

National Bureau of Standards

Report of Investigation

Reference Material 8511

Volatile Toxic Organics in Nitrogen (Ambient Air Standard)

A Reference Material (RM) as defined by ISO Guide 30-1981(E), is a material or substance, one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

RM 8511 was developed to provide a preliminary multi-component, NBS-traceable standard in the low part-per-billion (ppb) range, in the absence of SRM's, for the calibration of instruments used for the measurement of volatile toxic organic compounds in state ambient air monitoring programs. RM 8511 contains the seven organic compounds listed below in a pure nitrogen gas matrix contained in a high-pressure aluminum cylinder. While RM 8511 is not an SRM, the concentration values of the organic compounds that it contains were determined using the same analytical methods used to prepare NBS-traceable mixtures of volatile toxic organic compounds in the ppb range and by the same analytical methods that will be used to certify future SRM's of such mixtures.

| | |
|---------------------------|--------------------------|
| Benzene..... | 5.0 ± 0.8 nanomoles/mole |
| Chloroform | 5.2 ± 0.3 nanomoles/mole |
| Methylene Chloride..... | 3.5 ± 0.2 nanomoles/mole |
| Trichloroethylene..... | 5.3 ± 0.3 nanomoles/mole |
| Tetrachloroethylene..... | 5.2 ± 0.3 nanomoles/mole |
| 1,2-Dichloroethane | 5.1 ± 0.2 nanomoles/mole |
| Carbon Tetrachloride..... | 5.2 ± 0.2 nanomoles/mole |

The cylinder number and cylinder pressure for each of the five RM's in this lot are as follows:

| <u>Cylinder Number</u> | <u>Pressure (psi)</u> |
|------------------------|-----------------------|
| CAL-11232 | 1625 |
| CAL-11239 | 1600 |
| CAL-11240 | 1450 |
| CAL-11252 | 1500 |
| CAL-11253 | 1600 |

The preparation and analysis of RM 8511 were carried out by G.C. Rhoderick, with the overall coordination for its development provided by W.L. Zielinski, Jr., of the NBS Gas and Particulate Science Division.

Support aspects for the issuance of RM 8511 were coordinated through the Office of Standard Reference Materials by T.E. Gills.

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Gaithersburg, MD 20899

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Background

RM 8511 was developed through joint support of the Quality Assurance Division of the Environmental Monitoring Systems Laboratory of the U.S. Environmental Protection Agency in Research Triangle Park, NC (EPA/EMSL/RTP), and the NBS Gas and Particulate Science Division. The seven volatile organic compounds in this RM were identified and selected for inclusion in this reference mixture in discussions with staff of EPA/EMSL/RTP and state monitoring programs.

Preparation and Analysis

RM 8511 was prepared as a homogenous set of five 30 cubic feet aluminum cylinders by the simultaneous transfer from a 150 cubic feet aluminum cylinder that contained a gravimetrically prepared mixture of the seven volatile toxic organics in pure nitrogen at a nominal concentration level of 11 ppb. Following this transfer, additional pure nitrogen was added to all six cylinders to achieve a target nominal 5 ppb concentration level for the organic compounds in all six cylinders. Analyses of the organic compounds in each of these cylinder mixtures were carried out against three independently prepared gravimetric standards containing all seven organics in the 2-8 ppb range. The analytical results confirmed that the concentration of each organic compound was the same throughout the six cylinder set, within their stated uncertainties.

The uncertainty of each concentration was determined at the 95% confidence limit by summing, in quadrature, the uncertainty in the preparation of the gravimetric standards, the analytical imprecision in comparing the samples with the gravimetric standards, and the analytical imprecision of replicate analyses of each organic compound.

All analyses for RM 8511 were carried out by gas chromatography (GC) using peak height measurements. Typical analytical conditions were as follows:

For benzene, analyses were carried out using a GC system containing a flame-ionization detector, a gas sampling valve having a 10-mL sample loop, and a 10-foot by 1/8-inch O.D. stainless-steel column packed with 5% SP-1000 on 100-120 mesh Supelcoport* operated at a column oven temperature of 80°C and a nitrogen carrier gas flow rate of 30 mL/minute.

For the remaining six organic compounds, analyses were carried out using a GC system containing an electron-capture detector, a gas sampling valve having a 0.5 mL sample loop, and a 20-foot by 1/8-inch O.D. stainless-steel column packed with a 10% SP-1000 on 100-120 mesh Supelcoport* operated at a column oven temperature of 100°C and a nitrogen carrier gas flow rate of 30 mL/minute.

Stability

Based on experience with these and numerous similar mixtures of these compounds, the concentration values assigned to the seven organic compounds in RM 8511 should be stable within their stated uncertainties for a period of at least one year from date of receipt. The sixth cylinder (150-cubic feet) will be retained at NBS for long-term stability measurements, and will be included in any future reanalyses of the five RM's to verify their long-term stability.

*Commercial products are identified to adequately describe materials and experimental procedures used in the preparation and analysis of RM 8511. In no instance does such identification imply endorsement by NBS of the particular product or equipment as being the best available for that purpose.