



A Guide for the Assessment of EPA Method 1664A

N-Hexane Extractable Material (HEM; Oil and Grease) by Extraction and Gravimetry

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Aid To
Laboratories
For Use In
Internal Audits

Guide For
NELAC
Assessors

Provide A
Template To
Document
Assessment
Findings

Beta Test Of
The Format
For Use In
Preparing
Future Guides

Purpose and Approach

- Built on NELAC Chapter 5 Appendix D-1 Checklist for Chemical Testing
- Omits unrelated topics, e.g. surrogates
- Eliminates redundancies
- Addresses method-specific issues not covered in D-1 checklist e.g. reporting
- Provides method-specific technical criteria
- Provides method citations for all items



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Summary of Special Requirements For Compliance-Related Results

- Mandatory QC criteria if results are used in compliance reporting (Table1)
- Limits on method modifications (Guide, Section 4)
- Explicit requirements for method modifications (9.2.3)
- Emphasis on matrix types in QC steps (9.3)
- Special requirements for sample containers (6.1.2)
- Control of sample preservation confirmation (11.2)



Acceptance Criteria for Performance Tests – Table 1

Table 1. Acceptance Criterion for Performance Tests

Acceptance Criterion	Section	Limit (%)
<u>Initial precision and recovery</u>	9.2.2	
HEM Precision (s)	9.2.2.2	11
HEM Recovery (X)	9.2.2.2	83 – 101
SGT-HEM Precision (s)	9.2.2.2	28
SGT-HEM Recovery (X)	9.2.2.2	83 – 116
<u>Matrix spike/matrix spike duplicate</u>	9.3	
HEM Recovery	9.3.4	78 – 114
HEM RPD	9.3.5	18
SGT-HEM Recovery	9.3.4	64 – 132
SGT-HEM RPD	9.3.5	34
<u>Ongoing precision and recovery</u>	9.6	
HEM Recovery	9.6	78 – 114
SGT-HEM Recovery	9.6	64 – 132



QC Criteria For Compliance Reporting

- All QC and method modifications must be evaluated on each industrial waste subcategory (IWS)
- Must not report a result associated with:
 - failed method blank
 - failed matrix spike
 - failed ongoing precision & recovery sample (OPR)
- Laboratory may provide results to the client if flagged with statement saying results can't be used for compliance reporting.



Limits On Method Modifications

Simple change

e.g. change of a beaker to an Erlenmeyer flask or change of a boiling flask to a Kuderna-Danish is allowed, after a demonstration of equivalency

Changes not Allowed

Extraction Solvent
Standards
Determinative Step

Solid Phase Extraction Allowed

see Guide, Appendix C for details



Matrix-Related Issues

- Each IWS must be evaluated separately (see 40 CFR 403 – 500)
- Laboratory must track and perform an MS at a frequency of 1 in 20 for each IWS (9.3)
- Method modifications, including SPE, must be evaluated on each IWS (9.1.2; 9.2.3; Guide Section 3)



Transportation Equipment Cleaning Subcategories

- Tank Trucks and Intermodal Tank Containers Transporting Chemical and Petroleum Cargos
- Rail Tank Cars Transporting Chemical and Petroleum Cargos
- Tank Barges and Ocean/Sea Tankers Transporting Chemical and Petroleum Cargos
- Tanks Transporting Food Grade Cargos (direct discharging facilities only)



Suggest Reading

- Provides many details and rational for technical elements of the method for application in the CWA & RCRA programs <http://www.epa.gov/fedrgstr/EPA-WATER/1999/May/Day-14/w12163.htm>
- Technical Background Documents available from: National Service Center for Environmental Publications (NSCEP)
<http://nepis.epa.gov/EPA/html/Pubs/pubtitleOW.htm>
 - Phase I Freon Replacement Study (EPA-821-R-93-011)
 - Phase II Freon Replacement Study (EPA-820-R-95-003)



Summary of Major Issues for Assessment of Method 1664A

- Definition of sample matrix is tightly controlled by regulation
- Method 1664A introduces mandatory QC requirements not normally found in EPA methods
 - Method blank, matrix spikes, and on going precision and recovery samples
- Method procedure is mandatory and any change must be rigorously documented using prescribed procedures
- Reporting of results associated with failed QC is forbidden for compliance purposes –will impact clients and laboratories who provide reporting support for clients

