National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2907

Trace Terrorist Explosives Simulants

This Standard Reference Material (SRM) is a surrogate for explosives residues and is intended for use in evaluating analytical equipment used for the detection of trace explosives, which may include those based on ion mobility spectrometry. SRM 2907 consists of two non-explosive materials prepared from inert particles that are coated with trace levels of explosives. One material contains the plastic explosive Semtex 1A with a nominal mass fraction of 0.04 % and a second material contains TATP (triacetone triperoxide, 3,3,6,6,9,9-hexamethyl-1,2,4,5,7,8-hexaoxacyclononane) with a nominal mass fraction of 0.4 %. The 0.04 % Semtex is certified for the mass fraction of pentaerythritol tetranitrate (PETN) and the 0.4 % TATP is certified for the mass fraction of TATP. A unit of SRM 2907 consists of individual plastic squeeze bottles each containing approximately 1 g of the two trace terrorist explosives simulant materials.

Certified Mass Fraction Values: Certified values, expressed as mass fractions, for the two explosives are provided in Tables 1 and 2. The certified values for the two explosives are based on the agreement of results obtained at NIST using two independent analytical techniques [1]. 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 [1].

Expiration of Certification: The certification of **SRM 2907** is valid, within the measurement uncertainties specified, until **31 December 2016**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see “Instructions for Use”). This 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 coordination of the technical measurements leading to the certification of SRM 2907 was under the leadership of W.A. MacCrehan of the NIST Analytical Chemistry Division.

Analytical measurements for the certification of SRM 2907 were performed at NIST by 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.

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

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

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Certificate Issue Date: 30 January 2012

Robert L. Watters Jr., Chief
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NOTICE AND WARNING TO USERS

Storage: To maintain the certified values for long periods, SRM 2907 must be stored in its original bottle at temperatures of approximately –20 °C (freezer temperature) away from direct sunlight. The material may be stored for moderate periods (up to 1 week) at room temperature with no loss of the analytes. The caps of the 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. The use of a particle filter mask is recommended.

INSTRUCTIONS FOR USE

Following a period to allow the packaging to reach room temperature, open the package and remove the individual bottles of material. Prior to removal of subsamples for analysis, the contents of the bottle should be mixed by inverting the container at least three times. It may be necessary to enlarge the nozzle of the bottle with a pin or other small, sharp object to ensure free flow of the contents.

PREPARATION AND ANALYSIS[1]

The preparation and analysis of this SRM material has been described in detail [2].

Sample Preparation: The simulant material for Semtex was prepared by dissolution of neat Semtex in diethylether with ultrasonic agitation. The TATP simulant material was prepared by dissolution of the crystalline material in 2-methylbutane. Each of the respective solutions was diluted with a sufficient volume of solvent to cover the respective substrates to be coated. The 0.04 % Semtex substrate employed an octadecylsilyl-modified silica (Protein and Peptide C18, GraceVydac, Hesperia, CA) with a 20 µm to 30 µm particle size. The 0.4 % TATP substrate was poly(styrene-divinylbenzene) polymer (PRP-1, Hamilton, Reno, NV) with a particle size between 12 µm and 20 µm. A rotary evaporator was used to mix the materials and for the removal of the solvents. The two materials were dried for an extended period at elevated temperature under vacuum. The materials have been 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 2907, liquid chromatography (LC) with ultraviolet absorbance detection (UV) and mass spectrometric detection (MS). The LCUV method for both PETN and TATP utilized an octadecylsilyl-modified silica separation column (MACMOD, Chadds Ford, PA) ACE3 C18 (3 µm, 3.0 mm × 150 mm) with a gradient elution solvent program using water and acetonitrile. Detection was at 210 nm. All samples were solvent extracted with ultrasonic agitation. For LCUV determination of TATP in the 0.4 % TATP material, ≈50 mg samples were extracted with three fresh volumes of acetonitrile (1 mL) for 30 min each and combined. For the determination of PETN in the 0.04 % Semtex material, a single extraction of ≈50 mg with 1 mL of acetonitrile for 99 min was used. The LCMS separations used an octadecylsilyl-modified silica separation column (Agilent Technologies, Santa Clara, CA), Eclipse XDB-C18 (5 µm, 4.6 mm × 150 mm). Atmospheric pressure chemical ionization (APCI) mass spectrometry was used for determination of TATP and PETN with an isocratic methanol and water mobile phase. For the determination of TATP and PETN by LCMS, ≈50 mg sample was extracted with 1 mL of isopropanol for 99 min. For TATP MS, ammonium acetate (10 mmol/L, pH 5.5), was added as a mobile phase additive [3]. An adduct ion, corresponding to $m/z\ (M + NH_4)^+$ was monitored for APCI MS detection. In both the LCUV and LCMS determinations of TATP, the two conformational isomers [4] were separated and the sum of the two peak areas was taken for the determination of the TATP content. For the APCI MS determination of PETN a mobile phase additive, 0.2 mmol/L ammonium nitrate, was included. This produced an adduct ion corresponding to $m/z\ (M + NO_3)^-$, which was monitored for PETN determination.

Internal Standards Used: For the LCUV determinations of both TATP and PETN, 4-nitrobenzonitrile (recrystallized from 50 % volume fraction pentane/acetone) was used as the internal standard. For the LCMS determination of TATP, the stable isotope-labeled analog ^{13}C-TATP was used. For PETN determination, BTTN (1,2,4-butanetriol trinitrate) was used as the internal standard. The analogous $m/z\ (M + NO_3)^-$ ion was monitored.

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[1] Certain commercial equipment, instrumentation, or materials are identified in this report to specify adequately 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.

Homogeneity Assessment: The homogeneity of SRM 2907 was assessed by analyzing duplicate samples of 50 mg from 16 bottles selected by stratified random sampling. Samples were processed and analyzed as described above using the LCUV determination. No statistically significant differences among bottles were observed for the two explosives at the 50 mg sample size within the specified uncertainties.

Certified Values: Each certified value is a weighted mean of the results from two analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) calculated by combining a between-method variance with a pooled, within-method variance [5-7] following the ISO Guide [8].

Table 1. Certified Mass Fraction for TATP in SRM 2907, Nominally 0.4 % TATP

Explosive	Mass Fraction (mg/g)
TATP	3.628 ± 0.070

Table 2. Certified Mass Fraction for PETN in SRM 2907, Nominally 0.04 % Semtex

Explosive	Mass Fraction (mg/g)
PETN	0.291 ± 0.014

REFERENCES

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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.