National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2905

Trace Particulate Explosive 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 [1]. SRM 2905 consists of four non-explosive materials prepared from inert particles having a diameter of approximately 20 µm to 30 µm that are coated with trace levels of explosives. Two materials contain military-grade trinitrotoluene (TNT) with nominal concentrations of 0.01 % and 0.1 % mass fractions, and two materials contain Composition C-4 plastic explosive with nominal concentrations of 0.01 % and 0.1 % mass fractions. The Composition C-4 materials contain the explosives hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) and octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX). Certified concentrations are provided for TNT, RDX, and HMX. A unit of SRM 2905 consists of four individual plastic squeeze bottles each containing approximately 1 g of the trace particulate explosives materials.

Certified Concentration Values: Certified concentration values, expressed as mass fractions, for the three explosives are provided in Tables 1 through 4. The certified concentration values for the three explosives are based on the agreement of results obtained at NIST using two independent analytical techniques [2]. 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 2905** is valid, within the measurement uncertainty specified, until **31 August 2020**, 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 coordination of the technical measurements leading to the certification of SRM 2905 was under the leadership of W.A. MacCrehan of the NIST Chemical Sciences Division.

Analytical measurements for the certification of SRM 2905 were performed at NIST by W.A. MacCrehan of the NIST Chemical Sciences 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 Office of Reference Materials.

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

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Gaithersburg, MD 20899
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INSTRUCTIONS FOR STORAGE, HANDLING, AND USE

Storage: To maintain the certified values for long periods, SRM 2905 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 50 days) 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.

Use: Following a period to allow the packaging to reach room temperature, cut 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 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)

Sample Preparation: The simulant materials for TNT were prepared by dissolution of pure TNT into methanol with ultrasonic agitation. The Composition C-4 materials were prepared by dissolution of the plastic explosive Holston AAP M112 in a small volume of unstabilized tetrahydrofuran using ultrasonic agitation. Each of the respective solutions was diluted with a sufficient volume of methanol to cover the octadecylsilyl-modified (C_{18}) silica substrate to be coated (Protein and Peptide C18, GraceVydac, Hesperia, CA). A rotary evaporator was used to mix the materials and for the removal of the solvents. The four materials were dried at 70 °C for 1 h 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 2905, liquid chromatography (LC) with ultraviolet absorbance detection (UV) and mass spectrometric detection (MS), respectively. The LC/UV method utilized an octadecylsilyl-modified silica separation column (Zorbax SB-C18, Agilent Technologies, Santa Clara, CA, 5 µm particles, 3.0 mm × 250 mm) with a gradient elution solvent program using a water methanol mobile phase. Detection was at 230 nm. For this method, 5 mg to 10 mg samples were extracted with 0.4 mL to 1.0 mL of 50 % volume fraction acetonitrile in isopropanol for 2 h using ultrasonic agitation. The LC/MS method used a pentafluorphenyl propylsilane-modified silica column (Pinnacle DB PFP Propyl, Restek, Bellefonte, PA, 5 µm particles, 3.2 mm × 250 mm) with a water methanol mobile phase. For the determination of RDX and HMX, a mobile phase additive, ammonium acetate pH 5.5, was added to each solvent at a concentration of 0.05 mmol/L. An adduct ion, corresponding to m/z $(M + OAc)^-$ was monitored for MS detection of these analytes. For the extraction of the Composition C-4 samples, 50 mg to 100 mg of sample were extracted with 0.4 mL to 1.0 mL of 50 % volume fraction methanol in *n*-propanol for 2 h using ultrasonic agitation. A similar protocol was used for the TNT samples except that the extraction solvent was *n*-propanol.

Internal Standards Used: For the LC/UV determinations, 4-nitrobenzonitrile (recrystallized from 50 % volume fraction pentane:acetone) was used as the internal standard. For the LC/MS determinations, stable isotope-labeled analogs of the explosives were used. HMX ($^{13}C_4$ 99 %; $^{15}N_4$ 98 %) 1 mg/mL in CH_3CN, 2,4,6-trinitrotoluene ($^{13}C_7$ 99 %; $^{15}N_3$ 98 %) 1 mg/mL in benzene, and RDX ($^{13}C_3$ 99 %) 1 mg/mL in CH_3CN were obtained from Cambridge Isotope Laboratories (Andover, MA).

Homogeneity Assessment: The homogeneity of SRM 2905 was assessed by analyzing duplicate samples of 5 mg from ten bottles selected by stratified random sampling. Samples were processed and analyzed as described above using the LC/UV determination. No statistically significant differences among bottles were observed for the three explosives at the 5 mg sample size within the specified uncertainties.

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(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 and HMX in SRM 2905, 0.01 % Composition C-4 (nominal)

Explosive	Mass Fraction[(a)] (mg/g)
RDX	0.0777 ± 0.0009
HMX	0.00866 ± 0.00008

Table 2. Certified Concentrations for RDX and HMX in SRM 2905, 0.1 % Composition C-4 (nominal)

Explosive	Mass Fraction[(a)] (mg/g)
RDX	0.687 ± 0.014
HMX	0.0757 ± 0.0009

Table 3. Certified Concentrations for TNT in SRM 2905, 0.01 % TNT (nominal)

Explosive	Mass Fraction[(a)] (mg/g)
TNT	0.097 ± 0.001

Table 4. Certified Concentrations for TNT in SRM 2905, 0.1 % TNT (nominal)

Explosive	Mass Fraction[(a)] (mg/g)
TNT	0.976 ± 0.007

(a) The certified value is an unweighted 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 [4] following the ISO Guide [5].

REFERENCES

[1] MacCrehan, W.; *Development of a NIST Trace Particulate Explosives Reference Material to Evaluate IMS Detectors*, Int. J. Ion Mobil. Spectrom. 9:13–23, (2006).

[2] MacCrehan, W.; *A NIST Standard Reference Material (SRM) to Support the Detection of Trace Explosives,* Anal. Chem., Vol. 81, No. 17, pp. 7189–7196; available at http://pubs.acs.org (accessed March 2013) doi: 10.1021/ac900641v (2009).

[3] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements;* NIST Special Publication 260-136, U.S. Government Printing Office, Gaithersburg, MD (2000); available at http://www.nist.gov/srm/publications.cfm (accessed March 2013)

[4] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.-K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, No. 4, p. 571–579 (2000).

[5] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed March 2013); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/index.cfm (accessed March 2013).

Certificate Revision History: 25 March 2013 (Extension of the certification period; editorial changes); 13 August 2009 (Original certificate date)

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) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.