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Method 1671, Revision A: Volatile Organic Compounds Specific to the Pharmaceutical Manufacturing Industry by GC/FID

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1.0 Scope and Application

1.1 This method is for surveying and monitoring under the Clean Water Act. The method is used to determine certain non-purgeable volatile organic pollutants specific to the pharmaceutical manufacturing industry (PMI) that are amenable to direct aqueous injection gas chromatography (GC) and detection by a flame ionization detector (FID).

1.2 The PMI analytes listed in Table 1 may be determined in waters, soils, and municipal sludges by this method.

1.3 The detection limits of Method 1671 are usually dependent on the level of interferences rather than instrumental limitations. The minimum levels (MLs) in Table 2 are the level that can be attained with no interferences present.

1.4 This method is recommended for use by, or under the supervision of, analysts experienced in the operation of gas chromatographs and in the interpretation of chromatograms.

1.5 This method is performance-based. The analyst is permitted to modify the method to overcome interferences or to lower the cost of measurements, provided that all performance criteria in this method are met. The requirements for establishing method equivalency are given in Section 9.2.

2.0 Summary of the Method

2.1 The percent solids content of the sample is determined. If the solids content is less than 1%, an internal standard(s) is added to a 5-mL sample. If the solids content of the sample is greater than 1%, 5 mL of reagent water and an internal standard(s) is added to a 5-g aliquot of sample.

The mixture is sonicated in a centrifuge tube with little or no headspace for 5 minutes. During this period the analytes and the internal standard will equilibrate between the solid and aqueous phases. In some cases, additional sonication will be necessary to establish equilibrium. The resulting suspension is centrifuged and the supernatant liquid analyzed.

2.2 An appropriate amount of the aqueous solution (or supernate) is injected into the GC. The compounds are separated by the GC and detected by the FID.

3.0 Definitions

There are no definitions specific to this method.

4.0 Interferences

4.1 Impurities in the carrier gas, organic compounds outgassing from the GC plumbing, and solvent vapors in the laboratory account for the majority of contamination problems encountered with this method. The analytical system is demonstrated to be free from interferences under conditions of the analysis by analyzing reagent water blanks initially and with each sample batch (samples analyzed on the same 12-hour shift), as described in Section 9.4.

4.2 Samples can be contaminated by diffusion of volatile organic compounds through the bottle seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol may serve as a check on such contamination.

4.3 Contamination by carryover can occur when high-level and low-level samples are analyzed sequentially. To reduce carryover, the syringe is cleaned or replaced with a new syringe after each sample is analyzed. When an unusually concentrated sample is encountered, it is followed by analysis of a reagent water blank to check for carryover. Syringes are cleaned by washing with soap solution, rinsing with tap and distilled water, and drying in an oven at 100-125°C. Other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4.4 Interferences resulting from samples will vary considerably from source to source, depending on the diversity of the site being sampled.

5.0 Safety

The toxicity or carcinogenicity of each analyte, compound, or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets should also be made available to all personnel involved in these analyses. Additional information on laboratory safety can be found in References 2-4.

6.0 Equipment and Supplies

6.1 Sample bottles and septa

6.1.1 Bottles**S**25- to 40-mL with polytetrafluoroethylene (PTFE)-lined screw-cap (Pierce 13075, or equivalent). Detergent wash, rinse with tap and distilled water, and dry at >105°C for a minimum of 1 hour before use.

6.1.2 Septa**S**PTFE-faced silicone (Pierce 12722, or equivalent), cleaned as above and baked at 100-200°C for a minimum of 1 hour.

6.2 Gas chromatograph**S**Shall be linearly temperature programmable with initial and final holds, and shall produce results which meet the calibration (Section 10), quality assurance (Section 9), and performance tests (Section 13) of this method.

6.2.1 Column**S**30 m long × 0.32 mm i.d. fused-silica microbore column coated with 4-µm of bonded poly(dimethylpolysiloxane) (Supelco SPB-1 Sulfur, or equivalent).

6.2.2 GC operating conditions.

Temperatures:

Column**S**2 minutes at 40°C, 10°C per minute to 180°C.

Injection port**S**200°C

FID**S**300°C

Carrier gas**S**Hydrogen at a head pressure of 10 psig.

An injector split may be used in order to optimize peak shape and repeatability.

6.3 Syringes**S**5-mL, gas-tight glass hypodermic, with Luer-lok tips.

6.4 Micro syringes**S**10-, 25-, and 100-µL.

6.5 Syringe valves**S**2-way with Luer ends, PTFE.

6.6 Bottles**S**15-mL, screw-cap with PTFE liner.

6.7 Balances.

6.7.1 Analytical, capable of weighing 0.1 mg.

6.7.2 Top-loading, capable of weighing 10 mg.

6.8 Equipment for determining percent moisture.

6.8.1 Oven, capable of being temperature-controlled at 110°C (±5°C).

6.8.2 Desiccator.

6.8.3 Beakers**S**50-, 100-mL.

6.9 Centrifuge apparatus.

6.9.1 Centrifuge capable of rotating 10-mL centrifuge tubes at 5000 rpm.

6.9.2 Centrifuge tubes, 10-mL, with screw-caps (PTFE-lined) to fit centrifuge.

6.10 Sonication apparatus capable of sonicating 10-mL centrifuge tubes and thoroughly agitating contents.

7.0 Reagents and Standards

7.1 Reagent water: Water in which the compounds of interest and interfering compounds are not detected by this method. It may be generated by any of the following methods:

7.1.1 Activated carbon**S**pass tap water through a carbon bed (Calgon Filtrasorb-300, or equivalent).

7.1.2 Water purifier**S**Pass tap water through a purifier (Millipore Super Q, or equivalent).

7.1.3 Boil and purge**S**Heat tap water to between 90 and 100°C and bubble contaminant-free inert gas through it for approximately 1 hour. While still hot, transfer the water to screw-cap bottles and seal with a PTFE-lined cap.

7.2 Sodium thiosulfate**S**ACS granular.

7.3 Standard solutions**S**Purchased as solutions or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If compound purity is 96% or greater, the weight may be used without correction to calculate the concentration of the standard.

7.3.1 Place approximately 8 mL of reagent water in a 10-mL ground-glass-stoppered volumetric flask. Allow the flask to stand unstoppered for approximately 10 minutes or until all wetted surfaces have dried. For each analyte, weigh the stoppered flask, add the compound, restopper, then immediately reweigh to prevent evaporation losses from affecting the measurement.

7.3.2 Liquids**S**Using a microsyringe, add sufficient liquid (about 100 mg) so that the final solution will have a concentration of about 10 mg/mL.

7.3.3 Gases**S**Fill a valved 5-mL gas-tight syringe with the compound. Lower the needle to approximately 5 mm above the meniscus. Slowly introduce the compound above the surface

of the meniscus. The gas will dissolve in the solvent. Repeat if necessary to reach desired concentration.

7.3.4 Fill the flask to volume, stopper, then mix by inverting several times. Calculate the concentration in milligrams per milliliter (mg/mL, equivalent to micrograms per microliter [µg/µL]) from the weight gain.

7.3.5 Transfer the stock solution to a PTFE-sealed screw-cap bottle. Store, with minimal headspace, in the dark at approximately 4°C. Do not freeze.

7.3.6 Replace standards after one month, or sooner if comparison with check standards indicate a change in concentration. Quality control check standards that can be used to determine the accuracy of calibration standards may be available from the National Institute of Standards and Technology, Gaithersburg, MD.

7.4 Secondary standards**S**Using standard solutions (Section 7.3), prepare a secondary standard to contain each pollutant at a concentration of 100 mg/L or 500 mg/L for compounds with higher MLs. Where necessary, a concentration of 1000 mg/L may be used.

7.4.1 Aqueous calibration standards**S**Using a syringe or a microsyringe, add sufficient secondary standard (Section 7.4) to five reagent water aliquots to produce concentrations in the range of interest.

7.4.2 Aqueous performance standard**S**An aqueous standard containing all pollutants and internal standard(s) is prepared daily, and analyzed each shift to demonstrate performance (Section 13). This standard shall contain concentrations of pollutants and internal standard(s), as appropriate, within a factor of 1 to 5 times the MLs of the pollutants listed in Table 1. It may be one of the aqueous calibration standards described in Section 7.4.1.

8.0 Sample Collection, Preservation, and Handling

8.1 Grab samples are collected in glass containers having a total volume greater than 20 mL. For aqueous samples that pour freely, fill sample bottles so that no air bubbles pass through the sample as the bottle is filled and seal each bottle so that no air bubbles are entrapped. Maintain the hermetic seal on the sample bottle until time of analysis.

8.2 Maintain samples at 4°C from the time of collection until analysis. Do not freeze. If an aqueous sample contains residual chlorine, add sodium thiosulfate preservative (10 mg/40 mL) to the empty sample bottles just prior to shipment to the sample site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine (Reference 5). If preservative has been added, shake the bottle vigorously for 1 minute immediately after filling.

8.3 For aqueous samples, experimental evidence indicates that some PMI analytes are susceptible to rapid biological degradation under certain environmental conditions. Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days. For this reason, a separate sample should be collected, acidified, and analyzed when compounds susceptible to rapid biological degradation are to be determined. Collect about 500 mL of sample in a clean container. Adjust the pH of the sample to about 2 by adding hydrochloric acid (1:1) while stirring. Check pH with narrow range (1.4 to 2.8) pH paper. Fill a sample bottle as described in Section 8.1. If residual chlorine is present, add sodium thiosulfate to a separate sample bottle and fill as in Section 8.1.

8.4 All samples shall be analyzed within 14 days of collection.

9.0 Quality Assurance/Quality Control

9.1 Each laboratory that uses this method is required to operate a formal quality assurance program (Reference 6). The minimum requirements of this program consist of an initial demonstration of laboratory capability (Section 9.5) and analysis of standards (Sections 9.6 and 13) and blanks (Section 9.4) as tests of continued performance. Each time a batch of samples is analyzed or there is a change in reagents or procedures, a method blank must be analyzed as a safeguard against contamination.

9.2 In recognition of advances that are occurring in analytical technology, and to allow the analyst to overcome sample matrix interferences, the analyst is permitted certain options to improve separations or lower the costs of measurements. These options include alternative concentration and cleanup procedures, and changes in columns and detectors. Alternative techniques, such as the substitution of spectroscopy or immunoassay, and changes that degrade method performance are not allowed. If an analytical technique other than the techniques specified in this method is used, that technique must have a specificity equal to or better than the specificity of the techniques in this method for the analytes of interest.

9.2.1 If the detection limit of the method will be affected by the change, the laboratory is required to demonstrate that the method detection limit (MDL; 40 CFR 136, Appendix B) is lower than one-third the regulatory compliance level. If calibration will be affected by the change, the analyst must recalibrate the instrument per Section 10.

9.2.2 The laboratory is required to maintain records of modifications made to this method. These records include the information in this subsection, at a minimum.

9.2.2.1 The names, titles, addresses, and telephone numbers of the analyst(s) who performed the analyses and modification, and of the quality control officer who witnessed and will verify the analyses and modification.

9.2.2.2 A listing of pollutant(s) measured, by name and CAS Registry Number.

9.2.2.3 A narrative stating the reason(s) for the modification.

9.2.2.4 Results from all quality control (QC) tests comparing the modified method to this method including:

(a) calibration (Section 10);
(b) calibration verification (Section 13);
(c) initial precision and accuracy (Section 9.5);
(d) analysis of blanks (Section 9.4); and
(e) accuracy assessment (Section 9.6 and 13).

9.2.2.5 Data that will allow an independent reviewer to validate each determination by tracing the instrument output (peak height, area, or other signal) to the final result. These data are to include:

(a) sample numbers and other identifiers;
(b) analysis dates and times;
(c) injection logs;
(d) analysis sequence/run chronology;
(e) sample weight or volume;
(f) sample volume prior to each cleanup step, if applicable;
(g) sample volume after each cleanup step, if applicable;
(h) final sample volume prior to injection;
(I) injection volume;
(j) dilution data, differentiating between dilution of a sample or an extract;
(k) instrument and operating conditions;
(l) column (dimensions, liquid phase, solid support, film thickness, etc.);
(m) operating conditions (temperature, temperature program, flow rates, etc.);
(n) detector (type, operating condition, etc.);
(o) chromatograms, printer tapes, and other recording of raw data; and
(p) quantitation reports, data system outputs, and other data necessary to link raw data to the results reported.

9.3 With each sample batch, a matrix spike (MS) and matrix spike duplicate (MSD) are analyzed to assess precision and accuracy of the analysis. The relative percent difference (RPD) between the MS and MSD shall be less than 30% and compound recoveries shall fall within the limits specified in Table 3. If the recovery of any compound falls outside its warning limit, method performance is unacceptable for that compound in that sample and the results may not be reported for regulatory compliance purposes.

9.4 Analyses of blanks are required to demonstrate freedom from contamination and that the compounds of interest and interfering compounds have not been carried over from a previous analysis (Section 4.3).

9.4.1 With each sample batch (samples analyzed on the same 12-hour shift), a blank shall be analyzed immediately after analysis of the aqueous performance standard (Sections 9.6 and 13) to demonstrate freedom from contamination. If any of the compounds of interest or any potentially interfering compound is found in a blank at greater than the ML (assuming a response factor of 1 relative to the nearest-eluted internal standard for compounds not listed in Table 1), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

9.5 Initial precision and recovery—To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations for compounds to be calibrated.

9.5.1 Analyze two sets of four 5-mL aliquots (eight aliquots total) of the aqueous performance standard (Section 7.4.2) containing the PMI analytes listed in Table 1.

9.5.2 Using the first set of four analyses, compute the average recovery (X) in percent of spike level and standard deviation of the recovery (s) in percent of spike level, for each compound.

9.5.3 For each compound, compare s and X with the corresponding limits for initial precision and accuracy found in Table 3. If s and X for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If, however, any individual s exceeds the precision limit or any individual X falls outside the range for accuracy, system performance is unacceptable for that compound.

9.5.4 Using the results of the second set of analyses, compute s and X for only those compounds that failed the test of the first set of four analyses (Section 9.5.3). If these compounds now pass, the system performance is acceptable for all compounds, and analysis of blanks and samples may begin. If, however, any of the same compounds fail again, the analysis system is not performing properly for the compound(s) in question. In this event, correct the problem and repeat the entire test (Section 9.5).

9.6 The laboratory shall, on an ongoing basis, demonstrate through the analysis of the aqueous performance standard (Section 7.4.2) that the analysis system is in control. This procedure is described in Section 13.

9.7 Where available, field replicates may be used to validate the precision of the sampling technique.

9.8 The laboratory shall maintain records to define the quality of data that is generated.

10.0 Calibration

10.1 Inject standards into the GC and adjust the sensitivity to detect an amount of each compound less than or equal to one-third of the ML listed in Table 2 for the analyte.

10.2 Internal standard calibration procedure. The analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard(s) is not affected by method or matrix interferences. Because of these limitations, no internal standard that would be applicable to all samples can be required in the method. The method was developed using tetrahydrofuran (THF) as an internal standard. Where THF is not present in the sample matrix and no interference precludes its use, THF is to be used as an internal standard for application of this method. If interferences preclude use of THF and other internal standards, external standard calibration may be used.

10.2.1 Prepare aqueous calibration standards at a minimum of five concentration levels for each analyte by carefully adding an appropriate amount of secondary standard to reagent water or to the matrix under study. One of the concentrations should be at or below the ML. The concentration range should bracket the concentrations expected in the samples and should not exceed the dynamic range of the GC/FID instrument. These aqueous standards must be prepared daily.

10.2.2 Prepare a spiking solution containing the internal standard(s) using the procedures described in Sections 7.3 and 7.4 and add an appropriate amount of internal standard to each aqueous calibration standard.

10.2.3 Using injections appropriate to optimize system sensitivity and separation of the analytes, analyze each calibration standard and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:
A_s = *Response for the analyte to be measured*
A_{is} = *Response for the nearest eluting internal standard*
C_{is} = *Concentration of the nearest eluting internal standard*
C_s = *Concentration of the analyte to be measured*

If the RF value over the working range is a constant (less than 10% relative standard deviation), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of relative response, A_s*C_{is}/A_{is}, against analyte concentration (C_s).

11.0 Sample Preparation

Samples containing less than 1% solids are analyzed directly as aqueous samples. Samples containing 1% solids or greater are analyzed after equilibration with reagent water containing internal standard(s).

11.1 Determination of percent solids.

11.1.1 Weigh 5-10 g of sample into a tared beaker.

11.1.2 Dry overnight (12 hours minimum) at 110±5°C, and cool in a desiccator.

11.1.3 Determine the percent solids as follows:

$$\%\ solids = \frac{weight\ of\ sample\ dry}{weight\ of\ sample\ wet}\ x\ 100$$

11.2 Remove standards and samples from cold storage and bring to 20-25 °C.

11.3 Samples containing less than 1% solids.

11.3.1 Allow solids to settle and remove 5 mL of sample.

11.3.2 Add an appropriate amount of internal standard spiking solution.

11.3.3 Inject a sample directly into the GC. The temperature of the injection block should be great enough to immediately vaporize the entire sample. An example of the separations achieved by the column listed is shown in Figure 1.

Note: Use of a 0.2-µL injection has been found to improve method sensitivity over a larger injection combined with a split sample. Where possible, splitless injection should be used. All requirements of this Method must be met regardless of type of injection used.

11.4 Samples containing 1% solids or greater.

11.4.1 Mix the sample thoroughly using a clean spatula and remove rocks, twigs, sticks, and other foreign matter.

11.4.2 Add 5±1 g of sample to a tared 10-mL centrifuge tube. Using a clean metal spatula, break up any lumps of sample. Record the sample weight to three significant figures.

11.4.3 Add an appropriate amount of internal standard spiking solution to the sample in the centrifuge tube.

11.4.4 Add a measured quantity (Y ± 0.1 mL) of reagent water to the tube so as to minimize head space.

11.4.5 Place a cap on the centrifuge tube leaving little or no headspace. Place the tube in the sonicator for a minimum of 5 minutes, turning occasionally. For most samples this should be sufficient time to distribute the analytes and standard(s) between the solid and aqueous phases and to establish equilibrium. Some sample matrices may require more sonication.

11.4.6 On completion of sonication, centrifuge the sample and inject the same amount of supernate into the GC that was injected for the calibration standards.

11.5 For liquid samples containing high-solids concentrations, such as sludges or muds, weigh approximately 5 g (to three significant figures) into a 10-mL centrifuge tube, add an appropriate amount of internal standard solution, sonicate, centrifuge, and inject as in Section 11.4.6.

12.0 Quantitative Determination

12.1 The calibration curve or averaged response factor determined during calibration is used to calculate the concentration. For calculation using the averaged RF, the equation below is used, and the terms are as defined in Section 10.2.3.

$$Concentration = \frac{A_s \times C_{is}}{A_{is} \times RF}$$

12.2 The concentration of the pollutant in the solid phase of the sample is computed using the concentration of the pollutant detected in the aqueous solution, as follows:

$$Concentration\ in\ solid\ (mg/kg) = \frac{YL \times aqueous\ conc\ (mg/L)}{sample\ wt\ (kg)} \times percent\ solids \times DF$$

where:
percent solids is from Section 11.1
Y = Volume of water in liters (L) from 11.4.4
DF = Dilution factor (as a decimal number), if necessary

12.3 Sample dilution—If the calibration range of the system is exceeded, the sample is diluted by successive factors of 10 until the sample concentration is within the calibration range.

12.4 Report results for all pollutants found in standards, blanks, and samples to three significant figures. For samples containing less than 1% solids, the units are milligrams per liter (mg/L); and for samples containing 1% solids or greater, units are milligrams per kilogram (mg/kg).

13.0 System Performance

13.1 At the beginning of each 12-hour shift during which analyses are performed, system calibration and performance shall be verified. Acceptance criteria for each compound (R) are found in Table 3. Adjustment and/or recalibration shall be performed until all performance criteria are met. Only after all performance criteria are met may blanks and samples be analyzed.

13.2 Where THF is used as the internal standard, the absolute retention time of THF shall be 416 seconds (± 30 seconds). The relative retention times of all pollutants shall fall within 10% of the value given in Table 2.

14.0 Method Performance

14.1 This method was developed and validated in a single laboratory.

14.2 A chromatogram of the aqueous performance standard is shown in Figure 1.

15.0 Pollution Prevention

15.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Many opportunities for pollution prevention exist in laboratory operation. 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 reduced feasibly at the source, the Agency recommends recycling as the next best option. The acids used in this Method should be reused as practicable by purifying by electrochemical techniques. The only other chemicals used in this Method are the neat materials used in preparing standards. These standards are used in extremely small amounts and pose little threat to the environment when managed properly. Standards should be prepared in volumes consistent with laboratory use to minimize the disposal of excess volumes of expired standards.

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

16.0 Waste Management

16.1 It is the laboratory's responsibility to comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land-disposal restrictions. In addition, it is the laboratory's responsibility to protect air, water, and land resources by minimizing and controlling all releases from fume hoods and bench operations. Also, compliance is required with any sewage discharge permits and regulations.

16.2 Samples containing acids at a pH of less than 2 are hazardous and must be neutralized before being poured down a drain or must be handled as hazardous waste.

16.3 For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* and *Less is Better: Laboratory Chemical Management for Waste Reduction*, both available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street NW, Washington, DC 20036.

17.0 References

1. "Standard Test Method for Volatile Alcohols in Water by Direct Aqueous-Injection Gas Chromatography." 1994 Annual Book of ASTM Standards, Volume 11.02 (Water (II)). ASTM, 1916 Race Street, Philadelphia, PA 19103-1187.

2. "Working with Carcinogens," DHEW, PHS, NIOSH, Publication 77-206 (1977).

3. "OSHA Safety and Health Standards, General Industry," 29 CFR 1910, OSHA 2206 (1976).

4. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety (1979).

5. "Methods 330.4 and 330.5 for Total Residual Chlorine," USEPA, EMSL Cincinnati, OH 45268.

6. "Method of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL Cincinnati, OH 45268, EPA-4-79-019 (March, 1979).

7. Technical Report to PhRMA from Tichler & Kocurek by Malcolm Pirnie Laboratory, EPA Water Docket for Pharmaceutical Manufacturing Industry rule proposed May 2, 1995 (60 FR 21592), Document Control Number 8166 at Record Section 13.2.4. (February 13, 1997).

18.0 Tables

Table 1. Non-purgeable Water Soluble PMI Analytes to be Analyzed by Direct Aqueous Injection GC/FID and Internal Standard Techniques

PMI Analyte	CASRN[1]	EPA-EGD
Acetonitrile	75-05-8	972
Diethylamine	109-89-7	986
Dimethylamine	124-40-3	987
Dimethyl sulfoxide	67-68-5	1037
Ethanol	64-17-5	134
Ethylene glycol	107-21-1	1038
Formamide	75-12-7	988
Methanol	67-56-1	135
Methylamine	74-89-5	989
Methyl Cellosolve® (2-methoxyethanol)	109-86-4	1040
n-Propanol	71-23-8	955
Triethylamine	121-44-8	990

[1] Chemical Abstracts Service Registry Number

Table 2. Gas Chromatographic Retention Times and Minimum Levels for Non-purgeable Water Soluble PMI Analytes by Direct Aqueous Injection GC/FID

EGD No.	PMI Analyte	Retention Time			ML[1]
		Mean (sec)	EGD Ref	Relative	(mg/L)
989	Methylamine	128	975	0.307	50
135	Methanol	139	975	0.334	2[(2)]
987	Dimethylamine	165	975	0.396	50
134	Ethanol	188	975	0.452	2[(2)]
972	Acetonitrile	203	975	0.488	50
955	n-Propanol	307	975	0.737	50
986	Diethylamine	341	975	0.819	50
975	Tetrahydrofuran (int std)	416	975	1.000	
1040	Methyl Cellosolve®	429	975	1.030	20
988	Formamide	473	975	1.136	100
1038	Ethylene glycol	495	975	1.189	100
990	Triethylamine	518	975	1.244	50
1037	Dimethyl sulfoxide	676	975	1.624	20

[1] This is a minimum level at which the entire analytical system shall give an acceptable calibration point, taking into account method-specific sample and injection volumes. The concentration in the aqueous or solid phase is determined using the equations in Section 12.

[2] The minimum level for this analyte was developed from data provided in Reference 7.

Table 3. Acceptance Criteria for Performance Tests

			Acceptance Criteria (% of Spike Level)		
			Initial Precision and Accuracy		On-going Accuracy
EGD No.	**PMI Analyte**	**Spike Level**	**s**	**X**	**R**
972	Acetonitrile	50	30	70 - 146	70 - 148
986	Diethylamine	50	20	65 - 130	70 - 130
987	Dimethylamine	50	27	70 - 153	70 - 155
1037	Dimethyl sulfoxide	50	20	31 - 130	30 - 130
134	Ethanol	50	20	70 - 131	70 - 132
1038	Ethylene glycol	100	22	70 - 149	70 - 150
988	Formamide	200	20	70 - 130	70 - 130
135	Methanol	50	21	70 - 130	70 - 130
989	Methylamine	50	20	70 - 130	70 - 130
1040	Methyl Cellosolve®	50	20	64 - 130	64 - 130
955	n-Propanol	50	25	70 - 137	70 - 139
990	Triethylamine	50	47	70 - 165	68 - 168

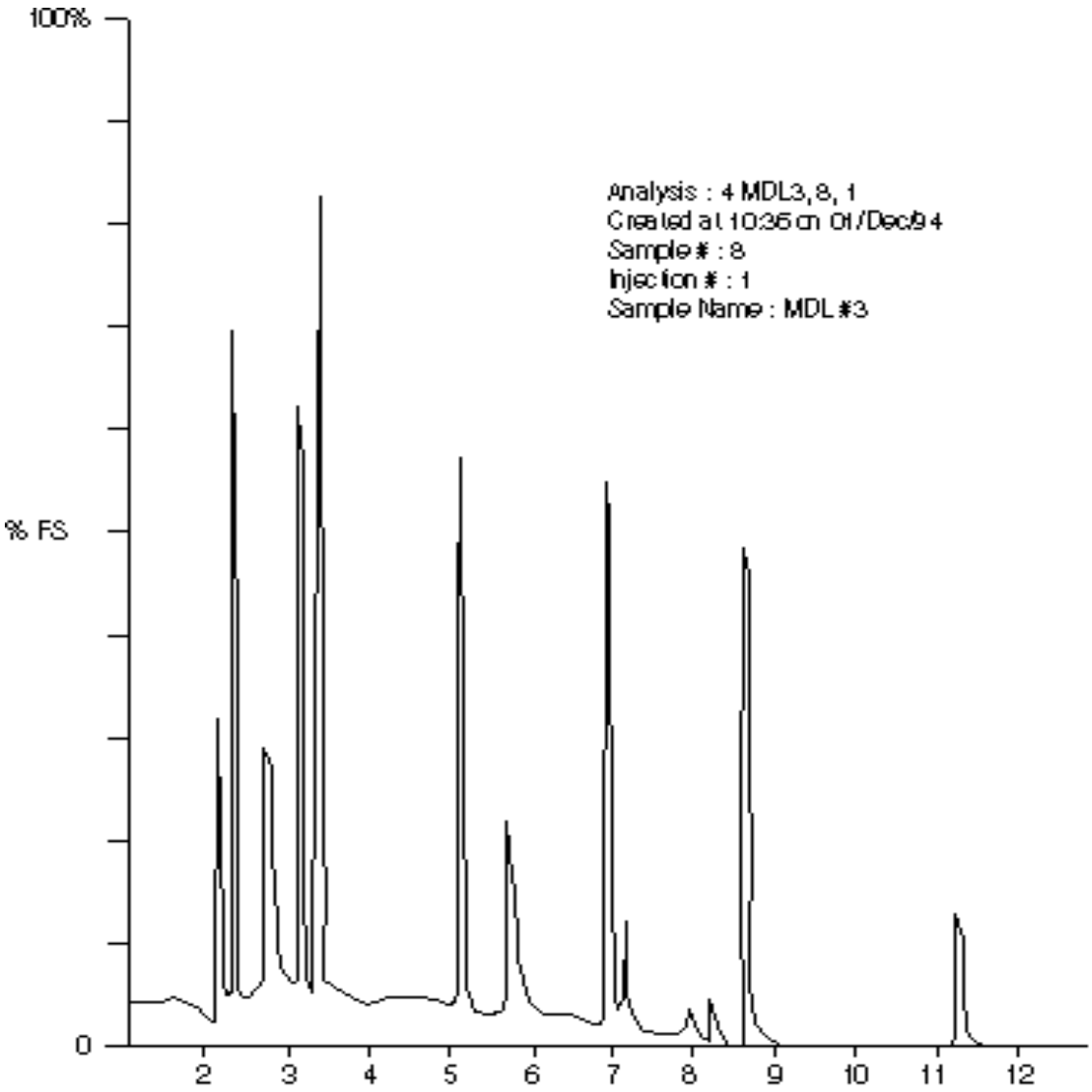

Figure 1. Chromatogram of Aqueous Performance Standard of Analytes from Table 1