Designation: ~~D5830 – 95 (Reapproved 2006)~~ D5830 – 14

Standard Test Method for Solvents Analysis in Hazardous Waste Using Gas Chromatography[1]

This standard is issued under the fixed designation D5830; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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 designed for use as a screening method with a typical reporting level of 0.1 %.

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

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

1.2 The scope of this test method may be expanded to include other volatile and semivolatile organic constituents.

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.

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[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.

Current edition approved ~~Feb. 1, 2006~~May 1, 2014. Published ~~March 2006~~June 2014. Originally approved in 1995. Last previous edition approved in ~~2001~~2006 as D5830 – 95(~~2001~~).(2006). DOI: ~~10.1520/D5830-95R06.~~10.1520/D5830-14.

1.3 Gas chromatographic methods are recommended for use only by, or under close supervision of, an experienced analyst.

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

2.2 *EPA Document:*

Gas Chromatography/Mass Spectrometry Method 8260, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition, Final Update 1, July 1992[3]

3. Summary of Test Method

3.1 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.

4. Significance and Use

4.1 This test method is useful in identifying the major 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.

5. Interferences

5.1 Interferences may be encountered from any number of organic compounds that respond in the detector. 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:

5.1.1 Gas chromatography/mass spectrometric (GC/MS) confirmation, see EPA Method 8260, direct injection technique;

5.1.2 Use of confirmation column, or confirmatory detector;

5.1.2.1 This method identifies one column (DB1701) 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, 8.1) and QC daily check, one for each confirmatory column or detector, or both.

5.1.3 Use of varying temperature programs or standard comparison, or both;

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

5.1.4 Sample history, for example, any information available from the waste generator; and,

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

5.2 Interferences may also be encountered from syringe carryover. Immediately following each injection, the syringe should be 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 be aware that carryover and possible interferences may occur if the rinse solvent is not completely cleaned from the syringe before reuse. Before each injection the syringe must be thoroughly rinsed with the sample to be injected, where the first two pumps are flushed into a separate waste receptacle.

5.3 When carbon disulfide (CS_2) is used to extract solids or sludges that contain significant amounts of water, low recovery of the water miscible solvents may result.

5.4 Some grades of CS_2 may contain trace amounts of benzene.

5.5 *M*-Pyrol seems to degrade slowly with time. The low-level degradation products interfere with some late eluting compounds on some columns (approximately five small peaks).

5.6 Interference from the CS_2 solvent peak may occur if using a TCD.

5.7 When using a TCD, be aware that water, as well as oxygenated compounds, for example, MEK, MIBK, may suppress detector response.

[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.

[3] Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

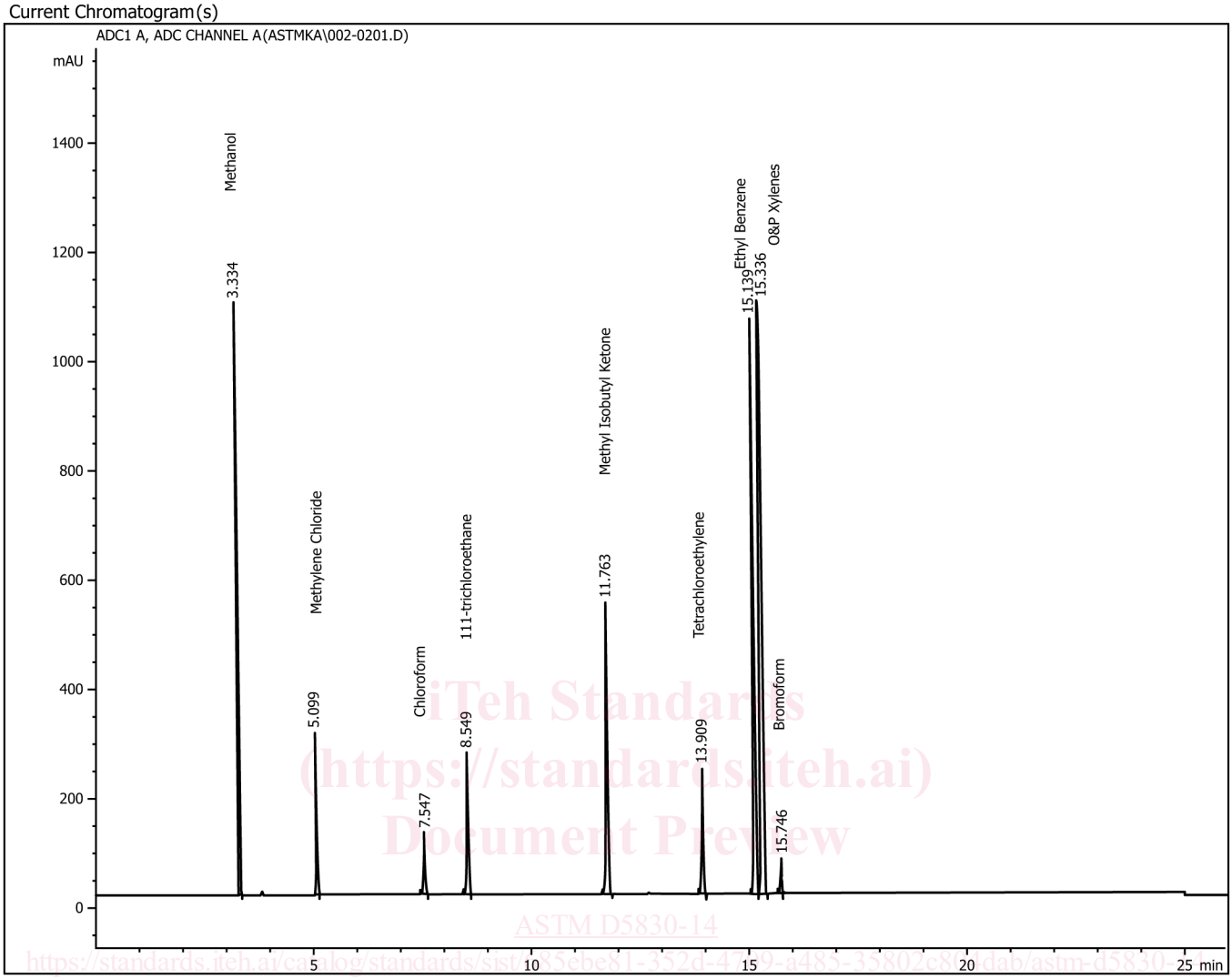

FIG. 1 Daily QC Standard FID/DB-1701

5.8 If an electrolytic conductivity detector (ELCD) or electron capture detector (ECD) must be used, be aware that CS_2, ~~*M*-Pyrol,~~ -Pyrol (required for an ELCD), and high concentrations of halogenated compounds may overload and possibly damage ~~the detector.~~both detectors. It is recommended that ~~the ECD~~these detectors be used only when very low detection levels of halogenated compounds are expected and direct injection of the sample is possible.

6. Apparatus

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

6.2 *Recommended Chromatographic Columns:*

6.2.1 *Capillary; Microbore or Megabore.*

6.2.1.1 DB-1701, 30M × 0.25-mm inside diameter, 0.25-µm film thickness.

6.2.1.2 DB-624, 30M × 0.3-mm inside diameter, 1.8-µm film thickness.

6.2.2 *Packed: Stainless Steel or Glass.*

6.2.2.1 1 % SP-1000, 60/80 Carbopak B, 8-ft by ⅛-in. inside diameter.

6.2.2.2 10 % SP-2100, 100/120 Chromosorb WHP, 2M × 2 mm ID.

NOTE 1—These columns are recommended and have shown to give good results. Operating conditions for each is listed in Section 10. Equivalent or alternative columns, or both, may be used depending on application.

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

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

6.5 *Analytical Balance,* accurate to 0.0001 g.

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

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

6.8 *Centrifuge,* optional.

6.9 *Vortex-Type Mixer.*

7. Reagents and Materials

7.1 *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 first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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

7.3 *Nitrogen or Helium (High Purity)*—For carrier and makeup gases. Air and hydrogen (high purity) for fuel gases. Gases may be obtained from a gas generator if available, through purification of a lower grade, or from a high-purity tank supply.

7.4 *Carbon Disulfide, CS_2* —Chromatography grade.

7.5 *M-Pyrol, C_5H_9NO*—Available through several chemical suppliers and sources as 1-methyl-2-pyrrolidone.

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

8. Standard Preparation

8.1 *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 7.

NOTE 2—Due to the incompatibility of some standard compounds, that is, some compounds are not miscible with each other, and also because of the number of compounds typically looked for in a single chromatographic run, it is advisable to prepare 3 or 4 standard solutions each composed of 10 to 15 compounds. A set of standard chromatograms and a retention timetable should be available for reference.

8.2 *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 must be established with all detectors and chromatography systems used for quantitation. All calibration and sample analysis must be done within the established linear range.

8.3 *Calibration Check Standard*—A calibration check standard should be prepared. The standard mixture should provide a good overall check of the GC/detector system. The compounds should cover the major compound types, for example, alcohols, aromatics, aliphatics, ketones, and halogenates. A typical calibration check standard flame ionization detector (FID) chromatogram is shown in Fig. 1.

9. Sample Collection, Preservation, and Handling

9.1 Sample collection should be in accordance with appropriate sampling protocols.

9.2 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.

9.3 *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.

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

9.5 Unused sample material, laboratory dilutions, and waste from the samples may be regulated. Consult your specialist or the regulations, or both, for guidance in the proper handling and disposal of laboratory wastes.

[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.

10. Procedure

10.1 *Sample Preparation:*

10.1.1 Analyze liquid matrices with relatively low viscosity using direct injection into the GC, either as received or after dilution with CS_2, *M*-Pyrol, or other suitable solvent.

10.1.2 Analyze solid or semisolid samples as follows:

10.1.2.1 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 CS_2 or *M*-Pyrol extract into the GC.

10.1.2.2 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 11.

10.1.3 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.

10.2 *Recommended GC Operating Conditions:*

10.2.1 For Capillary DB-1701 with FID

Column flow rate	1–1.5 mL/min
Make-up 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.

10.2.2 For Capillary DB-624 with FID

Column flow rate	3.5 mL/min
Make-up 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

10.2.3 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

10.2.4 For packed SP-2100 with FID

Carrier gas flow	30 mL/min
~~Injector temperatuare~~	~~250°C~~
Injector temperature	250°C

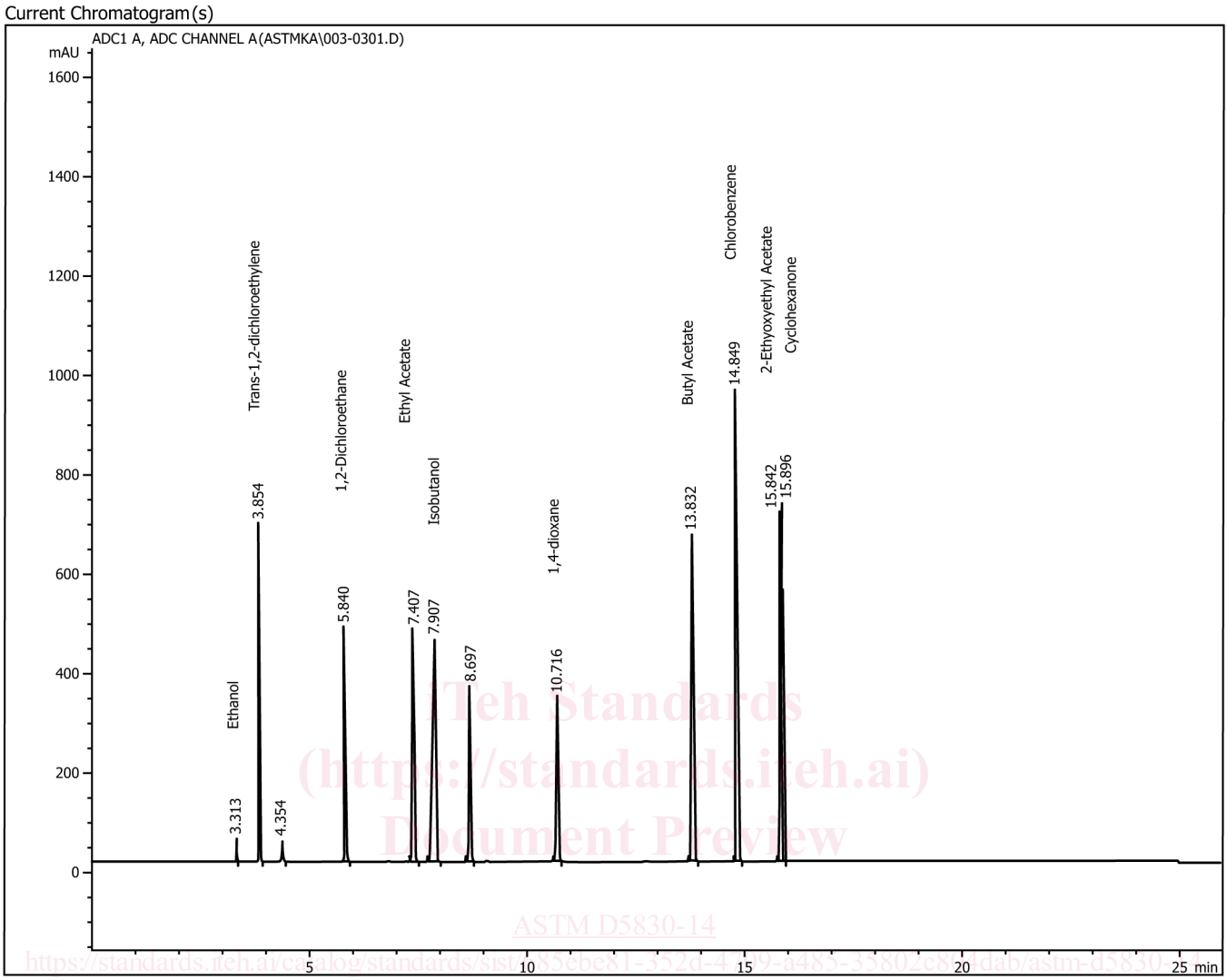

FIG. 2 Solvent Standard #1 FID/DB-1701

Detector temperature	300°C
Airflow (FID)	Approximately 300 mL/min
Hydrogen flow (FID)	Approximately 30 mL/min
Initial oven temperature	45°C
Initial hold time	3 min
Level 1 rate	15°C/min
Level 1 final value	90°C
Level 2 rate	10°C/min
Level 2 final value	195°C
Run time	16.5 min

10.3 *Linear Range Determination*—The linearity and linear range for each compound must be established on any GC system used for quantitation. This must be done on an annual basis or after any major maintenance or alteration of the system configuration, for example, detector replacement. Final quantitation for each compound must be done within the linear range of that compound.

10.4 *Calibration (External Standard Procedure):*

10.4.1 A single-point initial calibration of all compounds must be performed monthly. Inject 0.5 to 2.0 µL of the working standards that were prepared in 8.2. Tabulate peak area against concentration and express response factors (RF) for each component. This calculation is shown in 11.1. It is recommended for ease of calculation that the response factors be expressed as area counts per 1 % by weight.

10.4.2 The response factors must be verified daily or after every 20 samples, whichever is more frequent, by injecting 0.5 to 2.0 µL of the calibration check standard. This must be done for every column used for quantitation. If the predicted response varies