	t	39	45	73	38
									87	48
						b			234	78
									188	48
					3	t			83	38
									96	47
						b			136	39
									165	55
					6	t			32	42
									65	45
						b			94	48
									81	44
					7	t			30	36
									38	54
						b			104	54
									88	50
					mean				100	48
2	830×4 h	43.3 ± 2.4	0	15.0 ± 2.2	1	t	0	0	2416	7.3
									2546	5.6
						b			2697	5.9
									2711	6.2
					3	t			1948	5.8
									2076	5.3
						b			2903	7.7
									3269	6.5
					6	t			1823	9.1
									2017	6.3
						b			1938	6.4
									2071	6.7
					7	t			1880	6.1
									1955	6.0
						b			2493	8.2
									2478	8.8
					mean				*2326*	*6.7*
			6		1	t	0	6	236	29
									261	26
						b			339	31
									512	42
					3	t			310	26
									234	23
						b			468	31
									311	30
					6	t			253	27
									287	28
						b			371	31
									341	32
					7	t			290	36
									310	30
						b			327	28

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	placement (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
									335	35
					mean				*324*	*30*
					1	t	4	10	180	28
									194	25
						b			414	37
									365	38
					3	t			195	33
									200	24
						b			334	36
									201	29
					6	t			208	34
									213	32
						b			299	36
									318	40
					7	t			246	33
									247	36
						b			291	36
									240	34
					mean				*259*	*33*
					1	t	9	15	167	30
									149	22
						b			292	43
									357	41
					3	t			177	26
									168	35
						b			238	25
									236	34
					6	t			136	20
									175	26
						b			218	28
									198	27
					7	t			224	32
									284	38
						b			217	31
									203	32
					mean				*215*	*31*
					1	t	24	30	93	49
									65	44
						b			137	46
									162	61
					3	t			72	35
									78	44
						b			106	37
									136	46
					6	t			95	43
									123	38
						b			113	52
									170	45
					7	t			106	30
									109	36
						b			120	34
									136	41
					mean				*114*	*43*
					1	t	39	45	64	46
									44	45
						b			117	47
									137	61
					3	t			62	42
									57	45
						b			90	40
									108	48

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	placement (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
					6	t			83	46
									85	38
						b			109	54
									115	42
					7	t			73	32
									72	36
						b			128	37
									134	44
					mean				*92*	*44*
3	830×4 h	43.3 ± 2.4	0	15.0 ± 2.2	1	t	0	0	2284	23
									2200	24
						b			3292	24
									3504	25
					3	t			1885	25
									2037	25
						b			3530	26
									4424	24
					6	t			1740	25
									3307	24
						b			2190	25
									3068	25
					7	t			1819	24
									2271	24
						b			3323	27
									2986	26
					mean				*2741*	*25*
			6		1	t	0	6	76	42
									87	42
						b			232	46
									288	51
					3	t			62	40
									77	43
						b			280	48
									302	55
					6	t			94	37
									128	41
						b			239	48
									342	49
					7	t			92	37
									105	39
						b			235	49
									196	43
					mean				*177*	*44*
					1	t	4	10	64	38
									40	41
						b			158	53
									198	48
					3	t			72	36
									32	40
						b			233	50
									200	45
					6	t			38	41
									78	41
						b			160	42
									157	43
					7	t			50	38
									105	38
						b			100	44
									174	45
					mean				*116*	*43*

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	placement (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
					1	t	9	15	49	43
									18	43
						b			198	51
									126	49
					3	t			57	52
									28	53
						b			171	42
									196	46
					6	t			44	42
									56	42
						b			140	50
									174	48
					7	t			6.8	40
									32	46
						b			79	50
									83	52
					mean				*91*	*47*
					1	t	24	30	120	66
									44	60
						b			119	62
									82	51
					3	t			51	51
									32	50
						b			76	42
									68	50
					6	t			36	46
									38	51
						b			90	47
									86	49
					7	t			55	43
									50	49
						b			108	53
									104	55
					mean				*72*	*52*
					1	t	39	45	18	44
									40	48
						b			93	61
									88	49
					3	t			<1	47
									<1	50
						b			43	42
									80	53
					6	t			<1	45
									10	50
						b			40	48
									38	50
					7	t			9.4	42
									4.1	49
						b			17	52
									22	58
					mean				*39*	*49*
4	830×4 h	43.3 ± 2.4	0	15.0 ± 2.2	1	t	0	0	3116	26
									3086	25
						b			3007	25
									3166	26
					3	t			2125	25
									2174	25
						b			3316	25
									3683	34
					6	t			2146	25

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	placement (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
									2369	27
						b			2446	25
									2319	24
					7	t			2001	26
									2070	27
						b			2840	26
									2888	25
					mean				*2672*	*26*
			6		1	t	0	6	197	42
									237	41
						b			288	43
									578	55
					3	t			266	39
									184	37
						b			465	43
									248	42
					6	t			226	40
									268	40
						b			368	44
									336	45
					7	t			241	46
									266	41
						b			293	40
									339	47
					mean				*300*	*43*
					1	t	4	10	145	42
									163	39
						b			439	50
									382	49
					3	t			121	43
									177	38
						b			318	44
									108	38
					6	t			147	42
									120	43
						b			230	47
									280	48
					7	t			127	41
									158	44
						b			251	44
									172	45
					mean				*209*	*44*
					1	t	9	15	81	42
									88	36
						b			228	52
									341	53
					3	t			89	38
									89	44
						b			139	37
									143	44
					6	t			69	34
									111	39
						b			165	41
									159	40
					7	t			142	42
									170	44
						b			119	41
									130	41
					mean				*142*	*42*
					1	t	24	30	59	44
									50	47

trial	Rate (mg ai/L)	Chamber temp during treatment (°C)	incubation days @ 32 °C	storage temp (°C)	pallet	placement (top vs. bottom)	days of storage	PFI (days)	PPO (mg/kg)	PCH-1 (mg/kg)
						b			31	48
									50	57
					3	t			92	45
									91	48
						b			81	43
									91	49
					6	t			68	47
									67	42
						b			26	50
									60	45
					7	t			81	40
									73	42
						b			107	43
									77	45
					mean				*69*	*46*
					1	t	39	45	<1	45
									13	47
						b			20	50
									35	58
					3	t			<1	41
									<1	44
						b			<1	43
									<1	48
					6	t			<1	45
									<1	42
						b			40	51
									20	44
					7	t			<1	40
									<1	42
						b			44	42
									38	45
					mean				*30*	*45*

In a separate series of trials conducted in 2016 (Walse and Jimenez 2016) almonds and walnuts were treated at rates approximating the maximum approved in the USA.

Commercial-scale fumigations were conducted in a 3760 L steel chamber (137×137×203 cm). Prior to fumigation, the chamber was loaded to 41.9% capacity with a single triwall bin lined (102×122×129 cm) with a polyethylene bag that was volume-filled with *ca.* 900 kg of shelled almonds "conditioned" for 10 hours to *ca.* 32 °C. Pressure in the chamber was reduced to *ca*. 100 mm Hg. The vaporizer for fumigant delivery was heated to 60–71 °C and PPO was added, to achieve the requisite dose, 2 000 g/m^3 or 1,500 g/m^3. After the application of PPO, pressure in the chamber was increased to *ca*. 600 mm Hg marking the beginning of the exposure period. Fumigations with 2 000 g/m^3 PPO were conducted for 4.5 h while those with 1,500 g/m^3 were conducted for 6 hours. After the exposure, normal atmospheric pressure was established in the chamber via a gaseous nitrogen balance. Pressure was again reduced to *ca.* 100 mm Hg via vacuum, and immediately thereafter, aeration valves were opened to bring chamber pressure to *ca.* 600 mm Hg. Four consecutive vacuum-aeration cycles were conducted. At the end of the aeration cycles the bin of treated almonds was removed from the chamber. For the initial (day 0) sample *ca.* 1 kg of almonds was collected with a scooper from the top as well as the bottom of the bin (*ca*. 500 g from each location) and transferred to a Ziploc bag and the contents were mixed and subsequently nuts selected from the Ziplock bag to produce two 400 g composite samples. The composite samples were then immediately transferred to a cooler filled with dry-ice. The bin was then transferred to off-gas PPO within an on-site storage facility maintained at *ca.* 25 °C and 65% RH. The contents of the bin were emptied into cardboard cases with no plastic poly-liner, each containing *ca.* 11.3 kg of almonds. Samples were collected at various intervals, through to a 28 day period at the storage facility. For each sample, two cases were randomly selected and 500 g of nuts collected from different locations in the

case and the nuts (*ca.* 1 kg) transferred to a Ziploc bag and the contents were mixed and subsequently nuts selected from the Ziplock bag to produce two 400 g composite samples.

For walnuts, commercial-scale fumigations were conducted in the vacuum-chamber described above set to a treatment temperature of 43 °C. Prior to fumigation, the chamber was loaded to 42.5% capacity with a palletized load comprised of 60 cardboard cases (each *ca.* 11.3 kg, 39×29×24 cm) of shelled or in-shell Hartley variety walnuts "conditioned" for 10 hours to *ca*. 32 °C. The walnuts were commercially-packed into the cases, whereby shelled walnuts were contained within a plastic poly-liner (folded over the top of the walnuts) that is not used in the case of in-shell nuts. After completion of the exposure, four aeration cycles were conducted as described for almonds. At the end of the aeration cycles, the pallets of treated walnuts were removed from the chamber and a sample collected. For the initial (day 0) post-fumigation residue measurement, a case of walnuts from the top as well as the middle of the pallet was retrieved. Retrieved boxes were opened, where necessary, the poly-liners unfolded and then a total of *ca.* 1 kg walnuts (*ca.* 500 g from each case) were transferred to a Ziploc bag and mixed. Bags were immediately transferred to a cooler that was filled with dry-ice. The liner was refolded (if necessary), the sampled-boxes were then resealed as well as incorporated back into the respective pallet, and the pallets were transferred to the on-site storage facility maintained at *ca.* 25 °C and 65% RH. Samples were collected at various intervals, through to a 28 day period at the storage facility.

Commercial-scale fumigations on mixed loads of almonds and walnuts were conducted in a 94238 L steel chamber set to a treatment temperature of 49 °C. Prior to fumigation, the chamber was loaded to an estimated 50.1% capacity with twenty 80-case (each case: ~11.3 kg, 39×29×24 cm) pallets of shelled walnut halves situated in two 10-pallet rows. Three cases each of shelled almonds, shelled walnuts, and in-shell walnuts, packaged as described above, were placed on top the 1st, 5th, and 10th pallets. The load was "conditioned" for 16 hours to *ca*. 32 °C and the chamber was set to treatment temperature of 43 °C. Pressure in the chamber was reduced to ca.100 mmHg. The vaporizer for fumigant delivery was heated to a temperature of 60–71 °C and PPO was added, to achieve the requisite dose of 2000 or 1500 mg ai/L. Replicate fumigations were conducted with each of the two dose-duration scenarios described above. After completion of the exposure, and four aeration cycles, the nine non-palletized cases were removed from the chamber. For the initial post-fumigation sample, all retrieved boxes were opened and then about 1 kg of each nut type (*ca.* 333 g from each case), was transferred to respective Ziploc bags and then mixed. Respective bags were immediately transferred to a cooler that was filled with dry-ice. The sampled-boxes were then resealed, and then transferred to an on-site storage facility maintained under the conditions, and for the duration, described above *ca.* 25 °C and 65% RH. Samples were collected at various intervals, through to a 28 day period at the storage facility.

PPO and PCH were quantified using or GC-EIMS and PBH using GC-ECD. Acceptable concurrent recovery data were obtained for samples fortified at 2 or 10 mg/kg for PPO, PCH-1 and PCH-2 and using GC-EIMS and at 0.08 and 1 mg/kg for PBH-1 and PBH-2 using GC-ECD with 20-fold MTBE concentration.

The laboratory sample for analysis was a 25 g subsample for almonds and a 15 g subsample of nutmeat for walnuts. The laboratory sample sizes do not correspond to the generally accepted or prescribed sample size of 1 kg for tree nuts, see the "*FAO manual on the submission and evaluation of pesticide residues data for the estimation of maximum residue levels in food and feed*, 3rd edition" (2016), p 168. Justification as to the validity of using a smaller laboratory sample size was not presented, for example see "*Guidelines on Good Laboratory Practice in Pesticide Residue Analysis*", CAC/GL 40-1993.

An indication of the variability in results from the use of 15 and 25 g sample sizes can be obtained from the CVs for the sample sets from the data in the 2013 trial (Walse *et al.,* 2013) where results for 8 to 12 individual subsamples were reported for each time point. For the almond subsample size of 25 g from the 2013 trial, CVs were in the range 19–112%, mean 41% for PPO and 2.7–26%, mean 16% for PCH. For walnuts with a sample size 15 g, CVs were in the range 18–79%, mean 39% for PPO and 4.0–22%, mean 14% for PCH. The small sample sizes are inadequate to obtain a reliable

measure of the average residue in the lot and the data is not suitable for use in estimating maximum residue levels.

Less than 25 minutes elapsed between sampling, processing and preparing MTBE extracts for GC-EIMS and GC-ECD analysis. The analysis of PBH by GC-ECD included a 20-fold concentration of extracts.

In all cases PBH was not detected (LOQ 0.04 mg/kg).

The same laboratory conducted the analyses as for the 2013 trials and it was not possible to verify the appropriate use of the calibration curves for this dataset. Clarification is required to verify the validity of the reported results.

The half-lives for PPO loss for shelled walnuts, in-shell walnuts and almonds were *ca.* 7.0, 6.6 and 8.8 days, respectively.

There was a significant difference in PPO levels at t=0 following treatment with 2 000 g/m^3 dose for 4.5 h, relative to 1,500 g/m^3 dose for 6.0 h with higher levels observed in nuts treated at 2 000 g/m^3 dose for 4.5 h. Single factor ANOVAs applied to each nut type at respective temporal intervals of analysis ≥ 7 d (i.e. PPO(t_7 – t_{28})) however were not significant; a finding that reflects congruency in overall mean PPO levels across fumigation trials, independent of the fumigation type (*i.e.* mixed load fumigation or not mixed) and the two dose-duration scenarios (2 000g/m^3 for 4.5 h or 1,500g/m^3 for 6 h). The study authors proposed the greater initial PPO(t_0) levels in nuts to be a result of relatively greater dose, 2 000 g/m^3 versus 1,500 g/m^3. It was proposed that since air is nearly saturated with PPO at a 2 000 g/m^3 loading at 125 ºC and ~600 mmHg, that PPO sorption to the nut meat surface exceeds monolayer coverage for the two applied doses. PPO sorbed to the adlayer, which is reflected in PPO levels at t_0, is no longer present in subsequent temporal intervals of sampling. This interpretation is supported by the observation of ~500 g m^{-3} h greater C×t exposures for fumigations conducted with 2 000 g/m^3 dose for 4.5 h, relative to 1,500 g/m^3 dose for 6.0 h, indicating nearly equivalent amounts of PPO was sorbed by the load for the respective dose-duration scenario; ~ 50% of 2 000 g/m^3 dose was sorbed in for 4.5 h and ~65% of 1,500 g/m^3 dose was sorbed in 6.0 h. Residue levels of PCH-1 and PCH-2 (*vide infra*), which form as a function of sorbed PPO and its hydrolysis thereafter, were similar across the dose-duration scenarios for each nut type, a finding that is consistent with the above interpretation.

Table 7 Residues of PPO and PCH in commercially treated shelled almonds following fumigation (Walse and Jimenez 2016)

trial	chamber type	Rate (mg ai/L)	temp during treatment (°C)	incubation (days @ 38 °C)	storage temp (°C)	days of storage	PPO (mg/kg)	PCH-1 (mg/kg)	PCH-2 (mg/kg)	Total PCH (mg/kg)
1	1	1500×6h	49.2±0.8	0	25.2±0.4	0	1766	35	3.2	38
							1935	34	7.0	41
						Mean	1851	35	5.1	40
						7	1342	35	7.3	43
							1429	29	7.5	37
						Mean	1386	32	7.4	40
						14	554	40	15	54
							554	34	9.0	43
						Mean	554	37	12	49
						21	413	24	8.9	33
							368	21	9.3	30
						Mean	391	23	9.1	32
						28	185	14	9.1	24
							260	23	8.5	31
						Mean	222	19	8.8	27
2	1	1500×6h	50.1±0.5	0	25.0±0.3	0	2140	31	11	42
							1933	27	4.2	31
						Mean	2036	29	7.6	37
						7	1979	45	10	56
							1331	43	9.9	53

trial	chamber type	Rate (mg ai/L)	temp during treatment (°C)	incubation (days @ 38 °C)	storage temp (°C)	days of storage	PPO (mg/kg)	PCH-1 (mg/kg)	PCH-2 (mg/kg)	Total PCH (mg/kg)
						Mean	1655	44	10	54
						14	722	27	8.4	36
							707	41	3.7	44
						Mean	714	34	6.0	40
						21	386	31	7.6	39
							356	33	9.3	43
						Mean	371	32	8.5	41
						28	249	25	8.3	33
							337	25	9.0	34
						Mean	293	25	8.6	34
3	1	1500×6h	48.9±0.4	0	25.6±0.5	0	2585	40	5.7	45
							2524	35	1.8	37
						Mean	2555	37	3.8	41
						7	1383	38	7.0	45
							1276	37	10	47
						Mean	1330	37	8.6	46
						14	573	37	8.4	45
							637	44	6.0	50
						Mean	605	41	7.2	48
						21	384	21	11	32
							322	19	6.1	25
						Mean	353	20	8.6	28
						28	201	37	8.6	46
							245	24	10	34
						Mean	223	30	9.4	40
4	1	1500×6h	49.2±0.5	0	25.2±0.6	0	1706	31	4.5	35
							1892	32	6.8	39
						Mean	1799	32	5.6	37
						7	1182	41	11	52
							1002	38	7.2	45
						Mean	1092	39	9.3	48
						14	560	39	6.7	45
							508	29	7.2	36
						Mean	534	34	6.9	41
						21	352	31	9.6	40
							342	24	8.1	32
						Mean	347	27	8.9	36
						28	215	25	8.1	33
							304	28	9.5	37
						Mean	259	26	8.8	35
5	1	1500×6h	49.6±0.6	0	25.3±0.5	0	2202	30	7.0	37
							2519	34	6.7	41
						Mean	2361	32	6.8	39
						7	1274	30	6.3	36
							1413	30	6.8	36
						Mean	1344	30	6.5	36
						14	631	37	4.3	41
							702	44	4.5	49
						Mean	666	40	4.4	45
						21	538	44	8.4	52
							549	34	12	46
						Mean	543	39	10	49
						28	260	31	8.7	40
							243	26	8.7	35
						Mean	252	29	8.7	38
11	2	1500×6h	49.7±0.4	0	25.7±0.5	0	2412	38	8.7	47
							2488	41	6.2	47
						Mean	2450	39	7.4	47
						7	1555	31	7.1	38
							1396	44	5.1	49

trial	chamber type	Rate (mg ai/L)	temp during treatment (°C)	incubation (days @ 38 °C)	storage temp (°C)	days of storage	PPO (mg/kg)	PCH-1 (mg/kg)	PCH-2 (mg/kg)	Total PCH (mg/kg)
						Mean	1476	37	6.1	43
						14	646	34	7.0	41
							718	28	2.9	31
						Mean	682	31	5.0	36
						21	442	32	11	43
							442	46	8.1	54
						Mean	442	39	9.4	48
						28	237	26	7.7	34
							248	25	2.6	28
						Mean	242	26	5.2	31
12	2	1500×6h	49.2±0.3	0	25.4±0.5	0	2315	31	4.6	36
							2200	40	7.6	47
						Mean	2258	35	6.1	41
						7	1672	32	8.4	40
							1652	31	5.3	36
						Mean	1662	31	6.8	38
						14	645	26	<2	28
							879	23	6.8	30
						Mean	762	24	4.2	29
						21	438	40	10	50
							413	27	6.6	33
						Mean	426	33	8.5	42
						28	285	28	3.6	31
							285	21	6.7	28
						Mean	285	24	5.2	30
6	1	2000×4.5h	48.5±1.0	0	25.4±0.7	0	2548	30	3.7	34
							2858	27	9.5	37
						Mean	2703	29	6.6	36
						7	1217	38	3.2	41
							1280	22	2.9	24
						Mean	1249	30	3.1	33
						14	686	41	11	52
							640	38	11	49
						Mean	663	40	11	50
						21	363	27	6.3	33
							370	25	4.1	29
						Mean	367	26	5.2	31
						28	249	15	7.3	22
							264	22	4.2	27
						Mean	256	19	5.7	25
7	1	2000×4.5h	47.6±0.7	0	25.1±0.5	0	2706	26	4.0	30
							2682	22	8.1	30
						Mean	2694	24	6.0	30
						7	1284	24	9.6	33
							1174	19	6.8	25
						Mean	1229	21	8.2	29
						14	823	47	13	60
							832	47	8.9	56
						Mean	827	47	11	58
						21	396	18	3.4	21
							365	22	7.1	29
						Mean	380	20	5.3	25
						28	343	27	5.9	33
							314	22	4.5	27
						Mean	329	25	5.2	30
8	1	2000×4.5h	49.2±0.5	0	25.3±0.4	0	2217	36	2.8	39
							2140	25	5.1	30
						Mean	2178	30	4.0	34
						7	1426	37	6.7	44
							1220	48	5.4	54

trial	chamber type	Rate (mg ai/L)	temp during treatment (°C)	incubation (days @ 38 °C)	storage temp (°C)	days of storage	PPO (mg/kg)	PCH-1 (mg/kg)	PCH-2 (mg/kg)	Total PCH (mg/kg)
						Mean	1323	43	6.0	49
						14	555	35	12	47
							530	36	7.5	44
						Mean	543	36	9.8	45
						21	397	17	4.2	21
							380	29	2.8	32
						Mean	389	23	3.5	26
						28	302	9.3	8.3	18
							301	15	5.3	20
						Mean	301	12	6.8	19
9	1	2000×4.5h	49.5±0.6	0	25.7±0.7	0	2724	46	2.7	49
							2863	46	9.1	55
						Mean	2794	46	5.9	52
						7	1659	24	3.2	27
							1573	32	9.6	42
						Mean	1616	28	6.4	35
						14	498	40	11	51
							531	36	8.3	44
						Mean	514	38	9.8	48
						21	449	38	6.7	45
							435	23	3.9	27
						Mean	442	31	5.3	36
						28	242	22	6.8	29
							235	26	4.3	30
						Mean	238	24	5.6	30
10	1	2000×4.5h	49.3±0.4	0	25.4±0.6	0	2806	26	4.0	49
							2686	36	12	30
						Mean	2746	31	7.9	48
						7	1168	24	6.2	39
							1243	33	2.9	30
						Mean	1206	28	4.5	36
						14	736	29	7.6	33
							803	37	9.6	37
						Mean	770	33	8.6	47
						21	439	20	8.5	42
							455	24	5.7	29
						Mean	447	22	7.1	29
						28	294	16	6.2	29
							320	17	5.0	23
						Mean	307	17	5.6	22
13	2	2000×4.5h	49.0±0.4	0	25.2±0.5	0	2745	34	5.8	40
							2986	37	4.3	41
						Mean	2865	35	5.0	40
						7	1363	51	9.7	60
							1469	44	7.4	51
						Mean	1416	47	8.5	56
						14	741	34	5.9	40
							727	39	9.7	49
						Mean	734	36	7.8	44
						21	390	28	3.7	31
							494	30	3.6	34
						Mean	442	29	3.6	32
						28	272	28	10	38
							232	17	4.1	21
						Mean	252	22	7.2	30
14	2	2000×4.5h	48.9±0.3	0	25.6±0.3	0	2903	36	7.6	44
							2665	40	4.8	45
						Mean	2784	38	6.2	44
						7	1630	42	9.2	51
							1685	38	10	48

trial	chamber type	Rate (mg ai/L)	temp during treatment (°C)	incubation (days @ 38 °C)	storage temp (°C)	days of storage	PPO (mg/kg)	PCH-1 (mg/kg)	PCH-2 (mg/kg)	Total PCH (mg/kg)
						Mean	1658	40	9.6	50
						14	672	30	11	41
							743	32	8.3	40
						Mean	707	31	9.7	41
						21	471	42	5.2	48
							477	44	10	55
						Mean	474	44	7.8	51
						28	261	22	<2	23
							280	28	4.4	32
						Mean	270	25	2.9	28

Table 8 Residues of PPO and PCH in commercially treated shelled walnuts following fumigation (Walse and Jimenez 2016)

trial	chamber type	rate (mg ai/L)	temp during treatment (°C)	incubation (days @ 38 °C)	storage temp (°C)	days of storage	PPO (mg/kg)	PCH-1 (mg/kg)	PCH-2 (mg/kg)	Total PCH (mg/kg)
1	1	1500×6h	49.5±1.2	0	25.2±0.5	0	2475	42	4.1	47
							2656	46	15	61
						Mean	2566	44	9.6	54
						7	1265	34	8.7	43
							1101	44	12	56
						Mean	1183	39	10	50
						14	624	29	5	34
							729	34	4.5	38
						Mean	676	32	4.8	36
						21	278	30	9.7	40
							318	34	9.1	43
						Mean	298	32	9.4	42
						28	178	22	2.4	24
							137	31	3.3	34
						Mean	158	26	2.9	29
2	1	1500×6h	50.2±0.8	0	25.3±0.5	0	2818	43	8	51
							2654	37	4.1	41
						Mean	2736	40	6	46
						7	1405	41	5.4	46
							1652	34	4.3	39
						Mean	1528	38	4.8	42
						14	715	35	6.8	42
							715	39	12	51
						Mean	715	37	9.5	47
						21	347	27	7.1	34
							384	32	13	45
						Mean	366	30	9.9	40
						28	63.1	38	7.9	46
							111	30	13	42
						Mean	87.2	34	10	44
3	1	1500×6h	50.1±0.6	0	25.6±0.4	0	1996	35	4.6	40
							2225	47	10	57
						Mean	2111	41	7.3	48
						7	1357	44	3.3	48
							1531	40	7.3	48
						Mean	1444	42	5.3	48
						14	804	30	15	45
							762	34	7.8	42
						Mean	783	32	11	43
						21	345	38	12	50
							386	29	6.4	35

trial	chamber type	rate (mg ai/L)	temp during treatment (°C)	incubation (days @ 38 °C)	storage temp (°C)	days of storage	PPO (mg/kg)	PCH-1 (mg/kg)	PCH-2 (mg/kg)	Total PCH (mg/kg)
						Mean	365	33	9.2	42
						28	103	28	5.3	33
							101	32	2.5	35
						Mean	102	30	3.9	34
4	1	1500×6h	49.2±0.4	0	25.0±0.3	0	2698	31	5.3	36
							2434	35	9.5	45
						Mean	2566	33	7.4	41
						7	1322	46	13	59
							1360	37	8.3	45
						Mean	1341	41	11	52
						14	642	33	4.9	38
							679	34	12	46
						Mean	660	33	8.4	42
						21	371	44	6.1	50
							385	36	12	47
						Mean	378	40	8.8	49
						28	158	40	2.1	42
							85.4	33	2.7	36
						Mean	122	36	2.4	39
5	1	1500×6h	50.1±0.6	0	25.6±0.4	0	2339	37	2	39
							2266	43	4.7	48
						Mean	2302	40	3.4	43
						7	1548	27	9.5	36
							1455	25	16	41
						Mean	1502	26	13	39
						14	716	37	5.9	42
							844	41	2.2	43
						Mean	780	39	4.1	43
						21	373	30	6.6	37
							288	25	8.2	34
						Mean	330	28	7.4	35
						28	133	41	8.8	50
							167	35	2.7	38
						Mean	150	38	5.7	44
11	2	1500×6h	50.2±0.6	0	25.2±0.4	0	2260	28	8	36
							2381	33	10	43
						Mean	2320	30	9.1	40
						7	1745	33	8.2	41
							1688	27	9.2	36
						Mean	1717	30	8.7	39
						14	595	27	5.8	33
							722	22	3.3	25
						Mean	658	24	4.6	29
						21	213	28	2	30
							340	33	<2	33
						Mean	277	31	<2	32
						28	116	16	7.9	24
							150	22	8.8	31
						Mean	133	19	8.3	28
12	2	1500×6h	50.7±0.6	0	25.2±0.4	0	2659	35	8.3	43
							2780	41	7.7	49
						Mean	2720	38	8	46
						7	1176	34	9	43
							1564	37	7.6	44
						Mean	1370	35	8.3	44
						14	769	26	3.8	30
							838	16	4.5	21
						Mean	804	21	4.2	26
						21	313	37	8	45
							189	44	3	46

trial	chamber type	rate (mg ai/L)	temp during treatment (°C)	incubation (days @ 38 °C)	storage temp (°C)	days of storage	PPO (mg/kg)	PCH-1 (mg/kg)	PCH-2 (mg/kg)	Total PCH (mg/kg)
						Mean	251	40	5.5	46
						28	126	31	7.7	39
							136	17	7.5	24
						Mean	131	24	7.6	32
6	1	2000×4.5h	48.7±0.8	0	25.4±0.8	0	3661	26	4.6	31
							3352	32	6.8	39
						Mean	3507	29	5.7	35
						7	1531	24	<2	25
							1480	34	6.3	40
						Mean	1505	29	3.6	33
						14	779	46	9.7	56
							810	33	7.4	40
						Mean	794	39	8.6	48
						21	353	29	6.7	36
							317	32	8.1	40
						Mean	335	31	7.4	38
						28	160	28	6.9	35
							110	24	10	34
						Mean	135	26	8.5	34
7	1	2000×4.5h	48.9±0.7	0	25.3±0.5	0	2932	18	4.8	22
							2941	16	6.8	23
						Mean	2936	17	5.8	22
						7	1660	36	10	46
							1646	39	<2	40
						Mean	1653	37	5.5	43
						14	765	36	6.9	43
							758	35	5.3	40
						Mean	762	36	6.1	42
						21	371	34	13	47
							384	39	9.3	48
						Mean	378	37	11	48
						28	187	22	14	36
							189	35	7.2	42
						Mean	188	28	11	39
8	1	2000×4.5h	49.7±0.7	0	25.4±0.4	0	3055	40	4.7	44
							2667	41	6.1	48
						Mean	2861	41	5.4	46
						7	1634	37	7.2	44
							1560	26	13	39
						Mean	1597	31	10	42
						14	785	18	2.5	20
							782	10	8.6	18
						Mean	783	14	5.5	19
						21	355	32	6	38
							389	46	6.5	53
						Mean	372	39	6.3	46
						28	111	30	5.3	36
							116	15	14	29
						Mean	114	23	9.9	32
9	1	2000×4.5h	49.2±1.2	0	25.3±0.4	0	2966	19	4.6	24
							3260	33	6.4	39
						Mean	3113	26	5.5	32
						7	1532	21	9.5	30
							1858	22	8.7	31
						Mean	1695	22	9.1	31
						14	751	24	6	30
							720	20	6.7	27
						Mean	735	22	6.4	29
						21	288	34	6.5	40
							305	36	9.9	46

trial	chamber type	rate (mg ai/L)	temp during treatment (°C)	incubation (days @ 38 °C)	storage temp (°C)	days of storage	PPO (mg/kg)	PCH-1 (mg/kg)	PCH-2 (mg/kg)	Total PCH (mg/kg)
						Mean	296	35	8.2	43
						28	56.6	26	5.9	32
							87.8	21	14	35
						Mean	72.2	24	10	34
10	1	2000×4.5h	48.9±0.9	0	25.7±0.3	0	3126	33	6.7	40
							2920	27	4.6	32
						Mean	3023	30	5.7	36
						7	1562	20	2.4	22
							1579	24	4.8	29
						Mean	1571	22	3.6	26
						14	702	26	4.6	30
							853	21	9.5	31
						Mean	778	24	7	30
						21	283	43	6.5	50
							298	34	5.4	39
						Mean	290	38	5.9	44
						28	82.9	29	8.5	37
							124	28	6.8	35
						Mean	103	28	7.7	36
13	2	2000×4.5h	50.0±0.8	0	25.4±0.5	0	2907	36	9.6	46
							2736	26	4.8	31
						Mean	2821	31	7.2	38
						7	1128	43	6.8	50
							1287	33	7.7	41
						Mean	1207	38	7.2	45
						14	814	24	6.5	31
							809	30	7.9	38
						Mean	811	27	7.2	34
						21	385	24	9.7	34
							395	16	8.1	24
						Mean	390	20	8.9	29
						28	136	31	8.8	40
							106	26	9.6	36
						Mean	121	29	9.2	38
14	2	2000×4.5h	49.7±0.7	0	25.4±0.7	0	3006	30	3.3	33
							3242	20	12	33
						Mean	3124	25	7.8	33
						7	1568	41	8.2	50
							1738	31	6.5	38
						Mean	1653	36	7.3	44
						14	808	28	7.5	36
							860	47	8.9	56
						Mean	834	38	8.2	46
						21	268	35	6.1	41
							283	32	4.2	36
						Mean	275	34	5.2	39
						28	170	42	11	52
							149	41	9.1	50
						Mean	159	41	9.9	51

Chamber type 1 = 3760 L, 2 = 94238 L.

Table 9 Residues of PPO and PCH in commercially treated in-shell walnuts following fumigation (Walse and Jimenez 2016)

trial	chamber type	Rate (mg ai/L)	temp during treatment (°C)	incubation (days @ 38 °C)	storage temp (°C)	days of storage	PPO (mg/kg)	PCH-1 (mg/kg)	PCH-2 (mg/kg)	Total PCH (mg/kg)
1	1	1500×6h	49.8±0.9	0	25.1±1.0	0	2833	39	12	50
							2703	33	6.4	40
						Mean	2768	36	9	45
						7	1722	33	3.9	37
							1599	42	5.5	48
						Mean	1660	38	4.7	42
						14	724	21	3.7	25
							715	14	<2	15
						Mean	719	17	2.6	20
						21	392	52	5.6	57
							375	38	6.7	45
						Mean	384	45	6.1	51
						28	132	39	5.8	45
							163	48	3.6	52
						Mean	147	44	4.7	48
2	1	1500×6h	48.9±1.2	0	25.1±0.5	0	2439	46	9.1	55
							2556	33	7.2	40
						Mean	2497	40	8.2	48
						7	1811	42	5.4	48
							1996	34	11	45
						Mean	1904	38	8.1	46
						14	991	31	6.8	38
							982	23	6	29
						Mean	986	27	6.4	33
						21	413	42	7	49
							513	36	7.8	44
						Mean	463	39	7.4	47
						28	135	30	6.5	36
							169	25	11	36
						Mean	152	27	8.6	36
3	1	1500×6h	50.2±1.6	0	25.2±0.6	0	2760	21	5.7	27
							2506	24	5.8	30
						Mean	2633	22	5.8	28
						7	2022	44	5.3	50
							1695	33	4.2	37
						Mean	1858	39	4.8	44
						14	788	34	5.8	40
							755	21	8.4	30
						Mean	771	28	7.1	35
						21	336	28	5.1	33
							323	36	5.4	42
						Mean	330	32	5.3	37
						28	146	42	5.9	47
							132	38	5.8	44
						Mean	139	40	5.8	46
4	1	1500×6h	50.3±1.1	0	25.3±0.7	0	3098	39	13	52
							2777	30	15	45
						Mean	2938	35	14	49
						7	1493	30	4	34
							1824	38	2.7	40
						Mean	1658	34	3.3	37
						14	791	18	1.7	20
							841	28	4.5	33
						Mean	816	23	3.1	26
						21	372	29	6.9	36
							403	31	7.5	39
						Mean	387	30	7.2	37

trial	chamber type	Rate (mg ai/L)	temp during treatment (°C)	incubation (days @ 38 °C)	storage temp (°C)	days of storage	PPO (mg/kg)	PCH-1 (mg/kg)	PCH-2 (mg/kg)	Total PCH (mg/kg)
						28	152	33	5.1	38
							77.8	28	7.6	35
						Mean	115	30	6.3	36
5	1	1500×6h	49.6±1.0	0	25.4±0.9	0	2508	45	5.9	51
							2388	38	8.5	46
						Mean	2448	41	6.8	48
						7	1773	30	11	41
							1762	41	4.6	45
						Mean	1767	35	6.5	42
						14	808	36	4.9	40
							650	25	<2	27
						Mean	729	30	4.4	35
						21	337	32	4.9	37
							343	29	5.4	34
						Mean	340	31	10	41
						28	70.4	32	3.3	36
							114	44	3.4	48
						Mean	92.4	38	8.7	47
11	2	1500×6h	49.5±0.8	0	25.0±0.4	0	2162	32	<2	33
							2411	24	2.4	26
						Mean	2286	28	<2	30
						7	1773	43	5.3	48
							1681	40	2.7	42
						Mean	1727	41	4	45
						14	635	12	7.4	19
							623	25	3.4	29
						Mean	629	18	5.4	24
						21	361	23	8.5	31
							304	28	7.6	35
						Mean	332	25	8	33
						28	157	22	3.8	26
							160	29	<2	30
						Mean	158	25	2.8	28
12	2	1500×6h	50.1±0.8	0	25.1±0.5	0	2598	17	5.4	22
							2444	25	4.3	29
						Mean	2521	21	4.8	26
						7	1469	39	6.6	45
							1909	36	11	47
						Mean	1689	38	8.7	46
						14	776	36	5.9	42
							1018	13	2.2	15
						Mean	897	24	4.1	28
						21	384	20	8.6	28
							403	40	7.8	48
						Mean	394	30	8.2	38
						28	149	14	10	24
							142	23	<2	25
						Mean	145	19	6	25
6	1	2000×4.5h	50.1±1.0	0	25.1±0.9	0	3270	37	6.6	44
							3471	34	2.6	36
						Mean	3371	36	4.6	40
						7	1997	37	7.9	45
							1887	28	3.1	32
						Mean	1942	33	5.5	38
						14	821	36	5.3	41
							765	48	8.1	56
						Mean	793	42	6.7	48
						21	367	31	4.7	36
							348	19	5	24
						Mean	358	25	4.9	30

trial	chamber type	Rate (mg ai/L)	temp during treatment (°C)	incubation (days @ 38 °C)	storage temp (°C)	days of storage	PPO (mg/kg)	PCH-1 (mg/kg)	PCH-2 (mg/kg)	Total PCH (mg/kg)
						28	81.2	21	<2	21
							129	26	7.8	33
						Mean	105	23	4	27
7	1	2000×4.5h	49.9±0.9	0	25.1±0.9	0	3183	40	8.3	49
							3368	31	4.5	36
						Mean	3275	36	6.4	42
						7	1353	34	6.6	41
							1281	23	2.7	26
						Mean	1317	29	4.7	33
						14	1040	45	4.4	49
							942	30	5.7	36
						Mean	991	37	5.1	42
						21	337	31	6.4	37
							303	18	4.6	23
						Mean	320	24	5.5	30
						28	164	33	9.6	42
							237	44	5.6	50
						Mean	200	38	7.6	46
8	1	2000×4.5h	50.0±0.6	0	25.0±0.9	0	2794	37	7.5	45
							2572	48	6.8	54
						Mean	2683	42	7.2	50
						7	2495	26	3.8	30
							2493	34	6	40
						Mean	2494	30	4.9	35
						14	706	36	9	45
							637	39	4.5	43
						Mean	671	38	6.7	44
						21	379	26	9.1	35
							314	12	<2	13
						Mean	347	19	5.3	24
						28	129	7	<2	9
							31.9	24	<2	26
						Mean	80.4	16	<2	17
9	1	2000×4.5h	50.2±0.7	0	25.3±1.0	0	3304	35	3.8	39
							3857	41	8	49
						Mean	3581	38	5.9	44
						7	2497	19	4.6	23
							2169	28	6.8	35
						Mean	2333	23	5.7	29
						14	883	35	5.9	40
							890	33	12	44
						Mean	887	34	8.7	42
						21	377	9	4.1	13
							373	16	5	21
						Mean	375	13	4.5	17
						28	96.9	36	<2	38
							86.6	21	2.8	24
						Mean	91.8	29	2.1	31
10	1	2000×4.5h	48.9±0.8	0	25.2±1.1	0	3994	29	4.7	34
							3582	40	9.5	49
						Mean	3788	34	7.1	42
						7	1892	18	5.3	24
							1854	32	7.9	40
						Mean	1873	25	6.6	32
						14	903	33	8.5	42
							842	34	4	38
						Mean	872	34	6.2	40
						21	332	31	6	37
							344	21	<2	23
						Mean	338	26	4	30

trial	chamber type	Rate (mg ai/L)	temp during treatment (°C)	incubation (days @ 38 °C)	storage temp (°C)	days of storage	PPO (mg/kg)	PCH-1 (mg/kg)	PCH-2 (mg/kg)	Total PCH (mg/kg)
						28	74.8	8	<2	9
							153	24	9.7	33
						Mean	114	16	5.8	21
13	2	2000×4.5h	50.1±1.0	0	25.4±1.0	0	3259	22	7	28
							2963	26	6.9	33
						Mean	3111	24	6.9	31
						7	1432	29	7.9	37
							1523	34	8.2	43
						Mean	1478	32	8.1	40
						14	1051	42	9.6	52
							924	37	6.1	43
						Mean	987	40	7.8	48
						21	262	11	9.4	20
							313	20	3.1	23
						Mean	287	15	6.3	22
						28	98.4	40	3	44
							108	33	5.6	38
						Mean	103	37	4.3	41
14	2	2000×4.5h	50.2±1.0	0	25.2±0.9	0	3267	31	4.9	36
							2932	24	5	29
						Mean	3100	27	5	32
						7	2154	41	5.9	47
							2351	33	6.1	39
						Mean	2252	37	6	43
						14	827	20	3.5	23
							755	22	7.4	29
						Mean	791	20	5.5	26
						21	379	30	<2	32
							344	17	8.4	26
						Mean	361	24	5	29
						28	104	41	9.9	51
							128	36	5.7	42
						Mean	116	38	7.8	46

Chamber type 1 = 3760 L, 2 = 94238 L.

APPRAISAL

Propylene oxide is used in agriculture as an insecticidal fumigant and sterilant, to control bacterial contamination, mould contamination, insect infestations, and microbial spoilage of food products, as well as to control insects in non-food products. Propylene oxide is also a commercially important industrial chemical finding application as an intermediate for a wide array of products. It was first evaluated by JMPR in 2011. Residue definitions established by the 2011 JMPR are:

Definition of the residue (for compliance with MRL) for plant comodities: *propylene oxide*.

Definition of the residue (for estimation of dietary intake) for plant commodities: *propylene oxide, propylene chlorohydrin and propylene bromohydrin. Propylene chlorohydrin and propylene bromohydrin to be considered separately from propylene oxide.*

The residue is not considered fat soluble.

Propylene oxide was scheduled at the 48th Session of the CCPR for the evaluation of additional MRLs by the 2017 JMPR.

Residue studies were submitted for tree nuts.

Methods of analysis

Descriptions of analytical methods together with validation data for residues of propylene oxide (PPO), the two isomers of propylene chlorohydrin (PCH) (1-chloropropan-2-ol (PCH-1), 2-chloropropan-1-ol (PCH-2)) and the two isomers of propylene bromohydrin (PBH) (1-bromopropan-2-ol (PBH-1) and 2-bromopropan-1-ol (PBH-2)) in tree nuts were made available to the Meeting.

Residues of PPO, PCH, and PBH were measured using GC-MS. Gas chromatographic retention time and/or mass spectrometry were used for confirmation of chemical identity. Respective GC-EIMS LOQs for PPO, PCH-1 and PCH-2, in non-concentrated MTBE extracts based on the lowest fortification level for which satisfactory recoveries were obtained are 1, 2 and 2 mg/kg for PPO, PCH-1 and PCH-2 respectively in almonds and walnuts.

GC-ECD: With a 10-fold concentration of the MTBE extract, LOQs for PBH-1 and PBH-2 were 0.08 mg/kg for almonds and walnuts and for a 20-fold concentration were 0.04 mg/kg. PPO is not amenable to analysis by GC-ECD.

Stability of residues in stored analytical samples

PPO is particularly volatile. In the supervised residue trials samples were extracted on the day of collection.

Results of supervised residue trials on crops

The Meeting received information on supervised field trials on post-harvest fumigation of almonds (shelled) and walnuts (in-shell and shelled).

The critical GAP in the USA for post-harvest treatment of tree nuts (includes almonds and walnuts) is fumigation at 2 g ai/L in a sealed chamber and at temperatures of less than 52 °C for up to 6 hours. This is followed by four flushes of the chamber with air or an inert gas and storage for a post-fumigation interval of 28 days at 25° or 35 °C or until the residues of propylene oxide decline to below 300 mg/kg.

It was observed that although residues in nuts fumigated at 2 g ai/L chamber for 4.5 hours were greater than those fumigated at 1.5 g ai/L for 6 hours at the first sampling time, 0 hours after fumigation, at ≥ 7 days off-gassing there was no statistical difference in residue levels. It was proposed that since air is nearly saturated with propylene oxide on fumigation at 2 g ai/L and at ~600 mmHg, that propylene oxide sorption to the nutmeat surface exceeds monolayer coverage for the two applied doses. Propylene oxide sorbed to the adsorption layer, which is reflected in the levels immediately after fumigation, is no longer present in samples during off-gassing at ≥ 7 days. The Meeting agreed the results for fumigation conducted at 1.5 and 2 g ai/L could be considered to approximate critical GAP and the results could be combined for the purposes of estimating maximum residue levels.

However, the Meeting also noted issues with the residue data. The laboratory sample for analysis was a 25 g subsample for almonds and a 15 g subsample of nutmeat for walnuts. The laboratory sample sizes do not correspond to the generally accepted prescribed sample size of 1 kg for tree nuts, see the "*FAO manual on the submission and evaluation of pesticide residues data for the estimation of maximum residue levels in food and feed*, 3rd edition" (2016), p 168. There is a concern that variability in residues, even for post-harvest fumigation, is such that the results may not adequately represent the average residue in the lot sampled. While the Meeting noted the difficulties presented by the analysis of a volatile fumigant such as PPO the small sample size was considered unacceptable.

In addition, clarification is required as to the calculation of residue values in the analytical method.

In summary, the trials approximating cGAP for almonds and walnuts were not considered valid as the sample analysed was too small (2×15+/–25 g). Additionally clarification regarding the calculations of residue levels is required.

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