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NATIONAL WATER QUALITY LABORATORY TECHNICAL MEMORANDUM 1996.06A

April 15, 1996

To: Assistant Chief Hydrologist for Technical Support
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Employees, National Water Quality Laboratory

From: Peter F. Rogerson, Chief
National Water Quality Laboratory
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Subject: Revised Method Detection Limits for Pesticide Schedules 2001/2010 and 2050/2051

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Revision: This tech memo replaces NWQL Tech Memo 96.06 in its entirety

Purpose

The National Water Quality Laboratory (NWQL) has provided inconsistent "less than" concentration values for analytes that were not detected for Laboratory Pesticide Schedules 2001/2010 and 2050/2051. This technical memorandum announces new sets of method detection limits (MDLs) for these analytical methods.

Background

Schedules 2001/2010 and 2050/2051 were developed in response to specific needs of the National Water Quality Assessment (NAWQA) Program. These methods are designed to be broad based and

cover a wide variety of classes of pesticides (organochlorine, organophosphorous, chlorophenoxy acid herbicides, carbamate, and organonitrogen).

Schedules 2001/2010 analyze for pesticides in filtered water by utilizing Solid Phase Extraction (SPE) technology on a C-18 cartridge and Gas Chromatography/Mass Spectrometer (GC/MS). This method has been published in Open-File Report (OFR) 95-181, "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory -- Determination of Pesticides in Water by C-18 Solid-Phase Extraction and Capillary-column Gas Chromatography/Mass Spectrometry with Selected Ion Monitoring," Zaugg and Others.

Schedules 2050/2051 analyze for pesticides in filtered water by utilizing Solid Phase Extraction (SPE) technology on a Carboapak-B cartridge with High-Performance Liquid Chromatography-Diode Array Detection (HPLC-DAD). This method is currently a "special method" as it is not yet published in an approved OFR describing the method, precision, and accuracy. This documentation is required before the method can be considered for general use as an approved U.S. Geological Survey technique. Guidance on this method has been developed jointly by the NWQL and the NAWQA Program and distributed by Bob Gilliom (RGILLIOM) ((916) 979-2609 x348). Initially this method was implemented with a reporting limit (RL) of 0.05 micrograms per liter ($\mu\text{g/L}$). MDLs as defined by the U.S. Environmental Protection Agency (USEPA) in 40-CFR-136, Appendix B, were not available.

As these methods evolved, several different RLs have been used to indicate that a compound was not detected and was not present above these limits. Changes in the RLs did not represent shifts in the sensitivity of the methods used over time--but rather represented the way in which the NWQL reported "nondetects" for these schedules. These methods were implemented without an artificial lower RL censor in order to provide as much information as possible to our customers (NWQL Technical Memorandum 94-12). If criteria for qualitative identification were met, the result was quantitated and reported to the user. If the criteria were not met, the <RL was reported to indicate the compound was not detected. It is these <RL values that are being superseded by the following tables of MDLs. These changing RLs complicated data interpretation for WRD scientists.

Guidelines

The following tables of MDLs will be used by the NWQL to indicate the compound was not detected by the respective method. In some instances, a compound may be detected below its established MDL. These detections will be reported back to the user with the addition of an "E" or "0" (zero) remark code as a data qualifier to indicate that the reported concentration is an estimated value. The NWIS-I software replaced the "E" remark code with a "0" remark code on the WATLIST report. When an "E" remark is reported, the data user can be assured that the analyte has passed all qualitative criteria and only the concentration is estimated, not the presence.

Table 1.--Lab Schedules 2001 and 2010 Method Detection Limits (from Open-File Report 95-181, Table 9)

[MDL, method detection limit; $\mu\text{g/L}$, microgram per liter]

Parameter Code	Compound Name	MDL	Unit
49260	Acetochlor	0.002	$\mu\text{g/L}$
46342	Alachlor (Lasso)	0.002	$\mu\text{g/L}$

04040	Atrazine, desethyl-	0.002	µg/L
39632	Atrazine	0.001	µg/L
82686	Azinphos-methyl (Guthion)	0.001	µg/L
82673	Benfluralin (Benefin, Balan, Bonalin)	0.002	µg/L
04028	Butylate (Genate Plus, Suntan+)	0.002	µg/L
82680	Carbaryl (Sevin)	0.003	µg/L
82674	Carbofuran (Furandan)	0.003	µg/L
38933	Chlorpyrifos	0.004	µg/L
04041	Cyanazine	0.004	µg/L
82682	Dacthal (DCPA, Chlorthal-dimethyl)	0.002	µg/L
34653	DDE, p,p'-	0.006	µg/L
39572	Diazinon	0.002	µg/L
39381	Dieldrin	0.001	µg/L
82660	Diethylaniline, 2,6-	0.003	µg/L
82667	Disulfoton	0.017	µg/L
82668	EPTC (Eptam)	0.002	µg/L
82663	Ethalfuralin (Sonalin)	0.004	µg/L
82672	Ethoprop (Mocap, Ethoprophos)	0.003	µg/L
04095	Fonofos	0.003	µg/L
34253	HCH, alpha-	0.002	µg/L
39341	HCH, gamma- (Lindane)	0.004	µg/L
82666	Linuron (Lorox, Linex)	0.002	µg/L
39532	Malathion	0.005	µg/L
39415	Metolachlor (Dual)	0.002	µg/L
82630	Metribuzin (Lexone, Sencor)	0.004	µg/L
82671	Molinate (Ordram)	0.004	µg/L
82684	Napropamide (Devrinol)	0.003	µg/L
39542	Parathion, Ethyl-	0.004	µg/L
82667	Parathion, Methyl- (Pennacap-M)	0.006	µg/L
82669	Pebulate (Tillam)	0.004	µg/L
82683	Pendimethalin	0.004	µg/L
82687	Permethrin, cis-	0.005	µg/L
82664	Phorate (Thimet)	0.002	µg/L
82676	Pronamide (Kerb, Propyzamid)	0.003	µg/L
04037	Prometon	0.018	µg/L
04024	Propachlor (Ramrod)	0.007	µg/L
82679	Propanil (Stampede)	0.004	µg/L
82685	Propargite (Omite, alkyl sulfite)	0.013	µg/L
04035	Simazine (Aquazine, Princep)	0.005	µg/L
82681	Thiobencarb (Bolero)	0.002	µg/L
82670	Tebuthiuron (Spike)	0.010	µg/L
82665	Terbacil (Sinbar)	0.007	µg/L
82675	Terbufos (Counter)	0.013	µg/L
82678	Triallate (Avadex BW, Far-Go)	0.001	µg/L
82661	Trifluralin (Treflan)	0.002	µg/L

Table 2.--Lab Schedules 2050 and 2051 Method Detection Limits

[MDL, method detection limit; µg/l, microgram per liter]

Parameter			
Code	Compound Name	MDL	Unit
39732	2,4-D	0.035	µg/L
38746	2,4-DB	0.035	µg/L
39742	2,4,5-T	0.035	µg/L
49315	Acifluorfen	0.035	µg/L
49312	Aldicarb	0.016	µg/L
49314	Aldicarb Sulfoxide	0.021	µg/L
49313	Aldicarb Sulfone	0.016	µg/L

38711	Bentazon	0.014	µg/L
04029	Bromacil	0.035	µg/L
49311	Bromoxynil	0.035	µg/L
49310	Carbaryl	0.008	µg/L
49309	Carbofuran	0.028	µg/L
49305	Clopyralid	0.050	µg/L
49307	Chloramben	0.011	µg/L
49306	Chlorthalonil	0.035	µg/L
49304	Dacthal, mono-acid (DCPA)	0.017	µg/L
38442	Dicamba	0.035	µg/L
49303	Dichlobenil	0.020	µg/L
49302	Dichlorprop	0.032	µg/L
49301	Dinoseb	0.035	µg/L
49300	Diuron	0.020	µg/L
49299	DNOC	0.035	µg/L
49298	Esfenvalerate	0.019	µg/L
49297	Fenuron	0.013	µg/L
38811	Fluometuron	0.035	µg/L
49308	Hydroxycarbofuran, 3-	0.014	µg/L
38478	Linuron	0.018	µg/L
38482	MCPA	0.050	µg/L
38487	MCPB	0.035	µg/L
38501	Methiocarb	0.026	µg/L
49296	Methomyl	0.017	µg/L
49295	Naphthol, 1-	0.007	µg/L
49294	Neburon	0.015	µg/L
49293	Norflurazon	0.024	µg/L
49292	Oryzalin	0.019	µg/L
38866	Oxamyl	0.018	µg/L
49291	Picloram	0.050	µg/L
38538	Propoxur	0.035	µg/L
49236	Propham	0.035	µg/L
39762	Silvex	0.021	µg/L
49235	Triclopyr	0.050	µg/L

For each District, the NWQL will reload all samples with the updated "less than" values--unless requested not to do so by the District QW Specialist. All data with actual concentrations reported, including estimated values, will not be affected. An announcement will be made to affected Districts prior to the reload.

In the future, the NWQL will recalculate MDLs as part of its routine operation. Under those conditions, MDLs are expected to change because they are a statistical estimate of a property of the method. When this occurs, the NWQL will not update all data to the new values because each estimate is valid for an appropriate period of time. In this instance, the values shown in the above tables are considered the first estimate of a true MDL, so it is appropriate to update all other values.

Supersedes: NWQL Technical Memorandum 1996.06

Key Words: 2001, 2010, 2050, 2051, SPE, MDL, Pesticides

Distribution: See above plus the netnews USGS.labnews & .waterquality; WRD Secretaries; Field and Project Offices; Hydrologic Technicians; and <http://wwwnwql.cr.usgs.gov/>

Impact on

Data base: All current "less than" values of varying levels will be changed to reflect the above values for each analyte.