U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1829

Alcohols in Reference Fuel

This Standard Reference Material (SRM) is intended for use in calibrating instruments and evaluating techniques used for the determination of specific alcohols in gasoline. SRM 1829 consists of three different alcohols in reference fuel solutions: one is nominally 9 volume percent methanol and 6 volume percent t-butanol (Solution 1); another is nominally 10 volume percent ethanol (Solution 2); and a third is nominally 0.3 volume percent methanol (Solution 3).

Certified Concentrations: The certified alcohol concentrations, in grams of alcohol per 100 grams of solution (weight percent) and in milliliters of alcohol per 100 milliliters of solution (volume percent), are shown in Table 1. For solutions 1 and 2, the certified concentrations, in weight percent, are based on the mass of alcohol added to the mass of reference fuel and experimental measurements by gas chromatography (GC), and for solution 3, the certified concentration is based in addition on measurements by isotope dilution mass spectrometry (ID-MS). These results are shown in Table 2. The certified concentration, in mL/100 mL at 23 °C, was calculated by dividing the certified concentration in g/100 g by the density of the appropriate alcohol at 23 °C and multiplying by the density of the solution at 23 °C.

Table 1

Certified Concentrations

Solution	Alcohol	Concentration	
		Weight Percent, g/100 g	Volume Percent, mL/100 mL at 23±2 °C
1	Methanol	10.33 ± 0.09	9.18 ± 0.08
	t-Butanol	6.63 ± 0.02	5.92 ± 0.02
2	Ethanol	11.39 ± 0.04	10.08 ± 0.04
3	Methanol	0.335 ± 0.014	0.292 ± 0.012

The uncertainties listed represent two standard deviations of the certified concentrations and include all observable variabilities. For concentrations in mL/100 mL, the uncertainties include allowances for changes in density in the 21 to 25 °C range.

The densities of the alcohols in g/mL at 23 °C used in calculating the concentrations in volume percent are: ethanol 0.7868, methanol 0.7885, and t-butanol 0.7834 (Thermodynamics Research Center Table, Values of Properties of Chemical Compounds, Volume 1, Table 23-2-1-(1-1020)-d). The densities of the solutions, in g/mL, at 23 °C are: solution 1, 0.7005; solution 2, 0.6965; and solution 3, 0.6884.

Analyses leading to certification were performed at the NBS Center for Analytical Chemistry, Organic Analytical Research Division by M. (Miller) Schantz, R.E. Rebbert, and M.J. Welch.

The statistical analysis of the data was performed by R.C. Paule of the NBS National Measurement Laboratory.

The coordination of the technical measurements leading to certification was under the direction of S.N. Chesler, W.E. May, and E. White V.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Gaithersburg, MD 20899
March 19, 1986

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(Over)

NOTICE AND WARNINGS TO USER

FLAMMABLE: Caution, these materials are flammable and should be given the same care in handling as gasoline. See Material Safety Data Sheet.

Expiration of Certification: This certification is valid, within the limit certified, for two years from the date of purchase. In the event that the certification should become invalid before then, purchasers will be notified by NBS.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures between 10 and 30 °C.

Use: Samples for analysis should be withdrawn immediately after opening ampoules and should be processed without delay for certified values in Table 1 to be valid within the stated uncertainty. For use of the volume percent certified values, the temperature of the samples should be 23 ± 2 °C. Certified values are not applicable to material stored in ampoules that have been opened, even if they are resealed.

Extra precautions should be taken with ampoules of solution 3 because the low concentration and high volatility of methanol make this solution particularly susceptible to handling errors. In particular, trace concentrations of water (approximately 0.01 wt%) can drastically decrease the solubility of methanol in the reference fuel causing phase separation.

Table 2

Summary of Results (g/100 g)

Solution	Alcohol	Calculated[a]	GC	ID/MS
1	Methanol	10.29	10.38 ±0.25[b]	- - -
	t-Butanol	6.63	6.63 ±0.07	- - -
2	Ethanol	11.40	11.38 ±0.18	- - -
3	Methanol	0.345	0.321 ±0.011	0.336 ±0.010[b]

[a]Calculated from the mass of alcohol, corrected for purity, added to the mass of reference fuel.

[b]One standard deviation of a single measurement.

Preparation and Analysis: The reference fuel for the three solutions was supplied by Phillips Petroleum Company. It is a 91-octane (Research Octane Number) fuel, which is a mixture of 91 volume percent (91.09 weight percent) 2,2,4-trimethylpentane (iso-octane) and 9 volume percent (8.91 weight percent) n-heptane. The methanol and ethanol used had a determined purity of greater than 99.9% while the t-butanol had a determined purity of 99.3%. The alcohol-reference fuel solutions were gravimetrically prepared (the calculated concentration in Table 2) at NBS. All three solutions were analyzed by capillary gas chromatography with flame ionization detection using t-amyl alcohol as an internal standard. A fused silica column (30 m x 0.25 mm I.D. x 0.25 μm film of bonded dimethyl polysiloxane) was used with a split injection system. Solution 3 was also analyzed by isotope dilution mass spectrometry using methanol-^{18}O as the isotope diluent.