METHOD 8410

GAS CHROMATOGRAPHY/FOURIER TRANSFORM INFRARED SPECTROMETRY FOR SEMIVOLATILE ORGANICS: CAPILLARY COLUMN

1.0 SCOPE AND APPLICATION

1.1 This method covers the automated identification, or compound class assignment of unidentifiable compounds, of solvent-extractable semivolatile organic compounds which are amenable to analysis by gas chromatograph/Fourier transform-infrared spectrometer (GC/FT-IR). GC/FT-IR can be a useful complement to gas chromatograph/mass spectrometer (GC/MS) analysis (Method 8270). It is particularly well suited for the identification of specific isomers that are not differentiated using GC/MS. Compound class assignments are made using infrared (IR) group absorption frequencies. The presence of an IR band in the appropriate group frequency region may be taken as evidence of the possible presence of a particular compound class, while its absence may be construed as evidence that the compound class in question is not present. This evidence will be further strengthened by the presence of confirmatory group frequency bands. Identification limits of the following compounds have been demonstrated by this method.

Compound Name	CAS No.[a]
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo(a)anthracene	56-55-3
Benzo(a)pyrene	50-32-8
Benzoic acid	65-85-0
Bis(2-chloroethoxy)methane	111-91-1
Bis(2-chloroethyl)ether	111-44-4
Bis(2-chloro-1-methylethyl)ether[b]	108-60-1
Bis(2-ethylhexyl)phthalate	117-81-7
4-Bromophenyl phenyl ether	101-55-3
Butyl benzyl phthalate	85-68-7
4-Chloroaniline	106-47-8
4-Chloro-3-methylphenol	59-50-7
2-Chloronaphthalene	91-58-7
2-Chlorophenol	95-57-8
4-Chlorophenol	106-48-9
4-Chlorophenyl phenyl ether	7005-72-3
Chrysene	218-01-9
Dibenzofuran	132-64-9
Di-n-butyl phthalate	84-74-2
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
2,4-Dichlorophenol	120-83-2
Diethyl phthalate	84-66-2
Dimethyl phthalate	131-11-3

Compound Name	CAS No.[a]
4,6-Dinitro-2-methylphenol	534-52-1
2,4-Dinitrophenol	51-28-5
2,4-Dinitrotoluene	121-14-2
2,6-Dinitrotoluene	606-20-2
Di-n-octyl phthalate	117-84-0
Di-n-propyl phthalate	131-16-8
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
1,3-Hexachlorobutadiene	87-68-3
Hexachlorocyclopentadiene	77-47-4
Hexachloroethane	67-72-1
Isophorone	78-59-1
2-Methylnaphthalene	91-57-6
2-Methylphenol	95-48-7
4-Methylphenol	106-44-5
Naphthalene	91-20-3
2-Nitroaniline	88-74-4
3-Nitroaniline	99-09-2
4-Nitroaniline	100-01-6
Nitrobenzene	98-95-3
2-Nitrophenol	88-75-5
4-Nitrophenol	100-02-7
N-Nitrosodimethylamine	62-75-9
N-Nitrosodiphenylamine	86-30-9
N-Nitroso-di-n-propylamine	621-64-7
Pentachlorophenol	87-86-5
Phenanthrene	85-01-8
Phenol	108-95-2
Pyrene	129-00-0
1,2,4-Trichlorobenzene	120-82-1
2,4,5-Trichlorophenol	95-95-4
2,4,6-Trichlorophenol	88-06-2

[a] Chemical Abstract Services (CAS) Registry Number.
[b] Chemical name was changed by the Integrated Risk Information System (IRIS) on November 30, 2007 from Bis(2-chloroisopropyl)ether to Bis(2-chloro-1-methylethyl)ether (common name). This compound is also known as 2,2'-oxybis(1-chloropropane) (CAS index name). See the link at http://www.epa.gov/iris/subst/0407.htm, Section VII for the "Revision History" and Section VIII, for "Synonyms" of this chemical.

1.2 This method is applicable to the determination of most extractable, semivolatile-organic compounds in wastewater, soils and sediments, and solid wastes. Benzidine can be subject to losses during solvent concentration and analysis by gas chromatograph (GC); α-BHC, β-BHC, Endosulfan I and II, and Endrin are subject to decomposition under the alkaline conditions of the extraction step; Endrin is subject to decomposition during GC analysis; and Hexachlorocyclopentadiene and N-Nitrosodiphenylamine may decompose during extraction and

GC analysis. Other extraction and/or instrumentation procedures should be considered for unstable analytes.

1.3 The identification limit of this method may depend strongly upon the level and type of semivolatile extractants amenable to GC analysis. The values listed in Tables 1 and 2 represent the minimum quantities of semivolatile organic compounds which have been identified by the specified GC/FT-IR system, using this method and under routine environmental analysis conditions. Capillary GC/FT-IR wastewater identification limits of 25 µg/L may be achieved for weak IR absorbers with this method, while the corresponding identification limit for strong IR absorbers is 2 µg/L. Identification limits for other sample matrices can be calculated from the wastewater values after choice of the proper sample workup procedure (see Sec. 11.1).

2.0 SUMMARY OF METHOD

Prior to using this method, the samples should be prepared for chromatography using the appropriate sample preparation and cleanup methods. This method describes chromatographic conditions that will allow for the separation of the compounds in the extract and uses FT-IR for detection and quantitation of the target analytes.

3.0 DEFINITIONS

Refer to Chapter One, the individual determinative methods, and the manufacturer's instructions for definitions that may be relevant.

4.0 INTERFERENCES

4.1 Glassware and other sample processing hardware must be thoroughly cleaned to prevent contamination and misinterpretation. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. Specific selection of reagents or purification of solvents by distillation in all-glass systems may be required.

4.2 Matrix interference will vary considerably from source to source, depending upon the diversity of the residual waste being sampled. While general cleanup techniques are provided as part of this method, unique samples may require additional cleanup to isolate the analytes of interest from interferences in order to achieve maximum sensitivity.

4.3 4-Chlorophenol and 2-nitrophenol are subject to interference from coeluting compounds.

4.4 Clean all glassware as soon as possible after use by rinsing with the last solvent used. Glassware should be sealed/stored in a clean environment immediately after drying to prevent any accumulation of dust or other contaminants.

5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling

of the chemicals and instrumentation included in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6.0 EQUIPMENT AND SUPPLIES

6.1 GC/FT-IR Spectrometric Equipment

6.1.1 FT- IR spectrometer – A spectrometer capable of collecting at least one scan set per second at 8 cm^{-1} resolution is required. In general, a spectrometer purchased after 1985, or retrofitted to meet post-1985 FT-IR improvements, will be necessary to meet needed sensitivity of this protocol. A state-of-the-art analog/digital (A/D) converter is required, since it has been shown that the signal-to-noise ratio of single-beam GC/FT-IR systems is A/D converter limited.

6.1.2 GC/FT-IR interface – The interface should be lightpipe volume-optimized for the selected chromatographic conditions (lightpipe volume of 100-200 µL for capillary columns). The shortest possible inert transfer line (preferably fused silica) should be used to interface the end of the chromatographic column to the lightpipe. If fused silica capillary columns are employed, the end of the GC column can serve as the transfer line if it is adequately heated. It has been widely demonstrated that the optimum lightpipe volume is equal to the full width at half height of the GC eluate peak.

6.1.3 Capillary column – A fused silica DB-5 30 m x 0.32 mm capillary column with 1.0 µm film thickness (or equivalent)

6.1.4 Data Acquisition – A computer system dedicated to the GC/FT-IR system to allow the continuous acquisition of scan sets for a full chromatographic run. Peripheral data storage systems should be available (magnetic tape and/or disk) for the storage of all acquired data. Software should be available to allow the acquisition and storage of every scan set to locate the file numbers and transform high signal/noise (S/N) scan sets, and to provide a real-time reconstructed chromatogram.

6.1.5 Detector – A cryoscopic, medium-band mercury/cadmium/tellurium (HgCdTe or MCT) detector with the smallest practical focal area. Typical narrow-band MCT detectors operate from 3800-800 cm^{-1}, but medium-band MCT detectors can reach 650 cm^{-1}. A 750 cm^{-1} cutoff (or lower) is desirable since it allows the detection of typical carbon-chlorine stretch and aromatic out-of-plane carbon-hydrogen vibrations of environmentally important organo-chlorine and polynuclear aromatic compounds. The MCT detector sensitivity (D)* should be $\geq 1 \times 10^{10}$ cm.

6.1.6 Lightpipe – Constructed of inert materials, gold-coated, and volume-optimized for the desired chromatographic conditions (see Sec. 11.3)

6.1.7 GC – The FT-IR spectrometer should be interfaced to a temperature programmable GC equipped with a Grob-type (or equivalent) purged splitless injection system suitable for capillary glass columns or an on-column injector system.

A short, inert transfer line should interface the GC to the FT-IR lightpipe and, if applicable, to the GC detector. Fused silica GC columns may be directly interfaced to the lightpipe inlet and outlet.

6.2 Dry purge gas – If the spectrometer is the purge-type, provisions should be made to provide a suitable continuous source of dry purge-gas to the FT-IR spectrometer.

6.3 Dry carrier gas – The carrier gas should be passed through an efficient cartridge-type drier.

6.4 Syringes – 1 µL, 10 µL

7.0 REAGENTS AND STANDARDS

7.1 Reagent-grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS), where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Organic-free reagent water. All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

7.3 Solvents

7.3.1 Acetone, CH_3COCH_3 – Pesticide quality, or equivalent

7.3.2 Methylene chloride, CH_2Cl_2 – Pesticide quality, or equivalent

7.4 Stock standard solutions (1000 mg/L) – Standard solutions can be prepared from pure standard materials or purchased as a certified solution.

7.4.1 Prepare stock standard solutions by accurately weighing 0.1000±0.0010 g of pure material. Dissolve the material in pesticide-quality acetone or other suitable solvent and dilute to volume in a 100-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96 percent or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

7.4.2 Transfer the stock standard solutions into bottles with Teflon-lined screw caps. Store at ≤6°C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

7.4.3 Stock standard solutions must be replaced after 6 months or sooner if comparison with quality control (QC) reference samples indicates a problem.

7.5 Calibration standards and internal standards - For use in situations where GC/FT-IR will be used for primary quantitation of analytes rather than confirmation of GC/MS identification.

7.5.1 Prepare calibration standards that contain the compounds of interest, either singly or mixed together. The standards should be prepared at concentrations that will completely bracket the working range of the chromatographic system (at least one order of magnitude is suggested).

7.5.2 Prepare internal standard solutions. Suggested internal standards are 1-fluoronaphthalene and p-terphenyl-d14, or deuterated versions of 2-chlorophenol, phenol,

bis(2-chloroethoxy)methane, 2,4-dichlorophenol, phenanthrene, anthracene, and butyl benzyl phthalate. If deuterated versions aren't used, compounds that are on the method target analyte list may be used as internal standards as long as they aren't an analyte for a given site, and historical data are available to ensure their absence at a given site. Determine the internal standard concentration levels from the minimum identifiable quantities. See Tables 1 and 2.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

See Chapter 4, Organic Analytes, Sec. 4.1 for more information on this topic.

9.0 QUALITY CONTROL

9.1 General guidance

Refer to Chapter One for guidance on quality assurance (QA) and QC protocols. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific and Chapter One criteria; technique-specific QC criteria take precedence over Chapter One criteria. Any effort involving collection of analytical data should include development of a structured and systematic planning document, such as a quality assurance project plan (QAPP) or a sampling and analysis plan (SAP), which translates project objectives and specifications into directions for those who will implement the project and assess the results.

Each laboratory should maintain a formal QA program. The laboratory should also maintain records to document the quality of the data generated. Development of in-house QC limits for each method is encouraged, as described in Sec. 9.6 of Method 8000. Use of instrument-specific QC limits is encouraged, provided such limits will generate data appropriate for use in the intended application. All data sheets and QC data should be maintained for reference or inspection. QC to validate sample extraction is covered in Method 3500 and in the extraction method utilized. If extract cleanup was performed, follow the QC in Method 3600 and in the specific cleanup method.

9.2 Initial demonstration of proficiency (IDP)

Prior to implementation of a method, each laboratory must perform an IDP consisting of at least four replicate reference samples spiked into a clean matrix taken through the entire sample preparation and analysis. Whenever a significant change to instrumentation or a procedure occurs, the laboratory must demonstrate that acceptable precision and bias can still be obtained by the changed conditions. Whenever new staff members are trained, an analyst IDP must be performed.

9.2.1 Demonstration of proficiency for new analysts

Each laboratory should have a training program which documents that a new analyst is capable of performing the method, or portion of the method, for which the analyst is responsible. This demonstration should document that the new analyst is capable of successfully following the standard operating procedure (SOP) established by the laboratory.

9.3 It is recommended that the laboratory adopt additional QA practices for use with this method. The specific practices that are most productive depend upon the needs of the

laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.4 Lower limit of quantitation (LLOQ)

The laboratory shall establish the LLOQ as the lowest point of quantitation, which in most cases, is the lowest concentration in the calibration curve. LLOQ verification is recommended for each project application to validate quantitation capability at low analyte concentration levels. This verification may be accomplished with either clean control material (e.g., reagent water, solvent blank, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix, free of target compounds. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated data quality objectives (DQOs).

9.5 One hundred percent line test – Set the GC/FT-IR operating conditions to those employed for the sensitivity test (see Sec. 11.5). Collect 16 scans over the entire detector spectral range. Plot the test and measure the peak-to-peak noise between 1800 and 2000 cm^{-1}. This noise should be ≤ 0.15%. Store this plot for future reference.

9.6 Single beam test – With the GC/FT-IR at analysis conditions, collect 16 scans in the single-beam mode. Plot the co-added file and compare with a subsequent file acquired in the same fashion several minutes later. Note if the spectrometer is at purge equilibrium. Also, check the plot for signs of deterioration of the lightpipe potassium bromide windows. Store this plot for future reference.

9.7 Align test – With the lightpipe and MCT detector at thermal equilibrium, check the intensity of the centerburst versus the signal temperature calibration curve. Signal intensity deviation from the predicted intensity may mean thermal equilibrium has not yet been achieved, loss of detector coolant, decrease in source output, or a loss in signal throughput resulting from lightpipe deterioration.

9.8 Mirror alignment – Adjust the interferometer mirrors to attain the most intense signal. Data collection should not be initiated until the interferogram is stable. If necessary, align the mirrors prior to each GC/FT-IR run.

9.9 Lightpipe – The lightpipe and lightpipe windows should be protected from moisture and other corrosive substances at all times. For this purpose, maintain the lightpipe temperature above the maximum GC program temperature but below its thermal degradation limit. When not in use, maintain the lightpipe temperature slightly above ambient. At all times, maintain a flow of dry, inert, carrier gas through the lightpipe.

9.10 Beamsplitter – If the spectrometer is thermostated, maintain the beamsplitter at a temperature slightly above ambient at all times. If the spectrometer is not thermostated, minimize exposure of the beamsplitter to atmospheric water vapor.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibration – Refer to Method 8000 for proper calibration techniques.

10.2 Calibration must take place using the same sample introduction method that will be used to analyze actual samples.

11.0 PROCEDURE

11.1 Sample Preparation – Samples must be prepared by one of the following methods prior to GC/FT-IR analysis.

Matrix	Methods
Water	3510, 3520
Soil/sediment	3540, 3541, 3550
Waste	3540, 3541, 3550, 3580

11.2 Extracts may be cleaned up by Method 3640, Gel-Permeation Cleanup.

11.3 Initial Calibration

Recommended GC/FT-IR conditions:

Scan time:	At least 2 scan/sec
Initial column temperature and hold time:	40 °C for 1 minute
Column temperature program:	40-280 °C at 10 °C/min
Final column temperature to hold:	280 °C
Injector temperature:	280-300 °C
Transfer line temperature:	270 °C
Lightpipe:	280 °C
Injector:	Grob-type, splitless or on-column
Sample volume:	2-3 µL
Carrier gas:	Dry helium at about 1 mL/min

11.4 With an oscilloscope, check the detector centerburst intensity versus the manufacturer's specifications. Increase the source voltage, if necessary, to meet these specifications. For reference purposes, laboratories should prepare a plot of time versus detector voltage over at least a five-day period.

11.5 Capillary column interface sensitivity test – Install a 30 m x 0.32 mm fused silica capillary column coated with 1.0 µm of DB-5 (or equivalent). Set the lightpipe and transfer lines at 280 °C (if used). Under splitless Grob-type or on-column injection conditions, inject 25 ng of nitrobenzene, dissolved in 1 µL of methylene chloride. The nitrobenzene should be identified by the on-line library software search within the first five hits (nitrobenzene should be contained within the search library).

11.6 Interferometer – If the interferometer is air driven, adjust the interferometer drive air pressure to manufacturer's specifications.

11.7 MCT detector check – If the centerburst intensity is 75 percent or less of the mean intensity of the plot maximum obtained by the procedure of Sec. 11.4, install a new source and check the MCT centerburst with an oscilloscope versus the manufacturer's specifications (if available). Allow at least five hours of new source operation before data acquisition.

11.8 Frequency calibration – At the present time, no consensus exists within the spectroscopic community on a suitable frequency reference standard for vapor phase FT-IR. One reviewer has suggested the use of indene as an on-the-fly standard.

11.9 Minimum identifiable quantities – Using the GC/FT-IR operating parameters specified in Sec. 11.3, determine the minimum identifiable quantities for the compounds of interest.

11.9.1 Prepare a plot of lightpipe temperature versus MCT centerburst intensity (in volts or other vertical height units). This plot should span the temperature range between ambient and the lightpipe thermal limit in increments of about 20 °C. Use this plot for daily QA/QC (see Sec. 9.7). Note that modern GC/FT-IR interfaces (1985 and later) may have eliminated most of this temperature effect.

11.10 GC/FT-IR extract analysis

11.10.1 Analysis – Analyze the dried methylene chloride extract using the chromatographic conditions specified in Sec. 11.3 for capillary column interfaces.

11.10.2 GC/FT-IR identification – Visually compare the analyte IR spectrum versus the search library spectrum of the most promising on-line library search hits. Report, as identified, those analytes with IR frequencies for the five (maximum number) most intense IR bands (S/N ≥ 5) which are within ± 5.0 cm^{-1} of the corresponding bands in the library spectrum. Choose IR bands which are sharp and well resolved. The software used to locate spectral peaks should employ the peak "center of gravity" technique. In addition, the IR frequencies of the analyte and library spectra should be determined with the same computer software.

11.10.3 Retention time confirmation – After visual comparison of the analyte and library spectra as described in Sec. 11.10.2, compare the relative retention times (RRT) of the analyte and an authentic standard of the most promising library search hit. The standard and analyte RRT should agree within ± 0.01 RRT units when both are determined at the same chromatographic conditions.

11.10.4 Compound class or functionality assignment – If the analyte cannot be unequivocally identified, report its compound class or functionality. See Table 3 for gas-phase group frequencies to be used as an aid for compound class assignment. It should be noted that FT-IR gas-phase group stretching frequencies are 0-30 cm^{-1} higher in frequency than those of the condensed phase.

11.10.5 Quantitation – This protocol can be used to confirm GC/MS identifications, with subsequent quantitation. Two FT-IR quantitation techniques and a supplemental GC detector technique are also provided.

11.10.5.1 Integrated absorbance technique - After analyte identification, construct a standard calibration curve of concentration versus integrated IR absorbance. For this purpose, choose for integration only those FT-IR scans which are at or above the peak half-height. The calibration curve should span at least one order of magnitude and the working range should bracket the analyte concentration.

11.10.5.2 Maximum absorbance IR band technique – Following analyte identification, construct a standard calibration curve of concentration versus

maximum IR band intensity. For this purpose, choose an intense, symmetrical and well resolved IR absorbance band.

NOTE: IR transmission is not proportional to concentration. Select the FT-IR scan with the highest absorbance to plot against concentration. The calibration curve should span at least one order of magnitude and the working range should bracket the analyte concentration. This method is most practical for repetitive, target compound analyses. It is more sensitive than the integrated absorbance technique.

11.10.5.3 Supplemental GC detector technique – If a GC detector is used in tandem with the FT-IR detector, the following technique may be used: following analyte identification, construct a standard calibration curve of concentration versus integrated peak area. The calibration curve should span at least one order of magnitude and the working range should bracket the analyte concentration. This method is most practical for repetitive, target compound analyses.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 See Method 8000, Sec. 11 for information regarding data analysis and calculations.

12.2 Results must be reported in units commensurate with their intended use and all dilutions must be taken into account when computing final results.

13.0 METHOD PERFORMANCE

13.1 Method 8410 has been in use at the U.S. Environmental Protection Agency (EPA or the Agency) Environmental Monitoring Systems Laboratory for many years. Portions of it have been reviewed by key members of the FT-IR spectroscopic community (Ref 9). Side-by-side comparisons with GC/MS sample analyses indicate similar demands upon analytical personnel for the two techniques. Extracts previously subjected to GC/MS analysis are generally compatible with GC/FT-IR. However, it should be kept in mind that lightpipe windows are typically water soluble. Thus, extracts must be vigorously dried prior to analysis.

13.2 Table 4 provides performance data for this method.

14.0 POLLUTION PREVENTION

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

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste*

Reduction, a free publication available from the ACS, Committee on Chemical Safety, http://portal.acs.org/portal/fileFetch/C/WPCP_012290/pdf/WPCP_012290.pdf.

15.0 WASTE MANAGEMENT

The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Laboratories are urged to protect air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations and complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the ACS at the web address listed in Sec. 14.2 above.

16.0 REFERENCES

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17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method.

TABLE 1

FUSED SILICA CAPILLARY COLUMN GC/FT-IR IDENTIFICATION LIMITS FOR BASE/NEUTRAL EXTRACTABLES

Compound	Identification Limit		
	ng injected[a]	µg/L[b]	ν max, cm^{-1}[c]
Acenaphthene	40(25)	20(12.5)	799
Acenaphthylene	50(50)	25(25)	799
Anthracene	40(50)	20(25)	874
Benzo(a)anthracene	(50)	(25)	745
Benzo(a)pyrene	(100)	(50)	756
Bis(2-chloroethyl)ether	70(10)	35(5)	1115
Bis(2-chloroethoxy)methane	50(10)	25(5)	1084
Bis(2-chloro-1-methylethyl)ether	50(10)	25(5)	1088
Butyl benzyl phthalate	25(10)	12.5(5)	1748
4-Bromophenyl phenyl ether	40(5)	20(2.5)	1238
2-Chloronaphthalene	110	55	851
4-Chloroaniline	40	20	1543
4-Chlorophenyl phenyl ether	20(5)	10(2.5)	1242
Chrysene	(100)	(50)	757
Di-n-butyl phthalate	20(5)	10(2.5)	1748
Dibenzofuran	40	20	1192
Diethyl phthalate	20(5)	10(2.5)	1748
Dimethyl phthalate	20(5)	10(2.5)	1751
Di-n-octyl phthalate	25(10)	12.5(5)	1748
Di-n-propyl phthalate	25(5)	12.5(2.5)	1748
1,2-Dichlorobenzene	50	25	1458
1,3-Dichlorobenzene	50	25	779
1,4-Dichlorobenzene	50	25	1474
2,4-Dinitrotoluene	20	10	1547
2,6-Dinitrotoluene	20	10	1551
Bis-(2-ethylhexyl)phthalate	25(10)	12.5(5)	1748
Fluoranthene	100(50)	50(25)	773
Fluorene	40(50)	20(25)	737
Hexachlorobenzene	40	20	1346
Hexachlorocyclopentadiene	120	60	814
Hexachloroethane	50	25	783
1,3-Hexachlorobutadiene	120	60	853
Isophorone	40	20	1690
2-Methylnaphthalene	110	55	3069
Naphthalene	40(25)	20(12.5)	779
Nitrobenzene	25	12.5	1539
N-Nitrosodimethylamine	20(5)	10(2.5)	1483
N-Nitrosodi-n-propylamine	50(5)	25(2.5)	1485
N-Nitrosodiphenylamine[d]	40	20	1501
2-Nitroaniline	40	20	1564
3-Nitroaniline	40	20	1583

Compound	Identification Limit		ν max, cm^{-1c}
	ng injected[a]	µg/L[b]	
4-Nitroaniline	40	20	1362
Phenanthrene	50(50)	25(25)	729
Pyrene	100(50)	50(25)	820
1,2,4-Trichlorobenzene	50(25)	25(12.5)	750
1-Fluoronaphthalene[e]	(100)	(50)	770
p-Terphenyl-d14[e]	(100)	(50)	712

[a] Determined using on-column injection and the conditions of Sec. 11.3. A medium-band HgCdTe detector [3800-700 cm^{-1}; D*value (λpeak 1000 Hz, 1) 4.5 x 10^{10} cm $Hz^{1/2}W^{-1}$] type with a 0.25 mm^2 focal chip was used. The GC/FT-IR system is a 1976 retrofitted model. Values in parentheses were determined with a newer (1986) GC/FT-IR system. A narrow band HgCdTe detector [3800-750 cm^{-1}; D*value (λpeak 1000 Hz, 1) 4 x 10^{10} cm $Hz^{1/2}W^{-1}$] was used. Chromatographic conditions are those of Sec. 11.3.

[b] Based on a 2-µL injection of a 1-L sample that has been extracted and concentrated to a volume of 1.0 mL. Values in parentheses were determined with a newer (1986) GC/FT-IR system. A narrow band HgCdTe detector [3800-750 cm^{-1}; D*value (λpeak 1000 Hz, 1) 4 x 10^{10} cm $Hz^{1/2}W^{-1}$] was used. Chromatographic conditions are those of Sec. 11.3.

[c] Most intense IR peak and suggested quantitation peak.

[d] Detected as diphenylamine.

[e] Suggested internal standard. Identification limit data is not actual, but is estimated high to assure sufficient amount or concentration.

TABLE 2

FUSED SILICA CAPILLARY COLUMN GC/FT-IR ON-LINE AUTOMATED IDENTIFICATION LIMITS FOR ACIDIC EXTRACTABLES

Compound	Identification Limit		νmax, cm^{-1}[c]
	ng injected[a]	µg/L[b]	
Benzoic acid	70	35	1751
2-Chlorophenol	50	25	1485
4-Chlorophenol[d]	100	50	1500
4-Chloro-3-methylphenol	25	12.5	1177
2-Methylphenol	50	25	748
4-Methylphenol	50	25	1177
2,4-Dichlorophenol	50	25	1481
2,4-Dinitrophenol	60	30	1346
4,6-Dinitro-2-methylphenol	60	30	1346
2-Nitrophenol[d]	40	20	1335
4-Nitrophenol	50	25	1350
Pentachlorophenol	50	25	1381
Phenol	70	35	1184
2,4,6-Trichlorophenol	120	60	1470
2,4,5-Trichlorophenol	120	60	1458

[a] Operating conditions are the same as those cited in Sec. 11.3.

[b] Based on a 2-µL injection of a 1-L sample that has been extracted and concentrated to a volume of 1.0 mL.

[c] Most intense IR peak and suggested quantitation peak.

[d] Subject to interference from co-eluting compounds.

TABLE 3

GAS-PHASE GROUP FREQUENCIES

Functionality	Class	Number of Compounds	Frequency Range, ν cm^{-1}
Ether	Aryl, Alkyl	14	1215-1275
	Benzyl, Alkyl	3	1103-1117
	Diaryl	5	1238-1250
	Dialkyl	12	1084-1130
	Alkyl, Vinyl	3	1204-1207
			1128-1142
Ester	Unsubstituted Aliphatic	29	1748-1761
	Aromatic	11	1703-1759
	Monosubstituted Acetate	34	1753-1788
Nitro	Aliphatic	5	1566-1594
			1548-1589
			1377-1408
			1327-1381
	Aromatic	18	1535-1566
			1335-1358
Nitrile	Aliphatic	9	2240-2265
	Aromatic	9	2234-2245
Ketone	Aliphatic (acyclic)	13	1726-1732
	(α,β unsaturated)	2	1638-1699
	Aromatic	16	1701-1722
Amide	Substituted Acetamides	8	1710-1724
Alkyne	Aliphatic	8	3323-3329
Acid	Aliphatic	24	3574-3580
		22	1770-1782
	Dimerized-Aliphatic	2	3586-3595
	Aromatic	10	3574-3586
		10	1757-1774
Phenol	1,4-Disubstituted	15	3645-3657
		15	1233-1269
		15	1171-1190
	1,3-Disubstituted	10	3643-3655
		10	1256-1315
		10	1157-1198
	1,2-Disubstituted	6	3582-3595
			1255-1274

(continued)

TABLE 3

(Continued)

Functionality	Class	Number of Compounds	Frequency Range, ν cm^{-1}
Alcohol	Primary Aliphatic	20	3630-3680
		11	1206-1270
		16	1026-1094
	Secondary Aliphatic	17	3604-3665
		10	1231-1270
	Tertiary Aliphatic	10	3640-3670
		6	1213-1245
Amine	Primary Aromatic	15	3480-3532
	Secondary Aromatic	5	3387-3480
	Aliphatic	10	760-785
Alkane		14	2930-2970
			2851-2884
			1450-1475
			1355-1389
Aldehyde	Aromatic	12	1703-1749
		12	2820-2866
		12	2720-2760
	Aliphatic	6	1742-1744
		6	2802-2877
		6	2698-2712
Benzene	Monosubstituted	7	1707-1737
		24	1582-1630
		24	1470-1510
		11	831-893
		23	735-790
		25	675-698

TABLE 4

FUSED SILICA CAPILLARY COLUMN GC/FT-IR QUANTITATION RESULTS

Compound	Concentration Range, and Identification Limit, ng[a]	Maximum Absorbance[b] Correlation Coefficient[d]	Integrated Absorbance[c] Correlation Coefficient[d]
Acenaphthene	25-250	0.9995	0.9985
Acenaphthylene	25-250	0.9959	0.9985
Anthracene	50-250	0.9969	0.9971
Benzo(a)anthracene	50-250	0.9918	0.9921
Benzoic acid	50-250	0.9864	0.9892
Benzo(a)pyrene	100-250	0.9966	0.9074
Bis(2-chloroethoxy)methane	25-250	0.9992	0.9991
Bis(2-chloroethyl)ether	25-250	0.9955	0.9992
Bis(2-chloro-1-methylethyl)ether	50-250	0.9981	0.9998
4-Bromophenyl phenyl ether	25-250	0.9995	0.9996
Butyl benzyl phthalate	25-250	0.9999	0.9994
4-Chloroaniline	25-250	0.9991	0.9965
4-Chloro-3-methylphenol	25-250	0.9975	0.9946
2-Chloronaphthalene	100-250	0.9897	0.9988
2-Chlorophenol	25-250	0.9976	0.9965
4-Chlorophenol[e]	-	-	-
4-Chlorophenyl phenyl ether	25-250	0.9999	0.9997
Chrysene	100-250	0.9985	0.9984
Dibenzofuran	25-250	0.9697	0.8579
Di-n-butyl phthalate	25-250	0.9998	0.9996
1,2-Dichlorobenzene	25-250	0.9937	0.9947
1,3-Dichlorobenzene	25-250	0.9985	0.9950
1,4-Dichlorobenzene	25-250	0.9994	0.9994
2,4-Dichlorophenol	25-250	0.9964	0.9969
Dimethyl phthalate	25-250	0.9998	0.9996
Dimethyl phthalate	25-250	0.9998	0.9997
Dinitro-2-methylphenol	50-250	0.9936	0.9967
2,4-Dinitrophenol	50-250	0.9920	0.9916
2,4-Dinitrotoluene	25-250	0.9966	0.9928
2,6-Dinitrotoluene	25-250	0.9947	0.9966
Di-n-octyl phthalate	25-250	0.9983	0.9991
Bis(2-ethylhexyl)phthalate	25-250	0.9991	0.9993
Fluoranthene	25-250	0.9983	0.9966
Fluorene	25-250	0.9987	0.9989
Hexachlorobenzene	50-250	0.9981	0.9995
1,3-Hexachlorobutadiene	50-250	0.9960	0.9979
Hexachlorocyclopentadiene	100-250	0.9862	0.9845
Hexachloroethane	25-250	0.9986	0.9992
Isophorone	25-250	0.9984	0.9990

Compound	Concentration Range, and Identification Limit, ng[a]	Maximum Absorbance[b] Correlation Coefficient[d]	Integrated Absorbance[c] Correlation Coefficient[d]
2-Methylnaphthalene	50-250	0.9981	0.9950
2-Methylphenol	25-250	0.9972	0.9964
4-Methylphenol	25-250	0.9972	0.9959
Naphthalene	25-250	0.9956	0.9954
2-Nitroaniline	25-250	0.9996	0.9994
3-Nitroaniline	25-250	0.9985	0.9990
4-Nitroaniline	25-250	0.9936	0.9992
Nitrobenzene	25-250	0.9997	0.9979
2-Nitrophenol[e]	-	-	-
4-Nitrophenol	50-250	0.9951	0.9953
N-Nitrosodimethylamine	25-250	0.9982	0.9993
N-Nitrosodiphenylamine	25-250	0.9994	0.9971
N-Nitrosodi-n-propylamine	25-250	0.9991	0.9995
Pentachlorophenol	50-250	0.9859	0.9883
Phenanthrene	25-250	0.9941	0.9989
Phenol	25-250	0.9978	0.9966
Pyrene	50-250	0.9971	0.9977
1,2,4-Trichlorobenzene	50-250	0.9969	0.991
2,4,5-Trichlorophenol	25-250	0.9952	0.9966
2,4,6-Trichlorophenol	25-250	0.9969	0.9965

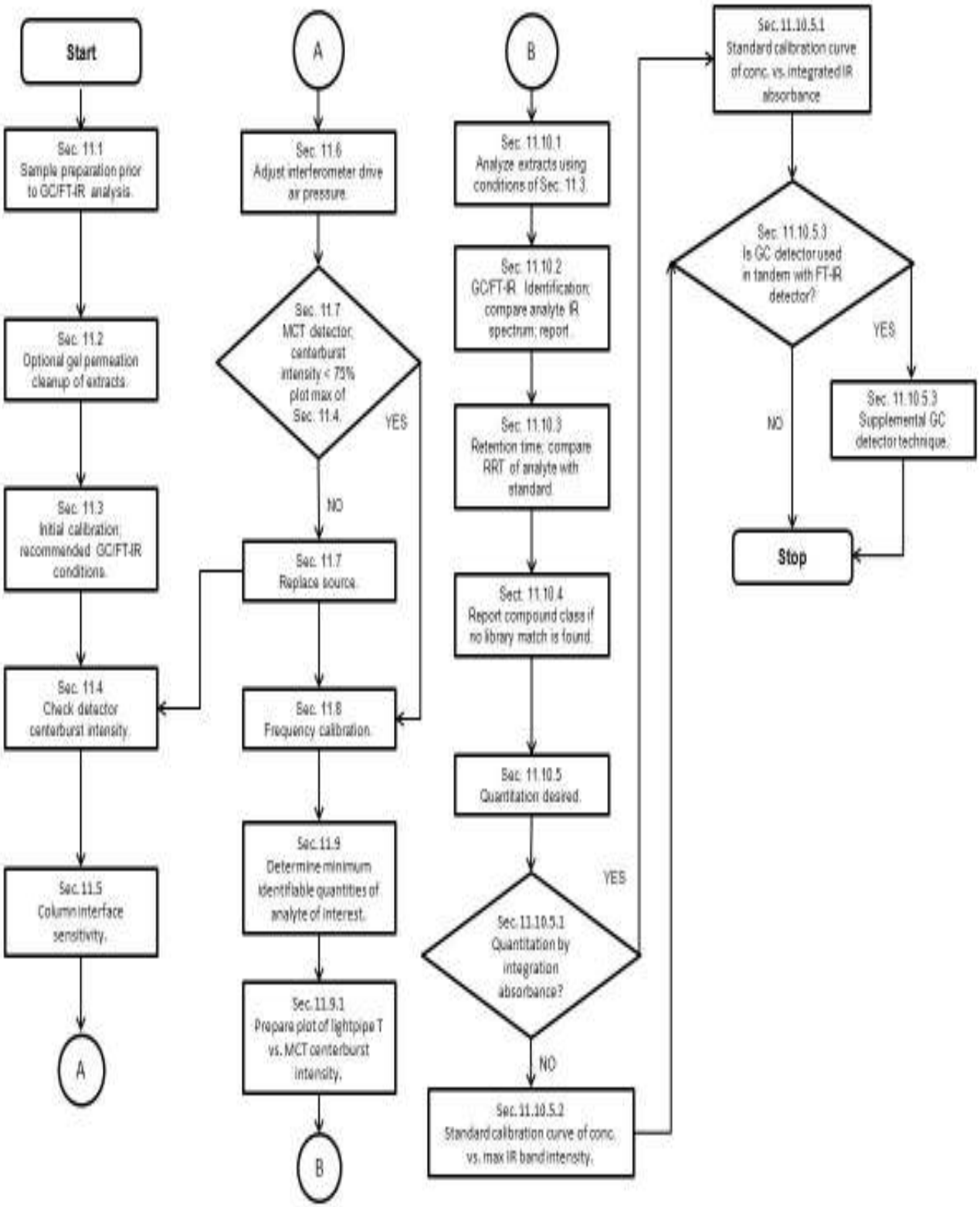
[a] Lower end of range is at or near the identification limit.

[b] FT-IR scan with highest absorbance plotted against concentration.

[c] Integrated absorbance of combined FT-IR scans which occur at or above the chromatogram peak half-height.

[d] Regression analysis carried out at four concentration levels. Each level analyzed in duplicate. Chromatographic conditions are stated in Sec. 11.3.

[e] Subject to interference from co-eluting compounds.


Start
Sec. 11.1 Sample preparation prior to GC/FT-IR analysis.
Sec. 11.2 Optional gel permeation cleanup of extracts.
Sec. 11.3 Initial calibration; recommended GC/FT-IR conditions.
Sec. 11.4 Check detector centerburst intensity.
Sec. 11.5 Column interface sensitivity.
A
A
Sec. 11.6 Adjust interferometer drive air pressure.
Sec. 11.7 MCT detector; centerburst intensity < 75% plot max of Sec. 11.4.
YES
NO
Sec. 11.7 Replace source.
Sec. 11.8 Frequency calibration.
Sec. 11.9 Determine minimum identifiable quantities of analyte of interest.
Sec. 11.9.1 Prepare plot of lightpipe T vs. MCT centerburst intensity.
B
B
Sec. 11.10.1 Analyze extracts using conditions of Sec. 11.3.
Sec. 11.10.2 GC/FT-IR Identification; compare analyte IR spectrum; report.
Sec. 11.10.3 Retention time; compare RRT of analyte with standard.
Sect. 11.10.4 Report compound class if no library match is found.
Sec. 11.10.5 Quantitation desired.
YES
Sec. 11.10.5.1 Quantitation by integration absorbance?
NO
Sec. 11.10.5.2 Standard calibration curve of conc. vs. max IR band intensity.
Sec. 11.10.5.1 Standard calibration curve of conc. vs. integrated IR absorbance
Sec. 11.10.5.3 Is GC detector used in tandem with FT-IR detector?
YES
NO
Sec. 11.10.5.3 Supplemental GC detector technique.
Stop

Appendix A
Changes in this version from the December 1994, Revision 0.

1. Improved overall method formatting for consistency with new SW-846 methods style guidance. The format was updated to Microsoft Word .docx. This includes the additions of all sections required by the new format.

2. Many minor editorial and technical revisions were made throughout to improve method clarity.

3. The revision number was changed to one and the date published was changed to July 2014.

4. This appendix was added showing changes from the previous revision.

5. Chemical name was changed by the Integrated Risk Information System (IRIS) on November 30, 2007 from Bis(2-chloroisopropyl)ether to Bis(2-chloro-1-methylethyl)ether (common name). This compound is also known as 2,2'-oxybis(1-chloropropane) (CAS index name). See the link at http://www.epa.gov/iris/subst/0407.htm, Section VII for the "Revision History" and Section VIII, for "Synonyms" of this chemical.

6. This appendix was added showing changes from the previous revision.

7. The flowchart on page 19 was updated to reflect the current section numbers.

8. Section 7.5.2 and Table 1 was updated to clarify use of internal standards.