CAPTAN (007)

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EXPLANATION

Captan is a contact fungicide with a multi-site mode of action. It is used to control a broad range of diseases on a variety of crops. Captan was first reviewed by JMPR in 1963 with subsequent residue reviews between 1969 and 1997. In 2000, a periodic review for residues was conducted, with subsequent reviews for toxicology conducted in 2004 and 2007.

The 1995 JMPR established an ADI of 0–0.1 mg/kg bw/day for captan. This same year, the residue definition established for plant and animal commodities, for both compliance with MRLs and for dietary intake assessment was captan. In 2004, the WHO established an ARfD of 0.3 mg/kg bw for women of childbearing age and unnecessary for the general population.

Specifications for captan technical material and relevant formulations have been established by the JMPS in 1990 and published on the AGP-FAO Specifications webpage.

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METHODS OF RESIDUE ANALYSIS

Analytical methods

The 2000 JMPR reviewed and summarized analytical method descriptions and validation data for captan, generally involving maceration of the sample with a solvent, which is usually ethyl acetate or acetone. As captan is readily hydrolysed at high pH, a small quantity of phosphoric acid is often added at the extraction step in order to lower the pH. Extracts of captan are cleaned-up on a silica column prior to analysis on a gas chromatograph equipped with an electron capture detector (GC-ECD). Typical LOQs are 0.01 mg/kg.

The ginseng samples from the supervised field trials were analysed using a modified version of the GC-ECD analytical method titled "Residue Analysis of Captan on Ginseng by GC/Electron Capture Detection. Version #2A" which is derived from "Determination by Gas Chromatography of Captan and Tetrahydrophthalimide Residues in Raw and Processed Agricultural Crops" (Report No. WRC 89-51, ICI Americas Inc., Richmond, CA 94804-0023, June 1, 1989). Briefly, the method involved blending samples with 85% phosphoric acid and ethyl acetate. The extract was then filtered and shaken with 1% phosphoric acid. The mixture was passed through sodium sulfate and dried. The residue was dissolved in methylene chloride and cleaned up on a silica gel column eluted with 5% ethyl acetate in methylene chloride. The captan residues were then analysed by GC-ECD. The lowest limit of method validation (LLMV) was established at 0.05 mg/kg.

Table 2 Summary of recoveries of captan from ginseng

Type of Recovery	Fortification Levels (mg/kg)	Average Recoveries (%)	Range of Recoveries (%)	% RSD	Number of Recoveries
Method Validation	0.05	83	70-92	13.2	5
	0.5	77	68-84	7.7	5
Concurrent Validation	0.05	80	68-110	22.5	8
	0.5	55	48-58	10.6	3

Note: Some of the concurrent recoveries were re-injections of the same extract after the GC column was changed

USE PATTERN

Different types of formulations of captan are currently registered in the USA and Canada for use on various fruits, vegetables, legumes, pulses and oilseeds. These end-use products are typically applied as foliar sprays using ground and aerial equipment.

Table 2 Registered uses of captan on ginseng

Country	Form	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	Maximum Rate (kg ai/ha/season)	PHI (days)
Canada	80% DF	2.0	935-1871	8	7	16.0	20
USA	80% WDG	3.4	935-1871	8	6-8	27.0	14

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised field trials for captan on ginseng.

Trials were generally well documented with laboratory and field reports. Laboratory reports included method validation with procedural recoveries from spiking at levels similar to those occurring in samples from the supervised field trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables as residues in control samples did not exceed the LOQ. Unless stated otherwise, residue data are recorded unadjusted for recovery. Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Conditions of the supervised field trials were well reported in detailed field reports. Trial designs used non-replicated plots. Field reports provided data on the sprayers used, plot size, field sample size and sampling date.

A total of four crop field trials were conducted in Canada and the USA on ginseng during the 2005-2006 growing seasons (Corley, 2009, 07997.05-NYR10). The treated plots received eight foliar broadcast applications, of a water dispersible granule formulation containing 80% captan, at a nominal rate of 3 kg ai/ha/application. The spray volumes ranged from 919 to 1930 L/ha. No tank mix adjuvants were added to the spray mixes. Ginseng was harvested 12–15 days after the last application (DALA). The roots were dried to a commercially acceptable texture, in drying facilities or research facilities simulating commercial drying practices. No decline samples were collected from the trials.

The ginseng samples were analysed using the validated GC-ECD analytical method. Captan residues found in the treated samples from the 2006 BC trial varied between < 0.050 and 1.0 mg/kg. The first analytical set had an average recovery of 68% (0.050 mg/kg fortification level) but the two injections were inconsistent (58 and 78%) and residues were found to be between < 0.050 and 0.070 mg/kg. A second set of samples were ground, extracted and analysed and residues between < 0.050 and 0.46 mg/kg were found with a spike recovery of 110%. A third set of samples were ground, extracted and analysed and residues between 0.31 and 1.0 mg/kg were found with a spike recovery of 70%. This set was reinjected resulting in residues of 0.24 and 0.66 mg/kg and a spike recovery of 102%. Due to the inconsistency in results a new column was installed on the GC and the samples were again injected and residues between 0.45 and 1.0 mg/kg were found with a spike recovery of 76%. A fourth set of samples were ground, extracted and analysed and residues between 0.096 and 0.16 mg/kg were found with a spike recovery of 58% (0.50 mg/kg fortification level). This set was re-injected together with the set above after installation of the new column and residues between 0.070 and 0.14 mg/kg were found with a fortification recovery of 48%. The latter set was injected a third time and residues between 0.11 and 0.17 mg/kg were found with a fortification recovery of 58%. There was no clear explanation to the low spike recovery of the fourth set.

All harvested samples were ground up immediately prior to extraction and analysis which occurred within 6–9 days of sampling except for the re-analysis of the BC01 (re-extracted up to 31 days after sampling), therefore, no storage stability information was required.

Table 3 Captan residues in ginseng from supervised field trials in the USA and Canada

Location Year Trial ID (variety)	Application						DALA	Captan Residues [average] (mg/kg)
	Form	Spray Volume (L/ha)	Rate (kg ai/ha)	No.	RTI (days)	Maximum rate (kg ai/ha/season)		
USA GAP	80% WDG	935-1871	3.36	8	6-8	27.0	14	
Coldstream, BC, Canada, 2006 BC01 (American Ginseng)	80% WDG	1868 1808 1784 1809 1824 1877 1930 1789	3.48 3.37 3.32 3.37 3.40 3.50 3.60 3.22	8	- 7 8 7 7 8 6 6	27.4	15	Initial set:{0.072, 0.069}, {< 0.05, < 0.05} Mean [0.06] 2nd set:{0.51, 0.41}, {< 0.05, < 0.05} Mean [0.255] 3rd set: {**0.99, 1.0**, 0.67[a], 0.64[a], 0.98[a], 1.1[a]}, {0.32, 0.30, 0.24, 0.23, 0.48, 0.42} Mean [0.538] 4th set: {0.092, 0.099, 0.072, 0.068, 0.11, 0.11}, {0.16, 0.16, 0.14, 0.13, 0.19, 0.15} Mean [0.123] Mean of all 4 sets: [0.244]
Athens, WI, USA, 2005 MI23 (American Ginseng)	80 WDG	919 957 967 970 994 940 970 992	3.29 3.43 3.46 3.48 3.56 3.37 3.48 3.56	8	- 8 7 7 8 6 7 7	27.7	15	< 0.05, 0.05 [0.05]
Athens, WI, USA, 2005 WI17 (American Ginseng)	80 WDG	1622 1645 1674 1602 1600 1533 1682 1600	3.46 3.34 3.48 3.30 3.32 3.19 3.41 3.39	8	- 13 13 13 6 8 6 6	26.9	12	< 0.05, < 0.05 [< 0.05]
Otterville, ON, Canada, 2005 ON05 (Land Race)	80 WDG	998 1035 992 993 1017 1009 995 1035	3.34 3.47 3.33 3.33 3.41 3.39 3.34 3.47	8	- 7 7 7 8 7 6 7	27.11	13	< 0.05, < 0.05 [< 0.05]

THPI residues were < 0.05 mg/kg in all trials.

{values in parentheses} represent the same analytical set *i.e.* the same sample re-injected

The values in **bold** (0.99 and 1.0 mg/kg) were from a sample which was re-injected undiluted. These values are not included in the mean calculation

[a] These 4 values are from a sample which was diluted 2×

Note: As application dates were ≥ 18 days between trials MI23 and WI17, these were considered independent.

APPRAISAL

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A summary report of recovery data of the GC-ECD method for the analysis of captan in ginseng was provided. Mean recoveries from the method validation at 0.05 and 0.5 mg/kg levels ranged from 77 to 83% (n=5/fortification level, range of recoveries: 70–92% and 68–84%, respectively) with % RSD < 14%. For the concurrent method validation at the same fortification levels, mean recoveries ranged from 55–80% (n=5/fortification level, range of recoveries: 68–110% and 48–58%, respectively) with % RSD < 23%.

Results of supervised residue trials on crops

In the USA, the critical GAP for captan on ginseng is 3.36 kg ai/ha with a maximum of 8 applications or 27 kg ai/ha/season and a PHI of 14 days.

A total of four independent crop field trials were conducted in Canada and the USA on ginseng during the 2005-2006 growing seasons. All trials were conducted in accordance with the USA critical GAP of 26.88 kg ai/ha/season. Residues in ginseng samples were (n=4): < 0.05 (2), 0.05 and 0.54 mg/kg.

The Meeting concluded that there were analytical issues that precluded sufficient confidence in the representativeness of the captan residues for estimating a maximum residue level.

REFERENCES

Code	Author	Year	Title, Institute
IR-4 Project number 07997	Johannes Corley	2008	Captan: Magnitude Of The Residue On Ginseng; IR-4 Project HQ, Rutgers, The State University of New Jersey, Princeton, NJ 08540; dated: 10 November 2009. GLP, not published