



Designation: D5830 – 22

Standard Test Method for Solvents Analysis in Hazardous Waste Using Gas Chromatography¹

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1. Scope

1.1 This test method is used to determine qualitatively and quantitatively the presence of the following compounds in waste samples using gas chromatography. This test method is intended for use as a screening method with a typical reporting level of 0.1 %.

| | |
|---------------------------------------|--------------------------|
| Dichodifluoromethane | Tetrahydrofuran |
| Trichlorofluoromethane | Acetone |
| 1,1,2-Trichloro-1,2,2-trifluoroethane | Methyl Ethyl Ketone |
| Methanol | MIBK |
| Ethanol | Cyclohexanone |
| Isopropanol | Ethyl Acetate |
| <i>n</i> -Propanol | Propyl Acetate |
| Isobutanol | Butyl Acetate |
| <i>n</i> -Butanol | Benzene |
| <i>tert</i> -Butanol | Toluene |
| Methylene Chloride | Ethylbenzene |
| Chloroform | Xylenes |
| Carbon Tetrachloride | Styrene |
| 1,1-Dichloroethane | Chlorobenzene |
| 1,2-Dichloroethane | Dichlorobenzenes |
| 1,2-Dichloropropane | Nitrobenzene |
| 1,1-Dichloroethylene | Fluorobenzene |
| 1,2-Dichloroethene | <i>n</i> -Propyl Benzene |
| 1,1,1-Trichloroethane | Isopropyl Benzene |
| Tetrachloroethylene | Isobutyl Benzene |
| Trichloroethylene | <i>n</i> -Butyl Benzene |
| Tetrachloroethane | 2-Ethoxyethanol |
| Cyclopentane | 2-Butoxyethanol |
| Pentane | 2-Ethoxyethanol Acetate |
| Hexane | 2-Methoxyethanol |
| Heptane | Bromoform |
| Cyclohexane | Carbitol |
| <i>Is</i> ooctane | Ethyl Ether |
| Nitropropane | 1,4-Dioxane |
| Ethanolamine | Diacetone Alcohol |
| Nitromethane | Acetonitrile |
| Ethylene Chloride | Pyridine |
| Benzyl Chloride | Toluidine |
| | Ethylene Glycol |
| | Propylene Glycol |

1.1.1 This compound list is a compilation of hazardous solvents and other constituents that are commonly seen in hazardous waste samples.

¹ This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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1.2 The scope of this test method may be expanded to include other volatile and semivolatile organic constituents such as but not limited to those described below, provided the intended use data quality objectives including sampling, recovery, and analytic data quality are demonstrated as satisfied by the user.

1.2.1 Hydrocarbon mixtures such as kerosene and mineral spirits.

1.2.2 High-boiling organics, defined here as compounds which boil above *n*-Hexadecane.

1.2.3 Other organics that the analyst is able to identify, either through retention time data or gas chromatography/mass spectrometric (GC/MS) analysis.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

[D1193 Specification for Reagent Water](#)

[D4547 Guide for Sampling Waste and Soils for Volatile Organic Compounds](#)

[D4687 Guide for General Planning of Waste Sampling](#)

[D5013 Practices for Sampling Wastes from Pipes and Other Point Discharges](#)

[D5681 Terminology for Waste and Waste Management](#)

[D5743 Practice for Sampling Single or Multilayered Liquids, with or Without Solids, in Drums or Similar Containers](#)

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

2.2 EPA Documents:³

Test Method 8000D Determinative Chromatographic Separations, SW-846, Revision 5, 2018

Test Method 8015D Nonhalogenated Organics Using GC/FID, SW-846, Revision 4, 2003

Test Method 8021B Aromatic and Halogenated Volatiles By Gas Chromatography Using Photoionization and/or Electrolytic Conductivity Detectors, SW-846, Revision 3, 2014

Test Method 8260D Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW-846, Revision 4, 2018

3. Terminology

3.1 *Definitions*—For definitions of terms used in this standard, refer to Terminology **D5681**.

4. Summary of Test Method

4.1 Waste samples are analyzed by direct injection, or by carbon disulfide, *M*-Pyrol, or other suitable solvent extraction and injection of the extract into a gas chromatograph. Detection is achieved using a detector which is specific for the needed application, for example, flame ionization detector (FID), electron capture detector (ECD), thermal conductivity detector (TCD), photoionization detector (PID), or mass selective detector (MSD). This test method may be expanded to utilize other detector types not previously mentioned.

5. Significance and Use

5.1 This test method is useful in identifying the common solvent constituents in hazardous waste samples. This test method is designed to support field or site assessments, recycling operations, plant operations, or pollution control programs.

6. Interferences

6.1 Interferences may be encountered from any number of organic compounds to which a specific detector responds. Also, closely eluting components may complicate identification based solely on retention time. When these types of interferences are encountered, the analyst must rely on other sources of information for positive identification, such as:

6.1.1 GC/MS confirmation; see EPA Method 8260D, direct injection technique.

6.1.2 Gas chromatography with an analyte class selective detector such as PID or electrolytic conductivity (ELCD) detectors or both; see EPA Method 8021B.

6.1.3 Use of confirmation column, or confirmatory detector.

6.1.3.1 This method identifies one column (DB-1701) and one detector (FID) and utilizes three solvent standards and one QC daily check. Use of confirmatory columns or detectors, or both, will also require the use of the three solvent standards (see **Note 2, 9.1**) and QC daily check, one for each confirma-

tory column or detector, or both. EPA Method 8000D provides useful guidance on calibration and QC requirements applicable to this activity.

6.1.4 Use of varying temperature programs or standard comparison, or both.

6.1.4.1 Use of varying analytical programs will also require the use of three solvent standards and QC daily check for each variation.

6.1.5 Sample history, for example, any information available from the waste generator.

6.1.6 Physical characteristics, for example, flammability, specific gravity, or miscibility with water.

6.2 Interferences may also be encountered from syringe carryover. Immediately following each injection, the syringe is thoroughly rinsed with carbon disulfide or *M*-Pyrol. Other solvents such as methanol may be used as rinse solvents if sample types necessitate their use, but carryover and possible interferences may occur if rinse solvent is still present in the syringe at reuse. Before each injection the syringe is rinsed with the sample to be injected, where the first two pumps are flushed into a waste receptacle.

6.3 When carbon disulfide (CS₂) is used to extract solids or sludges that contain significant amounts of water, low recovery of the water-miscible solvents may result.

6.4 Some grades of CS₂ may contain trace amounts of benzene.

6.5 *M*-Pyrol degrades with time. The degradation products interfere with some late eluting compounds on some columns (approximately five small peaks).

6.6 Interference from the CS₂ solvent peak may occur if using a TCD.

6.7 When using a TCD, water as well as oxygenated compounds such as MEK, MIBK, or both, may suppress detector response.

6.8 If an ELCD or ECD is used, CS₂, *M*-Pyrol (required for an ELCD), and high concentrations of halogenated compounds may overload and damage these detectors. It is recommended that these detectors be used only when very low detection levels of halogenated compounds are expected and direct injection of the sample is possible.

7. Apparatus

7.1 *Gas Chromatograph System*—Equipped with capillary or packed column injection ports, or both, detector, and data system.

7.2 *Suggested Chromatographic Columns*:

7.2.1 *Capillary*, microbore or megabore.

7.2.1.1 DB-1701, 30M by 0.25 mm inside diameter, 0.25 μm film thickness.

7.2.1.2 DB-624, 30M by 0.3 mm inside diameter, 1.8 μm film thickness.

7.2.2 *Packed: Stainless Steel or Glass*.

7.2.2.1 1 % SP-1000, 60/80 Carbowax B, 8 ft by 1/8 in. inside diameter.

7.2.2.2 10 % SP-2100, 100/120 Chromosorb WHP, 2M by 2 mm ID.

³ Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

NOTE 1—These columns are recommended as they have demonstrated acceptable results in many applications. Operating conditions for each are listed in Section 11. Equivalent or alternative columns may be used when they are demonstrated as fulfilling the needs of a particular application.

7.3 *Glass Screw-Cap Vials or Equivalent*—To collect samples and store standards. Polytetrafluoroethylene or other inert material should be used for the cap liner.

7.4 *Microsyringes*, 1.0, 10, and 100 μL .

7.5 *Analytical Balance*, accurate to 0.0001 g.

7.6 *Pipettes*, glass, disposable, or volumetric micropipettor or equivalent.

7.7 *Microdisk Filters*, 0.45, 1.0, or 5.0 μm , optional.

7.8 *Centrifuge*, optional.

7.9 *Vortex-Type Mixer*.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is determined that the reagent is of sufficiently high purity to retain an acceptable accuracy for the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D1193.

8.3 *Nitrogen or Helium (UHP)*—For carrier and makeup gases. Air and hydrogen (UHP) for fuel gases. Gases may be obtained from a gas generator providing gases of sufficient purity, or from commercially available high-purity gas cylinders.

8.4 *Carbon Disulfide, CS₂*—Chromatography grade.

8.5 *M-Pyrol, C₅H₉NO*—Available through chemical suppliers and sources as 1-methyl-2-pyrrolidone.

8.6 *Individual Standards for Each Component of Interest*—99 % purity, available from many vendors.

9. Standard Preparation

9.1 *Stock Standard Solutions*—Stock standards are prepared from pure standard materials. It is recommended that the standards be prepared so that each component is 5 to 10 % by weight. The stock standards must be prepared by directly weighing each component. For extremely volatile components such as ether and freons, it is recommended that a new stock standard be prepared daily or as needed. If a dilution solvent is needed when preparing the stock standards, use the same solvent used for sample extraction or dilution in Section 11.

NOTE 2—Due to the incompatibility of some standard compounds, such

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

as immiscible substances, or when a large number of compounds are determined, it is advisable to prepare three or four standard solutions each composed of ten to 15 compatible compounds. In addition, a set of standard chromatograms and a retention timetable are typically retained as references.

9.2 *Secondary Working Standards*—These are prepared from stock standard solutions using the appropriate solvent. Secondary standards should encompass the linear range of the GC system.

NOTE 3—Linear response and range shall be established with all detectors and chromatography systems used for quantitation. All calibration and sample analysis shall be done within the established linear range.

9.3 *Calibration Check Standard*—A calibration check standard is prepared. The standard mixture should provide a good overall check of the GC/detector system with respect to analytic accuracy and precision. These criteria are established prior to commencing sample analysis and are based upon the data quality needs of the investigation. The compounds selected should cover the major compound types, for example, alcohols, aromatics, aliphatics, ketones, and halogenates. A typical calibration check standard FID chromatogram is shown in Fig. 1.

10. Sample Collection, Preservation, and Handling

10.1 Sample collection should be in accordance with appropriate sampling protocols. Sampling procedure guidance for common waste sources can be found in Guides D4547 and D4687, and in Practices D5013 and D5743.

10.2 Samples should be collected in glass containers that have tightly sealing caps. If very volatile organics are of particular interest, the headspace in the container should be kept to a minimum.

10.3 *Sample Transfer Implements*—Implements are required to transfer portions of waste samples from the sample containers to the laboratory containers. Liquid samples may be transferred using disposable pipets. Solids and semisolids may be transferred using a conventional laboratory spatula.

10.4 Samples shall be handled maintaining safe laboratory practices. Any samples with special hazards must be appropriately labeled.

10.5 Unused sample material, laboratory dilutions, and waste from the samples may be regulated. Consult the appropriate specialist and/or local, state, provincial, or national regulations for guidance in the proper handling and disposal of laboratory wastes.

11. Procedure

11.1 *Sample Preparation:*

11.1.1 Analyze liquid matrices with relatively low viscosity using direct injection into a GC, either as received or after dilution with CS₂, M-Pyrol, or other suitable solvent.

11.1.2 Analyze solid or semisolid samples as follows:

11.1.2.1 For carbon disulfide or M-Pyrol preparation, weigh 3 g of the waste sample in a 15-mL glass vial. Add 3 g of carbon disulfide or M-Pyrol to the vial and the mixture is vortexed vigorously. After allowing the solids to settle, inject the CS₂ or M-Pyrol extract into the GC.

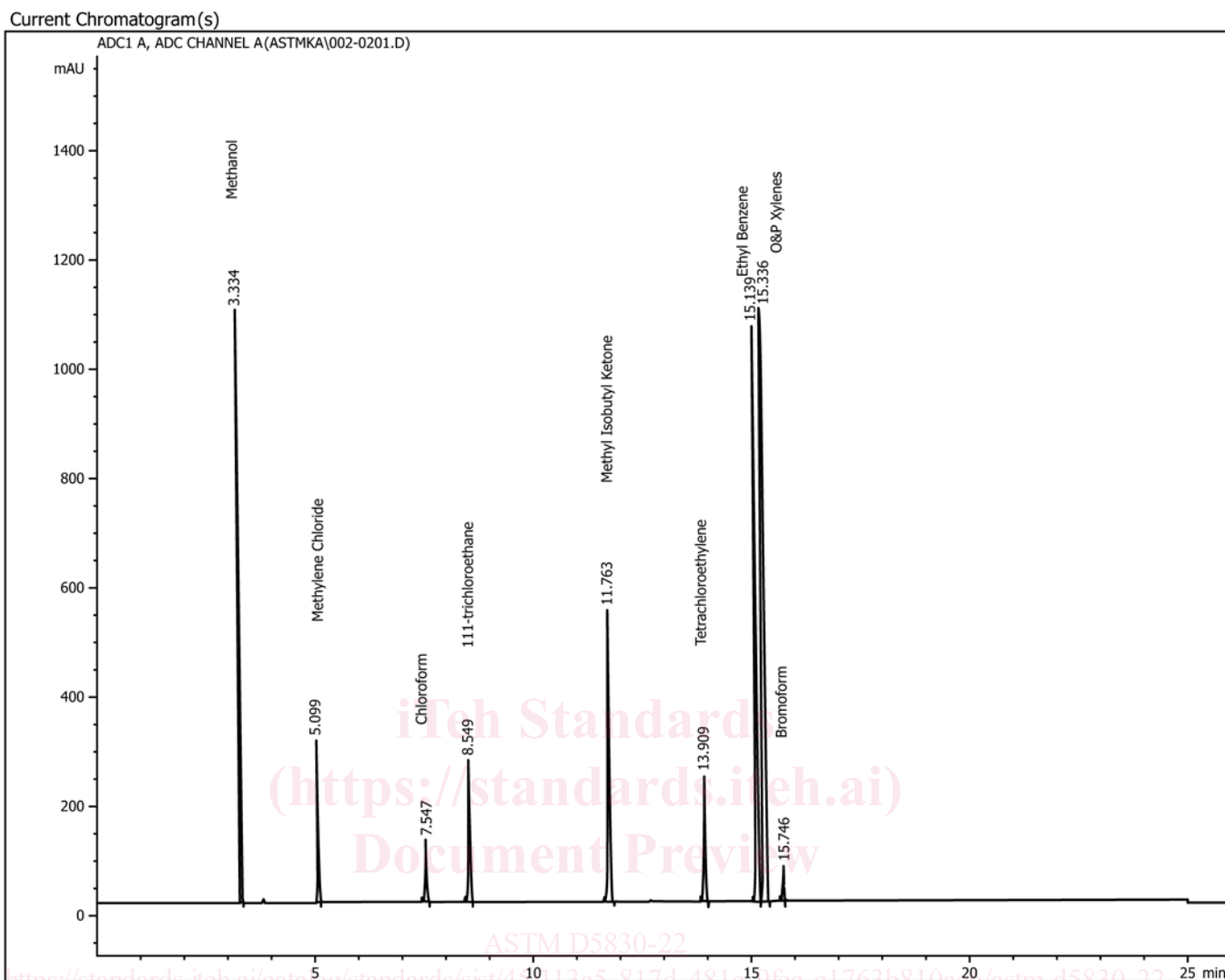


FIG. 1 Daily QC Standard FID/DB-1701

11.1.2.2 Use alternate sample sizes and extraction solvent weights if necessary. Actual sample size and solvent weight must be recorded in the appropriate sample preparation log book. It is essential for accurate waste sample analysis that sample size be sufficient to ensure a representative sample. If alternate sample size or extraction solvent volumes, or both, are used, this must be reflected in the calculations under the dilution factor in Section 12.

11.1.3 Multiple phases or layers are typically present in hazardous waste samples. Depending on treatment or process requirements, it may be necessary to analyze each phase or layer individually.

11.2 Suggested GC Operating Conditions—A number of GC operating parameters can be used to achieve determination of hazardous waste components by GC and any particular detector. The following conditions have been successfully used in hazardous waste VOC analysis by this standard.

11.2.1 For Capillary DB-1701 with FID:

| | |
|--------------------------|--------------------------|
| Column flow rate | 1–1.5 mL/min |
| Makeup gas flow rate | 30–60 mL/min |
| Split flow | 60 cm ³ /min |
| Injector temperature | 250 °C |
| Detector temperature | 250 °C |
| Airflow (FID) | Approximately 300 mL/min |
| Hydrogen flow (FID) | Approximately 30 mL/min |
| Initial oven temperature | 35 °C |
| Initial time | 6 min |
| Level 1 rate | 6 °C/min |
| Level 1 final value | 180 °C |
| Level 2 rate | 10 °C/min |
| Level 2 final value | 230 °C |
| Run time | 40 min |
| Threshold | 4 units |
| Peak width | 0.04 min |

NOTE 4—Typical chromatograms are shown in Figs. 2-5.

Current Chromatogram(s)

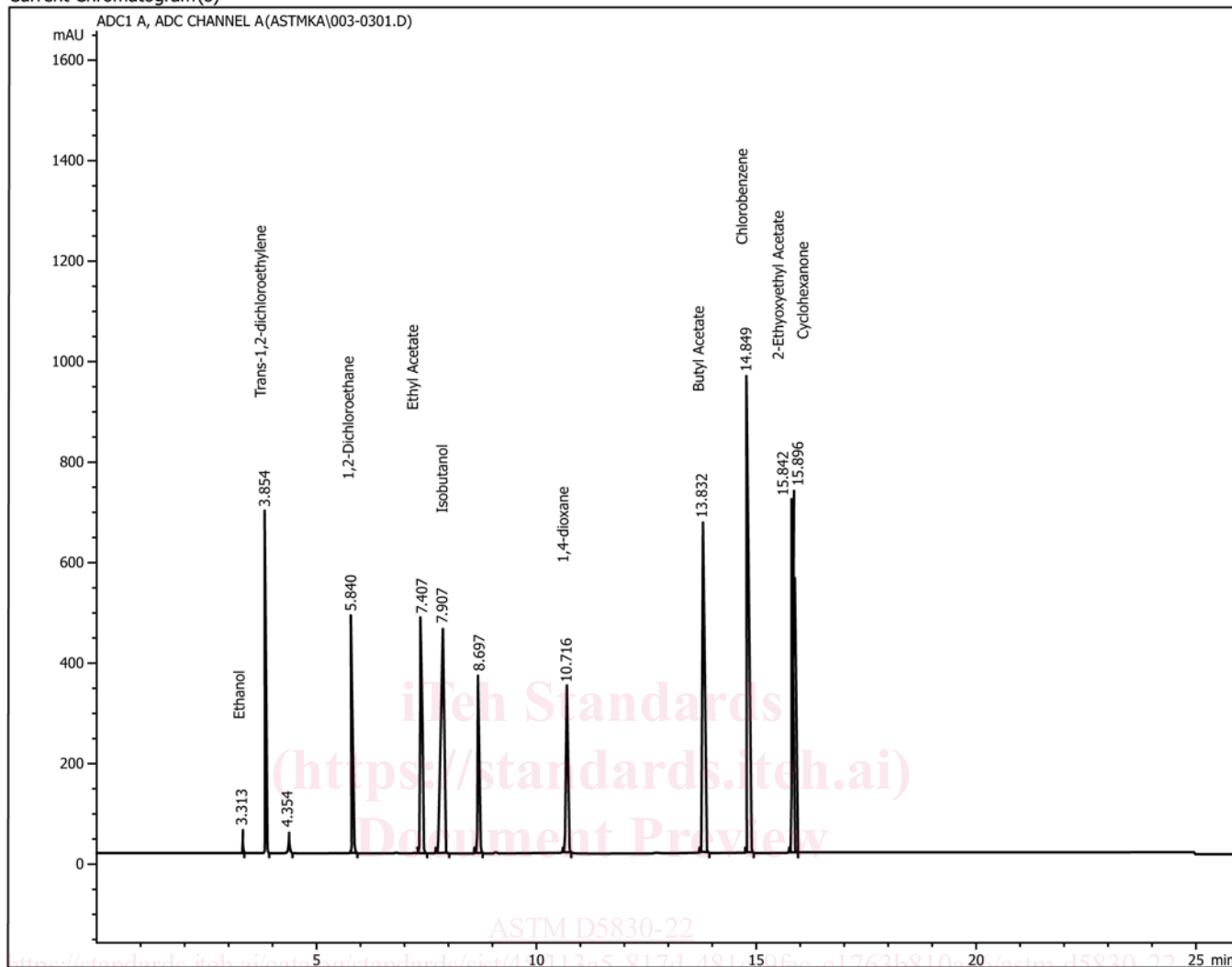


FIG. 2 Solvent Standard No. 1 FID/DB-1701

11.2.2 For Capillary DB-624 with FID:

| | |
|--------------------------|--------------------------|
| Column flow rate | 3.5 mL/min |
| Makeup gas flow | 29 mL/min |
| Airflow (FID) | Approximately 300 mL/min |
| Hydrogen flow (FID) | Approximately 30 mL/min |
| Injector temperature | 275 °C |
| Detector temperature | 275 °C |
| Initial oven temperature | 35 °C |
| Initial time | 5 min |
| Level 1 rate | 5 °C/min |
| Level 1 final value | 150 °C |
| Level 1 hold time | 4 min |
| Level 2 rate | 20 °C/min |
| Level 2 final value | 225 °C |
| Run time | 45 min |

11.2.3 For Packed SP-1000 with FID:

| | |
|--------------------------|-----------|
| Column flow rate | 40 mL/min |
| Air pressure (FID) | 300 kPa |
| Hydrogen pressure (FID) | 130 kPa |
| Injector temperature | 250 °C |
| Detector temperature | 250 °C |
| Initial oven temperature | 90 °C |
| Initial time | 6 min |
| Level 1 rate | 3 °C/min |
| Level 1 final value | 120 °C |
| Level 2 rate | 5 °C/min |
| Level 2 final value | 180 °C |
| Level 3 rate | 10 °C/min |
| Level 3 final value | 230 °C |
| Run time | 46 min |