

NO. 94293-5
Court of Appeals, Division II Case No. 48267-3-II

SUPREME COURT OF THE STATE OF WASHINGTON

PUGET SOUNDKEEPER ALLIANCE,

Petitioner,

v.

STATE OF WASHINGTON, DEPARTMENT OF ECOLOGY; and
STATE OF WASHINGTON POLLUTION CONTROL HEARINGS
BOARD,

Respondents.

**PUGET SOUNDKEEPER ALLIANCE'S ANSWER TO AMICUS
CURIAE BRIEFS OF SPOKANE COUNTY AND NORTHWEST
PULP & PAPER ASSOCIATION, ASSOCIATION OF
WASHINGTON BUSINESS, AND ASSOCIATION OF
WASHINGTON CITIES**

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I. INTRODUCTION

Arguments offered by *amicus curiae* briefs submitted by Spokane County and by Northwest Pulp and Paper Association, Association of Washington Business, and Association of Washington Cities (collectively, “Associations”) provide no basis for the Court to deny the relief requested by petitioner Puget Soundkeeper Alliance. This brief presents Soundkeeper’s responses to new arguments raised in these *amicus* briefs. As an initial matter, Soundkeeper notes that no party has offered any argument contradicting Soundkeeper’s basic assertion that RCW 90.48.520 prohibits the issuance of the Seattle Iron and Metals National Pollutant Discharge Elimination System (“NPDES”) permit (“SIM permit”) with an effective total PCB effluent limitation set at the practical quantitation level (“PQL”) of Method 608, which is orders magnitude higher than the PCB discharge concentration found necessary to prevent violation of applicable water quality standards.¹

¹ No matter that opposition briefs filed with this Court sometimes misidentify the PQL of Method 608 as 0.1 or 0.25 µg/L or something else, the PQL of Method 608 is 0.5 µg/L. RP 68:7 – 25; AR 3305 (SIM Permit at p.52) (table giving PQL of Method 608); *Puget Soundkeeper Alliance v. State of Washington, Department of Ecology, et al.*, No. 48267-3-II, Unpublished Opinion (Feb. 22, 2017) (“Decision”) at 10. Soundkeeper suspects that a cause of confusion is discussion of a third lab analysis method, Method 8082, which was also at one time required by the SIM permit and so the subject of evidence and testimony, and which has a PQL between those of Method 608 and Method 1668C. E.g., *Puget Soundkeeper Alliance v. Ecology and SIM*, PCHB No. 13-137c, Findings of Fact, Concl. of Law, and Order (July 23, 2015) (“Board Order”) at 26 (discussing Method 8082 and correctly reciting the *minimum detection level* (not PQL) of Method 608 as 0.25 µg/L).

II. ARGUMENTS RESPONDING TO *AMICUS CURIAE* BRIEFS

- A. Ecology has taken a legal position, not a technical one, about use of Method 1668C for compliance monitoring.

Contrary to Spokane County’s assertion, the Department of Ecology (“Ecology”) has made no determination whatsoever about the merits or utility of Method 1668C for compliance monitoring in SIM’s permit or otherwise. The basis for Ecology’s rejection has exclusively been its *legal* position that WAC 173-201A-260(3)(h) strictly requires the use of a method listed in 40 C.F.R. § 136.3.² As to practicality or efficacy, Ecology did not even consider imposing Method 1668C for compliance monitoring because it believed that its use was legally precluded.³

There is thus no basis for Spokane County’s argument that the Court should defer to “Ecology’s water quality technical expertise”⁴ – none was exercised.⁵ As Soundkeeper has argued, Ecology’s legal interpretation of WAC 173-201A-260(3)(h) should be rejected as conflicting with the letter and underlying intent of state statute, RCW 90.48.010 and .520, in this Court’s *de novo* review.⁶ As the court of

² Board Order at 27 and 34; Decision at 10 and 12 – 15.

³ Board Order at 26 (“Ecology did not consider the use of Model (sic) 1668 in the 2013 Permit.”); RP 53:22 – 55:10 (argument by Ecology’s counsel); RP 63:18 – 64:14); RP 664:13 – 19; RP 649:5 – 649:11 (against Ecology policy); RP 702:17 – 22.

⁴ *Amicus Curiae* Brief of Spokane County (“County Brief”) at 12.

⁵ RP 664:13 – 19.

⁶ Puget Soundkeeper Alliance’s Supplemental Brief at 12 - 20; *Puget Soundkeeper Alliance v. Pollution Control Hearings Board*, 189 Wn.App. 127, 135 – 136, 356 P.3d 753 (2015).

appeals has explained, “[a]llowing violations of water quality standards, especially for the convenience of permittees and regulators, does not provide a rational basis for disregarding the plain language of [Ecology’s] rules and is an abdication of its responsibility to implement those rules.”⁷

B. Spokane County’s concerns about use of Method 1668C under its permit are not properly at issue here.

The Court’s Administrative Procedure Act review is limited to the record before the Pollution Control Hearings Board.⁸ The Court’s ability to receive evidence in addition to that contained in the agency record is tightly restricted by statute.⁹ Since none of the circumstances in which supplementation of the administrative record with additional facts is present, the Court should not consider Spokane County’s extra-record factual assertions in deciding this case, including the declaration testimony of Spokane County’s engineer and its quotations of non-record, non-produced comments submitted to the Environmental Protection Agency (“EPA”) by Environmental Standards, Inc.¹⁰

⁷ *Puget Soundkeeper Alliance*, 189 Wn.App. at 148.

⁸ RCW 34.05.566(1); *Samson v. City of Bainbridge Island*, 149 Wn.App. 33, 43 (2009) (citing *Wash. Indep. Tel. Ass’n v. Wash. Utils. & Transp. Comm’n*, 110 Wn. App. 498, 518, 41 P.3d 1212 (2002), *aff’d*, 149 Wn.2d 17 (2003)).

⁹ RCW 34.05.562; *Port of Seattle v. Pollution Control Hearings Board*, 151 Wn.2d 568, 603, n. 11, 90 P.3d 659 (2004).

¹⁰ *US W. Communs. v. Utils. and Transp. Comm’n*, 134 Wn2d 48, 72 – 73, 949 P.2d 1321 (1997); County Brief at 8. Additionally, the purported comments of Environmental Standards, Inc. constitute hearsay and lack any indicia of authenticity or reliability, never mind any indication that they were prepared or reviewed by a qualified expert.

Spokane County is concerned about the possible requirement to use Method 1668C for PCB effluent limit compliance monitoring in the pending reissuance of the NPDES permit for its sewage treatment plant.¹¹ If Spokane County has an objection to such a requirement, or to any other condition of its pending permit upon reissuance, it will have the opportunity to appeal the permit to the Pollution Control Hearings Board and make its case that the permit is invalid.¹² That is the proper forum for its factual and technical arguments about Method 1668C as they pertain to its permit.

The record for this case contains testimony and evidence about the efficacy and practicality of using Method 1668C for compliance monitoring, to which the Court must limit its consideration in determining the particular legal issues presented here. Soundkeeper's well-qualified expert, Ann Bailey, testified at length about the various methods available to conduct lab analysis of total PCB concentrations in water samples and their comparative merits.¹³ Ms. Bailey explained to the Board why, due to

¹¹ County Brief at 1 – 2.

¹² RCW 43.21B.110; WAC 173-220-225; *Spokane County v. Sierra Club*, No. 47158-2-II, 2016 Wash.App.LEXIS 1941, *16 (Feb. 23, 2016).

¹³ RP 51:5 – 86:7; Ecology's Jerry Shervey also provided limited testimony about what he has heard about Method 1668C, albeit after clarifying that he not a "chemist" (i.e., not an expert in analysis techniques). RP 648:9 – 14.

its improved and modern technique, Method 1668C is superior to Method 608 in terms of precision and accuracy.¹⁴

As Ms. Bailey also explained, the EPA's 2012 deferral of its proposal to add Method 1668C to the 40 C.F.R. § 136.3 list of approved NPDES compliance monitoring methods does not support assertions now offered by *Amici* that the method is defective or deficient.¹⁵ The EPA deferral notice explains that EPA's development of Method 1668 to address the manifest inadequacy of Method 608 has included several revisions to refine the method since starting work on it in 1995.¹⁶ EPA acknowledges that Method 1668C "is being used in some states in their regulatory programs and by other groups for some projects with good success," which "shows that recoveries and precision for this method are within the performance achievable with other approved methods."¹⁷ EPA deferred approval to allow it to continue to evaluate comments, and concluded that its deferral decision "does not negate the merits of [Method 1668C] for the determination of PCB congeners in regulatory programs or

¹⁴ RP 70:8 – 71:20, 75:21 – 76:23 (citing 64 Fed.Reg. 61186)

¹⁵ RP 76:24 – 77:21 ("Those reasons [given by EPA for deferral] do not discount the validity of the method and [EPA] actually state[s] that in there."); RP 82:6 – 83:9.

¹⁶ AR 3586 (77 Fed.Reg. at 29763); Method 608 was developed in the 1970's. RP 66:17 – 67:10.

¹⁷ AR 3586 (77 Fed.Reg. at 29763).

for other purposes when analyses are performed by an experienced laboratory.”¹⁸ EPA has taken no further action on the deferral.

Ms. Bailey also addressed the types of concerns Spokane County seeks to raise about Method 1668C quantitation, reporting, and blank contamination issues.¹⁹ The answer to these is in the development of rules, to be incorporated in the terms of an NPDES permit, governing how to correct for blank contamination, how to sum PCB congeners quantified by the lab, and how to report laboratory results.²⁰ That Spokane County identified no consensus among different quantification methods used in various studies of Method 1668C quantification methods does not mean that Ecology cannot chose or construct one that appropriately serves the objectives of RCW 90.48.010.²¹ Of course, such rules would – along with the rest of an NPDES permit issued to the County or any other discharger – be appealable, within thirty days of issuance, by the discharger to the Pollution Control Hearings Board if asserted to be inappropriate.²²

¹⁸ Id.

¹⁹ County Brief at 3 – 5.

²⁰ RP 79:6 – 80:3; *see also, e.g.*, “Clean Water Act Methods Update Rule for the Analysis of Effluent,” EPA, Aug. 28, 2017, 82 Fed.Reg. 40836, 40889, 40907, and 40924 – 40925 (Appendix 1) (examples of specified data reporting practices for various laboratory methods that address the types of quantification concerns raised by Spokane County).

²¹ County Brief at 5.

²² WAC 371-08-335.

C. Soundkeeper's request for relief is appropriate.

Contrary to the Associations' arguments, nowhere in its appellate briefing does Soundkeeper ask the Court, implicitly or explicitly, to require Ecology to petition EPA for approval of Method 1668C under 40 C.F.R. § 136. The relief actually sought – an order that Ecology must deny issuance of the SIM NPDES permit as violating RCW 90.48.520 and its progeny WAC sections unless the SIM permit includes conditions adequate to ensure compliance with toxicity water quality standards²³ (specifically, the approval and use of Method 1668C to determine compliance with total PCB numeric effluent limitations) – is squarely within the scope of relief available under law.²⁴ Soundkeeper makes no argument whatsoever that Ecology must be required to petition EPA; it instead argues that Ecology cannot issue the SIM NPDES permit unless limited use approval for Method 1668C is obtained.²⁵

This situation is very similar to that considered by the Ninth Circuit in *Friends of Pinto Creek v. United States EPA*, 504 F.3d 1007 (9th Cir. 2007). There, environmentalists appealed an EPA-issued NPDES permit on the grounds that 40 C.F.R. § 122.4(i)(2) prohibited its issuance absent the adoption of plans or compliance schedules to control non-point

²³ WAC 173-201A-240(1) and (2), -510(1); WAC 173-220-130(1)(b)(i), -150(1)(c).

²⁴ RCW 30.05.574.

²⁵ RCW 90.48.520; WAC 173-201A-510(1); WAC 173-220-130(1)(b)(i).

sources of pollution that caused the receiving waters to violate water quality standards for the pollutant at issue.²⁶ The Ninth Circuit agreed with the plaintiff, and rejected EPA’s argument that the plaintiff was seeking to force EPA to act against non-point source dischargers.²⁷ It held that relief prohibiting the issuance of the permit until EPA complied with the § 122.4(i)(2) planning requirement was just that – a prohibition on the issuance of a single NPDES permit.²⁸ As here, the plaintiff there sought to compel the permitting authority to do nothing, and only to prevent an illegal permit issuance. There is no compulsion in the order sought; Ecology “remains free to establish its priorities,” and to draft and offer, or support or oppose a petition to EPA for Method 1668C limited use approval.²⁹ Soundkeeper simply seeks an order prohibiting SIM permit issuance unless the limited use approval is obtained.

Unlike EPA’s nationwide approval process for a test method’s inclusion in the 40 C.F.R. § 136.3 list³⁰, the § 136.5 process for limited use approval entails no rulemaking. Under § 136.5, “[a]ny person” may request approval for NPDES compliance monitoring use of a method not

²⁶ 504 F.3d at 1009 and 1011-15.

²⁷ *Id.* at 1014 – 15.

²⁸ *Id.*

²⁹ *Id.*

³⁰ 40 C.F.R. § 136.4(c)(2) (rule-making procedures needed to conclude approval of alternative test procedure for nationwide use)

included in the § 136.3 list. Given EPA's extensive work in developing and documenting Method 1668C, the specific information required by § 136.5(c) for such a request is readily available for this method.³¹ The process involves the submission of the requestor's application to the state NPDES permitting authority (here, Ecology), after which the state permitting authority forwards the application to EPA along with its recommendation for or against approval.³² EPA then issues its decision on limited use approval for the proposed method, specifying the scope of the approval, without rulemaking.³³ Ecology staff testified at the hearing about employing the § 136.5 limited use approval process in cooperation with another permittee without noting any difficulties with it.³⁴ The Associations are simply wrong to assert that limited use approval requires rulemaking.³⁵

Soundkeeper's unanswered argument here is that issuance of the SIM NPDES permit with provision for monitoring compliance with total PCB effluent limitations by Method 608 is barred by RCW 90.48.520 and its implementing regulations. No one disputes that the law allows – indeed, requires – Ecology to deny permit issuance if the permit cannot be

³¹ RP 78:3 – 7.

³² 40 C.F.R. § 136.5(b).

³³ 40 C.F.R. § 136.5(d).

³⁴ RP 711:15 – 712:1, 714:5 – 9 (EPA issued approval in 45 days).

³⁵ Associations' Brief at 6 – 8.

conditioned to ensure against violations of water quality standards.³⁶

Ecology is required to impose monitoring requirements adequate to determine compliance with water quality criteria for toxics.³⁷

If SIM wants its discharge permit to be issued, it should bear the burden and be required to obtain EPA's limited use approval for Method 1668C. There is no reason that its NPDES permit cannot require it to do so, and to prohibit its discharge if it does not. Alternatively, Ecology could require the limited use approval for Method 1668C to be obtained for the SIM permit before issuing the permit.³⁸ There is nothing in Soundkeeper's requested relief that would violate any provision of federal law.

D. Soundkeeper's position is entirely consistent with the CWA.

The objective of the federal CWA is "to restore and maintain the chemical, physical, and biological integrity of the Nation's waters."³⁹ It asserts national goals "that the discharge of pollutants into the navigable waters be eliminated by 1985," and that water quality protective of fish, shellfish, and wildlife be achieved by 1983.⁴⁰ Further, it establishes a

³⁶ RCW 90.48.520; WAC 173-201A-240(1), -510(1); WAC 173-220-130(1)(b)(i).

³⁷ WAC 173-201A-240(2).

³⁸ It is standard for a permit applicant to undertake performance of technical and other work, subject to agency review and approval, needed for permit issuance, rather than for Ecology to do this work itself. *E.g.*, Board Decision at 18 (mixing zone study prepared by SIM's consultant and adopted by Ecology).

³⁹ 33 U.S.C. § 1251(a); *S.D. Warren Co. v. Me. Bd. of Ent'l Prot.*, 547 U.S. 370, 385, 126 S. Ct. 1843, 164 L. Ed. 2d 625 (2006).

⁴⁰ 33 U.S.C. § 1251(a)(1) and (2).

national policy “that the discharge of toxic pollutants in toxic amounts be prohibited.”⁴¹ Section 301 prohibits point source pollutant discharges not in compliance with the terms of an NPDES permit, and demands that NPDES permits include effluent limitations stringent enough to meet water quality standards.⁴² This demand is firm – NPDES permits must ensure compliance with water quality standards regardless of “economic and technological restraints.”⁴³

To accomplish this, federal regulations require the imposition in an NPDES permit of water quality-based effluent limitation whenever the discharge of a pollutant poses a “reasonable potential” to contribute to violation of water quality standards.⁴⁴ “More stringent” effluent limitations established by state law must also be imposed.⁴⁵ Water quality-based effluent limitations must be numeric limits on pollution discharge mass or concentration, unless the calculation of numerical limits is “infeasible.”^{46,47}

⁴¹ 33 U.S.C. § 1251(a)(3).

⁴² 33 U.S.C. § 1311(a) and (b)(1)(C).

⁴³ *Ackels v. United States EPA*, 7 F.3d 862, 865 – 866 (9th Cir. 1993); *see also*, *Defenders of Wildlife v. Browner*, 191 F.3d 1159, 1164 – 1165 (9th Cir. 1999) (“industrial dischargers must comply strictly with state water quality standards”).

⁴⁴ 40 C.F.R. § 122.44(d)(1)(i) – (iii).

⁴⁵ 40 C.F.R. § 122.44(d)(5).

⁴⁶ 40 C.F.R. § 122.44(k)(3); *NRDC v. EPA*, 804 F.3d 149, 170 fn. 16 (2nd Cir. 2015); *Communities for a Better Environment v. State Water Res. Control Bd.*, 109 Cal.App.4th 1089, 1104 fn. 9 (2003); *see also*, WAC 173-220-130(3)(a) (requiring numeric limits).

⁴⁷ Conditions included in the SIM permit by Ecology to ensure compliance with water quality standards comprise the numeric limits on PCBs and those on total suspended

On top of all this, the CWA expressly authorizes states to impose yet more stringent standards or limitations on discharges of pollutants.⁴⁸ Indeed, for its cooperative federalism, states' rights in controlling pollutant sources hold a prominent place in the CWA.⁴⁹

Nothing about Soundkeeper's argument or request for relief contradicts the letter or intent of the CWA whatsoever, despite the Associations' arguments to the contrary.

The Associations' citations to requirements for use of particularly described, "best available" science are entirely misplaced.⁵⁰ These requirements are particular to a state's development of water quality criteria, and do not concern NPDES permitting decisions or requirements.⁵¹ Indeed, the very existence of the 40 C.F.R. § 136.5 procedure for limited use approval of alternative test methods renders

solids. RP 605:24 – 608:1, 677:10 – 678:1. Initially, Ecology required the use of a third PCB analysis method, Method 8082, to somewhat address the gross difference between the Method 608 PQL and the required PCB effluent limitation. RP 646:8 – 647:13, 658:12 – 660:21.

⁴⁸ 33 U.S.C. § 1370. *Milwaukee v. Illinois*, 451 U.S. 304, 327 – 328 (1981).

⁴⁹ 33 U.S.C. § 1251(a)(5) and (b) ("It is the policy of the Congress to recognize, preserve, and protect the primary responsibilities and rights of States to prevent, reduce, and eliminate pollution . . ."); *S.D. Warren Co.*, 547 U.S. at 386.

⁵⁰ Associations' Brief at 9.

⁵¹ 33 U.S.C. § 1314(a)(1); 40 C.F.R. § 131.1; *Mississippi Comm'n on Nat. Res. v. Costle*, 625 F.2d 1269, 1275 – 78 (5th Cir. 1980) (discussing operation of § 1314(a)(1) and distinguishing EPA requirements for state establishment of water quality criteria from EPA authority over state-issued NPDES permits).

nonsensical the Associations' argument that Soundkeeper would have Ecology "simply ignore" EPA regulations.⁵²

E. There is no "slippery slope."

The Court may safely disregard the Associations' assertions that granting the relief requested by Soundkeeper could "bring Ecology's NPDES permit to a virtual standstill," or cause the mass invalidation of existing NPDES permit discharge authorizations.⁵³ These are without factual or legal bases.

First, none of the general NPDES permits identified by the Associations includes any numeric effluent limitation for PCBs, so it is utterly irrelevant that "[n]one of these permits requires compliance with water quality standards using Method 1668C."⁵⁴ Indeed, the Associations, with all of their concerned members subject to the terms of NPDES permits, identify not a single member of theirs, or any other NPDES permittee that is currently subject to numeric effluent limitations for total PCBs. Any discharger in whose NPDES permit Ecology may in the future impose PCB numeric effluent limitations and Method 1668C monitoring requirements will have the opportunity to challenge the validity of the

⁵² Associations' Brief at 10.

⁵³ *Id.* at 11 – 13.

⁵⁴ *Id.* at 11. General NPDES permits issued by Ecology are available for review at <http://www.ecy.wa.gov/programs/wq/permits/genpermits.html>.

conditions of their permit to the Pollution Control Hearings Board and make their case, whatever it may be, about these issues.⁵⁵ This case, however, is about the SIM permit, SIM's discharge of process wastewater and commingled industrial stormwater, and the administrative record before the Court.

Second, the opportunity of a permittee or any concerned person, such as Soundkeeper, to challenge the conditions of an NPDES permit is limited to the thirty-day period following permit issuance.⁵⁶ Appeal to the Pollution Control Hearings Board within this period is the only way that conditions of an NPDES permit may be challenged.⁵⁷ Once the thirty-day period has passed, there is no ability for anyone to collaterally attack permit terms.⁵⁸ Thus, there is no forum in which Soundkeeper could possibly argue, as the Associations fear, that “any permits currently requiring Method 608 are invalid.”⁵⁹

⁵⁵ RCW 43.21B.110.

⁵⁶ WAC 371-08-335.

⁵⁷ *Dioxin/Organochlorine Center v. Dep't of Ecology*, 119 Wn.2d 761, 774, 837 P.2d 1007 (1992) (resort to Pollution Control Hearings Board is exclusive means to appeal NPDES permit issued by Ecology).

⁵⁸ 33 U.S.C. § 1369(b)(2); *GMC v. EPA*, 168 F.3d 1377, 1382 (D.C. Cir. 1999); *Public Interest Research Group v. Powell-Duffryn Terminals*, 913 F.2d 64, 77 – 78 (3rd Cir. 1990); *Ohio Valley Env'tl Coalition, Inc. v. Fola Coal Co.*, No. 2:12-3750, 2013 U.S. Dist. LEXIS 178319, *35 – 36 (W.V. S.D. Dec. 19, 2013) (“well-settled”); *United States v. Gulf States Steel, Inc.*, 54 F.Supp.2d 1233, 1241 – 45 (N.D. Ala. 1999) (discussing cases).

⁵⁹ Associations' Brief at 11.

Third, there is no reason to think that Ecology's NPDES permitting program would be brought to a standstill if the Court grants the relief Soundkeeper seeks.⁶⁰ As noted, there is no evidence (and it is not true) that there are numerous NPDES permits, individual or general, that impose numeric effluent limitations for total PCBs. So the vast majority of permitting decisions that Ecology makes will be unaffected by the outcome of this case. The evidence before the Court is that the information needed for a 40 C.F.R. § 136.5 application for limited use approval is readily available and that there is no apparent reason that EPA would find an application using this information to be inadequate.⁶¹

Finally, if there were any merit to the Associations' "slippery slope/standstill" argument, it would have been asserted in some form by Ecology as well. Ecology has offered no such arguments about possible cascade effects from a ruling in Soundkeeper's favor.

RESPECTFULLY SUBMITTED this 29th day of September, 2017

SMITH & LOWNEY, P.L.L.C.

By: 
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Puget Soundkeeper Alliance

⁶⁰ Id at 12.

⁶¹ RP 78:3 – 7, 79:3 – 5.

APPENDIX A

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 136

[EPA-HQ-OW-2014-0797; FRL-9957-24-OW]

RIN 2040-AF48

Clean Water Act Methods Update Rule for the Analysis of Effluent

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule.

SUMMARY: This rule modifies the testing procedures approved for analysis and sampling under the Clean Water Act. The changes adopted in this final rule fall into the following categories: New and revised EPA methods (including new and/or revised methods published by voluntary consensus standard bodies (VCSB), such as ASTM International and the Standard Methods Committee); updated versions of currently approved methods; methods reviewed under the alternate test procedures (ATP) program; clarifications to the procedures for EPA approval of nationwide and limited use ATPs; and amendments to the procedure for determination of the method detection limit to address laboratory contamination and to better account for intra-laboratory variability. **DATES:** This regulation is effective on September 27, 2017. The incorporation by reference of certain publications listed in the rule is approved by the Director of the Federal Register as of

September 27, 2017. For judicial review purposes, this final rule is promulgated as of 1:00 p.m. (Eastern time) on September 12, 2017 as provided at 40 CFR 23.2 and 23.7.

ADDRESSES: EPA has established a docket for this action under Docket ID No. EPA-HQ-OW-2014-0797. All documents in the docket are listed on the www.regulations.gov Web site. Although listed in the index, some information is not publicly available, e.g., confidential business information (CBI) or other information whose disclosure is restricted by statute. Certain other materials, such as copyrighted material are not placed on the Internet and will be publicly available only in hard copy form. Publicly available docket materials are available either electronically through www.regulations.gov or in hard copy at the Water Docket in EPA Docket Center, EPA/DC, EPA West William J. Clinton Building, Room 3334, 1301 Constitution Ave. NW., Washington, DC. The Public Reading Room is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number for the Public Reading Room is 202-566-1744 and the telephone number for the Water Docket is 202-566-2426.

FOR FURTHER INFORMATION CONTACT: Adrian Hanley, Engineering and Analysis Division (4303T), Office of Water, Environmental Protection Agency, 1200 Pennsylvania Ave. NW., Washington, DC 20460-0001; telephone:

202-564-1564; email: hanley.adrian@epa.gov.

SUPPLEMENTARY INFORMATION:

A. General Information

1. Does this Action apply to me?

EPA proposed the changes in this method update rule for public comment on February 19, 2015 (80 FR 8956).

EPA Regions, as well as States, Territories and Tribes authorized to implement the National Pollutant Discharge Elimination System (NPDES) program, issue permits with conditions designed to ensure compliance with the technology-based and water quality-based requirements of the Clean Water Act (CWA). These permits may include restrictions on the quantity of pollutants that may be discharged as well as pollutant measurement and reporting requirements. If EPA has approved a test procedure for analysis of a specific pollutant, the NPDES permittee must use an approved test procedure (or an approved alternate test procedure if specified by the permitting authority) for the specific pollutant when measuring the required waste constituent. Similarly, if EPA has established sampling requirements, measurements taken under an NPDES permit must comply with these requirements. Therefore, entities with NPDES permits will potentially be affected by the actions in this rulemaking.

Entities potentially affected by the requirements of this rule include:

Category	Examples of potentially affected entities
State, Territorial, and Indian Tribal Governments	States, territories, and tribes authorized to administer the National Pollutant Discharge Elimination System (NPDES) permitting program; states, territories, and tribes providing certification under CWA section 401; state, territorial, and tribal owned facilities that must conduct monitoring to comply with NPDES permits.
Industry	Facilities that must conduct monitoring to comply with NPDES permits.
Municipalities	Publicly Owned Treatment Works (POTWs) or other municipality owned facilities that must conduct monitoring to comply with NPDES permits.

This table is not exhaustive, but rather provides a guide for readers regarding entities likely to be affected by this action. This table lists types of entities that EPA is now aware of that could potentially be affected by this action. Other types of entities not listed in the table could also be affected. To determine whether your facility is affected by this action, you should carefully examine the applicability language at 40 CFR 122.1 (NPDES purpose and scope), 40 CFR 136.1 (NPDES permits and CWA) and 40 CFR 403.1 (pretreatment standards purpose and applicability). If you have questions regarding the applicability of this action

to a particular entity, consult the appropriate person listed in the preceding **FOR FURTHER INFORMATION CONTACT** section.

B. What process governs judicial review of this rule?

Under Section 509(b)(1) of the Clean Water Act (CWA), judicial review of this CWA rule may be obtained by filing a petition for review in a United States Circuit Court of Appeals within 120 days from the date of promulgation of this rule. For judicial review purposes, this final rule is promulgated as of 1 p.m. (Eastern time) on September 12, 2017 as provided at 40 CFR 23.2.

Section 509(b)(2) provides that any rule (or requirements of any rule) for which review could have been obtained under Section 509(b)(1) may also not be challenged later in civil or criminal proceedings for enforcement.

C. Abbreviations and Acronyms Used in the Preamble and Final Rule Text

- 4AAP: 4-Aminoantipyrine
- AA: Atomic Absorption
- ADMI: American Dye Manufacturers Institute
- AOAC: AOAC International
- ASTM: ASTM International
- ATP: Alternate Test Procedure
- BOD₅: 5-day Biochemical Oxygen Demand test
- CAS: Chemical Abstract Services

CATC: Cyanide Amenable to Chlorination
 CFR: Code of Federal Regulations
 CIE/UV: Capillary Ion Electrophoresis/
 Ultraviolet
 COD: Chemical Oxygen Demand
 CWA: Clean Water Act
 DPD: N,N-diethyl-*p*-phenylenediamine
 DPD-FAS: N,N-diethyl-*p*-phenylenediamine
 with ferrous ammonium sulfate
 EDTA: Ethylenediamine tetraacetic acid
 EPA: Environmental Protection Agency
 FLAA: Flame Atomic Absorption
 Spectroscopy
 GC: Gas Chromatograph/Chromatography
 GC/HSD: Gas chromatography/halogen-
 specific detector
 GC/MS: Gas chromatography/mass
 spectrometry
 HEM: Hexane extractable material
 HPLC: High performance liquid
 chromatography
 HRGC: High Resolution Gas Chromatography
 HRMS: High Resolution Mass Spectrometry
 HSD: Halogen-specific detector
 ICP: Inductively coupled plasma
 ICP/AES: Inductively Coupled Plasma-
 Atomic Emission Spectroscopy
 ICP/MS: Inductively Coupled Plasma-Mass
 Spectrometry
 LCS: Laboratory Control Sample
 MDL: Method Detection Limit
 MS: Mass Spectrometry
 MPN: Most Probable Number
 MS/MSD: Matrix Spike/Matrix Spike
 Duplicate
 NARA: National Archives and Records
 Administration
 NPDES: National Pollutant Discharge
 Elimination System
 NIST: National Institute of Standards and
 Technology
 PAH: Polynuclear aromatic hydrocarbons
 POTW: Publicly Owned Treatment Works
 QA: Quality Assurance
 QC: Quality Control
 RRT: Relative retention time
 SDDC: Silver diethyldithiocarbamate
 SGT-HEM: Silica gel treated-hexane
 extractable material
 SM: Standard Methods
 SPADNS: Common name for fluoride dye
 reagent which is a mixture of chemicals
 STGFAA: Stabilized Temperature Graphite
 Furnace Atomic Absorption Spectroscopy
 TKN: Total Kjeldahl Nitrogen
 TOC: Total Organic Carbon
 USGS: United States Geological Survey
 UV: Ultraviolet
 VCSB: Voluntary Consensus Standards Body
 WET: Whole Effluent Toxicity

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I. Statutory Authority

EPA is promulgating this rule pursuant to the authority of sections 301(a), 304(h), and 501(a) of the Clean Water Act (“CWA”) 33 U.S.C. 1311(a), 1314(h), and 1361(a). Section 301(a) of the CWA prohibits the discharge of any pollutant into navigable waters unless the discharge complies with, among other provisions, a National Pollutant Discharge Elimination System (NPDES) permit issued under section 402 of the CWA. Section 304(h) of the CWA requires the Administrator of the EPA to “* * * promulgate guidelines establishing test procedures for the analysis of pollutants that shall include the factors which must be provided in any certification pursuant to [section

401 of the CWA] or permit application pursuant to [section 402 of the CWA].” Section 501(a) of the CWA authorizes the Administrator to “* * * prescribe such regulations as are necessary to carry out this function under [the CWA].” EPA generally has codified its test procedure regulations (including analysis and sampling requirements) for CWA programs at 40 CFR part 136, though some requirements are codified in other Parts (e.g., 40 CFR chapter I, subchapters N and O).

II. Summary of Final Rule

The following sections describe the changes EPA is making in this final rule. In addition, further information concerning the rule may be found in a document prepared for this rule providing EPA’s responses to comments it received on the proposed rule. That document (“Response to Comments Document for the Methods Update Rule Proposal (80 CFR 8956, February 19, 2015”) is available in the electronic docket listed in the **ADDRESSES** section at the beginning of this document. The following sections describe changes EPA is making in this final rule.

A. New Versions of Previously Approved EPA Methods in 40 CFR 136.3 and Appendix A

This rule approves new versions of already approved EPA methods and corrects typographical errors in the methods. The following briefly describes the EPA methods added to part 136.

1. EPA Methods 608.3, 611, 624.1 and 625.1

Method 608.3, Organochlorine Pesticides and PCBs by GC/HSD. This method measures organochlorine pesticides and polychlorinated biphenyls (PCBs) in industrial discharges and other environmental samples by gas chromatography (GC) combined with a halogen-specific detector (HSD: e.g., electron capture, electrolytic conductivity), as provided under 40 CFR 136.1.

EPA Method 611, Haloethers. This method measures the following haloethers: Bis(2-chloroethyl) ether, bis(2-chloroethoxy) methane, 2, 2'-oxybis (1-chloropropane), 4-bromophenyl phenyl ether, and 4-chlorophenyl phenyl ether in municipal and industrial discharges by gas chromatography (GC) as provided under 40 CFR 136.1. The only change EPA has made is correcting a typographical error in the list of parameters by changing “4-Chlorophenyl phenyl ether” to “4-Chlorophenyl phenyl ether” and has

A_{is} = Area of the internal standard
 RF = Response factor, as defined in section 7.6.1

15.3 Calculate the concentration of the analyte in the sample using the concentration in the extract, the extract volume, the sample volume, and the dilution factor, per the following equation:

$$C_s = \frac{C_{ex} \times V_{ex} \times DF}{V_s \times 1000}$$

where:

C_s = Concentration of the analyte in the sample ($\mu\text{g/L}$)
 V_{ex} = Final extract volume (mL)
 C_{ex} = Concentration in the extract (ng/mL)
 V_s = Volume of sample (L)
 DF = Dilution factor

and the factor of 1,000 in the denominator converts the final units from ng/L to $\mu\text{g/L}$

15.4 If the concentration of any target analyte exceeds the calibration range, either extract and analyze a smaller sample volume, or dilute and analyze the diluted extract.

15.5 Quantitation of multi-component analytes.

15.5.1 PCBs as Aroclors. Quantify an Aroclor by comparing the sample chromatogram to that of the most similar Aroclor standard as indicated in section 14.3.2. Compare the responses of 3 to 5 major peaks in the calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 3 to 5 characteristic peaks chosen in section 7.5.1. Determine the concentration of each of the characteristic peaks, using the average calibration factor calculated for that peak in section 7.5.2, and then those 3 to 5 concentrations are averaged to determine the concentration of that Aroclor.

15.5.2 Other multi-component analytes. Quantify any other multi-component analytes (technical chlordane or toxaphene) using the same peaks used to develop the average calibration factors in section 7.5.2. Determine the concentration of each of the characteristic peaks, and then the concentrations represented by those characteristic peaks are averaged to determine the concentration of the analyte. Alternatively, for toxaphene, the analyst may determine the calibration factor in section 7.5.2 by summing the areas of all of the peaks for the analyte and using the

summed of the peak areas in the sample chromatogram to determine the concentration. However, the approach used for toxaphene must be the same for the calibration and the sample analyses.

15.6 Reporting of results. As noted in section 1.6.1, EPA has promulgated this method at 40 CFR part 136 for use in wastewater compliance monitoring under the National Pollutant Discharge Elimination System (NPDES). The data reporting practices described here are focused on such monitoring needs and may not be relevant to other uses of the method.

15.6.1 Report results for wastewater samples in $\mu\text{g/L}$ without correction for recovery. (Other units may be used if required by in a permit.) Report all QC data with the sample results.

15.6.2 Reporting level. Unless specified otherwise by a regulatory authority or in a discharge permit, results for analytes that meet the identification criteria are reported down to the concentration of the ML established by the laboratory through calibration of the instrument (see section 7.5 or 7.6 and the glossary for the derivation of the ML). EPA considers the terms "reporting limit," "quantitation limit," and "minimum level" to be synonymous.

15.6.2.1 Report the lower result from the two columns (see section 15.7 below) for each analyte in each sample or QC standard at or above the ML to 3 significant figures. Report a result for each analyte in each sample or QC standard below the ML as "<ML," where "ML" is the concentration of the analyte at the ML (e.g., if the ML is 10 $\mu\text{g/L}$, then report the result as <10 $\mu\text{g/L}$), or as required by the regulatory authority or permit. Report a result for each analyte in a blank at or above the MDL to 2 significant figures. Report a result for each analyte found in a blank below the MDL as "<MDL," where MDL is the concentration of the analyte at the MDL, or as required by the regulatory/control authority or permit.

15.6.2.2 In addition to reporting results for samples and blank(s) separately, the concentration of each analyte in a blank or field blank associated with that sample may be subtracted from the result for that sample, but only if requested or required by a regulatory authority or in a permit. In this case, both the sample result and the blank results must be reported together.

15.6.2.3 Report the result for an analyte in a sample or extract that has been diluted

at the least dilute level at which the peak area is within the calibration range (i.e., above the ML for the analyte) and the MS/MSD recovery and RPD are within their respective QC acceptance criteria (Table 4). This may require reporting results for some analytes from different analyses. Results for each analyte in MS/MSD samples should be reported from the same GC column as used to report the results for that analyte in the unspiked sample. If the MS/MSD recoveries and RPDs calculated in this manner do not meet the acceptance criteria in Table 4, the analyst may use the results from the other GC column to determine if the MS/MSD results meet the acceptance criteria. If such a situation occurs, the results for the sample should be recalculated using the same GC column data as used for the MS/MSD samples, and reported with appropriate annotations that alert the data user of the issue.

15.6.2.4 Results from tests performed with an analytical system that is not in control (i.e., that does not meet acceptance criteria for all of QC tests in this method) must not be reported or otherwise used for permitting or regulatory compliance purposes, but do not relieve a discharger or permittee of reporting timely results. See section 8.1.7 for dispositions of failures. If the holding time would be exceeded for a re-analysis of the sample, the regulatory/control authority should be consulted for disposition.

15.6.3 Analyze the sample by GC/MS or on a third column when analytes have co-eluted or interfere with determination on both columns.

Note: Dichlone and kepone do not elute from the DB-1701 column and must be confirmed on a DB-5 column, or by GC/MS.

15.7 Quantitative information that may aid in the confirmation of the presence of an analyte.

15.7.1 As noted in Section 14.3, the relative agreement between the numerical results from the two GC columns may be used to support the identification of the target analyte by providing evidence that co-eluting interferences are not present at the retention time of the target analyte. Calculate the percent difference (%D) between the results for the analyte from both columns, as follows:

$$\%D = \frac{\text{Higher result} - \text{Lower result}}{\text{Higher result}} \times 100$$

In general, if the %D of the two results is less than 50% (e.g., a factor of 2), then the pesticide is present. This %D is generous and allows for the pesticide that has the largest measurement error.

Note: Laboratories may employ metrics less than 50% for this comparison, including those specified in other analytical methods for these pesticides (e.g., CLP or SW-846).

15.7.2 If the amounts do not agree, and the RT data indicate the presence of the

analyte (per Section 14), it is likely that a positive interference is present on the column that yielded the higher result. That interferent may be represented by a separate peak on the other column that does not coincide with the retention time of any of the target analytes. If the interfering peak is evident on the other column, report the result from that column and advise the data user that the interference resulted in a %D value greater than 50%. If an interferent is not

identifiable on the second column, then the results must be reported as "not detected" at the lower concentration. In this event, the pesticide is not confirmed and the reporting limit is elevated. See section 8.1.7 for disposition of problem results.

Note: The resulting elevation of the reporting limit may not meet the requirements for compliance monitoring and the use of additional cleanup procedures may be required.

$$C_s (\mu\text{g/L}) = \frac{A_s \times C_{is} \times DF}{A_{is} \times RF}$$

Equation 2

Where:

C_s = Concentration of the analyte in the sample, and the other terms are as defined in Section 7.3.3.

13.2 Reporting of results

As noted in section 1.4.1, EPA has promulgated this method at 40 CFR part 136 for use in wastewater compliance monitoring under the National Pollutant Discharge Elimination System (NPDES). The data reporting practices described here are focused on such monitoring needs and may not be relevant to other uses of this method.

13.2.1 Report results for wastewater samples in $\mu\text{g/L}$ without correction for recovery. (Other units may be used if required by a permit.) Report all QC data with the sample results.

13.2.2 Reporting level. Unless otherwise specified in by a regulatory authority or in a discharge permit, results for analytes that meet the identification criteria are reported down to the concentration of the ML established by the laboratory through calibration of the instrument (see section 7.3.2 and the glossary for the derivation of the ML). EPA considers the terms "reporting limit," "limit of quantitation," "quantitation limit," and "minimum level" to be synonymous.

13.2.2.1 Report a result for each analyte in each field sample or QC standard at or above the ML to 3 significant figures. Report a result for each analyte found in each field sample or QC standard below the ML as "<ML," where ML is the concentration of the analyte at the ML, or as required by the regulatory/control authority or permit. Report a result for each analyte in a blank at or above the MDL to 2 significant figures. Report a result for each analyte found in a blank below the MDL as "<MDL," where MDL is the concentration of the analyte at the MDL, or as required by the regulatory/control authority or permit.

13.2.2.2 In addition to reporting results for samples and blanks separately, the concentration of each analyte in a blank associated with the sample may be subtracted from the result for that sample, but only if requested or required by a regulatory authority or in a permit. In this case, both the sample result and the blank result must be reported together.

13.2.2.3 Report a result for an analyte found in a sample that has been diluted at the least dilute level at which the area at the quantitation m/z is within the calibration range (*i.e.*, above the ML for the analyte) and the MS/MSD recovery and RPD are within their respective QC acceptance criteria (Table 7). This may require reporting results for some analytes from different analyses.

13.2.3 Results from tests performed with an analytical system that is not in control (*i.e.*, that does not meet acceptance criteria for any of the QC test in this method) must be documented and reported (*e.g.*, as a qualifier on results), unless the failure is not required to be reported as determined by the regulatory/control authority. Results

associated with a QC failure cannot be used to demonstrate regulatory compliance. QC failures do not relieve a discharger or permittee of reporting timely results. If the holding time would be exceeded for a re-analysis of the sample, the regulatory/control authority should be consulted for disposition.

14. Method Performance

14.1 This method was tested by 15 laboratories using reagent water, drinking water, surface water, and industrial wastewaters spiked at six concentrations over the range 5–600 $\mu\text{g/L}$ (References 4 and 16). Single-operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the analyte and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 8.

14.2 As noted in section 1.1, this method was validated through an interlaboratory study conducted in the early 1980s. However, the fundamental chemistry principles used in this method remain sound and continue to apply.

15. Pollution Prevention

15.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Many opportunities for pollution prevention exist in laboratory operations. EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, the laboratory should use pollution prevention techniques to address waste generation. When wastes cannot be reduced at the source, the Agency recommends recycling as the next best option.

15.2 The analytes in this method are used in extremely small amounts and pose little threat to the environment when managed properly. Standards should be prepared in volumes consistent with laboratory use to minimize the disposal of excess volumes of expired standards.

15.3 For information about pollution prevention that may be applied to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Governmental Relations and Science Policy, 1155 16th Street NW., Washington, DC 20036, 202-872-4477.

16. Waste Management

16.1 The laboratory is responsible for complying with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance is also required with any sewage discharge permits and regulations. An overview of requirements can

be found in Environmental Management Guide for Small Laboratories (EPA 233-B-98-001).

16.2 Samples at $\text{pH} < 2$, or $\text{pH} > 12$, are hazardous and must be handled and disposed of as hazardous waste, or neutralized and disposed of in accordance with all federal, state, and local regulations. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions. The laboratory using this method has the responsibility to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance is also required with any sewage discharge permits and regulations. For further information on waste management, see "The Waste Management Manual for Laboratory Personnel," also available from the American Chemical Society at the address in Section 15.3.

16.3 Many analytes in this method decompose above 500 °C. Low-level waste such as absorbent paper, tissues, and plastic gloves may be burned in an appropriate incinerator. Gross quantities of neat or highly concentrated solutions of toxic or hazardous chemicals should be packaged securely and disposed of through commercial or governmental channels that are capable of handling these types of wastes.

16.4 For further information on waste management, consult "Waste Management Manual for Laboratory Personnel and Less is Better-Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Governmental Relations and Science Policy, 1155 16th Street NW., Washington, DC 20036, 202-872-4477.

17. References

1. Bellar, T.A. and Lichtenberg, J.J. "Determining Volatile Organics at Microgram-per-Litre Levels by Gas Chromatography," Journal American Water Works Association, 66: 739 (1974).
2. "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants," U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1977, Revised April 1977.
3. Bellar, T.A. and Lichtenberg, J.J. "Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds," Measurement of Organic Pollutants in Water and Wastewater, C.E. Van Hall, editor, American Society for Testing and Materials, Philadelphia, PA. Special Technical Publication 686, 1978.
4. "EPA Method Study 29 EPA Method 624-Purgeables," EPA 600/4-84-054, National Technical Information Service, PB84-209915, Springfield, Virginia 22161, June 1984.
5. 40 CFR part 136, appendix B.

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} \times 100$$

% breakdown of Endrin

$$= \frac{\text{sum of degradation peak areas (Endrin aldehyde + Endrin ketone)}}{\text{sum of all peak areas (Endrin + Endrin aldehyde + Endrin ketone)}} \times 100$$

13.8.3 Both the % breakdown of DDT and of Endrin must be less than 20%, otherwise the system is not performing acceptably for DDT and endrin. In this case, repair the GC column system that failed and repeat the performance tests (sections 13.2 to 13.6) until the specification is met.

Note: DDT and endrin decomposition are usually caused by accumulation of particulates in the injector and in the front end of the column. Cleaning and silanizing the injection port liner, and breaking off a short section of the front end of the column will usually eliminate the decomposition problem. Either of these corrective actions may affect retention times, GC resolution, and calibration linearity.

14. Qualitative Identification

14.1 Identification is accomplished by comparison of data from analysis of a sample or blank with data stored in the GC/MS data system (sections 5.6.5 and 7.2.1.2). Identification of an analyte is confirmed per sections 14.1.1 through 14.1.4.

14.1.1 The signals for the quantitation and secondary m/z's stored in the data system for each analyte of interest must be present and must maximize within the same two consecutive scans.

14.1.2 The retention time for the analyte should be within ± 10 seconds of the analyte

in the calibration verification run at the beginning of the shift (section 7.3 or 13.4).

Note: Retention time windows other than ± 10 seconds may be appropriate depending on the performance of the gas chromatograph or observed retention time drifts due to certain types of matrix effects. Relative retention time (RRT) may be used as an alternative to absolute retention times if retention time drift is a concern. RRT is a unitless quantity (see Sec. 22.2), although some procedures refer to "RRT units" in providing the specification for the agreement between the RRT values in the sample and the calibration verification or other standard. When significant retention time drifts are observed, dilutions or spiked samples may help the analyst determine the effects of the matrix on elution of the target analytes and to assist in qualitative identification.

14.1.3 Either the background corrected EICP areas, or the corrected relative intensities of the mass spectral peaks at the GC peak maximum, must agree within 50% to 200% (1/2 to 2 times) for the quantitation and secondary m/z's in the reference mass spectrum stored in the data system (section 7.2.1.2), or from a reference library. For example, if a peak has an intensity of 20% relative to the base peak, the analyte is identified if the intensity of the peak in the sample is in the range of 10% to 40% of the

base peak. If identification is ambiguous, an experienced spectrometrist (section 1.7) must determine the presence or absence of the compound.

14.2 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different gas chromatographic retention times. Sufficient gas chromatographic resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

15. Calculations

15.1 When an analyte has been identified, quantitation of that analyte is based on the integrated abundance from the EICP of the primary characteristic m/z in Table 4 or 5. Calculate the concentration in the extract using the response factor (RF) determined in Section 7.2.2 and Equation 2. If the concentration of an analyte exceeds the calibration range, dilute the extract by the minimum amount to bring the concentration into the calibration range, and re-analyze the extract. Determine a dilution factor (DF) from the amount of the dilution. For example, if the extract is diluted by a factor of 2, DF = 2.

$$C_{ex} (\mu\text{g/mL}) = \frac{A_s \times C_{is}}{A_{is} \times RF}$$

Equation 2

where:

C_{ex} = Concentration of the analyte in the extract, in µg/mL, and the other terms are as defined in section 7.2.2.

Calculate the concentration of the analyte in the sample using the concentration in the extract, the extract volume, the sample

volume, and the dilution factor, per Equation 3:

$$C_{samp} (\mu\text{g/L}) = \frac{C_{ex} \times V_{ex} \times DF}{V_s}$$

Equation 3

where:

C_{samp} = Concentration of the analyte in the sample
 C_{ex} = Concentration of the analyte in the extract, in µg/mL
 V_{ex} = Volume of extract (mL)
 V_s = Volume of sample (L)
 DF = Dilution factor

15.2 Reporting of results. As noted in section 1.4.1, EPA has promulgated this method at 40 CFR part 136 for use in wastewater compliance monitoring under the National Pollutant Discharge Elimination System (NPDES). The data reporting practices described here are focused on such monitoring needs and may not be relevant to other uses of the method.

15.2.1 Report results for wastewater samples in µg/L without correction for recovery. (Other units may be used if required by in a permit.) Report all QC data with the sample results.

15.2.2 Reporting level. Unless specified otherwise by a regulatory authority or in a discharge permit, results for analytes that meet the identification criteria are reported

down to the concentration of the ML established by the laboratory through calibration of the instrument (see section 7.3.2 and the glossary for the derivation of the ML). EPA considers the terms "reporting limit," "quantitation limit," "limit of quantitation," and "minimum level" to be synonymous.

15.2.2.1 Report a result for each analyte in each field sample or QC standard at or above the ML to 3 significant figures. Report a result for each analyte found in each field sample or QC standard below the ML as "ML" where ML is the concentration of the analyte at the ML, or as required by the regulatory/control authority or permit. Report a result for each analyte in a blank at or above the MDL to 2 significant figures. Report a result for each analyte found in a blank below the MDL as "MDL," where MDL is the concentration of the analyte at the MDL, or as required by the regulatory/control authority or permit.

15.2.2.2 In addition to reporting results for samples and blanks separately, the concentration of each analyte in a blank associated with the sample may be subtracted from the result for that sample, but only if requested or required by a regulatory authority or in a permit. In this case, both the sample result and the blank results must be reported together.

15.2.2.3 Report a result for an analyte found in a sample or extract that has been diluted at the least dilute level at which the area at the quantitation m/z is within the calibration range (*i.e.*, above the ML for the analyte) and the MS/MSD recovery and RPD are within their respective QC acceptance criteria (Table 6). This may require reporting results for some analytes from different analyses.

15.2.3 Results from tests performed with an analytical system that is not in control (*i.e.*, that does not meet acceptance criteria for any QC test in this method) must be documented and reported (*e.g.*, as a qualifier on results), unless the failure is not required to be reported as determined by the regulatory/control authority. Results associated with a QC failure cannot be used to demonstrate regulatory compliance. QC failures do not relieve a discharger or permittee of reporting timely results. If the holding time would be exceeded for a re-analysis of the sample, the regulatory/control authority should be consulted for disposition.

16. Method Performance

16.1 The basic version of this method was tested by 15 laboratories using reagent water, drinking water, surface water, and industrial wastewaters spiked at six concentrations over the range 5–1300 $\mu\text{g/L}$ (Reference 2). Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the analyte and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 7.

16.2 As noted in section 1.1, this method was validated through an interlaboratory study in the early 1980s. However, the fundamental chemistry principles used in this method remain sound and continue to apply.

16.3 A chromatogram of the combined acid/base/neutral calibration standard is shown in Figure 2.

17. Pollution Prevention

17.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Many opportunities for pollution prevention exist in laboratory operations. EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, the laboratory should use pollution prevention techniques to address waste generation. When wastes cannot be reduced at the source, the Agency recommends recycling as the next best option.

17.2 The analytes in this method are used in extremely small amounts and pose little threat to the environment when managed properly. Standards should be prepared in volumes consistent with laboratory use to minimize the disposal of excess volumes of expired standards. This method utilizes significant quantities of methylene chloride. Laboratories are encouraged to recover and recycle this and other solvents during extract concentration.

17.3 For information about pollution prevention that may be applied to laboratories and research institutions, consult *Less is Better: Laboratory Chemical Management for Waste Reduction*, available from the American Chemical Society's Department of Governmental Relations and Science Policy, 1155 16th Street NW., Washington DC 20036, 202–872–4477.

18. Waste Management

18.1 The laboratory is responsible for complying with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance is also required with any sewage discharge permits and regulations. An overview of requirements can be found in *Environmental Management Guide for Small Laboratories* (EPA 233–B–98–001).

18.2 Samples at $\text{pH} < 2$, or $\text{pH} > 12$, are hazardous and must be handled and disposed of as hazardous waste, or neutralized and disposed of in accordance with all federal, state, and local regulations. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions. The laboratory using this method has the responsibility to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance is also required with any sewage discharge permits and regulations. For further information on waste management, see "*The Waste Management Manual for Laboratory Personnel*," also available from the American Chemical Society at the address in section 17.3.

18.3 Many analytes in this method decompose above 500 °C. Low-level waste

such as absorbent paper, tissues, and plastic gloves may be burned in an appropriate incinerator. Gross quantities of neat or highly concentrated solutions of toxic or hazardous chemicals should be packaged securely and disposed of through commercial or governmental channels that are capable of handling these types of wastes.

18.4 For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel and Less is Better-Laboratory Chemical Management for Waste Reduction*, available from the American Chemical Society's Department of Governmental Relations and Science Policy, 1155 16th Street NW., Washington, DC 20036, 202–872–4477.

19. References

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