CHLORPROPHAM (201)

IDENTITY

ISO common name: chlorpropham

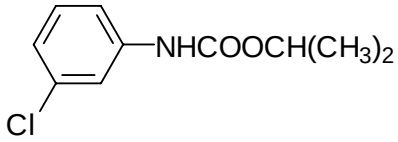
Chemical name:
IUPAC: isopropyl 3-chlorocarbanilate
CA: 1-methylethyl (3-chorophenyl)carbamate

CAS Registry no.: 101-21-3

CIPAC no.: 0043

Synonyms: CIPC

Structural formula:



Molecular formula: $C_{10}H_{12}ClNO_2$

Molecular weight: 213.7

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Appearance:	Light cream coloured crystalline solid with slight sweet ester odour (Wojcieck, 1993)		
Density:		1.17 g/cm^3 at 24°C	(Wojcieck, 1993)
Vapour pressure:		$2.46 \cdot 10^{-2}$ Pa at 25°C	(Lorence, 1993a)
		$8.02 \cdot 10^{-2}$ Pa at 35°C	(Lorence, 1993a)
		$2.65 \cdot 10^{-1}$ Pa at 45°C	(Lorence, 1993a)
Melting point:		38-41°C	(Wojcieck, 1993)
Octanol/water partition coefficient:		log P_{ow} = 3.4	(Lorence, 1993b)
Solubility:	water	0.017 g/100 g at 25°C	(Lorence, 1993c)
	n-octanol	>95 g/100 g at 25°C	(Lorence, 1993c)
	acetonitrile	>95 g/100 g at 25°C	(Lorence, 1993c)
	acetone	>95 g/100 g at 25°C	(Lorence, 1993c)
Hydrolysis:		no data submitted	
Photolysis:		no data submitted	
Dissociation constant:		pKa 13.3 at 20 ± 1°C in 19% ethanol (Hambrick, 1993)	
Thermal stability:		25-150°C range without decomposition (Malone, 1993)	

Technical material

Minimum purity:	>98%
Colour:	off-white to light brown
Physical State:	solid

Melting point: 38-40°C
Stability: stable indefinitely (Lorence, 1993d; Dewitt and Lorence, 1994)

FORMULATIONS

Commercially available formulations: DP, HN, TC, EC, SL

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

Metabolism in rats (Robinson and Liu, 1991), lactating goats (Wu, 1991a) and laying hens (Wu, 1991b) was evaluated for toxicology by the 2000 JMPR. The same trials were reported to the 2001 Meeting for residue evaluation.

Rats (Robinson and Liu, 1991). Groups of male and female Sprague-Dawley rats were given single oral low and high doses, and single intravenous injections of ^{14}C-ring-labelled chlorpropham at 5, 200, and 0.5 mg/kg bw respectively. An additional group was dosed orally once daily for 14 days with 5 mg/kg of unlabelled chlorpropham, followed by single doses of the radiolabelled compound on day 15. An open test system was used because a negligible amount of $^{14}CO_2$ elimination was observed in the preliminary range-finding study. Urine and faeces were collected over 7-day intervals. 89-97% of the dose was excreted in the urine and 4-7% in the faeces over 7 days, most within 24 hours and minor amounts during days 2 and 3. Excretion did not vary significantly according to dose or sex. No significant levels of the administered dose were released into respired gases after oral dosing.

Analysis of tissues and organs showed that none of the low-dosed groups showed ^{14}C residues exceeding 0.05 mg/kg as chlorpropham. No ^{14}C residues were detected in the tissues or organs of the intravenous-dosed group. Tissues from the high-dose group showed higher residues than the other groups as expected.

Urine and faeces samples collected over the first 24 h were pooled according to sex, excreta type, and dose regimen. Filtered urine and acetonitrile extracts of faeces homogenates were analysed by reverse-phase HPLC. Major radioactive peaks of representative excreta samples were also isolated and compared with radioactive metabolite standards by normal-phase TLC for qualitative confirmation of structures.

A total of 21 metabolites, plus the parent chemical, were detected. Thirteen metabolites accounting for 88-95% of the administered dose were identified (Table 1). Most of the metabolites were found in the urine. No appreciable differences in the metabolite profiles were seen between dose groups or between males and females. In the urine, aryl *O*-sulfate conjugates accounted for approximately 58-70% of the administered dose. The main metabolites were *p*-hydroxy-chlorpropham *O*-sulfate, 3-chloro-4-hydroxyacetanilide *O*-sulfate and *p*-hydroxy-chlorpropham. The structures are shown in Figure 1. Most of the radioactivity in faeces was detected as free metabolites. Unchanged chlorpropham was detected in some faecal samples but not in urine. Three major metabolic pathways were proposed. In addition to aromatic hydroxylation, there is oxidation of the isopropyl side chain and hydrolysis to 3-chloroaniline.

Table 1. Combined distribution of chlorpropham and its metabolites in rat urine and faeces. Mean percentages of orally administered dose (Robinson and Liu, 1991). Structures of compounds are shown in Figure 2.

Compound		%
No.	Name	
	Chlorpropham (parent compound)	0.3

Compound		%
No.	Name	
M1	Chlorpropham alcohol	0.4
M3	Chlorpropham carboxylic acid	4.0
M4	*p*-Hydroxychlorpropham alcohol	1.7
	M4 sulfate	6.2
M2	*p*-Hydroxychlorpropham	14.3
	M2 sulfate	39.0
	M2 glucuronide	1.7
M5	3-Chloroaniline	0.6
	3-Chloro-4-hydroxyaniline sulfate (M6 sulfate)	2.4
	3-Chloro-4-hydroxyaniline *N*-glucuronide *O*-sulfate	1.1
M9	3-Chloro-4-hydroxyacetanilide	1.0
	M9 sulfate	15.5
	M9 glucuronide	0.7
	Unknown (8 metabolites)	1.0
	Total sulfate conjugates	64.1
	TOTAL (parent compound and all metabolites)	90.0

Goats (Wu, 1991a). Two lactating goats were dosed with capsules containing ^{14}C-ring-labelled chlorpropham plus lactose at a rate of 75 mg/day (equivalent to dietary exposure levels of 31.5-36 ppm in the feed or 1.6–1.9 mg/kg bw) for seven days. A control goat received capsules containing only lactose. Urine and faeces were collected daily and milk twice daily, and blood samples were taken on days 1, 3 and 5 and before slaughter. The goats were killed 24 hours after the last dose and liver, kidneys, heart, loin muscle, leg muscle, omental and peripheral fat were collected for analysis.

Extraction and fractionation procedures were combined with combustion, liquid scintillation counting (LSC), HPLC, TLC including two-dimensional TLC, and radiochromatography to characterize significant metabolites.

Mean excretion of the radioactivity in the urine, faeces and milk for 7 days and up to 24 h after the last dose was approximately 99%, 6% and 1% of the cumulative dose respectively. Transfer to liver was about 1%; to fat and muscle lower by 1 or 2 orders of magnitude.

Radioactivity in the milk, expressed as mg chlorpropham equivalents/kg, was constant throughout the study. Residues ranged from 0.32 to 0.45 mg/kg in the milk, 0.18-0.32 mg/kg in the liver, and 0.05 mg/kg in the kidneys, and was below the limit of detection in the hearts, muscles, and fat (<0.03 mg/kg). Residues in the blood were <0.03 mg/kg on day 1, <0.03-0.046 mg/kg on day 3, 0.06 mg/kg on day 5 and 0.09 mg/kg on day 7. The main metabolites identified in the milk, liver and kidneys are shown in Table 2 (average values expressed as a percentage of the TRR and as mg chlorpropham/kg). An unknown metabolite which had been detected in rat urine was found in the goat kidneys (3.7% of the TRR). In the fat chlorpropham was 88.5% of the TRR (0.03 mg/kg). No data on metabolite identification in excreta or blood were reported. The metabolic pathways are shown in Figure 1.

Table 2. Distribution of chlorpropham and its metabolites in milk and tissues of goats (Wu, 1991a). Structures are shown in Figure 1.

Substance		Milk		Liver		Kidney	
		% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
1	3-Chloro-4-hydroxyaniline sulfate (M6 sulfate)		<0.001		<0.001		<0.001

	Substance	Milk		Liver		Kidney	
		% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
2	3-Chloro-4-hydroxyacetanilide *O*-glucuronide (M9 glucuronide)						<0.001
3	3-Chloro-4-hydroxyacetanilide (M9)					3.8	0.003
4	*p*-Hydroxychlorpropham alcohol (M4)			1.0	0.003	0.55	<0.001
5	*p*-Hydroxy-chlorpropham *O*-glucuronide (M2 glucuronide)	3.7	0.014			3.5	0.002
6	3-Chloro-4-hydroxyacetanilide *O*-sulfate (M9 sulfate)	4.5	0.02	0.4	0.001	4.1	0.003
7	M4 aryl sulfate	5.0	0.02			1.1	0.001
8	Chlorpropham alcohol (M1)						
9	*p*-Hydroxy-chlorpropham (M2)	0.89	0.005	3.2	0.008		
10	3-Chloro-4-hydroxyaniline *N*-glucuronide *O*-sulfate						
11	Chlorpropham carboxylic acid (M3)			0.5	0.001		<0.001
12	Chlorpropham (parent)					1.1	0.001
13	*p*-Hydroxy-chlorpropham *O*-sulfate (M2 sulfate)	81	0.3			16.5	0.01
14	3-Chloraniline (M5)						<0.001
15	3-Chloro-4-hydroxyaniline (M6)						<0.001
16	3-Chloroacetanilide (M7)			1.95	0.004	1.3	0.001
17	3-Chloro-6-hydroxyacetanilide (M10)						<0.001
18	3-Chloro-6-hydroxyaniline (M8)					0.65	<0.001
19	3-Chloroaniline-*N*-sulfamate, potassium salt	0.89	0.005	3.2	0.008		<0.001
20	*p*-Methoxy-chlorpropham					1.1	0.001
	B-6, structure unknown (found in rat urine)					3.7	0.003
	Unknowns	3.3	0.013	1.5	0.005	14	0.009
	Nonpolar lipids	0.46	0.002	2.2	0.007	1.7	0.001
	Aqueous			4.2	0.01		
	Protease-hydrolyzable			59	0.16	23	0.014
	Acid-hydrolyzable			22	0.06	16	0.01
	Bound residues	0.75	0.003	4.4	0.01	8.8	0.005
	Total	100.5	0.38	103.5	0.28	100.9	0.064

Poultry (Wu, 1991b). Ten laying hens were dosed once daily with gelatine capsules containing 6 mg of ^{14}C-chlorpropham for seven days (3.3-4.2 mg/kg bw or 50 ppm in the diet). Five control hens received lactose only by capsule. During treatment excreta were collected once daily and eggs twice daily. Eight hours after the last dose the hens were killed and blood, breast and thigh muscle, fat, heart, gizzard, kidney, liver and skin were collected for analysis. During the dosing period, eggs and excreta were also collected for analysis. The methods used were as described above for goats.

During the 7 days and for 8 hours after the last dose 83% of the cumulative dose was recovered from the excreta and 0.03% from the total of eggs laid (0.01% in the whites and 0.02% in the yolks). Radioactivity in the yolks, expressed as mg chlorpropham equivalents/kg, was undetectable during the first 3 days then increased from 0.1 mg/kg on day 4 to 0.23 mg/kg on day 7, when a steady state had not been reached. In the whites residues ranged from 0.007 to 0.074 mg/kg reaching a plateau on day 6. ^{14}C residues in the liver and kidneys were 0.47 and 0.46 mg/kg, in the

skin and fat 0.15 and 0.19 mg/kg, in the gizzard, heart and blood 0.09, 0.04 and 0.09 mg/kg, and in thigh and breast muscle 0.015 and 0.006 mg/kg respectively. The main compounds in the eggs, liver and kidney, expressed as a percentage of the TRR and in mg/kg as chlorpropham are shown in Table 3. Parent chlorpropham was the main compound identified in the fat (92% of the TRR) and skin (68% of the TRR). *p*-Hydroxy-chlorpropham *O*-sulfate constituted 19% of the TRR in the skin. The metabolic pathways are shown in Figure 1.

Table 3. Distribution of chlorpropham and its metabolites in the eggs and tissues of hens (Wu, 1991b).

	Substance	White		Yolk		Liver		Kidney	
		% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
1	3-Chloro-4-hydroxyaniline sulfate (M6 sulfate)	22	0.016					3.8	0.015
2	3-Chloro-4-hydroxyacetanilide *O*-glucuronide (M9 glucuronide)							8.1	0.037
3	3-Chloro-4-hydroxyacetanilide (M9)					0.35	0.002	0.4	0.002
4	*p*-Hydroxychlorpropham alcohol (M4)							5.0	0.023
5	*p*-Hydroxy-chlorpropham *O*-glucuronide (M2 glucuronide)							9.3	0.042
6	3-Chloro-4-hydroxyacetanilide *O*-sulfate (M9 sulfate)							3.7	0.017
7	M4 sulfate	1.1	0.001						
8	Chlorpropham alcohol (M1)	2.3	0.002	3.4	0.007				
9	*p*-Hydroxy-chlorpropham (M2)					3.7	0.017		
10	3-Chloro-4-hydroxyaniline *N*-glucuronide *O*-sulfate	3.9	0.003						
11	Chlorpropham carboxylic acid (M3)	3.3	0.002					3.0	0.014
12	Chlorpropham (parent)	3.1	0.002	20	0.04	0.5	0.002	7.4	0.033
13	*p*-Hydroxy-chlorpropham *O*-sulfate (M2 sulfate)	7.7	0.006	32	0.06	4.3	0.02		
14	3-Chloraniline (M5)								
15	3-Chloro-4-hydroxyaniline (M6)							3.4	0.015
16	3-Chloroacetanilide (M7)	1.4	0.001	1.5	0.003				
	B-6, Unknown found in rat urine							5.5	0.025
	Organosoluble and water-soluble unknowns	39[1]	0.03'	10	0.02	12	0.055	6	0.027
	Lipophilic radioactivity			13	0.025	2.5	0.012		
	Protease-hydrolyzable			21	0.04				
	Enzyme- or acid-hydrolyzable 3-chloro-4-hydroxyaniline-related metabolites					64	0.3	25	0.11
	Other unknowns	16	0.01			12.5	0.06	20	0.09
	Total	99.8	0.073	100.9	0.195	99.85	0.468	100.6	0.45

[1]Seven unknowns, none exceeding 0.014 mg/kg

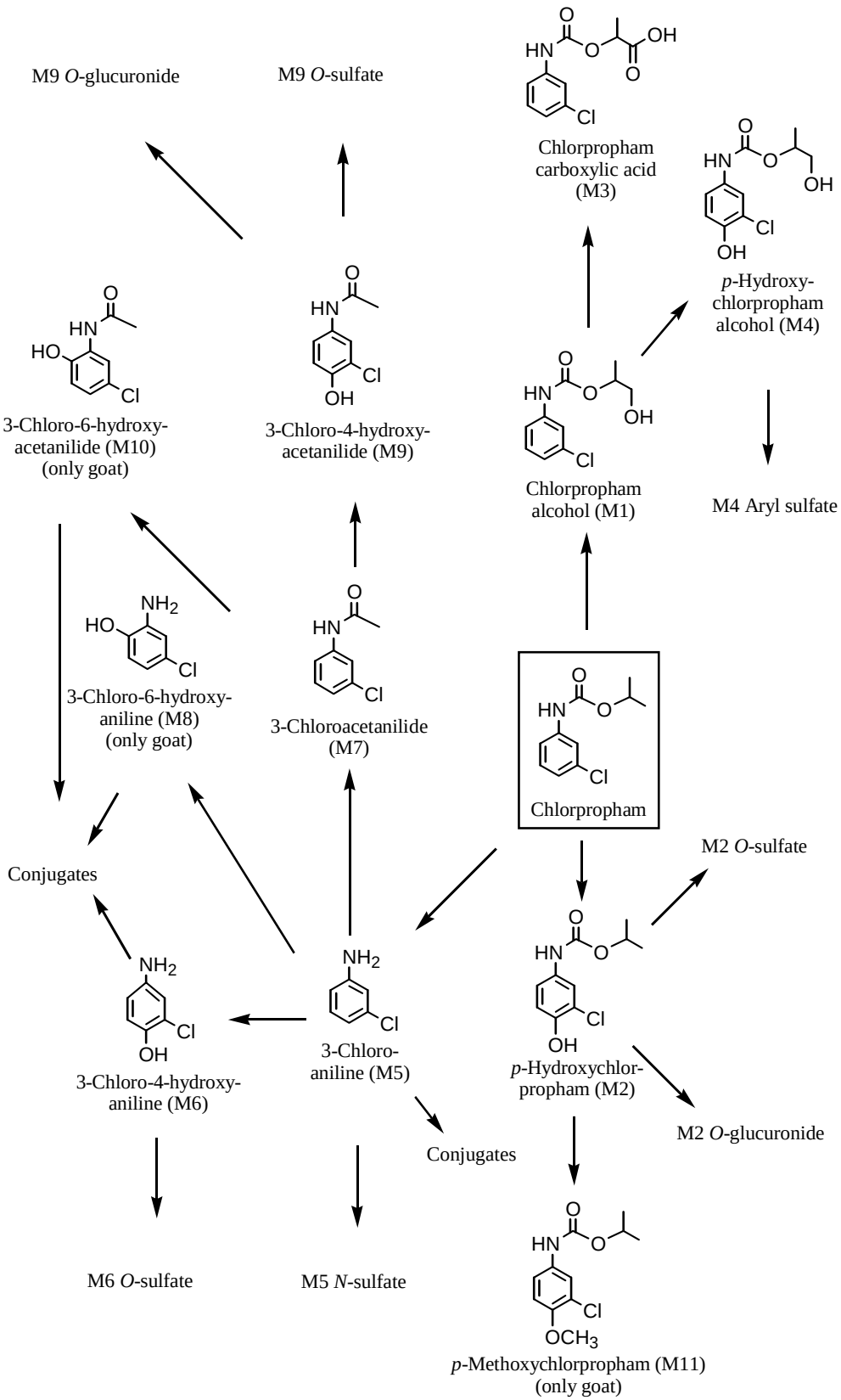


Figure 1. Proposed metabolic pathways in lactating goats and laying hens.

Plant metabolism

Potatoes (Kim-Kang, 1991). 164 potatoes, with an average size of 170 g, were treated with 1.5% ^{14}C-ring-labelled chlorpropham diluted from a 26% w/w emulsion at a nominal dose level of 40 mg/kg and stored in an incubator (8 ± 2°C) with circulating moist air simulating cold storage. Eight potatoes were sampled at 2 hours, 2 days, and 1, 3, 6, 9, 12, 16, 20, 24, 28, 32, 40, 44, 48 and 52 weeks after treatment, and the tubers washed with methanol to remove surface residues. Two potatoes from each sampling were homogenised as whole tubers, and 6 were peeled twice to yield peel, first layer and pulp. Each sample was diced, frozen in liquid nitrogen and homogenized for determination of the TRR.

Peel, pulp, first layer, and whole potatoes were extracted by blending with a modified Bligh-Dyer solvent mixture of methanol/water/chloroform ($MeOH/H_2O/CHCl_3$, 11:5:5). The $CHCl_3$ fraction was further partitioned with a mixture of acetonitrile (ACN) and hexane (1:1). The percentage of the TRR in each extracted fraction was determined for each sample. The final TRR levels after one year's storage (mg/kg as chlorpropham) were 1.2, 1.9, 20, and 4.2 mg/kg in the pulp, first layer, peel and whole potato respectively. A gradual decrease in the proportion of the TRR in the ACN fraction from the pulp and a gradual increase in the $MeOH/H_2O$ fraction was observed. Comprehensive analyses of the peel and pulp samples from the final harvest (52 weeks) showed 49.7% of the TRR in the ACN fraction, 39.5% in the $MeOH/H_2O$ fraction, 1.8% in the hexane fraction, and 9% in the post extraction solids (PES) from the pulp and 71%, 9.4%, 6.4%, and 13% in the ACN, MeO/H_2O, hexane, and PES fractions respectively from the peel.

Unextractable residues in the pulp PES digested with α-amyloglucosidase and yielded 6.7% of a non-starch material related to protein, cellulose, hemicellulose and lignin. The peel PES were treated with cellulase, then α-amyloglucosidase, followed by Bleidner distillation-extraction, giving ^{14}C residues of 2.75% in the cellulose, 1.25% in the starch and 8.8% in the cellulose/lignin subfractions.

In summary, under cold storage translocation of the radioactivity from the peel to the pulp was slow (Table 4). About 86% of the radioactivity remained on the surface of the potatoes after 364 days.

Table 4. Distribution of radioactivity in potatoes (Kim-Kang, 1991).

Days after treatment	^{14}C, % of TRR				Days after treatment	^{14}C, % of TRR	
	Methanol wash	Peel	1st layer	Pulp		Methanol wash	Whole potato
0 (2h)	98	1.9	0.02	0.04	0 (2h)	99	1.1
2	95.5	4.4	0.03	0.05	2	94	5.6
7	97	2.6	0.03	0.05	7	97	3.2
21	97	3.1	0.06	0.11	21	97	2.85
42	96	3.4	0.11	0.27	42	96	3.9
63	95	4.3	0.13	0.35	63	96	4.2
84	94	5.6	0.16	0.56	84	94	6.2
112	92	7.0	0.24	0.68	112	93	7.2
140	92	7.2	0.21	0.57	140	90.5	9.5
168	91.5	6.9	0.42	1.1	168	91.5	8.55
196	92	6.3	0.54	1.6	196	91	8.9
224	89.5	8.3	0.51	1.7	224	88.5	11.5
252	87	9.2	0.87	3.1	252	88	12
280	85	13	0.63	2.0	280	79	21
308	87	10.5	0.70	2.2	308	87.5	12.5
336	85	11	0.75	2.8	336	78	22
364	86	9.8	0.90	2.9	364	85	15

The compounds in the peel and pulp samples were determined at 52 weeks by two-dimensional TLC and radiochromatography. Seven compounds, including the parent, were identified. Most of the residue remained as chlorpropham. The main metabolites identified were an oligosaccharide conjugate of *p*-hydroxy-chlorpropham, a novel amino acid conjugate of *p*-hydroxy-chlorpropham found in the pulp and as a minor metabolite in the peel, and also included 3-chloroaniline, *p*-methoxy-chlorpropham, 3-chloro-*N*-glucosylaniline and an oligosaccharide conjugate of chlorpropham alcohol. Enzyme hydrolysis of PES (cellulase and α-amyloglucosidase) yielded some of the parent compound indicating that part of the chlorpropham was probably physically bound to endogenous carbohydrate material unextractable by conventional solvent-solvent extraction. The MeOH washes were also analysed by HPLC and 2-D TLC; chlorpropham was the only surface residue identified. The results are shown in Table 5. The three potential metabolic pathways listed below are shown in Figure 2.

1) Decarboxylation to 3-chloroaniline, followed by conjugation with glucose and other biomolecules.
2) Hydroxylation and subsequent conjugation with either oligosaccharides or amino acids at the position para to the amino moiety or methylation of *p*-hydroxy-chlorpropham to *p*-methoxy-chlorpropham.
3) Oxidation of the isopropyl side chain and subsequent conjugation with oligosaccharide(s).

Table 5. Distribution of ^{14}C residues in potato peel and pulp (Kim-Kang, 1991).

Substance (52 weeks after treatment)	Peel		Pulp	
	% of TRR	mg/kg as chlorpropham	% of TRR	mg/kg as chlorpropham
identified				
Chlorpropham	85	17	42	0.52
p-methoxy-chlorpropham			1.9	0.023
3-Chloroaniline	3.6	0.72		
3-Chloro-*N*-glucosylaniline	0.54	0.11	6.1	0.075
Oligosaccharide conjugate of *p*-hydroxy-chlorpropham	7.6	1.5	18	0.22
Oligosaccharide conjugate of chlorpropham alcohol	0.27	0.05		
Amino acid conjugate of *p*-hydroxy-chlorpropham	0.58	0.12	18	0.22
unknowns				
Polar unidentified	1.2	0.24	5.0	0.062
Non polar hexane soluble			1.8	0.022
Enzyme-hydrolyzed water-soluble	1.7	0.34	1.3	0.016
Bound residues			6.7	0.083
Total	100	20	100	1.24

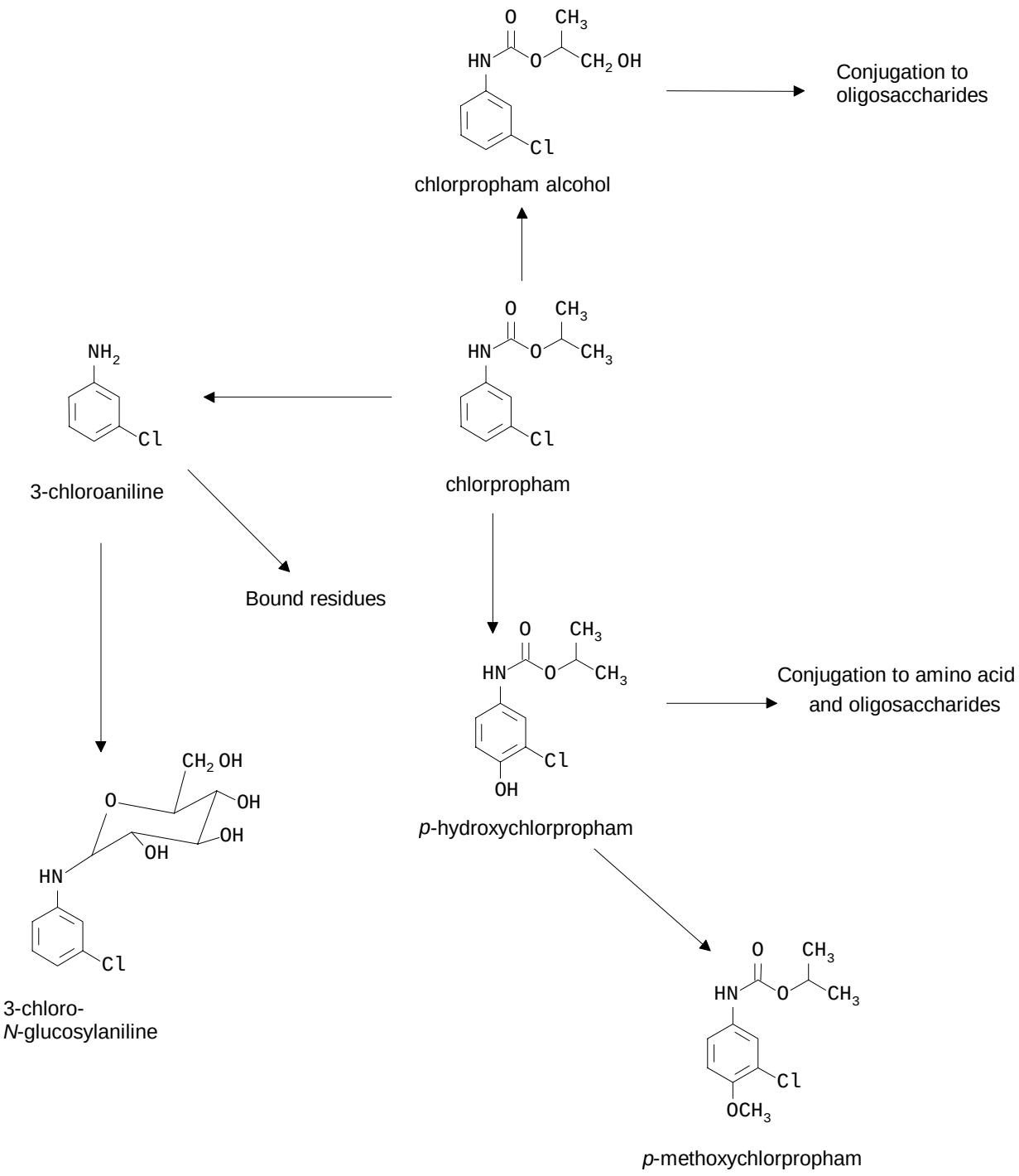


Figure 2. Proposed metabolic pathways in stored potatoes (Kim-Kang, 1991).

Environmental fate in soil

No information.

Environmental fate in water/sediment systems

No information.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Plant material

Potatoes. Analytical methods were reported for the determination of residues of chlorpropham alone or of the parent and three metabolites (3-chloroaniline, *p*-hydroxy-chlorpropham, *p*-methoxy-chlorpropham).

(Roland, 1998a,b). In the supervised trials (Tables 26-28) potatoes were rinsed in running water to remove any adhering soil and divided into representative parts before being completely thawed, to ensure subsampling homogeneity. The potatoes were peeled with a stainless steel paring knife as soon as the surfaces were sufficiently tenderized. The thickness of the peel was 1.3 ± 0.2 mm and fortification rates were 0.1, 1, 2 and 5 mg/kg for entire tubers, 0.02, 0.2, 0.5, 1 mg/kg for peeled and 0.02 and 0.1 mg/kg for cooked potatoes. Two independent analyses were made using separate subsamples of each part of the sample. 50 g of sample was homogenized with 200 ml of petroleum ether/acetone (1/1) and filtered. The apparatus and cake were then rinsed with 50 ml of the extraction mixture, and the extracts partitioned with 500 ml of water and 20 ml of saturated NaCl solution. The petroleum ether extract was filtered through anhydrous sodium sulfate and the aqueous phase re-extracted with 75 ml of methylene chloride. The methylene chloride extract was filtered through anhydrous sodium sulfate. The combined organic extracts were evaporated under vacuum and the dry residue dissolved in 5 ml of iso-octane. After further purification on a Florisil column, chlorpropham was eluted with 60 ml of hexane saturated with CH_3CN, the eluate evaporated under vacuum, and the dry residue dissolved in 2.5 ml of iso-octane. Determination was carried out by GLC with an NPD. The results are shown in Table 6.

Table 6. Recovery of chlorpropham from potatoes (Roland, 1998a,b).

Sample	Fortification level (mg/kg)	Chlorpropham recovered (mg/kg)	Recovery (%)
Entire tubers Untreated	0	<0.02	-
	0	<0.02	-
	0	<0.02	-
	0	<0.02	-
	0.1	0.106	106
	0.1	0.103	103
	0.1	0.104	104
	0.1	0.1	100
	2	1.9	95
	2	1.8	90
Peeled potatoes Untreated	0	<0.02	-
	0	<0.02	-
	0	<0.02	-
	0	<0.02	-
	0.02	0.02	100
	0.02	0.018	90
	0.02	0.021	105
	0.02	0.019	95
	0.5	0.5	100
	0.5	0.45	90
	0.5	0.49	98
	0.6	0.47	94

Sample	Fortification level (mg/kg)	Chlorpropham recovered (mg/kg)	Recovery (%)
Cooked potatoes Untreated	0	<0.02	-
	0	<0.02	-
	0.02	0.02	100
	0.02	0.018	90
	0.1	0.092	92
	0.1	0.089	89

(Brielbeck and Marx, 1996a,b). Recoveries of chlorpropham residues from unpeeled (1996a) and peeled potatoes (1996b) determined by GLC with an ECD after bromination were 88-94% at fortification levels of 0.3-0.4 mg/kg.

(Brielbeck and Marx, 1999c). Recoveries of chlorpropham residues from peeled and cooked and peeled and unpeeled raw potatoes determined by GLC with an NPD were 94-101% at fortification levels of 0.02-5 mg/kg.

(Moeller, 1991). In a method for the determination of chlorpropham and the three metabolites 3-chloroaniline, *p*-hydroxy-chlorpropham and *p*-methoxy-chlorpropham potatoes are chopped in a food processor, and 50 g subsamples in a tall square bottle are homogenized in 100 ml of 50:50 methanol/0.5 N HCl using a polytron insertion homogenizer. The bottle is then heated to near boiling for 3 min at 50% power in a 1500 W microwave oven, cooled and 200 ml of 50:50 hexane/ethyl acetate added and shaken, followed by 20 ml of pH 7 phosphate buffer plus 25 ml of a 1 N NaOH solution, and the bottle is capped and shaken. The emulsion separates rapidly especially when chilled or set on a slow orbital shaker. After separation 100 ml of the upper hexane-ethyl acetate layer is filtered through sodium sulfate, 100 ml of extract is collected and evaporated in a turbo-vap evaporator under nitrogen and twice exchanged with n-hexane without going to dryness. The final exchange is automatically reduced to 0.5 ml of n-hexane which is then brought to 5 ml with mixing to obtain a final concentration of 5 g of sample per ml of n-hexane. The extract is analysed by gas chromatography with a nitrogen-phosphorus detector on a 15 m DB-5 megabore column equipped with a 1 m uncoated guard.

A recovery study was carried out on whole potatoes spiked with the analytes at 0.4 mg/kg and 1.2 mg/kg (n = 6 or 7). The recoveries at 1.2 mg/kg level were 69% for chlorpropham, 41% for 3-chloroaniline, 83% for *p*-hydroxy-chlorpropham and 78% for *p*-methoxy-chlorpropham, and at 0.4 mg/kg were 68% for chlorpropham, 38% for 3-chloroaniline, 87% for *p*-hydroxy-chlorpropham and 78% for *p*-methoxy-chlorpropham. The low recovery of 3-chloroaniline is similar to the results of the metabolism study (Kim-Kang, 1991) and appears to confirm the reactive incorporation of this compound into the insoluble post-extraction solids. The stability of the compounds under acidic hydrolysis conditions is verified.

(Walker *et al.*, 1993). In a multi-residue method for the determination of chlorpropham, 3-chloroaniline, *p*-hydroxy-chlorpropham and *p*-methoxy-chlorpropham in potatoes, their processed products, and canola cooking oil the primary methanol-water extract is partitioned with methylene chloride. The post-extraction solids are filtered, mixed with a pH 6.5 NaCl-saturated phosphate buffer, sonicated, and partitioned with methylene chloride which is combined with the first extract. For oil-processed samples a GPC clean-up is necessary. The methylene chloride is evaporated in a stream of nitrogen and exchanged with n-hexane for analysis by capillary gas chromatography with a nitrogen-phosphorus detector. This method has been validated for the determination of chlorpropham and its metabolites in whole potatoes, potato peel and pulp, French fries and chips with and without skins, processed dried and wet peels, dehydrated granules and canola oil. The detection limits and practical limits of quantification for chlorpropham, *p*-hydroxy-chlorpropham, *p*-methoxy-chlorpropham and 3-chloroaniline were 0.08 and 0.45 mg/kg in whole potatoes, fresh pulp and peel, and processed wet peel, 0.2 and 1.1 mg-kg in French fries, 0.45 and 2.2 mg/kg in chips, 0.38 and 1.9 mg/kg in dehydrated granules and processed dried peel, and 2.9 and 14 mg/kg in canola oil.

Experimental recoveries (n = 6) at spiking levels of 5.3 and 13 mg/kg (8 and 20 mg/kg for canola oil) were above 60% with the exception of 3-chloroaniline in fresh potato peels which was not recovered. It is suggested that irreversible binding of 3-chloroaniline with the substrate could prevent extraction. Sonication gave recoveries of all analytes equal to or better than were achieved after 12 hours of refluxing at alkaline pH. The results are shown in Table 7.

Table 7. Recoveries of chlorpropham and its metabolites from fortified potato samples (Walker *et al*., 1993).

Analyte	Sample	Fortification level (mg/kg)	No. of samples[1]	Recovery, %
Chlorpropham	Whole potato	5.3	6 (1)	81-121
		13	6	70-95
	Potato pulp	5.3	6	72-94
		13	6 (1)	68-101
	Potato peel	5.3	6 (2)	36-126
		13	6 (1)	73-128
	Canola oil	8.0	6 (1)	68-82
		20	6	91-116
	French fries	5.3	6	77 -90
		13	6	77-11
	Potato chips	5.3	6	74-98
		13	6 (1)	80-151
	Dried peel	5.3	6 (2)	66-124
		13	6 (1)	80-140
	Wet peel	5.3	6	70-113
		13	6 (2)	65-92
	Dehydrated granule	5.3	6	81-104
		13	6	87-95
	MeOH/water	5.3	6 (1)	75-121
		13	6 (1)	86-121
p-Hydroxy-chlorpropham	Whole potato	5.3	6	74-104
		13	6	80-120
	Potato pulp	5.3	6 (3)	57-79
		13	6 (3)	62-94
	Potato peel	5.3	6 (3)	51-102
		13	6 (3)	57-97
	Canola oil	8.0	6 (1)	69-91
		20	6 (2)	94-124
	French fries	5.3	6	106-117
		13	6	74-112
	Potato chips	5.3	6	87-106
		13	6	83-104
	Dried peel	5.3	6 (1)	62-117
		13	6 (2)	65-134
	Wet peel	5.3	6	70-98
		13	6	80-97
	Dehydrated granule	5.3	6	77-102
		13	6	85-98
	MeOH/water	5.3	6	72-111
		13	6	80-113

Analyte	Sample	Fortification level (mg/kg)	No. of samples[1]	Recovery, %
p-methoxy-chlorpropham	Whole potato	5.3	6 (1)	81-123
		13	6	73-101
	Potato pulp	5.3	6	75-98
		13	6	72-98
	Potato peel	5.3	6 (3)	74-149
		13	6	72-119
	Canola oil	8.0	6 (1)	66-89
		20	6 (1)	94-126
	French fries	5.3	6	83-98
		13	6	77-114
	Potato chips	5.3	6	81-96
		13	6	88-113
	Dried peel	5.3	6	70-113
		13	6 (1)	80-132
	Wet peel	5.3	6	83-102
		13	6	87-102
	Dehydrated granule	5.3	6	90-115
		13	6	88-100
	MeOH/water	5.3	6 (1)	81-123
		13	6 (1)	88-131
3-Chloroaniline	Whole potato	5.3	6 (1)	68-77
		13	6 (5)	65-71
	Potato pulp	5.3	6 (5)	64-72
		13	6 (6)	51-68
	Potato peel	5.3	6 (6)	not detected
		13	6 (6)	not detected
	Canola oil	8.0	6 (6)	26-60
		20	6 (5)	54-72
	French fries	5.3	6	89-94
		13	6 (6)	59-68
	Potato chips	5.3	6	87-106
		13	6 (3)	62-77
	Dried peel	5.3	6 (1)	49-90
		13	6 (3)	55-108
	Wet peel	5.3	6 (1)	68-89
		13	6 (1)	60-85
	Dehydrated granule	5.3	6 (2)	34-89
		13	6	72-87
	MeOH/Water	5.3	6	75-117
		13	6 (1)	65-101

[1]Numbers in parentheses: nos. of samples with recoveries outside 70-120% range.

Goodrick *et al.* (1994) modified the method by using hexane-based calibration standards. (In the original method substrate-based standards were used with the aim of limiting the effects of interference.)

As before a methanol-water extract is partitioned with methylene chloride but the primary extractant is a 50:50 mixture of methanol and 0.5N HCl. The analysis is completed as before. The method has been validated for the determination of chlorpropham and 3-chloroaniline in whole potatoes, chips with peel, processed dried and wet peel, and dehydrated granules. The results are shown in Table 8.

Table 8. Recoveries of chlorpropham and 3-chloroaniline from fortified potato samples (Goodrick *et al.*, 1994).

Analyte	Sample	Fortification level (mg/kg)	No. of samples [1]	Recovery, %
Chlorpropham	Whole	0.5	6	82-106
		2	6	83-113
		8	6 (1)	43-105
	Chips with peel	4	6 (2)	94-141
		10	6 (4)	113-149
		20	6	70-105
	Processed wet peel	0.8	6	71-88
		2	6	85-104
		8	6 (3)	68-91
	Processed dried peel	2	6	73-93
		10	6 (1)	67 -92
		40	6 (1)	64-80
	Dehydrated granules	2	6	72-101
		10	6 (1)	ND-97
		40	6 (4)	41-73
3-Chloroaniline	Whole	0.5	6 (6)	20-26
		2	6 (6)	15-22
		8	6 (6)	17-37
	Chips with peel	4	6 (6)	34-67
		10	6 (6)	36-48
		20	6 (6)	8-28
	Processed wet peel	0.8	6 (6)	49-62
		2	6 (6)	36-50
		8	6 (6)	38-54
	Processed dried peel	2	6 (6)	46-62
		10	6 (6)	46-62
		40	6 (6)	24-55
	Dehydrated granules	2	6 (6)	46-57
		10	6 (6)	ND-62
		40	6 (5)	37-72

[1]Numbers in parentheses: nos. of samples with recoveries outside 70-120% range

Bogess (1993) validated a method for the determination of chlorpropham, 3-chloroaniline, *p*-hydroxy-chlorpropham, and *p*-methoxy-chlorpropham in whole potatoes, potato pulp, fresh and processed wet and dry peel, dehydrated granules, and chips and French fries with and without peel using gas chromatography with a specific nitrogen-phosphorus detector. The results are shown in Table 9.

Table 9. Recoveries of chlorpropham and metabolites from fortified potato samples (Bogess, 1993).

Analyte	Sample	Fortification level (mg/kg)	No. of samples[1]	Recovery, %
Chlorpropham	Whole	0.4	2 (2)	138-183
		1	2 (2)	169-177
	Pulp	0.4	2 (2)	122-155
		1	1	99
	Fresh peel	0.4	2 (1)	65-89
		1	2	76-78

Analyte	Sample	Fortification level (mg/kg)	No. of samples[1]	Recovery, %
	Processed wet peel	0.4	2 (1)	98-141
		1	2	80-81
	Processed dry peel	2	2	72-96
		5	2 (1)	108-126
	Dehydrated granules	2	2	83 -94
		5	2	89-94
	Potato chips	2	2	105-108
	French fries	0.4	2	76-78
		1	2	79-81
4'-Hydroxy-chlorpropham	Whole potato	0.4	2 (2)	59-126
		1	2 (2)	147-150
	Potato pulp	0.4	2 (2)	29-32
		1	1 (1)	40
	Fresh peel	0.4	2 (1)	60-78
		1	2 (1)	66-72
	Processed wet peel	0.4	2 (1)	56-113
		1	2	74-93
	Processed dry peel	2	2 (1)	56-113
		5	2 (2)	122-140
	Dehydrated granules	2	2	91-98
		5	2	96-98
	Potato chips	2	2	72-97
	French fries	0.4	2	86-89
		1	2	83-83
p-methoxy-chlorpropham	Whole potato	0.4	2 (1)	94-150
		1	2 (2)	153-158
	Potato pulp	0.4	2 (1)	109-134
		1	1	102
	Fresh peel	0.4	2 (1)	69-91
		1	2	79-82
	Processed wet peel	0.4	2 (1)	94-137
		1	2	89-92
	Processed dry peel	2	2 (1)	66-109
		5	2 (2)	132- 150
	Dehydrated granules	2	2	93-106
		5	2	95-99
	Potato chips	2	2	71-75
	French fries	0.4	2	83-87
		1	2	83-84
3-Chloroaniline	Whole potato	0.4	2 (2)	39-51
		1	2 (2)	38-55
	Potato pulp	0.4	2 (2)	39-40
		1	1 (1)	41
	Fresh peel	0.4	2 (2)	44-57
		1	2 (2)	48-68
	Processed wet peel	0.4	2 (2)	40-56
		1	2 (2)	41-43
	Processed dry peel	2	2 (2)	33-53
		5	2 (1)	57-77
	Dehydrated granules	2	2 (2)	51-63
		5	2 (2)	62-63

Analyte	Sample	Fortification level (mg/kg)	No. of samples[1]	Recovery, %
	Potato chips	2	2 (2)	36-38
	French fries	0.4	2 (2)	23-39
		1	2 (2)	27-29

[1]Numbers in parentheses: nos. of samples with recoveries outside 70-120% range.

Bogess (1994) conducted a supplementary study on whole potatoes spiked with chlorpropham at higher levels. The results are shown in Table 10.

Table 10. Recoveries of chlorpropham from fortified samples of whole potatoes (Bogess, 1994).

Analyte	Fortification level (mg/kg)	No. of samples	Recovery, %
Chlorpropham	2	2	58, 73
	5	2	81, 85

Animal material

Note: *p*-Hydroxy-chlorpropham *O*-sulfate has been widely referred to as 4'-hydroxy-chlorpropham-*O*-sulfonic acid, with the abbreviation HSA. In the following sections the compound will be named *p*-hydroxy-chlorpropham sulfate, but the abbreviation HSA will be used.

Daun (1995a,b, 1996a,b) developed and validated a method for the determination of residues of chlorpropham and *p*-hydroxy-chlorpropham sulfate (HSA) in meat and milk. Homogenized samples of whole milk, skimmed milk, cream, muscle, liver, kidney, and fat are ground in a glass mortar with 40 μm C-18 solid-phase material (Bakerbond Octadecyl (C_{18}) Prep LC Packing). The mixture is packed into a polymeric column and eluted sequentially with 1:1 hexane/dichloromethane (DCM), pure DCM, and methanol/water (5:1). The hexane/DCM eluate is evaporated to near dryness, reconstituted in hexane, partitioned with acetonitrile (ACN), concentrated under vacuum and, after water is added, partitioned back into hexane and diluted to known volume for determination of chlorpropham by gas chromatography with mass-selective detection (GLC-MSD). The DCM fraction is discarded.

The methanol/water eluate is passed through a solid-phase extraction (SPE) cartridge containing a quaternary ammonium bonded phase. The cartridge is washed with methanol and water and the *p*-hydroxy-chlorpropham-*O*-sulfonic acid eluted with dilute alkali. The fraction containing the *p*-hydroxy-chlorpropham-*O*-sulfonic acid is analysed by HPLC on a reverse-phase column with detection and measurement at 238 nm.

Table 11. Recoveries of chlorpropham and HSA from fortified control samples of milk and tissues of cattle (Daun, 1995a,b, 1996b).

Analyte	Sample	Fortification level (mg/kg)	No. of samples [1]	Recovery, %
Chlorpropham	Whole milk	0.01	3 (1)	113-130
		0.1	3 (1)	109-129
	Skimmed milk	0.01	3	72-86
		0.1	3	78-106
	Cream	0.01	3 (1)	12-108
		0.1	3	103-127
	Muscle	0.01	3	89-113
		0.1	3 (1)	85-122

Analyte	Sample	Fortification level (mg/kg)	No. of samples [1]	Recovery, %
	Liver	0.01	3 (3)	123-143
		0.1	3	106-114
	Kidney	0.01	3 (1)	100-165
		0.1	3	87-94
	Fat	0.01	3 (1)	25-118
		0.1	3	99-102
HSA	Whole milk	0.05	3	85-95
		0.5	3	83-87
	Skimmed milk	0.05	3	75-88
		0.5	3	81-83
	Cream	0.05	3 (1)	67-81
		0.5	3	82-97
	Muscle	0.05	3	87-95
		0.5	3 (1)	69-72
	Liver	0.05	3 (2)	66-80
		0.5	3	71-104
	Kidney	0.05	3	75-95
		0.5	3	70-105
	Fat	0.05	3	92-101
		0.5	2	79, 95

[1]Numbers in parentheses: nos. of samples with recoveries outside 70-120% range.

Daun and Zeller (1995) determined chlorpropham in the milk and tissues of dairy cows after solid-phase extraction followed by elution with organic solvents and further isolation through partition between immiscible solvents. Chlorpropham is determined in the purified extract by gas chromatography with mass selective detection (GC-MSD).

p-Hydroxy-chlorpropham-*O*-sulfonic acid (HSA) is determined in whole and skimmed milk by dilution with acetonitrile, selective precipitation of interfering substances and analysis of the resulting solution by reverse-phase HPLC with UV detection. HSA is isolated from tissues and cream using a single-phase extraction system. The aqueous phase is reduced in volume and further purified on a C-18 SPE cartridge. HSA is determined in the eluate by reverse-phase HPLC as before.

The limits of quantification (LOQs) of chlorpropham and HSA were 0.01 and 0.05 mg/kg respectively. The recoveries are shown in Tables 12-14.

Table 12. Recoveries of chlorpropham and HSA from whole milk (Daun and Zeller, 1995).

Day of treatment	Fortification level (mg/kg)	Recovery, %	
		Chlorpropham	HSA
0	0.01	100, 118	
	0.05	113	71, 77, 83
	0.1	87, 77, 119	
	0.5	85, 83, 77	
1	0.01	163, 191	
	0.05	116	87, 93
	0.1	101, 88, 57	
	0.5	98, 87, 75	
4	0.05	101, 91, 97	73, 70
	0.1	104, 82	101
	0.5	83	85, 82

Day of treatment	Fortification level (mg/kg)	Recovery, %	
		Chlorpropham	HSA
	10		72
	0.01	97	
	0.05	72, 65	83
	0.1	49, 78, 75	70, 87
	0.5		77
7	10		88, 73
	0.05	88, 92, 76	87, 74, 57
	0.1	95, 100, 96	
10	0.5		86, 76, 82
	0.05	118, 105	65, 90, 54
	0.1	132, 102, 98	
14	0.5		72, 77, 71
24	0.05	62	
	0.05	84	
	0.1	154	
28	0.5		72

Table 13. Recoveries from skimmed milk and cream samples fortified with chlorpropham or HSA (Daun and Zeller, 1995).

Sample	Fortification level (mg/kg)	Recovery, %	
		Chlorpropham	HSA
Skimmed milk	0.05	117, 116	68, 78
	0.1	63, 112	
	0.5		79, 85
Cream	0.05	106, 103	102, 86
	0.1	89, 82	
	0.5		89, 108

Table 14. Recoveries from tissue samples fortified with chlorpropham or HSA (Daun and Zeller, 1995).

Sample	Fortification level (mg/kg)	Recovery, %	
		Chlorpropham	HSA
Liver	0.01	261, 228	
	0.05		88, 108
	0.1	105, 88	
	0.5		78, 81
Muscle	0.01	134, 173, 209	
	0.05		86, 74
	0.1	91, 100, 80	
	0.5		77, 71
Kidney	0.01	128, 389, 273	
	0.05		96, 142
	0.1	99, 72, 71	
	0.5		83
	5		102
Fat	0.01	224	
	0.05	83, 129	97, 86

Sample	Fortification level (mg/kg)	Recovery, %	
		Chlorpropham	HSA
	0.1	109	
	0.5		100, 96
	5	110	
	10	73	

Stability of pesticide residues in stored analytical samples

Plants

Goodrick *et al.* (1993a) investigated the storage stability of chlorpropham and metabolites in fortified untreated tuber samples which were also processed into pulp, peel, French fries, chips, processed wet and dried peel and dehydrated granules. Subsamples of each were spiked with 2 or 20 mg/kg of chlorpropham, 3-chloroaniline, *p*-hydroxy-chlorpropham or *p*-methoxy-chlorpropham and stored frozen at –20 to -21°C before analysis at 0 and 14 days, 1 and 2 months, and monthly thereafter up to 390 days for whole potatoes and fresh peel, 360 days for pulp and processed dried peel, 272 days for French fries, 240 days for chips and dehydrated granules, and 180 days for processed wet peel. The results are shown in Tables 15 and 16.

The stabilities of all the analytes were broadly similar at the two fortification levels but were low for 3-chloroaniline and *p*-hydroxy-chlorpropham. Of the 64 sample-analyte combinations 40 decreased by 3% or less per month and 15 showed monthly negative rates from 3.4-6.8%. 3-Chloroaniline and *p*-hydroxy-chlorpropham were unstable in whole potatoes, pulp and peel after 90 days, and 3-chloroaniline in processed wet peel. The instability in the fresh samples may be because of reaction with the substrate.

Table 15. Highest and lowest percentages of chlorpropham and its metabolites remaining in processed potato products after frozen storage (Goodrick *et al*., 1993a).

Compound	Highest remainder		Lowest remainder	
	mean ± sd	Product/fort. level/days	mean ± sd	Product/fort. level/days
Chlorpropham	98 ± 26	Dried peel/20 mg/kg/360	60 ± 28	French fries/2 mg/kg/272
3-Chloroaniline	64 ± 21	Dried peel/20 mg/kg/360	24 ± 12	Fresh peel/2 mg/kg/390
p-Hydroxy-chlorpropham	91 ± 30	Dried peel/2 mg/kg/360	28 ± 19	Pulp/20 mg/kg/360
p-Methoxy-chlorpropham	105 ± 32	Dried peel/20 mg/kg/360	63 ± 24	French fries/2 mg/kg/272

Table 16. Storage stability of analytes in potatoes and their processed products fortified with chlorpropham or its metabolites (Goodrick *et al*., 1993a).

Sample	% Change per month[1]							
	Chlorpropham		3-chloroaniline		*p*-hydroxychlor-propham		*p*-methoxychlor-propham	
	2 mg/kg	20 mg/kg	2 mg/kg	20 mg/kg	2 mg/kg	20 mg/kg	2 mg/kg	20 mg/kg
Whole potato	-2.7	-1.2	-2.3	-8.7	-5.2	-9.1	-2.1	0.6
Pulp	-2.1	-0.1	-6.2	-9.1	-6.1	-10	-0.8	1.7
Fresh peel	-2.0	-2.4	1.5	-7.0	0.6	-8.8	-1.9	-2.1

Sample	% Change per month[1]							
	Chlorpropham		3-chloroaniline		*p*-hydroxychlor-propham		*p*-methoxychlor-propham	
	2 mg/kg	20 mg/kg	2 mg/kg	20 mg/kg	2 mg/kg	20 mg/kg	2 mg/kg	20 mg/kg
Dehydrated granules	-5.9	-4.2	-6.8	-5.2	-4.9	-4.9	-4.5	-2.5
French fries	-4.1	-4.7	-2.0	-1.4	-3.0	-3.0	-2.9	-3.4
Chips	-2.7	7.8	-4.4	3.2	-0.5	4.1	-1.1	2.5
Processed wet peel	0.8	-2.0	-13	-12	6.8	6.0	1.5	0.5
Processed dried peel	2.5	1.2	-5.3	-6.3	-0.5	3.5	1.0	0.7

[1] Calculated using secular trend analysis, based on expected 365 days value using a linear regression model. Positive rates of change are artefacts of the measurement system according to the author.

Haws *et al.* (1993a,b) investigated the storage stability of chlorpropham in field-treated potato tubers and their products. The potatoes were treated under practical conditions (aerosol fogging) and processed to produce fresh peel, chips and French fries with skin, dehydrated granules, and dried and wet peel, then homogenized and stored at -20 to -21°C. Residues were determined at intervals up to 272-391 days. As different field samples were analysed at successive storage times the results (Table 17) are of limited use. The initial concentrations of 3-chloroaniline, *p*-hydroxy-chlorpropham and *p*-methoxy-chlorpropham were below practical limits of quantification and detection and therefore not suitable for a stability study.

Table 17. Stability of chlorpropham in field-treated potatoes and their processed products stored at -20 to -21°C (Haws *et al.*, 1993a,b).

Sample	Chlorpropham (mg/kg), mean of two samples												
	Days in freezer at -20 to -21°C												
Whole potatoes	**9**	**20**	**30**	**62**	**91**	**121**	**184**	**218**	**283**	**305**	**333**	**361**	**391**
	11	9.9	8.7	10	10	8.6	6.5	5.1	12	6.1	8.0	6.7	11
Fresh peel	**Days in freezer at -20 to -21°C**												
	9	**20**	**30**	**62**	**91**	**121**	**184**	**218**	**283**	**300**	**333**	**361**	**391**
	62	51	46	58	61	17	61	39	59	43	58	46	56
Potato chips with skin	**Days in freezer at -20 to -21°C**												
	20	**62**	**91**			**121**	**156**	**182**	**215**	**231**	**282**		
	4.5	5.2	3.7			5.8	9.9	12	13	3.4	3.1		
French fries with skin	**Days in freezer at -20 to -21°C**												
	20	**30**	**62**	**91**	**121**	**154**	**182**	**215**	**282**	**304**			
	2.5	3.5	2.5	2.4	3.2	3.0	3.0	2.8	2.3	3.2			
Dehydrated granules	**Days in freezer at -20 to -21°C**												
	5	**13**	**29**	**61**	**95**	**152**	**179**	**209**	**272**				
	1.8	2.4	2.7	2.1	2.3	2.5	2.6	2.3	1.9				
Processed dried peels	**Days in freezer at -20 to -21°C**												
	6	**14**	**30**	**62**	**96**	**120**	**153**	**180**	**210**	**272**	**300**	**330**	**362**
	88	96	78	94	58	49	154	137	105	125	135	160	133
Processed wet peels	**Days in freezer at -20 to -21°C**												
	9	**42**	**62**	**91**	**121**	**153**	**183**	**217**		**272**			
	33	34	27	37	22	23	26	37		39			

Mammalian products

Storage stability studies were conducted on liver, muscle and milk (Daun and Zeller, 1995; Daun, 1996b). Samples were fortified with 0.1 mg/kg chlorpropham or HSA and stored under the same conditions as samples collected from treated cows (-20°C) to provide for a minimum of duplicate analyses at each of 6 time points plus several contingency samples. Samples were analysed on the day of fortification and after various periods of storage (Tables 18 and 19).

Table 18. Storage stability at -20°C of chlorpropham in fortified samples of milk, liver and muscle of cows (Daun and Zeller, 1995; Daun, 1996b).

Sample	Storage period (days)	Fortification level (mg/kg)	Chlorpropham found (mg/kg)	% remaining	% of fresh fortification
Whole milk	NA	Control	<0.01	NA	
	0	0.1	0.112	112	
	0	0.1	0.079	79	
	NA	Control	<0.01	NA	
	0	0.1	0.094	94	
	7	0.1	0.096	96	102
	7	0.1	0.087	87	92
	NA	Control	<0.01	NA	
	0	0.1	0.113	113	
	29	0.1	0.079	79	70
	29	0.1	0.083	83	73
	NA	Control	<0.01	NA	
	0	0.1	0.088	88	
	59	0.1	0.07	70	79
	59	0.1	0.062	62	70
	NA	Control	0.019	NA	
	0	0.1	0.093	93	
	127	0.1	0.078	78	84
	127	0.1	0.089	89	96
Liver	NA	Control	0.011	NA	
	0	0.1	0.081	81	
	0	0.1	0.073	73	
	NA	Control	0.018	NA	
	0	0.1	0.078	78	
	7	0.1	0.104	104	133
	7	0.1	0.057	57	73
	NA	Control	0.01	NA	
	0	0.1	0.103	103	
	14	0.1	0.089	89	87
	NA	Control	0.012	NA	
	0	0.1	0.094	94	
	28	0.1	0.085	85	91
	28	0.1	0.091	91	98
Muscle	NA	Control	0.022	NA	
	0	0.1	0.082	82	
	0	0.1	0.094	94	
	NA	Control	0.01	NA	
	0	0.1	0.102	102	
	7	0.1	0.104	104	102
	7	0.1	0.08	80	78
	NA	Control	0.021	NA	
	0	0.1	0.096	96	
	15	0.1	0.076	76	80
	15	0.1	0.077	77	80
	NA	Control	<0.01	NA	
	0	0.1	0.105	105	
	29	0.1	0.112	112	107
	29	0.1	0.126	126	120
	NA	Control	<0.01	NA	
	0	0.1	0.091	91	
	59	0.1	0.092	92	102
	59	0.1	0.084	84	93

NA: not applicable

Table 19. Storage stability at -20°C of HSA in fortified samples of milk, liver and muscle of cows (Daun and Zeller, 1995; Daun, 1996b).

Sample	Storage period (days)	Fortification level (mg/kg)	HSA found (mg/kg)	% remaining	% of fresh fortification
Whole milk	NA	Control	<0.05		
	0	0.1	0.077	77	
	0	0.1	0.075	75	
	NA	Control	<0.05	NA	
	0	0.1	0.076	76	
	7	0.1	0.064	64	86
	7	0.1	0.08	80	105
	NA	Control	<0.05	NA	
	0	0.1	0.085	85	
	14	0.1	0.086	86	101
	14	0.1	0.084	84	99
	NA	Control	<0.05	NA	
	0	0.1	0.078	78	
	31	0.1	0.083	83	107
	31	0.1	0.082	82	106
	NA	Control	<0.05	NA	
	0	0.1	0.081	81	
	59	0.1	0.046	46	57
	59	0.1	0.076	76	94
	NA	Control	<0.05	NA	
	0	0.1	0.076	76	
	133	0.1	0.073	73	96
	133	0.1	0.07	70	91
Liver	NA	Control	<0.05	NA	
	0	0.1	0.071	71	
	0	0.1	0.069	69	
	NA	Control	<0.05	NA	
	0	0.1	0.103	103	
	7	0.1	0.079	79	77
	7	0.1	0.075	75	73
	NA	Control	<0.05	NA	
	0	0.1	0.108	108	
	16	0.1	0.076	76	70
	16	0.1	0.07	70	64
	NA	Control	0.06	NA	
	0	0.1	0.155	155	
	30	0.1	0.111	111	72
	30	0.1	0.103	103	67
	NA	Control	<0.05	NA	
	0	0.1	0.135	135	
	59	0.1	0.102	102	76
	59	0.1	0.099	99	73
Muscle	NA	Control	<0.05	NA	
	0	0.1	0.04	40	
	0	0.1	0.054	54	
	NA	Control	<0.05	NA	
	0	0.1	0.066	66	
	7	0.1	0.06	60	91
	7	0.1	0.052	52	79
	NA	Control	0.001	NA	
	0	0.1	0.061	61	
	21	0.1	0.044	44	73
	21	0.1	0.052	52	85
	NA	Control	<0.05	NA	
	0	0.1	0.041	41	
	34	0.1	0.055	55	132

Sample	Storage period (days)	Fortification level (mg/kg)	HSA found (mg/kg)	% remaining	% of fresh fortification
	34	0.1	0.051	51	123
	NA	Control	<0.05	NA	
	0	0.1	0.069	69	
	41	0.1	0.047	47	68
	41	0.1	0.056	56	81
	NA	Control	<0.05	NA	
	0	0.1	0.066	66	
	63	0.1	0.028	28	43
	63	0.1	0.075	75	114
	NA	Control	0.003	NA	
	0	0.1	0.079	79	
	92	0.1	0.068	68	86
	92	0.1	0.066	66	84
	NA	Control	0.006	NA	
	0	0.1	0.066	66	
	122	0.1	0.059	59	90
	122	0.1	0.055	55	84

NA: not applicable

Definition of the residue

Plants (potatoes). Metabolism studies on stored potatoes established that 10% of the applied radioactivity was in the peel after washing and 3% in the pulp. 85% of the residue in the peel was chlorpropham and 3.5% 3-chloroaniline. In the pulp 42% was chlorpropham, two different conjugates of *p*-hydroxy-chlorpropham each accounted for 18%, and 6% was a conjugate of 3-chloroaniline. Thus the main metabolic pathway was hydroxylation at the *p*- position and subsequent conjugation. A minor pathway was decarboxylation to 3-chloroaniline.

In a supervised residue trial on stored potatoes the only metabolite detected was 3-chloroaniline at about 2% of the level found for chlorpropham but the analytical method for 3-chloroaniline showed insufficient recoveries (approximately 40-70% from whole potato, pulp and peel at a fortification level of 0.4 mg/kg). Residues of *p*-methoxy-chlorpropham and *p*-hydroxy-chlorpropham or its conjugates were not detected. Although the samples were kept frozen for several months before analysis, the absence of these metabolites was confirmed in the storage stability study included in this trial, and they were not detected shortly after sampling and processing.

On the basis of these findings the Meeting recommended that the definition of the residue in potatoes for enforcement and risk assessment purposes should be chlorpropham *per se*.

Animal products. In metabolism studies on rats, goats and hens chlorpropham was rapidly virtually fully absorbed, extensively metabolised and quickly excreted. However there were differences between the ultimate residue composition in the edible products of hens and goats.

In hens 92% and 68% of the residue in the fat and skin respectively was chlorpropham, and in other tissues and in eggs it was 3-chloro-4-hydroxyaniline conjugates ranging from 22-70%, in eggs the main compound is the *O*-sulfonic acid conjugate. Since these conjugates together are a major residue in these tissues and in eggs, they should be included in the definition of the residue. As potatoes are less than 10% of the feed for poultry (see FAO Manual p125), and no hen feeding study nor analytical method for poultry products were submitted, no definition of the residue in poultry products is recommended.

In goats, the main residue in milk and kidney is *p*-hydroxy-chlorpropham *O*-sulfonate (HSA; 81% and 16% of the TRR respectively), and in fat tissues it is chlorpropham (88%). No methods of analysis are available to determine the two residues simultaneously. As the metabolite was

considered to be of no toxicological significance by the 2000 JMPR, the Meeting agreed that the residue definition for animal products for compliance with the MRL and dietary risk assessment should be chlorpropham only.

The chlorpropham log P_{OW} of 3.4 and the presence of chlorpropham in fat and cream but not in muscle or skimmed milk in the dairy cow feeding study indicate fat-solubility. No octanol/water partition coefficient was reported for HSA but the chemical nature of the molecule suggests that its fat-solubility would be low.

USE PATTERN

Chlorpropham is used as a growth regulator to suppress potato sprouting during storage after harvest. This use is registered in Australia, Europe and the USA (Table 20). Labels were submitted by Australia (Simpson and Hamilton, 2001) and the Chlorpropham Task Force in the USA. Germany provided information on GAP, but without labels.

Further uses are for weed control as a pre- or post-emergence herbicide for vegetables and flower bulbs in Europe (Table 21). As the product is applied at an early stage, a post-harvest interval is not specified. Labels were submitted by the Chlorpropham Task Force in the USA.

Table 20. Registered uses of chlorpropham for the post-harvest treatment of ware potatoes for sprout control.

Country	Form, conc. ai	Application				WhP[1] (days)
		Method	Remarks/label information	Rate (kg ai/t)	No.	
Australia	DP 25 g/kg	dusting	Treatment must be managed so that potatoes removed from storage and sent for processing contain less than 30 mg/kg chlorpropham	0.038		
	SL 500 g/l	fogging	Application rate will depend upon storage conditions. Retreatment may be necessary if residues fall below 25 mg/kg.	0.03		
Belgium	DP 10 g/kg	dusting		0.018-0.02		
	HN 300 g/l	hot fogging	In air-cooled storage	0.018		
	EC 300 g/l	spraying or fogging	On the conveyor belt (no hot fogging)	0.018	1	
France	DP 10 g/kg	dusting		0.01		
Germany	DP 10 g/kg	dusting		0.01-0.02	1	
Netherlands	DP 10 g/kg	dusting	In air-cooled boxes up to 6°C Re-treatments are possible	0.01		60
		dusting	Normal cool storage	0.02	1	
	HN 300 g/l	fogging		0.018[2]	1-3	
UK	HN 500 g/l	hot fogging in boxes	Retreatments should be after 80-100 days, 45 days apart, if the storage period is uncertain, use half dose rate.	0.045[2] 0.015		21
		hot fogging in bulk	Minimum interval of 45 days between treatments	0.053[2] 0.018		
	M 300 g/l	fogging in boxes and bulk	Retreatments should be after 45-90 days	0.064[2] 0.014-0.021	3	21
	LF 500 g/l	fogging in boxes	Retreatments should be after 45-90 days	0.014-0.021	3	21
		fogging in bulk	1st application 2nd and 3rd application	0.021 0.014	3	21
	M 500 g/l	fogging in boxes	Retreatments should be after 45-90 days	0.014-0.021	3	21
		fogging in bulk	1st application 2nd and 3rd application	0.021 0.014	3	21

Country	Form, conc. ai	Application				WhP[1] (days)
		Method	Remarks/label information	Rate (kg ai/t)	No.	
	GR 50 g/kg	Sprinkling of granules over the top of boxes and bulk	Retreatments by fogging should be made using other formulations, 45-90 days between applications	0.025	3	21
	M 500 g/l	fogging boxes		0.021	5	21
		fogging n bulk		0.014	5	21
	M 600 g/l	hot fogging in boxes	Retreatments should be after 80-100 days.	0.018		21
		hot fogging in bulk	If the storage period is uncertain use half dose rate	0.015		21
USA	EC 250 g/l	spraying	Spraying on the conveyor belt during transport into storage	0.01		
	EC 240 g/l	spraying	Spraying on the conveyor belt during transport into storage	0.01		
	Aerosol 1000 g/l	fogging	Re-treatments are possible; adapt rate to storage period and temperature	0.015-0.025		
	Aerosol 840 g/l	fogging	Re-treatments are possible; adapt rate to storage period and temperature	0.015-0.022		

[1] Withholding period
[2] Maximum total dose

Table 21. Registered uses of chlorpropham for weed control.

Crop	Country	Form, conc. ai	Application			
			Method	Remarks	Rate (kg ai/ha)	No.
Carrots	UK	EC 400 g/l	spraying pre-emergence	within 3 days of drilling	1.1-1.7	1
Grassland	Netherlands	EC 400 g/l			1.2-1.6	
Leek	UK	EC 400 g/l	spraying pre-emergence		1.1-4.5 4.5	1
Lettuce	UK	EC 400 g/l	spraying pre-emergence		1.1	1
Onion	UK	EC 400 g/l	spraying pre-emergence post-emergence to 4 leaf stage		1.1-4.5 2.2	1
Flower bulbs	Netherlands	EC 400 g/l			1.6	
Flower bulbs	UK	EC 400 g/l	spraying pre-emergence	to weed-free soil	1.6-4.5	1
	UK	EC 400 g/l	spraying post-emergence	at plant height of 5 cm and post-flowering	2.2-3.4	1
	UK	EC 400 g/l	spraying post-emergence	before leaves unfold	2.2	1
Parsley	UK	EC 400 g/l	spraying pre-emergence	immediately after drilling	0.8-1.1	1

RESIDUES RESULTING FROM SUPERVISED TRIALS

Animal products

Cows (Daun and Zeller, 1995). In a feeding study on cows to determine chlorpropham and HSA (*p*-hydroxy-chlorpropham sulfate) residues in edible tissues and milk four groups consisting of three animals each were fed at nominally 0, 290, 870 and 2900 ppm in the feed for 28 days. Actual mean chlorpropham dietary burdens were 322 ppm, 955 ppm, and 3111 ppm based on feed consumption during the study. No changes in milk production or feed consumption, or any other adverse reactions were observed.

Samples of milk were collected twice daily from each animal from day -1. Afternoon samples were stored at approximately 5°C until combined with the next morning's sample, then stored frozen at -20°C. Tissue samples were collected within 16-24 hours after the last dose. Extreme care was taken to maintain tissue sample integrity through processing, extraction, and analysis. The tissue samples were kept on wet ice until ground with liquid nitrogen, and stored at -20°C until extraction.

Low levels of chlorpropham were found in the whole milk, muscle, liver, and kidneys. The fat contained levels from 0.09 mg/kg in one of the cows treated at 332 ppm to 2.8 mg/kg in one treated at 3111 ppm. Chlorpropham in the cream varied from 0.02 mg/kg (cow No. 5, 322 ppm) to 0.64 mg/kg (cow No. 11, 3111 ppm). Minor background chromatographic responses were present in many of the control chromatograms, representing mean apparent concentrations from 0.002 to 0.02 mg/kg.

Residues of HSA calculated as chlorpropham in the tissues, skimmed milk and cream ranged from below the limit of detection (<0.03 mg/kg) to 3.9 mg/kg in one skimmed milk sample (3111 ppm). Residues in the whole milk were higher and roughly proportional to feeding level, ranging from undetected in the samples from the control cows to 6.7 mg/kg in one of the 3111 ppm group. Residues of HSA in whole milk reached nearly maximum levels by the 4th day of dosing and fluctuated throughout the remainder of the dosing period. Although the concentration of HSA in whole milk varied between cows in a given treatment group, the cow producing the highest level of HSA did so consistently over the treatment period.

The levels of chlorpropham and HSA in whole milk and tissues (Tables 22-24) were consistent with those found in the ruminant metabolism study.

Table 22. Residues in whole milk from three treatment groups (3 animals per group) after various periods of treatment (Daun and Zeller, 1995).

Compound	Day of treatment	Treatment groups, residues calculated as chlorpropham[1] (mg/kg)			
		Control	322 ppm	955 ppm	3111 ppm
Chlorpropham	0	<0.01	<0.01	<0.01	<0.01
		<0.01	<0.01	<0.01	<0.01
		<0.01	<0.01	<0.01	<0.01
	1	<0.01	<0.01	<0.01	<0.01
		0.01	<0.01	<0.01	0.032
		<0.01	<0.01	<0.01	<0.01
	4	<0.01	<0.01	<0.01	0.03
		<0.01	<0.01	<0.01	0.06
		<0.01	<0.01	<0.01	0.04
	7	<0.01	<0.01	<0.01	0.03
		<0.01	<0.01	0.01	0.03
		<0.01	<0.01	<0.01	0.03
	10	<0.01	<0.01	<0.01	0.015
		<0.01	<0.01	<0.01	0.03
		<0.01	<0.01	<0.01	0.04
	13	<0.01	<0.01	<0.01	0.02
		<0.01	<0.01	<0.01	0.05
		0.01	<0.01	<0.01	0.03
	14	0.01	<0.01	<0.01	0.04
		<0.01	<0.01	<0.01	0.04
		<0.01	<0.01	<0.01	0.02
	18	<0.01	0.06	<0.01	0.02
		<0.01	0.04	<0.01	0.04
		<0.01	0.03	<0.01	0.02
	21	<0.01	0.03	0.01	0.02
		<0.01	0.03	<0.01	0.05
		<0.01	0.04	<0.01	0.02

Compound	Day of treatment	Treatment groups, residues calculated as chlorpropham[1] (mg/kg)			
		Control	322 ppm	955 ppm	3111 ppm
		<0.01	<0.01	<0.01	0.01
		<0.01	<0.01	<0.01	0.03
	24	<0.01	<0.01	<0.01	0.02
		<0.01	<0.01	<0.01	0.01
		<0.01	<0.01	<0.01	0.04
	28	<0.01	<0.01	0.014	0.02
		<0.03	<0.03	<0.03	<0.03
HSA		<0.03	<0.03	<0.03	<0.03
	0	<0.03	<0.03	<0.03	<0.03
		0.08	0.17	0.52	1.7
		<0.03	0.23	0.55	1.4
	1	<0.03	0.33	0.32	4.1
		<0.03	0.21	0.50	3.2
		<0.03	0.31	0.58	2.5
	4	<0.03	0.5	1.1	6.2
		<0.03	0.22	0.48	3.2
		<0.03	0.46	1.4	2.5
	7	<0.03	0.20	1.2	5.4
		<0.03	0.10	0.54	1.2
		<0.03	0.29	0.76	0.55
	10	<0.03	0.48	1.1	2.2
		<0.03	0.22	0.20	3.7
		<0.03	0.32	0.18	2.9
	13	<0.03	0.54	0.46	5.7
			0.23	0.43	1.0
			0.34	0.45	0.86
	14		0.59	1.1	2.9
		<0.03	0.24	0.46	2.6
		<0.03	0.37	0.52	2.5
	18	<0.03	0.57	1.1	6.7
		<0.03	0.20	0.33	0.50
		<0.03	0.26	0.79	0.55
	21	<0.03	0.46	0.57	3.2
		<0.03	0.22	0.21	0.83
		<0.03	0.52	0.20	0.37
	24	<0.03	0.58	0.26	3.0
		<0.03	0.61	0.56	0.83
		<0.03	0.15	0.46	0.55
	28	<0.03	0.44	0.64	3.4

[1] Conversion factor from HSA (MW 309.7) to chlorpropham (MW 213.7): 0.69

Table 23. Residues of chlorpropham and HSA in skimmed milk and cream from day 14 of treatment (Daun and Zeller, 1995).

Sample	Compound	Treatment groups, residues calculated as chlorpropham (mg/kg)			
		Control	322 ppm	955 ppm	3111 ppm
		0.01	<0.01	<0.01	<0.01
		<0.01	<0.01	<0.01	<0.01
Skimmed Milk	Chlorpropham	<0.01	<0.01	<0.01	<0.01
		<0.03	0.14	0.42	2.2
		<0.03	0.20	0.65	1.9
	HSA	<0.03	0.50	0.76	3.9
		<0.01	0.03	0.05	0.18
		0.01	0.02	0.05	0.64
Cream	Chlorpropham	<0.01	0.03	0.09	0.21
		<0.03	0.15	0.4	2.3
		<0.03	0.23	0.66	1.7
	HSA	<0.03	0.37	0.97	3.6

Table 24. Residues of chlorpropham and HSA in cattle tissues (Daun and Zeller, 1995).

Sample	Compound	Treatment groups, residues calculated as chlorpropham (mg/kg)			
		Control	322 ppm	955 ppm	3111 ppm
Liver	Chlorpropham		0.01	<0.01	0.02
		0.01	0.02	0.012	0.01
		0.02	<0.01	<0.01	0.02
	HSA	<0.03	<0.03	<0.03	0.06
		<0.03	<0.03	<0.03	0.04
		<0.03	<0.03	<0.03	<0.03
Kidney	Chlorpropham	0.01	<0.01	<0.01	<0.01
		<0.01	<0.01	<0.01	0.02
		0.02	<0.01	<0.01	<0.01
	HSA	<0.03	0.12	0.76	1.0
		<0.03	0.24	1.0	2.3
		<0.03	0.26	1.2	1.5
Muscle	Chlorpropham	<0.01	<0.01	<0.01	0.11
		0.01	0.01	0.01	0.02
		0.01	<0.01	<0.01	<0.01
	HSA	<0.03	<0.03	<0.03	<0.03
		<0.03	<0.03	<0.03	<0.03
		<0.03	<0.03	<0.03	<0.03
Fat	Chlorpropham	0.02	0.11	0.34	0.97
		<0.01	0.09	0.18	2.8
		0.02	0.13	0.26	0.15
	HSA	<0.03	<0.03	<0.03	<0.03
		<0.03	<0.03	<0.03	<0.03
		<0.03	<0.03	<0.03	<0.03

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

Potatoes. The results of the supervised trials on ware potatoes are shown in Tables 25-32. When residues were not detected, they are shown as below the method detection limit (MDL). The lowest validated fortification level, the limit of quantification (LOQ), was about 5 times higher. Residues of chlorpropham and its metabolites as well as application rates have generally been rounded to 2 significant figures or, for residues near the MDL, to 1 significant figure. Although all trials included control plots residues in control samples are recorded only when they exceeded the MDL. Values are not corrected for recoveries. Residues from trials according to GAP are underlined; results used to estimate STMRs are double underlined.

Kleinkopf and Thomson (1992); Goodrick *et al*. (1993b-d). An extensive trial on mature potato tubers stored under commercial conditions in bins was conducted according to GLP and EPA pesticide Assessment Guidelines Subdivision 0 - Residue Chemistry Series 171-4, Magnitude of the Residue. The storage capacity of the bins ranged from approximately 54 to 68 tonnes. Each bin had its own air ventilation equipment, refrigeration unit and computer-controlled monitoring systems to measure sampling pile conditions accurately, and was designed to allow tuber sampling during storage. Industry standards of relative humidity and temperature with continuous air flow were followed. Each bin was aerosol-fogged separately. Before placing the potatoes in the fumigation/storage bins, hand removal of rocks, dirt clods, vegetative debris and rotten tubers was attempted. Five bins were filled to 75% with potatoes and stored at 14°C for two weeks. Thereafter, bin temperatures were gradually reduced to:

a) 5°C in bin 1, the untreated control, to help prevent sprouting, and to 4°C in week 27 to prolong the sprout-free condition
b) 7.2°C in bins 2 and 3 for storage of potatoes for the fresh market or for processing into frozen or dehydrated products

c) 10°C in bins 4 and 5 for storage of potatoes for processing into chips.

Three commercial formulations of chlorpropham were applied to the stored potatoes at the prescribed maximum rates in a manner consistent with standard practices in the potato industry:

- Bins 2, 3, 4 and 5, each containing approximately 63.4 tonnes of potatoes, served as the fumigation chambers for thermal fogging with two aerosol formulations (Decco 273 Aerosol containing 50% ai, and Sprout Nip 4A Aerosol containing 47% ai).
- The other formulation, an emulsifiable concentrate (Decco 276 EC, containing 26% ai), was applied as 1% aqueous direct spray to samples of potatoes from bins 2 and 3 that were also thermally fogged. (The application of a 1% aqueous emulsion to potato tubers moving along a conveyor line is called a "direct spray".) The EC formulation was applied once before and once after thermal fogging at 5 different times to tubers collected from three sampling depths in storage bins 2 and 3. After collecting a 60-tuber composite sample, the tubers were washed in water and allowed to air-dry until damp. Rotten tubers were discarded, and those remaining were weighed and the weight used to calculate the amount of 1% chlorpropham emulsion required for an application rate of 0.01 kg ai/t potatoes. Some samples from the untreated control (bin 1) were treated once by direct spray.

Two sampling pipes were inserted into each bin before filling the bins with potatoes to facilitate sampling at various depths. The pipes allowed personnel access to selected parts of the potato pile for sampling at 0.3, 2.4 and 4.6 m above the air ducts in the bin floor.

Duplicate top, middle and bottom samples (A and B) were thus collected 0, 5, 91, 96, 140, 145 and 215 days after initial sampling from various locations within the piles, stored at 3.3-4.4 °C, and shipped as soon as possible under ambient conditions (shipment lasted for 2 days) to an analytical laboratory or processing plant. Samples were prepared, homogenized, and frozen until extraction. Upon arrival, samples were stored at 1.1-4.4 °C until composited and homogenized, then stored at -20 to -21°C for 3-12 months. Samples were extracted, and chlorpropham, 3-chloroaniline, conjugates of *p*-hydroxy-chlorpropham and *p*-methoxy-chlorpropham were quantified by GLC with an NPD. The results are shown in Table 25.

Table 25. Residues of chlorpropham and its metabolites in whole potatoes (Kleinkopf and Thomson, 1992; Goodrick *et al*., 1993b-d).

Treatment	Days after initial treatment	Bin no., location in pile[1]	Residue[2] (mg/kg]			
			chlorpropham	3-chloroaniline	4'-hydroxy-chlorpropham	*p*-methoxy-chlorpropham
Report 92CIPC04, 3 different treatments: 1 x direct spray; 1 x fogging + direct spray; 2 x fogging + direct spray						
EC direct spray 0.01 kg ai/t potatoes, applied 19-11-91	0	1, top A	4.3	<0.08	<0.08	<0.08
		1, top B	4.3	<0.08	<0.08	<0.08
		1, top C	6.4	<0.08	<0.08	<0.08
		1, top D	8.2	<0.08	<0.08	<0.08
EC direct spray 0.02 kg ai/t potatoes, applied 14-11-91	0	2, bottom A	3.8	<0.08	<0.08	<0.08
		2, bottom B	3.6	<0.08	<0.08	<0.08
		2, middle A	4.3	<0.08	<0.08	<0.08
		2, middle B	3.6	<0.08	<0.08	<0.08
		2, top A	3.9	<0.08	<0.08	<0.08
		2, top B	3.6	<0.08	<0.08	<0.08
		3, bottom A	2.7	<0.08	<0.08	<0.08
		3, bottom B	3.5	<0.08	0.10	<0.08
		3, middle A	3.3	<0.08	<0.08	<0.08
		3, middle B	2.9	<0.08	<0.08	<0.08
		3, top A	3.6	<0.08	<0.08	<0.08
		3, top B	3.8	<0.08	<0.08	<0.08

Treatment	Days after initial treatment	Bin no., location in pile[1]	Residue[2] (mg/kg]			
			chlorpropham	3-chloroaniline	4'-hydroxy-chlorpropham	*p*-methoxy-chlorpropham
Aerosol fogging 0.02 kg ai/t potatoes, applied 15-11-91 + EC direct spray 0.01 kg ai/t potatoes, applied 19-11-91	5	2, bottom A	9.5	<0.08	<0.08	<0.08
		2, bottom B	11	<0.08	<0.08	<0.08
		2, middle A	7.2	<0.08	<0.08	<0.08
		2, middle B	7.6	<0.08	<0.08	<0.08
		2, top A	6.9	<0.08	<0.08	<0.08
		2, top B	7.0	<0.08	<0.08	<0.08
		3, bottom A	7.5	<0.08	<0.08	<0.08
		3, bottom B	7.2	<0.08	<0.08	<0.08
		3, middle A	5.0	<0.08	<0.08	<0.08
		3, middle B	4.8	<0.08	<0.08	<0.08
		3, top A	9.1	0.08	<0.08	<0.08
		3, top B	8.0	<0.08	<0.08	<0.08
Aerosol fogging 0.02 kg ai/t potatoes, applied 15-11-91 + EC direct spray 0.01 kg ai/t potatoes, applied 13-02-92	91	2, bottom A	8.9	0.11	<0.08	<0.08
		2, bottom B	8.6	0.10	<0.08	<0.08
		2, middle A	9.3	0.10	<0.08	<0.08
		2, middle B	8.3	0.10	<0.08	<0.08
		2, top A	6.5	0.09	<0.08	<0.08
		2, top B	7.3	0.09	<0.08	<0.08
		3, bottom A	7.6	0.10	<0.08	<0.08
		3, bottom B	9.4	0.11	<0.08	<0.08
		3, middle A	6.1	0.10	<0.08	<0.08
		3, middle B	6.3	0.10	<0.08	<0.08
		3, top A	9.1	0.10	<0.08	<0.08
		3, top B	9.0	0.12	<0.08	<0.08
Aerosol fogging 0.02 kg ai/t potatoes, applied 15-11-91 + Aerosol fogging 0.02 kg ai/t potatoes, applied 14-02-92 + EC direct spray 0.01 kg ai/t potatoes, applied 18-02-92	96	2, bottom A	8.3	0.16	<0.08	<0.08
		2, bottom B	9.7	0.14	<0.08	<0.08
		2, middle A	8.5	0.12	<0.08	<0.08
		2, middle B	7.0	0.12	<0.08	<0.08
		2, top A	6.0	<0.08	<0.08	<0.08
		2, top B	7.2	<0.08	<0.08	<0.08
		3, bottom A	12	0.13	<0.08	<0.08
		3, bottom B	14	0.16	<0.08	<0.08
		3, middle A	9.3	0.12	<0.08	<0.08
		3, middle B	8.8	0.12	<0.08	<0.08
		3, top A	10	0.13	<0.08	<0.08
		3, top B	9.5	0.12	<0.08	<0.08
Aerosol fogging 0.02 kg ai/t potatoes, applied 15-11-91 + Aerosol fogging 0.02 kg ai/t potatoes applied 14-02-92 + EC direct spray 0.01 kg ai/t potatoes applied 02-04-92	140	2, bottom A	11	0.12	<0.08	<0.08
		2, bottom B	10.5	0.1	<0.08	<0.08
		2, middle A	8.4	0.12	<0.08	<0.08
		2, middle B	8.9	0.1	<0.08	<0.08
		2, top A	9.0	0.11	<0.08	<0.08
		2, top B	7.5	0.1	<0.08	<0.08
		3, bottom A	13	0.12	<0.08	<0.08
		3, bottom B	12	0.12	<0.08	<0.08
		3, middle A	12	0.12	<0.08	<0.08
		3, middle B	9.4	0.12	<0.08	<0.08
		3, top A	13	0.13	<0.08	<0.08
		3, top B	12	0.12	<0.08	<0.08
Aerosol fogging 0.02 kg ai/t potatoes, applied 15-11-91 + Aerosol fogging 0.02 kg ai/t potatoes, applied 14-02-92 + EC direct spray 0.01 kg ai/t potatoes, applied 16-06-92	215	2, bottom A	8.2	0.14	<0.08	<0.08
		2, bottom B	7.7	0.12	<0.08	<0.08
		2, middle A	7.5	0.12	<0.08	<0.08
		2, middle B	8.0	0.14	<0.08	<0.08
		2, top A	7.6	0.14	<0.08	<0.08
		2, top B	7.1	0.13	<0.08	<0.08
		3, bottom A	11	0.14	<0.08	<0.08
		3, bottom B	9.8	0.15	<0.08	<0.08
		3, middle A	7.6	0.13	<0.08	<0.08
		3, middle B	8.3	0.14	<0.08	<0.08

Treatment	Days after initial treatment	Bin no., location in pile[1]	Residue[2] (mg/kg]			
			chlorpropham	3-chloroaniline	4'-hydroxy-chlorpropham	*p*-methoxy-chlorpropham
		3, top A	7.4	0.12	<0.08	<0.08
		3, top B	8.2	0.13	<0.08	<0.08
Report 92CIPC05, 2 different treatments: 1 x fogging; 2 x fogging						
Aerosol fogging 0.02 kg ai/t potatoes, applied 15-11-91 sampling at 19-11-91	5	2, bottom A	8.7	<0.08	<0.08	<0.08
		2, bottom B	7.3	<0.08	<0.08	<0.08
		2, middle A	4.2	<0.08	<0.08	<0.08
		2, middle B	3.2	<0.08	<0.08	<0.08
		2, top A	3.6	<0.08	<0.08	<0.08
		2, top B	3.2	<0.08	<0.08	<0.08
		3, bottom A	7.9	0.2	<0.08	<0.08
		3, bottom B	8.9	0.21	<0.08	<0.08
		3, middle A	7.3	0.2	<0.08	<0.08
		3, middle B	6.1	0.19	<0.08	<0.08
		3, top A	6.7	0.19	<0.08	<0.08
		3, top B	6.3	0.19	<0.08	<0.08
Aerosol fogging 0.02 kg ai/t potatoes, applied 15-11-91 sampling at 13-02-91	91	2, bottom A	2.8	0.13	<0.08	<0.08
		2, bottom B	4.1	0.12	<0.08	<0.08
		2, middle A	3.6	0.12	<0.08	<0.08
		2, middle B	2.6	0.12	<0.08	<0.08
		2, top A	1.4	0.12	<0.08	<0.08
		2, top B	2.0	0.12	<0.08	<0.08
		3, bottom A	5.2	0.14	<0.08	<0.08
		3, bottom B	4.5	0.14	<0.08	<0.08
		3, middle A	4.3	0.15	<0.08	<0.08
		3, middle B	6.1	0.14	<0.08	<0.08
		3, top A	7.1	0.17	<0.08	<0.08
		3, top B	5.1	0.14	<0.08	<0.08
Aerosol fogging 0.02 kg ai/t potatoes applied 15-11-91 + Aerosol fogging 0.02 kg ai/t potatoes, applied 14-02-92 sampling at 18-02-92	96	2, bottom A	9.8	<0.08	<0.08	<0.08
		2, bottom B	9.9	<0.08	<0.08	<0.08
		2, middle A	6.9	<0.08	<0.08	<0.08
		2, middle B	6.4	<0.08	<0.08	<0.08
		2, top A	6.0	<0.08	<0.08	<0.08
		2, top B	4.5	<0.08	<0.08	<0.08
		3, bottom A	16	0.18	<0.08	<0.08
		3, bottom B	10	0.18	<0.08	<0.08
		3, middle A	9.4	0.14	<0.08	<0.08
		3, middle B	9.5	0.13	<0.08	<0.08
		3, top A	12	0.15	<0.08	<0.08
		3, top B	11	0.13	<0.08	<0.08
Aerosol fogging 0.02 kg ai/t potatoes, applied 15-11-91 + Aerosol fogging 0.02 kg ai/t potatoes, applied 14-02-92 sampling at 07-04-92	140	2, bottom A	7.1	0.1	<0.08	<0.08
		2, bottom B	9.7	0.12	<0.08	<0.08
		2, middle A	8.1	0.09	<0.08	<0.08
		2, middle B	9.2	0.1	<0.08	<0.08
		2, top A	4.7	0.08	<0.08	<0.08
		2, top B	4.4	0.09	<0.08	<0.08
		3, bottom A	11	0.1	<0.08	<0.08
		3, bottom B	18	0.12	<0.08	<0.08
		3, middle A	12	0.08	<0.08	<0.08
		3, middle B	11	0.08	<0.08	<0.08
		3, top A	6.8	0.09	<0.08	<0.08
		3, top B	9.4	0.09	<0.08	<0.08

Treatment	Days after initial treatment	Bin no., location in pile[1]	Residue[2] (mg/kg]			
			chlorpropham	3-chloroaniline	4'-hydroxy-chlorpropham	*p*-methoxy-chlorpropham
Aerosol fogging 0.02 kg ai/t potatoes, applied 15-11-91 + Aerosol fogging 0.02 kg ai/t potatoes, applied 14-02-92 sampling at 16-06-92	215	2, bottom A	7.8	<0.08	<0.08	<0.08
		2, bottom B	7.0	<0.08	<0.08	<0.08
		2, middle A	6.6	<0.08	<0.08	<0.08
		2, middle B	6.8	<0.08	<0.08	<0.08
		2, top A	6.8	<0.08	<0.08	<0.08
		2, top B	5.4	<0.08	<0.08	<0.08
		3, bottom A	8.2	0.09	<0.08	<0.08
		3, bottom B	8.8	0.08	<0.08	<0.08
		3, middle A	11	0.08	<0.08	<0.08
		3, middle B	8.2	<0.08	<0.08	<0.08
		3, top A	7.8	<0.08	<0.08	<0.08
		3, top B	8.2	<0.08	<0.08	<0.08
Report 92CIPC06, 2 different treatments: 1 x fogging; 2 x fogging						
Aerosol fogging 0.03 kg ai/t potatoes, applied 19-11-91 sampling at 19-11-92	5	4, bottom A	23	0.23	<0.08	<0.08
		4, bottom B	21	0.18	<0.08	<0.08
		4, middle A	7.6	0.11	<0.08	<0.08
		4, middle B	6.4	0.1	<0.08	<0.08
		4, top A	4.7	0.1	<0.08	<0.08
		4, top B	4.7	0.09	<0.08	<0.08
		5, bottom A	16	0.16	<0.08	<0.08
		5, bottom B	13	0.19	<0.08	<0.08
		5, middle A	11	0.15	<0.08	<0.08
		5, middle B	13	0.15	<0.08	<0.08
		5, top A	13	0.17	<0.08	<0.08
		5, top B	10	0.13	<0.08	<0.08
Aerosol fogging 0.03 kg ai/t potatoes, applied 19-11-91 sampling at 13-02-92	91	4, bottom A	7.8	0.15	<0.08	<0.08
		4, bottom B	11	0.15	<0.08	<0.08
		4, middle A	7.0	0.15	<0.08	<0.08
		4, middle B	11	0.16	<0.08	<0.08
		4, top A	5.4	0.16	<0.08	<0.08
		4, top B	3.2	0.14	<0.08	<0.08
		5, bottom A	7.0	<0.08	<0.08	<0.08
		5, bottom B	7.8	<0.08	<0.08	<0.08
		5, middle A	5.6	<0.08	<0.08	<0.08
		5, middle B	14	<0.08	<0.08	<0.08
		5, top A	9.2	<0.08	<0.08	<0.08
		5, top B	7.4	<0.08	<0.08	<0.08
Aerosol fogging 0.03 kg ai/t potatoes, applied 15-11-91 sampling at 02-04-92	140	4, bottom A	8.6	0.26	<0.08	<0.08
		4, bottom B	8.2	0.23	<0.08	<0.08
		4, middle A	10	0.22	<0.08	<0.08
		4, middle B	12	0.25	<0.08	<0.08
		4, top A	7.1	0.23	<0.08	<0.08
		4, top B	5.9	0.22	<0.08	<0.08
		5, bottom A	13	0.16	<0.08	<0.08
		5, bottom B	13	0.19	<0.08	<0.08
		5, middle A	10	0.15	<0.08	<0.08
		5, middle B	12	0.14	<0.08	<0.08
		5, top A	9.8	0.14	<0.08	<0.08
		5, top B	11	0.14	<0.08	<0.08
Aerosol fogging 0.03 kg ai/t potatoes, applied 15-11-91 + 0.015 kg ai/t potatoes, applied 03-04-92 sampling at 07-04-92	145	4, bottom A	13	0.22	<0.08	<0.08
		4, bottom B	12	0.22	<0.08	<0.08
		4, middle A	12	0.2	<0.08	<0.08
		4, middle B	14	0.19	<0.08	<0.08
		4, top A	5.5	0.17	<0.08	<0.08
		4, top B	6.0	0.19	<0.08	<0.08
		5, bottom A	14	0.23	<0.08	<0.08

Treatment	Days after initial treatment	Bin no., location in pile[1]	Residue[2] (mg/kg]			
			chlorpropham	3-chloroaniline	4'-hydroxy-chlorpropham	*p*-methoxy-chlorpropham
		5, bottom B	14	0.18	<0.08	<0.08
		5, middle A	12	0.19	<0.08	<0.08
		5, middle B	16	0.22	<0.08	<0.08
		5, top A	10	0.19	<0.08	<0.08
		5, top B	12	0.20	<0.08	<0.08
Aerosol fogging 0.03 kg ai/t potatoes, applied 15-11-91 + 0.015 kg ai/t potatoes, applied 03-04-92 sampling at 16-06-92	215	4, bottom A	7.8	0.09	<0.08	<0.08
		4, bottom B	8.2	0.09	<0.08	<0.08
		4, middle A	8.0	<0.08	<0.08	<0.08
		4, middle B	7.8	<0.08	<0.08	<0.08
		4, top A	6.7	<0.08	<0.08	<0.08
		4, top B	8.1	<0.08	<0.08	<0.08
		5, bottom A	8.9	0.13	<0.08	<0.08
		5, bottom B	10	0.13	<0.08	<0.08
		5, middle A	11	0.12	<0.08	<0.08
		5, middle B	15	0.13	<0.08	<0.08
		5, top A	11	0.12	<0.08	<0.08
		5, top B	9.5	0.11	<0.08	<0.08

[1] Bottom, middle, top: 0.3, 2.4, 4.6 m above floor ducts respectively
[2] 0.08 mg/kg is method detection limit (MDL), not LOQ

Roland (1998b). In a field study to determine chlorpropham residues in potatoes in France (field part) and Belgium (analytical part) two fogging applications were made, firstly in October 1997 at 7 g ai/t, and secondly in January 1998 at 6 g ai/t. Samples were taken 1 day before and 0, 30 and 60 days after the first application and 0, 30, 60, 90 and 120 days after the second. The potatoes were stored on wooden pallets in a warehouse in piles 5 or 6 pallets high each containing about 1 tonne. The samples were taken in four places sited diagonally in the warehouse from the bottom, the middle and top pallets of the piles. The whole tubers were frozen. After removal of adhering soil by rinsing in running water, the potatoes (not completely thawed) were divided into representative parts. They were peeled with a knife as soon as the surface part was sufficiently tenderised. The residues in tubers and pulp are shown in Tables 26 and 27 respectively. Each value is the mean of two analyses, obtained from separate sub-samples.

Table 26. Residues of chlorpropham in whole tubers after fogging (Roland, 1998b).

Treatment	Days after application	Pallet location, residues (mg/kg)		
		bottom	middle	top
Fogging, pile, NeoStop L 500 (HN, Chlorpropham 500 g/l) 1x 7 g ai/t at 31-10-1997 + 1x 6 g ai/t at 06-01-1998	-1 (before 1st application)	0.5, 0.18, 0..21, 0.29	0.24, 0.2, 0.18, 0.20	0.29, 0.44, 0.33, 0.34
	0 (1st application)	1.2, 1.6, 3.1, 1.7	1.1, 2.0, 1.6, 0.86	3.3, 6.4, 3.2, 2.0
	30 (1st application)	1.2, 1.7, 1.6, 2.0, 1.6	1.1, 2.1, 2.0, 1.7, 1.4	3.4, 13, 6.0, 1.9
	60 (1st application)	0.75, 0.92, 1.8, 1.1	0.67, 1.5, 1.1, 1.1	1.1, 6.1, 3.6, 1.8
	0 (2nd application)	1.5, 1.8, 3.4, 2.6	1.9, 4.0, 3.2, 3.3	4.1, 11, 7.1, 4.1
	30 (2nd application)	1.2, 1.7, 3.5, 2.5	1.6, 3.5, 3.3, 2.9	3.8, 9.0, 8.0, 3.4
	60 (2nd application)	1.4, 1.4, 2.2, 2.4	3.3, 3.1, 3.5, 2.3	1.4, 13, 5.1, 2.4
	90 (2nd application)	1.2, 1.1, 2.2, 1.6	1.1, 2.2, 0.99, 1.6	1.7, 8.3, 4.9, 1.9
	120 (2nd application)	1.2, 0.92, 2.3, 1.9	1.6, 1.9, 1.0, 1.6	1.7, 7.5, 5.6, 2.0

Table 27. Residues of chlorpropham in the pulp of peeled potatoes after fogging (Roland, 1998b).

Days after application	Pallet location, residues (mg/kg)		
	bottom	middle	top
-1 (before 1st application)	<0.02	<0.02	<0.02
0 (1st application)	0.02	0.04	0.05
30 (1st application)	0.03	<0.02	0.04
60 (1st application)	<0.02	0.02	0.03
0 (2nd application)	0.04	0.04	0.23
30 (2nd application)	0.07	0.05	0.08
60 (2nd application)	0.07	0.07	0.07
90 (2nd application)	0.06	0.06	0.14
120 (2nd application)	0.11	0.07	0.24

Roland (1998a). In a field trial in Belgium potatoes given one fogging application of NeoStop (DP 1% chlorpropham) at 150 g/100 kg (equal to 15 g ai/t potatoes) during storage were sampled after 0, 1, 3, 7, 14, 38 and 45 days, and 2, 3, 4, 6 and 8 months. The residues of chlorpropham are shown in Table 28.

Table 28. Residues of chlorpropham in potatoes (Roland, 1998a).

Treatment	Interval after application	Residues (mg/kg)		
		Whole tubers	Peeled potatoes	Cooked potatoes
Manually powdering of the exact quantity above each paper bag filled with potatoes, shaking of the bag. NeoStop (DP, 1% chlorpropham) 1x 15 g ai/t at 12-11-1997	Untreated before application	<0.02	<0.02	<0.02
	0 day following application	3.6	0.11	
	1 day after application	7.9	0.23	
	3 days after application	5.5	0.12	
	7 days after application	8.8	0.18	
	14 days after application	5.8	0.19	
	28 days after application	6.1	0.27	
	45 days after application	4.6	0.24	0.08
	2 months after application	5.3	0.22	
	3 months after application	4.9	0.37	
	4 months after application	3.1	0.36	
	6 months after application	3.2	0.45	
	8 months after application	2.6	0.33	

Brielbeck and Marx (1999a). Seven trials (one a decline trial) were conducted to determine residues of chlorpropham in peeled and unpeeled potato tubers following two fogging applications of Neo-Stop L 500 (chlorpropham 500 g/l HN) equal to 7 and 6 g ai/t in Belgium. For unpeeled samples, tubers were washed with water before weighing and freezing. For peeled samples, tubers were washed, weighed and peeled. Peels and peeled tubers were weighed separately and peels discarded. Peeled tubers were washed again with water, weighed and frozen. The residues in duplicate field samples, each the mean of duplicate analyses, are shown in Table 29.

Table 29. Residues of chlorpropham in potatoes, Saint-Amand, Belgium (Brielbeck and Marx, 1999a).

Treatment	Sample	Time of sampling	Residues (mg/kg)	
			Unpeeled potatoes	Peeled potatoes
Fogging, box, Neo-Stop L 500 (HN, chlorpropham 500 g/l) 1x 7 g ai/t at 17-11-1998 + 1x 6 g ai/t at 18-01-1999	G-09	before treatment 1	<0.02, <0.02	<0.02, <0.02
		1 day after treatment 1	0.49, 0.45	0.03, 0.08
		before treatment 2	0.64, 0.63	0.11, 0.03
		1 day after treatment 2	1.1, 1.2	0.09, 0.07
		29 days after treatment 2	0.70, 0.76	0.09, 0.07
		91 days after treatment 2	0.58, 0.65	0.08, 0.07
	G 10	before treatment 1	<0.02, <0.02	<0.02, <0.02
		7 days after treatment 2	0.70, 0.85	0.06, 0.05
	G 11	before treatment 1	<0.02, <0.02	<0.02, <0.02
		7 days after treatment 2	0.89, 0.67	0.08, 0.08
	G 12	before treatment 1	<0.02, <0.02	<0.02, <0.02
		7 days after treatment 2	0.61, 0.58	0.10, 0.10
	G 13	before treatment 1	<0.02, <0.02	<0.02, <0.02
		7 days after treatment 2	0.96, 0.68	0.14, 0.16
	G 14	before treatment 1	<0.02, <0.02	<0.02, <0.02
		7 days after treatment 2	1.1, 1.2	0.18, 0.22
	G 15	before treatment 1	<0.02, <0.02	<0.02, <0.02
		7 days after treatment 2	1.1, 0.77	<0.02, <0.02

Brielbeck and Marx (1999b). A similar set of seven trials was conducted in Germany with single applications of 1.5 kg/t Neo-Stop (chlorpropham 1% DP, equal to 15 g ai/t). The samples were prepared as above. The results (each the mean of duplicate analyses) are shown in Table 30.

Table 30. Residues of chlorpropham in potatoes, Goch Hülm, Germany (Brielbeck and Marx, 1999b).

Treatment	Sample, variety	Time of sampling	Residues (mg/kg)	
			Unpeeled potatoes	Peeled potatoes
Dusting, box, Neo-Stop, (DP, 1% chlorpropham) 1x 15 g ai/t at 28-10-98	ASU 52, Bintje	before treatment	<0.02, <0.02	<0.02, <0.02
		1 day after treatment	2.6, 2.3	0.10, 0.10
		7 days after treatment	3.8, 2.9	0.08, 0.08
		28 days after treatment	2.1, 1.9	0.14, 0.13
		61 days after treatment	2.7, 2.3	0.04, 0.05
		92 days after treatment	1.4, 1.3	0.11, 0.10
		118 days after treatment	2.0, 1.2	0.13, 0.15
		181 days after treatment	1.9, 2.4	0.07, 0.08
	ASU 46, Bintje	before treatment	<0.02, <0.02	<0.02, <0.02
		30 days after treatment	2.9, 3.4	0.09, 0.11
		61 days after treatment	2.9, 3.2	0.05, 0.06
		92 days after treatment	3.5, 2.8	0.09, 0.09
	ASU 47, Bintje	before treatment	<0.02, <0.02	<0.02, <0.02
		30 days after treatment	4.3, 4.3	0.09, 0.11
		61 days after treatment	4.8, 4.0	0.05, 0.06
		92 days after treatment	2.4, 1.7	0.09, 0.09
	ASU 48, Mentor	before treatment	<0.02, <0.02	<0.02, <0.02
		30 days after treatment	2.2, 2.9	0.04, <0.02
		61 days after treatment	3.0, 3.1	0.06, 0.05
		92 days after treatment	1.7, 1.7	0.06, 0.06

Treatment	Sample, variety	Time of sampling	Residues (mg/kg)	
			Unpeeled potatoes	Peeled potatoes
	ASU 49, Russet Burbank	before treatment	<0.02, <0.02	<0.02, <0.02
		30 days after treatment	3.2, 3.3	0.06, 0.07
		61 days after treatment	2.2, 2.7	0.09, 0.13
		92 days after treatment	2.6, 3.5	0.07, 0.08
	ASU 50, Helmond	before treatment	<0.02, <0.02	<0.02, <0.02
		30 days after treatment	4.6, 4.9	0.13, 0.14
		61 days after treatment	2.7, 2.8	0.11, 0.05
		92 days after treatment	3.9, 4.6	0.35, 0.34
	ASU 51, Nierswalde	before treatment	<0.02, <0.02	<0.02, <0.02
		30 days after treatment	3.0, 4.3	0.14, 0.17
		61 days after treatment	2.7, 2.0	0.48, 0.44
		92 days after treatment	2.8, 2.4	0.15, 0.17

Brielbeck and Marx (1996a,b). In further trials potatoes stored in separate boxes were treated with 1 kg CIPC 1% DP/t (10.6 g ai/t) which is equivalent to AU 95395 and NEO Stop. Samples were taken from the top, the middle and the bottom of the boxes and prepared as before. The results are shown in Table 31.

Table 31. Residues of chlorpropham in potatoes, Keppeln, Germany (Brielbeck and Marx, 1996a,b).

Treatment	Sample source, variety	Time of sampling	Chlorpropham (mg/kg)	
			Unpeeled potatoes	Peeled potatoes
Powdering, box, Neo-Stop, (DP, 1% chlorpropham), 1x 11 g ai/t at 28-02-96	ASU 32, Bintje	before treatment	<0.02, <0.02	<0.025, <0.025
		1-2 hours after treatment	2.9, 3.0	<0.025, <0.025
		30 days after treatment	1.9, 3.0	0.08, 0.07
	ASU 33, Gloria	before treatment	<0.02, <0.02	<0.025, <0.025
		1-2 hours after treatment	2.5, 1.2	<0.025, <0.025
		30 days after treatment	2.1, 2.2	0.06, 0.06
	ASU 34, Hansa	before treatment	<0.02, <0.02	<0.025, <0.025
		1-2 hours after treatment	1.8, 1.7	<0.025, <0.025
		30 days after treatment	1.9, 0.96	<0.025, 0.032
	ASU 35, Cilena	before treatment	<0.02, <0.02	<0.025, <0.025
		1-2 hours after treatment	1.3, 1.2	0.03, <0.025
		30 days after treatment	2.0, 1.4	0.03, 0.06

Brielbeck and Marx (1999c). In four trials potatoes were treated with 1.0 kg of Neo Stop 1% DP/t (10 g ai/t) by dusting immediately before being taken into the warehouse. After sampling, some of the tubers were washed with water and some were washed and peeled as before. The results are shown in Table 32.

Table 32. Residues of chlorpropham in potatoes, Goch Hülm, Germany (Brielbeck and Marx, 1999c).

Treatment	Sample, variety	Time of sampling	Residues (mg/kg)	
			Unpeeled	Peeled
Powdering, box, Neo-Stop (DP, 1% chlorpropham) 1x 10 g ai/t	ASU 42, Bintje	before treatment	<0.02, <0.02	<0.02, <0.02
		1 day after treatment	3.0, 2.9	0.21, 0.12
		30 days after treatment	2.9, 2.6	0.21, 0.30
	ASU 43, Cilena	before treatment	<0.02, <0.02	<0.02, <0.02
		1 day after treatment	1.5, 1.7	0.37, 0.13
		30 days after treatment	1.1, 1.2	0.24, 0.19
		before treatment	<0.02, <0.02	<0.02, <0.02

Treatment	Sample, variety	Time of sampling	Residues (mg/kg)	
			Unpeeled	Peeled
	ASU 44, Hansa	1 day after treatment	2.3, 2.5	0.08, 0.14
		30 days after treatment	2.2, 2.0	0.23, 0.21
	ASU 45, Secura	before treatment	<0.02, <0.02	<0.02, <0.02
		1 day after treatment	3.2, 2.7	0.07, 0.05
		30 days after treatment	2.8, 2.2	0.06, 0.06

Further incomplete residue data were reported by Germany (Anon., 2001) including the results of one trial in 1970 on stored potatoes treated twice with 6.4-13 kg ai/t, WhP 65 days. Chlorpropham residues were reported for peeled potatoes only and ranged from <0.05 to 0.3 mg/kg. No data were available for whole tubers. These results could not be used for evaluation.

In processing

Potatoes (Roland, 1998a). Potatoes stored for 45 days were peeled, put in boiling water and cooked for 20 minutes. The residues in fresh whole tubers were 4.6 mg/kg, in fresh peeled potatoes 0.24 mg/kg and in cooked peeled potatoes 0.08 mg/kg (see also Table 28).

(Swanson *et al.*, 1993; Haws *et al.*, 1993c). The storage conditions and chlorpropham treatments used in industry and in the study reported by Kleinkopf and Thomson (1992) vary with the intended use of the raw commodity for chips, or frozen and dehydrated products. The scheme used by Kleinkopf and Thomson is shown in Table 33.

Table 33. Storage conditions and treatment of potatoes for processing as chips, or frozen or dehydrated products (Kleinkopf and Thomson, 1992).

	For chips	For frozen or dehydrated products
Storage bins	4 and 5	2 and 3
Storage conditions	10°C, 5% relative humidity	7.2°C, 95% relative humidity
Chlorpropham treatments	aerosol fogging	aerosol fogging
Chlorpropham formulations	Sprout Nip 4A Aerosol	Decco 273 Aerosol
Chlorpropham rates	0.033 kg ai/t potatoes-initial fogging 0.017 kg ai/t potatoes-second fogging	0.02 kg ai/t potatoes-initial fogging 0.02 kg ai/t potatoes-second fogging
Treatment schedule	15-11-1991 initial aerosol fogging 03-04-1992 second aerosol fogging	15-11-1991 initial aerosol fogging 14-02-1992 second aerosol fogging

The tubers were processed by standard industrial procedures. Stored tubers were shipped intact and unfrozen to a pilot processing plant. They were stored at 3.3°C before being processed into frozen French fries and chips with and without skin, dehydrated granules and wet and dry peel. Thereafter the products were homogenized and stored at -20 to -21°C for 2-10 months. Chlorpropham and its metabolites 3-chloroaniline, *p*-hydroxy-chlorpropham (including conjugates) and *p*-methoxy-chlorpropham were determined in the French fries, chips and in the canola oil used during processing. The wet and dried peel removed during processing was retained for analysis (Swanson *et al.*, 1993).

Chips. Although commercial potato processing includes a water wash to remove starch the procedure did not include this, maximizing the potential residue of chlorpropham in the chips. Neither did it include salting the chips. Figure 3 shows the processing of chips at the pilot plant.

Table 34 shows the residues of chlorpropham and 3-chloroaniline in chips. Residues of *p*-hydroxy-chlorpropham and *p*-methoxy-chlorpropham were undetectable in any fresh or processed product. Processing factors could not be determined as different samples were used for the determination of residues in the raw agriculture commodity (RAC) and the processed product.

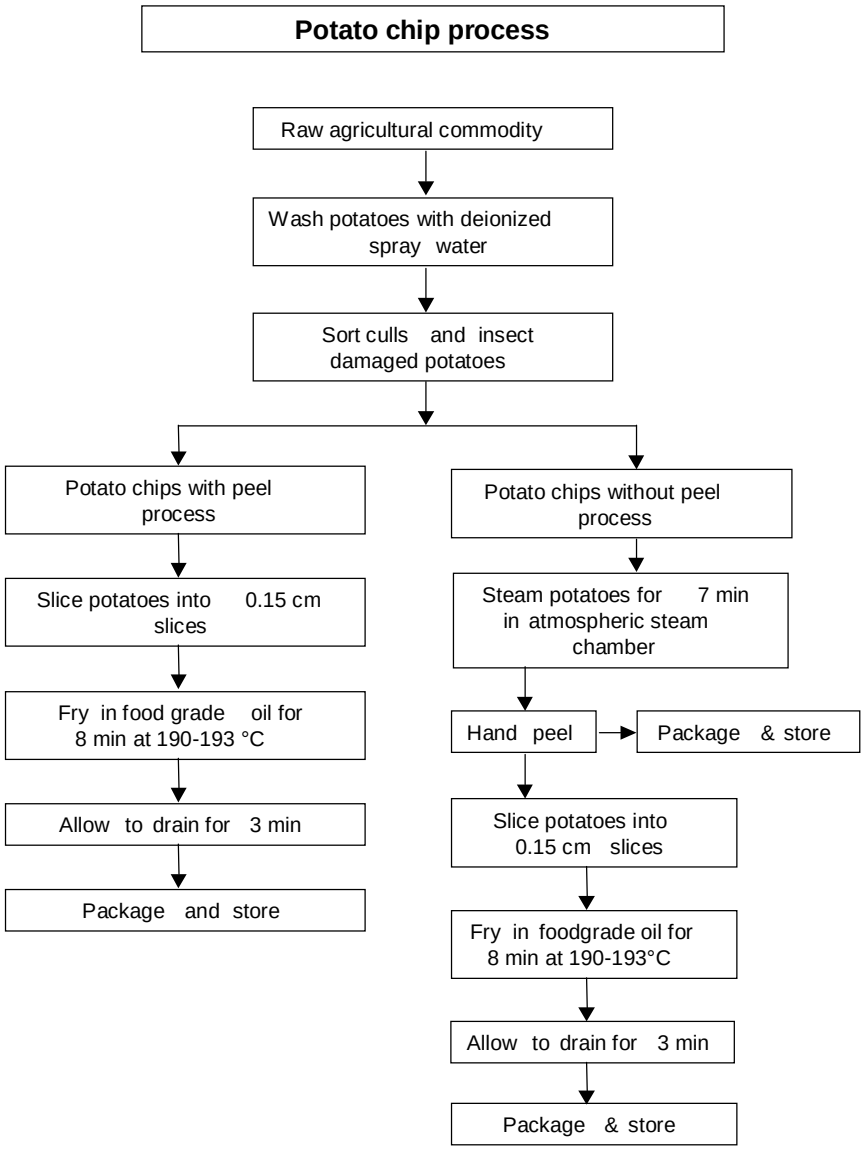


Figure 3. Processing of potatoes to chips at a pilot plant (Swanson *et al.*, 1993).

Table 34. Residues of chlorpropham and 3-chloroaniline in potato chips (Haws *et al.*, 1993c).

Treatment	Bin no. Location in pile	Days after first treatment	Chlorpropham residues (mg/kg)		3-Chloroaniline residues (mg/kg)	
			With skin	Without skin	With skin	Without skin
Aerosol fogging	4, bottom A	5	3.4	<0.45	<0.45	<0.45
at 15-11-91	4, bottom B	5	2.7	<0.45	<0.45	<0.45
0.03 kg ai/t	4, middle A	5	1.3	<0.45	<0.45	<0.45
potatoes	4, middle B	5	1.4	<0.45	<0.45	<0.45
	4, top A	5	0.72	<0.45	<0.45	<0.45
	4, top B	5	0.7	<0.45	<0.45	<0.45
sampling at	5, bottom A	5	3.6	<0.45	<0.45	<0.45

Treatment	Bin no. Location in pile	Days after first treatment	Chlorpropham residues (mg/kg)		3-Chloroaniline residues (mg/kg)	
			With skin	Without skin	With skin	Without skin
19-11-91	5, bottom B	5	3.3	<0.45	<0.45	<0.45
	5, middle A	5	1.5	<0.45	<0.45	<0.45
	5, middle B	5	1.6	<0.45	<0.45	<0.45
	5, top A	5	1.8	<0.45	<0.45	<0.45
	5, top B	5	1.7	<0.45	<0.45	<0.45
Aerosol fogging	4, bottom A	91	5.7	<0.45	<0.45	<0.45
at 15-11-91	4, bottom B	91	6.4	<0.45	<0.45	<0.45
0.03 kg ai/t	4, middle A	91	2.8	<0.45	<0.45	<0.45
potatoes	4, middle B	91	3.7	<0.45	<0.45	<0.45
	4, top A	91	2.9	<0.45	<0.45	<0.45
	4, top B	91	2.3	<0.45	<0.45	<0.45
sampling at	5, bottom A	91	4.0	<0.45	<0.45	<0.45
13-02-92	5, bottom B	91	5.1	<0.45	<0.45	<0.45
	5, middle A	91	4.7	<0.45	<0.45	<0.45
	5, middle B	91	5.0	<0.45	<0.45	<0.45
	5, top A	91	4.6	<0.45	<0.45	<0.45
	5, top B	91	4.9	<0.45	<0.45	<0.45
Aerosol fogging	4, bottom A	140	4.5	<0.45	<0.45	<0.45
at 15-11-91	4, bottom B	140	3.7	<0.45	<0.45	<0.45
0.03 kg ai/t	4, middle A	140	1.2	<0.45	<0.45	<0.45
potatoes	4, middle B	140	2.0	<0.45	<0.45	<0.45
	4, top A	140	2.4	<0.45	<0.45	<0.45
	4, top B	140	2.6	<0.45	<0.45	<0.45
sampling at	5, bottom A	140	3.8	<0.45	<0.45	<0.45
02-04-92	5, bottom B	140	4.0	<0.45	<0.45	<0.45
	5, middle A	140	3.8	<0.45	<0.45	<0.45
	5, middle B	140	4.1	<0.45	<0.45	<0.45
	5, top A	140	7.9	<0.45	<0.45	<0.45
	5, top B	140	6.4	<0.45	<0.45	<0.45
Aerosol fogging	4, bottom A	145	4.4	≤0.45	<0.45	<0.45
at 15-11-91	4, bottom B	145	4.2	≤0.45	<0.45	<0.45
0.03 kg ai/t	4, middle A	145	4.0	≤0.45	<0.45	<0.45
potatoes	4, middle B	145	8.1	≤0.45	<0.45	<0.45
	4, top A	145	4.1	≤0.45	<0.45	<0.45
+	4, top B	145	1.5	≤0.45	<0.45	<0.45
0.015 kg ai/t	5, bottom A	145	0.82	≤0.45	<0.45	<0.45
	5, bottom B	145	1.5	≤0.45	<0.45	<0.45
03-04-92	5, middle A	145	1.9	≤0.45	<0.45	<0.45
	5, middle B	145	1.7	≤0.45	<0.45	<0.45
sampling at	5, top A	145	1.2	≤0.45	<0.45	<0.45
07-04-92	5, top B	145	-		<0.45	<0.45
Aerosol fogging	4, bottom A	215	3.8	1.2	<0.45	<0.45
at 15-11-91	4, bottom B	215	5.0	1.4	<0.45	<0.45
0.03 kg ai/t	4, middle A	215	4.6	1.5	<0.45	<0.45
potatoes	4, middle B	215	6.3	1.1	<0.45	<0.45
	4, top A	215	4.6	1.4	<0.45	<0.45
+	4, top B	215	6.4	1.3	<0.45	<0.45
0.015 kg ai/t	5, bottom A	215	7.0	1.5	<0.45	<0.45
potatoes	5, bottom B	215	5.3	1.6	<0.45	<0.45

Treatment	Bin no. Location in pile	Days after first treatment	Chlorpropham residues (mg/kg)		3-Chloroaniline residues (mg/kg)	
			With skin	Without skin	With skin	Without skin
03-04-92	5, middle A	215	6.3	1.8	<0.45	<0.45
	5, middle B	215	7.9	1.4	<0.45	<0.45
sampling at	5, top A	215	4.6	1.5	<0.45	<0.45
16-06-92	5, top B	215	7.1	1.5	<0.45	<0.45

[1] 0.45 mg/kg is method detection limit (MDL), not LOQ

French fries. Commercial processing incorporates sequential water blanching, air-drying and a glucose dip to control colour and solid concentrations in pan-fried French fries. The experimental procedure included a minimal single water blanching to gelatinize starch which results in maximum chlorpropham residues. Figure 4 shows the process at a pilot plant.

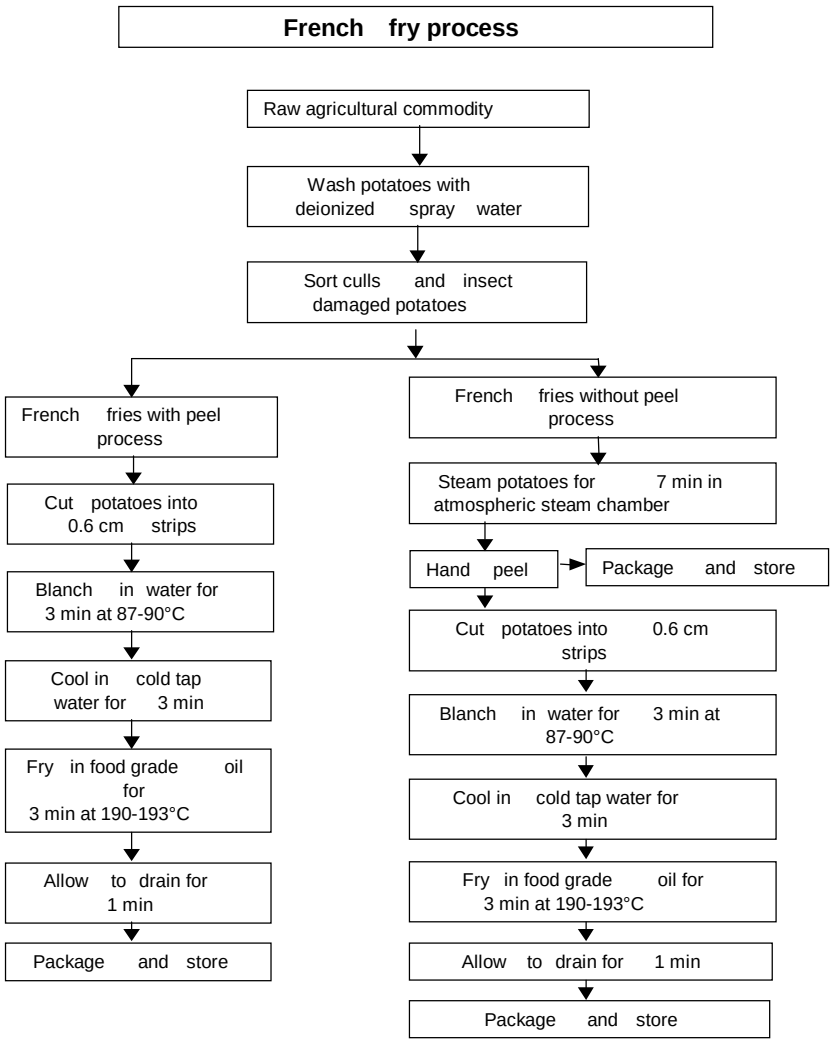


Figure 4. Processing of potatoes to French fries at a pilot plant (Swanson *et al.*, 1993).

Table 35 shows the residues of chlorpropham and 3-chloroaniline in fries with and without skin. Residues of *p*-hydroxy-chlorpropham and *p*-methoxy-chlorpropham were undetectable in any fresh or processed product. Processing factors could not be determined as different samples were used for determination of residues in the raw agriculture commodity (RAC) and the processed product.

Table 35. Residues of chlorpropham and 3-chloroaniline in French fries (Haws *et al.*, 1993c).

Treatment	Bin No.	Days after initial treatment	Chlorpropham residues[1] (mg/kg)		3-Chloroaniline residues[1] (mg/kg)	
	Location in pile		with skin	without skin	with skin	without skin
Aerosol fogging	2, bottom A	5	0.47	<0.2	<0.2	<0.2
at 15-11-91	2, bottom B	5	0.56	<0.2	<0.2	<0.2
0.02 kg ai/t	2, middle A	5	0.26	<0.2	<0.2	<0.2
potatoes	2, middle B	5	0.31	<0.2	<0.2	<0.2
	2, top A	5	<0.2	<0.2	<0.2	<0.2
	2, top B	5	<0.2	<0.2	<0.2	<0.2
sampling at	3, bottom A	5	0.41	<0.2	<0.2	<0.2
19-11-91	3, bottom B	5	0.49	<0.2	<0.2	<0.2
	3, middle A	5	0.39	<0.2	<0.2	<0.2
	3, middle B	5	0.42	<0.2	<0.2	<0.2
	3, top A	5	0.34	<0.2	<0.2	<0.2
	3, top B	5	0.37	<0.2	<0.2	<0.2
Aerosol fogging	2, bottom A	91	1.3	<0.2	<0.2	<0.2
at 15-11-91	2, bottom B	91	1.5	<0.2	<0.2	<0.2
0.02 kg ai/t	2, middle A	91	1.6	<0.2	<0.2	<0.2
potatoes	2, middle B	91	1.1	<0.2	<0.2	<0.2
	2, top A	91	0.73	<0.2	<0.2	<0.2
	2, top B	91	0.78	<0.2	<0.2	<0.2
sampling at	3, bottom A	91	1.1	<0.2	<0.2	<0.2
13-02-92	3, bottom B	91	1.4	<0.2	<0.2	<0.2
	3, middle A	91	2.0	<0.2	0.23	<0.2
	3, middle B	91	2.0	<0.2	0.23	<0.2
	3, top A	91	1.2	<0.2	<0.2	<0.2
	3, top B	91	1.5	<0.2	<0.2	<0.2
Aerosol fogging	2, bottom A	96	1.9	<0.2	<0.2	<0.2
at 15-11-91	2, bottom B	96	2.2	<0.2	<0.2	<0.2
0.02 kg ai/t	2, middle A	96	2.0	<0.2	<0.2	<0.2
potatoes	2, middle B	96	2.3	<0.2	<0.2	<0.2
	2, top A	96	1.4	<0.2	<0.2	<0.2
+	2, top B	96	1.4	<0.2	<0.2	<0.2
0.02 kg ai/t	3, bottom A	96	2.6	<0.2	<0.2	<0.2
potatoes	3, bottom B	96	2.7	<0.2	<0.2	<0.2
14-02-92	3, middle A	96	2.0	<0.2	<0.2	<0.2
	3, middle B	96	2.0	<0.2	<0.2	<0.2
sampling at	3, top A	96	1.3	<0.2	<0.2	<0.2
18-02-92	3, top B	96	1.6	<0.2	<0.2	<0.2
Aerosol fogging	2, bottom A	140	1.5	0.41	<0.2	<0.2
at 15-11-91	2, bottom B	140	0.97	0.54	<0.2	<0.2
0.02 kg ai/t	2, middle A	140	2.8	0.37	<0.2	<0.2
potatoes	2, middle B	140	4.0	0.31	<0.2	<0.2
	2, top A	140	1.1	0.37	<0.2	<0.2
+	2, top B	140	1.4	0.34	<0.2	<0.2
0.02 kg ai/t	3, bottom A	140	2.1	0.28	<0.2	<0.2
potatoes	3, bottom B	140	2.6	0.29	<0.2	<0.2
14-02-92	3, middle A	140	2.2	0.32	<0.2	<0.2
	3, middle B	140	2.2	0.40	<0.2	<0.2
sampling at	3, top A	140	1.3	0.36	<0.2	<0.2

Treatment	Bin No.	Days after initial treatment	Chlorpropham residues[1] (mg/kg)		3-Chloroaniline residues[1] (mg/kg)	
	Location in pile		with skin	without skin	with skin	without skin
07-04-92	3, top B	140	2.1	0.33	<0.2	<0.2
Aerosol fogging	2, bottom A	215	1.4	≤0.2	<0.2	<0.2
at 15-11-91	2, bottom B	215	1.6	<0.2	<0.2	<0.2
0.02 kg ai/t	2, middle A	215	1.6	≤0.2	<0.2	<0.2
potatoes	2, middle B	215	1.4	<0.2	<0.2	<0.2
	2, top A	215	1.7	≤0.2	<0.2	<0.2
+	2, top B	215	1.6	<0.2	<0.2	<0.2
0.02 kg ai/t	3, bottom A	215	1.6	0.34	<0.2	<0.2
potatoes	3, bottom B	215	1.5	0.35	<0.2	<0.2
14-02-92	3, middle A	215	1.5	≤0.2	<0.2	<0.2
	3, middle B	215	2.3	0.23	<0.2	<0.2
sampling at	3, top A	215	1.2	0.28	<0.2	<0.2
16-06-92	3, top B	215	1.2	<0.2	<0.2	<0.2

[1] 0.2 mg/kg is method detection limit (MDL), not LOQ

Peels and granules. Peeling was very similar to commercial practice. Steamed potatoes were peeled by hand in the experimental process because of the small sample size. Figure 5 shows the production of peel at a pilot plant.

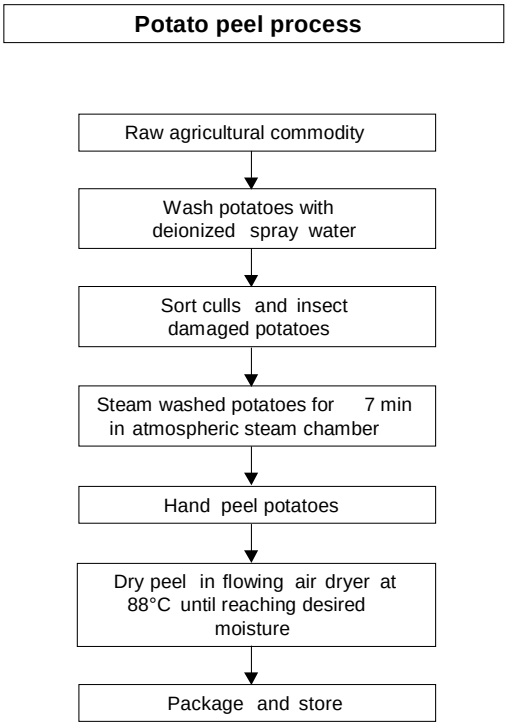


Figure 5. Potato peeling process at a pilot plant (Swanson *et al.*, 1993).

The commercial granule drying process was closely simulated in the experimental procedure. Figures 6a and 6b show granule production at a pilot plant.

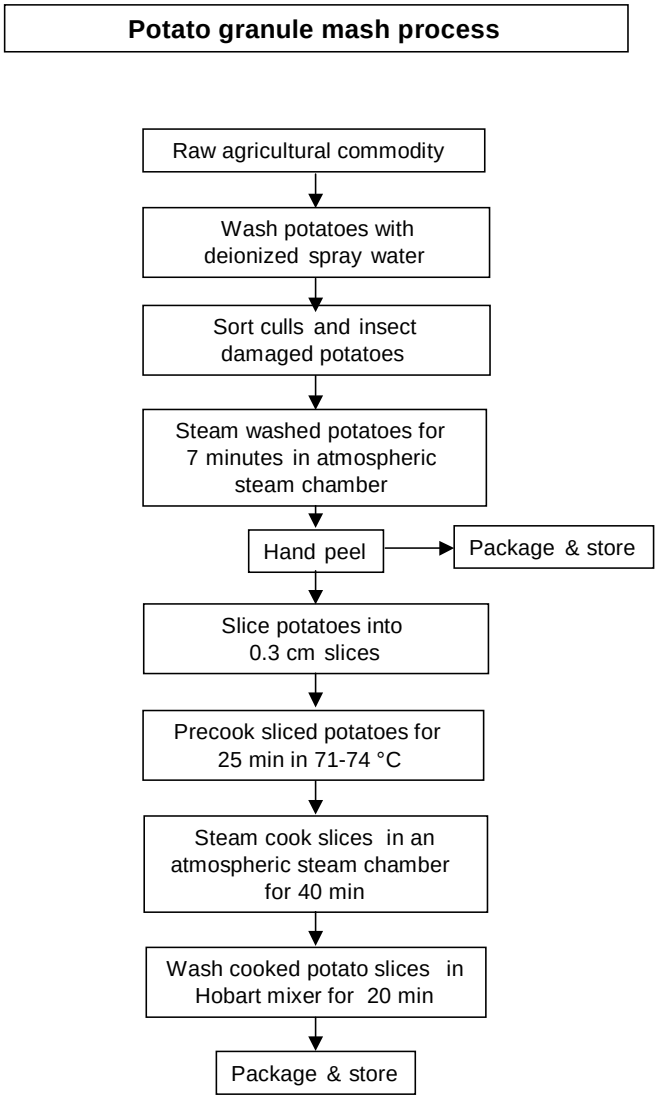


Figure 6a. Potato granule mash production at a pilot plant (Swanson *et al.*, 1993).

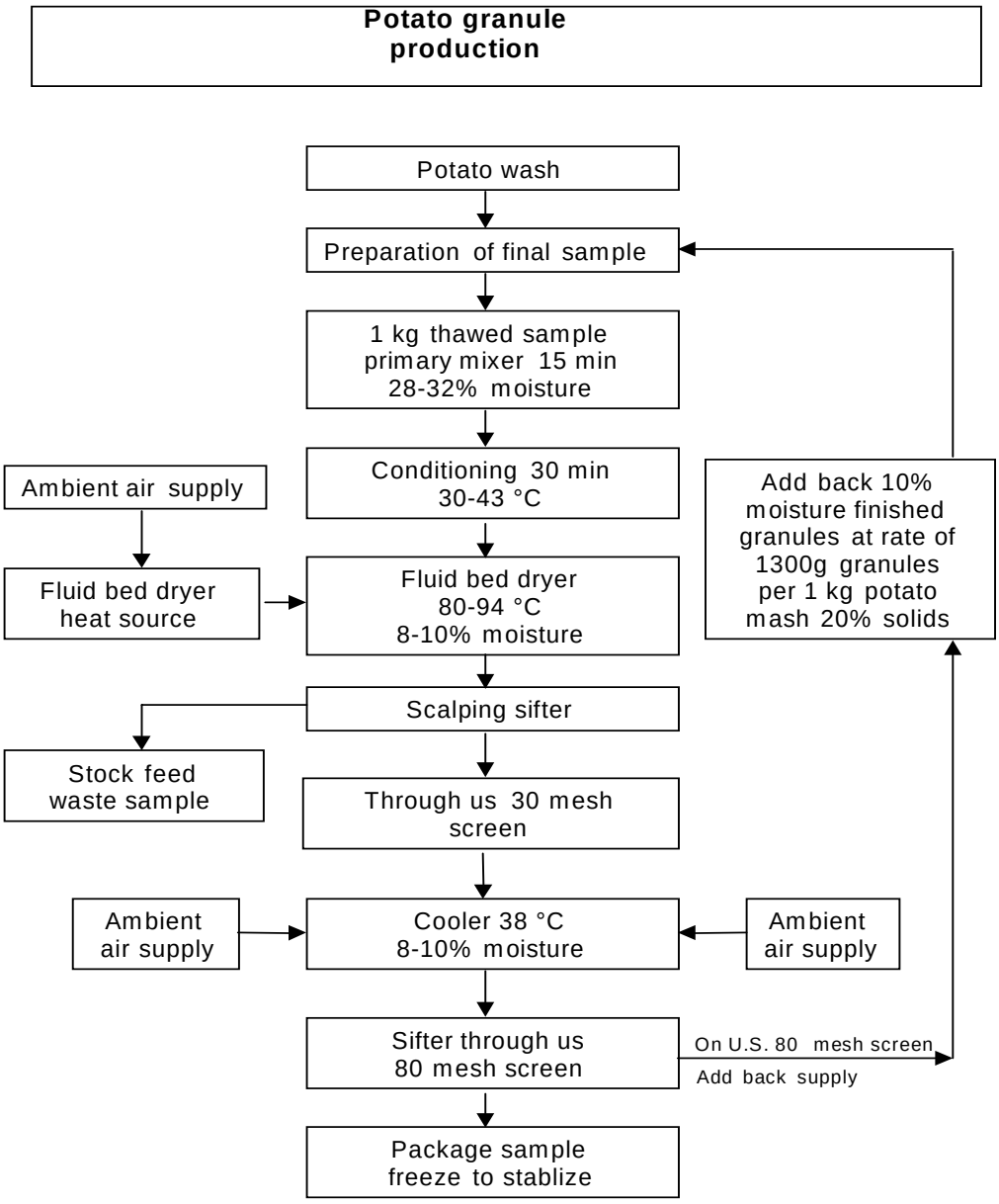


Figure 6b. Potato processing to dry granules at a pilot plant (Swanson *et al.*, 1993).

Table 36 shows the residues of chlorpropham and 3-chloroaniline in dried and wet potato peel as well as in dehydrated granules. Residues of *p*-hydroxy-chlorpropham and *p*-methoxy-chlorpropham were undetectable in any fresh or processed product. Processing factors could not be determined as different samples were used for determination of residues in the raw agriculture commodity (RAC) and the processed product.

Table 36. Residues of chlorpropham and 3-chloroaniline in potato peel and granules (Haws *et al.*, 1993c).

Treatment	Bin no., location in pile	Days after initial treatment	Chlorpropham residues[1] (mg/kg)			3-Chloroaniline residues[1] (mg/kg)		
			Dried peel	Wet peel	Dehydr. granules	Dried peel	Wet peel	Dehydr. granules
Aerosol fogging	2, bottom A	5	89	11	<0.38	0.69	0.12	<0.38
at 15-11-91	2, bottom B	5	61	12	<0.38	<0.38	0.18	<0.38
0.02 kg ai/t	2, middle A	5	38	8.8	<0.38	<0.38	0.11	<0.38
potatoes	2, middle B	5	32	9.0	<0.38	<0.38	0.08	<0.38
	2, top A	5	29	7.0	<0.38	<0.38	<0.08	<0.38
	2, top B	5	33	7.2	<0.38	<0.38	<0.08	<0.38
sampling at	3, bottom A	5	59	14	<0.38	2.7	0.18	<0.38
19-11-91	3, bottom B	5	60	13	<0.38	0.67	0.15	<0.38
	3, middle A	5	67	13	<0.38	0.50	1.16	<0.38
	3, middle B	5	76	9.7	<0.38	1.0	0.14	<0.38
	3, top A	5	60	10	<0.38	2.2	0.11	<0.38
	3, top B	5	42	10	<0.38	<0.38	0.12	<0.38
Aerosol fogging	2, bottom A	91	26	7.3	0.77	0.48	0.08	<0.38
at 15-11-91	2, bottom B	91	40	10	0.78	0.61	0.14	<0.38
0.02 kg ai/t	2, middle A	91	52	9.2	0.47	0.57	0.08	<0.38
potatoes	2, middle B	91	30	7.2	0.50	0.40	<0.08	<0.38
	2, top A	91	45	3.7	0.83	0.55	<0.08	<0.38
	2, top B	91	20	3.4	0.84	0.42	<0.08	<0.38
sampling at	3, bottom A	91	47	9.9	0.78	0.84	0.16	<0.38
13-02-92	3, bottom B	91	44	9.7	0.78	0.78	0.18	<0.38
	3, middle A	91	35	8.8	0.79	0.50	0.12	<0.38
	3, middle B	91	43	9.8	0.78	0.58	0.12	<0.38
	3, top A	91	56	11	0.76	0.68	0.13	<0.38
	3, top B	91	51	10	0.72	0.60	0.16	<0.38
Aerosol fogging	2, bottom A	96	81	31	1.2	1.6	0.31	<0.38
at 15-11-91	2, bottom B	96	76	33	1.0	1.8	0.28	<0.38
0.02 kg ai/t	2, middle A	96	65	17	<0.38	1.0	0.13	<0.38
potatoes	2, middle B	96	70	14	<0.38	1.3	0.15	<0.38
	2, top A	96	48	19	0.75	0.64	0.18	<0.38
+	2, top B	96	44	14	0.65	<0.38	0.15	<0.38
0.02 kg ai/t	3, bottom A	96	145	34	0.69	3.5	0.25	<0.38
potatoes	3, bottom B	96	90	34	0.57	1.6	0.39	<0.38
14-02-92	3, middle A	96	81	32	0.64	1.1	0.29	<0.38
	3, middle B	96	106	30	0.71	1.3	0.23	<0.38
sampling at	3, top A	96	93	33	0.41	1.1	0.24	<0.38
18-02-92	3, top B	96	102	26	<0.38	1.0	0.11	<0.38
Aerosol fogging	2, bottom A	140	60	35	0.67	0.38	0.36	<0.38
at 15-11-91	2, bottom B	140	57	26	0.81	0.38	0.23	<0.38
0.02 kg ai/t	2, middle A	140	51	26	0.63	0.38	0.23	<0.38
potatoes	2, middle B	140	41	31	0.87	0.38	0.26	<0.38
	2, top A	140	44	17	0.75	0.38	0.18	<0.38
+	2, top B	140	30	21	0.76	0.38	0.22	<0.38
0.02 kg ai/t	3, bottom A	140	61	45	0.75	0.38	0.34	<0.38
potatoes	3, bottom B	140	63	41	0.87	0.38	0.32	<0.38
14-02-92	3, middle A	140	67	35	0.95	0.38	0.25	<0.38

Treatment	Bin no., location in pile	Days after initial treatment	Chlorpropham residues[1] (mg/kg)			3-Chloroaniline residues[1] (mg/kg)		
			Dried peel	Wet peel	Dehydr. granules	Dried peel	Wet peel	Dehydr. granules
	3, middle B	140	78	42	0.96	0.53	0.27	<0.38
sampling at	3, top A	140	71	43	0.69	0.48	0.30	<0.38
07-04-92	3, top B	140	77	31	0.82	0.53	0.28	<0.38
Aerosol fogging	2, bottom A	215	56	11	1.2	1.0	0.21	<0.38
at 15-11-91	2, bottom B	215	59	12	0.91	0.93	0.2	<0.38
0.02 kg ai/t	2, middle A	215	53	14	1.3	1.1	0.18	<0.38
potatoes	2, middle B	215	57	14	1.4	1.1	0.16	<0.38
	2, top A	215	47	15	1.1	0.92	0.21	<0.38
+	2, top B	215	57	15	1.2	1.2	0.22	<0.38
0.02 kg ai/t	3, bottom A	215	26	14	1.5	1.4	0.22	<0.38
potatoes	3, bottom B	215	25	13	1.5	1.5	0.19	<0.38
14-02-92	3, middle A	215	24	15	1.9	1.1	0.19	<0.38
	3, middle B	215	25	10	2.1	1.1	0.21	<0.38
sampling at	3, top A	215	27	17	1.6	1.6	0.24	<0.38
16-06-92	3, top B	215	25	17	1.5	1.4	0.23	<0.38

[1] 0.38 mg/kg is method detection limit (MDL) for granules and dried peel; 0.08 mg/kg is MDL for wet peel

Canola oil. Samples of oil used in frying French fries and chips, with and without skins, were taken before and after each frying. The samples were delivered to the analytical laboratory and stored frozen. The oil did not show a residue above the method detection limit (MDL) of 2.9 mg/kg each for chlorpropham, 3-chloroaniline, *p*-hydroxy-chlorpropham and *p*-methoxy-chlorpropham.

Residues in the edible portion of food commodities

Potatoes (Kleinkopf and Thomson, 1992; Goodrick *et al*., 1993b-d). In a study on mature potato tubers stored in bins under commercial conditions (see Table 25 above) whole tubers (Kleinkopf and Thomson, 1992) were processed into pulp and peel. The results from the three types of treatment are reported by Goodrick *et al.* (1993b-d) in reports 92CIPC04, 92CIPC05 and 92CIPC06. The chlorpropham and 3-chloroaniline residues in whole potatoes, pulp and peel, and processing factors calculated for 3-chloroaniline only if residues in the raw agriculture commodity were higher than the MDL, are shown in Tables 37 and 38.

Table 37. Residues of chlorpropham in the edible portions of potatoes (Goodrick *et al*., 1993b-d).

Treatment, Report No.	Bin no. Location in pile	Days after initial treatment	Chlorpropham residues[1] (mg/kg)				
			Whole	Pulp	Process factor (pulp)	Peel	Process factor (peel)
Aerosol fogging	2, bottom A	5	9.5	0.22	0.023	31	3.3
at 15-11-91	2, bottom B	5	11	0.22	0.02	35	3.2
0.02 kg ai/t	2, middle A	5	7.2	0.23	0.031	28	3.9
potatoes	2, middle B	5	7.6	0.23	0.03	32	4.2
	2, top A	5	6.9	0.25	0.036	33	4.8
+	2, top B	5	7.0	0.26	0.037	34	4.8
EC direct spray	3, bottom A	5	7.5	<0.08	0.011	37	4.9
at 19-11-91	3, bottom B	5	7.2	<0.08	0.011	32	4.4
0.01 kg ai/t	3, middle A	5	5.0	<0.08	0.016	41	8.2
potatoes	3, middle B	5	4.8	<0.08	0.017	43	9
	3, top A	5	9.1	0.12	0.013	46	5.1

Treatment, Report No.	Bin no. Location in pile	Days after initial treatment	Chlorpropham residues[1] (mg/kg)				
			Whole	Pulp	Process factor (pulp)	Peel	Process factor (peel)
Rep. 92CIPC04	3, top B	5	8.0	0.17	0.021	40	5
Aerosol fogging	2, bottom A	91	8.9	0.14	0.016	37	4.2
at 15-11-91	2, bottom B	91	8.6	0.15	0.017	36	4.2
0.02 kg ai/t	2, middle A	91	9.3	0.15	0.016	47	5.1
potatoes	2, middle B	91	8.3	0.14	0.017	55	6.6
	2, top A	91	6.5	0.17	0.026	30	4.6
+	2, top B	91	7.3	0.15	0.021	32	4.4
EC direct spray	3, bottom A	91	7.6	0.18	0.024	52	6.8
at 13-02-92	3, bottom B	91	9.4	0.18	0.019	64	6.8
0.01 kg ai/t	3, middle A	91	6.1	0.12	0.02	60	9.8
potatoes	3, middle B	91	6.3	-	-	65	10.3
	3, top A	91	9.1	0.17	0.019	51	5.6
Rep. 92CIPC04	3, top B	91	9.0	-	-	59	6.6
Aerosol fogging	2, bottom A	96	8.3	0.12	0.014	95	11
at 15-11-91	2, bottom B	96	9.7	0.13	0.013	77	8
0.02 kg ai/t	2, middle A	96	8.5	0.09	0.011	65	7.6
potatoes	2, middle B	96	7.0	0.09	0.013	55	7.9
+	2, top A	96	6.0	<0.08	0.013	52	8.7
0.02 kg ai/t	2, top B	96	7.2	<0.08	0.011	50	6.9
potatoes	3, bottom A	96	12	0.13	0.011	68	5.7
14-02-92	3, bottom B	96	14	0.14	0.01	83	5.9
+	3, middle A	96	9.3	0.12	0.013	60	6.4
EC direct spray	3, middle B	96	8.8	0.18	0.02	62	7.1
at 18-02-92	3, top A	96	10	0.13	0.013	49	4.9
0.01 kg ai/t Rep. 92CIPC04	3, top B	96	9.5	0.15	0.016	58	6.1
Aerosol fogging	2, bottom A	140	11	0.15	0.014	14	1.3
at 15-11-91	2, bottom B	140	11	0.18	0.016	25	2.3
0.02 kg ai/t	2, middle A	140	8.4	0.12	0.014	15	1.8
potatoes	2, middle B	140	8.9	0.16	0.018	23	2.6
+	2, top A	140	9.0	0.14	0.016	20	2.2
0.02 kg ai/t	2, top B	140	7.5	0.13	0.017	18	2.4
potatoes	3, bottom A	140	13	0.42	0.032	19	1.5
14-02-92	3, bottom B	140	12	0.13	0.011	19	1.6
+	3, middle A	140	12	0.71	0.059	27	2.2
EC direct spray	3, middle B	140	9.4	0.73	0.078	18	1.9
at 02-04-92	3, top A	140	13	0.42	0.032	18	1.4
0.01 kg ai/t Rep. 92CIPC04	3, top B	140	12	0.34	0.041	-	-
Aerosol fogging	2, bottom A	215	8.2	0.39	0.048	30	3.6
at 15-11-91	2, bottom B	215	7.7	0.32	0.042	28	3.6
0.02 kg ai/t	2, middle A	215	7.5	0.24	0.032	30	4
potatoes	2, middle B	215	8.0	0.30	0.038	39	4.9
+	2, top A	215	7.6	0.28	0.037	28	3.7
0.02 kg ai/t	2, top B	215	7.1	0.33	0.046	32	4.5
14-02-92	3, bottom A	215	11	0.36	0.033	43	3.9
+	3, bottom B	215	9.8	0.30	0.031	48	4.9
EC direct spray	3, middle A	215	7.6	0.45	0.059	38	5
at 16-06-92	3, middle B	215	8.3	0.40	0.048	31	3.7

Treatment, Report No.	Bin no. Location in pile	Days after initial treatment	Chlorpropham residues[1] (mg/kg)				
			Whole	Pulp	Process factor (pulp)	Peel	Process factor (peel)
0.01 kg ai/t	3, top A	215	7.4	0.42	0.057	51	6.9
Rep. 92CIPC04	3, top B	215	8.2	0.44	0.054	54	6.6
Mean processing factor				pulp (n = 58)	0.03	peel (n-59)	**5.1**
Median processing factor				pulp (n = 58)	0.0195	peel (n=59)	**4.9**
Aerosol fogging	2, bottom A	5	8.7	<0.08	0.009	34	3.9
at 15-11-91	2, bottom B	5	7.3	-	-	40	5.5
0.02 kg ai/t	2, middle A	5	4.2	<0.08	0.019	16	3.8
potatoes	2, middle B	5	3.2	<0.08	0.025	21	6.6
	2, top A	5	3.6	<0.08	0.022	17	4.7
	2, top B	5	3.2	-	-	19	5.9
Rep. 92CIPC05	3, bottom A	5	7.8	-	-	47	6.0
	3, bottom B	5	8.9	0.11	0.012	62	7.0
	3, middle A	5	7.3	-	-	42	5.8
	3, middle B	5	6.1	0.12	0.02	33	5.4
	3, top A	5	6.7	-	-	33	4.9
	3, top B	5	6.3	0.13	0.021	40	6.3
Aerosol fogging	2, bottom A	91	2.8	<0.08	0.029	39	13.9
at 15-11-91	2, bottom B	91	4.1	<0.08	0.02	46	11.2
0.02 kg ai/t	2, middle A	91	3.6	<0.08	0.022	34	9.4
potatoes	2, middle B	91	2.6	<0.08	0.031	29	11.2
	2, top A	91	1.4	<0.08	0.057	25	17.9
	2, top B	91	2.0	<0.08	0.04	23	11.5
Rep. 92CIPC05	3, bottom A	91	5.2	-	-	47	9.0
	3, bottom B	91	4.5	<0.08	0.018	37	8.2
	3, middle A	91	4.3	<0.08	0.019	67	15.6
	3, middle B	91	6.1	<0.08	0.013	37	6.1
	3, top A	91	7.1	<0.08	0.011	43	6.1
	3, top B	91	5.1	<0.08	0.016	39	7.6
Aerosol fogging	2, bottom A	96	9.8	0.11	0.011	-	-
at 15-11-91	2, bottom B	96	9.9	0.16	0.016	78	8.0
0.02 kg ai/t	2, middle A	96	6.9	<0.08	0.012	37	5.4
potatoes	2, middle B	96	6.4	0.09	0.014	32	5.0
	2, top A	96	6.0	0.09	0.015	35	5.8
+	2, top B	96	4.5	<0.08	0.018	26	5.8
0.02 kg ai/t	3, bottom A	96	16	-	-	74	4.6
potatoes	3, bottom B	96	10	-	-	61	6.1
14-02-92	3, middle A	96	9.4	-	-	64	6.8
	3, middle B	96	9.5	<0.08	0.008	51	5.4
	3, top A	96	12	<0.08	0.007	58	4.8
Rep. 92CIPC05	3, top B	96	11	<0.08	0.007	52	4.7
Aerosol fogging	2, bottom A	140	7.1	<0.08	0.011	51	7.2
at 15-11-91	2, bottom B	140	9.7	<0.08	0.008	48	5.0
0.02 kg ai/t	2, middle A	140	8.1	<0.08	0.01	35	4.3
potatoes	2, middle B	140	9.2	-	-	38	4.2
	2, top A	140	4.7	0.15	0.032	36	7.7
+	2, top B	140	4.4	<0.08	0.018	23	5.2
0.02 kg ai/t	3, bottom A	140	11	0.23	0.021	52	4.7
potatoes	3, bottom B	140	18	0.24	0.013	45	2.4
14-02-92	3, middle A	140	12	0.25	0.021	40	3.3

Treatment, Report No.	Bin no. Location in pile	Days after initial treatment	Chlorpropham residues[1] (mg/kg)				
			Whole	Pulp	Process factor (pulp)	Peel	Process factor (peel)
	3, middle B	140	11	0.25	0.023	37	3.4
Rep. 92CIPC05	3, top A	140	6.8	0.25	0.037	52	7.6
	3, top B	140	9.4	0.32	0.034	76	8.1
Aerosol fogging	2, bottom A	215	7.8	0.53	0.068	40	5.1
at 15-11-91	2, bottom B	215	7.0	0.70	0.1	82	11.7
0.02 kg ai/t	2, middle A	215	6.6	0.53	0.08	32	4.8
potatoes	2, middle B	215	6.8	0.38	0.056	38	5.6
	2, top A	215	6.8	0.28	0.041	23	3.4
+	2, top B	215	5.4	0.35	0.065	27	5.0
0.02 kg ai/t	3, bottom A	215	8.2	0.41	0.05	43	5.2
potatoes	3, bottom B	215	8.8	0.61	0.069	-	-
14-02-92	3, middle A	215	11	0.49	0.045	53	4.8
	3, middle B	215	8.2	0.75	0.091	49	6.0
Rep. 92CIPC05	3, top A	215	7.8	0.52	0.067	58	7.4
	3, top B	215	8.2	0.73	0.089	50	6.1
Mean processing factor (n = 50)				pulp (n = 50)	0.03	peel (n=58)	**6.6**
Median processing factor (n = 50)				pulp (n = 50)	0.021	peel (n=58)	**5.8**
Aerosol fogging	4, bottom A	5	23	0.14	0.006	101	4.4
at 15-11-91	4, bottom B	5	21	0.12	0.006	152	7.2
0.03 kg ai/t	4, middle A	5	7.6	-	-	56	7.4
potatoes	4, middle B	5	6.4	<0.08	0.012	43	6.7
	4, top A	5	4.7	<0.08	0.017	39	8.3
	4, top B	5	4.7	<0.08	0.017	31	6.6
sampling at	5, bottom A	5	16	0.14	0.009	95	5.9
19-11-91	5, bottom B	5	13	<0.08	0.006	75	5.8
	5, middle A	5	11	<0.08	0.007	63	5.7
Rep. 92CIPC06	5, middle B	5	13	<0.08	0.006	67	5.2
	5, top A	5	13	0.16	0.012	85	6.5
	5, top B	5	10	<0.08	0.008	68	6.8
Aerosol fogging	4, bottom A	91	7.8	0.22	0.028	61	7.8
at 15-11-91	4, bottom B	91	11	0.16	0.014	64	5.8
0.03 kg ai/t	4, middle A	91	7.0	-	-	45	6.4
potatoes	4, middle B	91	11	0.16	0.014	48	4.4
	4, top A	91	5.4	0.22	0.041	28	5.2
sampling at	4, top B	91	3.2	<0.08	0.025	18	5.6
13-02-92	5, bottom A	91	7.0	0.23	0.033	60	8.6
	5, bottom B	91	7.8	0.26	0.033	52	6.7
Rep. 92CIPC06	5, middle A	91	5.6	0.24	0.043	52	9.3
	5, middle B	91	14	0.35	0.025	62	4.4
	5, top A	91	9.2	0.30	0.033	63	6.8
	5, top B	91	7.4	0.26	0.035	53	7.2
Aerosol fogging	4, bottom A	140	8.6	0.53	0.062	36	4.2
at 15-11-91	4, bottom B	140	8.2	0.54	0.066	60	7.3
0.03 kg ai/t	4, middle A	140	10	0.62	0.062	44	4.4
potatoes	4, middle B	140	12	0.52	0.043	57	4.8
	4, top A	140	7.1	0.35	0.049	45	6.3
	4, top B	140	5.9	0.28	0.047	46	7.8
sampling at	5, bottom A	140	13	0.49	0.038	58	4.5
02-04-92	5, bottom B	140	13	0.47	0.036	55	4.2

Treatment, Report No.	Bin no. Location in pile	Days after initial treatment	Chlorpropham residues[1] (mg/kg)				
			Whole	Pulp	Process factor (pulp)	Peel	Process factor (peel)
	5, middle A	140	10	0.57	0.057	58	5.8
Rep. 92CIPC06	5, middle B	140	12	0.55	0.046	78	6.5
	5, top A	140	9.8	0.64	0.065	58	5.9
	5, top B	140	11	0.54	0.049	90	8.2
Aerosol fogging	4, bottom A	145	13	0.52	0.04	90	6.9
at 15-11-91	4, bottom B	145	12	0.35	0.029	71	5.9
0.03 kg ai/t	4, middle A	145	12	0.57	0.048	81	6.8
potatoes	4, middle B	145	14	0.42	0.03	58	4.1
+	4, top A	145	5.5	0.35	0.064	53	9.6
0.015 kg ai/t	4, top B	145	6.0	0.30	0.05	51	8.5
potatoes	5, bottom A	145	14	0.55	0.039	69	4.9
03-04-92	5, bottom B	145	14	0.39	0.028	72	5.1
sampling at	5, middle A	145	12	0.54	0.045	74	6.2
07-04-92	5, middle B	145	16	0.52	0.032	73	4.6
	5, top A	145	10	0.48	0.048	50	0.5
Rep. 92CIPC06	5, top B	145	12	0.42	0.035	98	8.2
Aerosol fogging	4, bottom A	215	7.8	1.0	0.13	61	7.8
at 15-11-91	4, bottom B	215	8.2	1.2	0.15	73	8.9
0.03 kg ai/t	4, middle A	215	8.0	1.2	0.15	56	7
potatoes	4, middle B	215	7.8	1.1	0.14	46	5.9
+	4, top A	215	6.7	0.99	0.15	49	7.3
0.015 kg ai/t	4, top B	215	8.1	1.0	0.12	54	6.7
potatoes	5, bottom A	215	8.9	1.0	0.11	66	7.5
03-04-92	5, bottom B	215	10	1.0	0.1	61	6.1
sampling at	5, middle A	215	11	1.4	0.13	53	4.8
16-06-92	5, middle B	215	15	1.1	0.073	77	5.1
	5, top A	215	11(1)	1.3	0.12	74	6.7
Rep. 92CIPC06	5, top B	215	9.5	1.3	0.14	75	7.9
Mean processing factor				pulp (n = 58)	0.05	peel (n=59)	**6.3**
Median processing factor				pulp (n = 58)	0.041	peel (n=59)	**6.4**
Overall mean processing factor				pulp (n = 166)	0.037	peel (n=177)	**6.0**
Overall median processing factor				pulp (n = 166)	0.027	peel (n=177)	**5.8**

[1] 0.08 mg/kg is method detection limit (MDL) for pulp, not LOQ.

Table 38. Residues of 3-chloroaniline in edible portions of potatoes (Goodrick *et al.*, 1993b-d).

Treatment, Report No.	Bin no., location in pile	Days after initial treatment	3-Chloroaniline residues[1] (mg/kg)			
			Whole	Pulp	Peel	Processing factor (peel)
Aerosol fogging	2, bottom A	5	<0.08	<0.08	<0.08	-
at 15-11-91	2, bottom B	5	<0.08	<0.08	<0.08	-
0.02 kg ai/t	2, middle A	5	<0.08	<0.08	<0.08	-
potatoes	2, middle B	5	<0.08	<0.08	<0.08	-
	2, top A	5	<0.08	<0.08	<0.08	-
+	2, top B	5	<0.08	<0.08	<0.08	-
EC direct spray	3, bottom A	5	0.09	<0.08	<0.08	-
at 19-11-91	3, bottom B	5	0.08	<0.08	0.08	-
0.01 kg ai/t	3, middle A	5	<0.08	<0.08	<0.08	-

Treatment, Report No.	Bin no., location in pile	Days after initial treatment	3-Chloroaniline residues[1] (mg/kg)			
			Whole	Pulp	Peel	Processing factor (peel)
	3, middle B	5	<0.08	<0.08	<0.08	-
	3, top A	5	0.08	<0.08	<0.08	-
Rep. 92CIPC04	3, top B	5	<0.08	<0.08	0.11	-
Aerosol fogging	2, bottom A	91	0.11	<0.08	0.20	1.8
at 15-11-91	2, bottom B	91	0.10	<0.08	0.19	1.9
0.02 kg ai/t	2, middle A	91	0.10	<0.08	0.20	2.0
potatoes	2, middle B	91	0.10	<0.08	0.23	2.3
	2, top A	91	0.09	<0.08	0.21	2.3
+	2, top B	91	0.09	<0.08	0.20	2.2
EC direct spray	3, bottom A	91	0.10	<0.08	0.20	2.0
at 13-02-92	3, bottom B	91	0.11	<0.08	0.21	1.9
0.01 kg ai/t	3, middle A	91	0.10	-	0.20	2.0
	3, middle B	91	0.10	<0.08	0.21	2.1
	3, top A	91	0.10	-	0.23	2.3
Rep. 92CIPC04	3, top B	91	0.12	<0.08	0.20	1.7
Aerosol fogging	2, bottom A	96	0.16	<0.08	0.30	1.9
at 15-11-91	2, bottom B	96	0.14	<0.08	0.23	1.6
0.02 kg ai/t	2, middle A	96	0.12	<0.08	0.26	2.2
potatoes	2, middle B	96	0.12	<0.08	0.22	1.8
+	2, top A	96	<0.08	<0.08	0.20	-
0.02 kg ai/t	2, top B	96	<0.08	<0.08	0.20	-
potatoes	3, bottom A	96	0.13	<0.08	0.21	1.6
14-02-92	3, bottom B	96	0.16	<0.08	0.33	2.1
+	3, middle A	96	0.12	<0.08	0.19	1.6
EC direct spray	3, middle B	96	0.12	<0.08	0.19	1.6
at 18-02-92	3, top A	96	0.13	<0.08	0.21	1.6
0.01 kg ai/t Rep. 92CIPC04	3, top B	96	0.12	<0.08	0.21	1.8
Aerosol fogging	2, bottom A	140	0.12	<0.08	0.25	2.1
at 15-11-91	2, bottom B	140	0.10	<0.08	0.24	2.4
0.02 kg ai/t	2, middle A	140	0.12	<0.08	0.24	2.0
potatoes	2, middle B	140	0.10	<0.08	0.24	2.4
+	2, top A	140	0.11	<0.08	0.25	2.3
0.02 kg ai/t	2, top B	140	0.10	<0.08	0.25	2.5
potatoes	3, bottom A	140	0.12	<0.08	0.29	2.4
14-02-92	3, bottom B	140	0.12	<0.08	0.27	2.2
+	3, middle A	140	0.12	<0.08	0.25	2.1
EC direct spray	3, middle B	140	0.12	<0.08	-	-
at 02-04-92	3, top A	140	0.13	<0.08	0.29	2.2
0.01 kg ai/t Rep. 92CIPC04	3, top B	140	0.12	<0.08	0.29	2.4
Aerosol fogging	2, bottom A	215	0.14	<0.08	0.28	2.0
at 15-11-91	2, bottom B	215	0.12	<0.08	0.32	2.7
0.02 kg ai/t	2, middle A	215	0.12	<0.08	0.25	2.1
potatoes	2, middle B	215	0.14	<0.08	0.26	1.9
+	2, top A	215	0.14	<0.08	0.22	1.6
0.02 kg ai/t	2, top B	215	0.13	<0.08	0.23	1.8
14-02-92	3, bottom A	215	0.14	<0.08	0.33	2.4
+	3, bottom B	215	0.15	<0.08	0.31	2.1
EC direct spray	3, middle A	215	0.13	<0.08	0.24	1.8

Treatment, Report No.	Bin no., location in pile	Days after initial treatment	3-Chloroaniline residues[1] (mg/kg)			
			Whole	Pulp	Peel	Processing factor (peel)
at 16-06-92	3, middle B	215	0.14	<0.08	0.24	1.7
0.01 kg ai/t	3, top A	215	0.12	<0.08	0.29	2.4
Rep. 92CIPC04	3, top B	215	0.13	<0.08	0.29	2.2
Aerosol fogging	2, bottom A	5	<0.08	<0.08	0.22	-
at 15-11-91	2, bottom B	5	<0.08	-	0.21	-
0.02 kg ai/t	2, middle A	5	<0.08	<0.08	0.20	-
potatoes	2, middle B	5	<0.08	<0.08	0.19	-
	2, top A	5	<0.08	<0.08	0.19	-
	2, top B	5	<0.08	-	0.19	-
	3, bottom A	5	0.20	<0.08	0.18	0.90
Rep. 92CIPC05	3, bottom B	5	0.21	<0.08	0.19	0.90
	3, middle A	5	0.20	<0.08	0.17	0.85
	3, middle B	5	0.19	<0.08	0.24	1.3
	3, top A	5	0.19	<0.08	0.20	1.1
	3, top B	5	0.19	<0.08	0.16	0.84
Aerosol fogging	2, bottom A	91	0.13	<0.08	0.16	1.2
at 15-11-91	2, bottom B	91	0.12	<0.08	0.16	1.3
0.02 kg ai/t	2, middle A	91	-	<0.08	0.15	-
potatoes	2, middle B	91	0.12	<0.08	0.15	1.2
	2, top A	91	0.12	<0.08	0.15	1.2
	2, top B	91	0.12	<0.08	0.14	1.2
	3, bottom A	91	0.15	<0.08	0.31	2.1
Rep. 92CIPC05	3, bottom B	91	0.14	<0.08	0.30	2.1
	3, middle A	91	0.15	<0.08	0.32	2.1
	3, middle B	91	0.14	<0.08	0.34	2.4
	3, top A	91	0.17	<0.08	0.31	1.8
	3, top B	91	0.14	<0.08	0.31	2.2
Aerosol fogging	2, bottom A	96	<0.08	<0.08	-	-
at 15-11-91	2, bottom B	96	<0.08	<0.08	0.36	-
0.02 kg ai/t	2, middle A	96	<0.08	<0.08	0.26	-
potatoes	2, middle B	96	<0.08	<0.08	0.24	-
	2, top A	96	<0.08	<0.08	0.27	-
+	2, top B	96	<0.08	<0.08	0.28	-
0.02 kg ai/t	3, bottom A	96	0.18	-	0.34	1.9
potatoes	3, bottom B	96	0.18	-	0.28	1.6
14-02-92	3, middle A	96	0.14	<0.08	0.27	1.9
	3, middle B	96	0.13	<0.08	0.25	1.9
	3, top A	96	0.15	<0.08	0.24	1.6
Rep. 92CIPC05	3, top B	96	0.13	<0.08	0.22	1.7
Aerosol fogging	2, bottom A	140	0.10	<0.08	0.26	2.6
at 15-11-91	2, bottom B	140	0.12	<0.08	0.20	1.7
0.02 kg ai/t	2, middle A	140	0.09	<0.08	0.17	1.9
potatoes	2, middle B	140	0.10	<0.08	0.15	1.5
	2, top A	140	0.08	<0.08	0.17	2.1
+	2, top B	140	0.09	<0.08	0.15	1.7
0.02 kg ai/t	3, bottom A	140	0.10	<0.08	0.14	1.4
potatoes	3, bottom B	140	0.12	<0.08	0.13	1.1
14-02-92	3, middle A	140	-	<0.08	0.10	-
	3, middle B	140	-	<0.08	0.09	-

Treatment, Report No.	Bin no., location in pile	Days after initial treatment	3-Chloroaniline residues[1] (mg/kg)			
			Whole	Pulp	Peel	Processing factor (peel)
Rep. 92CIPC05	3, top A	140	0.09	<0.08	0.15	1.7
	3, top B	140	0.09	<0.08	0.19	2.1
Aerosol fogging	2, bottom A	215	<0.08	<0.08	0.33	-
at 15-11-91	2, bottom B	215	<0.08	<0.08	0.51	-
0.02 kg ai/t	2, middle A	215	<0.08	<0.08	0.29	-
potatoes	2, middle B	215	<0.08	<0.08	0.34	-
	2, top A	215	<0.08	<0.08	0.26	-
+	2, top B	215	<0.08	<0.08	0.57	-
0.02 kg ai/t	3, bottom A	215	0.09	<0.08	0.37	4.1
potatoes	3, bottom B	215	-	<0.08	-	-
14-02-92	3, middle A	215	-	<0.08	0.30	-
	3, middle B	215	<0.08	<0.08	0.57	-
Rep. 92CIPC05	3, top A	215	<0.08	<0.08	0.35	-
	3, top B	215	<0.08	<0.08	0.38	4.8
Aerosol fogging	4, bottom A	5	0.23	<0.08	0.64	2.8
at 15-11-91	4, bottom B	5	0.18	<0.08	0.62	3.4
0.03 kg ai/t	4, middle A	5	0.11	<0.08	0.27	2.4
potatoes	4, middle B	5	0.10	<0.08	0.23	2.3
	4, top A	5	0.10	<0.08	0.22	2.2
	4, top B	5	0.09	<0.08	0.22	2.4
sampling at	5, bottom A	5	0.16		0.31	1.9
19-11-91	5, bottom B	5	0.19	<0.08	0.31	1.6
	5, middle A	5	0.15	<0.08	0.32	2.1
	5, middle B	5	0.15	<0.08	0.29	1.9
Rep. 92CIPC06	5, top A	5	0.17	<0.08	0.31	1.8
	5, top B	5	0.13	<0.08	0.26	2.0
Aerosol fogging	4, bottom A	91	0.15	<0.08	0.50	3.3
at 15-11-91	4, bottom B	91	0.15	<0.08	0.73	4.9
0.03 kg ai/t	4, middle A	91	0.15	-	0.22	1.5
potatoes	4, middle B	91	0.16	<0.08	0.28	1.8
	4, top A	91	0.16	<0.08	0.24	1.5
	4, top B	91	0.14	<0.08	0.18	1.3
sampling at	5, bottom A	91	<0.08	<0.08	0.22	-
13-02-92	5, bottom B	91	<0.08	<0.08	-	-
	5, middle A	91	<0.08	<0.08	-	-
Rep. 92CIPC06	5, middle B	91	<0.08	<0.08	-	-
	5, top A	91	<0.08	<0.08	0.15	-
	5, top B	91	<0.08	<0.08	0.13	-
Aerosol fogging	4, bottom A	140	0.26	<0.08	0.44	1.7
at 15-11-91	4, bottom B	140	0.23	<0.08	1.1	4.8
0.03 kg ai/t	4, middle A	140	0.22	<0.08	0.18	0.82
potatoes	4, middle B	140	0.25	<0.08	0.20	0.80
	4, top A	140	0.23	<0.08	0.24	1.0
	4, top B	140	0.22	<0.08	0.29	1.3
sampling at	5, bottom A	140	0.16	<0.08	0.44	2.8
02-04-92	5, bottom B	140	0.19	<0.08	0.41	2.2
	5, middle A	140	0.15	<0.08	0.39	2.6
	5, middle B	140	0.14	<0.08	0.37	2.6
Rep. 92CIPC06	5, top A	140	0.14	<0.08	0.37	2.6

Treatment, Report No.	Bin no., location in pile	Days after initial treatment	3-Chloroaniline residues[1] (mg/kg)			
			Whole	Pulp	Peel	Processing factor (peel)
	5, top B	140	0.14	<0.08	0.36	2.6
Aerosol fogging	4, bottom A	145	0.22	<0.08	0.21	0.95
at 15-11-91	4, bottom B	145	0.22	<0.08	0.23	1.1
0.03 kg ai/t	4, middle A	145	0.20	<0.08	0.12	0.60
potatoes	4, middle B	145	0.19	<0.08	0.11	0.58
	4, top A	145	0.17	<0.08	0.10	0.59
+	4, top B	145	0.19	<0.08	0.11	0.58
0.015 kg ai/t	5, bottom A	145	0.23	<0.08	0.15	0.65
potatoes	5, bottom B	145	0.18	<0.08	0.17	0.94
03-04-92	5, middle A	145	0.19	<0.08	0.18	0.95
sampling at	5, middle B	145	0.22	<0.08	0.16	0.73
07-04-92	5, top A	145	0.19	<0.08	0.15	0.79
Rep. 92CIPC06	5, top B	145	0.20	<0.08	0.21	1.1
Aerosol fogging	4, bottom A	215	0.09	<0.08	0.28	-
at 15-11-91	4, bottom B	215	0.09	<0.08	0.31	-
0.03 kg ai/t	4, middle A	215	<0.08	<0.08	0.23	-
potatoes	4, middle B	215	<0.08	<0.08	0.24	-
	4, top A	215	<0.08	<0.08	0.32	-
+	4, top B	215	<0.08	<0.08	0.24	-
0.015 kg ai/t	5, bottom A	215	0.13	<0.08	0.49	3.8
potatoes	5, bottom B	215	0.13	<0.08	0.40	3.1
03-04-92	5, middle A	215	0.12	<0.08	0.55	4.6
sampling at	5, middle B	215	0.13	<0.08	0.30	2.3
16-06-92	5, top A	215	0.12	<0.08	0.48	4.0
Rep. 92CIPC06	5, top B	215	0.11	<0.08	0.33	3.0
Mean processing factor peel (n = 127)						1.94
Median processing factor (n = 127)						1.9

[1] 0.08 mg/kg is method detection limit (MDL) for whole potato, pulp, peel, not LOQ.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Table 39 shows a summary of results from the USDA Pesticide Data Program for 1992, 1993, 1994, 1995, 1996, 1997 and 1998.

Table 39. Chlorpropham monitoring data from the USDA Pesticide Data Program.

Year	No. of samples analysed	No. of residues detected	Number of samples in chlorpropham residue range (mg/kg)										
			>0.002 ≤0.01	>0.01 ≤0.05	>0.05 ≤0.1	>0.1 ≤0.5	>0.5 ≤1	>1 ≤2	>2 ≤3	>3 ≤4	>4 ≤5	>5 ≤10	>10 ≤20
Apples													
1992		1		1									
1993		2		2									
1994	687	2	2										
1995	693	0											
1996	530	2		2									
Apple juice													
1996	171	0											
1997	683	0											

Year	No. of samples analysed	No. of residues detected	Number of samples in chlorpropham residue range (mg/kg)										
			>0.002 ≤0.01	>0.01 ≤0.05	>0.05 ≤0.1	>0.1 ≤0.5	>0.5 ≤1	>1 ≤2	>2 ≤3	>3 ≤4	>4 ≤5	>5 ≤10	>10 ≤20
1998	694	0											
Bananas													
1994	640	0											
1995	486	0											
Broccoli													
1994	679	0											
Cantaloupe													
1998	408	0											
Carrots													
1994	687	4		4									
1995	701	0											
1996	500	0											
Celery													
1994	176	0											
Corn syrup													
1998	298	0											
Corn, sweet													
1994	462	0											
1995	671	0											
1996	173	0											
Green beans													
1993		1		1									
1994	591	0											
1995	587	19		14	3	2							
1996	531	0											
1997	707	0											
1998	359	0											
Grapes													
1994	669	0											
1995	689	1		1									
1996	525	1		1									
Grape juice													
1998	665	0											
Lettuce													
1994	691	0											
Milk													
1996	570	0											
1997	727	0											
1998	594	1	1										
Oranges													
1994	683	0											
1995	691	0											
1996	518	0											
Orange juice													
1997	692	0											
1998	700	0											
Peaches													
1994	396	0											
1995	367	1		1									
1996	324	0											
1997	756	0											
Pears													
1997	708	0											
1998	712	0											

Year	No. of samples analysed	No. of residues detected	Number of samples in chlorpropham residue range (mg/kg)										
			>0.002 ≤0.01	>0.01 ≤0.05	>0.05 ≤0.1	>0.1 ≤0.5	>0.5 ≤1	>1 ≤2	>2 ≤3	>3 ≤4	>4 ≤5	>5 ≤10	>10 ≤20
Peas, sweet													
1994	433	0											
1995	670	0											
1996	355	0											
Potatoes													
1992		337		52	32	62	42	71	30	17	17	14	
1993		399		60	25	64	62	74	59	20	9	26	
1994	693	419	1	68	43	63	52	67	45	35	19	24	2
1995	707	482		107	25	59	75	74	58	35	17	29	3
Potatoes, sweet													
1996	507	1		1									
1997	681	0											
1998	357	1		1									
Spinach													
1995	610	0											
1996	517	1		1									
1997	680	0											
1998	695	0											
Strawberries													
1998	656	0											
Tomatoes													
1996	174	0											
1997	722	0											
1998	717	3		2		1							
Winter squash													
1997	661	1		1									
1998	679	1			1								

Table 40 gives a summary of the results from a review of the US Food and Drug Administration Pesticide Monitoring Database for Fiscal years 1992, 1993, 1994, 1995, 1996, 1997 and 1998. This database only reports detected residues not analysed samples in which residues were not detected.

Table 40. Results of monitoring of chlorpropham in the USA.

Crop/year	No. of residues detected	Number of samples in chlorpropham residue range (mg/kg)										
		≤0.01	>0.01 ≤0.05	>0.05 ≤0.1	>0.1 ≤0.5	>0.5 ≤1	>1 ≤2	>2 ≤3	>3 ≤4	>4 ≤5	>5 ≤10	>10 ≤20
Carrots												
1998	1	1										
Peppers												
1998	1	1										
Potatoes												
1992	24	7	4		1		7	3	2			
1993	70	10	2	9	13	10	6	11	2	3	4	
1994	56	3	3	7	21	7	9	4	2			
1995	100	57	11	4	7	4	3	8	3	1	1	1
1996	43	1	2	1	14	3	8	2	3	3	6	
1997	43	1			6	3	6	5	5	4	12	1
1998	37	5	1	5	4	4	5	2		5	6	
Yams												
1992	3		1		2							

Crop/year	No. of residues detected	Number of samples in chlorpropham residue range (mg/kg)										
		≤0.01	>0.01 ≤0.05	>0.05 ≤0.1	>0.1 ≤0.5	>0.5 ≤1	>1 ≤2	>2 ≤3	>3 ≤4	>4 ≤5	>5 ≤10	>10 ≤20
1995	2		2									

Chlorpropham was included in the 1994 and 1996 Australian Market Basket Surveys (Marro, 1996; Hardy, 1998).

In the 1994 survey chlorpropham was not detected (limit of reporting not stated) in the only food examined which was potatoes.

In the 1996 survey chlorpropham was detected in one sample at 0.2 mg/kg. Potatoes were the only food examined. The calculated dietary intakes of chlorpropham were very low for both the mean energy diets and the 95th percentile energy diets (Table 41).

Table 41. Estimated dietary intakes of chlorpropham from the Australian Market Basket Survey (Hardy, 1998).

	Body weight, kg	Potato consumption, g/day		Intake, ng/kg bw/day	
		mean	95th percentile	mean	95th percentile
Adults males	75	151	244	1.9	3.1
Adults females	59.1	87	144	1.4	2.3
Boys aged 12	39.8	116	183	2.8	4.4
Girls aged 12	41.5	104	150	2.4	3.4
Toddlers aged 2	12.3	23	27	1.8	2.1
Infants 9 months	9.1	13	15	1.3	1.6

NATIONAL MAXIMUM RESIDUE LIMITS

The governments of Australia and Germany submitted their MRLs. Australian MRLs for chlorpropham were revised in December 2000 (Simpson and Hamilton, 2001).

National MRLs reported to the Meeting.

Country	Crop	MRL (mg/kg)
Australia	Potatoes	30
	Onion, bulb	0.05*
	Garlic	0.05*
Germany	Potatoes, washed	5
	Carrot, leaf of root celery, chervil, parsnips, parsley, celery stock	0.2
	Other foods of plant origin	0.1
USA	Post-harvest application on potatoes	50
	Soya beans	0.2

* MRL set at or about the LOQ

APPRAISAL

Chlorpropham (isopropyl 3-chlorophenylcarbamate) was reviewed only for toxicology by the JMPR in 1963, 1965 and 2000. The compound was identified as a candidate for evaluation of residues as a new compound by the JMPR 2001 by the CCPR at its Thirtieth Session (1998) (ALINORM 99/24).

Chlorpropham is used as a growth regulator to suppress the post-harvest sprouting of ware

potatoes during storage. As a herbicide, it controls a broad spectrum of annual weeds. Only information on its use as a growth regulator for ware potatoes was made available to the Meeting by the Chlorpropham Manufacturers Task Force in the USA. This comprised studies on metabolism in animals and plants, methods of residue analysis, stability of residues in stored analytical samples, uses, results of supervised residue trials under commercial storage conditions and processing data. Information on national trials conducted according to GAP was provided by the governments of Australia and Germany.

Pure chlorpropham is a cream-coloured, crystalline solid of moderate volatility. It has limited solubility in water but is highly soluble in certain organic solvents. The log P_{ow} of 3.4 suggests that bioaccumulation may occur.

The trials summarized below were based on post-harvest use of chlorpropham on stored potatoes only.

Metabolism

Animals

The metabolism of chlorpropham in rats, lactating goats and laying hens is qualitatively similar. In rats, chlorpropham was rapidly absorbed and essentially completely metabolized before excretion in urine and, in small amounts, in faeces. Within 24 h, 82–92% of the radiolabel was recovered in the urine and 3–5% in the faeces. Three major metabolic routes were proposed: (1) hydroxylation at the 4-position and subsequent conjugation with sulfate or glucuronide; (2) oxidation of the isopropyl side-chain to the alcohol and subsequently the acid; and (3) decarbanilation to form 3-chloroaniline followed by *N*-acetylation, 4-hydroxylation and conjugation.

After administration of [^{14}C-ring]chlorpropham in capsules at a dose of 1.6–1.9 mg/kg bw (32–36 ppm in the feed) to two lactating goats for 7 days, rapid absorption and elimination *via* urine and faeces were seen (about 99%). About 1% was transferred to milk and liver, and one or two orders of magnitude less to fat and muscle. The goats metabolized chlorpropham readily. The main metabolic pathways included hydroxylation at the 4-position and subsequent formation of conjugates of sulfate or glucuronide. The main residue in the milk and kidney was the metabolite 4-hydroxy-chlorpropham-*O*-sulfonic acid (81% and 16% of TRR, respectively), while the main residue in fat tissues was chlorpropham (88% of TRR).

In laying hens receiving a daily dose of 6 mg [^{14}C-ring]chlorpropham by capsule (3.3–4.2 mg/kg bw or 50 ppm in the feed) for 7 days, 83% of the cumulative dose was recovered from excreta and only 0.03% from the egg production. The maximum concentrations of residues were 0.07 mg/kg in egg white and 0.23 mg/kg in egg yolk. The concentrations of TRR in tissues and organs were low (~0.5 mg/kg in liver and kidneys, ~0.2 mg/kg in fat and skin; 0.015 and 0.006 mg/kg in thigh and breast muscle, respectively). Chlorpropham was the main residue in hen fat and skin (92% and 68% of TRR, respectively), while the main residues in liver and kidney were 3-chloro-4-hydroxyaniline conjugates (25–64%). The *O*-sulfonic acid conjugate of 3-chloro-4-hydroxyaniline was the main compound in eggs (22% of TRR).

Plants: potato

Studies on metabolism and residues in crops other than potato were not provided. Translocation and formation of metabolites in potatoes were investigated after treatment by surface coating with [^{14}C-ring]chlorpropham and simulation of cold-storage conditions. Translocation was slow; approximately 86% of the TRR still being present in the surface methanol-wash fraction as chlorpropham after 52 weeks of storage. About 10% of TRR was recovered from the peel and about 3% from the pulp, mainly as unchanged chlorpropham.

The main metabolite in peel was an oligosaccharide of 4-hydroxy-chlorpropham. 3-Chloroaniline was the second main metabolite in peel. It was not identified as a free metabolite in pulp but in conjugated form, as 3-chloroaniline-*N*-glucosylamine (6% of TRR in pulp). The main metabolites in pulp, both representing about 18% of TRR, were an oligosaccharide and an amino acid conjugate of 4-hydroxy-chlorpropham. About 10% of TRR in peel and pulp was not extractable. Three potential metabolic pathways in plants were proposed:

- hydroxylation and subsequent conjugation with glucose, oligosaccharides or amino acids at the 4-position (*para* to the amino moiety) or conjugation of 4-hydroxy-chlorpropham with a methyl moiety to *para*-methoxy-chlorpropham or to an *S*-cysteinyl-hydroxy-chlorpropham;
- decarboxylation to 3-chloroaniline, followed by conjugation with glucose and other biomolecules;
- oxidation of the isopropyl chain and subsequent conjugation with oligosaccharide(s).

Methods of analysis

Plant matrices: potato

Most of the methods submitted for the analysis of chlorpropham residues in potato involved homogenization with an organic solvent (e.g. methanol, petroleum ether/acetone, hexane/acetone) followed by partition into dichloromethane. For further purification of the extract, an adsorbent column (e.g. Florisil) can be used. Chlorpropham is determined by GLC–NPD or after bromination as the bromo derivative by GLC–ECD. The LOQ was validated as 0.02 mg/kg.

Methods for the determination of chlorpropham and its three metabolites 3-chloroaniline, 4-hydroxy-chlorpropham and *para*-methoxy-chlorpropham in potato and potato products were submitted. They involved methanol/water as the primary extraction solvent, sometimes acid or alkaline hydrolysis and sonication for splitting conjugates, with subsequent clean-up by liquid–liquid partition with other organic solvents or phosphate buffer. For oil-processed samples, GPC clean-up follows. Determination was made by GLC–NPD. The methods have been validated for analysis of the parent compound and metabolites in whole potato, fresh peel and pulp, fries with and without skins, canola oil, potato chips with and without skins, processed dried peels, processed wet peels and dehydrated granules.

The recoveries of chlorpropham, 4'-hydroxy-chlorpropham and *para*-methoxy-chlorpropham were satisfactory. 3-Chloroaniline was recovered from fortified samples with varying consistency (40–70% from whole potato, pulp, peel with a fortification level of 0.4 mg/kg), as a large proportion of the aniline moiety can remain bound on biological material and occur as e.g. *N*-glucosyl or *N*-malonyl conjugates. Therefore, for each batch of samples from supervised trials, three untreated samples of each matrix were extracted, two of which were fortified with chlorpropham and the three metabolites to document recovery levels. The third sample served as a blank matrix to monitor contamination and interfering background matrix. Furthermore, matrix-based calibration standards were used. The method detection limits (MDL) and the LOQ for chlorpropham, 3-chloroaniline, *para*-hydroxy-chlorpropham and *para*-methoxy-chlorpropham (MDL / LOQ) were:

– 0.08 / 0.45 mg/kg in whole potato, fresh pulp, fresh peel and processed wet peel,
– 0.2 / 1.1 mg/kg in fries,
– 0.45 / 2.2 mg/kg in chips,
– *0.38 / 1.9 mg/kg in dehydrated granules and processed dried peel,*
– 2.9 / 14 mg/kg in canola oil.

Animal matrices

The parent and the metabolite *p*-hydroxy-chlorpropham-*O*-sulfonic acid cannot be determined together in ruminant matrices. The method for chlorpropham involves solid phase matrix dispersion

followed by GLC–MS detection. The recoveries of the lowest fortification level of 0.01 mg/kg in whole milk, liver, muscle, kidney and fat were about 200% in some cases. Therefore, the LOQ for chlorpropham achievable in whole milk, skim milk and cream should be 0.05 mg/kg and that for liver, muscle, kidney and fat should be 0.1 mg/kg.

4-Hydroxy-chlorpropham-*O*-sulfonic acid is determined in whole and skim milk by dilution with acetonitrile, selective precipitation of interfering substances and analysis by reversed-phase HPLC with UV detection. In tissues and cream, 4-hydroxy-chlorpropham-*O*-sulfonic acid is isolated by solid phase extraction and is determined by reversed-phase HPLC and UV detection. The achievable LOQ for this metabolite in whole milk, skim milk, cream, liver, muscle, kidney and fat is 0.05 mg/kg.

Stability of residues in stored analytical samples

Plant matrices: potato

A study of stability in freezer storage at –20 to –21 °C with fresh whole tubers, pulp and peel and processed potato products (chips, fries, dehydrated granules, processed wet and dried peel), fortified at two levels with chlorpropham or one of the metabolites 3-chloroaniline, 4-hydroxy-chlorpropham or *para*-methoxy-chlorpropham, showed that 3-chloroaniline and 4-hydroxy-chlorpropham were unstable in whole potatoes, potato pulp and potato peel after 90 days of storage. 3-Chloroaniline was also unstable in processed wet peels. The low initial recoveries of these analytes and their instability in fresh products may be due to bioreactivity with the potato matrix. An acceptable stability of 5–6 months' storage was found for chlorpropham and *para*-methoxy-chlorpropham.

Animal matrices

Cow liver, muscle and milk were fortified with 0.1 mg/kg chlorpropham and 4′-hydroxy-chlorpropham-*O*-sulfonic acid and stored at –20 °C. There was no significant degradation of either compound in any of the matrices over the storage period: chlorpropham, 28 days in liver, 59 days in muscle and 127 days in milk; 4-hydroxy-chlorpropham-*O*-sulfonic acid, 59 days in liver, 122 days in muscle and 133 days in milk.

Definition of the residue

Plant material

Studies of metabolism in stored potatoes established that most of the radiolabel was in the peel (10% of the applied amount after washing) and only a small proportion (3% of the applied amount) in the pulp. Most of the residue in the peel consisted of chlorpropham (85%) and only a minor part (3.5%) was 3-chloroaniline. Chlorpropham made up 42% of the residue in pulp.

In a supervised trial with stored potatoes, the only metabolite detected was 3-chloroaniline, less than 2% of the chlorpropham residue. Residues of *para*-methoxy-chlorpropham and (conjugates of) 4-hydroxy-chlorpropham were not detected.

The 2000 JMPR identified 3-chloroaniline as a toxicologically significant compound, apart from the parent chlorpropham. As 3-chloroaniline forms only a minor part of the residue, the Meeting agreed that residues in potatoes can be defined as chlorpropham *per se* for enforcement and risk assessment purposes.

Animal products

Studies of metabolism were carried out in rats, goats and hens. Chlorpropham was rapidly and virtually completely absorbed, extensively metabolized and rapidly excreted in both domestic

animals and rats. As potatoes are a minor feed item for chicken (< 10% of feed, see *FAO Manual*, p. 125), the Meeting focused on the study of metabolism in goats.

The main residue in milk and kidney of goats was the low-fat-soluble metabolite 4-hydroxy-chlorpropham-*O*-sulfonic acid (81% and 16% of measured TRR), while the fat-soluble chlorpropham was the main residue in fat (88%). No methods of analysis are available to determine the two residues simultaneously. As the metabolite was considered to be of no toxicological significance by the 2000 JMPR, the Meeting agreed that the residue definition for animal products for compliance with MRLs and dietary risk assessment should be chlorpropham only.

The presence of chlorpropham in fat and cream but not in muscle or skim milk in the feeding study in dairy cows and its log P_{OW} of 3.4 imply solubility in fat. The Meeting agreed that the residue is fat-soluble.

Fate of residues during storage

Chlorpropham is registered in the USA for post-harvest treatment on potato as an emulsifiable concentrate used by direct spray of a 1% aqueous emulsion on potato tubers moving along a conveyor line or as an aerosol fog at a standard application rate of 0.015 kg ai/t. The rate should be adapted to the storage period and temperature. Re-treatments can be made with one of the following regimens:

- aerosol fog at 0.02 kg ai/t at each of two applications 90 days apart, followed by direct spray at 0.01 kg ai/t, or
- aerosol fog at 0.03 kg ai/t and a second aerosol fog at 0.015 kg ai/t 140 days later.

A withholding period in days was not identified.

Extensive data were provided from a supervised trial in the USA on various treatment schedules on ware potatoes stored in bins. Each bin had its own air ventilation, refrigeration unit and computer-controlled monitoring system for accurate measurement of sampling pile conditions. The bins, each containing approximately 63.5 t of potatoes, were designed to allow access for tuber sampling during storage. Industry standards for relative humidity and temperature with continuous air flow were followed. Each bin was fogged with aerosol separately. Each bin was therefore considered as a separate trial. Furthermore, applications carried out at different times and different rates were considered separate treatments and equal a separate trial. The residue values used for evaluation were selected as either the highest value of the six samples taken from each bin or, in the case of a decline study, only one value (the highest) was selected. The concentrations of residues of chlorpropham in whole unwashed tubers resulting from various treatments according to GAP were:

Treatment (kg ai/t potatoes)	Residues (mg/kg)	Time after initial treatment (days)
1 x EC direct spray 0.01	8.2	0
1 x aerosol fog 0.02 + 1 x EC direct spray 0.01	9.1, 9.3, 9.4, 11	5, 91, 96
1 x aerosol fog 0.02	8.7, 8.9	5
1 x aerosol fog 0.03	16, 23	5,
2 x aerosol fog 0.02	9.9, 18	96, 140
1 x aerosol fog 0.03 + aerosol fog 0.015	14, 16	145
2 x aerosol fog 0.02 + 1 x EC direct spray 0.01	8.2, 9.7, 11, 11, 13, 14	96, 140, 215

The concentrations, in ranked order (median underlined), were: 8.2 (2), 8.7, 8.9, 9.1, 9.3, 9.4, 9.7, 9.9, <u>11</u> (3), 13, 14 (2), 16 (2), 18 and 23 mg/kg.

Chlorpropham is registered in Belgium, France and Germany for spraying, dusting or hot fogging of ware potatoes at 0.01–0.02 kg ai/t without a withholding period in days. The same treatment rates are registered in The Netherlands, with a withholding period of 60 days. The potatoes

can be stored in boxes or in bulk.

One trial carried out in France in 1998 (1 x 0.007 + 1 x 0.006 kg ai/t, pile from pallox) and one trial from Belgium in 1997 (1 x 0.015 kg ai/t, manual treatment of potatoes in paper bags) resulted in maximum residue concentrations of 8.8 and 13 mg/kg. The tubers were not washed before freezing of the analytical samples.

Treatment of potatoes stored in boxes was investigated in several trials, in which some of the potatoes were washed and some were washed and peeled after sampling. Seven trials in Belgium (1997) with hot fogging application of 1 x 0.007 kg ai/t plus 1 x 0.006 kg ai/t resulted in values of 0.61, 0.85, 0.89, 0.96, 1.1 and 1.2 (2) mg/kg. Seven trials in Germany in 1998 (dusting, 1 x 0.015 kg ai/t) resulted in concentrations of 0.06, 0.11, 3.5 (2), 3.8, 4.3 and 4.9 mg/kg. Four trials carried out in Germany in 1996 and 1999 with powdering of 1 x 0.01 kg ai/t, resulted in values of 1.7, 1.9, 2.0, 2.5, 2.5, 3.0, 3.0 and 3.2 mg/kg. The concentrations in washed whole potato tubers were, in ranked order (median underlined), 0.61, 0.85, 0.89, 0.96, 1.1, 1.2 (2), 1.7, 1.9, 2.0, 2.5 (2), 3.0 (2), 3.1, 3.2, 3.5 (2), 3.8, 4.3, 4.8 and 4.9 mg/kg.

The data on residues received from the European studies of box-stored, washed potatoes are different from those from the study of bin storage of unwashed tubers in the USA. The MRL, STMR and highest residues were derived from the USA data on unwashed potatoes and the two trials with unwashed potatoes in France and Belgium. The residue concentrations, in ranked order, were: 8.2 (2), 8.7, 8.8, 8.9, 9.1, 9.3, 9.4, 9.7, 9.9, 11 (3), 13 (2), 14 (2), 16 (2), 18 and 23 mg/kg.

The Meeting estimated a maximum residue level of 30 mg/kg, an STMR value of 11 mg/kg and a highest residue of 23 mg/kg for ware potatoes.

Fate of residues during processing

No information on the fate or nature of the residue after hydrolysis under cooking conditions was submitted.

Cooked potatoes were prepared from one fresh whole tuber sample containing 4.6 mg/kg chlorpropham. The concentration of residues decreased to 0.24 mg/kg in peeled fresh potatoes and to 0.08 mg/kg in peeled cooked potatoes after cooking for 20 min. Cooking reduced the value to 33% (processing factor, 0.33). From the STMR and the HR values for fresh ware potatoes of 11 and 23 mg/kg, an STMR-P value of 3.6 mg/kg and an HR-P value of 7.6 mg/kg were calculated for cooked potatoes with skin.

Cooked and peeled potatoes: The median processing factor for chlorpropham on raw peeled potatoes, based on 166 samples, was 0.027. Application of this factor to the STMR of 11 mg/kg and the HR of 23 mg/kg for raw ware potatoes provided a median value of 0.297 mg/kg and a highest residue of 0.62 for raw peeled potatoes. With the processing factor for cooking (0.33), an STMR-P value of 0.098 mg/kg and a HR-P value of 0.2 mg/kg were calculated for cooked potatoes without skin.

An adequate, extensive study of potato processing by standard industrial procedures provided information on the distribution of residues of chlorpropham and 3-chloroaniline in whole potato, pulp and peel, chips, and frozen and dehydrated products. Processing factors could be derived for fresh peeled potato and fresh peel, but not for chips, fries, dehydrated granules or processed peel, as different samples were used for the determination of residues in the raw agricultural commodity and in the processed product. For this reason, the concentrations used for evaluation of chips, fries, dehydrated granules and processed peel were selected from the data in trials conducted according to GAP.

Chips[1]: The concentrations of chlorpropham residues in chips with and without skin were 0.82, 1.2, 1.5 (2), 1.7, 1.9, 3.8, 4.0, 4.1, 4.2, 4.4, 4.6 (3), 5.0, 5.3, 6.3 (2), 6.4, 7.0, 7.1, 7.9 and 8.1 mg/kg and < 0.045 (11), 1.1, 1.2, 1.3, 1.4 (3), 1.5 (4), 1.6 and 1.8 mg/kg. The Meeting estimated STMR-P values of 4.6 and 1.1 mg/kg for chips with and without skin, respectively.

Fries[2]: The concentrations of chlorpropham residues in fries with and without skin were 0.97, 1.1, 1.2 (2), 1.3 (2), 1.4 (5), 1.5 (3), 1.6 (5), 1.7, 1.9, 2.0 (3), 2.1 (2), 2.2 (3), 2.3 (2), 2.6 (2), 2.7, 2.8 and 4.0 mg/kg and ≤ 0.2 (20), 0.23, 0.28 (2), 0.29, 0.31, 0.32, 0.33, 0.34 (2), 0.35, 0.36, 0.37 (2), 0.4, 0.41 and 0.54 mg/kg, respectively. The Meeting estimated STMR-P values of 1.6 and 0.2 mg/kg for fries with and without skin, respectively.

Dehydrated granules[2]: The concentrations of residues in dehydrated granules were < 0.38 (3), 0.41, 0.57, 0.63, 0.64, 0.65, 0.67, 0.69 (2), 0.71, 0.75 (3), 0.76, 0.81, 0.82, 0.87 (2), 0.91, 0.95, 0.96, 1.0, 1.1, 1.2 (3), 1.3, 1.4, 1.5 (3), 1.6, 1.9 and 2.1 mg/kg. The Meeting estimated an STMR-P value for chlorpropham of 0.845 mg/kg in dehydrated granules.

Potato peel, processed[2]: The concentrations of residues in industrially produced wet peel were 10, 11, 12, 13, 14 (5), 15 (3), 17 (4), 19, 21, 26 (3), 30, 31 (3), 32, 33 (2), 34 (2), 35 (2), 41, 42, 43 and 45 mg/kg The Meeting estimated an STMR-P value of 23.5 mg/kg for processed potato wet peel.

Residues in animal commodities

The Meeting estimated the dietary burden of chlorpropham and 3-chloroaniline in farm animals on the basis of the feeds listed in Appendix IX of the *FAO Manual.* The Meeting agreed to use only the STMR value for calculation of the dietary burden from processed animal feed as wet potato peel. It is suitable for estimating MRLs and HRs for animal commodities.

Dietary burden of chlorpropham

Commodity	Residue (mg/kg)	Basis	Dry matter (%)	Residue, dry weight (mg/kg)	Choose diets (%)			Residue contribution (mg/kg)		
					Beef cattle	Dairy cattle	Poultry	Beef cattle	Dairy cows	Poultry
Potato wet peel, processed	23.5	STMR-P	15	157	75	40	–	118	63	–

The dietary burden of chlorpropham in ruminant commodities (expressed as dry weight) used to estimate the MRL and STMR value was 118 mg/kg for beef cattle and 63 mg/kg for dairy cows.

In a 28-day study of cows given chlorpropham by capsule at a level equivalent to 0, 322, 955 or 3111 ppm in the feed (dry weight basis), only minor concentrations of chlorpropham residues (< 0.01–0.06 mg/kg) were found in milk at the highest level tested. The concentrations of the metabolite 4-hydroxy-chlorpropham-*O*-sulfonic acid (calculated as chlorpropham) were higher and roughly proportional to the feeding level, ranging from 0.1 to 0.61 mg/kg at the lowest level to 0.37–6.7 mg/kg at the highest level. Chlorpropham residues could not be detected in skim milk, but in cream the concentrations were 0.02–0.03 at the lowest level and 0.21–0.64 at the highest level. The residues of 4-hydroxy-chlorpropham-*O*-sulfonic acid were nearly equally distributed in skim milk and cream, the concentrations (calculated as chlorpropham) being 1.9–3.9 mg/kg and 1.7–3.6 mg/kg, respectively, in the group given the highest dose.

[1] Treatment of potatoes intended for chips: aerosol fogging 0.03 + 0.015 kg ai/t, treatment interval, 4.5 months
[2] Treatment of potatoes intended for frozen or dehydrated products: aerosol fogging 0.02 + 0.02 kg ai/t, treatment interval, 3 months

Minor concentrations of parent chlorpropham were found in muscle, liver and kidney. In the group at the highest level, the maximum values were 0.02 mg/kg in liver and kidney and 0.1 mg/kg in muscle. In fat, the chlorpropham residue values were 0.1–0.13, 0.19–0.39 and 0.15–2.8 mg/kg at the lowest, intermediate and highest level, respectively. Residues of 4-hydroxy-chlorpropham-*O*-sulfonic acid (calculated as chlorpropham) were found predominantly in kidney, with concentrations of 0.12–0.26 mg/kg, 0.76–1.2 mg/kg and 1.0–2.3 mg/kg at the three levels, respectively. No residues of the metabolite were detected (< 0.03 mg/kg) in muscle or fat. In liver, it was found only in cows at the highest level, at a maximum of 0.06 mg/kg.

The MRL and the STMR value for chlorpropham in milk were calculated from the interpolated dietary burden of 63 mg/kg (based on the STMR) for dairy cows; and the MRLs and the STMRs for meat, liver and kidney were derived from the dietary burden of 118 mg/kg for beef cattle. The interpolation is based on the actual concentration of residue in the group at the lowest level (322 ppm). As the compound is fat-soluble, the maximum residue level and the STMR value for milk were based on the concentrations of residue in cream. The following table shows the highest and the mean actual and interpolated residues used for estimating MRLs and STMR values for chlorpropham.

Feeding level (ppm) *Interpolated* / Actual	Chlorpropham residues (mg/kg)							
	Cream (mean)	Liver		Kidney		Muscle		Fat	
		High	Mean	High	Mean	High	Mean	High	Mean
MRL Beef cattle		*0.007* / 0.02		*< 0.004* / < 0.01		*0.004* / 0.01		*0.048* / 0.13	
STMR Beef cattle			*0.005* / 0.013		*< 0.004* / < 0.01		*< 0.004* / < 0.01		*0.04* / 0.11
MRL Dairy cows	*0.006* / 0.03								
STMR Dairy cows	*0.006* / 0.03								

118 / 322 (Beef cattle); *63* / 322 (Dairy cows)

The Meeting estimated maximum residue levels for chlorpropham of 0.0005* mg/kg F for milk, 0.01* mg/kg for edible offal of cattle and 0.1 mg/kg (fat) for cattle meat. The estimated STMR values are 0.0003 mg/kg for cattle milk, 0.005 mg/kg for edible offal of cattle and 0.004 mg/kg for cattle meat. The estimated highest residues are 0.007 mg/kg for edible offal of cattle and 0.004 mg/kg for cattle meat.

Recommendations

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

Definition of residue (for compliance with MRLs and for estimation of dietary intake): Chlorpropham. The residue is fat-soluble.

Commodity		Recommendation (mg/kg)			
CCN	Name	MRL		STMR, STMR-P	HR, HR-P
		New	Previous		
MO 0812	Cattle, edible offal of	0.01*	-	0.005	0.007
MM 0812	Cattle meat	0.1 (fat)	-	0.004	0.004
ML 0812	Cattle milk	0.0005*F	-	0.0003	
VR 0589	Potato	30 Po	–	11	23
	Potato, cooked[1]			3.6	7.6
	Potato, peeled and cooked			0.098	0.2
	Potato chips with skin			4.6	
	Potato chips without skin			1.1	
	Potato French fries with skin			1.6	
	Potato French fries without skin			0.2	
	Potato dehydrated granules			0.845	

[1]The information provided to the JMPR precludes an estimate that the dietary intake would below the acute RfD.

Further work or information

Desirable

1. A study on hydrolysis with radiolabelled chlorpropham to clarify the effect of cooking on the nature of residues (Annex 5, reference 86, pp. 12–15)
2. Processing studies on cooked potatoes with skin and for microwaved and oven-baked potatoes

Dietary risk assessment

Long-term intake

STMR or STMR-P values for chlorpropham were estimated by the Meeting for animal products, potatoes and six processed potato commodities. When data on consumption were available, these values were used to estimate dietary intake. The results are shown in Annex 3 (Report 2001).

The IEDIs, based on the estimated STMR values, were 1–50% of the ADI for the five GEMS/Food regional diets. The Meeting concluded that long-term intake of residues of chlorpropham from use on potatoes is unlikely to present a public health concern.

Short-term intake

The IESTI for chlorpropham was calculated for animal products and for potatoes (and their processing fractions) for which maximum residue levels and STMR values were estimated and for which data on consumption were available. The results are shown in Annex 4 (Report 2001).

The 2000 JMPR established an acute RfD of 0.03 mg/kg bw, on the basis of a NOAEL of 10 mg/kg bw per day in a 90-day study of toxicity in rats and a safety factor of 300. This value includes an additional safety factor of 3 to take account of inadequacies in the assessment of

methaemoglobinaemia, the critical toxicological effect. The current Meeting stated that the assessment of acute risk might require refinement of the acute RfD by submission of new studies that more appropriately address the end-point of concern.

The IESTI represented 0–1600% of the acute RfD for the general population and 0–4600% of the acute RfD for children. The values of 510 and 1500% represent the estimated short-term intake of cooked potatoes with skin. Peeling and cooking of potatoes reduced the concentration of chlorpropham residue, resulting in IESTIs of 10% of the acute RfD for the general population and 40% of the acute RfD for children. The Meeting concluded that short-term intake of chlorpropham residues is unlikely to present a public health concern, when peeled potatoes are consumed.

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