

**PROPARGITE (113)**  
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**EXPLANATION**

Propargite is an acaricide. It was first evaluated for residues in 1977 and then in 1978, 1979, 1980 and 1982. The current definition of the residue is propargite. The residue is fat-soluble. The 1977, 1980, 1982 and 1999 JMPRs assessed the compound toxicologically. The present Meeting determined that the acceptable daily intake for humans is 0–0.01 mg/kg bw and that an acute reference dose is not necessary. The present review is part of the CCPR Periodic Review Programme.

The manufacturer has reported data on metabolism, analytical methods, animal feeding studies, supervised field trials, GAP, processing, frozen storage stability of residues and environmental fate. The government of Australia reported information on GAP, labels, residues in food in commerce or at consumption and national residue limits, and those of Thailand on GAP and Germany on GAP and national MRLs.

**IDENTITY**

ISO common name: propargite

Chemical name

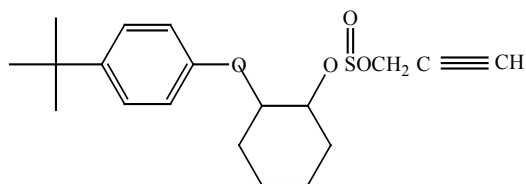
IUPAC: 2-(4-*tert*-butylphenoxy)cyclohexyl prop-2-ynyl sulfite

CA: 2-[4-1,1-dimethylethyl]phenoxy]cyclohexyl 2-propynyl sulfite

CAS No.: 2312-35-8

Molecular formula: C<sub>19</sub>H<sub>26</sub>O<sub>4</sub>S

Structural formula:



Propargite

Molecular weight: 350

**Physical and chemical properties**

Pure active ingredient

UV spectrum: Maximum absorbance at 276 nm with a mean molar extinction coefficient of 1387 (Gaydosh, 1988).

Mass spectrum (EI): Significant ions (m/z) were 350, 335, 201, 173, 150, 135, 107, 81, 57 and 39 (Yu, 2000).

Dissociation constant: pKa >12.0 (no ionization observed) (Tang and Rose, 1988)

Technical material

Purity: 90.6% (range 88.49-92.98%, for 20 lots from 2 plants) (Brown, 1994; Brown and Riggs, 1991)

Main impurities: Twelve impurities and solvent were identified in the technical material, ranging from 0.01–2.9% each.

Appearance: Light to dark brown viscous liquid (Judge and Smilo, 1987)

Odour: Faint odour of solvent (Judge and Smilo, 1987)

Vapour pressure: <math>3.12 \times 10^{-6}</math> torr at  $24 \pm 1^\circ\text{C}</math> (Judge and Smilo, 1987)  
 <math>4.49 \times 10^{-8}</math> mm Hg at  $25^\circ\text{C}</math> (Blasberg and Schofield, 1989)$$

Boiling point: decomposes before boiling. Estimate based on molecular mass:  $475^\circ\text{C}</math> (Judge and Smilo, 1987)$

Octanol/water partition coefficient: log  $P_{ow}$   $3.7 \pm 0.30$  (Smilo, 1987)  
 $5.8 \pm 0.17$  (Young, 1993)

Solubility: water: 1.93  $\mu\text{g/ml}$  ( $25^\circ\text{C}</math>) (Judge and Smilo, 1987; Spare, 1987a)  
 0.632 mg/l (in distilled water;  $25^\circ\text{C}</math>)  
 0.573 mg/l (pH 5;  $25^\circ\text{C}</math>)  
 0.701 mg/l (pH 7;  $25^\circ\text{C}</math>)  
 0.585 mg/l (pH 9;  $25^\circ\text{C}</math>) (Akhtar, 1988a)  
 acetone: >1 g/ml ( $25^\circ\text{C}</math>) (Judge and Smilo, 1987; Spare, 1987a)  
 hexane: >1 g/ml ( $25^\circ\text{C}</math>) (Judge and Smilo, 1987; Spare, 1987a)  
 hexane, toluene, dichloromethane, methanol, acetone: >200 mg/ml at  $20 \pm 1^\circ\text{C}</math> (Akhtar, 1988b)  
 calcium acetate (0.01M): 0.54 ppm at  $20^\circ\text{C}</math> (Tutty, 1995)  
 water solubility of the glycol ether metabolite: 3.5 mg/l ( $20^\circ\text{C}</math>) (Mitchell, 1992)$$$$$$$$$$

Relative density: 1.0818 (unstated temperature) (Tutty, 1993d)

pH: 3.99 (1% in aqueous solution) at  $25 \pm 1^\circ\text{C}</math> (Tutty, 1993a)$

Henry's Law constant:  $3.3 \times 10^{-8}$  atm.m<sup>3</sup>/mol at  $25^\circ\text{C}</math> (Pierce, 1995)$

Viscosity: 11.37 poise at  $25^\circ\text{C}</math> (Tutty, 1993b)$

Flash point:  $71.4^\circ\text{C}</math> (Tutty, 1993c)$

Storage stability: stable for 1 year at  $20^\circ\text{C}</math> and 50% relative humidity in commercial packaging (Young, 1992; Riggs, 1993b)  
 1.1% loss after storage for 14 days at  $55^\circ\text{C}</math> in water-saturated air (Riggs, 1993a)$$

Stability in the presence of 315L stainless steel and Hastalloy C-276: 1.4% loss and 1.5% loss respectively, after 16 weeks at  $20 \pm 0.5^\circ\text{C}$  (Riggs, 1993c)

Stability in the presence of simulated sunlight: 3.2% loss after continuous exposure to sunlight (long-wave UV/fluorescent lamp) for 7 days at  $20^\circ\text{C}$  (Riggs, 1993d)

Hydrolysis: rate increased with pH, with half-lives 2–3 days at pH 9, 48–78 days at pH 7 and 120–720 days at pH 5 (Nowakowski, 1987a).

### **Formulations**

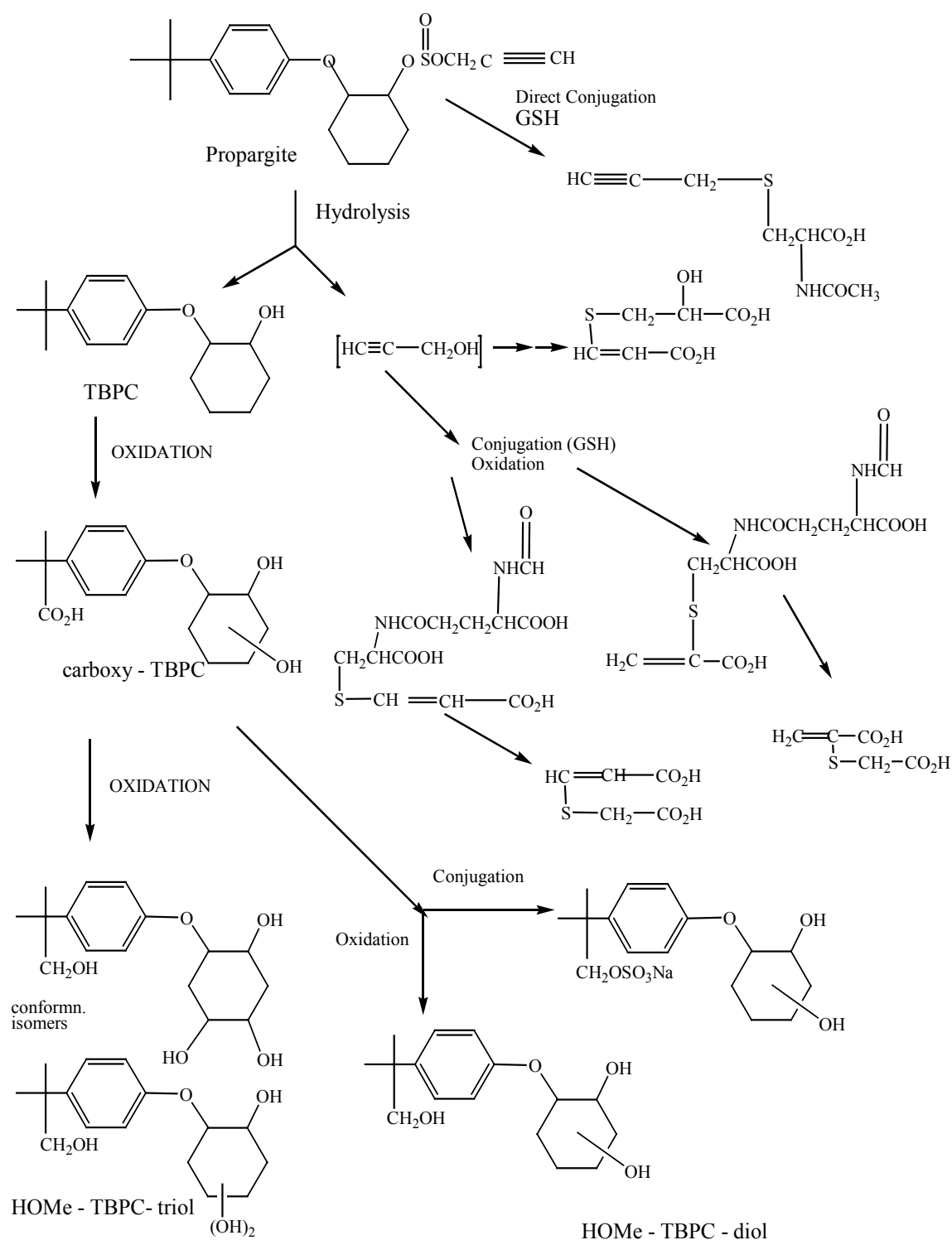
Propargite is formulated as a 4% dust (Omite 4D), a 30% wettable powder (Omite 30W) and emulsifiable concentrates containing 23, 57, 68 or 75% ai (OmiteEW, Omite57E, Omite 6E/Comite II and Comite).

## **METABOLISM AND ENVIRONMENTAL FATE**

### **Animal metabolism**

Metabolism studies on mice and rats were previously evaluated by the WHO Core Assessment Group at the 1999 Meeting (FAO/WHO, 2000c). Studies submitted to the FAO Panel are identical to those reviewed in 1999. Figure 1 shows the metabolic pathways of propargite in rats.

Figure 1. Metabolic pathways of propargite in rats.



**Note:** The compound labelled “carboxy-TBPC” should be labelled “carboxy-TBPC-diol”. Below this compound the words “OXIDATION” and “Oxidation” should be deleted. [It was not possible to edit the Figure.]

**Goats.** In a study in the USA in 1988 two Alpine dairy goats (approximately 36 kg each) were acclimatized for 14 days, then one animal was given oral doses of [ $^{14}\text{C}$ -phenyl]propargite at 19 mg/kg bw/day for 3 consecutive days, equivalent to 504, 868 and 2340 ppm in the feed on the 3 days, during

which time urine, faeces and milk were collected am and pm and stored at  $-20^{\circ}\text{C}$ . Feed consumption by the treated goat decreased by about 80% during the treatment period, but milk production remained constant both during acclimatization and treatment. Both goats were killed 8 h after the last dose and liver, kidney, muscle, fat and bile samples stored frozen until analysis. The highest internal concentration of  $^{14}\text{C}$  was found in bile (161 mg/kg as propargite, 0.29% of the administered dose), followed by liver (12 mg/kg, 0.59%), kidney (4.8 mg/kg, 0.04%), fat (1.8 mg/kg, 0.05%) and muscle (0.63 mg/kg, 0.03%), and 16% was found in the urine, 14% in the faeces and 0.086% in the milk. About 33% of the administered dose was accounted for (Byrd, 1988a).

Milk samples were extracted with acetonitrile, which recovered about 95% of the total radioactive residue (TRR), and purified by solid-phase extraction. Sequential extraction with chloroform and methanol/water accounted for between 85% from liver and 100% from muscle of the TRR. Unextracted residues in milk and tissues accounted for 0-8% of the TRR. The pellet from the liver extraction (8% of the TRR) was treated with protease, which released the radioactivity as 2-[4-(2-hydroxy-1,1-dimethylethyl)phenoxy]cyclohexane-1,x-diol (HOMe-TBPC-diol). Extracts were analysed by HPLC with radioactive and UV detectors in series. Identifications were by co-chromatography with compounds uniformly labelled with  $^{14}\text{C}$  in the phenyl ring and usually confirmed by MS (EI and CI), revealing hydrolytic loss of the sulfite and oxidation of the cyclohexyl and *tert*-butyl groups resulting in a number of polar metabolites, including 2-[4-(2,x-dihydroxycyclohexyloxy)phenyl]-2,2-dimethylacetic acid (carboxy-TBPC-diol), HoMe-TBPC-diol, 2-(4-*tert*-butylphenoxy)cyclohexane-1,x-diol (TPBC-diol), 1-[4-(2-hydroxycyclohexyloxy)phenyl]-2,2-dimethylacetic acid (carboxy-TBPC) and 2-(4-*tert*-butylphenoxy)cyclohexanol (*tert*-butylphenoxy)cyclohexanol, TBPC) in the milk and tissues. Small quantities of unchanged propargite were found in the milk, fat and liver, and bis(2-[4-*tert*-butylphenoxy]cyclohexyl) sulfite (BGES) was found in liver. The metabolites identified in the milk and tissues are shown in Table 1 (Banijamali, 1989a).

Table 1.  $^{14}\text{C}$  residues in goats dosed with [ $^{14}\text{C}$ ]propargite (Banijamali, 1989a).

Compound	Liver		Kidney		Muscle		Fat		Milk	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg <sup>6</sup>
Carboxy-TBPC-diol <sup>1</sup>			19							
TBPC-diol <sup>2</sup>					20	0.13			11	
Propargite	7.4	0.89					100	1.8	29	
BGES <sup>3</sup>	7.4	0.89								
HOMe-TBPC-diol <sup>4</sup>	56	6.7	64	3.1	80	0.50			41	
TBPC <sup>5</sup>	7.6	0.91							13	
Unextracted	8.0 <sup>7</sup>	0.96	2.0	0.10	2.0		0		0	
Total	86	-	85	-	102	-	100	-	94	

mg/kg as propargite

<sup>1</sup> 2-[4-(2,x-dihydroxycyclohexyloxy)phenyl]-2,2-dimethylacetic acid

<sup>2</sup> 2-(4-*tert*-butylphenoxy)cyclohexane-1,x-diol

<sup>3</sup> bis(2-[4-(*tert*-butyl)phenoxy]cyclohexyl) sulfite

<sup>4</sup> 2-[4-(2-hydroxy-1,1-dimethylethyl)phenoxy]cyclohexane-1,x-diol

<sup>5</sup> 2-(4-*tert*-butylphenoxy)cyclohexanol

<sup>6</sup> Insufficient data for calculation.

<sup>7</sup> Protease treatment released (100%) HOMe-TBPC-diol.

In a second study in 1996 two lactating goats were given oral doses of [ $^{14}\text{C}$ -phenyl]propargite in capsules at 65 or 325 mg/kg bw per day for 3 days, equivalent to 85 ppm and 460 ppm in the feed, and then slaughtered (Banijamali and Lau, 1996). Milk was collected twice daily and milk and tissues were stored frozen. Of the administered doses, 35% was recovered from the urine and 1% from the tissues at both levels, and 35% and 32% from the faeces, and 0.09% and 0.07% from the milk at the low and high doses respectively.

Tissues were extracted sequentially with acetonitrile, methanol, and methanol/water. Fat was first extracted with hexane. Milk, after treatment with acetonitrile to precipitate the proteins, was extracted sequentially with acetonitrile and methanol. The various post-extraction solids (PES) were treated with an equal mixture of proteases types I and XIV. Milk extracts contained 92-99% of the TRR, and enzyme hydrolysis of the milk PES released an additional 2-5%. Extraction of the tissues released about 99% of the TRR from the low-dose goat, with enzyme hydrolyses of the PES releasing an additional 0.6-1.7%, and 94-96% from the high-dose goat, with enzyme hydrolyses releasing an additional 1.4-3.6%. Extracts were purified by solid-phase extraction before HPLC analysis. LC-MS and LC-MS-MS were used to confirm the identifications of metabolites. The liver contained 14 metabolites, kidney 12, muscle 9, milk 7 and fat 6. Half of those in the liver were glucuronide or sulfate conjugates. Identifications and characterizations are shown in Tables 2 and 3.

Table 2. Propargite and its metabolites in the tissues and milk of goat dosed with 65 mg/kg bw/day (Banijamali and Lau, 1996).

Compound	Liver (4.1 mg/kg)		Kidney (2.0 mg/kg)		Muscle (0.17 mg/kg)		Fat (0.36 mg/kg)		Milk (0.15 mg/kg day 3)	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Carboxy-TBPC-diol <sup>1</sup>	10	0.42	23	0.47	13	0.023			4.5	0.0067
TBPC-diol <sup>2</sup>	7.8	0.32	1.8	0.037	15 <sup>14</sup>	0.026	7.7	0.028	1.8	0.0028
Propargite	0.51	0.021	0.33	0.007	5.3	0.009	66	0.024	48	0.072
BGES <sup>3</sup>							1.5	0.005		
HOMe-TBPC-diol <sup>4</sup>	20 <sup>7</sup>	0.81	27 <sup>11</sup>	0.56	13		2.8	0.010	22	0.034
TBPC <sup>5</sup>	10 <sup>10</sup>	0.41	5.7 <sup>12</sup>	0.076	2.5	0.004	8.4	0.030	2.0	0.0030
Carboxy-TBPC-triol <sup>6</sup>	7.5	0.31								
Carboxy-TBPC <sup>8</sup> , carboxy-TBPC glucuronide, HOMe- TBPC glucuronide (co-elution)	24	1.0	23	0.47	50 <sup>13</sup>	0.086				
HOMe-TBPC sulfate <sup>9</sup> , TBPC-diol glucuronide (co-elution)	15	0.61	5.9	0.12						
HOMe-TBPC <sup>15</sup>							1.8	0.006		
Total unknowns (HPLC)	7.5	0.31	3.6	0.074	8.1	0.014	5.7	0.020	20	0.031
Enzyme hydrolysis	0.65	0.027	0.96	0.020	1.7	0.003	2.4 <sup>16</sup>	0.009	2.6	0.0039
Unextracted	0.33	0.014	0.26	0.005	2.4	0.004	1.6	0.006	1.4	0.0021
Total	103		92		98		98		102	

mg/kg as propargite

<sup>1</sup> 2-[4-(2,x-dihydroxycyclohexyloxy)phenyl]-2,2-dimethylacetic acid

<sup>2</sup> 2-(4-*tert*-butylphenoxy)cyclohexane-1,x-diol

<sup>3</sup> bis(2-[4-(*tert*-butyl)phenoxy]cyclohexyl) sulfite

<sup>4</sup> 2-[4-(2-hydroxy-1,1-dimethylethyl)phenoxy]cyclohexane-1,x-diol

<sup>5</sup> 2-(4-*tert*-butylphenoxy)cyclohexanol

<sup>6</sup> 2-[4-(2,x,y-trihydroxycyclohexyloxy)phenyl]-2,2-dimethyl acetic acid

<sup>7</sup> 12% glucuronide and 7.9% sulfate

<sup>8</sup> 2-[4-(cyclohexyloxy)phenyl]-2,2-dimethyl acetic acid

<sup>9</sup> 2-[4-(2-hydroxy-1,1-dimethylethyl)phenoxy]cyclohexanol sulfate

<sup>10</sup> Includes 4.3% glucuronide, 5.8% TBPC

<sup>11</sup> 16% glucuronide and 11% sulfate

<sup>12</sup> 2.0%TBPC and 3.7% glucuronide

<sup>13</sup> Also includes HOMe-TBPC-diol

<sup>14</sup> 7% glucuronide + HOMe-TBPC and 7.8% diol

<sup>15</sup> 2-[4-(2-hydroxy-1,1-dimethylethyl)phenoxy]cyclohexanol

<sup>16</sup> Residual activity in the hexane

Table 3. Propargite and its metabolites in the tissues and milk of goat dosed with 325 mg/kg bw/day (Banijamali and Lau, 1996).

Compound	Liver (19 mg/kg)		Kidney (6.9 mg/kg)		Muscle (0.56 mg/kg)		Fat (1.4 mg/kg)		Milk (0.45 mg/kg day 3)	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/ kg	% of TRR	mg/ kg
Carboxy-TBPC-diol <sup>1</sup>	5.4	1.0	11	0.73	7.7	0.043			3.2	0.014
TBPC-diol <sup>2</sup>	6.4 <sup>10</sup>	1.2	2.3	0.16	26 <sup>16</sup>	0.14	7.3	0.10	6.3	0.028
Propargite	1.3	0.26	1.0	0.070			55	0.77	43	0.19
BGES <sup>3</sup>					4.6	0.026	2.9	0.041		
HOMe-TBPC-diol <sup>4</sup>	18 <sup>11</sup>	3.4	23 <sup>13</sup>	1.6	<sup>15</sup>		5.1	0.072	21	0.092
TBPC <sup>5</sup>	13 <sup>12</sup>	2.5	8.4 <sup>14</sup>	0.58	2.8	0.015	8.1	0.11	3.0	0.013
Carboxy-TBPC-triol <sup>8</sup>	3.5	0.66								
Carboxy-TBPC <sup>9</sup> , carboxy-TBPC glucuronide, HOMe-TBPC glucuronide (co-elution)	26	4.9	29	2.0	43 <sup>15</sup>	0.24				
HOMe-TBPC <sup>6</sup>	19	3.7					1.6	0.022	7.6 <sup>7</sup>	0.034
HOMe-TBPC sulfate, TBPC-diol glucuronide (co-elution)			7.6	0.52						
Total Unknowns (HPLC)	2.8	0.53	5.4	0.37	4.7	0.026	9.8	0.14	4.8	0.021
Enzyme hydrolysis	3.4	0.65	1.4	0.093	3.6	0.020	1.5 <sup>17</sup>	0.021	2.4	0.011
Unextracted	0.84	0.16	0.47	0.032	1.2	0.007	0.79	0.011	0.17	0.0008
Total	99		90		94		92		91	

<sup>1</sup> 2-[4-(2,x-dihydroxycyclohexyloxy)phenyl]-2,2-dimethylacetic acid

<sup>2</sup> 2-(4-*tert*-butylphenoxy)cyclohexane-1,x-diol

<sup>3</sup> bis(2-[4-(*tert*-butyl)phenoxy]cyclohexyl) sulfite

<sup>4</sup> 2-[4-(2-hydroxy-1,1-dimethylethyl)phenoxy]cyclohexane-1,x-diol

<sup>5</sup> 2-(4-*tert*-butylphenoxy)cyclohexanol

<sup>6</sup> 2-[4-(2-hydroxy-1,1-dimethylethyl)phenoxy]cyclohexanol

<sup>7</sup> Including 4% HOMe-TBPC-sulfate

<sup>8</sup> 2-[4-(2,x,y-trihydroxycyclohexyloxy)phenyl]-2,2-dimethylacetic acid

<sup>9</sup> 2-[4-(cyclohexyloxy)phenyl]-2,2-dimethylacetic acid

<sup>10</sup> Mixture of 2 diol isomers.

<sup>11</sup> 10% glucuronide and 7.3% sulfate.

<sup>12</sup> 7.6% TBPC and 5.4% glucuronide.

<sup>13</sup> 10% glucuronide and 12% sulfate.

<sup>14</sup> 1.8% TBPC and 6.6% glucuronide.

<sup>15</sup> Includes HOMe-TBPC-diol.

<sup>16</sup> 18% TBPC-diol and 8.1% TBPC-diol-glucuronide + HOMe-TBPC.

<sup>17</sup> Residual activity in the hexane.

**Hens.** Five white Leghorn hens (1.2-1.9 kg each) were dosed orally once a day for four days with 1.05 mg [<sup>14</sup>C-phenyl]propargite and 42 mg unlabelled propargite, equivalent to about 330 ppm in the diet based on total measured feed consumption. Eggs were collected daily and pooled for analysis. The hens were killed 8 hours after the last dose. About 65% of the total dose was found in the faeces and 1.4% in the tissues, with 0.59% in the liver, constituting in all about 67% of the administered dose. Average propargite concentrations (mg/kg) in the tissues were liver 31 (range 19-47); kidney, 18 (12-26); fat, 13 (6.6-26); and muscle 4.1 (2.9-5.0). The concentration in egg whites reached a plateau on day 2 at 1.9 mg/kg, but did not reach a plateau in the yolks and was 4.3 mg/kg on the last day (Byrd, 1988b).

Excreta, liver, kidney and muscle were extracted with chloroform and methanol/water with 88-106% of the radioactivity extracted, and egg whites and yolks and fat with acetonitrile/methanol

with 90-98% extracted. Analysis was by HPLC, with radiolabelled standards for comparison. Identified compounds are shown in Table 4 (Banijamali, 1989b).

Table 4.  $^{14}\text{C}$  residues in samples from hens dosed with [ $^{14}\text{C}$ ]propargite (Banijamali, 1989b).

Compound	Sample												
	Excreta	Liver (31 mg/kg)		Kidney (18 mg/kg)		Muscle (4.1 mg/kg)		Fat (13 mg/kg)		Egg white (1.9 mg/kg)		Egg yolk (4.3 mg/kg)	
	% re- covered	% of TRR	mg/k g	% of TRR	mg/k g	% of TRR	mg/k g	% of TRR	mg/ kg	% of TRR	mg/ kg	% of TRR	mg/ kg
Propargite	6.5							43	5.6			13	0.56
TBPC	1.5	6.0	1.9	10	1.8	4.1	0.17	18	2.3			7.9	0.34
HOMe-TBPC	2.8	14	4.3	7.5	1.4			6.8	0.88				
HOMe-TBPC- diol	21	39	12	41	7.4	67	2.7			59	1.1	41	1.8
Carboxy-TBPC- diol	33	6.2	1.9	13	2.3								
HOMe-TBPC- triol <sup>1</sup>	27									27	0.51		
TBPC-diol		19	5.9	12	2.2	20	0.82	14	0.27			18	0.77
HOMe-TBPC- triol													
Total		84 <sup>2</sup>		84		91		82		86		80	

<sup>1</sup>2-[4-(2-hydroxy-1,1-dimethylethyl)phenoxy]cyclohexane-1,x,y-triol

<sup>2</sup>An additional 6% of TRR was released by Pronase E and identified (see below).

Liver pellets remaining after the solvent extractions were digested with Pronase E to reveal by HPLC two peaks corresponding to two peaks in the solvent extracts. Subsequent analysis of released radioactivity indicated the metabolites HOMe-TBPC-diol, 1.3% of the TRR, and TBPC, 4.8%. Residues in samples stored frozen (-20°C) for 4 months were stable: neither the distribution of radiolabel among the extracts nor the HPLC chromatograms of those extracts from samples prepared and analysed within one month of necropsy changed appreciably when analysed four months after necropsy (Banijamali, 1991).

In a supplementary study two white Leghorn hens were each dosed orally once daily for three days with 46.5 mg propargite, including 1.25 mg [ $^{14}\text{C}$ -phenyl]propargite. About 90% of the total dose was recovered with 82% being excreted, and a further 7.8% in tissues and 0.1% in egg whites and yolks. In the pooled tissue samples liver contained 20 mg/kg propargite equivalents, kidney 14 mg/kg, fat 9.2 mg/kg and muscle 2.8 mg/kg. The residue in egg white reached a plateau of 1.2 mg/kg on day two, and in the yolks continued to increase, reaching 3.1 mg/kg on day three (Banijamali, 1991).

### Plant metabolism

Maize grown outdoors was sprayed with 2.8 kg/ha of a 5.2% [ $^{14}\text{C}$ -phenyl]propargite/94.8% unlabelled propargite formulation in water (Lengen, 1989a). Six weeks later plants were harvested and analysed for total  $^{14}\text{C}$  residues by combustion after drying. Husks, stalks and tassels were extracted with water/methanol/acetone and the  $^{14}\text{C}$  determined by LSC (Table 5).

Table 5. Distribution of  $^{14}\text{C}$  from [ $^{14}\text{C}$ ]propargite in field-grown maize (Lengen, 1989a).

Sample	% of total activity recovered from whole plant	mg/kg propargite equivalents (dry weight basis)
husk fraction	95.4	219
silks	4.4	205
kernels	0.07	0.09
cobs	0.05	0.29



Extraction with 20% aqueous methanol released 75% of the radioactivity from the husks and 91% from the silks. The extracts were analysed by reverse-phase HPLC, revealing 4 metabolites, which were analysed by MS (EI) (Table 6).

Table 6. Compounds identified [ $^{14}\text{C}$ ]propargite in field-grown maize treated with [ $^{14}\text{C}$ ]propargite at 2.8 kg ai/ha.

Compound	Husk		Silk	
	% of TRR	mg/kg	% of TRR	mg/kg
propargite	59	130	69	140
TBPC	8.5	19	7.2	15
HOMe-TBPC	2.4	5.2	4.8	9.8
unidentified			2.2	4.5

Extracted husk samples were subjected to acid and base hydrolysis (1 N, 1 h, 100°C) for analysis by LSC and HPLC. Acid released 13% of the TRR, and base all of the radioactivity in the extracted husk (25% of the original TRR). Each procedure produced a main metabolite but this was not investigated further.

In a glasshouse trial (Lengen, 1982a,b) Bush Blue Lake 274 green beans were sprayed with [ $^{14}\text{C}$ -phenyl]propargite mixed with a propargite formulation at a rate equivalent to 4.2 kg ai/ha, and harvested 7 days later. In a second experiment some pods were painted with 9.5 mg of a [ $^{14}\text{C}$ -phenyl]propargite formulation and harvested 7 days later, and in a third pieces of immature pods removed from plants and sterilized with ethanol were placed on Miller's modified media and callus tissue initiated. [ $^{14}\text{C}$ -phenyl]propargite (0.7 mg) was injected into the 2-week old tissue which was harvested 7 days later. The various samples were analysed by combustion and TLC of methanol extracts. Methanol extracted >95% of the radioactivity in all cases. 8.7% of the applied radioactivity (equivalent to 22 mg/kg propargite) was extracted from sprayed plant pods, 28% from painted pods, 87% from bean callus and 8.6% from agar media. MS was used for identification. No data were provided of the calculation of recoveries of radioactivity and quantification of metabolites.

Partially differentiated maize root callus (Golden Cross Bantam) was grown on Miller's modified medium and injected with 0.6 mg [ $^{14}\text{C}$ -phenyl]propargite. Samples were collected after 7 days for analysis by combustion and TLC. Recovered radioactivity was 99 or 104%. No details were provided.

Table 7. Distribution of  $^{14}\text{C}$  in propargite-treated beans and maize harvested 7 days after treatment (Lengen, 1982a,b).

Compound	$^{14}\text{C}$ , % of TRR			
	Sprayed pods	Painted pods	Bean callus	Maize callus
propargite	80	88	76.0	96.0
TBPC	1.0	0.8	2.4	<1.0
Polar (minimum of 6 compounds)	9.9	6.2	12.2	1.1
Other extractable products (4)	4.7		6.7	1.8
Post-extraction solid	4.1	3.5	2.8	<1.0

In another experiment Banijamali (1995) sprayed [ $^{14}\text{C}$ -phenyl]propargite and unlabelled propargite as a 73.8% mixture onto field-grown maize 1.2-1.5 m tall at a rate of 2.8 or 11.2 kg ai/ha. Samples were collected after three weeks (forage) and at normal harvest after 6 weeks and separated into ears, stover, husks, silks, cobs and kernels for extraction with acetonitrile, methanol, and methanol/water with 0.1-2% 1 N HCl. Total radioactivity in the various samples, as determined by

combustion and liquid scintillation counting, and radioactivities in the combined solvent extracts and hydrolysates are shown in Table 8.

Table 8. Radioactivity in maize treated with [ $^{14}\text{C}$ ]propargite (Banijamali, 1995).

Sample	% of TRR			
	Solvent	Enzyme hydrolysis <sup>1</sup>	Chemical hydrolysis <sup>2</sup>	Total
<i>2.8 kg ai/ha</i>				
Forage (8.0 mg/kg)	103	5.6	3.8	112
Stover (12 mg/kg)	101	4.3	6.9	112
Husks (4.8 mg/kg)	70 <sup>3</sup>	3.6	7.9	82
Silks (150 mg/kg)	96	1.0	5.3	102
<i>11.2 g ai/ha</i>				
Forage (39 mg/kg)	87	6.0		93
Stover (79 mg/kg)	82	4.6		86
Husks (16 mg/kg)	95	6.4		102
Cobs (0.22 mg/kg)	56	2.7	35	94
Kernels (0.17 mg/kg)	47	24	33	104

mg/kg as propargite

<sup>1</sup> With mixture of Pectinex Ultra SP-L, cellulase, hemicellulase, amylase and  $\beta$ -glucosidase of solids from solvent extraction; 37°C for 3-5 days.

<sup>2</sup> Acid and base hydrolysis (both 1 N at room temperature for 4 hours and/or 6N at reflux for 4 hours) of solids from enzyme hydrolysis.

<sup>3</sup> Modified extraction released 94% of TRR: chloroform 29%; methanol/water 61%; acetone 3.7%.

Identifications (Table 9) were by HPLC with UV and radioactivity detectors, TLC, GC-MS (capillary column, EI) and MS (direct exposure probe).

Table 9.  $^{14}\text{C}$  compounds in maize treated with [ $^{14}\text{C}$ ]propargite (Banijamali, 1995).

Compound	$^{14}\text{C}$ , % of TRR and (mg/kg as propargite)				
	Forage (2.8 kg ai/ha) solvent extract	Forage (2.8 kg ai/ha) enzyme hydrolysate	Stover (2.8 kg ai/ha) solvent extract	Stover (2.8 kg ai/ha) enzyme hydrolysate	
HOMe-TBPC-triol	5.7 (0.46)	1.7 (0.13)	7.7 (0.91)		
HOMe-TBPC-diol	7.8 (0.62)	1.3 (0.10)	11 (1.3)		1.0 (0.12)
HOMe-TBPC-diol isomer	17 (1.3)	0.96 (0.08)	15 (1.8)		0.14 (0.02)
HOMe-TBPC	3.4 (0.27)		2.9 (0.34)		1.6 (0.18)
TBPC-diol	1.4 (0.11)	0.68 (0.05)	6.5 (0.77)		0.13 (0.02)
TBPC	4.6 (0.37)		6.5 (0.77)		0.23 (0.03)
Propargite	40 (3.2)		26 (3.1)		0.40 (0.05)
Bis-TBPC	3.0 (0.24)		1.1 (0.13)		
Total	88		80		
	Kernels (11.2 kg ai/ha) combined extracts	Husks (2.8 kg ai/ha) solvent extract	Silks (2.8 kg ai/ha) solvent extract	Silks (2.8 kg ai/ha) enzyme hydrolysate	Cobs (11.2 kg ai/ha) combined extracts plus enzyme hydrolysate
HOMe-TBPC-triol		7.6 (0.36)			14 (0.03)
HOMe-TBPC-diol	45 (0.07)	11 (0.52)	14 (20)	0.2 (0.31)	6.7 (0.01)
HOMe-TBPC-diol isomer		13 (0.61)	9.4 (14)	0.1 (0.15)	11 (0.02)
HOMe-TBPC	7.5 (0.01)	6.1 (0.29)	18 (26)	0.06 (0.10)	3.0 (<0.01)

TBPC-diol		2.6 (0.13)		0.2 (0.32)	3.5 (<0.01)
TBPC	6.6 (0.01)	2.3 (0.11)	2.8 (4.2)	0.1 (0.16)	1.7 (<0.01)
Propargite	11 (0.02)	13 (0.63)	33(48)	0.06 (0.08)	7.0 (0.02)
Bis-TBPC		3.6 (0.17)	5.3 (7.8)		
unknown	5.4 (<0.01)			0.08 (0.11)	3.4 (<0.01)
Total	76	59	83		50

A forage extract was re-analysed after refrigerated storage for 1 year. The metabolic profile appeared unchanged and this was interpreted to indicate stability of the metabolites in the extracts.

A W formulation of unlabelled propargite (0.624 g) and [<sup>14</sup>C]propargite (0.0205 g) dissolved in acetonitrile and water was painted at a rate equivalent to 8.6 kg ai/ha onto apple fruit and leaves (Red Delicious), which were collected 23 days later and washed with acetone and water/acetone sequentially (Table 10, Lengen, 1989b).

Table 10. <sup>14</sup>C in apples and apple tree leaves treated with [<sup>14</sup>C]propargite (Lengen, 1989b).

Sample	% of TRR	mg/kg propargite equivalents
Leaf washes		
acetone	36	
acetone/water	13	
Washed leaves	52	440
Apple washes		
acetone	16	
acetone/water	15	
Apple peel after washing	68	110 ± 13
Apple fruit (less peel) after washing	1.1	0.35 ± 0.03
Apple (whole)		
Unwashed		27 <sup>1</sup>
After washing		19 <sup>1</sup>

<sup>1</sup> Calculated from peel and pulp.

At least 99% of the radioactivity was extracted with methanol from the washed peel and pulp, as shown by combustion analysis of the residual solids. The extracts were cleaned up by solid phase extraction. Leaves were also extracted with chloroform/methanol/water (1/2/0.8). The extracts were partitioned with chloroform and the fractions cleaned up on sulfonic acid solid-phase extraction columns. The washes and extracts were analysed by HPLC with UV and radioactivity detectors. Co-chromatography with standards was used to identify residues (Table 11).

Table 11. Compounds in apples treated with [<sup>14</sup>C]propargite (Lengen, 1989b).

Sample	<sup>14</sup> C, % of TRR and mg/kg (as propargite)				
	Propargite	TBPC	HOMe-TBPC	HOMe-TBPC-diol	Metabolite V <sup>1</sup>
Leaf wash	86	3.1		trace	
after washing	62 (270)	26 (113)		2.7 (12)	4.4 (19)
Apple wash	70-92				
peel	89 (98)	4.2 (4.6)			
fruit	31 (0.11)	14 (0.05)	28 (0.10)	14 (0.05)	4.4 (0.02)

<sup>1</sup> Polar unknown.

Lengen (1989c) sprayed the foliage of outdoor-grown potatoes with formulated [<sup>14</sup>C-phenyl]propargite at a rate equivalent to 1.91 kg ai/ha. Potatoes were harvested 3 weeks later and separated into vines, peels and tubers. The total radioactivity was determined by combustion and LSC.

Total radioactivity in the peels (fresh weight), tubers (fresh weight) and vines (dry weight) was 0.012, 0.004 and 270 mg/kg propargite equivalents respectively. Because of the low levels of radioactivity, no further analysis was carried out on peels or tubers. Aqueous methanol extraction of vines released 84% radioactivity. The extract was separated into a green chlorophyll precipitate and a yellow-brown filtrate which were analysed by HPLC. Identifications were made by co-elution with standards and for some metabolites by MS (CI) (Table 12). About 82% of the TRR in the vines was accounted for.

Table 12.  $^{14}\text{C}$  residues in potato vines treated with [ $^{14}\text{C}$ ]propargite (% of the TRR) (Lengen, 1989c).

Sample	Propargite	TBPC	HOMe-TBPC	D <sup>1</sup>	HOMe-TBPC-diol	HOMe-TBPC-triol	G <sup>1</sup>
Green chlorophyll	89	3.4					
Yellow-brown filtrate	14	7.2	12	22	23	8.5	12
Total mg/kg propargite equivalents in the vine and (% of TRR)	71 (26%)	15 (5.5)	22 (8.0)	39 (14)	40 (14)	15 (5.5)	22 (8.0)

<sup>1</sup> Unknown.

The compounds identified in animals and plants are summarized in Table 13 and the metabolic pathways are shown in Figures 2 and 3.

Table 13. Occurrence of propargite and metabolites in plants and animals.

Compound	Rats/mice (Banijamali and Nag, 1991)	Goat (Banijamali, 1989)	Chicken (Byrd, 1988b; Banijamali, 1989a)	Maize (Lengen, 1989a)	Maize (Banijamali, 1995)	Beans (Lengen, 1982 a,b)	Potato vines (Lengen, 1989c)	Apple (Lengen, 1989b)
Propargite		Milk, liver, fat	Fat, egg yolk	Husk, silk	Forage, stover, kernels, husks, silk, cobs	x	x	Wash, peel, fruit
TBPC (propargite glycol ether)	x	Milk, liver	Liver, kidney	Husk, silk	Forage, stover, kernels, husks, silk, cobs	x	x	Peel, fruit
HOMe-TBPC (OMT-B; propargite hydroxymethyl glycol ether)	pi		Liver, kidney, muscle, fat, egg yolk	Husk, silk	Forage, stover, kernels, husks, silk, cobs		x	
HOMe-TBPC-diol	x	Milk, liver, muscle, kidney	Liver, kidney, muscle, egg white and yolk		Forage, stover, kernels, husks, silk, cobs		x	fruit
Carboxy-TBPC-diol (Omite OGE acid)	x	kidney	Liver, kidney					
TBPC-diol (propargite hydroxy glycol ether)		Milk, muscle	Liver, kidney, muscle, fat, egg yolk		Forage, stover, husks, silk, cobs			fruit
HOMe-TBPC-triol	x		Egg white		Forage, stover, husks, cobs		x	
BGES		liver						
Bis-TBPC					Forage, stover, husks, silk			

x: detected compound

pi: postulated intermediate

\* impurity in radiolabelled material formed by dimerisation of TBPC

Figure 2. Metabolic pathways of propargite in poultry and ruminants.

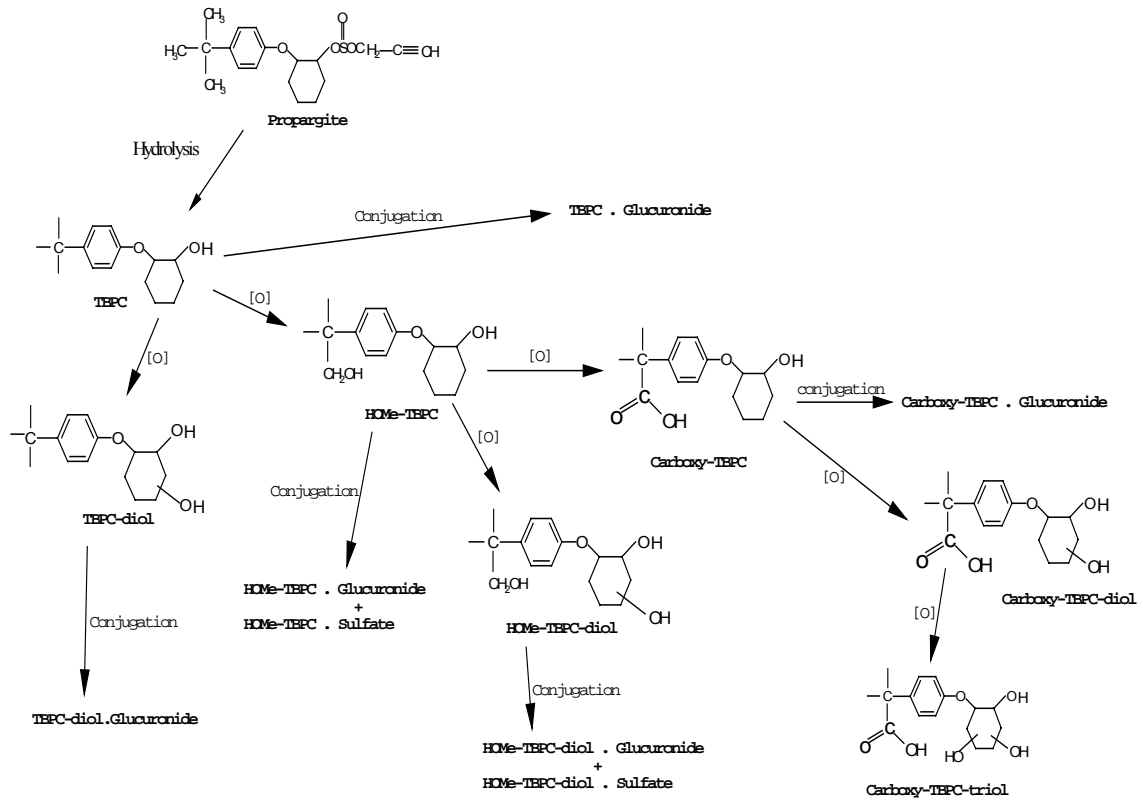
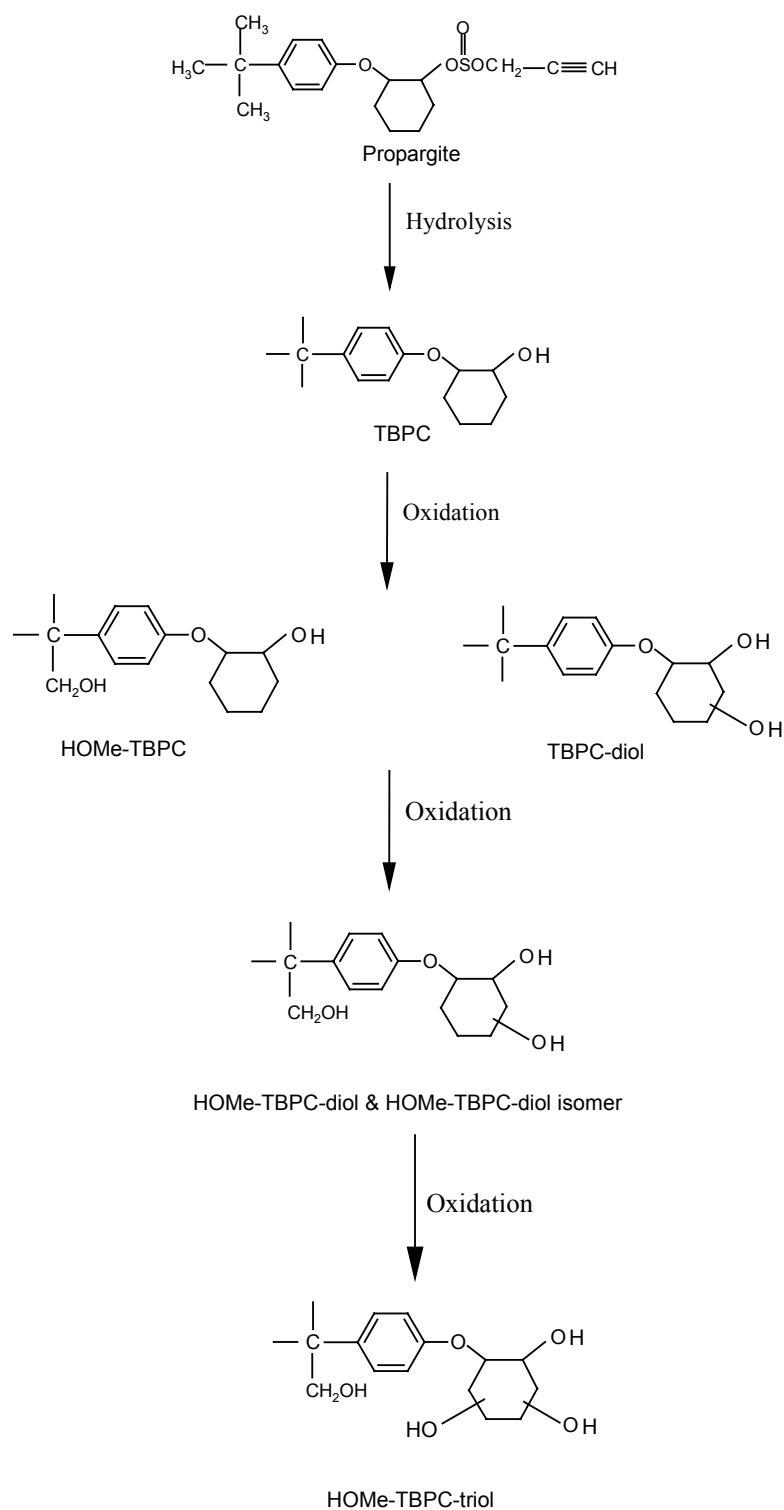


Figure 3. Metabolic pathways of propargite in plants.



### Environmental fate in soil

**Aerobic degradation.** Sandy clay loam soil treated with 4.9 mg/kg [ $^{14}\text{C}$ -phenyl]propargite was incubated in the dark at 25°C. The acetone-extractable radioactivity decreased from 94% of that applied on day 2 (85% propargite, 5% BEGS) to 31% (24% propargite, 4% BEGS) on day 90. BEGS,

or bis(2-[4-(*tert*-butyl)dimethylethylphenoxy]-cyclohexyl) sulfite, was an impurity in the starting radiolabelled mixture. The unextractable residue increased from 2% on day 2 to 30% on day 90; it was not further investigated.  $^{14}\text{CO}_2$  accounted for 31% of the applied radioactivity at day 90 but. The half-life was calculated to be 40 days by linear regression (Dzialo, 1987).

In another trial samples of a standard German loamy sand (Speyer 2.2) treated with 2.9 mg/kg [ $^{14}\text{C}$ -phenyl]propargite (equivalent to 2.4 kg ai/ha assuming a core depth of 5 cm and a bulk density of soil of 1.5 g/cm<sup>3</sup>) were incubated for 100 days at 22°C. Extractable residues (80% methanol) decreased from 107% of the applied radioactivity to 30% on day 100, unextractable increased from 0.4% to 26%, and  $^{14}\text{CO}_2$  accounted for 42%. The rates of degradation are shown in Table 14.

Table 14. Aerobic degradation of [ $^{14}\text{C}$ ]propargite in Speyer 2.2 loamy sand soil (Galiccia, 1990).

Assumption	Half-life	DT <sub>90</sub>	Rate constant
First-order kinetics	53	180	0.013 1/day
1.5 order kinetics	45	230	0.0052 (kg/mg) <sup>0.5</sup> 1/day

As well as propargite, which constituted 30% of the day 100 radioactivity, two unknown metabolites detected at a maximum on day 32 (about 1% each of the applied radioactivity) had each decreased to about 0.6% by day 64.

In a third trial by Comezoglu and Ly (1995) sandy loam soils treated with [ $^{14}\text{C}$ -phenyl]propargite (5.8 mg/kg) were incubated at 25°C for 1 year. On day 3 about 90% of the radioactivity was extracted into ethyl acetate, but this gradually decreased to 25% after 1 year. The radioactivity in the post-extraction solid after extraction with ethyl acetate, acetonitrile, chloroform and water) increased from 3% on day 3 to 33% on day 365, when up to 32% of the applied radioactivity was detected as  $^{14}\text{CO}_2$ . Propargite constituted 24% of the day 365 radioactive residue, with up to 9 degradation products accounting for <7% of the residue including TBPC (1.5%, day 365), *p-tert*-butylphenol (0.3%, day 365), BGES (0.65%, day 365; absent on days 0, 3 and 7) and TBPC sulfate (3.8%). The dissipation curve was biphasic with an estimated half-life of 67 days for the first 59 days and 231 days thereafter.

Anaerobic degradation. Dzialo (1988) treated sandy clay loam with 5.07 mg/kg [ $^{14}\text{C}$ -phenyl]propargite and aged it under aerobic conditions for 27 days. The soil was then flooded with oxygen-free water to establish anaerobic conditions (day 0). After 60 days 62% of the applied radioactivity was extracted from the soil by acetonitrile, contrasting with 83% on day 0, and propargite was the main residue (37% of the applied radioactivity) although TBPC was also significant (20% of the applied radioactivity). The aqueous filtrate and unextracted material accounted for 11 and 14% respectively, of the applied radioactivity.  $^{14}\text{CO}_2$  was <3% by day 60. Small amounts of TBPC (3.4%) and *p-tert*-butylphenol (0.7%) were detected in the aqueous filtrate. The half-life was calculated by linear regression to be 64 days.

Mobility. The adsorption/desorption of [ $^{14}\text{C}$ -phenyl]propargite at 25°C on various soils was reported by Spare (1993b). Adsorption was determined by shaking mixtures of soil and [ $^{14}\text{C}$ ]propargite in 0.01M calcium acetate (0–0.585 µg/ml) for 8 hours. The initial and equilibrium concentrations of propargite were determined in the solutions. Desorption was determined by equilibrating the soil samples remaining from adsorption with 0.01 M calcium ion solution for 8 hours. The radioactivity in the final solutions and residual soils was measured. All data were evaluated using the Freundlich equation and values for  $K_{oc}$  (adsorption coefficient),  $K_d$  (adsorption constant) and  $n$  (empirical exponent) were determined. Propargite was strongly adsorbed by all the soils and may be characterized as slightly mobile to immobile

Table 15. Adsorption coefficients and constants for propargite (Spare, 1993b).

Soil	adsorption			desorption		
	$K_d$	$K_{oc}$	n	$K_d$	$K_{oc}$	n
Mississippi clay	427	23400	0.72	1976	108000	0.74
Maryland sand	318	90100	0.63	111	31500	0.84
Connecticut loamy sand	165	5290	1.03	201	6430	1.10
California-sandy loam	162	39300	0.78	557	135000	0.74
Florida sand	172	48900	0.75	53	14900	1.13
Florida sediment	113	95900	0.67	15	12900	1.09

TBPC was detected in some adsorption solutions but propargite was the main residue. TBPC was not found in the solutions after desorption.

Spare (1987b, 1993a) and Korpalski (1989) also studied the absorption/desorption of [<sup>14</sup>C-phenyl]TBPC at 24-26°C. Concentrations were determined in both the aqueous and soil fractions after equilibration. All data were evaluated using the Freundlich equation and values for  $K_{oc}$ ,  $K_d$  and n were determined. The 1987 study was with autoclaved soils, which may explain the differences between the two studies.

Table 16. Adsorption coefficients and constants for TBPC.

Ref.	Soil	adsorption			desorption		
		$K_d$	$K_{oc}$	n	$K_d$	$K_{oc}$	n
Spare, 1987b	Mississippi clay	5.1	184.6	1.04	6.9	248.7	1.04
	Maryland sand	0.4	74.8	0.97	1.3	243.5	1.06
	Mississippi loam	2.2	308.5	1.07	3.2	455.6	1.06
	California sandy loam	0.6	190.4	1.06	1.1	373.1	1.13
Korpalski, 1989	Ducar clay loam	4.7	354.4	1.02	4.1	304.9	1.05
Spare, 1993a	Mississippi clay	8.4	460	1.04	13.3	728	1.05
	Mississippi loam	0.66	187	1.09	1.9	543	1.02
	Connecticut loamy sand	6.7	215	1.12	12.2	390	1.12
	California sandy loam	1.2	284	1.13	2.5	604	0.98
	Florida sand	1.5	418	1.2	2.5	723	1.17
	Florida sediment	0.65	551	0.99	0.77	651	1.28

TBPC was not strongly adsorbed and was easily desorbed and may therefore be characterized as very mobile. TBPC was the main compound detected.

**Field dissipation.** Many studies monitoring spray drift and run-off from treated fields to bodies of water were carried out. Spray drift cards were positioned to intercept drift during applications made so that a finite amount of propargite reached the water surface. Samples of water and sediment were taken at various intervals especially after significant rainfall (Table 17).

Table 17. Propargite run-off from treated fields in the USA.

Ref.	State	Crop/ha	Rate (kg ai/ha)	Test site	Propargite residues in sediment (mg/kg)	Propargite residues in water (mg/l)	Residues of TBPC (mg/kg)
Harned <i>et al.</i> , 1989	Missouri	Maize 8	2.76 (aerial application)	0.8 ha dammed pond (treated field draining on 3 sides of pond)	<0.025-0.10 for first 12 days after application; >12 days nd (<0.025)	nd (<0.005) over 4 months	nd in water (<0.005) or sediment (<0.025) over 4 months
Harned, 1990b	Georgia	Cotton 12	0.9 1.8 1.8 aerial	1.2 ha pond (treated field sloped toward pond)	dat 0, 0.077 dat 3–da3t, <0.025	Max 0.013, da3t 0 run-off water: 0.018 after 1st run-off 0.014 after 3rd and 4th run-off	Max 0.012, water da3t 0 <0.025-0.030 sediment



Ref.	State	Crop/ha	Rate (kg ai/ha)	Test site	Propargite residues in sediment (mg/kg)	Propargite residues in water (mg/l)	Residues of TBPC (mg/kg)
Harned, 1989b	Texas	Maize 21.5	1.88 (aerial application)	2.2 ha pond (treated field draining to one side of pond)	<0.025	1 dat 0.12 >6 dat <0.0025 field run-off: 1 dat 0.269. > 9 dat <0.0025	0 dat 0.015 in water, >9 dat <0.0025 sediment, <0.0025
Harned, 1989c	Florida	Orange grove 1.9	2 x 2.8 (airblast mist sprayer)	0.17 ha pond (treated field drained to all sides of pond)	12 dat 0.025 28 da2t 0.05 42 da2t 0.042	1 dat 0.009	Water: 19 dat 0.009 1 da2t 0.006 sediment: 12 dat 0.025
Harned, 1989d	Florida	Orange grove 21.1	2 x 2.8 (airblast mist blower)	1 ha pond (treated field drained to all sides of pond)	<0.025	<0.0025/nd Drainage water: 0.037 after 1st run-off ; 0.045 after 2nd run-off.	Water: 1 dat 0.005 Sediment: <0.0025 Drainage water: 0.073 after 1st run-off 0.08 1 da2t and day of 10th run-off
Harned, 1990a	Georgia	Cotton 4.7	3 X 1.8	0.20 ha pond (field drains on two sides into)	Max 0.12 dat 3	Max 0.058 dat 0	Water max :0.063 dat 0 Sediment: max 0.072 3 days after 3rd run-off (da3t 60+)

dat: days after treatment

da2t: days after 2nd treatment

da3t: days after 3rd treatment

nd: not detectable

Harned *et al.* (1994) modelled concentrations of propargite in the run-off from cotton fields and citrus groves using the US EPA models PRZM2 and EXAMS. The estimated environmental concentrations (EEC) ranged from 0.3 to 10 ppb. The theoretical calculations lead to the conclusion that the anticipated run-off levels of propargite in pond water from the treatment of fields will not seriously affect aquatic life.

Harned (1989a) treated a 1.9 ha orange grove (sandy soil) in central Florida, USA, twice with a 75% EC formulation at a rate of 2.8 kg ai/ha using an air-blast sprayer. Soil cores (0-15 cm, 15-30 cm, 30-60 cm and 60-90 cm depth) collected from the drip line of 3 trees at various intervals were analysed for propargite and TBPC. Residues of propargite were detected only in the 0-15 cm samples and on a dry weight basis were 0.31-0.34 mg/kg immediately after application and peaked 1 dat at 0.54 mg/kg. 28 dat residues of 0.15 mg/kg were detected; by day 151 residues were at or below the limit of quantification (0.05 mg/kg). The first-order rate constant was calculated as  $-0.0104 \text{ day}^{-1}$  and the half-life as 67 days. TBPC was not detected (<0.01 mg/kg) in any sample. About 120 cm rain fell during the 358 days of the trial. Both propargite and TBPC were shown to be stable (<30% loss of residues during freezer storage for 8.5 months).

Lengen (1989d) conducted a similar study in California, USA, applying a WP formulation (300 g/kg) twice to 2 unplanted sites at a rate of 5 kg ai/ha. Soil cores of 0-15 cm, 15-30 cm and 30-60 cm were collected at intervals for analysis. No residues were detected below 15 cm except in a single sample from site 1 at 15-30 cm which contained 0.16 mg/kg TBPC on the day of the second treatment. The half-life was calculated to be 64-122 days. Some 20-23 cm of rain fell during the study. Both propargite and TBPC were shown to be stable in freezer storage for 6 months (Table 18).

Table 18. Residues of propargite and TBPC in California soils (0-15 cm depth) (Lengen, 1989d).

Days after 1st treatment	Days after 2nd treatment	Site 1 (sandy clay loam), mg/kg dry wt		Site 2 (loamy sand), mg/kg dry wt	
		propargite	TBPC	propargite	TBPC
0		4.2	0.15	0.28	0.13
7		1.7	0.07	0.90	0.16
21		0.82	<0.1	0.45	<0.1
21	0	5.3	0.35	2.2	0.27

Days after 1st treatment	Days after 2nd treatment	Site 1 (sandy clay loam), mg/kg dry wt		Site 2 (loamy sand), mg/kg dry wt	
		propargite	TBPC	propargite	TBPC
31	10	2.8	0.10	2.0	0.30
45	24	3.4	0.07	1.0	0.15
178	157	0.42	0.11	1.6	0.21
375	354	0.14	0.16	0.14	0.16

Harned (1990a) conducted a third study in Georgia, USA. Cotton in 0.15 ha sandy loam soil was treated 3 times with a 75% EC formulation at sequential rates of 0.9, 1.8 and 1.8 kg ai/ha. Residues of TBPC were detected only in 0-15 cm core samples. The first-order rate constant for propargite or total residue was  $-0.014 \text{ day}^{-1}$  and the half-life was 50 days. 142 cm rain and irrigation occurred during the course of the study. Propargite was shown to be stable in freezer storage for 9.75 months, but TBPC varied from 61-75% at 1 month, 74-99% at 3 months, 79-86% at 6 months and 86-111% at 9.75 months.

Table 19. Residues<sup>1</sup> of propargite and TBPC in soils in Georgia, USA (Harned, 1990a).

	Residues, mg/kg dry wt.					
	0-15 cm (sandy loam)		15-30 cm (sandy clay loam)		30-60 cm (clay)	
	propargite	TBPC	propargite	TBPC	propargite	TBPC
post appl. 1	0.17	<0.1	<0.05	<0.1	<0.05	<0.1
post appl. 2	0.26	<0.1	0.11	<0.1	0.07	<0.1
post appl. 3	0.24	<0.1	0.1	<0.1	0.06	<0.1
4	0.38	0.21	<0.05	<0.1	<0.05	0.1
7	0.19	0.10	0.05	<0.1	0.05	0.1
27	0.11	<0.1	<0.05	<0.1	<0.05	<0.1
62	<0.05	<0.1	<0.05	<0.1	<0.05	<0.1
93	<0.05	<0.1	<0.05	<0.1	<0.05	<0.1
364	<0.05	<0.1	<0.05	<0.1	<0.05	<0.1

<sup>1</sup> Average of three samples.

In a similar study by Harned (1990c) 36 orange trees in California, USA (sandy loam soil, 0.12 ha) were treated twice at a rate of 3.5 kg ai/ha/treatment using airblast equipment. Samples of soil were collected at the tree dripline. Residues of TBPC were not detectable in the soil cores of 15-30 and 30-45 cm depth and were detected (below 0.1 mg/kg) in only a few samples of the 0-15 cm layer. About 139 cm rain and irrigation occurred throughout the study. Both propargite and TBPC were shown to be stable in freezer storage for 9 months.

Table 20. Residues<sup>1</sup> of propargite and TBPC in California soils (Harned, 1990c).

Dat	Residues, mg/kg dry wt.			
	0-15 cm		15-30 cm and 30-45 cm	30 cm and 30-45 cm p
	propargite	TBPC	Propargite	TBPC
post appl. 1	0.15	<0.1	<0.05	<0.1
pre appl. 2	0.017	<0.1	<0.05	.1
post appl. 2	0.47	<0.1	<0.05	.1
1	0.83	<0.1	<0.05	<0.1
3	0.63	<0.1	<0.05	.1
7	0.041	<0.1	<0.05	.1
30	0.10	<0.1	<0.05	<0.1
62	0.32	<0.1	<0.05	.1
90	0.12	<0.1	<0.05	.1
120	0.11	<0.1	<0.05	<0.1
190	<0.05	<0.1	<0.05	.1
370	<0.05	<0.1	<0.05	.1

<sup>1</sup> Average of three samples.

In a fourth study by Beevers and Harned (1991), cotton grown in California, USA (low organic sandy loam soil, 0.02 ha) was treated three times with a 75% EC formulation at sequential rates of 0.9, 1.8 and 1.8 kg ai/ha. Residues of propargite and TBPC were not detected in 15-30, 30-60 and 60-90 cm samples. The first-order rate constant for the total residue was calculated to be  $-0.0074 \text{ day}^{-1}$  with a half-life of 94 days. About 117 cm rain and irrigation occurred throughout the study. Both propargite and TBPC were shown to be stable in freezer storage for 8.5 months.

Table 21. Residues<sup>1</sup> of propargite and TBPC in California soils (Harned and Beevers, 1991)

DAT	Soil depth 0-15 cm, mg/kg dry wt.	
	propargite	TBPC
post appl. 1	0.54	<0.1
post appl. 2	0.26	<0.1
post appl. 3	0.28	<0.1
1	0.47	<0.1
4	0.30	<0.1
7	0.37	<0.1
29	0.32	<0.1
60	0.54	<0.1
91	0.38	0.1
122	0.22	<0.1
182	0.07	<0.1
283	<0.05	<0.1
367	<0.05	<0.1

Soil and aqueous photolysis. Nowakowski (1988a) exposed [<sup>14</sup>C-phenyl]propargite as a dilute aqueous solution (0.98 mg/l; pH 5) or coated on the surface of desiccated and sterilized sandy loam soil (about 300 mg/kg) to simulated natural sunlight (Xenon arc lamp; 720-800w/m<sup>2</sup>) for 650 hours in solution and 480 hours (soil). In aqueous solution, propargite was calculated to have a half-life of 134-140 days in full sunlight (equivalent to the hydrolysis half-life, see below). No unidentified residues above 10% of the total radioactivity were detected after 27 days and propargite accounted for 69% of the applied radioactivity, TBPC for 6.4% and PTBP for 4.2%, and the remaining 20% was unaccounted for. The half-life in soil was 64 full sunlight days. In the same period dark controls showed no change. TBPC was the only degradation product formed (16% of the applied radioactivity).

Nowakowski (1988b) carried out a further soil photolysis study, coating [<sup>14</sup>C-phenyl]propargite onto the surface of sterilized undesiccated sandy loam soil (114 mg/kg) and maintaining simulated natural sunlight (Xenon arc lamp; 700-750w/m<sup>2</sup>) at 25°C for 17 days. The half-life was calculated as 31 days (24-hour sunlight days) using first-order kinetics and correcting for the reaction constant for the dark control (24 days without correction) The half-life for the control was 113 days. The only product detected by HPLC was TBPC.

In a third study (Korpalski, 1990a) [<sup>14</sup>C-phenyl]propargite was applied to air-dried sandy loam soil at a concentration of 32 mg/kg, maintained at 25°C and irradiated with a Xenon arc lamp with an irradiance of 600 to 650 W/m<sup>2</sup>. After fifteen days of continuous irradiation the half-life was calculated by pseudo-first order kinetics to be 38 days (25 days without correction for the control) and 70 days for the dark control. The major photoproduct was TBPC. About 6% of the applied radioactivity was unextractable after 15 days.

### Environmental fate in water-sediment systems

Hydrolysis. The stability of dilute aqueous solutions of [<sup>14</sup>C-phenyl]propargite (0.6-0.7 mg/l) at pH 5, 7 and 9 at concentrations of 0.005 and 0.5 M at 25°C was assessed by Nowakowski (1987a). The only major hydrolysis product was TBPC. Half lives are shown in Table 22.

Table 22. Hydrolytic half lives (days) of [<sup>14</sup>C]propargite (% of the applied radioactivity as TBPC in parenthesis) (Nowakowski, 1987a).

Concentration	pH 5	pH 7	pH 9
0.005 M	702 (7.8)	48 (47)	2 (86)
0.5 M	120 (32)	78 (37)	3 (71)

Propargite was readily adsorbed onto glass and plastic surfaces and the differences in half lives was attributed to the sorption of propargite by the reaction vessel as a function of buffer strength.

Wright and Lacadie (1983) studied the hydrolysis of 719 g/l EC and 300 g/kg WP formulations in simulated spray tanks (pH 5, 7 and 9 at 37°C). At 0, 5, 24 and 120 hours no significant degradation of the EC formulation mixture (3 g/l) was observed, whereas the WP (1.8 g/l), formulation was degraded at all pHs, most rapidly at pH 9 with a half-life of 6 days.

**Aerobic degradation.** Comezoglu and Harned (1993) applied 5.2 mg/l [<sup>14</sup>C-phenyl]propargite to pond water and sand sediment collected from Lake Van, Florida, USA (equivalent to 11 kg ai/ha in the upper 15 cm soil layer). The system (5.56 g soil plus 9.4 ml lake water) was maintained at 25°C and samples were taken 0-30 days after treatment. In addition to propargite, 8 degradation products were detected in ethyl acetate-extractable fractions by day 30. Less than 1% of the applied radioactivity was recovered as volatiles. Sediment unextracted radioactivity increased from 0.18% at day 0 to 4.1% at day 3 but had not exceeded 6% by the end of the study. The half-life in the hydrosol system was calculated to be 38 days.

Table 23. Distribution of [<sup>14</sup>C]propargite in ethyl acetate extract<sup>1</sup> of pond water and sand sediment after aerobic degradation (Comezoglu and Harned, 1993).

Day	<sup>14</sup> C, % of applied									
	Pro-pargite	TBPC	PTBP	HOMe-TBPC	HOMe-TBPC diol (proposed)	Carboxy-TBPC-	Un-known	Carboxy-TBPC-diol (proposed)	Un-known	Total
0	105	nd	nd	nd	nd	nd	nd	nd	nd	105
3	94	3.3	0.08	0.01	0.26	0.09	nd	0.07	nd	98
7	86	6.2	0.12	nd	0.28	0.31	nd	0.15	nd	93
14	81	12	0.25	0.16	0.48	0.64	nd	0.35	nd	94
21	67	16	0.68	0.17	0.15	1.2	0.18	0.19	0.37	86
30	60	26	0.84	0.26	0.06	0.90	nd	0.07	0.46	88

<sup>1</sup> Ethyl acetate extracts of soil and water combined.

<sup>2</sup> Two compounds with identical mass spectra postulated to be stereoisomers.

**Anaerobic degradation.** The degradation of propargite was studied in a similar pond water/sand sediment mixture from Lake Van by Comezoglu (1994). Residual water and lake debris were removed from the pond sediment (hydrosol) by vacuum filtration, and the filtered hydrosol (22.3 g/biometer flask) combined with lake water (47.8 ml/flask) with glucose added to provide a substrate for metabolism was purged with nitrogen for 30 seconds, then incubated at 25°C in the dark for 30 days. The mixtures were then spiked with 5 mg/kg [<sup>14</sup>C-phenyl]propargite (equivalent to 11.2 kg ai/ha in the upper 15 cm soil layer). The flasks were purged with nitrogen every 14 days, and samples taken 0-365 days after treatment. Initially about 93-96% of the radioactivity was partitioned into ethyl acetate but this decreased to <50% by 1 year. The water-soluble radioactivity remained at about 4% for 9 months and then increased to 13% by 1 year. Unextractable residues slowly increased to >10%

after 90 days and reached a plateau at 120 days of 28%. At 30 days volatiles accounted for only 0.68% of the total applied radioactivity. The half-life in 'hydrosoil' under anaerobic aqueous conditions was 47 days calculated from pseudo-first-order kinetics (log of percentage propargite remaining against time).

Table 24. Distribution of [ $^{14}\text{C}$ ]propargite and its metabolites in organosoluble fractions after anaerobic degradation in pond water and sand sediment (Comezoglu, 1994).

Days	$^{14}\text{C}$ , % of applied											
	Pro-propargite	TBPC	PTBP	HOMe-TBPC	4 unknown	Carboxy TBPC	6 unknown	7 unknown	BGES	9 unknown	10 unknown	total
0	93	nd <sup>1</sup>	nd	nd	nd	nd	nd	nd	nd	nd	nd	93
3	94	5.6	nd	nd	nd	nd	nd	nd	nd	nd	nd	99
7	73	20.	nd	nd	nd	nd	nd	nd	nd	nd	nd	94
14	70	21	nd	nd	nd	nd	nd	nd	nd	nd	nd	91
30	41	39	0.13	0.07	nd	nd	nd	nd	nd	nd	nd	80
60	40	46	nd	0.17	nd	nd	nd	nd	1.0	nd	nd	86
90	29	48	0.11	0.40	0.17	nd	nd	nd	0.94	nd	nd	79
120	11	48	0.47	1.0	0.74	0.25	0.16	nd	0.93	0.50	0.28	63
179	25	45	0.73	0.35	0.26	0.22	0.10	nd	1.2	0.21	0.20	74
270	1.2	60	0.96	4.4	1.2	nd	0.61	nd	0.92	0.51	nd	70
365	0.30	34	0.84	3.5	1.2	nd	0.61	nd	0.34	0.92	0.02	42

<sup>1</sup> Not detected. Limit of detection estimated at 0.05 mg/kg

**Biodegradation.** De Kreuk *et al.* (1986) measured the degradation of [ $^{14}\text{C}$ -phenyl]propargite in polluted and unpolluted water-sediment systems from The Netherlands.

Table 25. Evolved  $^{14}\text{CO}_2$  from two water-sediment systems treated with [ $^{14}\text{C}$ ]propargite at 0.3 and 1 mg/l (De Kreuk *et al.*, 1986).

Days	$^{14}\text{C}$ % of applied evolved in $^{14}\text{CO}_2$			
	Unpolluted (0.3 mg/l)	Unpolluted (1.0 mg/l)	Polluted (0.3 mg/l)	Polluted (1.0 mg/l)
0	0	0	0	0
5	5	4	0	0
19	24	20	9	6
31	32	27	33	31
35	35	29	42	39
46	39	33	55	53
50	42	36	61	59
80	47	41	65	63

The extractability of the residues decreased with time in both systems. Unextractable radioactivity in unpolluted samples was highest in the final sample analysed 50 days after treatment, and in the polluted system reached a plateau of 32% after 35 to 50 days and decreased to 19% after 80 days.

Coenen (1989) conducted a further study using a modified Sturm test with technical propargite at pH 5.5 and 5.9 at 19-22°C. The inoculum was sludge from a municipal sewage treatment plant in The Netherlands. Less than 6% degradation occurred during the test period of 28 days.

### Residues in rotational crops

No information was reported on confined rotational crop studies with radiolabelled propargite.

Crops of small grains and root and leafy vegetables were rotated with cotton in Texas, USA (Korpalski, 1996f). Cotton plants were treated 3 times at a rate of 1.84 kg ai/ha with a propargite EC

formulation by pressurized boom sprayer at 230 l/ha, and the cotton harvested 27 days after the last application at boll-opening stage. After harvest and before planting the new crops beds and stubble were mown, disked and cultivated by hipping, disking, fertilizer application and harrowing. Wheat, carrots and lettuce were planted 82 days and 120 days after the last spraying, 55 and 93 days after harvest respectively. Crop ( $\geq 2.3$  kg) and soil samples were collected at maturity (wheat forage also before grain harvest) and analysed by GC with FPD for propargite, GC with MSD for TBPC, and LC with fluorescence for TBPC diol. No residues (propargite  $<0.01$ - $<0.05$  mg/kg, TBPC  $<0.01$ - $<0.04$  mg/kg, TBPC diol  $<0.025$ - $<0.03$  mg/kg) were found in any samples (wheat forage, grain and straw, carrot root and tops and lettuce) at either plant-back interval. Residues in the soil were 0.01-0.017 mg/kg propargite,  $<0.01$ -0.011 mg/kg TBPC and  $<0.015$  mg/kg TBPC diol.

In an earlier study in California, USA, cotton was treated three times, the third time at mature boll stage, with an EC formulation of propargite at rates of 1.8 or 3.7 kg ai/ha in 230 l water/ha (Popadic, 1993e). Barley, carrots, radish and lettuce were planted 60 or 119 days after the last application, and samples extracted with hexane/2-propanol were cleaned up by a combination of Florisil, gel permeation and alumina column chromatography and analysed by GLC with flame photometric detection for propargite (sulfur mode). The demonstrated limits of quantification were 0.01 mg/kg for barley forage and grain, carrot roots and tops, lettuce and soil, and 0.05 mg/kg for straw. Extracts were derivatized with heptafluorobutyric anhydride (HFBA), cleaned up on SAX and SCX ion-exchange SPE columns and analysed by GLC with an electron capture detector. The demonstrated limits of quantification were again 0.01 mg/kg for barley forage and grain, carrot roots and tops and soil, but 0.04 mg/kg for barley straw. Detectable residues of propargite and TBPC were found in some crop samples at normal maturity, and in barley forage before maturity (Table 68). Soils were collected after each treatment of the cotton, at planting of rotational crops and at rotational crop harvest. Residues of propargite and TBPC in soil were  $<0.01$ -1.2 mg/kg and  $<0.01$ -0.17 mg/kg respectively. The highest concentrations in soil were 0.66 mg/kg of propargite at planting and 0.03 mg/kg of TBPC both in carrots, 3.7 kg ai/ha with a 60-day plant-back.

Table 26. Residues of propargite and TBPC in rotational crops (Popadic, 1993e).

Sample <sup>1</sup>	Rate (kg ai/ha)	Plant-back interval (days)	Residues, mg/kg	
			propargite	TBPC
Barley forage	1.8	119	$<0.01$ -0.03	$<0.01$
Barley straw	1.8	119	$<0.05$ -0.09 (0.08 <sup>2</sup> )	$\leq 0.04$
Barley straw	3.7	119	0.06-0.08 (0.13 <sup>2</sup> )	$<0.04$
Carrot roots	1.8	60	$\leq 0.01$ (0.011 <sup>2</sup> )	$<0.01$
Carrot roots	3.7	60	0.06-0.10	$<0.01$
Carrot roots	1.8	119	0.01-0.03 (0.016 <sup>2</sup> )	$<0.01$
Carrot roots	3.7	119	0.07-0.16	$<0.01$ -0.02
Carrot tops	1.8	60	0.02-0.03 (0.015 <sup>2</sup> )	$<0.01$
Carrot tops	3.7	60	0.02-0.04 (0.024 <sup>2</sup> )	$<0.01$
Carrot tops	1.8	119	0.04-0.05 (0.072 <sup>2</sup> )	$<0.01$
Carrot tops	3.7	119	0.06-0.07 (0.10 <sup>2</sup> )	$<0.01$
Radish tops	3.7	60	$<0.01$ -0.02	$<0.01$
Radish tops	1.8	119	$<0.01$ -0.02	$<0.01$
Lettuce	3.7	119	$<0.01$ -0.01	$<0.01$

<sup>1</sup> In samples at rates and intervals not listed (for example, barley grain at both rates and both plant-back intervals) residues were below the limit of quantification.

<sup>2</sup> Residues in untreated samples

Two additional rotational crop studies on cotton were reported from the USA (Popadic, 1992d,e). In the first cotton (1992d) was sprayed three times with an EC formulation of propargite at rates of 1.8 or 3.7 kg ai/ha applied in water at 190-230 l/ha. Wheat and barley were planted 60 days after the third application in California and Texas and after 79 days in Mississippi. Wheat and barley

forage was harvested about 98 days after planting in California and about 150 days in Texas, when the grain and straw were mature. Samples were stored frozen until analysis by GLC with an FPD. The demonstrated quantification limits for propargite were 0.01 mg/kg for forage and soil and 0.05 mg/kg for grain and straw. TBPC was derivatized with heptafluorobutyric anhydride and determined by GLC with electron capture detection. The demonstrated limits of quantification were 0.04 mg/kg for wheat and barley straw and 0.01 mg/kg for forage, grain and soil. Residues of propargite or TBPC were detected only in the straw after the 3.7 kg ai/ha treatment, 60 days plant-back (<0.01-0.07 mg/kg propargite). Residues of propargite and TBPC in soil were <0.01-1.4 mg/kg and <0.01-0.3 mg/kg respectively.

In the second study (Popadic, 1992e) cotton at two US sites, one in California and the other in Georgia, was sprayed three times with an EC formulation of propargite at 1.8 or 3.7 kg ai/ha (230 l water/ha), the third at lay-by to boll opening. Barley, carrots and lettuce were planted 60 or 120 days after the last application and samples collected at maturity for analysis as above. Detectable residues of propargite and TBPC were found in some crop samples (Table 27). Residues of propargite and TBPC in soil were <0.01-2.2 mg/kg and <0.01-0.46 mg/kg respectively.

Table 27. Residues of propargite and TBPC in rotational crops (Popadic, 1992d,e).

Sample <sup>1</sup>	Rate (kg ai/ha)	Plant-back interval (days)	Residues mg/kg	
			propargite	TBPC
Barley grain California	1.8	60	0.03 (0.011 <sup>2</sup> )	<0.01
Barley grain California	3.7	60	0.01 (0.16 <sup>2</sup> )	<0.01
Barley grain California	1.8	120	0.03 (0.029 <sup>2</sup> )	0.01
Barley grain California	3.7	120	0.03 (no control)	<0.01
Barley straw California	1.8	60	0.6-1.1 (0.29 <sup>2</sup> )	-
Barley straw California	3.7	60	0.2 (0.21) <sup>2</sup>	-
Barley straw California	1.8	120	0.6-0.7 (0.33 <sup>2</sup> )	-
Barley straw California	3.7	120	0.4-0.7 (0.36 <sup>2</sup> )	-
Carrot roots Georgia/California	1.8	60	<0.01-0.02	<0.04
Carrot roots Georgia	3.7	60	0.06-0.08	<0.04
Carrot roots California	3.7	60	0.1	<0.04
Carrot roots California	1.8	120	0.02	<0.04
Carrot roots Georgia	3.7	120	0.04-0.06	<0.04
Carrot roots California	3.7	120	0.25-0.34	<0.04
Carrot tops Georgia	3.7	60	0.04-0.05	<0.04
Carrot tops California	1.8	120	0.10-0.17 (0.10 <sup>2</sup> )	0.01-0.02
Carrot tops Georgia	3.7	120	0.01-0.03	<0.04
Carrot tops California	3.7	120	0.03 (0.018 <sup>2</sup> )	<0.04
Lettuce Georgia	3.7	60	0.01-0.03	<0.01
Lettuce	3.7	120	0.02	<0.01-0.01

Sample <sup>1</sup>	Rate (kg ai/ha)	Plant-back interval (days)	Residues mg/kg	
			propargite	TBPC
Georgia				
Lettuce California	3.7	120	<0.01–0.01	<0.01

<sup>1</sup> Only samples at treatment rates and plant-back intervals with residues are listed.

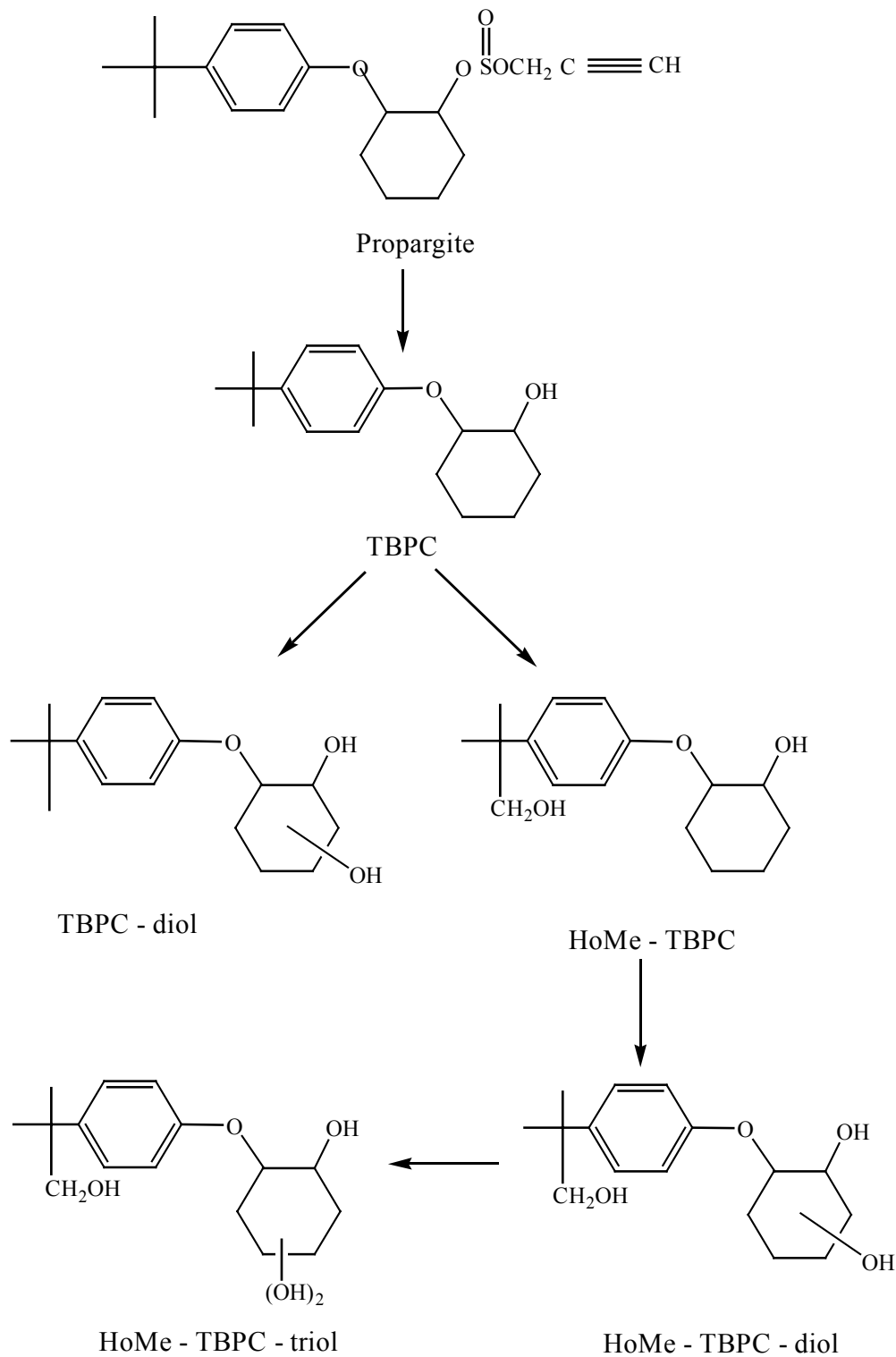
<sup>2</sup> Residues in untreated samples

In additional rotational crop studies reported from the USA (Popadic, 1993d, 1994a) maize was treated once at a rate of 2.8 kg ai/ha with a propargite EC formulation (190 l/ha) and harvested after 30 days. After sixty-two days wheat, carrots and lettuce were planted. Samples were stored frozen and analysed as above. No residues of propargite (<0.01 or <0.05 mg/kg), TBPC (<0.01 or <0.04 mg/kg) and TBPC-diol (<0.025 or <0.03 mg/kg) were detected in any samples (wheat forage, grain, and straw, carrot roots and tops, and lettuce). Residues in soil were <0.01-0.37 mg/kg propargite, <0.01-0.03 mg/kg TBPC and <0.025 mg/kg TBPC-diol.

The degradation pathways of propargite in the environment are shown in Figure 4.



Figure 4. Degradation of propargite in the environment.



### Bioaccumulation in fish

Kuc and Doebbler (1979) studied the bioaccumulation of propargite in channel catfish. Treated soil was placed in the tanks and the fish thus exposed to 0.007-0.03 mg/l [ $^{14}\text{C}$ -phenyl]propargite for 30 days. Maximum residue concentrations occurred on day 14.

Table 28. Residues of propargite and bioaccumulation factors in channel catfish (Kuc and Doebbler, 1979)

	Whole fish	Edible tissues	Inedible tissues
mg/kg propargite (maximum)	2.49	0.46	3.28
Bioaccumulation factor (maximum)	228	39.1	232
mg/kg propargite after 14 days depuration	0.16	0.05	0.20

Surprenant (1988) exposed bluegill sunfish to 3.1 (2-4)  $\mu\text{g/l}$  [ $^{14}\text{C}$ -phenyl]propargite continuously for 35 days. Residues had reached a plateau by day 7 in edible and by day 10 in inedible tissues.

Table 29. Residues of propargite and bioaccumulation factors in bluegill sunfish (Surprenant, 1988).

	Whole fish	Edible tissues	Inedible tissues
mg/kg propargite (maximum)	3.5	1.3	7.3
Bioaccumulation factor (mean)	775	260	1550
mg/kg propargite after 14 days depuration	0.36	0.19	0.64

## RESIDUE ANALYSIS

### Analytical methods

The earliest methods for the determination of propargite in plant and animal commodities and environmental samples used solvent extraction with hexane, nitromethane, hexane/propanol or acetone (for animal tissue), clean-up on a Florisil or alumina column and analysis by GLC with a flame photometric detector (FPD) with a sulfur filter (Devine and Sisken, 1972; Sisken, 1978). These formed the basis for the enforcement methods detailed in the US Pesticide Analytical Methods, Vol. 1 (updated 10/99). Smilo (1977) showed that benzene could be successfully replaced by toluene as the alumina or Florisil column elution solvent in the method described by Devine and Sisken (1972) for peaches and walnuts. Difficulties occurred with water samples where it was later demonstrated that propargite adheres to plastic surfaces (Akhtar, 1988d).

Later modifications include a 0.53 mm capillary column with splitless injection instead of a packed column (Beadle, 1990), although packed columns continue to be used (Popadic, 1993a). In the validation of the method for carrots, radishes and lettuce, clean-up was with a mixture of Florisil column and gel permeation chromatography (GPC), or GPC only (Popadic, 1993b). In the method of Webster (1998) potatoes, mint and stone fruit were extracted with acetonitrile. The acetonitrile fraction was washed with hexane, concentrated and exchanged to petroleum ether. The extract was cleaned up on a Florisil column (plus an Alumina-N column for mint oil) for final analysis by GLC with a mass selective detector. Both propargite (ions 350 and 335) and TBPC (ions 248 and 233) were determined. The column was a megabore (0.25 mm ID) capillary.

An HPLC method was used for the determination of propargite residues in apples, peaches and grapes (Barbina, 1993a,b; Imbroglini, 1995a). In this method (006/CRSA or 008/ISPV) 30 g of chopped fruit is extracted with 75 ml of acetonitrile in a blender for 1 min. The mixture is then filtered, added to water, transferred to a C-18 solid-phase extraction cartridge and eluted with 1 ml of acetonitrile/water (80/20). The HPLC column is a C-18 reverse phase and the mobile phase is isocratic acetonitrile/water 80/20 at 1 ml/min, with UV detection at 225 nm.

In a method developed specifically for TBPC by Popadic (1993a) the plant sample is extracted with acetonitrile and the extract solvent changed to hexane for clean-up on a Florisil column

(acetone/hexane eluant). The eluate is evaporated to dryness and derivatized with heptafluorobutyric anhydride (HFBA). The derivative may require additional clean-up with ion exchange solid phase extraction columns. The derivatized analyte is determined by GLC with an electron capture detector.

The various methods of analysis for propargite and TBPC in plant material, products of animal origin and environmental samples are shown in Tables 30-34. The range of recoveries is shown, usually with the mean  $\pm$  SD.

Table 30. Propargite in plant materials.

Ref.	Sample	Extraction	Clean-up/derivatization	Quantification	LOQ (mg/kg)	Fort. (mg/kg)	% recovery
Devine and Sisken, 1972	Plums, peaches, oranges, grapefruit, potatoes, apples, cherries, strawberries, apricots, grapes	Hexane/2-propanol (1:1), anhydrous sodium sulphate, evaporation (hexane fraction)	Florisil eluted with 2% acetone/hexane	GC-FPD (S)	0.1	0.25-10 Potatoes 0.1	70-120 130
Devine and Sisken, 1972, Sisken, 1978 (PAM II) Smilo, 1977	Nuts	Grind with sodium sulfate/sodium thiosulfate, nitromethane, hexane wash, exchange to toluene	Alumina eluted with benzene	GC-FPD (S)	0.1	Almonds 0.1	81
Sisken, 1978 (PAM II)	Cotton seed	Hexane, extract hexane with nitromethane, exchange to benzene	Alumina eluted with benzene	GC-FPD (S)	0.1	Cotton seed 0.1	99
Sisken, 1978 (PAM II)	Peanuts	Hexane/2-propanol (1:1), partition hexane with acetonitrile, exchange to benzene	Florisil eluted with hexane/acetone (97/3), followed by alumina eluted with benzene	GC-FPD (S)	Not specified		
Webster, 1998	Plums, potatoes, peaches, apricots, 'mint top'	Acetonitrile, hexane wash, evaporation, partitioning with petroleum ether/10% aqueous sodium chloride	Florisil eluted with 15% acetone/petroleum ether, dissolved in toluene	GC-MSD (ions 350, 335)	0.01	Potatoes 0.01 Potatoes 0.5 Potatoes 10 Plums 0.01 Plums 0.5 Plums 10 Peaches 0.01 Peaches 0.5 Peaches 10 Apricots 0.01 Apricots 0.5 Apricots 10	86-96 (n=8) 89 $\pm$ 3.1 92-113 (n=4) 95-101 (n=4) 76-102 (n=8) 74-84 (n=4) 99-107 (n=4) 65-103 (n=8) 96-106 (n=4) 87-95 (n=4) 94-117 (n=8) 91-101 (n=4) 83-101 (n=4)

Ref.	Sample	Extraction	Clean-up/derivatization	Quantification	LOQ (mg/kg)	Fort. (mg/kg)	% recovery
						Mint tops 0.01 Mint tops 0.5 Mint tops 10	100-108 (n=8) 90-109 (n=4) 93-104 (n=4)
Webster, 1998	Mint oil	Acetonitrile, hexane wash, evaporation, partitioning with petroleum ether/10% aqueous sodium chloride	Florisil eluted with acetone/petroleum ether, dissolved in hexane, alumina-N eluted with hexane for propargite, then ethyl acetate for TBPC	GC-MSD (ions 350, 335)	0.1	0.10 5 100	109-120 (n=8) 83-112 (n=4) 68-79 (n=4)
Beadle, 1990	Egg plant (aubergine)	Hexane/2-propanol, anhydrous sodium sulphate, evaporation	None	GC-FPD (S)	0.05	0.05 1 2	68-100 (n=4) 73-84 (n=4) 71-85 (n=4)
Popadic, 1992d <sup>1</sup>	Wheat and barley grain	Hexane/2-propanol (3/2), wash with 3% sodium chloride, partition with acetonitrile	Florisil eluted with 5% acetone/hexane, followed by alumina eluted with toluene	GC-FPD (S)	0.01	Wheat 0.01 Wheat 0.05 Wheat 0.1  Barley 0.01 Barley 0.05 Barley 0.1	90-93 (n=4) 82-99 (n=4) 87-103 (n=4)  77-120 (n=4) 74-101 (n=4) 72-120 (n=4)
Popadic, 1993n <sup>1</sup>	Maize grain Maize forage/ silage Maize fodder	Hexane/2-propanol (1.5/2.0), wash with 3% sodium chloride, partition with acetonitrile.	Florisil eluted with 10% acetone/hexane, followed by alumina eluted with toluene.	GC-FPD (S)	0.05	Maize forage 0.05 Maize forage 5 Maize forage 10 Maize grain 0.05 Maize grain 0.075 Maize grain 0.1 Maize fodder 0.05 Maize fodder 5 Maize fodder 10	90-110 (n=4) 76-97 (n=4) 78-102 (n=4) 107-113 (n=4) 89-113 (n=4) 83-110 (n=4) 93-103 (n=4) 94-112 (n=4) 85-118 (n=4)
Popadic, 1993a <sup>1</sup>	Carrots, radishes, lettuce	2-propanol/hexane (1:1), 3% sodium chloride wash	Florisil eluted with 5% acetone/hexane and GPC	GC-FPD (S)	0.01	Carrot root 0.01 Carrot root 0.05 Carrot root 0.1 Carrot top 0.01 Carrot top 0.05 Carrot top 0.1	83-97 (n=4) 88-104 (n=4) 77-93 (n=4) 87-93 (n=4) 79-99 (n=4) 79-86 (n=4)

Ref.	Sample	Extraction	Clean-up/derivatization	Quantification	LOQ (mg/kg)	Fort. (mg/kg)	% recovery
						Radish root 0.01	70–107 (n=4)
						Radish root 0.05	76–97 (n=4)
						Radish root 0.1	72–102 (n=4)
						Radish top 0.01	80–107 (n=4)
						Radish top 0.05	84–101 (n=4)
						Radish top 0.1	80–92 (n=4)
						Lettuce 0.01	93–100 (n=4)
						Lettuce 0.05	80–117 (n=4)
						Lettuce 0.1	95–100 (n=4)
Barbina, 1993a,b; Imbroglini, 1995	Peaches Grapes Apples	Acetonitrile/ water	C-18 SPE	HPLC (225 nm)	0.1	Grape 1 Grape 2 Apple 1 Apple 2 Peach 1 Peach 2	83 86 88 85 91 83

<sup>1</sup>: validations performed in two sets on different days.

Table 31. Propargite in products of animal origin

Ref.	Sample	Extraction	Clean up/derivatization	Quantification	LOQ, mg/kg	Fort., mg/kg	% recovery
Sisken, 1978 (PAM II)	Animal tissues	Acetone, sodium sulphate, hexane, nitromethane, evaporation	Alumina eluted with benzene, Florisil eluted with hexane/acetone. For fat, clean-up residue dissolved in petroleum ether and extracted with acetonitrile.	GC-FPD (S)	0.1	Liver 0.1 Liver 0.2	90, 100 100, 85
Sisken, 1978 (PAM II)	Eggs	Isopropanol/hexane, sodium sulfate, nitromethane, hexane, toluene, evaporation	Alumina eluted with benzene, Florisil eluted with hexane/acetone. Dissolved in petroleum ether and extracted with acetonitrile.	GC-FPD (S)			
Sisken, 1978 (PAM II)	Milk	Sodium oxalate/ethanol, ethyl ether, petroleum ether, water washing, sodium sulphate, sodium chloride, partition into acetonitrile, evaporate	Florisil eluted with benzene	GC-FPD (S)	0.08	0.08 0.16	75 (n=6) 81–106 (n=6)
Singh and Batorewicz, 1991	Liver Kidney Muscle Fat	Hexane, acetonitrile partition	Florisil eluted with 5% acetone in hexane	GC-FPD(S)	0.01	Beef fat 0.01  Beef liver 0.01	84–102 (n=5) 93+7.0 89–102 (n=6)

Ref.	Sample	Extraction	Clean up/derivatization	Quantification	LOQ, mg/kg	Fort., mg/kg	% recovery
						Beef muscle 0.01 Beef kidney 0.01	95±5.9 72-115 (n=6) 90±15 72-100 (n=5) 88±8.9
Singh and Batorewicz, 1993; Batorewicz, 1993; Bat and Singh, 1991	Milk	Hexane (3X), sodium sulfate	Florisil eluted with 5% acetone in hexane	GC-FPD(S) GC-MS confirmatory	0.01	Milk 0.01 Milk 0.05	82-123 (n=6) 101±16 78-98 (n=6) 88±6.9
Singh and Batorewicz, 1993; Batorewicz, 1993; Bat and Singh, 1991	Eggs	Hexane/2-propanol (1/1), sodium sulfate,	Florisil eluted with 5% acetone in hexane	GC-FPD(S) GC-MS confirmatory	0.01	Eggs 0.01 Eggs 0.05	73-118 (n=6) 98±13 74-100 (n=6) 88±9.3

Table 32. TBPC in plant material, LOQ 0.01 mg/kg.

Ref.	Sample	Extraction	Clean up/derivatization	Quantification	Fort. (mg/kg)	% recovery
Xu and Arjmand, 1994	Wheat straw and grain, raisins, lettuce, carrots, apples	Water/acetonitrile (1/1.9), hexane, sodium chloride solution, water, sodium sulfate, evaporated	Florisil eluted with toluene, HFBA derivatization, ion exchange, eluted with 5% ether in hexane	GC-ECD, capillary column	0.01 (n=3) Grain straw raisins lettuce carrot apple 0.05 (n=3) Grain straw raisins lettuce carrot apple	90-93 80-88 71-100 85-98 78-110 75-91 86-101 93-105 91-101 82-109 101-122 84-86
Webster, 1998	Plums, potatoes, peaches, apricots, 'mint top'	Acetonitrile, hexane (waste), evaporation, partitioning with petroleum ether/10% sodium chloride	Florisil eluted with 15% acetone/petroleum ether, dissolved in toluene. Alumina-N eluted with hexane, dissolved in toluene (mint oil only)	GC-MSD (ions 248 and 233) Megabore capillary	Potato 0.01 Potato 0.5 Potato 10 Plum 0.01 Plum 0.5 Plum 10 Peach 0.01 Peach 0.5 Peach 10 Apricot 0.01	75-90 (n=8) 81±4.8 84-93 (n=4) 90-94 (n=4) 72-91 (n=8) 81±5.2 69-85 (n=4) 93-99 (n=4) 55-87 (n=8) 73±9.5 88-93 (n=4) 81-89 (n=4) 73-91 (n=8) 83±6.0

Ref.	Sample	Extraction	Clean up/derivatization	Quantification	Fort. (mg/kg)	% recovery
					Apricot 0.5 Apricot 0.5 Apricot 10 Mint tops 0.01 Mint tops 0.5 Mint 10	85-94 (n=4) 85-94 (n=4) 73-90 (n=4) 83-91 (n=8) 87±2.4 78-104 (n=4) 85-91 (n=4)
Webster, 1998	Mint oil	Acetonitrile, hexane, evaporation, partitioning with petroleum ether/10% sodium chloride	Florisil eluted with 15% acetone/petroleum ether, dissolved in hexane, alumina-N eluted with hexane for propargite, then ethyl acetate for TBPC	GC-MSD (ions 248 and 233) Megabore capillary	Mint oil 0.01 Mint oil 5 Mint oil 100	109-121 (n=8) 115±5.1 83-112 (n=4) 68-79 (n=4)
Popadic, 1993b	Carrots, radish, lettuce	acetonitrile, hexane, saturated sodium chloride solution, water, sodium sulfate, evaporated, dissolved in hexane	Florisil eluted with toluene, HFBA derivatization with triethylamine (TEA) catalyst, ion exchange	GC-ECD, capillary column	Carrot roots 0.01 Carrot roots 0.05 Carrot roots 0.1 Carrot tops 0.01 Carrot tops 0.05 Carrot tops 0.1 Radish roots 0.01 Radish roots 0.05 Radish roots 0.1 Radish tops 0.01 Radish tops 0.05 Radish tops 0.1 Lettuce 0.01 Lettuce 0.05 Lettuce 0.1	106-113 (n=4) 99-110 (n=4) 89-101 (n=4) 101-118 (n=4) 94-103 (n=4) 94-103 (n=4) 97-111 (n=4) 93-99 (n=4) 94-98 (n=4) 96-113 (n=4) 90-120 (n=4) 98-110 (n=4) 109-119 (n=4) 93-111 (n=4) 97-110 (n=4)

Table 33 TBPC and TBPC-diol in products of animal origin.

Ref.	Sample	Extraction	Clean up/derivatization	Quantification	LOQ (mg/kg)	Fortification (mg/kg)	% recovery
TBPC							
Batore-wiecz and Noon, 1991	Egg Milk Liver Muscle Fat	Acetonitrile, hexane and salt water	Florisil eluted with acetone/hexane. Derivatization with HFBA and	GC-FPD (S)	Egg, milk, muscle, kidney 0.02 Fat, liver 0.04	Egg 0.02 Muscle 0.02 Fat .04	98,99,103 94-121 (n=4) 92-107 (n=5)

Ref.	Sample	Extraction	Clean up/derivatization	Quantification	LOQ (mg/kg)	Fortification (mg/kg)	% recovery
			TEA in benzene			Liver 0.01 Milk 0.02 Kidney 0.02	73-77 (n=4) 103,106, 110 90, 91
TBPC-Diol							
Batorewicz, 1993	Egg Milk Muscle	Acetonitrile	C-18 SPE, Florisil, derivatized with trifluoroacetic anhydride, ether cleaved with BBr <sub>3</sub>	HPLC with fluorescence or amperometric detector	0.02	Egg 0.023 Milk 0.02 Muscle 0.02 Liver 0.02 Kidney 0.02	85-116 (n=10) 89-111 (n=5, ampero) 89-115 (n=10, fluor) 85,94,107 83,102,107 93,107,124

Table 34. Propargite and TBPC in soil and water

Ref.	sample	Extraction	Quantification	LOQ (mg/kg)	Fortification (mg/kg)	% recovery
Akhtar, 1988c	Ground water	Hexane. Glass only (no plastic)	GC-MS (SIM <sup>1</sup> : 64, 108, 135, 150, 211) Megabore capillary column	0.1 µg/l	Propargite 0.1 µg/l TBPC 0.1 µg/l	115-124 (n=3) 84-115 (n=3)
Akhtar, 1988d	Ground water	Hexane. Glass only (no plastic)	GC-FPD (S), megabore capillary column	0.1 µg/l	Propargite 0.1 Propargite 1.0	88, 102 133, 136
Sisken, 1973	Soil	Acetone, chloroform, evaporation	GC- FID (for TBPC) GC-FPD (S) (for propargite)	0.1	Propargite 0.1-5 TBPC 0.1-3	78-104 (n=13) 75-110 (n=7)
Pierce, 1999	Soil	Acetone, chloroform, evaporation, dilution with hexane	GC- FPD (for propargite) or FID (for TBPC), meabore capillary column	0.1	Propargite 0.1 Propargite 1.0 TBPC 0.1 TBPC 1.0	74-98 86±7.9 (n=12) 72-96 84±6.4 (n=11) 71-120 100±6.4 (n=9) 72-89 82±6.4 (n=7)

<sup>1</sup> Single-ion monitoring

### Radio-validation of methods of analysis

Samples from metabolic studies with labelled compounds were analysed to assess the recoveries of residues (accuracy) by the various methods.

Table 35. Radiovalidation of selected methods of analysis.

Reference	Sample	Analyte	Method	TRR (mg/kg)	Distribution of <sup>14</sup> C in TRR	
					Fraction	%
Xu, 1995a,b	Maize forage	propargite	Devine and Sisken, 1972 <sup>1</sup>	0.58	Post-extraction solid	35
					Partition waste	32
					Florisil waste	1.9
					Final extract	32
					Propargite (final extract GC)	26
					Propargite (metabolism study; Banijamali, 1995)	40



Reference	Sample	Analyte	Method	TRR (mg/kg)	Distribution of <sup>14</sup> C in TRR	
					Fraction	%
Xu, 1995a,b	Maize forage	TBPC	Xu and Arjmand, 1994	1.2	Post-extraction solid	36
					Filtrate	68
					Partition waste	32
					Florisil (used)	1.4
					Florisil rinse waste	2.1
					Ion exchange column waste	6.7
					Final extract	3.8
					TBPC (final extract GC)	2.9
					TBPC (metabolism study; Banijamali, 1995)	4.6
Xu, 1996a	Goat milk	propargite	Sisken, 1978 <sup>2</sup>	0.15	Sodium sulfate	15
					Partition waste	32
					Hexane	3.0
					Florisil	4.6
					Florisil waste	22
					Final extract	34
					Propargite (final extract GC)	35
					Propargite (metabolism study; Banijamali and Lau, 1996)	43
Xu, 1996a	Goat liver	propargite	Sisken, 1978 <sup>2</sup>	5.7	Post-extraction solid	53 <sup>3</sup>
					Hexane after acetonitrile extraction	5.2
					Florisil	4.6
					Florisil waste	0.20
					Final extract	35 (28, 28, 49)
					Propargite (final extract GC)	1.1
					Propargite (metabolism study, Banijamali and Lau, 1996)	1.3
Xu, 1996b	Goat milk	TBPC	Xu and Arjmand, 1994 <sup>4</sup>	0.45	Initial extract	97
					Alumina milk residue	3.5
					Partition waste	48
					Water rinse	0.37
					Partitioned extract	55
					Florisil	1.4
					Florisil waste	0.30
					Derivatization waste	0.16
					Final extract	44
					TBPC (final extract GC)	2.2
					TBPC (metabolism study; Banijamali and Lau, 1996)	3.0
					Xu, 1996b	Goat liver
Partition waste	15					
Water rinse	0.02					
Partitioned extract	10					
Florisil	0.84					
Florisil waste	0.01					
Derivatization waste	0.01					
Final extract	9					
TBPC (final extract GC)	5.7					
TBPC (metabolism study; Banijamali and Lau, 1996)	5.8					

<sup>1</sup>Water/methanol/hexane extraction and megabore capillary column. Demonstrated 0.01 mg/kg limit of quantification.

<sup>2</sup>Extraction with hexane. Florisil clean-up only.

<sup>3</sup>Average of three independent sample preparations and analyses.

<sup>4</sup>Similar to method for plant samples. Alumina added to the initial acetonitrile extraction.

### Stability of pesticide residues in stored analytical samples

The Meeting received data on the stability of residues of propargite, TBPC and in some cases TBPC-diol in numerous fortified plant and animal commodities stored frozen and analysed at various intervals. The results are shown in Table 36. Values were uncorrected for analytical method recovery unless indicated.

Table 36. Stability of propargite residues during storage before analysis

Reference	sample	Temp (°C)	Storage <sup>1</sup>	Fort. (mg/kg)	% remaining <sup>2</sup> (Propargite)	% remaining <sup>2</sup> (TBPC)	% remaining <sup>2</sup> (TBPC-diol)
Popadic, 1994f.	Alfalfa regrowth hay (dry)	-20	0d 1m 3m 6m 8m 12m	1.0	100, 100 (92, 97) 90, 104 (98, 100) 93, 90 (88, 72) 88, 77 (87, 87) 90, 78 (88, 97) 92, 82 (100, 95)		
Popadic, 1994f.	Alfalfa regrowth hay (fresh)	-20	0d 1m 3m 6m 8m 12m	1.0	112, 104 (100, 104) 117, 104 (104, 121) 92, 92 (92, 88) 88, 88 (88, 92) 100, 100 (100, 104) 97, 85 (97, 97)		
Popadic, 1994f.	Alfalfa seed screenings	-20	0d 1m 3m 6m 8m 12m	1.0	112, 108 (108, 104) 108, 104 (104, 97) 72, 112 (112, 108) 88, 92 (87, 92) 112, 112 (112, 104) 100, 90 (104, 97)		
Popadic, 1994f.	Alfalfa seed	-20	0d 1m 3m 6m 8m 12m	1.0	113, 96 (104, 112) 100, 98 (112, 104) 88, 92 (92, 85) 92, 97 (112, 112) 98, 83 (112, 104) 88, 85 (104, 104)		
Popadic, 1993g.	Almond hulls	-20	0d 1m 3m 6m 8m 12m	1.0	104, 104 (102, 110) 85, 78 (80, 88) 87, 85 (100, 102) 63, 70 (87, 90) 83, 82 (87, 87) 93, 90 (95, 100)		
Popadic, 1993g.	Almond kernels	-20	0d 1m 3m 6m 8m 12m	1.0	104, 104 (112, 98) 88, 95 (97, 97) 88, 88 (95, 92) 87, 67 (82, 87) 88, 93 (100, 98) 82, 87 (100, 98)		
Popadic, 1993f.	Apple	-20	0m 2m 4m 6m 8m	5.0		(93, 118) 87, 84 (79, 90) 96, 98 (92, 94) 95, 104 (104, 105) 112, 99 (101, 111)	
Popadic, 1994d	Apple	-20	4m	0.05	63-128		
Popadic, 1991e.	Apple	-20	0d 4m 8m 12m	5.0	82 (93) 97 (103) 87 (99) 96 (102)		
Popadic, 1994b.	Apple	-20	0d 2w 1m	0.05	78, 72 (75) 96, 86 (80) 76, 76 (120)		

Reference	sample	Temp (°C)	Storage <sup>1</sup>	Fort. (mg/kg)	% remaining <sup>2</sup> (Propargite)	% remaining <sup>2</sup> (TBPC)	% remaining <sup>2</sup> (TBPC-diol)
			2m 3m 4m		116, 80 (96) 91, 74 (86) 91, 85 (71)		
Popadic, 1994b	Apple juice	-20	0d 2w 4w 8w 12w	0.05	101, 101 (99) 93, 96 (93) 101, 104 (107) 96, 96 (96) 104, 96 (101)		
Popadic, 1994b.	Apple juice concentrate	-20	0d 2w 4w 8w 12w	0.2	104, 99 (107) 93, 93 101, 104 (93) 101, 91 (104) 93, 99 (107)		
Popadic, 1994b.	Apple sauce	-20	0d 2w 4w 8w 12w	0.05	88, 93 (93) 104, 93 (101) 99, 107 (107) 96, 99 (101) 96, 93 (99)		
Korpalski, 1996b	Avocado	-20	90d	1-3	75-95		
Korpalski, 1996b	Avocado	-12	0d 1m 3m 7m 14m	0.1	112, 108 (103) 115, 111 (107) 102, 104 (96) 108, 106 (93) 99, 95 (92)		
Popadic, 1993e.	Barley (grain)	-20	0 1m 2m 6m 7m 9m 12m 13m	0.1	71, 83 (74) 54, 57 (69) 56, 68 (90)  51, 63 (71) 78, 81 (89) 49, 57 (72)	77, 88 (86) 100, 102 (79) 46, 47 (65) 59, 68 (67)  57, 59 (70)  38, 37 (60)	
Popadic, 1993m.	Barley (straw)	-20	0d 1m 2m 7m 9m 12m	0.1	83, 79 (84) 78, 74 (75) 92, 91 (106) 100, 103 (113) 99, 87 (114) 48, 54 (83)		
Popadic, 1993l	Barley forage	-20	604d	0.1			84, 86 (109, 96)
Popadic, 1993m.	Barley forage	-20	0d 2m 4m 6m 8m 12m	0.1	85, 93 (92, 94) 81, 110 (85, 78) 74, 75 (97, 89) 81, 100 (91, 94) 91, 94 (98, 76) 60, 75 (96, 88)		
Popadic, 1993l.	Barley grain	-20	640d	0.1			57, 64 (86, 90)
Popadic, 1993l.	Barley straw	-20	637d	0.4			76, 81 (82, 83)
Popadic, 1993k.	Barley straw	-20	0d 2m 4m 8m 12m	0.2		(79, 88) 85, 88 (91, 95) 114, 113 (99, 108) 90, 77 (82, 82) 70, 90 (89, 93)	
Popadic, 1993e.	Carrot (root)	-20	0d 1m 2m 7m	0.1	106, 104 (121) 87, 85 (112) 81, 76 (123) 88, 87 (113)	98, 87 (84) 79, 81 (86) 92, 98 (91)	

Reference	sample	Temp (°C)	Storage <sup>1</sup>	Fort. (mg/kg)	% remaining <sup>2</sup> (Propargite)	% remaining <sup>2</sup> (TBPC)	% remaining <sup>2</sup> (TBPC-diol)
			9m 12m 13m 20m		72, 83 (107) 76, 76 (120)	72, 74 (77) 72, 79 (65) 52, 62 (59)	
Popadic, 1993e.	Carrot (top)	-20	0 1m 2m 7m 9m 12m 13m 20m	0.1	97, 94 (113) 85, 76 (94) 84, 96 (113) 91, 95 (103) 85, 70 (80) 44, 42 (70)	86, 90 (93) 76, 82 (75) 66, 69 (84) 46, 47 (54) 71, 76 (75) 24, 25 (36)	
Popadic, 1993l.	Carrot roots	-20	685d	0.4			81, 85 (89, 82)
Popadic, 1993l.	Carrot tops	-20	686	0.4			77, 78 (80, 80)
Singh, 1991a; Batorewicz and Noon, 1992.	Eggs	-20	0d 15d 30d 90d  0d 21d  93d	5.0     0.1	94, 104 (88, 105) 104, 96 (92, 90) 102, 98 (86, 110) 90, 94 (100, 84)	(108, 108) 115, 112 (108, 108) 84, 89 (84, 82)	
Singh, 1991a; Batorewicz and Noon, 1992.	Fat (bovine)	-20	0d 15d 30d 90d  0d 44d 100d	5.0     0.2	98, 100 (83, 84) 96, 106 (96, 90) 100, 110 (80, 76) 104, 102 (90, 90)	(86, 85) 79, 69 ((80, 80) 89, 95 (98, 93)	
Singh, 1991a; Batorewicz and Noon, 1992.	Fat (chicken)	-20	0d 30d 81d  0d 15d 30d 90d	0.2     5.0	108, 100 (85, 100) 106, 96 (82, 83) 104, 102 (82, 82) 110, 96 (94, 92)	(82, 91) 79, 74 (101, 84) 80, 85 (90, 80)	
Popadic, 1994b.	Grapefruit	-20	0d 2w 1m 2m 3m 4m	0.05	120, 106 (91) 78, 98 (92) 72, 76 (92) 104, 96 (78) 72, 68 (120) 94, 70 (94)		
Popadic, 1994b.	Grapes	-20	0d 2w 1m 2m 3m 4m	0.05	84, 91 (115) 104, 116 (116) 96, 194 (96) 88, 88 (98) 67, 72 (74) 80, 82 (84)		
Popadic, 1991e	Hops (dry)	-20	12m	10.0	82-92		
Korpalski, 1991a	Hops (dry)	-20	7d 14d 21d	0.1, 5.0	0 <sup>4</sup> , 96 86, 11 0 <sup>4</sup> , 90		
Popadic, 1991f	Hops (dry)	-20	0d 4m 8m	10.	92 (87) 82 (74) 86 (75)		

Reference	sample	Temp (°C)	Storage <sup>1</sup>	Fort. (mg/kg)	% remaining <sup>2</sup> (Propargite)	% remaining <sup>2</sup> (TBPC)	% remaining <sup>2</sup> (TBPC-diol)
			12m		82 (92)		
Popadic, 1991f	Hops (green)	-20	0d 4m 8m 12m	10.	89 (85) 76 (83) 84 (87) 60 (82)		
Korpalski, 1991a	Hops (green)	-20	8d 21d 26d	0.1, 5.0	38, 64 0 <sup>4</sup> , 48 42, 94		
Singh, 1991a; Batorewicz and Noon, 1992.	Kidney (bovine)	-20	0d 15d 30d 90d 180d  0d 68d 121d	5.0      0.1	102, 104 (110, 97) 104, 104 (84, 87) 94, 102 (85, 90) 84, 82 (97, 97) 102, 104 (82, 91)		(88, 87) 90, 88 (91, 89) 110, 101 (114, 114)
Popadic, 1993e.	Lettuce	-20	0d 1m 2m 6m 7m 9m 12m	0.1	101, 112 (111) 87, 109 (114) 93, 104 (97)  106, 104 (103) 102, 103 (109) 93, 95 (112)	91, 112 (99) 98, 101 (103) 96, 101 (103) 101, 103 (104)	
Popadic, 1993l.	Lettuce	-20	685d  810d	0.1			84, 88 (93, 97) 111, 103 (109, 105)
Singh, 1991a; Batorewicz and Noon, 1992.	Liver (bovine)	-20	0d 15d 30d 90d  0d 29d 133d	5.0     0.2	98, 106 (91, 100) 110, 98 (78, 104) 102, 92 (93, 80) 98, 96 (87, 86)		(100, 83) 74, 72 (64, 73) 101, 111 (100, 90)
Batorewicz and Noon, 1992	Liver (chicken)	-20	0d 36d 76d	0.2		(102, 124) 94, 105 (84, 83) 111, 112 (107, 112)	
Popadic, 1993i.	Maize fodder	-20	0 2m 4m 6m 8m 12m	0.1	77, 82 (93, 100) 77, 77 (87, 90) 65, 68 (77, 78) 72, 73 (88, 88) 57, 67 (75, 78) 60, 67 (82, 87)		
Popadic, 1993i.	Maize forage/silage	-20	0d 2m 4m 6m 8m 12m	0.1	77, 85 (97) 87, 90 (92, 100) 65, 70 (80, 80) 62, 63 (70, 70) 55, 62 (70, 97) 50, 62 (77, 83)		
Gaydosh, 1990.	Maize grain (whole)	-20	0m 4m 8m 12m	0.5	89 (94) 100 (102) 84 (87) 86 (95)		
Korpalski, 1990a	Maize grain	-20	2m	0.1	66-110		

Reference	sample	Temp (°C)	Storage <sup>1</sup>	Fort. (mg/kg)	% remaining <sup>2</sup> (Propargite)	% remaining <sup>2</sup> (TBPC)	% remaining <sup>2</sup> (TBPC-diol)
Popadic, 1994b.	Meat (beef)	-20	0d 2w 4w 8w 12w	0.05	91, 93 (88) 88, 85 (88) 77, 77 (80) 75, 73 (79) 76, 73 (85)		
Popadic, 1994b.	Milk	-20	0d 2w 4w 6w 8w	0.05	83, 91 (93) 93, 99 (99) 99, 88 (96) 83, 83 (88) 80, 83 (83)		
Singh, 1991a; Batorewicz and Noon, 1992.	Milk (bovine)	-20	0d 15d 30d 90d  0d 46d 140d	5.0     0.1	104, 96 (92, 80) 100, 102 (83, 80) 92, 102 (94, 88) 96, 88 (100, 92)	(98, 100) 75, 93 (93, 99) 108, 109 (110, 102)	
Singh, 1991a; Batorewicz and Noon, 1992.	Muscle (bovine)	-20	0d 15d 30d 90d 180d  0d 62d 139d	5.0     0.1	106, 104 (102, 92) 94, 102 (83, 107) 104, 100 (98, 94) 88, 88 (86, 91) 64, 68 (114, 95)	(116, 114) 89, 89 (88, 90) 107, 99 (102, 101)	
Batorewicz and Noon, 1992.	Muscle (chicken)	-20	0d 46d  63d	0.1		(96, 96) 105, 108 (100, 113) 109, 107 (108, 110)	
Popadic, 1991b.	Orange	-20	0d 4m 8m 12m	5.0	76 (81) 102 (108) 82 (93) 94 (106)		
Popadic, 1994b.	Orange juice	-20	0d 2w 1m 2m 3m 4m	0.05	119, 96 (98) 120, 94 (98) 84, 98 (96) 88, 106 (98) 82, 82 (91) 91, 96 (96)		
Popadic, 1994b.	Orange juice concentrate	-20	0d 2w 1m 2m 3m 4m	0.2	120, 98 (72) 92, 76 (116) 97, 93 (117) 114, 92 (82) 82, 78 (102) 72, 79 (87)		
Popadic, 1994b.	Oranges	-20	0d 2w 1m 2m 3m 4m	0.05	86, 86 (106) 99, 101 (95) 89, 93 (105) 98, 124 (92) 82, 87 (75) 73, 85 (75)		
Popadic, 1994a.	Peach infant food	-20	0d 2w 4w 8w 12w	0.05	101, 104 (101) 93, 96 (101) 99, 104 (104) 73, 99 (96) 101, 101 (109)		

Reference	sample	Temp (°C)	Storage <sup>1</sup>	Fort. (mg/kg)	% remaining <sup>2</sup> (Propargite)	% remaining <sup>2</sup> (TBPC)	% remaining <sup>2</sup> (TBPC-diol)
Korpalski, 1995c	peaches	-20	171d	0.1, 1.0	89-109		
Popadic, 1994b.	Peaches	-20	0d 2w 1m 2m 3m 4m 5m	0.05	84, 101 (103) 92, 86 (92) 79, 91 (91) 100, 98 (86) 86, 69 (93) 96, 98 (108) 80, 78 (75)		
Popadic, 1992c	potatoes	-20	23d	0.05, 1.0	90-94		
Popadic and Smudin, 1995	Potato (tubers)	-20	121d	0.5	109-110		
Popadic and Smudin, 1995	Potato (flakes)	-20	121d	0.5	103-118		
Popadic and Smudin, 1995	Potato (wet peel)	-20	144d	0.5	94-105		
Popadic and Smudin, 1995	Potato (dry peel)	-20	150d	0.5	76-79		
Popadic, 1994b.	Raisins (dried grapes)	-20	0d 2w 1m 2m 3m 4m	0.05	82, 78 (97) 85, 95 (85) 98, 94 (94) 87, 89 (93) 81, 71 (105) 80, 78 (82)		
Popadic, 1994d.	Raisins (dried grapes)	-20	0d 2m 4m 6m 8m 12m	0.1	(106, 104) 102, 104 (111, 110) 92, 99 (100, 104) 103, 102 (93, 93) 102, 100 (95, 95) 90, 67 (89, 95)	67-204	
Popadic, 1993e.	Soil	-20	0 1m 2m 7m 9m 12m 16m 19m	0.1	101, 96 (95) 93, 100 (117) 77, 89 (94) 81, 81 (92) 93, 94 (93) 79, 80 (88) 86, 78 (86)	80, 92 (75) 63, 84 (80) 71, 72 (82)  55, 61 (76) 63, 72 (75)  72 (84)	
Popadic, 1993f.	Soil	-20	699d  838	0.1			72, 75 (77, 77) 81, 85 (94, 95)
Gaydosch, 1991.	Sorghum grain	-20	0m 4m 8m 12m	10	80 (79) 74 (74) 72 (75) 94 (89)		
Ball, 1988a.	Strawberries	-10 to -15	0d 2m 3m 6m	7.0	81, 78 (79) 111, 108 (109) 103, 107 (91) 104, 97 (92)		
Ball, 1988a.	Strawberries	-10 to -15	8m	2.6, 5.8, 12.0 <sup>3</sup>	108, 88, 79		
Popadic, 1994b.	Strawberries	-20	0d 2w 4w 8w 12w	0.05	107, 107 (107) 91, 93 (96) 93, 99 (96) 96, 101 (104) 99, 93 (104)		
Lalko <i>et al.</i> , 1997.	Tea (black)	-20	0d 2m	1.0	(113, 115) 95, 98 (117, 96) <sup>5</sup>		

Reference	sample	Temp (°C)	Storage <sup>1</sup>	Fort. (mg/kg)	% remaining <sup>2</sup> (Propargite)	% remaining <sup>2</sup> (TBPC)	% remaining <sup>2</sup> (TBPC-diol)
			4m 8m		98, 101 (93, 94) <sup>6</sup> 78, 99 (91, 84) <sup>7</sup>		
Lalko <i>et al.</i> , 1997.	Tea (fresh leaves)	-20	0d 3m 4m 8m	1.0	(106, 106) 111, 91 (86, 101) 105, 100 (92, 93) 90, 98 (94, 110) <sup>8</sup>		
Lalko <i>et al.</i> , 1997.	Tea (green)	-20	0d 2m  4m  8m	1.0	(122, 115) 112, 84 (112, 102) <sup>9</sup> 105, 102 (81, 84) <sup>10</sup> 79, 80 (93, 80) <sup>11</sup>		
Akhtar, 1988c	water (pond)	4	20d	20 µg/l	90-98	95-99	
Akhtar, 1988d	water (pond)	4	18d	20 µg/l	97		

<sup>1</sup> d: days, w: weeks, m: months

<sup>2</sup> values in parenthesis are analytical recoveries from control samples fortified and analysed on same day

<sup>3</sup> incurred residues

<sup>4</sup> corrected for controls

<sup>5</sup> control 0.016 mg/kg

<sup>6</sup> control 0.051 mg/kg

<sup>7</sup> control 0.048 mg/kg

<sup>8</sup> control 0.016 mg/kg

<sup>9</sup> control 0.048 mg/kg

<sup>10</sup> control 0.024 mg/kg

<sup>11</sup> control 0.041 mg/kg

### Definition of the residue

The results of the metabolism studies on maize, apples and potato vines indicate that propargite is a significant proportion of the metabolic residue. In washed apples propargite was 31% of the residue; in maize forage 40% and kernels 11%, and in potato vines 26%. Propargite is hydrolysed to generate the phenoxycyclohexanol, TBPC. TBPC undergoes oxidation to diols and triols.

Studies on chickens and goats demonstrate that propargite is a major proportion of the metabolic residue in milk, fat and eggs, but minor or absent in kidney, liver and muscle. The presence in fat is predicted by the octanol/water partition coefficient of 4–6. As with plants, propargite is hydrolysed to the TBPC and TBPC oxidized to diols and triols. However further oxidation occurs to carboxy-TBPC and this metabolite and its conjugates are a major fraction of the residue in the liver, kidney and muscle of ruminants.

Additionally the metabolites in rats include the plant and animal metabolites and no specific toxicological concern was expressed for these metabolites (*Evaluations 1999. Part II–Toxicology*).

Analytical methods are available for the determination of propargite. A few methods are also capable of determining TBPC, but these require separate sample preparation, derivatization and/or GC-MS.

The Meeting therefore concluded that the following definition of the residue is appropriate.



For compliance with MRLs and estimation of dietary intake in plant and animal commodities:  
propargite.

The residue is fat-soluble.

## USE PATTERN

The manufacturer provided information on GAP and labels. Non-English language labels were not translated but summary sheets were provided. The governments of Australia and Thailand also supplied information on GAP. Germany and The Netherlands have no registered uses of propargite. The use patterns relevant to crop field trials reported to this Meeting are shown in Table 37.

Table 37. Registered uses of propargite.

Crop	Country	Formulation	Rate kg/ai ha	Spray vol. kg ai/hl	Water, l/ha min.	No.	PHI (days)	Remarks
Almond	Greece	EC800g/l EW570g/l	1.1	0.06	1500	3	21	
Almond	Greece	WP300g/kg	1.5	0.06	1500	2		Apply after mid-May
Almond	USA	EC719g/l	3.4		470 ground 140 aerial	2	28	
Almond	USA	WP320g/kg	3.6		470 ground	2	28	California and Arizona only.
Apple	Australia	WP300g/kg	2.6	0.06			7	
Apple	Chile	TD360g/l		0.072			7, 15	Plus tetradifon (60g/l)
Apple	France	WP300g/kg	1.5	0.3	500	1	7	Apply end 06 to end 08
Apple	Greece	WP300g/kg	1.2	0.06	1000	3		After mid-June
Apple	Greece	EC800g/l EW570g/l	1.4	0.06	2000	3	7	After mid-June
Apple	Hungary	EC570g/l	1.1		800 1200		14 10	
Apple	Hungary	WP300g/kg	1.8		1000		10	
Apple	Iran	EW570g/l	1.1	0.057	2000	2		During vegetation period
Apple	Indonesia	EW570g/l	0.57					
Apple	Italy	EW570g/l	0.86	0.086	1000	1	15	
Apple	Italy	EC587g/l	0.88	0.088	1000	1	15	
Apple	Italy	WP300g/kg	0.90	0.090	1000	1	15	
Apple	Japan	WP300g/kg		0.04		1	14	
Apple	Moldova	EC 570 g/l	1.7	0.17	1000	45		
Apple	Portugal	EW570g/l	0.86	0.085	1000		21	
Apple	Ukraine	WP300g/kg	1.2		500	2	45	
Apple	Ukraine	WE570g/l EC570g/l	1.1		1000	2	45	
Apricot	Greece	WP300g/kg	1.5	0.06	1500	2		Apply after mid-May
Banana	Australia	WP300g/kg	0.38	0.03			7	
Beans	Australia	WP300g/kg		0.03			7	
Beans (dry)	Czech Republic	EW 570g/l WP 300g/kg	0.15		500		14	
Beans (dry)	USA	EC719g/l EC785g/l	2.8		190 ground 47 aerial	2	14	West of the Rocky Mountains only. Do not feed or forage vines or bean trash.

Crop	Country	Formulation	Rate kg/ai ha	Spray vol. kg ai/hl	Water, l/ha min.	No.	PHI (days)	Remarks
Cherry	Japan	WP300g/kg	0.40					After harvest and leaf fall.
Cherry	USA	CR320g/kg	2.2		3700			After harvest only. Not in California.
		WP320g/kg			470			After harvest only. West of Rocky Mountains only.
Citrus	Greece	WP300g/kg	2.4	0.06	200	1		Citrus for juices
Citrus	Italy	WP300g/kg	0.90	0.090	1000	1	15	
Citrus	Japan	WP300g/kg	0.04			2	14	
Citrus	Spain	EW380g/l	0.91	0.023	4000	2	14	Plus 17g/l hexithiazox
Citrus	Spain	EC800g/l WP300g/kg EW570g/l	1.1	0.028	4000	2	14	
Citrus	South Africa	WP300g/kg	3.6	0.06	6000	2	14	
Citrus	USA	EC785g/l	2.8		230 ground 94 aerial	2	21	Oranges and grapefruit, Florida and Texas only.
Cotton	Australia	EC600g/l	1.5		100 ground 20 aerial	2	28	
Cotton	Greece	WP300g/kg	1.1	0.11	500	3		
Cotton	Greece	EC800g/l EW570g/l	1.4	0.36	400	3	14	Apply when balls are at final size
Cotton	Spain	EC800g/l WP300g/kg EW570g/l	0.86	0.085	1000	2	14	
Cotton	Kenya	EC570g/l	0.86 ground 1.1 aerial		500 50			
Cotton	USA	EC719g/l	1.9		230 ground 47 aerial	3	50	East of Rocky Mountains only. Apply only before bolls open. Do not feed treated foliage or gin trash.
Cotton	USA	EC785g/l	1.9		230 ground 47 aerial	3	50	California and Arizona only. Ground application through lay-by only. Apply only before bolls open. Do not feed treated foliage or gin trash.
Cucumber	Czech Republic	EW 570 g/l WP300g/kg	0.3		1000		5	Includes cucumber type vegetables. Field and glasshouse.
Cucurbits	Spain	EC800g/l WP300g/kg EW570g/l	0.86	0.085	1000	2	7	
Currant	Czech Republic	EW 570g/l WP300g/kg	0.6		1000			Before blossom and after harvest.
Durian	Thailand	WP300g/kg		0.045			14	20 l/plant
Egg plant (aubergine)	Italy	EW570g/l	0.86	0.086	1000	1	15	
Egg plant	Italy	EC587g/l	0.88	0.088	1000	1	15	
Egg plant	Italy	WP300g/kg	0.90	0.090	1000	1	15	

Crop	Country	Formulation	Rate kg/ai ha	Spray vol. kg ai/hl	Water, l/ha min.	No.	PHI (days)	Remarks
Grapes	Czech Republic Republic	EW 570g/l WP300g/kg	0.88 0.6		1000	2	28	WP after budding
Grapes	France	WP300g/kg	0.9	0.45	200		30	
Grapes	France	EW380g/l + 16.7g/l hexy- thiazole	0.57 + 0.25	0.28 + 0.12	200		30	
Grapes	France	EW570g/l	0.85	0.43	200	1	21	Table and wine grapes.
Grapes	Greece	WP300g/kg	0.9	0.06	500	1		
Grapes	Greece	EC800g/l EW570g/l	0.84	0.06	1500	3	21	15-20 days after bloom
Grapes	Hungary	EC570g/l	1.1		800 1200		14 10	
Grapes	Hungary	WP300g/kg	0.90	200			14	
Grapes	Italy	EW570g/l	0.86	0.086	1000	1	15	No application before flowering
Grapes	Italy	EC587g/l	0.88	0.088	1000	1	15	
Grapes	Italy	WP300g/kg	0.90	0.090	1000	1	15	
Grapes	Japan	WP300g/kg		0.03		1	21 small grain 14 large grain	
Grapes	Spain	EC800g/kg WP300g/kg EW570g/l	0.86	0.085	1000	2	21	
Grapes	Ukraine	WP300g/kg	0.9		1000	2	60	
Grapes	Ukraine	WE570g/l EC570g/l	1.1		1000	2	45	
Grapes	USA	WP320g/kg	3.2		370 ground	2	21	West of Rocky Mountains
Grapefruit	USA	CR320g/kg WP320g/kg	3.8		9400 940	2 1	28	California only West of Rocky Mountains only and use after harvest only.
Green bean	Italy	WP300g/kg	0.90	0.090	1000	1	15	
Hazelnut	Italy	WP300g/kg	0.90	0.090	1000	1	15	
Hops	Australia	WP300g/kg		0.06			7	
Hops	Czech Republic	WP 300g/kg	1.2		2000		10	
Hops	France	WP300g/kg	0.6	0.09	1000	1	21	
Hops	USA	CR320g/kg EC719g/l	1.8		1900	2	14	
Jojoba	USA	EC785g/l	1.8		190 ground 47 aerial	2		
Lemon	Italy	EW570g/l	0.86	0.086	1000	1	15	
Lemon	Italy	EC587g/l	0.88	0.088	1000	1	15	
Lemon	USA	CR320g/kg	3.6		5600	2	28	Arizona only
Lemon	USA	CR320g/kg	3.8		9400	2	28	California only
Maize	Spain	EC800g/l WP300g/kg EW570g/l	1.4	0.42	1000	2	14	
Maize	USA	EC719g/l EC785g/l	2.8		190 ground 19 aerial	1	30	
Maize	USA	EC719g/l	1.7		190 ground	1	56	California only.

Crop	Country	Formulation	Rate kg/ai ha	Spray vol. kg ai/hl	Water, l/ha min.	No.	PHI (days)	Remarks
					94 aerial			
Melon	Italy	EW570g/l	0.86	0.086	1000	1	15	
Melon	Italy	EC587g/l	0.88	0.088	1000	1	15	
Melon	Italy	WP300g/kg	0.90	0.090	1000	1	15	
Mint	USA	EC719g/l	2.5		190 ground 94 aerial	2	14	
Nectarine	Chile	TD360g/l		0.072			1,3,5,10	Plus tetradifon (60g/l)
Nectarine	USA	WP320 g/kg	3.2		470 ground 190 aerial	2	14	West of Rocky Mountains.
Non-bearing crops	USA	CR320g/kg EC719g/l WP320g/kg	2.2 1.7 3.2		470	2		Apply to crops with no fruit within one year: berries, citrus, currants, dates, figs, nut trees, persimmons, tree fruit (stone and pome)
Nut	Italy	WP300g/kg	0.90	0.090	1000	1	15	
Oranges	USA	CR320g/kg WP320g/kg	3.8		9400 940	2 1	28	California only.  After harvest use only. Navel oranges only. West of Rocky Mountains only.
Passion fruit	Australia	WP300g/kg		0.03		2	7	
Peach	Chile	TD360g/l		0.072			1,3,7,10	Plus tetradifon (60g/l)
Peach	France	WP300g/kg	1.5	0.3	500	1	14	Apply end 06 to end 08
Peach	Greece	WP300g/kg	1.5	0.06	1500	2		Apply after mid-May
Peach	Hungary	EC570g/l	1.1		800 1200		14 10	
Peach	Italy	EW570g/l	0.86	0.086	1000		15	
Peach	Italy	EC587g/l	0.88	0.088	1000	1	15	
Peach	Japan	WP300g/kg		0.04		2	21	
Peanuts	USA	EC719g/l EC785g/l WP320g/kg	1.9		190 ground 47 aerial (EC)	1 2 (WP)	14	Do not graze or feed livestock on treated areas or cut treated forage for hay
Pears	Australia	WP300g/kg		0.06			7	
Pepper	Czech Republic	EW 570g/l WP300g/kg	0.14 0.22		500 750		14	Field and glasshouse.
Pepper	Hungary	EC570g/l	1.1		600		7	
Pepper	Italy	EW570g/l	0.86	0.086	1000	1	15	
Pepper	Italy	EC587g/l	0.88	0.088	1000	1	15	
Pepper	Italy	WP300g/kg	0.90	0.090	1000	1	15	
Plum	Chile	TD360g/l		0.072			12,18,25,28	Plus tetradifon (60g/l)
Plum	France	WP300g/kg	1.2	0.24	500	1	21	Apply end 06 to end 08
Plum	Greece	WP300g/kg	1.5	0.06	1500	2		Apply after mid-May
Plum	Hungary	EC570g/l	1.1		800 1200		14 10	
Pome fruit (except pear)	Spain	EC800 g/l WP300g/kg EW570g/l	1.3	0.085	1500	2	21	
Pomelo	Thailand	WP300g/kg		0.045			14	10 l/plant
Popcorn	USA	EC719g/l	2.8		190 ground	1	30	

Crop	Country	Formulation	Rate kg/ai ha	Spray vol. kg ai/hl	Water, l/ha min.	No.	PHI (days)	Remarks
		EC785g/l			19 aerial			
Potato	USA	EC719g/l EC785g/l	2.3		190 ground 94 aerial	2	14	Pacific Northwest only. No. 400-89 (Omite 6; EC719g/l) may be applied through sprinkler irrigation. NO 400-154 (Comite II; EC719g/l) may NOT be applied via irrigation.
Sorghum	USA	EC719g/l EC785g/l	1.9		190 ground 47 aerial	1	30 silage 60 grain	
Soya	Czech Republic	WP300g/kg	0.3		500		21	
Soya	Hungary	EC570g/l	0.86		600		28	
Soya	Italy	EW570g/l	0.86	0.086	1000	1	30	
Soya	Italy	EC587g/l	0.88	0.088	1000	1	15	
Stone fruit	Australia	WP300g/kg	1.6	0.06			7	
Stone fruit	Czech Republic	EW 570g/l WP 300 g/kg	0.6		1000		21	
Stone fruit	Greece	EC800g/l EW570g/l	1.4	0.06	2000	2	7	
Stone fruit	Spain	EC800 g/l WP300g/kg EW570g/l	1.3	0.085	1500	2	7	
Strawberry	Australia	WP300g/kg		0.03			7	
Strawberry	Czech Republic	EW 570 g/l WP 300 g/kg	0.22		750		21	
Strawberry	Greece	WP300g/kg	0.6	0.06	500	2		15 days after transplanting
Sweet corn	USA	EC785g/l	2.8		190 ground 19 aerial	1	30	California only.
Tangerine	Thailand	WP300g/kg		0.045			14	5 l/plant
Tea	Indonesia	EC570g/l		0.11				
Tea	India	EC570g/l	0.81	0.2	400		7	
Tea	Japan	EW570g/l		0.04		2+	14	
Tea	Japan	WP300g/kg		0.04		2	14	
Tea	Kenya	EC570g/l	0.86		500			
Tea	Thailand	EC200g/l		0.05				Repeat at 10–15 day interval
Tea	Thailand	WP300g/kg		0.06				Repeat at 10–15 day interval
Tea	Thailand	EC570g/l	0.57	0.11	500			
Tomato	Australia	WP300g/kg		0.03			7	
Tomato	Italy	EW570g/l	0.86	0.086	1000	1	15	
Tomato	Italy	EC587g/l	0.88	0.088	1000	1	15	
Tomato	Italy	WP300g/kg	0.90	0.090	1000	1	15	
Tomato	Spain	EC800g/l WP300g/kg EW570g/l	0.86	0.085	1000	2	7	

Crop	Country	Formulation	Rate kg/ai ha	Spray vol. kg ai/hl	Water, l/ha min.	No.	PHI (days)	Remarks
Tomato	Portugal	EW570g/l	0.86	0.086	1000		21	
Vegetables	Australia	WP300g/kg		0.03			7	
Vegetables	Greece	WP300g/kg	0.6F 0.9G	0.06G	500	2		F: field G: glasshouse 15 days after transplanting
Vegetables	Iran	EW570g/l	0.57	1000	0.057	2		During vegetation period
Walnuts	Greece	EC800g/l EW570g/l	1.1	0.06	1500	3	21	
Walnuts	USA	EC719g/l	5.0 ground 3.4 aerial		940 ground 190 aerial	2	21	
Walnuts	USA	WP320g/kg	4.5		940 ground 47 aerial	2	21	

### RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The results of trials are shown in Tables 35-67. Where multiple samples were taken from a single plot or multiple analyses conducted on a single sample, the average value is reported. Where results from separate plots with distinguishing characteristics such as different formulations, varieties or treatment schedules were reported, results are listed for each plot. Underlined values were used for the estimation of MRLs and STMRs. Results have not been corrected for concurrent method recoveries unless indicated.

Class	Table no.	Commodity	Class	Table no.	Commodity	
Fruit	38	Orange and Mandarin	Animal feeds	68	Alfalfa	
	39	Orange		69	Peanut hay	
	40	Orange		70	Maize forage	
	41	Lemon		71	Maize dust and fodder	
	42	Grapefruit		72	Sorghum dust and fodder	
	43	Apple		Miscellaneous	73	Hops
	44	Pear			74	Tea
	45	Cherry				
	46	Plum				
		47	Nectarine			
		48	Peach			
		49	Strawberry			
		50	Currant			
		51	Grape			
	52	Avocado				
Vegetables	53	Melon				
	53	Cucumber				
	54	Pepper				
	55	Tomato				
	56	Soya bean				
	57	Dry bean				
	58	Potato				
Cereal grains	59	Maize grain				
	60	Sorghum grain				
Nuts	61	Almond				
	62	Filbert (Hazel nut)				
	63	Pecan				
	64	Walnut				
Oil seed	65	Cotton seed				
	66	Peanut				
Herbs and spice	67	Mint				

Citrus fruits

Oranges. Eight foliar trials were carried out in 2000 in Spain, four on mandarins and four on oranges. Single applications of a WP formulation (300 g/kg) were applied at 1.2 kg ai/ha and 12 fruits sampled from day 0 to day 14 were separated into peel and pulp, extracted with acetonitrile and analysed by GC-MS. The limit of quantification was 0.01 mg/kg. Procedural recoveries from orange peel fortified at 0.01 mg/kg were 75-87%, from pulp, 102-107%. The frozen storage periods before analysis ranged from 127 to 229 days. All controls were <0.01 mg/kg (Table 38).

Table 38. Residues of propargite in oranges and mandarin oranges after single foliar treatments with a WP formulation at 300 g/kg in Spain in 2000 (Harrison, 2002a,b).

Location	Application		PHI (days)	Propargite (mg/kg)		
	kg ai/ha	kg ai/hl		Peel	Pulp	Whole <sup>1</sup>
<i>Orange</i>						
Coria del Rio, Sevilla	1.2	0.17	0	1.3	<0.01	0.37
			3	1.5	0.02	0.46
			7	2.0	0.03	0.58
			10	1.7	<0.01	0.48
			14	0.96	<0.01	<u>0.28</u>
Utrera, Sevilla	1.2	0.18	0	1.2	0.07	0.47
			3	1.8	0.03	0.66
			7	2.2	<0.01	0.70
			10	2.0	0.02	0.70
			14	1.7	0.01	<u>0.61</u>
Tocina, Sevilla	1.2	0.27	14	2.1	0.02	<u>0.55</u>
Palma del Rio, Cordoba	1.2	0.08	14	0.69	<0.01	<u>0.22</u>
<i>Mandarin</i>						
Tocina, Sevilla	1.2	0.18	0	2.1	0.08	0.57
			3	4.4	0.07	1.3
			7	3.9	0.07	1.1
			10	2.3	0.01	0.63
			14	2.7	<0.01	<u>0.71</u>
El Rocio, Almonte	1.1	0.13	0	4.0	0.12	0.84
			3	3.0	0.09	0.70
			7	1.7	0.04	0.36
			10	1.8	0.01	0.38
			14	1.6	<0.01	<u>0.33</u>
Coria del Rio, Sevilla	1.3	0.26	14	3.8	0.02	<u>0.77</u>
Palma del Rio, Cordoba	1.3	0.07	14	0.80	<0.01	<u>0.19</u>

<sup>1</sup> Calculated from peel and pulp results and masses of the two fractions. For oranges, the pulp to peel ratio was 2.4-2.7 for Coria del Rio; from 1.7-2.1 for Utrera; 2.8 for Tocina; and 2.1-2.5 for Palma del Rio; and for mandarins 2.7-3.0 for Tocina, 3.6-4.1 for El Rocio, 4.0 for Coria del Rio and 3.3 for Palma del Rio.

In field trials in the USA foliar applications of EC and WP formulations were made to oranges in Florida, Texas and California in 1986 and 1987. All samples were harvested 7 days after the last treatment. Whole oranges were prepared by solvent extraction and gel permeation chromatography. Final extracts were analysed by gas chromatography with a flame photometric detector. The results are shown in Table 39 (Polakoff, 1988e).

Table 39: Residues of propargite in whole oranges after foliar treatment with EC or WP formulations in the USA (Polakoff, 1988e).

Location Year	Application				PHI Days	Residues, mg/kg
	Form.	No.	kg ai/ha	kg ai/hl		
Orosi, California, 1987	WP 300 g/kg	2	5	0.9	7	2.9
Reedley, California, 1987	WP 300 g/kg	2	5	1.0	7	1.1
Weslaco, Texas, 1987	WP 300 g/kg	2	5	0.7	7	3.2
Orosi, California 1987	WP 300 g/kg (CR)	2	5	0.9	7	2.6
Reedley, California, 1987	WP 300 g/kg (CR)	2	5	1	7	2.7
Weslaco, Texas 1987	WP 300 g/kg (CR)	2	5	0.7	7	2.1
Sanford, Florida 1987	WP 300 g/kg (CR)	2	3.4	0.07	7	1.7
Sanford, Florida 1987	WP 300 g/kg (CR)	2	6.7	0.14	7	2.6
Orosi, California 1987	EC 750 g/kg	2	2.8	0.5	7	2.4
Reedley, California 1987	EC 750 g/kg	2	2.8	0.5	7	2.2
Weslaco, Texas 1987	75EC	2	2.8	0.4	7	2.1
Lake County, Florida 1986	WP 300 k/kg (CR)	2	5	0.21	7	0.75
Ft Pierce, Florida 1986	WP 300 g/kg (CR)	2	5	0.21	7	3.1
Lake Wales, Florida, 1986	WP 300 g/kg (CR)	2	5	0.21	7	1.3
Exeter, California, 1986	WP 300 g/kg (CR)	2	5	0.53	7	4.4
Riverside, California, 1986	WP 300 g/kg (CR)	2	5	0.21	7	1.4
Sanger, California, 1986	WP 300 g/kg (CR)	2	5	0.53	7	3.8
Exeter, California, 1986	WP 300 g/kg (CR)	2	5	0.53 0.21	7	1.6
Lake County, Florida, 1986	EC 570 g/kg	2	2.8	0.12	7	1.1
Lake Wales, Florida, 1986	EC 570 g/kg	2	2.8	0.12	7	2.7

In field trials in South Africa orange trees at three locations (Cairn Trust, Visagie en Seun and Japie Lubbe) were treated twice with a WP formulation (300 g/kg) at 60 or 120 g ai/hl applied by high pressure guns at 5–8 l per tree. Samples taken 0 to 42 days after the last treatment were stored frozen, and separated into peel and pulp before extraction with hexane and partitioning with acetonitrile. Extracts were analysed by gas chromatography with a flame photometric detector. Acceptable recoveries were demonstrated at 0.1 mg/kg for pulp (108%) and at 0.5 mg/kg for peel (88, 98, 114%). The results are shown in Table 40 (Anon., 1997).



Table 40. Residues in oranges after two applications of a WP formulation, South Africa, 1997 (Anon., 1997).

Location	Application		PHI (days)	Propargite (mg/kg)		
	Formulation	kg ai/hl		Peel	Pulp	Whole <sup>1</sup>
Cairn Trust	WP 300 g/kg	0.06	0	2.0	<0.1	0.76
			1	3.0	0.14	1.1
			7	3.1	0.12	0.83
			14	2.4	<0.1	0.87
			21	3.0	<0.1	0.98
			28	4.0	<0.1	1.5
			42	1.4	<0.1	0.52
	WP 300 g/kg	0.12	0	1.8	<0.1	0.58
			1	6.2	0.18	2.1
			7	4.8	0.20	1.7
			14	5.7	0.23	1.9
			21	3.8	0.22	1.3
			28	4.0	0.12	1.4
			42	3.2	0.12	1.1
Visagie en Seun	WP 300 g/kg	0.06	0	0.50	<0.1	0.13
			1	1.4	<0.1	0.38
			7	1.4	<0.1	0.36
			14	0.94	<0.1	0.26
			21	0.26	<0.1	<0.1
	WP 300 g/kg	0.12	0	1.5	<0.1	0.45
			1	3.4	0.18	1.2
			7	3.2	0.16	0.98
			14	5.6	0.10	1.8
			21	1.4	<0.1	0.51
Japie Lubbe	WP 300 g/kg	0.06	0	1.0	<0.1	0.35
			1	1.6	0.28	0.58
			7	4.1	0.16	1.3
			14	3.4	0.30	1.2
			21	6.4	0.34	2.1
			28	1.4	0.14	0.53
			42	3.2	0.17	1.1
	WP 300 g/kg	0.12	0	2.1	<0.1	0.61
			1	5.6	0.10	1.6
			7	7.2	<0.1	2.1
			14	6.2	<0.1	1.8
			21	4.8	<0.1	1.4
			28	4.4	<0.1	1.2
			42	5.8	<0.1	1.7

<sup>1</sup> Calculated from the residues in peel and pulp.

**Lemons.** In field trials on lemons in California (4 trees) and Florida (1 tree), USA, two applications were made at about 5 kg ai/ha and 540 or 9400 l/ha and fruits harvested 7 days later. The whole fruits were extracted and analysed by gas chromatography with a flame photometric detector. The limit of quantification was 0.05 mg/kg. The results are shown in Table 41 (Polakoff, 1988h).

Table 41. Residues of propargite in lemons after the foliar application of a WP formulation in the USA, 1987 (Polakoff, 1988h).

Location, Year	Application				PHI Days	Residues, mg/kg
	Form.	No.	kg ai/ha	kg ai/hl		
Lemon Cove, California	WP 300 g/kg (CR)	2	5	0.92	7	1.7
Nocatee, Florida	WP 300 g/kg (CR)	2	4.7	0.05	7	0.84

Grapefruit. In trials in the USA at three locations a WP formulation at 300 g/kg or EC at 570 g/kg were applied twice at 5 kg ai/ha before a 7-day PHI. The California site consisted of 25 trees on 0.36 ha, while single trees were sprayed in Texas and Florida. Whole fruits were analysed by gas chromatography with a flame photometric detector in the sulfur mode. Controls did not contain propargite (<0.05 mg/kg). The results are shown in Table 42 (Polakoff, 1988i).

Table 42. Residues of propargite in grapefruit from foliar treatment in the USA (7-day PHI) (Polakoff, 1988i).

Location Year	Application				Residues, mg/kg
	Form.	No.	kg ai/ha	kg ai/hl	
Pomona, California, 1987	WP 300 g/kg	2	5	1.0	4.4
Weslaco, Texas 1987	WP 300 g/kg	2	5	0.68	5.9
Lakeland, Florida, 1987	WP 300 g/kg	2	5	0.05	0.61
Weslaco, Texas, 1987	EC 570 g/kg	2	2.8	0.37	2.6
Ft Pierce, Florida, 1987	WP 300 g/kg	2	5	0.21	1.6
Blue Goose Groves, Florida, 1987	WP 300 g/kg	2	5	0.21	2.2
Sanford, Florida, 1987	WP 300 g/kg	2	5	0.11	1.6

### Pome fruits

Apples. Numerous field trials were reported from the USA. In 1991 WP formulations of propargite were applied to apple trees at two locations in Washington state as a concentrate spray (750 l/ha). Ripe apples were harvested 14 and 21 days after the second of two applications for analysis by gas chromatography with a flame photometric detector (Popadic, 1992b).

In 1994 in field trials on apples in California, Washington, New York and Michigan, WP and EC formulations were tested at various application rates. In all cases commercial ground airblast equipment was used to apply the spray at 470 l/ha. Apple samples were frozen immediately after harvest and analysed by gas chromatography with a flame photometric detector (Korpalski, 1995a,b).

In additional field trials in 1994 in Michigan and New York (Popadic, 1995) apple trees were sprayed twice at intervals of 5 to 6 weeks with concentrated EC (570 g/l, 700 g/kg) and WP (300 g/kg) formulations using ground equipment at 2.1 and 3.0 kg ai/ha respectively in 750 l/ha of water. Apples were harvested 14 days after the second application (about 12 apples per sample), stored frozen and analysed by gas chromatography with a flame photometric detector. Recovery from a fortified control apple (0.05 mg/kg) was 71%.

Field trials in the USA in 1987 included side-by-side trials of WP, CR plus WP and EC formulations in six states. Apple trees were sprayed three times with the WP and/or CR formulations at 4.0 kg ai/ha or 2 or 3 times with the EC formulation at 2.1 kg ai/ha. The spray volume was 230 l/ha of water in all cases with PHIs of 7 or 14 days. The apples were analysed by gas chromatography with a flame photometric detector. The method was validated in the concentration range 0.05-20 mg/kg (Polakoff, 1988a).

Summary information of a trial in the Czech Republic was submitted (Anon., 1991). Analyses were by gas chromatography with an electron capture detector.

In a field trial on apples in Vacaria, Brazil, in 1986, an EC formulation (720 g/kg) was sprayed at concentrations of 0.05 kg ai/hl and 0.10 kg ai/hl, with PHIs of 7-30 days. Analysis was by gas chromatography with a flame photometric detector. The limit of quantification was 0.1 mg/kg (Anon., 1986).

Field trials in Hungary in 1991 and 1992 were reported by O'Connell (1992a). In 1991 a WP formulation (300 g/kg) was applied to maturing apples at a rate of 0.9 kg ai/ha to duplicate plots on a 0.5 ha site. Samples taken 10 days later, chopped and stored frozen, were mixed with Florisil and eluted with methylene chloride/acetone, before purification by thin-layer chromatography (Kieselgel 60). Analysis was by gas chromatography with an electron capture detector. In 1992 an EC formulation (570 g/kg) was sprayed on maturing fruit grown on a 0.2 ha plot at 1 kg ai/ha. Samples were taken after 14 days and analysed as in 1991.

In the Republic of Moldova apple trees were treated with an EC formulation (570 g/kg) at a rate of 1.1 kg ai/ha in 1998. The spray volume and method of analysis were not specified. Apples were sampled from day 0 to day 47 (Vasilos, 1998).

Field trials were conducted in Italy in 1992, 1994 and 1995. In 1992 trees in Risano, Udine, six plots of apple trees were treated with either WP (three plots, 0.06 kg ai/hl in 15 hl/ha of water, 0.9 kg ai/ha) or EC formulations (three plots, 1.1 kg ai/ha in 15 hl/ha of water). Samples of apples were stored frozen until analysis by HPLC with a UV detector.

In further trials in 1994 in Risano, EC and EW formulations, each at 570 g/l, were applied at a rate of 1.5 l/ha (0.86 kg ai/ha) in either 10 or 15 hl/ha of water. Apples (12-24 per sample) were harvested at random from 8 or 4 trees and stored frozen until analysis by HPLC with a UV detector.

In trials in Codroipo, Udine, in 1995 ten plots of trees were treated, five with EC and five with EW formulations at a rate of 1.5 l/ha (0.86 kg ai/ha) in 10 hl/ha of water. There were 14 or 15 trees in each plot plus control plots. At harvest, 12-24 apples were collected at random from 5 central trees in each plot and stored frozen until analysis by HPLC (Partington, 1996a).

Several foliar trials on apple trees were reported from France. The WP formulation was applied to trees at two locations, Chateaurenard and La Française, in 1991 and in 1990 to another at Mazieres en Gatine. The plot size was 7 trees. Single applications were made at rates of 0.12 and 0.15 kg ai/hl, and samples taken at 0-14 days. Extracts were analysed by GC-MS, with monitoring of m/z 350. The limit of quantification was 0.05 mg/kg.

In additional field trials in 1992 in France a WP spray formulation was applied to run-off with a motorized knapsack sprayer in commercial orchards at Loiret and Indre et Loire at a rate of 1.5 kg ai/ha in 10 hl/ha of water. Each site consisted of one plot of 5 trees. Apples were sampled from days 0-14 and analysed by GC-MS (Partington, 1993a).

In 1993 in France, commercial orchards at Cheille and Semoy in the Loire valley (central west) were sprayed with single WP or EW formulations to run-off in 10 hl/ha about 14-21 days before harvest. At Cheille the plot consisted of 10 trees and at Semoy of 5 trees. Sampled apples were stored frozen for analysis by GC-MS, monitoring m/z 173. Procedural recoveries at 0.02 mg/kg averaged 76% (range 74-80%) (Partington, 1994d).

The EW, EC and WP formulations were tested at two locations, Aquitaine-Vincent de Pertignas and Midi-Pyrenees- Montauban, France, in 1994. The EC and EW formulations were applied at a rate of 0.86 kg ai/ha and the WP at 1.8 or 3.3 kg ai/ha, all in 500 or 800 l water/ha. One

foliar application was made at the mature fruit stage. Samples were analysed by GC-MS. Recoveries ranged from 72 to 83% for fortifications of 0.05-0.24 mg/kg (Mestres *et al.*, 1996).

The EW formulation was sprayed at 5.0 l/ha (2.8 kg ai/ha) with a motorized knapsack unit overall to run-off in two field trials in France in 1995, one in the Northern Region (St Mesmin, 1132 l/ha) and the other in the Southern Region (Meauzac, 950 l/ha. The plot sizes were 6 trees in a straight row. Apple samples were analysed by GC-MS within 157 days of harvest. Recoveries of 0.01-1.0 mg/kg propargite ranged from 69 to 120%, average 94% (Partington, 1996b).

In two field trials in France for the 1996 season, one in the Northern Region (Saint Hilaire) and the Southern Region (Meauzac), the EW formulation (240 g/l propargite + 40 g/l tetradifon) was applied with a mistblower at the former and with a hydraulic knapsack sprayer at the latter. Analyses were by GC-MS (Partington, 1997a).

In additional trials with the EW formulation (240 g/l propargite + 40 g/l tetradifon) at 4 locations in France in 1996 an air-blast manual sprayer was used to simulate commercial application and one application was made with a 7 day PHI at 1.3 kg ai/ha. Application volumes ranged from 425 to 1087 l/ha in water. Samples for analysis were stored frozen before GC-MS determination. Large samples for processing were kept in chilled storage (Partington, 1997b).

Table 43. Residues of propargite in apples.

Location, Year	Application				PHI Days	Residues, mg/kg	Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl			
Sodus, New York, US, 1994	WP 300 g/kg	1	3.0	0.4	14	2.0	Popadic, 1995
	EC 570 g/kg	1	2.1	0.3	14	2.9	Popadic, 1995
Conklin, Michigan, USA, 1994	WP 300 g/kg	1	3.0	0.4	14	2.0	Popadic, 1995
	EC 570 g/kg	1	2.1	0.3	14	2.4	Popadic, 1995
Winchester, Virginia, USA, 1987	WP 300 g/kg	3	4.0	1.7	7	1.2	Polakoff, 1988a
Sodus, New York, USA, 1987	WP 300 g/kg	3	4.0	1.7	7	2.2	Polakoff, 1988a (c<0.05 mg/kg)
Fennville, Michigan, USA, 1987	WP 300 g/kg	3	4.0	1.7	7	7.4	Polakoff, 1988a
Hartford, Michigan, USA, 1987	WP 300 g/kg	3	4.0	1.7	7	2.1	Polakoff, 1988a
Lindsay, California, USA, 1987	WP 300 g/kg	3	4.0	0.85	7	3.6	Polakoff, 1988a
Sawyer, Washington, USA 1987	WP 300 g/kg	3	4.0	1.7	7	8.3	Polakoff, 1988a
Fennville, Michigan, USA, 1987	WP + 300 g/kg CR	3	4.0	1.7	7	11	Polakoff, 1988a (c<0.05 mg/kg)
Hartford, Michigan, USA 1987	WP + 300 g/kg CR	3	4.0	1.7	7	4.0	Polakoff, 1988a
Lindsay, California, USA 1987	WP + 300 g/kg CR	3	4.0	0.85	7	3.3	Polakoff, 1988a
Sawyer, Washington, USA 1987	WP + 300 g/kg CR	3	4.0	1.7	7	5.0	Polakoff, 1988a
Winchester, Virginia, USA 1987	WP + 300 g/kg CR	3	4.0	1.7	7	1.8	Polakoff, 1988a
Sodus, New York, USA, 1987	WP + 300 g/kg CR	3	4.0	1.7	7	2.6	Polakoff, 1988a

Location, Year	Application				PHI Days	Residues, mg/kg	Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl			
Winchester, Virginia, USA, 1987	EC 570 g/l	3	2.1	0.9	7	1.5	Polakoff, 1988a (c0.96 mg/kg)
Sodus, New York, USA, 1987	EC 570 g/l	2	2.1	0.9	7 14	1.5 3.0	Polakoff, 1988a
Fennville, Michigan, USA, 1987	EC 570 g/l	2	2.1	0.9	14	3.0	Polakoff, 1988a
Hartford, Michigan, USA, 1987	EC 570 g/l	2	2.1	0.9	7 14	4.4 3.8	Polakoff, 1988a
Lindsay, California, USA, 1987	EC 570 g/l	2	2.1	0.45	7 14	2.9 1.8	Polakoff, 1988a
Sawyer, Washington, USA, 1987	EC 570 g/l	2	2.1	0.9	7	2.9	Polakoff, 1988a
Sawyer, Washington, USA, 1987	EC 570 g/l	1	2.1	0.9	14	2.2	Polakoff, 1988a
Bennington, Vermont, USA, 1987	EC 570 g/l	2	2.1	1.7	7	12	Polakoff, 1988a
Ephrata, Washington USA, 1991	WP 300 g/kg	2	2.5	0.33	14	1.0	Popadic, 1992b
					21	0.92	
Ephrata, Washington, USA, 1991	WP 300 g/kg	2	3.0	0.40	14	1.1	Popadic, 1992b
					21	0.65	
Ephrata, Washington USA, 1991	WP 300 g/kg (CR)	2	2.5	0.33	14	0.46	Popadic, 1992b
					21	0.38	
Ephrata, Washington, USA, 1991	WP 300 g/kg (CR)	2	3.0	0.40	14	0.82	Popadic, 1992b
					21	0.41	
Yakima, Washington USA, 1991	WP 300 g/kg	2	2.5	0.33	14	0.79	Popadic, 1992b
					21	1.0	
Yakima, Washington, USA, 1991	WP 300 g/kg	2	3.0	0.40	14	1.4	Popadic, 1992b
					21	2.8	
Yakima, Washington, USA, 1991	WP 300 g/kg (CR)	2	2.5	0.33	14	0.70	Popadic, 1992b
					21	0.74	
Yakima, Washington, USA, 1991	WP 300 g/kg (CR)	2	3.0	0.40	14	1.3	Popadic, 1992b
					21	1.2	
Michigan, USA, 1994	WP 300 g/kg	3	1.7	0.36	14	1.7	Korpalski, 1995b
					21	3.1	
New York, USA, 1994	WP 300 g/kg	3	1.7	0.36	14	1.8	Korpalski, 1995b
					21	2.3	
Michigan, USA, 1994	WP 300 g/kg	3	2.5	0.53	14	4.9	Korpalski, 1995b
					21	4.3	
New York, USA, 1994	WP 300 g/kg	3	2.5	0.53	14	4.0	Korpalski, 1995b

Location, Year	Application				PHI Days	Residues, mg/kg	Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl			
					21	2.9	
Michigan USA, 1994	EC 680 g/kg	2	1.7	0.35	21	3.9	Korpalski, 1995b
					28	3.4	
New York, USA, 1994	EC 680 g/kg	2	1.7	0.35	21	2.6	Korpalski, 1995b
					28	2.9	
New York, USA, 1994	EC 680 g/kg	2	0.85	0.18	27	2.3	Korpalski, 1995b
California, USA, 1994	WP 300 g/kg	2	2.5	0.54	14	0.91	Korpalski, 1995a
					21	0.80	
Washington, USA, 1994	WP 300 g/kg	2	2.5	0.54	14	1.1	Korpalski, 1995a
					21	1.9	
California, USA, 1994	WP 300 g/kg	2	3.0	0.64	14	1.1	Korpalski, 1995a
					21	1.2	
Washington, USA, 1994	WP 300 g/kg	2	3.0	0.64	14	1.7	Korpalski, 1995a
					21	1.2	
California, USA, 1994	WP 300 g/kg (CR)	2	2.5	0.54	14	1.1	Korpalski, 1995a
					21	0.74	
Washington, USA, 1994	WP 300 g/kg (CR)	2	2.5	0.54	14	1.2	Korpalski, 1995a
					21	0.72	
California, USA, 1994	WP 300 g/kg (CR)	2	3.0	0.64	14	1.2	Korpalski, 1995a
					21	0.81	
Washington, USA, 1994	WP 300 g/kg (CR)	2	3.0	0.64	14	1.2	Korpalski, 1995a
					21	0.77	
Czech Republic, 1990 <sup>1</sup>	EC 570 g/kg	1	0.1% ai	Run-off 2.5 l/tree	8	0.48, 3.4	Anon., 1991
					15	2.6	(c0.30-0.65)
					23	1.5	
					29	1.1	
					37	1.2	
					44	1.6	
					49	1.4	
	EC 570 g/kg	1	1.7	0.1	0	2.2	(c0.07-0.20)
					7	2.6	
					14	3.2	
					21	2.8	
					26	3.8	
					33	1.9	
					39	3.6	

Location, Year	Application				PHI Days	Residues, mg/kg	Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl			
Vacaria, Brazil 1986	EC 750 g/kg	1		0.05	7	0.86	Anon., 1986
					15	3.2	
					21	1.7	
					30	0.44	
		1		0.10	7	3.8	
					15	4.4	
					21	3.6	
					30	2.3	
Kalmanhaza, Hungary, 1991	WP 300 g/kg	1	0.9	0.09	10	<0.5	O'Connell, 1992a
Ujfeherto, Hungary, 1992	EC 570 g/kg	1	0.97	0.1	14	2.8	O'Connell, 1992a Control<0.45 mg/kg
Ujfeherto, Hungary, 1992	EC 240 g/kg + tetradifon 40 g/kg	1	0.84	0.08	14	0.88	O'Connell, 1992a Control<0.45 mg/kg
Moldova, 1998	EC 570 g/kg	1	1.1	Unknown	0	0.33	Vasilos, 1998
					10	0.27	
					17	0.22	
					22	0.15	
					27	0.12	
					42	0.1	
					47	0.1	
Risano, Udine, Italy, 1992	WP 300 g/kg	1	0.9	0.06	0	0.15,0.36,0.10	Barbina, 1992a
					7	<0.10(3)	
					14	<0.10(3)	
					21	<0.10(3)	
Risano, Udine, Italy, 1992	EC 570 g/l	1	1.1	0.07	0	0.10,0.19,0.37	Barbina, 1992a
					7	<0.10(3)	
					14	<0.10(3)	
					21	<0.10(3)	
Risano, Udine, Italy, 1994	EW 570 g/l	1	0.86	0.086	0	0.32,0.27,1.1	Barbina, 1994a
					3	0.14,0.18,0.49	
					7	0.16,0.16,0.21	
					14	<0.10(2),0.22	
		1	0.86	0.057	0	0.63,0.40,0.68	Barbina, 1994a
					3	0.26,0.41,0.40	
					7	0.19,0.13,0.42	
					14	<0.10(3)	
	EC 570 g/l	1	0.86	0.086	0	0.39,0.64,0.34	Barbina, 1994a
					3	0.37,0.17,0.33	
					7	0.37,0.16,0.19	
					14	<0.10(3)	
	EC 570 g/l	1	0.86	0.057	0	0.44, 0.57, 0.68	Barbina, 1994a
					3	0.16,0.19,0.32	
					7	0.11,0.18,0.24	
					14	<0.10(3)	
Codroipo, Udine, Italy 1995	EC 570 g/l	1	0.86	0.086	1	0.90, 1.1, 0.70	Partington, 1996a Controls<0.01 mg/kg
					3	1.0, 1.1, 0.89	
					7	0.90, 0.72, 0.49	
					14	<0.01 (3)	
Codroipo, Udine, Italy 1995	EW 570 g/l	1	0.86	0.086	1	0.66, 0.69, 0.92	Partington, 1996a Controls<0.01 mg/kg
					3	0.96, 0.52, 0.77	
					7	0.53, 0.45, 0.76	
					14	0.58, 0.23, 0.55	
Codroipo, Udine, Italy, 1995	EC 570 g/l	1	0.86	0.086	7	0.89, 1.1, 0.90	Partington, 1996a
					14	0.01 (3)	
Codroipo, Udine, Italy, 1995	EW 570 g/l	1	0.86	0.086	7	0.87, 0.53, 0.57	Partington, 1996a
					14	0.65, 0.41, 0.39	

Location, Year	Application				PHI Days	Residues, mg/kg	Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl			
Chareaurenard, France, 1991	WP 300 g/kg	1	1.3	0.12	0	<0.05	O'Connell, 1992b
					3	<0.05	
					7	0.1	
					10	<u>0.2</u>	
					14	0.1	
Chareaurenard, France, 1991	WP 300 g/kg	1	1.6	0.15	0	0.3	O'Connell, 1992b
					3	0.4	
					7	0.3	
					10	<u>0.8</u>	
					14	<0.05	
La Francaise, France, 1991	WP 300 g/kg	1	1.2	0.12	0	0.3	O'Connell, 1992b
					3	0.6	
					7	0.6	
					10	0.3	
					14	<u>1.2</u>	
La Francaise, France, 1991	WP 300 g/kg	1	1.5	0.15	0	1.4	O'Connell, 1992b
					3	1.6	
					7	<u>1.7</u>	
					10	1.7	
					14	1.3	
Mazieres en Gatine, France, 1990	WP 300 g/kg	1	1.8	0.12	0	1.7	O'Connell, 1992b
					3	0.8	
					7	1.0	
					10	1.0	
					14	<u>1.8</u>	
Mazieres en Gatine, France, 1990	WP 300 g/kg	1	2.2	0.15	0	1.4	O'Connell, 1992b
					3	1.9	
					7	1.7	
					10	3.2	
					14	3.7	
Loiret, France, 1992	WP 300 g/kg	1	1.5	0.15	0	0.95	Partington, 1993a
					3	0.66	
					7	0.48	
					10	0.45	
					14	<u>0.60</u>	
Indre et Loire, France, 1992	WP 300 g/kg	1	1.5	0.15	0	0.73	Partington, 1993a
					3	0.69	
					7	<u>0.55</u>	
					10	0.54	
					14	0.37	
Cheille, France, 1993	WP 300 g/kg	1	1.5	0.15	0	1.2	Partington, 1994d
					3	1.2	
					7	<u>1.1</u>	
					14	0.90	
					21	0.83	
					28	0.68	
Cheille, France, 1993	EW 570 g/l	1	1.2	0.12	0	0.77	Partington, 1994d
					3	<0.01	
					7	<u>0.94</u>	
					14	0.69	
					21	0.56	
					28	0.66	
Semoy, France, 1993	WP 300 g/kg	1	1.5	0.15	0	0.80	Partington, 1994d
					3	0.67	
					7	<u>0.73</u>	
					14	0.47	
					21	0.38	
					28	0.36	



Location, Year	Application				PHI Days	Residues, mg/kg	Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl			
Semoy, France, 1993	EW 570g/l	1	1.2	0.12	0	0.81	Partington, 1994d
					3	0.73	
					7	<u>0.64</u>	
					14	0.47	
					21	0.57	
					28	0.45	
Aquitaine, France, 1994	WP 360 g/kg	1	3.3	0.66	7	0.51	Mestres <i>et al.</i> , 1996
					14	0.54	
Montauban, France, 1994	WP 360 g/kg	1	1.8	0.23	7	0.20	Mestres <i>et al.</i> , 1996
Aquitaine, France, 1994	EC 560 g/l	1	0.86	0.17	7	0.23	Mestres <i>et al.</i> , 1996
					14	<u>0.21</u>	
Montauban, France, 1994	EC 560 g/l	1	0.86	0.11	7	0.13	Mestres <i>et al.</i> , 1996
					14	<u>0.11</u>	
Aquitaine, France, 1994	EW 570 g/l	1	0.86	0.17	7	0.29	Mestres <i>et al.</i> , 1996
					14	<u>0.24</u>	
Montauban, France, 1994	EW 570 g/l	1	0.86	0.11	7	0.34	Mestres <i>et al.</i> , 1996
					14	<u>0.16</u>	
St. Mesmin, France, 1995	EW 240 g/l + 40 g/l tetradifon	1	1.2	0.11	0	0.66	Partington, 1996b
					7	<u>0.73</u>	
					14	0.60	
					21	0.53	
					28	0.39	
Meauzac, France 1995	EW 240 g/l + 40 g/l tetradifon	1	1.2	0.13	0	1.6	Partington, 1996b
					7	<u>0.81</u>	
					14	0.60	
					21	0.54	
					28	0.48	
Saint Hilaire (North), France, 1996	EW 240 g/l + 40 g/l tetradifon	1	1.2	0.12	0	0.63	Partington, 1997a
					1	0.66	
					3	0.74	
					7	0.42	
					14	<u>0.47</u>	
Meauzac (South), France, 1996	EW 240 g/l + 40 g/l tetradifon	1	1.2	0.12	0	0.57	Partington, 1997a
					1	0.57	
					3	0.51	
					8	<u>0.29</u>	
					15	0.28	
Thomeer La Sogne (North), France, 1996	EW 240 g/l + 40 g/l tetradifon	1	1.2	0.16	0	0.70	Partington, 1997b
					7	<u>0.55</u>	
Azay Le Rideau (North), France, 1996	EW 240 g/l + 40 g/l tetradifon	1	1.2	0.28	0	1.0	Partington, 1997b
					7	<u>0.64</u>	
Saint Porchaire (South), France, 1996	EW 240 g/l + 40 g/l tetradifon	1	1.2	0.14	0	0.75	Partington, 1997b
					7	<u>0.44</u>	
Fregimont, France (South), 1996	EW 240 g/l + 40 g/l tetradifon	1	1.2	0.11	0	0.94	Partington, 1997b
					7	<u>0.79</u>	

c: control

<sup>1</sup> trials in Czech Republic were reported in summarized form.

Pears. Field trials in Washington, Oregon, California, Minnesota and Pennsylvania, USA, in 1980, 1982 and 1987 were reported as summaries (Polakoff, 1988j). Typically two applications of a WP formulation were made at 2.5 kg ai/ha/application with 14 day PHIs. Samples were analysed by gas chromatography with a flame photometric detector (Table 44).

Table 44. Residues of propargite in pears, USA (Polakoff, 1988j). All two applications of WP 300 g/kg.

Year	Application		PHI Days	Residues, mg/kg
	kg ai/ha	kg ai/hl		
1987	2.5	1.1	14	2.5
1987	2.5	1.1	14	1.2
1987	2.5	1.1	14 21	0.63 0.63
1987	2.5	0.55	14 21	1.0 0.5
1981	2.5	0.05-0.55	14	0.66
1981	3.4	0.05-0.35	14	0.79
1981	5	0.05-0.55	14	0.83
1981	6.5	0.1-0.7	14	0.96
1981	2.5	0.05-0.55	13	1.2
1981	3.4	0.05-0.35	13	1.7
1981	5	0.05-0.55	13	1.5
1981	6.5	0.1-0.7	13	1.9
1980	2.5	0.05-0.25	14	1.6
1980	3.4	0.05-0.35	14	1.5
1980	5	0.05-0.65	14	2.3
1980	6.5	0.1-0.7	14	4.1
1980	2.5	0.05-0.25	14	1.1
1980	3.4	0.05-0.35	14	1.6
1980	5	0.05-0.65	14	1.6
1980	6.5	0.1-0.7	14	2.5
1980	3.4	0.05-0.35	14	1.1
1980	6.5	0.1-0.7	14	2.5
1980	2.5	0.05-0.25	14	1.4
1980	3.4	0.05-0.35	14	1.5
1980	5	0.05-0.65	14	2.3
1980	6.5	0.1-0.7	14	3.2
1980	2.5	0.05-0.25	14	1.6
1980	3.4	0.05-0.35	14	1.7
1980	5	0.05-0.65	14	3.1
1980	6.5	0.1-0.7	14	3.4
1980	2.5	0.05-0.25	14	2.3
1980	3.4	0.05-0.35	14	3.9
1980	5	0.05-0.65	14	7.8
1980	6.5	0.1-0.7	14	12.0
1980	2.5	0.05-0.25	14	2.2
1980	3.4	0.05-0.35	14	2.1
1980	5	0.05-0.65	14	2.7
1980	6.5	0.1-0.7	14	4.2

### Stone Fruits

**Cherries.** A summary of residue trials for cherries in the USA was submitted (Polakoff, 1988f). The WP formulation (300 g/kg) was applied to cherry trees as the cherries were ripening. Most treatments were at 2.5 kg ai/ha with a 7-day PHI. Cherries were pitted before analysis and the residue in the whole fruit was from the weights of stone and pulp. Propargite residues were determined by gas chromatography with a flame photometric detector. The nominal limit of quantification was 0.05 mg/kg (Table 45).

Table 45. Residues of propargite in sweet and sour cherries, USA (Polakoff, 1988f). All WP 300 g/kg with 7-day PHI.

Year	Application			Residues, mg/kg <sup>1</sup>
	No.	kg ai/ha	kg ai/hl	
1987	2	2.5	0.09	7
1980	1	2.5	0.06	1.4
1980	1	2.5	0.09	2.0
1980	1	2.5	0.09	3.0
1980	1	2.5	0.09	3.0
1980	1	2.5	0.09	3.0
1980	1	2.5	0.07	4.1
1980	1	2.5	0.07	4.0
1980	1	2.5	0.1	4.5
1980	1	2.5	0.5	4.5
1980	1	2.5	0.5	7.0
1980	1	2.5	0.07	9.0
1980	1	5.0	0.14	2.3
1980	1	5.0	0.18	4.0
1980	1	5.0	0.13	4.2
1980	1	5.0	0.18	5.0
1980	1	5.0	0.18	6.0
1980	1	5.0	0.14	5.9
1980	1	5.0	0.14	5.8
1980	1	5.0	1.1	5.8
1980	1	5.0	1.1	10
1980	1	5.0	0.14	15
1968	2	2.5	0.05	6
1968	2	5	0.1	11
1968	2	5	0.1	14
1968	1	2.5	0.05	0.2
1968	1	2.5	0.1	0.5
1968	1	2.5	0.05	0.92
1968	1	2.5	0.1	4.0
1968	1	5.0	0.2	0.5
1968	1	5.0	0.1	1.7
1968	1	5.0	0.1	4.0
1968	1	5.0	0.1	9.0

<sup>1</sup> whole weight including stone

**Plums.** In trials in South and North France (two in 1995 and two 1996, two or three treated and one untreated plot at each location) spray applications were made of a WP formulation (nominal 300 g/kg) at a rate of 0.15 kg ai/hl to run-off or an EC or EW formulation (nominal 570 g/l; 590 g/kg and 520 g/kg respectively) at 2.5 l/ha to below run-off but up to 1000 l/ha. Samples were taken 0-28 days after application and stored frozen for up to 235 days before analysis. They were extracted with methanol/hexane, cleaned up by liquid-liquid partition, further purified by gel permeation chromatography and analysed by GC-MS. The method was validated in the range 0.01-1 mg/kg (69-

90% recovery). Analyses were on pitted fruits, but the results (Table 46) were calculated for whole fruit from the weights of stone and pulp (Partington, 1996c,d).

An additional study was reported from France for the 1997 crop. Commercial crops of plums at 2 locations, one in the North and one in the South, were treated with an EW formulation (570 g/l applied to just below run-off at 1.4 kg ai/ha) or a WP (306 g/kg, at 1.5 kg ai/hl to run-off) applied by an airblast manual sprayer to simulate commercial application. Samples taken at intervals of 0-28 days were stored frozen and analysed by GC-MS. A large sample from a WP treatment was dispatched for processing. Stones were removed before analysis, but results were recorded on a whole fruit basis. Procedural recoveries in the concentration range 0.01-5 mg/kg were acceptable at 69-117% (Partington, 1998).

In US trials plums at three locations in California were treated twice at intervals of 50-60 days with WP or EC formulations in 1987 and 1979, and harvested 14 or 28 days later for analysis by gas chromatography with a flame photometric detector. Adequate recovery was demonstrated with fortified controls at 0.1 mg/kg (Polakoff, 1988d). In an additional trial in Madera, California in 1993 a 300 g/kg WP formulation was applied by airblast sprayer at 470 l/ha, with a second application at fruit maturity. Plum samples were picked 21 days after the last application. Quantification was by gas chromatography with a flame photometric detector. The limit of quantification was 0.05 mg/kg (Popadic, 1994, Table 46).

Table 46. Residues of propargite in plums and prunes.

Location, Year	Application				PHI Days	Residues, mg/kg fresh plums <sup>1</sup>	Residues, mg/kg prunes <sup>1,2</sup>	Reference
	Form.	No.	kg ai/ha	kg ai/hl				
La Cage, France (south), 1995	WP 300 g/kg	1	1.5	0.15	0	1.2		Partington, 1996c
					3	1.1		
					7	1.0		
					14	0.86(c0.02)		
					21	<u>0.65</u>		
					28	<u>0.48</u>		
La Cage, France (south), 1995	EC 570 g/l	1	1.4	0.16	0	1.2		Partington, 1996c
					3	1.4		
					7	0.98		
					14	1.0(c0.02)		
					21	<u>0.63</u>		
					28	<u>0.55</u>		
La Cage, France (south), 1995	EW 570 g/l	1	1.4	0.15	0	0.68		Partington, 1996c
					3	1.1		
					7	0.58		
					14	0.57(c0.020)		
					21	<u>0.59</u>		
					28	<u>0.41</u>		
Le Pech a Meuzac, France (south), 1995	WP 300 g/kg	1	2.0	0.15	0	1.8(c0.03)		Partington, 1996c
					3	1.8(c0.02)		
					7	1.9(c0.03)		
					14	1.4(c0.04)		
					21	1.0(c0.02)		
					28	0.84(c0.03)		
Le Pech a Meuzac, France (south), 1995	EC 570 g/l	1	1.4	0.14	0	1.1(c0.03)		Partington, 1996c
					3	1.1(c0.02)		
					7	0.86(c0.03)		
					14	1.1(c0.04)		
					21	<u>1.1(c0.02)</u>		
					28	<u>0.69(c0.03)</u>		

Location, Year	Application				PHI Days	Residues, mg/kg fresh plums <sup>1</sup>	Residues, mg/kg prunes <sup>1,2</sup>	Reference
	Form.	No.	kg ai/ha	kg ai/hl				
Le Pech a Meuzac, France (south), 1995	EW 570 g/l	1	1.4	0.15	0 3 7 14 21 28	0.89(c0.03) 0.95(c0.02) 0.84(c0.03) 0.70(c0.04) <u>0.74</u> (c0.02) 0.51(c0.03)		Partington, 1996c
Mezieres Lez Clery, France (north), 1996	WP 300 g/kg	1	1.8	0.15	0 3 7 14 21 28	0.78 0.59 0.74 0.23 0.32 0.27		Partington, 1996d
Mezieres Lez Clery, France (north), 1996	EC 570 g/l	1	1.4	0.14	0 3 7 14 21 28	0.53 0.56 1.0 0.58 0.38 <u>0.81</u>		Partington, 1996d
LeBlance, France (north). 1996	WP 300 g/kg	1	0.75	0.15	0 3 7 14 21 28	3.9 4.2(c0.04) 2.4 2.2(c0.02) 1.7 2.0		Partington, 1996d
LeBlance, France (north). 1996	EC 570 g/l	1	1.4	0.26	0 3 7 14 21 28	7.4 5.9(c0.04) 3.8 2.4(c0.02) <u>3.0</u> 2.0		Partington, 1996d
Valleres, France (north), 1997	WP 300 g/l	1	0.67	0.15	0 7 14 21 28	0.52 0.46 0.37 0.25 0.34		Partington, 1998
Valleres, France (north), 1997	EW 570 g/l	1	1.4	0.33	0 7 14 21 28	1.1 1.2 0.95 <u>0.97</u> 0.96		Partington, 1998
Saint-Maurin, France (south), 1997	WP 300 g/kg	1	1.0	0.15	0 7 14 21 28	1.0 1.2 0.68 <u>0.39</u> 0.29		Partington, 1998
Saint-Maurin, France (south), 1997	EW 570 g/l	1	1.4	0.26	0 7 14 21 28	1.5 1.8 0.94 0.71 <u>1.2</u>		Partington, 1998
Madera, California, USA, 1987	WP 300 g/kg	2	5.0	1.1	14	1.7(c0.26)	1.5(c0.18)	Polakoff, 1988d
Easton, California, USA, 1987	WP 300 g/kg	2	5.0	1.1	14	3.4(c0.08)	3.4(c0.30)	Polakoff, 1988d
Wheatland, California, USA, 1987	WP 300 g/kg	2	5.0	1.1	14	2.6	1.5(c0.16)	Polakoff, 1988d
Madera, California, USA, 1987	EC 570 g/kg	2	5.2	1.2	28	1.3(c0.24)	1.0(c0.14)	Polakoff, 1988d
Easton, California, USA, 1987	EC 570 g/kg	2	5.2	1.2	28	3.0	2.9(c0.30)	Polakoff, 1988d

Location, Year	Application				PHI Days	Residues, mg/kg fresh plums <sup>1</sup>	Residues, mg/kg prunes <sup>1,2</sup>	Reference
	Form.	No.	kg ai/ha	kg ai/hl				
Wheatland, California, USA, 1987	EC 570 g/kg	2	5.2	1.2	28	1.6	0.98(c0.16)	Polakoff, 1988d
Reedley, California USA, 1979	WP 300 g/kg	2	3.4	1.8 aerial 0.12 ground	14	0.5 0.8	1.4 1.4	Polakoff, 1988d
Reedley, California, USA, 1979	WP 300 g/kg	2	3.4	1.8 aerial	14	1.6	1.2	Polakoff, 1988d
Madera, California, USA, 1993	WP 300 g/kg	2	3.0	0.64	21	4.0, 4.0(c0.08)	1.0, 0.76(c0.08)	Popadic, 1994

<sup>1</sup>. whole including stone

<sup>2</sup>. c: control.

Nectarines. In a field trial in California, USA, a WP formulation (300 g/kg) was applied at 3.4 kg ai/ha by ground (2800 l/ha) and aerial (190 l/ha) equipment to trees, and the fruit harvested 14 days after the application. The pitted fruit was analysed by GLC with a flame photometric detector and the residues calculated on a whole fruit basis (Polakoff, 1988m, Table 47).

In trials in France in 1986 a WP formulation (300 g/kg) was applied once at 1.5 kg ai/ha, spray volume 1000 l/ha, and the ripe fruit sampled (1 kg) at 1-21 day intervals for analysis by gas chromatography with a flame photometric detector (Tomkins, 1987). Additional trials at two locations in South-West France were reported for the 1993 crop. Single applications of a WP formulation (300 g/kg, 1000 l/ha and 1.5 kg ai/ha) were made by air-blast manual sprayer 14-21 days before harvest. Samples were taken 0-21 days after application and stored frozen until analysis by GC-MS. The procedure was validated by control fortifications at 0.02, 0.10 and 1.9 mg/kg with recoveries of 73%, 94% and 93% (Partington, 1994a, Table 47).

Table 47. Residues of propargite in nectarines.

Location, Year	Application				PHI Days	Residues, mg/kg <sup>1</sup>	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
Reedley, California, US, 1979	WP 300 g/kg	2	3.4 aerial	1.8	14	<u>1.3</u>	Polakoff, 1988m
Reedley, California, US, 1979	WP 300 g/kg	2	3.4 ground	0.12	14	<u>1.4</u>	Polakoff, 1988m
Domain de Capou Montauban, France, 1986	WP 300 g/kg	1	1.5	0.15	0 7 14 21	3.2 2.6 <u>0.94</u> 0.39	Tomkins, 1987
Lizac, France 1993	WP 300 g/kg	1	1.5	0.15	0 3 7 14 21 28	1.2 1.2 1.0 0.89 <u>1.2</u> 0.63	Partington, 1994a
Meauzac, France 1993	WP 300 g/kg	1	1.5	0.15	0 3 7 14 21 28	1.5 1.6 1.4 <u>1.0</u> 0.82 0.81	Partington, 1994a

<sup>1</sup> whole including stone

Peaches (Table 48). In US field trials in California, Georgia and South Carolina a WP formulation (300 g/kg) of propargite was applied to trees twice at a rate of 3.0 kg ai/ha with ground airblast equipment at about 470 l/ha (Korpalski, 1995c). Each site consisted of three plots (two treated and one control) containing a minimum of 16 trees. Duplicate samples of 16 fruits each taken 14 days after the second application were stored frozen until analysis by gas chromatography with a flame photometric detector (Table 48). The method was validated at 0.05–7.0 mg/kg.

In US field trials in 1987 a WP formulation (300 g/kg) was applied twice to Autumn Glow, Fayette and O’Henrys peaches at 5.0 kg ai/ha in 470 l water/ha with ground airblast equipment (Polakoff, 1988g, summary report). Peaches were pitted before analysis, but results were calculated on the whole fruit.

In trials in Italy in 1992 an EC formulation (570 g/l; 590 g/kg) was applied once to trees at a rate of 2 l/ha (1.1 kg ai/ha) in 15 hl water/ha (Barbina, 1992c, 1993a).

In two trials in South France in 1995 (three treated and one untreated plot at each site) each plot was treated with a WP formulation (300 k/kg) at a rate of 0.15 kg ai/hl applied to run-off or an EC or EW formulation (590 g/kg and 520 g/kg respectively) at 2.5 l/ha, below run-off but up to 1000 l/ha. Samples taken at intervals of 0-28 days were stored frozen for 235 days before analysis of pitted fruits, extracted with methanol/hexane and cleaned up by liquid-liquid partition. The extracts were purified by gel permeation chromatography and analysed by GC-MS. The method was validated for peaches in the range 0.01-1 mg/kg (93-123%). The results were calculated for whole fruit (Partington, 1996c).

In additional trials in South France an EW or WP formulation was applied by airblast manual sprayer to two plots of trees at each of two locations (EW 0.15 kg ai/hl to run-off; EW at 1.4 kg ai/ha to less than run-off, maximum 1000 l/ha). Samples (24 fruits) were taken at intervals of 0-28 days and stored frozen until analysis by GC-MS. The stones were removed before analysis, but the results were expressed on the whole fruit (Partington, 1996d).

In trials in Hungary an EW formulation (nominal 570 g/l) was sprayed on a 0.3 ha plot of peach trees at 2 l/ha (1.1 kg ai/ha, volume 800 l/ha) 10 days before harvest (Toth, 1994; O’Connell, 1992f). Triplicate samples (2 kg) were stored frozen until analysis. The samples without stones were extracted with hexane and purified on a silica gel column. The final extracts were analysed by gas chromatography with an electron capture detector and the results calculated on a whole fruit basis. In a separate trial in 1991 a WP formulation (300 g/kg) was applied to ripening peaches at 1.1 kg ai/ha. A gas chromatograph with flame photometric detector was used to analyse the samples.

Table 48. Residues of propargite in peaches.

Location, Year	Application				PHI Days	Residues, mg/kg	Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl			
South Carolina, USA, 1994	WP 300 g/kg	2	3.1 3.0	0.6	14	3.0, 1.6	Korpalski, 1995c pitted
Georgia, USA, 1994	WP 300 g/kg	2	3.1 3.1	0.6	14	2.3, 1.8	Korpalski, 1995c pitted
California, USA, 1994	WP 300 g/kg	2	3.0 3.1	0.6	14	1.5, 2.4 (c0.41)	Korpalski, 1995c pitted
Fresno, California, USA, 1987	WP 300 g/kg	2	5.0	0.98	14	3.6	Polakoff, 1988g
Hanford, California, USA, 1987	WP 300 g/kg	2	5.0	1.1	14	5.7 (c0.06)	Polakoff, 1988g

Location, Year	Application				PHI Days	Residues, mg/kg	Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl			
Upper Black Eddy, Pennsylvania, USA, 1987	WP 300 g/kg	2	5.0	1.1	1	22	Polakoff, 1988g
					7	13.	
					14	6.4	
					21	5.8	
					28	4.4	
Hanford, California, USA, 1987	WP 300 g/kg	2	5.0	1.1	0	1.9 (c0.07)	Polakoff, 1988g
					1	7.6 (c0.05)	
					7	12	
					14	5.1 (c0.05)	
					28	2.3	
Fresno, California, USA, 1987	WP 300 g/kg	2	5.0	0.98	0	3.5	Polakoff, 1988g
					1	14	
					7	6.4	
					14	7.2	
					28	2.9	
Mortegliano, Italy, 1992	EC 570 g/l	1	1.1	0.07	0	0.24	Barbina, 1992c Barbina, 1993a
					7	<0.10	
					14	<0.10	
					21	<u>0.11</u>	
Lacieze, France (south), 1995	WP 300 g/kg	1	2.0	0.15	0	3.4	Partington, 1996c
					3	2.8(c0.03)	
					7	1.7	
					14	<u>1.2</u>	
					21	0.77	
					28	0.58(c0.04)	
Lacieze, France (south), 1995	EC 570 g/l	1	1.4	0.14	0	3.2	Partington, 1996c
					3	3.0(c0.03)	
					7	2.2	
					14	<u>1.9</u>	
					21	1.2	
					28	0.65(c0.04)	
Lacieze, France (south), 1995	EW 570 g/l	1	1.4	0.15	0	2.2	Partington, 1996c
					3	1.9(c0.03)	
					7	0.90	
					14	<u>0.99</u>	
					21	0.99	
					28	0.56(c0.04)	
Guarrigues, France (south), 1995	WP 300 g/kg	1	2.0	0.15	0	1.5(c0.17)	Partington, 1996c
					3	1.9(c0.13)	
					7	0.96(c0.12)	
					14	0.72(c0.08)	
					21	<u>0.73</u> (c0.08)	
					28	0.58(c0.07)	
Guarrigues, France (south), 1995	EC 570 g/l	1	1.4	0.14	0	1.3(c0.17)	Partington, 1996c
					3	1.2(c0.13)	
					7	0.76(c0.12)	
					14	<u>0.57</u> (c0.08)	
					21	0.42(c0.08)	
					28	0.23(c0.07)	
Guarrigues, France (south), 1995	EW 570 g/l	1	1.4	0.15	0	1.4(c0.17)	Partington, 1996c
					3	1.8(c0.13)	
					7	0.82(c0.12)	
					14	0.62(c0.08)	
					21	<u>0.80</u> (c0.08)	
					28	0.44(c0.07)	
Les Barthes, France (south), 1996	WP 315 g/kg	1	1.6	0.15	0	1.9	Partington, 1996d
					3	1.2	
					7	1.4	
					14	<u>0.89</u>	
					21	0.50	
					28	0.43	



Location, Year	Application				PHI Days	Residues, mg/kg	Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl			
Lagarrigue, France (south), 1966	WP 315 g/kg	1	1.8	0.15	0	2.7	Partington, 1996d
					3	2.4	
					7	2.1	
					14	<u>0.82</u>	
					21	0.67	
28	0.47						
Les Barthes, France (south), 1996	EW 524 g/kg	1	1.4	0.16	0	1.7	Partington, 1996d
					3	1.0	
					7	1.3	
					14	<u>0.86</u>	
					21	0.64	
28	0.51						
Lagarrigue, France (south), 1966	EW 524 g/kg	1	1.4	0.15	0	1.8	Partington, 1996d
					3	1.5	
					7	1.3	
					14	<u>0.87</u>	
					21	0.69	
28	0.51						
Hungary, 1994	EW 570 g/l	1	1.1	0.14	10	0.94	Toth, 1994
Hungary, 1991	WP 300 g/kg	1	1.1	0.18	7	0.69	O'Connell, 1992f

c: control

### Berries and other small fruits

**Strawberries.** In field trials at multiple locations in California, Florida and New York, USA, in 1985-1987 a WP formulation (300 kg/kg) was applied at rates of 1 or 3.4 kg ai/ha with PHIs of 1 or 3 days. Strawberries were analysed by gas chromatography with a flame photometric detector. The nominal limit of quantification was 0.05 mg/kg (Polakoff, 1988b).

In two further US trials in 1995 a WP formulation (30 g/kg) was applied twice at 14-day intervals at 1.5 kg ai/ha using tractor-mounted boom sprayers. Samples (1.3 kg) of fruit were harvested 3 days after the second treatment and stored frozen. The fruit was homogenized and extracted with hexane/2-propanol, and the extracts purified on a Florisil column for analysis by gas chromatography with a flame photometric detector. In a separate determination of TBPC, homogenized fruit was extracted with acetonitrile, derivatized, and purified on a SAX ion exchange column. The final extract was analysed by gas chromatography with an electron capture detector. The method was validated for strawberries at 0.1-7.5 mg/kg (78-92% recovery) for propargite and at 0.02-2.0 mg/kg (58-109%) for TBPC. The results are shown in Table 49 (Schuster and Korpalski, 1997).

Table 49. Residues of propargite and TBPC in strawberries, USA.

Location, Year	Application				PHI Days	Residues, mg/kg		Reference
	Form.	No.	kg ai/ha	kg ai/hl		propargite	TBPC	
Fresno, California, 1995	WP 300 g/kg	2	1.5	0.16	3	2.9 (c0.03)	0.20 (c0.02)	Schuster and Korpalski, 1997; propargite
Watsonville, California, 1995	WP 300 g/kg	2	1.5	0.16	3	3.0	0.15	Schuster and Korpalski, 1997; propargite
1987	WP 300 g/kg	3	1.0	0.10	1	2.9 (c0.07)	-	Polakoff, 1988b
					3	2.9		
1987	WP 300 g/kg	3	3.3	0.35	1	10. (c0.07)	-	Polakoff, 1988b
					3	15.		
1987	WP 300 g/kg	3	1.0	0.10	1	2.0	-	Polakoff, 1988b

Location, Year	Application				PHI Days	Residues, mg/kg		Reference
	Form.	No.	kg ai/ha	kg ai/hl		propargite	TBPC	
1987	WP 300 g/kg	3	1.0	0.10	1 3	1.1 1.1	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	3	1.0	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	1 3	4.2 3.0	-	Polakoff, 1988b
1987	WP 300 g/kg	3	1.0	0.10	1 3	2.6 (c0.11) 4.5	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	1 3	17. (c0.11) 14.1	-	Polakoff, 1988b
1987	WP 300 g/kg	3	1.0	0.10	1 3	9.0 6.1	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	1 3	16 23.	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	1 3	4.7 (c0.13) 4.7 (c0.13)	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	1 3	4.0 3.1	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	1 3	2.6 (c0.06) 1.2 (c0.06)	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	1 3	10. (c0.09) 14 (c0.09)	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	1 3	9.1 (c0.07) 12. (c0.07)	-	Polakoff, 1988b
1987	WP 300 g/kg	3	1.0	0.10	1 3	2.2 3.3	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	1 3	9.5 16	-	Polakoff, 1988b
1987	WP 300 g/kg	3	1.0	0.10	1	1.9 (c0.09)	-	Polakoff, 1988b;
1987	WP 300 g/kg	3	3.3	0.35	1	1.1	-	Polakoff, 1988b
1987	WP 300 g/kg	3	1.0	0.10	1 3	1.2 1.5	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	1 3	4.9 5.7	-	Polakoff, 1988b
1987	WP 300 g/kg	3	1.0	0.10	1 3	3.5 4.3	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	1 3	20 14	-	Polakoff, 1988b
1987	WP 300 g/kg	3	1.0	0.10	1 3	6.3 4.1	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	1 3	34. (c0.08) 22	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.3	0.35	1 3	4.4 4.7	-	Polakoff, 1988b
1987	WP 300 g/kg	3	7.0	0.75	1 3	5.7 9.3	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.33	0.35	1 3	7.8 7.1	-	Polakoff, 1988b
1987	WP 300 g/kg	3	7.0	0.65	1 3	4.4 5.9 (c0.10)	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.33	0.35	1 3	4.2 2.6	-	Polakoff, 1988b
1987	WP 300 g/kg	3	7.0	0.75	1 3	8.6 9.0	-	Polakoff, 1988b
1987	WP 300 g/kg	3	3.33	0.35	1 3	9.2 12.	-	Polakoff, 1988b
1987	WP 300 g/kg	3	7.0	0.75	1 3	21 24(c0.06)	-	Polakoff, 1988b

Location, Year	Application				PHI Days	Residues, mg/kg		Reference
	Form.	No.	kg ai/ha	kg ai/hl		propargite	TBPC	
1987	WP	3	3.3	0.35	1	10.	-	Polakoff, 1988b
	300 g/kg				3	22.		
1987	WP	3	7.0	0.75	1	15	-	Polakoff, 1988b
	300 g/kg				3	20		
1986	WP	3	3.3	0.54	3	27	-	Polakoff, 1988b
	300 g/kg							
1986	WP	3	3.3	0.54	3	15(c0.32)	-	Polakoff, 1988b
	300 g/kg							
1986	WP	3	1.0	0.15	1	0.93 (c0.10)	-	Polakoff, 1988b
	300 g/kg				3	0.65		
1986	WP	3	1.0	0.15	1	1.3	-	Polakoff, 1988b
	300 g/kg				3	0.66		
1986	WP	3	1.0	0.15	1	1.5	-	Polakoff, 1988b
	300 g/kg				3	0.48		
1986	WP	3	1.0	0.15	1	1.1	-	Polakoff, 1988b
	300 g/kg				3	0.55		
1985	WP	3	1.0	0.21	1	1.4	-	Polakoff, 1988b
	300 g/kg							
1985	WP	3	1.0	0.21	1	2.2	-	Polakoff, 1988b
	300 g/kg							
1985	WP	3	3.3	0.70	1	1.2	-	Polakoff, 1988b
	300 g/kg			3	0.54	18		
1985	WP	3	3.33	0.70	1	3.8	-	Polakoff, 1988b
	300 g/kg							
1985	WP	3	3.33	0.70	1	12.0	-	Polakoff, 1988b
	300 g/kg							
1985	WP	3	3.3	0.54	3	27 (c0.30)	-	Polakoff, 1988b
	300 g/kg							

c: control

**Blackcurrants.** In field trials in the UK in 1980 two plots in Rolvenden, Kent were treated twice with an EC formulation (570 g/l nominal) at a rate of 0.86 kg ai/ha. Currants were picked at intervals of 7-21 days after treatment and analysed by an HPLC procedure (Anon., 1980). In trials in 1981 at Bracklenham, Norfolk, UK, two plots were treated once with an unspecified formulation at 0.86 kg ai/ha. Samples were analysed by gas chromatography with a flame photometric detector. Recoveries from fortified controls (0.1-5.0 mg/kg) were acceptable (79-93%) (Anon., 1981, Table 50).

Table 50. Residues in blackcurrants, UK (Anon., 1980, 1981).

Location, Year	Application				PHI Days	Residues, mg/kg
	Form.	No.	kg ai/ha	kg ai/hl		
Rolvenden, Kent 1980	EC 570 g/kg	2	0.86		7	7.2 (c2.6)
					14	8.2 (c0.16)
					21	5.9 (c0.02)
						8.2 (c0.03)
Bracklenham, Norfolk, 1981	?	1	0.86	0.04	0	4.0
					7	1.8
					14	2.2
					21	0.81
						1.1
	0.71					
	2.8					
	0.25					

Grapes (Table 51). Field trials on grapes were reported from the Czech Republic, France, Hungary, Italy and the USA.

In the Czech Republic a 0.1% solution from an EC formulation (570 g/kg) was sprayed on vines until drip (1000 l/ha). Grape samples were taken on the day of treatment and at intervals up to 50 days. Sample extracts were analysed by gas chromatography with a flame photometric or electron capture detector (Anon., 1991).

In field trials in France in 1990-1996 vines were sprayed with EC or WP formulations, grapes were harvested at various intervals and stored frozen. The samples were analysed by GC-MS. The limit of quantification was 0.01 or 0.05 mg/kg.

In Hungary a WP formulation was applied once to the ripening crop, and grapes sampled at intervals from days 0-14 for analysis by gas chromatography with an electron capture detector (O'Connell, 1991a). Controls fortified at 0.1 mg/kg yielded recoveries in the range 76-106% (Table 51).

Field trials were also reported from Risano, Udine, Italy, in 1992 and 1993 (Barbina, 1992b, 1994b). Samples were analysed by HPLC with UV detection (225 nm). In additional trials at two locations in the province of Rome in 1994 HPLC was again used (Imbroglini, 1995a). The limit of quantification was 0.1 mg/kg.

In three field trials in the USA in 1987 a WP formulation was applied twice to vines in California (Polakoff, 1988k). The sites ranged from 100 to 700 vines. About 90 kg of grapes were harvested from each treated plot 21 days after the second application, and extracted for analysis by gas chromatography with a flame photometric detector. The limit of quantification was 0.05 mg/kg.

Two applications of a WP formulation of propargite were made 21 days apart to two plots of Thompson Seedless vines, when the grapes were ripening, in the San Joaquin Valley, California, USA, in 1998 at a rate of 3.0 kg ai/ha each (Korpalski, 1999a). A control plot was also maintained. Grapes were sampled at intervals until 28 days after the last treatment, and analysed by GC-MS for propargite and TBPC.

Additional field trials in California, USA, 1968-1970, were reported but insufficient information was supplied for evaluation.

Table 51. Residues of propargite in grapes.

Country, Year	Application				PHI Days	Residues		Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl		mg/kg propargite	mg/kg TBPC	
Czech Republic, 1987 <sup>1</sup>	EC 570 g/kg	1	1.0	0.1	0	0.89		Anon., 1991 c ≤ 0.05 mg/kg
					7	1.3		
					15	0.11		
					21	0.80		
					28	0.12		
					37	0.26		
					46	0.10		
52	<u>0.29</u>							
Theziers, France, 1991	EC 570 g/kg	1	0.86	0.43	0	1.2		O'Connell, 1992c.
					7	2.5		
					14	2.8		
					21	<0.05		
					28	0.7		
34	<u>2.4</u>							

Country, Year	Application				PHI Days	Residues		Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl		mg/kg propargite	mg/kg TBPC	
Theziers, France, 1991	WP 300 g/kg	1	0.92	0.46	0	1.4	O'Connell, 1992c.	
					7	2.1		
					14	<0.05		
					21	0.2		
					28	0.2		
					34	<u>0.3</u>		
Theziers, France, 1991	EW 570 g/kg	1	0.86	0.43	0	1.3	O'Connell, 1992c.	
					7	<0.05		
					14	1.4		
					21	<u>0.7</u>		
					28	0.3		
					34	0.6		
Fronton, France, 1991	EC 570 g/kg	1	0.86	0.43	0	0.5	O'Connell, 1992c.	
					7	1.6		
					14	0.6		
					21	<0.05		
					28	<0.05		
					34	<u>1.1</u>		
Fronton, France, 1991	WP 300 g/kg	1	0.92	0.46	0	0.4	O'Connell, 1992c.	
					7	<0.05		
					14	0.8		
					21	0.5		
					28	<u>0.8</u>		
					34	0.4		
Fronton, France, 1991	EW 570 g/kg	1	0.86	0.43	0	0.7	O'Connell, 1992c.	
					7	1.3		
					14	2.0		
					21	<u>0.6</u>		
					28	0.4		
					34	0.6		
Maraussan, France, 1990	WP 300 g/kg	1	0.90	0.45	0	2.1	Anon., 1990	
					7	1.1		
					14	2.4		
					21	1.1		
					28	<u>1.9</u>		
					34	0.9		
Maraussan, France, 1990	EC 570 g/kg	1	0.86	0.43	0	12	Anon., 1990	
					7	6.6		
					14	9.7		
					21	2.1		
					28	<u>2.7</u>		
					34	1.2		
Chancay, Indre et Loire, France, 1992	WP 300 g/kg	1	0.92	0.46	0	0.47	Partington, 1993b. 40 plants per plot; clay soil. Early ripening. Average of 3 replicates	
					7	0.41		
					14	0.34		
					21	<u>0.35</u>		
					28	0.21		
					35	0.18		
Centre Viti- Vinicole, Indre et Loire, France, 1992	WP 300 g/kg	1	0.92	0.46	0	0.31	Partington, 1993b 40 plants per plot; silt loam. Early ripening. Average of 3 replicates.	
					7	0.23		
					14	0.24		
					21	0.19		
					28	<u>0.23</u>		
					35	0.22		
Chancay, Indre et Loire, France, 1992 (north)	WP 300 g/kg	2	0.92	0.46	0	0.48	Partington, 1993b 40 plants per plot; clay soil. Ripe. Average of 3 replicates	
					7	0.51		
					14	0.50		
	EC 570 g/kg		21	<u>0.30</u>				
			28	0.20				
			35	0.19				

Country, Year	Application				PHI Days	Residues		Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl		mg/kg	mg/kg	
						propargite	TBPC	
Centre Viti- Vinicole, Indre et Loire, France, 1992 (north)	WP 300 g/kg EC 570 g/kg	2	0.92	0.46	0	0.34	Partington, 1993b 40 plants per plot; silt loam Ripe. Average of 3 replicates.	
					7	0.53		
					14	0.30		
					21	<u>0.29</u>		
					28	0.29		
35	0.24							
Chancay, Indre et Loire, France, 1992	WP 300 g/kg EW 570 g/kg	2	0.92	0.46	0	0.60	Partington, 1993b 40 plants per plot; clay soil. Ripe. Average of 3 replicates	
					7	0.71		
					14	0.40		
					21	<u>0.38</u>		
					28	0.20		
35	0.22							
Centre Viti- Vinicole, Indre et Loire, France, 1992	WP 300 g/kg EW 570 g/kg	2	0.92	0.46	0	0.30	Partington, 1993b 40 plants per plot; silt loam. Ripe. Average of 3 replicates.	
					7	0.28		
					14	0.21		
					21	<u>0.18</u>		
					28	0.15		
35	0.14							
Chinon, France, 1993 (north)	WP 300 g/kg	1	0.99	0.1	0	1.4	Partington, 1994b. Average of 3 replicates. 80 vines.	
					7	0.69		
					14	0.66		
					21	<u>0.67</u>		
					28	0.44		
35	0.47							
Panzoult, France, 1993 (north)	WP 300 g/kg	1	0.86	0.09	0	0.65	Partington, 1994b. Average of 3 replicates. 80 vines.	
					7	0.48		
					14	0.26		
					21	<u>0.28</u>		
					28	0.27		
35	0.19							
Chinon, France, 1993 (north)	EW 570 g/kg	1	0.82	0.08	0	1.6	Partington, 1994b. Average of 3 replicates. 80 vines. 80 vines.	
					7	1.3		
					14	0.89		
					21	0.83		
					28	0.84		
35	<u>0.93</u>							
Panzoult, France, 1993 (north)	EW 570 g/kg	1	0.95	0.09	0	0.70	Partington, 1994b Average of 3 replicates. 80 vines.	
					7	0.65		
					14	0.43		
					21	<u>0.51</u>		
					28	0.39		
35	0.38							
Chancy, France, 1995 (north)	EW 226 g/kg propargite 38 g/kg tetradifon	1	0.86	0.43	0	0.94	Partington, 1996e. Average of 3 replicates. 40 vines in single row.	
					7	0.76		
					14	0.50		
					21	<u>0.45</u>		
					28	0.41		
35	0.23							
Le Bois Vieux, France, 1995 (south)	EW 240 g/l propargite 40 g/l tetradifon	1	0.86	0.43	0	1.9	Partington, 1996e Average of 3 replicates. 40 vines in single row.	
					7	1.4		
					14	1.1		
					21	<u>0.96</u>		
					28	0.93		
35	0.92							
Alsace (Westhalten), France, 1996 (north)	EW 226 g/kg (240 g/l nominal) propargite 38 g/kg tetradifon	1	0.7	0.44	0	0.33	Milbach, 1997. Average of 3 replicates.	
					21	<u>0.11</u>		

Country, Year	Application				PHI Days	Residues		Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl		mg/kg propargite	mg/kg TBPC	
Franche-Comte, France, 1996 (north)	EW 226 g/kg (240 g/l nominal) propargite 38 g/kg tetradifon	1	0.9	0.45	0 21	0.62 <u>0.18</u>		Milbach, 1997 Average of 3 replicates.
Bordeaux, France, 1996 (south)	EW 226 g/kg (240 g/l nominal) propargite 38 g/kg tetradifon	1	0.9	0.50	0 21	1.3 <u>0.93</u>		Milbach, 1997 Average of 3 replicates.
Rhone Valley, France, 1996 (south)	EW 226 g/kg (240 g/l nominal) propargite 38 g/kg tetradifon	1	0.8		0 21	0.62 <u>0.29</u>		Milbach, 1997. Average of 3 replicates.
Balatonboglár, H ungary, 1991	WP 300 g/kg	1	0.9	0.13	0 7 14	1.1 0.65 <u>0.36</u>		O'Connell, 1991a
Risano, Udine, Italy 1992	EC 578 g/kg	1	1.1	0.11	0 7 14 21	1.2, 0.40, 0.59 <0.10, 0.14, <0.10 <u>&lt;0.10 (3)</u> <0.10, 0.34, <0.10		Barbina, 1993b. Three replicate plots.
Risano, Udine, Italy 1993	EC 570 g/kg	1	0.86	0.09	1 3 7 14	0.39, 1.1, 1.6 0.50, 0.59, 0.68 0.23, <0.10, 0.14 <0.10 (2), <u>0.26</u>		Barbina, 1994b. 8 plants/plot. Three replicate plots.
Risano, Udine, Italy 1993	EW 570 g/kg	1	0.86	0.09	1 3 7 14	1.4, 1.7, 2.0 0.98, 1.1, 1.2 1.2, 0.33, 0.21 <u>0.31</u> , 0.18, <0.10		Barbina, 1994b. 8 plants/plot. Three replicate plots.
Tormancina- Monterotondo, Rome, Italy	EC 570 g/kg	1	0.86	0.06	7 14	1.0, 0.97 0.46, <u>0.48</u>		Imbroglini, 1995a Plot 10 x 1.5 m Two replicate plots
Campo Marinaro- Anguillara Sabazia, Rome, Italy	EC 570 g/kg	1	0.86	0.06	7 14	0.88, 0.72 0.30, <u>0.33</u>		Imbroglini, 1995a. Plot 10 x 1.5 m Two replicate plots
California, USA, 1998	WP 300 g/kg	2	3.0	0.80	7 14 21 28	1.4 0.34 <u>0.49</u> 0.29	0.11 0.40 0.31 0.05	Korpalski, 1999a
California, USA, 1987	WP 300 g/kg	2	3.0	0.80	21	<u>1.3</u>		Polakoff, 1988k
California, USA, 1987	WP 300 g/kg	2	3.0	0.80	21	<u>3.4</u>		Polakoff, 1988k

Country, Year	Application				PHI Days	Residues		Reference, remarks
	Form.	No.	kg ai/ha	kg ai/hl		mg/kg propargite	mg/kg TBPC	
California, USA, 1987	WP 300 g/kg	2	3.0	0.80	21	4.8		Polakoff, 1988k

<sup>1</sup> data reported in a summarized form

### Assorted tropical and sub-tropical fruits

Avocados. In US field trials in 1995 three applications of a WP formulation (300 g/kg) were made at 5.0 kg ai/ha (1900 l/ha) to two sites in California at 14-day intervals, using commercial ground airblast equipment. Each plot contained a minimum of 16 trees. 28 days after the last application four replicate samples were harvested from each treated plot (minimum of 24 avocados picked from all sections of the tree). Samples were frozen for up to 3 months, then subdivided in the laboratory, half being pitted and half peeled and pitted, for extraction with hexane. The extracts were partitioned with acetonitrile and cleaned up by gel permeation chromatography on a Florisil column. Analysis was by gas chromatography with a flame photometric detector. The demonstrated limit of quantification was 0.05 mg/kg propargite (Korpalski, 1996b, Table 52).

Table 52. Residues of propargite in avocados without stone, California, USA, 1995 (Korpalski, 1996b).

Location	Application				PHI Days	Residues, mg/kg	
	Form.	No.	kg ai/ha	kg ai/hl		Whole	Peeled
Riverside County	WS <sup>1</sup>	3	5.1	0.26	29	0.19	0.05
	300 g/kg		5.0				
			5.0				
Ventura County	WS <sup>1</sup>	3	5.2	0.26	28	1.2	0.14
	300 g/kg		5.0				
			5.0				

<sup>1</sup> WP in a water-soluble bag

### Fruiting vegetables, cucurbits

Cucumbers. Field trials were reported from Italy and Hungary but information on the Italian trials was contradictory and could not be evaluated (Barbina, 1993c). Field conditions (EC or WP, field or glasshouse, year of trial) did not match the analytical reports. In Hungary vines in a glasshouse were treated with a WP formulation (300 g ai/kg) at a rate of 1.1 kg ai/ha in 1991 (spray volume unknown). Samples were collected 3 and 5 days after treatment and stored at -20°C. The sample extracts were analysed by gas chromatography with a flame photometric detector in the sulfur mode. The demonstrated limit of quantification was 0.25 mg/kg (O'Connell, 1992g).

The laboratory analysis of field trial samples was reported from the USA, but no information was supplied on the field phase (Richards, 1987).

Melons. In a field trial on melons (Talma variety) in France one application of a WP formulation (300 g/kg) was made with a boom sprayer to a commercial crop in the Loire valley (Cowley, 1994; Gill, 1994). The plants were at various growth stages from mid-flower to 15 cm diameter melons on the 2 m x 30 m plot. Samples (6 melons) taken at various intervals were stored at -15 to -22°C for about 200 days, extracted with methanol/hexane, purified by partition and analysed by GC-MS with a capillary column in the splitless mode. The ions monitored were 173, 201 and 350. The procedure was



validated at control fortifications of 0.02 (75%), 0.10 (77%) and 1.0 (81%) mg/kg propargite. All controls were <0.02 mg/kg.

Table 53. Residues of propargite in cucumbers and melons.

Location, Year	Application				PHI Days	Residues, mg/kg	Reference, comments
	Form.	No.	kg ai/ha	kg ai/hl			
<i>Cucumber</i>							
Hungary, 1991	WP 300 g/kg	1	1.1		3	1.7	O'Connell, 1992g Glasshouse
					5	0.78	
<i>Melon</i>							
Loire valley, North, France, 1993	WP 300 g/kg	1	0.86	0.17	0	0.38	Cowley, 1994; Gill, 1994
					3	0.16	
					7	0.10	
					10	0.06	
					14	<u>0.05</u>	

#### Fruiting vegetables other than cucurbits

Peppers. A WP (300 g/kg) formulation of propargite was applied to pepper (paprika) plants in Hungary in 1991 at a rate of 1.1 kg ai/ha. Samples were taken 3 and 5 days after treatment and stored frozen until analysed by gas chromatography with a flame photometric detector. The demonstrated limit of quantification was 0.25 mg/kg, with 60% recovery (Table 54, O'Connell, 1991b).

Table 54. Residues of propargite in peppers, Hungary, 1991 (O'Connell, 1991b).

Application				PHI Days	Residues, mg/kg <sup>1</sup>
Form.	No.	kg ai/ha	kg ai/hl		
WP 300 g/kg	1	1.1	0.18	3	4.2
				5	<u>2.7</u>

<sup>1</sup> corrected for recovery

Tomatoes. In a glasshouse trial in central west France (Tours, Loire Valley) a WP formulation (306 g/kg) was applied with a small plot sprayer to a 60 square m section of plants in 500 l water/ha at 0.99 kg ai/ha. Tomato samples were taken on the day of application and at various intervals and analysed by GC-MS (Partington, 1994c).

Several trials were reported from Italy for the period 1992-1999. In a trial in 1992 at Scaluninco-Lestizza, Province of Udine, two plots were treated with a WP and two with an EW formulation applied with a backpack with 5 hl of water/ha, 0.22 kg ai/hl (WP) and 5 hl of water/ha, 1.1 kg ai/ha (EC). One plot with each formulation was treated once and the other received two treatments at a 14-day interval. Tomato samples were stored frozen and analysed by HPLC with a UV detector. The method was validated at 0.8 mg/kg for tomatoes, but a limit of quantification of 0.1 mg/kg was claimed (Barbina, 1992d,e).

In trials in 1994 at Monterotondo and Anguillara Sabazia, in Rome, Italy, two applications of an EC formulation (570 g/kg) were applied by backpack sprayer (1500 l/ha of water, spray concentration 0.057 kg ai/hl) (Imbroglini, 1995b). 12-24 tomatoes from 12 randomly selected plants were picked 7 and 14 days after the second application, which was made at early fruit ripening. The samples were stored frozen for analysis by HPLC with UV detector. The method was validated at 0.40-1.9 mg/kg, with a recovery at 0.40 mg/kg of 70%. Additional Italian trials were conducted at Mortigliano and Lestizza, Udine, also in 1994 (Barbina, 1994c). Each site consisted of three plots

with 20 plants in each. The plots were sprayed twice by means of a backpack pump unit at a rate of 0.86 kg ai/ha in 10 hl/ha of water: one plot at each site with an EW (570 g/kg) and one plot with an EC formulation (570 g/kg) at intervals of 14 or 19 days. Samples of 24 fruits were collected randomly from 12 central plants and stored frozen until analysis by HPLC with a UV detector. The method was validated with 0.93 and 1.9 mg/kg fortified controls.

In trials in Lestizza, Udine, Italy, in 1995, EC, EW and WP formulations were applied twice with a backpack pump at an interval of 14 days to separate plots, each consisting of 20 plants. The EC and EW formulations were applied at a rate of 0.86 kg ai/ha, and the WP at 0.90 kg ai/ha (all 10 hl/ha spray mixture). Samples (12-24 fruits) were collected at random from the 12 central plants and stored frozen until analysis. A GC-MS procedure was employed for quantification of the propargite residues. The method was validated at 0.01 mg/kg and 1.0 mg/kg, 98-116% recovery (Partington, 1996f).

Two Italian trials were reported from Provincia d'ell'Emilia Romagna in 1997 (Cawkwell, 1999) and Mezzano di Alfonsine in 1998 (Harrison, 1999). Two applications of an EW formulation were made at 0.86 kg ai/ha in 1500 l/ha of water by means of a plot boom sprayer with an interval of 14 days. Samples (12 fruits minimum) were taken at random and stored frozen. Samples were extracted with an acetonitrile/hexane solvent mixture and the extracts purified by liquid-liquid partition and gel permeation chromatography. Propargite was determined by GC-MS. Procedural recoveries were run concurrently with the samples over a fortification range of 0.01-5 mg/kg, mean recovery 88%. A large sample was taken for processing.

The analytical part only of a field trial was reported by Blango (1993). Apparently an EC formulation was applied in a single trial somewhere in the USA at a rate of 1.0 kg ai/ha and tomatoes were harvested 3, 5 and 7 days later. The single treated plot was subdivided into 4 subplots for sampling purposes and there was a control plot. Samples were stored frozen in the laboratory for 8 months, then solvent-extracted and the extracts cleaned up on a Florisil column before analysis by gas chromatography with a flame photometric detector in the sulfur mode. Recoveries at 0.05 mg/kg fortification were 78 and 92% and at 1 mg/kg 95, 63 and 70%. The interval from harvest to arrival at the laboratory was not reported.

Table 55. Residues of propargite in tomatoes.

Location, Year	Application				PHI Days	Residues, mg/kg	Reference, comment
	Form.	No.	kg ai/ha	kg ai/hl			
Tours, France, 1993	WP 300 g/kg	1	0.92	0.18	0	0.99	Partington, 1994c Glasshouse Controls <0.01 mg/kg
					3	1.0	
					7	0.48	
					10	0.54	
					14	0.67	
Scalunicco- Lestizza, Italy, 1992	WP 300 g/kg	1	1.1	0.22	0	0.65	Barbina, 1992d Controls <0.1 mg/kg
					7	0.14	
					14	<0.10	
					21	<u>0.14</u>	
Scalunicco- Lestizza, Italy, 1992	WP 300 g/kg	2	1.1	0.22	0	0.32	Barbina, 1992d Controls <0.1 mg/kg. Sampled 14 d after 1st treatment. GAP is single treatment.
					7	<0.10	
					14	<u>&lt;0.10</u>	
					21	<0.10	
Scalunicco- Lestizza, Italy, 1992	EW 570 g/kg	1	1.1	0.22	0	1.1	Barbina, 1992e Controls <0.1 mg/kg
					7	0.50	
					14	<u>0.28</u>	
					21	<0.10	

Location, Year	Application				PHI Days	Residues, mg/kg	Reference, comment
	Form.	No.	kg ai/ha	kg ai/hl			
Scalunicco- Lestizza, Italy, 1992	EW 570 g/kg	2	1.1	0.22	0	0.27	Barbina, 1992e Controls <0.1 mg/kg Sampled 14 d after 1st treatment. GAP is single treatment.
					7	<0.10	
					14	<0.10	
					21	<0.10	
Monterotondo, Rome, Italy, 1994	EC 570 g/kg	2	0.86	0.057	7	1.7	Imbroglini, 1995b Controls <0.1 mg/kg
					14	<u>1.4</u>	
Anguillara Sabazia, Rome, 1994	EC 570 g/kg	2	0.86	0.057	7	1.8	Imbroglini, 1995b Controls <0.1 mg/kg
					14	<u>1.4</u>	
Mortegliano, Udine, Italy 1994	EC 570 g/kg	2	0.86	0.086	1	0.68	Barbina, 1994c Controls <0.1 mg/kg
					3	0.30	
					7	<u>0.14</u>	
					14	<0.10	
Mortegliano, Udine, Italy 1994	EW 570 g/kg	2	0.86	0.086	1	0.95	Barbina, 1994c Controls <0.1 mg/kg
					3	0.52	
					7	0.17	
					14	<0.10	
Lestizza, Udine Italy, 1994	EC 570 g/kg	2	0.86	0.086	1	0.43	Barbina, 1994c Controls <0.1 mg/kg
					3	<0.10	
					7	<0.10	
					14	<0.10	
Lestizza, Udine Italy, 1994	EW 570 g/kg	2	0.86	0.086	1	0.41	Barbina, 1994c Controls <0.1 mg/kg
					3	<0.10	
					7	<0.10	
					14	<0.10	
Lestizza, Udine Italy, 1995	EC 570 g/kg	2	0.86	0.086	-0	0.05	Partington, 1996f -0: just before second treatment Controls <0.01 mg/kg
					1	0.93	
					3	0.33	
					7	0.43	
					14	<u>0.27</u>	
Lestizza, Udine Italy, 1995	EW 570 g/kg	2	0.86	0.086	-0	0.07	Partington, 1996f -0: just before second treatment Controls <0.01 mg/kg
					1	0.79	
					3	0.51	
					7	0.44	
					14	<u>0.29</u>	
Lestizza, Udine Italy, 1995	WP 300 g/kg	2	0.90	0.09	-0	0.04	Partington, 1996f -0: just before second treatment Controls <0.01 mg/kg
					1	0.52	
					3	0.38	
					7	0.43	
					14	<u>0.27</u>	
Provincia dell'Emilia Romagna, Italy, 1997	EW 570 g/kg	2	0.86	0.06	15	<u>0.23</u>	Cawkwell, 1999 Control 0.02 mg/kg
Mezzano di Alfonsine, Italy 1998	EW 570 g/kg	2	0.86	0.06	15	<u>0.17</u>	Harrison, 1999 Control <0.01 mg/kg
US, 1992	EC	3	1.0	-	3 5 7	0.33, 1.5, 1.1, 1.3 (1.0) 0.22, 0.23, 1.2 (0.55) <0.050, 1.2, 0.37, 0.12 (0.44)	Blango, 1993. No field data. Replicate samples

Egg plant. Laboratory work on eight samples was conducted in 1988-1990 in the USA, but no information was supplied on the field part of the study (Beadle, 1990).

## Pulses

Soya beans (Table 56). In a trial in Hungary an EC formulation (570 g/kg) was applied at 0.91 kg ai/ha to green pods, and triplicate samples taken 10, 14 and 21 days later for analysis by gas chromatography with an electron capture detector (O'Connell, 1992d).

In a trial in the Czech Republic a single spray of an EC formulation (570 g/kg) at 0.25 kg ai/ha was applied to plants 3 weeks after flowering. Samples were taken at intervals of 0-14 days and analysed by gas chromatography with a flame photometric or electron capture detector (Anon., 1991).

A field trial was reported from Italy, where an EC formulation (570 g/kg) was applied in the Veneto region with a knapsack sprayer to 20 m<sup>2</sup> plots. Applications were made in 2000 l/ha at rates of 0.09 kg ai/hl, 0.20 kg ai/hl and 0.36 kg ai/hl (Anon., 1989).

Field trials at eight locations were reported from the USA, where an EC formulation (730 g/kg, 0.78 kg ai/l) was applied once to the beans in 1989, either with a pressurized hand-held or tractor-mounted boom sprayer at the rate of 1.8 kg ai/ha, 190-240 l/ha. The beans were harvested 59-60 days later and seed samples were immediately frozen. Samples were solvent-extracted, cleaned up by partitioning and Florisil column chromatography and analysed by gas chromatography with a flame photometric detector. Adequate recovery was demonstrated with fortified controls, 0.05-1 mg/kg. All control samples were <0.05 mg/kg (Popadic, 1991c).

Table 56. Residues of propargite in soya bean seed, all single applications.

Location, Year	Application			PHI Days	Residues, mg/kg	Reference
	Form.	kg ai/ha	kg ai/hl			
Hollandale, Minnesota, USA, 1989	EC 750 g/kg	1.8	0.97	60	<0.05	Popadic, 1991c
Ames, Iowa, USA, 1989	EC 750 g/kg	1.8	0.96	60	<0.05	Popadic, 1991c
Muscatine, Iowa, USA, 1989	EC 750 g/kg	1.8	0.96	59	<0.05	Popadic, 1991c
Noblesville, Indiana, USA, 1989	EC 750 g/kg	1.8	0.75	59	<0.05	Popadic, 1991c
Hawkinsville, Georgia, 1989	EC 750 g/kg	1.8	0.84	60	<0.05	Popadic, 1991c
Senatobia, Mississippi, USA, 1989	EC 750 g/kg	1.8	0.96	60	<0.05	Popadic, 1991c
Steele, Missouri, USA, 1989	EC 750 g/kg	1.8	0.94	60	<0.05	Popadic, 1991c
Bethany, Illinois, USA, 1989	EC 750 g/kg	1.8	0.96	60	<0.05	Popadic, 1991c
Czech Republic, 1990	EC 570g/kg	0.25	0.05	0	3.9	Anon., 1991
				7	2.0	
				14	<0.05	
Hungary, 1991	EC, 570g/kg	0.91	0.25	10	0.53*	O'Connell, 1992d
				14	0.31*	
				21	0.18*	
Italy, 1989	EC, 570 g/kg	1.8	0.09	14	0.28	Anon., 1989
		3.9	0.20	14	0.20	
		7.2	0.36	14	0.26	

\* corrected for recovery (75-80%).

Kidney beans. An EC formulation (730 g/kg) was twice sprayed on Red Kidney beans at a rate of 2.8 kg ai/ha to two plots of 10 rows (18 m long) in California, USA, in 1989. The first application was at lay-by and the second 22 days later. The spray volume was 190 l/ha and the plants were harvested 14 days after the second treatment, field-dried for 14 days and thrashed. Beans were analysed by gas chromatography with a flame photometric detector. The limit of quantification was 0.05 mg/kg, as demonstrated by a fortification recovery of 76% (Popadic, 1991a).

Table 57. Residues of propargite in dry beans from the application of an EC formulation in California, USA (Popadic, 1991a).

Year	Application				PHI Days	Residues, mg/kg
	Form.	No.	kg ai/ha	kg ai/hl		
1995	75EC	2	2.8	1.5	14	<u>0.11</u>

#### Root and tuber vegetables

Potatoes. Two applications of an EC formulation of propargite at a rate of 2.5 kg ai/ha were applied by irrigation to Russel Burbank potatoes in trials in Washington and Idaho, USA, and the potatoes were dug 14 days later. The treated plots were a minimum of 0.5 ha. The vines were irrigated (0.25 to 0.51 cm water) by sprinkler during each application. The stage at the second treatment was vine casting in Washington and near maturity in Idaho. Potatoes were analysed by gas chromatography with a flame photometric detector within 55 days of harvest. The limit of quantification was 0.05 mg/kg (recovery 100, 104%). The results are shown in Table 58 (Popadic, 1992c).

Table 58. Residues of propargite in potatoes from chemigation in the USA in 1990 (Popadic, 1992c)

Location	Application				PHI Days	Residues, mg/kg
	Form.	No.	kg ai/ha	kg ai/hl		
Washington	EC 730 g/kg	2	2.5	Chemigation	14	<u>&lt;0.05</u>
Idaho	EC 730 g/kg	2	2.5	Chemigation	14	<u>&lt;0.05</u>

#### Cereal grains

Maize. In a field trial in France plants were treated twice with an EC formulation (570 g/l) of propargite at 1.4 kg ai/ha. Four plants per plot of 6 rows (84 m<sup>2</sup>) were harvested 41 days after the second treatment (pre-flowering) (Truchot, 1988).

In field trials in the USA in 1989 an EC formulation (785 g ai/l, 750 g/kg) was applied once by pivot irrigation during the milk stage of growth to one site in Georgia and one in Texas at 2.8 kg ai/ha. The crop was harvested 30 or 31 days later and 3 samples of 1.1 kg each were taken at each site and stored frozen. The grain was analysed by gas chromatography with a flame photometric detector. Recoveries at 0.05 mg/kg fortification were 100 and 101% (Korpalski, 1990a).

In 1990 single applications of an EC formulation (750 g/kg) were made to sites in Washington and Nebraska, USA, at 2.8 kg ai/ha by overhead irrigation at the hard-dough growth stage in 0.9 cm water/ha. Thirty days later three maize samples (2.3 kg each) were taken at each location and frozen for analysis by gas chromatography with a flame photometric detector (Popadic, 1991d).

In 1989 a single application of an EC formulation (750 g/kg) was made to maize in Wisconsin, USA, at a rate of 2.8 kg ai/ha in 190 l/ha of water by ground boom. Thirty days later, the crop was harvested by hand and stored frozen until analysis by gas chromatography with a flame photometric detector (Polakoff, 1990).

In two trials in the USA in 1995 at two field sites, one in Colorado and the other in Kansas, an EC formulation (680 g/kg) was applied twice at a rate of 1.9 kg ai/ha. The first application was with a

ground boom sprayer in a volume of 190 l/ha; the second aerial in 50 l/ha at the early dent growth stage. Grain was collected at normal harvest, 30 days after the second application and stored frozen for about 180 days. The grain was extracted with hexane/2-propanol, exchanged to acetonitrile, cleaned up on Florisil and alumina columns and analysed on a gas chromatograph with flame photometric detector. The method was validated at 0.05 mg/kg (Korpalski, 1997c).

In trials on maize at two locations in Texas, USA, in 1995 two applications were made of an EC formulation (680 g/kg, 720 kg/l), the first by ground-boom sprayer in 190 l/ha of water at 1.3 kg ai/ha and the second aerial in 50 l/ha of water at 1.9 kg ai/ha. The grain was harvested 30 days after the second application, and samples frozen for extraction with hexane/2-propanol. After Florisil column chromatography, the extracts were analysed on a gas chromatograph with a flame photometric detector. The demonstrated limit of quantification was 0.05 mg/kg (Korpalski, 1996c).

Numerous trials on maize in the USA in 1979 and 1980 were reported. No details were provided and the PHIs were generally 60 days or more (Anon., 1998).

Table 59. Residues of propargite in maize grain after foliar treatment.

Location Year	Application				PHI Days	Residues, mg/kg	Reference; analytical method
	Form.	No.	kg ai/ha	kg ai/hl			
USA (Colorado), 1995	EC 680 g/kg	2	1.9	1 ground 3.8 aerial	30	<0.05	Korpalski, 1997c; GC/FPD
USA (Kansas), 1995	EC 680 g/kg	2	1.9	1 ground 3.8 aerial	30	<0.05	Korpalski, 1997c; GC/FPD
USA (Uvalde, Texas), 1995	EC 680 g/kg	2	1.3 1.9	0.68 ground 3.8 aerial	29	<0.05	Korpalski, 1996c; GC/FPD
USA (Lockney, Texas), 1995	EC 680 g/kg	2	1.3 1.9	0.68 ground 3.8 aerial	28	<0.05	Korpalski, 1996c; GC/FPD
USA (Washington), 1991	EC 750 g/kg	1	2.8	chemigation	30	<0.05	Popadic, 1991d; GC/FPD
USA (Nebraska), 1991	EC 750 g/kg	1	2.8	chemigation	30	<0.05	Popadic, 1991d; GC/FPD
USA (Georgia), 1989	EC 785 g/l 750 g/kg	1	2.8	chemigation	30	0.06	Korpalski, 1990a; GC/FPD
USA (Texas), 1989	EC 785 g/l 750 g/kg	1	2.8	chemigation	31	<0.05	Korpalski, 1990a; GC/FPD
USA (Wisconsin), 1989	EC 750 g/kg	1	2.8	1.5	30	<0.05	Polakoff, 1990; GC/FPD
France, 1988	EC 570 g/l	2	1.4	0.26	41	<0.01	Truchot, 1988; GC/ECD

**Sorghum.** In a 1990 US trial an EC formulation (730 g/kg) was applied at a rate of 1.8 kg ai/ha in 190 l of water per ha and the grain harvested at maturity 30 days later (Popadic, 1991b), and in 1991 the same formulation was applied once at the same rates and the beans harvested again 30 days later (Popadic, 1993o). In a 1995 trial in Texas an EC formulation was applied to sorghum at the same rate of 1.8 kg ai/ha in 190 l of water per ha and the beans harvested 60 days later (Korpalski, 1995d). All samples from the three studies were stored frozen and analysed by gas chromatography with a flame photometric detector in the sulfur mode (Table 60).

Table 60. Residues of propargite in sorghum in US trials.

Location, Year	Application				PHI Days	Residues, mg/kg grain	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
Texas, 1995	EC 750 g/kg	1	1.8	0.95	59	<0.05	Korpalski, 1995d

Location, Year	Application				PHI Days	Residues, mg/kg grain	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
Nebraska, 1991	EC 750 g/kg	1	1.8	0.95	30	3.0	Popadic, 1993o
Texas, 1990	EC 750 g/kg	1	1.8	1.0	30	0.77	Popadic, 1991b

### Tree nuts

**Almonds.** Trees at various sites in California, USA, were treated with a CR or E formulation of propargite in 1976–1987 (Polakoff, 1988a). Plots were typically 0.4 ha and two applications were made at 3.4 or 5.0 kg ai/ha, with PHIs of 14 and/or 28 days and both kernels and hulls were analysed. Recovery information was supplied for the 1986–1987 trials only, and only summary information was provided for trials before 1986. The results are shown in Table 61.

In two trials in California, USA, one in 1994 and the other in 1995, trees were sprayed twice by commercial airblast equipment with an aqueous mixture at about 470 l/ha with an interval of 21–27 days (Korpalski, 1997b). Each trial was with 1 control plot and two treated plots of at least 12 trees. Samples were taken at the normal PHI, separated by hand into hulls and kernels and stored frozen (minimum  $-8^{\circ}\text{C}$ ). Two samples were taken at each PHI from the treated plots and one from the control plot, each sample at least 1 kg. The analytical procedure included GPC and Florisil clean-up of the extracts. The final extracts were analysed by GLC with a flame photometric detector in the sulfur mode. A 0.53 mm capillary column was used for some of the work. Limits of quantification of 0.05 mg/kg propargite for kernels and 2 mg/kg for hulls were demonstrated. The results are shown in Table 61.

Table 61. Residues of propargite in almonds after two treatments of trees in California, USA.

Year	Application			PHI Days	Residues, mg/kg		Reference; analytical method
	Form.	kg ai/ha	kg ai/hl		kernels	hulls	
1994	EC 680g/kg	3.4	0.7	15	0.066 0.082	44 (c2.0) 27	Korpalski, 1997b; GC/FPD
				22	<u>0.05</u> <0.05	<u>30</u> (c0.53) 18	
1995	EC 680g/kg	3.4	0.7	21	0.060 0.068	30 (c0.06) 39	Korpalski, 1997b; GC/FPD
				28	<u>0.076</u> <0.05	<u>35</u> (c0.31) 37	
1987	WP 300g/kg	5.0	0.5	14	<0.05	28	Polakoff, 1988a; GC/FPD
				28	<u>&lt;0.05</u>	<u>14</u>	
1987	EC 680g/kg	3.4	0.7	14	<0.05	27	Polakoff, 1988a; GC/FPD
				28	<u>&lt;0.05</u>	<u>12</u>	
1986	WP 300g/kg	5.0	0.5	14	<u>&lt;0.05</u>	12 (c0.12)	Polakoff, 1988a; GC/FPD
				28	<u>&lt;0.05</u>	<u>15</u> (c0.12)	
1986	WP 300g/kg	5.0	1.3	28	0.06	13 (c0.13)	Polakoff, 1988a; GC/FPD
1979	EC 680g/kg	3.4	0.2	14	<u>0.05</u>	26	Polakoff, 1988a; GC/FPD
1979	EC 680g/kg	3.4	0.7	14	<u>&lt;0.05</u>	33	Polakoff, 1988a; GC/FPD
1979	EC 680g/kg	3.4	0.7	14	<u>&lt;0.05</u>	12	Polakoff, 1988a; GC/FPD
1979	EC 680g/kg	3.4	0.7	14	<u>&lt;0.05</u>	12	Polakoff, 1988a; GC/FPD

Year	Application			PHI Days	Residues, mg/kg		Reference; analytical method
	Form.	kg ai/ha	kg ai/hl		kernels	hulls	
1979	EC 680g/kg	3.4	0.7	14	<0.05	12	Polakoff, 1988a; GC/FPD
1979	EC 680g/kg	6.7	1.4	14	<0.05	27	Polakoff, 1988a; GC/FPD
1979	EC 680g/kg	3.4	0.7	14	0.08	27	Polakoff, 1988a; GC/FPD
1979	EC 680g/kg	3.4	0.7	14	0.07	40	Polakoff, 1988a; GC/FPD
1978	EC 680g/kg	3.4	0.7	0	0.58	64	Polakoff, 1988a; GC/FPD
1978	EC 680g/kg	3.4	0.7	0	0.97	95	Polakoff, 1988a; GC/FPD
1977	EC 680g/kg	3.4	0.7	14	0.09	32	Polakoff, 1988a; GC/FPD
1977	EC 680g/kg	3.4	0.7	14	<0.05	21 <sup>1</sup>	Polakoff, 1988a; GC/FPD
1976	EC 680g/kg	3.4	0.7	14	<0.05	29	Polakoff, 1988a; GC/FPD

c: control

<sup>1</sup> value of 150 mg/kg from a replicate sample is treated as an outlier.

**Filberts.** Three field trials were reported for the foliar application of an EC formulation to trees in Oregon, USA, in the 1996 growing season (Korpalski, 1998). Each site consisted of a control plot and two treated plots. The treated plots were sprayed twice by ground air-blast equipment, with 470 or 940 l/ha at 2.5 kg ai/ha or 5.0 kg ai/ha at 17–27 day intervals, in early spring when no nuts were on the trees. Harvest was 163–188 days after the second application. Two samples of nuts from each plot were cracked open about 13 days after harvest, yielding about 1 kg kernel per sample. The kernels were frozen immediately, stored for 8 months, and extracted with hexane. The extract was partitioned with acetonitrile to remove oils and cleaned up by GPC and Florisil chromatography. The acetone/hexane eluate was analysed by GLC using a flame photometric detector in the sulfur mode. The demonstrated limit of quantification was 0.05 mg/kg propargite in the kernels (recoveries 107, 103, 102%). The results are shown in Table 62.

Table 62. Residues of propargite in filbert kernels after two foliar treatments in the USA in 1996 with an EC formulation, 680g/kg (Korpalski, 1998).

Location	Application		PHI (days)	Propargite (mg/kg)
	kg ai/ha	kg ai/hl		
Mulino, Oregon	2.5	0.53	188	<0.05
		0.27		
	5.0	0.53	188	<0.05
		0.27		
Hubbard, Oregon	2.5	0.53	163	<0.05
		0.27		
	5.0	0.53	163	<0.05
		0.27		
Eugene, Oregon	2.5	0.53	174	<0.05
		0.27		
	5.0	0.53	174	<0.05
		0.27		

**Pecans.** A 680 g/kg EC formulation of propargite was applied twice by ground airblast equipment at 3.4 kg ai/ha, 470 l/ha, PHI 14 days, to pecan trees in Georgia (2 locations), Louisiana, Alabama and Texas, USA, in 1990 (Popadic, 1992g). The nuts were cracked mechanically, shelled mechanically or



manually, and the kernels stored frozen. Samples were extracted and the extracts cleaned up by Florisil column chromatography before analysis by gas chromatography with a flame photometric detector in the sulfur mode. A limit of quantification of 0.1 mg/kg was demonstrated (recoveries 93, 91 and 77%). The samples that had quantifiable residues were from crops that were treated for the second time during the shuck split maturity stage. The results are shown in Table 63.

Table 63. Residues of propargite in pecan kernels after two foliar applications of an EC formulation at 680 g ai/kg, 14-day PHI, in the USA in 1990 (Popadic, 1992g).

Location	Application		Propargite mg/kg	Comments
	kg ai/ha	kg ai/hl		
Georgia	3.4	0.72	0.55, 0.90	Shuck split at second appl
Georgia	3.4	0.72	<0.10 (2)	Late nut fill at second appl
Louisiana	3.4	0.72	<0.10 (2)	Shuck split at second appl
Alabama	3.4	0.72	<0.10 (2)	Late nut fill at second appl
Texas	3.4	0.72	0.18, 0.14	80% shuck split at second appl

Walnuts. An EC formulation (680 g/kg) of propargite was applied twice at a rate of 7.6 kg ai/ha in 940 l/ha with an airblast sprayer to trees at two locations in California, USA, in 1988 (Popadic, 1988). Each site consisted of two treated plots and one untreated plot, each containing 24 trees. Nuts were harvested, shelled manually 14 days after the second application, and stored frozen for some 6 months. Kernel extracts were analysed by gas chromatography with a flame photometric detector in the sulfur mode. A limit of quantification of 0.05 mg/kg was demonstrated (recoveries 80 and 137%). Fortified control kernels (0.05 mg/kg) analysed with the samples gave recoveries of 83 and 106%.

In a trial in France a WP formulation was applied once at 0.15 kg ai/ha with a 23-day PHI (Malet and Allard, 1997).

Table 64. Residues of propargite in walnut kernels after foliar applications of an EC formulation in California, USA in 1988 (Popadic, 1988) and a WP formulation in France in 1997 (Malet and Allard, 1997).

Location	Application				PHI Days	Residues, mg/kg
	Form.	No.	kg ai/ha	kg ai/hl		
Fresno, California	EC 680 g/kg	2	7.6	0.8	14	<0.05 (2)
Denair, California	EC 680 g/kg	2	7.6	0.8	14	0.07, 0.06
France (South) 1997	WP 300 g/kg	1	0.15	0.015	23	<0.2

### Oilseed

Cotton. EC formulations (740 g/kg or 700 g/kg) were applied at 6 locations in the USA in 1999 three times at 1.8 kg ai/ha or 1.9 kg ai/ha, the third 34–93 days before harvest (Belcher, 2001). The aqueous spray volume was a nominal 230 l/ha, applied with ground boom spray equipment. Seed cotton (unginned fresh cotton) was harvested by spindle picker or stripper and ginned within 48 hours to produce undelinted cotton seed and gin trash samples. The samples were immediately frozen, stored for 8–9 months, extracted with acetonitrile and partitioned with hexane. The extract was cleaned up on a Florisil column and derivatized with heptafluorobutyric anhydride (HFBA). Analysis was by

GC-MS in the selected ion mode. The demonstrated limit of quantification for both propargite and TBPC was 0.01 mg/kg. The results are shown in Table 65.

Additional trials were reported from the USA for 1980 and 1987 (Table 65). The 1987 trial was at an exaggerated rate with a short PHI to generate cotton seed for processing (Polakoff, 1988c). In the six trials in 1980 at various locations in California cotton was treated three times with an EC formulation (730 g/kg, 78 kg/hl) (Popadic, 1993c). The three plots at each location were treated at different rates (1.8, 3.7, or 7.3 kg ai/ha), the last 37-58 days before harvest and before boll opening. All applications were with aerial equipment at 47-94 l/ha. Samples were stored frozen for about 8 months. The seed was extracted with a mixture of hexane and 2-propanol, and the extracts partitioned with acetonitrile and cleaned up by gel permeation chromatography. The final extract was analysed by gas chromatography with a flame photometric detector in the sulfur mode. The demonstrated limit of quantification was 0.1 mg/kg (recoveries 89 and 95%).

Table 65. Residues of propargite and TBPC in cotton seed and gin trash after three foliar applications of EC formulations in the USA.

Location Year	Application			PHI Days	Propargite mg/kg	TBPC mg/kg	Reference
	Method <sup>1</sup>	kg ai/ha	kg ai/hl				
<i>Gin trash</i>							
Oklahoma 1999		1.8	0.76	49	1.0	0.28	Belcher, 2001
		1.9	0.81		<u>1.0</u>	0.28	
Texas 1999		1.8	0.76	34	3.7	0.42	Belcher, 2001
		1.9	0.81		3.8	0.48	
Texas 1999		1.8	0.76	48	5.5	0.46	Belcher, 2001
		1.9	0.81		<u>5.8</u>	0.58	
New Mexico 1999		1.8	0.76	49	5.0	0.49	Belcher, 2001
		1.9	0.81		<u>8.4</u>	0.75	
California 1999		1.8	0.76	50	7.7	1.5	Belcher, 2001
		1.9	0.81		<u>16</u>	2.1	
California 1999		1.8	0.76	48	11	2.0	Belcher, 2001
		1.9	0.81		<u>16</u>	2.4	
<i>Cotton seed</i>							
Oklahoma 1999		1.8	0.76	49	<u>0.12</u>	0.027	Belcher, 2001
		1.9	0.81		<0.1	0.018	
Texas 1999		1.8	0.76	34	0.15	0.030	Belcher, 2001
		1.9	0.81		0.17	0.031	
Texas 1999		1.8	0.76	48	0.32	0.042	Belcher, 2001
		1.9	0.81		<u>0.44</u>	0.053	
New Mexico 1999		1.8	0.76	49	0.36	0.051	Belcher, 2001
		1.9	0.81		<u>0.42</u>	0.045	
California 1999		1.8	0.76	50	0.054	0.01	Belcher, 2001
		1.9	0.81		<u>0.095</u>	0.012	
California 1999		1.8	0.76	48	<0.1	0.016	Belcher, 2001
		1.9	0.81		<u>0.10</u>	0.015	
Mississippi 1987		11	5.9	2	4.2	-	Polakoff 1988k
California 1980	aerial	1.8	1.9	51	<u>0.11</u>	-	Popadic 1993c
		3.7	3.9		<0.1		
		7.3	7.8		<0.1		
California 1980	aerial	1.8	3.9	37	0.10	-	Popadic 1993c
		3.7	8.0		<0.1		
		7.3	16		<0.1		
California 1980	aerial	1.8	3.9	44	<0.1	-	Popadic 1993c
		3.7	8.0		<0.1		
		7.3	16		<0.1		
California 1980	aerial	1.8	1.8	58	<0.1	-	Popadic 1993c
		3.7	3.7		<0.1		
		7.3	7.3		<0.1		
California	aerial	1.8	1.8	58	<0.1	-	Popadic

Location Year	Application			PHI Days	Propargite mg/kg	TBPC mg/kg	Reference
	Method <sup>1</sup>	kg ai/ha	kg ai/hl				
1980		3.7 7.3	3.7 7.3		<0.1 <0.1		1993c
California 1980	aerial	1.8 3.7 7.3	3.9 8.0 16	58	<0.1 <0.1 0.12	-	Popadic 1993c

<sup>1</sup> Ground spray unless shown as aerial

Peanuts. A WP formulation (300 g/kg) and an EC formulation (750 g/kg) were applied with a pressurized backpack sprayer to peanuts at five locations in the USA in 1988 (Popadic, 1992f). Two applications of each formulation were made at rates of 1.8 kg ai/ha (EC) and 1.7 kg ai/ha (WP), 200 l/ha. Peanuts and hay were then harvested by hand or with a commercial digger 14 days later. The plants were allowed to dry for 7 days before sampling. Samples were stored frozen (4 months kernels; 5 months hulls; 7 months hay), and the cleaned up extracts analysed by gas chromatography with a flame photometric detector in the sulfur mode. Demonstrated limits of quantification were 0.05 mg/kg for kernels ( $92 \pm 22\%$  at 0.05 mg/kg, n=10, range 62–126%) and hulls and 0.1 mg/kg for hay. The results are shown in Tables 66 and 69.

Table 66. Residues of propargite in peanuts after two foliar applications of EC or WP formulations in the USA in 1988 (Popadic, 1992f).

Location	Application			PHI Days	Propargite, mg/kg	
	Form.	kg ai/ha	kg ai/hl		hulls	kernels
Virginia	EC 750 g/kg	1.8	0.9	14	0.21	<0.05
Georgia	EC 750 g/kg	1.8	0.9	14	0.45	<0.05
Georgia	EC 750 g/kg	1.8	0.9	14	0.25	<0.05
Oklahoma	EC 750 g/kg	1.8	0.9	14	0.81	<0.05
Alabama	EC 50 g/kg	1.8	0.9	14	0.84	<0.05
Virginia	WP 300 g/kg	1.7	0.8	14	0.20	<0.05
Georgia	WP 300 g/kg	1.7	0.8	14	0.49	<0.05
Georgia	WP 300 g/kg	1.7	0.8	14	0.19	<0.05
Oklahoma	WP 300 g/kg	1.7	0.8	14	0.48	<0.05
Alabama	WP 300 g/kg	1.7	0.8	14	0.62	<0.05

### Herbs and spices

Mint. In US trials in Washington and Idaho an EC formulation of propargite (740 g/kg) was applied in 200 l water/ha as a broadcast spray at 2.3 kg ai/ha at three locations, and the tops harvested 14 days later. Samples were frozen in plastic bags for 106-165 days, and also about 25 kg per replicate at each site was dispatched under ambient conditions for processing into oil. Propargite and TBPC were extracted from the tops with acetonitrile and the acetonitrile fraction was partitioned with hexane, exchanged to petroleum ether and cleaned up on a Florisil column. The analysis was by GC-MS in the selected ion mode. Concurrent procedural recoveries at 0.01 mg/kg, 0.50 mg/kg and 10 mg/kg for both analytes were propargite 90-112%, TBPC 78-104%. The results are shown in Table 67 (Korpalski, 2001).

Table 67. Residues of propargite in fresh mint tops in the USA (Korpalski, 2001).

Location Year	Application				PHI Days	Residues, mg/kg	
	Form.	No.	kg ai/ha	kg ai/hl		Propargite	TBPC
Eden, Idaho, 1997	EC 750 g/l	2	2.3	1.2	14	<u>5.6</u>	0.39
Harrah, Washington, 1997	EC 750 g/l	2	2.4	1.3	14	<u>5.2</u>	0.78
Ephrata, Washington, 1997	EC 750 g/l	2	2.3	1.2	14	<u>1.6</u>	0.15

### Legume animal feeds

Alfalfa. Summaries of US trials in the 1970s were reported but did not include critical information on the field work or analyses. In a field trial on alfalfa grown for seed three aerial applications of an EC formulation (730 g ai/kg) at 0.28 kg ai/ha were made to 0.2-0.4 ha sites in Washington, California and Nevada, the third at bloom to post-bloom stage in 1991 (Popadic, 1993b). Alfalfa was harvested at maturity, 27-28 days later and the uncleaned seed separated into seed and seed screenings. Straw was taken in the field or during processing and regrowth hay collected by hand 18-121 days after seed harvest in accordance with local practice. All samples were stored frozen for one year, extracted with hexane/2-propanol and the extract partitioned with acetonitrile. Clean-up was by gel permeation and Florisil column chromatography. The final extracts were analysed by gas chromatography with a flame photometric detector. The demonstrated limit of quantification was 0.05 mg/kg in all samples except straw, in which it was 0.25 mg/kg (Table 68).

Table 68. Residues of propargite in alfalfa seed, screenings, straw and regrowth hay in the USA in 1991 after three aerial applications of a 750 g/kg EC formulation (Popadic, 1993b).

Location, Year	Application			PHI (days)	Residues, mg/kg				
	Form.	kg ai/ha	kg ai/hl		seed	seed screenings	straw	regrowth hay, fresh	regrowth hay, dry
Ephrata, Washington	EC 750 g/kg	2.8	3.0	27	<u>1.24</u>	38. (c0.10)	25 (c0.14)	0.10	0.34
Riverdale, California,	EC 750 g/kg	2.8	3.0	28	<u>1.24</u>	56. (c0.06)	15. (c0.10)	0.15	0.36
Orovada, Nevada	EC 750 g/kg	2.8	3.0	28	<u>0.35</u> 7.4 (c0.06)		25. (c0.13)	0.14	0.17

c: control

Table 69. Residues of propargite in peanut hay after two foliar application of EC and WP formulations in the USA in 1988. PHI 14 days (See also Table 66).

Location	Application			Propargite, mg/kg
	Form.	kg ai/ha	kg ai/hl	
Virginia	EC 750 g/kg	1.8	0.9	<u>5.6</u>
Georgia	EC 750 g/kg	1.8	0.9	<u>5.8</u>
Georgia	EC 750 g/kg	1.8	0.9	<u>8.2</u>
Oklahoma	EC 750 g/kg	1.8	0.9	<u>8.5</u>
Alabama	EC 50 g/kg	1.8	0.9	<u>3.6</u>

Virginia	WP 300 g/kg	1.7	0.8	<u>4.0</u>
Georgia	WP 300 g/kg	1.7	0.8	<u>14.</u>
Georgia	WP 300 g/kg	1.7	0.8	<u>7.5</u>
Oklahoma	WP 300 g/kg	1.7	0.8	<u>5.6</u>
Alabama	WP 300 g/kg	1.7	0.8	<u>3.9</u>

### Straw, fodder and forage of cereal grains

**Maize.** In a field trial in France plants were treated twice with an EC formulation (570 g/l) of propargite at 1.4 kg ai/ha. Four plants per plot of 6 rows (84 m<sup>2</sup>) were harvested 41 days later (pre-flowering, Table 70) (Truchot, 1988).

In two US field trials in 1995 at sites in Colorado and Kansas, an EC formulation (680 g/kg) was applied once at 1.9 kg ai/ha with a ground boom sprayer in a volume of 190 l/ha. Forage samples were taken after 30 days and 57-60 days (early dent stage). Duplicate samples of about 2.3 kg, consisting of a minimum of twelve plants sheared off about 15 cm above ground, were stored frozen until analysis by gas chromatography with a flame photometric detector. The method was validated for maize forage (silage) in the range 0.05-10 mg/kg (Table 70, Korpalski, 1997c).

An EC formulation (680 g/kg, 720 kg/l) was applied to maize at two separate locations in Texas, USA, in 1995. Two applications were made, the first by boom sprayer at 1.3 kg ai/ha in 190 l/ha of water and the second aerial at 1.9 kg ai/ha in 50 l/ha of water. Forage samples of 3.2-4.5 kg were taken 30 days and 38 or 59 days after the first application (early dent stage), and stored frozen until extracted with hexane/2-propanol. After Florisil column chromatography, the extracts were analysed on a gas chromatograph with a flame photometric detector. The demonstrated range of quantification was 0.1 to 10 mg/kg (Table 70 (Korpalski, 1996c).

Table 70. Residues of propargite in maize forage after applications of EC formulations.

Location, Year	Application				PHI Days	Residues, mg/kg	Reference, analytical method
	Form.	No.	kg ai/ha	kg ai/hl			
USA (Colorado), 1995	680 g/kg	1	1.9	1.0	30	0.08	Korpalski, 1997c; GC/FPD
USA (Kansas), 1995	680 g/kg	1	1.9	1.0	30	0.16	Korpalski, 1997c; GC/FPD
USA (Colorado), 1995	680 g/kg	1	1.9	1.0	60	<0.05	Korpalski, 1997c; GC/FPD
USA (Kansas), 1995	680 g/kg	1	1.9	1.0	57	0.05	Korpalski, 1997c; GC/FPD
USA(Uvalde, Texas), 1995	680 g/kg	1*	1.3	0.7	29	<0.05	Korpalski, 1996c; GC/FPD
USA(Lockney, Texas), 1995	680 g/kg	1*	1.3	0.7	31	<0.05	Korpalski, 1996c; GC/FPD
USA(Uvalde, Texas), 1995	680 g/kg	1*	1.3	0.7	38	<0.05	Korpalski, 1996c; GC/FPD
USA(Lockney, Texas), 1995	680 g/kg	1*	1.3	0.7	59	<0.05	Korpalski, 1996c; GC/FPD
France, 1988	570 g/l	2	1.4	0.26	41	<u>2.2</u>	Truchot, 1988; GC/EC

\* Two applications, but samples were taken before the second

In two further US field trials in 1995 in Colorado and Kansas the 680 g/kg EC was sprayed twice at 1.9 kg ai/ha, first by ground boom in 190 l/ha, then aerial in 50 l/ha at the early dent stage. Fodder (stalks, leaves and cobs) was collected 30 days after the second treatment and stored frozen, and 110 kg of grain was sent to an aspirating facility where the moisture content was determined to be 20-30%. The grain was then dried with forced warm air at 63°C to a 10-15% moisture content, put

through a Kice grain aspirator to remove light impurities and the aspirated fraction sieved to obtain the desired grain dust fraction (<2540  $\mu\text{m}$ ). The sample yielded 0.2 kg final dust. The samples of fodder and dust were extracted and analysed by gas chromatography with a flame photometric detector. The method was validated from 0.05 to 10 mg/kg for fodder only. Fodder samples were stored for about 200 days, and grain dust samples analysed within 30-60 days of generation (Table 71, Korpalski, 1997c).

Fodder and grain from the trials at two sites in Texas with two applications as described above (Korpalski, 1996c) was sampled 28-29 days after the second application. The fodder samples of about 1.1 kg were frozen pending extraction and analysis, and grain samples of 113 kg dispatched for aspiration, and the moisture content reduced from 30% to 14-15% by forced warm air drying at 68°C. The grain was aspirated and sieved as above. About 0.8 kg final dust was obtained from the 113 kg of grain. The fodder and dust samples were extracted with hexane/2-propanol, and, after Florisil column chromatography, the extracts analysed by GLC with a flame photometric detector. Recoveries from grain dust fortified at 0.05-0.5 mg/kg ranged from 98 to 114%, and from fodder fortified at 0.1 and 10 mg/kg from 87 to 97% (Table 71).

Table 71. Residues of propargite in maize dust (aspirated grain fractions) and fodder in the USA.

Location Year	Application				PHI Days	Residues, mg/kg dust	Residues, mg/kg fodder
	Form.	No.	kg ai/ha	kg ai/hl			
Korpalski, 1997c							
Colorado, 1995	EC 680 g/kg	2	1.9	1 ground aerial	30	0.05	7.4 7.0
Kansas, 1995	EC 680 g/kg	2	1.9	1 ground 4 aerial	30	0.36	
Korpalski, 1996c							
Lockney, Texas 1995	EC 680 g/kg	2	1.3	0.7 ground 4 aerial	28	<0.05	3.7
Uvalde, Texas 1995	EC 680 g/kg	2	1.3	0.7 ground 4 aerial	29	0.13	9.8

Sorghum. In a 1995 trial in Texas, USA, an EC formulation was applied at a rate of 1.8 kg ai/ha in 170 l/ha of water, and grain and fodder harvested 60 days later. Large samples (170 kg) were taken to allow for grain dust collection. Fodder was cut about 8 cm above the ground by hand using a sickle after the grain had been harvested. All samples were stored frozen. At an aspirating facility, the grain was dried at 43-66°C until the moisture content was reduced to 10-13%. The grain was then cycled repeatedly through holding bins, drag conveyors and a bucket conveyor for 2 hours. Dust was removed at each transfer point by vacuum aspiration. The samples were next put through a Kice grain aspirator to remove any additional dust, and the dusts collected for analysis (Table 72).

Table 72. Residues of propargite in sorghum fodder and grain dust after the application of an EC formulation in Texas, USA, 1995.

Formulation	Application			PHI (days)	Residue (mg/kg)	
	No.	kg ai/ha	kg ai/hl		Fodder	Grain dust
EC 750 g/kg	1	1.8	1.0	59	0.05	<0.05

## FATE OF RESIDUES IN STORAGE AND PROCESSING

Hops. Trials were reported from the USA, but only summary information was provided for the 1969 trials in Washington (Anon., 1969) which gave no details of sample handling, methods of analysis or control values.

Summary information was also reported for the US field trials in 1979-1987 (Ball, 1988b). Both WP and EC formulations were applied in Washington State, at rates of 1.7 and 1.5 kg ai/ha. At maturity, the vines were harvested, cones removed with a hop picking machine, and some kiln-dried. Fresh and dried samples were analysed by gas chromatography with a flame photometric detector. The nominal limit of quantification was 0.05 mg/kg (Table 73).

In additional US trials in 1989 and 1990 Korpalski (1991a,b) applied a WP formulation (300 g/kg) three times with an airblast sprayer at pre-bloom, small cone and large cone stages at a rate of 1.5 kg ai/ha (1989) or 2.0 and 2.5 kg ai/ha (1990) at various locations in Oregon, Idaho and Washington. Cones were harvested at maturity, 14 and 21 days after the last application. Two or three samples (about 0.9 kg each) were hand-picked at each site. About half of each sample was dried in a commercial kiln (Washington), a food dryer (Oregon), or by air-drying (Idaho). Samples of fresh and dried cones were stored frozen until analysis by gas chromatography with a flame photometric detector. Recoveries from fortified control samples were acceptable.

In a UK field trial in 1981, Wye Target and WVG hops were treated once with an EC formulation (570 g/kg) at 0.86 kg ai/ha (Anon., 1982). Green hops were stored frozen. Some were commercially dried. Samples extracts were cleaned up by gel permeation chromatography and analysed by gas chromatography with a flame photometric detector (Table 73).

In field trials in Germany in 1989 a WP formulation (300 g/kg) was applied at 2.4 kg ai/ha at five locations (O'Connell, 1992e). Cones were collected 0-28 days after the last application and analysed by gas chromatography with a flame-photometric detector. Acceptable recoveries (70-93%) were demonstrated for fortified control green cones, concentration range 0.25-50 mg/kg (Table 73).

Table 73. Residues of propargite in hop cones after foliar application.

Location, Year	Application				PHI Days	Residues, mg/kg		Reference
	Form.	No.	kg ai/ha	kg ai/hl		dry hops	green hops	
Moxee City, Washington, USA 1969	EC 690 g/kg	1	1.5	0.75	0	33	40	Anon., 1969
					6	27	5.7	
					13	<u>15</u>	<u>8.4</u>	
					20	3.8	2.5	
	EC 690 g/kg	1	3.0	1.5	0	200	63	Anon., 1969
					6	40	40	
					13	62	42	
					20	63	26	
	WP 300 g/kg	1	1.7	0.85	0	108	32	Anon., 1969
					6	29	15	
					13	<u>28</u>	<u>3.4</u>	
					20	13	0.7	
	WP 300 g/kg	1	3.4	1.7	0	90	77	Anon., 1969
					6	94	37	
					13	35	14	
					20	50	14	
Toppehnish, Washington, USA 1969	EC 690 g/kg	1	1.5	0.75	0	79	41	Anon., 1969
					7	40	14	
					14	<u>33</u>	<u>7.2</u>	
					21	8.9	3.3	

Location, Year	Application				PHI Days	Residues, mg/kg		Reference
	Form.	No.	kg ai/ha	kg ai/hl		dry hops	green hops	
	EC 690 g/kg	1	3.0	1.5	0 7 14 21	170 140 23 31	75 54 16 11	Anon., 1969
	WP 300 g/kg	1	1.7	0.85	0 7 14 21	74 32 <u>18</u> 18	36 14 <u>5.4</u> 4.1	Anon., 1969
	WP 300 g/kg	1	3.4	1.7	0 7 14 21	110 55 24 14	30 24 9.3 5.0	Anon., 1969
Mabton, Washington, USA, 1969	WP 300 g/kg	1	1.7	0.85	0 7 14 21	120 35 <u>20</u> 27	31 16 <u>3.5</u> 18	Anon., 1969
	WP 300 g/kg	1	3.4	1.7	0 7 14 21	140 44 44 58	48 31 20 8.6	Anon., 1969
Yakima, Washington, USA, 1987	EC 600 g/kg	2	1.8	0.19	14	<u>15</u> 17(c0.06)	<u>4.0</u> 3.6	Ball, 1988b
	WP 300 g/kg	2	1.7	0.18	14	<u>12</u> 12(c0.06)	<u>8.8</u> 4.0	Ball, 1988b
Prosser, Washington, USA, 1984	EC 600 g/kg	2	2.0	0.22	15	<u>16</u> (c0.49)	<u>5.0</u> (c0.15)	Ball, 1988b
Prosser, Washington, USA, 1984	EC 600 g/kg	2	2.0	0.11	14	<u>25</u> (c0.49)	<u>7.0</u> (c0.15)	Ball, 1988b
Toppenish, Washington, USA, 1984	EC 600 g/kg	2	2.0	0.07	14	<u>18</u> (c0.27)	<u>6.0</u> (c0.26)	Ball, 1988b
Moxee, Washington, USA, 1983	WP 300 g/kg	2	1.7	0.18	14 14 15	30 25 <u>90</u>		Ball, 1988b
Prosser, Washington, USA, 1981	WP 300 g/kg	2	1.7	0.09 0.09 0.18	14	22(c0.22) 19 <u>46</u>		Ball, 1988b
Prosser, Washington, USA, 1981	WP 300 g/kg	2	3.4	0.09 0.09 0.18	14	40 51(c0.22) <u>75</u>		Ball, 1988b
Harrah, Washington, USA, 1979	WP 300 g/kg	2	2.2	0.12	14	<u>19</u>		Ball, 1988b
Granger, Washington, USA 1990	WP 300 k/kg	3	2.0	0.21	14	<u>6.9</u> (c0.25)	2.5	Korpalski, 1991b
Harrah, Washington, USA 1990	WP 300 k/kg	3	2.0	0.21	14	<u>14.</u> (c0.35)	<u>2.4</u> (c0.11)	Korpalski, 1991b
Moxee, Washington, USA, 1990	WP 300 k/kg	3	2.0	0.21	14	<u>14</u> (c0.24)	<u>1.7</u> (c0.17)	Korpalski, 1991b
Salem, Oregon, USA, 1990	WP 300 k/kg	3	2.0	0.21	14	1.3 <sup>1</sup>	13.0 <sup>1</sup>	Korpalski, 1991b
Wilder, Idaho, USA, 1990	WP 300 k/kg	3	2.0	0.21	14	<u>15</u> (c1.9)	<u>2.2</u> (c0.34)	Korpalski, 1991b
Granger, Washington, USA, 1990	WP 300 k/kg	3	2.5	0.27	14	17.0 (c0.25)	4.3	Korpalski, 1991b
Harrah, Washington, USA, 1990	WP 300 k/kg	3	2.5	0.27	14	22.0 (c0.35)	2.9 (c0.11)	Korpalski, 1991b
Moxee, Washington, USA, 1990	WP 300 k/kg	3	2.5	0.27	14	16.0 (c0.24)	1.7 (c0.17)	Korpalski, 1991b
Salem, Oregon, USA, 1990	WP 300 k/kg	3	2.5	0.27	14	2.6	21	Korpalski, 1991b
Wilder, Idaho, USA, 1990	WP 300 k/kg	3	2.5	0.27	14	5.8 (c1.9)	1.7 (c0.34)	Korpalski, 1991b



Location, Year	Application				PHI Days	Residues, mg/kg		Reference
	Form.	No.	kg ai/ha	kg ai/ha		dry hops	green hops	
Hubbard, Oregon, USA, 1989	WP 300 k/kg	3	1.5	0.16	14	-	3.8	Korpalski, 1991a
					21	-	3.8	
Hubbard, Oregon, USA, 1989	WP 300 k/kg	3	1.5	0.16	14	-	16	Korpalski, 1991a
					21	-	12	
Greenleaf, Idaho, USA, 1989	WP 300 k/kg	3	1.5	0.16	14	9.1(c0.39)	1.2	Korpalski, 1991a
					21	9.0	2.6 <sup>2</sup>	
Wilder, Idaho, US, 1989	WP 300 k/kg	3	1.5	0.16	14	18(c0.17)	3.1	Korpalski, 1991a
					21	18	3.8	
Kent, UK, 1981	EC 750 g/kg	1	0.86		0		3.9	Anon., 1982
					7		3.1	
					14		3.1	
					17	2.7		
Kent, UK 1981	EC 750 g/kg	1	0.86		0		12	Anon., 1982
					7		1.2	
					14		1.4	
					29	0.61		
Tett nang, Germany 1989	WP 300 g/kg	2	2.4	0.05	21	16	0.86	O'Connell, 1992e
					28	4.4	0.80	
Tett nang, Germany 1989	WP 300 g/kg	2	2.4	0.05	21	76	14	O'Connell, 1992e
					28	60	10	
Hull, Germany 1989	WP 300 g/kg	2	2.4	0.05	21	2.6	0.63	O'Connell, 1992e
					28	1.7	0.87	
Hull, Germany 1989	WP 300 g/kg	2	2.4	0.05	21	4.4	2.6	O'Connell, 1992e
					28	10	0.87	
Hull, Germany 1989	WP 300 g/kg	2	2.4	0.05	21	1.2	0.48	O'Connell, 1992e
					28	1.3	0.38	

c: control

<sup>1</sup> Possible confusion of samples. Higher residues in green than dry hops.

<sup>2</sup> Average of two samples, 5.1 and 0.14 mg/kg.

Tea (Table 74). Field trials were reported from Kenya, India, Indonesia and Japan.

In western Kenya an EW formulation (570 g/l) was applied twice by means of a backpack pump sprayer with hand-held boom to mature plants at two sites in 1996. Each site consisted of three treated and one control plot, one site was at 2000 m and the other below 1000 m, and the rows were nearly closed by the growth of the bushes. Fresh tealeaf samples of 125 g were picked 7-28 days after the second application and stored frozen until analysis. A sub-sample (20 g) was hydrated with water, extracted with hexane/2-propanol, and the hexane fraction purified on a Florisil column and further cleaned up by gel permeation chromatography. The final extract was analysed by gas chromatography with a flame photometric detector. The limit of quantification was 0.05 mg/kg (Korpalski, 1997a).

In two field trials, each consisting of six plots, in the island of Java, Indonesia, an EC formulation (570 g/l) was sprayed three times onto mature tea plants at about 0.57 and 1.1 kg ai/ha with a knapsack pump sprayer with hand wand. About 400 l of solution was applied per ha. Two of the plots at each location were treated at each rate and two were controls. Seven days after the third application two samples (300 g) were taken from each site and frozen immediately for shipment to the analytical laboratory, where the sample were prepared and analysed in a procedure analogous to that used for the samples from Kenya (above) (Table 74, Korpalski, 1996e).

In two field trials in Japan an EC formulation (570 g/kg) of propargite was applied with a backpack pump sprayer to mature plants at two sites. The first trial took place in Koyu-gun, Miyazaki, on the island of Honshu, a cooler tea cultivation region north of Tokyo, and the second at a subtropical cultivation area, Iruma-shi, Saitama, Kyushu. Two applications were made at 14-day intervals to duplicate plots at a rate of 1.5 and 3.0 kg ai/ha, or 2.7 and 5.3 l/ha in a spray volume of

about 4000 l/ha. Leaves (250-350 g) were sampled 14-42 days after the second application, and stored frozen until prepared for analysis as above (Korpalski, 1996a).

In a field trial in Valparai, India, bushes at seven plots were treated once with an EC formulation (570 g/kg) at rates of 0.57 kg ai/ha and 1.1 kg ai/ha in 2001, applied at 400 l/ha with a hand-operated knapsack sprayer (Muraleedharan, 2001). Shoots with three leaves and a bud were picked 1-14 days (i.e. 7 pre-harvest intervals) later and converted to black tea. Fresh tea samples were not retained. The black teas were stored frozen until extracted and analysed by HPLC. The method was validated with fortified control tea samples at 3.3 and 6.6 mg/kg.

Table 74. Residues of propargite in tea.

Location, Year	Application				PHI Days	Residues, mg/kg fresh tea leaves	Reference Comment
	Form.	No.	kg ai/ha	kg ai/hl			
Kericho, Kenya, 1996	EW 570 g/kg	2	1.1	0.45	7	19, 18, 13	Korpalski, 1997a
					10	13, 15, 14	
					14	5.8, 4.6, 2.7	
					21	0.72, 0.91, 0.73	
					28	0.33, 0.38, 0.34, 0.15	
Sotik, Kenya, 1996	EW 570 g/kg	2	0.85	0.17	7	3.6, 3.2, 2.9	Korpalski, 1997a
					10	1.3, 0.84, 0.93	
					14	0.10, 0.21, 0.10	
					21	0.05, 0.09, 0.25	
					28	0.06, 0.05, 0.09	
Gambung, Indonesia, 1994	EC 570 g/kg	3	0.57	0.14	7	0.29, 0.60	Korpalski, 1996e
Gambung, Indonesia, 1994	EC 570 g/kg	3	1.1	0.28	7	2.1, 2.0	Korpalski, 1996e
Pasir Sarongge, Indonesia, 1994	EC 570 g/kg	3	0.57	0.14	7	1.0, 1.2	Korpalski, 1996e
Pasir Sarongge, Indonesia, 1994	EC 570 g/kg	3	1.1	0.28	7	2.2, 3.8	Korpalski, 1996e
Kyushu, Japan, 1994	EC 570 g/kg	2	1.5	0.04	14	0.26, 0.24	Korpalski, 1996a
					21	0.09, 0.09	
					28	0.07, 0.06	
					35	0.05, 0.05	
					42	<0.05, <0.05	
Kyushu, Japan, 1994	EC 570 g/kg	2	3.0	0.08	14	0.50, 0.88	Korpalski, 1996a
					21	0.17, 0.17	
					28	0.09, 0.12	
					35	0.07, 0.06	
					42	<0.05, <0.05	
Honshu, Japan, 1994	EC 570 g/kg	2	1.5	0.04	14	0.14, 0.16	Korpalski, 1996a
					21	0.08, 0.05	
					28	<0.05, <0.05	
					35	<0.05, <0.05	
					42	<0.05, <0.05	
Honshu, Japan, 1994	EC 570 g/kg	2	3.0	0.08	14	0.27, 0.22	Korpalski, 1996a
					21	0.09, 0.10	
					28	<0.05, <0.05	
					35	<0.05, <0.05	
					42	<0.05, <0.05	

Location, Year	Application				PHI Days	Residues, mg/kg fresh tea leaves	Reference Comment
	Form.	No.	kg ai/ha	kg ai/hl			
Valparai, India 2001	EC 570 g/kg	1	0.57	0.14	0	140	Muraleedharan, 2001 Black tea (not fresh) ND: not detected.
					1	110	
					3	5.2	
					5	2.4	
					7	ND	
					10	ND	
					14	ND	
Valparai, India 2001	EC 570 g/kg	1	1.1	0.28	0	250	Muraleedharan, 2001 Black tea (not fresh) ND: not detected.
					1	240	
					3	10	
					5	5.5	
					7	1.7	
					10	ND	
					14	ND	

## FATE OF RESIDUES IN STORAGE AND PROCESSING

### In processing

Studies generally simulated commercial production with the exception of peeling avocados and brewing tea. The results of all the trials are shown in Table 75. Multiple entries represent distinct processing runs and not multiple samples from one run.

Table 75. The effect of processing on residues of propargite.

Ref.	RAC	Residue in RAC (mg/kg)	Product	Residue in product (mg/kg)	Processing factor	Mean processing factor
Partington, 1997b	Apple (whole)	0.55, 0.64, 0.44, 0.79 (procedure 1)	Apple purée (sauce)	0.01, 0.01, <0.01, <0.01 (procedure 1)	0.018, 0.016, <0.023, <0.013 (procedure 1)	0.02
		0.55, 0.64, 0.44, 0.79 (procedure 2)		1.4, 1.8, 1.2, 1.8 (procedure 2)	2.54, 2.81, 2.73, 2.28 (procedure 2)	2.6
Korpalski, 1995a	Apple (whole)	0.61, 0.70, 0.67, 0.84	Apple juice	<0.05 (4)	<0.082, <0.071, <0.075, <0.060	<0.07
Korpalski, 1995b	Apple (whole)	1.9, 2.0, 2.0	Apple juice	<0.05, 0.06, <0.05	<0.026, 0.030, <0.025	≤0.03
Korpalski, 1995a	Apple (whole)	0.61, 0.70, 0.67, 0.84	Wet pomace	2.45, 3.59, 2.10, 3.76	4.02, 5.13, 3.13, 4.48	4.2
Korpalski, 1995b	Apple (whole)	1.9, 2.0, 2.0	Wet pomace	8.53, 8.27, 7.11	4.49, 4.14, 3.56	4.1
Korpalski, 1996b	Avocado (whole)	0.12-0.28 (0.19)	Peeled avocado	<0.05-0.06 (0.05)	0.263	0.19
Korpalski, 1996b	Avocado (whole)	1.0-1.28 (1.16)	Peeled avocado	0.08-0.19 (0.14)	0.121	
Polakoff, 1988k	Cotton seed (undelinted whole)	3.7	Delinted seed	0.68	0.183	0.18
Polakoff, 1988c	Cotton seed	3.7	Hulls	2.1	0.567	0.57
Polakoff, 1988c	Cotton seed	3.7	Meal	<0.05	<0.014	<0.014
Polakoff, 1988c	Cotton seed	3.7	Refined oil	0.80	0.216	0.22
Polakoff,	Cotton seed	3.7	Soapstock	<0.05	<0.0135	<0.014

Ref.	RAC	Residue in RAC (mg/kg)	Product	Residue in product (mg/kg)	Processing factor	Mean processing factor
1988c						
Polakoff, 1988c	Cotton seed	3.7	Solvent extracted oil	0.75	0.203	0.20
Polakoff, 1988k <sup>3</sup>	Grapes	1.3, 3.4, 4.8	Dry pomace	4.4, 17.2, 20.3 <sup>2</sup>	3.38, 5.06, 4.23	4.2
Polakoff, 1988k <sup>3</sup>	Grapes	1.3, 3.4, 4.8	Juice	0.2, 0.82, 0.88 <sup>3</sup>	0.154, 0.241, 0.183	0.19
Popadic, 1994e	Grapes	1.16	Raisins	1.40	1.20	1.2
Polakoff, 1988k <sup>2</sup>	Grapes	1.3, 3.4, 4.8	Raisins	0.81, 5.2, 16.9 <sup>2,3</sup>	0.623, 1.53, 3.52	1.9
Polakoff, 1988k <sup>2</sup>	Grapes	1.3, 3.4, 4.8	Raisin waste	3.4 <sup>3</sup> , 11.2 <sup>3</sup> , 11.9 <sup>2,3</sup>	2.62, 3.29, 2.48	2.8
Korpalski, 1999b	Grapes (propargite)	0.46	Juice	<0.01	<0.022	<0.02
Korpalski, 1999b	Grapes (propargite)	0.46	Wine	<0.01	<0.022	<0.02
Korpalski, 1999b	Grapes (TBPC)	0.11	Juice	<0.01	<0.091	<0.09
Korpalski, 1999b	Grapes (TBPC)	0.11	Wine	0.032	0.29	0.3
Korpalski, 1999b	Grapes (TBPC-diol)	0.12	Wine	0.069	0.58	0.6
Ball, 1992	Hops (dry cones)	8.3, 9.0, 20.8, 22.5, 12.6, 13.7, 26.9, 24.4	Hops (beer)	<0.01	<0.012, <0.011, <0.048, <0.044, <0.079, <0.072, <0.037, <0.041	<0.043
Ball, 1992	Hops (dry cones)	8.3, 9.0, 20.8, 22.5, 12.6, 13.7, 26.9, 24.4	Hops (wort)	0.01, <0.01 (7)	0.012, <0.011, <0.048, <0.044, <0.079, <0.072, <0.037, <0.041	<0.043
Ball, 1992	Hops (dry cones)	8.3, 9.0, 20.8, 22.5, 12.6, 13.7, 26.9, 24.4	Hops (spent hops)	0.64, 0.71, 1.6, 1.5, 0.38, 0.31, 1.5, 1.5	0.077, 0.079, 0.077, 0.067, 0.030, 0.023, 0.056, 0.061	0.059
Ball, 1992	Hops (green cones) <sup>4</sup>	4.5, 4.1, 9.7, 9.9, 5.2, 4.9, 8.3, 7.6	Hops (dry cones)	8.3, 9.0, 20.8, 22.5, 12.6, 13.7, 26.9, 24.4	1.84, 2.19, 2.14, 2.27, 2.42, 2.80, 3.24, 3.21	2.5
Smudin, 1995a	Maize (grain)	0.05, 0.06	Crude oil (dry milled)	0.13, 0.19	2.60, 3.17	2.9
Smudin, 1995a	Maize (grain)	0.05, 0.06	Crude oil (wet milled)	0.33, 0.28	6.60, 4.67	5.6
Smudin, 1995a	Maize (grain)	0.05, 0.06	Dust (dry milled)	1.5, 1.9	30.00, 31.67	31
Smudin, 1995a	Maize (grain)	0.05, 0.06	Flour (dry milled)	0.08, 0.10	1.60, 1.67	1.6
Smudin, 1995a	Maize (grain)	0.05, 0.06	Grits (dry milled)	<0.05	<1.00, <0.83	<0.92
Smudin, 1995a	Maize (grain)	0.05, 0.06	Meal (dry milled)	0.05, 0.07	1.00, 1.17	1.1
Smudin, 1995a	Maize (grain)	0.05, 0.06	Refined oil (dry milled)	0.12, 0.20	2.40, 3.33	2.9
Smudin, 1995a	Maize (grain)	0.05, 0.06	Refined oil (wet milled)	0.29, 0.28	5.80, 4.67	5.2
Smudin, 1995a	Maize (grain)	0.05, 0.06	Starch (wet milled)	<0.05, <0.05	<1.00, <0.83	<0.92

Ref.	RAC	Residue in RAC (mg/kg)	Product	Residue in product (mg/kg)	Processing factor	Mean processing factor
Korpalski, 2001	Mint tops (propargite)	5.2, 3.7, 1.3, 1.6	Mint oil	10.5, 12.5, 16.0, 31.2	2.02, 3.38, 12.31, 19.50	9.3
Korpalski, 2001	Mint tops (TBPC)	0.46, 0.56, 0.17, 0.20	Mint oil	11.4, 11.3, 12.6, 21.9	24.78, 20.18, 74.12, 109.50	57
Polakoff, 1988e	Orange (washed whole)	0.46, 0.75	Dried peel	1.5, 1.5	3.26, 2.00	2.6
Polakoff, 1988e	Orange (washed whole)	0.46, 0.75	Juice	<0.05, <0.05	<0.109, <0.067	<0.09
Polakoff, 1988e	Orange (washed whole)	0.46, 0.75	Molasses	0.12, 0.18	0.261, 0.240	0.25
Polakoff, 1988e	Orange (washed whole)	0.46, 0.75	Orange oil	10.5, 17.7	22.8, 23.6	23.
Smudin, 1995b	Peanut (kernel)	0.22 <sup>2</sup> , 0.062 <sup>2</sup>	Crude oil	0.47 <sup>2,3</sup> , 0.24 <sup>2,3</sup>	2.14, 3.87	3.0
Smudin, 1995b	Peanut (kernel)	0.22 <sup>2</sup> , 0.062 <sup>2</sup>	Meal	0.07 <sup>2</sup> , <0.05 <sup>2</sup>	0.318, <0.806	≤0.56
Smudin, 1995b	Peanut (kernel)	0.22 <sup>2</sup> , 0.062 <sup>2</sup>	Refined oil	0.34 <sup>2,3</sup> , 0.22 <sup>2,3</sup>	1.54, 3.55	2.5
Smudin, 1995b	Peanut (kernel)	0.22 <sup>2</sup> , 0.062 <sup>2</sup>	Soapstock	<0.05	<0.227, <0.806	<0.52
Popadic, 1994c	Plums (fresh)	4.0 <sup>2,3</sup> , 4.0 <sup>2,3</sup>	Dried prunes	1.0 <sup>2,3</sup> , 0.76 <sup>2,3</sup>	0.25, 0.19	0.22
Polakoff, 1988d	Plums (fresh)	1.7, 3.4, 2.6, 1.3, 3.0, 1.6, 0.8, 1.6	Dried prunes	1.6, 3.4, 1.5, 1.0, 3.0, 1.0, 1.4, 1.2	0.941, 1.00, 0.577, 0.769, 1.00, 0.625, 1.75, 0.750	0.93
Partington, 1998	Plums (fresh)	0.68	Dried prunes	1.4	2.06	2.1
Partington, 1998	Plums (fresh)	0.68	Canned prunes	0.83	1.2	1.2
Popadic and Smudin, 1995	Potatoes	<0.05	Chips	<0.05	<1.00	
Popadic and Smudin, 1995	Potatoes	<0.05	Dry peel	0.45, 0.42, 0.05, 0.15, 0.25	>9.00, >8.40, >1.00, >3.00, >5.00	
Popadic and Smudin, 1995	Potatoes	<0.05	Flakes	<0.05	<1.00	
Popadic and Smudin, 1995	Potatoes	<0.05	Wet peel	<0.05	<1.00	
Polakoff, 1988l	Sorghum (whole grain)	0.10	Bran (dry milled)	0.16	1.6	1.6
Polakoff, 1988l	Sorghum (whole grain)	0.10	Bran (wet milled)	0.12	1.2	1.2
Polakoff, 1988l	Sorghum (whole grain)	0.10	Fines	<0.05	<0.5	<0.5
Polakoff, 1988l	Sorghum (whole grain)	0.10	Flour	<0.05	<0.5	<0.5
Polakoff, 1988l	Sorghum (whole grain)	0.10	Paste	<0.05	<0.5	<0.5
Polakoff, 1988l	Sorghum (whole grain)	0.10	Starch	<0.05	<0.5	<0.5
Korpalski, 1996a	Tea (black)	88.2, 21.4, 5.4, 16.1, 3.4, 1.2	Brewed tea	1.6, 0.32, 0.09, 0.24, 0.06, 0.04	0.018, 0.015, 0.017, 0.015, 0.018, 0.033	0.019
Korpalski, 1996a	Tea (black)	88.2, 21.4, 5.4, 16.1, 3.4, 1.2	Instant tea	0.96, 0.33, 0.08, 0.27, 0.13, 0.04	0.011, 0.015, 0.015, 0.017, 0.038, 0.033	0.026
Korpalski, 1997a	Tea (black)	88.2, 21.4, 5.4, 16.1, 3.4, 1.2	Brewed black tea	1.64, 0.321, 0.087, 0.24, 0.061, 0.040	0.018, 0.015, 0.016, 0.015, 0.018, 0.034	0.019
Korpalski, 1996a	Tea (black)	1.4, 1.4, 1.8, 1.6, 8.2, 8.4, 3.0, 3.0,	Brewed black tea	0.024, 0.028, 0.040, 0.038,	0.017, 0.020, 0.022, 0.024,	0.020

Ref.	RAC	Residue in RAC (mg/kg)	Product	Residue in product (mg/kg)	Processing factor	Mean processing factor
		3.1, 2.6, 2.9, 3.0, 11, 14, 14, 10		0.15, 0.15, 0.054, 0.053, 0.060, 0.063, 0.058, 0.068, 0.20, 0.20, 0.24, 0.26	0.018, 0.018, 0.018, 0.018, 0.019, 0.024, 0.020, 0.023, 0.018, 0.014, 0.017, 0.026	
Korpalski, 1996e	Tea (fresh) <sup>4</sup>	0.10, 0.48, 0.58, 0.61, 2.4, 1.8, 1.5, 2.4, 1.3, 0.78, 1.2, 1.2, 1.6, 2.9, 4.0, 3.6	Green tea	2.2, 2.9, 1.1, 1.2, 5.9, 5.4, 3.3, 4.3, 3.6, 3.4, 2.9, 2.7, 4.3, 5.2, 11, 9.2	22, 6.0, 1.9, 2.0, 2.4, 3.0, 2.2, 1.8, 2.8, 4.4, 2.4, 2.2, 2.7, 1.8, 2.8, 2.6	3.9
Korpalski, 1997a	Tea (fresh) <sup>4</sup>	19.2, 5.0, 0.86, 3.4, 0.17, 0.11	Black tea	88.2, 21.4, 5.4, 16.1, 3.4, 1.2	4.59, 4.28, 6.33, 4.74, 20.00, 10.91	8.5
Korpalski, 1997a	Tea (fresh) <sup>4</sup>	19.2, 5.0, 0.86, 3.4, 0.17, 0.11	Instant tea	0.96, 0.33, 0.078, 0.27, 0.13, 0.044	0.050, 0.066, 0.091, 0.079, 0.76, 0.40	0.24
Korpalski, 1996a	Tea (fresh) <sup>4</sup>	0.10, 0.48, 0.58, 0.61, 2.4, 1.8, 1.5, 2.4, 1.3, 0.78, 1.2, 1.2, 1.6, 2.9, 4.0, 3.6	Black tea	1.4, 1.4, 1.8, 1.6, 8.2, 8.4, 3.0, 3.0, 3.1, 2.6, 2.9, 3.0, 11, 14, 14, 10	14, 2.9, 3.1, 2.6, 3.4, 4.7, 2.0, 1.2, 2.4, 3.4, 2.4, 2.5, 6.9, 4.8, 3.5, 2.8	3.9
Korpalski, 1996e	Tea (fresh) <sup>4</sup>	0.06, 0.09, 0.18, 0.19, 0.06, 0.05, 0.07, 0.06, 0.10, 0.06, 0.10, 0.11	Green tea	0.15, 0.24, 0.54, 0.50, 0.10, 0.10, 0.19, 0.18, 0.11, 0.11, 0.21, 0.21,	2.50, 2.67, 3.00, 2.63, 1.67, 2.00, 2.71, 3.00, 1.10, 1.83, 2.10, 1.91,	2.3
Korpalski, 1996e	Tea (green)	2.2, 2.9, 1.1, 1.2, 5.9, 5.4, 3.3, 4.3, 3.6, 3.4, 2.9, 2.7, 4.3, 5.2, 11, 9.2	Brewed green tea	0.044, 0.013, 0.015, 0.015, 0.071, 0.084, 0.040, 0.047, 0.036, 0.044, 0.037, 0.033, 0.067, 0.088, 0.16, 0.16	0.02, 0.004, 0.014, 0.012, 0.012, 0.016, 0.012, 0.011, 0.010, 0.013, 0.013, 0.012, 0.016, 0.017, 0.014, 0.017	0.013
Korpalski, 1996e	Tea (green)	0.15, 0.24, 0.54, 0.50, 0.10, 0.10, 0.19, 0.18, 0.11, 0.11, 0.21, 0.21,	Brewed green tea	<0.01, <0.01, 0.010, 0.013, <0.01 (8)	<0.067, <0.042, 0.018, 0.026, <0.1, <0.1, <0.052, <0.056, <0.091, <0.091, <0.048, <0.048	0.06
Cawkwell, 1999	Tomato	0.23	Tomato purée	0.21	0.91	0.9
Cawkwell, 1999	Tomato	0.23	Tomato canned (skinless)	<0.01	0.043	0.04
Cawkwell, 1999	Tomato	0.23	Tomato skins from canning (tomato pomace)	3.6	15.6	16
Harrison, 1999	Tomato	0.17	Tomato purée	0.23	1.35	1.4
Harrison, 1999	Tomato	0.17	Tomato canned (skinless)	<0.01	0.058	0.06

<sup>1</sup> several treated and untreated samples wrongly labelled

<sup>2</sup> average of 2 analyses

<sup>3</sup> labelled untreated

<sup>4</sup> not strictly processing. The dried commodity is considered the raw agricultural commodity.

Oranges. An orange grove (10 Valencia trees) in Florida, USA, was treated sequentially with a WP formulation of propargite at rates of 3.4 kg ai/ha and 6.7 kg ai/ha in 1987, and the oranges were picked 7 days after the last application and stored at 4.5°C for 4 days before processing at the University of Florida Institute of Food and Agricultural Sciences, but no details were reported. The oranges were washed, and the processed fractions stored frozen (-23°C) for 30-90 days. Extracts were purified by gel permeation chromatography and analysed by GLC with a photometric detector in the sulfur mode. The method was validated for whole oranges, pulp, peel and molasses at 0.05-5 or 10 mg/kg and for oil at 10-50 mg/kg (Polakoff, 1988e).

Apples. In four field trials in France in 1996 an EW formulation (240 g/l propargite + 40 g/l tetradifon) was applied once with a manual air-blast sprayer with a 7-day PHI at 1.3 kg ai/ha at application volumes of 425-1087 l/ha in water. Large samples for processing were kept in chilled storage. Whole apples (about 13 kg) were sorted to remove debris and rotten fruit. In the first procedure apples were then washed, drained for 2 min, peeled, cored, sliced, heated in a steam pan (80°C) until soft, water was added and the mixture puréed and placed in cans, which were sealed and pasteurised in boiling water. In the second procedure apples were washed, sliced, heated, sieved (to remove peel and core), puréed (80° C) and canned with pasteurization. Apples, peel and purée were analysed by GC-MS. Recoveries from fortified control samples at 0.01 mg/kg were purée 80, 100%, core/peel 100, 100%, and apple 100% (Partington, 1997b).

Two US trials were reported from the USA. In one apples from a commercial field in California that had been treated with a WP formulation (300 g/kg) were processed into juice and pomace at a New York facility (Korpalski, 1995a,b). The samples were refrigerated (not frozen) for a maximum of 11 days before simulated commercial processing, except that a batch process was used with the 23 kg samples. An unwashed sub-sample of each batch was retained for analysis. The remaining apples were ground in a Hammer mill and the resulting mash loaded into cloth sacks and pressed in a hydraulic press at 2200 to 3000 psi for 5 min. The juice, pomace and apple samples were stored frozen. The pasteurization step was omitted. Samples were extracted with hexane/2-propanol and the purified extracts were analysed by gas chromatography with a flame photometric detector.

In the second study apples treated with a WP (300 g/kg) or EC formulation (570 g/kg) in New York were processed into juice and pomace after 5 days' storage at 2°C. Processing was at the same facility as above and samples were stored and analysed in the same manner.

Avocados. Residues were measured in whole and peeled avocados without stones (see avocado field trials, Table 52, for details) (Korpalski, 1996b).

Plums. A WP formulation of propargite was applied twice to French prune plums at 3.0 kg ai/ha in California, USA, in 1993 (Popadic, 1994c). The 11-year old plum trees were planted 378 trees/ha. The plums were picked at maturity with a mechanical harvester 21 days after the second treatment. About 22 t were obtained from each plot (2 treated plots and 1 control plot, each plot five rows and 24.4 m x 368 m with 5 m row spacings) and stored at ambient conditions to be dried. A subsample was taken at harvest and stored frozen.

The entire crop was dried at a commercial facility. The prunes were rinsed with water, placed on trays and put into a drying tunnel at 86°C. The trays advanced one cycle every two hours until drying was complete (18-19 h). The dried fruit in wooden bins were turned periodically to facilitate moisture equilibration. The prunes were stored for 2 months, fumigated with methyl bromide, placed on a shaker to remove sticks and other debris, and air-vacuumed to remove light prunes and more debris. The prunes were then sized on stainless steel screens, passed over a computerized colour sorter to remove off-grade fruit, and regraded to simulate commercial blending. Finally the fruits were washed, steamed to 99°C to rehydrate and pitted, and analytical samples collected and stored frozen. The whole processing phase took about 90 days to complete.

Sample extracts were cleaned up by Florisil and gel permeation chromatographies, and analysed by GLC with a flame photometric detector. The limit of quantification, 0.05 mg/kg, was verified through fortified control recoveries. The control plum and prune samples contained propargite: 0.077 mg/kg in fresh plums and 0.076 mg/kg in the dried prunes was 0.076 mg/kg. Fresh plums and prunes were purchased to use for the fortified controls. The analyses were conducted over a 6-month period after the completion of the processing study.

A summary of plum processing studies from 1987 and 1979 in California, USA, was reported. Plums were treated with an EC or WP formulation and harvested 14 or 28 days later. Plums from the 1987 trials were dehydrated at 35% relative humidity and 71°C. Processing of the 1979 samples was not described (Polakoff, 1988d). The results are for whole fruit with stone.

In a processing study in Saint-Maurin, France, in 1997 a commercial crop of Prune d'Ente plums was treated with a WP formulation (306 g/kg) applied to run-off at 1.5 kg ai/hl using an airblast manual sprayer to simulate commercial practice. A large sample of plums was harvested 14 days later, cleaned and tailed, then fast-dried for 5 hours at 60°C and 60% humidity, with additional drying at 75°C and 30% humidity. This produced extra dried prunes (21-24% moisture). The prunes were rehydrated in hot water at 60°C with 3 g/l sorbic acid to a moisture content of 33-35%. Some prunes were canned by grading, addition of saccharose syrup to plums in the can and sterilization at 100°C for 20 min. Samples of final dried prunes and canned prunes were analysed by GC-MS. Residues were expressed on the whole fruit with stone (Partington, 1998).

Grapes. Two applications of a WP formulation at 3.0 kg ai/ha were made 21 days apart to two plots of Thompson Seedless vines in the San Joaquin Valley, California, USA, in 1998, when grapes were ripening. An untreated control plot was also maintained. 21 days after the last treatment 45 kg of grapes from each plot were transported to the processing facility, where they were stored at 5°C for 3 months. At harvest, subsamples for analysis were frozen.

Each batch of grapes was fed manually into a crusher/stemmer from which pulp was collected. The stems were discarded. About half of the pulp was pressed for juice in a hydraulic fruit press, the juice filtered and frozen, and the pomace discarded. About 15 kg of grape juice was obtained from 24 kg of grapes.

The remainder of the pulp was processed into red wine. Potassium metabisulfite, pectic enzyme and champagne yeast were gradually added to the pulp and mixed periodically during storage for 6 days. Solids were removed in a hydraulic press and the wine was racked and stored for 6 days at 10 to 13°C, then racked again and stored for 11 days at 5°, shipped to the analytical laboratory and refrigerated but not frozen. Commercial storage intervals (secondary fermentation) would be considerably longer. About 14 kg of wine was obtained from 24 kg of grapes.

The grapes, juice and wine were analysed for propargite, TPBC and TBPC-diol. Samples were extracted with acetonitrile, cleaned up on Florisil followed by an SPE column of alumina, silica and carbon and analysed for propargite and TBPC by GC-MSD, with a demonstrated limit of quantification of 0.01 mg/kg. For the determination of TBPC-diol, the extract was derivatized with trifluoroacetic anhydride, treated with boron tribromide and analysed as 4-(2-bromomethyl-2-propyl)phenol by HPLC with fluorescence detection. The demonstrated limit of quantification was 0.03 mg/kg in wine and grapes. Approximately two months elapsed from generation to analysis of the processed fractions. The grape samples were stored frozen for about 90 days before analysis for propargite and TBPC and for 210 days before analysis for TPBC-diol. Grape juice samples were stored frozen for 88 days (propargite and TBPC) and 105 days (TBPC-diol) and wine samples refrigerated for 63-83 days (Korpalski, 1999b).

In a study in 1993 a WP formulation (300 g/kg) was applied twice with an airblast sprayer in 370 l/ha to Thompson Seedless grapes in California, USA, at the rate of 3.0 kg ai/ha, and the grapes



were picked 30 days later. 25 t were field-dried to raisins on paper trays for seventeen days and were turned once during this time. The raisins were placed on a shaking machine to remove dirt and shipped in wood bins (twenty-five days after harvest) under ambient conditions to a commercial raisin facility. The raisins were again shaken on a machine, and then fumigated with phosphine gas for two months. After additional cleaning including water wash, samples were taken for analysis. Propargite was determined in both grapes and raisins. The fresh grape analytical sample was frozen (-10°C) within two hours of harvest. Propargite was extracted with hexane/2-propanol and the extracts cleaned up by Florisil and gel permeation chromatography. Detection and quantification was by gas chromatography with a flame ionisation detector in the sulfur mode. The demonstrated limit of quantification was 0.05 mg/kg (grapes 97, 100, 100%; raisins 100, 103, 107% recovery). Four months elapsed from harvest (<2 months from completion of processing) to completion of the analyses. (Popadic, 1994e).

Tomatoes. Two processing trials were reported from Italy. In the first in 1997 tomatoes that had been treated with an EW formulation of propargite 15 days before harvest were canned or processed into purée. For canning and for purée the tomatoes were sorted and washed (10 kg samples). For canning they were then peeled, canned in tomato juice, and the sealed cans pasteurised at 100°C for 50 min and cooled. For purée they were pulped, heated at 90°C, screened to remove skins and seeds, and concentrated (85°C, 4% solids to 24% solids) at atmospheric pressure in a steam-jacketed pan (rather than under vacuum as in commercial practice) and canned. The product was therefore subjected to elevated temperatures with unknown effects on the residues. Samples of the tomatoes and finished products were stored frozen until analysis by GC-MS. Recoveries were adequate: control tomatoes at 0.1 mg/kg 105%, canned at 0.01 mg/kg 88%, purée at 0.2 mg/kg 93% and skins at 0.2 and 5 mg/kg 86%, 79% (Cawkwell, 1999).

In the second trial tomatoes that had been treated with an EW formulation 15 days before harvest were processed into canned tomatoes and tomato purée. The processes were as described for the first trial. For tomato purée, about 20 kg of tomatoes yielded 900 g of purée. The solids content was reduced from 5% to 26-32% during concentration. For canning, 10 kg yielded 2.8 kg of canned tomatoes. Skins weighed 1.2-1.5 kg (Harrison, 1999).

Maize. Maize with field-incurred weathered residues was processed by dry milling into grits, meal, flour, and crude and refined oils, and by wet milling into starch and crude and refined oils. The leaves of plants at two separate locations in Texas, USA, were sprayed once in 1993 from fixed-wing aircraft at a rate of 17 kg ai/ha (60-day PHI, blister-growth stage) or 15 kg ai/ha (30-day PHI, dent stage). Maize was harvested in the September by a commercial combine and the ears stored frozen. Processing of the 30-day PHI maize was carried out within 2 months and of the 60-day PHI within 6 months of harvest (Smudin, 1995a).

In the wet milling process, maize samples were dried and cleaned by aspiration and screening, then steeped in water and milled to recover sequentially germ, hulls, coarse gluten-starch, gluten and starch. After drying, the germ was heat-conditioned, flaked and pressed in an expeller to release most of the crude oil. The residual oil in the presscake was extracted with hexane. The two crude oils were combined and refined (NaOH treatment excluding bleaching and deodorizing). The process differed from commercial practice in that it was batch and not continuous. In a typical run, 180 kg of shelled maize yielded 160 kg of dried maize, 4.5 kg germ, 6.7 kg hulls, 3.7 kg gluten, 41 kg starch, 0.92 kg crude oil from pressing, 0.78 kg crude oil from solvent extraction, 0.62 kg refined oil and 0.058 kg soapstock.

In the dry milling process, the maize was dried and cleaned by aspiration and screening, then moisture-adjusted and impact-milled to produce hulls, grits, meal, flour and germ. The germ was heat-conditioned, flaked and pressed to release the oil. Additional oil was recovered from the presscake by hexane extraction. The crude oils were combined and refined by NaOH treatment. A batch process was used, whereas commercial processes are continuous. In a typical run 180 kg of maize yielded 160

kg of dried grain, 30 kg large, 13 kg medium and 8.4 kg small grits, 8.2 kg coarse meal, 8.2 kg meal, 6.0 kg flour, 11 kg germ, 0.86 kg oil by pressing, 0.16 kg oil by solvent extraction, 0.98 kg refined oil and 0.13 kg soapstock.

Samples were analysed by a GLC method, validated at 0.05-0.5 mg/kg. Recoveries were 75-80% for grain, 96-120% for starch, 82-106% for crude oil, 93-101% for meal, 93-110% for flour. In addition, the recoveries of propargite from fortified control samples prepared and analysed with treated samples were acceptable. The interval from harvest to analysis was one year for grain, one year for oil (wet and dry) and two years for other fractions (starch, flour, etc).

Cotton seed. Plants in Mississippi, USA, were treated three times with a propargite EC formulation at a rate of 11 kg ai/ha in 190 l/ha, the third application 2 days before harvest, and seed samples stored for 46 days at 29°C before shipping to the processing facility. The samples were then stored frozen until about 3 months after harvest, when the raw agricultural commodity, undelinted seed, was obtained and stored with the processed commodities at -10 to -20°C until analysed (Polakoff, 1988c).

Processing simulated commercial practice. A gin was used to separate the seed cotton into cotton seed, lint and gin trash, a Carver saw delinter to remove most of the lint from the ginned seed, yielding linters, motes and delinted seed, and a Carver bar huller to decorticate the seed. The cracked seed was passed over a shaker screen to separate hull and kernel fractions, the hulls frozen and the kernels pre-heated to 74°C, flaked and extracted at 63°C with hexane for 3 hours. The solvent was drained and the residual flake dried with warm air, collected and frozen. The crude oil was recovered from the hexane in a laboratory evaporator at 85°C. The crude oil was treated with NaOH, the amount depending on the free acid content of the crude oil. Refined oil and soapstock were collected and frozen.

Some 10.4 kg of cotton seed yielded 9.4 kg delinted seed, 0.64 kg linters, 0.18 kg motes, 6.3 kg kernels, 3.0 kg hulls, 1.9 kg crude oil, 4.4 kg meal, 0.20 kg soapstock and 1.6 kg refined oil.

The cotton seed and processed fractions were analysed by a GLC procedure with a sulfur-phosphorus emission detector in the sulfur mode. The method was validated for oil (0.1-1.0 mg/kg) and hulls (0.05-5.0 mg/kg). Fortified seed controls were prepared and analysed with the treated samples, with recoveries ranging from 76 to 92%.

Sorghum. Sorghum was treated with an EC formulation at 1.8 kg ai/ha and 19 l/ha by air in Illinois, USA. About 150 grain heads (16 kg) were harvested by hand 30 days later, and milled by wet and dry procedures. The dry milling consisted of abrasive decortication to remove the pericarp and germ. The major fractions were starch endosperm (flour, grits, or meal, depending on particle size), bran (extreme outer layer of the pericarp) and shorts. The wet milling procedure consists of steeping the grain at 50°C for 40 hours in an aqueous solution of sodium bisulfite and lactic acid. The drained grain is blended with water, which completes the disintegration of the tissue and filtered, with protein and starch passing through the filter and germ and bran being retained. A gravity flow table is used to separate protein and starch, with the starch remaining on the table.

Grain samples were shipped directly from field to laboratory and stored frozen within 2 days at 4°C for 4 months before processing. The processed fractions were stored frozen until analysis, less than one month later, by the method used for cotton seed fractions. The method was validated for grain, bran and starch at concentrations of 0.05-10 mg/kg (Polakoff, 1988l).

Potatoes. An EC formulation was applied to potatoes at a rate of 11.4 kg ai/ha in Washington, USA, in 1993, and the potatoes harvested 14 and 21 days later for processing into chips, flakes and wet peel. No propargite residues were found in the fresh tubers (<0.05 mg/kg) or in the chips and flakes, but in the dried peel residues were 0.15-0.45 mg/kg. Processing factors could not be determined (Popadic and Smudin, 1995).

Peanuts. An EC formulation of propargite was applied to a plot of plants at a rate of 9.1 kg ai/ha in North Carolina, USA, in 1993. The PHI was 14 days, and a second plot was treated at a rate of 9.5 kg ai/ha and the peanuts were dug 21 days later and stored frozen until processed.

The peanuts were dried and then cleaned by aspiration and screening. A mechanical sheller was used to crack the hulls and aspiration to separate the hull particles from the kernels. The kernels were heat-conditioned and pressed in an expeller to release most of the crude oil. The presscake was flaked and extracted with hexane. The crude oils were combined and refined by treatment with NaOH. All processed fractions were immediately frozen. 46 kg of unshelled peanuts yielded 31 kg of kernels and ultimately 3.1 kg crude oil from expelling and 2.0 kg of oil from solvent extraction.

Samples were extracted, cleaned up (including gel permeation for oils) and analysed by GLC (FPD). The validated limit of quantification was 0.05 mg/kg. Processing and analyses were completed within 10 months of harvest (Smudin, 1995b).

Mint. In a processing trial in Washington, USA, an EC formulation of propargite (740 g/kg) was applied in 200 l water/ha as a broadcast spray at 2.3 kg ai/ha at two separate locations, and the tops of the mint harvested 14 days later, and frozen in plastic bags. The remainder (about 25 kg from each replicate at each site) was dispatched under ambient conditions for processing into oil. At the processing facility, the mint tops were kept in cold storage (4.5°C).

Simulated commercial processes were used. Fresh tops in cloth mesh bags (3.2 kg) were placed in a cooker (eight cookers per run), compressed, and steam passed through to distil the oil. The distillate was passed through a condenser connected to a collection tube, where oil was separated from the co-distilled water. The process required one hour. The oil was immediately frozen. Two runs were conducted for each of the two locations. Processed samples ranged from 25 kg to 44 kg and the ratio of the weight of the oil to that of the mint tops ranged from 0.0014 to 0.0037.

The mint top and oil samples were extracted with acetonitrile and partitioned with hexane. The acetonitrile was extracted with petroleum ether and the latter fraction was cleaned up on a Florisil column. Mint oil extracts were cleaned up on an Alumina-N column. Final extracts were analysed for propargite and TBPC by GC-MS in the selective ion mode. The method was validated for both analytes at 0.01 mg/kg in mint tops and at 0.10 mg/kg in mint oil. Analyses were completed within 6 months of harvest. The results are shown in Table 75 (Korpalski, 2001).

Hops. In a 1990 US processing study hops at two locations in Washington were sprayed with a WP formulation of propargite (300 g/kg) at 2 x 5.0 kg ai/ha or 3 x 8.4 kg ai/ha from pre-bloom through the large cone growth stages with a tractor-mounted airblast sprayer at 940 l/ha and harvested 14 days after the last application with a hop-picking machine. Approximately 1 kg samples of green hops were immediately frozen for later analysis and the remaining hops transported to a commercial kiln and dried.

Processing was at the Anheuser-Busch Pilot Brewery within 3 months. A mass of standard production barley malt and rice was boiled with the treated dried hops and strained to produce the wort and recover the spent hops. The resulting liquor was fermented and finished into beer according to standard brewing practices. The highest hopping rate was used, 125 g/hl. Samples of dry cones, wort, and spent hops were stored frozen for analysis. Samples of bottled beer were refrigerated.

Green, dry and spent hops were extracted with hexane, beer and wort with hexane/2-propanol, and the extracts cleaned up by gel permeation chromatography. The demonstrated limits of determination were 0.01 mg/kg for beer and wort, 0.1 mg/kg for green and spent hops and 1.0 mg/kg for dry hops (Ball, 1992).

Tea. In a trial in Japan two applications of an EC formulation (570 g/kg) were made with a backpack pump sprayer at an interval of 14 days to mature plants at a rate of 1.5 and 3.0 kg ai/ha, or 2.7 and 5.3 l/ha. 21 and 35 days later 2.5 to 4 kg samples of leaves were taken, steam treated and frozen for one week. The leaves were rolled for 30 min at 90°C, rolled and twisted for 15 min at ambient temperature, middle rolled for 30 min at 34-38°C, fine rolled for 28-38 min at 75-80°C, and hot-air dried for 20-25 min at 80-90°C, simulating commercial Japanese practice. The final green tea samples were stored frozen in plastic bags.

In the laboratory, 20 g of dried tea was steeped in two 600 ml portions of boiled deionized water. Brewed tea, fresh tea and dried green tea were extracted with hexane/2-propanol and cleaned up by Florisil and gel permeation chromatography. Brewed tea was similarly extracted, but GPC was omitted. The extracts were analysed by gas chromatography with a flame photometric detector. The limit of quantification for the fresh and dried teas was 0.05 mg/kg and for brewed tea 0.01 mg/kg (Korpalski, 1996e).

In another study in Kenya an EC formulation (570 g/kg) of propargite was applied using a backpack pump sprayer to mature plants at two locations. At the first the application rate was 1.14 kg ai/ha and 250 l/ha, and at the second 0.86 kg ai/ha in 500 l/ha. 7, 14 and 21 days after treatment, a large sample (15 kg) of fresh leaves was taken at each location. A small sample was frozen and the remainder processed into black tea.

The tea leaves were withered at ambient temperature for 14-18 h, macerated and fermented at ambient temperature for 90 min, then dried at 80-120°C to stop fermentation and to reduce the moisture content to commercial standards.

Instant tea was generated from the black tea by creating a strong brew, concentrating by evaporation and freeze drying. The concentrate was milled.

The fresh and dry teas were extracted with hexane/2-propanol and the extracts cleaned up by Florisil and gel permeation chromatography. For brewed tea, dried tea (20g) was steeped in two 600 ml portions of boiled water and then extracted with hexane/2-propanol. Only Florisil column clean-up was used. A similar procedure was used for instant tea, but gel permeation chromatography was necessary. Final extracts were analysed by gas chromatography with a flame photometric detector. The limits of determination were 0.05 mg/kg for fresh and black tea and 0.01 mg/kg for instant and brewed tea (Korpalski, 1997a).

At two locations in Indonesia an EC formulation of propargite (570 g/kg) was applied three times to plants at 0.57 and 1.14 kg ai/ha in an aqueous mixture of 400 l/ha using a knapsack pump sprayer. Two large samples of 12 to 21 kg were taken from each plot 7 days after the third spray. A sub-sample of fresh leaves was immediately frozen. One large sample was dispatched to a black tea processing facility where the leaves were withered with warm air for 18 h, rolled (30 min), ground in a rotorvane (0.25-1 h), fermented by spreading on a tray at ambient temperature (1 h) and dried under hot air (0.67-1 h). The other large sample was sent to a green tea processing facility and the leaves withered in a rotary dryer (7 min), rolled (15-20 min) and dried in a rotary dryer (1-2 h). Processed samples of about 600 g each of both black and green teas were taken for analysis.

At the laboratory the green and black teas were brewed. About 20 g of dried tea was steeped in two 600 ml fractions of boiling water. The combined 1200 ml was extracted with hexane/2-propanol. Fresh, green and black teas were also extracted with hexane/2-propanol. The extracts were cleaned up by Florisil and GPC chromatography and analysed by gas chromatography with a flame photometric detector. The limits of determination were 0.05 mg/kg for the teas and 0.01 mg/kg for the brew (Korpalski, 1996a).

## **RESIDUES IN ANIMAL COMMODITIES**

### Farm animal feeding studies

Dairy cows. Twelve Holstein dairy cows were divided into four groups of three and dosed orally with gelatine capsules at rates equivalent to 0, 50, 150, or 500 ppm in the diet for 28 consecutive days (Singh, 1991b; Batorewicz and Noon, 1991; Batorewicz, 1993). The doses were based on feed consumption measured during the acclimation period before commencement of the study. Milk was collected morning and night and the cows slaughtered 24 hours after the last dose and tissue samples collected. Residues of TBPC in the milk were detected only at the 500 ppm dose.

Table 76. Propargite, TBPC and TBPC-diol residues (mg/kg) in milk (Singh, 1991b; Batorewicz and Noon, 1991; Batorewicz, 1993).

Feeding level (ppm)	Residue, mg/kg										
	Day										
	0	1	2	3	4	6	7	8	9	11	
<i>Propargite</i>											
0	<0.01	<0.01						<0.01		<0.01	
50	<0.01	<0.01						<0.01		<0.01	
150	<0.01	<0.01						<0.01		<0.01-0.02	
500	<0.01	0.02-0.07						0.05-0.24		0.1-0.22	
<i>TBPC</i>											
500 <sup>1</sup>		<0.02-0.02	0.02-0.08		0.02	0.08			0.03-0.09	0.04-0.10	0.03-0.12
<i>TBPC-diol</i>											
50		<0.02									
150		0.02		0.05		0.07					
500		0.08			0.11						
Feeding level (ppm)	Day										
	11	12	13	14	16	19	20	21	24	27	28
<i>Propargite</i>											
0		<0.01		<0.01					<0.01		<0.01
50		<0.01-0.01		<0.01-0.01					<0.01-0.01		<0.01-0.01
150		0.02		0.02					0.02		0.02
500		0.22-0.42		0.26-0.39					0.30-1.6		0.38-2.7
<i>TBPC</i>											
500	0.03-0.07			0.05-1.2	0.04-0.57	0.19-0.43			0.05-0.97	0.42-0.63	0.08-1.4
<i>TBPC-diol</i>											
50				<0.02					<0.02		0.02
150			0.06					0.04		0.07	
500				0.20					0.16		0.28

<sup>1</sup> <0.02 mg/kg TBPC in all samples from 50 and 150 ppm feeding levels.

Table 77. Propargite, TBPC and TBPC-diol residues (mg/kg) in cattle tissues (Singh, 1991b; Batorewicz and Noon, 1991; Singh and Batorewicz, 1993; Batorewicz, 1993).

Sample	Feeding level (ppm)	Residues, mg/kg		
		Propargite	TBPC	TBPC-diol
muscle	50	<0.01-0.02	<0.02	na
	150	0.02-0.03	<0.02	0.021
	500	0.14	0.12-2.1	0.22
liver	50	<0.01	0.16-0.24	0.19
	150	0.02-0.04	0.29-1.8	0.62

Sample	Feeding level (ppm)	Residues, mg/kg		
		Propargite	TBPC	TBPC-diol
	500	0.09-0.52	3.4-9.7	4.3
kidney	50	<0.01	0.03-0.06	na
	150	<0.01	0.14-0.16	0.11
	500	<0.01-0.01	0.97-4.3	1.5
fat	50	0.09-0.20	<0.04-0.05	na
	150	0.55-0.84	0.12-0.13	na
	500	6.8-30.	1.3-14.0	na

na: not analysed

No abnormalities were detected in the cows at the end of the study. Two of the three cows dosed with 500 ppm lost weight, and showed reduced feed consumption and milk production as the study progressed. The identity of the propargite residues in milk and fat and TBPC residues in milk, muscle, fat, kidney and liver was confirmed by GC-MS (Burger, 1992; Singh and Batorewicz, 1993).

Poultry. Eighty White Leghorn hens divided into four groups of 20 were dosed with gelatine capsules at rates equivalent to 0, 5, 15, or 50 ppm propargite in the feed for 28 consecutive days (Singh, 1991b; Batorewicz and Noon, 1991; Batorewicz, 1993; Batorewicz and Singh, 1993). The levels were based on measurements of feed consumption during an acclimatization period, with an average value of 110 g feed/bird/day. Similar feed consumption were noted during the study. Eggs were collected on days 1,2, 7, 14 and 28 and the hens killed 24 hours after the last dose. Residues of TBPC were not detected in muscle (<0.02 mg/kg), fat (<0.04 mg/kg) or liver (<0.04 mg/kg).

Table 78. Propargite, TBPC and TBPC-diol residues in the tissues and eggs of hens (Singh, 1990; Batorewicz and Noon, 1991; Batorewicz, 1993; Singh and Batorewicz, 1993) (not all samples analysed for all analytes).

Sample	Day	Dose, ppm in feed	Residues, mg/kg		
			propargite	TBPC	TBPC-diol
eggs	1	15	<0.01	na	<0.02
	1	50	<0.01	<0.02	<0.02
	4	50	na	na	0.03
	7	50	<0.01	<0.02	0.06
	8	15	na	na	na
	14	50	<0.01	<0.02-0.02	0.06
	15	15	na	na	<0.02
	21	50	<0.01	na	0.03
	27	15	na	na	<0.02
	28	50	<0.01	<0.02	0.04
fat		0, 5	<0.01	na	na
		15	0.01-0.02	na	na
		50	0.08	<0.04	na
liver		15	na	na	<0.02
		50	na	<0.04	0.042
muscle		50	na	<0.02	<0.02

na: not analysed

No abnormalities were detected in the hens at the end of the study, nor any changes in body weight or feed consumption during it although some hens laid soft-shelled eggs which were discarded.

## RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Data on residues in 7 fruit crops were reported from Australia (Table 79).

Table 79. Residues of propargite detected during monitoring in Australia (Victoria) 1987-2000 (Simpson and Hamilton, 2001).

Sample	No. of samples with residues below LOQ (0.05 mg/kg)	Residues above LOQ, mg/kg
Grape	20	
Blackberry	2	
Blueberry	5	
Raspberry	1	
Strawberry	51	0.88, 1.5
Apple	66	0.28, 0.3, 0.31, 0.39, 0.49, 0.5(4), 0.53, 0.58, 0.7, 1.0, 1.2, 1.24, 1.4
Pear	64	0.09, 0.12

## NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

Commodity	Australian MRL (mg/kg)	German MRL (mg/kg)
Apple	3	
Banana	3	
Cotton seed	0.2	0.1
Blackcurrants	3T	
Cucumber including gherkins		0.5
Edible offal (mammalian)	0.1*	
Eggs	0.1*	
Hops, dry	3	30
Mangosteen	3T	
Meat (mammalian, fat)	0.1*	
Milks	0.1*	
Other fruit		3
Other food of plant origin		0.01*
Passion fruit	3	
Pear	3	
Poultry meat (fat)	0.1*	
Poultry (edible offal)	0.1*	
Stone fruit	3	
Strawberries	7	
Tea		5
Vegetables	3	

\* at or about the analytical limit of quantification

T: temporary

## APPRAISAL

Propargite [2-(4-*tert*-butylphenoxy)cyclohexyl prop-2-ynyl sulfite] is an acaricide. It is widely registered for foliar use, primarily on fruits, cotton, hops and tea. It was first evaluated for residues in 1977, followed by additional considerations in 1978, 1979, 1980, and 1982. Toxicological assessments of propargite were performed by the 1977, 1980, 1982, and 1999 JMPRs. The 1999 JMPR session determined that the acceptable daily intake for humans is 0 – 0.01 mg/kg bw and that an acute reference dose is not necessary. The present review of residues is part of the periodic review program.

The manufacturer has submitted data on metabolism, analytical methods of analysis, animal transfer (feeding) studies, supervised field trials, GAP, processing, frozen storage stability of residues,

and environmental fate. Australia submitted information on GAPs, labels, and residues in food in commerce or at consumption and national residue limits. Thailand submitted information on GAPs and Germany submitted information on GAPs and national MRLs.

Propargite is currently formulated as wettable powders and as emulsifiable concentrates. It is a viscous liquid with low solubility in water (<1 mg/l). Its octanol/water partition coefficient (4 - 6) suggests that it is fat soluble.

### **Animal metabolism**

The metabolism of <sup>14</sup>C-propargite has been studied in the rat, goat, and hen. The radiolabel is uniformly distributed in the phenyl ring. In ruminants and poultry, propargite undergoes hydrolysis, thereby losing the propynyl sulfite side chain and generating 2-(4-*tert*-butylphenoxy)cyclohexanol (TBPC). The TBPC undergoes oxidation on the *tert*-butyl group and/or on the cyclohexanol ring, yielding diols and triols. The hydroxymethyl-TBPC is further oxidized to carboxy-TBPC, carboxy-TBPC-diol, and carboxy-TBPC-triol. The various carboxy and hydroxy compounds were found to form sulfate and glucuronide conjugates. For goats, the major residue in fat and milk was propargite, about 60% and 45%, respectively. The major metabolites in muscle were TBPC-diol (20%) and free and conjugated carboxy-TBPC (45%). The major metabolite in liver and kidney was carboxy-TBPC, free and conjugated, about 25%. Propargite was minor to absent in liver, kidney, and muscle.

A similar situation was found with chickens. From the oral administration of radiolabelled material, propargite was found in egg yolk (10%) and fat (50%), but was absent in kidney, muscle, and egg white. The major metabolite in these matrices was hydroxymethyl-TBPC-diol, 40%, 40%, 60%, respectively.

The rat metabolism study was reviewed by the 1999 JMPR. The same metabolites were found in the rat studies previously considered as those reported for goats and hens.

### **Plant metabolism**

The metabolism of <sup>14</sup>C-propargite has been studied on corn, apple, potato, and beans. The radiolabel is uniformly distributed in the phenyl ring. In corn, the major metabolite on kernels harvested six weeks after application was hydroxymethyl-TBPC-diol (45%), although propargite was present (10%), whereas in forage (3 weeks after application) and stover propargite was the major component of the residue, 40% and 25%, respectively.

Apple fruits and leaves were painted with radiolabelled propargite and harvested 23 days later. About 30% of the total radioactive residue on the apple was removable with acetone or acetone/water wash of the whole fruit. The pulp (peeled fruit) contained about 1% of the total radioactive residue in/on the fruit. The remaining 68% was on the (washed) peel. In the pulp, 30% of the residue present was propargite, and the major metabolite was hydroxymethyl-TBPC at 30%. Some 90% of the residue on the peel was propargite. On washed leaves, 60% of the remaining residue was propargite and 25% was TBPC.

Potato vines were sprayed with a radiolabelled formulation and harvested 3 weeks later. The total radioactivity on potato peels (fresh weight) was 0.012 mg/kg and on tubers (fresh weight) 0.004 mg/kg. The radioactivity on the vines (270 mg/kg, dry weight) was examined. Propargite comprised 30% of the total residue on vines, hydroxymethyl TBPC-diol comprised 15%, and hydroxymethyl TBPC comprised 10%.



Green bean pods were painted or sprayed with radiolabelled propargite and harvested 7 days later. About 80 -90% of the total radioactive residue was propargite. TBPC was a minor component (1%).

The studies are consistent with a metabolism that involves hydrolysis to TBPC and oxidation of TBPC to hydroxymethyl-TBPC and hydroxymethyl-TBPCdiol. TBPC diol and hydroxymethyl-TBPC triol were also found in some studies, but carboxy-TBPC derivatives were never found. Also, the potato and apple studies indicate that propargite does not translocate.

## **Environmental fate**

### *Soil*

Confined rotational crop studies with radiolabelled propargite were not provided. However, field rotational crop studies with propargite were submitted. Wheat, carrot, and lettuce were rotated with cotton that had been treated 3 times at 1.8 kg ai/ha with propargite. With plantback intervals of 82 and 120 days, no propargite (<0.05 mg/kg), no TBPC (<0.04 mg/kg), and no TBPC diol (<0.02 mg/kg) were found in any commodity at normal harvest. In another study, barley, carrot, radish, and lettuce were rotated with cotton that had been treated three times at rates of 1.8 or 3.7 kg ai/ha. The plantback intervals were 60 days and 119 days. The maximum residues found were in carrot root, 0.16 mg/kg for propargite and 0.02 mg/kg for TBPC at 119 days and 3.7 kg ai/ha. In all other cases, propargite residues were  $\leq 0.05$  mg/kg and TBPC residues were  $\leq 0.01$  mg/kg, with the exception of barley straw, 0.09 mg/kg propargite. These findings were confirmed by additional similar studies.

The Meeting concluded that propargite may persist in root type rotational crops for plantback intervals of 120 days or less, with potential residues at longer plantback intervals unknown. Residues in other food crops are none or minimal (<0.05 mg/kg) at plantback intervals of 60 days or greater.

The aerobic degradation of propargite in sandy loam soils proceeded with a calculated first order kinetics half-life of 40 - 60 days. Extractable residue (acetone or methanol) decreases from about 100% on the day of application to 30% by day 90 - 100. At day 100, carbon dioxide accounted for 40% of the applied radioactivity. After 365 days, 9 metabolites were detected, including TBPC, p-tertiarybutyl phenol (PTBP), and TBPC-sulfate.

The anaerobic degradation of propargite in sandy loam soils yielded propargite (40% applied radioactivity) and TBPC (20% applied radioactivity) as the major components after 60 days. The time to 50% degradation was calculated by linear regression to be 65 days.

The mobility of propargite in 6 soil types was studied. Propargite was strongly adsorbed by all soil types and may be considered only slightly mobile. The mobility of TBPC was also measured in numerous soil types. It was not adsorbed and was easily desorbed. The metabolite TBPC may be classified as very mobile.

When propargite was applied to orange trees with an airblast sprayer and soil samples were taken at various intervals and depths, neither propargite nor TBPC were detected beyond the first 15 cm for post-treatment intervals up to one year. In a study with cotton, propargite was found in the 15 - 30 cm cores (0.1 mg/kg) and 30 - 60 cm cores (0.07 mg/kg) within less than 4 days of application, but declined to <0.05 mg/kg by day 7. TPBC was found (0.1 mg/kg) at the 30- 60 cm depth at 4 - 7 days after application. Again, propargite appears not to be mobile.

Numerous field dissipation studies were reported, wherein crops bordering bodies of water were sprayed with propargite and the residue of propargite in the water and sediment were determined as a function of time. Generally, residues were as great as 0.1 mg/kg in sediment and 0.12 mg/kg in

water immediately after the treatments. Sediment residues declined to <0.025 mg/kg after 10 days, and concentrations in water declined to <0.005 mg/kg over 10 days to 4 months.

The photolysis of propargite on soil showed a half-life of about 60 days with full sunlight (no dark periods) based on a 20-day study. TPBC was identified as a degradate.

#### *Water-sediment systems*

The hydrolysis of radiolabelled propargite at various pHs revealed that propargite's stability decreases with increasing pH, with a half life of 100 - 700 hours at pH 5 and 2 - 3 hours at pH 9.

The aerobic degradation of radiolabelled propargite in a pond water/sand sediment mixture led to a calculated 50% loss of propargite in 38 days. The composition of the water/sand extract as a percentage of the applied radioactivity on day 30 was 60% propargite, 26% TBPC, 0.1% carboxy-TBPC compounds, 0.3% hydroxymethyl-TBPC, and 1% PTBP. Less than 1% of the applied radioactivity was recovered as volatiles.

The anaerobic degradation of propargite was studied in a lake water/pond sediment system spiked with glucose and purged with nitrogen. The radioactivity extractable with ethyl acetate decreased from 96% on day 0 to <50% after one year. The levels of radioactivity in the water fraction remained low (13% maximum). TBPC maximized at 60% of the applied dose on day 270. The calculated half-life in "hydrosol" was about 50 days.

The Meeting concluded that propargite is not mobile in soils and that it degrades under various conditions in soil and sediment/water with half-lives of 40 - 60 days, forming TBPC, which may further degrade to various diols. Under aerobic conditions in soil, significant degradation to carbon dioxide may occur. Because it is not mobile, propargite may accumulate in rotational root crops such as carrots when short plantback intervals are used.

#### **Methods of analysis**

Several methods were provided for the determination of propargite in raw and processed agricultural commodities. The method most frequently used entails sample maceration, extraction with solvent, purification on Florisil and/or alumina columns and/or gel permeation chromatography, followed by determination of the extracts by gas chromatography with a flame photometric detector in the sulfur mode. This method, with modifications such as the use of capillary columns and different extraction solvents, has been traditionally used for data collection in field trials and animal feeding studies. It is also the basis of the enforcement method in the United States, with limits of quantification of 0.1 mg/kg, except 0.08 mg/kg for milk. Where used for data collection with modifications, the demonstrated limits of quantification are 0.01 - 0.05 mg/kg.

More recently, gas chromatography/mass spectrometry (GC/MS) has been substituted for the flame photometric detector. Usually the MS is operated to monitor ions specific to propargite. The limits of quantification are generally 0.01 - 0.05 mg/kg.

An HPLC method has also been used for residue determinations in field trials, especially for fruits. Extracts are purified on solid phase extraction cartridges and analyzed on HPLC, isocratic mode, with a UV detector (225 nm). Acceptable recoveries are reported for 1 - 2 mg/kg fortifications, although a limit of quantification of 0.1 mg/kg is claimed.

The metabolite TBPC has been determined in plant commodities by heptafluorobutyric anhydride (HFBA) derivatization and analysis by GC/ECD. Direct analysis of the extract by GC/MS has also been reported. The limit of quantification is 0.01 mg/kg in both methods. For animal

commodities, the derivatization procedure with GC/FPD has been used, with a 0.02 mg/kg limit of quantification.

The GC/FPD method has been radiovalidated. The method recovered 26% of the total radioactive residue (TRR) from corn forage as propargite, whereas the metabolism study yielded 40%. For milk, the values were 35% from the GC/FPD method and 43% from the metabolism study. The Meeting concluded that the method provided adequate extraction of the target analyte, propargite.

The Meeting concluded that adequate methods exist for the collection of data for the residues of propargite in/on raw and processed agricultural commodities both for monitoring and MRL enforcement purposes.

### **Stability of pesticide residues in stored analytical samples**

Storage stability studies were conducted on about 51 commodities in support of the storage intervals encountered in the various field trials and feeding studies. Most studies indicated stability (>70% remaining) for the longest period studied, typically one year. There were exceptions, mainly forages and fodders. Maize forage and fodder had a 40% loss at 6 - 8 months, barley straw lost 50% of the propargite residue between 9 and 12 months. Study periods for animal commodities were shorter. Thus, propargite in muscle and in kidney was stable for the period studied, 6 months, and stable for the 3 month period in milk, fat (bovine and chicken), liver (bovine and chicken), and eggs.

The Meeting concluded that propargite is stable in frozen plant commodities for about one year, but that animal commodities should be analyzed within 3 months because of the lack of adequate storage stability data for longer intervals.

### **Residue definition**

Whereas propargite forms the majority portion of the residues in the plant metabolism studies, whereas propargite is the major residue component in fat and milk, and a significant portion of the residue in egg yolk, as ascertained from the animal metabolism studies, whereas analytical methods suitable for use by national authorities exist for the determination of propargite in raw and processed plant and animal commodities, whereas analytical methods for the major metabolite TBPC have not been validated as enforcement methods by national authorities and require extensive additional efforts beyond the determination of the parent (derivatization, use of GC/MS), and whereas the 1999 JMPR noted no special concern for the metabolites of propargite, the Meeting concluded that the appropriate residue definition for monitoring and risk assessment was propargite. The Meeting noted that propargite will most likely not be found in the lean muscle, offal, and egg white of animals exposed to propargite in the diet, based on the results of the metabolism studies.

Definition of the residue for compliance with MRLs for plant and animal commodities and for estimation of dietary intake:

Propargite. The residue is fat-soluble.

### **Results of the supervised trials**

Supervised trials were conducted for the foliar application of WP and EC and EW formulations to many crops, primarily in Europe and the USA. Trials were also reported from Asia and Africa for tea.

Trial data were not submitted for several crops with current maximum residue level recommendations: apricot, common bean, cranberry, and fig. The Meeting agreed to withdraw the previous maximum residue level recommendations for these commodities.

Oranges and mandarins. Field trial data was received from Spain, California (USA), and South Africa. The GAP for Spain is 1.1 kg ai/ha in at least 4000 l water/ha with a 14 day PHI for the EC, WP, and EW formulations. Four trials each for oranges and mandarins were at GAP. Oranges: 0.22, 0.28, 0.55, and 0.61 mg/kg; Mandarins: 0.19, 0.33, 0.71, and 0.77. The GAP for the USA is use of the WP (CR) formulation at 3.8 kg ai/ha in 9400 l water/ha, 28 day PHI. No trials were at the GAP conditions. The GAP for South Africa is 3.6 kg ai/ha in 6000 l/ha of the WP formulation, or 0.06 kg ai/hl, 14 day PHI. Three trials were at GAP for oranges: 0.26, 1.5, and 2.1 mg/kg. Combining the values for oranges and mandarins for mutual support, the residues in ranked order are: 0.19, 0.22, 0.26, 0.28, 0.33, 0.55, 0.61, 0.71, 0.77, 1.5, and 2.1.

Residue values for pulp were supplied for the trials from Spain (<0.01 (5), 0.01, 0.02 (2) mg/kg) and South Africa (<0.1 (2), 0.34 mg/kg). The ranked order of the residues is: <0.01 (5), 0.01, 0.02 (2), <0.1 (2), 0.34. The Meeting estimated an STMR of 0.01 mg/kg for orange and mandarin pulps.

Lemons. Field trial data were reported from the USA. However, the data did not support the current GAP: 3.8 kg ai/ha and 28 day PHI. All data were for a 7 day PHI and a 5 kg ai/ha application rate.

Grapefruit. Field trial data were reported from the USA. However, the data did not support the current GAP: 3.8 kg ai/ha and 28 day PHI. All data were for a 7 day PHI and a 5 kg ai/ha application rate.

Citrus. The Meeting agreed to withdraw the previous maximum residue level recommendation for citrus fruits (5 mg/kg), to be replaced by a new recommendation for citrus (3 mg/kg).

Apple. Field trial data were received from the Czech Republic, Brazil, Hungary, Moldova, Italy, France, and the USA. The USA has no GAP for apples, and the trials are discarded. No GAP was available for the Czech Republic, but the GAP of Hungary may be applied (1.1 kg ai/ha, 10 or 14 day PHI). The data for the two trials do not support this GAP.

Two trials were submitted from Brazil, but no relevant GAP was available.

Three trials from Hungary may be evaluated against the critical GAP of Hungary: WP, 1.8 kg ai/ha, 10 day PHI. No trials support the GAP

One trial from Moldova is not supported by the Moldova GAP: 1.7 kg ai/ha, 45 day PHI.

Ten trials from Italy support the Italian GAP: EC, EW, WP 0.9 kg ai/ha, 1000 l/ha water minimum, 15 day PHI. The residues are: <0.01, 0.01, <0.10 (5), 0.22, 0.58, 0.65 mg/kg. In addition, four trials from France may be evaluated against the Italian GAP: 0.11, 0.16, 0.21, and 0.24 mg/kg.

Twenty trials from France support the French GAP: WP, 1.5 kg ai/ha, 500 l/ha water minimum, 7 day PHI. The residues are: 0.2, 0.21, 0.29, 0.44, 0.47, 0.55(2), 0.60, 0.64(2), 0.73(2), 0.79, 0.8, 0.81, 0.94, 1.1, 1.2, 1.7, and 1.8 mg/kg.

Combining the values from Italy and France gives the following ranked order for 34 trials: <0.01, 0.01, <0.10 (5), 0.11, 0.16, 0.2, 0.21(2), 0.22, 0.24, 0.29, 0.44, 0.47, 0.55(2), 0.58, 0.60, 0.64(2), 0.65, 0.73(2), 0.79, 0.8, 0.81, 0.94, 1.1, 1.2, 1.7, and 1.8 mg/kg. The Meeting estimated an STMR of 0.51 mg/kg. The Meeting agreed to withdraw the previous maximum residue level recommendation level for apple (5 mg/kg), to be replaced by a new recommendation for apple (3 mg/kg).

Pear. Numerous trials for pears were submitted from the USA, but the USA does not have a current GAP for the use of propargite on pears. The Meeting recommended withdrawal of its previous recommendation for a maximum residue level on pears (5 mg/kg).

Cherry. Numerous field trials were submitted from the USA, but the USA does not have a current GAP for the use of propargite on cherries (sweet and sour). The Meeting could not make a recommendation for a maximum residue level on cherries.

Plum. Field trials for the use of propargite on plums (prunes) were submitted from France and the USA. The USA has no current GAP for plums. The GAP for France is: WP, 1.2 kg ai/ha or 0.24 kg ai/hl, 21 day PHI. Ten trials support this GAP: 0.38, 0.39, 0.59, 0.63, 0.65, 0.71, 0.74, 0.97, 1.1, 3.0 mg/kg.

Nectarine. Nectarine field trial studies were made available from France and the USA. The GAP in France, using the Peach GAP, is: WP, 1.5 kg ai/ha or 0.3 kg ai/hl, 14 day PHI. Three trials support the GAP: 0.94, 1.0, 1.2 mg/kg. The GAP in the USA is: WP, 3.2 kg ai/ha, 14 day PHI. Two trials support this GAP: 1.3, 1.4 mg/kg. Combining residue values, the ranked order is: 0.94, 1.0, 1.2, 1.3, 1.4 mg/kg.

Peach. Peach field trial studies were reported from France, Hungary, Italy, and the USA. The USA has no current GAP for peaches. The GAP for France is: WP, 1.5 kg ai/ha or 0.3 kg ai/hl, 14 day PHI. Ten trials support this GAP: 0.57, 0.73, 0.80, 0.86, 0.82, 0.87, 0.89, 0.99, 1.2, and 1.9 mg/kg. The GAP for Hungary is: EC, 1.1 kg ai/ha, 10 day PHI at 0.09 kg ai/ha and 14 day PHI at 0.14 kg ai/ha. The two available trials do not support the GAP. The GAP for Italy is: EW, EC, 0.9 kg ai/ha, 0.09 kg ai/hl, 15 day PHI. The one available trial supports the GAP: 0.11 mg/kg. However, the Meeting concluded that the value from Italy is not from the same population as the data of France.

The Meeting agreed that the residue data for peach, nectarine, and plum were from the same population and could be combined. The GAPs are similar, 1.5 – 3.2 kg ai/ha, PHI 14 or 21 days. The 25 values in ranked order are: 0.38, 0.39, 0.57, 0.59, 0.63, 0.65, 0.71, 0.73, 0.74, 0.80, 0.82, 0.86, 0.87, 0.89, 0.94, 0.97, 0.99, 1.0, 1.1, 1.2 (2), 1.3, 1.4, 1.9, 3.0 mg/kg. The Meeting estimated a maximum residue level of 4 mg/kg for stone fruit (excluding cherry). The Meeting further agreed to recommend the withdrawal of previous maximum residue level recommendations for peach (7 mg/kg) and plums (7 mg/kg). The Meeting estimated an STMR of 0.87 mg/kg for stone fruit (excluding cherry) with stone.

Strawberry. Numerous field trials for strawberries were reported from the USA, but the USA does not have a current GAP. The Meeting could not estimate an STMR or maximum residue level. The Meeting recommended withdrawal of the previous recommendation for a maximum residue level for strawberry (7 mg/kg).

Currant. Field trial reports for black currants were supplied from the UK, but no GAP was available. The Meeting could not estimate an STMR or maximum residue level.

Grape. Field trial reports for grapes were provided from the Czech Republic, France, Hungary, Italy, and the USA. The GAP for the Czech Republic is: EW, 0.88 kg ai/ha; WP, 0.6 kg ai/ha, 28 day PHI. The one trial supported the GAP: 0.29 mg/kg.

The GAP for France is: EW, 0.85 kg ai/ha or 0.43 kg ai/hl, 21 day PHI. Twenty-four trials support this GAP: 0.11, 0.18 (2), 0.23, 0.28, 0.29 (2), 0.30 (2), 0.35, 0.38, 0.45, 0.51, 0.6, 0.67, 0.7, 0.8, 0.83, 0.93, 0.96, 1.1, 1.9, 2.4, 2.7 mg/kg.

The GAP for Hungary is: EC, 1.1 kg ai/ha, 10 day PHI with 0.09 kg ai/hl and 14 day PHI with 0.14 kg ai/ha; WP, 0.9 kg ai/ha, 14 day PHI. The one trial supports the GAP: 0.36 mg/kg.

The GAP for Italy is: EW, EC, WP, 0.9 kg ai/ha or 0.09 kg ai/hl, 15 day PHI. Five trials support the GAP: <0.10, 0.26, 0.31, 0.33, 0.48 mg/kg.

The GAP for the USA is: WP, 3.8 kg ai/ha, 28 day PHI. Four trials support this GAP: 0.49, 1.3, 3.4, 4.8 mg/kg.

The combined residue results in ranked order are: <0.10, 0.11, 0.18 (2), 0.23, 0.26, 0.28, 0.29 (3), 0.30 (2), 0.31, 0.33, 0.35, 0.36, 0.38, 0.45, 0.48, 0.49, 0.51, 0.6, 0.67, 0.7, 0.8, 0.83, 0.93, 0.96, 1.1, 1.3, 1.9, 2.4, 2.7, 3.4, 4.8 mg/kg.

The Meeting agree to withdraw the previous maximum residue level recommendation for grape (10 mg/kg), to be replaced by a new recommendation for grape (7 mg/kg). The Meeting also estimated an STMR of 0.45 mg/kg.

Avocado. Two trials were received from the USA on avocado. However, there is no current GAP in the USA for the use of propargite on avocado. Therefore, the Meeting could not estimate a maximum residue level or STMR for avocado.

Cucumber. One field trial study was made available from Hungary. The GAP for Hungary was not available, and the trial does not support the GAP of the Czech Republic: EW, WP, 0.3 kg ai/ha, 5 day PHI. The Meeting could not estimate a maximum residue level or STMR for cucumber. The Meeting agreed to withdraw the previous maximum residue level recommendation (0.5 mg/kg).

Melon. One field trial study was received from France. The GAP for France was not available, but the GAP of Italy may be applied: WP, 0.9 kg ai/ha, 0.09 kg ai/ha, 15 day PHI. The one field trial supports the GAP: 0.05 mg/kg. The Meeting concluded that one field trial was an insufficient data base upon which to estimate the maximum residue level and STMR.

Pepper. One field trial was received from Hungary, but the GAP for the WP formulation was not available. The Meeting could not estimate a maximum residue level or STMR.

Tomato. Field trial studies on tomatoes were received from France, Italy, and the USA. The GAP for France was not available for the one trial from France.

The GAP for Italy is: EW, EC, WP, 0.9 kg ai/ha, 0.09 kg ai/hl, 15 day PHI. Fifteen trials support the GAP: <0.10 (5), 0.14 (2), 0.17, 0.23, 0.27 (2), 0.28, 0.29, 1.4 (2) mg/kg.

One trial study was submitted from the US, but the information was incomplete and the US has no GAP for tomatoes.

Based on the 15 trials from Italy, the Meeting confirmed the previous maximum residue level recommendation for tomato (2 mg/kg). The Meeting also estimated an STMR of 0.17 mg/kg.

Soya bean. Field trial studies were submitted from the USA, but the USA does not currently have a GAP for the use of propargite on soybeans. The Meeting could not estimate a maximum residue level or STMR.

Bean (dry). A single study was submitted from the USA, but the USA does not currently have a GAP for beans (dry). The Meeting could not estimate a maximum residue level and STMR for beans (dry). The Meeting agreed to withdraw the previous maximum residue level recommendation (0.2 mg/kg) for beans (dry).

Potato. The details of two studies in the USA were submitted. The GAP in the USA is: EC, 2.3 kg ai/ha, 14 day PHI, chemigation. The trials support the GAP: <0.05 (2). The Meeting decided that 2 trials provide an insufficient data base upon which to estimate a maximum residue level or an STMR. The Meeting agreed to withdraw the previous recommendation for a maximum residue level of 0.1 (\*) mg/kg.

Maize. A field trial study was submitted from France. The GAP in France was not provided, and available GAPs do not match the trial condition (1.4 kg ai/ha, 41 day PHI).

Field trial studies were submitted from the USA on the foliar application of propargite to corn (maize). The GAP is: EC, 2.8 kg ai/ha, 30 day PHI; California, 1.7 kg ai/ha, 56 day PHI. Nine trials support the GAP, including 4 trials conducted in the USA under the GAP for California: <0.05 (8), 0.06 mg/kg. The Meeting agreed to withdraw the previous recommendation for a maximum residue level (0.1 mg/kg(\*)) and recommended a new maximum residue level (0.1 mg/kg). The Meeting also estimated an STMR of 0.05 mg/kg.

Sorghum. Grain sorghum trials were reported for the USA. The GAP in the USA is: EC, 1.9 kg ai/ha, 30 day PHI silage, 60 day PHI grain. One of the three trials supported the GAP: <0.05 mg/kg. The Meeting concluded that the data base was insufficient to estimate a maximum residue level or STMR and agreed to withdraw the previous recommendation for a maximum residue level for sorghum (5 mg/kg).

Almond. Field trial studies were submitted from the USA. The GAP in the USA is: WP, 3.6 kg ai/ha, 28 day PHI (California and Arizona only). Fourteen trials support the GAP. The ranked order of residues on almond kernels (nutmeats) is: <0.05 (11), 0.05 (2), 0.076 mg/kg. The Meeting agreed to withdraw the previous recommendation for a maximum residue level for almond 0.1 (\*) mg/kg and recommended a new maximum residue level for almonds (0.1 mg/kg). The Meeting also estimated an STMR of 0.05 mg/kg.

Filbert nuts (Hazel nuts). Field trial studies from the USA for the application of propargite to filbert nuts were presented, but the USA currently does not have a GAP for filbert nuts. The Meetings could not estimate a maximum residue level or STMR.

Pecan. Field trial studies from the USA for the application of propargite to pecans were presented, but the USA currently does not have a GAP for pecans. The Meetings could not estimate a maximum residue level or STMR.

Walnut. Field trials were provided for France and the USA. The GAP in France was not provided, but the GAP in Italy for nuts is: WP, 0.9 kg ai/ha, 0.09 kg ai/hl, 15 day PHI.. The single field trial does not support this GAP.

Two trials were reported from the USA, but there is no current GAP for walnuts in the USA. The Meeting could not estimate a maximum residue level or STMR for walnuts. The Meeting agreed to withdraw the previous recommendation for a maximum residue level (0.1 mg/kg (\*)).

Cotton seed. Field trial studies on cotton seed were provided for the USA. The GAP in the USA is: EC, 1.9 kg ai/ha, 50 day PHI. Ten studies support the GAP, and the residues on undelinted cottonseed are in ranked order: 0.095, <0.1 (4), 0.10, 0.11, 0.12, 0.42, 0.44 mg/kg. A single processing study (see below) yielded a processing factor of 0.18 for the delinting process. Delinted cottonseed values in ranked order are: ≤0.02 (5), 0.02 (3), 0.08 (2) mg/kg. The Meeting agreed to withdraw its previous recommendation for a maximum residue level (0.1 mg/kg (\*)) and recommended a new maximum residue level (0.1 mg/kg). The Meeting also estimated an STMR (0.02 mg/kg).

Peanut. Field trials for peanuts were provided for the USA. The GAP in the USA is: EC, WP, 1.9 kg ai/ha, 14 day PHI, with a restriction against grazing and haying. Ten trials support the GAP: <0.05 mg/kg (10). The Meeting confirmed the previous recommendation for a maximum residue level (0.1 mg/kg (\*)) and estimated an STMR (0.05 mg/kg).

Mint. Trials on mint were reported from the USA. The GAP is: EC, 2.5 kg ai/ha, 14 day PHI. The three trials (fresh mint tops) support the GAP: 1.6, 5.2, 5.6 mg/kg. Data were not provided on mint hay. The Meeting agreed to withdraw the previous recommendation for mint hay (50 mg/kg).

Alfalfa. A single trial for alfalfa (fodder, forage) was provided from the USA. The USA has no current GAP for alfalfa. The Meeting decided to withdraw the previous recommendation for maximum residue levels on alfalfa fodder (75 mg/kg) and alfalfa forage (green) (50 mg/kg).

Peanut hay (fodder). Trial studies for the foliar application of propargite to peanut plants were reported for the USA. The GAP is: EC, WP, 1.9 kg ai/ha, 14 day PHI, no grazing or cutting forage for hay. Ten trials support the GAP: 3.6, 3.9, 4.0, 5.6 (2), 5.8, 7.5, 8.2, 8.5, 14 mg/kg. The Meeting agreed to withdraw the previous recommendation for a maximum residue level for peanut fodder (10 mg/kg) and declined to recommend a new maximum residue level for peanut fodder because the US GAP forbids the production of fodder from treated peanuts. Thus, the commodity ought not be available in trade. The Meeting also agreed to withdraw the previous recommendation for a maximum residue level for peanut forage (green) (10 mg/kg).

Maize forage. Field trials were presented from France and the USA. The GAP in France was not provided.

Field trial studies were submitted from the USA on the foliar application of propargite to corn (maize). The GAP is: EC, 2.8 kg ai/ha, 30 day PHI; California, 1.7 kg ai/ha, 56 day PHI. The trials do not support the GAP.

The Meeting agreed to withdraw the previous recommendation for maximum residue levels for maize forage (10 mg/kg).

Maize fodder. Field trial studies were submitted from the USA on the foliar application of propargite to corn (maize). The GAP is: EC, 2.8 kg ai/ha, 30 day PHI; California, 1.7 kg ai/ha, 56 day PHI. The four trials do not support the GAP.

The Meeting agreed to withdraw the previous recommendation for a maximum residue level for maize fodder (10 mg/kg).

Sorghum fodder. One trial was provided for the USA. The GAP in the USA is: EC, 1.9 kg ai/ha, 30 day PHI silage, 60 day PHI grain. The trial supports the GAP: 0.05 mg/kg.

The Meeting concluded that one trial provided an insufficient data base upon which to estimate a maximum residue level and an STMR. The Meeting agreed to withdraw the previous recommendation for a maximum residue level for sorghum straw and fodder, dry (10 mg/kg).

Almond hulls. Field trial studies were submitted from the USA. The GAP in the USA is: WP, 3.6 kg ai/ha, 28 day PHI (California and Arizona only). Fourteen trials support the GAP. The ranked order of residues on almond hulls is: 12, 14, 15, 30, 35 mg/kg. The Meeting estimated an STMR (15 mg/kg) for almond hulls. The Meeting estimated a maximum residue level of 50 mg/kg for almond hulls.

Cotton gin byproducts. Field trial studies were submitted from the USA. The GAP in the USA is: EC, 1.9 kg ai/ha, 50 day PHI. Five trials support the GAP: 1.0, 5.8, 8.4, 16 (2) mg/kg. The Meeting estimated an STMR of 8.4 mg/kg for cotton gin byproducts.

Hops. Field trials on hops were reported for Germany, the UK, and the USA. The GAP for Germany was not available, and the trials do not support the GAPs of France or the Czech Republic. Likewise, the GAP for the UK was not available.

The GAP for the USA is: EC, CR (WP), 1.8 kg ai/ha, 14 day PHI. Twenty trials support the GAP: 6.9, 9.1, 12, 14 (2), 15 (2), 16, 17, 18 (3), 19, 20, 25, 28, 33, 46, 75, 90 mg/kg. The Meeting agreed to withdraw the recommendation for the previous maximum residue level (30 mg/kg) and to recommend a new maximum residue level for hops (dry) (100 mg/kg). The Meeting also estimated an STMR for hops (dry) (18 mg/kg).



Tea. Field trials for the foliar application of propargite to tea were provided for India, Indonesia, Japan, and Kenya. Two trials from India support the GAP of India (0.81 kg ai/ha, 7 day PHI): <0.05, 1.7 mg/kg for black tea. Two trials from Indonesia do not support the Indonesia GAP (0.11 kg ai/hl, no PHI specified) because of no data for post treatment day 0 – 1.. The GAP for Japan is: EW, WP, 0.04 kg ai/hl, 14 day PHI. Two trials support the GAP: 0.16, 0.26 mg/kg on fresh tea leaves. The GAP for Kenya is: EC, 0.86 kg ai/ha, with no PHI specified. No field trial data were available for a 0 or 1 day PHI.. Processing studies (see below) for the production of black tea and green tea yielded processing factors of 8.5 and 3.9 for black tea and 3.9 and 2.3 for green tea. The average factor is 5.0. Using this factor for the Japan samples, the ranked order of residues for tea, black and green, is: 0.05, 0.8, 1.3, 1.7 mg/kg. The Meeting agreed to withdraw the previous recommendation for a maximum residue level for tea, green, black (10 mg/kg) and to replace it with a recommendation for a maximum residue level for tea, green, black (5 mg/kg). The Meeting also estimated an STMR of 1.0 mg/kg.

### **Fate of residues during processing**

Processing studies were presented for 13 raw agricultural commodities. All studies were conducted with field-incurred residues of propargite, typically from application rates in excess of the GAP, and the processing studies simulated commercial practices, except where consumer practices are indicated, i.e., tea brewing and avocado peeling. Propargite concentrated in three types of commodities: oils (peanut, orange, mint, maize), surface residues (sorghum bran, orange peel, apple pomace, maize dust, grape pomace, raisin waste, cotton gin byproducts), and dried commodities (plum prune, grape raisin). This confirms that propargite does not translocate and that it is fat/oil soluble.

The STMRs and MRLs determined above are multiplied by the relevant processing factor to obtain the STMR-Ps and MRL-Ps (where appropriate) for the processed commodities of raw agricultural commodities.

### Orange

Orange, in a single study, was processed into juice, molasses, oil, and dried peel (pulp). The factors were <0.09, 0.25, 23, and 2.6. Using the maximum residue level estimates and STMR estimates for whole orange, the Meeting calculated maximum residue level estimates and STMR-Ps, as appropriate, for juice and orange pulp dry. The STMR for orange juice is 0.05 mg/kg (0.09 X 0.55) and the maximum residue level is 0.3 mg/kg (0.09 X 3).

The Meeting agreed to withdraw the previous recommendation for a maximum residue level for citrus pulp, dry (40 mg/kg) and recommended a new maximum residue level for citrus pulp, dry (10 mg/kg), based on the 2.6 factor and a maximum residue level of 3 mg/kg. The STMR for citrus pulp, dry is 1.4 mg/kg (2.6 X 0.55).

### Apple

Two studies were provided for the processing of apple to apple juice and wet pomace, and one study, with two variants, was presented for the processing of apple to apple pomace (sauce). The factors for apple to juice were <0.07 and <0.03, average 0.05. Applying this factor to the recommendations for apple maximum residue level and STMR yields maximum residue level and STMR-P estimates for apple juice of 0.2 ( 3 X 0.05) and 0.03 mg/kg (0.51 X 0.05), respectively..

Two variations were conducted on the processing of apples to sauce. In one, the apples were peeled before crushing and in the second, the apples were crushed and the peel was strained. The factors were 0.02 and 2.6, respectively. This confirms the presence of the residue on the peel. Using factor 2.6, the STMR-P for apple sauce is estimated as 1.4 mg/kg (2.6 X 0.51).

The processing factors for apple pomace (wet) were 4.2 and 4.1, average 4.2. Applying this factor to the STMR for apple (0.51 mg/kg) yields the STMR-P for apple pomace (wet), 2.2 mg/kg. No information was supplied on water content and/or the study was not extended to a drying process. The Meeting agreed to withdraw the previous recommendation for apple pomace (dry) (80 mg/kg).

### Grapes

Two studies were provided on the processing of grapes into raisins, and two studies were provided on the processing into juice. One study was provided for wine. The STMR for grapes is 0.45 mg/kg and the maximum residue level is 7 mg/kg. Based on average processing factors, the STMR-P for grape juice is 0.05 mg/kg (0.10 X 0.45), and the STMR-P for raisins is 0.72 mg/kg (1.6 X 0.45), and the STMR-P for wine is 0.01 mg/kg (0.02 X 0.45).

The Meeting estimated maximum residue levels for dried grapes (12 mg/kg, 1.6 X 7), for grape pomace dry (40 mg/kg, 4.2 X 7), for grape juice (1 mg/kg, 0.10 X 7), and for wine (0.2 mg/kg, 0.02 X 7). The Meeting confirmed the previous recommendation of a maximum residue level for grape pomace dry (40 mg/kg) and agreed to withdraw the previous recommendation for a maximum residue level for dried grapes (10 mg/kg).

### Tomato

Two studies were provided for the processing of tomatoes to canned tomatoes (skinless) and tomato purée, with average factors of 0.05 and 1.2, respectively. Applying these factors to the STMR for tomatoes (0.17 mg/kg), the Meeting estimated STMR-Ps of 0.01 mg/kg for canned tomatoes and 0.2 mg/kg for tomato purée.

### Maize

Maize was subjected to both dry milling and wet milling processes. The processing factors for refined oil from dry and wet milling were 2.9 and 5.2, respectively. Using the higher factor and the STMR and maximum residue level for maize (0.05, 0.1 mg/kg(\*)), the Meeting estimated an STMR-P and a maximum residue level for maize oil edible of 0.26 mg/kg and 0.5 mg/kg, respectively. The factors for crude oil from dry and wet milling were 2.9 and 5.6, respectively. Using the higher factor and the maximum residue level for maize (0.1 mg/kg (\*)), the Meeting estimated a maximum residue level for maize oil crude of 0.7 mg/kg.

The processing factors for aspirated grain fractions (dust), flour, grits, and meal were 31, 1.6, 0.9, and 1.1. The Meeting estimated STMR-Ps for aspirated grain fractions, flour, grits, and meal of 1.6, 0.08, 0.05, 0.06 mg/kg, respectively. The Meeting recommended maximum residue levels of 0.2 mg/kg for maize flour.

### Cotton seed

A processing study for cottonseed gave processing factors from delinted cottonseed of 3.1 for hulls, <0.07 for meal, and 1.2 for refined oil. Using these factors and the STMR and maximum residue level for cotton seed, 0.02 and 0.1 mg/kg, respectively, the Meeting estimated STMR-Ps for hulls (0.06 mg/kg), meal (0.002 mg/kg), and refined oil (0.02 mg/kg), and the Meeting recommended a maximum residue level processed for cotton seed oil, edible, 0.2 mg/kg.

### Peanut

A processing study for peanuts gave processing factors of 3.0 for crude oil, 2.5 for refined oil, and 0.56 for meal. Using the STMR and maximum residue level for peanut kernels, 0.05 and 0.1 (\*)

mg/kg, respectively, the Meeting estimated STMR-Ps for refined oil (0.12 mg/kg), and meal (0.03 mg/kg) and recommended maximum residue levels processed for peanut oil crude (0.3 mg/kg) and peanut oil edible (0.3 mg/kg).

### Hops

A study was provided on the use of hops (dry cones) to brew beer. The overall factor was <0.043 at both the wort and beer stages. However, this factor exceeds the maximum theoretical factor of 0.001. This discrepancy arises from the lack of a quantifiable residue in the beer from the processing study, i.e., less than the limit of quantitation. Using the STMR for dried hops, 18 mg/kg, the Meeting estimated an STMR-P for propargite in beer (0.02 mg/kg).

## **Residues in animal commodities**

### Dietary burden in animals

The plateau concentration of propargite in cow milk and in eggs was attained slowly (> 2 weeks). Therefore, the STMR and STMR-P values for commodities were used in calculating the dietary burden of dairy and beef cattle and chickens. This burden was then compared with the results of the feeding studies at various exposure levels (ppm) to estimate the maximum residue levels and STMRs in animal commodities (meat, milk, poultry, eggs, etc).

Commodity	Group	STMR or STMR-P (mg/kg)	Dry matter (%)	Residue, dry weight (mg/kg)	Diet Selection (%)			Residue concentration (mg/kg)		
					Maximum/Selected	Beef cattle	Dairy cattle	Poultry	Beef Cattle	Dairy Cattle
Almond hulls	AM	15	90	20	10/10	10/10		1.7	1.7	
Citrus pulp, dry	AB	1.4	91	1.5	20/20	20/20		0.30	0.30	
Cotton seed	SO	0.10	88	0.11	25/25	25/25		0.03	0.03	
Cotton seed hulls	AM	0.06								
Cotton gin byproducts	AM	8.4	90	9.3	20/20	20/20		1.9	1.9	
Cotton seed meal	-	0.002	89	0.002	15/0	15/0	20			
Maize	GC	0.05	88	0.06	80/5	40/5	80/80	0.003	0.003	0.048
Maize grain dust	CF	1.6	85	1.9	20/20	20/20		0.38	0.38	
Peanut meal	-	0.03	85	0.04	15/0	51/0	25/20			0.008
TOTAL					/100	/100	/100	4.3	4.3	0.06

Feeding studies were provided for both chickens and cows. Dairy cattle received daily oral doses of propargite equivalent to feed levels of 0, 50, 150, and 500 ppm for 28 consecutive days. The residue range in milk at the 50 ppm level was <0.01 - 0.01 mg/kg. At the 500 ppm feeding rate, the residues in milk had not attained a plateau by day 28, with a maximum value of 2.7 mg/kg. At the 500 ppm feeding rate, the residue in kidney ranged from <0.01 to 0.01 mg/kg. At the 150 mg/kg feeding rate, the residue in liver ranged from 0.02 – 0.04 mg/kg. At the 50 ppm feeding rate, the residues in tissues were: muscle, <0.01 - 0.02; liver, 0.02 - 0.04 mg/kg; kidney, <0.01 mg/kg; fat, 0.09 - 0.20 mg/kg. Extrapolating from the maximum values at the 50 ppm feeding level to the exposure level of 4.3 ppm, yields the following residue levels: milk, 0.001 mg/kg; muscle, 0.002 mg/kg; liver, 0.004 mg/kg; kidney, <0.001 mg/kg; fat, 0.02 mg/kg

As the current enforcement methods for animal commodities typically rely upon GC/FPD with established limits of quantification of 0.1 mg/kg, except milk at 0.08 mg/kg, the Meeting agreed to recommend maximum residue levels for milks at 0.1 mg/kg (\*) (F) and for meat (from mammals

other than marine animals) at 0.1 mg/kg (\*) (fat). This confirms the previous recommendations for maximum residue levels. The Meeting also estimated a maximum residue level for offal of mammals at 0.1 (\*) mg/kg.

The Meeting estimated STMRs as the residues levels from extrapolation, using the fat value (0.02 mg/kg) for meat. Because the extrapolation was over an order of magnitude, it seemed prudent to use the more conservative maximum values rather than median values for estimating STMRs for mammalian commodities. The estimated STMRs are: meat (fat), 0.02 mg/kg; milk, 0.001 mg/kg; offal, 0.004 mg/kg. The calculations are summarized in the following table:

Dietary burden (mg/kg) Feeding level [ppm]	Propargite total residue, mg/kg				
	Milk Mean	Muscle Highest	Liver Highest	Kidney Highest	Fat Highest
MRL/STMR beef cattle (4.3) [50]		0.0017 0.02	0.0034 0.04	<0.0009 <sup>1</sup> <0.01	0.017 0.20
MRL/STMR dairy cattle (4.3) [50]	0.0009 0.01	0.0017 0.02	0.0034 0.04	<0.0009 <sup>1</sup> <0.01	0.017 0.20

<sup>1</sup> Effectively 0.000 mg/kg. Note results at 500 ppm feeding level.

Laying hens received daily oral doses of propargite equivalent to feed levels of 0, 5, 15, and 50 ppm for 28 consecutive days. After 28 days, the propargite concentration in eggs at all feeding levels was <0.01 mg/kg. The propargite concentration in fat from the 5 ppm feeding level was <0.01 mg/kg. Liver and muscle were not analyzed for propargite, as the metabolism studies indicated that propargite would not be found. The poultry dietary burden is estimated as 0.06 mg/kg. The Meeting confirmed the existing maximum residue levels for poultry meat (0.1 mg/kg \*(fat)) and eggs (0.1 mg/kg \*), and estimated a maximum residue level of 0.1 mg/kg \* for poultry offal. The Meeting estimated the STMRs for poultry meat, offal, and eggs as 0.000 mg/kg each, based on extrapolation from the 5 ppm feed level to the estimated exposure at 0.06 ppm.

The calculations are summarized in the following table:

Dietary burden (mg/kg) Feeding level (ppm) MRL/STMR	Propargite total residue, mg/kg			
	Eggs Highest	Muscle Highest	Liver Highest	Fat Highest
MRL/STMR (0.06) [5]	<0.00012 <sup>1</sup> <0.01	ND <sup>2</sup>	ND <sup>2</sup>	<0.00012 <sup>1</sup> <0.01

<sup>1</sup> Effectively 0.000

<sup>2</sup> Not determined. No expectation of residue (see metabolism).

## RECOMMENDATIONS

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

Definition of the residue for compliance with MRLs and estimation of dietary intake: propargite.

The residue is fat-soluble.

Commodity		MRL, mg/kg		STMR or STMR-P, mg/kg
CCN	Name	New	Previous	
AL 1020	Alfalfa fodder	W	75	
AL 1021	Alfalfa forage	W	50	
AM 738	Almond hulls	50	-	15
TN 0660	Almonds	0.1	0.1*	0.05
FP 0226	Apple	3	5	0.51
JF 0226	Apple juice	0.2	-	0.03
AB 0226	Apple pomace,dry	W	80	
	Apple purée (sauce)			1.4
FS 0240	Apricot	W	7	
VD 0071	Beans (dry)	W	0.2	
	Beer			0.02
FC 0001	Citrus fruits	3	5	0.01
AB 0001	Citrus pulp, dry	10	40	1.4
VP 0526	Common bean (pods and/or immature seeds)	W	20	
	Cotton gin byproducts	-	-	8.4
SO 0691	Cotton seed	0.1	0.1 *	0.02
	Cotton seed hulls	-	-	0.06
	Cotton seed meal	-	-	0.002
OR 0691	Cotton seed oil, edible	0.2	-	0.02
FB 0265	Cranberry	W	10	
VC 0424	Cucumber	W	0.5	
DF 0269	Dried grapes (= currants, raisins and sultanas)	12	10	0.72
MO 0105	Edible offal (mammalian)	0.1 *	-	0.004
PE 0112	Eggs	0.1*	0.1	0
FT 0297	Fig	W	2	
JF 0269	Grape juice	1	-	0.05
AB 0269	Grape pomace, dry	40	40	
FB 0269	Grapes	7	10	0.45
DH 1100	Hops, dry	100	30	18
GC 0645	Maize	0.1	0.1*	0.05
CF 1255	Maize flour	0.2	-	0.08
AS 0645	Maize fodder	W	10	
AF 0645	Maize forage	W	10	
	Maize grain dust	-	-	1.6
	Maize grits	-	-	0.05
CF 0645	Maize meal	-	-	0.06
OC 0645	Maize oil, crude	0.7	-	-
OR 0645	Maize oil, edible	0.5	-	0.26
MM 0095	Meat (from mammals other than marine mammals)	0.1* (fat)	0.1 (fat)	0.02 (fat)
ML 0106	Milks	0.1* F	0.1 F	0.001 F
AM 0738	Mint hay	W	50	
FS 0245	Nectarine	W	7	
JF 0004	Orange juice	0.3	-	0.05
FS 0247	Peach	W	7	
SO 0697	Peanut	0.1 *	0.1 *	0.05
AL 0697	Peanut fodder	W	10	
AL 1270	Peanut forage (green)	W	10 fresh weight	
	Peanut meal	-	-	0.03
OC 0697	Peanut oil, crude	0.3	-	
OR 0691	Peanut oil, edible	0.3	-	0.12
FP 0230	Pear	W	5	
FD 0014	Plums (including Prunes)	W	7	
VR 0589	Potato	W	0.1 *	
PM 0110	Poultry meat	0.1* (fat)	0.1 (fat)	0
PO 0111	Poultry, Edible offal of	0.1 *	-	0
GC 0651	Sorghum	W	5	
AF 0651	Sorghum forage (green)	W	10 fresh	

Commodity		MRL, mg/kg		STMR or STMR-P, mg/kg
CCN	Name	New	Previous	
			weight	
ASb0651	Sorghum straw and fodder, dry	W	10	
FS 0012	Stone fruits	4	-	0.87
FB 0275	Strawberry	W	7	
DT 1114	Tea, Green, Black	5	10	1.0
VO 0448	Tomato	2	2	0.17
	Tomato purée	-	-	0.20
TN 0678	Walnuts	W	0.1 *	
	Wine		-	0.01

## DIETARY RISK ASSESSMENT

### Long-term intake

The International Estimated Daily Intakes of propargite, based on the STMRs estimated for 19 commodities, for the five GEMS/Food regional diets were in the range of 2% to 10% of the ADI (Annex 3). The Meeting concluded that the long-term intake of residues of propargite resulting from its uses that have been considered by JMPR is unlikely to present a public health concern.

### Short-term intake

The 1999 JMPR decided that an acute RfD is unnecessary. The Meeting therefore concluded that the short-term intake of propargite residues is unlikely to present a public health concern.

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