## ENDOSULFAN (032)

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### **EXPLANATION**

Endosulfan is a synthetic cyclodiene non-systemic insecticide and acaricide with both contact and stomach activity. It has been widely used in agriculture to control a range of insects and mites on a broad spectrum of crops. It has been evaluated several times by the JMPR; the initial evaluation for residues was in 1967 and the latest in 2006. The 1998 JMPR established an ADI and an acute reference dose for endosulfan. A periodic review for residues was performed by the 2006 JMPR.

At the Thirty-ninth Session of CCPR in 2007, on the proposal of the delegations of China and India the CXL for tea (green, black) was retained for four years under the Periodic Review Program. The Forty-first Session of CCPR in 2009 scheduled the review of data on tea from China by the 2010 JMPR. Information was submitted by the Government of China.

### METHODS OF RESIDUE ANALYSIS

## Analytical methods for plant materials used in trials and studies

Fresh tea leaves, made tea samples<sup>14</sup>, and tea infusions were analysed for endosulfan. The analytical method for fresh tea leaves involved homogenisation in petroleum ether-acetone, made tea first involved homogenisation with hexene-acetone, then extraction with hexene-acetone-toluene. Tea infusion was extracted with n-hexene-acetone. After clean-up by column chromatography over a Florisil column, the residues were detected by GC-ECD. Retention times of alpha endosulfan, beta endosulfan and endosulfan sulfate were 6.0 minutes, 9.5 minutes and 10.7 minutes, respectively. The residue of endosulfan was quantitated using an external standard method, and the total of residue of alpha endosulfan, beta endosulfan and endosulfan sulfate was regarded as the residue of endosulfan.

In Table 1 the recovery results are shown for endosulfan (total residue) in fresh leaves of tea.

Table 1 Validation of analytical method for endosulfan (total residue) in fresh leaves of tea

commodity	reported LOQ mg/kg	spike level mg/kg	no	% recovery mean range	RSD <sub>r</sub>	control samples mg/kg (n)	Calibration	reference, method
Fresh tea leaves	0.005	0.005 0.01 0.1 1.0	5 5 5	95.43 89.12-98.16 95.57 94.55-96.21 95.73 91.94-99.75	- 4.02 0.69 3.67	-	-	unknown

- : not available

In Table 2 the recovery results are shown for endosulfan (total residue) in made tea.

<sup>&</sup>lt;sup>14</sup> Made tea: directly after picking the tea leaves are transported to an indoor cool place and then processed (a process of cooling and frying) within one day to the end-product 'made tea'. After processing the tea samples are frozen until analysis.

Table 2 Validation of analytical method for endosulfan (individual residue components) in 'made' tea

commodity	reported LOQ mg/kg	spike level mg/kg	Isomer/metabolite	no	% recovery mean (range)	RSD <sub>r</sub>	control samples mg/kg (n)	Calibration	reference. method
Made tea	0.005	0.005	α-endosulfan β-endosulfan endosulfan- sulfate	-	-	-	-	-	unknown
		0.01	α-endosulfan β-endosulfan endosulfan- sulfate	5	108.8 (98.3–117) 109.8 (101.5–118.5) 112.4	7.02 5.88 7.24			
		0.1	α-endosulfan	5	(103.5–122.2) 100.9				
		0.1	α-endosulfan β-endosulfan endosulfan- sulfate	3	(99.4–103.5) 100.3 (95–102.6) 100.5 (98.3–103.9)	1.58 3.02 3.60			
		1	α-endosulfan β-endosulfan endosulfan- sulfate	5	103.3 (98.7–107) 94.56 (89.7–101.1) 95.44 (93.8–99.3)	3.17 4.37 3.60			
		5	α-endosulfan β-endosulfan endosulfan- sulfate	5	100.4 (93.75–105.3) 106.3 (100.5–109.9) 97.5 (90.5–103.3)	4.36 3.93 6.56			
		50	α-endosulfan β-endosulfan endosulfan- sulfate	5	99.13 (93.68–110) 106.6 (101.3–115.3) 98.59 (90.32–103.9)	7.10 6.17 8.77			

# Stability of pesticide residues in stored analytical samples

For the purpose of studying the storage stability of endosulfan residue in made tea, samples of made tea from field trials were collected. Samples were stored at room temperature (25 °C) and at cold storage conditions (4 °C), and the samples were analysed every 30 days. The results are listed in Table 3. No concurrent recoveries were available.

Table 3 Storage stability of endosulfan residue in 'made tea'

	Content of	Storage Conditions	Content of Endosulfan residue in 'made tea' (mg/kg) after storage for 1–							
	Endosulfan in		4 months							
	'Made Tea'		1 month	2 months	3 months	4 months				
	(mg/kg)		(% remaining)	(% remaining)	(% remaining)	(% remaining)				
Tea Sample	3.645	25 °C	3.524	3.518	3.512	3.486				
No.1			(97)	(96)	(96)	(96)				
		4 °C	3.626	3.612	3.586	3.589				
			(99)	(99)	(98)	(98)				
Tea Sample	10.842	25 °C	10.816	10.796	10.668	10.432				
No.2			(100)	(100)	(98)	(96)				
		4 °C	10.832	10.812	10.794	10.632				
			(100)	(100)	(100)	(98)				

The frozen samples of made tea were analysed 2 to maximally 31 days after harvest. Therefore the storage stability studies cover the results for 'made tea'.

### Residue definition

The 2006 Meeting concluded that the residue definition in plant and animal commodities for enforcement and dietary intake purposes is: the sum of  $\alpha$ - and  $\beta$ - isomer and its main metabolite endosulfan sulphate.

#### **USE PATTERN**

For this year's evaluation new GAP information on green tea was received from China and is shown in Table 4.

Crop	Country	Formu	ılation		Application					
		type	Conc g/L	Method	Water L/ha	Rate kg ai/hL	Spray conc., kg ai/ha			
'Made tea'	China	EC	350	spray	750	0.089	0.668	7		

## RESIDUSE RESULTING FROM SUPERVISED TRIALS ON CROPS

According to the Codex Food Classification, tea is defined as follows. Newly grown vegetative shoots (terminal bud and 2–3 leaves) of tea are plucked, withered, twisted and comminuted and thereafter, in general, fermented and dried. Portion of commodity to which the MRL applies (and which is analysed): Whole commodity as prepared for wholesale or retail distribution.

The present Meeting received a report on field trials on tea which were all performed in China in the years 2004–2007 on four different sites. The residue trials were performed in 30 years old crops in the open field on plots of 0.067 ha with one treated and one control plot (10 rows per plot).

Seventeen decline trials were conducted to determine the endosulfan residue in tea at Hangzhou city (Zhejiang province), Changsha city (Hunan province), Jianou county, (Fujian province) and Huangshan city (Anhui province). Trials were done both at the recommended dosage and at double dosage. During the tea growing stage (one-bud and two-leaf stage), 35% endosulfan EC was sprayed on the tea plant by using a PB-16 hand-operated knapsack sprayer. The area of each plot was 60 square meter, each treatment was done with 3 replicates, blank samples were taken from a control area, the test level included two dose group: recommended dosage (0.668 kg ai/ha) and double dosage (1.226 kg ai/ha). Half an hour after spraying one-bud and two or three-leaf shoot of tea plant was plucked as the 0-day sample of fresh tea leaves, further sampling was performed 1, 3, 5, 7, (10)14, (17) 21 and 25 days later. After collecting the fresh leaves and manufacturing to made tea, the residue level in the fresh leaves was determined, as well as the water content. The rest of the tea was processed to made tea (Longjing Green Tea in Hangzhou, Broken black tea in Hunan, Oolong tea in Fujian and Baked green tea in Anhui). See Table 5.

In addition eight trials were performed to investigate the influence of an additional treatment at the recommended dosage (0.668 kg ai/ha) and double dosage (1.226 kg ai/ha) of 35% endosulfan EC. Tea plant were sprayed either one or two times, the interval between the first spraying and the

second spraying was 7 days. After spraying, the tea fresh leaves were collected after 5, 7 and 14 days, and manufactured into made tea. See Table 6.

Soil samples were tested as well. The results however are not summarised here.

Table 5 Residues of endosulfan in fresh tea leaves and 'made tea' after pre-harvest treatment (decline trials)

Location	Eama	Ma	Luton	1	1	m oth od	DAT	manidaan	manidaan	ma famous a a
Location, year,	Form	No	Inter- val	kg ai/ha	kg ai/hL	method, timing	DAT	residues, mg/kg	residues, mg/kg	reference
,				ai/iia	ai/IIL	tilling		in fresh		
(variety) Trial no.			(days)					tea	in 'made tea' a	
THAI IIO.								leaves	ica	
Hananhau	EC	1		0.668	0.089	Vacancials	0	29	48	TRI/LD/2004/001
Hangzhou	EC	1	-	0.008	0.089	Knapsack	1		26	1 K1/LD/2004/001
Zhejiang province						sprayer; one bud and	4	13 4.4	9.5	
China						2 or 3 leaf	7	1.9	9.3 4.1	
2004						shoot	10	1.9	$\frac{4.1}{3.4}$	
(Longjing 43)						SHOOL	14	0.91	3.4	
(Longjing 43)							20	0.69	2.0	
Hangzhou	EC	1	_	0.668	0.089	Knapsack	0	0.07	26	TRI/LD/2004/002
Zhejiang	LC	1		0.000	0.007	sprayer;	1		14	TRI/LD/2004/002
province						one bud and	4		5.0	
China						2 or 3 leaf	7		2.3	
2004						shoot	10		0.67	
(Longjing 43)						SHOOT	14		0.18	
Hangzhou	EC	1	_	1.226	0.178	Knapsack	0		57	TRI/LD/2004/002
Zhejiang						sprayer;	1		35	
province						one bud and	4		6.5	
China						2 or 3 leaf	7		2.6	
2004						shoot	10		0.78	
(Longjing 43)							14		0.31	
Changsha	EC	1	-	0.668	0.089	Knapsack	0		25	TRI/LD/2004/003
Hunang						sprayer;	1		4.0	
province						one bud and	4		3.6	
China						2 or 3 leaf	7		<u>2.5</u>	
2004						shoot	10		1.3	
Chingjing 43)							14		0.86	
Changsha	EC	1	-	1.226	0.178	Knapsack	0		72	TRI/LD/2004/003
Hunang						sprayer;	1		8.6	
province						one bud and	4		5.1	
China						2 or 3 leaf	7		4.1	
2004						shoot	10		2.7	
Chingjing 43)	EC	1		0.660	0.000	IZ	14	21	2.4 36	TRI/LD/2005/004
Changsha	EC	1	-	0.668	0.089	Knapsack	1	5.8	13	1 K1/LD/2005/004
Hunang province						sprayer; one bud and	4	2.4	4.9	
China						2 or 3 leaf	7	1.1	2.0	
2005						shoot	10	0.46	1.5	
(Longjing 43;						SHOOL	14	0.46	0.90	
Hunan black							20	0.23	0.45	
tea)							20	0.12	0.45	
Changsha	EC	1	_	1.226	0.178	Knapsack	0	37	60	TRI/LD/2005/004
Hunang		-				sprayer;	1	11	23	2000,001
province						one bud and	4	3.8	11	
China						2 or 3 leaf	7	1.9	4.4	
2005						shoot	10	1.0	2.2	
(Longjing 43)							14	0.38	1.5	
							20	0.21	0.68	

Location, year, (variety) Trial no.	Form	No	Interval (days)	kg ai/ha	kg ai/hL	method, timing	DAT	residues, mg/kg in fresh tea leaves	residues, mg/kg in 'made tea' <sup>a</sup>	reference
Hangzhou Zhejiang province China 2006 (Longjing 43)	EC	1	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3 leaf shoot	0 1 3 5 7 10 14 17 20 25	31 29 7.6 3.3 3.1 1.5 0.68 0.47 0.42 0.11	23 12 7.4 5.4 3.4 2.1 1.0 0.89 0.75 0.28	TRI/LD/2006/005
Hangzhou Zhejiang province China 2006 (Longjing 43)	EC	1	-	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	0 1 4 7 10 14 20	45 16 5.3 2.6 1.8 1.3 0.76	79 32 12 6.8 4.7 3.7 2.0	TRI/LD/2006/005
Changsha Hunang province China 2006 (Longjing 43)	EC	1	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3 leaf shoot	0 1 3 5 7 10 14 17 20		29 21 7.5 5.7 <u>4.3</u> 2.7 2.2 1.3 0.82	TRI/LD/2006/006
Hangzhou Zhejiang province China 2007 (Longjing 43)	EC	1	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3 leaf shoot	0 1 3 5 7 10 14 21	15 9.8 5.9 3.5 2.3 1.5 0.68 0.22	30 22 11 8.5 4.2 3.3 1.8 0.42	TRI/LD/2007/007
Changsha Hunang province China 2007 (Longjing 43; Hunan black tea)	EC	1	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3 leaf shoot	0 1 3 5 7 14	25 16 7.3 4.0 0.59 0.10	41 33 13 9.4 2.0 0.33	TRI/LD/2007/008
Changsha Hunang province China 2007 (Longjing 43)	EC	1	-	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	0 1 3 5 7 14 21	41 32 9.6 4.4 0.61 0.11 0.089	76 51 13 11 3.5 0.42 0.23	TRI/LD/2007/008
Huangshan city Anhui Province China 2007 (Longjing 43)	EC	1	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3 leaf shoot	0 1 3 5 7 14 21	21 9.3 5.3 3.1 2.1 0.99 0.32	43 16 8.7 5.4 <u>3.2</u> 1.0 0.34	TRI/LD/2007/009

Location, year, (variety) Trial no.	Form	No	Interval (days)	kg ai/ha	kg ai/hL	method, timing	DAT	residues, mg/kg in fresh tea leaves	residues, mg/kg in 'made tea' <sup>a</sup>	reference
Huangshan city Anhui Province China 2007 (Longjing 43)	EC	1	-	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	0 1 3 5 7 14 21 28	33 22 6.4 3.5 2.9 1.3 0.45 0.26	101 43 9.8 6.8 4.4 1.4 0.54 0.22	TRI/LD/2007/009
Jianou county Fujian Province China 2007 (Longjing 43)	EC	1	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3 leaf shoot	0 1 3 5 7 14 21	14 9.2 2.5 1.9 1.2 0.54 0.22	20 15 7.4 3.1 2.5 1.0 0.3	TRI/LD/2007/010
Jianou county Fujian Province China 2007 (Longjing 43)	EC	1	ı	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	0 1 3 5 7 14 21	22 9.7 2.6 2.2 1.5 0.59 0.40	22 17 9.2 2.6 2.6 1.2 0.54	TRI/LD/2007/010

<sup>&</sup>lt;sup>a</sup> The residues are measured in 'made tea', i.e.: the fresh green tea leaves (one bud and 2 or 3 leaf shoots from the tea plant; 5-6 units) are picked on the required pre harvest interval (days 0 up to day 21 after treatment), and are further manufactured to 'made tea' on the same day; then the samples are stored in the freezer until analysis (at -15 °C for 2 to max 32 days). For each trial all samples were analysed on the same day (several days after the last sample picking). Control plots were sampled on days 0, 7 and 20 and the results of all control plots were below the LOQ (0.005 mg/kg).

Table 6 Residues of endosulfan in fresh tea leaves and 'made tea' after pre-harvest treatment

Location, year, (variety) Trial no.	Form	No	Interval (days)	kg ai/ha	kg ai/hL	method, timing	DAT	residues, mg/kg in 'made tea' a	reference
Hangzhou Zhejiang province China 2004 (Longjing 43)	EC	1	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3 leaf shoot	5 7 14	8.2 4.1 1.4	Report 2008
Hangzhou Zhejiang province China 2004 (Longjing 43)	EC	2	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3 leaf shoot	5 7 14	11 5.4 2.8	Report 2008
Hangzhou Zhejiang province China 2004 (Longjing 43)	EC	1	-	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	5 7 14	12 6.8 2.7	Report 2008
Hangzhou Zhejiang province China 2004 (Longjing 43)	EC	2	-	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	5 7 14	12 9.5 3.2	Report 2008
Changsha Hunang province China 2004 (Chingjing 43)	EC	1	-	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	5 7 14	12 4.4 1.7	Report 2008

Location,	Form	No	Inter-	kg	kg ai/hL	method, timing	DAT	residues,	reference
year, (variety)			val (days)	ai/ha		, ,		mg/kg in 'made tea' a	
Trial no. Changsha	EC	2	_	1.226	0.178	Knapsack sprayer;	5	14	Report
Hunang province China 2004 (Chingjing 43)	EC	2		1.220	0.178	one bud and 2 or 3 leaf shoot	7 14	8.2 4.3	2008
Hangzhou	EC	1	-	0.668	0.089	Knapsack sprayer;	7	4.7	Report
Zhejiang province China 2006 (Longjing 43)						one bud and 2 or 3 leaf shoot	14	<u>4.7</u> 1.7	2008
Hangzhou Zhejiang province China 2006 (Longjing 43)	EC	2	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3 leaf shoot	7 14	9.1 2.4	Report 2008
Hangzhou Zhejiang province China 2006 (Longjing 43)	EC	1	-	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	7 14	8.6 4.7	Report 2008
Hangzhou Zhejiang province China 2006 (Longjing 43)	EC	2	-	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	7 14	14 4.0	Report 2008
Changsha Hunang province China 2006 (Chingjing 43)	EC	1	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3 leaf shoot	7 14	4.3 2.2	Report 2008
Changsha Hunang province China 2006 (Chingjing 43)	EC	2	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3 leaf shoot	7 14	11 4.0	Report 2008
Changsha Hunang province China 2006 (Chingjing 43)	EC	1	-	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	7 14	8.9	Report 2008
Changsha Hunang province China 2006 (Chingjing 43)	EC	2	-	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	7 14	16 5.6	Report 2008
Hangzhou Zhejiang province China 2007 (Longjing 43)	EC	1	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3 leaf shoot	5 7 14	5.3 4.3 2.7	Report 2008
Hangzhou Zhejiang province China 2007 (Longjing 43)	EC	2	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3 leaf shoot	5 7 14	6.8 4.8 3.4	Report 2008
Hangzhou Zhejiang province China 2007 (Longjing 43)	EC	1	-	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	5 7 14	10 9.6 3.9	Report 2008

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Location, year,	Form	No	Inter- val	kg ai/ha	kg ai/hL	method, timing	DAT	residues, mg/kg in	reference
(variety)			(days)	ai/iia				'made	
Trial no.			(days)					tea' a	
Hangzhou	EC	2	-	1.226	0.178	Knapsack sprayer;	5	15	Report
Zhejiang province	LC	_		1.220	0.170	one bud and 2 or 3	7	12	2008
China						leaf shoot	14	4.6	2000
2007									
(Longjing 43)									
Changsha	EC	1	-	0.668	0.089	Knapsack sprayer;	5	6.8	Report
Hunang province						one bud and 2 or 3	7	<u>4.1</u>	2008
China						leaf shoot	14	1.1	
2007									
(Chingjing 43)									
Changsha	EC	2	-	0.668	0.089	Knapsack sprayer;	5	8.4	Report
Hunang province						one bud and 2 or 3	7	5.3	2008
China						leaf shoot	14	2.6	
2007 (Chinging 42)									
(Chingjing 43) Changsha	EC	1	_	1.226	0.178	Knapsack sprayer;	5	11	Report
Hunang province	EC	1	-	1.220	0.1/8	one bud and 2 or 3	7	3.5	2008
China						leaf shoot	14	1.4	2000
2007						icai siloot	17	1.4	
(Chingjing 43)									
Changsha	EC	2	-	1.226	0.178	Knapsack sprayer;	5	16	Report
Hunang province						one bud and 2 or 3	7	9.4	2008
China						leaf shoot	14	1.5	
2007									
(Chingjing 43)									
Huangshan city	EC	1	-	0.668	0.089	Knapsack sprayer;	5	3.0	Report
Anhui Province						one bud and 2 or 3	7	<u>2.2</u>	2008
China						leaf shoot	14	1.0	
2007									
(Longjing 43)	EC	2		0.669	0.000	V	-	4.0	Donost
Huangshan city Anhui Province	EC	2	-	0.668	0.089	Knapsack sprayer; one bud and 2 or 3	5 7	4.9 3.8	Report 2008
China						leaf shoot	14	1.8	2008
2007						icai siloot	17	1.0	
(Longjing 43)									
Huangshan city	EC	1	-	1.226	0.178	Knapsack sprayer;	5	6.9	Report
Anhui Province						one bud and 2 or 3	7	4.4	2008
China						leaf shoot	14	1.4	
2007									
(Longjing 43)									
Huangshan city	EC	2	-	1.226	0.178	Knapsack sprayer;	5	8.4	Report
Anhui Province						one bud and 2 or 3	7	6.7	2008
China						leaf shoot	14	2.2	
2007 (Longjing 43)									
Jianou county	EC	1	_	0.668	0.089	Knapsack sprayer;	5	2.3	Report
Fujian Province	EC	1	-	0.008	0.009	one bud and 2 or 3	7	1.9	2008
China						leaf shoot	14	$\frac{1.9}{0.50}$	2000
2007							1		
(Longjing 43)									
Jianou county	EC	2	-	0.668	0.089	Knapsack sprayer;	5	3.9	Report
Fujian Province						one bud and 2 or 3	7	3.1	2008
China						leaf shoot	14	0.90	
2007									
(Longjing 43)									
<u> </u>					I .				

Location, year, (variety) Trial no.	Form	No	Inter- val (days)	kg ai/ha	kg ai/hL	method, timing	DAT	residues, mg/kg in 'made tea' <sup>a</sup>	reference
Jianou county Fujian Province China 2007 (Longjing 43)	EC	1	-	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	5 7 14	2.6 2.6 0.54	Report 2008
Jianou county Fujian Province China 2007 (Longjing 43)	EC	2	-	1.226	0.178	Knapsack sprayer; one bud and 2 or 3 leaf shoot	5 7 14	4.9 4.0 1.3	Report 2008

<sup>&</sup>lt;sup>a</sup> The residues are measured in 'made tea', i.e.: the fresh green tea leaves (one bud and 2 or 3 leaf shoots from the tea plant; 5-6 units) are picked on the required pre harvest interval (days 0 up to day 21 after treatment), and are further manufactured to 'made tea' on the same day.

## Fate of residues in storage and processing

The Meeting received information on the fate of incurred residues of endosulfan during the processing of fresh tea leaves to 'made tea'. Since the commodity moving in trade is 'made tea', these results are not summarised. In addition, information was available on residues in tea infusions.

## Residues in the edible portion of food commodities

To 3.0g crushed and blended made tea 150 mL boiling water was added. After waiting for 20 minutes, the infusion was removed. This was repeated twice and the infusions were extracted with n-hexane-acetone and analysed after clean-up.

Table 7 Extraction rate of endosulfan residue during tea infusion

Samples	Endosulfan residue in 'made tea' (mg/kg)	Extraction rate – first infusion (%)	Extraction rate - second infusion (%)	Extraction rate - third infusion (%)	Sum of Extraction rates (%)
Hangzhou Green Tea (No.1)	50.36	4.49	2.47	1.31	8.27
Hangzhou Green Tea (No.2)	40.86	3.83	2.34	1.81	7.98
Hangzhou Green Tea (No.3)	28.1	3.25	2.01	1.87	7.13
Hangzhou Green Tea (No.4)	13.6	3.45	2.13	1.93	7.51
Mean		3.755	2.238	1.730	7.720
Fujian Oolong Tea (No.1)	66.2	3.80	2.78	1.80	8.38
Fujian Oolong Tea (No.2)	34.5	4.04	2.24	1.51	7.79
Fujian Oolong Tea (No.3)	12.45	3.13	3.05	2.14	8.32
Fujian Oolong Tea (No.4)	9.36	3.42	2.19	2.83	8.44
Mean		3.598	2.565	2.07	8.233
Hunan Black Tea (No.1)	57.1	3.57	3.08	1.81	8.46
Hunan Black Tea (No.2)	19.25	2.76	2.62	2.65	8.03
Hunan Black Tea (No.3)	12.31	3.30	3.24	3.19	9.73
Mean		3.210	2.980	2.550	8.740
Anhui Baked Green Tea (No.1)	57.8	3.24	3.33	1.22	7.79
Anhui Baked Green Tea (No.2)	31.9	3.63	2.26	1.52	7.41
Anhui Baked Green Tea	10.11	3.86	2.27	2.51	8.64

Samples	Endosulfan residue in 'made tea' (mg/kg)	Extraction rate – first infusion (%)	Extraction rate - second infusion (%)	Extraction rate - third infusion (%)	Sum of Extraction rates (%)
(No.3)					
Anhui Baked Green Tea (No.4)	7.68	3.52	2.30	2.25	8.07
Mean		3.563	2.540	1.875	7.978

### Summary table extraction rates.

Commodity	Extraction Rate of Endosulfan (%)
Green Tea (Hangzhou and Anhui)	7.85
Black Tea (Hunan)	8.74
Oolong Tea (Fujian)	8.23
Mean	8.27

#### APPRAISAL

Endosulfan is a synthetic cyclodiene non-systemic insecticide and acaricide with both contact and stomach activity. It is has been widely used in agriculture to control a range of insects and mites on a broad spectrum of crops. It has been evaluated several times by the JMPR; the initial evaluation for residues was in 1967 and the latest in 2006. Under the CCPR Periodic Review Programme the toxicology was re-evaluated in 1998. The Meeting established an ADI of 0–0.006 mg/kg bw and an acute reference dose (ARfD) of 0.02 mg/kg bw. In 2006, a Periodic Review of the residue-analytical aspects was completed.

In the 2006 review, the Meeting was not able to recommend a maximum residue level for tea, as trials from India could not be matched against the provided GAPs from China, Japan or Malaysia. At the Thirty-ninth Session of CCPR in 2007, on the proposal of the delegations of China and India the CXL for tea (green, black) was retained for four years under the Periodic Review Programme. The Forty-first Session of CCPR in 2009 scheduled the review of data on tea from China by the 2010 JMPR. GAP for tea in China and new trials in tea performed over several years (2004–2007) were submitted by the Government of China.

### Methods of analysis

The Meeting received a description and validation data of a GC-ECD analytical method for residues of total endosulfan (alpha endosulfan, beta endosulfan and endosulfan sulphate) in fresh leaves of tea. in made tea, and in tea infusions. The recoveries of the method for endosulfan (total residue) are satisfactorily over a range of 0.01-50 mg/kg in made tea.

## Results of supervised trials on tea

The present Meeting received 17 decay trials and 30 residue trials (2-point decline) on tea, green and black, which were performed over a period of four years in four different provinces within China. GAP in China is one treatment at an application rate of 0.668 kg ai/ha (0.089 kg ai/hL, 750 L water/ha) with a pre-harvest interval of 7 days.

Ten of the decay trials were at GAP. Total endosulfan residue levels in 'made tea', were, in ranked order: 2.0(2), 2.3, 2.5, 2.5, 3.2, 3.4, 4.1, 4.2 and 4.3 mg/kg.

Seven of the 2-point decline trials were at GAP. Total endosulfan residue levels in 'made tea', in ranked order, were: 1.9, 2.2, 4.1(2), 4.3(2), 4.7 mg/kg. Since the decay trials and the decline trials

appear not to be independent, the Meeting decided to estimate a maximum residue level based on the data set yielding the highest STMR, the terminal residue trials.

The Meeting estimated a maximum residue level of 10 mg/kg for 'made tea', and an STMR of 4.1 mg/kg.

The maximum residue level estimate derived from use of the NAFTA calculator was 6 mg/kg.

## Fate of residues during processing

The Meeting received information on the extraction rate of endosulfan residue during tea infusion. Tea infusions were prepared by adding boiling water to dried and processed tea leaves ('made tea') and allowed to stand for 20 minutes. This was repeated twice. The infusion was filtered and the % infusion (% of residue extracted in the boiling water) was calculated. This was done  $4\times$  for Hangzhou Green Tea,  $4\times$  for Fujian Oolong Tea,  $3\times$  for Hunan Black Tea,  $4\times$  for Anhui Baked Tea. After 3 infusions for endosulfan (total residue) the mean %infusion (or extraction rate) was 8.3%, range 7.1-9.7%, n=15.

The Meeting estimated an STMR-P in tea infusion of 0.34 mg/kg.

#### Residues in animal commodities

Since tea is not an animal feed item, the recommendations for animal commodities as made by the 2006 Meeting are still valid.

#### RECOMMENDATIONS

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

Definition of the residue for compliance with MRLs and for estimation of dietary intake for plant and animal commodities: *sum of alpha endosulfan, beta endosulfan and endosulfan sulfate*.

The residue is fat soluble.

Codex Code	Commodity	MRL (mg/kg	()	STMR (mg/kg)
		New	Previous	
DT 1114	Tea, Green, Black	10	W	4.1
	Tea infusion			0.34

### **DIETARY RISK ASSESSMENT**

## Long-term intake

In 2006 the Meeting concluded that the long term intake of residues of endosulfan from uses that have been considered by the JMPR is unlikely to present a public health concern. The IEDI in the thirteen GEMS/Food regional diets, on the basis of the estimated STMRs, represented 2–20% of the maximum ADI of 0.006 mg/kg bw.

Due to the low contribution of tea in the entire diet, no revision of the chronic dietary exposure assessment has been carried out.

### Short-term intake

In the 2006 evaluation no short-term intake for tea was calculated.

Based on the STMR-P of 0.34 mg/kg for tea (green, black), the short-term intake for both children  $\leq 6$  years and for the general population represented 1% of the ARfD. The Meeting concluded that the short-term intake of endosulfan from its use on tea (green, black) was unlikely to present a public health concern.

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