National Institute of Standards & Technology

Report of Investigation

Reference Material 8107

Additives in Smokeless Powder

This Reference Material (RM) is a smokeless powder of the type used as the propellant in small arms ammunition. RM 8107 is intended to support analytical measurements of nitroglycerin (NG), diphenylamine (DPA), *N*-nitroso-diphenylamine (NnDPA [a nitration product of DPA]), and ethyl centralite (EC [*N,N*'-diethyl-*N,N*'-diphenylurea]), including qualitative additive identification and quantitative compositional measurements. A unit of RM 8107 consists of one bottle containing 5 g of smokeless powder.

Reference Concentration Values: The reference values for NG, DPA, NnDPA and EC content of the powder, expressed as mass fraction, are provided in Table 1. The reference values were determined by a single analytical technique – solvent extraction and liquid chromatography. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty.

Expiration of Value Assignment: The reference values of RM 8107 are valid, within the measurement uncertainty specified, for one year from the date of sale, provided the RM is handled and stored in accordance with instructions given in this report (see "Notice and Warning to Users"). However, the reference values are nullified if the RM is damaged, contaminated, or modified.

Maintenance of RM Value Assignment: NIST will monitor the reference values for this RM over the period of its validity. If substantive changes occur that affect the reference values before expiration, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical project leading to the investigation of this RM was performed by W.A. MacCrehan of the NIST Analytical Chemistry Division and S. Ballou of the NIST Office of Law Enforcement Standards.

The reference values in Table 1 were provided by M. Bedner, contractor of the NIST Analytical Chemistry Division. Design of the calibration protocol was done by D.L. Duewer of the NIST Analytical Chemistry Division.

Statistical consultation leading to the reference values was performed by D.D. Leber of the NIST Statistical Engineering Division.

The technical support aspects involved in the issuance of this RM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

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Certificate Issue Date: 20 August 2004

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NOTICE AND WARNING TO USERS

Warning: This material is for laboratory use only; it is **NOT** for human consumption. Refer to MSDS 8107 for the chemical hazards associated with this material. Smokeless powder is a highly flammable solid; keep it away from sparks and flames. Avoid storage at elevated temperatures as the material may become unstable and may detonate. Dispose of unused material properly.

Storage: Material must be stored at room temperature (23 °C) or below. Refrigeration may prolong the stability of the material and is recommended. After opening the packaging for use, the bottle should be tightly resealed. Since the smokeless powder composition may be moisture-sensitive, the remaining powder should be stored with a desiccant, such as in a zip-enclosure bag containing a silica-drying packet. If the powder is stored at sub-ambient temperatures, be sure to allow the powder to reach room temperature prior to sampling.

Stability: An investigation of the reference values of additives NG, DPA, NnDPA, and EC was conducted for stability under sub-ambient, room temperature, and elevated temperature (35 °C) for a period of approximately one year. The investigation revealed adequate stability for storage at or below room temperature (23 °C). Some loss of DPA was noted for storage at the elevated temperature.

INSTRUCTIONS FOR USE

Samples may be withdrawn from the bottle at room temperature. A sample size of 20 mg or greater is recommended. Care should be taken to avoid ignition of the powder during handling (see "Warning").

PREPARATION AND ANALYSIS

Source of Material: A ball-type, smokeless rifle powder from a single manufacturing lot was obtained from St. Marks Powder[1] (Crawfordville, FL).

Preparation: The commercial smokeless powder for this RM was prepared from a single manufacturing lot and blended prior to unit bottling.

Method of Analysis Used in Value Assignment: Approximately 20-milligram samples of the powder were solvent extracted with ultrasonic agitation for 75 min. The extraction solvent was a 25 % (volume fraction) solution of 2-butanol in methanol and incorporated an internal standard, diethylphthalate. This solvent recovers the four additives yet inhibits the complete dissolution of the nitrocellulose polymer [1].

The additives were determined by reversed-phase liquid chromatography with ultraviolet absorbance detection. The four additives and the internal standard were separated using a Luna C18 analytical column, 250 mm × 4.6 mm, Phenomenex[1] (Torrance, CA), with a 66 % (volume fraction) methanol/water mobile phase. The absorbance at 214 nm was used for the additive determination. Sample extracts were determined against in-house calibration solutions using the internal standard method.

Homogeneity Assessment: The effect of sample mass (5 mg, 20 mg, and 100 mg) on the reference values was determined. For the three additives NG, DPA, and EC, no statistical difference was obtained for the mean value with sample mass. NnDPA did appear to be lower for the 5 mg samples. For each of the four additives, the 5 mg measurements had greater variances than the other sample weights. Sample masses of 20 mg were used to produce the reference values and uncertainties.

[1]Certain equipment, instruments, or materials are identified in this certificate 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.

Reference Concentration Value Assignment: The reference concentration values summarized in Table 1 are the means of the measurement results obtained by NIST using the method previously described. The expanded uncertainty displayed is the tolerance limit associated with the 16-bottle averages calculated as $U = ks$. The standard deviation of the 16-bottle averages is given by s. The coverage factor, k, is the tolerance expansion factor of 2.903 associated with 95 % confidence that 95 % of the population lies within the tolerance limits [2].

Table 1. Reference Concentration Values for Additives in Smokeless Powder

Additive	Mass Fraction (mg/g)		
Nitroglycerin	129.1	±	2.1
Diphenylamine	7.80	±	0.18
N-nitrosodiphenylamine	3.05	±	0.09
Ethyl Centralite	36.4	±	1.3

REFERENCES

[1] Reardon, M.R.; MacCrehan, W.A.; *Developing a Quantitative Extraction Technique for Determining the Organic Additives in Smokeless Handgun Powder*; J. Forens. Sci., Vol. 46, No. 4, pp. 802-807 (2001).

[2] Montgomery, D.C.; *Introduction to Statistical Quality Control,* 3rd ed.; John Wiley & Sons, Inc.: New York (1997).

Users of this RM should ensure that the report in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.