United States Department of the Interior

U.S. GEOLOGICAL SURVEY
Box 25046 M.S. 407
Denver Federal Center
Denver, Colorado 80225

IN REPLY REFER TO:

NATIONAL WATER QUALITY LABORATORY TECHNICAL MEMORANDUM 2002.03

22 May 2002

Subject: Permanent Application of the Estimated (E) Remark Code for Malathion and Disulfoton Data Reported by Lab Schedule 1319 (Organophosphate Pesticides in Water)

Distribution: E

Effective date of changes: July 9, 1998

Author: Duane Wydoski, GC/Waters Unit Supervisor
Organic Chemistry Program; NWQL; 303-236-3240
Email: dwydoski@usgs.gov

Revision: None

Supplemental: None

PURPOSE

This technical memorandum is intended to report the variable performance of two constituents, malathion and disulfoton, in the National Water Quality Laboratory lab schedule 1319 and the application of a permanent estimated (E) remark code for all reported concentrations of these two compounds. Some key method changes also are outlined in this document.

BACKGROUND

The organophosphorus pesticides in whole-water schedule 1319 include two constituents that have shown variable recoveries in laboratory reagent-water spike samples and in spike samples from the Branch of Quality System's Organic Blind Sample Program. Laboratory spike samples are used to monitor the method for bias and variability in the recovery of all compounds from reagent water. Malathion has shown lower than expected mean recoveries compared to performance data described in Wershaw and others (1987), where malathion had 50% mean recovery and 20% standard deviation (see table 1). Similarly, disulfoton has shown low mean recovery of about 40% and a standard deviation of about 20% in schedule 1319. The low recoveries are believed to be the result of volatilization losses during sample preparation and variable performance during gas chromatographic analysis. The

performance data from laboratory spike samples for all of the compounds reported in lab schedule 1319 for the past 4 years are listed in table 1.

The method was modified in 1991, when packed-column isothermal gas chromatography was replaced with capillary-column temperature-programmed chromatography. This modification improved resolution of compounds and reduced analysis time. Methyl trithion was dropped from the analysis because of difficulty obtaining an analytical standard, and three new compounds -- disulfoton, phorate, and fonofos -- were added to the analysis.

Benzene was removed as the solvent for calibration standards and replaced with hexane. This change was designed to limit exposure of laboratory personnel to benzene, a known carcinogen.

The surrogate standard for schedule 1319 is isofenphos, which is added to all field and quality-control samples. A surrogate standard is a compound that ideally has the same relative method performance as the compounds determined by the method, but is not likely to be found in environmental samples. Isofenphos shows better performance in environmental samples than in laboratory reagent-water spike samples.

Table 1 - - Percent recovery and relative standard deviation in lab spikes by year (reagent water)

Compound name	Parameter code	Year 1997	Year 1998	Year 1999	Year 2000	Year 2001
Chlorpyrifos	38932A	74±13%	77±12%	75±14%	66±10%	71±8%
DEF	39040A	52±20%	50±20%	55±21%	54±21%	45±16%
Diazinon	39570B	68±17%	73±17%	73±17%	69±16%	64±12%
Disulfoton	39011A	38±15%	40±21%	41±25%	41±21%	33±14%
Ethion	39398B	73±13%	76±13%	77±14%	70±9%	72±9%
Fonofos	82614C	75±13%	77±12%	77±12%	69±8%	71±10%
Isophenfos (Surrogate)	90712A	58±18%	62±21%	56±21%	54±21%	57±17%
Malathion	39530B	38±20%	36±20%	42±21%	32±13%	33±19%
Methylparathion	39600B	76±13%	77±13%	75±13%	67±10%	69±11%
Parathion	39540B	73±14%	77±13%	77±12%	69±11%	68±12%
Phorate	39023A	56±13%	44±22%	55±20%	52±15%	46±16%
Trition	39786B	63±18%	59±17%	69±15%	66±10%	59±12%

REPORTING CHANGES

Malathion and disulfoton will continue to be reported in lab schedule 1319. However, if either compound is not detected in environmental samples, the reporting level for the nondetected compound might be increased to "greater than" the current laboratory reporting level based on the performance of the compound in the lab spike samples or possibly be reported as "delete-ruined." All detections of malathion and disulfoton in field samples have been reported with an estimated (E) remark code since June 1998 to reflect the variable method performance of these two compounds. Disulfoton had been reported sporadically with an estimated (E) remark code prior to June 1998.

CHANGES TO DATA BASE

No changes to historic data base.

REFERENCE

Wershaw, R.L., Fishman, M.J., Grabbe, R.R., and Lowe, L.E., eds., 1987, Methods for the determination of organic substances in water and fluvial sediments: U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A3, p. 50-58.

// signed //

Gregory B. Mohrman, Chief
National Water Quality Laboratory
Branch of Analytical Services

This memorandum does not supersede any other NWQL Technical Memorandum.

Key Words: Schedule 1319, Insecticides, Malathion, Disulfoton, Surrogate

Distribution: All WRD employees plus the Netnews usgs.labnews, usgs.water.quality, and http://wwwnwql.cr.usgs.gov/