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An American National Standard

Standard Specification for Fuel Methanol (M70-M85) for Automotive Spark-Ignition Engines[1]

This standard is issued under the fixed designation D 5797; 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 specification covers a fuel blend, nominally 70 to 85 volume % methanol and 30 to 14 volume % hydrocarbons for use in ground vehicles with automotive spark-ignition engines. Appendix X1 discusses the significance of the properties specified. Appendix X2 presents the current status in the development of a luminosity test procedure for M70-M85.

1.2 The values stated in SI units are to be regarded as the standard. Values given in parentheses are provided for information only.

1.3 The following precautionary caveat pertains only to the test method portions–Annex A1, Annex A2, Annex A3, and Appendix X2 of this specification. *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]

2. Referenced Documents

2.1 *ASTM Standards:*

D 86 Test Method for Distillation of Petroleum Products[3]

D 130 Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test[3]

D 381 Test Method for Existent Gum in Fuels by Jet Evaporation[3]

D 512 Test Methods for Chloride Ion in Water[4]

D 525 Test Method for Oxidation Stability of Gasoline (Induction Period Method)[3]

D 872 Test Method for Sulfonation Index of Road Tars[5]

D 1193 Specification for Reagent Water[4]

D 1266 Test Method for Sulfur in Petroleum Products (Lamp Method)[3]

D 1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products[6]

D 2622 Test Method for Sulfur in Petroleum Products by X-ray Spectrometry Method[3]

D 2988 Test Method for Water-Soluble Halide Ion in Halogenated Organic Solvents and Their Admixtures[7]

D 3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry[3]

D 3231 Test Method for Phosphorus in Gasoline[8]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[8]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[8]

D 4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards[8]

D 4626 Practice for Calculation of Gas Chromatographic Response Factors[8]

D 4814 Specification for Automotive Spark-Ignition Engine Fuel[8]

D 4815 Test Method for Determination of MTBE, ETBE, TAME, DIPE, *tertiary*-Amyl Alcohol and C_1 to C_4 Alcohols in Gasoline by Gas Chromatography[8]

D 4929 Test Methods for Determination of Organic Chloride Content in Crude Oil[8]

D 4953 Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method)[8]

D 5059 Test Method for Lead in Gasoline by X-ray Spectroscopy[8]

D 5190 Test Method for Vapor Pressure of Petroleum Products (Automatic Method)[8]

D 5191 Test Method for Vapor Pressure of Petroleum Products (Mini Method)[8]

D 5453 Test Method for Determination of Total Sulfur in Light Hydrocarbons, Motor Fuels and Oils by Ultraviolet Fluorescence[9]

E 203 Test Method for Water Using Karl Fischer Reagent[7]

[1] This specification is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is under the direct responsibility of Subcommittee D02.A on Gasoline and Oxygenated Fuels.

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[2] Reference to the following documents is to be the latest issue unless otherwise specified.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 11.01.

[5] *Annual Book of ASTM Standards*, Vol 04.03.

[6] *Annual Book of ASTM Standards*, Vol 06.04.

[7] *Annual Book of ASTM Standards*, Vol 15.05.

[8] *Annual Book of ASTM Standards*, Vol 05.02.

[9] *Annual Book of ASTM Standards*, Vol 05.03.

E 355 Practice for Gas Chromatography Terms and Relationships[10]

E 1145 Specification for Denatured Ethyl Alcohol, Formula 3A[7]

3. Terminology

3.1 *Definitions:*

3.1.1 *methanol, n*—methyl alcohol, the chemical compound CH_3OH.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aliphatic ether*—an oxygen-containing, ashless, organic compound in which the oxygen atom is interposed betweeen two carbon atoms (organic groups), has the general formula $C_nH_{2n+2}O$ with n being 5 to 8, and in which the carbon atoms are connected in open chains and not closed rings.

3.2.1.1 *Discussion*—Aliphatic compounds can be straight or branched chains and saturated or unsaturated. The term aliphatic ether, as used in this specification, refers only to the saturated compounds.

3.2.2 *fuel methanol (M70-M85)*—a blend of methanol and hydrocarbons of which the methanol portion is nominally 70 to 85 volume% .

3.2.3 *higher alcohols*—aliphatic alcohols of the general formula $C_nH_{2\ n+1}OH$ with *n* being 2 to 8.

3.2.4 *hydrocarbon*—those components in a methanol-hydrocarbon blend that contain only hydrogen and carbon.

4. Fuel Methanol (M70-M85) Performance Requirements

4.1 Fuel methanol (M70-M85) shall conform to the requirements in Table 1.

NOTE 1—Most of the requirements cited in Table 1 are based on the best technical information currently available regarding the performance of these fuels in current technology vehicles. Requirements for sulfur, phosphorus, and lead are based on the use of gasoline defined in Specification D 4814 understanding that control of these elements will affect catalyst lifetime. The lead maximum is limited for Class 1 and Class 2 fuels to the lower limit of the test method. As greater experience is gained from field use of M70-M85 vehicles, and further vehicle hardware developments for the use of higher methanol content fuels occurs, it is expected that many of these requirements will change.

4.1.1 Vapor pressure is varied for seasonal and climatic changes by providing three vapor pressure classes for M70-M85. The seasonal and geographic distribution for the three vapor pressure classes is shown in Table 2. Class 1 encompasses geographical areas with 6-h tenth-percentile minimum ambient temperature of greater than 5°C (41°F). Class 2 encompasses geographical areas with 6-h tenth-percentile minimum temperatures of greater than −5°C (23°F) but less than +5°C. Class 3 encompasses geographical areas with 6-h tenth-percentile minimum ambient temperature less than or equal to −5°C.

[10] *Annual Book of ASTM Standards,* Vol 14.02.

TABLE 1 Requirements for Fuel Methanol (M70-M85)

Properties	Class 1[A]	Class 2	Class 3
Methanol + higher alcohols, min, volume%	84	80	70
Hydrocarbon/aliphatic ether, volume%	14–16	14–20	14–30
Vapor pressure, kPa	48–62	62–83	83–103
(psi)	7.0–9.0	9.0–12.0	12.0–15.0
Lead, max, mg/L	2.6	2.6	3.9
Phosphorus, max, mg/L	0.2	0.3	0.4
Sulfur, max, mg/kg	160	200	300
	All Classes		
Higher alcohols (C_2–C_8), max, volume %		2	
Acidity, as acetic acid, max, mg/kg		50	
Solvent washed gum content, max, mg/100 mL		5	
Unwashed gum content, max, mg/100 mL		20	
Total chlorine as chlorides, max, mg/kg		2	
Inorganic chloride, max, mg/kg		1	
Water, max, mass%		0.5	
Appearance	This product shall be visibly free of suspended or precipitated contaminants (clear and bright). This shall be determined at indoor ambient temperatures unless otherwise agreed upon between the supplier and the purchaser.		

[A] See 4.1.1 for volatility class criteria.

TABLE 2 Seasonal and Geographical Volatility Specifications for Fuel Methanol (M70-M85)

NOTE 1—This schedule subject to agreement between the purchaser and the seller denotes the vapor pressure class of the fuel at the time and place of bulk delivery to fuel dispensing facilities for the end user. Shipments should anticipate this schedule.

State	January	February	March	April	May	June	July	August	September	October	November	December
Alabama	2	2	2	2	2/1	1	1	1	1	1/2	2	2
Alaska												
Southern Region	3	3	3	3	3/2	2/1	1	1/2	2/3	3	3	3
South Mainland	3	3	3	3	3/2	2/1	1/2	2	2/3	3	3	3
Arizona												
N of 34° Latitude	3	3	3	3/2	2	2/1	1	1	1/2	2/3	3	3
S of 34° Latitude	2	2	2	2/1	1	1	1	1	1	1/2	2	2
Arkansas	3	3	3/2	2/1	1	1	1	1	1/2	2	2/3	3
California[A]												
North Coast	2	2	2	2	2	2/1	1	1	1	1/2	2	2
South Coast	3/2	2	2	2	2/1	1	1	1	1	1/2	2/3	3
Southeast	3	3/2	2	2	2/1	1	1	1	1/2	2	2/3	3
Interior	2	2	2	2	2	2/1	1	1	1	1/2	2	2
Colorado												
E of 105° Longitude	3	3	3	3/2	2	2/1	1	1	1/2	2/3	3	3
W of 105° Longitude	3	3	3	3	3/2	2	2/1	1/2	2/3	3	3	3
Connecticut	3	3	3	3/2	2	2/1	1	1	1/2	2	2/3	3
Delaware	3	3	3/2	2	2/1	1	1	1	1/2	2	2/3	3
District of Columbia	3	3	3/2	2	2/1	1	1	1	1/2	2	2/3	3
Florida												
N of 29° Latitude	2	2	2	2/1	1	1	1	1	1	1/2	2	2
S of 29° Latitude	2	2/1	1	1	1	1	1	1	1	1	1/2	2
Georgia	3	3/2	2	2/1	1	1	1	1	1	1/2	2	2/3
Hawaii	1	1	1	1	1	1	1	1	1	1	1	1
Idaho	3	3	3	3/2	2	2	2/1	1/2	2	2/3	3	3
Illinois												
N of 40° Latitude	3	3	3	3/2	2	2/1	1	1	1/2	2/3	3	3
S of 40° Latitude	3	3	3	3/2	2/1	1	1	1	1/2	2/3	3	3
Indiana	3	3	3	3/2	2/1	1	1	1	1/2	2/3	3	3
Iowa	3	3	3	3/2	2	2/1	1	1	1/2	2/3	3	3
Kansas	3	3	3	3/2	2	2/1	1	1	1/2	2/3	3	3
Kentucky	3	3	3/2	2	2/1	1	1	1	1/2	2	2/3	3
Louisiana	2	2	2	2/1	1	1	1	1	1	1/2	2	2
Maine	3	3	3	3/2	2	2/1	1	1/2	2	2/3	3	3
Maryland	3	3	3/2	2	2/1	1	1	1	1/2	2	2/3	3
Massachusetts	3	3	3	3/2	2	2/1	1	1	1/2	2	2/3	3
Michigan												
Lower Michigan	3	3	3	3/2	2	2/1	1	1/2	2	2/3	3	3
Upper Michigan	3	3	3	3	3/2	2/1	1	1/2	2	2/3	3	3
Minnesota	3	3	3	3	3/2	2/1	1	1/2	2	2/3	3	3
Mississippi	2	2	2	2/1	1	1	1	1	1	1/2	2	2
Missouri	3	3	3	3/2	2/1	1	1	1	1/2	2/3	3	3
Montana	3	3	3	3	3/2	2	2/1	1/2	2/3	3	3	3
Nebraska	3	3	3	3/2	2	2/1	1	1/2	2	2/3	3	3
Nevada												
N of 38° Latitude	3	3	3	3/2	2	2	2/1	1/2	2	2/3	3	3
S of 38° Latitude	3	3	3/2	2	2/1	1	1	1	1/2	2	2/3	3
New Hampshire	3	3	3	3/2	2	2/1	1	1/2	2	2/3	3	3
New Jersey	3	3	3/2	2	2/1	1	1	1	1/2	2	2/3	3
New Mexico												
N of 34° Latitude	3	3	3	3/2	2	2/1	1	1	1/2	2/3	3	3
S of 34° Latitude	3	3	3/2	2/1	1	1	1	1	1	1/2	2/3	3
New York												
N of 42° Latitude	3	3	3	3/2	2	2/1	1	1/2	2	2/3	3	3
S of 42° Latitude	3	3	3	3/2	2/1	1	1	1	1/2	2	2/3	3
North Carolina	3	3	3/2	2	2/1	1	1	1	1/2	2/3	3	3
North Dakota	3	3	3	3	3/2	2/1	1	1/2	2	2/3	3	3
Ohio	3	3	3	3/2	2/1	1	1	1	1/2	2/3	3	3
Oklahoma	3	3	3	3/2	2/1	1	1	1	1/2	2	2/3	3
Oregon												
E of 122° Longitude	3	3	3	3/2	2	2	2/1	1/2	2	2/3	3	3
W of 122° Longitude	3	3/2	2	2	2	2/1	1	1	1/2	2	2	2/3
Pennsylvania												
N of 41° Latitude	3	3	3	3/2	2	2/1	1	1/2	2	2/3	3	3
S of 41° Latitude	3	3	3	3/2	2	2/1	1	1	1/2	2	2/3	3
Rhode Island	3	3	3	3/2	2/1	1	1	1	1/2	2	2/3	3
South Carolina	2	2	2	2/1	1	1	1	1	1	1/2	2	2
South Dakota	3	3	3	3/2	2	2/1	1	1/2	2	2/3	3	3
Tennessee	3	3	3/2	2	2/1	1	1	1	1/2	2	2/3	3
Texas												
N of 31° Latitude	3	3	3/2	2	2/1	1	1	1	1/2	2	2/3	3

TABLE 2 *Continued*

State	January	February	March	April	May	June	July	August	September	October	November	December
S of 31° Latitude	2	2	2	2/1	1	1	1	1	1	1/2	2	2
Utah	3	3	3	3/2	2	2/1	1	1	1/2	2/3	3	3
Vermont	3	3	3	3/2	2	2/1	1	1/2	2	2/3	3	3
Virginia	3	3	3/2	2	2/1	1	1	1	1/2	2	2/3	3
Washington												
E of 122° Longitude	3	3	3/2	2	2	2/1	1	1	1/2	2/3	3	3
W of 122° Longitude	3	3/2	2	2	2	2/1	1	1	1/2	2	2	2/3
West Virginia	3	3	3	3/2	2	2/1	1	1/2	2	2/3	3	3
Wisconsin	3	3	3	3/2	2	2/1	1	1/2	2	2/3	3	3
Wyoming	3	3	3	3	3/2	2	2/1	1/2	2	2/3	3	3

[A] Details of State Climatological Division by county as indicated:

California, North Coast—Alameda, Contra Costa, Del Norte, Humbolt, Lake, Marin, Mendocino, Monterey, Napa, San Benito, San Francisco, San Mateo, Santa Clara, Santa Cruz, Solano, Sonoma, Trinity

California, Interior—Lassen, Modoc, Plumas, Sierra, Siskiyou, Alpine, Amador, Butte, Calaveras, Colusa, El Dorado, Fresno, Glenn, Kern (except that portion lying east of Los Angeles County Aqueduct), Kings, Madera, Mariposa, Marced, Placer, Sacramento, San Joaquin, Shasta, Stanislaus, Sutter, Tehama, Tulare, Tuolumne, Yolo, Yuba, Nevada

California, South Coast—Orange, San Diego, San Luis Obispo, Santa Barbara, Ventura, Los Angeles (except that portion north of the San Gabriel Mountain range and east of the Los Angeles County Aqueduct)

California, Southeast—Imperial, Riverside, San Bernadino, Los Angeles (that portion north of the San Gabriel Mountain range and east of the Los Angeles County Aqueduct), Mono, Inyo, Kern (that portion lying east of the Los Angeles County Aqueduct)

4.1.2 The hydrocarbons used shall have a final maximum boiling point of 225°C (437°F) by Test Method D 86, oxidation stability of 240-min minimum by Test Method D 525, and No. 1 maximum copper strip corrosion by Test Method D 130. The hydrocarbons may contain aliphatic ethers as blending components as are customarily used for automotive fuel.

4.1.3 Use of unprotected aluminum in fuel methanol (M70-M85) distribution and dispensing equipment will introduce insoluble aluminum compounds into the fuel causing plugged vehicle fuel filters. Furthermore, this effect can be exaggerated even with protected aluminum by elevated fuel conductivity caused by contact with a nitrile rubber dispensing hose. Therefore, unprotected aluminum and an unlined nitrile rubber dispensing hose should be avoided in fuel methanol (M70-M85) distribution and dispensing systems.[11]

5. Sampling

5.1 Sample in accordance with Practice D 4057, except that water displacement (10.3.1.8 of Practice D 4057) shall not be used.

5.2 Where practical, M70-M85 should be sampled in glass containers. If samples must be collected in metal containers, do not use soldered metal containers. This is because the soldering flux in the containers and lead in the solder can contaminate the sample. Plastic containers should be avoided.

5.3 A minimum sample size of about 1 L (1 qt) is recommended.

6. Test Methods

6.1 Determine the requirements enumerated in this specification in accordance with the following test methods:

NOTE 2—The appropriateness of ASTM test methods cited has not been demonstrated for use with M70-M85. In addition, test methods contained in the annexes and appendixes are in the developmental stages or lack precision and bias determinations.

6.1.1 *Methanol*—A procedure for a test method for methanol content of fuel methanol (M70-M85) is included as Annex A1. Verification of the appropriateness of this test method has indicated that the precision of this method may not be adequate. As work continues to develop a method, this procedure remains the best available.

6.1.2 *Hydrocarbon/Aliphatic Ether*—Use Test Method D 4815 to determine higher alcohols, methyl *tert*-butyl ether (MTBE), and other ethers. Water may also be determined if the gas chromatograph is equipped with a thermal conductivity detector. As an alternative, water can be determined by the Karl Fischer test method (see 6.1.9). The concentration of methanol, other alcohols, and water can be added, and the sum subtracted from 100 to get the percent of hydrocarbons/aliphatic ethers. An alternative test method is contained in Annex A2.

6.1.3 *Vapor Pressure*— Test Methods D 4953, D 5190, or D 5191.

6.1.4 *Acidity*—Test Method D 1613.

6.1.5 *Gum Content, Solvent Washed and Unwashed*—Test Method D 381.

6.1.6 *Total Chlorine as Chloride*—Test Methods D 4929, Method B.

6.1.7 *Lead*—Test Method D 5059. With Test Method D 5059, prepare the calibration standards using methanol (reagent grade) as the solvent to prevent errors caused by large differences in carbon-hydrogen ratios.

6.1.8 *Phosphorus*—Test Method D 3231.

6.1.9 *Water*—Test Method E 203.

6.1.10 *Sulfur*—Test Methods D 1266, D 2622, D 3120, or D 5453. With Test Method D 2622, prepare the calibration standards using methanol (reagent grade) as the solvent to prevent errors caused by large differences in carbon-hydrogen ratios.

6.1.11 *Inorganic Chloride*—Inorganic chloride can be determined by Test Methods D 512 (Method C) or D2988. Also, see the test method in Annex A3. Another test method is under development.

[11] American Automobile Manufacturers Association, "Fuel Methanol Compatibility Standards and Dispensing Equipment List for M85 Fueled Vehicles," October 1994.

7. Keywords

7.1 acidity; alcohol; automotive spark-ignition engine fuel; chloride; copper corrosion; ether; fuel methanol (M70-M85) for automotive spark-ignition engines; gum content; solvent washed; hydrocarbon; inorganic chloride; lead; MTBE; M70-M85; methanol; oxidation stability; oxygenates; phosphorus; sulfur; total chlorine; vapor pressure; volatility; water

ANNEXES

(Mandatory Information)

A1. TEST METHOD FOR DETERMINATION OF METHANOL IN FUEL METHANOL (M70-M85) FOR AUTOMOTIVE SPARK-IGNITION ENGINES

A1.1 Scope

A1.1.1 This test method covers a procedure for determination of methanol in fuel methanol (M70-M85) by gas chromatography. This test method is appropriate for fuels containing 70 to 95 volume % methanol.

A1.1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

A1.2 Referenced Documents

A1.2.1 *ASTM Standards*:

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[8]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[8]

D 4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards[8]

D 4626 Practice for Calculation of Gas Chromatographic Response Factors[8]

E 355 Practice for Gas Chromatography Terms and Relationships[10]

A1.3. Terminology

A1.3.1 *Definitions of Terms Specific to This Standard:*

A1.3.1.1 *low-volume connector*—special union for connecting two lengths of tubing 1.6 mm inside diameter and smaller. Also referred to as a zero dead-volume union.

A1.3.1.2 *split ratio—in gas chromatography using capillary columns*—the ratio of the total flow of the carrier gas to the sample inlet versus the flow of carrier gas to the capillary column.

A1.3.1.3 *TCEP*—1,2,3-tris-2-cyanoethoxypropane. A gas chromatographic liquid phase.

A1.3.1.4 *WCOT*—abbreviation for a type of capillary column, wall-coated open tubular, used in gas chromatography. This type of column is prepared by coating the inside of the capillary with a thin film of stationary phase

A1.4 Summary of Test Method

A1.4.1 An internal standard, *tert*-amyl alcohol, is added to the sample that is then introduced into a gas chromatograph equipped with two columns and a column switching valve. The sample passes into the first column, a polar TCEP column that elutes lighter hydrocarbons to vent and retains the oxygenated and heavier hydrocarbons.

A1.4.2 After methylcyclopentane, but before methanol elutes from the polar column, the valve is switched to backflush the oxygenates onto a WCOT nonpolar column. The methanol and internal standard elute from the nonpolar column in boiling point order, before elution of any major hydrocarbon constituents.

A1.4.3 After the internal standard elutes from the non-polar column, the column switching valve is switched back to its original position to backflush the heavy hydrocarbons. The eluted components are detected by a flame ionization or thermal conductivity detector. The detector response, proportional to the component concentration, is recorded; the peak areas are measured; and the concentration of methanol is calculated with reference to the internal standard.

A1.5 Significance and Use

A1.5.1 The production of fuel methanol (M70-M85) requires knowledge of the methanol content to ensure acceptable commercial fuel quality. The methanol content of fuel methanol (M70-M85) affects the performance of an automobile designed to run on such fuel.

A1.5.2 This test method is applicable to both quality control in the production of fuel methanol (M70-M85) and for the determination of fuel contamination.

A1.6 Apparatus

A1.6.1 *Chromatograph*— See Practice E 355 for specific descriptions and definitions.

A1.6.1.1 *Gas Chromatographic Instrument*, operable at the conditions given in Table A1.1 and having a column switching and backflushing system equivalent to Fig. A1.1. Carrier gas

TABLE A1.1 Chromatographic Operating Conditions

	Temperatures, °C	Flows	mL/min
Column Oven	60	to injector	75
Injector	200	column	5
Detector		auxiliary	3
TCD	200	makeup	18
FID	250		
Valve	60		
Carrier Gas—Helium			
Sample size, µL		1	
Split ratio		15:1	
Backflush, min		0.2–0.3	
Valve reset time, min		8–10	
Total analysis time, min		18–20	

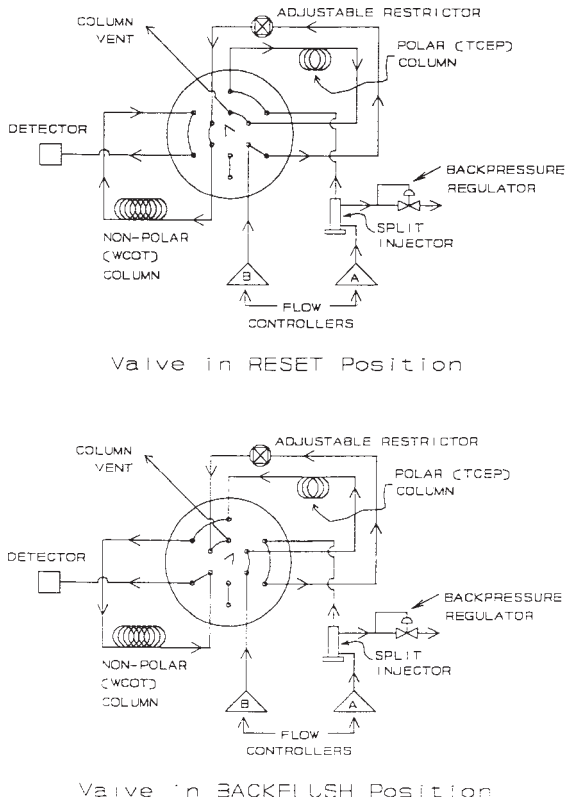

FIG. A1.1 Schematic of Chromatographic System

flow controllers must be designed for use at the required flow rates (see Table A1.1). Pressure control devices and gages must be designed for use at the pressures required. Table A1.2

A1.6.1.2 *Detector*, either a thermal conductivity detector (TCD) or flame ionization detector (FID) may be used. The system must have sufficient sensitivity and stability to sense absolute concentration changes of 0.01 volume % of methanol or internal standard at the 50 volume % level.

A1.6.1.3 *Switching and Backflushing Valve*, a ten-port valve, to be located within the gas chromatographic column oven, capable of performing the functions described in A1.10 and illustrated in Fig. A1.1. The valve must be of lowvolume design and not contribute significantly to chromatographic deterioration.[12]

A1.6.1.4 *Automatic Valve Switching Device*, (strongly recommended to ensure repeatable switching times) a device synchronized with injection and data collection times. If no such device is available, a stopwatch, started at the time of injection, should be used to indicate the proper valve switching time.

A1.6.1.5 *Injection System*, a splitting-type inlet device. Split injection is necessary to maintain the actual chromatographed sample size within the limits of column and detector optimum efficiency and linearity.

A1.6.1.6 *Sample Introduction System*, any system capable of introducing a representative sample into the split inlet device.

NOTE A1.1—Microlitre syringes, automatic syringe injectors, and liquid sampling valves have been used successfully.

A1.6.2 *Data Presentation or Calculation System*:

A1.6.2.1 *Recorder*, a recording potentiometer or equivalent with a full-scale deflection of 1 mV or less, and full-scale response time of 1 s or less, with sufficient sensitivity and stability to meet the requirements of A1.6.1.2.

A1.6.2.2 *Integrator or Computer Devices*, capable of meeting the requirements of A1.6.1.2, and providing graphic and digital presentation of the chromatographic data. Peak heights or areas can be measured by computer, electronic integration, or manual techniques.

A1.6.3 *Columns*—Two columns are used as follows:

A1.6.3.1 *Polar Column*— Any column with equivalent or better chromatographic efficiency and selectivity to that described in A1.6.3.1(*a*) can be used. The column must perform at the same temperature as required for the column in A1.6.3.2. This column performs a pre-separation of the oxygenates from volatile hydrocarbons in the same boiling point range. The oxygenates and remaining hydrocarbons are backflushed onto the nonpolar column in A1.6.3.2.

(a) (a) TCEP Micro-Packed Column,[13] 560-mm (22-in.) by 1.6-mm (1⁄16-in.) outside diameter by 0.38-mm (0.015-in.) inside diameter stainless steel tube packed with 0.14 to 0.15 g of 20 % by mass TCEP on 80/100 mesh Chromosorb P(AW). This column is being used to develop precision and bias data for A1.15.

A1.6.3.2 *Nonpolar (Analytical) Column*—Any column with equivalent or better chromatographic efficiency and selectivity to that described in A1.6.3.2(*a*) and illustrated in Fig. A1.2 can be used.

(a) (*a*) *WCOT Methyl Silicone Column*, 30-mm (1.181-in.) long by 0.53-mm (0.021-in.) inside diameter fused silica WCOT column with a 2.65-µm film thickness of crosslinked methyl siloxane. This column is being used to develop precision and bias data for A1.15.

A1.7 Reagents and Materials

A1.7.1 *Carrier Gas*, carrier gas appropriate to the type of detector used. The minimum purity of the carrier gas shall be 99.995 mol %.

NOTE A1.2—Helium has been used successfully.

A1.7.2 *Methanol, 99.9 % Purity*, required to establish identification by retention time and for calibration. Shall be of

[12] A Valco Model No. CM-VSV-10-HT valve with 1.6-mm (1⁄16-in.) fittings has been found satisfactory for this purpose. This is the valve being used in the majority of the analyses for the development of the data for A1.15. A Valco Model No. C10W with 0.8-mm (1⁄31-in.) fittings is recommended for use with columns of 0.32-mm inside diameter and smaller.

TABLE A1.2 Retention Characteristics for TCEP/WCOT Column Set Conditions as in Table A1.1

Component	Retention Time, min	Relative Retention Time (*t*-Amyl Alcohol = 1.00)
Methanol	3.21	0.44
t-Amyl Alcohol	7.30	1.00

[13] Available from Hewlett Packard Co., Wilmington, DE.

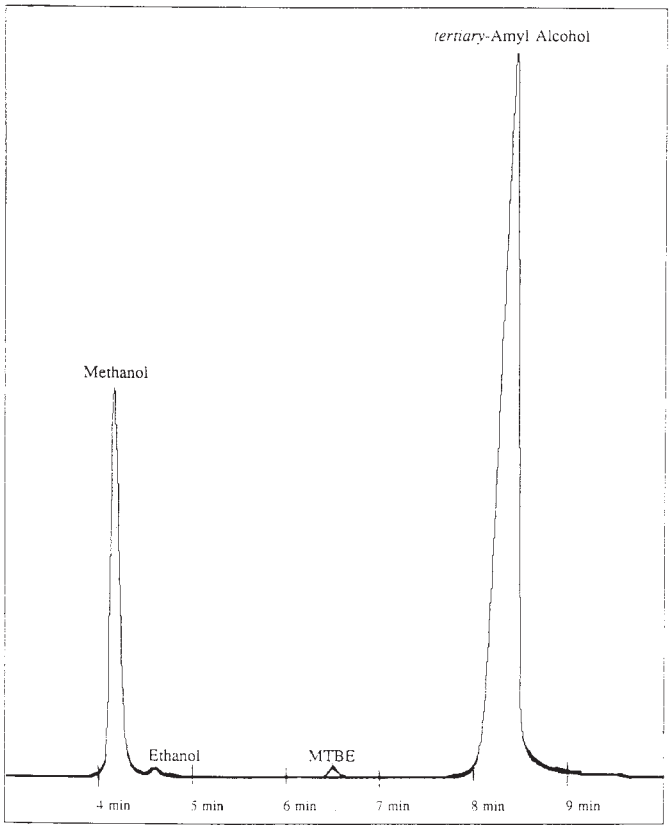

FIG. A1.2 Example of Chromatographic Results

known purity and free of the other components to be analyzed. (**Warning**—Flammable. Health hazard.)

A1.7.3 *Methylene Chloride*, used for column preparation. Reagent grade, free of nonvolatile residue. (**Warning**—Health hazard.)

A1.7.4 *Nitrogen, 99.998 mol %*, used to prepare tubing for the micro-packed TCEP column. (**Warning**—Gas under pressure.)

A1.7.5 *Tert-Amyl Alcohol (2-Methyl-2-Butanol), 99 % Purity*, used as the internal standard. (**Warning**—Flammable. Health hazard.)

A1.8 Preparation of Column Packing

A1.8.1 *Preparation of TCEP Column Packing*:

A1.8.1.1 Any satisfactory method, used in the practice of the art that will produce a column capable of retaining methanol and *tert*-amyl alcohol (internal standard) from hydrocarbon components of the same boiling point range in a fuel methanol (M70-M85) sample, may be used. The following procedure has been used successfully.

A1.8.1.2 Completely dissolve 10 g of TCEP in 100 mL of methylene chloride. Next add 40 g of 80/100 mesh Chromosorb P(AW) to the TCEP solution. Quickly transfer this mixture to an evaporating dish, in a fume hood, without scraping any of the residual packing from the sides of the container. Constantly, but gently, stir the packing until all of the solvent has evaporated. This column packing can be used immediately to prepare the TCEP column.

A1.8.2 *Packing of Micro-Packed TCEP Column*:

A1.8.2.1 Wash a straight 560-mm length of 1.6-mm outside diameter (0.38-mm inside diameter) stainless steel tubing with methanol and dry with compressed nitrogen.

A1.8.2.2 Insert six to twelve strands of silvered wire, a small mesh screen, or stainless steel frit inside one end of the tube. Slowly add 0.14 to 0.15 g of packing material to the column and gently vibrate to settle the packing inside the column. When strands of wire are used to retain the packing material inside the column, leave 6.4 mm (0.25 in.) of space at the top of the column. Place wire or screen in the column to retain packing.

A1.8.2.3 *Column Conditioning*—Both the TCEP and WCOT columns are to be briefly conditioned before use. Connect the columns to the valve (see A1.10.1) in the chromatographic oven. Adjust the carrier gas flows as in A1.10.3 and place the valve in the RESET position. After several minutes, increase the column oven temperature to 120°C and maintain these conditions for 5 to 10 min. Cool the columns below 60°C before shutting off the carrier flow.

A1.9 Sampling

A1.9.1 To obtain samples for this test method, use the procedures outlined in Practice D 4057 or Practice D 4177 except that water displacement (see section 10.3.1.8 of Practice D 4057) shall not be used.

A1.9.2 Fuel samples shall be stored under refrigeration until the laboratory subsample is taken for analysis. Any laboratory subsamples shall also be refrigerated if they are not to be analyzed immediately.

A1.9.3 Thoroughly shake the sample container prior to withdrawing any subsample for analysis. Allow any particulate matter to settle to the bottom of the subsample container. Inspect the subsample for evidence of phase separation. If phase separation is detected, the sample is invalid and a new sample shall be requested.

A1.10 Preparation of Apparatus and Establishment of Conditions

A1.10.1 *Assembly*—Connect the WCOT column to the valve system using low-volume connectors and narrow bore tubing. It is important to minimize the volume of the chromatographic system that comes in contact with the sample, otherwise peak broadening will occur.

A1.10.2 Adjust the operating conditions to those listed in Table A1.1. (If a TCD is being used, do not turn on the detector circuits.) Check the system for leaks before proceeding further.

A1.10.3 *Flow Rate Adjustment*—See Fig. A1.1.

A1.10.3.1 Attach a flow-measuring device to the column vent with the valve in the RESET position and adjust the pressure to the injection port to give 5.0-mL/min flow (14 psig).

NOTE A1.3—Soap-bubble flowmeters are suitable.

A1.10.3.2 Attach a flow-measuring device to the split injector vent and adjust the flow from the split vent using the A Flow Controller to give a flow of 70 mL/min. Recheck the column vent flow set in A1.10.3.1 and adjust, if necessary.

A1.10.3.3 Switch the valve to the BACKFLUSH position and adjust the variable restrictor to give the same column vent

flow set in A1.10.3.1. This is necessary to minimize flow changes when the valve is switched.

A1.10.3.4 Switch the valve to the inject position RESET and adjust the B Flow Controller to give a flow of 3.0 to 3.2 mL/min at the detector exit. When required for the particular instrumentation used, add makeup flow or TCD switching flow to give a total of 21 mL/min at the detector exit.

A1.10.4 When a thermal conductivity detector is used, turn on the filament current and allow the detector to equilibrate. When a flame ionization detector is used, set the hydrogen and air flows, ignite the flame, and turn on the electrometer.

A1.10.5 *Valve Switch Times*—The times for switching the valve to the BACKFLUSH position and then back to the RESET position will vary slightly for each column system and shall be determined experimentally as follows. The start time of the integrator and valve timer shall be synchronized with the injection to accurately reproduce the backflush time.

A1.10.5.1 Prepare a blend consisting of approximately, 40 volume % methanol, 10 volume % gasoline (methanol-free), and 50 volume % *tert*-amyl alcohol (internal standard.)

A1.10.5.2 Initially assume a valve BACKFLUSH time of 0.23 min. With the valve in the RESET position, inject 1 µL of the blend prepared above. At 0.23 min after the injection, rotate the valve to the BACKFLUSH position and leave it there until the complete elution of internal standard is realized. Note this time as the RESET time and return the valve to the RESET position. When all of the remaining hydrocarbons are backflushed, the signal will return to a stable baseline and the system is ready for another analysis. The chromatogram should appear similar to that illustrated in Fig. A1.2.

A1.10.5.3 It is necessary to optimize the valve BACKFLUSH time by analyzing the blend prepared in A1.10.5.1. The correct BACKFLUSH time is determined experimentally by using valve switching times between 0.2 and 0.3 min. When the valve is switched too soon, C_5 and lighter hydrocarbons will be backflushed and will co-elute in the oxygenate section of the chromatogram. When the valve BACKFLUSH is switched too late, part or all of the methanol will be vented resulting in an incorrect methanol measurement.

A1.11 Calibration

A1.11.1 Prepare at least three different blends of methanol in gasoline that is known to be free of methanol and *tert*-amyl alcohol. Refer to Practice D 4307 for preparation of liquid blends. Volume % concentrations for each component shall be calculated at the temperature of interest. These blends shall be at known concentrations bracketing the methanol concentrations of interest.

A1.11.2 Analyze each calibration blend as described in A1.12.

A1.11.3 Measure the peak areas of methanol and of the internal standard by either manual methods or an electronic integrator. Calculate the relative volume response factor for methanol relative to the internal standard, in accordance with Practice D 4626.

A1.12 Procedure

A1.12.1 *Preparation of Sample*—Remove the sample from refrigeration and withdraw a subsample for analysis as described in A1.9.3. Allow the subsample, internal standard, and any volumetric apparatus employed to equilibrate at the temperature of interest. Accurately add a known volume of the internal standard, *tert*-amyl alcohol, to an accurately measured volume of sample.

NOTE A1.4—Concentrations of 40 to 60 volume % have been used successfully.

A1.12.2 *Chromatographic Analysis*—Introduce a representative aliquot of the sample, containing internal standard, into the chromatograph and use the valve switch times determined in A1.10.5.

NOTE A1.5—An injection volume of 1 µL with a 15:1 split ratio has been used successfully.

A1.12.3 *Interpretation of Chromatogram*—Compare the results of sample analyses to those of calibration analyses to determine identification of methanol and internal standard present.

A1.13 Calculation

A1.13.1 After identifying the methanol peak, measure its area and that of the internal standard. Calculate the volume % of methanol as follows:

$$V(M) = 100 \times \frac{RR_v(M) \times V(S) \times A(M)}{A(S) \times V(F)} \quad \text{(A1.1)}$$

where:

$V(M)$	=	volume of methanol, %,
$V(S)$	=	volume of internal standard (*tert*-amyl alcohol) added,
$V(F)$	=	volume of fuel sample taken,
$A(M)$	=	peak area or height of methanol,
$A(S)$	=	peak area or height of the internal standard (*tert*-amyl alcohol), and
$RR_v(M)$	=	relative volume response factor for methanol (relative to the internal standard).

A1.14 Report

A1.14.1 Report the volume % of methanol to the nearest 0.1 volume %.

A1.15 Precision and Bias

A1.15.1 *Precision*—The precision of this test method for measuring methanol in methanol-based fuels is being determined.

A1.15.2 *Bias*—Since there is no accepted reference material suitable for determining bias for the procedure in this test method for measuring methanol in M70-M85, bias has not been determined.

A1.16 Keywords

A1.16.1 alcohols; fuel methanol; gas chromatography; M70-M85; methanol

A2. TEST METHOD FOR DETERMINATION OF HYDROCARBON/ALIPHATIC ETHER CONTENT OF FUEL METHANOL (M70-M85) FOR SPARK-IGNITION ENGINES

A2.1 Scope

A2.1.1 This test method covers a procedure to determine the hydrocarbon/aliphatic ether content of fuel methanol (M70-M85).

A2.1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

A2.2 Referenced Document

A2.2.1 *ASTM Standard*:
D 1193 Specification for Reagent Water[4]

A2.3 Summary of Test Method

A2.3.1 This test method consists of the addition of fuel methanol (M70-M85) to a sodium chloride solution followed by the addition of water to cause phase separation of the hydrocarbon/aliphatic ethers which is then visually measured in the Babcock flask.

A2.4 Significance and Use

A2.4.1 Maintenance of a specified proportion of hydrocarbon/aliphatic ether to methanol has importance in the starting, operation, and emission performance of vehicles designed to operate on fuel methanol (M70-M85) and the production of a visible flame in the event of a fuel fire involving fuel methanol (M70-M85).

A2.5 Apparatus

A2.5.1 *Babcock Flask (Babcock Milk Bottle)*, borosilicate glass, 165 mm, 18 g, as specified in Test Method D 872.

A2.5.2 *Disposable Syringes or Clean Pipettes*, 10 ± 0.1 mL.

A2.5.3 *Centrifuge*, capable of holding Babcock flasks and operating at 1500 rpm.

A2.6 Reagents and Materials

A2.6.1 *Distilled Water*, Specification D 1193, Type IV.

A2.6.2 *Sodium Chloride (NaCl) Solution*, 10 % by mass.

A2.7 Procedure

A2.7.1 Place 20 mL of a 10 % by mass NaCl solution into an 8 % Babcock flask. Introduce 10 mL of the test fuel methanol (M70-M85) with use of a pipette or syringe. Stopper and shake for 1 min. Add water sufficient to read between 7.5 and 8.0 on the Babcock scale. Place the stoppered flask into a centrifuge for 5 min at 1500 rpm. Read and record the volume percentage directly from the neck of the Babcock bottle. This is done by subtracting the reading at the liquid/liquid interface from that of the upper meniscus. Multiply by two to obtain the percent hydrocarbon/aliphatic ether by volume in the test fuel methanol (M70-M85).

A2.8 Precision and Bias

A2.8.1 The precision of this test method as obtained by statistical examination of interlaboratory test results is as follows:

A2.8.1.1 *Repeatability*— The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the following value only in one case in twenty:

$$1.4 \text{ volume } \% \qquad \text{(A2.1)}$$

A2.8.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the following value only in one case in twenty:

$$3.6 \text{ volume } \% \qquad \text{(A2.2)}$$

A2.8.2 *Bias*—No significant bias was observed between the measured and the prepared methanol content of the fuel methanol blends tested in the cooperative study used to evaluate this test method. The accuracy of results shall not differ from the established true value by more than the stated precision.

A2.9 Keywords

A2.9.1 Babcock flask; fuel methanol (M70-M85); hydrocarbon/aliphatic ether content; phase separation; spark-ignition engines

A3. TEST METHOD FOR DETERMINATION OF INORGANIC CHLORIDE IN FUEL METHANOL (M70-M85) FOR AUTOMOTIVE SPARK-IGNITION ENGINES

A3.1 Scope

A3.1.1 This test method covers a procedure to determine the inorganic chloride in fuel methanol in the range of concentrations from 0.4 to 2.0 mg/kg.

A3.1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

A3.2 Summary of Test Method

A3.2.1 The sample is concentrated, acidified, and treated with silver nitrate. The turbidity is visually compared with standards.

A3.3 Significance and Use

A3.3.1 Because of the corrosive nature of inorganic chloride to the fuel systems of internal combustion engines, a means to measure low levels of inorganic chloride in fuel methanol is required.

A3.4 Apparatus

A3.4.1 *Distillation Apparatus*, 500-mL distillation flask, condenser, and 250-mL graduated cylinder as collector.

A3.4.2 *Nessler Tubes*, 100 mL, matched, tall form.

A3.5 Reagents and Materials

A3.5.1 *Demineralized Water*, halide- and sulfide-free.

A3.5.2 *Methanol*, halide- and sulfide-free by distillation. (**Warning**—Flammable. Health hazard.)

A3.5.3 *Nitric Acid (HNO_3) Solution*, 1 part 15.7 *M* acid to 1 part demineralized water. (**Warning**—Corrosive. Health hazard.)

A3.5.4 *Silver Nitrate Solution*, 0.1 *M*. (**Warning**—Health hazard.)

A3.5.5 *Sodium Chloride (NaCl)*.

A3.6 Standards

A3.6.1 Dissolve 0.845 g of dry sodium chloride (NaCl) in halide- and sulfide-free water and dilute to 1 L in a volumetric flask. Mix thoroughly and label Solution A (0.5 mg Cl/mL).

A3.6.2 Pipette 10 mL of Solution A into a 1-L volumetric flask. Dilute to volume with halide- and sulfide-free water. Mix thoroughly and label Solution B (0.005 mg Cl/mL).

A3.6.3 In matching Nessler tubes, prepare the following standards:

	Blank	1	2	3	4	5
Solution B, mL (pipette)	0.0	1.0	2.0	3.0	4.0	5.0
Methanol, mL (Cl-free)	80	80	80	80	80	80

A3.6.4 For each standard follow A3.7.4 to A3.7.8.

A3.6.5 The turbidity standards are affected by light and are not stable. Prepare fresh standards (from Solution B) for each group of samples.

A3.7 Procedure

A3.7.1 Clean all glassware with 1 *M* HNO_3 and rinse with demineralized water and halide- and sulfide-free methanol.

A3.7.2 Measure 320 mL of sample in a graduated cylinder and put into the distillation flask. Add boiling beads. Distill the sample into a graduated cylinder until 240 mL of the distillate is obtained. Use 4 mL of the concentrated residue from the distillation flask as follows.

A3.7.3 Add a 4-mL sample to a 100-mL Nessler tube.

A3.7.4 Add 80 mL of halide- and sulfide-free methanol to the sample in the Nessler tube.

A3.7.5 Dilute the contents of all tubes (sample and standards) to the 100-mL mark with halide- and sulfide-free water.

A3.7.6 Pipette 2 mL of nitric acid solution (1 part 15.7 *M* acid to 1 part demineralized water) into each tube.

A3.7.7 Pipette 1 mL of 0.1 *M* silver nitrate solution into each tube.

A3.7.8 Stopper and mix thoroughly by inverting.

A3.7.9 Allow the tubes to stand in the dark for 5 min. Visually compare the sample to the standard solutions while looking vertically against a black background. Record the millilitres of standard Solution B that match the sample.

A3.8 Calculation

A3.8.1 Calculate the results as follows:

$$(A)(B)(0.001)(1\,000\,000)/(320/80)(4) \qquad \text{(A3.1)}$$

$$(0.794) = \text{Cl, mg/kg}$$

where:

A	=	Solution B that matched sample, mL,
B	=	Solution B, mg Cl/mL,
0.001	=	mg to g,
320/80	=	concentration factor,
4	=	sample, mL, and
0.794	=	relative density of methanol at 20°C (68°F) compared to water at 4°C (39°F).

A3.9 Precision and Bias

A3.9.1 *Precision*—The precision of this test method for measuring inorganic chloride in fuel methanol is being determined.

A3.9.2 *Bias*—Since there is no accepted reference material suitable for determining bias for the procedure in this test method for measuring inorganic chloride in M70-M85, bias has not been determined.

A3.10 Keywords

A3.10.1 chloride; fuel methanol (M70–M85); inorganic chloride; spark-ignition engines; silver nitrate; turbidity; turbidity standards

APPENDIXES

(Nonmandatory Information)

X1. SIGNIFICANCE OF STANDARD FOR FUEL METHANOL (M70-M85) FOR AUTOMOTIVE SPARK-IGNITION ENGINES

X1.1 Methanol

X1.1.1 The methanol content of M70-M85 is a crucial parameter as it affects the capability of the fuel metering system of the M70-M85 vehicle to establish the proper air-fuel ratio for optimum vehicle operation. This is much less of a concern for multi-fuel-capable vehicles than for dedicated M70-M85 vehicles. Methanol content affects the lubrication properties of the fuel and affects the water tolerance of the M70-M85.

X1.1.2 The inclusion of impurities and contaminants, except for the deliberately added hydrocarbons or additives, or both, can impact adversely on the properties and performance of fuel methanol (M70-M85) as an automotive spark-ignition engine fuel. The quantities of some of these materials are limited by specified property limits. Trace amounts of unspecified materials including higher alcohols, methyl formate, acetone, and dimethyl ether can be present. The maximum limit on water, the maximum limit on higher alcohols, and minimum-maximum limits on hydrocarbon/aliphatic ether content control the amount of some impurities and contaminants.

X1.2 Hydrocarbon

X1.2.1 Hydrocarbons are deliberately added to provide improved cold startability and cold-start and warm-up driveability. The addition of hydrocarbons also contributes to flame visibility (luminous flame), nonexplosive air-fuel mixtures in storage tanks (rich mixture vapor space), and denaturation (malodorant and taste deterrent). The hydrocarbon portion of the fuel must be unleaded.

X1.2.2 This specification does not control the composition of the hydrocarbons added to the fuel methanol. However, the hydrocarbons shall be stable, noncorrosive, and be in the boiling range of spark-ignition engine fuel as specified in Specification D 4814.

X1.3 Vapor Pressure

X1.3.1 The addition of volatile hydrocarbons improves cold startability. The addition of too much volatile hydrocarbons can cause hot fuel handling problems. When blending with gasoline as the hydrocarbon portion during the wintertime, a higher hydrocarbon content may be necessary to obtain required volatility. Higher vapor pressures are required in the wintertime for cold starting, and lower vapor pressures are needed in the summertime to prevent hot fuel handling problems. Excessive vapor pressure for a given ambient condition can contribute to evaporative emissions. Lower and upper limits on vapor pressure for three volatility classes are used to define the acceptable range of the volatile components to ensure proper vehicle performance.

X1.3.2 Three vapor pressure classes of fuel are provided to satisfy vehicle performance requirements under different climatic conditions. The schedule for seasonal and geographical distribution in Table 2 indicates the appropriate vapor pressure class (Class 1 through Class 3) for each month in all areas of the United States, based on altitude and expected air temperatures.

X1.4 Luminosity

X1.4.1 When pure methanol burns, it produces a blue, smokeless, nonluminous flame that is nearly invisible in daylight. Thus, it is difficult to know when a fire exists and to fight such a fire. A desirable property for M70-M85 fuel is that it maintain a clearly visible flame throughout the duration of a burn. It would be very hazardous for the visible flame to disappear before the fire was extinguished. For lack of a suitable test method for determining luminosity of M70-M85, luminosity is not controlled by this specification. (See X1.4.3.)

X1.4.2 To make a methanol flame visible, materials such as aromatic hydrocarbons are added to methanol. In general, it has been established that unleaded gasoline having greater than 30 volume % aromatics content when used as the hydrocarbon portion of M70-M85 will result in an M70-M85 fuel that will meet a requirement of a clearly visible flame throughout most of a burn. However, the luminosity performance is dependent on the types of aromatics present in the hydrocarbon portion.

X1.4.3 Appendix X2 contains a suggested procedure for measuring the luminosity of M70-M85 fuel. However, lack of a suitable criteria for establishing the relevancy of the procedure makes it unusable as the basis of a specification at this time. It is intended that a test be developed that will ensure adequate luminosity of M70-M85 based on performance, rather than on composition.

X1.5 Acidity

X1.5.1 Very dilute aqueous solutions of low molecular weight organic acids such as formic acid are highly corrosive to many metals. It is, therefore, necessary to keep such acids at a very low level.

X1.6 Gum Content, Solvent-Washed and Unwashed

X1.6.1 The test for solvent-washed gum content, measures the amount of residue after evaporation of the fuel and following a heptane wash. The heptane wash removes the heptane-soluble, nonvolatile material such as additives, carrier oils used with additives, and diesel fuels. Unwashed gum consists of fuel-insoluble gum and fuel-soluble gum. The fuel-insoluble portion can clog fuel filters. Both can be deposited on surfaces when the fuel evaporates.

X1.6.2 Solvent-washed gum content can contribute to deposits on the surfaces of carburetors, fuel injectors, and intake manifolds, ports, valves, and valve guides. The impact of solvent-washed gum on malfunctions of modern engines which can operate on fuel methanol (M70-M85) has not been fully

established but is based on limited experience gained with M70-M85 fuels in field tests and from historic gasoline limits. Performance effects depend on where the deposits form, the presence of other deposit precursors such as airborne debris, blowby and exhaust gas recirculation gases, oxidized engine oil, and the amount of deposit.

X1.6.3 The difference between the unwashed and solvent-washed gum content values can be used to assess the presence and amount of nonvolatile material in the fuel. Additional analytical testing is required to determine if the material is additive, carrier oil, diesel fuel, and so forth.

X1.6.4 The unwashed gum content limit is intended to limit high-boiling contaminants, like diesel fuel, that can affect engine performance, yet allow the proper dosage of deposit-control additives with carrier oils normally added to the hydrocarbon portion of the fuel methanol (M70-M85).

X1.6.5 Because the precision statements for Test Method D 381 were developed using only data on hydrocarbons, they may not be applicable to fuel methanol (M70-M85).

X1.7 Total Chlorine

X1.7.1 Ionic (inorganic) and organic chlorine are corrosive to many metals. It is desirable to minimize these compounds in fuel methanol (M70-M85).

X1.7.2 A total chlorine as chloride limit of 2 mg/kg, maximum, has been found to be inadequate in protecting some fuel system components. An inorganic chloride limit of 1 mg/kg, maximum, is specified to provide additional protection.

X1.8 Lead

X1.8.1 Most modern gasoline-powered vehicles are equipped with exhaust catalytic converters to control emissions of hydrocarbons, carbon monoxide, and oxides of nitrogen. Most fuel methanol vehicles are also equipped with exhaust catalysts that control emissions of formaldehyde as well as the regulated emissions. Lead compounds deactivate the catalysts and are limited to trace amounts to prevent this problem.

X1.9 Phosphorus

X1.9.1 Like lead, phosphorus deactivates exhaust catalysts and is limited to trace amounts.

X1.10 Appearance

X1.10.1 Turbidity, phase separation, or evidence of precipitation normally indicates contamination.

X1.11 Water

X1.11.1 The solubility of hydrocarbons in fuel methanol decreases with lowering temperature and increasing water content. Separation of the hydrocarbon from the fuel will adversely affect cold starting and driveability, luminosity, and taste-deterrence. Water may affect the calibration of some types of composition sensors of flexible-fuel vehicles. Water also reduces the energy content of the fuel and thus adversely affects fuel economy and power. Because some degree of water contamination is practically unavoidable in transport and handling, and because the fuel methanol is miscible with water, the water content of fuel methanol is limited to reduce the potential for problems.

X1.12 Sulfur

X1.12.1 The limit on sulfur content is included to protect against engine wear, deterioration of engine oil, corrosion of exhaust system parts, and exhaust catalyst deactivation.

X2. TEST METHOD FOR LUMINOSITY OF FUEL METHANOL (M70-M85) FOR AUTOMOTIVE SPARK-IGNITION ENGINES

X2.1 Scope

X2.1.1 This test method covers a procedure to determine if a fuel methanol (M70-M85) composition produces a luminous flame throughout the duration of a burn by comparing its luminosity performance under controlled conditions to that of ethanol. The test method in this appendix, while not adequate for use in its present form, is included here for information as it represents the current state of development for a luminosity procedure. It is hoped that its inclusion will solicit positive efforts toward development of a viable luminosity test procedure.

X2.1.2 The values stated in SI units are to be regarded as the standard. An exception is footcandles, lumens per square foot, that is a hybrid unit and the unit used for the calibration of the optometer.

X2.2 Referenced Document

X2.2.1 *ASTM Standard*:

E 1145 Specification for Denatured Ethyl Alcohol, Formula 3A[7]

X2.3 Summary of Test Method

X2.3.1 The sample of fuel methanol is measured into a Petri dish resting on a digital scale under a daylight spectrum source (achieved by the use of two daylight spectrum fluorescent bulbs) in a fume hood. The sample is ignited and the output of an optometer and the decreasing output of the scale are recorded on a two pen recorder. A video tape recording of the event is also recommended. A comparison is then made to ethanol under the same conditions.

X2.4 Significance and Use

X2.4.1 Potentially low-luminosity (invisible) methanol fires are a major safety consideration. Ensuring that the fuel methanol burns with a visible flame over the entire duration of a burn allows visual recognition of a hazardous situation. The test method in this appendix measures a property related to such visual recognition.

X2.5 Apparatus

X2.5.1 *Digital Scale*, having an accuracy of 0.1 g.

X2.5.2 *Borosilicate Petri Dishes*, clean, 100-mm diameter by 20 mm high, one per sample.

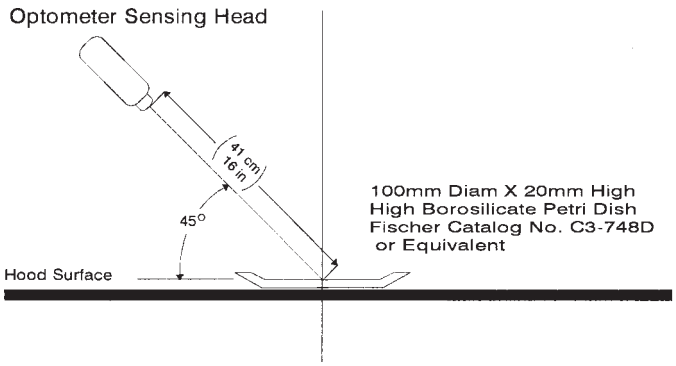

FIG. X2.1 Optometer Sensing Head Position

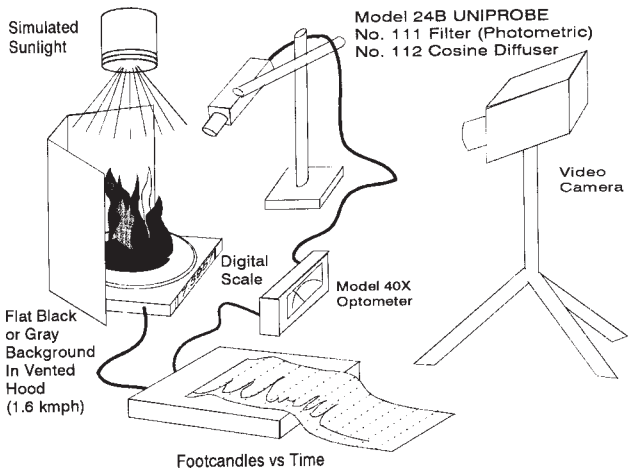

FIG. X2.2 Luminosity Test Apparatus Schematic

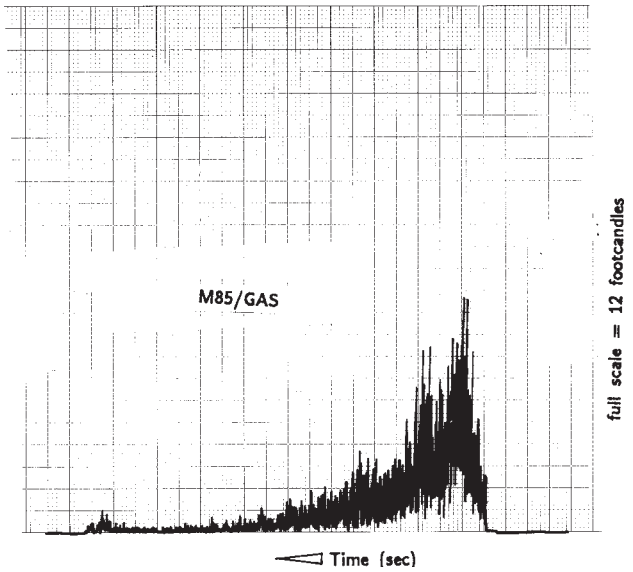

FIG. X2.3 Luminosity Trace of M85, 30 % Aromatic Gasoline

X2.5.3 *Disposable Syringes or Clean Pipettes*, 10 ± 0.1 mL.

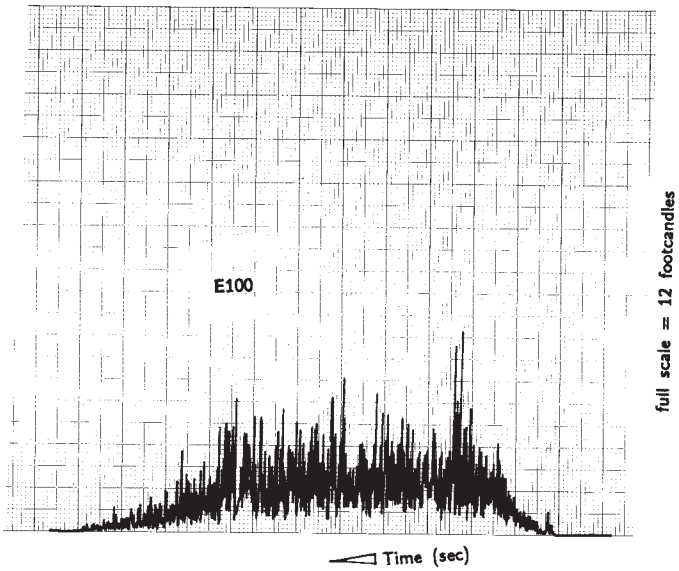

FIG. X2.4 Luminosity Trace of E100

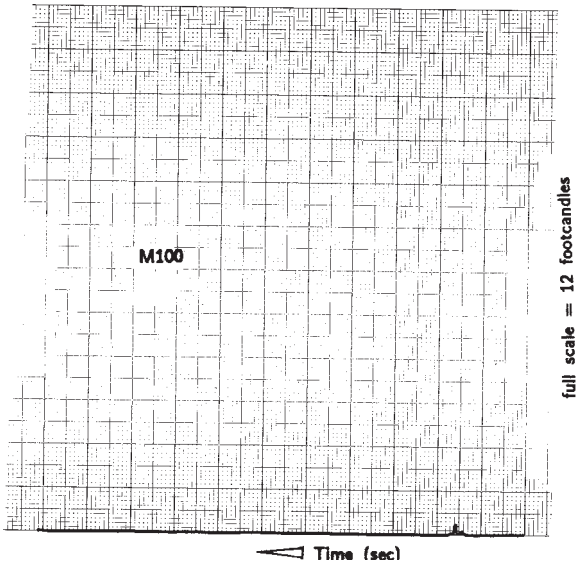

FIG. X2.5 Luminosity Trace of M100

X2.5.4 *Optometer*, with photometric (400 to 700-nm) filter.[14]

X2.5.5 *Two-Channel Pen Recorder*.

X2.5.6 *Color Video Camera and Video Cassettes*.

X2.5.7 *Daylight Spectrum Fluorescent Bulbs*.

X2.6 Reagents and Materials

X2.6.1 *Ethanol*—See Specification E 1145. (**Warning**—Flammable. Health hazard. Denatured alcohol cannot be made nontoxic.)

[14] The United Detector Technology Model 40× optometer, or an equivalent, has been found suitable for this purpose.

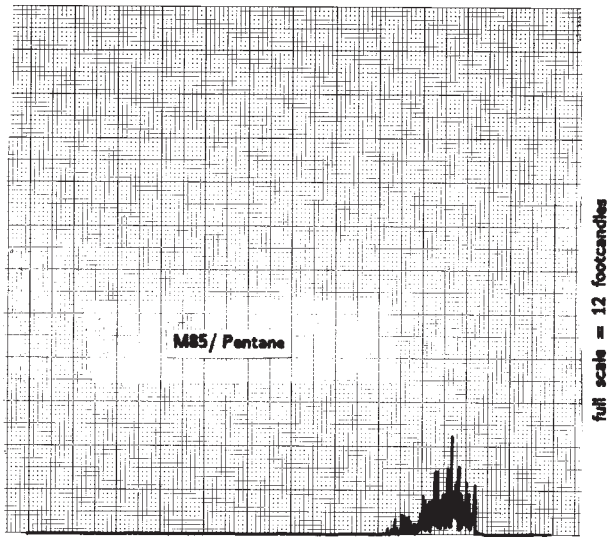

FIG. X2.6 Luminosity Trace of M85/Pentane

X2.7 Procedure

X2.7.1 In a laboratory hood equipped with two 30-W daylight spectrum fluorescent bulbs, introduce a 10-mL fuel sample from a clean 10-mL pipette or syringe into a borosilicate Petri dish resting on the pan of a tared digital balance. A standard grey background is recommended if a video recorder is used. Position the optometer sensing head on a 45° angle pointed toward the center of the Petri dish, 41 cm (16.1 in.) from the vertical center line of the Petri dish (see Fig. X2.1). After zeroing the amplifier with the daylight spectrum fluorescent bulbs on, adjust the amplifier scale to 1.0 fc (10.8 lm/m^2) for burning ethanol (denatured ethyl alcohol, Formula 3A, see Specification E 1145) in the lighted hood.

X2.7.2 Strike an ordinary wooden match and wave over the test sample, initiating burning at time zero, at which point simultaneously start the video timer graphics and chart feed. Record flame luminosity, measured in footcandles ($lm/ft^2 = 10.8\ lm/m^2$), and weight decrease concurrently on the chart. Termination of the burn is evidenced by the scale's return to zero weight. Obtain the base luminosity value by continuing the trace after the burn. The entire apparatus is shown in Fig. X2.2.

X2.8 Interpretation of Results

X2.8.1 Review the output of the optometer and scale as recorded on the dual-pen recorder (and the video, if recorded) to ensure acceptable luminosity over the entire burn duration. Figs. X2.3-X2.6 illustrate traces that are typical of the results obtained with this technique.

X2.8.2 A numerical result obtained by mathematical comparison of the ethanol standard burn to that of the M70-M85 burn, that would ensure a visible flame for the entire duration of the burn is desirable but unavailable at this time.

X2.8.3 The video recording of the burn is important because luminosity by the suggested test method in this appendix does not measure the flame color that is available upon review of the video. Flame color, in addition to luminosity, are important in assessing the visibility of a flame.

X2.8.4 Correlation with actual vehicle fires has not been established.

X2.9 Precision and Bias

X2.9.1 *Precision*—The precision of the suggested test method in this appendix for measuring the luminosity of fuel methanol (M70-M85) has not been determined.

X2.9.2 *Bias*—The bias of the suggested test method in this appendix has not been determined.

X2.10 Keywords

X2.10.1 automotive spark-ignition engine fuel; daylight spectrum fluorescent bulb; duration of a burn; ethanol; flame; illuminance; luminosity; luminous flame; M70-M85; methanol; standard daylight spectrum source; video