FENBUTATIN OXIDE (109)

EXPLANATION

Fenbutatin oxide was first reviewed by the 1977 JMPR for both toxicology and residues. It was scheduled for a periodic re-evaluation by the 1992 JMPR at which time the toxicology re-evaluation was conducted. Owing to the work load and late receipt of data, the residue re-evaluation was postponed until 1993. The 1993 Meeting received residue data and information on GAP for most of the commodities with current CXLs (although in some cases only summary data) and for several commodities currently without CXLs. Extensive GAP information for Europe and some from many other parts of the world was also provided.

The data available to the Meeting included a joint submission to the 1992 JMPR and a 1992 submission to the European Commission (EC). Except for certain studies on apples, grapes, plums and cucumbers in the joint submission to the 1992 JMPR, the EC submission includes (reformatted) most of the residue information available for review by the Meeting. Less comprehensive information was provided for critical supporting studies except processing studies,.

IDENTITY

ISO common name: fenbutatin oxide

Chemical name:
IUPAC: bis[tris(2-methyl-2-phenylpropyl)tin] oxide

Chemical Abstracts: hexakis(2-methyl-2-phenylpropyl)distannoxane

Synonyms: SD 14114, SD 14114-U, "Torque", "Vendex", "Osadan"

Structural formula:

Molecular formula: $C_{60}H_{78}OSn_2$

Molecular weight: 1053

Physical and chemical properties

Pure active ingredient

Vapour pressure: Not volatile. 8.5×10^{-8} Nm^{-2} at 20°C (Shell)

Melting point:	140-145 °C (Fisk, 1991)	
Octanol/water partition coefficient:	1.4 x 10^5 (log K_{ow}	5.15) (Melander, 1988)
Solubility (g/l):	water	5 x 10^{-6} at 23°C (1977 JMPR)
at 20°C:	hexane	3.49 (MacDonald *et al.*, 1992a)
	ethyl acetate	11.4 (MacDonald *et al.*, 1992a)
	propan-2-ol	25.3 (MacDonald *et al.*, 1992a)
	toluene	70.1 (MacDonald *et al.*, 1992a)
	benzene	140 (1977 JMPR)
	methanol	182 (MacDonald *et al.*, 1992a)
	dichloromethane	310 (MacDonald *et al.*, 1992a)
Absolute density (powder):	1.31 g cm^{-3} (Fisk, 1991)	
Hydrolysis:		None up to 30 days in 25°C dark sterile aqueous solutions buffered at pH 5, 7 or 9 (Horne, 1988)

Other properties: (MacDonald *et al.*, 1992b):

EEC Method	Test	Result
A7	Fat solubility	1.1 g/100g at 37 °C
A10	Flammability	none (melts)
A16	Auto-flammability	none

Technical material

Purity: ≥ 97% w/w (1977 JMPR). February-May 1990 5-batch composite yielded 98.8% fenbutatin oxide, other components ≤0.014% except one up to 0.8%. The Meeting was provided with the identity of the components (Broadbent and Smith, undated).

Formulations

Wettable powder: 250 and 500 g ai/kg. Suspension concentrate: 500 and 550 g ai/l
Dusting powder: 20 g ai/kg. 4L Liquid: 42% ai by weight (4 lb. ai/gallon = 479 g ai/l)

USE PATTERN

Several formulations are registered for a variety of crops around the world, including suspension concentrate (SC) formulations at 550 g ai/l, dustable powders (DP) at 2%, wettable powders (WP) at 25 and 50%, and liquid formulations (4L) at 42% by weight. These may act as contact and stomach acaricides for the control of various mites in a variety of fruit, vegetable and field crops. Registered or approved uses were provided in the form of labels, label translations or GAP provided by national authorities. Table 1 summarizes registered and approved uses in countries for which supervised trials data were provided or countries whose GAP is most relevant to the conditions of the reported trials.

Table 1. Summary of fenbutatin oxide approved and registered uses.

Crop Country	Application			PHI, days	Comments
	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.		
Apples					
Australia	SC	(22)	as reqd.	2	
Austria	SC	(50)	1	21	≥1000 l/ha
Belgium	WP SC	(25) (27.5)	multi multi	28 28	
Canada	SU	0.3-0.9	≤4*	14	*incl. pre-bloom
Cyprus	WP	(30-50)	repeat*	14	*after 10-15 d.
Germany	WP	0.45 (30)	multi*	14	*Reregistration expected 1993
Greece	SC	(22-27.5)		14	
New Zealand	WP	0.5-0.6[1] (20)	repeat* 2-3	14	*after 7-10 d.
Spain (top fruit) pome fruit	WP FL	0.4-1 (25-50) 0.4-0.8 (28-55)	 1-2	21	
USA (same for pears)	4L (liq.)	0.6-1.7 (conc. spray) (15-30) (dil. spray)	≤4*	14	* ≤3 petal fall to harvest
	WP	0.8-1.7 (conc. spray) (15-30) (dil. spray)	same	14	same
Bananas					
Australia	SC	0.2 (≤50)	as reqd.	1	≥400l/ha
Guatemala	WP	(21)		14-21	
Spain	SC	0.4-1.1(27.5)		21	
	WP	0.4-1.1(25)		21	
	FL	0.4-0.8 (28-55)	1	21	
Beans					
France	SC	(50)		7* 3*	*"vines" *veg. crops
Germany (legume veg.)	WP	0.2-0.3 (30)		21	
Netherlands (glass)	WP	(25)	repeat*	7	*after 10-12 d
Spain (green beans)	WP	0.25-0.5 (25-50)	1-3	10	Field or greenhouse

Table 1 (contd.)

Crop/ Country	Application			PHI (days)	Comments
	Formu-lation	Rate per applicn. k gai/ha (g ai/hl)	No.		
Citrus					
Australia	SC	(11-25)	repeat*	7	*as required
Brazil	SC	(30)	repeat*	14	*as required
Cyprus	WP	(30-50)		7	
Greece	SC	(22-27.5)		**1**	
Guatemala	WP	(28-50)		14	
Italy	SC WP	(50) (30-40)		60 60	
Spain	SC DP WP FL	(28-55) 0.4-0.6 (25-50) 1.1-3.3 (28-55)	 1	21 15 21 21	1500-2000l/ha 1500-2000l/ha 4000-6000l/ha
Uruguay	SC	(30-40)		14	
USA	WP	1.1-2.2 [conc. spray] (15-30) [dil. spray]	2 (max./ 12 mo.[2]	7	≥60 days between sprays
	4L (liq.)	same as WP	same as WP	7	same as WP
Orange or tangerine					
Brazil	SC	(40)		14	
Cucumber					
Belgium (under glass)	WP	(25)	repeat*	3	*after 10-12 days
Canada (glass)	WP	(25)	repeat*	3	* as required
Denmark (under glass)	WP	(25)		3	
France	SC	(50)	*	3	*flowering and during honey dew
Germany (fruiting veg.)	WP	0.2-0.3 (30)		4	
Hungary (field or glass)	WP	0.25-0.3 (20-50)*		3	*600-1500l/ha (more dilute in greenhouse)

Crop/	Application			PHI,	

Country	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.	days	Comments
Cucumber					
Italy	SC WP	(50) (30-40)		30 30	
Netherlands (glass)	WP	(25)		3	
Poland (under cover)	WP	0.25-0.5 (25)	repeat*	3	*if necessary
Spain (cucurbits)	SC	0.4-1.1 (28-55)		8	
	DP WP	0.4-0.6 0.4-1 (25-50)		21 10	
Switzerland (glass)	FL WP	0.28-0.55* (25)	1-3 repeat*	10 3	* 28-55 g ai/hl *after 3-7 days
UK		(25)	*	3	greenhouse *No information
Egg plant					
Belgium (glass)	WP	(25)	repeat*	3	*after 10-12 days
Netherlands (glass)	WP	(25)		3	
USA	WP or 4L liq.	1.1-2.2	≤6	3	≤6.7 kg ai/ha per annum
Grapes					
Austria	SC	0.2-0.3 (28)		21	
Cyprus	WP	(30-50)		7	
France	550g/l	0.35		28	Dual pack applied in mix (no label)
	SC	(50)	*	7	* flowering and honeydew
	WP	0.5 (50)		7	(no label)
Germany	WP	(25)	≤2	28	Reregistration expected 1993
Hungary	SC	0.6-0.7		10	1200-1500 l/ha

Table 1 (contd.)

Crop/	Application	PHI,	

Country	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.	days	Comments
Grapes cont'd					
Italy	SC	(50)		45	
	WP	(30-40)		45	
Netherlands	SC	(25)	multi*	42	* with egg hatch blossoming after harvest
Spain	SC	0.4-1.1 (28-56)		21	
	WP	0.4-1 (25-50)		21	Field
	FL	0.17-0.33 (28-55)	1	21	*up to 56 g ai/hl
Switzerland	SC	0.6-1.1 (28-33)*		21	* same
	WP	0.5-1 (25-30)		21	
	WS*	0.5 (23)			* mix, 4.5% f. oxide
USA	WP	0.7-1.4 (30-60)	≤2*	28	* 28 d. between applications
	4L liq.	same	same	same	same
Hops					
Australia	SC	(22)	repeat*	2	*as required
Germany	WP	0.2-0.9 (30)		14	
Greece	SC	(23-28)		1	
Japan	WP	(16-25)		45	

Table 1 (contd.)

Crop/ Country	Application			PHI, days	Comments
	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.		
Melons					
Belgium	WP	(25)		3	
France	SC	(50)	*	3	*during flowering and honeydew
Germany (fruiting veg.)	WP	0.2-0.3 (30)		4	
Netherlands (incl. glass)	WP	(25)		3	
Spain (cucurbits)	SC WP	see cucumber see cucumber		8 10	
Switzerland (glass)	WP	(25)		3	
Nuts					
Almonds, walnuts					
Cyprus	WP	(30-50)		14	
USA	WP or 4L liq.	0.7-1.4 [conc. sprays] (15-30) [dil. sprays]	≤2*	14	* per season; 4700 l/ha max.
Pecans					
USA	WP or 4L liq./	see almonds/ walnuts	≤2*	14	* same
Peppers					
Belgium (glass)	WP	(25)	repeat*	3	* as required after 10-12 d.
Denmark (glass)	WP	(25)		3	
Germany (fruiting veg.)	WP	0.2-0.3 (30)		4	
Hungary (paprika, incl. glass)	WP	0.3*			* 600 l/ha open, 1000- 1500 glass
Netherlands (incl. glass)	WP	(25)		3	

Table 1 (contd.)

Crop/ Country	Application			PHI, days	Comments
	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.		
Pears					
Australia	SC	(11-22)	repeat*	2	*as required
Belgium	WP	(25)	repeat*	28	*after 15 d.
	SC	(28)	repeat*	28	*after 15 d.
Germany (pome fruit)	WP	(25)	repeat*	14	*after 1-2wk. Reregistration expected 1993
Netherlands	WP or SC	(25)		28	
New Zealand (pome fruit)	WP	0.5-0.9 (20)	repeat*	14	*after 7-10 d.
Spain (top fruit)	WP	0.4-1 (25-50)		21	
USA see apples					
Raspberries					
Netherlands	SC	(25)		*	*pre-bloom spray
Poland	WP	0.5*			*750-1000 l/ha; pre-bloom and post-harvest
Soya beans					
France (beans)	SC	(50)	*	7	* flowering & during honeydew
Germany (legumes)	WP	0.2-0.3 (30)		21	
Spain (beans)	SC	0.4-1.1 (28-55)		8	
	WP	same		10	
Stone fruits					
Germany	WP	0.5 (25)	repeat*	14	*after 1-2 wk
New Zealand	WP	0.5-0.6[1] (20)	2-3*	21	*as reqd. Oct.-Dec.
Poland	WP	0.5		14	500-1000 l/ha

Table 1 (contd.)

Crop/ Country	Application			PHI, days	Comments
	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.		
Stone fruits cont'd					
Cherries					
Germany	WP	(25)	repeat*	14	* after 1-2 wk Reregistration expected 1993
Italy	SC	(50)		30	
	WP	(30-40)		30	
Netherlands	SC	(25)		42	incl. Morrello
Spain	FL	0.42-0.83 (28-55)	12	21	Field
USA (sweet and sour)	WP or 4L liq.	0.8-1.7 [conc. spray] (15-30) [dilute spray]	≤2*	14	* with fruit on tree; ≤ 5640 l/ha for dilute spray
Peaches					
Australia	SC	(11-22)	repeat*	14	* as required
Denmark (glass)	WP	(25)		28	
Germany	WP	(25)	repeat*	21	*after 1-2 wk; Reregistration expected 1993
	WP	0.5 (30)		21	
Hungary	SC	0.6-0.7		14	1200-1500 l/ha
Italy	SC & WP	see cherries		30	
USA	WP or 4L liq.	0.6-1.1 [conc. spray] (15-30) [dilute spray]	≤2	14	≤4760 l/ha for dilute spray
Plums					
Germany	WP	(25)	repeat*	14	*after 1-2 wk Reregistration expected 1993
Hungary	SC	0.6-0.7		21	1200-1500 l/ha
Italy	WP	see cherries		30	
Netherlands	SC	(25)		42	
USA	WP or 4L Liq	see peaches	see peaches	14	see peaches

Table 1 (contd.)

Crop/ Country	Application			PHI, days	Comments
	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.		
Stone fruits contd.					
Nectarines					
Australia	SC	(11-22)		14	
USA	WP & 4L liq.	see peaches	see peaches	14	see peaches
Strawberries					
Australia	SC	0.17(<8.5)* 0.22(8.5-55)* 0.39(96-193)* (40)[3]	repeat as req. (up to 3)	1	* >2000 l/ha * 400-2000 l/ha * 200-400 l/ha
France	SC	(50)	*	5	* flowering & during honeydew
Germany	WP	(25)		*	* pre-bloom & post-harvest registration pending
	WP	0.5 (30)		*	* pre-bloom & post-harvest; 1500 l/ha
Netherlands	SC	(25)		*	* pre-bloom & post-harvest
UK (glass)	WP	(25)		7*	* 3 for "tunnel" grown
USA	WP or 4L Liq.	0.6-1.1 [conc. spray] (45-60) [dilute spray]	≤4*	1	* per season
Tomatoes					
Belgium (glass)	WP	(25)	repeat*	3	*after 10-12 d.
Canada (glass)	WP	(25)	repeat*	5	* as required
Denmark (glass)	WP	(25)		3	
Germany (fruiting veg.)	WP	0.2-0.3 (30)		4	600-1000 l/ha

Table 1 (contd.).

Crop/ Country	Application			PHI, days	Comments
	Formu-lation	Rate per applicn. kg ai/ha (g ai/hl)	No.		
Tomatoes cont'd					
Hungary (incl. glass)	WP	0.25-0.3		4	600 l/ha in open; 1000-1500 l/ha glass
Italy	WP SC	(30-40) (50)		30 30	
Netherlands (glass)	WP	(25)	repeat*	3	* after 10-12 d.
UK (glass)	WP	(25)	as required	3	≥10 d. between applicns.
Tea					
Japan	WP	(16-25)	1	21	excludes tea grown under cover

[1] Label rate cites 20 ai/hl and 2500-4500 l/ha (0.5-0.9 kg ai/ha). New Zealand government submission cites maximum GAP of 20 g ai/hl and 0.6 kg ai/ha.
[2] Previous US labels permitted up to 4 applications per season.
[3] Label rates given in ml product/ha (converted to kg ai/ha in Table 1), with repeat applications. The g ai/hl rates are calculated from the range of label volumes cited for each g ai/ha rate. A 1992 JMPR submission cites 0.039% ai and maximum of 3 applications.

RESIDUES RESULTING FROM SUPERVISED TRIALS

In plants

The Meeting received 251 individual studies or reports representing 456 supervised trials on 29 different commodities (and processed products from some of them) in many countries. Supervised trials data are summarized in 7 tables:

Table
2. Selected crops - avocado, bananas, beans, cucumbers, egg plant, hops, melons, peppers, soya beans and tomatoes.
3. Citrus
4. Grapes
5. Tree Nuts
6. Pome fruit
7. Raspberries and strawberries.
8. Stone fruit

Underlined residues in the Tables are from treatments approximating GAP.

The analytical methods for fenbutatin oxide and the metabolites dihydroxybis(2-methyl-2-phenylpropyl)stannane (SD 31723) and 2-methyl-2-phenylpropylstannoic acid (SD 33608) are described in the section "Methods of residue analysis". Methods used in individual trials are referred to by their code numbers in the paragraphs on the commodities concerned.

Avocado. Supervised trials data (Table 2) were available from Australia and the United States. Trials on single trees with 3 replicates/tree and application by boom sprayer were carried out in Australia and trials with

multi-tree replicates and ground hydraulic sprayers in the USA. Results were generally uncorrected for controls or recoveries (mostly >80%) and samples were shipped and stored in an acceptable manner.

The analytical method SAMS 215-1 was used in the Australian trials and MMS-R-494-2 in the US trials.

Maximum residues ranged from 0.23 mg/kg at day 0 to 0.08 mg/kg after 28 days from maximum rates and 0.18 and 0.11 mg/kg at days 0 and 14 respectively from rates comparable to GAP for "stone fruit" in other countries. In two of the three studies samples were also analyzed for the metabolites dihydroxybis(2-methyl-2-phenylpropyl)stannane (SD 31723) and 2-methyl-2-phenylpropylstannoic acid (SD 33608). In the representative data provided neither was detected at a 0.01 mg/kg limit of detection, except in one sample with a residue of 0.02 mg/kg SD 33608.

Bananas. Results of supervised trials which appeared to be according to GAP were available from three locations and two years in Australia. Although application rates were in terms of spray concentration whereas GAP information was in g ai/ha, information on minimum spray volumes indicated that GAP rates were used. Applications were by backpack mister to run-off and transit and storage conditions were acceptable. The analytical method was SAMS 215-1. Recoveries were generally $\geq$90%. The interval from the last sampling to receipt at the laboratory was less than 2 months. The period of storage before analysis was not stated, but a period of less than 6 months can be deduced. No sample chromatograms were provided.

Results (Table 2) were not corrected for recovery or controls and reflect 1 to 4 applications at 20-40 g ai/hl. In one study peel weights averaged 39% of the total fruit weight. Most of the residues were in the peel (pulp residues were approximately 1-2% of the whole banana level). Whole banana residues are proportional to the spray concentrations used and increase with the number of applications. Residue levels in whole bananas on the day of application ranged from 1.6 mg/kg after 4 applications at 10 g ai/hl to 5.7 mg/kg from four applications at 40 g ai/hl. Residues from single applications at the same two rates ranged from 0.6 to 3.4 mg/kg after 2 days. Maximum pulp residues under all conditions were 0.14 mg/kg.

Beans. Supervised trials data were available from outdoor use of a mixed EC formulation on green beans in France and from two glasshouse trials with a WP formulation on French beans in The Netherlands (Table 2).

The French trial was on a 5 x 2.4 m plot with no replicates. Sample storage conditions were acceptable. One month elapsed between the last sampling and receipt of frozen samples, and extraction was three weeks later. Although the period from extraction to analysis was not stated, the date of the report indicated that it would have been less than three months. The method was MMS-R-494-2 for which the limit of detection in beans was reported as 0.01 mg/kg. The mean recovery was 91% a 0.1 mg/kg fortification level. Results were not corrected for recovery. Sample chromatograms were not provided.

The maximum residue from the single application to green beans was 0.5 mg/kg at the French PHI of 3 days for vegetables, although the field trial application rate of 0.4 kg ai/ha (75 g ai/hl) was higher than the 50 g ai/hl French GAP rate for SC formulations (no French GAP provided for mixed EC formulations). The ai/ha rate is comparable to the German 0.3 kg ai/ha GAP rate for a WP on legume vegetables, although the German rate is only 30 g ai/hl and the German PHI is 21 days. The g ai/hl field rate in the French trials is 3 times that for The Netherlands glasshouse WP use.

Four replicates were taken in glasshouse trials on French beans in The Netherlands. Samples were received in cool condition 1-2 months after the last sampling and were stored under acceptable conditions. The interval from laboratory receipt to analysis was not reported, but a period of less than one month can be deduced. The analytical method for fenbutatin oxide was SAMS 215-1 and for SD 31723 SAMS 121-1. Recoveries were 95% of fenbutatin oxide and 65% of SD 31723, both at 0.5 mg/kg fortification.

Maximum residues in whole French beans after 6 days in The Netherlands glasshouse trials were 0.4 mg/kg from applications at 20 g ai/hl compared to The Netherlands GAP of 25 g ai/hl with a 7-day PHI. No sample chromatograms were provided. No residues of metabolite SD 31723 were detected by TLC at a 0.1 mg/kg limit of detection.

Cucumber. Supervised trials data were available from four European countries and the United States, although available information indicates that the use is not GAP in the USA where the outdoor application rates in terms of both kg ai/ha and concentration were greater and PHI intervals shorter than European GAP. Generally the interval from harvest to laboratory receipt or extraction in the European trials was less than 4 months and sample handling and storage appear to have been adequate. However, the interval from receipt or extraction to analysis was not reported. This information was provided for the US studies, for which the sampling-to-analysis intervals were generally $\leq$ 2 months. While the US results may not reflect current GAP, they are useful to illustrate the effect of varying application rates or numbers of applications under the US trial conditions. While Table 2 summarizes only the results of US applications at 2.2 kg ai/ha, data from trials at 4.4 kg ai/ha were also provided and showed an increase in residue levels roughly proportional to the application rate.

Table 2 summarizes the data for single European applications (with one exception), with 9 trials representing GAP. Residues reflecting GAP ranged from 0.03 to 0.3 mg/kg fenbutatin oxide from the single applications. Residues of fenbutatin oxide in peeled cucumbers at PHIs near GAP were $\geq$33% of the whole- fruit residues. The US trials showed no residues of metabolites SD 31723 or SD 33608 after 2 days.

Egg plant. Residues were 0.15, 0.11 and 0.07 mg/kg 0, 7 and 14 days after a single application to egg plants in a French glasshouse trial (Table 2, reference 15) from 50 g ai/hl applications of an SC formulation. Although no GAP information was provided for France, one or more applications at 25 g ai/hl with a PHI of 3 days is GAP in other European countries.

Gherkins. Summary data were provided from The Netherlands on a trial in which a single application of a 50% WP formulation to glasshouse gherkins at 0.75 kg ai/ha gave rise to residues of 1.8, 0.8, 0.7 and 0.9 mg/kg fenbutatin oxide after 0, 3, 5, and 10 days respectively (Shell Chemie, 1974).

Hops. Supervised trials data were available from Australia and Germany (Table 2). No results from applications at GAP rate were available at the Australian 2-day PHI, although they were available for dry cones at 1 day, 3 days and later intervals. Maximum residues were 5.2 mg/kg from GAP application rates and up to 16 mg/kg from double rates. Maximum residues in green cones in the German trials were 0.3 mg/kg at the German 14-day GAP PHI, but from exaggerated application rates.

The analytical method used in the Australian trial was SAMS 215-1 with recoveries of 110% at 0.2 mg/kg fortification and recoveries were <0.2 mg/kg. The analytical method in the German trial was SAMS 345-1 with recoveries of 102% at 1.5 mg/kg. Controls were 0.01 mg/kg.

Melons. In a supervised French trial on Vedrantais melons in 1976 no residues of fenbutatin oxide or its metabolite SD 31723 (<0.01 mg/kg fenbutatin oxide, <0.1 mg/kg SD 31723) were detected 7 or 14 days after a single application of a WP formulation at 0.09 g ai/ha (30 g ai/hl). The French GAP PHI is 3 days. The analytical method was SAMS 215-1 for which fenbutatin oxide recoveries were 80% at 0.2 mg/kg with control values <0.01 mg/kg (Table 2, reference 18).

Peppers. Two supervised trials were available, one outdoor trial on red peppers in Belgium and one glasshouse trial on paprika peppers in The Netherlands. In the Belgian trial residues were 1 mg/kg at the Belgian glasshouse 3-day PHI from a single application (repeat permitted) at

glasshouse GAP rates. Residues in The Netherlands trial were 0.6 mg/kg at the 3-day GAP PHI from a single application at 0.5 kg ai/ha compared to GAP of 25 g ai/hl (Table 2 references 19 and 20).

Soya beans. In three supervised trials in France, 1 or 2 applications at 0.5 kg ai/ha (100 g ai/hl), twice the GAP spray concentration, gave no detectable residues (<0.01 mg/kg) 67-80 days after application. The French GAP PHI is 7 days however (Table 2, reference 21).

Tomatoes. Seven studies in 6 countries, involving 12 supervised trials, were available to the Meeting. Eight of the 12 trials were in glasshouses. The maximum residues reflecting GAP were 0.4 mg/kg in Denmark (glasshouse), 0.3 mg/kg in Italy (field), and 0.3 mg/kg in the UK (glasshouse). In the French glasshouse trial residues were up to 0.08 mg/kg 14 days after applications in accordance with the Italian application rate (no French GAP was provided). Glasshouse trials in The Netherlands were at 0.5 kg ai/ha whereas GAP in The Netherlands is 25 g ai/hl. Information was not sufficient to determine whether the trials reflected GAP. Data from South African field trials could also not readily be related to the GAP information available. No tomato processing data were provided.

In the South African trials no residues (<0.1 mg/kg) were found of the metabolite SD 31723.

The interval from laboratory receipt of the samples to analysis was ≤8 months except for 11 months in the French trials and 15 months in the South African trials. Samples were generally received and/or stored in an acceptable manner. The analytical methods were SAMS-215-1, MMS-R-345-1 and SAMS-332-1. The last included methylation with methyllithium/lithium bromide and GLC determination with an FPD. This appears to be similar to MMS-R-494-2. Analytical recoveries ranged from 80 to 110% at fortification levels varying from 0.1 to 1 mg/kg and apparent residues in untreated samples reported as ≤0.1 mg/kg.

Table 2. Residues of fenbutatin oxide and metabolites SD 31723[1] and SD 33608[2] in selected crops resulting from supervised trials.

Crop Country Year	Application			Residues, mg/kg, at intervals (days) after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
Avocado			Day	0 7 14 21 28	
				One tree (3 replicates) (mature, flesh):	
Australia 1982	55 SC	2	(20)	0.11 0.09 0.11	1
(avocado pears)			(40)	0.18 0.11 0.10	
				controls 0.02	
USA 1983				Single trees replicated 5 times (mature, flesh):[3]	2
	WP	3	2.8	0.09 0.18 0.09 0.05 0.03	
			(30)	0.08 0.07 0.09 0.08 0.03	
				0.07 0.08 0.08 0.04 0.02	
				0.07 0.10 0.07 0.03 0.02	
				0.06 0.06 0.12 0.04 0.04	
				5 trees/replicate; 3 replicates (mature, flesh):[3]	
1983	SC	3	2.8 (83)	0.07 0.07 0.05 0.08 0.07	3
			5.6 (165)	0.23 0.12 0.13 0.12 0.08	

Crop Country Year	Application			Residues, mg/kg, at intervals (days) after last application						Ref
	Form	No	Rate kg ai/ha (g ai/hl)							
				controls <0.01						
Banana			Day	0	7/8	14	16/17	21	Sample	
Australia	SC	4	(20)	0.05	0.03		0.04		pulp	4
Tully 1982			(40)	0.07	0.14		0.13		pulp	
				controls 0.02						
Euramo 1984	SC	4	(10)	1.6	1.7	1.6	1.0		whole	5
			(20)	3.2	3.5	3.1		2.2	whole	
				0.02	0.02	0.01		0.02	pulp	
			(40)	5.7	6.3	4.8		4.6	whole	
			GAP (≤50)	controls 0.01 pulp, 0.12 whole						
			Day	0	1	2	7/8	14	21	
Australia	SC	1	(10)			0.6	0.4	0.2	whole	6
Yanding 1981						<0.01	<0.01	<0.01	pulp	
			(20)			1.2	0.8	1.0	whole	
						0.02	<0.01	<0.01	pulp	
			(40)			3.4	2.7	2.3	whole	
						0.08	0.04	0.02	pulp	
				controls <0.01 whole and pulp						
Beans, green (whole)			Day	3		6		7	10	
France 1982 (outdoor)	EC	1	0.4 (75)	0.5				0.4	0.4	7

French beans (whole)				3 days	6 days					
Netherlands 1978	WP	2	1 (20)	0.8	0.6					8
(glasshouse)				0.7	0.4	Replicate sub-samples				
				0.7	0.3	Replicate				
				0.7	0.4	Replicate				
1978	WP	2	0.5 (45)	0.6	0.2	Controls <0.02				
Cucumbers			Day		0	2	3/4	7/10		
Denmark 1977	WP	1	0.63 (25)				0.03			9
		2					0.07			
				controls		<0.01				
				SD 31723			<0.1			
France 1980	WP	1	0.75 (50)	whole	0.1	0.1		0.03		10
glasshouse				pulp	0.03	0.03		<0.01		
				control <0.01						
			Day		0	1	2	3/4	5/6	7/10
France 1980	WP	1	0.5 (50)	whole	0.1			0.08		0.04	10
glasshouse				pulp	0.03			<0.01		<0.01	
			0.95 (50)	whole	0.4			0.1		0.07	
			0.95 (50)	pulp	0.08			0.03		<0.01	
				untreated <0.01							
1982	EC	1	0.64 (38)	whole	0.08	0.09				0.07	
glasshouse				pulp	<0.01	<0.01				<0.01	
				untreated <0.01							
Netherlands	WP	1	(25)	whole	0.2			0.1	0.1	<0.1	12
1974		1	(25)	whole	0.2			0.2	0.2	<0.1	
				untreated <0.1							
UK 1980	WP	1	(50)	whole	0.3	0.3	0.2	0.2	0.2		13
		1	(50)	whole	0.3	0.3	0.4	0.3	0.1		
		1	(100)	whole	1			0.5	0.3		
			Day		1		2				
USA 1988 FL	SC (4L)	2	2.2 (1800)		1		0.87				14
		3			1.1		0.6				
PA		2	2.2 (790)		0.4		0.2				
		3			0.3		0.3				
IN		2	2.2 (600)		0.2		0.4				
		3			0.5		0.2				
USA 1988 WI	SC (4L)	2	2.2(980)		0.3		<0.05				
		3			0.5		0.2				
CA	WP	2	2.2 (620)		0.5		0.2				
		3			0.4		0.2				
		2	2.2 (600)		0.7		0.2				

		3		0.9		0.5				
	SC (4L)	2	2.2 (480)	0.5		0.4				
		3		0.7		0.3				
		2	2.2(650)	0.3		0.09				
		3		0.3		0.2				
TX	SC (4L)	3	2.2 (480)	0.9		1				
		2	2.2 (610)	0.8		0.8				
			3	1		1.3				
				Controls ≤0.07 mg/kg SD 31723 & SD 33608 not detected (<0.05 mg/kg)						
Egg plant			Day	0	7	14				
France 1983 glasshouse	SC	1	1 (50)	0.15	0.11	0.07				15
Hops			Day	0	1*	3*	7	22		
Australia	SC	2	0.4 (22)	7.5	5.8	3.5	5.2	5.1		
1988 dry cones		1	0.8 (44)	11.2	16	16	14		* GAP = 2 days	16
			Day	0	7	10	14	23		
Germany 1989 green cones[4]	SC	3	4.5 (150)[5]	2.8	2.6	1.4	0.3	0.01		17

Melons			Day	0 3 7 14	
France 1976 Vedrantais	WP	1	0.09 (30)	<0.01 <0.01	18
Peppers					
Belgium 1975	WP	1	(25)	1.2 1 0.9 0.6	19
red pepper				controls <0.1	
Netherlands 1975 paprika (glasshouse)	WP	1	0.5	0.4 0.6 0.4 0.1 controls <0.1	20
Soya beans					
France 1988 Kador	SC	1-2	0.5 (100) [500l/ha]	65-70 days <0.01 (3) [3 trials, three locations]	21
Tomato			Day	0 1 3 7 14 30	
Denmark 1981	WP	1	0.75 (25)	0.4 0.4 0.5 (5 d.)	22
Ida glasshouse				0.3 (7 d.) controls <0.01	
France 1983 Foxy glasshouse	C	1	0.5 (50)	0.15 0.04 0.08	23
1983 Pyros HFI	SC	1	0.75 (50)	0.1 0.06 0.06 controls <0.02	
Italy 1974 Tonda Liscia	WP	1	(30)	0.4 0.2	24
1974 S. Marzano				0.5 0.3	
				controls <0.1	
Netherlands 1975 Sonate glasshouse	WP	1	0.5 [GAP= (25)]	0.2 0.4[2] 0.4 0.4	25
				controls <0.1	
S. Africa 1976	WP	1	1.5 (20)	0.5 0.4,4d. 0.4 0.2	
Heinz			3 (40)	1 0.7 0.8 0.6 control <0.1; SD 31723 <0.1	
UK glasshouse			Day	0 1 2 3 4 6 7	
Solatine 1980	WP	1	(50)	0.7 0.6 0.9 0.7 0.7	27
				0.3 0.2 0.3 0.4 0.3	
Eurocross 1974	WP	1	(100)	0.8 1 0.8 0.9 1.1 controls <0.01	28
			(25) [GAP]	0.2 0.3 0.4 controls <0.1	

Unless otherwise indicated, residues are parent compound only.

Notes to Table 2
[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane
[2] SD 33608 = 2-methyl-2-phenylpropylstannoic acid
[3] Representative data
[4] The Volume 9 detailed report gave no information on stage of crop, while the Volume 1 summary stated green cones.
[5] Rate is from Volume 1 of 1992 EC summary submission. The volume 9 detailed report gives application rates of 0.075% ai or 0.225 and 0.375 g ai/plant. Insufficient information was available to relate g ai/plant to g ai/ha. No explanation was provided for the discrepancy between the ai concentrations.

Citrus (Table 3). There were 27 studies in 5 countries representing 53 trials (38 from the USA) on oranges (29), lemons (9), limes (1), grapefruit (12) and mandarins (2). Of these trials 6 on oranges, 2 on grapefruit, 3 on lemons and 1 on mandarins reflect current GAP. Most US trials include 4 applications within 12 months compared with current GAP of 1 or 2/year with 60-day intervals. The excessive number of applications provide information for processing considerations, but not necessarily for estimates of maximum residue levels. Most but not all trials were on only single trees. Some low-volume applications are included. Generally sample transit and laboratory storage conditions were acceptable and sampling-to-analysis intervals were generally <1 year. Several analytical methods were

described, but most were validated at near tolerance levels (e.g. 5 mg/kg). Few limits of determination were provided, although the limits of detection were generally reported as 0.02 mg/kg for the parent compound. In several cases residues of two metabolites were also determined and in some trials processing fractions were analysed. Trials were with both SC and WP formulations, although generally similar residues resulted where comparisons could be made.

Maximum residue level reflecting GAP were 0.14, 0.2, 0.3, 0.7, 0.8, 1.3, and 3.2 mg/kg in oranges, 2.4 mg/kg in mandarins, 0.5, 2.4 and 4 mg/kg in lemons (the last at 21 days compared to GAP of 7 days) and 0.7, 0.9 and 1.5 mg/kg in grapefruit. Other trials at GAP rates but with twice the number of applications resulted in residues ranging up to 14 mg/kg, although most were $\leq$10 mg/kg. Although there were few trials on lemons reflecting GAP, one of them resulted in the highest residue from GAP rates, and at a 21-day PHI compared with the 7-day GAP PHI.

Table 3 shows residues of fenbutatin oxide in citrus pulp typically to be $\leq$5% of the whole fruit residue. It also shows that residues of the metabolites SD 31723 and SD 33608 were about 2-10% and 1-5% or less of the parent compound. However, the trials in which the metabolites were determined were not in accordance with current GAP.

Table 3. Residues of fenbutatin oxide and metabolites SD 31723[1] and SD 33608[2] in citrus and citrus by-products resulting from supervised trials.

Crop, Country, state, year, variety	Application			Residues, mg/kg, at intervals(days) after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
Oranges			Day	071428-30	
USA, FLA 1972	WP	4	1.1 (16)	whole0.60.50.80.8	1
Valencia		GAP=2		Replicate0.60.80.70.6	
	WP	4	2.1 (30)	whole1.51.521.5	
				Replicate1.61.51.51.7	
	WP	4	4.2 (60)	whole3.83.23.13.6	
			2xGAP rate	Replicate44.13.23.1	
				controls <0.05 mg/kg	
Oranges				Washing and processing (30 day samples):	
USA, FLA 1972				WholeWholeFinisherDry	
Valencia				unwashedwashedJuicepulppulp	
(contd.)	WP	4	1.1 (15)	0.24<0.05<0.05<0.05 0.3	
			2.1 (30)	1.30.05<0.05<0.05 1.2	
			4.2 (60)	1.40.4<0.05<0.05 2.2	
				SD 31723 (TLC method): <0.2 mg/kg all samples.	
				Method: MMS-R-345-1 GLC and TLC - not provided.	
CA 1978	SC	1	1.7 (30)	whole full size1.314 days	2
Navel			3.4 (60)	whole full size3.9 14 days	
				controls <0.02; SD 31723 and SD 33608 <0.02 mg/kg each	
	WP	1	1.7 (30)	whole full size0.8 7 days	3
			3.4 (60)	whole full size2.5 7 days	
				controls<0.02	
				SD 33608 0.02 both applicn. rates	
				SD 31723<0.02 low rate; 0.08 high rate	
				Distribution of residues (7-day PHI, replicates)	
				Peeled Whole orangePeel	
AZ 1978	SC	4	2.2 (130)	Parent5.5, 4 0.23, 0.275.6, 4.8	4
Valencia		(GAP		SD 317230.08, 0.07 <0.02(2)0.07,0.06	
		= 2)		SD 33608<0.02,<0.02 <0.02(2)≤0.02(2)	
				Total[3]5.6, 4.1 0.29, 0.235.7,4.9	
			4.5 (270)	Parent17, 9.6 0.82, 0.2623, 9.6	
				SD 31723[1]0.31, 0.26 <0.02(2)0.51,0.31	
				SD 33608[2]0.03, <0.02 <0.02(2)0.07,0.05	
				Total[3]17, 10 0.88, 0.4224, 10.1	
				Controls: Parent ≤0.04 in all fractions	
				SD 3172 and SD 33608<0.02 in all fractions	
				7 days	
FLA 1980	SC or	4	2.2 (48)	SC WP	
Hamlin	WP			Trial 1Trial 2Trial 1Trial 2	
				Parent 8.6, 8.79.33.7, 4 3	5
				SD 31723 0.7, 0.60.8 0.3, 0.33 0.4	
				SD 33608 0.4, 0.40.40.13, 0.15 0.13	
				Total[3] 10, 10114.4 , 4.7 4	
				controls <0.02 mg/kg in all compounds	
1980	SC or	4	2.2 (120)	SC WP	
Navel	WP	(GAP=2)		Rep 1Rep 2Rep 1Rep 2	
				Parent 1010711	6
				SD 31723 0.50.60.30.6	
USA, CA 1980				SD 33608 0.20.30.20.2	

Crop, Country, state, year, variety	Application			Residues, mg/kg, at intervals(days) after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
Navel contd.				Total[3] 1111812	
				controls <0.02 in all compounds	
				7 days	
				ParentSD 31723SD 33608Controls	
FLA 1979	SC	4	2.2 (70)	7, 5.40.7, 0.50.2, 0.2<0.02 all cpds.	
Hamlin			4.4 (140)	12, 141.1, 1.20.3, 0.4<0.02 all cpds.	7
				Parent SD 31723 SD 33608 (control)(control)(control)	
FLA 1987	SC	4	2.2 (80)	unwashed fruit3.3 0.19 <0.05	8
Hamlin		GAP		(0.21)(<0.05)(<0.05)	
		= 2		washed fruit2.10.1<0.05	
				(0.12)(<0.05)(<0.05)	
				oil emulsion0.05<0.05<0.05	
				(<0.05)(<0.05)(<0.05)	
				juice0.06<0.05<0.05	
				(<0.05)(<0.05)(<0.05)	
				chopped peel/4.30.190.1	
				pulp(0.1)(<0.05)(<0.05)	
				dried peel/160.70.2	
				pulp(0.4)(<0.05)(<0.05)	
				press liquor1.60.06<0.05	
				(0.07)(<0.05)(<0.05)	
				molasses0.6<0.05<0.05	
				(0.11)(<0.05)(<0.05)	
				peel frits8.80.60.2	
				(0.14)(<0.05)(<0.05)	
				orange oil231.2<0.05	
				(0.9)(<0.5)(<0.5)	
				finisher pulp<0.05<0.05<0.05	
				(<0.05)(<0.05)(<0.05)	
			Day	0714	
Australia 1982	SC	3	(20)	whole4.43.03.2	9
Navel				peel13 99	
				pulp<0.05<0.05<0.05	
	SC	3	(40)	whole7.44.84	
				peel211412	
				pulp0.10.050.1	
				controls<0.05 whole	
			Day	0 2 7 14	
Australia 1981	SC	1	(10)	whole0.47 0.210.230.14	10
Valencia				pulp<0.02<0.02<0.02<0.02	
				juice<0.02<0.02 <0.02<0.02	
	SC		(20)	whole1 0.80.60.7	
			[GAP]	pulp<0.02<0.02 <0.02 <0.02	
				juice<0.02<0.02 <0.02 <0.02	
				controls<0.02<0.02 <0.02<0.02	
				14 days	
				whole pulp	
Brazil 1985				Adjusted	
Natal				to GAP rate	
	SC	2	(45) [1.5XGAP]	0.40.27<0.03	11
			(60)	0.30.15<0.03	
			(90)	0.4<0.03	

Crop, Country, state, year, variety	Application			Residues, mg/kg, at intervals(days) after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
			(120)	0.9<0.03	
				controls<0.3 whole	
				115 days (GAP=21 days)	
				WholePulp (peeled orange)	
Spain 1973	WP	1	0.6	<0.05	12
Clementino			1.2	<0.05	
				controls<0.05 whole	
				156 days	13
Navel	WP	1	(15)	0.16<0.05	
			(30)	0.1<0.05	
			[GAP=25-50]	controls <0.1 whole	
Mandarins					
Australia 1982	SC	3	(20)	Day0 714	
Hickson			GAP=25	whole3.42.42.1	9
				peel149.58	
				pulp0.10.10.05	
	SC	3	(40)	whole6.23.82.6	
				peel251611	
				pulp0.250.10.15	
				controls0.05	
Lemons				Days314461	
Italy 1974	WP	1	(30)	whole0.90.60.5	14, 15
Nostrana				pulp0.1<0.02<0.02	
				juice0.08<0.02<0.02	
				controls <0.02<0.02<0.02 all fractions	
				Metabolite SD 31723 <0.1 mg/kg in all fractions	
				Days071421	
USA, CA 1981	WP	4	2.2 (20)	Parent3.4	16
Lisbon		GAP		SD 336080.05	
				SD 317230.2	
		=2		Total[3]3.7	
Eureka		1	1.7 (33)	<0.02	17
Eureka			1.7 (33)	whole4	18
				peeled fruit0.14	
				peel5.8	
				controls0.05 whole	
Eureka		1	1.7 (33)	whole2.12.41.51.3	19
				peeled fruit0.140.230.030.08	
				peel3.66.31.53	
				controls0.05 parent	
				7 days (the 2 results are replicates)	
				whole pulp peel	
USA, CA 1981	SC	4	2.2 (130)	Parent 11, 9.30.42, 0.3411, 10	20
Lisbon		GAP		SD 317230.3, 0.3<0.020.4, 0.3	
		=2		SD 336080.06, 0.07<0.020.08,0.06	
				controls<0.02 all compounds whole	
Lisbon	SC	4	4.5 (270)	Parent16, 321.2, 1.426, 28	
				SD 317230.6, 10.03, 0.041.3, 1	
				SD 336080.14, 0.17<0.020.3, 0.3	
				Controls	
AZ --	SC	4	4.5 (480)	Parent5.4, 3.80.03, 0.07	21
				SD 317230.3, 0.2<0.02	
				SD 336080.1, 0.08<0.02	

Crop, Country, state, year, variety	Application			Residues, mg/kg, at intervals(days) after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
CA Lisbon	SC	4	2.2 (20)	Parent4.9	16
				SD 317230.3	
				SD 336080.08	
Limes				Controls	
USA, FLA 1981	SC	4	4.5 (190)	whole25, 260.04	22
				SD 317230.9, 0.8<0.02	
				SD 336080.3, 0.3<0.02	
				7 days	
Grapefruit				ParentSD 31723SD 33608	
USA, TX 1979	WP	4	2.2 (96)	whole1.30.10.03	23
Red Blush		GAP=2		controls0.30.03<0.02	
FLA 1980	WP	4	2.2 (48)	whole2.5, 2.30.2, 0.20.1, 0.08	24
Marsh seedless				controls0.02<0.02 <o.o2	
TX 1977	WP	4	8.4 (60)	whole20.130.08	25
Ruby Red		4	16.8(120)	3.20.30.13	
				controls<0.02 <0.02<0.02	
				Days 0 7 142845	
CA 1972	SC	2	0.21 (15)	whole0.8,0.5, 0.9,0.4,0.4, 0.70.5 0.70.40.4	26
Red Blush			[GAP=(15)-(30)]		
				pulp0.14,0.02, 0.07,0.04,<0.02 0.10.05 0.04<0.02--	
				peel1.5,0.7, 1.6,0.8,0.5, 1.31.1 1.30.50.5	
	SC	2	0.4(30)	whole1.6,1, 1.3,0.9,0.9, 1.70.8 1.50.70.7	
				pulp0.23,0.09, 0.12,0.08,0.1, 0.20.1 0.20.060.02	
				peel2.5,1.7, 3,2,2.1, 3.3,2.3 2.51.61.4	
	SC		2	whole3.2,3.1, 2.5,1.9,1.7, 2.92.7 2.61.52.5	
			0.84 (60)	pulp0.5,0.2, 0.3,0.3,0.1, 0.40.09 0.30.20.1	
				peel5,6, 4,4,4, 44 534	
				SD 31723 <0.2 mg/kg each matrix and interval	
				7 days	
				(2 results are replicates)	
				wholepulppeel	
AZ 1979	SC	4	2.2 (130)	Parent10, 2.61.6, 0.19 21, 4.8	27
Ruby Marsh				SD 137230.2, 0.08≤0.020.2, 0.5	
				SD 33608≤0.02≤0.020.05,0.03	
		4	4.5 (260)	Parent6.6, 141.4, 1.721, 27	
				317230.2, 0.30.02, 0.050.5, 0.6	
				33608<0.02,0.03<0.020.04,0.07	
				controls<0.02 all compounds whole	
TX 1979	SC	4	2.2 (96)	Parent1.4	23
Red Blush				SD 137230.1	
				SD 336080.03	
		4	4.5 (192)	Parent3.7	
				SD 137230.3	

Crop, Country, state, year, variety	Application			Residues, mg/kg, at intervals(days) after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
				SD 336080.1	
				controls0.3 Parent, 0.03 SD 13723, <0.02 SD 33608	
FLA 1980				Parent5.2, 4.5	
Marsh seedless	SC	4	2.2 (48)	SD 137230.4, 0.4	24
				SD 336080.3, 0.2	
				controls<0.02 all compounds	

Unless otherwise indicated, residues are parent compound only.
[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane
[2] SD 33608 = 2-methyl-2-phenylpropylstannoic acid
[3] Total organotin residues of fenbutatin oxide and its two metabolites calculated as fenbutatin oxide.

Grapes. Studies were available from 5 countries representing over 60 supervised trials, two thirds of which were from the United States (Table 4). Several varieties of grape and WP and SC formulations were covered and many of the US studies also included analyses for the two major metabolites SD 31723 and SD 33608. Several studies included analyses of various processed fractions and one was a simulated processing study. Generally plot sizes and transit/storage conditions gave credence to the data. Analysis was typically <7 months after sampling. Various analytical procedures were used and described, but not provided (e.g. MMS-R-494-2, MMS-R-345-1, SAMS-345-1, MMS-391-1), with recoveries generally >70%. Limits of detection for fenbutatin oxide were <0.02 mg/kg in grapes, when reported. Control values were generally $\leq$0.05 mg/kg in grapes and often <0.02 mg/kg, depending on the study, but sometimes only recorded as 0.1 mg/kg. Residues have not been corrected for recoveries or control values.

Maximum residues in grapes from the French trials (Table 4 references 1-4) were approximately 1 mg/kg after 28 days or 55 days; the French PHI is 7 days for the WP formulation used. Twenty one to 28 days are common PHIs in other countries which generally have comparable GAP rates, at least on an ai/ha basis. In one trial (Table 4 reference 2) no residues (<0.02 mg/kg) were found in wine from grapes treated in accordance with GAP. Maximum residues reflecting German GAP from the German results were 0.5 mg/kg after 35 days compared to a 28-day PHI. Most of the German results were from application rates higher than GAP or from more than the recommended 2 applications. Residues were up to 1.9 mg/kg after 21 days at rates similar to the GAP rates of other European countries. Maximum residues from the Italian trials were 1.1 mg/kg under the GAP conditions of other European countries, although the PHI of 21 days is shorter than the Italian GAP PHI.

Maximum residues reflecting US GAP were 4.1 mg/kg, a level found in two different locations on two varieties in different years (Table 4 references 18 and 24). Maximum residues of SD 31723 and SD 33608 from uses according to GAP were 0.2 and 0.04 mg/kg respectively. Generally SD 31723 residues were $\leq$6% of fenbutatin oxide residues and in individual samples SD 33608 residues were about half those of SD 31723, as observed for citrus.

Table 4. Residues of fenbutatin oxide and metabolites SD 31723[1] and SD 33608[2] in grapes and grape processed products from supervised trials.

Country, Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
			Days	28*4855 136	1
France 1974	WP	1	0.5 (50)	0.3 * French WP GAP is 7 days	
Lavallet				0.5	
Grenache			0.5 (125)	1.1Controls <0.02	
			[GAP=(50)]		
1974 Grenache	WP	1	0.5 (50) or	wine<0.02 (whole grape values not given)	2
			(125)		
1972 Semillon	WP	1	(30)	0.7	3
			(50)	0.9	
				Controls<0.1	
1974 Grenache	WP	1	0.5	0.5	4
Merlot Rouge				0.13	
Merlot Rouge				<0.05	
				Controls <0.05	
Germany			Days	0 4 7 10 14 21 28 [GAP PHI]	
1979 Riesling	WP	2	0.4 (25)[GAP]	0.10.10.080.070.08	5
4 Trials				0.50.30.30.30.3	
				0.40.50.20.20.2	
				0.30.30.30.20.1	
				Controls <0.01	
1980 Riesling	WP	4	0.3 (20)	2.62.22.51.91.2	6
		4	0.3 (75)	2.21.42.41.41.8	
1980 Müller-Thurgau		5	0.3 (20)	1.60.90.40.40.4	
		5	0.3 (75)	0.50.50.50.50.4	
1980 Riesling		4	0.3 (20)	0.40.30.140.30.1	
		4	0.3 (75)	0.20.10.30.20.3	
				Controls <0.01	
			Days	014212835	
1987 Müller-Thurgau	SC	2	0.45 (56)	0.60.60.70.50.14	7
Portugieser			0.45 (50)	0.50.40.40.20.2	
Müller-Thurgau				0.80.20.70.2	
Spätburgunder			0.45 (25)	0.70.40.60.40.5	
Riesling			0.45 (50)	0.90.70.60.60.4	
				2.521.91.51.3	
				In each study filtered and unfiltered juice and	

Country, Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
				bottled and unbottled wine contained <0.01 mg/kg from 35-day grapes	
Italy 1974			Days	142131	
Barbera	WP	1	(30)	1.31.10.5	8
Nebbiolo	WP	1	(30)	1.81.10.7	
				Controls <0.02	
Switzerland 1974			Days	59127	
Pinot Noir	WP	1	(100)	<0.02	9
Riesling				<0.02	
Chasselas				2.1	
				Controls <0.1 mg/kg	
				Day 12 (GAP=28)	
USA				GrapeMaturing wine	
1974 Mission	WP	2	1.1	2.8<0.02	10/11
		2	2.2	2.90.02Controls <0.02 mg/kg both	
			Days	07142852	
1973 Rabier	WP	3	0.5 (30)	0.90.70.20.2	12
		3	1 (60)	20.632.3	
				07142852	
1973 Mission	WP	3	0.5 (30)	10.510.7	12
			1 (60)	2.20.50.71.2	
			0.5 (30)	1.51.51.81 raisin 4.2	
Thompson			1 (60)	3.91.81.40.7 raisin 3.2	
				Day 14	
				ParentSD 31723SD 33608	
Valdepeñas	SC	3	1.4 (75)	2.1, 3.1, 2.8, 0.06,0.07, <0.02 (5)	13
				4.5, 4.5 reps.0.09, 0.15 reps.	
	WP	3	1.4 (75)	7, 6, 5, 6, 50.2, 0.1, 0.2,≤0.04 (5)	
				replicates0.2 replicates	
				Controls 0.05	
	SC	3	1.4 (75)	0.5, 1.3,<0.02<0.02	14
				2 (re-analysis)0.12, 0.06<0.02	
	WP	3	1.4 (75)	0.4, 0.7≤0.03 (2)<0.02 (2)	
				Controls 0.13, 0.31 (contamination suspected, not	
				subtracted from treated sample values).	
				Day0142128	
Concord	WP	3	1.1 (60)	3.122.21.8	15/16
				Pomace5.2	

Country, Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
				dry pomace11 (55% moisture)	
				Wine<0.02	
		3	1.7 (90)	5.62.83.42.7	
				Pomace8.9	
				dry pomace15 (55% moisture)	
				Wine<0.02	
				Controls ≤0.04 mg/kg grape, 0.07 mg/kg pomace	
				Day14	
1975 Niagara	WP	1	0.56 (60)	1.2	17
			1.12(120)	1.6	
			2.2 (240)	1.3	
				Controls 0.02 mg/kg	
				Day30	
				wholeraisinwine dry pomace	
1976 Thompson	WP	2	1.1 (60)	4 15<0.02 45, 65 (10% water)	18
			2.2 (120)	13 51<0.02 240, 190	
				Controls: grape 0.04, raisin 0.12, wine <0.02,	
				dry pomace 0.6 mg/kg.	
				Day 28	
				ParentSD 31723 SD 33608	
1980 Thompson	SC	2	1.4 (50)	1, 1.1,0.08, 0.08, 0.02, 0.03	
				1.4, 2.10.08, 0.1 <0.02(2)reps	
	WP	2	1.4 (50)	1.4, 1.6,0.06, 0.06, <0.02 (2)	19
				1.9, 2.20.15, 0.16 0.04(2)	
				Controls: ≤0.06, <0.02<0.02	
1980 Thompson	WP	2	1.4 (50)	grapes2.3, 1.70.1, 0.09, 0.04, <0.02	20
				2.2, 1.80.03, 0.02	
				raisins2.70.1 <0.02	
	SC	2	1.4 (50)	grapes0.8, 0.6,0.03, 0.02, <0.02(2)	
				1, 0.90.02, <0.02	
				raisins1.70.04 <0.02	
				Controls: <0.02 ≤0.03<0.02	
				Day 28	
				ParentSD 31723SD 33608	
1980 Thompson	WP	2	1.4 (50)	grapes 1.5, 1.60.09, 0.060.04, 0.03	21
				raisins 3.2, 3.30.14, 0.150.03	
	SC	2	1.4 (50)	grapes 1.6, 0.70.05, 0.04<0.02(2)	

Country, Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
				raisins 2.8 2.90.1, 0.10.02	
1980 Carignan	WP	2	1.4 (50)	grapes 0.6, 0.5, 0.3,<0.02(4)<0.02(3)	22[3]
				0.2, 0.3	
				Controls <0.02 mg/kg, all compounds in all substrates	
	SC	2	1.4 (50)	grape 0.4, 0.3, 0.4≤0.02 (2)<0.02 (2)	
				Controls <0.02 mg/kg all compounds in all substrates	
				Day 14	
1978 Thompson	WP	3	1.1 (60)	grapes 0.7<0.02<0.02	23
			2.2 (120)	60.20.06	
			1.1 (60)	dry pomace*50.10.05	
			2.2 (120)	391.40.4	
				* lab dried to 10% moisture	
			1.1 (60)	juice<0.02<0.02<0.02	
			2.2 (120)	0.05<0.02<0.02	
			1.1 (60)	wine<0.02<0.02<0.02	
			2.2 (120)	<0.02,0.02<0.02	
			1.1 (60)	raisins40.120.08	
			2.2 (120	70.160.06	
			1.1 (60)	raisin waste80.30.2	
			2.2 (120)	140.50.3	
				Controls <0.02 all compounds in all substrates	
				Day 33	
				parentSD 31723SD 33608	
1981 Concord	SC	2	1.4 (75)	4.1, 4.10.09, 0.080.04, 0.03	24
			(1.3X)	DayUnrinsedRinsedRemoved from grapes[4]grapesgrapes by rinse	
1977 Emperor	WP	1	0.7 (30)	01.9,1.2,0.74,	25
				2,1.3,0.7,	
				2.41.70.66	
				13 1.6,1.3,0.33,	
				1.2,0.98,0.18,	
				0.90.720.16	
				Controls <0.02 mg/kg	
			Days	03	
1974 Concord	WP	2	2.2 (60)	9.14.6 Control <0.02	26
				Day 14	
				ParentSD 31723SD 33608	

Country, Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
1980 Thompson	WP	3	1.4 (150)	4.9, 50.2 (2)0.06, 0.04	27
				3.8, 4.20.2, 0.10.04, 0.05	
				2.5, 2.20.08, 0.05<0.02 (2)	
				15, 14*0.6, 0.4*0.2, 0.08*	
				Controls≤0.13<0.02<0.02	
				*Apparent outliers	
1980 Thompson	WP	3	1.4 (50)	4, 3.7<0.1, 0.08<0.02 (2)	28
	SC			5.3, 4<0.2, 0.10.04 (2)	
				Controls<0.02 <0.02<0.02	
				Day 14	
				ParentSD 31723SD 33608	
1979 Aurora	WP	3	1.4 (740)	duplicate analyses19, 181.2, 1.40.57, 0.46	29
				replicate sample130.60.2	
	SC	3	1.4 (740)	duplicate analysis7.2, 6.80.27, 0.390.09 (2)	
				replicate sample4.70.20.08	
				Controls <0.02 all compounds	
				Parent SD 31723 SD 33608	
1979 Thompson	WP	3	1.4 (75)	grapes21, 12,0.3, 0.2, 0.06, <0.02,	30
			(replicates)	(23,12.2)*(0.4, 0.2)*,0.04 (2)	
				130.2	
				controls≤0.09<0.02<0.02	
f				raisins(27, 13)*(0.9, 0.5)*(0.18,0.08)*	
				controls<0.02 <0.02<0.02	
1979 Thompson	SC	3	1.4 (75)	grapes14, 7.9,0.2, 0.2,<0.02 (2),	
			(replicates)	(14, 17)*0.1, (0.2,0.03, (0.03,	
				0.3)* 0.04)*	
				raisins(39, 46)*(1.4, 2.6)*(0.2, 0.6)*	
				controls≤0.04<0.02<0.02	
				* duplicate analyses	
1976 Concord	SC	3	1.1(60)	3.8, 5.2	31
			2.2(120)	14, 17	
				Day 7 Grape processing	
				Concentration	
				Residue factor	
1989 Thompson	WP	2	1.4 (180)	grapes, unwashed0.54	32

Country, Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No	Rate kg ai/ha (g ai/hl)		
				grapes, de-stemmed,	
				washed0.330.6	
				stems11.9	
				wet pomace2.34.3	
				dry pomace9.818	
				juice, unclarified0.070.13	
				juice, clarified, canned<0.05<0.09	
				sediment0.10.19	
				raisins2.44.4	
				stem waste1018.5	
				SD 31723 0.13 mg/kg in dry pomace, 0.2 mg/kg in stem waste, <0.05 mg/kg in other processed fractions. SD 33608 ≤0.2 mg/kg in stem waste, <0.05 mg/kg in other processed fractions.	

Unless otherwise indicated residues are parent compound only in grapes.
[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane.
[2] SD 33608 = 2-methyl-2-phenylpropylstannoic acid.
[3] Reference 22 also included analyses of juice and pomace and one additional set of data for the SC formulation, none of which were legible in the submission.
[4] Calculated by adding two right-hand columns. "Removed from grapes by rinse" assumed to be on a grape basis.

Tree Nuts. There are no current Codex MRLs for fenbutatin oxide in nuts. Twenty four studies representing 43 supervised trials in various years on several varieties of almonds, pecans, filberts and walnuts were available from the major nut-growing areas of the United States. Both WP and SC formulations and both dilute and low-volume sprays were included (Table 5). Residues of fenbutatin oxide in the nut-meats reflecting GAP application rates, PHIs and number of applications included residues of ≤0.02, 0.04, 0.05, 0.13, 0.16, and 0.3 mg/kg (the last at a 1.5-fold application rate). More results were provided at the GAP application rates and PHI, but with 3 applications instead of the approved 2 per season. These included residues of ≤0.02 (10 results), 0.03 (4), 0.04 (2), 0.05, 0.07, 0.08, 0.1, 0.2 (3) and 0.3 mg/kg. Other results were provided from exaggerated rates or at PHIs which do not closely reflect GAP. Apparent residues in untreated samples were <0.02 mg/kg except in one trial with apparent residues up to 0.04 mg/kg.

The shells and hulls of nuts were also analyzed, showing maximum fenbutatin oxide residues reflecting GAP of 8.3 mg/kg in the shells and 56 mg/kg in the hulls of almonds, 1 and 12 mg/kg respectively in walnuts and 1.9 mg/kg in pecan shells, although 3 applications were made to pecans instead of 2. Residues in pecan and walnut shells were generally some 20-30 times the levels in the meat, although in one study only about 3 to 4 times. In almonds, hull residues were generally between 60 and several hundred times the level in the meat.

In many cases samples of nut-meats, shells and hulls were also analyzed for metabolites SD 31723 and SD 33608. Results are summarized in Table 5. In all cases the metabolite residues were <0.02 mg/kg in nut-meats. Maximum residues reflecting GAP of SD 31723 and SD 33608 in almonds were 1.3 and 0.3 mg/kg respectively in the shells and 7.1 and 1.3 mg/kg in the hulls. Generally residues in the hulls were ≥3 times those in the shells.

Table 5. Residues of Fenbutatin oxide and its metabolites SD 31723[1] and SD

33608[2] in tree nuts resulting from supervised trials[3,4] in the USA.

Crop Year, Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
				Day 14	
Pecans				ParentSD 31723SD 33608Controls (parent)	1
1981 Success	SC	3	1.4 (50)	meat<0.02<0.02<0.02<0.02	
	WP	3	1.4 (50)	<0.02<0.02<0.02<0.02	
1981 Stuarts	SC	3	1.4 (150)	meat<0.02(2)<0.02<0.02<0.02	2
				shell0.5,0.4,<0.02<0.02<0.02	
				0.3(2)	
	WP	3	1.4 (150)	meat0.03,0.04<0.02<0.02<0.02	
				shell0.6(2),<0.02<0.02<0.02	
				0.5,0.4	
1979 various	SC	3	0.7 (150)	meat0.02,0.04<0.02<0.02≤0.02	3
				shell0.08,0.09<0.02<0.02≤0.02	
			1.4 (300)	meat0.05,0.08<0.02<0.02≤0.02	
				shell0.2,0.3≤0.02<0.02≤0.02	
	WP	3	0.7 (150)	meat0.2, 0.3<0.02<0.02<0.02	
				shell1.9, 1.5 0.06(2)0.03,0.02 <0.02	
				14 35 42 70 76	
1974 Schley	WP	1	0.4 (15)	meat <0.02<0.02	4
		1	0.7 (30)	meat <0.02	
1974 Native	WP	1	1.4 (30)	meat + shell 0.03 0.02 <0.02	5
				meat<0.02	
1974 Money maker	WP	3	1.4 (30) or	meat≤0.02(4)<0.02	6
			2.8 (30)		
				Day 14	

Crop Year, Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
				ParentSD 31723SD 33608Control	
Walnuts				(parent)	
1980 Payne	WP	3	1.4 (60)	meat<0.02(2)<0.02<0.02<0.02	7
				shells 0.3,0.5≤0.02<0.020.05,0.13	
	SC	3	1.4 (60)	meat<0.02,0.03<0.02<0.02same	
				shells 0.5,0.4<0.02,0.05<0.02,0.03same	
1979 Payne	SC	3	1.4 (75)	meat<0.02,0.04<0.02,0.03<0.02<0.02	9
		3	2.8 (150)	0.06(2)<0.02<0.02<0.02	
1981 Hartley	SC	3	1.4 (75)	meat<0.02 <0.02<0.02<0.02	10
1981 Franquette	SC	3	1.4 (424)	meat0.02,0.03<0.02<0.02<0.02	11
				(green)	
1977 Franquette	WP	2	1.4 (30)	meat 0.04<0.02<0.02<0.02	12
				shells 10.05<0.02<0.02	
				hulls 120.30.130.03	
				(nuts 1/2 size)	
				meat <0.02 <0.02<0.02<0.02	
				shells 0.5<0.02<0.02<0.02	
		2	2.8 (60)	hulls 5.10.20.070.03	
1977 Franquette				meat 0.05<0.0l2 <0.012<0.012	13
				shell 1.50.06<0.02<0.02	
	WP	2	1.4 (30)	hulls 5.80.20.09<0.02	
				(nuts 1/2 size)	
		2	2.8 (60)	meat 0.05<0.02 <0.02<0.02	
				shell 0.90.03<0.02<0.02	
				hulls 60.120.05 0.02	
Filberts				ParentSD 31723SD 33608Controls	
1979 Barcelona	WP	3	1.4 (70)	meat<0.02<0.02<0.02<0.02	8
	SC	3	1.4 (70) or	<0.02 <0.02<0.02<0.02	
			2.8 (140)		
1974 Barcelona	WP	2	0.4 (15) 0.8 (30) 1.7 (60)	meat0.02 0.02 0.02 0.02 0.02 0.02	14
Almonds					
1980 Nonpareil	WP	3	1.4 (150)	meat0.2(2)<0.02 <0.02<0.02	15
				hulls 12(2)0.4(2)0.08(2)<0.02	
	SC	3	1.4 (150)	meat0.1,0.07<0.02 <0.02<0.02	
				hulls 5,60.1,0.1<0.02<0.02	
1981 Nonpareil	SC	3	1.4 (250)	meat<0.02 <0.02 <0.02<0.02	16
				hulls 60.80.3<0.02	
1981 Carmel	SC	3	1.4 (300)	meat≤0.02 ≤0.02≤0.02≤0.02	17
				hulls 9,80.7,0.80.3,0.30.2,0.3	
1981 Mission	SC	3	1.4 (300)	meat0.03(2)<0.02<0.02 <0.02	18
				hulls 20,360.7,1.60.2,0.54,6	
			Day	0714 28	
				(Parent compound)	

Crop Year, Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
1973 Mission	WP	2	1.7 (45)	meat 0.90.70.3 0.5	19
1973 Mission	WP	4	0.6 (30)	nut in shell 3.32.11.6 1.3	20
			1.1 (60)	nut in shell 1.91.92.4 1.6	
				Controls <0.02	
				Day 14	
				ParentSD 31723SD 33608Controls (parent)	
1977 Nonpareil	WP	2	1.3 (30)	meat 0.13<0.02 <0.02<0.02	21
				shell 130.30.10.06	
				hulls 562.70.80.3	
		2	2.5 (60)	meat 0.6<0.02 <0.02<0.02	
				shell 361.30.30.06	
				hulls 1707.11.30.3	
1977 Nonpareil	WP	2	1.3 (30)	meat 0.16<0.02 <0.02<0.02	22
				shell 8.30.30.20.06	
				hulls 241.10.30.4	
	WP	2	2.5 (60)	meat 0.12<0.02 <0.02<0.02	
				shell 3.50.10.070.06	
				hulls 391.70.60.4	
			Day	0 514	
				(Parent compound)Control	
1976 Merced	WP	1	1.4 (60)	meat 0.050.050.07<0.03,04	23
				shell1.50.70.10.03,0.1	
				hulls1412210.3,0.4	
		1	2.8 (120)	meat0.20.070.05as above	
				shell6.62.84.4	
				hulls 283357	
				Day 14	
				ParentSD 31723SD 33608Controls (parent)	
1978 Nonpareil	WP	2	1.4 (60)	meat 0.2<0.02<0.02<0.02	24
				shells 40 1.90.3<0.02	
				hulls 29 1.50.3<0.02	
	WP	2	2.8 (120)	meat 0.2<0.02<0.02<0.02	
				shells 45 2.20.4<0.02	
				hulls 36 1.70.3<0.02	

[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane
[2] 33608 = 2-methyl-2-phenylpropylstannoic acid
[3] Unless otherwise indicated, residues are parent compound only in nut-meats without shell.
[4] For evaluation purposes, spray volumes of ≥935l/ha (≥100 gal/acre) were treated as dilute sprays for comparing with GAP information.

Pome fruit (Table 6)

Apples. Fifty one studies were conducted in 10 countries, representing 103 supervised trials, about half of them in the United States. Both WP and SC formulations and about 20 varieties of apple were included. Plot sizes ranged from 1 to as many as 10 trees, although in many trials the number was not stated. The interval from sampling to laboratory receipt, extraction or analysis was generally ≤9 months and samples were generally

shipped and stored satisfactorily. The analytical methods were SAMS 215-1, MMS-R-494-2 and MMS-R-345-1, although the methods were not provided to the Meeting. Limits of detection ranged from 0.01 to 0.05 mg/kg and recoveries were typically 80-110% at 0.1 to 5 mg/kg fortification levels. Controls were generally <0.05 mg/kg but occasionally as high as about 0.1 mg/kg, and one was recorded as 0.5 mg/kg.

Maximum residues approximately reflecting GAP were 2.9 mg/kg in Australian trials (after 6 days compared to a 2-day GAP PHI) and ≤1.6 mg/kg in other non-US trials. All but four of the US apple trials were with dilute sprays, with maximum residues of 4.3 mg/kg from GAP applications. Three of the four trials with concentrated sprays reflected GAP, a WP application resulting in maximum residues of 9.6 mg/kg and an SC 12 mg/kg.

Pears. Twelve studies including 17 supervised trials were conducted in 5 countries, again about half of them in the United States. Maximum residues approximately reflecting GAP were 2.3 mg/kg in Australian trials and 2.7 mg/kg in South African trials (referred to Australian GAP). Conditions in other non-US trials were not according to available GAP. Maximum residues in the US trials from dilute sprays were 2.3 mg/kg and from concentrated sprays 5.6 mg/kg for WP and 3 mg/kg for SC formulations. Pulp residues were generally about 10 to 30% of those in the whole fruit.

Some pome fruit samples (mostly USA trials) were also analyzed for the metabolites SD 31723 and SD 33608. Maximum residues resulting from GAP were 0.3 and 0.05 mg/kg respectively (in pears from concentrated spray applications). Generally residues were ≤0.1 mg/kg SD13723 and <0.02 mg/kg SD 33608, both being <10% of the fenbutatin oxide residue in whole fruit and SD 31723 the higher.

Table 6. Residues of fenbutatin oxide and its metabolites sd 31723[1] and sd 33608[2] in pome fruit and processed products of pome fruit resulting from supervised trials[3]

Crop/ Country (State), Year, Variety	Application	Residues, mg/kg, at intervals after last application	Ref

	Form	No.	Rate, kg ai/ha (g ai/hl)		
			Days	1(GAP=2)61321 28	
Apples				Whole fruit (Pulp)	
Australia 1979	SC	5	(10)	1.6 1.11.10.8 0.05	1
			(GAP=20)	(0.05) (0.06)(0.07)(0.07)(0.06)	
		3	(20)	2.7 2.92.61.91.1	
				(0.2) (0.2)(0.2)(0.1)(0.1)	
				Controls <0.02 (<0.01)	
			Day	127	
1974 Josephine	WP	1	(19)	0.90.60.8	2
				(0.1)(0.1)(<0.1)	
				Controls <0.1 (whole)	
			Day	3 9 19 29	
1975 Granny Smith	WP	2	(20)	1.3 1.52.21.6	3
	SC	2	(20)	1.4 1.41.10.9	
		2	(30)	1.8 22.12	
				Controls <0.1	
			Day	56(GAP=28) 70	
Belgium 1973	WP	1	(25)	0.6	4
G. Delicious		2	(25)	0.8	
		1	(30)	1.3	
		2	(30)	1.2	
				Controls 0.12	
				Day14	
Brazil 1986	SC	2	(60)	2.1 (<0.01)	5
Fuji			(120)	2.5 (<0.01)	
			[1.1 or 2.1 g ai/tree]	Controls <0.01	
			Day 0 3 7 1435 70		
				Whole fruit (pulp)	
Canada 1975	WP	1	(13)	0.2,	6
Cortland				0.5	
				(0.02,	
G. Delicious				0.1)	
		1	(25)	1 0.9 0.7 0.5	
				(0.3) (0.2)(0.2)(0.1)	
		1	(13)	0.2	
				(0.01)	
				Controls <0.01	
			Day	7 14 3241 5972	
				Whole fruit (pulp)	

Crop/ Country (State), Year, Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate, kg ai/ha (g ai/hl)		
France 1974	WP	1	0.3 (30)	0.3 0.04	7
Cardinal				(0.02) (<0.01)	
			0.5 (50)	0.4 0.09	
				(0.03) (0.01)	
G. Delicious	WP	1	0.6 (75)	0.08	8
				(<0.02)	
Melrose		1	0.9 (50)	0.07	
				(<0.02)	
G. Delicious			0.6 (75)	0.2	
				(<0.02)	
				Controls <0.02	
				Day 41	
1977	WP	1	0.3 (30)	0.02 (<0.02), 0.6 (<0.02)*, 0.12 (<0.02)*	9
G. Delicious				* separate trials using different anti-dry-off oils	
				Controls <0.02	
				SD 31723 <0.2 mg/kg in Whole fruit and pulp.	
				Day 7 14 21 117	
				Whole fruit (pulp)	
France 1982	EC	1	0.45 (45)	0.2(<0.01)0.3(<0.01)0.1(<0.01)	10
G. Delicious					
			0.45 (90)	1.5(0.02)0.6(0.02)	
				Controls <0.01	
1972	WP	1	(30)	<0.1	11
			(50)	0.1	
				Controls <0.1	
1973			Day	55/56 65102	
G. Delicious	WP	2	0.5	0.4	12
Cardinal		1		0.08	
Cardinal		2		0.4	
Richard		1		0.3	
			Day	0 7 101421	
Germany 1977	WP	3	0.25 (**25**)	1.7 1.2 1.1 1	13
G. Delicious				1.3 1.1 1 0.5	
	SC	3	0.25 (25)	2.6 1.5 0.7 0.5	
				2.3 1.5 1.3 1	
				Controls <0.01	
				(pulp <0.01 in all samples)	
				SD 31723 <0.1 in all whole fruit and pulp samples	
1989	SC	3	0.75 (50)	0.4 0.40 0.30.25	14
Melrose				puree0.22	
				juice0.27	
				0.5 0.20 0.140.3	15
1989 Jonathan	SC	3	0.75(100)	0.7 0.50 0.5	16

Crop/ Country (State), Year, Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate, kg ai/ha (g ai/hl)		
				0 7 101421	
1989 G.Delicious	SC	3	0.5 (50)	1.5 1.10.90.60.4	17
1989 Melrose	SC	3	0.75 (100)	0.3 0.30.30.30.23	18
				puree0.23	
	SC		3	juice0.28	
			Day	0 7 10 1421 28	
Germany				Whole fruit (pulp)	
1989 Jonagold	SC	3	0.75 (100)	0.4 0.4 0.3 0.30.2	19
1989 Melrose	SC	3	0.38 (25)	0.4 0.4 0.4 0.30.25	20
				mashed apple0.22	
				juice 0.27	
Jonagold				0.5 0.2 0.2 0.140.3	
Jonathan				0.7 0.5 -- 0.50.5	
G. Delicious			0.5 (50)	1.4 1.1 0.9 0.60.4	
Melrose			0.38 (50)	0.3 0.3 0.3 0.30.23	
				mashed apple 0.23	
				juice 0.28	
Jonagold			0.38 (50)	0.4 0.4 0.30.30.2	
				Controls 0.01	
1978 G. Delicious	SC	3	0.025[4]	1.8 1.3 1.81.2 1	21
				(<0.01)(0.02)(0.03)(0.01) (0.01)	
				Control 0.15 (parent in Whole fruit apple)	
				SD 31723 ≤0.1 in all Whole fruit and pulp samples	
1980 Jamba	WP	5	0.3 (20)	0.2 0.1 0.090.01	22
				(pulp <0.01 all intervals)	
Melrose	WP	5	0.3 (20)	1.4 10.90.7	
				(0.3) (0.1) (0.1) (0.07)	
G. Delicious	WP	5	0.3 (20)	1.6 1.7 1.61.1	
				(0.2) (0.07) (0.05) (<0.01)	
				Controls <0.01	
1973	WP	4	1	1.9 1.1 1.10.7 1.1	23
Gold Parmene		3	2X	0.8 0.8 0.40.3 0.3	
				Controls <0.05	
			Day	0 237 14	
South Africa				Whole fruit (Pulp)	
1975 Granny Smith	WP	1	(30)	1.8 1.311	24
				(0.14) (0.1) (0.1) (0.07)	
			(50)	2.5 2.41.51.2	
				(0.4) (0.2) (0.2) (0.1)	
				Controls <0.01	
1974	WP	1	(100)	2 2.42.9 (4 days)	25
Granny Smith				Controls <0.05	

Switzerland			Day	10 19 26/27 35 61	
1973 Jonathan	WP	3	0.5	0.6 0.4 0.50.4 0.3	26
UK 1979				Controls 0.1	
Worcester	SC	2	(12.5)	0.15	
			(25)	0.33	
Bramley			(12.5)	0.2	27
			(25)	0.16	
				SD 33723 <0.02. Controls <0.02	
				Days 626598	
1973 James Grieve	WP	2	(25)	Controls 0.081.30.25	28
1975 G. Delicious		1	0.36(30)	<0.1	29
				Controls <0.1	
				SD 31723 <0.2	
			Days	0 63	
USA 1973	WP	4	3.4 (45)[5]	1.6 Controls <0.08	30
G. Delicious			1.5X	0.7 washed[6]	
1972 York Imperial	WP	1	0.6 (15)	0.6, 0.7, 1	31
			1.1 (30)	1.5, 1.3, 1.7Controls <0.05	
				SD 31723 <0.1	
			Day	0 71428/30 45	
1972 G. Delicious	WP	4	0.84 (15)	1.6 1.51.5 1.3	32
			1.7 (30)	3.5 2.53.1 3.2	
			3.4 (60)	7.9 4.75 3.3 4.1	
				Controls <0.05	
				SD 31723 <0.1	
1972	WP	4	0.84 (15)	2.3,2.6 2.2,2.22.1,1.51.3, 0.9,	33
Ben Davis				1.6 0.9	
			1.7 (30)	5.6,5.4 3.5,4.22.8,3.63, 2.6,	
				2.5 2.6	
			3.4 (60)	8.5,8 5.6,65.6,6.23.8, 5.4,	
				3.8 5.6	
				Controls <0.05	
				SD 31723 <0.1	
1972	WP	4	0.84 (15)	sauce*<0.05	34
G. Delicious				peel+core 4.3	
			1.7 (30)	sauce<0.05	
				peel+core 6.9	
			3.4(60)	sauce<0.05	
				peel+core16	
				Controls <0.05	
				SD 31723 <0.1 sauce, <0.2 peel+core	
				* washed, peeled apples pre-cooked, passed through finisher. Whole fruit apple residue not stated.	
			Day	0 56	
Apples cont'd				juice wet pom. dry pom. juice wet pom. dry pom.	
USA (MD) 1972	WP	1	0.56 (15)	<0.02 2 11	
Winesap			1.1 (30)	<0.02 4 18	

(VA) 1972	WP	2	0.56 (15)	<0.02 3.7 14	
Winesap			1.1 (30)	<0.02 6.7 23	
				All samples washed before processing, whole apple residues not stated.	
				Wet pomaces ca. 74% moisture, dry pomace ca. 1.5%	
				Controls ≤0.08	
				SD 31723 ≤0.2 all samples	
(NY) 1972	WP	4	0.84 (15)	<0.02 3 10.4	36
Ben Davis			1.7 (30)	<0.02 5.7 22	
			3.4 (60)	<0.02 10 40	
				Controls <0.02 juice, <0.05 wet pomace, 0.14 dry pomace	
				Whole apple residues not stated.	
				Wet pomace ca. 71% moisture, dry pomace ca. 1.7%	
				SD 31723 <0.1 juice or wet pomace, <0.2 dry pomace	
			Day	0 7 14 21 45	
(WA) 1971	WP	4	0.84 (15)	0.9 0.8 0.8 0.7 0.7	37
G. Delicious			1.7 (30)	1.7 1.4 1.5 1	
			3.4 (60)	3.7 3.5 3.6 2.8	
				Controls <0.05	
				SD 31723 <0.1 all samples	
				14 days	
				juice* wet pomace dry pomace	38
(NY) 1972	WP	4	0.84 (15)	0.4 3.7 11, 13	
Ben Davis			1.7 (30)	0.1 7.5 21, 24	
			3.4 (60)	0.09 12 45, 39	
				* Sample mislabelling suspected.	
				Controls <0.05 all matrices	
				Wet pomace ca. 69% moisture, dry pomace ca. 3.2%	
				Whole apple residues not stated.	
				SD 31723 <0.1 juice, <0.2 pomaces	
				30 days	
				juice* wet pomace dry pomace	
USA (NY) 1972	WP	4	0.84 (15)	0.17 3, 3.5 8.2, 7.8, 6.3	39
Ben Davis			1.7 (30)	0.08 4.2, 5.7 13, 11, 12	
			3.4 (60)	0.05 9.2, 10 25, 25, 23, 23	
				* mislabelling suspected	
				Controls 0.2 juice, <0.05 pomaces	
				Whole apple residues not stated.	
				Wet pomace ca. 69% moisture, dry pomace 2.3%	
				SD 31723 <0.1 juice, <0.2 pomaces	
			Day	54/56 65 98	
(PA) 1972	WP	1	0.56 (15)	0.2, 0.2	40
G. Delicious			1.1 (30)	0.5, 0.3	
				Controls <0.05, SD 31 723 <0.1	
(MD) 1972			0.56 (15)	0.5, 0.3, 0.2	41
R. Del.			1.1 (30)	0.7, 0.6, 0.8	
				Controls <0.05SD 31723 <0.1	
(VT) 1978	WP	1	0.45 (16)	0.05	42

R. Del.				Control<0.02	
				SD 31723<0.02	
(VA) 1972	WP	2	0.56 (15)	0.7, 0.9, 0.9,	43
Winesap				1, 0.8 Controls <0.05	
			1.1 (30)	1.5, 1.8, 1.7, SD 31723 <0.1	
				1.7, 1.7	
			Day	0 714	
(CA) 1973	WP	3	1.4 (30)	2.7 3.94.3	44
Gravenstein			2.8 (60)	4 3.92.2	
				Controls <0.05	
				14 days	
Apples cont'd				Whole fruit JuiceW.pomaceD.pomace	
USA 1973	WP	5	1.4 (300)	0.9, 1.3 <0.1(2)2.3, 1.59, 9.2,	45
York Imperial		(GAP		3.1, 3.39.5, 8.3	
		=4)	2.8 (600)	2.6, 2.2 <0.1(2)5.2, 5.613, 16	
				6.4, 617, 16	
				Controls <0.2 dry pomace, <0.1 other matrices	
				moisture: wet pomace ca. 74%, dry pomace ca. 1.8%	
				Day 14 48 98	
(Me) 1978	WP	2	0.8 (15)	0.3 Controls:	46
Red Del.	SC	2	0.8 (15)	0.3 all compounds <0.02	
(CA) 1979	WP	1	1.7 (45)	0.5	47
Red/G. Delicious	SC	1	1.7 (45)	0.5 Controls parent <0.05 SD 317243 & SD 33608 <0.02	
(CA) 1980 Red delicious	WP	3	1.7 (180}	9.6, 8.3	48
Newton Pippins	SC	3	1.7 (180)	10, 12	
		(GAP	935l/ha	Controls: all compounds <0.02	
1978		=4)			
Red Delicious	SC	1	0.45 (16)	0.03	49
				Controls, SD 31723 and SD 33608 <0.02	
			Days	0 7 14 28 43 52	
USA (NY) 1973	WP	4	(15)	1.8 1 1.1 1.4 juice <0.1 0.6	50
Red Rome				wet pomace 2.6,3.5	
				dry pomace 7.8,6.6,	
				11	
			(30)	2.2 1.4 1.6 1.2 juice <0.1 1	
				wet pomace 3.6,3.3	
				dry pomace 12,13	
			(60)	2.3 2.8 3.2 2.7 juice <0.1 2.2	
			[76 l/tree]	wet pomace 7.3, 6.2	
				dry pomace 20,15	
				Controls, <0.1 apple and juice, 0.25 dry pomace, 0.12 wet pomace.	
				Day 7 (GAP=14)	
				parent* SD 31723* SD 33608*	
				residue (concn. factor)**	
(NY) 1988	SC	4	(85)	apples, unwashed 3 (1)0.080.05	51
Red Rome			2.8X	apples, washed 2.5 (0.83)0.060.05	
				wet pomace 5 (1.7) 0.1 0.1	

				dry pomace 18 (6) 0.31 0.36	
				juice, unclarified 1.9 (0.6) 0.05<0.05	
				slices 0.07 (0.02) <0.05<0.05	
				* Each value the average of duplicate samples	
				** Factors relative to unwashed apples	
				Controls <0.05 all compounds in all samles	
Pears				Day 1 2 6/7 14 27/28	
Australia 1979	SC	5	(10)	Whole fruit 1 0.6 0.5	52
Beurre Bosc				pulp 0.06 0.04 0.02	
				Controls <0.01	
1974	WP	2	(19)	Whole 0.5 <0.1 0.4 0.30.5	53
Josephine				pulp <0.1<0.1	
				Controls <0.1	
				Day 3 9 19 29	
1975	SC	2	(20)	1.5 1.61.51.3	54
Packman			(20)	1.4 1.21.31.2	
			(30)	2.3 1.41.61.2	
				Controls <0.5 (confirmed by GC, different column)	
				113 or 125 days	
Belgium 1974 Durondeau	WP	1	0.75 (50)	Whole fruit and pulp, two trials<0.02	55
				0 371421 35	
France 1975	WP	1	0.3 (60)	Whole 0.20.10.03	56
Williams				pulp <0.02 <0.02<0.02	
			0.5 (100)	Whole 1.70.2 0.1	
				pulp 0.05 <0.02<0.05	
				Controls <0.02	
S. Africa 1975	WP	1	(30)	Whole 1.7 1.61.40.9	57
Packman's Triumph			15l/tree	pulp 0.2 0.10.10.05	
			(50)	Whole 3.6 3.12.71.6	
			15l/tree	pulp 0.3 0.30.20.2	
				Controls ≤0.07 Whole fruit	
			Days	0 2 5	
S. Africa 1974	WP	1	(10)	3.8 2.7 2.5	58
Wintermelis				Controls 0.11	
				14 days	
				Parent SD 31723SD 33608	
USA (WA) 1980	SC	4	1.7	2.1,2.10.1 (2)0.04 (2)	59
Bartlett	WP		(30-45)	2.3,2.20.1 (2)0.03,0.04	
(CA) 1980	WP	3	1.7 (180)	5.1, 2.90.2,0.10.05,0.03	60
Bartlett	SC	3		5.6,5.50.4,0.30.08,0.06	
				Controls: parent 0.04, metabolites <0.02	
(MI) 1980	WP	4	1.7 (700)	4.5,4.50.3,0.30.08,0.1	61
Bartlett	SC			2.5,3 0.2 (2)0.06,0.05	
				Controls: parent ≤0.04, metabolites <0.02	
				7 days	
(CA) 1979	SC	1	1.7 (45)	ParentSD 31723SD 33608	62
Bartlett				10 0.20.05	

				Controls: parent 0.05, metabolites 0.02	
			Day	0 7 1430	
(MI) 1971	WP	1	0.42 (15)	3 1.71.61	63
Kieffer			0.84 (30)	6 511.1	
			1.7 (60)	4.743	
				Controls: parent <0.05, SD 31723 <0.2	

[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane
[2] SD 33608 = 2-methyl-2-phenylpropylstannoic acid
[3] Unless otherwise indicated, residues are parent compound only in whole fruit.
[4] Residue levels suggest a rate error, but reported as 0.025 kg "am"/ha in several places in the report.
[5] For evaluation purposes, spray volumes of ≥935l/ha (≥100 gal/acre) were treated as dilute sprays for comparing with GAP information.
[6] 5 min. water soak, soap solution rinse, 30 sec. brush, fresh water rinse

Raspberries. Supervised trials information was available from only one country (Germany, Table 7, reference 28). Residues ranged from 19 mg/kg on the day of application to 0.8 mg/kg after 21 days at the spray concentration approved in The Netherlands, but no German GAP was provided for raspberries and the sampling intervals could not be related to the pre-bloom applications in the GAP of The Netherlands or Poland.

Strawberries. Twenty seven reports were available, representing 48 supervised trials in 7 countries: Australia (5), France (5), Mexico (2), The Netherlands (1), South Africa (2), the UK (2) and the USA (31) (Table 7). Plots ranged typically from 9 to 60 metre rows. Both SC and WP formulations were used on several varieties of strawberry. Most sprays were dilute, with a few concentrated sprays at GAP rates. Sampling-to-analysis intervals were as high as a year, but generally less than 9 months. Samples were generally shipped and stored satisfactorily. Several analytical methods were used (SAMS-215-1, MMS-R-391-1, MMS-R-345-1, MMS-R-494-2), although none of them were provided. Limits of detection were generally reported as 0.02 mg/kg, a few as 0.01 mg/kg. Control values were mostly ≤0.02 mg/kg, but one (Australia, 1982) was as high as 0.25 mg/kg.

The PHI in the USA and Australia is 1 day compared with 5 days in France and 7 days in the UK. In The Netherlands and Germany fenbutatin oxide is used pre-bloom and post-harvest. The results of the trials in The Netherlands and South Africa could not readily be related to available GAP information.

The highest residues from uses approximating GAP were 1.3 mg/kg in the Australian trials, 0.4 mg/kg in the French trials, 0.5 mg/kg in the UK trials and 7 mg/kg in the Mexican trials (referred to US GAP). The more numerous US trials resulted in a fairly evenly spaced range of residues, except two values, up to 9.9 mg/kg (this from a 1.2-fold application rate). The two exceptions were residues of 12 and 18 mg/kg from two trials at one site (Table 7, reference 27). The report mentioned difficulties in getting a reliable project history from the co-operator in these trials. For this reason and because the results were inconsistent with those of many other similar trials the author of the report doubted their validity.

Some samples were also analyzed for the metabolites SD 31723 and SD 33608 (expressed as parent), mostly in the US trials. Maximum residues from treatments according to GAP were 0.1 and 0.05 mg/kg respectively. Residues of SD 31723 were generally ≤5% of the fenbutatin oxide residues and SD 33608 was usually half of SD 31723 or less. Most analyses were after one day.

Table 7. Residues of Fenbutatin oxide and its metabolites SD 31723[1] and SD 33608[2] in strawberries and raspberries resulting from supervised trials.[3]

Crop, Country (State), Year Variety	Application	Residues, mg/kg, at intervals after last application	Ref.

	Form	No.	Rate kg ai/ha (g ai/hl)		
Strawberries			Day	0123579	
Australia 1982	SC	3	(20)	0.40.3	1
Early sweet		3	(40)	0.60.6	
				Control 0.25	
Australia 1981	SC	1	(10)	0.40.20.1	4
Redlands,			(20)	0.90.40.3	
Early sweet			(40)	1.30.50.4	
				Controls <0.01	
France 1976	WP	1	0.3 (30)	0.40.2 at 14 days	5
Gorela				SD 31723 <0.1	
				Control <0.01	
				Day 035712/1314	
1986	SC	1	0.55 (73)	0.040.02	6
Belle rubis				0.50.1	
				0.70.06	
Bogotta	SC	1	0.55(110)	0.060.03	
Gorella			0.5 (50)	0.80.140.12	
Gariguette			0.5 (50)	0.40.080.05	
				Controls <0.01	
			Days	0 1 23/4 5714	
Mexico 1976	SC	1	0.55	2.3[4] 220.9[4]	7
Fresa			1.1	7 4.1[4]1.9[4]5.9	

Crop, Country (State), Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref.
	Form	No.	Rate kg ai/ha (g ai/hl)		
			(US GAP)	Controls <0.1[4]	
Netherlands 1980	WP	2	0.25 (25)	0.7,1.5,1.1,	8
				2.1,1.6,1.5,	
				2.2,1.0,1.1,	
				1 (reps.)1.2 (reps)1 (reps)	
				Controls <0.05	
S. Africa 1976	WP	1	0.13 (25)	0.50.20.2	9
Parfaite			0.19 (25)	0.80.50.5 Control <0.1	
UK 1974			Day	23/471481/84	10
Favourite	WP	1	(25)	<0.1	
Red Gauntlet				0.70.70.5<0.1	
Favourite			(30)	0.40.20.50.2	
				Day 1 3 7	
USA (WI) 1975	WP	2	1.1 (120)	1.60.5	2
Catskil			2.2 (240)	2.21.5	
			[930l/ha]	Controls <0.02	
(WA) 1975	WP	1	0.56 (60)	0.71.11	3
North West			1.1 (120)	1.11.20.8	
				2.44.33.1	
				Controls <0.02	
				Day 1 3 7	
				parent=A SD 31723=B SD 33608=C	
				ABCAA	
USA (CA) 1983	SC	4	1.7 (72)	9.90.10.05	11
			(GAP=60)	50.080.02 (reps)	
				6.50.10.03	
				5.20.080.02	
				Controls <0.02 (parent)	
(CA) 1984	SC	4	1.7 (72)	3.10.030.02	12
Heidi				2.4<0.010.04(reps)	
				Controls parent <0.01	
USA (NJ) 1975	WP	2	0.56 (60)	8.60.90.6	13
Raritan			1.1 (120)	1.41.30.6	
			2.2 (240)	51.9	
			[930l/ha]	Controls (parent) 0.07	
(FL) 1981	WP	6	1.1 (60)	1.70.04<0.02	14
Tuffs		GAP		1.60.04<0.02 (replicates)	
		= 4		30.10.05	
	SC	6	1.1 (60)	2.30.090.03	
				2.70.070.03 (replicates)	
				2.70.10.06	
USA cont.				1.90.10.07 Controls ≤0.02 (parent)	
(CA) 1976	WP	1	0.56 (30)	2.3	15
Heidi			1.1 (60)	3.6Control <0.02 (parent)	

Crop, Country (State), Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref.
	Form	No.	Rate kg ai/ha (g ai/hl)		
(FL) 1979	WP	10	1.1 (60)	8.90.20.1	16
Tioga				7.50.20.09 (replicates)	
		10	2.2 (120)	190.50.3	
				180.40.2 (replicates)	
(KY) 1976 Tenn Beauty	WP	2	0.56 (60)	2.9 Control <0.02 (parent)	17
(CA) 1979 Shasta	SC	10	1.1 (67)	4.60.30.1Control <0.02 (parent)	18
(FL) 1979	SC	10	1.1 (60)	6.80.20.1	19
Tioga				7.70.30.1Control <0.02 (parent)	
(CA) 1981	SC	6	1.1 (200)	0.90.03<0.02	20
Shasta			[560l/ha]	1.20.04<0.02 (replicates)Control <0.02 (parent)	
				Note: 1 yr. between sampling and analysis, 57% recovery of parent. (Not stated whether results corrected for recovery).	
USA (CA) 1984	SC	4	1.7 (72)	4.40.040.02	21
G-4		=GAP	[GAP=(60)]	4.70.050.04 (replicates)	
				Controls 0.01 all compounds	
(CA) 1981	SC	6	1.1 (48)	1.70.04<0.02Note: 11.5 months between sampling and analysis	22
Heidi				2.80.060.02 (replicates)	
				Controls <0.02 all compounds	
			Day	0137 (parent) (parent)	
(CA) 1973	WP	4	4.5 (190)	4.243.33	23
G-4				Control 0.02	
				Note: 1.3 yr. between sampling and analysis	
				1 day	
				ParentSD 31723SD 33608	
(CA) 1979	WP	10	1.1 (67)	1.60.10.04	24
Shasta			2.2 (130)	2.70.20.07	
				Controls 0.03 parent, 0.02 metabolites	
(KY) 1974	WP	3	0.56 (60)	2.4	25
Tennessee Beauty			1.1 (120)	5Control <0.02	
			[930l/ha]		
				Day0 1	
(CA) 1974	WP	1	1.1 (60)	2.31.4	26
Toiga			2.2 (120)	3.82.4	
				Control 0.02	
				Note: 11 months between sampling and analysis	
(MI) 1974	WP	3	0.56 (60)	12^5	27
Everberring			1.1 (120)	18^5 Control 0.31	
			[930l/ha]	Note: 9 months between sampling and analysis	
Raspberries				Day 0714 21	
Germany1980	WP	2	0.38 (25)	198.310.8	28
Schoenemann				Control <0.01	

[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane
[2] SD 33608 = 2-methyl-2-phenylpropylstannoic acid
[3] Unless otherwise indicated, residues are parent compound only in the whole fruit.
[4] Samples arrived at laboratory cold but unfrozen, and consisted of fruit and some separated juice.
[5] Doubtful result. See text.

Stone fruit (Table 8)

Forty seven studies on stone fruit were available from 8 countries representing 76 supervised trials on cherries, plums, peaches and nectarines. In some samples of peaches the whole fruit and pulp were analysed separately, and plums and peaches were analysed before and after drying. In some cases the metabolites SD 31723 and SD 33608 were determined. Most of the results were for de-stoned fruit, but in a few cases it was not clear whether the stones had been removed. No attempt was made to calculate the result on a whole-fruit basis since the stone weights were only about 6% of the whole fruit weights.

The analytical methods were SAMS-215-1, MMS-R-391-1, and MMS-R-494-1 or -2. Limits of detection were generally reported as 0.02 mg/kg. Analytical recoveries were generally 80%, although in some studies as low as 55-65% with no indication of whether the results were corrected for analytical recoveries. In general plot sizes ranged from 1 tree (2-3 replicates) to 5 trees and samples were stored satisfactorily.

Cherries. The highest individual residue from trials approximately reflecting GAP was 0.6 mg/kg in Germany (6 trials) and 5.1 mg/kg in the United States (14 trials) (it was not stated whether results were adjusted for 69% recoveries). The 2 trials in The Netherlands were not according to GAP. Analytical recoveries ranged from 58 to 110% and controls were $\leq$0.3 mg/kg, except in the trials in The Netherlands where they were up to 0.6 mg/kg. Maximum residues of SD 31723 and 33608 were 0.9 mg/kg and 0.04 mg/kg respectively from treatments according to GAP. The ratios of the residues of fenbutatin oxide to those of the metabolites were similar to those found in other commodities.

Peaches. The higheset residues from treatments close to GAP in Australia (4 trials) was 2.5 mg/kg. The highest in the 9 United States trials was 8 mg/kg from a 1.5-fold rate (corresponding to 5.3 mg/kg at the GAP rate) and 5.8 mg/kg from 3 instead of the permitted 2 applications at approximately the GAP rate, and after a PHI of 21 instead of 14 days. The intervals from sampling to analysis were 34 to 44 months in most of the US trials (9 months in one and 18 months in another). Maximum residues from 3 Canadian trials according to GAP were 0.8 mg/kg, from 5 French trials according to German GAP 1.3 mg/kg, and from 3 trials in Germany 3.3 mg/kg from the approved spray concentration but after 28 days rather than the GAP interval of 21 days. Three trials in South Africa resulted in residues up to 7.8 mg/kg after 10 days and 6 mg/kg after 13 days (a 14-day PHI is common in other countries), although information on GAP in South Africa was not provided. Apparent residues were up to 0.2 mg/kg in untreated samples, depending on the method or trial, and in at least one case the residue was confirmed as fenbutatin oxide.

Fenbutatin oxide residues in the pulp were 5 to 10% or less of the whole fruit residues. Maximum residues of SD 31723 and SD 33608 from GAP treatments were <0.1 mg/kg. The ratios of the residues of the metabolites to those of fenbutatin oxide in whole fresh fruit were again similar to those in other commodities.

Plums. Maximum residues of fenbutatin oxide from uses approximating GAP were 0.7 mg/kg in 9 German, and 2.1 mg/kg in 12 United States trials, while residues of SD 31723 were $\leq$0.07 (or <0.1) mg/kg and of SD 33608 $\leq$0.03 mg/kg except for 1 value of 0.1 mg/kg. SD 31723 residues were usually <5% of the fenbutatin oxide residues and SD 33608 similar to or lower than SD 31723. Control values for fenbutatin oxide ranged from <0.01 to 0.1 mg/kg, depending on the analytical method used. Fenbutatin oxide residues were concentrated in prunes (dried plums).

Table 8. Residues of Fenbutatin oxide and its metabolites SD 31723[1] and SD 33608[2] in stone fruit resulting from supervised trials[3,4].

Crop, Country (State), Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
Cherries (de-stoned)[4]			Day	04710142128	
Germany 1976					
Biggarau	WP	3	(25)	0.70.20.09	1
Rote Leber			[GAP]	1.50.20.040.02	
Schattenmorelle (Morello)				1.10.40.20.10.09	
				Controls <0.01-0.3 SD 31723 <0.1 all samples	
1980		3	0.3 (20)	3.41.71.5 0.9 0.6	2
Schattenmorelle				1.61.30.9 0.8 0.2	
Rubin				3.51.4 1 0.7 0.1	
				Controls <0.01	
USA (OR) 1974	WP	2	0.4 (15)	0.3	3
Sweet			0.8 (30)	0.5	
			1.7 (60)	Control 0.04 1.2	
(NY) 1974	WP	1	0.7 (18)	0.4	4
Montmorency			1.3 (36)	1.3	
			2.7 (71)	1.9	
				Control <0.02	
				14 days	
				ParentSD 31723**SD 33608**	
(OR) 1981	SC	2	1.7[6] (45)[7]	1.7,0.02,<0.02	5
Royal Ann				1.8 (repl.)*0.02 (repl.)<0.02	
				* Not stated whether corrected for recoveries 58% parent, 61% metabolite. ** Calculated as parent	
				Controls <0.02 all compounds	
(CA) 1981	SC	2	1.7[6] (71)[7]	3.5, 0.05,	6
				3 (repl.)*0.04*	
				* Not stated whether corrected for recoveries 75% parent, 73% metabolite	
				Controls <0.02 all compounds	
USA (MI) 1980					
Montmorency	SC	2	1.7 (180)	4.7, 5.1*0.2, 0.10.04, 0.03*	7,9
	WP	2	1.7 (180)	2.7, 2.2*0.07, 0.050.02*, <0.02	
			[930l/ha]	* Not stated whether corrected for recoveries 69% parent, 53% metabolite	
				Controls: parent 0.02-0.17, metabolites <0.02	
(CA) 1979	SC	2	1.7 (90)	3.80.90.04	8
Bings			3.4 (180)	100.30.05	
				Controls parent 0.08, metabolites <0.02	
(OR) 1977 Royal Ann/Bings	WP	1-2	1.1-2.2 (30-60)	<0.02 parent at 292-305 days	10, 11,
			post-harvest		12
			Days	2128 (NL GAP=42 days, Italian 30)	
Netherlands 1976	WP	1	0.3 (25)	0.9*0.5 * German GAP applicn.	13

Crop, Country (State),Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
Morello			0.6 (50)	1.1*1.2 * Italian GAP applicn.	
				Controls 0.4, 0.6 (confirmed)	
Plums[4]			Days	07142128	
Germany 1977 Auerbacher	WP	3	0.25 (25) [GAP]	0.080.060.04	14
Zwetschge (Victoria)				0.40.30.30.2	
Hauspflaume				0.50.50.70.50.1	
				Controls: parent <0.01, SD 31723 <0.1	
1978 Hauspflaume	WP	3	0.25 (25)	0.50.50.70.50.1	15
				Controls: parent <0.05, S31723 D <0.1	
1980 Auerbacher	WP	5	0.3 (20)	0.20.40.30.1	16
Ortenberg				0.10.070.05<0.01	
				0.50.70.40.3	
				Controls <0.01	
1976 Auerbacher	WP	3	0.25 (25)	0.20.20.20.1	17
Buehler				0.30.10.20.1	
				Control <0.1	
Netherlands 1976	SC	1	0.18 (25)	0.30.20.1<0.1<0.1	18
Czar			0.35 (50)	0.50.40.20.1 0.1	
				Control <0.1	
			Day	04 714	
S. Africa 1975	WP	1	(30)	1.2 1.30.80.9	19
Kelsey			(50)	2.22.21.81	
				Control <0.01	
				14 days	
				ParentSD 31723SD 33608	
USA (MI) 1981	SC	2	1.1[6] (60)[7]	0.2, 0.04<0.02<0.02	20
Lumbard				Controls <0.02 all compounds	
(NY) 1980	SC	2	1.1 (80)	1, 1.5*0.05, 0.07<0.02, 0.03	21
Stanley			2.2 (160)	6.7, 3.1*0.3, 0.10.1, 0.05	
				* Not stated whether corrected for 68% recoveries. Controls <0.02 all compounds	
(MI) 1979	SC	2	1.1 (340)	1.3, 2.10.03, 0.060.02, 0.1	22
Stanley			2.2 (680)	5.9, 6.10.1, 0.090.1, 0.06	
			[330 l/ha]	Controls parent 0.06, metabolites <0.02	
				Prunes, dried	
(CA) 1981	SC	2	1.1 (60)	2.5*0.070.07	23
Prunes, dried (from treated plum trees)				*Not stated whether corrected for 67% recoveries Controls: parent 0.15, metabolites 0.02	
USA (CA) 1981					
Prunes (from treated sugar plum trees)	SC [GAP=2]	3	1.1 (120)	3.1*0.070.04	24
			[930l/ha]	* Not stated whether corrected for 55% recovery. Controls: parent 0.18, metabolites <0.02	
				24 days	
USA (CA) 1981	SC	2	1.1 (120)	0.7, 0.8<0.02<0.02	25
Santa Rosa			[930l/ha]	Controls: parent 0.06, metabolites <0.02	

Crop, Country (State),Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
(NY) 1974	WP	2	0.56 (15)	0.3	26
Stanley/Italian			1.1 (30)	0.5	
			2.2 (60)	0.5 Controls <0.02	
				7 days (GAP=14)	
				unwashed plums driedrehydrated prunesprunes	
(CA) 1988 domestica	WP	2	1.1 (60)[5]	(80% moisture) (18.5% moisture)(33% moisture)	27
			[426l/ha]	0.18, 0.08* 0.37, 0.280.08, 0.22	
				(0.13)* (0.32)*(0.15)*	
				Concentration factor, fresh to dry = 2.5	
				Concentration factor, fresh to rehydrated = 1.2	
				* duplicate assays, average in parentheses	
				SD 31723, SD 33608 and Controls <0.05 all matrices	
Peaches[4]			Day	1 6 13(GAP=14) 2128	

Crop, Country (State), Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
				whole (pulp)	
Australia 1979	SC	5	(10)	2.8 2.4 1.7 0.90.9	28
Golden Queen				(0.1)(0.1)(0.08)(0.08)(0.07)	
		3	(20)	5.6 2.9 2.5 1.81.2	
				(0.2)(0.1)(0.1)(0.09)(0.07)	
				Controls <0.01	
				15 daysParent SD 331723 SD 33608	
USA (CA) 1978	WP	2	1.1[6] (60)[7]	fresh*1.1** 0.08**<0.02	29
Hale Haven				controls<0.02 <0.02 <0.02	
				dried*** 8.2 0.08 0.03	
				Controls 0.09 <0.02 <0.02	
				14 days	
				fresh*1.9** 0.06**<0.02	30
Riosa Gems				Controls≤0.03 <0.02<0.02	
				dried***17 0.07 0.08	
				Controls 0.08 <0.02<0.02	
				*90% moisture **Not stated whether corrected for 62-65% recoveries ***10% moisture, lab air-dried	
			Day	0 712/14 21 28	
(OH) 1973	WP	1	1.7[8] (45)[7]	2.7 (12 days, corresponds to 1.1 at GAP rate)	31
Cumberland				Control<0.02	
(CA) 1973	WP	3	0.31 (20)	3.9 2.81.9 1.5	32
Key Stone		(GAP =2)	0.62[8] (40)[7]	7.7 8.24.6 5.8	
				Controls 0.04, 0.18	
(CA) 1973	WP	3	0.56 (30)	4.31	33
Sunblest			1.1 (60)	7.32.6	
				Controls ≤0.02	
(OR) 1973	WP	2	1.7[8] (45)[7]	5.7, 8 (corresponds to 3.1, 5.3 at GAP rate)	34
Red Haven				Controls 0.02	
(CA) 1973	WP	3	1.1[6] (60)[7]	4.33	35
Sunblest				Controls <0.02	
Peaches cont'd			Day	0 3 71421 28	
Australia 1975	SC	2	(20)	2.2	36
Tatura Aurora			(30)	3.6	
	WP	2	(20)	2.2 Controls 0.2 (confirmed)	
Canada 1975	WP	2	(25)	2.6 0.6	37
Alberta			[U.S GAP]	pulp 0.05 0.03	
				Controls <0.01	
1981	SC	2	0.63 (19)	0.3, 0.4,	38
Baby Golden				0.3, 0.2	
				pulp 0.02, 0.04 (3)	
				Controls <0.01	
1982	SC	1	0.89 (26)	0.6 (2),	39
Madison				0.5 (2),	

Crop, Country (State), Year Variety	Application			Residues, mg/kg, at intervals after last application	Ref
	Form	No.	Rate kg ai/ha (g ai/hl)		
				0.7 (3),	
				0.8 (2),	
				0.4, 1, 0.3	
				pulp<0.01 (10)	
				Controls <0.01	
France 1976	WP	1	0.75 (50)	0.9 (8 days)	40
Royal Gold				pulp<0.01	
				SD 31723<0.01	
				Controls<0.01	
				0 3 71421 28	
1973 Audenot	WP	1	0.5	3.72.6 1.9 (2 days) Control 0.2	41
1982	SC	1	0.45 (90)	1.4	42
Merril s.d.				pulp0.04Control 0.01	
1973	WP	1	0.5	1.61.6 1.51.30.7	43
Early Alberta			[German GAP]	Controls<0.05	
				Day 2 815	
France 1973	WP	1	0.5	21.70.4	44
Red Aven				Controls 0.04-0.1	
			Days	01 3714212835	
New Zealand 1975	WP	4	(19)	0.80.60.10.70.5 Control <0.1	45
Golden Queen					
Germany 1976					
Rote Ingelheim	WP	3	0.25 (25)	9 3.43.421.81.3	46
Rekord aus Alf.				8.1 4.72.52.21.5	
M. Rochiat				8.13.12.52.33.3	
				Controls <0.1	
				Days 013671013	
S. Africa 1976	WP	1	(30)	76.3 5.94	47
Kakamas				pulp0.20.10.060.02	
			(50)	128.276	
				pulp0.20.20.10.1	
				Controls 0.01	
1976 Kakamas	WP	1	(30-50) [2 trials]	canned <0.01<0.01 <0.01	48
1974	WP	1	(30)	4.23.1	49
			(50)	6.47.8	
Nectarines				14 days	
				ParentSD 31723 SD 33608	
USA (CA) 1981	SC	2	1.1(230) [480l/ha]	0.2 (2) <0.02 <0.02	50
Sunglo	SC	2	1.1[6] (60)[7]	2, 3.50.05, 0.08 <0.02	51
				Controls: parent 0.1, metabolites <0.02	

[1] SD 31723 = dihydroxybis(2-methyl-2-phenylpropyl)stannane
[2] SD 33608 = 2-methyl-2-phenylpropylstannoic acid
[3] Unless otherwise indicated, residues are parent compound only in the whole fruit.

[4] All residues refer to de-stoned fruit, except references 14, 32, and 36 in which it was not indicated whether stones were removed. The Meeting did not recalculate residues on a whole-fruit basis since the stones constituted on average only about 6% of the whole fruit weight (the range was 3.8-11%). Reference 3 was an exception where the stone weighed 30-40% of the fruit in early samples and 10-15% in later samples.
[5] In reference 27 the summary (Volume 1) cites the application rate as 0.06% (60 g ai/hl). This is consistent with the text of volume 3, page 13 (based on about 200 G/A (1892 l/ha) but not with Table V, page 24, which lists the volume as 45 G/A (170 l/A = 426 l/ha). Airblast sprayer was used.
[6] GAP rate for concentrated sprays.
[7] GAP rate for dilute sprays = (15-30).
[8] GAP rate for concentrated sprays = 0.6-1.1.

Animals

The Meeting received feeding studies on cows and chickens which had also been provided to the 1979 JMPR.

Cows. Six lactating Guernsey cows were fed daily, three at 11 and three at 96 ppm unlabelled fenbutatin oxide in the diet (equivalent to 0.37 and 3.7 mg/kg body weight) for 21 or 22 days, with handling procedures similar to those described below under "Fate of residues" (Shell, 1973). In this study (Potter and Nugent, 1978), samples of milk were taken periodically and animals slaughtered within 24 hours of the last feeding for analysis of tissues.

Analyses were for fenbutatin oxide, SD 31723 and SD 33608 by method MMS-R-494-1, described later under "Methods of residue analysis", because "a previous trial, 11-112-74" was reported to have shown residues to be mainly fenbutatin oxide and SD 31723. That report was not provided to the Meeting and appears not to have been reviewed by the 1977 or 1979 Meetings.

No residues (<0.02 mg/kg) of fenbutatin oxide or its metabolites were detected in any tissue or in cream from the 11 ppm feeding level, or in skim milk or brain from either level. Residues of fenbutatin oxide were found in tissues and cream from feeding at 96 ppm, but SD 33608 was not found in any samples and SD 31723 only in liver and kidney (Table 9). Analytical recoveries of all three compounds averaged ≥84% in skim milk and cream at 0.1 and 0.2 mg/kg fortification levels, ranged between 79 and 118% in liver and kidney at 0.2 mg/kg and were ≥81% in fat and muscle at 0.1 mg/kg.

The greatest potential for residues in cattle products would be from feeding dry apple pomace, dry citrus pulp, dry grape pomace and almond hulls. These might be fed to beef cattle at 50, 33, 30 and 25% of the dry feed matter respectively, and to dairy cattle at 25, 33, 20 and 25%. The implications are discussed in the Appraisal.

Table 9. Residues of fenbutatin oxide, SD 31723 and SD 33608 in cream and in tissues of dairy cattle from feeding fenbutatin oxide for 21 or 22 days at 96 ppm in the diet (Potter and Nugent, 1978).

Sample	Residues, mg/kg		
	Fenbutatin oxide	SD 31723	SD 33608
Cream	day 1 <0.02 day 3 0.03-0.04 day 13 0.06-0.11 day 21 0.04-0.06	<0.02	<0.02
Cream fat[1]	0.08-0.14	--	--
Liver	0.04-0.07	0.09-0.12	<0.02
Kidney	0.13-0.18	0.02-0.04	<0.02
Subcutaneous fat	0.04-0.06	<0.02	<0.02
Mesenteric fat	0.05-0.06	<0.02	<0.02
Quadriceps muscle	0.03-0.04	<0.02	<0.02

[1] Calculated from levels in cream and the proportion of milk fat in cream

Chickens. In the chicken feeding study two groups of 27 White Leghorn hens were fed for periods up to 28 days with fenbutatin oxide in the total feed at either 5 or 25 ppm. Eggs were collected every 24 hours and after intervals of 7, 14, 21 or 28 days tissue samples were taken for determination of fenbutatin oxide, SD 31723 and SD 33608 by method MMS-R-494-1. Some birds were also fed untreated feed for various periods up to 28 days after withdrawal of the treated feed before slaughter.

In the 5 ppm feeding group, no residues (<0.02 mg/kg) of fenbutatin oxide were detected in any samples except egg yolks, one sample of whole egg with 0.09 or 0.15 mg/kg (suspected of being contamination) and two samples of whole egg with 0.02 mg/kg. Neither metabolite was found in any sample. Residues of both fenbuatin oxide and the metabolites were found in liver and kidney from the 25 ppm feeding level and of the parent compound only in whole egg and egg yolk but not egg white (Table 10). No residues were found in light or dark meat or fat. Analytical recoveries were ≥85% in all matrices from 0.1 to 0.5 mg/kg fortifications.

The greatest potential for residues in chicken eggs or tissues would come from dry grape pomace, which may constitute up to 5% of poultry diets.

Table 10. Residues of fenbutatin oxide and metabolites SD 31723 and SD 33608 in eggs and tissues of chickens from feeding fenbutatin oxide at 25 ppm dietary levels for intervals up to 28 days and for periods thereafter with untreated feed (Potter and Nugent, 1979)

Sample	Residues, mg/kg		
	Fenbutatin oxide	SD 31723	SD 33608
Liver	Day 7 0.02 Day 14 0.04 Day 21 0.02 Day 28 0.02 Day 31[1] <0.02 Day 36 <0.02 Day 42 <0.02	0.05 0.12 0.1 0.07 0.08[1] 0.03 0.02	<0.02 0.02 0.04 <0.02 0.03[1] 0.02 0.02
Kidney	Day 7 0.03 Day 14 0.02 Day 21 <0.02 Day 28 <0.02 Days 31[1] <0.02 Days 36 <0.02	0.03 0.03 0.03 0.02 0.04[1] <0.02	<0.02 all intervals
Whole Egg	Days 1-3 <0.02 Days 4-7 0.05 Days 8-22 0.1 Days 23-28 0.12 Days 29-30[1] 0.1 Days 33-35 0.04 Days 36-42 <0.02	<0.02 all intervals	<0.02 all intervals
Egg yolk	Days 8-15[2] 0.17 Days 23-28[2] 0.25	<0.02 <0.02	<0.02 <0.02

[1] Untreated feed after 28 days for tissues or 29 days for whole eggs
[2] 8-15 days value from 5 ppm feeding level, 23-28 days from 25 ppm

FATE OF RESIDUES

Two cow feeding studies with the radiolabelled compound (at least one of which was previously submitted to the 1977 JMPR) were available, together with processing information for apples, citrus, grapes, plums and to some extent tree nuts, cucumbers and peaches. The processing information is summarized below, but the results are included in the supervised trials

Tables 2 to 8.

In animals

In one study three lactating Guernsey cows were fed [^{119}Sn]fenbutatin oxide at 170 ppm in grain concentrates, giving a dietary equivalent of 34 ppm, for 21 days (Shell, 1973). The 34 ppm was linked to a mean feed consumption of 16 kg/day/cow so that the cows received on average 540 mg/day of labelled fenbutatin oxide. The main weight of the cows was 425 kg, so that the dosage was approximately 1.3 mg/kg body weight/day.

Milk, urine, and faeces were taken for analysis daily. The animals were slaughtered 12 hours after the last dose and tissue samples analysed by liquid scintillation counting. Faeces accounted typically for about 65-85% of the daily dose, and urine for 0.2 to 1%, but some samples were outside these ranges. The R_f of the major TLC spot from faeces extracts corresponded to fenbutatin oxide.

No radioactivity was found in milk at a 0.02 mg/kg limit of detection, nor in brain, bone, bone marrow, mesenteric fat, subcutaneous fat or quadriceps muscle at a 0.04 mg/kg limit. Residues equivalent to 0.04 mg/kg of fenbutatin oxide were reported in gastrocnemius muscle from one of the three cows but <0.04 mg/kg in the other two. Fenbutatin oxide equivalents in the kidneys of the three cows were 0.27, 0.38 and 0.15 mg/kg and in the livers 0.4, 0.41 and 0.22 mg/kg. The residues were not identified or characterized.

In the second study (Koos, 1973) three Guernsey cows were also fed twice daily with ^{119}Sn-labelled fenbutatin oxide for 21 days, at a total dietary equivalent reported to be 30 ppm. 337 mg of the compound was applied to 1 kg of grain concentrate. The daily feed was 15 kg (5 kg concentrate plus 10 kg alfalfa cubes) feed/cow/day, although the actual feed consumption was not recorded in the report. Milk and tissue samples were analysed by analytical method MMS-R-345-2. The interval from last feeding to slaughter was not recorded in the report. The analytical method was not provided, but is based on EC-GLC and TLC procedures for the determination of the parent compound and SD 31723.

No residues of fenbutatin oxide (<0.01 mg/kg) were found in milk, brain, quadriceps, gastroenemius, subcutaneous fat, mesenteric fat or liver, nor in the kidney or heart of two of the three cows. One cow had 0.03 mg/kg fenbutatin oxide in kidney and 0.05 mg/kg in heart. Limits of determination were 0.01 mg/kg in milk and 0.02 mg/kg in tissues.

No information was submitted on the fate in plants, soil, or water/sediment systems. Information on the fate in plants had been submitted to the 1977 Meeting.

In processing

Apples. The most comprehensive processing study received was a simulated commercial process with apples treated at 85 g ai/hl (an exaggerated rate) and harvested after 7 days (compared with GAP 14 days) to ensure sufficient residues in the processed fractions (Table 6, reference 51). Apples were washed, peeled and cored. Peeled and cored apples were sliced and cooked. Peels and cores were ground to produce unclarified juice and wet pomace in the finisher step. Wet pomace was dried in a forced-air dryer for 24 hours at 77°C. Concentration factors were washed apples 0.83, unclarified juice 0.6, wet pomace 1.7, dry pomace 6 and slices 0.02. Residues of SD 31723 and SD 33608 were $\leq$0.1 mg/kg in all fractions. In a separate study water washing (including soap rinse and brushing) reduced the residue level by approximately 56% (Table 6, reference 30).

Additional data were provided from processing apples treated in accordance with GAP into juice, wet pulp and dry pulp, but with fewer details of the procedure (Table 6, references 45 and 50). Concentration factors were juice <0.1, wet pomace 2.4-3.5, and dry pomace 6.9-12. In other studies apples treated in accordance with GAP were processed into juice and wet and dry

pomace, although residue levels in the whole apples were not provided to permit the estimation of concentration factors (Table 6 references 35, 36, 38, 39). Maximum residues from treatments according to GAP were 0.17 mg/kg in juice, 7.5 mg/kg in wet pomace and 24 mg/kg in dry pomace.

Many of the studies included analyses of whole apples and pulp. Pulp residues were generally ≤20% of the whole fruit residues and in most cases <10%. Similar results were observed for pears with residues in the pulp <10% of the whole fruit residue.

Oranges. In one processing study on Valencia oranges about 70% of the field-incurred residue of fenbutatin oxide was removed by "normal" washing 30 days after treatment, although details of the washing procedure were not provided. No residues (<0.05 mg/kg) were found in the juice from whole fruit with field-incurred residues up to 1.4 mg/kg, but residues in the dry pulp were 1-1.6 times the level in unwashed whole oranges (Table 3, reference 1).

A more comprehensive processing study was conducted on Hamlin oranges to simulate commercial processing in accordance with University of Florida Circular 2-266. It consisted of standard washing procedures and "F.M.C. in-line extraction" resulting in three fractions: (1) peel, seed and rag (2) oil emulsion crude and (3) unfinished juice. Fraction 1 was chopped, limed and pressed to give a peel press liquor which was concentrated into molasses and a press residue which was dried into dry citrus pulp. Fraction 2 was further divided into orange oil and peel frit, and fraction 3 into single strength juice and finisher pulp.
Washing whole Hamlin oranges harvested after only 7 days (the GAP PHI) removed 36% of the fenbutatin oxide residue of 3.3 mg/kg from applications in excess of GAP. In this case only trace levels (0.06 mg/kg) were found in the juice. Residues were concentrated in orange oil (6.6 times), dried peel/pulp (4.6 times), peel frits (2.5 times) and in chopped peel/pulp (1.2 times). Residues in the oil emulsion, press liquor, molasses, and finisher pulp were ≤50% of those in the unwashed whole fruit (Table 3, reference 8).

The levels of the total residue of the parent and the two metabolites (calculated as parent) in orange peel and peeled oranges were found to be about 100-120% and 3-7% respectively of the level in whole oranges after 7 days (Table 3, reference 4). Another study showed residues in Navel and Hickson orange pulp to be about 1-3% of those in whole oranges and the residue level in the peel to be about 2-3 times that in the whole orange. A somewhat similar distribution was observed in mandarins, but peel residue levels were a little higher relative to levels in whole oranges (Table 3, reference 9). Other results in Table 3 reveal a similar trend in the distribution of residues in lemons and grapefruit.

Cucumbers. Residues in cucumbers were also largely in the peel with pulp residues ≤1/3 of those in the whole commodity (Table 2).

Grapes (Table 4). Rinsing removed about 30-40% of the grape residue on the day of application, but only about 15-20% of the residue remaining after 13 days (Table 4 reference 25). In a simulated commercial processing study washing and destemming grapes removed approximately 40% of the fenbutatin oxide present after a PHI of 7 days (Table 4, reference 32). No fenbutatin oxide residues (<0.02 mg/kg) were detected in wine or juice from grapes treated according to GAP. The highest residues of fenbutatin oxide from treatments reflecting GAP were 15 mg/kg in raisins and 65 mg/kg in dry pomace (Table 4, reference 18).

Grapes were treated twice with a WP formulation at 1.4 kg ai/ha (180 g ai/hl) with a 14-day interval between treatments, harvested 7 days after the second treatment and subjected to simulated commercial processing. Residues of 0.54 mg/kg were reduced to 0.3 mg/kg in de-stemmed, washed fruit. Residues became concentrated in wet pomace (4.3 times), raisins (4.3 times), dry pomace (18 times), stems (1.8 times) and stem waste, (19 times). The metabolite SD 31723 was found only in dry pomace (0.13 mg/kg) and stem waste, and SD 33608 only in stem waste (Table 4, reference 32). In other studies similar concentration factors were found for raisins (Table

4, references 18 and 30), but lower factors for dry pomace (Table 4, reference 23).

Nuts. Fenbutatin oxide residues in nut shells are typically some 10-50 times those in the meats and residues in hulls ≥3 times those in shells (Table 5).

Peaches. When peaches containing 90% moisture were air-dried in the laboratory to 10% moisture, residues were concentrated from 1.1 mg/kg to 8.2 mg/kg (7.5 times) (Table 8, reference 29) and from 1.9 to 17 mg/kg (9 times) (reference 30). If the residues in the fresh samples were not corrected for 62-65% analytical recoveries the corrected concentration factors would be about 4.8 and 5.5 respectively.

Plums. Plums containing 80% moisture were treated at 1.1 kg ai/ha (60 g ai/hl), harvested after 7 days (GAP PHI 14 days), processed by a simulated commercial procedure to prunes (18.5% moisture) and rehydrated to 33% moisture (Table 8, reference 27). The plums were washed in a spray washer for 15-30 seconds with water at 165-185°F, dried at about 150°F in a forced-circulation air dryer and rehydrated in water at 165-185°F for 3-5 minutes. Pitted plums, dried prunes and rehydrated prunes were analyzed for fenbutatin oxide by analytical method AMR-720-87 (MMS-R-494-2). Samples were also analysed for metabolites SD 31723 and 33608. Mean concentration factors were 2.5 for prunes with 18.5% moisture and 1.2 for prunes with 33% moisture.

Residues in prunes from plums treated approximately in accordance with GAP have been reported up to 3.1 mg/kg (Table 8, reference 24). It is not known whether this value was corrected for the 55% recovery: if not, the "true" residue would be 5.6 mg/kg.

Stability of pesticide residues in stored analytical samples

The 1977 monograph cited a study (TIR-26-116-74) as reporting that residues were stable for 18 months when stored at -15°C, but no details were given. No reports on the stability of residues in stored analytical samples were provided for the present review. A summary discussion on "Freezer storage stability study of fenbutatin oxide (Vendex®) and metabolites in strawberries, egg plants, and almonds" is included in several reports provided to the Meeting (e.g. Table 3, reference 8). This summary reports that residues in all target samples are stable up to 8.5 months when stored at -20°C. Intervals before analysis were generally ≤1 year for the supervised trials data provided, except in some studies on stone fruit where the interval was up to 3 years or more.

Residues in the edible portion of food commodities (see also "Fate of residues in processing", above)

Washing apples was shown to remove about 15% of the residue in whole apples, although under some conditions (soap rinsing and brushing) over 50% may be removed (see processing above) (Table 6, reference 30). Generally ≤20% of the residue in whole apples will be found in the pulp and residues in juice from treatments approximating GAP were generally ≤10% of those in the whole apple (see processing above), although a residue in unclarified juice was 60% of the whole fruit residue.

Residues in banana pulp have been shown to be 2-8% of field-incurred residues in the whole banana (Table 2, reference 6). Washing has been shown to remove 40-70% of fenbutatin residues from whole oranges and it has been demonstrated that residues in peeled oranges are less than 10% of the level in whole unpeeled oranges (Table 3, e.g. reference 4). Residues may be concentrated in orange oil (6.6 times), dried peel/pulp (4.6 times), peel frits (2.5 times) and chopped peel/pulp (1.2 times).

Washing or washing and destemming grapes can remove about 20-40% of the fenbutatin oxide residues, depending on the PHI (Table 4, references 25 and 32). No residues (<0.02 mg/kg) are expected in wine or grapes from

applicatins according to GAP, although concentrations up to about 5-fold in raisins and 18-fold in dry pomace may result from processing.

Canning whole destoned peaches (no reference was made to peeling) field-treated in a manner expected to result in residues up to about 10 mg/kg in whole peaches or 0.2 mg/kg in peeled peaches (similar trials, same time, same place, same variety, same application and similar PHIs) left no detectable residues of fenbutatin oxide (<0.01 mg/kg) or SD 31723 (<0.2 mg/kg) in the canned fruit. The destoned peaches were halved, immersed in boiling sodium hydroxide for 30 seconds, washed with water, steam de-aired for 10 minutes at 85°C for 10 minutes, sealed and heated at boiling point for 20 minutes (Table 8 reference 48).

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No information was provided.

METHODS OF RESIDUE ANALYSIS

Although various analytical methods have been used in the numerous supervised trials only two were provided to the Meeting, one as an appendix to Potter and Nugent, 1978 (method MMS-R-494-1). The principles of this and other methods, control values and analytical recoveries are included in individual field trial reports. The other was method MMS-R-345-1 (Shell, 1972) which was among thosereviewed by the 1977 JMPR (see below). It also includes a TLC procedure for the determination of metabolite SD 31723.

Two basic approaches underly the methods used in most of the trials. The first, reviewed by the 1977 JMPR, includes methods MMS-R-345-1, MMS-R-391-1 or (-2) and WAMS 215-1 (SAMS-1). The second was described in the 1979 monograph but not specifically referenced.

In the first approach extraction is by dichloromethane, chloro-derivatization by HCL digestion, clean-up on an alumina column and determination by GLC with EC detection. For method MMS-R-345-1 the limit of detection was reported as 0.05 mg/kg for the parent compound and 0.1 mg/kg for SD 31723.

In the other procedure (method MMS-R-494-1) the macerated sample is extracted by shaking for two hours with chloroform and HCl, the mixture is allowed to stand overnight, the chloroform evaporated, and the residue taken up in hexane which is partitioned with acetonitrile containing 0.05% tropolone. The acetonitrile solution is evaporated to dryness and the residue taken up in diethyl ether. The residue of the three compounds (parent, SD 31723 and SD 33608) is methylated with methyllithium. After further partitioning and clean-up on a Florisil column determination is by GLC with an FPD (6100A Sn-selective interference filter) (Shell, 1979). From summary descriptions method MMS-4-494-2 appears to be a variation of this procedure.

Although not presented to show details for individual commodities, summary validation results for MMS-494-1 in a variety of crops and animal tissues show mean recoveries of the order of ≥90% for the parent, SD 31723 and Sd 33608, but ranging from 50 to 133% from fortifications of 0.1 to 50 mg/kg of the parent and 0.1 to 0.5 mg/kg of the metabolites. Sample chromatograms suggest that limits of determination for the parent would be 0.02-0.05 mg/kg in cream and 0.1 mg/kg in liver, for SD 31723 0.1-0.2 mg/kg in grapes, 0.1 mg/kg in liver and 0.02-0.05 mg/kg in cream, and for SD 33608 0.02-0.05 mg/kg in grapes, 0.1 mg/kg in cow liver and 0.02-0.05 mg/kg in cream.

With a few exceptions discussed under individual supervised trials, analytical recoveries by the various analytical methods in the crops studied were generally ≥80%. Recoveries were quoted over a range of fortification levels, but generally near the MRLs. Limits of detection (usually expressed as a percentage of full scale deflection, e.g. 4% FS)

were reported for most methods and were generally ≤0.02 or ≤0.05 mg/kg, but few limits of determination were given. Descriptions of analytical methods for the two primary metabolites SD 31723 and SD 33608 were also provided in individual study reports, but again were not provided for review.

A proposed HPLC method for fenbutatin oxide in fruit matrices was also provided to the Meeting (Nicolas *et al.*, undated). Laboratory-fortified samples were extracted with dichloromethane, centrifuged, and filtered through a phase separator. The solvent was evaporated and the sample dissolved in the mobile phase for analysis. An octadecyl silica column was used with methanol containing triethylamine (2-5%) to minimize tailing as the mobile phase. Detection was by UV absorption at 205 nm or by fluorescence (excitation at 410 nm, emission at 490nm) after post-column derivatization with a 254 nm mercury lamp. Analytical recoveries were 56% from apples, 77% from grapes and 48% from oranges at 0.1 mg/kg. The limit of determination was reported as 0.2 mg/kg for apples and grapes with UV detection, but analysis of oranges was not possible owing to lack of specificity. The limit of determination was 0.04 mg/kg in all three matrices with fluorometric detection.

NATIONAL MAXIMUM RESIDUE LIMITS

National maximum residue limits reported to the Meeting are summarized below
(US limits include fenbutatin oxide and its organotin metabolites).

Crop/country	MRL, mg/kg	Notes
ALMONDS		
USA	0.5	
ALMOND HULLS		
USA	80	
APPLES		
Australia	3	
Austria	1.5	
Belgium	2	
Germany	2	registration in progress
Italy	0.5	
Japan	5	
Netherlands	2	
New Zealand	1	pip fruit
S. Africa	2	
Spain	2	
Switzerland	1.5	for fruits
USA	15	
APPLE POMACE, dry		
USA	75	
APRICOTS		
Germany	1	
Japan	5	
BANANAS		
Australia	5	
Spain	1	
BEANS		
Japan	0.5	
Spain	0.5	
Netherlands	0.5	French, scarlet runner, slicing beans
CHERRIES		
Australia	3	stone fruit
Belgium	1	stone fruit
Germany	1	registration in progress
Italy	0.5	stone fruit
Japan	5	
Netherlands	5	
New Zealand	1	stone fruit
Spain	2	stone fruit
Switzerland	1.5	fruits
USA	6	
CITRUS FRUITS		
Australia	5	
Brazil	0.4	peeled
China	5	
Italy	0.5	
Japan	5	
Netherlands	5	
S. Africa	1	
Spain	2	

Crop/country	MRL, mg/kg	Notes
Taiwan	2	
USA	20	
CITRUS POMACE		
USA	35	
CITRUS PULP, dry		
USA	7	
CUCUMBERS		
Belgium	0.5	fruiting vegetables
Denmark	1	greenhouse
Italy	0.5	
Japan	2	
Netherlands	1	gherkins
Spain	1	gherkins
Belgium	1	gherkins, fruiting vegetables
Switzerland	0.2	gherkins
USA	4	
EGG PLANT		
Belgium	o.5	fruiting vegetables
Japan	2	
Netherlands	1	
Spain	0.5	solanaceae
FRUIT		
Austria	1.5	
Switzerland	1.5	
FRUITING VEGETABLES		
Netherlands	1	cucumbers, egg plant, gherkins, melons, okra, peppers, pumpkins (patisson, summer squash), vegetable spaghetti, tomatoes, water melons
GRAPES		
Austria	0.01	
Belgium	2	
France	2	
Germany	4	registration in progress
Italy	0.5	
Japan	5	
Netherlands	5	
Spain	1	
Switzerland	1.5	fruits
USA	5	
GRAPE POMACE, dry		
USA	100	
HOPS		
Australia	20	
Japan	30	
NUTS		
USA	0.5	almonds, walnuts, pecans
NECTARINES		
Australia	3	
OTHER BERRIES		
Netherlands	0.2	
OTHER SMALL FRUIT		
Netherlands	0.2	
OTHER STONE FRUIT		
Netherlands	1	
PEACHES		

Crop/country	MRL, mg/kg	Notes
Australia	3	
Austria	1.5	fruit
Belgium	1	stone fruit
Denmark	5	greenhouse
Germany	4	registration in progress
Italy	0.5	stone fruit
Japan	7	
Netherlands	1	stone fruit
New Zealand	1	stone fruit
S. Africa	2	
Spain	2	
Switzerland	1.5	fruits
USA	10	
PEARS		
Austrlia	3	
Austria	0.5	
Belgium	2	
Germany	2	registration in progress
Italy	0.5	
Japan	5	
Netherlands	2	
New Zealand	1	pip fruit
S. Africa	2	
Spain	2	
Switzerland	1.5	fruits
Taiwan	2	
USA	15	
PLUMS		
Germany	1	
Netherlands	3	
USA	4	
PRUNES		
Japan	3	
USA	4	
PRUNES, dry		
USA	20	
RASPBERRIES		
USA	10	
STONE FRUIT		
Belgium	1	
New Zealand	1	
STRAWBERRIES		
Australia	1	
Japan	3	
Netherlands	3	
USA	10	
TOMATOES		
Belgium	0.5	fruiting vegetables
Denmark	1	greenhouse
Italy	0.5	
Netherlands	1	
Spain	0.5	solanaceae
RAISINS		
USA	20	

Crop/country	MRL, mg/kg	Notes
OTHER FOOD COMMODITIES		
Netherlands	0* (0.05)	
ANIMAL PRODUCTS		
USA	0.5	cattle meat, fat or meat byproducts
Netherlands	0.2	liver
Netherlands	0.2	kidney
Netherlands	0.02*	other meat
USA	0.5	hogs, meat, fat or meat byproducts
USA	0.5	horses or sheep meat, fat or meat byproducts
MILK FAT		
USA	0.1	
Netherlands	0.02*	milks

*At or about the limit of determination

APPRAISAL

Fenbutatin oxide, a miticide registered for use on many crops world-wide, was first reviewed by the 1977 JMPR for both toxicology and residues. A toxicological re-evaluation in the periodic review programme of the CCPR was conducted in 1992, but the corresponding residue review was postponed to 1993 owing to the late arrival of data. Although the present Meeting reviewed over 250 individual reports or studies containing residue data and GAP information, little or no information was provided on some critical supporting studies (e.g. plant, goat and hen metabolism studies, processing studies for tomatoes, freezer storage stability of analytical samples, analytical methods, etc.). The Meeting received processing studies for apples, grapes and citrus and, on request, cow metabolism and transfer studies as well as a chicken feeding study. The Meeting was informed that rat, hen and goat metabolism studies had been submitted to WHO and could be submitted to FAO for future review. A proposed LC analytical method for fenbutatin was also reviewed.

GAP and summary residue data received from Spain and The Netherlands for a number of commodities were received too late for full consideration. The GAP information has been added to the 1993 Monograph. Most if not all of the summary residue information appears to have been included in earlier submissions for 1993 review and has therefore been considered.

There was still a lack of critical supporting information, with the exception of the cow and chicken feeding studies and processing studies provided. Accordingly, the Meeting limited this periodic review primarily to evaluating supervised trials data and/or evaluating data in the context of the available information on current GAP.

Supervised trials data show fenbutatin oxide residues in general to be primarily on the surface or in the peel. Residues in banana pulp are 1-2% of the level in the whole banana, peeled cucumber residues are $\leq$33% of whole cucumber residues, citrus pulp residues are <5% of the whole fruit residues. In nuts residues in the shell are typically 25 times those in the nut meat, although in a few cases only 3 to 4 times. In almonds the hull residues were of the order of 60 times the level in nut meat.

Processing information was provided on a number of commodities. Washing may remove 20 to 40% of the residues on fruits and an even higher proportion in some cases on citrus fruits. Concentration occurs in some processed fractions, with concentration factors of 1.7 in wet apple pomace, 6 in dry apple pomace, 5 in dry citrus pulp, 6.7 in citrus oil, 4.3 in wet grape pomace, 18 in dry grape pomace, 4.3 in dried grapes, 2.4 in dried prunes, and up to 9 in dried peaches. Residues from GAP applications to grapes are $\leq$0.02 mg/kg in wine or grape juice.

In a number of studies samples were also analysed for residues of the metabolites dihydroxybis(2-methyl-2-phenylpropyl)stannane (SD 31723) and 2-

methyl-2-phenylpropylstannoic acid (SD 33608). The former is with few exceptions ≤10% of the fenbutatin oxide residue and the latter usually ≤1/2 the level of the SD 31723. There is some evidence that the canning process may reduce residues near the MRL to non-detectable levels, at least in stone fruit.

Avocado. There is no MRL for avocado. Because data were available only for the flesh and no GAP was available for the countries in which trials were conducted, the Meeting concluded that information was insufficient to support a limit.

Banana. There is currently no MRL for bananas. Maximum residues reflecting GAP were 6.3 mg/kg at 7 days, 3.4 mg/kg at 2 days and 5.7 mg/kg at 0 days. The GAP PHI is 1 day. About 1-8% of the whole fruit residue has been found in the pulp (maximum level 0.14 mg/kg), although ≤2% is likely to be a more reliable estimate taking into account analytical factors. Although few of the results were exactly at the GAP PHI, residues show little decline over 7 days from application, and the Meeting concluded that data over this period were relevant to estimating a maximum residue level. Because results were available from only one country, the Meeting considered additional data reflecting the GAP of other countries desirable. However, because results were available from three locations in three different years, the Meeting concluded that they were sufficient to estimate a 10 mg/kg limit.

Beans. There is no MRL for beans. Residues in green beans from a single application in a single trial in one country were 0.5 mg/kg after 3 days. Data were available from another country at a slightly exaggerated application rate (75 g ai/hl instead of 50 g ai/hl) from a formulation which is not recognised as GAP. The plot size was only 12 m^2. The Meeting could not recommend a limit for green beans.

Residues in French beans from 2 applications at GAP rates under glasshouse conditions in one country resulted in maximum residues of 0.4 mg/kg after 6 days compared with a GAP PHI of 7 days. At twice the 25 g ai/hl GAP rate residues were 0.15 mg/kg after 7 days. The Meeting concluded that the data were insufficient to support a limit for beans.

Citrus. The CXL for citrus fruits is 5 mg/kg. Although 43 trials were conducted in 5 countries, 3/4 of these were in one country and only 12 of the trials represented current GAP (6 on oranges, 2 on grapefruit, 3 on lemons and one on mandarins). Maximum residues resulting from GAP were 1.5 mg/kg in grapefruit, 2.4 mg/kg in mandarins, 3.3 mg/kg in oranges and 4 mg/kg in lemons, the last at a 21-day PHI compared with the GAP 7-day PHI. Other trials at GAP application rates, but at twice the GAP number of applications resulted in residues up to 14 mg/kg, but generally less than 10 mg/kg. Residues of the metabolite SD 31723 were typically 2-10% of the parent compound in whole oranges and in all the fruits the residue of SD 33608 tends to be about half that of SD 31723.

While additional data reflecting GAP for oranges, grapefruit and mandarins are desirable, the Meeting concluded that the available results were marginally sufficient in a mutually supportive way to confirm the existing 5 mg/kg citrus group CXL for these individual citrus fruits. This does not apply to lemons or limes, because relatively few trials representing GAP were available for lemons. As noted, the highest GAP residue (4 mg/kg) in lemons was at a 21-day PHI compared with a GAP 7-day PHI, and the next highest residue of 2.4 mg/kg was from only single applications whereas two are permitted. The Meeting concluded that additional data reflecting current GAP for lemons and/or limes with the minimum PHI and maximum application rates would be required before a limit could be recommended for lemons or limes or for citrus fruits as a group. Additional data reflecting GAP for oranges, grapefruit and mandarins are also desirable.

The available information from the simulated commercial processing of oranges with field-incurred residues indicates that residues in dry orange pulp are 2-4.6 times those in whole unwashed oranges. Assuming a fivefold concentration and a residue at the 5 mg/kg MRL level in the unprocessed fruit, the maximum dry pulp residue would be 25 mg/kg compared with the

current 7 mg/kg limit. Although there is currently no Codex MRL for citrus oil, the 6.6 concentration factor from whole unwashed oranges indicates that an MRL of 30 mg/kg would be needed.

Cucumbers. The CXL for cucumbers is 1 mg/kg. Data were available from 4 European countries (open and glasshouse) and the USA (open), although the US data do not correspond to current GAP. While application rates in the US trials were higher and PHI intervals shorter than required by GAP in Europe, the data are useful for illustrating the dependence of residues on the application rate and giving some indication of differences in residues between 2 and 3 applications. Residues from single applications representing European GAP ranged from 0.03 to 0.3 mg/kg. The Meeting concluded that a 0.5 mg/kg limit was supported.

Egg plant. The CXL for egg plant is 1 mg/kg. Because data were available from only a single glasshouse trial in one country, because no GAP information was provided for that country and because the trials did not conform to GAP application rates or PHIs of neighbouring countries the Meeting concluded that the information was insufficient to support the limit, and recommended its withdrawal.

Gherkins. The CXL for gherkins is 1 mg/kg. Because only summary information from a single glasshouse trial was available, the Meeting concluded that it was insufficient to support the limit, and recommended its withdrawal.

Grapes. The CXL for grapes is 5 mg/kg. Data were available from over 60 supervised trials in 5 countries. Maximum residues of about 2 mg/kg resulted from European GAP and 4 mg/kg from US GAP. While US GAP is comparable to that of several European countries, many more US trials were available. The Meeting confirmed the existing 5 mg/kg limit. Currently there is no Codex limit for processed grape products. Because the concentration of residues from grapes to raisins is about fourfold, the Meeting concluded that an MRL of 20 mg/kg for raisins would be appropriate, based on 5 mg/kg in the whole fruit. Similarly, concentration of the order of 18 times in dry pomace (or stem waste) supports a limit of 100 mg/kg for dry pomace.

Hops. No MRL is proposed for fenbutatin oxide in hops. Supervised trials data reflecting GAP were available from a single trial in one country, resulting in residues of the order of 5 mg/kg. The Meeting concluded that these data were inadequate for estimating a maximum residue level.

Melons. Currently there is a 1 mg/kg CXL for melons, except watermelons. Although no residues (<0.01 mg/kg) resulted 7 or 14 days after applications at GAP rates in a single trial in one country whose GAP PHI is 3 days, the Meeting concluded that the data were insufficient to support the limit, and recommended its withdrawal.

Nuts. No MRL is established for nuts. Maximum residues of fenbutatin oxide in the nut meats of almonds, pecans, walnuts and filberts from treatments at US GAP application rates, PHIs and number of applications included residues of $\leq$0.02, 0.04, 0.05, 0.13, 0.16, and 0.3 mg/kg (the last at a 1.5 times application rate). Appreciably more data were provided which were at GAP application rates and PHI, but with 3 applications instead of the maximum 2 per season which is GAP. These included residues of $\leq$0.02 (10), 0.03 (4), 0.04 (2), 0.05, 0.07, 0.08, 0.1, 0.2 (3) and 0.3 mg/kg.

Although there were no side-by-side comparisons, the overall data suggest similar residues from 2 or 3 applications at GAP rates. The Meeting therefore concluded that a 0.5 mg/kg limit could be supported for almonds, pecans, and walnuts. Residues were observed up to 56 mg/kg in almond hulls (an animal feed item).

Data were too limited to support a limit for filberts, for which no GAP information was provided.

Data were also available for metabolites SD 31723 and SD 33608 with levels of <0.02 mg/kg in nut meats.

Peppers. The CXL for peppers (sweet) is 1 mg/kg. Data were available from two supervised trials. The single application (2 are permitted) in the Belgian trial was outdoors, although the provided Belgian GAP was for glasshouse uses. The Netherlands glasshouse trial was at 0.5 kg ai/ha, which could not be related to the GAP rate of 25 g ai/hl. Although residues in two supervised trials (1 and 0.6 mg/kg after the 3-day PHIs) suggest that residues may not exceed 1 mg/kg from GAP, the Meeting did not consider two data points reflecting GAP sufficient to support the MRL, and recommended its withdrawal.

Pome fruit. The CXLs are 5 mg/kg for apples and pears and 20 mg/kg for dry apple pomace. The data base for pome fruit included 103 supervised trials for apples and 17 for pears. In many trials residues decreased little during 2-3 weeks after application. Maximum residues in apples were 2.9 mg/kg from GAP applications in non-US trials. In US trials the highest residues from GAP applications were 4.3 mg/kg from dilute sprays, 9.6 mg/kg from concentrated SC sprays and 12 mg/kg from concentrated WP sprays, although only three of the many trials on apples were with concentrated sprays at GAP application rates. The two apple trials which resulted in the higher residues were on a different variety from those in other trials, but it could not be concluded that the variety influenced the residue. While the trial plot was a single tree, this was also true of other trials. Higher residues from concentrated spray applications are also suggested by pear trials where maximum residues reflecting US GAP were 2.3 mg/kg for dilute sprays and 5.6 and 3 mg/kg for WP and SC concentrated spray applications respectively. These dilute and concentrated spray applications were all on the same variety of pear.

While the Meeting concluded that the current 5 mg/kg Codex limit is adequate for dilute spray applications, it would not accommodate the USA concentrated spray uses. The Meeting concluded that additional data reflecting GAP would be needed to accommodate these.

The Codex limit for dry apple pomace is 20 mg/kg to accommodate the current 5 mg/kg limit on apples (fourfold concentration factor). Concentration and reduction factors in apple processed products estimated from studies provided to this Meeting varied, depending on the study: juice (unclarified) 0.6 times, wet pulp 1.7-3.5 times, and dry pulp 6-12 times. Clearly the fourfold factor previously used by the JMPR is too low in view of this information. Putting greater weight on the most comprehensive processing study provided, the Meeting concluded that for estimating maximum residue levels a factor of 7 would be reasonable for whole fruit to dry pomace. With an MRL of 5 mg/kg and a concentration factor of 7 a 40 mg/kg limit can be recommended for dry apple pomace.

Raspberries. No MRL exists. Supervised trials information was available for only one country, for which no GAP information was provided and the GAP of other countries could not be used. The Meeting concluded that insufficient information was available to estimate a maximum residue level.

Soya beans. Because residue results (<0.01 mg/kg) were available from only three supervised trials in a single country 67-80 days after application compared with the GAP 7-day PHI, and because analytical recoveries by analytical method SAMS 345-1 were highly variable at a 0.2 mg/kg fortification level (50-110%), the Meeting concluded that data reflective of GAP were insufficient to support a limit.

Stone fruit

Forty-seven studies from 8 countries were available for stone fruit, representing 76 supervised trials. Most of the results referred to de-stoned fruit. No attempt was made to calculate residue in the whole fruit including stone, since average stone weights were only about 6% of the whole fruit weight.

Cherries. The CXL is 5 mg/kg. Maximum residues reflecting approximate GAP in Germany were 0.6 mg/kg and in the USA 5.1 mg/kg (whether results were adjusted for 69% recoveries was not stated). If not corrected, a maximum

residue of 7.4 mg/kg would be indicated. Other US data also did not indicate whether corrections had been made for low recoveries. If not, other residues when adjusted for recoveries would be of the order of 7 mg/kg. Results from The Netherlands data did not reflect the national 42-day PHI. However, the residues up to 1.2 mg/kg were from applications consistent with German or Italian GAP PHIs, although German GAP was reported to be due to expire in 1993. Maximum residues of SD 31723 were 0.9 mg/kg and SD 33608 0.04 mg/kg from GAP. The former was ≤25% of the fenbutatin oxide residue and SD 33608 is usually less than half of the level of SD 31723.

The Meeting was particularly concerned at the lack of information on whether results from several US studies (the major portion of the data base) were corrected for analytical recoveries less than 70% (58% in one case), and at the information that German reregistration is to expire in 1993 (the German GAP is relevant to other European trials for which no GAP was provided). The Meeting concluded that a 10 mg/kg limit could be supported for cherries.

Peaches, nectarines. The CXL is 7 mg/kg. Maximum residues in peaches reflecting approximate GAP in Australia were 2.5 mg/kg, and in the USA 8 mg/kg from a 1.5 times rate (5.3 mg/kg adjusted to the GAP rate) and up to 5.8 mg/kg from approximate GAP rates, but with 3 instead of the permitted 2 applications. Residues were up to 3.5 mg/kg in two US trials reflecting GAP on nectarines. Maximum peach residues were 0.8 mg/kg in Canada at US GAP rates, 1.3 mg/kg in France at German GAP rates, and 3.3 mg/kg in Germany. Three trials in South Africa also resulted in residues up to 3.1 mg/kg after 10 days and 4 mg/kg after 13 days (14 day-PHIs are common in other countries) at application rates which are GAP in other countries, although GAP information for South Africa was not provided. Residues were up to 6 and 7.8 mg/kg after 13 and 10 days respectively at higher application rates. The Meeting concluded that the data supported the current 7 mg/kg limit for peaches and in a mutually supportive way could support a limit at the same level for nectarines.

Plums. The CXL is 3 mg/kg. Maximum residues approximating GAP were: German trials 0.7 mg/kg, United States trials 2.1 mg/kg, Netherlands trials <0.1 mg/kg. No GAP information was available for South Africa. Residues were 0.9 and 1 mg/kg after 14 days. From GAP applications, maximum residues of SD 31723 were 0.07 mg/kg and of SD 33608 0.04 mg/kg. SD 31723 is usually <5% of the fenbutatin oxide residue and SD 33608 is similar to or lower than SD 31723. Control values for fenbutatin oxide range from <0.01 to 0.1 mg/kg, depending on the analytical method used.

Although recoveries in some US trials were below 70% and no information was provided on whether the results were corrected, recoveries were acceptable in the trial with the highest GAP residue (2.1 mg/kg). Furthermore, assuming that the results in the trials with low recoveries are uncorrected, maximum residues would be about 2.2 mg/kg. The Meeting concluded that the data were sufficient to support the CXL for plums.

There is no MRL for prunes (dried plums). Data provided indicate that fenbutatin oxide residues are concentrated in drying plums by a factor as high as 2.5. Applying this to the 3 mg/kg limit for fresh plums would imply an MRL of 7.5 or 10 mg/kg for dried prunes.

Residue levels in dried plums from trees treated in accordance with GAP were provided, although no data were included for the fresh fruit from which a concentration factor could be estimated. Maximum residues were 3.1 mg/kg. Analytical recoveries for this study were only 55% and it was not indicated whether the result had been corrected for the low recovery. If not, a residue of 5.7 mg/kg would be indicated. This would be consistent with the theoretical 7.5 mg/kg estimated above.

Strawberries. The CXL is 3 mg/kg. Twenty-seven reports were available from 7 countries representing 47 supervised trials (32 from the USA). Data from two countries could not be related to the available GAP. Maximum residues approximating GAP were 1.3 mg/kg from Australian trials, 0.4 mg/kg from French trials, 0.5 mg/kg from UK trials, and 7 mg/kg from Mexican trials

(based on US GAP). The more numerous US trials resulted in a fairly continuous distribution of residues, except for two values, up to 9.9 mg/kg (the last from a 1.2 times application rate). The exceptions were at one site with residues of 12 and 18 mg/kg. Because information on the project history for these trials was in question and because the residues (especially 18 mg/kg) were not consistent with those found in numerous other similar trials, even at exaggerated rates, the Meeting gave little weight to these two values.

Maximum residues from GAP of the metabolites SD 31723 and SD 33608 were respectively 0.1 and 0.05 mg/kg after 1 day. Generally residues of SD 31723 were $\leq$5% of fenbutatin oxide residues and SD 33608 residues were about half or less of the SD 31723 residues (after one day). The Meeting concluded that the data supported an increase in the current 3 mg/kg CXL to 10 mg/kg.

Tomato. The CXL is 1 mg/kg. Four of the 12 supervised trials were according to GAP, and the maximum residues in these: Denmark 0.4 mg/kg (glasshouse), Italy 0.3 mg/kg (field), the UK 0.3 mg/kg (glasshouse). Although results were available from 3 additional countries, they could not be related to the GAP information provided. Residues were up to 0.8 mg/kg after 3 or 4 days in two trials that could not be confirmed to reflect GAP. No tomato processing data were provided. No residues (<0.1 mg/kg) of metabolite SD 31723 were found in the two trials in which it was determined.

The Meeting concluded that the data were adequate to confirm the current limit for tomatoes, but only for glasshouse uses.

Animals. Feeding studies with labelled fenbutatin oxide at 34 ppm dietary feeding levels indicate that the greatest potential for residues is in the kidney and liver of cattle, with possible low residues in muscle. Conventional feeding studies were also conducted at 11 or 96 ppm in the cattle diet for 21 or 22 days. No residues (<0.02 mg/kg) were found in milk, cream or tissues from the lower feeding level. Residues of fenbutatin oxide were found in all cream and tissue samples from the higher feeding level, while SD 31723 was found only in the liver and kidney. SD 33608 was not detected in any sample (<0.02 mg/kg).

Depending on the assumptions used, a dietary intake of the order of 20 ppm could be estimated, about twice the level in the lower feeding level trial. Adjusting data from the highest feeding level trial to a 20 ppm dietary burden results in maximum fenbutatin oxide residues in liver of 0.02, kidney 0.05, fat of meat 0.01, muscle 0.01 and milk fat 0.05 mg/kg.

Again depending on what assumptions are made, a case could be made for a slight lowering of the previously estimated 0.2 mg/kg limits for liver and kidney, but since the levels are not much greater than the validated limits of determination for these organs, and because more than one of the feed items could be fed at one time, the Meeting concluded that the liver and kidney limits previously estimated for cattle, goats, pigs, horses and sheep could be confirmed. They have been combined under a new proposal at the same level for edible offal.

The Meeting had some reservations about the previous estimates for cattle meat and milk of 0.02 mg/kg at the limit of determination. There was no evidence that the levels would be exceeded in practice, but the analytical method had not been validated below 0.1 mg/kg for any animal matrix in studies provided to the Meeting. For this reason the Meeting recommended increasing the stated limits of determination and hence the MRLs for these commodities to 0.05 mg/kg and limits for the meat of cattle, dogs, horses and sheep have been combined at the same level as a new proposal for meat.

The Meeting also observed that residues of SD 31723 can be about twice those of fenbutatin oxide in cattle liver. Because residues of fenbutatin oxide are found in liver and because it is the only matrix in which SD 31723 exceeds fenbutatin oxide, the Meeting concluded that definition of the residue solely as fenbutatin oxide is satisfactory.

Residues in skim milk and cream indicate a propensity for fenbutatin oxide to accumulate in lipid rather than aqueous media, but levels in muscle do

not differ from those in mesenteric or subcutaneous fat sufficiently to regard fenbutatin oxide as a fat-soluble pesticide.

Feeding chickens at 5 ppm dietary levels produced no residues of fenbutatin oxide or its two metabolites in tissues or eggs, except 0.02 mg/kg fenbutatin oxide in two whole egg samples. From the 25 ppm dietary feeding level the maximum residues of fenbutatin oxide were 0.04 mg/kg in liver, 0.03 mg/kg in kidney and 0.12 mg/kg in whole eggs. These decreased to <0.02 mg/kg in liver and kidney 3 days after cessation of feeding, but the decrease was slower in whole eggs. No residues of either parent compound or metabolites were found in other tissues or organs. As in the case of cattle, residues of SD 13723 were greater than those of fenbutatin oxide in liver (3 to 5 times as high in this case) and residues of SD 33608 were generally comparable to those of the parent compound.

If it is assumed that the greatest dietary intake from feed items for which there are MRLs would be from dry grape pomace (100 mg/kg MRL) and that it is fed at a maximum of 5% of the diet, a dietary intake of about 5 ppm can be estimated. Maximum fenbutatin oxide residues of 0.02 mg/kg in whole egg from the 5 mg/kg feeding level and 0.12 mg/kg from the 25 ppm level support 0.02 mg/kg as a maximum residue level for whole eggs. While SD 317243 might occur near 0.02 mg/kg in liver (0.12 mg/kg from 25 ppm feeding), residues of fenbutatin oxide *per se* would not be expected to be above 0.02 mg/kg. Although residues would be likely not to exceed 0.02 mg/kg in whole eggs, kidney or liver, the same considerations as those mentioned above regarding the levels of method validation for cattle products led the Meeting to conclude that a limit of 0.05 mg/kg (not a limit of determination, because residues around 0.02 mg/kg may occur) would be more appropriate in whole eggs and 0.05 mg/kg (as a limit of determination) in liver and kidney. There would be no compelling need for a limit in poultry meat or fat. Because limits are proposed for eggs and chicken edible offal, 0.05 mg/kg is recommended for chicken meat as a limit of determination level.

Only one of the two analytical methods used in the supervised trials was provided, although the principles were summarized and recoveries and limits of detection were usually provided with field trials data. The two basic methods were both described in earlier monographs. The first is based on chloro-derivatization in a solvent containing HCl followed by GLC determination. The second (e.g. method MMS-R-494-1 provided to the Meeting) includes methylation of fenbutatin oxide, SD 31723 and SD 33608 with methyllithium and determination by GLC with flame-photometric detection of tin. In general determination at 0.02 to 0.05 mg/kg of each compound in cream, 0.1 mg/kg of each in cow liver, and 0.1 to 0.2 mg/kg SD 31723, 0.05 mg/kg SD 33608 and probably $\geq$0.1 mg/kg parent compound in grapes appears to be supported by sample chromatograms. Recoveries from the various substrates were generally $\geq$80%, but at near MRL levels, especially for fenbutatin oxide.

The submitted method may be adequate for regulatory analysis at proposed MRL levels, although submission of all of the analytical methods with sufficient information to permit estimation of the limits of determination and of any information on multi-residue methods suitable for enforcement is desirable.

A proposed liquid chromatographic procedure was also provided, but it was not validated sufficiently for the Meeting to recommend its use.

RECOMMENDATIONS

On the basis of the data on residues from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits. The Meeting could not confirm some current CXLs at this time. They are also indicated below.

Definition of the residue: fenbutatin oxide

Commodity		Recommended MRL (mg/kg)		PHI, days
CCN	Name	New or confirmed	Previous	
TN 0660	Almonds	0.5	-	14
FP 0226	Apple	W (Note 1)	5	14
AB 0226	Apple pomace, dry	40	20	14
FI 0237	Banana	10	-	7
FS 0013	Cherries	10	5	
PO 0840	Chicken, edible offal of	0.05*	-	
PM 0840	Chicken meat	0.05*	-	
FC 0001	Citrus fruits	W (Note 2)	5	
AB 0001	Citrus pulp, dry	25	7	7
VC 0424	Cucumber	0.5	1	
MO 0105	Edible offal (mammalian)	0.2	-	
V0 0440	Egg plant	W	1	
PE 0112	Eggs	0.05	-	
VC 0425	Gherkin	W	1	
FB 0269	Grapes	5	5	
FC 0203	Grapefruit	5	5 for citrus	7
AB 0269	Grape pomace, dry	100	-	
MO 1292	Horse, kidney	W (Note 3)	0.2	
MO 1293	Horse, liver	W (Note 3)	0.2	
MO 0098	Kidney of cattle, goats, pigs and sheep	W (Note 3)	0.2	
MO 0099	Liver of cattle, goats, pigs and sheep	W (Note 3)	0.2	
FC 0206	Mandarin	5	5 for citrus	7
MM 0095	Meat	0.05*	-	
MO 0096	Meat of cattle, goats, horses, pigs and sheep	W (Note 4)	0.02* 5 for citrus	
VC 0046	Melons, except Watermelon	W	1	
ML 0106	Milks	0.05*	0.02*	
FC 0208	Oranges, Sweet	5	5 for citrus	7
FS 0247	Peaches	7	7	14
FP 0230	Pear	W (Note 1)	5	14
TN 0672	Pecans	0.5	-	14
VO 0445	Peppers, Sweet	W	1	
FS 0014	Plums (including Prunes)	3	3	14
FP 0009	Pome fruits	5	Apple 5, Pear 5	14
DF 0014	Prunes (dried plums)	10	-	
DG 5623	Raisins	20	-	
FB 0275	Strawberry	10	3	1
VO 0448	Tomato	1	1	
TN 0678	Walnuts	0.5	-	14

* At or about the limit of determination W: The previous recommendation is withdrawn

Notes 1. Replaced by limit for Pome fruit
2. Replaced by separate limits for Grapefruit, Mandarin, and Orange, sweet
3. Replaced by Edible offal (mammalian)
4. Replaced by revised limit for Meat

FURTHER WORK OR INFORMATION

Desirable

1. Information on whether residues in US stone fruit trials in 1993 Monograph Table 8 references 5,6,7,9 (cherries), 21, 23, 24, (plums), and 29, 30 (peaches), were corrected for analytical recoveries.

2. Information on South African GAP for the use of fenbutatin oxide on peaches.

3. Submission of the analytical methods used in the supervised field trials and in the cow feeding study TIR-26-119-73, with validation information.

4. Current information on analytical methods suitable for enforcement for both plant and animal foods, including multi-residue methods.

5. Current information on the stability of residues in stored analytical samples.

6. Current information on the fate of residues in poultry, plants, soil and water/sediment systems. Metabolism studies on rats, goats and hens reportedly submitted to WHO are specifically requested .

7. Information on residues in food in commerce or at consumption.

8. Information on the interval between the last feeding and slaughter in

cow feeding study TIR-26-119-73 (Koos, 1973).

9. Submission of Report 22-112-74 (on the fate of residues), referenced in Potter and Nugent (1978), as the basis for analyses of animal products for fenbutatin oxide, SD 31723 and SD 33608.

10. Additional pome fruit data reflecting US concentrated spray GAP.

11. Tomato processing information.

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Table 2 (miscellaneous commodities)

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23. Shell. 1978. Residue Data For Vendex® Miticide and Its Organotin Metabolites SD 31723 and SD 31723 in Grapes and Processed Products Resulting From Three Applications of Vendex® to Grape Vines, a California Study. Unpublished Shell Report No. TIR-24-254-78. 1992 Vol. 6 Shell Submission to the EC.

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Table 5 (tree nuts)

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2. Shell 1981a. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Pecans Following Three Applications of Vendex to Pecan Trees, a Georgia Study. Unpublished Shell Report RIR-24-306-81. 1992 Shell Volume 8 submission to the European Commission.

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5. Nugent, K., Doern, B. and Krudop, W. 1976a. Residue Levels of Vendex(R) Miticide in Pecans Resulting From the Application of Vendex, a Texas Study. Unpublished Shell Report TIR-24-186-74. 1992 Shell Volume 8 submission to the European Commission.

6. Nugent, K., Doern, B. and Krudop, W. 1976b. Residue Levels of Vendex(R) Miticide in Pecans Resulting From the Application of Vendex, a Louisiana Study. Unpublished Shell Report TIR-24-273-74. 1992 Shell Volume 8 submission to the European Commission.

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9. Shell. 1979c. Residue Data For Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Walnuts Following Three Applications of Vendex to Walnut Trees, a California Study. Unpublished Shell Report RIR-24-257-79. 1992 Shell Volume 8 submission to the European Commission.

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Metabolites SD 31723 and SD 33608 in Walnut Hulls, Shells and Meats Resulting From Two Applications of Vendex to Walnut Trees, a California Study. Unpublished Shell Report TIR-24-301-77. 1992 Shell Volume 8 submission to the European Commission.

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19. Shell. 1973. Residue Data For Vendex(R) Miticide in Almonds Resulting From the Application of Vendex, California Study. Unpublished Shell Report TIR-24-708-73. 1992 Shell Volume 8 submission to the European Commission.

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24. Nugent, K. and Matsuyama, H. 1979. Residue Levels of Vendex(R) Miticide and its Organotin Metabolites SD 31723 and SD 33608 in Almond Hulls, Shells and Meats Resulting From Two Applications of Vendex to Almond Trees, a California Study. Unpublished Shell Report TIR-24-255-78. 1992 Shell Volume 8 submission to the European Commission.

Table 6 (pome fruit)

Unpublished Shell International Chemical Co. Ltd. Reports submitted (unless otherwise indicated) to the 1993 JMPR in the form of a 1992 Volume 3 submission (summarized in Volume 1) to the European Commission.

Apples

1. DEGR.80.114
2. WKGR.0125.74
3. WKGR.0093.75
4. WKGR.0014.74
5. BETR.86.035
6. BEGR.0043.76
7. BEGR.0105.74
8. BEGR.0016.75
9. BEGR.0004.78
10. BEGR.83.005
11. WKGR.0089.73
12. WKGR.0142.73
13. BEGR.0016.78
14. R-139-89[1]
15. R-140-89[1]
16. R141-89[1]
17. R142-89[1]
18. R143-89[1]
19. R144-89[1]
20. BETR.90.018[2]
21. BEGR.79.106
22. BEGR.81.026
23. WKGR.0027
24. BEGR.0030.76
25. WKGR.0111.74
26. WKGR.0034.74
27. BLGR.80.1116
28. WKGR.0013.74
29. WKGR.0044.76
30. TIR-26-201-73
31. TIR-26-148-72
32. RIR-26-137-72
33. TIR-26-147-72
34. TIR-26-145-72
35. TIR-26-143-72
36. TIR-26-141-72
37. TIR-26-125-71
38. TIR-26-129-72
39. TIR-26-131-72
40. TIR-26-134-72
41. TIR-26-133-72
42. RIR-24-361-78
43. TIR-26-135-72
44. TIR-26-157-73
45. TIR-26-101-74
46. RIR-24-371-78
47. RIR-25-348-79
48. RIR-24-234-80
49. RIR-24-362-78
50. TIR-26-197-73[3]
51. AMR-112588[4]

Pears

52. BEGR.80.116
53. WKGR.0180.74
54. WKGR.1202.75
55. BEGR.0011.75
56. BEGR.0029.76
57. BEGR.0031.76
58. WKGR.0107.74
59. RIR-24-283-80
60. RIR-24-233-80
61. RIR-24-274-80
62. RIR-24-351-79
63. TIR-26-139-71B

[1] Field reports, in German. Table 6 summary based primarily on Volume 1 summary data.
[2] Included in 1992 Volume 3 EC submission, but not in Volume 1 summary.
[3] Included in June 7, 1993 Shell submission, not in EC submission
[4] Included in June 4, 1992 Volume 3 Shell International/E.I Du Pont submission to the 1992 JMPR.

Table 7 (strawberries and raspberries)

Unpublished Shell International Chemical Co. Ltd. Reports submitted (unless otherwise indicated) to the 1993 JMPR in the form of a 1992 Volume 7 submission (summarized in Volume 1) to the European Commission.

Strawberries

1. ASTL.83.005
2. TIR-24-167-25
3. TIR-24-164-75
4. BEGR.82.007
5. BEGR.0014.77
6. BETR.87.004
7. BLGR.0087.77
8. BEGR.81.043
9. BLGR.0086.77
10. WKGR.0068.75
11. RIR-24-240-83
12. RIR-24-168-84
13. TIR-24-195-75
14. RIR-24-158-8L1
15. TIR-14-158-76
16. TIR-24-142-79
17. TIR-24-274-76
18. TIR-24-164-79
19. TIR-24-143-79
20. RIR-24-159-81
21. RIR-24-16L7-84
22. RIR-24-214-81
23. TIR-24-707-73
24. TIR-24-163-79
25. TIR-24-155-74
26. TIR-24-140-74
27. TIR-24-278-74

Raspberries

28. BEGR.81.122

Table 8 (stone fruit)

Unpublished Shell International Chemical Co. Ltd. Reports submitted (unless otherwise indicated) to the 1993 JMPR in the form of a 1992 Volume 5

submission (summarized in Volume 1) to the European Commission.

Cherries

1. BEGR.0066.77
2. BEGR.81.033
3. TIR-24-250-74
4. TIR-24-243-74
5. RIR-24-282-81
6. RIR-24-175-81
7,9 RIR-24-201-l80
8. RIR-24-165-79
7,9 RIR-24-201-80
10. TIR-24-205-78
11. TIR-24-204-78
12. TIR-24-177-78
13. BLGR.0066.77

Plums

14. BEGR.0015.78
15. BEGR.79.107
16. BEGR.81.037
17. BLGR.0088.77
18. BLGR.0050.77
19. BEGR.0014.76
20. RIR-24-128-82
21. RIR-24-108-81
22. RIR-24-102-80
23. RIR-24-333-81
24. RIR-24-226-81
25. RIR-24-182-81
26. TIR-24-242-74
27. AMR-1127-88*
29. TIR-24-216-78
30. TIR-24-223-77
31. TIR-24-700-74
32. TIR-24-714-73
33. TIR-24-702-73
34. TIR-24-713-73

Plums cont'd

35. TIR-24-702-73-B
36. WKGR.0101.75
37. BEGR.0044.76
38. BEGR.82.048
39. BEGR.83.028
40. BEGR.0020.77
41. WKGR.0093.73
42. BEGR.83.008
43. WKGR.0139.73
44. WKGR.0015.74
45. WKGR.0065.75
46. BLGR.0089.77
47. BEGR.0035.76
48. BLGR.79.142
49. WKGR.0108.74

Peaches

28. BEGR.80.115

Peaches cont'd

Nectarines

50. RIR-24-204-81
51. RIR-24-353-81

* Reference 27 included in June 4, 1992 Volume 3 Shell International/E.I Du Pont submission to the 1992 JMPR.