National Institute of Standards & Technology

# Certificate of Analysis

## Standard Reference Material® 2906

### Trace Explosives Calibration Solutions

This Standard Reference Material (SRM) is intended for use in calibrating and evaluating analytical equipment used for the detection of trace explosives, which may include those based on ion mobility spectrometry [1]. SRM 2906 consists of three dilute solutions of hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX), 2,4,6-trinitrotoluene (TNT), and pentaerythritol tetranitrate (PETN) A unit of SRM 2906 consists of four glass ampoules of each of the three explosives containing approximately 1 mL of the solution and four vials of the 2-propanol solvent used to prepare the solutions. In addition, there are four labeled dropper bottles for temporary storage of the solutions once opened.

The concentrations of the explosives in SRM 2906 were prepared to approximately satisfy the requirements of ASTM Standard E2520-07 *Standard Practice for Verifying Minimum Acceptable Performance of Trace Explosive Detectors* [2].

**Certified Values:** Certified values, expressed as mass fractions (μg/g) and as concentrations (μg/mL) at 23 °C for the three explosives are provided in Table 1. The certified concentration values for the three explosives are based on the agreement of results obtained at NIST using two independent analytical techniques [3,4]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [3].

**Expiration of Certification:** The certification of **SRM 2906** is valid, within the measurement uncertainties specified, until **31 December 2015**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage, Handling, and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

**Maintenance of SRM Certification:** NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination of the technical measurements leading to the certification of this SRM were performed by W.A. MacCrehan of the NIST Analytical Chemistry Division.

Analytical measurements for the certification of SRM 2906 were performed at NIST by A. Spessard and W.A. MacCrehan of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh of the NIST Statistical Engineering Division.

This SRM was produced with partial support from the Office of Standards at the Department of Homeland Security.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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Certificate Issue Date: 01 October 2010

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## INSTRUCTIONS FOR STORAGE, HANDLING, AND USE

**Storage:** To maintain the certified values for long periods, SRM 2906 must be stored in its original ampoules at temperatures of approximately –20 °C (freezer temperature) away from direct sunlight. Once transferred to the dropper bottles, the material may be stored for short periods (up to 1 day) at room temperature with minimal changes in analytical levels. The caps of the dropper bottles should be screwed tightly onto the bottles at all times to avoid the influx of moisture between uses.

**Handling:** This is a fabricated material containing constituents of known and unknown toxicities. Caution should be exercised during its handling and use. When handling the materials, the use of chemically resistant gloves is recommended.

**Use:** Following a period to allow the material to reach room temperature (between 20 °C and 25 °C), open the package and remove the individual vials of material. Prior to removal of subsamples for analysis, the contents of the ampoule should be mixed by inverting the container three times. The ampoule is pre-scored.

If desired, to follow the ASTM E2520-07 protocol, the entirety of the ampoule may be transferred to the clean plastic dropper bottle labeled for the specific explosive. For transfer, the dispenser tip of the plastic bottle may be removed; the entirety of the contents of the ampoule may be transferred by placing the plastic receiver bottle over the opened ampoule and quickly inverting. The tip may then be replaced for dispensing. Once transferred to the dropper bottle, the solution is stable in a tightly-capped bottle for about 1 day at room temperature. Drops should be expressed by continuous application of pressure, assuring that none of the drop was drawn back into the bottle following dispensation. The estimated mass of each of the three explosives dispensed in a single drop is provided in Table 2. Before refilling used dropper bottles with fresh vials of solution, the bottles should be rinsed with rubbing or other alcohol and dried. *For precise measurements achieving the certified values provided in Table 1, the exact mass or volume of the solution should always be determined.*

## PREPARATION AND ANALYSIS[1]

**Sample Preparation:** The solution calibrants were prepared by dissolution of pure RDX, TNT, and PETN into separate volumes of 2-propanol with stirring for 8 hours. The materials were stored at –20 °C prior to shipment.

### Composition of the Explosives

Two approaches were used to determine the explosives content of the materials in SRM 2906, gravimetry and liquid chromatography with absorbance detection (LC/UV). The LC/UV method utilized an octadecylsilyl-modified silica separation column (Restek Pinnacle DB C-18, Bellefonte, PA, 5 µm particles, 4.6 mm × 250 mm) with an isocratic elution using a water:acetonitrile mobile phase. Detection was at 230 nm for RDX and TNT and 210 nm for PETN.

**Internal Standards Used:** For the LC/UV determinations of RDX and TNT, 4-nitrobenzonitrile (recrystallized from 50/50 (volume fractions) pentane:acetone) was used as the internal standard. For the determinations of PETN, external calibration was used.

**Homogeneity Assessment:** The homogeneity of SRM 2906 was assessed by analyzing duplicate samples from each of 18 ampoules selected by stratified random sampling. No statistically significant differences among bottles were observed for the three explosives within the specified uncertainties.

[1]Certain commercial equipment, instruments, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 1. Certified Concentrations for RDX, PETN, and TNT in SRM 2906

| Explosive | Mass Fraction[(a)] (μg/g) | Mass Concentration[(b)] (μg/mL) |
|---|---|---|
| RDX | 0.189 ± 0.005 | 0.148 ± 0.004 |
| PETN | 2.15 ± 0.06[(c)] | 1.68 ± 0.05[(c)] |
| TNT | 1.27 ± 0.07 | 0.99 ± 0.05 |

[(a)] The results are expressed as the certified value ± the expanded uncertainty. The certified value is a weighted average of concentrations [4,5] determined by gravimetric and chromatographic measurements, corrected for purity. The expanded 95 % uncertainty uses a coverage factor of $t_{(1,0.975)}$ and includes both correction of estimated purity and allowance for differences between the concentrations determined by gravimetric preparation and LC/UV measurements by combining a between-method variance with a pooled, within-method variance [6] following the ISO Guide [7].

[(b)] Determined utilizing an estimated density of isopropanol of 0.7827 g/mL at 23 °C (73 °F).

[(c)] The expanded uncertainty includes the components listed in footnote a as well as a larger component for type B uncertainty in purity and calibration.

Table 2. Information Values for the Mass of RDX, PETN, and TNT Dispensed per Drop of SRM 2906 Using the Provided Dropper Bottles

| Explosive | Mass[(a)] (ng) |
|---|---|
| RDX | 3.66 ± 0.50 |
| PETN | 41.6 ± 5.7 |
| TNT | 24.6 ± 3.4 |

[(a)] The associated uncertainties provide a 95 % confidence that 95 % of the true drop mass values are within the stated interval of the mean mass given [8,9].

## REFERENCES


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[3] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260–136 (2000); available at http://ts.nist.gov/MeasurementServices/ReferenceMaterials/PUBLICATIONS.cfm (accessed Oct 2010).

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[8] Gardiner, D.A.; Hull, N.C.; *An Approximation to Two-Sided Tolerance Limits for Normal Populations*; Technometrics, Vol. 8, No. 1, pp. 115–122 (1966).

[9] Hahn, G.J.; Meeker, W.O.; *Statistical Intervals: A Guide to Practitioners*; John Wiley and Sons: New York (1991).


*Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.*