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Chemical Reactivity Test: Assessing Thermal Stability and Chemical Compatibility

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Abstract—The thermal stability of high explosive (HE) and its compatibility with other materials are of critical importance in storage and handling practices. These properties are measured at Lawrence Livermore National Laboratory using the chemical reactivity test (CRT). The CRT measures the total amount of gas evolved from a material or combination of materials after being heat treated for a designated period of time. When the test result is compared to a threshold value, the relative thermal stability of an HE or the compatibility of an HE with other materials is determined. We describe the CRT testing apparatus, the experimental procedure, and the comparison methodology and provide examples and discussion of results.

Index Terms—chemical reactivity test, compatibility, high explosive, thermal stability

I. INTRODUCTION

PRISTENE high explosive (HE) will violently deflagrate or detonate to produce hot, rapidly expanding gasses under appropriate energy input in the form of impact, friction, electrical shock, or heat. These reactions are predictable in nature, and therefore, are straightforward to manage and control.

However, HE may slowly decompose when exposed to elevated temperature or other material, leading to thermal stability or compatibility concerns. These slow decomposition reactions, which are usually imperceptible to human senses, may decrease chemical stability and cause HE to deflagrate or detonate more readily and less predictably. As such, an accurate, standardized test is required to assess HE thermal stability and HE compatibility with other materials to ensure safe handling procedures are implemented and proper storage conditions are met.

The chemical reactivity test (CRT) conducted at Lawrence Livermore National Laboratory is designed to assess the thermal stability of HE or its compatibility with another material under many conditions, namely, elevated thermal conditions. As HE decomposes, it evolves gas. The CRT measures the total amount of gas evolved from a material or combination of materials after exposure to an elevated temperature for a designated period of time.

Depending on how the CRT is used, two types of information may be obtained. When HE is tested separately, the amount of gas evolved is used to provide a relative measure of *thermal stability*. When HE is tested after being mixed with another material, the amount of gas evolved is used to provide a relative measure of *compatibility*. We describe and clarify the CRT testing apparatus, the experimental procedure, and the characterization methodology for both uses as described in an earlier paper, “Chemical

Reactivity Test for Thermal Stability” [1]. Additionally, sample results are shown and discussed.

II. EXPERIMENTAL APPARATUS AND PROCEDURE

The LLNL CRT sample loop assembly, shown schematically in Fig. 1, is a robust, stainless steel vessel that holds a 0.25g explosive sample and captures gas evolved from it. The HE sample can be a single material or mixture of materials in powder or pressed form.

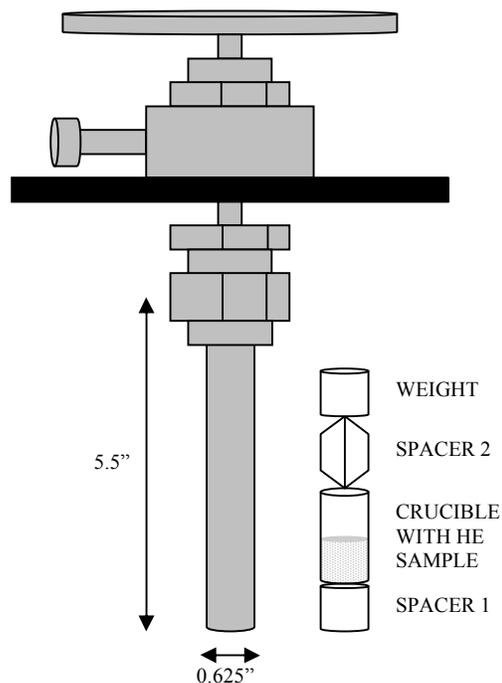


Fig. 1: Stainless steel CRT sample holder loop assembly and internal loop components.

To conduct a measurement, the sample loop is assembled and attached to a vacuum rack. The loop is evacuated to remove atmospheric gasses that contaminate future gas chromatograph (GC) measurements. The loop is then backfilled with helium, sealed, and submerged in a 120°C silicon fluid bath for 22 hours. After 22 hours, it is removed, allowed to cool to room temperature, and attached to a GC. After evacuating air from the GC, the captured gas that evolved from the HE is allowed to flow directly from the loop into the GC. The GC separates the gas into individual components, and a thermal conductivity detector measures the quantities of the components. The GC is regularly calibrated

by analyzing a gas mixture with known concentrations of N_2 , O_2 , Ar, CO, NO, CO_2 , and N_2O .

In addition to calibration, the stability of the GC and the accuracy of the experimental technique are verified by measuring gas volume generation from PBX-9404, a predetermined explosive standard. From many previous experiments, it is known that PBX-9404 generates approximately two cubic centimeters of gas per gram of explosive after being heat treated for 22 hours. Therefore, a problem with the GC or with the experimental technique is indicated if a PBX-9404 gas volume measurement deviates significantly from 2cc/g. If this occurs, the experimenter corrects the problem and repeats the PBX-9404 measurement. If a 2cc/g measurement is obtained, thermal stability or compatibility tests are resumed.

III. CHARACTERIZATION METHODOLOGY

A. Thermal Stability

Thermal stability test results are “thermally unstable” or “thermally stable”. The experimenter measures the total amount of gas evolved with the GC and compares it to a 4cc/g threshold value based on earlier work by Prokosch and Garcia [2]. If the volume of gas generated by the HE during heating is less than the 4cc/g threshold value, the HE is generally

considered “thermally stable”. If the volume of gas generated by the HE during heating is greater than the 4cc/g threshold value, the HE is generally considered “thermally unstable”. Additional kinetic experiments may be needed to gain more understanding.

B. Compatibility

Compatibility results are “incompatible” or “compatible”. The experimenter measures the total amount of gas evolved from the mixture with the GC and compares it to a 1cc/g threshold value. If the gas volume generated by the mixture is less than 1cc/g, the HEs are generally considered “compatible”. If the gas volume generated from the mixture is greater than 1cc/g, the HEs are generally considered “incompatible”. Additional kinetic experiments may be needed to gain more understanding. This lower threshold reflects stricter compatibility characterization criteria. Such an approach allows scientists and engineers a broader safety envelope when working with mixtures containing HE. A flowchart that summarizes the thermal stability and compatibility characterization methodology described above is shown in Fig. 2.

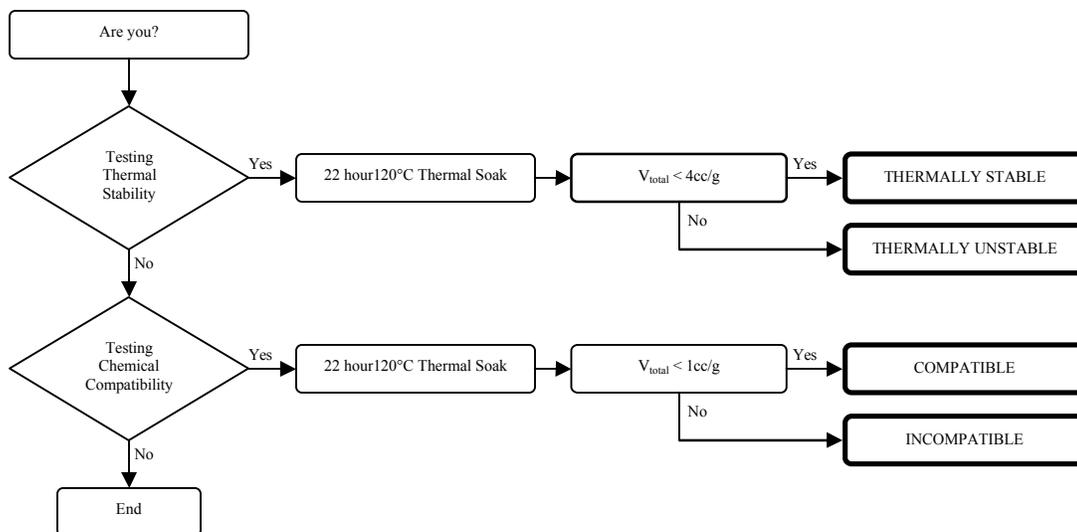


Fig. 2: Thermal Stability and Compatibility Characterization Methodology Flowchart

IV. RESULTS AND DISCUSSION

Examples of two specific data sets illustrate the test utility. Corresponding data are found in Tables I and II.

A. Example 1 – Thermal Stability of Common Explosives

Fig. 3 shows the amount of gas evolved per gram of explosive over the 22 hour heating period for PBX-9404, LX-14, and LX-17 in both powder and pellet form [3]. HE compositions are listed in Table III.

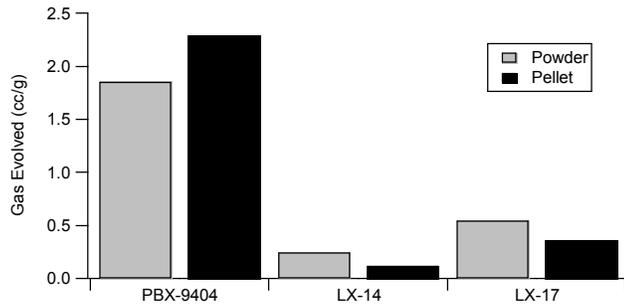


Fig. 3: PBX-9404, LX-14, and LX-17 gas volume generation characterizing thermal stability.

The gas generation value for PBX-9404 is consistent with past measurements of 2cc/g. Subsequent LX-14 and LX-17 gas volume generation measurements are significantly less than the 4.0cc/g threshold value. These explosives are considered thermally stable under normal operating conditions.

B. Example 2 – PETN and Ceramabond 503 Incompatibility

Fig. 4 shows the amount of gas evolved per gram of explosive over the 22 hour heating period for HE PETN and epoxy Ceramabond 503 [4]. The gray portion of the third bar labeled “Mixed” represents the amount of gas evolved from the 50/50 weight percent mixture of HE and epoxy after undergoing a 22 hour thermal soak. The black portion represents the sum of the component gas volumes depicted in the first and second bars.

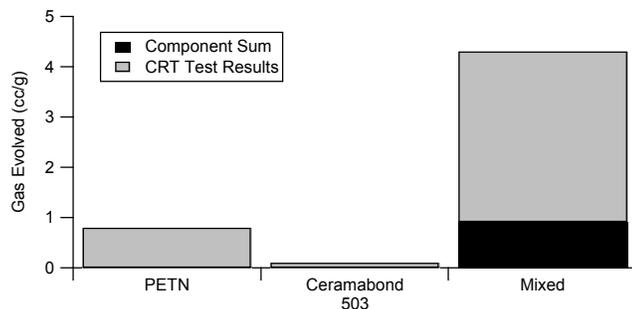


Fig. 4: Explosive PETN, epoxy Ceramabond 503, component sum and mixture gas volume generation characterizing incompatibility

The total gas volume is 4.3cc/g far exceeds the compatibility threshold of 1cc/g specified. This suggests a significant reaction between the two components occurred during heating. The results in this case clearly indicate that the two materials are incompatible.

V. SUMMARY

The CRT testing apparatus, the experimental procedure, and the characterization methodology for the thermal stability and compatibility tests were described. Examples with several common HEs show the utility of these simple characterization techniques. Fig. 3 shows that PBX-9404, LX-14, and LX-17 are individually thermally stable, and Fig. 4 shows that the PETN/Ceramabond 503 mixture is incompatible. The analysis techniques described can be applied to measure thermal stability of nearly any material and the compatibility of countless combinations of materials.

APPENDIX

TABLE I
CRT RESULTS FOR PBX-9404, LX-14, AND LX-17

MATERIAL	Gas Evolved (cc/g)
PBX-9404, Powder	1.86
PBX-9404 Pellet	2.29
LX-14 Powder	0.25
LX-14 Pellet	0.12
LX-17 Powder	0.55
LX-17 Pellet	0.36

TABLE II
CRT RESULTS FOR PETN AND CERAMABOND 503

MATERIAL 1	MATERIAL 2	Gas Evolved (cc/g)
PETN	-	0.80
Ceramabond #503 (cured)	-	0.10
PETN	Ceramabond #503 (cured)	4.31

TABLE III
EXPLOSIVE COMPOSITIONS

Explosive	Composition
PBX-9404	95.5% HMX (tetranitro-tetraazo-cyclooctane)
	2.9% Nitrocellulose
	3.0% Tris-β-chloroethyl-phosphate plasticizer (CEF)
	0.1% Diphenylamine
LX-14	95.5% HMX
	4.5% Estane
LX-17	92.5% TATB (triaminotrinitrobenzene)
	7.5% Kel-F 800 (fluoropolymer binder)
PETN	Pentaerythrol tetranitrate

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