National Institute of Standards & Technology

# Certificate of Analysis

## Standard Reference Material 2289

### t-Amyl-methyl Ether in Reference Gasoline
(Nominal 2.7 Weight Percent Oxygen)

This Standard Reference Material (SRM) is intended primarily for use as a primary standard for the calibration of instruments used for the determination of the oxygen content of motor fuels. It consists of a solution of t-amyl-methyl ether (Chemical Abstracts Service (CAS) Registry Number 994-05-8) in a reference gasoline. Certified concentrations are provided for t-amyl-methyl ether and oxygen. A unit of SRM 2289 consists of two 20 mL ampoules of the t-amyl-methyl ether in reference gasoline solution, each containing approximately 18 mL, and one 20 mL ampoule containing approximately 18 mL of the reference gasoline, intended for use as a measurement blank.

**Certified Concentrations:** The certified concentrations and estimated uncertainties for t-amyl-methyl ether and oxygen are given below. These concentrations are based on results obtained from the gravimetric preparation of the solution of t-amyl-methyl ether in gasoline and from the analytical results determined using gas chromatography, corrected for impurities.

| Component | Wt %<br>(g/100 g Solution) |
|---|---|
| t-Amyl-methyl Ether | 17.30 ± 0.20 |
| Oxygen | 2.73 ± 0.04 |

**Uncertainty:** The uncertainty of each value on this document is the numerical value of an expanded uncertainty $U = ku_c$, with $U$ determined from a combined standard uncertainty $u_c$ and a coverage factor, k = 2. The expanded uncertainty defines a range of values for the certified value within which the true value is believed to lie, at a level of confidence of 95% [1].

**NOTICE AND WARNING TO USERS**

**Handling and Storage:** Provide local exhaust or general dilution ventilation to meet published exposure limits. Those handling gasoline should wear appropriate clothing and gloves to prevent skin contact with this material and splash-proof or dust resistant safety goggles to prevent eye contact. Store in a cool, dry, and well-ventilated area away from incompatible materials and prevent contact with heat sparks or open flame. Protect ampoules from physical damage. Please read the MSDS for this material before use.

Analytical determinations were performed by S.N. Chesler and T.L. Green of the NIST Analytical Chemistry Division. The coordination of the technical measurements leading to certification was under the direction of S.N. Chesler and F.R. Guenther of the NIST Analytical Chemistry Division. Data analysis was provided by D. Duewer of the NIST Analytical Chemistry Division and reviewed by K. Eberhart of the NIST Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.E. Gills and J.S. Kane.

Gaithersburg, MD 20899
January 25, 1995

Thomas E. Gills, Chief
Standard Reference Materials Program

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**Expiration of Certification:** The certified values are valid, within the limits specified, for 5 years from the date of shipment from NIST. In the event that the certification should become invalid before then, purchasers will be notified by NIST. Please return the attached registration card to facilitate notification.

**Use:** Sample aliquots for analysis should be withdrawn at 20 to 25 °C immediately after opening the ampoules and should be processed without delay for the certified values to be valid within the stated uncertainty. Because of the volatility of some of the components in the reference gasoline, certified values are not applicable to material stored in ampoules that have been opened for more than 5 min, even if they are resealed.

**Preparation and Gravimetric Analysis:** This SRM was prepared by G. Lew of Scott Specialty Gases Inc. at Scott's facilities in Houston, TX. All chemicals used in the preparation of this material were obtained from commercial sources. The reference gasoline was obtained from Phillips Petroleum, Bartlesville, OK and was identified as RF-A. HPLC Grade t-amyl-methyl ether was obtained from Aldrich Chemical Co., Milwaukee, WI.

This SRM was prepared by additive weighing of the RF-A gasoline and t-amyl-methyl ether. After preparation and homogenization, the solution was aliquoted into 20 mL glass ampoules which were subsequently flame sealed. The gravimetrically determined concentration of t-amyl-methyl ether in the SRM solution was (17.50 $\pm$ 0.02) g/100 g solution.

**Gas Chromatographic Analysis:** The concentration of the t-amyl-methyl ether in the SRM solution was determined by capillary column gas chromatography using flame ionization detection (GC-FID). Analysis was based upon the ratio of the elution peak area of t-amyl-methyl ether and that of a gasoline component. The gasoline component selected had a large, well-resolved peak with a similar retention time to t-amyl-methyl ether. t-Amyl-methyl ether solution concentration was calculated from a calibration curve constructed from peak area ratios for unmodified RF-A gasoline blank and NIST gravimetrically prepared standards. This concentration of t-amyl-methyl ether in the SRM solution was (17.55 $\pm$ 0.12) g/100 g solution.

The organic purity of the t-amyl-methyl ether was also determined using GC-FID. Based on 12 replicate analyses, it was determined to be (98.79 $\pm$ 0.04)%.

The water concentration of the SRM solution was determined by capillary column gas chromatography using atomic emission detection (GC-AED) in oxygen-specific mode. Based on 3 replicate analyses, it was determined to be (0.02 $\pm$ 0.01) g/100 g SRM solution. The concentration of oxygen from trace organic impurities was also determined using GC-AED. No significant oxygen-containing impurities other than water were found in this SRM.

The t-amyl-methyl ether content in the SRM solution was calculated from the consensus concentration of t-amyl-methyl ether in the SRM solution, with corrections for both water and organic purity. The molecular oxygen content in the SRM was calculated from the sum of the corrected t-amyl-methyl ether concentration and water concentration.

## REFERENCE

[1] *"Guide to the Expression of Uncertainty in Measurement"*, ISBN 92-67-10188-9 1st Ed. ISO, Geneva, Switzerland, (1993).