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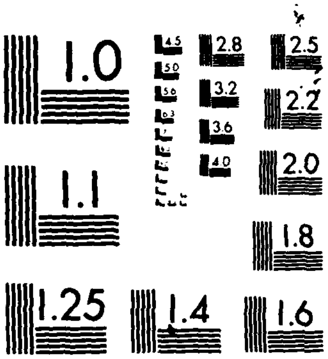

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DETAILED CHEMICAL ANALYSIS OF FUEL METHANOL FOR TRACE IMPURITIES

INTERIM REPORT
BFLRF No. 216

By
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Under Contract to
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Fort Belvoir, Virginia

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September 1986

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19. ABSTRACT *(Continue on reverse if necessary and identify by block number)*

Two samples of methanol, one a coal-derived product and the other a natural gas-derived product, were obtained for analysis. The coal-derived methanol was received in a 5-gallon steel can and the natural gas-derived methanol was received in 1-gallon glass bottles. The detailed analysis reported in this interim report provides an initial assessment of the compositional differences between the two methanols produced from different raw materials.

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FOREWORD

This report was prepared at the Belvoir Fuels and Lubricants Research Facility (SwRI) located at Southwest Research Institute, San Antonio, TX, under Contract No. DAAK70-85-C-0007, for the period 1 January 1986 through 1 August 1986. Work was funded by the U.S. Army Belvoir Research, Development and Engineering Center, Ft. Belvoir, VA, with Mr. F.W. Schaekel (STRBE-VF) serving as contracting officer's representative. Project technical monitor was Mr. M.E. LePera, STRBE-VF.

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I. INTRODUCTION

Section 202(a) of the 1985 Department of Defense Authorization Act requested that 50 percent of the methanol used in the U.S. Army Methanol-Fueled Administrative Vehicle Demonstration Program be derived from coal. The remaining 50 percent would be derived from natural gas. The methanol fuel used in the demonstration program contains approximately 85 vol% methanol with the remaining 15 percent being primarily unleaded gasoline. Methanol fuel specifications obtained from three different sources are presented in the Appendix.

The detailed analysis reported here provides an initial assessment of the compositional differences between the two methanols produced from different raw materials.

Two samples of methanol designated for use as fuel were obtained for analysis. Methanol produced from coal was assigned sample number AL-14571-F. Methanol derived from natural gas was assigned sample number AL-14737-F. The coal-derived product, AL-14571-F, was received in a 5-gallon steel can, and the natural gas-derived product, AL-14737-F, was received in 1-gallon glass bottles.

II. EXPERIMENTAL

The fuel methanol samples were analyzed for trace contaminants considered possible based on the chemical processes involved in production and those contaminants from shipping and handling.

- Water Content - Water was determined by Karl Fischer method using a Photovolt Aquatest IV automatic analyzer.
- Chemically Bound Nitrogen - Nitrogen was determined using an Antek Chemiluminescence Nitrogen Analyzer.
- Total Acidity - Total acid number was determined using a Fisher Scientific Computer Aided Titrimeter to perform ASTM D 664.
- Phosphorus Content - Phosphorus was determined by ASTM D 3231.
- Sulfur Content - Sulfur was determined using a Dohrmann Microcoulometric system, ASTM Method D 3120.

- Organic Chloride Content - Organic chloride was determined using modified ASTM Method D 3120.
- Trace Metals - Trace metals were determined using Atomic Absorption Spectroscopy with both flame and graphite furnace capabilities.
- Lead Content - Atomic absorption was used to determine lead content.

The results of these analyses are shown in Table 1 and are discussed in the following section.

TABLE 1. ANALYSIS OF FUEL METHANOL FOR TRACE CONTAMINANTS

Determination	AL-14571-F	AL-14737-F	Limits of Detection
Water, Karl Fischer, wt%	0.023	0.049	0.001
Nitrogen, Chemiluminescence, ppm	<5	<5	5
Titratable Acid, meq/g	0.0003	0.00004	
Phosphorus, g/L	ND*	ND	0.001
Sulfur, Microcoulometric, ppm	ND	ND	10
Chlorine, Microcoulometric, ppm	ND	ND	10
Trace Metals, AAS, ppb			
Cr	ND	ND	5
Cu	2.5	12	5
Zn	130	10	5
Si	ND	ND	10
Mg	ND	ND	5
Al	ND	ND	5
Na	ND	ND	125
Pb, AAS, g/L	ND	ND	0.001

*ND = None Detected.

III. RESULTS/DISCUSSION

Water content of the two samples as-received is not high for methanol samples. Reagent grade American Chemical Society certified methanol contains approximately 0.02 wt% water. The difference between the two samples is not enough to be considered significant.

Nitrogen values below 5 ppm obtained by chemiluminescence are useful only as estimates. Sample AL-14737-F contained an estimated 0.1 ppm nitrogen and AL-14571 approximately 0.4 ppm nitrogen. Since these are estimated values, the difference is not significant.

Total acidity was calculated from the total acid numbers obtained by ASTM D 664. The calculated total acidity for the samples are 0.0003 meq/g for AL-14571-F and 0.00004 meq/g for AL-14737-F. American Chemical Society certified reagent grade methanol contains approximately 0.0003 meq/g titratable acid, which is similar to the higher acidity value found in these samples.

Sulfur was not detected in either sample using ASTM Method D 3120. Detection limit for the sulfur is 10 ppm. No chlorine was detected in either sample using the Dohrmann microcoulometric system in a modified Method D 3120. This method has a limit of detection of 10 ppm, and detects organic chlorides.

Trace metals were determined using atomic absorption spectroscopy (AAS). Sodium (Na) was determined using flame atomization of the sample. Limit of detection for sodium using this method is 125 parts per billion (ppb). The other trace metals were determined using a graphite furnace to atomize the sample. Limits of detection for the elements using this method range from 5 ppb for chromium, magnesium, and aluminum to 10 ppb for silicon. Note in Table 1 that the sample with higher zinc content was received in a steel can, while the other sample, AL-14737-F, was received in a glass container. This could account for the higher zinc content if the can was zinc coated. Lead was determined using AAS with a lead in gasoline reference for calibration.

IV. ANALYSIS BY GAS CHROMATOGRAPHY AND GAS CHROMATOGRAPHY/MASS SELECTIVE DETECTION

A. High-Resolution Gas Chromatography

Each methanol sample was analyzed using high-resolution gas chromatography (HRGC) with a 50-meter narrow bore fused silica capillary column. The neat sample was injected using a split mode injection technique and a flame ionization detector (FID) which responds to organic compounds. The HRGC analytical conditions are shown in Table 2.

TABLE 2. HIGH-RESOLUTION CAPILLARY GAS CHROMATOGRAPHY CONDITIONS

Column Oven Temperature Program: 0°C, 1 min; 0° to 150°C at 2°/min;
150°C for 10 min

Flame Ionization Detector Temperature: 300°C
Injector: Split Mode
Split Ratio 100:1
Temperature: 250°C

Column: 50 meter, 0.2 mm ID, SE-54
Carrier Gas: Helium
Flow Rate: 1 cc/min

Injection Size: 1 or 2 microliters

Chromatograms of the methanol fuels did not indicate any component other than methanol. Since the methanol peak was large and broad due to the large injection amount necessary to detect very small amounts of contaminants, the possibility of other components coeluting with the methanol was considered. Individual mixtures of possible contaminants in methanol were prepared (Table 3). Using the listed analytical conditions, each of the mixtures was analyzed to determine the retention time of each in relation to the major component, methanol.

TABLE 3. POSSIBLE METHANOL CONTAMINANTS

Contaminant	Dilution in Methanol
Formic Acid	0.1%, 10% Vol/Vol
Acetic Acid	0.1%, 10% Vol/Vol
Formaldehyde	0.1%, 10% Vol/Vol
Acetaldehyde	0.1%, 10% Vol/Vol
Acetone	0.1%, 10% Vol/Vol
Ethanol	0.1%, 10% Vol/Vol

Clear indication and separation of the added components at 0.1 percent using the 200:1 split ratios were not adequate, so the analytical task was redirected to a gas chromatograph with a mass selective detector.

B. Gas Chromatography/Mass Selective Detection

The two methanol samples were examined using mass spectroscopic detection following a high-resolution gas chromatographic separation (HRGC/MSD). The HRGC serves as the inlet into the MSD. The HRGC conditions are shown in Table 4. The MS data acquisition parameters are shown in Table 5.

TABLE 4. HIGH-RESOLUTION GAS CHROMATOGRAPH CONDITIONS FOR MASS SELECTIVE DETECTOR

Column Oven Temperature Program

Run Time: 30.00 min	
Equilibration Time:	2.00 min
	0°C, 1 min
	0°C to 100°C at 4°C/min
	100°C for 4 min

Heated Zones	Set Pt., °C	Limit, °C
Oven (Standby)	0	325
Inj. Port B	150	400
Transfer Line	280	300

TABLE 5. MASS SELECTIVE DETECTION DATA ACQUISITION PARAMETERS

Solvent Delay:	1.00 min.
eM Volts:	0 Relative
Resulting Voltage:	1600
Start Time:	1.00 min
Low Mass:	10.0
High Mass:	100.0
Scan Threshold:	10
a/d samples (2N):	2
Scans per Second:	4.75

A detailed analysis of the data obtained for each sample was made by examining the total ion chromatogram (TIC), as well as a scan-by-scan search for compounds other than methanol. The compounds searched for by comparison to the retention time of standards, as well as mass spectral comparisons, are shown in Table 2. However, the search was not limited to those compounds only, but was carefully examined for any possible contaminant in the methanol.

The TIC (Figure 1) is a reconstructed chromatogram generated by merging all the ions detected in the spectrometer at a given time. Discrete, user-selected increments of time may be selected by the analyst for closer scrutiny by enlargement of the details in both the X and Y axes (Figure 2).

Figure 3 shows the mass spectrum of methanol. Subtraction of the mass spectrum generated by the instrument background (Figure 4) from the mass spectrum of the methanol (Figure 5) yields a "pure" spectrum of methanol. The large methanol peak for each sample shown in the TIC, Figure 1, was searched scan by scan for any differences that might indicate the presence of something other than background and methanol within the methanol peak and therefore not visible in the TIC.

In addition, the entire time frame of the analysis was searched for other components by enlargement of discrete time sections for the possible appearance of very small peaks not readily discernible at the normal presentation size.

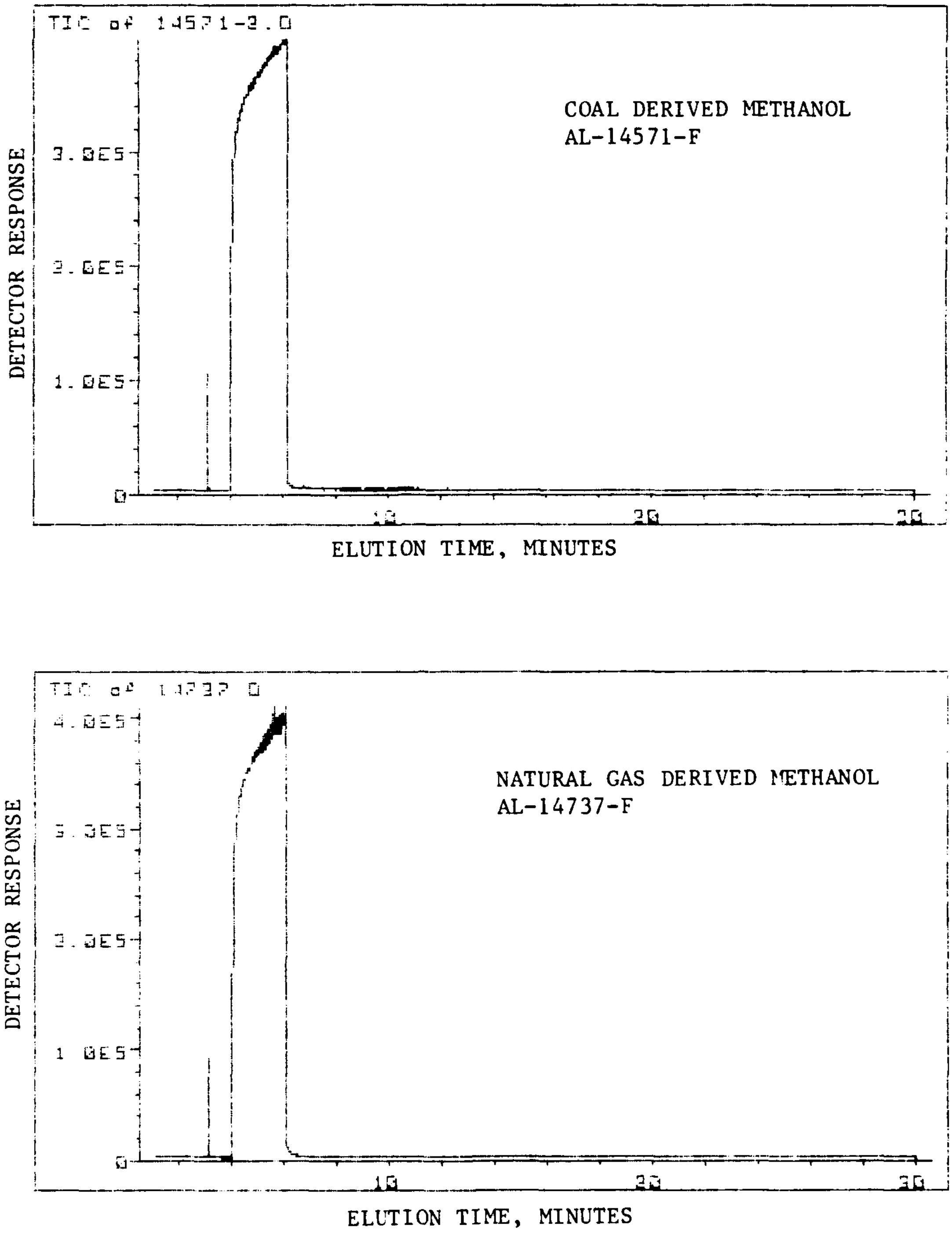

FIGURE 1. RECONSTRUCTED TOTAL ION CHROMATOGRAM OF TWO METHANOL SAMPLES

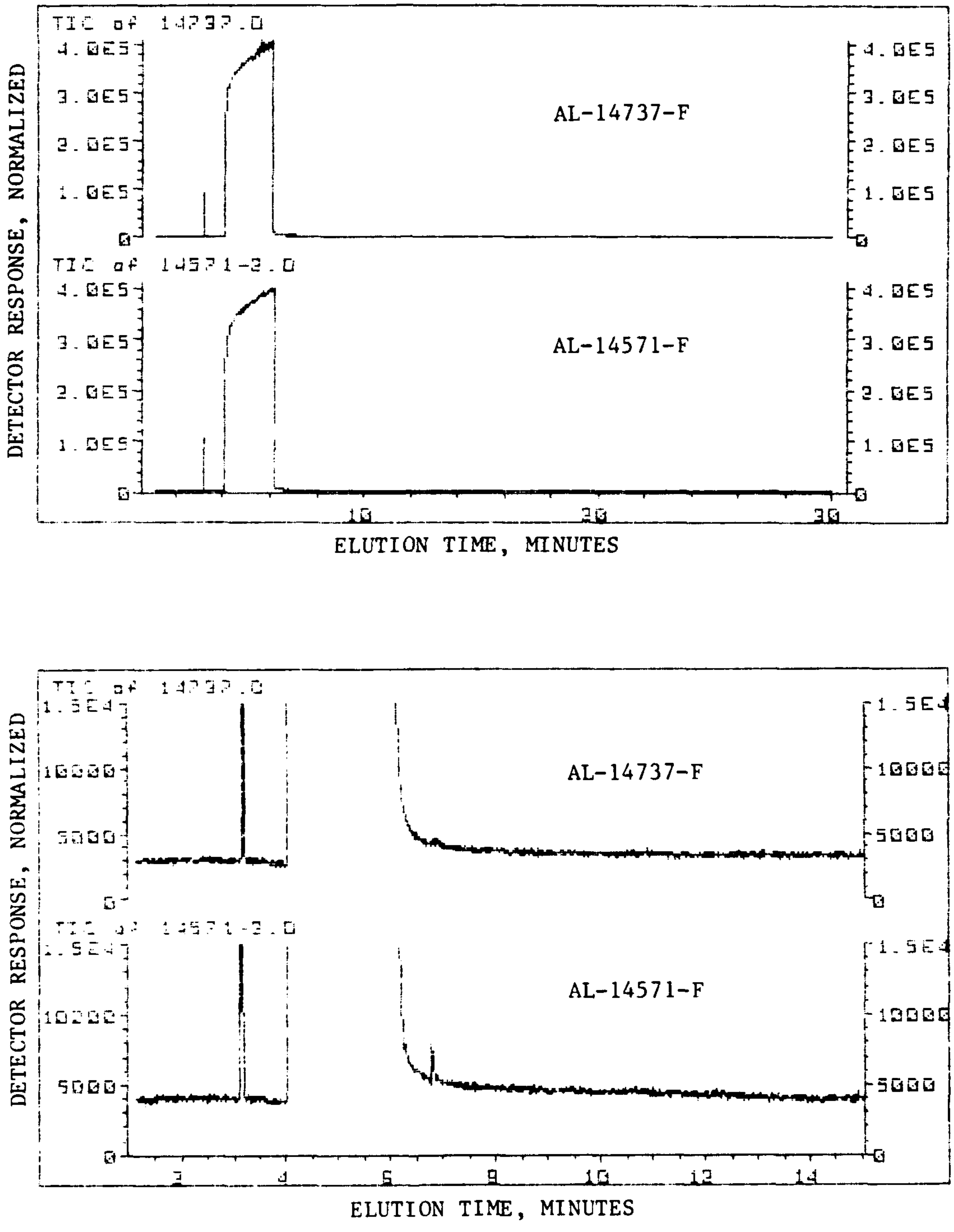

FIGURE 2. RECONSTRUCTED TOTAL ION CHROMATOGRAM OF TWO METHANOL SAMPLES WITH X/Y AXIS ENLARGEMENT

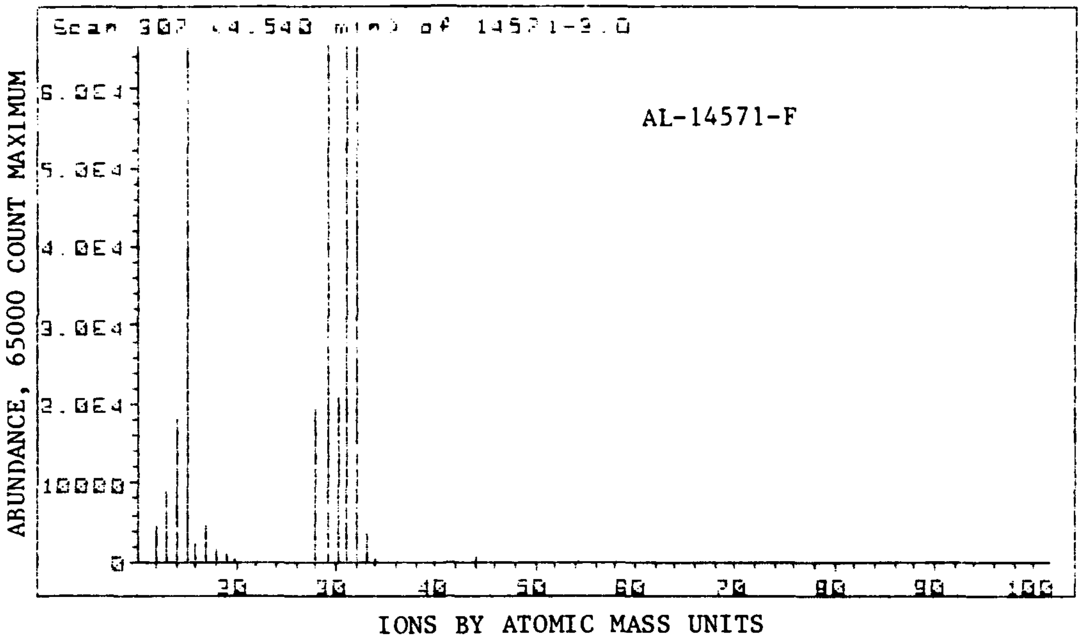

FIGURE 3. MASS SPECTRUM OF METHANOL FROM SAMPLE AL-14571-F

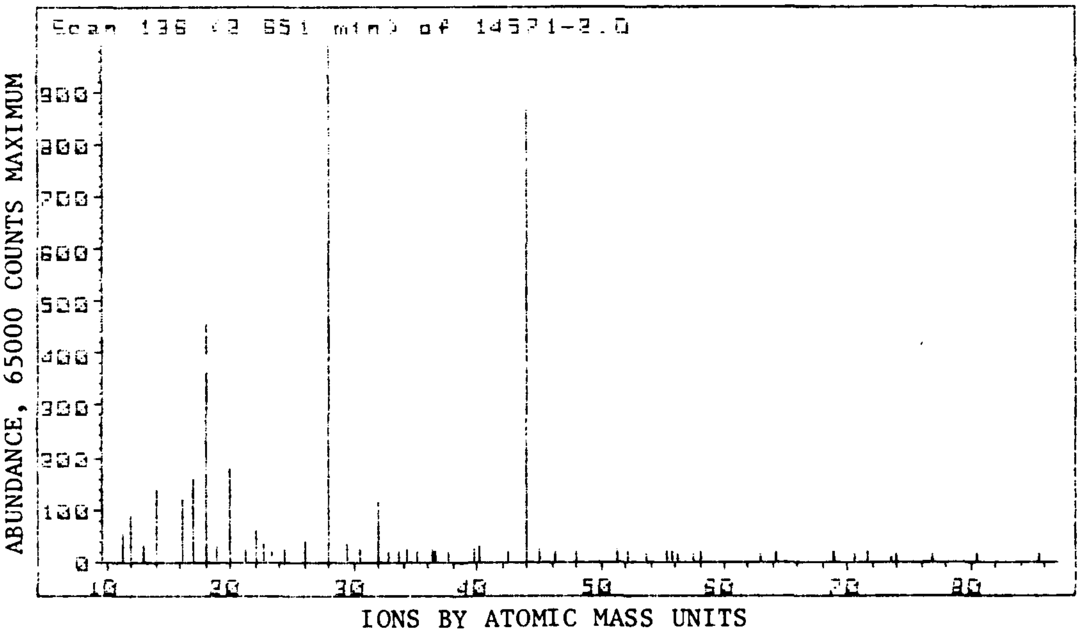

FIGURE 4. MASS SPECTRUM OF INSTRUMENT BACKGROUND

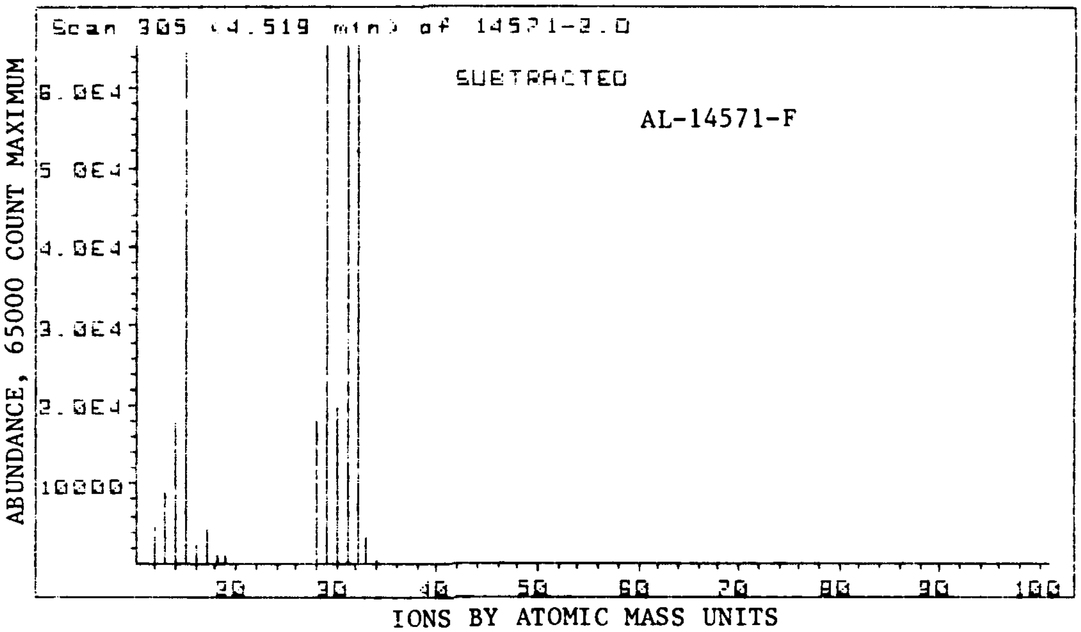

FIGURE 5. MASS SPECTRUM OF METHANOL FROM SAMPLE AL-14571-F WITH INSTRUMENT BACKGROUND SUBTRACTED

V. GC RESULTS AND DISCUSSION

The HRGC/FID results showed that this method was not effective for the detection of contaminants in concentrations less than 100 ppm. This technique did confirm that there were no contaminants present above 0.1 percent. Therefore, the investigation was shifted to the HRGC/MSD. This technique provides greater sensitivity, but requires a thorough knowledge of organic chemistry for the interpretation of the mass spectral data. The HRGC/MSD analysis showed the two methanol samples to be very pure. The only impurity detected was ethanol, and this only in the coal-derived methanol, AL-14571-F. This is graphically shown, by comparison, in Figure 6, the TIC of the two methanol samples, and Figure 7, which shows the enlargement of the time segment containing the impurity. The mass spectrum of the small peak confirmed that the impurity was ethanol (Figure 8). Figure 9 shows the mass spectrum of ethanol. A series of standards of ethanol in ethanol-free methanol was prepared. Detector response to ethanol was graphically plotted, peak area versus concentration. The ethanol in AL-14571-F was determined to be present at 15 ppm (0.0015%). The reagent grade methanol stocked in the chemical laboratory and used to prepare the standards was found to be less pure than the samples in question when analyzed by HRGC/MSD. The reagent methanol was found to contain trace amounts of ethanolamine, which is used in industry to scrub CO_2 and H_2S from the gas streams used to produce methanol. Ethanolamine was not detected in either of the two methanol samples in question.

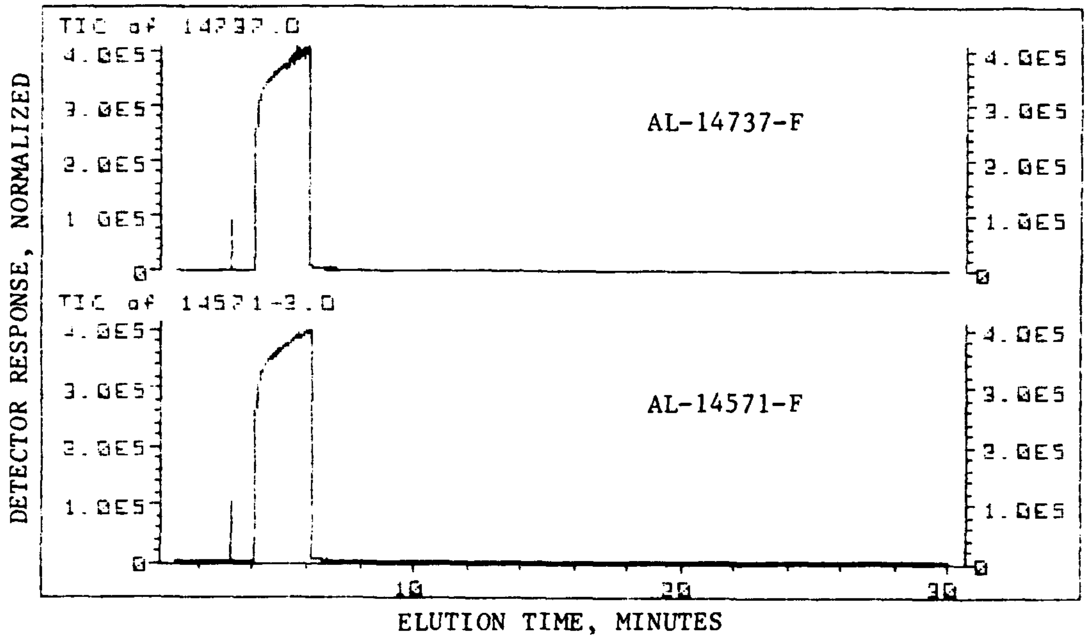

FIGURE 6. TIC OF TWO METHANOL SAMPLES

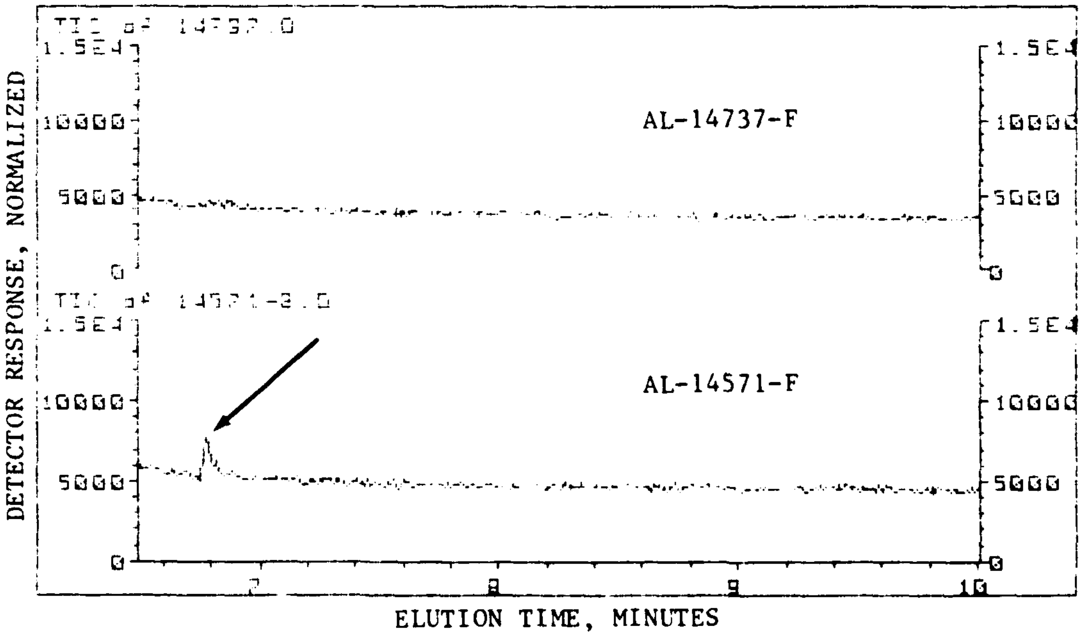

FIGURE 7. ENLARGEMENT OF 6- TO 10-MINUTE TIME SEGMENT

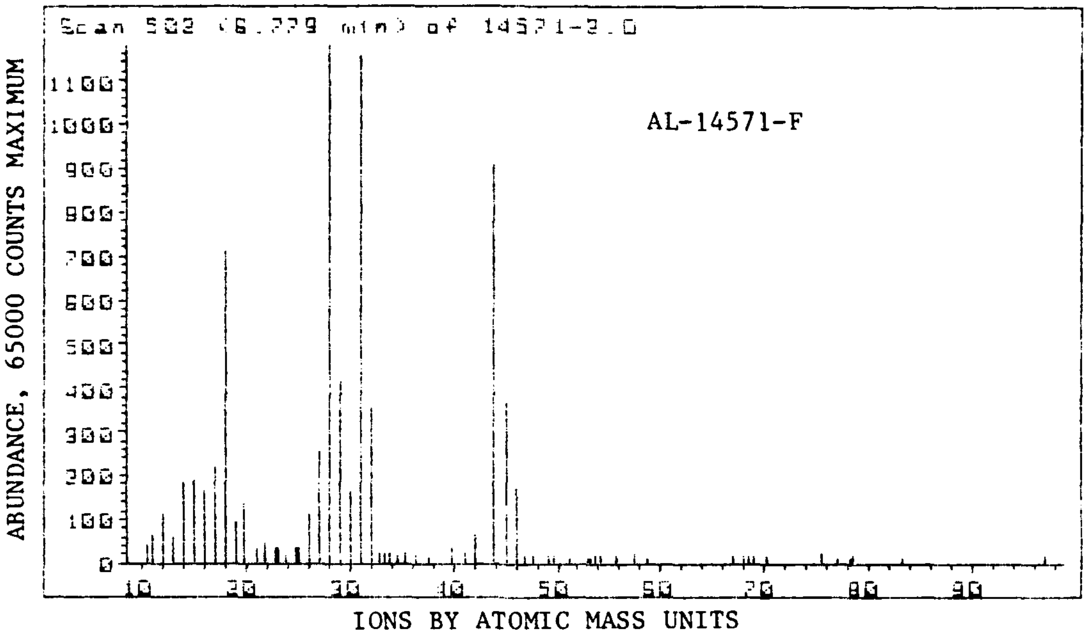

FIGURE 8. MASS SPECTRUM OF SMALL PEAK AT 6.8 MINUTES

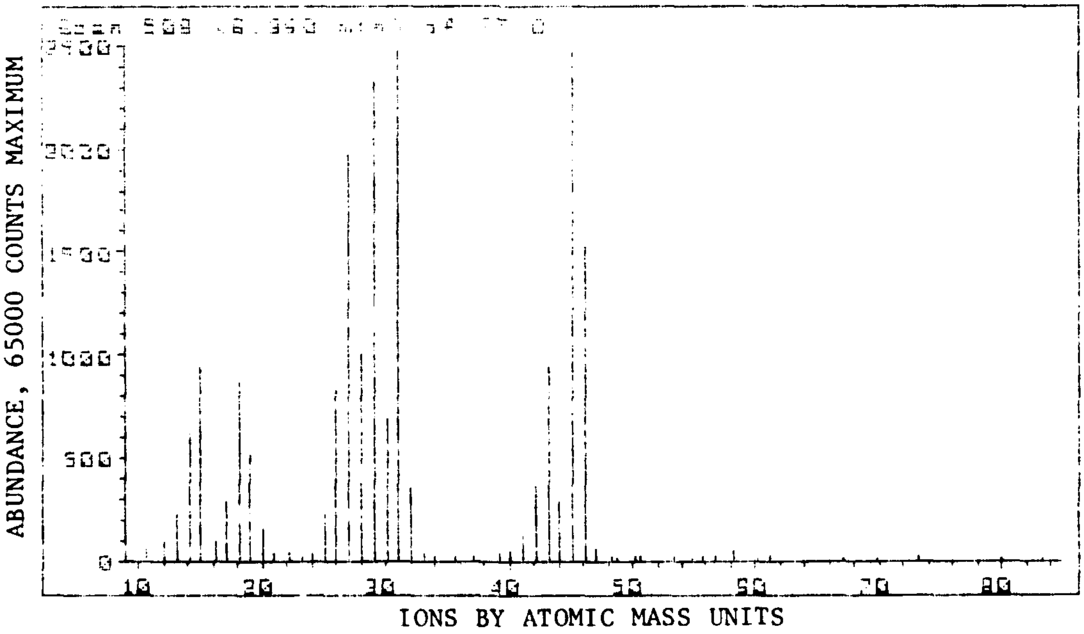

FIGURE 9. MASS SPECTRUM OF ETHANOL

VI. SUMMARY

Table 6 summarizes the analysis of fuel methanol for trace contaminants.

TABLE 6. SUMMARY OF ANALYSIS OF FUEL METHANOL FOR TRACE CONTAMINANTS

Determination	AL-14571-F	AL-14737-F	Limits of Detection
Water, Karl Fischer, wt%	0.023	0.049	0.001
Nitrogen, Chemiluminescence, ppm	<5	<5	5
Titratable Acid, meq/g	0.0003	0.00004	
Phosphorus, g/L	ND*	ND	0.001
Sulfur, Microcoulometric, ppm	ND	ND	10
Chlorine, Microcoulometric, ppm	ND	ND	10
Trace Metals, AAS, ppb			
Cr	ND	ND	5
Cu	2.5	12	5
Zn	130	10	5
Si	ND	ND	10
Mg	ND	ND	5
Al	ND	ND	5
Na	ND	ND	125
Pb, AAS, g/L	ND	ND	0.001
Mass Spectral Analysis, Impurities			
Ethanol, ppm	15	ND	

*ND = None Detected.

Examination of the two methanol samples in question by HRGC showed no significant amounts of impurities present (greater than 100 ppm), but was not sensitive enough to detect trace amounts.

Further analysis by HRGC/MSD showed a trace amount of ethanol, 15 ppm, present in sample AL-14571-F, the coal-derived methanol. In-depth examination of the MS analytical data did not show any other impurities in either of the samples. Trace contaminant analyses showed no significant difference between the two methanols. Concentrations of water and titratable acid were within acceptable limits for reagent grade ACS certified methanol. Trace metal contaminants were found only in low part per billion concentrations. The observed trace metal concentrations could be due to contamination from metal containers used in shipping and handling.

It should be pointed out that all the analyses performed were on the samples as-received. Water content and other trace contamination in methanols for use as fuels will vary according to shipping and handling procedures.

VII. CONCLUSION

Both methanol samples were very pure, with only the coal-derived sample, AL-14571-F having 15 ppm of ethanol present.

No contamination was detected which would affect use of either methanol as a fuel for internal combustion engines.

APPENDIX

METHANOL FUEL SPECIFICATIONS

Engineering Material Specification

Material Name	Specification Number
FUEL, METHANOL - GASOLINE MODIFIED - INITIAL FILL	ESE-M4C97-B

1. SCOPE

The material defined by this specification is a gasoline-modified methanol blend with controlled acidity, chloride, lead, phosphorus and water levels.

2. APPLICATION

This specification was released originally for a methanol fuel used for initial fill and general purpose use.

3. REQUIREMENTS

3.1	METHANOL, By Volume, min (ASTM D 1152, 99.85% grade)	84%*
3.2	GASOLINE, UNLEADED, By Volume (ASTM D 439)	14 ± 1%*
3.2.1	Hydrocarbon Composition (of Gasoline) (ASTM D 1319)	
	Aromatics, min	40%
3.3	VAPOR PRESSURE (ASTM D 323)	40 - 65 kPa
3.4	ACIDITY, max (ASTM D 1613)	0.003%
3.5	DISTILLATION RESIDUE, max (ASTM D 86)	0.5%

*The following test method shall be used when determining methanol-gasoline content.

ASTM D 3545 is to be modified for the determination of methanol, utilizing the gas chromatograph described therein. Acidity and water content results determined in Para 3.4 and 3.11 are to be used to normalize values obtained by gas chromatography. Note that the remaining portion of the fuel mixture is to be reported as gasoline.

Date	Release No.	Release/Revision
1984 11-19		Released CE3P-RD 863100-E131 CSherwood

3.6	TOTAL CHLORIDE CONTENT, ORGANIC AND INORGANIC, max (ASTM D 3120, Modified & ASTM D 2988)	0.0002%

ASTM D 3120 is modified for the determination of organic chlorides.

3.7	LEAD CONTENT, max (ASTM D 3237)	0.003 g/L
3.8	PHOSPHORUS CONTENT, max (ASTM D 3231)	0.001 g/L
3.9	SULFUR CONTENT, max (ASTM D 3120)	0.01%
3.10	PARTICULATE CONTAMINANTS, max (ASTM D 2276)	0.1 g/L
3.11	WATER, By Volume, max (ASTM D 1744)	0.5%

3.12 ODOR

Shall have the usual characteristic odor of methanol and gasoline.

3.13 COLOR

May include a dye in accordance with standard practice for purpose of vendor and user identification.

3.14 CERTIFICATION

Supplier shall certify that the methanol fuel is a formula that has been tested and meets Ford Motor Company requirements as stated in this specification. Technical evidence of these data and manufacturing limits must be tabulated on the attached form. This certification shall be by the supplier's Director of Research or other authorized official and submitted to the responsible Ford purchasing activity. This information will then be forwarded to the materials activity of the Engine Product Engineering Office and the Fuels Analysis Laboratory, Research Staff.

3.15 SUPPLIER'S RESPONSIBILITY

All materials supplied to this specification must be equivalent in all characteristics to the material upon which approval was originally granted.

Prior to making any change in the properties, composition, construction, color, processing or labelling of the material originally approved under this specification, whether or not such changes affect the material's ability to meet the specification requirements, the Supplier shall notify Purchasing, Toxicology and the affected Materials Engineering activity of the proposed changes and obtain the written approval of the Materials Engineering activity. Test data, test samples and a new code identification are to be submitted with the request.

Substance restrictions imposed by law, regulations or Ford, apply to the materials addressed by this document. The restrictions are defined in Engineering Material Specification WSS-M99P9999-A1, unless a different suffix (e.g., A2 or A3) is specified on the engineering document for the application.

4. APPROVAL OF MATERIALS

Materials defined by this specification must have prior approval by the responsible Materials Engineering activity. Suppliers desiring approval of their materials shall first obtain an expression of interest from the affected Purchasing, Design and Materials Engineering activity. Upon request, the Supplier shall submit to the affected Materials Engineering activity its own laboratory report to the specification (test results, not nominal values), the material designation and code number, and test specimens for Ford evaluation. Upon approval, the material will be added to the Engineering Material Approved Source List.

5. GENERAL INFORMATION

5.1 FLAMMABILITY

This material represents a potential fire hazard. The Manufacturing Engineering Department or other responsible activity shall notify both the Security and Safety Units of its presence to assure that proper fire precautions are provided. The location in which it is used or stored shall be included in the notifications.

5.2 SAMPLING AND SUBMISSION OF SAMPLES

(ASTM D 270)

5.3 Quality Control records must be made available to the responsible product engineering office upon request.

Company ______________________ Date ______________________

Refinery Location ______________________ Specification ______________________

	Actual Test Results (Typical)	Manufacturing Limits
Methanol, Vol. %		
Gasoline, Vol. %		
Vapor Pressure, kPa, (ASTM D 323)		
Acidity, %, (ASTM D 1613)		
Distillation Residue, % (ASTM D 86)		
Chloride Content		
Organic, %, (ASTM D 3120 Modified)		
Inorganic, %, (ASTM D 2988)		
Lead Content, g/L, (ASTM D 3237)		
Permanganate, min., (ASTM D 1363)		
Phosphorus Content, g/L, (ASTM D 3231)		
Sulfur Content, %, (ASTM D 3120)		
Particulate Contaminants, g/L, (ASTM D 2276)		
Water, %, (ASTM D 1744)		

Additives	Type and Formula	Manufacturing Limits
Corrosion Inhibitor		
Detergent		
Dye		
Upper Cylinder Lubricant		
Miscellaneous		

Signature ______________________

Title ______________________

09/09/85

BANK OF AMERICA
METHANOL FUEL SPECIFICATIONS
AND TESTING METHODS

1. METHANOL, By Volume for a) M-85, b) M-86, & c) M-88 (ASTM D 1152, 99.85% grade)
   a) 85.0 + 1.0, -0.0%
   b) 86.0 + 1.0, -0.0%
   c) 88.0 + 1.0, -0.0%

   ASTM D 3545 is to be modified* for the determination of methanol, utilizing the gas chromatograph described therein. Acidity and water content results determined in 5 and 12 are to be used to normalize values obtained by gas chromatography. Note that the remaining portion of the fuel mixture is to be reported as gasoline.

2. GASOLINE, PREMIUM UNLEADED, By Volume (ASTM D 439), 9-11 psi RVP Aromatics in Gasoline, 40% min. by volume, ASTM D1319
   a) 15.0 +0.0, -1.0%
   b) 14.0 +0.0, -1.0%
   c) 12.0 +0.0, -1.0%

3. ADDITIVE, FA5**, By Weight — 2 lb/1000 gal

4. VAPOR PRESSURE, dry (ASTM D 323) — 40-65 kPa

5. ACIDITY, wt%, max. (ASTM D 1613) — 0.003%

6. DISTILLATION RESIDUE, max. (ASTM D 86) — 0.5%

7. TOTAL CHLORIDE CONTENT, ORGANIC AND INORGANIC, max. (ASTM D 3120, Modified & ASTM D 2988) — 0.0002%

   ASTM D 3120 is modified for the determination of organic chlorides .

8. LEAD CONTENT, max. (ASTM D 3237) — 0.003 g/L

9. PHOSPHORUS CONTENT, max. (ASTM D 3231) — 0.001 g/L

10. SULFUR CONTENT, max. (ASTM D 3120) — 0.015%

11. PARTICULATE CONTAMINANTS, max. (ASTM D 2276) — 0.1 g/L

12. WATER, wt%, max. (ASTM D 1744) — 0.5%

* The specified test method shall be used when determining methanol-gasoline content.

** FA5 is a proprietary fuel additive for use with methanol. It is available from the Bank of America.

STATE OF CALIFORNIA
FUEL SPECIFICATION/TESTING METHODS
(Subject to change)
Rev. 12/12/84

1.	METHANOL, By Volume, Min (ASTM D 1152,99.85% grade)	85.0%*

*The following test method shall be used when determining methanol-gasoline content.

ASTM D 3545 is to be modified for the determination of methanol, utilizing the gas chromatograph described therein. Acidity and water content results determined in 4 and 11 are to be used to normalize values obtained by gas chromatography. Note that the remaining portion of the fuel mixture is to be reported as gasoline.

2.	GASOLINE, PREMIUM UNLEADED, By Volume (ASTM D 439), 9-11 psi RVP Aromatics in Gasoline, 40% min. by volume ASTM D1319	14.5±0.5%
3.	VAPOR PRESSURE, dry (ASTM D 323)	40-65 kPa
4.	ACIDITY, wt%, max (ASTM D 1613)	0.003%
5.	DISTILLATION RESIDUE, max (ASTM D 86)	0.5%
6.	TOTAL CHLORIDE CONTENT, ORGANIC AND INORGANIC max (ASTM D 3120, Modified & ASTM D 2988)	0.0002%
7.	LEAD CONTENT, Max (ASTM D 3237)	0.003 g/L
8.	PHOSPHOROUS CONTENT, max (ASTM D 3231)	0.001 g/L
9.	SULFUR CONTENT, max (ASTM D 3120)	0.015%
10.	PARTICULATE CONTAMINANTS, max (ASTM D 2276)	0.1 g/L
11.	WATER. wt%, max (ASTM D 1744)	0.5%

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