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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
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OFFICE OF  
PREVENTION, PESTICIDES AND  
TOXIC SUBSTANCES

**MEMORANDUM**

**SUBJECT:** Response to the Malathion Reregistration Standard: Independent Laboratory Validation of a Malathion Analytical Method for Crop Matrices. (MRID No. 43630301, CBRS No. 15600, Barcode: D215369).

**FROM:** David E. Hrdy, Biologist  
Reregistration Section 2  
Chemistry Branch II: Reregistration Support  
Health Effects Division (7509C)

**THRU:** Robert Quick, Acting Section Head  
Reregistration Section 2  
Chemistry Branch II: Reregistration Support  
Health Effects Division (7509C)

**TO:** Larry Schnaubelt PM 72/Susan Jennings  
Reregistration Branch  
Special Review & Reregistration Division (7508W)

**BACKGROUND**

Tolerances for residues in/on food/feed commodities are expressed in terms of malathion (*O,O*-dimethyl dithiophosphate of diethyl mercaptosuccinate) [40 CFR §180.111, §185.3850, §185.7000, and §186.3850]. The HED Metabolism Committee has determined that the parent compound malathion and the malaoxon metabolite are the compounds to be regulated. Codex MRLs exist for residues of malathion *per se* in/on various plant and processed commodities. The Codex MRLs and the U.S. tolerances will be incompatible when the U.S. tolerance expression for plant commodities is revised to include both residues of malathion and the metabolite malaoxon.

The qualitative nature of malathion residues in alfalfa, lettuce, cotton, and wheat is adequately understood. The qualitative nature of the residue resulting from oral dosing of ruminants and poultry is adequately understood. However, if the direct livestock treatment uses of malathion are supported, appropriate dermal metabolism studies are required.

An Agency memorandum (CBRS 9969, DP Barcode D178988, 7/30/92, R. Perfetti) required that the plant substrate(s) in the metabolism study be analyzed by the analytical methods developed for data collection and tolerance enforcement to assure that these methods are capable of adequately recovering malathion and malaoxon. In response, Cheminova Agro A/S has submitted (1995; MRID 43630301) a description of independent laboratory validation data for a proposed enforcement method, Method M-1886, for the determination of malathion and malaoxon in/on plant matrices. The four matrices analyzed by method M-1886 were (1) a dry matrix, alfalfa hay, (2) an oily matrix, cottonseed, (3) a sugary matrix, whole orange, and (4) a watery matrix leaf lettuce. The independently performing laboratory was AGVISE Laboratories, Inc. The Conclusions and Recommendations stated below apply only to the independent laboratory

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validation of the proposed method M-1886. All other residue chemistry data requirements stated in the Malathion Reregistration Standard Guidance Document are not addressed herein.

Adequate methods are available for the enforcement of established malathion tolerances. The Pesticide Analytical Manual (PAM) Vol. II lists a TLC method, a GLC method with KCl thermionic detection, and a spectrophotometric method as Methods I, II, and III, respectively, for the enforcement of malathion tolerances. The Residue Chemistry Science Chapter of the Malathion Reregistration Standard, dated 7/87, recommends use of the GLC method for tolerance enforcement.

**CONCLUSIONS/RECOMMENDATIONS**

1. The independent laboratory validation data for the proposed GLC/FPD method (Method M-1886) are adequate to satisfy validation data requirements in plants. The method adequately recovered residues of malathion and malaoxon from samples of alfalfa, oranges, cottonseed, and lettuce. All four matrices achieved recoveries (<70%). Please refer to the Table below for recovery data. No additional validation data are required.
2. The matrices and their resultant recoveries are shown in Table 1.
3. The fortification levels ranged from 0.0103 to 40.3 ppm for malathion and 0.0102 to 4.51 ppm for malaoxon in leaf lettuce and whole oranges. Alfalfa hay had fortifications of 0.0510 to 504 ppm of malathion and 0.0150 to 56.4 ppm of malaoxon. Un-delinted cottonseed had fortifications of 0.0515 to 50.4 ppm of malathion and 0.0510 to 5.64 ppm of malaoxon.
4. When the tolerance expression is revised to include both malathion and malaoxon, an adequate enforcement method to determine both malathion and malaoxon in plant commodities will be needed. Before the proposed method can be considered suitable for enforcement purposes, a method tryout, is required. Once the method has been successfully validated by the Agency, it will be designated as an enforcement method.

**DETAILED CONSIDERATIONS**

Table 1. **Average Malathion and Malaoxon Recoveries in Various Plant Matrices Using Method M-1886**

Sample Description	Malathion Recovery Ranges	Average Malathion Recovery	Malaoxon Recovery Ranges	Average Malaoxon Recovery
Leaf Lettuce	96 to 164%	114 ±25%	100 to 118%	106 ±5.8%
Whole Oranges	81 to 111%	94 ±12%	97 to 126%	109 ±12%
Alfalfa Hay	74 to 113%	90 ±14%	75 to 117%	93 ±16%
Un-delinted Cottonseed	93 to 101%	92 ±7.6%	85 to 113%	103 ±11%

The LOQ of the method, as specified by developers, is 0.05 ppm.

The three analytical methods evaluated were:

- (1) *En-Cas Method No. ENC-10/94* entitled, "Analytical Method for the Gas Chromatographic Determination of Malathion and Malaoxon Residues in/on Leaf Lettuce When Using Continuous Automated Sample

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Injections" (see MRID #43367201).

(2)*En-Cas Method No. Enc-4/93* entitled "Analytical Method for the Gas Chromatographic Determination of Malathion and Malaoxon Residues in Oranges When Using Continuous Automated Sample Injections".

(3)*En-Cas Method No. Enc-19/94* entitled, "Analytical Method for the Gas Chromatographic Determination of Malathion and Malaoxon Residues in/on Alfalfa When Using Continuous Automated Sample Injections" (MRID #43546101).

(4)*En-Cas Method No. Enc-22/94* entitled, "Analytical Method for the Gas Chromatographic Determination of Malathion and Malaoxon Residues in/on Cottonseed When Using Continuous Automated Sample Injections"(MRID #43596601).

A method tryout will be requested by CBRS. The tryout request will be the subject of a separate memo.

**cc : Malathion Reregistration Standard File & Malathion Subject File, Circulation**  
**7509C:CBRS:DEH:CM#2:Rm804:703 305-6990:8/18/95**  
**RDI: RQuick:8/29/95 RBPerfetti:9/5/95 EZager:8/29/95**  
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