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# United States Department of the Interior

U.S. GEOLOGICAL SURVEY

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## **NATIONAL WATER QUALITY LABORATORY TECHNICAL MEMORANDUM 1998.02**

December 8, 1997

**Subject:** Reporting Changes for Selected Organochlorine Constituents from Schedule 1325 (Insecticides and Gross PCBs in Bottom Material) and Schedule 1335 (Organochlorine Insecticides, Gross PCBs, and Organophosphate Insecticides in Bottom Material)- Minimum Reporting Levels, Qualified Data, Surrogates, and Elimination of Two Constituents

Effective date

of changes: December 15, 1997

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**Revision:** None

**Supplemental:** None

### **SCOPE**

This technical memorandum announces changes in data reporting for all constituents in NWQL schedule 1325 (Insecticides and Gross PCBs in Bottom Material; Wershaw and others, 1987) effective December 15, 1997. The changes also are applicable to schedule 1335 (Organochlorine Insecticides, Gross PCBs, and Organophosphate Insecticides in Bottom Material) which is a combination of schedules 1325 and 1320. Minimum reporting levels are changed for 15 method constituents, all detections of methoxychlor are qualified as "estimated", additional surrogates are included, and two constituents (perthane, lab code 342, and gross polychlorinated naphthalenes, lab code 395) are deleted. Reporting changes are listed in table 1. No changes are being made to constituents in schedule 1320 (Organophosphate Insecticides in Bottom Material).

Table 1. Reporting level changes for constituents in schedules 1325 and 1335 at the NWQL

[WATSTORE, Water Data Storage and Retrieval System]

Constituent (in alphabetical order)	Parameter (WATSTORE) code*	NWQL lab code	Minimum reporting level (microgram/kilogram)		Fraction
			Current	Effective Dec. 15, 1997	
Aldrin	39333A	361	0.1	0.2	1
p,p'-DDD	39363A	363	0.1	0.5	2
p,p'-DDE	39368A	364	0.1	0.2	1
p,p'-DDT	39373A	365	0.1	0.5	2
Dieldrin	39383A	366	0.1	0.2	2
Endosulfan I	39389A	346	0.1	0.2	2
Endrin	39393A	367	0.1	0.2	2
Heptachlor	39413A	368	0.1	0.2	1
Heptachlor epoxide	39423A	369	0.1	0.2	2
Lindane (gamma-HCH)	39343A	370	0.1	0.2	2
Methoxychlor**	39481A	401	0.1	2.5	3
Mirex	39758A	545	0.1	0.2	1
Gross PCBs	39519A	394	1.0	5.0	1
Gross PCNs	39251A	395	1.0	deleted	
Perthane	81886A	342	1.0	deleted	
Technical chlordane	39351A	362	1.0	3.0	2
Toxaphene	39403A	371	10	50	2
Organochlorine Surrogates					
Isodrin	90568A	1487	--	--	1 & 2
Nonachlorobiphenyl (PCB #207)	--	2155	--	--	1
alpha-HCH-d6	--	2154	--	--	2

\*Letter following 5 digits represents method code.

\*\*Detections of methoxychlor will be reported as "estimated" in every case.

## BACKGROUND

Reporting levels for schedule 1325 initially were derived from reagent-water spike data using a different method. As a result, the reporting levels for organochlorine constituents in bottom material (sediments) determined by schedules 1325 and 1335 previously were set too low. Historically, schedule 1325 did not use data qualifiers. Analysts would raise reporting levels when the constituent could not be identified, or when interferences were present. Occasionally, the analyst would delete a constituent because of severe interference. The changes in reporting levels announced in this memorandum are based on a limited determination of the method detection limit for bottom material and on experience with many difficult matrices.

Isodrin has been the only organochlorine surrogate compound in schedules 1325 and 1335 despite generally low recovery relative to most method constituents and wide variance. Two additional organochlorine surrogates are added to better represent the range of constituents in this method.

The constituent list for schedules 1325 and 1335 includes two constituents that are rarely adequately measured or identified by this method; perthane and gross polychlorinated naphthalenes (PCNs). Perthane has not been detected in any environmental sample for many years. PCNs are not well suited for these gas chromatographic/electron-capture detection schedules because of their complexity and coelution with PCBs, which are much more abundant in most sediment samples. Additionally, no historical quality-control data exist for PCNs by these schedules.

## **REPORTING CHANGES**

The NWQL will qualify detections of methoxychlor in schedules 1325 and 1335 because of highly variable performance. All reported concentrations of methoxychlor will now be accompanied by an "E" remark code to indicate that the detected methoxychlor concentration is a quantitative estimate. The "E" remark code also will be used to flag "estimated" concentrations for other method constituents on an "as needed" basis when quality-control samples exceed acceptance limits (for example, matrix-induced loss of instrument sensitivity), or the quantitation is judged by the analyst to be an estimation.

The preparation of sediments for analysis by schedules 1325 and 1335 involves a fractionation of the extract to separate complex compounds and to simplify analytical interpretation. Currently three fractions are produced and independently analyzed per sample, with constituents observed in the fractions shown in table 1. Because of fraction specific interferences, reporting levels may be further raised to a value greater than the new higher minimum reporting levels listed in table 1 on a sample-by-sample and constituent-specific basis. Knowledge of constituent fractionation will help the data user interpret their data when reporting levels are further raised in these limited situations. For example, severe interferences might be observed in fraction 2, requiring raised reporting levels for a number of or all fraction 2 constituents. However, fraction 1 from the same sample might be free of interferences, thus, requiring no raised reporting level adjustment for constituents in fraction 1.

For consistency, isodrin will continue to be reported as an organochlorine surrogate for these two schedules. Isodrin is observed in both fraction 1 (primarily) and fraction 2, and its recovery is calculated as the sum of the amount quantitated in both fractions. In 1997, additional organochlorine surrogates were tested and two were proven to provide reliable results for permanent inclusion in schedules 1325 and 1335. These surrogates are nonachlorobiphenyl (PCB-207), which is quantitated in and representative of constituents found in fraction 1, and alpha-HCH-d6, which is quantitated in and representative of constituents in fraction 2. Unlike isodrin, which splits fractions, the new surrogates normally are found in a single fraction as listed in table 1, and thus are reflective of a fraction-specific surrogate recovery. No organochlorine surrogate is representative of fraction 3 (which contains only methoxychlor of the organochlorine constituents). The NWQL has requested parameter codes for these new surrogates. All recovery data for the two surrogates will be stored in the NWQL's data base beginning December 15, 1997, and these data will be transmitted electronically following assignment of the new codes. Until then, recovery information for these two new surrogates will be provided to customers in mailed data reports.

Two constituents, perthane and gross PCNs, are deleted from schedules 1325 and 1335 as of December 15, 1997, for reasons outlined above. These constituents may be requested on a custom basis. Contact Brooke Connor for custom analyses related to schedules 1325 or 1335.

## **EFFECT ON DATA BASE**

Minimum reporting level changes for the constituents in table 1 will cause a shift in nondetection (less than) concentrations in the data base. Minimum reporting levels for historical data will not be changed in the data base. During FY1998, the Office of Water Quality, along with other Water Resources Division representatives, will be developing guidance on interpreting analytical results, both new and historical, for the effected determinations. Data users should be cautious when interpreting historical data for these constituents in light of new higher reporting levels.

## **REFERENCES**

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, Laboratory Analysis, chap. A3, p. 59-67.

/signed/

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This memorandum does not supersede any other NWQL Technical Memorandum.

Key Words: Schedule 1325, Schedule 1335, Reporting levels, Insecticides, Estimated concentration, Polychlorinated naphthalenes, PCNs, Polychlorinated biphenyls, PCBs, Perthane, Methoxychlor, Surrogates

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