METHOD 8430

ANALYSIS OF BIS(2-CHLOROETHYL) ETHER AND HYDROLYSIS PRODUCTS BY DIRECT AQUEOUS INJECTION GC/FT-IR

1.0 SCOPE AND APPLICATION

1.1 This method provides procedures for the identification and quantitation of bis(2-chloroethyl) ether and its hydrolysis compounds in aqueous matrices by direct aqueous injection (DAI) and gas chromatography with detection by a fourier transform infrared spectrometer (GC/FT-IR). The following compounds can be determined by this method:

Compound Name	Abbreviation	CAS Number[a]
Bis(2-chloroethyl) ether	BCEE	111-44-4
2-Chloroethanol	CE	107-07-3
2-(2-Chloroethoxy)ethanol	2CEE	628-89-7
Diethylene glycol	DEG	111-46-6
Ethylene glycol	EG	107-21-1

[a] Chemical Abstract Service Registry Number.

1.2 Although the initial study upon which this method is based targeted only the bis(2-chloroethyl) ether and its hydrolysis compounds, its has been suggested that this method can be used for the identification of compounds that are generally non-extractable, highly water soluble, thermally stable, and do not co-elute with water from the GC. Possible analytes include ethers and alcohols.

1.3 The minimum identifiable quantities (MIQ) for the five compounds in organic-free reagent water range from a low of 46 ng for BCEE, to a high of 120 ng for EG (See Sec. 9.2 for MIQ definition). The MIQ for a specific sample may differ depending on the nature of the interferences in the sample matrix and the amount of sample used for the analysis.

1.4 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatography (GC), the interpretation of FT-IR spectra and the use of continuous data collection systems. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Water samples are filtered through a 0.45 μm filter, and 1 μL aliquots are injected directly into a GC. The GC is equipped with 2 detectors, a thermal conductivity detector (TCD) and an FT-IR. During analysis, the analyst disconnects the FT-IR from the system to prevent aqueous degradation of the KBr window, using an 8 port switching valve. Further analysis of either the solid precipitate from the filtering step or the aqueous filtrate for trace amounts of non-water soluble compounds may be done by extracting the samples using appropriate 3500 series methods.

3.0 INTERFERENCES

3.1 Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks.

    3.1.1 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. It should then be drained dry, and heated in a laboratory oven at 130°C for several hours before use. Solvent rinsing with methanol may be substituted for the oven heating. After drying and cooling, glassware should be stored in a clean environment to prevent any accumulation of dust or other contaminants.

    3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are in the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the matrix being sampled. If significant interferences occur in subsequent samples, additional cleanup may be necessary.

3.3 The extent of interferences that may be encountered in this method has not been fully assessed. Although the GC conditions described allow for a unique resolution of the specific compounds covered by this method, other matrix components may interfere.

4.0 APPARATUS AND MATERIALS

4.1 GC/FT-IR System

    4.1.1 GC - Temperature programmable oven equipped with a cool on-column injection port, a purged splitless injection port, or an equivalent suitable for capillary glass columns.

    4.1.2 Column - 30 m DB-wax, 1.0 µm film, Megabore (J&W Scientific), or equivalent.

    4.1.3 Detectors

        4.1.3.1 Thermal Conductivity Detector (TCD) - Must be able to handle temperatures up to 300°C.

        4.1.3.2 FT-IR Spectrometer - System should be equipped with a mercury-cadmium-telluride detector, using a light-pipe interface with KBr windows (available from Digilab Model GC/C32 or equivalent). Resolution of 8 cm^{-1} and a range of 4,000 to 7,000 cm^{-1} is required. The light-pipe interface and transfer lines should be contained in a heated system (up to 250°C) to prevent sample condensation. Extra transfer lines will be needed for the switching system.

    4.1.4 Detector Switching System - 8 port stainless steel GC rotary switching valve, with an inert interval surface (such as FEP) and capable of withstanding temperatures up to 300°C. Ideally, the valve should be mounted inside the GC oven with external control. If internal

mounting is not possible then use of an external heated valve enclosure may be employed. This allows direct injection of aqueous samples by using the valve system to route water away from the FT-IR KBr window which water rapidly destroys.

    4.1.5   Data Collection - Each detector should have its own signal recorder.

        4.1.5.1   TCD Signal - Either an analog strip chart recorder or a digital computerized data collection system is acceptable.

        4.1.5.2   FT-IR Signal - The continuous collection of the spectrometer's signal will require a computerized data system with the ability to continuously collect spectra at a rate of 4 scans/sec and add the 4 scans to produce a data point for each second. To confirm the identity of target compounds, a library of vapor phase spectra should be established.

    4.2   Glassware

        4.2.1   Glass Funnels

        4.2.2   Volumetric Flasks (square) - various sizes.

        4.2.3   Pipettes (A grade) - various sizes.

        4.2.4   Sample Vial with polytetrafluoroethylene (PTFE)-lined lids.

    4.3   Syringes - 10 µL, suitable for GC work.

    4.4   Analytical Balance, accurate to ±0.0001 g.

    4.5   Vacuum Filtration Apparatus - 0.45 µm filter and clean glass flasks that are able to hold at least 1 L of liquid.

5.0 REAGENTS

    5.1   Reagent grade 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, 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.

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

    5.3   Helium Gas - Suitable for gas chromatography.

    5.4   Methanol, CH_3OH - Pesticide reagent grade, nanograde, or equivalent.

    5.5   Stock Standard Solutions

5.5.1   Prepare, in organic-free reagent water, a stock standard solution containing all of the target analytes at a concentration of 1000 mg/L. Record the actual weight of each compound added and calculate the actual concentration of each component of the solution.

5.5.2   Transfer the stock standard solution into a PTFE-sealed screw-cap bottle for storage. Store at 4°C and protect from light. Check the solution periodically for signs of degradation or evaporation. This solution must be replaced every 3 months, or when any sign of degradation or evaporation is observed.

5.6   Calibration Standards

5.6.1   Calibration standards at 500, 250, 100, 50, and 25 mg/L, from the aqueous stock standard solution, by appropriate volumetric dilutions with water are suggested. Store as in Sec. 5.5.2. The calibration solutions should not be made by serial dilution of a single solution.

5.6.2   Since the MDL for the target compounds in water has not been established, the suggested calibration curve concentrations may be modified, depending on matrix interferences and sensitivity of equipment. Generally speaking, the calibration curve should span at least one order of magnitude and the working range should bracket the analyte concentration.

5.7   Spiking Solution - The analyst should monitor the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix, by spiking a known amount of the target analytes into blanks or into matrix spike samples. A suggested preliminary concentration of the spiking stock solution is 100 g/L. It is recommended that the spiking concentration should be set at 1 to 5 times higher than the background concentration determined for that matrix.

5.8   Surrogate Spiking Solution: To monitor the performance of the method for all samples, a minimum of one surrogate compound should be chosen by the laboratory. This compound should be diluted with water to an appropriate concentration and added to all samples, method blanks, matrix spikes, and calibration standards.

6.0   SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1   See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

6.2   Samples should be stored at 4°C and protected from light.

6.3   Samples should be filtered and analyzed within 14 days of collection.

7.0   PROCEDURE

7.1   GC/FT-IR Conditions (Recommended):

Flow Rate:	2.4 mL/minute of helium carrier gas.
Run Time:	approximately 15 minutes
Injection Volume:	1 µL
Valve Switch Time:	4 minutes

Temperature Program:	80°C to 220°C at 15°C/minute, hold at 220°C for 10 minutes.
TCD Temperature:	290°C
GC/FT-IR Interface, Transfer Lines, and Light Pipe Temperature:	220°C
Scan Time:	4 scans/second

NOTE: Modifications of these parameters may be necessary to facilitate the separation of certain compounds depending on matrix interferences encountered.

7.2   External Calibration

7.2.1   Calibration standards may be prepared using the suggested concentrations in Sec. 5.6. Matrix interferences may prevent quantitation at the suggested concentrations. When necessary, the lowest concentration of the calibration curve should be adjusted to be at, or near, the method detection limit. Refer to Method 8000, Sec. 7.0 for proper external calibration procedures.

7.2.2   Refer to Method 8000, Sec. 7.0 for the establishment of retention time windows.

7.2.3   Analyze a solvent blank to ensure that the system is clean and interference free.

7.2.4   Analyze the 5 calibration standards, starting with the lowest concentration and ending with highest concentration.

7.2.5   Tabulate the IR absorbance peak area along with the calibration factor (CF) for the analyte at each concentration. Refer to Sec. 7.0 of Method 8000 for linear and non-linear calibration acceptance criteria. It should be noted that IR transmission is not directly proportional to concentration.

7.2.6   Recheck the instrument calibration each day, before and after an analysis is performed, by analyzing one or more calibration standards. The response obtained should fall within ±15 percent of the expected value or the instrument must be recalibrated.

7.2.7   After the analysis of 10 or fewer samples, one of the calibration standards must be reanalyzed to ensure that the retention times and the CFs of the target analytes remain within the QC requirements.

7.3   Sample Spiking and Filtering

7.3.1   Allow the sample to come to ambient temperature. Mark the water meniscus on the side of the 1 L sample bottle for later determination of exact sample volume.

7.3.2   Add 2 mL of the spiking solution to the spiked blank and the matrix spike sample. The final concentration of the added analytes should be about 200 mg/L.

7.3.3   Vacuum filter the sample through a 0.45 µm filter that has been rinsed with organic-free reagent water. The filtrate should be collected in a clean glass bottle. Any particulate collected by the filtering process may be discarded or extracted for analysis of trace amounts of non-water soluble compounds using an appropriate 3500 series method.

7.3.4   Using a pipette, withdraw a 5 mL aliquot of the sample (aqueous filtrate) and place it into a clean glass sample vial with a PTFE-lined lid. This 5 mL aliquot will be the sample used for the direct aqueous injection of the water sample. Store at 4°C in the dark. The remainder of the aqueous filtrate may be saved in a glass bottle with a PTFE-lined lid until after the analysis is complete or extracted for analysis of trace amounts of non-water soluble compounds using an appropriate 3500 series method.

7.4   GC Analysis

7.4.1   Method 8000, Sec. 7.0 provides instructions on calibration, establishing retention time windows, the analysis sequence, appropriate dilutions, and chromatopgraphic identification criteria.

7.4.2   Determining the Valve Switching Time - The valve switch time must be determined before proceeding with the analysis of samples.

7.4.2.1   Place the GC switching valve in the position which routes the sample to the TCD and away from the FT-IR spectrometer and inject a 1 µL aliquot of a reagent blank to determine the retention time of water in the system. Use this retention time to determine the optimum valve switch time to cycle water away from the KBr window loop. At the optimum valve switch time, the 8 port valve may be moved to the position which allows use of the FT-IR detector and the TCD in tandem after the bulk of the aqueous portion of the sample has been diverted away from the KBr window (See Figure 1).

7.4.2.2   Once the optimum valve switch time is established, the system may be re-evaluated with one of the aqueous calibration standards to assure complete separation of all target analytes.

NOTE: Traces of water in the GC/FT-IR interface are acceptable. However, the repeated injection of 1.0 µL aqueous samples may eventually destroy the KBr window used for FT-IR detection. Care should be taken in deciding the valve switching time so that the KBr window is exposed to only trace amounts of water and target analytes that elute with retention times just after water are not missed.

7.4.2.3   If the TCD is proven to have adequate sensitivity for a particular analysis, it may be used as the prime detector once the target analytes are identified using the FT-IR detector. When the TCD is used as the prime detector, the switching valve should remain in the position which diverts the sample away from the KBr window.

7.4.3   Sample Analysis - Analyze the samples, blanks, spiked blanks, and spiked matrix samples by injecting 1 µL aliquots into the GC and switching to the FT-IR spectrometer at the previously determined time. Dilution of the sample may be necessary to adjust the analyte concentration to within the working range of the calibration curve. If dilution is necessary, note which samples were diluted in the final report and make the appropriate calculation adjustments.

7.4.4   GC/FT-IR Identification - Visually compare the analyte infrared (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 $\geq$ 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.

7.5   Calculations

   7.5.1   Calculate the calibration factor for each calibration standard and determine the percent relative standard deviation (%RSD) using the external standard calibration procedure in Sec. 7.0 of Method 8000.

   7.5.2   Calculation of the concentration of analytes using the external standard calibration procedure is provided in Sec. 7.0 of Method 8000.

8.0   QUALITY CONTROL

   8.1   Refer to Chapter One and Method 8000 for specific quality control procedures. Quality control procedures to validate sample extraction is covered in Method 3500. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated.

   8.2   Quality control necessary to evaluate the GC system operation is found in Method 8000, Sec. 7.0 under Retention Time Windows, Calibration Verification and Chromatographic Analysis of Samples. Refer to Appendix A for FT-IR spectrometer QC requirements.

   8.3   Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

   8.4   Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes a method blank, matrix spike, a duplicate, a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

      8.4.1   Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike and matrix spike duplicate pair.

      8.4.2   A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.4.3   See Method 8000, Sec. 8.0 for the details on carrying out sample quality control for preparation and analysis.

8.5   Surrogate recoveries: The laboratory should evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. Currently, surrogate compounds have not been selected for this procedure. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

8.6   It is recommended that the laboratory adopt additional quality assurance 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.

9.0   METHOD PERFORMANCE

9.1   No MDL data are available.

9.2   Minimum Identifiable Quantities (MIQ)

9.2.1   The MIQ is defined as the minimum quantity that must be injected to result in a spectral match that has the correct compound identification in the top 5 spectral matches. The MIQ will vary depending on instrument sensitivity and sample matrix effects.
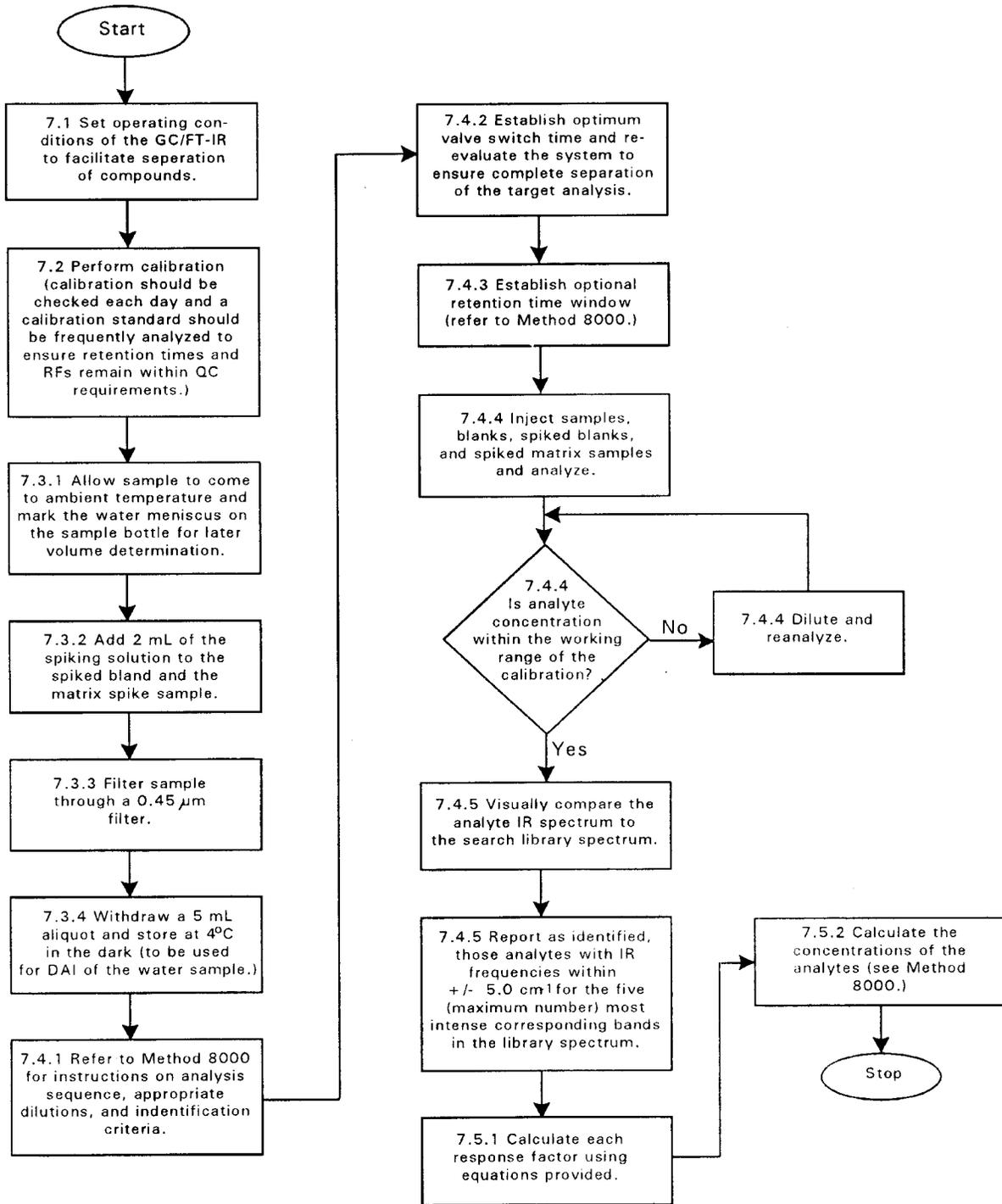
9.2.2   The MIQ range for CE, BCEE, EG, 2CEE, and DEG in organic-free reagent water by direct aqueous injection goes from a low of 46 ng for BCEE to a high of 120 ng for EG.[1]

10.0   REFERENCES

1.   Payne, W.D. and Collette, T.W., "Identification of Bis(2-chloroethyl) ether Hydrolysis Products by Direct Aqueous Injection GC/FT-IR," J. of High Res. Chrom., 12, 693-696, 1989.

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ANALYSIS OF BIS(2-CHLOROETHYL) ETHER AND HYDROLYSIS PRODUCTS BY DIRECT AQUEOUS INJECTION

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                          ┌─────────┐
                          │  Start  │
                          └─────────┘
                               │
                               ▼
┌──────────────────────────┐       ┌──────────────────────────┐
│ 7.1 Set operating con-   │       │ 7.4.2 Establish optimum  │
│ ditions of the GC/FT-IR  │       │ valve switch time and re-│
│ to facilitate seperation │       │ evaluate the system to   │
│ of compounds.            │       │ ensure complete separation│
└──────────────────────────┘       │ of the target analysis.  │
             │                     └──────────────────────────┘
             ▼                                  │
┌──────────────────────────┐                   ▼
│ 7.2 Perform calibration  │       ┌──────────────────────────┐
│ (calibration should be   │       │ 7.4.3 Establish optional │
│ checked each day and a   │       │ retention time window    │
│ calibration standard     │       │ (refer to Method 8000.)  │
│ should be frequently     │       └──────────────────────────┘
│ analyzed to ensure       │                   │
│ retention times and      │                   ▼
│ RFs remain within QC     │       ┌──────────────────────────┐
│ requirements.)           │       │ 7.4.4 Inject samples,    │
└──────────────────────────┘       │ blanks, spiked blanks,   │
             │                     │ and spiked matrix samples│
             ▼                     │ and analyze.             │
┌──────────────────────────┐       └──────────────────────────┘
│ 7.3.1 Allow sample to    │                   │
│ come to ambient          │                   ▼
│ temperature and mark     │              ╱7.4.4╲
│ the water meniscus on    │            ╱ Is analyte ╲   No  ┌──────────────┐
│ the sample bottle for    │           ╱ concentration ╲───▶│ 7.4.4 Dilute │
│ later volume             │           ╲ within the   ╱     │ and reanalyze│
│ determination.           │            ╲ working range╱     └──────────────┘
└──────────────────────────┘            ╲ of the     ╱
             │                           ╲calibration?╱
             ▼                               │ Yes
┌──────────────────────────┐                 ▼
│ 7.3.2 Add 2 mL of the    │       ┌──────────────────────────┐
│ spiking solution to the  │       │ 7.4.5 Visually compare   │
│ spiked bland and the     │       │ the analyte IR spectrum  │
│ matrix spike sample.     │       │ to the search library    │
└──────────────────────────┘       │ spectrum.                │
             │                     └──────────────────────────┘
             ▼                                  │
┌──────────────────────────┐                   ▼
│ 7.3.3 Filter sample      │       ┌──────────────────────────┐   ┌──────────────────┐
│ through a 0.45 μm        │       │ 7.4.5 Report as          │   │ 7.5.2 Calculate  │
│ filter.                  │       │ identified, those        │   │ the concentrations│
└──────────────────────────┘       │ analytes with IR         │──▶│ of the analytes  │
             │                     │ frequencies within       │   │ (see Method 8000.)│
             ▼                     │ +/- 5.0 cm-1 for the     │   └──────────────────┘
┌──────────────────────────┐       │ five (maximum number)    │            │
│ 7.3.4 Withdraw a 5 mL    │       │ most intense             │            ▼
│ aliquot and store at 4°C │       │ corresponding bands in   │       ┌─────────┐
│ in the dark (to be used  │       │ the library spectrum.    │       │  Stop   │
│ for DAI of the water     │       └──────────────────────────┘       └─────────┘
│ sample.)                 │                   │
└──────────────────────────┘                   ▼
             │                     ┌──────────────────────────┐
             ▼                     │ 7.5.1 Calculate each     │
┌──────────────────────────┐       │ response factor using    │
│ 7.4.1 Refer to Method    │       │ equations provided.      │
│ 8000 for instructions on │       └──────────────────────────┘
│ analysis sequence,       │
│ appropriate dilutions,   │
│ and indentification      │
│ criteria.                │
└──────────────────────────┘
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APPENDIX A
Quality Control of FT-IR Spectrometer

A.1   Equipment Adjustments and Maintenance

A.1.1   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.

A.1.2   Interferometer - If the interferometer is air-driven, adjust the interferometer drive air pressure to manufacturer's specifications.

A.1.3   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.

A.1.4   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.

A.2   Centerburst Intensity and MCT Detector Check

A.2.1   With an oscilloscope, check the MCT 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 5 day period.

A.2.2   If the centerburst intensity is 75 percent or less of the mean intensity of the plot maximum obtained by the procedure, 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.

A.2.3   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.

A.3   GC/FT-IR Sensitivity

A.3.1   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, the injector at 225°C and the GC detector 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).

A.3.2   One Hundred Percent Line Test - Set the GC/FT-IR operating conditions to those employed for the Sensitivity Test (see Sec. A.3.1). 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 less than or equal to 0.15 percent. Store this plot for future reference.

A.3.3   If the GC/FT-IR was purchased before 1985, there may be a temperature effect at the interface. To account for this, 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. A.2.3). Note that modern GC/FT-IR interfaces (1985 and later) may have eliminated most of this temperature effect.

A.4   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.

A.5   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.