UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF PESTICIDE PROGRAMS
ENVIRONMENTAL CHEMISTRY LABORATORY
Mail Code 7503ECB
Stennis Space Center, MS 39529-6000
(228) 688-3216

October 27, 2008

MEMORANDUM

SUBJECT: Oxamyl - ECM0243W1-W2 DP # D351881

FROM: Joseph Ferrario, Branch Chief
BEAD/Environmental Chemistry Laboratory

TO: Margaret Ervin ECM Gatekeeper
OPP/Environmental Fate and Effects Division
EISB 7507P

The EFED/Environmental Fate and Effects Division has requested an Environmental Chemistry Method Review of a method for the determination of Oxamyl and Oxime in water (MRID No. 455916-01). The method was submitted by E.I. du Pont de Nemours and Company in accordance with the registration of the above mentioned analytes. The method validation data was reviewed and the conclusions included in the attached Environmental Chemistry Method Review Report.

The following report includes an overview of the method and the method completeness, statements of adherence to EPA regulations, a presentation of results and a discussion of problems found in the registrant method and those discovered by the independent laboratory. A statement of method acceptability is also included.

If you have any questions concerning this report, please contact Elizabeth Flynt at (228) 688-2410 or me at (228) 688-3212.

Attachments
cc: Dr. Christian Byrne, QA Officer
BEAD/Environmental Chemistry Laboratory

Elizabeth C. Flynt
BEAD/ECL

Oxamyl in Water//E.I. du Pont de Nemours and Company/71654

ENVIRONMENTAL CHEMISTRY METHOD REVIEW REPORT

Data Requirement: PMRA Data Code: NA
EPA DP Barcode: D351881
OECD Data Point: NA
EPA Guideline: ECM Method Review

Test material:
Common name: Oxamyl
Chemical name: Methyl 2-(dimethylamino)-N-[[(methylamino)carbonyl]oxy]-2-oxoethanimidothioate
IUPAC name: (EZ)-*N,N*-dimethyl-2-methylcarbamoyloxyimino-2-(methylthio)acetamide

Primary Evaluator: Elizabeth Flynt **Date:** 10/27/08
Elizabeth Flynt, Chemist

Peer Reviewer: Charles Kennedy **Date:** 10/27/08
Charles Kennedy, Chemist

QA Officer: **Date:** 10/27/08
Dr. Christian Byrne, QA Officer

ANALYTICAL METHOD: 455916-01, E. Wickremesinhe, J. Brisbin, J. Ruhl, June 17, 1999, *Analytical Method for the Determination of Oxamyl and its Oxime Metabolite in Water Using LC/MS/MS Analysis"*. The unpublished method was developed by Centre Analytical Laboratory, Inc. of State College, PA and sponsored by E.I. du Pont de Nemours of Wilmington, Delaware. Pages 1-74.

EXECUTIVE SUMMARY

The method is applicable for the quantitative determination of residues of Oxamyl and its Oxime metabolite in water. Although the method was not created in accordance with EPA's Good Laboratory Practice Standards, Title 40 Code of Federal Regulations Part 160. After a thorough review, the ECB found that this Environmental Chemistry Method (ECM) and its associated independent laboratory validation (ILV) met all criteria for a scientifically valid method.

Method Summary

Ten milliliter water samples are fortified and adjusted to volume with a dilute formic acid solution. An aliquot is removed and analyzed via LC/MS/MS. Only water containing sediment is filtered using a PTFE filter.

The reported limit of quantification was found to be 1.0 ppb for all analytes.

METHOD ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS

This method was well documented and meets all the requirements for a scientifically valid method. Minor omissions included lack of characterization data for the water samples used and lack of a method blank in the validation data.

COMPLIANCE

Signed and dated statements that this method was conducted in accordance with the requirements for Good Laboratory Practice Standards, 40 CFR 160 were not present in the method. The laboratory did state that although the method development work presented in this paper was not done under GLP (they stated that it wasn't applicable), the analytical procedures, documentation, and archiving of the validation were done under GLP's as a part of DuPont Study No. AMR 4318-97. A statement of non-confidentiality on the basis of the method falling within the scope of FIFRA Section 10 (d)(1)(A), (B), or (C) was present.

A. BACKGROUND INFORMATION

Oxamyl is a carbamate insecticide/acaracide/nematicide that controls a broad spectrum of insects, mites, and/or nematodes on field crops, vegetables and fruits.

TABLE A.1. Test Compound Nomenclature for Oxamyl

Compound	Chemical Structure ***See Appendix A for the chemical structure information**
Common name	Oxamyl
Company experimental name	Not provided
IUPAC name	(EZ)-N,N-dimethyl-2-methylcarbamoyloxyimino-2-(methylthio)acetamide
CAS Name	methyl 2-(dimethylamino)-N-[[(methylamino)carbonyl]oxy]-2-oxoethanimidothioate
CAS #	23135-22-0

TABLE A.2. Physicochemical Properties of the Technical Grade Test Compound

Parameter	Value
Melting point/range	100 – 102 °C
pH	Not available
Density	Not available
Water solubility (25 °C)	280 g/L @25°C
Solvent solubility (mg/ml at 20 °C)	Very soluble in acetone, ethanol, 2-propanol, and methanol; soluble in toluene
Vapor pressure	31 mPa @ 25° C
Dissociation constant (pK_a)	Not available
Octanol/water partition coefficient	Not available
UV/visible absorption spectrum	Not available

MATERIALS AND METHODS

B.1. Principle of Method

Ten milliliter water samples are fortified with an appropriate spiking solution, acidified, and adjusted to 20 ml final volumes with a 0.1% aqueous formic acid solution. An aliquot of the sample is removed and analyzed via LC/MS/MS. Water samples containing sediment may be filtered using 0.2 µm Acrodisc®13 PTFE filters."

TABLE B.1.1.	**Summary Parameters for the Analytical Method Used for the Quantitation of Chemical Residues in Matrices Studied**
Method ID	ECM0243S1-S2
Analyte(s)	Oxamyl, Oxime
Extraction solvent/technique	No extraction is required
Cleanup strategies	Filtration with Acrodisc only if water contains sediment
Instrument/Detector	Shimadzu LC interfaced with PE Sciex API III Tandem Mass Spectrometer

C. RESULTS AND DISCUSSION

C.1.Recovery Results Summary

TABLE C.1.1. Recovery Results from Method Validation of Soil			
Matrix	Spiking Level (ppb)	Mean% Recoveries	Relative Standard Deviation
Oxamyl	1.0	96.0	6.2
	10.0	103	5.1
Oxime	1.0	97.0	2.4
	10.0	101	4.0

C.1.2. Method Characteristics

TABLE C.1.2. Method Characteristics	
Analyte	Oxamyl, Oxime
Limit of Quantitation	1.0 ppb
Limit of Detection (LOD)	0.1 ppb
Accuracy/Precision at LOQ	All recoveries and RSDs were acceptable.
Reliability of the Method/ [ILV]	An ILV was performed for this method. MRID No. 4455916-02
Linearity	Linear curves were prepared for each analyte. The correlation coefficient was > 0.998 for all compounds.
Specificity	The method is very specific due to the use of MS-MS which is the most highly specific method for detection of residues at low concentration.

C.2. Independent Laboratory Validation (ILV)

TABLE C.2.1. Recovery Results Obtained by an Independent Validation of the Method for the Determination of Oxamyl and Oxime in Water

Compound	Spiking Level (conc. units)	Oxamyl Mean Recovery (RSDs)	Oxime Mean Recovery (%RSDs)
Ground Water	1.0 ppb	97% (3)	115% (1)
Ground Water	10.0 ppb	108% (3)	109% (3)
Lums Pond Water	1.0 ppb	97% (4)	99% (5)
Lums Pond Water	10.0 ppb	90% (2)	101% (3)
Brandywine River Water	1.0 ppb	105% (5)	93% (4)
Brandywine River Water	10.0 ppb	96% (6)	93% (3)
Delaware River Water	1.0 ppb	92% (6)	112% (2)
Delaware River Water	10.0 ppb	99% (3)	111% (4)
Drinking Water	1.0 ppb	99% (5)	111% (3)
Drinking Water	10.0 ppb	97% (3)	102% (4)

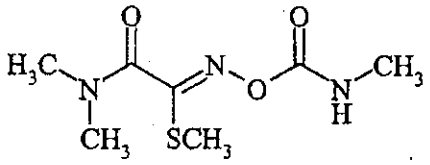
D. CONCLUSION

This is a well documented method which was confirmed by an independent laboratory validation. ECB finds the method acceptable.

Appendix A: Molecular Structure of Oxamyl and Oxime

Structures

Oxamyl (DPX-D1410)

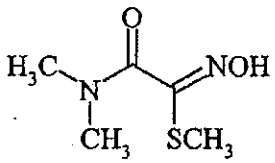


Monoisotopic Mass 219.07

CAS name: Methyl 2-(dimethylamino)-*N*-[[(methylamino)carbonyl]oxy]-2-oxoethanimidothioate

CAS Number 23135-22-0

Oxime metabolite of Oxamyl (IN-A2213)



Monoisotopic Mass 162.05

Methyl 2-(dimethylamino)-*N*-hydroxy-2-oxoethanimidothioate

CAS Number 66344-33-0