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**ENVIRONMENTAL PROTECTION AGENCY**

**40 CFR Parts 136 and 260**

**[FRL–6341–9]**

**RIN 2040–AC63**

## Guidelines Establishing Test Procedures for the Analysis of Oil and Grease and Non-Polar Material Under the Clean Water Act and Resource Conservation and Recovery Act; Final Rule

**AGENCY:** Environmental Protection Agency (EPA).

**ACTION:** Final rule.

**SUMMARY:** This action approves use of EPA Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry (hereafter Method 1664) for use in EPA's Clean Water Act (CWA) programs. This action also deletes Method 9070, adds revised Method 9071B, and incorporates Method 1664 by reference for use in EPA's Resource Conservation and Recovery Act (RCRA) programs. Method 1664 is also approved for determination of non-polar material (NPM) as silica gel treated n-hexane extractable material (SGT-HEM) to support phaseout of use of CFC–113 for determination of NPM in EPA's CWA and RCRA programs.

These actions are being taken as a part of EPA's effort to reduce dependency on use of chlorofluorocarbons (CFCs) to protect Earth's ozone layer and to meet the CFC phaseout agreed to in the Montreal Protocol and required by the Clean Air Act Amendments of 1990. Method 1664 uses normal hexane (n-hexane) as the extraction solvent in place of 1,1,2-trichloro-1,2,2-trifluoroethane (CFC–113; Freon-113), a Class 1 CFC.

**DATES:** This regulation is effective June 14, 1999. For judicial review purposes, this final rule is promulgated as of 1:00 p.m. Eastern Standard Time on May 28, 1999 in accordance with 40 CFR 23.7.

The incorporation by reference of Method 1664 is approved by the Director of the Federal Register May 14, 1999.

**ADDRESSES:** Copies of the public comments received, EPA responses, and all other supporting documents (including references included in this notice) are available for review at the U.S. Environmental Protection Agency, Water Docket, 401 M Street SW, Washington, DC 20460. For access to docket materials, call 202–260–3027 on Monday through Friday, excluding Federal holidays, between 9:00 a.m. and 3:30 p.m. Eastern Time for an appointment.

Copies of Method 1664 are available from the National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161, (703) 605–6000 or (800) 553–6847. The NTIS publication number is PB99–121949.

Copies of the Third Edition of SW–846 and Updates I, II, IIA, IIB, and III (document number 955–001–00000–1) are available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, (202) 512–1800. Update IIIA is available through EPA's Methods Information Communication Exchange (MICE) Service. MICE can be contacted by phone at (703) 821–4690. Update IIIA can also be obtained by contacting the U.S. Environmental Protection Agency, Office of Solid Waste (5307W), OSW Methods Team, 401 M Street, SW, Washington, DC, 20460. Copies of the Third Edition and all of its updates are also available from the National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161, (703) 605–6000 or (800) 553–6847. In addition, a CD-ROM version of SW–846, Third Edition, as amended by Updates I through III is available from NTIS (PB97–501928). In the future, the CD-ROM will be updated by NTIS to include additional updates, including Update IIIA.

An electronic version of Method 1664 and Method 9071B are also available via the Internet at http://www.epa.gov/OST.

**FOR FURTHER INFORMATION CONTACT:** For information regarding Method 1664 and its use in Clean Water Act programs, contact Maria Gomez-Taylor, Engineering and Analysis Division (4303), USEPA Office of Science and Technology, 401 M Street, SW, Washington, DC 20460, or call (202) 260–1639. For information regarding Update IIIA and the use of Method 1664 in the Resource Conservation And Recovery Act programs, contact Gail Hansen, Office of Solid Waste (5307W), USEPA, 401 M Street, SW, Washington, DC 20460, or call (703) 308–8855.

**SUPPLEMENTARY INFORMATION:**

### Potentially Regulated Entities

EPA Regions, as well as States, Territories and Tribes authorized to implement the National Pollutant Discharge Elimination System (NPDES) program, issue permits that comply with the technology-based and water quality-based requirements of the Clean Water Act. In doing so, the NPDES permitting authority, including authorized States, Territories, and Tribes, make a number of discretionary choices associated with permit writing, including the selection of pollutants to be measured and, in many cases, limited in permits. If EPA has ''approved'' standardized testing procedures (i.e., promulgated through rulemaking) for a given pollutant, the NPDES permit must include one of the approved testing procedures or an approved alternate test procedure. Therefore, entities with NPDES permits could be affected by the standardization of testing procedures in this rulemaking. These entities may be affected because NPDES permits may incorporate the standardized testing procedure approved for use in today's rulemaking. In addition, when a State, Territory, or authorized Tribe provides certification of federal licenses under Clean Water Act section 401, States, Territories and Tribes are directed to use the standardized testing procedures. Under the RCRA program, this method may be required as part of a hazardous waste delisting petition. Categories and entities that may ultimately be affected include:

| Category | Examples of potentially regulated entities |
|---|---|
| Regional, State and Territorial Governments and Indian Tribes. | States, Territories, and Tribes authorized to administer the NPDES permitting program; States, Territories, and Tribes providing certification under Clean Water Act section 401; Governmental NPDES permittees; Regional and State offices implementing delisting petitions. |
| Industry | Industrial NPDES permittees; delisting petitioners. |
| Municipalities. | Publicly-owned treatment works with NPDES permits. |

This table is not intended to be exhaustive, but rather provides a guide for readers regarding entities likely to be affected by this action. This table lists the types of entities that EPA is now aware could potentially be affected by this action. Other types of entities not listed in the table could also be affected. If you have questions regarding the applicability of this action to a particular entity, consult the person listed in the preceding **FOR FURTHER INFORMATION CONTACT** section.

### Outline of Preamble

I. Authorities
A. Clean Water Act
B. Resource Conservation and Recovery Act
C. Clean Air Act Amendments of 1990
II. Background and History

A. Regulatory Background
B. Data Gathering
1. EPA's Freon Replacement Studies
2. Water Pollution Performance Evaluation Data
3. Data Received from Commenters
III. Explanation of Today's Action
A. Application of Method 1664 in Clean Water Act Program
1. General Use
2. Search for Other Uses of CFCs in Clean Water Act Program
B. Application of Method 1664 in Resource Conservation and Recovery Act Program
C. Use of Solid-phase Extraction (SPE)
D. Differences in Results Produced by CFC–113 and n-Hexane and Determinations of Compliance
IV. Timing of Required Use of Method 1664 and Phaseout of Use of CFC–113
V. Improvements and Changes to Method 1664 Since Proposal
A. Names and Name Changes
1. Oil and Grease
2. Non-polar Material
B. Other Changes and Improvements
1. Changes to Quality Control
2. Miscellaneous Changes and Improvements
VI. Public Participation and Response to Comments
A. Regulatory Issues
B. Health and Safety Concerns
C. Economic Concerns
D. Solid-phase Extraction (SPE)
1. Comments Supporting Use of SPE
2. Comments Expressing Concern About the Use of SPE
E. Grace period for CFC–113
F. Use of Silica-gel Treated n-Hexane Extractable Material Procedure
G. Detection and Quantitation
H. Matrix Effects
I. Method Modifications
J. Matrix Spike/Matrix Spike Duplicate
K. Precision and Recovery
L. Differences in Results Produced by *n*-Hexane and CFC–113
M. Method Validation and QC Acceptance Criteria
N. Quality Control
O. Sample Collection and Preservation
P. Miscellaneous Issues
VII. Regulatory Requirements
A. Executive Order 12866
B. Unfunded Mandates Reform Act
C. Regulatory Flexibility Act
D. Paperwork Reduction Act
E. Submission to Congress and the General Accounting Office
F. National Technology Transfer and Advancement Act
G. Executive Order 13045
H. Executive Order 12805
I. Executive Order 13084

## I. Authorities

### *A. Clean Water Act*

These regulations are being promulgated under the authority of sections 301, 304, and 501(a) of the Clean Water Act (CWA), 33 U.S.C. 1311, 1314(h), 1361(a).

### *B. Resource Conservation and Recovery Act*

These regulations are being promulgated under the authority of sections 1006, 2002(a), 3001–3007, 3010, 3013–3018, and 7004 of the Solid Waste Disposal Act (SWDA), as amended by the Resource Conservation and Recovery Act of 1976 (RCRA), as amended (42 U.S.C. 6905, 6912(a), 6921–6927, 6930, 6934–6930, and 6974).

### *C. Clean Air Act Amendments of 1990*

These regulations are consistent with intent of sections 604, 606, and 608 of the 1990 Clean Air Act Amendments (CAAA) to phase out production of Class I CFCs and reduce use and emissions of Class I CFCs to the lowest achievable level, and with section 613 of CAAA to reduce the Federal procurement of products and services that employ CFCs.

## II. Background and History

### *A. Regulatory Background*

This final rule affects regulations implementing analytical methods under the Clean Water Act and the Resource Conservation and Recovery Act. This final rule supports, in part, EPA's Stratospheric Ozone Protection Program in the Office of Air and Radiation (OAR). The regulatory background for each of these programs is not given in detail in this rule; rather, pertinent proposals and rules are referenced, as follows:

EPA provided a history of analytical methods under 40 CFR part 136 on February 7, 1991 (56 FR 5090) in the proposal of EPA Method 1613. The Agency presented a brief background on the use of analytical methods as support for effluent limitations, permit applications, and compliance monitoring, and a brief description of the alternate test procedure program (ATP) in the proposal of several new 40 CFR part 136 methods on October 18, 1995 (60 FR 53988). The details of Method 1664 and issues surrounding its use were given at proposal on January 23, 1996 (61 FR 1730).

The EPA Office of Solid Waste gave a brief description of the regulatory framework for SW–846 methods in a final rule promulgating the third update to the SW–846 methods on June 13, 1997 (62 FR 32452). Method 1664 is approved as ''guidance'' under the RCRA programs (see Section III.B.). Other methods may be used under the conditions stated in the RCRA regulations (see 40 CFR part 268).

The regulatory background for development of effluent guidelines and standards is given in proposed and final rules for these guidelines and standards. See, for example, Section III of the final rule for the Coastal Subcategory of Oil and Gas Extraction promulgated on December 16, 1996 (61 FR 66086).

The background and history of applicability of EPA's Stratospheric Ozone Protection Program to analytical methods requiring use of CFCs in EPA's CWA programs was given in a proposal for an earlier EPA method for determination of oil and grease on July 3, 1991 (56 FR 30519). The Montreal Protocol and Section 604 of the Clean Air Act Amendments of 1990 impose limits on the production and consumption of certain ozone-depleting substances including CFC–113 (see 40 CFR part 82).

### *B. Data Gathering*

Data gathered to support today's final rule were Freon replacement studies and other data gathered prior to proposal, data collected in EPA's Water Pollution (WP) Performance Evaluation (PE) Study 038, and data received from commenters.

#### 1. EPA's Freon Replacement Studies

EPA summarized details of EPA studies that support today's final rule at proposal (see 61 FR 1730) and presented information in reports of EPA's Phase I Freon Replacement Study (EPA–821–R–93–011), Phase II Freon Replacement Study (EPA–820–R–95–003), and Method Validation Study (821–R–95–036). These reports are included in the Water Docket for the proposed rule.

#### 2. Water Pollution Performance Evaluation Data

EPA has collected data on the performance of laboratories conducting routine analyses of wastewaters through water pollution performance evaluation (PE) studies. For water pollution PE study 038 conducted in late calendar year 1997, EPA accepted data from laboratories determining oil and grease using either CFC–113 or n-hexane. The results of this study are summarized in Table 1 and show that, for this study, CFC–113 and n-hexane extracted identical average amounts of oil and grease.

TABLE 1.—RESULTS OF PERFORMANCE EVALUATION STUDY 038 FOR DETERMINATION OF OIL AND GREASE

| Solvent | Number of samples | Mean (mg/L) | Standard deviation |
|---|---|---|---|
| CFC–113 .... | 1,101 | 11.4 | 2.1 |
| n-hexane ..... | 353 | 11.4 | 2.4 |

US EPA ARCHIVE DOCUMENT

3. Data Received From Commenters

As detailed in other sections of this preamble and in the detailed comments and responses included in the Docket, EPA utilized data submitted in response to the proposal of Method 1664 for improvements included in the version of Method 1664 being approved for use today. All comments are included in the Water Docket.

**III. Explanation of Today's Action**

Today's final rule approves Method 1664 determination of n-hexane extractable material (HEM; oil and grease) and silica-gel treated n-hexane extractable material (SGT–HEM; non-polar material) in EPA's CWA and RCRA programs. Today's action has an economic benefit to regulated entities measuring oil and grease and non-polar material (NPM) in that the cost of CFC–113 is considerably greater than the cost of n-hexane as a result of the production phase-out of CFCs and increased tariffs on their use. The costs for CFCs being phased out can be expected to rise as production ceases and tariffs increase further. Because n-hexane is a product of oil refining on a large scale, the impact of the additional demand for n-hexane is expected to be insignificant. As pointed out by commenters, however, laboratory use of CFC–113 was small in comparison to other uses. Indeed, many commenters claimed that because of safety problems and the disparity in results produced by use of CFC–113 vs. n-hexane, use of CFC–113 should be continued indefinitely. Based on these comments and an extension of the laboratory use exemption to 2005, EPA has decided to allow continued use of methods that use CFC–113 and to approve use of Method 1664 for those regulated entities that desire to switch to use of n-hexane.

*A. Application of Method 1664 in EPA's Clean Water Act Programs*

1. General Use

Method 1664 will be used in EPA's wastewater program for regulation development, permit applications, and compliance monitoring. More than 600 industrial subcategories are regulated under Clean Water Act pollution control programs (see the rules for these industrial subcategories at 40 CFR parts 400–510). Nearly all of these regulations contain nationwide effluent guidelines and standards limiting the amount of oil and grease that may be discharged from facilities in these subcategories. EPA estimates that more that 10,000 National Pollutant Discharge Elimination System (NPDES) permits contain a limit for oil and grease, potentially necessitating an estimated minimum of 25,000 measurements annually.

2. Search for Other Uses of CFCs in Clean Water Act Program

EPA performed computerized string searches of 40 CFR parts 100–149, and of Subchapter N, ''Effluent Guidelines and Standards,'' at 40 CFR parts 400–500 for ''TPH'', ''Freon-113'', ''CFC–113'', ''chlorofluoro'', ''413.1'', and related terms in an attempt to locate references to CFC–113 and Method 413.1 for determination of oil and grease. EPA could find no references other than in 40 CFR part 136 and to the procedure for petroleum hydrocarbons in the Coil Coating standards at 40 CFR 465.03(c). EPA intends to consider allowing use of Method 1664 through subsequent rulemaking for the Coil Coating Point Source Category.

*B. Application of Method 1664 in Resource Conservation and Recovery Act Program*

Analytical methods found acceptable for testing under Subtitle C of RCRA are contained in OSW publication SW–846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. Use of some of these methods is required by some of the hazardous waste regulations under Subtitle C of RCRA. In other situations, SW–846 functions as a guidance document setting forth acceptable, although not required, methods to be implemented by the user, as appropriate, in satisfying RCRA-related sampling and analysis requirements. As of Update III to SW–846, the two SW–846 methods for determination of oil and grease have been Method 9070 for waters and aqueous wastes, and Method 9071A for solid and semi-solid material such as soil, sediment, and sludge. Method 9070 is virtually identical to presently approved CWA methods for determination of oil and grease. Method 9071A employs drying of the sample with magnesium sulfate and Soxhlet extraction with CFC–113 for the determination. These methods are not specifically required by any RCRA regulation, although they can be required as part of a hazardous waste de-listing demonstration.

In today's final rule, SW–846 is being amended further to delete Method 9070 and to include revised Method 9071B as Update IIIA. Specifically, Method 9071B addresses the use of n-hexane instead of CFC–113 as the extraction solvent, in a manner consistent with the use of n-hexane in Method 1664. In addition, in place of Method 9070, which uses CFC–113 as the extraction solvent in the testing of waters and aqueous wastes, the Agency is incorporating by reference Method 1664 in the RCRA regulations. As part of Update IIIA, SW–846 refers the regulated community to Method 1664 for testing previously conducted using Method 9070.

EPA compared results of Soxhlet extraction of solids and sludges with various solvents, including CFC–113 and n-hexane, in the Phase I Freon Replacement Study (EPA–821–R–93–011). Results of this study showed that, as with testing of waters which was described in the Phase I study report that was included in the Docket at proposal, CFC–113 and n-hexane extract different amounts of material. However, for petroleum-based samples, the amount of material extracted by CFC–113 and n-hexane was not significantly different. This is the same conclusion that was reached regarding extraction of waters. As a result, and for the other reasons allowing use of Method 1664 detailed at proposal and in other sections of this preamble as supported by the information contained in the Water Docket, EPA believes that changes to SW–846 as a result of Update IIIA, i.e., the use of Method 1664 in place of Method 9070 and the addition of Method 9071B to SW–846, are appropriate and logical outgrowths of the Agency's efforts to reduce dependency on the use of CFCs.

*C. Use of Solid-Phase Extraction (SPE)*

SPE uses a cartridge or disk for removal of the oil and grease from the sample. A detailed description of the SPE technique was provided at proposal (61 FR 1730). Even prior to proposal of Method 1664, vendors of SPE devices had requested that SPE be an allowed technique in the Method. Proposed Method 1664 allowed use of SPE, but required a demonstration that SPE produced results equivalent to results produced by the separatory funnel liquid-liquid extraction technique (LLE) written in Method 1664. Vendors and other commenters objected to this requirement, claiming that SPE provided sufficient advantages in solvent reduction, reduced analysis time, reduced emulsion formation, and other advantages so that its use should be allowed without prior demonstration of equivalency. EPA discussed the issue extensively at proposal and in public workshops and meetings, and specifically solicited data demonstrating equivalency of results produced by SPE and LLE. Data received were mixed, with some data demonstrating that results produced are equivalent and other data demonstrating that results produced are significantly different. EPA reopened the comment period (61

US EPA ARCHIVE DOCUMENT

FR 26149) to allow submission of further data, and EPA provided a notice of availability (62 FR 51621) of these and other data so that EPA could consider these data for today's final rule.

Discussions of the detailed issues on SPE are summarized in Section VI of this preamble and given in the detailed comments and responses included in the Docket. Based on comments received and supporting data, EPA is allowing the use of SPE in the version of Method 1664 being approved today without a prior demonstration of equivalency. However, EPA has added a note at the beginning of the extraction procedure (Section 11.3) in Method 1664 to indicate that it is the discharger/generator's responsibility to assure that the results produced are equivalent. If there is doubt about this equivalency, liquid/liquid extraction is definitive for the measurement.

EPA also acknowledges that if a Region, State, or other permitting authority has concerns about the difference in results produced by SPE and LLE, that authority may specify in the permit the use of one of the two techniques.

*D. Differences in Results Produced by CFC–113 and n-Hexane and Determinations of Compliance*

Since EPA announced results of the Phase I Freon Replacement Study in 1993, several commenters expressed concerns about the impact of differences resulting from substitution of CFC–113 with *n*-hexane on determinations of compliance under the NPDES program and pretreatment programs. EPA discussed this issue at proposal (61 FR 1730; January 23, 1996), and discussed the issue in workshops, conferences, and seminars between proposal and development of today's final rule.

After proposal, EPA received numerous requests from States and EPA Regions for guidance on implementation of Method 1664. On July 9, 1996, EPA issued guidance to Pretreatment Coordinators and Regional NPDES Contacts. A copy of the memorandum is included in the Docket for today's final rule. In part, this memorandum states the following:

''EPA acknowledges that, due to the diverse nature of discharges, there may be instances in which *n*-hexane will extract an amount of oil and grease greater or less than the amount extracted by Freon-113. If these instances affect compliance, the permitting authority may wish to consider establishing a conversion factor, multiplier, or divisor to account for these differences in the permit. EPA emphasizes that few, if any, instances will likely be found in which the differences affect compliance and, therefore, urges direct substitution of the presently approved methods with Method 1664 when the date of substitution is announced in the Federal Register.''

By today's final rule, EPA still believes that the approach outlined in the memorandum appropriately accommodates any significant discrepancies that could arise in determining compliance with limitations or standards for oil and grease using the new method. Based on the results from the Freon Replacement Studies, EPA found that, on average, *n*-hexane extracted approximately 96% of the material extracted using CFC–113. Therefore, while there may be some effluent matrices where *n*-hexane will extract more material than CFC–113, on the whole, most dischargers would have little risk of a determination of non-compliance with existing limits. The slightly smaller amount of oil and grease extracted by *n*-hexane (96% versus 100% by CFC–113) is not statistically significant because errors in oil and grease measurement are in the order of 10 percent relative standard deviation. A coarse estimate of 95% confidence limits around the 96% recovery by *n*-hexane is 96 plus or minus 20%, or the true difference lies somewhere between 76–116%. This encompasses 100% or no difference. Given the lack of significance of the 4% difference, the measurement error that would be encountered in the side-by-side comparison (estimated at 10% for each measurement), the potentially significant cost of a side-by-side comparison with each discharge and the low anticipated likelihood that a significant difference would be found (based on EPA's studies), EPA does not recommend a side-by-side comparison for each discharge. Instead, EPA continues to recommend a direct replacement of the approved Freon methods with Method 1664.

However, to accommodate regulated entities concerned about differences produced, EPA is not withdrawing approved use of methods employing CFC–113, a Class I ozone depleting substance. If a discharger/industrial user has concerns about measuring oil and grease, the discharger/industrial user may choose to perform a side-by-side comparison of Method 1664 and any of the approved methods that it previously used to measure compliance with the limitation or standard for oil and grease. For the side-by-side comparison, EPA suggests, at a minimum, analysis of three replicates of each sample by each method on any seven days over a minimum 30-day period, for a total of 42 analyses (21 by the previously used method and 21 by Method 1664). For this side-by-side comparison the laboratory should use the LLE procedure (not the optional SPE procedure) in Method 1664 because of the possible confounding of results that could occur when two variables (SPE and the solvent) are changed simultaneously. EPA suggests that all six results associated with any result less than the minimum level (<ML) not be used in the comparison because it is desirable to have actual measured values to test equivalency of results using the different methods. In the event that a test result less than the ML is obtained, the number of tests should be increased to provide a minimum of seven paired triplicate results for the comparison.

Statistical significance should be tested according to procedures for development of the root-mean-square deviation (RMSD) detailed in EPA's Freon Replacement Study reports using results obtained with CFC–113 as the reference. If the RMSD is within the acceptance limit, the results obtained using the different methods are equivalent.

## IV. Timing of Required Use of Method 1664 and Phaseout of Use of CFC–113

EPA proposed to withdraw approved use of previously approved methods and require use of Method 1664 on a date exactly six months after the date of publication of a final rule. This time lag was to allow for existing supplies of CFC–113 to be used, for laboratories to become familiar with Method 1664, and for dischargers/generators/industrial users and regulatory authorities to determine if a conversion factor based on a difference in the amount of material extracted is appropriate. Commenters suggested alternate dates for withdrawal, ranging between ''immediate withdrawal of presently approved methods'' and ''continue use of CFC–113 methods indefinitely'' (see Section VI below and the detailed comments and responses in the Docket for today's final rule). To accommodate regulated entities' concern that the requirement to change from CFC-based methods to Method 1664 could result in non-compliance, and based on an extension of the time for laboratory use of CFC–113 to 2005, EPA has decided to approve use of Method 1664 but not withdraw approved use of the CFC-based methods.

EPA strongly encourages dischargers/generators/industrial users to substitute use of Method 1664 beginning on the effective date of today's rulemaking rather than awaiting reissuance of the existing permit that currently requires

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use of a CFC–113 method. Also, instead of awaiting permit reissuance, EPA encourages prompt modification of the existing permit to specify the use of Method 1664. To accommodate those permittees wishing to use Method 1664 once today's rule becomes effective and to expedite reduction in CFC–113 use, EPA will exercise enforcement discretion with respect to the method used for compliance. This enforcement discretion does not extend, however, to liability for any violation of a permit limitation or condition, including the oil and grease limitation, only to a requirement to use a CFC–113 method to determine compliance or non-compliance. If non-compliance results from the use Method 1664, the permitting authority may establish a conversion factor, as detailed earlier in this preamble.

## V. Improvements and Changes to Method 1664 Since Proposal

The Agency has revised Method 1664 (''Method 1664, Revision A'') to indicate that it is different from previous versions. The significant changes resulting in this revision are the change of name from ''total petroleum hydrocarbons'' to ''non-polar material,'' the change of the status of the matrix spike duplicate (MSD) from a requirement to a suggestion, the change of an analytical batch to a maximum of 20 samples, and the allowed use of solid-phase extraction (SPE) without a demonstration of equivalency.

### *A. Names and Name Changes*

Confusion of the names used for the analytes determined by Method 1664 prompted EPA to examine these names. As a result, the name ''*n*-hexane extractable material'' (HEM) has been retained to be synonymous with ''oil and grease,'' but the name ''total petroleum hydrocarbons (TPH)'' has been dropped in favor of ''non-polar material'' (NPM) to indicate ''silica-gel treated *n*-hexane extractable material'' (SGT–HEM), as detailed below.

#### 1. Oil and Grease′

The EPA Administrator designated ''oil and grease'' as a conventional pollutant under the Clean Water Act (see 40 CFR 401.16). Oil and grease consists of those chemical substances extracted from water or wastes using a solvent. The nature of the substances extracted are determined by the extracting solvent and the extraction technique. Both CFC–113 and n-hexane extract many pure materials at nearly 100 percent efficiency. When mixtures of substances that typically occur in complex wastewater discharges and in complex solid and semi-solid wastes are present, the two solvents may extract different amounts of material. For most wastes and wastewaters, materials commonly extracted are relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases, and related materials. Because the nature and amount of material extracted is defined by the solvent and, to a lesser degree, by the details of the procedure used for extraction, EPA uses the term ''method-defined analyte'' to identify oil and grease.

Today's final rule approves use of Revision A of EPA Method 1664: n-Hexane Extractable Material (HEM; Oil and Grease) and Silica-gel Treated n-Hexane Extractable Material (SGT–HEM; Non-polar Material) by Extraction and Gravimetry, in which n-hexane is used as the extracting solvent, in addition to currently approved methods in which CFC–113 is used as the extracting solvent. The name ''n-hexane extractable material'' (HEM) reflects that it is the material extracted by normal hexane (n-hexane) that is being measured using Method 1664. The common name ''oil and grease'' is being retained because of its familiarity to the analytical community.

#### 2. Non-Polar Material

At proposal, EPA used the term ''total petroleum hydrocarbons'' (TPH) to designate the substances that remain after n-hexane extractable material is exposed to silica gel. Use of the term ''total petroleum hydrocarbons'' and the abbreviation TPH was confusing to the analytical community because the term is used in other analytical methods that measure a different property or material, in some instances by a different analytical technique. For example, EPA Method 418.1 and Standard Method 5520 F measure polar materials termed ''petroleum hydrocarbons'' using infrared spectroscopy, and certain SW–846 and State methods measure TPH by gas chromatography. The term ''non-polar material'' (NPM) was chosen for use in today's version of Method 1664 to avoid confusion with these uses of the term TPH.

Silica gel has the property that it removes ''polar'' material. Polar material includes aromatic compounds (those containing one or more benzene rings), unsaturated compounds (those containing one or more double bonds), and compounds containing atoms other than carbon and hydrogen (e.g., chlorine, oxygen, nitrogen, sulfur), and other compounds. Polar material also includes aromatic, phenolic, and heterocyclic compounds in petroleum and petroleum products, soaps, and animal fats. Silica gel adsorbs these polar materials, so the material that remains is ''non-polar material.'' Non-polar material contains straight and branched chain hydrocarbons (aliphatic hydrocarbons) and other chemical substances in which there are either no mixture of atoms of different types (hetero-atoms; e.g., chlorine, oxygen, nitrogen, sulfur) or these mixtures are ''balanced'' in the molecule. For example, the pollutant hexachloroethane, although containing chlorine atoms, does not exhibit sufficient polarity to be adsorbed by silica gel. As with HEM, the exact nature and amount of substances that will be adsorbed by silica gel is defined by the adsorption process. Therefore, as with oil and grease (HEM), SGT–HEM (NPM) is a ''method-defined analyte.''

### B. Other Changes and Improvements

#### 1. Changes to Quality Control (QC)

EPA has made two changes to the QC in Method 1664 in response to comments that the QC was onerous to laboratories: (1) the requirement for a matrix spike duplicate (MSD) has been changed to a suggestion; and (2) the size of an analytical batch has been increased to a maximum of 20 samples. This QC is consistent with the QC in methods for use under the CWA that are published at 40 CFR part 136, Appendix A. These methods use a single matrix spike (termed a ''QC check sample'' in those methods) to evaluate matrix effects and require matrix spikes at a minimum frequency of five percent (1 in 20) of samples from a given discharge/waste stream. The on-going precision and recovery (OPR) sample and blank are also at a minimum frequency of five percent (1 in 20). For those laboratories wishing to evaluate precision with each analytical batch, an MSD is suggested but not required.

#### 2. Miscellaneous Changes and Improvements

Nearly all of the other improvements to Method 1664 are minor technical improvements that correct or clarify language in the Method. Most improvements were incorporated in response to a comment or comments. EPA refers readers to the comments and responses detailed in the Docket for additional information. EPA believes that none of these changes or improvements or the other changes and improvements warrant re-proposal of Method 1664. Miscellaneous changes and improvements include:

- The hexadecane/stearic acid standard solution has been diluted by a factor of 2 and twice as much standard

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is spiked to avoid reported precipitation problems with this standard.

- Instructions have been amplified to rinse all glassware surfaces with solvent after transfer of sample or standards from one container to another to avoid reported problems that sample and standards cannot be recovered quantitatively.
- Performance data from EPA's validation study have been added to Section 13.
- A procedure was added for drying the sample to constant weight. The procedure was provided by the American Petroleum Institute.
- The section on safety was expanded to address in greater detail personnel monitoring and the hazards of handling n-hexane.
- A requirement was added that certain pieces of equipment such as the hot plate, centrifuge, and fume hood be specified as explosion proof.
- The QC acceptance criteria have been widened based on EPA's validation study.
- A procedure for collecting four grab samples over the course of a day for laboratory compositing was added.
- The term ''discharge'' was defined to be consistent with the words ''discharge'' and ''matrix type'' in EPA's Streamlining Initiative proposed on March 28, 1997 (62 FR 14976).
- A suggestion for back-extraction was added to aid in removal of salt from extracts of produced water samples to address concerns expressed by the American Petroleum Institute.
- References to the solvent removal process were changed from ''evaporation'' to ''distillation'' to indicate that the process recovers the solvent. Similarly, the word ''waste'' was changed to ''distillate'' to preclude indications that the distillate may be hazardous waste.
- Use of a greater amount of silica gel is now allowed so that greater amounts of polar material can be adsorbed in the SGT–HEM procedure, and the ratio of amount of silica gel to the amount HEM has been clarified.
- The top-loading analytical balance and centrifuge are made optional because they may not be needed.
- A limit has been placed on the amount of spiking solution that may be added to a sample for the matrix spike and matrix spike duplicate. The purpose of adding this limit is to preclude adding large amounts of acetone to the sample, thus possibly allowing the spiking material to be dissolved in the aqueous phase and not recovered in the extraction.

## VI. Public Participation and Response to Comments

The Agency proposed Method 1664 for use on January 23, 1996 (61 FR 1730). The comment period at proposal closed on March 25, 1996. On May 24, 1996 (61 FR 26149), EPA reopened the comment period for the purpose of accepting additional data and inviting comments. The reopened comment period closed on July 23, 1996. EPA continued to receive data and comments after the close of the reopened comment period. Because EPA desired to use some of these data to support the information and decisions in today's final rule, EPA issued a notice of data availability and request for comment on October 2, 1997 (62 FR 51621). The comment period on the notice closed on November 3, 1997.

In the proposal and for the reopened comment period, EPA solicited data comparing various extraction solvents, data comparing use of SPE with the LLE procedures in Method 1664, and comments on the other operational aspects of Method 1664. EPA is pleased with the content and nature of the comments received. Many contained analytical data and/or constructive comments for improvement of the Method. As a result, EPA has modified Method 1664, where appropriate, to respond to commenters' suggestions. Significant comments received are summarized below, along with EPA's response. To the extent practicable, the comments have been categorized by subject. Detailed comments and their accompanying responses are included in the Docket for today's final rule.

EPA thanks commenters for data and constructive suggestions and believes that the version of Method 1664 being promulgated today will provide reliable data for compliance monitoring.

### *A. Regulatory Issues*

*Comment:* Oil and grease is not a viable parameter for National Pollutant Discharge Elimination System (NPDES) compliance and there is no objectivity in permitting with an empirically based method.

*Response:* Oil and grease is a conventional pollutant designated pursuant to section 304(a)(4) of the Federal Water Pollution Control Act (FWPCA) and codified at 40 CFR 401.16, and therefore must be monitored for NPDES compliance under an effluent guideline or when deemed appropriate by a regulatory authority.

*Comment:* As with Method 413.1, Method 1664 measures non-oil and grease substances such as surfactants, soaps, and emulsifiers that will unduly subject dischargers to continual permitting and compliance difficulties.

*Response:* EPA recognizes that it may be inappropriate to include certain substances in the determination of oil and grease. However, discharges of pollutants, including surfactants, soaps, emulsifiers, and other substances, is prohibited under the Clean Water Act unless in compliance with an NPDES permit. EPA has provided the SGT–HEM (NPM) procedure in Method 1664 to allow development of effluent guidelines and in permitting situations for those instances in which removal of these substances is appropriate, and to allow a regulatory authority to specify measurement of SGT–HEM for compliance monitoring.

### *B. Health and Safety Concerns*

*Comment:* *n*-Hexane is a safety hazard compared to CFC–113. *n*-Hexane has a flash point of −23°C (−9 °F), has explosive limits in air in the range of 1.2–6.9 percent, and poses a serious fire risk when heated or exposed to flame. There are multiple ignition sources in a laboratory, including Bunsen burners and high temperature furnaces. Method 1664 should be performed in an explosion-proof hood.

*Response:* EPA agrees that *n*-hexane is comparatively more hazardous than CFC–113. Proposed EPA Method 1664 contained explicit precautions concerning the handling of *n*-hexane and the recommendation that material safety data sheets (MSDSs) be made available to laboratory personnel. EPA also included references to information on laboratory safety. EPA has expanded and re-emphasized these precautions in the version of Method 1664 being approved today.

*Comment:* EPA needs to modify Method 1664 to give information on the toxicity of *n*-hexane and on safety precautions required for safe handling and storage. *n*-Hexane is a known neurotoxin.

*Response:* EPA has expanded the section on safety and the health effects of *n*-hexane in the version of Method 1664 being approved for use in today's final rule.

### *C. Economic Concerns*

*Comment:* Method 1664 is more complicated and more labor intensive, requires a greater analysis time, and will be more expensive to practice.

*Response:* EPA agrees that Method 1664 will require a somewhat longer analysis time because of the increase in time required for extraction, *n*-hexane evaporation, and QC. However, regulated entities and their laboratories will benefit from lower costs for the *n*-

US EPA ARCHIVE DOCUMENT

hexane extraction solvent because CFC–113 is becoming difficult to obtain as well as expensive. EPA believes that any cost increases will be small and that the benefit outweighs the cost because it will reduce the use of ozone-depleting chlorofluorocarbons, thus protecting the Earth's ozone layer. Further, EPA is not withdrawing allowed use of methods that employ CFCs. If a regulated entity desires to continue use of a Freon-based method for economic reasons, the entity may continue to use that method.

*Comment:* Disadvantages of using *n*-hexane include: the lower density that causes *n*-hexane to float on the water sample making extraction more difficult and time consuming than with CFC–113 that sinks; the higher water solubility of *n*-hexane requiring more drying agent or use of phase-separation paper; the greater tendency to form emulsions than CFC–113; and the higher boiling point of *n*-hexane requiring an evaporation time longer than 30 minutes at 85 °C.

*Response:* EPA pointed out the disadvantages of the use of *n*-hexane, including some of the disadvantages above, when EPA Method 1664 was proposed. None of these disadvantages precludes *n*-hexane from being used as the extraction solvent in Method 1664. Indeed, and as pointed out by a commenter, *n*-hexane was used as the extraction solvent for oil and grease prior to the advent of CFC–113 in laboratories that were, undoubtedly, less well equipped to handle toxic and flammable substances. Tests performed to date using *n*-hexane in Method 1664 reveal that none of the disadvantages prevent use of this solvent. EPA agrees that the techniques in Method 1664 and the other methods that the Agency publishes must be performed carefully in order to ensure reliable results.

*D. Solid-phase Extraction (SPE)*

1. Comments Supporting Use of SPE

*Comment:* Many commenters stated that SPE should be a standard procedure in Method 1664. Some suggested that SPE should be the standard procedure and that LLE should be optional.

*Response:* EPA believes that LLE should remain the standard procedure because, except for a change necessitated by the change from CFC–113 to *n*-hexane, the procedures in Method 1664 are virtually identical to procedures in existing methods, the equipment used is the same, and because *n*-hexane with LLE produced results closest to results produced by CFC–113 in EPA's Freon replacement studies.

*Comment:* Method 1664, as proposed, requires a demonstration of equivalency of SPE and other method modifications on each and every discharge. This requirement is a barrier in the way of laboratories that receive samples from different sources and a barrier to use of innovative technologies on a national level. EPA should allow use of SPE and other modifications without this demonstration or should allow nationwide application to the matrices for which applicability has been demonstrated.

*Response:* EPA has allowed use of SPE without a required demonstration of equivalency in the version of Method 1664 approved today. However, EPA has added a note to Method 1664 that it is the discharger's responsibility to assure that results produced are equivalent. Nearly all permits were developed using LLE and CFC–113. Method 1664 allows the use of SPE. However, two things change when SPE is used: the solvent and the extraction technique. EPA is concerned that this double change may cause a discharger to violate a permit limit simply because the results obtained are not equivalent (i.e., SPE may produce different results). Therefore, if there is any doubt about SPE with *n*-hexane producing results significantly different from results produced by LLE with *n*-hexane, dischargers and laboratories should perform a side-by-side test to demonstrate that equivalent results are produced.

2. Comments Expressing Concerns about the Use of SPE

*Comment:* Results produced by SPE and LLE are not equivalent. Comparisons of results produced by LLE, SPE cartridge, and SPE disk in our laboratory showed statistically significant differences on 3 of 4 discharges tested. Further, infrared (IR) spectra and gas chromatography with a flame ionization detector (GC/FID) demonstrate that different material is being extracted by each of the extraction techniques.

*Response:* EPA has seen data that demonstrate differences and other data that demonstrate equivalence in results produced by LLE and SPE, and EPA has decided, based on comments and data received, that SPE should be allowed in the version of Method 1664 approved today. However, EPA has added the note to Method 1664 that, although SPE may be used, it is the discharger/industrial user's responsibility to assure that results produced using SPE are equivalent to results produced using liquid-liquid extraction (LLE).

*E. Grace Period for CFC–113*

*Comments:* Six months is too long to allow existing stocks of CFC–113 to be used up. Use should cease immediately. Use of CFC–113 should be allowed until existing stocks are used up, regardless of how long it takes. Six months is the correct period for stocks of CFC–113 to be used up. The date of the changeover should be the first day of the month to simplify compliance monitoring. NPDES permittees should be given 36 months to determine if they can be compliant with the new method. Use of Method 1664 should not be required for at least one year after the method is approved. Method 413.1 should not be withdrawn for at least two years after the effective date of Method 1664. The additional time should be used to generate data and establish new permit limits as needed.

*Response:* The comments on this issue are diverse but most commenters supported a grace period for switching to Method 1664. Based on comments received, on EPA's desire to allow existing stocks of CFC–113 to be used up, on EPA's desire not to mandate the use of Method 1664 if a CFC-based method is specified in the permit, and on extension of the laboratory exemption for use of CFCs until 2005, approved methods employing CFC–113 remain approved.

*F. Use of Silica-gel Treated, n*-Hexane Extractable Material (SGT–HEM) Procedure

*Comment:* Hexane-extractable material and silica-gel treated, *n*-hexane-extractable material (SGT–HEM) should be better defined.

*Response:* HEM and SGT–HEM are method-defined analytes, i.e., they are defined by the procedure used to measure them, in this case Method 1664.

*Comment:* When will it be necessary to perform the (SGT–HEM) procedure for total petroleum hydrocarbons (TPH)?

*Response:* Monitoring of TPH (now SGT–HEM or ''non-polar material;'' NPM) is presently required in the monitoring and reporting requirements under the Coil Coating point source category at 40 CFR 465.03(c). EPA intends to consider allowing use of Method 1664 through subsequent rulemaking for that category and in other categorical effluent guidelines. In today's rulemaking, EPA is also making the SGT–HEM procedure available to permitting authorities for instances in which only the non-polar material component of oil and grease needs to be monitored. The SGT–HEM procedure allows monitoring of these substances.

US EPA ARCHIVE DOCUMENT

*G. Detection and Quantitation*

*Comment:* Many commenters provided MDL data.

*Response:* The MDLs that the commenters provided and the resulting MLs are consistent with the range of MDLs and MLs that EPA obtained in the Agency's MDL studies. The average (mean) of these MDLs is 2.1 mg/L and the median is 1.4 mg/L. The pooled single-operator MDL, using the 34 MDLs listed above plus the 5 MDLs EPA reported at proposal (61 FR 1736–1737, January 23, 1996), and calculated as the root-mean-square of the standard deviations multiplied by a student's t value of 2.33 for 234 degrees of freedom, is 2.0 mg/L. These MDLs are all equal or close to the value of 1.4 mg/L that EPA proposed and support an ML in the range of 5—10 mg/L. Based on EPA's data and data provided by commenters, EPA has retained the MDL at 1.4 mg/L and the ML at 5 mg/L for both HEM and for SGT–HEM in the version of Method 1664 approved for use in today's final rule.

*Comment:* Several commenters state that estimates of detection and quantitation in Method 1664 are one or more of the following: they cannot be achieved; are scientifically unsound; are neither realistic nor reproducible; are flawed; were developed in an arbitrary and capricious manner; use an inappropriate multiplication factor; are based on spikes into reagent water instead of wastewaters; do not consider effluent characteristics; were developed using analytical standards; are based on a protocol that has never been subjected to peer review and public comment; are not representative of expected performance by qualified laboratories; represent performance of ''expert'' or ''research-grade'' laboratories; are not a statistical predictor of laboratory performance; and were not validated on an interlaboratory basis.

*Response:* EPA disagrees that the MDLs and MLs in Method 1664 were developed inappropriately. EPA has received nearly identical sets of comments from many of the same industry organizations on many recent methods that EPA has proposed for use in its wastewater programs. (See, for examples, responses to comments in the final rules promulgating use of Method 1613 (57 FR 31805, 62 FR 48394) and use of Method 1650 and 1653 (63 FR 18503 )). EPA responds to these comments briefly and collectively here with the same responses provided in those comments and responses. Responses to some of the individual issues raised by commenters are further amplified in other responses.

EPA has used the MDL successfully for estimating the lowest level at which a substance can be detected since 1984. The MDL procedure was subjected to peer review when the original article on the MDL was published in Environmental Science and Technology in 1981 (ES&T 15, 1426–1435). The MDL procedure is subjected to public comment with every MDL that EPA publishes in test methods proposed in the **Federal Register** for use in EPA programs. EPA believes that the MDL procedure is viable and provides an estimate of the lowest concentration of an analyte that can be detected. For Method 1664, EPA did not select the lowest or highest MDL from the five MDL studies that EPA performed prior to proposal; rather, the Agency selected the central value of the five MDLs determined, and provided the rationale for this selection process at proposal (61 FR 1736—1737). This MDL is supported by MDLs in comments received from laboratories that do not necessarily represent ''expert'' or ''research-grade'' laboratories.

With respect to the comment on lack of interlaboratory validation of the MDL and ML, EPA performed an interlaboratory validation of Method 1664 at levels consistent with historical interlaboratory method validation studies performed by EPA, ASTM, AOAC-International, the organizations that publish Standard Methods for the Examination of Water and Wastewater (Standard Methods), and by other organizations that validate methods. EPA and all of these organizations have not historically performed interlaboratory studies to estimate detection and quantitation limits. EPA used data from multiple single laboratory studies instead to support the MDL and ML. Commenters making this comment did not perform interlaboratory detection limit studies to demonstrate that EPA's estimates are flawed.

EPA will continue to examine the issues of detection and quantitation. The Agency initiated a study recently to evaluate these concepts and plans to involve the public on these issues.

*H. Matrix Effects*

*Comment:* Method 1664 produces severe emulsion problems. These emulsions were not formed when using Method 413.1. Breaking these emulsions requires additional handling, increasing the potential for inaccuracy. Bad emulsions can never be completely broken.

*Response:* Section 11.3.5 of Method 1664 contains the following suggestions for overcoming emulsions: ''stirring, filtration through glass wool, use of solvent phase separation paper, centrifugation, use of an ultrasonic bath with ice, addition of NaCl, or other physical methods. Alternatively, solid-phase, continuous, or other extraction techniques may be used to prevent emulsion formation, provided that the requirements in Section 9.1.2 are met.''

*Comment:* EPA must recognize the problems that the high salt content of produced water creates.

*Response:* A small amount of water may be soluble in the *n*-hexane used as the extracting solvent in Method 1664. In turn, a small amount of salt may be dissolved in the hexane/water mixture. However, after extraction, the solution is passed through granular, anhydrous sodium sulfate to remove all traces of water. In turn, this process should remove the residual salt. If not, the extract can be back-extracted with reagent water to remove all traces of residual salt. After back-extraction, the solution can be again filtered through sodium sulfate to remove residual traces of water.

*I. Method Modifications*

*Comment:* We endorse the concept of performance-based methods to allow for advances in technology and reductions in the cost of analyses and encourage EPA to continue to move in this direction.

*Response:* In response to this and similar requests, EPA proposed an implementation of a performance-based measurement system (PBMS) in the Streamlining Initiative on March 28, 1997 (62 FR 14976) and solicited comment on an alternative PBMS approach on October 6, 1997 (62 FR 52098). The Streamlining Initiative allows modification of a reference method so long as equivalent or superior performance can be demonstrated. The alternative PBMS approach allows modification of a method or use of any other method based on performance demonstrated equal or superior to a reference method (as with Streamlining) or to a set of data quality objectives (DQOs). The alternative PBMS approach does not allow modification of methods for method-defined analytes, such as oil and grease, because the analyte being measured is defined by the method used. EPA expects to make a final determination on PBMS for method-defined analytes in the future, as the Agency gains experience in dealing with any potential issues, and as comments from stakeholders on these and other PBMS approaches are received.

*Comment:* Please clarify the ''specific discharge'' as it relates to ''the

US EPA ARCHIVE DOCUMENT

discharger must demonstrate that the modified method produces results equivalent to those produced by Method 1664 for each specific discharge.''

*Response:* Specific discharge is equivalent to ''matrix type'' defined in the regulatory language proposed in EPA's Streamlining Initiative (62 FR 14994, March 28, 1997) and means a sample medium with common characteristics across a given industrial subcategory. Examples include: C-stage effluents from chlorine bleach mills in the Pulp, Paper, and Paperboard industrial category; effluent from the continuous casting subcategory of the Iron and Steel industrial category; publicly owned treatment work (POTW) sludge; and in-process streams in the Atlantic and Gulf Coast Hand-shucked Oyster Processing subcategory. For further explanation of this definition, please see the proposed Streamlining Initiative.

*Comment:* Declaring that performance-based modifications can be made perpetuates the incorrect notion that empirically determined analytes are not affected when the practice and manner of determining them changes. When a protocol defines an analyte, deviation from that protocol should not be permitted.

*Response:* EPA agrees with this comment, but only in part. The commenter presumably would not argue that the result of the analysis will be affected by the size of the funnel that contains the sodium sulfate used for removal of residual water. In allowing modification of Method 1664, the Agency identified and distinguished changes that would not adversely affect method performance (and analyte measurement) from changes that would. As a result, laboratories may modify extraction and concentration procedures, but not allow changes to the determinative technique (gravimetry), provided that equivalent or superior performance of the modification is demonstrated on a reference matrix (reagent water) and on the discharge to which the modification will be applied. EPA believes that this middle ground is the best that can be done to allow modifications and protect the reliability of the data produced with a modification.

*J. Matrix Spike/Matrix Spike Duplicate (MS/MSD)*

*Comment:* The relative percent difference criteria for the MS/MSD are too stringent and do not account for natural variations in grab samples.

*Response:* EPA changed the requirement for an MSD to a suggestion but believes that the MS/MSD will work with flowing streams. In the Phase I and Phase II studies that EPA performed in support of development of EPA Method 1664, flowing streams were split using the procedures given in the note in Section 8.2 of EPA Method 1664 and provided reliable replicates for testing. For the Phase II study, the discharge streams sampled were adjusted to provide background concentrations of oil and grease. No difficulty was encountered recovering spikes into these samples or achieving precise results with replicates.

*Comment:* The requirement for an MS/MSD at a frequency of 10 percent and per sample batch is excessive and unnecessarily burdensome.

*Response:* EPA agrees that this requirement is unnecessarily restrictive and has reduced the frequency of the MS to 5 percent and has changed the requirement for the MSD to a suggestion.

*K. Precision and Recovery*

*Comment:* The precision and recovery criteria are unrealistic and will not be achievable by most laboratories.

*Response:* EPA believes that the difficulties in achieving the precision and recovery criteria in EPA Method 1664 are attributable to precipitation of hexadecane and stearic acid from the standard solution and to failure of laboratories to adequately rinse all traces of the standard from glassware. EPA has modified the version of EPA Method 1664 being approved today to halve the concentration of hexadecane and stearic acid in the standard solution and require spiking twice as much. EPA also has noted that the sample container and other surfaces that the sample contacts must be carefully rinsed with solvent to effect quantitative transfer of oil and grease and NPM from the sample to the extract.

*L. Differences in Results Produced by n-Hexane and CFC–113*

*Comment:* Changing to *n*-hexane may cause dischargers to exceed permit limitations because the new method may result in higher oil and grease values. EPA should provide guidance to permit writers and enforcement staff for dealing with this positive bias.

*Response:* EPA believes that the possibility that the change in solvent will result in non-compliance is minimal based on the results from the Freon Replacement Studies conducted prior to proposal. If a discharger believes that the change to *n*-hexane will cause false readings of noncompliance, the discharger should discuss the situation with the permitting authority. EPA cautions that to demonstrate that the noncompliance is the result of the change to *n*-hexane alone, comparative data must be obtained using CFC–113 and *n*-hexane on a sufficient number of real-world samples and the difference must be statistically significant. For guidance in this demonstration, the commenter is referred to Section III.D. of this preamble and the statistical tests for significance in reports for EPA's Phase I and Phase II Freon Replacement studies that were included in the Docket at proposal.

*Comment:* EPA's proposal to replace previously approved methods with EPA Method 1664 is arbitrary and capricious and will render obsolete all effluent limitation guidelines and permit limitations for oil and grease that were based on the Freon method.

*Response:* First, EPA is not withdrawing the previously approved methods in today's rulemaking. Second, as discussed earlier, EPA believes that the use of *n*-hexane will generally not affect the oil and grease results significantly and, therefore, the possibility that the change in solvent will result in non-compliance is minimal. Third, in studies comparing different extraction solvents for measurement of oil and grease, and in the proposal of Method 1664, EPA acknowledged and again acknowledges that no two extraction solvents will produce exactly the same results for a method-defined analyte such as oil and grease. By providing for recalculation of compliance targets based on side-by-side data, EPA believes that the Agency has taken reasonable steps to minimize the impact of using Method 1664 rather than the Freon method in those cases when the difference in results may cause a non-compliance with a permit limitation.

*Comment:* Two commenters stated that samples collected and analyzed under the previously approved methods are used to determine a discharger's compliance with local and Federal categorical limits and that it may be appropriate for EPA to re-evaluate all of these limits.

*Response:* There are more than 600 industrial subcategories and nearly all contain a limit for oil and grease. Extensive time and expense would be required for re-evaluation of all limits in all of these subcategories. EPA conducted side-by-side evaluations on 39 facilities in 24 industrial categories in the Phase I Freon Replacement Study and on 25 facilities in 16 industrial categories in the Phase II study. Based on the results from these studies, EPA does not believe that a re-evaluation of the categorical limits is neccesary at this

US EPA ARCHIVE DOCUMENT

time and supports the use of Method 1664 instead of the Freon method to determine compliance with the categorical limits. However, EPA periodically re-evaluates categorical effluent limitations and plans to use Method 1664 to support this effort. Until each categorical standard is re-evaluated, EPA has decided to allowed the continued use of the CFC–113 methods. However, because compliance targets can be adjusted on a case-by-case basis, EPA encourages the replacement of CFC–113 with *n*-hexane consistent with EPA's efforts to reduce dependency on the use of chlorofluorocarbons.

*M. Method Validation and QC Acceptance Criteria*

*Comment:* EPA Method 1664 was not validated properly because inappropriate sample concentrations of 57 and 170 mg/L were used in the Twin Cities Round-robin study (TCRR).

*Response:* EPA disagrees that EPA Method 1664 was validated improperly. Laboratories that participated in the TCRR study validated the Method at concentrations of 40 mg/L for HEM and 20 mg/L for NPM in the initial precision and recovery (IPR) and ongoing precision and recovery (OPR) tests, and at concentrations of 57 and 170 mg/L for ''real-world'' samples from a petroleum and non-petroleum source, respectively. It is customary to validate analytical methods at concentrations in the middle of the concentration range to avoid attempting to compare results for which HEM is not detected and to allow lowered recoveries to be measured reliably, should lowered recoveries occur. For additional information on this issue, see the response to comments on the detection/quantitation issue.

*N. Quality Control*

*Comment:* The QC specified in Section 9 is excessive, especially for every discharge point.

*Response:* As stated elsewhere in these comment responses, the frequency requirement for an MSD has been changed to a suggestion and the frequency of the MS and OPR have been reduced to a minimum of 5 percent. EPA does not believe that a requirement to assess the precision and recovery on every 20th sample is excessive.

*O. Sample Collection and Preservation*

*Comment:* EPA should add a compositing procedure to EPA Method 1664. Samples can be composited in the laboratory by collecting individual 250-mL samples over the course of a day, pouring each 250-mL sample into the separatory funnel, rinsing each of the four bottles (and caps) sequentially with 30 mL of *n*-hexane, and using the 30 mL of *n*-hexane for the extraction.

*Response:* EPA has added the above compositing procedure to EPA Method 1664.

*Comment:* Change the temperature requirement to 4 ± 2 °C with the note that a lower storage temperature may be used so long as the sample is not frozen.

*Response:* EPA chose a temperature range of 0–4 °C to be consistent with holding time study data and to allow storage at 0 °C.

*P. Miscellaneous Issues*

*Comment:* The frequency of verification of balance calibration is excessive. One commenter suggests verification before and after every 20 samples. Another commenter suggests verification before and after daily measurements. A third commenter suggests that calibration verification is unnecessary. A fourth commenter suggests monthly verification.

*Response:* Calibration is verified prior to the first batch, between batches, and after the last batch. EPA does not believe that this is excessive. (An analytical batch is between 4 and 23 measurements.)

*Comment:* Method ruggedness has not been established, method validity has not been confirmed on a wide range of sample matrices, and the method is still empirical. The method needs further review and validation on real-world samples.

*Response:* Based on EPA's Phase II and method validation studies, and data supplied by commenters, EPA believes that Method 1664 has been adequately validated and is sufficiently rugged for its intended use. Method 1664 will always be empirical because oil and grease and NPM are method-defined analytes.

*Comment:* The flask used for collection of distillate should be referred to as a ''distillate collection flask'' rather than a ''waste collection flask'' because the word ''waste'' can imply hazardous waste.

*Response:* EPA agrees and has changed ''waste collection flask'' to ''distillate collection flask''.

*Comment:* Method 1664 requires that a smaller sample volume should be extracted when a sample contains >1000 mg/L of oil and grease. Should the smaller volume be diluted to one liter for extraction, or should the smaller volume be extracted without dilution?

*Response:* The smaller volume should be diluted to one liter so that changes to the Method are minimized. EPA has clarified this dilution in Method 1664.

*Comment:* The higher boiling point of n-hexane will cause loss of the more volatile constituents of oil and grease and therefore produce greater variability in results.

*Response:* The average amount of oil and grease determined when using *n*-hexane vs CFC–113 (96%), as detailed in other responses to comments, may be attributable to loss of some volatile constituents.

*Comment:* What is to be the fate of Method 418.1? Method 418.1 and SW–846 Method 9071 should not be used if continued use of Method 413.1 is disallowed.

*Response:* Method 418.1 is a CFC–113/infrared (IR) method for determination of oil and grease. Although listed in Methods for Chemical Analysis of Water and Wastes (EPA 600/4–79–020; NTIS PB84–128677), Method 418.1 has not been approved for use at 40 CFR Part 136. EPA plans a collaborative study with Canada for development of an IR method that does not use CFC–113 and may propose an IR method depending on the outcome of that study.

A solvent change from CFC–113 to n-hexane is being made in SW–846 Method 9071. Method 1664 replaces Method 9070 as the approved SW–846 method for determination of oil and grease in water, as detailed in section III.B of this preamble.

*Comment:* EPA should include a thorough discussion of oil and grease as a ''method-defined analyte'' in the final rule so that all stakeholders clearly understand the results generated by Method 1664.

*Response:* EPA explained in section V of the proposal (61 FR 1737, January 23, 1996) that determination of oil and grease is dependent on how the measurement is made and cited examples of biochemical oxygen demand and total suspended solids as other method-defined analytes. Method-defined analytes are those analytes that are defined by the procedure used to measure them. For oil and grease, the nature and the amount of the substances extracted from complex wastewater discharges and then measured is determined by the extracting solvent and technique.

*Comment:* Many commenters suggested that Section 11.4.4 be changed to require solvent evaporation and desiccation to constant weight. One commenter suggested specific wording for this change. Another commenter suggested a desiccation time of 24 hours.

*Response:* Section 11.4.4 has been changed to the specific wording suggested by the first commenter but

US EPA ARCHIVE DOCUMENT

has not been modified to include a 24-hour time requirement so that constant weight achieved in a shorter time can be considered valid.

*Comment:* It should be EPA's responsibility to certify each laboratory and not the responsibility of each and every company that uses the laboratory.

*Response:* EPA does not certify laboratories under the Clean Water Act and RCRA analytical programs. The States have this responsibility. EPA is working with the States and other interested parties under the auspices of the National Environmental Laboratory Accreditation Program (NELAP) to accredit laboratory auditing organizations.

## VII. Regulatory Requirements

### *A. Executive Order 12866*

Under Executive Order 12866, (58 FR 51735 (October 4, 1993)) the Agency must determine whether a regulatory action is ''significant'' and therefore subject to OMB review and the requirements of the Executive Order. The Order defines ''significant regulatory action'' as one that is likely to result in a rule that may: (1) Have an annual effect on the economy of $100 million or more or adversely affect in a material way the economy, a sector of the economy, productivity, competition, jobs, the environment, public health or safety, or State, local, or tribal governments or communities; (2) create a serious inconsistency or otherwise interfere with an action taken or planned by another agency; (3) materially alter the budgetary impact of entitlements, grants, user fees, or loan programs or the rights and obligations of recipients thereof; or (4) raise novel legal or policy issues arising out of legal mandates, the President's priorities, or the principles set forth in the Executive Order.''

It has been determined that this rule is not a ''significant regulatory action'' under the terms of Executive Order 12866 and is therefore not subject to OMB review.

### *B. Unfunded Mandates Reform Act*

Title II of the Unfunded Mandates Reform Act of 1995 (UMRA), Pub. L. 104–4, establishes requirements for Federal agencies to assess the effects of their regulatory actions on State, local, and tribal governments and the private sector. Under section 202 of the UMRA, EPA generally must prepare a written statement, including a cost-benefit analysis, for proposed and final rules with ''Federal mandates'' that may result in expenditures to State, local, and tribal governments, in the aggregate, or to the private sector, of $100 million or more in any one year. Before promulgating an EPA rule for which a written statement is needed, section 205 of the UMRA generally requires EPA to identify and consider a reasonable number of regulatory alternatives and adopt the least costly, most cost-effective or least burdensome alternative that achieves the objectives of the rule. The provisions of section 205 do not apply when they are inconsistent with applicable law. Moreover, section 205 allows EPA to adopt an alternative other than the least costly, most cost-effective or least burdensome alternative if the Administrator publishes with the final rule an explanation why that alternative was not adopted. Before EPA establishes any regulatory requirements that may significantly or uniquely affect small governments, including tribal governments, it must have developed under section 203 of UMRA a small government agency plan. The plan must provide for notifying potentially affected small governments, enabling officials of affected small governments to have meaningful and timely input in the development of EPA regulatory proposals with significant Federal intergovernmental mandates, and informing, educating, and advising small governments on compliance with the regulatory requirements.

Today's final rule contains no Federal mandates (under the regulatory provisions of Title II of UMRA) for State, local, or tribal governments or the private sector. EPA has determined that this rule contains no regulatory requirements that might significantly or uniquely affect small governments. This rule would impose no enforceable duty on any State, local or Tribal governments or the private sector, nor would it significantly or uniquely affect them. This rule makes available an additional testing procedure which would merely standardize the procedures when testing is otherwise required by a regulatory agency. Therefore, today's rule is not subject to the requirements of sections 202, 203 and 205 of UMRA.

### *C. Regulatory Flexibility Act*

Under the Regulatory Flexibility Act (RFA), 5 U.S.C. 601 *et seq.*, as amended by the Small Business Regulatory Enforcement Fairness Act (SBREFA), EPA generally is required to conduct a regulatory flexibility analysis describing the impact of the regulatory action on small entities as part of rulemaking. However, under section 605(b) of the RFA, if EPA certifies that the rule will not have a significant economic impact on a substantial number of small entities, EPA is not required to prepare a regulatory flexibility analysis. Pursuant to section 605(b) of the Regulatory Flexibility Act, 5 U.S.C. 605(b), the Administrator certifies that this rule will not have a significant economic impact on a substantial number of small entities. This regulation merely approves an additional testing procedure for the measurement of oil and grease and non-polar material but does not require its use. The new approved method uses n-hexane which has a much lower cost than Freon-113, which is used in the currently approved methods.

### *D. Paperwork Reduction Act*

This rule contains no information collection requirements. Therefore, no information collection request has been submitted to the Office of Management and Budget (OMB) for review and approval under the Paperwork Reduction Act of 1980, 44 U.S.C. 3501 *et seq.*

### *E. Submission to Congress and the General Accounting Office*

The Congressional Review Act, 5 U.S.C. section 801 *et seq.*, as added by the Small Business Regulatory Enforcement Fairness Act of 1996, generally provides that before a rule may take effect, the agency promulgating the rule must submit a rule report, which includes a copy of the rule, to each House of the Congress and to the Comptroller General of the United States. EPA will submit a report containing this rule and other required information to the U.S. Senate, the U.S. House of Representatives and the Comptroller General of the United States prior to publication of the rule in the **Federal Register**. A major rule cannot take effect until 60 days after it is published in the **Federal Register**. This rule is not a ''major rule'' as defined by 5 U.S.C. section 804(2). This rule will take effect on the effective date shown at the beginning of this preamble.

### *F. National Technology Transfer and Advancement Act*

Under Section 12(d) of the National Technology Transfer and Advancement Act (NTTAA), the Agency is required to use voluntary consensus standards in its regulatory activities unless to do so would be inconsistent with applicable law or otherwise impractical. Voluntary consensus standards are technical standards (e.g., materials specifications, test methods, sampling procedures, business practices, etc.) that are developed or adopted by voluntary consensus standards bodies. Where

US EPA ARCHIVE DOCUMENT

available and potentially applicable consensus standards are not used by EPA, the Act requires the Agency to provide Congress, through the Office of Management and Budget (OMB), an explanation of the reasons for not using such standards. EPA's search of the technical literature has revealed that there are no consensus methods for determination of hexane extractable material (HEM) and silica gel treated hexane extractable material (SGT–HEM), although the American Society of Testing and Materials (ASTM) is in the process of developing an analytical method for the determination of HEM. If ASTM or another voluntary consensus standard body approves such a method and EPA believes that the method is suitable for compliance monitoring and other purposes, EPA will promulgate the method in a subsequent rule.

*G. Executive Order 13045*

The Executive Order, ''Protection of Children from Environmental Health Risks and Safety Risks,'' (62 FR 19885), applies to any rule initiated after April 21, 1997, or proposed after April 21, 1998, that: (1) is determined to be ''economically significant'' as defined under E.O. 12866, and (2) concerns an environmental health or safety risk that EPA has reason to believe may have a disproportionate effect on children. If the regulatory action meets both criteria, the Agency must evaluate the environmental health or safety effects of the planned rule on children, and explain why the planned regulation is preferable to other potentially effective and reasonably feasible alternatives considered by the Agency.

This regulation is not subject to the Executive Order because EPA published a notice of proposed rulemaking before April 21, 1998 and further because this is not an economically significant rule as defined under E.O. 12866. However, EPA's policy since November 1, 1995, has been to consistently and explicitly consider risks to infants and children in all risk assessments generated during its decision making process including the setting of standards to protect public health and the environment.

EPA's Office of Water has historically considered risks to sensitive populations (including fetuses, infants, and children) in establishing risk assessments for setting health or safety standards. This regulation does not involve the development of a standard to mitigate environmental health or safety risks. This regulation instead approves an additional analytical method for compliance monitoring. However, because the extraction solvent used in Method 1664, *n*-hexane, has been associated with neurotoxic effects, EPA investigated the available health information to determine whether the fetus may be adversely affected as a result of pregnant women being exposed to *n*-hexane in the laboratory environment. Based on animal studies, the available information on developmental effects does not indicate any potential risks to the fetus due to exposure to *n*-hexane.

*H. Executive Order 12875*

Under Executive Order 12875, ''Enhancing the Intergovernmental Partnership,'' EPA may not issue a regulation that is not required by statute and that creates a mandate upon a State, local or tribal government, unless the Federal government provides the funds necessary to pay the direct compliance costs incurred by those governments, or EPA consults with those governments. If EPA complies by consulting, E.O. 12875 requires EPA to provide to the Office of Management and Budget a description of the extent of EPA's prior consultation with representatives of affected State, local and tribal governments, the nature of their concerns, any written communications from the governments, and a statement supporting the need to issue the regulation. In addition, Executive Order 12875 requires EPA to develop an effective process permitting elected officials and other representatives of State, local and tribal governments ''to provide meaningful and timely input in the development of regulatory proposals containing significant unfunded mandates.''

Today's rule does not create a mandate on State, local or tribal governments. The rule does not impose any enforceable duties on these entities. This rule makes available an additional testing procedure that would merely standardize the procedures when testing is otherwise required by a regulatory agency. Accordingly, the requirements of section 1(a) of Executive Order 12875 do not apply to this rule.

*I. Executive Order 13084*

Under Executive Order 13084, ''Consultation and Coordination with Indian Tribal Governments,'' EPA may not issue a regulation that is not required by statute, that significantly or uniquely affects the communities of Indian tribal governments, and that imposes substantial direct compliance costs on those communities, unless the Federal government provides the funds necessary to pay the direct compliance costs incurred by the tribal governments, or EPA consults with those governments. If EPA complies by consulting, Executive Order 13084 requires EPA to provide to the Office of Management and Budget, in a separately identified section of the preamble to the rule, a description of the extent of EPA's prior consultation with representatives of affected tribal governments, a summary of the nature of their concerns, and a statement supporting the need to issue the regulation. In addition, E.O. 13084 requires EPA to develop an effective process permitting elected and other representatives of Indian tribal governments ''to provide meaningful and timely input in the development of regulatory policies on matters that significantly or uniquely affect their communities.''

Today's rule does not significantly or uniquely affect the communities of Indian tribal governments. Further, this rule does not impose substantial direct compliance costs on tribal governments. This rule makes available an additional testing procedure which would merely standardize the procedures when testing is otherwise required by a regulatory agency. Accordingly, the requirements of section 3(b) of Executive Order 13084 do not apply to this rule.

**List of Subjects**

*40 CFR Part 136*

Environmental protection, Analytical methods, Incorporation by reference, Monitoring, Reporting and recordkeeping requirements, Waste treatment and disposal, Water pollution control.

*40 CFR Part 260*

Environmental protection, Administrative practice and procedure, Analytical methods, Confidential business information, Hazardous waste, Incorporation by reference, Reporting and recordkeeping requirements.

Dated: May 7, 1999.

**Carol M. Browner,**

*Administrator.*

For the reasons set out in the preamble, title 40, chapter I of the Code of Federal Regulations, is amended as follows:

## PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

1. The authority for part 136 continues to read as follows:

**Authority:** Secs. 301, 304(h), 307, and 501(a) Pub. L. 95–217, 91 Stat. 1566, *et seq.* (33 U.S.C. 1251, *et seq.*) (the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977.)

2. In § 136.3, paragraph (a), Table IB is amended by revising entry 41 to read as follows:

US EPA ARCHIVE DOCUMENT

**§ 136.3 Identification of test procedures.**

(a) * * *

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TABLE IB—LIST OF APPROVED INORGANIC TEST PROCEDURES

| Parameter, units and method | Reference (method number or page) | | | | |
|---|---|---|---|---|---|
| | EPA [1, 35] | STD methods 18th ed. | ASTM | USGS[2] | Other |
| * * * | * | * | * | | * |
| 41. Oil and grease—Total recoverable, mg/L: Gravimetric (extraction). | 413.1 ..... | 5520 B[38] | | | |
| Oil and grease and non-polar material, mg/L: Hexane extractable material (HEM): *n*-Hexane extraction and gravimetry[42]. | 1664, Rev. A. | | | | |
| Silica gel treated HEM (SGT–HEM): Silica gel treatment and gravimetry[42]. | 1664, Rev. A. | | | | |
| * * * | * | * | * | | * |

**Table 1B Notes:**

[1] ''Methods for Chemical Analysis of Water and Wastes'', Environmental Protection Agency, Environmental Monitoring Systems Laboratory-Cincinnati (EMSL–Ci), EPA–600/4–79–020, Revised March 1983 and 1979 where applicable.

[2] Fishman, M.J., et al, ''Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments,'' U.S. Department of the Interior, Techniques of Water—Resource Investigations of the U.S. Geological Survey, Denver, CO, Revised 1989, unless otherwise stated.

* * * * * * *

[35] Precision and recovery statements for the atomic absorption direct aspiration and graphite furnace methods, and for the spectrophotometric SDDC method for arsenic are provided in Appendix D of the part titled, ''Precision and Recovery Statements for Methods for Measuring Metals.''

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[38] Only the trichlorofluoromethane extraction solvent is approved.

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[42] Method 1664, Revision A ''n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractablke Material (SGT–HEM; Non-polar Material) by Extraction and Gravimetry'' EPA–821–R–98–002, February 1999. Available at NTIS, PB–121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, Virginia 22161.

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## PART 260—HAZARDOUS WASTE MANAGEMENT SYSTEM: GENERAL

1. The authority for part 260 continues to read as follows:

**Authority:** 42 U.S.C. 6905, 6912(a), 6921–6927, 6930, 6934, 6935, 6937, 6939, and 6974.

### Subpart B—Definitions

2. Section 260.11 is amended by revising paragraph (a)(11) and by adding paragraph (a)(16) to read as follows:

**§ 260.11 References.**

* * * * *

(a) * * *

(11) ''Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,'' EPA Publication SW–846 [Third Edition (November 1986), as amended by Updates I (dated July 1992), II (dated September 1994), IIA (dated August 1993), IIB (dated January 1995), III (dated December 1996) and IIIA (dated April 1998)]. The Third Edition of SW–846 and Updates I, II, IIA, IIB, and III (document number 955–001–00000–1) are available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, (202) 512–1800. Update IIIA is available through EPA's Methods Information Communication Exchange (MICE) Service. MICE can be contacted by phone at (703) 821–4690. Update IIIA can also be obtained by contacting the U.S. Environmental Protection Agency, Office of Solid Waste (5307W), OSW Methods Team, 401 M Street, SW, Washington, DC, 20460. Copies of the Third Edition and all of its updates are also available from the National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161, (703) 605–6000 or (800) 553–6847. Copies may be inspected at the Library, U.S. Environmental Protection Agency, 401 M Street, SW, Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street, NW, suite 700, Washington, DC.

* * * * *

(16) Method 1664, Revision A, *n*-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated *n*-Hexane Extractable Material (SGT–HEM; Non-polar Material) by Extraction and Gravimetry. Available at NTIS, PB99–121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, Virginia 22161.

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## FEDERAL COMMUNICATIONS COMMISSION

### 47 CFR Part 73

### Radio Broadcast Services

*CFR Correction*

In Title 47 of the Code of Federal Regulations, parts 70 to 79, revised as of Oct. 1, 1998, page 193, § 73.624 (c) introductory text, last sentence, is corrected by adding ''not'' after ''shall'' and before ''be''.

[FR Doc. 99–55519 Filed 5–13–99; 8:45 am]

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