Pesticide residues in food 2004

Evaluations
Part I – Residues

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PAPER

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Joint meeting of the
FAO Panel of Experts on Pesticide Residues
in Food and the Environment
and the
WHO Core Assessment Group
Rome, Italy, 20–29 September 2004

WORLD HEALTH ORGANIZATION
FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS
Rome, 2005

Monographs containing summaries or residue data and toxicological data considered at the 2004 JMPR, together with recommendations, are available upon request from FAO or WHO under the title:

Pesticide residues in food 2004
Evaluations
Part I: Residues
FAO Plant Production and Protection Paper
and
Part II: Toxicology
WHO

This report contains the collective views of two international groups of experts and does not necessarily represent the decisions or the stated policy of the Food and Agriculture Organization of the United Nations or of the World Health Organization.

INTERNATIONAL PROGRAMME ON CHEMICAL SAFETY

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CONTENTS

[1/] Evaluated for the Periodic Review Programme of the Codex Committee on Pesticide Residues.
[2/] New compound.

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Rome, 20–29 September 2004

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ABBREVIATIONS

(Well-known abbreviations in general use are not included. Specific abbreviations for pesticide degradation products, etc., may be used in the monographs and these are either identified where first used or in a table within the monograph. Two-letter codes for pesticide formulations are given in the Manual on development and use of FAO and WHO specifications for pesticides, 1st Ed., FAO Plant Production and Protection Paper 173, FAO, Rome, 2002.)

ADI	acceptable daily intake
AFID	alkali flame-ionization detection or detector (equivalent to TSD, forerunner of NPD)
ai	active ingredient
AR	applied radioactivity
bw	body weight
CA	Chemical Abstracts
CAC	Codex Alimentarius Commission
CAS	Chemical Abstracts Services
CCN	Codex classification number (for compounds or commodities)
CCPR	Codex Committee on Pesticide Residues
CCRVDF	Codex Committee on Residue of Veterinary Drugs in Food
CI	chemical ionization
CV	coefficient of variation (RSD)
CXL	Codex Maximum Residue Limit (Codex MRL). See MRL
DAT	days after (last) treatment
DFG	Deutsche Forschungsgemeinschaft
DT_{50}	time for 50% decomposition (i.e. half-life)
DT_{90}	time for 90% decomposition
ECD	electron capture detection or detector
EI	electron-impact (ionization), now more usually electron ionization
EPA	Environmental Protection Agency (usually US EPA)
F_1	first filial generation
F_2	second filial generation
FAO	Food and Agriculture Organization of the United Nations
FID	flame-ionization detection or detector
FPD	flame-photometric detection or detector
GAP	good agricultural practice(s)
GC	gas chromatography; the detector system used is usually also abbreviated as a suffix
GEMS/Food	Global Environment Monitoring System–Food Contamination Monitoring and Assessment Programme
GLP	good laboratory practice (i.e. the defined system, not in the general sense)
GPC	gel-permeation chromatography
GSH	glutathione
HPLC	high-performance liquid chromatography
HPLC-MS	high-performance liquid chromatography – mass spectrometry
HPLC-UV	high-performance liquid chromatography with UV absorption detection
HR	highest residue in the edible portion of a commodity found in trials used to estimate a maximum residue level in the commodity
HR-P	highest residue in a processed commodity calculated by multiplying the HR of the raw commodity by the corresponding processing factor

IARC	International Agency for Research on Cancer
IEDI	international estimated daily intake
IESTI	international estimate of short-term dietary intake
IPCS	International Programme on Chemical Safety
	ITD ion-trap detector or detection
JECFA	Joint Expert Committee on Food Additives
JMPR	Joint Meeting on Pesticide Residues
LC	liquid chromatography
LC-MS	liquid chromatography – mass spectromety
LC_{50}	median lethal concentration
LD_{50}	median lethal dose
LOAEL	lowest-observed-adverse-effect level
LOAEC	lowest-observed-adverse-effect concentration
LOD	limit of detection
LOQ	limit of quantification
LSC	liquid scintillation counting or counter
MID	multiple ion detection (mass spectrometric)
MRL	Maximum Residue Limit. MRLs include draft MRLs and Codex MRLs (CXLs). The MRLs recommended by the JMPR on the basis of its estimates of maximum residue levels enter the Codex procedure as draft MRLs. They become Codex MRLs when they have passed through the procedure and have been adopted by the Codex Alimentarius Commission.
MS	mass spectrometry or mass spectrometric detector (suffix to GC- or LC-)
MSD	mass-selective detection or detector
MS/MS	tandem mass spectrometry
NOAEL	no-observed-adverse-effect level
NPD	nitrogen/phosphorus detector
OECD	Organization for Economic Co-operation and Development
PES	post-extraction solids
PF	processing factor
PHI	pre-harvest interval
ppm	parts per million (used only with reference to the concentration of a pesticide in a diet, in all other contexts the terms mg/kg or mg/l are used)
P_{ow}	octanol–water partition coefficient
RAC	raw agricultural commodity
r.d.	relative density (formerly called specific gravity)
RfD	reference dose (usually in phrase “acute RfD”)
RSD	relative standard deviation
SD	standard deviation
SPE	solid-phase extraction (may also describe a post-extraction clean-up process)
STMR	supervised trials median residue
STMR-P	supervised trials median residue in a processed commodity calculated by multiplying the STMR of the raw commodity by the corresponding processing factor
t	tonne (metric ton)
TAR	total applied (or administered) radioactivity
TLC	thin-layer chromatography
TRR	total radioactive residue

TMDI	theoretical maximum daily intake
TSD	thermionic specific detection or detector (equivalent to AFID, forerunners of NPD)
USDA	US Department of Agriculture
US FDA	US Food and Drug Administration
UV	ultraviolet (radiation)
W	the previous recommendation is withdrawn, or withdrawal of the existing Codex or draft MRL is recommended
WHO	World Health Organization

USE OF JMPR REPORTS AND EVALUATIONS BY REGISTRATION AUTHORITIES

Most of the summaries and evaluations contained in this report are based on unpublished proprietary data submitted for use by JMPR in making its assessments. A registration authority should not grant a registration on the basis of an evaluation unless it has first received authorization for such use from the owner of the data submitted for the JMPR review or has received the data on which the summaries are based, either from the owner of the data or from a second party that has obtained permission from the owner of the data for this purpose.

INTRODUCTION

The Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Core Assessment Group (JMPR), held in Rome, 20-29 September 2004, contains a summary of the evaluations of residues in foods of the various pesticides considered, as well as information on the general principles followed by the Meeting (JMPR, 2004). The present document contains summaries of the residues data considered, together with the recommendations made.

The Evaluations are issued in two parts:
Part I: Residues (by FAO);
Part II: Toxicology (by WHO).

For those interested in both aspects of pesticide evaluation, both parts and the Report containing summaries of residues and toxicological considerations are available.

Some of the compounds considered at the Meeting were previously evaluated and reported on in earlier publications. In general, only new information is summarized in the relevant monographs but reference is made to previously published evaluations, which should also be consulted. In the case of older compounds which are re-evaluated as part of the periodic review programme of the CCPR, a review of all available data, including data which may have previously been submitted, is carried out. Compounds evaluated for the first time are indicated by a single asterisk and those evaluated in the CCPR periodic review programme by double asterisks in the Table of Contents.

Summaries of recommended MRLs, STMR and HR levels and assessments of dietary intake, are published as Annexes 1, 3 and 4 in the Report, and reference is made to this report.

The name of the compound appearing as the title of each monograph is followed by its Codex Classification Number in parentheses.

References to previous Reports and Evaluations of Joint Meetings are listed in Annex I.

Acknowledgements

The monographs in these Evaluations were prepared by the following participants in the 2004 JMPR, for the FAO Panel of Experts on Pesticide Residues in Food and the Environment:

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JMPR, 2004. Pesticide residues in Food – 2004. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Core Assessment Group on Pesticide Residues, Rome, Italy, 20-29 September 2004. WHO and FAO, Rome, 2004.

CORRIGENDUM TO THE 2003 RESIDUE EVALUATIONS OF JMPR

The 2003 Meeting of JMPR evaluated carbendazim/thiophanate-methyl and drafted two residue monographs and one joint appraisal. In the *Evaluations Part I - Residues* (FAO Plant Production and Protection Paper Vol. 177, 2004) under the title "Carbendazim/thiophanate-methyl" only the evaluation for carbendazim and the appraisal for both compounds were published, the evaluation for thiophanate-methyl was omitted. This evaluation is published in the present volume as corrigendum (see page 1403).

CARBOFURAN (096)/CARBOSULFAN (145)

No new data were available for evaluation. The further review of the data originally evaluated by the Meeting and reviewed in 1999 is described in Section 4.3 of the report of the present Meeting.

CHLORPYRIFOS (017)

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EXPLANATION

At the 25th Session of the CCPR in 1993 (ALINORM 93/24A para. 251) chlorpyrifos was identified for a periodic review, and at the 29th Session in 1997, was scheduled for toxicology in 1999 and for residue chemistry in 2000. The 1999 review confirmed the ADI of 0.01 mg/kg bw and established an acute reference dose (acute RfD) of 0.1 mg/kg bw. For the 2000 residue review, information was reported on the identity and physical properties of the active ingredient and technical material, metabolism in plants and animals, environmental fate, storage stability, animal feeding studies, field trials, GAP (national labels) and fate of residues in processing. The governments of Australia, Germany, The Netherlands, Poland, Thailand and the USA reported additional information, and the Meeting recommended MRLs for several commodities.

Chlorpyrifos was again scheduled for re-evaluation in 2004 to estimate maximum residue levels in cotton, potatoes, rice and soya beans as none had been recommended by the 2000 JMPR for these commodities because of insufficient information on GAP and/or residue data. The 36th Session of the Codex Committee on Pesticide Residues in 2004 agreed that the 2004 JMPR would review data from India to support the establishment of MRLs for tea, and more information was reported to the Meeting by the Government of India.

Information on relevant GAP labels and additional residue data on these commodities were reported to the present Meeting.

RESIDUE ANALYSIS

Analytical methods

Previously submitted methods

The following methods were submitted to the 2000 JMPR:

ACR 73.5.S1 (Wetters, 1976) for the analysis of raw agricultural commodities from supervised trials on cotton (Wetters, 1987) and soya beans (Miller, 1979).

ACR 74.4 (McKellar, 1975a) for the analysis of cotton seed, cotton gin trash and cotton processed products from supervised trials (McKellar and Dishburger, 1974; McKellar, 1975b).

BRC 93.1 (Gagnotto and Balderrama, 1993) for the analysis of raw agricultural commodities from supervised trials on cotton (Balderrama and Matos, 1994b,c), potatoes (Pinheiro *et al.*, 2001a,b) and soya beans (Balderrama and Matos, 1994d).

ERC 94.1 (Khoshab and Hastings, 1994) for the analysis of raw agriculatural commodities from supervised trials (Cowles, 2002).

STM CR 435 (based on ERC 94.1 submitted to 2000 JMPR) for the analysis of seed from supervised trials on cotton seed (Cowles, 2002).

Additional methods

The following methods were used to analyse samples from relevant supervised trials but were not reported to the 2000 JMPR.

BRC 94.3 (Balderrama and Matos, 1994a) was used to analyse potatoes from supervised trials (Balderrama and Matos, 1994e). Chopped samples of potatoes were extracted with acetone and an aliquot of the acetone filtrate was evaporated. A 5% aqueous sodium chloride solution was added and the mixture partitioned twice with hexane. The hexane layers were combined and evaporated to dryness. The residue was reconstituted in hexane and passed through a silica solid-phase extraction column, washed with hexane and the chlorpyrifos eluted with a solution of 5% ether in hexane. The eluate was evaporated to dryness, the residue taken up in hexane and residues of chlorpyrifos determined by gas chromatography with flame photometric detection.

BRC 94.4 (Catta-Preta *et al.*, 1994). The acetone extraction in the above method was slightly modified for the analysis of potatoes (do Amaral, 1999).

BRC 94.4.S1 (Catta-Perrera and Rampazzo, 1995). The above method was slightly modified by incorporating an acetonitrile partition and was used to analyse rice grain from supervised trials (Pinheiro and de Vito, 1999).

GRM 01.22 (Pinheiro and Oliviera, 2001) was used to analyse cotton seed from supervised trials on cotton (Pinheiro *et al.,* 2001e).

BRC 00.2 (Pinheiro, 2000). The method was used to analyse potatoes from supervised trials (Pinheiro and Santos, 2000; Pinheiro, *et al.,* 2001c,d). Chopped samples of potatoes were extracted by maceration with acetone/water (3:1). After centrifuging, an aliquot of the extract was diluted with water to a known volume, then partitioned with hexane. Residues of chlorpyrifos from the hexane extract were determined by gas chromatography with a flame photometric detector.

PA-RM-98-07 (Quin and Utting, 1999). This method was used to analyse raw agricultural commodities and processed products of rice in supervised trials (Cowles 2003a-d; Cowles, *et al.*, 1999a-d).

Ground samples were extracted with acetone. After the addition of water, the chlorpyrifos was partitioned into hexane. An aliquot of the hexane layer was passed through a silica solid-phase extraction column and additional hexane was added to elute chlorpyrifos. All hexane eluates were evaporated and the residue reconstituted with hexane and residues of chlorpyrifos determined by gas chromatography with a flame photometric detector.

"A General Method for Organophosphorous Pesticide Residues in Non-fatty Foods" (Storherr *et al.*, 1971) was used to analyse soya beans from supervised trials (de Baptista, 1996). The acetonitrile extraction procedure used by FDA for chlorinated hydrocarbon pesticides is used. An aliquot of the acetonitrile extract is partitioned three times with methylene chloride. The methylene chloride phase is passed through a short column containing an adsorbent mixture of 1 part acid-treated charcoal, 2 parts deactivated Sea Sorb 43 and 4 parts Celite 545. The eluates are evaporated and reconstituted with ethyl acetate, and chlorpyrifos is determined by gas chromatography using a flame photometric detector.

Recoveries and limits of quantification are given in Table 1.

Table 1. Summary of methods for determination of residues of chlorpyrifos.

Method		Sample	Fortification,	Recovery (%)			LOQ	Field trial ref.
No.	Type		mg/kg	Min	Max	Mean	mg/kg	
Cotton seed								
ACR 73.5.S1	GC/FPD	Cotton seed	0.01-1.0	74	96	86 ± 6	0.01	GH-C 1893
ACR 74.4	GC/FPD	Cotton seed	0.01-0.10	82	120	94 ± 10	0.01	GH-C 739
ACR 74.4	GC/FPD	Cotton seed	0.01-0.10	82	120	93 ± 10	0.01	GH-C 840
		Hulls	0.10	60	73	66 ± 9	0.01	
		Linters	0.01	68	96	85 ± 15	0.01	
		Meal	0.01-0.10	85	112	94 ± 7	0.01	
		Gin trash	0.01-1.0	60	128	88 ± 18	0.01	
ACR 75.1	GC/FPD	Oil	0.01-0.20	68	92	78 ± 8	0.01	
BRC 93.1	GC/EC	Cotton seed	0.01-1.0	72	97	81 ± 3	0.01	GHB-P 195 GHB-P 196
ERC 94.1 (STM CR 435)	GC/MSD	Cotton seed	0.05-0.5	79	89	82 ± 5	0.02	GHF-P 2477
ERC 94.1 (STM CR 435)	GC/MSD	Gin trash	0.05-6	94	121	108 ± 14	0.05	GHF-P 2477
GRM 01.22	GC/FPD	Cotton seed	0.01-3.0	75	110	89 ± 3	0.01	GHB-P 743
Potatoes								
BRC 93.1	GC/FPD	Potato	0.01-1.0	84	95	89 ± 3	0.01	GHB-P 710 GHB-P 711
BRC 94.3	GC/FPD	Potato	0.01-2.0	71	103	89 ± 4	0.01	GHB-P 218
BRC 94.4	GC/FPD	Potato	0.01-0.1	75	102	91 ± 4	0.01	GHB-P 349
BRC 00.2	GC/FPD	Potato	0.01-2.0	98	117	104 ± 4	0.01	GHB-P 462
BRC 00.2	GC/FPD	Potato	0.01-3.0	83	103	90 ± 5	0.01	GHB-P 650 GHB-P 678
Rice								
BRC 94.4.S1	GC/FPD	Rice grain	0.01-1.0	73	88	81 ± 3	0.01	GHB-P 406
PA-RM-98-07	GC/FPD	Rice grain	0.01-0.6	78	122	91 ± 11	0.01	GHF-P 1791
		Rice straw	0.01-10	64	139	96 ± 26	0.01	GHF-P 1792
PA-RM-98-07	GC/FPD	Rice grain	0.10	80	-	-	0.01	GHF-P 1794
		Rice hulls	1.0	110	-	-	0.01	GHF-P 1795
		Rice bran	1.0	72	102	87 ± 21	0.01	
		Brown rice	0.20	74	-	-	0.01	
		White rice	0.10	95	-	-	0.01	
PA-RM-98-07	GC/FPD	Rice grain	0.01-1.0	80	131	96	0.01	GHF-P 2670
		Rice straw	0.01-1.0	83	121	98	0.01	GHF-P 2671 GHF-P 2672
Soya beans								
ACR 73.5.S1	GC/FPD	Soya beans	0.01-0.1	70	110	88 ± 6	0.01	GH-C 1224
		Hulls	0.05	80	-	-	0.01	
		Meal	0.05	82	-	-	0.01	
		Crude oil	0.01	90	-	-	0.01	
		Refined oil	0.01	80	-	-	0.01	
		Soap stock	0.02	100	-	-	0.01	
BRC 93.1	GC/EC	Soya beans	0.01-0.5	85	102	91 ± 2	0.01	GHB-P 189
Manual[1]	HPLC	Soya beans	0.05	78	-	-	0.01	GHB-P 621
Storherr[2]	GC/FPD	Soya beans	0.01	63	-	-	0.01	GHB-P 534

[1] *Manual of Pesticide Residue Analysis*, Vol. I, p. 383, VCH Publishers Inc., New York, NY.

[2] Storrherr *et al.* "A general method for organophosphorous pesticide residues in non-fatty foods", J. Assoc. Off. Anal. Chem., 54(3): 513-516.

Enforcement and multi-residue methods

Enforcement and multi-residue methods were reported to the 2000 JMPR. No additional information was provided.

In a trial in India (Report No. CP-tea-1-04) hand-picked green leaf samples of tea (minimum 70% two leaves and a bud) of about 1–2 kg from treated plots were processed in the tea factory under controlled condition using normal black tea manufacturing processes involving withering, rolling, oxidation (fermentation) and drying. 0.2 kg samples of the raw and processed tea were used for analysis. The method involves re-hydration of dry 10 g tea samples with 40 ml distilled water and extraction with 200 ml mixtures of n-hexane and acetone (4:1) by blending in a high-speed top-mounted blender. The contents were allowed to stand for 5 min. and a 50 ml aliquot of extract was washed with aqueous sodium chloride solution. The residues were partitioned into hexane and the extracts cleaned up with alumina, for quantification by GLC-NPD or ECD on a capillary column, and the procedure was validated by analysing spiked samples fortified at levels of 0.1 to 5 mg/kg. The sensitivity of the method was 0.01 ng and the limit of quantification was 0.02 mg/kg. Information on recoveries was not reported.

Stability of residues in stored analytical samples

No additional information was provided.

USE PATTERN

Chlorpyrifos is an insecticide used as a pre-plant and at-planting seed treatment and as a foliar, directed and dormant spray.

Registered uses of chlorpyrifos are shown in Table 2 for cotton, potatoes, rice, soya beans and tea for which new MRLs are requested.

Table 2. Registered uses of chlorpyrifos on commodities for new MRLs

Crop	Country	Formulation		Application						PHI, days	Comment
		Type	Conc. of ai	Method	Growth stage	No.	kg ai/hl	Water l/ha	kg ai/ha (form./ha)		
Cotton	*Argentina	EC	480 g/l	Broadcast	Post-emergence			80-100 10 aerial	0.96 (2.0 l/ha)	21	
Cotton	Australia	EC	500 g/l	Foliar spray Or in-furrow		Repeat as needed		50 ground 20 aerial	0.15-0.75 (0.3-1.5 l/ha)	28	Label available to support residue trials. In-furrow: row spacing 1 m (QLD, NSW only)
Cotton	Australia	EC	300 g/l	Foliar spray		Repeat as needed 3 max.		50 ground 20 aerial	0.15-1.5 (0.5-5.0 l/ha)	28	Label available to support residue trials. 7-10 days between 2 sprays.
Cotton	Australia	WG	750 g/kg	Foliar spray		Repeat as needed 3 max.		50 ground 20 aerial	0.15-1.5 (0.2-2.0 kg/ha)	28	Label available to support residue trials. 7-10 days between 2 sprays.
Cotton	Brazil	EC	480 g/l	Foliar spray		1-3		100-300	0.14-0.96 (0.3-2.0 l/ha)	21	Label available to support residue trials. 7-14 days between 2 sprays.
Cotton	Columbia	EC	480 g/l	Foliar spray					0.96 2.0 l/ha	20	
Cotton	*Mexico	EC	480 g/l or 445 g/kg	Foliar spray			0.17 (0.35 l/hl)		0.96 (2.0 l/ha)	21	
Cotton	*Spain	EC	480 g/l	Foliar spray			0.096			21	
Cotton	*Spain	WP	250 g/kg	Foliar high volume	Early stage	1	0.10	600 (300 alt label)		21	
Cotton	*Spain	EC	480 g/l	Foliar spray high volume	Flowering	1	0.096	600		21	
Cotton	*Spain	EC	260 g/l	Foliar spray high volume	Flowering	1	0.072	500		21	Formulation with cypermethrin (50 g/kg).
Cotton	*Spain	DP	30 g/kg	Dusting	Flowering	1			0.9	-	

Crop	Country	Formulation		Application						PHI, days	Comment
		Type	Conc. of ai	Method	Growth stage	No.	kg ai/hl	Water l/ha	kg ai/ha (form./ha)		
Cotton	*Spain	GR	50 g/kg	Row Broadcast and incorporated	At planting	1			0.75 row 4.0 broadcast	-	
Cotton	USA	EC	480 g/l (4 lb/gal)	Foliar spray		Repeat as needed 6 max.		15 gal/A min.	0.2-1.12 (0.4-2 pt/A)	14	Label available to support residue trials. Grazing restriction. Do not feed gin trash. May be applied through irrigation sprinklers.
Cotton	USA	WG	750 g/kg (75%w/w)	Foliar spray		Repeat as needed Max. per season 3 lb ai/A		15 gal/A min.	0.2-1.12 (0.25-1.33 lb/A)	14	Label available to support residue trials. Grazing restriction. Do not feed gin trash. May be applied through irrigation sprinklers. 10 days min. between 2 applications
Potatoes	*Argentina	EC	480 g/l	Broadcast	Post-emergence			80-100 10 aerial	0.72 (1.5 l/ha)	21	
Potatoes	*Argentina	EC	480 g/l	Soil treatment, incorporated	Pre-plant			100-150	2.9 (6 l/ha)		
Potatoes	*Argentina	EC	480 g/l	Soil treatment, incorpo-rated	Pre-plant and immediate -ly after hilling up	2		100-150	1.9 (4 l/ha) 1.0 (2 l/ha		
Potatoes	Australia	EC	500 g/l	Soil treatment	Pre-plant, incorpo-rated; at hilling-up	2			0.5-3.0 (1-6l/ha); 0.45 (0.9l/ha)	N/A	
Potatoes	*Australia	EC	500 g/l	Foliar spray					0.25 (0.5-l/ha)		
Potatoes	Brazil	EC	480 g/l	Foliar spray		1-2		100-300	0.72 (1.5 l/ha)	14	Label available to support residue trials. 14 days between 2 sprays.
Potatoes	Brazil	EC	450 g/l	Foliar spray	During any phase of crop developme nt	as require d with 7 days interva ls			0.35-0.90 (0.8-2.0 l/ha)	21	Label available to support residue trials. 7 days between 2 sprays.
Potatoes	Brazil	GR	100 g/kg (10%w/w)	Broadcast along sowing furrows	During planting	1			2.0-3.0 (20-30 kg/ha)	21	Label available to support residue trials.
Potatoes	*Canada	EC	480 g/l	Ground spray, no incorpo-ration	Pre-transplant	1		200	2.4 l/ha (1.15)	7	
Potatoes	*Canada	EC	480 g/l	Ground spray	2-5 leaf	1		400	2.4 l/ha (1.15)	7	
Potatoes	*Canada	EC	480 g/l	Foliar spray		9		400-800	1.0 l/h (0.48)	7	
Potatoes	*Canada	WP	50%	Spray	Seedling 2-5 leaf	1		400	1.125	7	
Potatoes	*Chile	EC	500 g/l	Foliar spray					0.4 l/ha (0.2)	14	Formulation is a mix with cypermethrin (50 g/l).
Potatoes	*Chile	GR	150 g/kg	Broadcast or band (15-18 cm)	Pre-plant/ At planting				3		
Potatoes	*Chile	D	120 g/kg	Mix with fertilizer					0.12		
Potatoes	Columbia	EC	480 g/kg						0.48-1.92 (1-4l/ha)	20	
Potatoes	*France	G	50 g/kg	Broadcast	Pre-plant	1			1.25		
Potatoes	*Italy	EC	480 g/l	Foliar spray		Repeat		600	1.6 l/ha	15	

Crop	Country	Formulation		Application						PHI, days	Comment
		Type	Conc. of ai	Method	Growth stage	No.	kg ai/hl	Water l/ha	kg ai/ha (form./ha)		
						as needed			(0.77)		
Potatoes	*Italy	EC	225 g/l	Foliar spray	Post-flowering	2		600	0.56	30	
Potatoes	*India	EC	200 g/l	Foliar spray				500	0.50		35 days PHI pending
Potatoes	Philippines	EC	300 g/l	Foliar spray			0.047-0.066 (25-35 ml per 16l water)				
Potatoes	*Poland	EC	268 g/kg	Foliar, high volume		2	0.28 l/hl (0.075)	150	0.42 l/ha (0.11)	30	Formulation with dimethoate (22.2%)
Potatoes	*Poland	EC	500 g/l	Foliar spray		2	0.20 l/hl (0.1)	150	0.3 l/ha (0.15)	30	Formulation with cypermethrin (50 g/kg).
Potatoes	*Portugal	EC	480 g/l	Foliar spray, high volume	Fruiting	2	0.096	500 (1000 alt label)		14	
Potatoes	*Portugal	GR	50 g/kg	Broadcast or in-row	At planting	1		5.0 broad-cast 1.25 in-row			
Potatoes	*South Africa	EC	480 g/l	Spray, with good ground coverage	Pre-plant; Post-plant	1 pre; multiple post at 2-3 week intervals	(0.24)pre-plant	500 post	(0.72) pre-plant for 1 m row spacing. 0.5 l/ha post plant (0.24)	7	Apply pre-plant in a 100 mm band just before closing furrows, 15 ml/100m row length in 3 l water.
Potatoes	*South Africa	EC	480 g/l	Spray	Immediate-ly before tuber initiation and at 2 week intervals	Multiple		500 increase with crop density	1l/ha (0.48)	7	
Potatoes	*Spain	EC	480 g/l	Foliar spray			0.096	300		21	
Potatoes	*Spain	EC	260 g/l	Foliar spray high volume	40 cm high	1	0.072	500		21	Formulation with cypermethrin (50 g/kg).
Potatoes	*Spain	WP	250 g/kg	Broadcast spray, high volume			0.10	300		21	
Potatoes	*Spain	WP	240 g/kg	Foliar high volume	40 cm high	1	0.048	750		21	Formulation with carbaryl (375 g/kg).
Potatoes	*Spain	EC	480 g/l	Foliar spray high volume	20-40 cm high	1	0.096	500		21	
Potatoes	*Spain	GR	50 g/kg	Row Broadcast	At planting	1			1.5 row 5 broadcast	-	
Potatoes	*UK	EC	480 g/l	Overall volume spray				200	0.72	21	
Potatoes	*Uruguay	EC	480 g/l	Soil treatment, band/furrow	At planting				1.5 l/ha (0.72)		
Potatoes	*Uruguay	EC	480 g/l	Broadcast soil treatment, incorporate 10 cm	Pre-plant				3.5 l/ha (1.7)		
Potatoes	*Uruguay	EC	480 g/l	Foliar spray		15-20 day repeat interval			1.5 l/ha (0.72)		
Rice	Australia	EC	500 g/l	Foliar spray					0.03-0.75 (0.6-1.5L)	10	Maximum rate when water >15 cm or high amount of

Crop	Country	Formulation		Application						PHI, days	Comment
		Type	Conc. of ai	Method	Growth stage	No.	kg ai/hl	Water l/ha	kg ai/ha (form./ha)		
											decaying material.
Rice	Columbia	EC	480 g/l	Foliar spray		Repeat as needed 3 max.			0.34-0.96 (0.7-2.0 l/ha)	20	Label available to support residue trials.
Rice	India	EC	200 g/l	Foliar spray		1		500-1000	0.25-0.375 (1.25-1.875 l/ha)	14	Label available to support residue trials.
Rice	*India	EC	200 g/l	Seedling root dip			0.02% solution				
Rice	India	G	10 g/kg	Broadcast					1.0 (10 kg/ha)		Allow 2-3 cm standing in the field and keep water impounded for 2-3 days after application.
Rice	*Mexico	EC	480 g/l or 445 g/kg	Foliar spray				250 ground 50 aerial	0.6 (1.25 l/ha)		21 day grazing restriction
Rice	Philippines	EC	300 g/l	Foliar spray		Repeat as needed 3 max.	0.047-0.066 (25-35ml per 16l water)			25	Label available to support residue trials.
Rice	Thailand	EC	400 g/l (40%w/v)	Foliar spray		Repeat as needed 3 max.	0.08 (40cc per 20l water)			7-14	Label available to support residue trials.
Rice	Thailand	EC	400 g/l (40%w/v)	Seed treatment (for breeding only)		Coat rice or soak rice sack	0.2-0.3% solution				
Rice	Vietnam	EC	300 g/l	Foliar spray		1		500-800	0.3-0.42	10	Label available to support residue trials.
Soya beans	Australia	EC	500 g/l	Bait	At planting				0.1 L per 2.5 kg bait/ha		Bait is sorghum or wheat
Soya beans	*Argentina	EC	480 g/l	Broadcast	Post-emergence			80-100 10 aerial	0.96 (2.0 l/ha)	45	
Soya beans	Brazil	EC	480 g/l	Foliar spray		1-2		100-300	0.12-0.48 (0.25-1.0 l/ha)	21	Label available to support residue trials. 7-14 days between applications.
Soya beans	*France	EC	300 g/l	Low volume broadcast, incorporate	Pre-plant	1		150	1.5		
Soya beans	*Italy	EC	480 g/l 225 g/l	Foliar spray	Milky ripe			800	0.58 (1.2 l/ha)	120	
Soya beans	*Italy	GR	75 g/kg	Row localized. Broadcast	At planting At-transplant At earthing up	1			1.2; 3 broadcast	120	
Soya beans	*Mexico	EC	480 g/l or 445 g/kg	Foliar spray					0.72 (1.5 l/ha)	21	
Soya beans	Thailand	EC	400 g/l	Foliar spray		2-3	0.1 (50cc per 20L water)			7	7-10 days retreatment interval. 14 day PHI for undefined conditions.
Soya beans	*Uruguay	EC	500 g/l	Foliar spray		15-20 day repeat interval		80 ground 25 aerial	0.38 (0.75 l/ha)	45	Formulation includes cypermethrin (50 g/l)
Soya beans	USA	EC	480 g/l (4 lb/gal)	Soil spray. 4-6 in. band	Pre-plant through			94 (10	0.56-1.12 (1-2 pt/A)		Do not apply in-furrow. 36 in. row spacing requires

Crop	Country	Formulation		Application						PHI, days	Comment
		Type	Conc. of ai	Method	Growth stage	No.	kg ai/hl	Water l/ha	kg ai/ha (form./ha)		
				at planting; 9-12 in. band at post-emergence.	post-emergence			gal/A)			8.8 oz of spray per 100 feet or row.
Soya beans	USA	EC	480 g/l (4 lb/gal)	Foliar spray		Max. per season 3 lb ai/A (6 pt/A)			0.28-1.12 (0.5-2 pt/A)	28	Label available to support residue trials. Last two treatments minimum of 14 days apart. Grazing /feeding restriction. Only 1 application after pod set on determinate soya beans. May be applied with sprinkler irrigation.
Soya beans	USA	WG	750g/kg (75%w/w)	Soil spray. 4-6 in. band at planting; 9-12 in. band at post-emergence.	Pre-plant through post-emergence			94 (10 gal/A)	0.56-1.12 (0.67-1.33 lb/A)		Do not apply in-furrow. 36 in. row spacing requires 8.8 oz of spray per 100 feet or row.
Soya beans	*USA	WG	750g/kg (75%w/w)	Foliar spray		Max. per season 3 lb ai/A (6 pt/A)			0.28-1.12 (0.33-1.33 lb/A)		Last two treatments minimum of 14 days apart. Grazing /feeding restriction. Only 1 application after pod set on determinate soya beans. May be applied with sprinkler irrigation.
Soya beans	USA	GR	150g/kg (15%w/w)	Band incorporate	At planting Post-plant	1			8 oz per 1000 ft row, 8.7 lb/a (1.5) for 30 in row spacing.		Do not apply as an in-furrow treatment.
Tea	India	EC	200 g/l	Foliar spray, high volume	Active vegetative growth stage	1	0.05	400	0.200	7	

*: Data from 2000 JMPR monograph.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Cotton

Trials on cotton were reported to the 2000 JMPR but no MRLs were recommended by the Meeting because there were not enough trials. Additional residue data from six supervised trials on cotton in Australia and Brazil were reported to the present Meeting and the information combined with that from the trials previously evaluated by the 2000 JMPR.

Australia. In field trials in 2000 at three sites (Cowles, 2002: Report No. GHF-P 2477) bridging studies were conducted using a water-dispersible granule containing 750 g ai/kg and an emulsifiable concentrate containing 300 g ai/l. Three foliar broadcast applications of each formulation at the rate of 1.5 kg ai/ha per application were made at 5-8 day intervals at growth stages from late flowering to 15% of the bolls being open. The last application was made 17 or 28 days before harvest. A double rate of 3.0 kg ai/ha per application was also included in the trials.

Seed cotton and cotton trash were hand-harvested from the control and treated plots with a 17-day PHI at 2 sites and 28-day PHI at the third. The seed cotton was ginned to generate the undelinted samples, and seed and cotton trash samples were placed in polyethylene bags and stored frozen. The interval before analysis was from 7 to 8 months, and residues of chlorpyrifos were determined by GC-

MS using Amdel in-house method STM CR 435 (based on Dow AgroSciences Method ERC 94.1). Procedural recoveries were 79-89% (82% average) for cotton seed and 94-121% (108% average) for cotton trash. The limits of quantification were 0.02 mg/kg for cotton seed and 0.05 mg/kg for cotton trash.

Brazil. Two field trials were conducted in 1992 at two sites (Balderrama and Matos, 1994b,c: Report Nos. GHB-P 195 and 196), the results of which were reported to the 2000 JMPR and accepted as being according to GAP. In summary, foliar broadcast applications of chlorpyrifos EC 480 g ai/l were applied at the rate of either 0.96 kg ai/ha or 1.92 kg ai/ha (double the maximum GAP rate) per application, twice in one trial and three times in the other.

Seed samples from the control and treated plots were harvested 21 days after the final application and stored frozen at –20°C for 4 to 5 months. Residues of chlorpyrifos were determined by GC/EC using Dow AgroSciences Method BRC 93.1. Procedural recoveries were 72-97% (81% average) with a limit of quantification of 0.01 mg/kg.

In three further field trialsin 2000 at three sites (Pinheiro *et al.*, 2001e: Report No. GHB-P 743) three foliar broadcast applications of chlorpyrifos EC 480 g ai/l were made at the rate of 0.96 kg ai/ha or 1.92 kg ai/ha (double GAP rate) per application at growth stages from plant emergence to 30% of the bolls being open. Two replicate plots were treated at each field site.

Samples were hand-harvested from control and treated plots at 0, 7 (or 5), 14 (or 15), 21 (or 22) and 28 days after the last application and ginned to generate the undelinted cotton seed samples. The samples were placed in polyethylene bags and stored frozen at –20°C for a maximum of about 3 months. Residues of chlorpyrifos were determined by GC/FPD Dow AgroSciences Method GRM 01.22. The procedural recoveries were 75-100% (89% average) with a limit of quantification of 0.01 mg/kg.

USA. Field trials on cotton were conducted in 1973, 1974 and 1986 (McKellar and Disburger, 1974: Report No. GH-C 739; McKellar, 1975b: Report No. GH-C 840; Wetters, 1987: Report No. GH-C 1893), the results of which were reported to the 2000 JMPR and three trials were accepted as being conducted according to GAP. In summary, chlorpyrifos EC 480 g ai/l or (4 lb ai/gal) was applied as a foliar broadcast. At a Mississippi site in 1973 nine applications were made at the rate of either 1.12 kg ai/ha or 2.24 kg ai/ha per application. Samples were hand-harvested 0, 3, 7 and 14 days after the last application and ginned to generate undelinted cotton seeds. Samples of gin trash were also collected 3 days after the last application. At another Mississipi site in 1974 two applications were made at the rate of 0.28 kg ai/ha per application followed by twelve at 1.12 kg ai/ha per application. Undelinted cotton seed samples were taken 15 days after the last application. At a California site in 1986 5 applications were made at 1.12 kg ai/ha and undelinted seed samples were taken 14 days after the last application.

All samples were stored frozen until analysis, and residues of chlorpyrifos were determined by GC/FPD using either Dow AgroSciences Method ACR 73.5.S1 or Method ACR 74.4 with procedural recoveries of 74-96% (86% average) and 82-120% (94% average) respectively. The limit of quantification was 0.01 mg/kg for both methods.

Table 3. Chlorpyrifos residues in cotton from supervised trials in Australia, Brazil and the USA.

Country, year (variety)	Application (rate per application)				PHI		Residue	
	Form.	kg ai/ha	Water, l/ha	No.	days	Sample	mg/kg	Report No.
GAP – Australia	**EC 300 EC 500 WG 750**	**0.15-1.5 0.15-0.75 0.15-1.5**		**3**	**28**			
Australia,	WG 750	1.5	100	3	28	seed	0.05	GHF-P 2477

Country, year (variety)	Application (rate per application)				PHI		Residue	
	Form.	kg ai/ha	Water, l/ha	No.	days	Sample	mg/kg	Report No.
2000 (Not recorded)					28	trash	8.80	
		3.0	100	3	28	seed	0.11	
					28	trash	11.0	
	EC 300	1.5	100	3	28	seed	0.03	GHF-P 2477
					28	trash	10.3	
		3.0	100	3	28	seed	0.08	
					28	trash	21.6	
Australia, 2000 (Sioka V161)	WG 750	1.5	100	3	17	seed	0.07	GHF-P 2477
					17	trash	23.9	
		3.0	100	3	17	seed	0.15	
					17	trash	18.3	
	EC 300	1.5	100	3	17	seed	0.12	GHF-P 2477
					17	trash	19.0	
		3.0	100	3	17	seed	0.36	
					17	trash	sample missing	
Australia, 2000 (Sicot 198i)	WG 750	1.5	100	3	17	seed	0.11	GHF-P 2477
					17	trash	29.7	
		3.0	100	3	17	seed	0.09	
					17	trash	32.5	
	EC 300	1.5	100	3	17	seed	0.07	GHF-P 2477
					17	trash	19.4	
		3.0	100	3	17	seed	0.20	
					17	trash	28.2	
GAP – Brazil	EC 480	0.12-0.96		**1-3**	**21**			
Brazil, 1992 (IAPAR-45)	EC 480	0.96	100	3	21	seed	0.07	GHB-P 195
		1.92	100		21	seed	0.44	(Previously submitted[1])
Brazil, 1992 (IAC-20)	EC 480	0.96	630	2	21	seed	0.02	GHB-P 196
		1.92	630	2	21	seed	0.04	(Previously submitted[1])
Brazil, 2001 (IAC-22)	EC 480	0.96	100	3	0	seed	0.22, 0.14	GHB-P 743
					7	seed	0.03, 0.07	
					14	seed	0.03, 0.04	
					21	seed	0.02, 0.03	
					28	seed	0.02, 0.02	
		1.92	100	3	0	seed	0.27, 0.42	
					7	seed	0.06, 0.07	
					14	seed	0.07, 0.05	
					21	seed	0.06, 0.06	
					28	seed	0.06, 0.04	
Brazil, 2001, (Delta Opal)	EC 480	0.96	100	3	0	seed	0.24, 0.48	GHB-P 743
					5	seed	0.03, 0.04	
					15	seed	0.01, 0.01	
					21	seed	0.01, 0.01	
					28	seed	<0.01, <0.01	
		1.92	100	3	0	seed	0.77, 0.48	
					5	seed	0.05, 0.05	
					15	seed	0.03, 0.02	
					21	seed	1.15[2], 1.07[2]	
					28	seed	0.02, 0.01	
Brazil, 2001 (CD 404)	EC 480	0.96	137-149	3	0	seed	0.03, 0.03	GHB-P 743
					7	seed	<0.01, <0.01	
					14	seed	<0.01, 0.02	
					22	seed	<0.01, 0.01	

Country, year (variety)	Application (rate per application)				PHI		Residue	
	Form.	kg ai/ha	Water, l/ha	No.	days	Sample	mg/kg	Report No.
			137-149		28	seed	0.01, <0.01	
		1.92		3	0	seed	0.05, 0.02	
					7	seed	0.01, 0.02	
					14	seed	<0.01, <0.01	
					22	seed	0.02, <0.01	
					28	seed	<0.01, 0.01	
GAP – USA	**EC 480**	**0.21-1.12**		**2-6**	**14**			
USA, 1973 Wayside, MS (DPL 16)	EC 480	1.12	94	9	0	seed	1.4, 0.94, 0.97, 1.1	GH-C 739 (Previously submitted[1])
					3	seed	0.28, 0.37, 0.48, 0.47	
					7	seed	0.02, 0.02, 0.02	
					14	seed	0.16, 0.02, 0.03	
		2.24	94		0	seed		
					3	seed	3.0, 3.7, 1.8, 2.7	
					7	seed	0.63, 0.54, 0.66, 1.1	
					14	seed	0.07, 0.04, 0.07	
							0.05, 0.14, 0.04	
USA, 1974 Wayside, MS (Stoneville 213)	EC 480	0.28 x 2 1.12 x 12	94	14	15	seed	0.04, 0.07, 0.03, 0.04	GH-C 840 (Previously submitted[1])
USA 1986 CA (Acala SJ-5)	EC 480	1.12	280	5	14	seed	0.13, 0.18, 0.17, 0.11	GH-C 1893 (Previously submitted[1])

The highest residue from replicate plots was chosen for STMR estimation as a worst-case scenario
[1] Previously submitted to 2000 JMPR and accepted as being according to GAP.
[2] Suspected as outliers, residues were high and inconsistent with results from other locations.

Potatoes

Brazil. At the 2000 JMPR, the results of trials on potatoes were considered but no MRLs were recommended because there were too few trials. Additional data from supervised trials in Brazil in which applications were at-plant (soil in-furrow) have now been reported. Proposed MRLs based on the residue data from at-plant application would also cover residues from the foliar application. The previous data for at-plant application are provided again here to enable evaluation of residues from all supervised trials conducted in Brazil.

Four field trials were conducted in 1993-1994 (Balderrama and Matos, 1994e Report No. GHB-P 218; do Amaral, 1999 Report No. GHB-P 349) and reported to the 2000 JMPR were accepted as being conducted according to GAP. Either a granular formulation containing 100 g ai/kg or EC containing 450 g ai/l was used. A single application was made to the soil in-furrow at planting at the rate of 1.5, 3.0 or 6.0 kg ai/ha for the granular formulation and 2.9 or 5.9 kg ai/ha for the EC formulation.

Potatoes were collected manually from the control and treated plots at normal harvest 103-124 days after application. The samples were stored frozen at –20°C for 1 to 13 months. Residues of chlorpyrifos were determined by GC/FPD using Dow AgroSciences Method BRC 94.3 or BRC 94.4. Procedural recoveries were 71-103% (89% or 91% average) with a limit of quantification of 0.01 mg/kg. Details are given in Table 4.

In 1999-2000 seven field trials were conducted to provide additional data. In all seven trials a single application to the soil in furrow at planting was made. In four a granular formulation was

applied at the rate of 3 kg ai/ha or 6.0 kg ai/ha (Pinheiro and Santos, 2000 Report No. GHB-P 462; Pinheiro *et al.*, 2001b,d Report Nos. GHB-P 711 and 678), and in the other three EC 450 g ai/l was applied at 2.7 or 5.4 kg ai/ha (Pinheiro *et al.,* 2001a Report No. GHB-P 710; 2001c Report No. GHB-P 650). There were three replicate plots for each treatment.

Three samples of potatoes were collected manually at normal harvest from the control and treated plots 100-124 days after application and stored frozen at –20°C for a maximum of about 7 months. Residues of chlorpyrifos were determined using Dow AgroSciences Method BRC 93.1, BRC 94.3 or 00.2. The procedural recoveries were 71-111% (89%, 90% or 104% average) with a limit of quantification of 0.01 mg/kg. Details are given in Table 4.

Table 4. Chlorpyrifos residues in potatoes after single at-plant applications in supervised trials in Brazil.

Country, year (variety)	Application			PHI, days	Residue, mg/kg	Report No.
	Form.	kg ai/ha	Water, l/ha			
GAP – Brazil	**GR 100 EC 450**	**2 – 3**				
Brazil, 1993 (Achat)	GR 100	1.5		124	0.20, 0.03, 0.19	GHB-P 218 (Previously submitted[1])
		3.0		124	0.29, 0.10, 0.14	
		6.0		124	0.29, 0.08, 0.05	
Brazil, 1993 (Chatti)	GR 100	1.5		105	0.17, 0.13, 0.07	GHB-P 218 (Previously submitted[1])
		3.0		105	0.12, 0.51, 0.23	
		6.0		105	0.96, 0.66, 0.46	
Brazil, 1994 (Blutje)	EC 450	2.93	300	105	0.02, <0.01, 0.02	GHB-P 349 (Previously submitted[1])
		5.85	300	105	0.03, 0.06, 0.01	
Brazil, 1995 (Contenda)	EC 450	2.93	200	103	0.03, 0.13, 0.08	GHB-P 349 (Previously submitted[1])
		5.85	200	103	0.27, 0.18, 0.22	
Brazil, 1999 (Bintje)	GR 100	3.0		102	0.30, 0.56, 0.53	GHB-P 462
				112	0.22, 0.65, 0.26	
				123	0.18, 0.13, 0.51	
		6.0		102	0.22, 0.09, 0.50	
				112	0.42, 0.28, 0.43	
				123	0.61, 0.32, 0.71	
Brazil, 2000 (Bentje)	EC 450	2.7	200	100	0.53, 0.58, 0.31	GHB-P 650
				110	0.43, 0.43, 0.36	
				120	0.04, 0.05, 0.43	
		5.4	200	100	0.47, 0.78, 1.03	
				110	0.97, 0.72, 0.91	
				120	0.94, 0.47, 0.58	
Brazil, 2000 (Monalisa)	EC 450	2.7	200	101	0.22, 0.35, 0.59	GHB-P 650
				113	0.19, 0.11, 0.29	
				121	0.57, 0.29, 0.35	
		5.4	200	101	0.68, 0.50, 0.69	
				113	0.24, 0.25, 0.23	
				121	0.23, 0.09, 0.21	
Brazil, 2000 (Bintje)	GR 100	3.0		100	0.80, 0.67, 0.87	GHB-P 678
				110	0.71, 0.71, 0.78	
				120	0.03, 0.06, 0.07	
		6.0		100	0.86, 0.83, 1.03	
				110	1.07, 0.93, 0.92	
				120	0.06, 0.13, 0.14	

Country, year (variety)	Application			PHI, days	Residue, mg/kg	Report No.
	Form.	kg ai/ha	Water, l/ha			
Brazil, 2000 (Monalisa)	GR 100	3.0		101 113 121	0.42, 0.24, 0.64 0.57, 0.69, 0.31 0.10, 0.08, 0.26	GHB-P 678
		6.0		101 113 121	0.16, 0.11, 0.68 0.76, 1.56, 1.06 0.83, 0.79, 0.37	
Brazil, 2000 (Monalisa)	EC 450	2.7	100	104 115 124	0.10, 0.04, 0.10 0.05, 0.06, 0.05 0.09, 0.06, 0.09	GHB-P 710
		5.4	100	104 115 124	0.63, 0.20, 0.20 0.11, 0.28, 0.18 0.25, 0.14, 0.12	
Brazil, 2000 (Monalisa)	GR 100	3.0		104 115 124	0.01, <0.01, <0. ND, 0.01, <0.01 <0.01, 0.03, 0.02	GHB-P 711
		6.0		104 115 124	0.06, 0.03, <0.01 0.04, 0.09, 0.01 0.07, 0.01, 0.10	

The highest residue from replicate plots was chosen for STMR estimation as a worst-case scenario.
[1] Previously submitted to JMPR and accepted as meeting GAP.

Rice

The 2000 JMPR was not able to recommend an MRL for rice because no residue trials reported to it were conducted according to available relevant GAP. However those results from Colombia, the Philippines and Vietnam which accorded with current GAP are included here for ease of reference. Data from more recent supervised trials in India and Thailand were reported to the present Meeting.

Colombia. In two field trials in 1998 at two sites (triplicate plots at each) (Pinheiro and De Vito, 1999 Report No. GHB-P 406) chlorpyrifos EC 480 g ai/l was applied three times to upland rice. The first application was after germination at the rate of 0.96 kg ai/ha, the second at the tillering crop growth stage at 0.72 kg ai/ha and the third 20-21 days before harvest at 0.384 kg ai/ha when 100% of pinnacles were visible.

Rice samples from the control and treated plots were hand-harvested 20-21 days after the last application. Soon after harvest, the samples were hand-threshed, packed in polyethylene bags and stored at about –20°C. The interval between sampling and analysis ranged from 4 to 5 months. Residues of chlorpyrifos were determined by GC/FPD using Dow AgroSciences Method BRC 94.4.S1. The procedural recoveries were 73-88% (81% average) with a limit of quantification of 0.01 mg/kg (Table 5).

Philippines. In two field trials in 1998 at two sites (Cowles, *et al.*, 1999a Report No. GHF-P 1791) chlorpyrifos EC 300 g ai/l was applied three times at the rate of 0.3 kg ai/ha per application, the first two applications 25 days and 40 days after transplanting, and the last 25 days before harvest.

Grain and straw samples from the control and treated plots were harvested 25 days after the last application and the samples stored frozen. The interval between sampling and analysis was a maximum of 3 months. Residues of chlorpyrifos were determined by GC/FPD using Dow AgroSciences Method PA-RM-98-07. The procedural recoveries were 78-122% (91% average) for grain and 64-139% (96% average) for straw with a limit of quantification of 0.01 mg/kg. In addition, moisture was determined in the straw and the residues were reported on a dry basis (Table 5).

Vietnam. In two field trials in 1998 at two sites (Cowles, *et al*. 1999b Report No. GHF-P 1792) chlorpyrifos EC 300 g ai/l was applied at a rate of 0.42 kg ai/ha.

Grain and straw samples from the control and treated plots were harvested 10 days after the last application and stored frozen until analysis. The interval between sampling and analysis was a maximum of 4 months. Residues of chlorpyrifos were determined by GC/FPD using Dow AgroSciences Method PA-RM-98-07. The procedural recoveries were 78-122% (91% average) for grain and 64-139% (96% average) for rice straw with a limit of quantification of 0.01 mg/kg. Moisture was determined in the straw and the residues were reported on a dry-weight basis (Table 5).

Thailand. In 2002 in three field trials on rice (Cowles, 2003a-c Report Nos. GHF-P 2670-2672) chlorpyrifos EC 400 g ai/l was sprayed three times as a foliar application at the rate of 400 g ai/ha per application. The first application was made at mid-booting and the final application was at seed formation.

Samples of grain and straw from the control and treated plots were hand-harvested 7, 14 and 21 days after the last application, placed in double polyethylene bags, and stored frozen until analysis. The interval between sampling and analysis ranged from <1 to 3 months. Residues of chlorpyrifos were determined by GC/FPD using Dow AgroSciences Method PA-RM-98-07. The procedural recoveries were 80-131% (average 96%) for grain and 83-121% (average 98%) for straw with a limit of quantification of 0.01 mg/kg for both. The results are shown in Table 5.

India. In 2002 in three supervised field trials at three sites (Cowles, 2003d Report No. INDIA-CHP-RICE-1) single foliar applications of chlorpyrifos EC 200 g ai/l were made at a rate of 375 g ai/ha. Samples of grain and straw from the control and treated plots were collected 14 or 15, 21 and 30 days after application. In one of the trials processed fractions of husk, polished grain and bran were also collected. The results are discussed in the section on the effects of processing.

All samples were stored frozen at approximately –20°C and the interval between sampling and analysis was a maximum of 2 months. Residues of chlorpyrifos were determined by GC/ECD or GC/NPD. The procedural recoveries were 80-102% for grain and 84-116% for straw. The limit of quantification was 0.01 mg/kg for all samples. The results are shown in Table 5.

Table 5. Chlorpyrifos residues in rice from supervised trials in Colombia, the Philippines, Vietnam, Thailand and India.

Country, year (variety)	Rate per application					PHI days	Sample	Residue mg/kg	Reference (Report No.)
	Form.	kg ai/ha	kg ai/hl	Water l/ha	No.				
GAP – Colombia	**EC 480**	**0.34-0.96**				**20**			
Colombia, 1998 (Oryzica)	EC 480	0.96+0.72 +0.38		250	3	20	grain	0.09, 0.09, 0.19	GHB-P 406 (Previously submitted[I])
250	EC 480	0.96+0.72 +0.38			3	21	grain	0.08, 0.08, 0.08	GHB-P 406 (Previously submitted[I])
GAP – Philippines	**EC 300**	**0.30**	**0.047 - 0.066**			**25**			
Philippines, 1998 (Not recorded)	EC 300	0.30			3	25 25	grain straw	0.02 0.19	GHF-P 1791 (Previously submitted[I])
Philippines, 1998 (Not recorded)	EC 300	0.30			3	25 25	grain straw	0.16 0.45	GHF-P 1791 (Previously submitted[I])

Country, year (variety)	Rate per application					PHI days	Sample	Residue mg/kg	Reference (Report No.)
	Form.	kg ai/ha	kg ai/hl	Water l/ha	No.				
GAP – Vietnam	**EC 300**	**0.3-0.42**			**1**	**10**			
Vietnam, 1998 (OMCS 85)	EC 300	0.42		500	1	10 10	grain straw (dry)	0.15 1.83	GHF-P 1792 (Previously submitted[1])
Vietnam, 1998 (Not recorded)	EC 300	0.42		500	1	10 10	grain straw (dry)	0.28 2.33	(GHF-P 1792 (Previously submitted[1])
GAP – Thailand	**EC 400**	**0.40**	**0.08**			**7-14**			
Thailand, 2002 (Suphan)	EC 400	0.40		200	3	7 14 21 7 14 21	grain grain grain straw (dry) straw (dry) straw (dry)	0.08 0.02 0.01 3.10 0.78 0.37	GHF-P 2670
Thailand, 2002 (Suphanburi 50)	EC 400	0.40		200	3	7 14 21 7 14 21	grain grain grain straw (dry) straw (dry) straw (dry)	0.06 0.04 0.04 2.52 1.46 0.66	GHF-P 2671
Thailand, 2002 (Pathumthani)	EC 400	0.40		500	3	7 14 21 7 14 21	grain grain grain straw (dry) straw (dry) straw (dry)	0.09 0.04 0.05 2.21 0.64 0.89	GHF-P 2672
GAP – India	**EC 200**				**1**	**14**			
India, 2002 (Khitish)	EC 200	0.375 0.375 0.375		500 500 500	1 1 1	14 14 21 21 30 30	grain straw grain straw grain straw	0.40 4.53 0.30 2.53 0.21 1.70	INDIA-CHP-RICE-1
India, 2002 (Tella Hamsa)	EC 200	0.375 0.375 0.375		500 500 500	1 1 1	15 15 21 21 30 30	grain straw grain straw grain straw	0.06 0.07 0.04 0.05 0.03 0.03	INDIA-CHP-RICE-1
India, 2002 (PR 117)	EC 200	0.375 0.375 0.375		500 500 500	1 1 1	14 14 21 21 30 30	grain straw grain straw grain straw	0.26 1.73 0.19 0.92 0.17 0.87	INDIA-CHP-RICE-1

The highest residue from replicate plots was chosen for STMR consideration as a worst-case scenario.
[1] Previously submitted to JMPR and accepted as meeting GAP

Soya beans.

At the 2000 JMPR data from five supervised trials were considered but no MRLs or STMRs were established by the Meeting because five trials are insufficient. However the results are included for convenience as are those from previously unreported trials from Brazil conducted in 1994-1996, and those conducted in 1975-76 in the USA and reported in 2000.

Brazil. In 1992-1993 in two field trials chlorpyrifos EC 480 g ai/l was applied three times at either 0.48 kg ai/ha or 0.96 kg ai/ha (2x label rate) per application (Balderrama and Matos, 1994d Report No. GHB-P 189). Soya beans from the control and treated plots were hand-harvested 20-21 days after the third application and stored at –20° C for <1 to 3 months until analysis. Residues of chlorpyrifos were determined by GC/EC using Dow AgroSciences Method BRC 93.1. The procedural recoveries were 85-102% (91% average) with a limit of quantification of 0.01 mg/kg. The results are shown in Table 6.

In 1992-1993 an additional field trial was conducted using chlorpyrifos EC 480 g ai/l applied three times at either 0.48 kg ai/ha or 0.96 kg ai/ha (2x rate) per application (de Baptista, 1996 Report No. GHB-P 534). Soya beans from the control and treated plots were hand-harvested 21 days after application and stored at –18°C until analysis. Residues of chlorpyrifos were determined by GC/FPD using a general method for organophosphorus pesticides by Storherr, R.W. *et al.,* 1971. The procedural recovery was 63% with a limit of quantification of 0.01 mg/kg. The results are shown in Table 6.

In 1995 in a single field trial chlorpyrifos EC 480 g ai/l applied at either 0.34 kg ai/ha or 0.77 kg ai/ha (Tornisielo, 1995 Report No. GHB-P 621). Soya beans from the control and treated plots were harvested 21 days after application and stored at –18°C until analysis. Residues of chlorpyrifos were determined by HPLC using a method from the "Manual of Pesticide Residue Analysis", Vol. I p. 383. The procedural recovery was 78% with a limit of quantification of 0.01 mg/kg. The results are shown in Table 6.

USA. Five supervised field trials conducted according to GAP in 1975-1976 (Miller, P.W. 1979 Report No. GH-C 1224) using chlorpyrifos EC 480 g ai/l (4 lb ai/gal) applied once as a directed broadcast spray at crop emergence (0.56-2.2 kg ai/ha) followed by 3-4 foliar broadcast applications (0.56-1.1 kg ai/ha) during the growing season.

Soya beans were collected from the control and treated plots at normal harvest, 28-31 days after the last application and stored at –18°C until analysis. In some trials, green forage and straw were also collected but the residue data were insufficient for evaluation. Residues of chlorpyrifos were determined by GC/FPD Dow AgroSciences Method ACR 73.5.S1. The procedural recoveries were 70-110% (88% average) with a limit of quantification of 0.01 mg/kg. The results are shown in Table 6.

Table 6. Chlorpyrifos residues in soya beans from supervised trials in Brazil and the USA.

Country, year (variety)	Application				PHI	Residue[1]	Report No.
	Form.	kg ai/ha	Water l/ha	No.	days	mg/kg	
GAP – Brazil	**EC 480**	**0.12-0.48**		**1-2**	**21**		
Brazil, 1993 (Paranaiba)	EC 480	0.48	100	3	20	<0.01, <0.01, <0.01	GHB-P 189
		0.96	100	3	20	<0.01, <0.01, <0.01	
Brazil, 1993 (BR-16)	EC 480	0.48	100	3	21	<0.01, <0.01, <0.01	GHB-P 189
		0.96	100	3	21	<0.01, <0.01, <0.01	
Brazil, 1993 (BR-16)	EC 480	0.48	100	3	21	<0.01	GHB-P 534
		0.96	100	3	21	<0.01	

Country, year (variety)	Application				PHI	Residue[1]	Report No.
	Form.	kg ai/ha	Water l/ha	No.	days	mg/kg	
Brazil, 1995 (LAC-20)	EC 480	0.384 0.768		1 1	21 21	<u><0.01</u> <0.01	GHB-P 621
GAP – USA	**EC 480** **WG 750**	**0.28-1.12**		**Total 3.37 kg ai/ha**	**28**		
USA/MS, 1975 (Tracy)	EC 480	0.56 (2x) + 1.1 (2x)	94	4	28	<u><0.01,</u> <0.01, <0.01	GH-C 1224 Previously submitted[2]
USA/GA, 1975 (Hutton)	EC 480	2.2[3] + 0.56 (2x) + 1.1 (2x)	234	5	30	<u><0.01,</u> <0.01, <0.01	GH-C 1224 Previously submitted[2]
USA/IL, 1975 (Corsey)	EC 480 g/l	2.2[3] + 0.56 (2x) + 1.1 (2x)	281	5	28	<u>0.01,</u> 0.01, <0.01	(GH-C 1224) Previously submitted[2]
USA/IA, 1975 (Amsoy)	EC 480 g/l	2.2[3] + 0.56 (2x) + 1.1 (2x)	-	5	30	0.02, <u>0.05</u>, 0.02	GH-C 1224 Previously submitted[2]
USA/NE, 1975 (Amsoy)	EC 480 g/l	2.2[3] + 0.56 (2x) + 1.1 (2x)	234	5	31	<u>0.01,</u> 0.01, 0.01	GH-C 1224 Previously submitted[2]

The highest residue from replicate plots was chosen for STMR consideration as a worst-case scenario.
[1] LOQ: 0.01 mg/kg.
[2] Previously submitted to JMPR and accepted as meeting GAP.
[3]Applied at crop emergence.

<u>Tea</u>

<u>India</u>. In six field trials in 1995, 1996, 1998 and 1999 at different sites (Report No. CP-tea-1-04) chlorpyrifos EC 200 g/l was applied once at a rate of 0.200 kg ai/ha (0.05 kg ai/hl, 400 l/ha water) at the active vegetative growth stage. Samples of green leaves were collected after 5, 7 and 14 days. Samples of about 1-2 kg consisted of a minimum 70% two leaves and a bud and were hand picked. They were treated in the factory under controlled conditions following normal black tea manufacturing processes involving withering (reduction of moisture in the plucked shoots), rolling, oxidation (fermentation) and drying. The MRLs for tea apply to processed tea (black or green). Residues of chlorpyrifos were determined using GLC with NPD or ECD. The LOQ was 0.02 mg/kg. Validation was with samples fortified at 0.1-5 mg/kg.

Table 7. Chlorpyrifos residues in black tea from supervised trials in India.

Year (sample)	Application (rate per application)					PHI days	Residue [1] mg/kg	Reference (Report No.)
	Form.	kg ai/ha	kg ai/hl	Water l/ha	No.			
GAP - India	**EC 200**	**0.200**	**0.05**	**400**		**7**		
1996 (mixed seeds)	EC 200 g/l	0.200	0.05	400	1	5 7 14	0.24 <u>0.03</u> <0.02	CP-tea-1-04
1995 (mixed clones)	EC 200 g/l	0.200	0.05	400	1	5 7 14	0.38 <u>0.19</u> <0.02	CP-tea-1-04
1995 (mixed seed and clones)	EC 200 g/l	0.200	0.05	400	1	5 7 14	0.78 <u>0.57</u> 0.21	CP-tea-1-04
1995 (mixed clones)	EC 200 g/l	0.200	0.05	400	1	5 7 14	0.44 <u>0.15</u> 0.03	CP-tea-1-04
1998 (mixed	EC 200 g/l	0.200	0.05	400	1	5 7	1.40 <u>0.77</u>	CP-tea-1-04

Year (sample)	Application (rate per application)					PHI days	Residue [1] mg/kg	Reference (Report No.)
	Form.	kg ai/ha	kg ai/hl	Water l/ha	No.			
cultivars)						14	0.08	
1999 (mixed cultivars)	EC 200 g/l	0.200	0.05	400	1	5 7 14	3.48 1.13 0.05	CP-tea-1-04

[1]LOQ: 0.02 mg/kg

RESIDUES IN ANIMAL PRODUCTS

No additional information was provided.

FATE OF RESIDUES IN PROCESSING

Cotton

USA. Cotton was treated twice or four times with chlorpyrifos EC 480 g ai/l at a rate of 0.28 kg ai/ha and 12 times at 1.12 kg ai/ha, either by ground (Mississippi) or aerial (Texas) application at various intervals during May-October at all stages of crop growth. Cotton was picked by hand from the control and treated plots at normal harvest, 15 days after ground and 18 days after aerial applications. The cotton samples were ginned, and seeds and gin trash collected.

Fifty pounds (23 kg) of undelinted cotton seeds were sent to the Oilseed Products Laboratory, Texas A&M University, and processed into linters, hulls, solvent-extracted meal, crude oil and refined bleached oil (

Figure 1). Seeds were hulled in a mill and the kernels were separated by screening. The kernels were flaked and extracted in a batch solvent extractor using Skelly solve F or B solvent and heating just under the boiling point of the solvent. The solvent oil mixture (miscella) was drawn off from the bottom of the extractor at the rate of approximately 7.5 l/hour. The extracted flakes were dried to give meal and crude oil was recovered from the miscella by evaporating the solvent before being refined.

All samples were stored frozen until analysis, and, except crude oil and refined bleached oil, analysed by GC/FPD using Dow AgroSciences Method ACR 74.4 with average procedural recoveries of 93% for cotton seed, 85% for linters, 66% for hulls and 97% for meal. Crude oil and refined bleached oil were analysed using Dow AgroSciences Method ACR 75.1 with average procedural recoveries of 78%. The limit of quantification was 0.01 mg/kg for all samples.

The results showed that residues of chlorpyrifos do not concentrate in hulls, meal or refined oil and only slightly in crude oil (Table 8). Processing factors for linters were 1.0-5.0.

Table 8. Residues of chlorpyrifos in cotton seed and its processed fractions, with processing factors.

Sample	**Residues[1] mg/kg**	**Processing factor**	**Reference (Report No.)**
Ground application (Mississippi): 0.28 kg ai/ha (2x) + 1.12 kg ai/ha (12x) 15-day PHI			
Cotton seed (RAC)	0.043 (0.031,0.035,0.040, 0.066)		GH-C 840
Hulls	<0.01	<0.2[2]	
Linters	0.04	0.9	
Meal	<0.01 (ND)	<0.1[3]	
Crude oil	0.01	0.2[2]	
Refined oil	0.01	0.2	

Sample	Residues[1] mg/kg	Processing factor	Reference (Report No.)
Aerial application (Texas): 0.28 kg ai/ha (4x) + 1.12 kg ai/ha (12x) 18-day PHI			GH-C 840
Cotton seed (RAC)	0.103 (0.078,0.098,0.112, 0.123)		
Hulls	0.07	0.7	
Linters	0.52	5.0	
Meal	0.01	0.1	
Crude oil	0.14	1.4	
Refined oil	<0.01 (ND)	<0.1	
Average processing factor			
Hulls		0.7	
Linters		3.0	
Meal		0.1	
Crude oil		1.4	
Refined oil		0.2	

[1] LOQ: 0.01 mg/kg.
[2] Not included in the average calculation

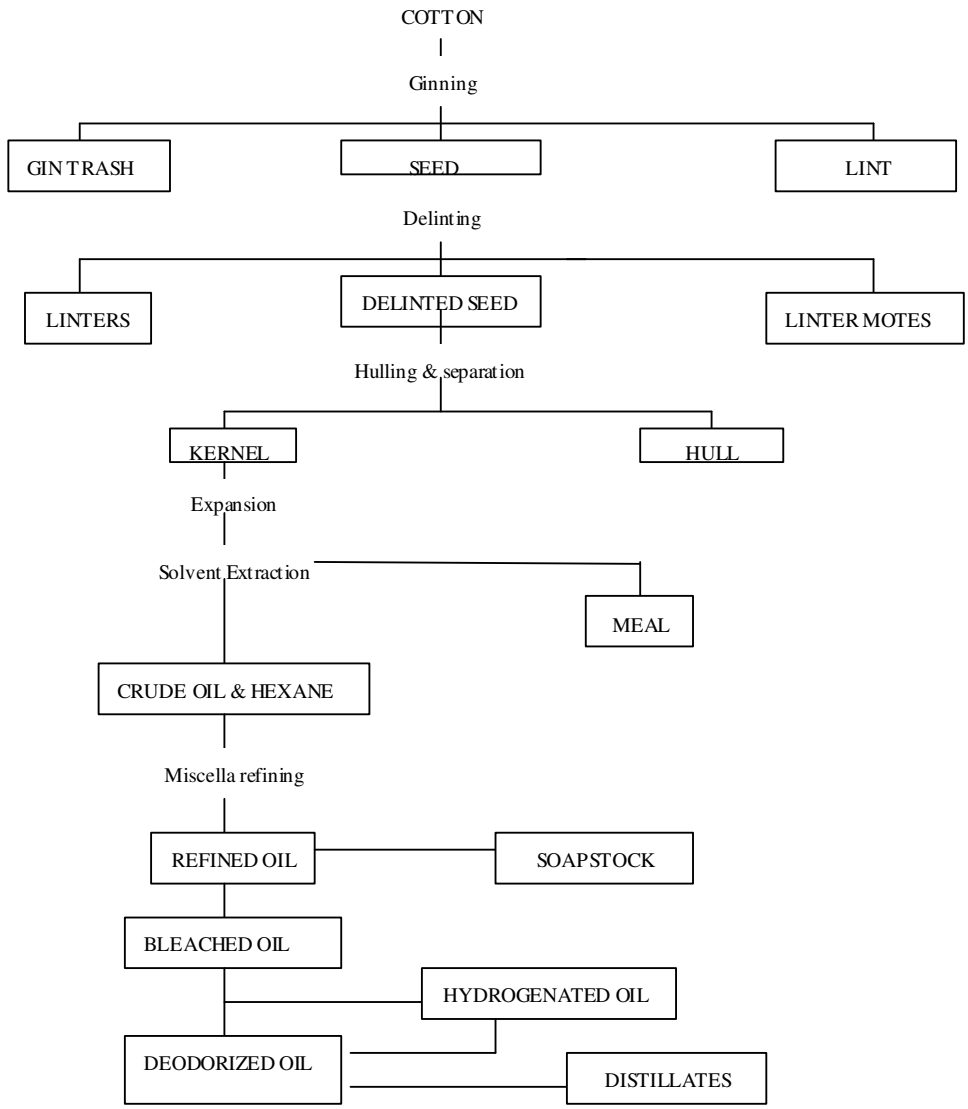

Figure 1. Cotton processing.

Rice

Australia. In a supervised trial to generate samples for a processing study (Cowles *et al.*, 1999d Report No. GHF-P 1795) chlorpyrifos EC 500 g ai/l was applied as a single broadcast application at the rate of 1.0 kg ai/ha. Paddy rice (whole rice grain) from the control and treated plots was harvested using a small plot harvester 10 days after application. Samples were processed into hulls, pollard/bran, brown rice, white rice, and trash.

All samples were stored at –20°C for about 9 months and analysed by GC/FPD using Dow AgroSciences Method PA-RM-98-07 with procedural recoveries of 80% for paddy rice, 110% for hulls, 87% for pollard/bran, 74% for brown rice, 95% for white rice and 85% for trash. The limit of quantification was 0.01 mg/kg for all samples.

Philippines. In a supervised trial to generate samples for a processing study (Cowles *et al.*, 1999c Report No. GHF-P 1794) chlorpyrifos EC 300 g ai/l was applied as a single broadcast application at the rate of 1.05 kg ai/ha. Paddy rice (whole rice grain) samples from the control and treated plots were harvested 10 days after treatment and processed into hulls, bran, and hulled rice (brown rice).

All samples were stored at –20°C for about 2 months and analysed by GC/FPD using Dow AgroSciences Method PA-RM-98-07 with procedural recoveries of 95% for unhulled rice, 84% for hulled rice, 110% for hulls, 87% for bran. The limit of quantification was 0.01 mg/kg.

India. In a supervised trial (Cowles, 2003d Report No. INDIA-CHP-RICE-1) chlorpyrifos EC 200 g ai/l was applied as a single broadcast application at a rate of 0.375 kg ai/ha. Paddy rice samples (whole rice grain) from the control and treated plots were harvested 14, 21 and 30 days after application and processed into husk (hulls), bran, and polished rice.

All samples were stored frozen at –20°C until analysis. Residues of chlorpyrifos were determined by procedures described in the report using GC/NPD. The average procedural recoveries were 90% for whole grain (paddy), 112% for husk, and 106% for polished rice with the limit of quantification 0.01 mg/kg for all samples.

The results of the processing studies showed that residues of chlorpyrifos were mainly on the outer layer of rice with an approximate average processing factor of 2.5 for the hulls. No residue concentration was observed in brown or polished rice (PF <1.0). The residues of chlorpyrifos in rice processed fractions and processing factors are shown in Table 9.

Table 9. Residues of chlorpyrifos in rice and its processed fractions with processing factors.

Sample	Residues[1] mg/kg	Processing factor	Reference (Report No.)
Australia: 1.0 kg ai/ha 10-day PHI			GHF-P 1795
Paddy rice (whole grain-RAC)	0.33		
Hulls	1.41	4.3	
Pollard (rice bran)	0.36	1.1	
Rice trash	1.85	5.6	
Brown rice	0.06	0.2	
White rice (polished)	0.01	0.03	
Philippines: 1.05 kg ai/ha 10-day PHI			GHF-P 1794
Unhulled rice (whole grain-RAC)	1.55		
Hulls	4.06	2.6	
Bran	3.89	2.5	
Hulled rice (brown rice)	0.10	0.06	
India: 0.375 kg ai/ha 14-day PHI			INDIA-CHP-RICE-1
Rice paddy (whole grain-RAC)	0.26		
Husk (hulls)	0.46	1.8	
Bran	<0.01	<0.05	
Polished rice	0.02	0.08	
India: 0.375 kg ai/ha 21-day PHI			INDIA-CHP-RICE-1
Rice paddy (whole grain-RAC)	0.19		
Husk (hulls)	0.34	1.8	
Bran	<0.01	<0.05	
Polished rice	0.02	0.11	
India: 0.375 kg ai/ha 30-day PHI			INDIA-CHP-RICE-1
Rice paddy (whole grain)	0.17		
Husk (hulls)	0.29	1.7	
Bran	<0.01	<0.05	

Sample	Residues[1] mg/kg	Processing factor	Reference (Report No.)
Polished rice	0.01	0.06	
Average processing factor			
Rice hulls		2.44	
Bran		1.80	
Brown rice		0.13	
Polished rice		0.07	

[1] LOQ: 0.01 mg/kg.

Soya bean

USA. A single processing trial reported to the 2000 JMPR (Miller, 1979 Report No. GH-C 1224) chlorpyrifos EC 480 g ai/l was applied as a broadcast spray directed at emergence at the rate of 4.48 kg ai/ha followed by foliar applications at 1.12 kg ai/ha (twice) and 2.24 kg ai/ha (twice). Soya beans were combine-harvested from the control and treated plots at normal harvest, 14 days after the last applications. Soya bean samples were sent to Oilseed Products Laboratory, Texas A&M University and processed into hulls, extracted meal, crude, refined and refined bleached oil and soap stock.

Samples were frozen at –18°C until analysed by GC/FPD using Dow AgroSciences Method ACR 73.5.S1. Average procedural recoveries were 80% for soya bean, 80% for hulls, 82% for extracted meal, 90% for crude oil, 80% for refined oil, 90% for refined bleached oil and 100% for soap stock. The limit of quantification for all samples was 0.01 mg/kg. Details are shown in Table 10.

The results showed that residues of chlorpyrifos do not concentrate in hulls, extracted meal, crude and refined oils or soapstock. Processing factors for all processed products were below 1.0.

Table 10. Residues of chlorpyrifos in soya beans and processed fractions with processing factors.

Sample	Residues[1] mg/kg	Processing factor	Report No.
Soya bean (RAC)	0.042		GH-C 1224
Hulls	0.022	0.5	
Extracted meal	<0.01	<0.2	
Crude oil	0.017	0.4	
Refined oil	0.018	0.4	
Refined bleached oil	0.019	0.5	
Soap stock	<0.01	<0.2	

[1] LOQ: 0.01 mg/kg.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Chlorpyrifos is determined in several pesticide monitoring programmes. Monitoring data from the USA (California Environmental Protection Agency Food Safety Program, the Food and Drug Administration Enforcement Monitoring Program, the US Department of Agriculture Pesticide Data Program and the Pesticide Residues Information System), the Government of The Netherlands, and the Government of Poland were included in the 2000 JMPR Residue Evaluation. No additional information was provided.

NATIONAL RESIDUE LIMITS

An extensive list of national MRLs for chlorpyrifos was provided in the JMPR 2000 evaluation. Table 11 lists national MRLs only for commodities for which MRLs were recommended by the present Meeting.

Table 11. Commodities with national MRLs for which Codex MRLs are recommended.

Commodity	Country	MRL, mg/kg
Cotton seed	Argentina	0.05
	Australia	0.05
	Brazil	0.05
	India	0.05
	Israel	0.05
	Mexico	0.5
	USA	0.2
	Venezuela	0.05
Cotton seed, oil	Argentina	0.05
	Australia	0.2
	Brazil	0.05
	India	0.025
Cotton fodder, dry	Australia	30
Cotton meal and hulls	Australia	0.05
Potato	Argentina	0.05
	Australia	0.05
	Brazil	0.01
	Chile	0.05
	Denmark	0.05
	India	0.01
	Italy	0.2
	Poland	0.05
	South Africa	0.3
	Spain	0.05
	Sweden	0.05
	Ukraine	0.05
	Venezuela	0.1
	Zimbabwe	0.05
Rice	Chile	0.1
	Mexico	0.1
	Taiwan	0.1
	Venezuela	0.1
Soya bean	Argentina	0.01
	Brazil	0.01
	France	0.05
	Mexico	0.5
	USA	0.3
Tea	EU	0.1

APPRAISAL

At its Twenty-fifth Session in 1993 (ALINORM 93/24A para. 251), the CCPR identified chlorpyrifos as a candidate for periodic review. At its Twenty-ninth Session in 1997, it scheduled periodic reviews for toxicology in 1999 and for residue chemistry in 2000. The 1999 toxicology review confirmed the ADI of 0.01 mg/kg bw and also established an ARfD of 0.1 mg/kg bw. In the 2000 residue chemistry review, information was supplied on the identity and physical properties of the active ingredient and technical material, metabolism in plants and animals, environmental fate, storage stability, animal feeding studies, field trials, GAP (national labels) and fate of residues in processing.

Chlorpyrifos was scheduled for re-evaluation in 2004 for consideration of maximum residue levels in cotton, potato, rice and soya bean. No MRLs were recommended by the 2000 JMPR on these commodities because of lack of relevant GAP labels or insufficient residue data. Relevant GAP labels and additional residue data to support proposed Codex MRLs in these commodities were submitted to the Meeting for evaluation. The CCPR at its Thirty-sixth Session in 2004 agreed that JMPR would

review data from India to support establishment of a maximum residue level on tea. Information was submitted by the Government of India for this purpose.

Methods of analysis

Methods for enforcement, data collection and monitoring of chlorpyrifos in different matrices were submitted and evaluated by JMPR in 2000. Additional methods were submitted to the present Meeting for the analysis of cotton-seed, potato, rice and soya bean. Various extraction and clean-up methods are followed by gas chromatography with flame photometric detection. The LOQ for chlorpyrifos is 0.01 mg/kg in all matrices.

The method of analysis for chlorpyrifos in tea was submitted by the Government of India. Residues in the cleaned extract were quantified by gas chromatography with flame-photometric or electron-capture detection. The LOQ is 0.02 mg/kg tea.

Results of supervised trials on crops

Cotton-seed

Supervised field trials on cotton were conducted at three sites in Australia in 2000 to provide additional residue data for consideration of MRLs for this crop. Bridging studies were conducted with a water-dispersible granule containing 750 g ai/kg and with an emulsifiable concentrate containing 300 g ai/l. Three foliar broadcast applications of each formulation were made at a rate of 1.5 kg ai/ha per application at a 5–8-day interval. A twofold rate of 3.0 kg ai/ha per application was also included. The applications were made at growth stages from late flowering to 15% of the bolls opened, with the last application 17 or 28 days before harvest. Although two sites in Australia have an early PHI (17 days), the residue levels in cotton-seed in these trials were included for MRL consideration because they were within the range of those in all trials. The trials conformed with Australian GAP (0.15–1.5 kg ai/ha in three applications and a 28-day PHI with either formulation). The highest residue level in replicate plots was chosen for estimating the STMR as a worst-case scenario.

Two supervised field trials on cotton were conducted at two sites in Brazil in 1992. Data on residues from the trials were submitted to JMPR in 2000 and accepted as meeting GAP. Briefly, the trials were conducted with an emulsifiable concentrate containing 480 g ai/l at a rate of either 0.96 or 1.92 kg ai/ha (twofold rate) per application. Foliar broadcast applications were made twice in one trial and three times in the other. In 2000, three additional supervised field trials on cotton were conducted at three sites in Brazil, with three foliar broadcast applications of the same emulsifiable concentrate at a rate of 0.96 or 1.92 kg ai/ha per application. The applications were made at growth stages from plant emergence to 30% of the bolls opened. Two replicate plots were used per treatment in each field site. Samples were hand-harvested from control and treated plots at 0, 7 (or 5), 14 (or 15), 21 (or 22) and 28 days after the last application and ginned to generate the cotton-seed (undelinted) samples. The trials conformed with Brazilian GAP (0.14–0.96 kg ai/ha in one to three applications and a 21-day PHI).

Supervised field trials on cotton were conducted in the USA in 1973, 1974 and 1986. Data on residue levels from these trials were submitted to JMPR 2000, and three of the trials were accepted as meeting GAP. Briefly, the trials were conducted with an emulsifiable concentrate containing 480 g ai/l applied as foliar broadcast applications. At a site in Mississippi in 1973, nine applications were made at a rate of either 1.12 or 2.24 kg ai/ha per application. Samples were hand-harvested 0, 3, 7 and 14 days after the last application and ginned to generate undelinted cotton-seed. At another site in Mississippi in 1974, two applications were made at a rate of 0.28 kg ai/ha per application, followed by 12 applications each at 1.12 kg ai/ha. Undelinted cotton-seed samples were generated similarly 15 days after the last application. At a site in California in 1986, five applications were made at 1.12 kg ai/ha, and undelinted cotton-seed samples were generated 14 days after the last application.

On the basis of the trials that conformed to GAP, the chlorpyrifos residue levels, in ranked order (median underlined), was: 0.01 (two), 0.02, 0.03 (two), 0.05, <u>0.07</u> (four), 0.11, 0.12, 0.16 and

0.18 mg/kg. The Meeting estimated a maximum residue level of 0.3 mg/kg for cotton-seed, an STMR of 0.07 mg/kg and a highest residue level of 0.18 mg/kg from the supervised trial in the USA in 1986.

Potato

The 2000 JMPR considered data on residues from supervised trials on potatoes, but did not establish MRLs because there were insufficient trials at GAP to estimate the STMR or maximum residue level. Additional data from supervised trials after at-plant application (soil in-furrow) on potatoes were generated in Brazil and submitted to the present Meeting. The proposed MRL from the data on residues after at-plant application would also cover residue levels after foliar application. The data previously summarized for at-plant application were provided again to allow evaluation of residue levels in all supervised trials conducted in Brazil. Four supervised field trials on potatoes were conducted in 1993–94 in Brazil. The data from these trials were submitted to the JMPR in 2000 and accepted as meeting GAP. The trials were conducted with either a granular formulation containing 100 g ai/kg or an emulsifiable concentrate containing 450 g ai/l. A single application was made to soil in-furrow at planting, at a rate of 1.5, 3.0 or 6.0 kg ai/ha for the granular formulation and 2.9 or 5.9 kg ai/ha for the emulsifiable concentrate.

In 1999–2000, seven supervised field trials on potatoes were conducted in Brazil to provide additional data on residue levels for MRLs. Four trials were conducted with the granular formulation at a rate of 3 or 6 kg ai/ha, and three trials were conducted with the emulsifiable concentrate at a rate of 2.7 or 5.4 kg ai/ha. Three replicate plots were maintained for each treatment. A single application of each formulation was made to soil in-furrow at planting. Three samples of potatoes were collected manually at normal harvest from the control and treated plots 100–124 days after application. The highest residue level in replicate plots was chosen for estimating the STMR as a worst-case scenario.

On the basis of at-plant treatment in trials conforming to GAP, the chlorpyrifos residue levels, in ranked order, were: 0.02, 0.03, 0.10, 0.13, 0.29, 0.51, 0.57, 0.58, 0.65, 0.69 and 0.87 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg for potato, an STMR of 0.51 mg/kg and a highest residue level of 0.87 mg/kg.

Rice

The 2000 JMPR considered data on residues from supervised trials on rice, but did not establish MRLs because no trials were conducted at the relevant GAP. Relevant GAP in Colombia, the Philippines and Viet Nam was made available to support the results of the supervised trials submitted to the JMPR in 2000. Some additional data from supervised trials on rice, generated in India and Thailand since 2000, were submitted to the present Meeting. Supervised trials were thus conducted in Colombia, India, the Philippines, Thailand and Viet Nam.

In Colombia, two supervised field trials on rice were conducted in 1998 at two sites. Data on residue levels from these two trials were submitted to the JMPR in 2000. Briefly, the trials were conducted with an emulsifiable concentrate containing 480 g ai/l, applied three times to upland rice. The first application was made at a rate of 0.96 kg ai/ha after germination, followed by a second application at 0.72 kg ai/ha when the plants were at tillering crop growth stage; the final application was made at a rate of 0.34 kg ai/ha 20–21 days before harvest, when 100% of pinnacles were present. Three replicate plots were maintained for each trial. Rice samples from the control and treated plots were hand-harvested 20–21 days after the last application. The supervised trials conformed to Colombian GAP (0.34–0.96 kg ai/ha in a maximum of three applications and 20-day PHI).

In the Philippines, two supervised field trials on rice were conducted in 1998 at two sites. An emulsifiable concentrate containing 300 g ai/l was applied three times at a rate of 0.3 kg ai/ha per application. Data on residue levels in these two trials were submitted to JMPR 2000. The first two applications were made 25 and 40 days after transplantation, and the last application was made 25 days before harvest. Rice grain and straw samples from the control and treated plots were harvested 25 days after the last application. The supervised trials conformed to GAP in the Philippines, which is 0.30 kg ai/ha in a maximum of three applications and 25 days' PHI.

In Viet Nam, two supervised field trials on rice were conducted in 1998 at two sites. Data on residue levels in these two trials were submitted to the JMPR in 2000. Briefly, the trials were conducted with an emulsifiable concentrate containing 300 g ai/l, applied at a rate of 0.42 kg ai/ha. Rice grain and straw samples from the control and treated plots were harvested 10 days after the last application. The supervised trials conformed to GAP in Viet Nam, which is 0.3–0.42 kg ai/ha in one application and 28 days' PHI.

In Thailand, three supervised field trials on rice were conducted in 2002. An emulsifiable concentrate of chlorpyrifos containing 400 g ai/l was applied three times as a foliar application at a rate of 400 g ai/ha per application. The first application was made at mid-booting, and the final one at seed formation. Samples of rice grain and straw from the control and treated plots were hand-harvested 7, 14 and 21 days after the last application. The GAP in Thailand is 0.40 kg ai/ha, with an unspecified number of applications and 7–14 days' PHI.

In India, three supervised field trials on rice were conducted at three sites in 2002. An emulsifiable concentrate of chlorpyrifos containing 200 g ai/l was applied as a foliar application at a single rate of 375 g ai/ha. Samples of grain and straw from the control and treated plots were taken 14 (or 15), 21 and 30 days after application. As the labels were not available in English, the Meeting did not evaluate the data from India.

On the basis of trials on rice conforming to GAP, the chlorpyrifos residue levels, in ranked order, were: 0.02 (two), 0.08 (two), 0.09, 0.15, 0.16, 0.19 and 0.28 mg/kg. The Meeting estimated a maximum residue level of 0.5 mg/kg for rice, an STMR of 0.12 mg/kg and a highest residue level of 0.28 mg/kg.

Soya bean

The 2000 JMPR considered data from supervised trials on soya beans, but did not establish MRLs because the data from accepted GAP trials were insufficient for estimating the STMR or maximum residue level. Additional data from supervised trials conducted in 1994–96 on soya beans in Brazil were submitted to the Meeting. The data from the five trials conducted in the USA according to GAP were provided again to the Meeting.

In Brazil, two field trials were conducted in 1992–93 with an emulsifiable concentrate containing 480 g ai/l, applied three times at either 0.48 or 0.96 kg ai/ha (twofold label rate) per application. Soya beans from the control and treated plots were hand-harvested 20–21 days after the last application. In 1992–93, an additional supervised field trial was conducted with the emulsifiable concentrate applied three times at 0.48 or 0.96 kg ai/ha per application. Soya beans from the control and treated plots were hand-harvested 21 days after application. In 1995, one supervised field trial was conducted in Brazil with the emulsifiable concentrate applied at either 0.34 or 0.77 kg ai/ha. Soya beans from the control and treated plots were harvested 21 days after application. Brazilian GAP is 0.12–0.48 kg ai/ha with one to two applications and 21 days' PHI. The supervised trials represent the worst-case scenario. The residue levels were below the LOQ (< 0.01 mg/kg) in all trials conducted at either the maximum or twice the maximum label rate or in single or triple applications.

In the USA, supervised field trials on soya beans were conducted in 1975–76. Data on residue levels from these trials were submitted to the JMPR in 2000, and five trials were accepted as meeting GAP. Briefly, the trials were conducted with an emulsifiable concentrate containing 480 g ai/l applied once as a directed broadcast spray at crop emergence, followed by three to four foliar broadcast applications during the growing season. The application rates were 0.56–2.2 kg ai/ha at emergence and 0.56–1.1 kg ai/ha at each foliar application. Soya beans were collected from the control and treated plots at normal harvest, 28–31 days after the last application. For replicate plots, the highest residue level was chosen for consideration of the MRL, as a worst-case scenario.

On the basis of trials conforming to GAP, the chlorpyrifos residue levels, in ranked order, were: < 0.01 (six), 0.01 (two) and 0.05 mg/kg. The Meeting estimated a maximum residue level of 0.1 mg/kg for soya bean, an STMR of 0.01 mg/kg and a highest residue level of 0.05 mg/kg.

Tea

Six supervised field trials were conducted in 1995, 1996, 1998 and 1999 at various sites in India. A chlorpyrifos emulsifiable concentrate containing 200 g/l was applied once at a rate of 0.20 kg ai/ha (0.05 kg ai/hl, 400 l/ha water), which complied with GAP for chlorpyrifos on tea as submitted by the Government.

On the basis of trials conforming to GAP in India, the chlorpyrifos residue levels in tea, in ranked order, were: 0.03, 0.15, 0.19, 0.57, 0.77 and 1.13 mg/kg. The Meeting estimated a maximum residue level of 2.0 mg/kg for tea, an STMR of 0.34 mg/kg and a highest residue levelof 1.13 mg/kg.

Fate of residues during processing

Studies on processing of cotton-seed, rice and soya beans were submitted but not evaluated by the JMPR in 2000 because no MRLs were established for the raw agricultural commodities of these crops. The processing studies were resubmitted to the present Meeting for evaluation of residue levels in processed products of these raw agricultural commodities. The processing factors and estimated STMR-Ps for cotton-seed, rice and soya bean are summarized below:

Processed commodity	Processing factor	STMR (mg/kg) (RAC)	STMR-P (mg/kg)
Cotton hulls	0.7	0.07	0.05
Cotton-seed meal	0.1	0.07	< 0.01
Cotton-seed oil, crude	1.4	0.07	0.10
Cotton-seed oil, refined	0.2	0.07	0.01
Rice hulls	2.44	0.12	0.29
Rice bran	1.80	0.12	0.22
Rice husked	0.13	0.12	0.016
Polished rice	0.07	0.12	0.008
Soya bean meal	< 0.2	0.01	< 0.002
Soya bean crude oil	0.4	0.01	0.004
Soya bean refined oil	0.4	0.01	0.004
Soya bean refined	0.5	0.01	0.005

STMR-P, STMR of raw agricultural commodity × processing factor of processed product

Residues in animal commodities

The 2000 JMPR estimated the dietary burden of chlorpyrifos in farm animals and poultry in cases in which calculations from the MRLs yielded maximum theoretical dietary intakes, and calculations from STMR values for feed allowed estimation of STMR values for animal commodities. The present Meeting concluded that the contribution of residues to feed, calculated for the uses considered this year, would not increase the dietary burden assessed by the 2000 JMPR. The Meeting maintained the recommendations of the 2000 JMPR.

RECOMMENDATIONS

On the basis of data from supervised trials the Meeting estimated that the residue levels for chlorpyrifos in raw agricultural commodities of cotton seed, potatoes, rice, soya beans and tea and in processed products of cotton seed, rice and soya beans listed below are suitable for establishing maximum residue limits and for IEDI and IESTI:

Definition of the residue for compliance with MRLs: chlorpyrifos
Definition of the residue for estimation of dietary intake: chlorpyrifos

The residues are fat-soluble.

Commodity		MRL	STMR/STMR-P	HR/HRP
CCN	Name	mg/kg	mg/kg	mg/kg
SO 0691	Cotton seed	0.3	0.07	
	Cotton seed meal		<0.01	
	Cotton seed hulls		0.05	
OC 0691	Cotton seed, crude oil		0.10	
OR 0691	Cotton seed, refined oil	0.05	0.01	
VR 0589	Potato	2.0	0.51	0.87
GC 0649	Rice	0.5	0.12	
CM 1205	Rice, polished		0.008	
CM 649	Rice, husked		0.016	
	Rice hulls		0.29	
CM 1206	Rice bran (unprocessed)		0.22	
VD 0541	Soya bean	0.1	0.01	
	Soya bean meal		<0.002	
OC 0541	Soya bean, crude oil		0.004	
OR 0541	Soya bean, refined oil	0.03	0.004	
DT 1114	Tea, green, black	2.0	0.34	

DIETARY RISK ASSESSMENT

Long-term intake

IEDIs for chlorpyrifos were calculated for the five GEMS/Food regional diets from the STMRs and STMR-Ps estimated by this Meeting, in addition to those for 61 commodities from the JMPR 2000 evaluation. The IEDIs were 3–30% of the maximum ADI (0–0.01 mg/kg bw), as shown in Annex 3. The Meeting concluded that the intake of residues of chlorpyrifos resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The IESTI for chlorpyrifos was calculated for the commodities for which MRLs, STMR values and highest residue values were estimated and for which data on consumption (large portion and unit weight) were available. The results are shown in Annex 4.

The ARfD for chlorpyrifos is 0.1 mg/kg bw. The short-term intakes were calculated for commodities for which highest residues or HR-Ps were estimated by the present Meeting. The calculated short-term intakes were < 100% of the ARfDs for children (0–40%) and for the general population (0–10%). The Meeting concluded that the intake of residues of chlorpyrifos resulting from uses that have been considered by the JMPR is unlikely to present a public health concern for consumers.

REFERENCES

do Amaral, L.C. 1999. Residues of chlorpyrifos in potatoes after treatment with Sabre at planting -Brazil, 1994-95. GHB-P 349. Dow AgroSciences. Unpublished.

Balderrama, O.P. and Matos, J.C. 1994a. Determination of residues of chlorpyrifos in orange whole fruit and juice. Method BRC 94.3. Dow AgroSciences. Unpublished.

Balderrama, O.P. and Matos, J.C.G. 1994b. Residues of chlorpyrifos in cotton following multiple applications of Lorsban* 480 BR-Brazil, 1992-94. GHB-P 195. Dow AgroSciences. Unpublished.

Balderrama, O.P. and Matos, J.C.G. 1994c. Residues of Chlorpyrifos in cotton Following Applications of Lorsban* 480 BR-Brazil, 1992-94. GHB-P 196. Dow AgroSciences. Unpublished.

Balderrama, O.P. and Matos, J.C.G. 1994d. Residues of chlorpyrifos in soybeans following multiple applications

of Lorsban 480 BR-Brazil, 1992-94. GHB-P 189. Dow AgroSciences. Unpublished.

Balderrama, O.P. and Matos, J.C.G. 1994e. Residues of chlorpyrifos in potatoes following treatments with Lorsban* 10G BR-Brazil, 1993-94. GHB-P 218. Dow AgroSciences. Unpublished.

de Baptista, G. C. 1996. Residues of chlorpyrifos in soybeans after treatment with Lorsban 480 – Brazil. Report of Contract Laboratory – ESALQ. GHB-P 534. Dow AgroSciences. Unpublished.

Catta-Preta, R.F., Balderrama, O.P. and Rampazzo, P.E. 1994. Determination of chlorpyrifos in pears, peaches and plums. Method BRC 94.4. Dow AgroSciences. Unpublished.

Catta-Perera, R.F. and Rampazzo, P.E. 1995. Determination of of chlorpyrifos in pears, peaches and plums – Supplement for dried coffee beans. Method BRC 94.4.S1. Dow AgroSciences. Unpublished.

Cowles, J., 2002. A comparison of chlorpyrifos residues in Australian cottonseed and trash after three applications of Lorsban* 75 WG Insecticide or three applications of Predator 300 Insecticide in the 2000-2001 season. GHF-P 2477. Dow AgroSciences. Unpublished.

Cowles, J. 2003a. Magnitude of chlorpyrifos residues in Sulphan 1 rice treated with three applications of Lorsban 40EC Insecticide in Suphanburi Thailand in the 2002 season. GHF-P 2670. Dow AgroSciences. Unpublished.

Cowles, J. 2003b. Magnitude of chlorpyrifos residues in Sulphanburi 50 rice treated with three applications of Lorsban 40EC Insecticide in Suphanburi Thailand in the 2002 season. GHF-P 2671. Dow AgroSciences. Unpublished.

Cowles, J. 2003c. Magnitude of chlorpyrifos residues in Pathumthani 1 rice treated with three applications of Lorsban 40EC Insecticide in Lamphoon Thailand in the 2002 season. GHF-P 2672. Dow AgroSciences. Unpublished.

Cowles, J. 2003d. Residues of chlorpyrifos following rice paddy applications in India. INDIA-CHP-RICE-1. Dow AgroSciences. Unpublished.

Cowles, J., Penn, M. and Quin R. 1999a. Magnitude of chlorpyrifos residues in rice treated with Lorsban 3E Insecticide in the Philippines, 1998. GHF-P 1791. Dow AgroSciences. Unpublished.

Cowles, J., Penn, M. and Quin R. 1999b. Magnitude of chlorpyrifos residues in rice treated with Lorsban 30EC Insecticide in Vietnam, 1998. GHF-P 1792. Dow AgroSciences. Unpublished.

Cowles, J., Quin, R. and Taylor, J. 1999c. Magnitude of chlorpyrifos residues in rice processing fractions after treatment with Lorsban 3E in the Philippines, 1998. GHF-P 1794. Dow AgroSciences. Unpublished.

Cowles, J., Quin, R. and Taylor, J. 1999d. Magnitude of chlorpyrifos residues in rice processing fractions after treatment with Lorsban 500EC in Australia, 1998. GHF-P 1795. Dow AgroSciences. Unpublished.

Gagnotto, S.R. and Balderrama, O.P. 1993. Determination of residues of chlorpyrifos in tomatoes. Method BRC 93.1. Dow AgroSciences. Unpublished.

Khoshab, A. and Hastings, M. 1994. Determination of chlorpyrifos in grapes. Method ERC 94.1 (also as Amdel In-house Method No. STM CR 435). Dow AgroSciences. Unpublished.

"Manual of Pesticide Residue Analysis" Vol. I, p. 383, VCH Publishers Inc., New York, NY.

McKellar, R.L. 1975a. Determination of residues of O,O,-diethyl O-(3,5,6-trichloro-2-pyridyl)phosphorothioate in cottonseed and cotton gin trash by gas chromatography. Method ACR 74.4. Dow AgroSciences. Unpublished.

McKellar, R.L. 1975b. Determination of residues of chlorpyrifos and 3,5,6-Trichloro-2-pyridinol in cottonseed**,** gin trash and process fractions following multiple treatments of cotton plants with Lorsban Insecticide. GH-C 840. Dow Chemical USA. Unpublished.

McKellar, R.L and Dishburger, H.J. 1974. Determination of residues of chlorpyrifos and 3,5,6-Trichloro-2-pyridinol in cottonseed and gin trash following multiple treatments of cotton plants with Lorsban Insecticide. GH-C 739. Dow Chemical USA. Unpublished.

Miller, P.W. 1979. Residues of chlorpyrifos and 3,5,6-trichloro-2-pyridinol in or on soybeans receiving multiple applications of Lorsban Insecticide. GH-C 1224. Dow AgroSciences. Unpublished.

Pinheiro, A.C.T. 2000 Determination of residues of chlorpyrifos residues in potatoes. Method BRC 00.2. Dow AgroSciences. Unpublished.

Pinheiro, A.C. and De Vito, R. 1999. Magnitude of residues of chlorpyrifos in rice treated with Lorsban 4E-Colombia, 1998. GHB-P 406. Dow AgroSciences. Unpublished.

Pinheiro, A.C.T. and Oliviera, R. 2001. Determination of residues of chlorpyrifos in cottonseeds. Method GRM 01.22. Dow AgroSciences. Unpublished.

Pinheiro, A.C.T. and Santos, A.C. 2000. Residues of chlorpyrifos in potatoes after treatment with Lorsban* 10G Insecticide-Brazil, 1999. GHB-P 462. Dow AgroSciences. Unpublished.

Pinheiro, A.C.T., Rosseto, J. and Rampazzo, P. 2001a. Residues of chlorpyrifos in potatoes after treatment with Sabre Insecticide-Brazil, 2000a. GHB-P 710. Dow AgroSciences. Unpublished.

Pinheiro, A.C.T., Rosseto, J., and Rampazzo, P. 2001b. Residues of chlorpyrifos in potatoes after treatment with Lorsban* 10G Insecticide-Brazil, 2000. GHB-P 711. Dow AgroSciences. Unpublished.

Pinheiro, A.C.T., Santos, A.C., Rampazzo, P. and Pavan, L.A. 2001c. Residues of chlorpyrifos in potatoes after treatment with Sabre Insecticide-Brazil, 2000. GHB-P 650. Dow AgroSciences. Unpublished.

Pinheiro, A.C.T., Santos, A.C., Rampazzo, P. and Pavan, L.A. 2001d. Residues of chlorpyrifos in potatoes after treatment with Lorsban* 10G Insecticide-Brazil, 2000. GHB-P 678. Dow AgroSciences. Unpublished.

Pinheiro, A.C.T., Oliviera, R., Pavan, L.A., Schmidt, F. and Cason, J. 2001e. Residues of chlorpyrifos in cottonseeds after treatment with Lorsban* 480 BR Insecticide-Brazil, 2000-01. GHB-P 743. Dow AgroSciences. Unpublished.

Quin, R. and Utting, A.J. 1999. Determination of residues of chlorpyrifos in rice grain and straw. Method PA-RM-98.07. GHF-P 1800. Dow AgroSciences. Unpublished.

Storherr, R.W., Ott, P. and Watts, R.R. 1971 "A general method for organophosphorous pesticide residues in non-fatty foods", *J. Assoc. Off. Anal. Chem*., Washington, 54 (3): 513-516.

Tornisielo, V.L. 1995. Residues of chlorpyrifos in soybeans after treatment with Sanachem 480CE – Brazil. Report of Contract Laboratory – CENA. GHB-P 621. Dow AgroSciences. Unpublished.

Wetters, J.H. 1976 Determination of residues of O,O,-diethyl O-(3,5,6-trichloro-2-pyridinyl)phosphorothioate in sugar beets and solid process fractions by gas chromatography, Method ACR 73.5. Supplement for sorghum green plant and dry plant Method ACR 73.5.S1. Dow AgroSciences. Unpublished.

Wetters, J.H. 1987. Residues of chlorpyrifos and 3,5,6-trichloro-2-pyridinol in or on cottonseed following five foliar applications of Lorsban 4E Insecticide. GH-C 1893. Dow Chemical USA. Unpublished.

By report numbers:

ACR 73.5.S1, Wetters 1976
ACR 74.4, McKellar 1975a
BRC 93.1**,** Gagnotto and Balderrama 1993
BRC 94.3, Balderrama and Matos 1994a
BRC 94.4, Catta-Preta *et al.* 1994
BRC 94.4.S1 Catta-Perera and Rampazzo 1995
BRC 00.2, Pinheiro 2000
ERC 94.1, Khoshab and Hastings 1994
GH-C 739, McKellar and Dishburger 1974
GH-C 840, McKellar 1975b
GH-C 1224, Miller 1979
GH-C 1893, Wetters 1987
GHB-P 189, Balderrama and Matos 1994d
GHB-P 195, Balderrama and Matos 1994b
GHB-P 196, Balderrama and Matos 1994c
GHB-P 218, Balderrama and Matos 1994e
GHB-P 349, do Amaral 1999
GHB-P 406, Pinheiro and De Vito 1999
GHB-P 534, de Baptista 1996
GHB-P 462, Pinheiro and Santos 2000
GHB-P 621, Tornisielo 1995
GHB-P 650, Pinheiro *et al.* 2001c
GHB-P 678, Pinheiro *et al.* 2001d
GHB-P 710, Pinheiro *et al.* 2001a
GHB-P 711, Pinheiro *et al.* 2001b,
GHB-P 743, Pinheiro *et al.* 2001e
GHF-P 1791, Cowles *et al.* 1999a
GHF-P 1792, Cowles *et al.* 1999b
GHF-P 1794, Cowles *et al.* 1999c
GHF-P 1795, Cowles *et al.* 1999d
GHF-P 2477, Cowles 2002
GHF-P 2670, Cowles 2003a
GHF-P 2671, Cowles 2003b
GHF-P 2672, Cowles 2003c
GRM 01.22, Pinheiro and Oliviera 2001
INDIA-CHP-RICE-1, Cowles 2003d
PA-RM-98.07, Quin and Utting 1999
Government of INDIA (Report No. CP-tea-1-04)

DITHIOCARBAMATES (105)

Propineb was evaluated by the present Meeting within the CCPR Periodic Review Programme, and the recommendations for estimates of MRLs, STMRs and highest residue levels are discussed in the appraisal of that compound. Recommended MRLs for dithiocarbamates arising from use of propineb are consolidated here.

The 1996 JMPR recommended MRLs for dithiocarbamates in almond hulls, almond, pecan, pome fruits and stone fruits which were based on residue data for ziram. The 1996 JMPR also recommended that estimates of maximum residue levels for dithiocarbamates, which relied primarily on data for ziram, should be temporary until the relevant data on environmental fate had been evaluated.

In view of the decision of the 2003 JMPR that data on environmental fate need be reviewed only when they directly affect estimation of maximum residue levels, the Meeting decided to withdraw its requirement for information on the environmental fate of ziram in soil and in water-sediment systems.

RECOMMENDATIONS

Definition of the residue (for compliance with MRLs): *total dithiocarbamates, determined as CS_2, evolved during acid digestion and expressed as mg/CS_2*

		Commodity	Recommended MRL[1]	
	CCN		New[2]	Previous
Dithiocarbamates	VC 0424	Cucumber	2 c, N, p	2 c, N
Ferbam	MO 0105	Edible offal (mammalian)	0.1 C, m, p	0.1 C, m
ADI: 0–0.003 mg/kg bw	PE 0112	Eggs	0.05(*) C, p	0.05(*) C
Propineb	FB 0269	Grapes	5 C, m, n	5 C, m, n, p
ADI: 0–0.007 mg/kg bw	MM 0095	Meat (from mammals other than marine mammals)	0.05(*) c, m, p	0.05(*) c, m
Thiram	VC 0046	Melon (except watermelon)	0.5 C	0.5 C, p
ADI: 0–0.01 mg/kg bw	ML 0106	Milks	0.05(*) c, m, p	0.05(*) c, m
Ziram	VA 0385	Onion, bulb	0.5 C	0.5 C, p
ADI: 0–0.003 mg/kg bw	TN 0672	Pecan	0.1(*) Z	0.1(*) T Z
	VO 0445	Peppers, sweet	7 c, m, P	1 c, m,
	FP 0009	Pome fruits	5 C, M, H, Z	5 C, M, p, H, Z
	VR 0587	Potato	0.2 c, m, n, p	0.2 c, m, n
	PM 0110	Poultry meat	0.1 c, p	0.1 C
	PO 0111	Poultry, edible offal of	0.1 c, p	0.1 C
	FS 0012	Stone fruit	7 h, p, Z	7 T h, Z

[1] Recommended MRLs refer to the total residues from the use of any or each of the dithiocarbamates.
[2].Based on trials with; n, maneb; m, metiram; c, mancozeb; p, propineb; h, thiram; z, ziram. Compounds shown in upper case are those on which the estimates of maximum residue levels are mainly based.
[8] At or about the LOQ
T: the 1996 JMPR recommended that the listed MRLs be designated as temporary pending review of data on environmental fate.