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Chemical Reactivity Test (CRT)

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Chemical Reactivity Test (CRT)

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SCOPE & APPLICATION

The Chemical Reactivity Test (CRT) is used to determine *the thermal stability of* High Explosives (HEs) and *chemical compatibility* between (HEs) and alien materials. The CRT is one of the small-scale safety tests performed on HE at the High Explosives Applications Facility (HEAF).

INTRODUCTION

The CRT assesses *thermal stability* of HE and the *chemical compatibility* between the HE and alien materials (1-3). Alien materials are defined here as substances which do not contain HE. Samples are weighed using an analytical balance and transferred into crucibles. A crucible is placed into a reactor vessel, which is evacuated and backfilled with helium (He). The reactor vessel is heated for 22 h (normally) at either 80°C or 120°C temperature. The gases generated from the decomposition of the sample during heating are injected into a gas chromatograph (GC) for identification of evolving gases. A typical GC run goes from -60°C to 60°C for 16 min. In addition, there is a 4-min post run bake out at 100°C. The GC uses a Thermal Conductivity Detector (TCD) to analyze the component gases generated by the sample.

The *thermal stability* of an HE is determined by comparing the sum of the volumes of gases generated to the standard volume of gases generated from a sample of PBX 9404 (1.5 - 2 cc/g when heated for 22 h at 120°C). A HE sample that generates a little more than twice the amount of gases as PBX 9404 (4 cc/g of HE) is considered thermally unstable.

The *compatibility* of an HE mixed with alien material is determined by comparing the sum of the volumes of gases generated by the mixture to the sum of the volumes of gases generated separately by the HE alone and the alien material alone. If a mixture of HE and alien material generates gases in excess of those generated by the individual materials, the HE and the alien material are considered to be incompatible.

The CRT measures the relative volume amounts of nitrogen (N₂), carbon monoxide (CO), nitric oxide (NO), carbon dioxide (CO₂) and nitrous oxide (N₂O) produced by each sample. The gases are separated by GC and detected and measured by a TCD. Oxygen (O₂) and argon (Ar) are also measured and used to evaluate air leaks.

SUMMARY

The CRT procedure is divided into three parts: sample preparation, gas chromatography, and data analysis. This document has two appendices, which explain calibration and quality control processes.

Samples are weighed, placed into reactor vessels, and heated isothermally for 22 h at 80°C or 120°C or any other requested temperature. When the reactor vessel containing the sample returns to ambient temperature, a fixed volume of the evolved gas from the 17 cc reactor vessel is injected into the GC for analysis. The injected sample gas components

are carried by the He gas, (mobile phase) across the stationary phase of the column. The gas components have differential solubility with the stationary phase of the column and pass through the column at different rates. The resulting mobility difference of the gas components separates the gas components from each other as they pass through the stationary phase. The carrier gas composition, pressure, and flowrate and the column diameter and temperature all affect the amount of separation achieved.

As each individual gas component elutes from the column, it enters the TCD. The TCD responds to the difference in the thermal conductivity between the He carrier gas containing the sample gas components and a He reference gas. As a sample gas component flows through the detector and displaces the He carrier gas conductivity, the resulting change in thermal conductivity specific to that gas component around the detector element, raises the temperature and resistance of the element. A Wheatstone resistance bridge circuit with the He reference arm, producing a voltage signal, measures the change in resistance of the element. The signal is recorded as a function of time as the gases elute from the GC, is recorded by the data system and plotted against elapsed time to produce the gas chromatogram.

The relative amounts of each gas component produced by the sample are determined from their respective peak areas in the gas chromatogram and reported as volume cc eluted. This value for each gas is then entered into the CRT spreadsheet. A sample report is then generated, given to the requestor and entered into the LLNL Explosives Reference Guide (5).

CRT PROCEDURE

Part 1: Sample Preparation

Sample submission and safety review

Upon receiving a sample, the first step taken is to check the safety profile of the HE submitted. The LLNL Explosives Reference Guide (5) and the HEAF Facility Safety Plan (FSP) can be used to obtain safety information. Current results from other small-scale safety tests can also be used. The CRT is never performed on primary HE because of potential safety issues. The CRT work performed is authorized by IWS 12275. The temperatures for heating material are 80°C or 120°C unless otherwise indicated by safety or other testing requirements by the requester (120°C is the maximum for HE based materials). Most samples analyzed are powder and/or solid material. Methods for preparing samples which contain epoxies are also outlined below.

Sample preparation

For a thermal stability test of an HE or an alien material, samples are prepared in duplicate. Two sample aliquots of 0.25 g are weighed in the crucibles on an analytical balance.

For a chemical compatibility test of one HE and one alien material, duplicate samples of the HE and alien material mixture are prepared. Two sample aliquots of 0.25 g of the HE are weighed in the crucibles on an analytical balance. Two sample aliquots of 0.25 g of the alien material are weighed in the crucibles on an analytical balance. Duplicate samples of the HE and alien material mixture are prepared. Each sample comprised of

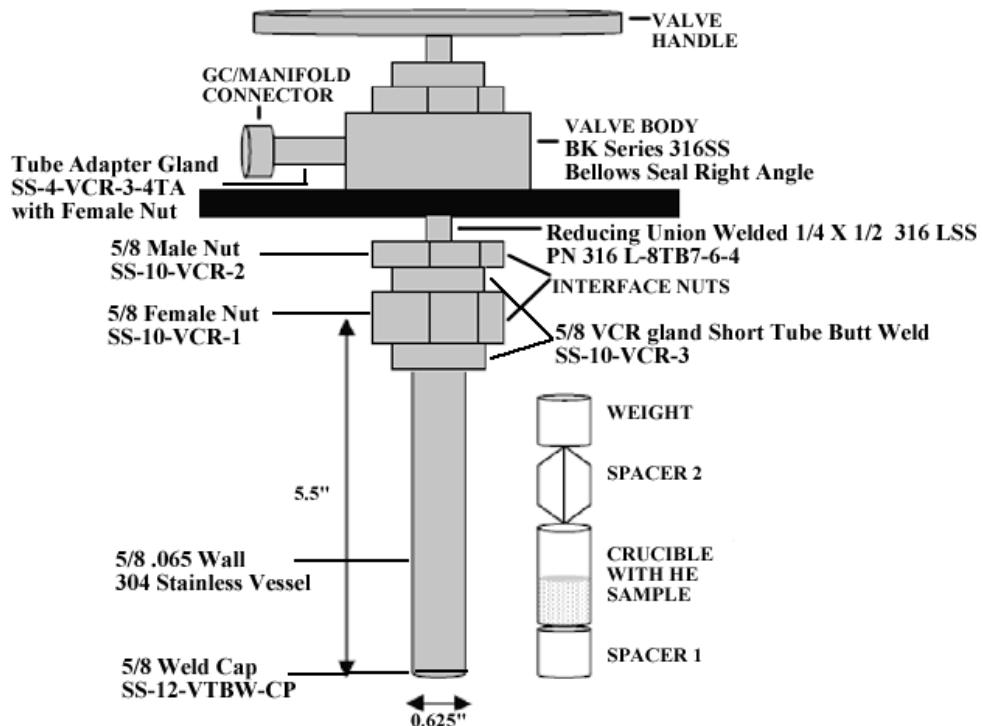
an HE/alien mixture contains 0.25 g of HE and 0.25 g of alien material for a total sample mass of 0.50 g.

For a chemical compatibility test of two HE materials (e.g. a & b), duplicate samples are prepared as follows: Two sample aliquots of 0.125 g of each HE are weighed in the crucibles on an analytical balance. Duplicate HE(a)-HE(b) mixture samples are then prepared by combining one 0.125 g sample aliquot of HE(a) and one 0.125g sample aliquot of HE(b) for a total sample mass of 0.25 g.

All individual sample aliquot weights recorded to the nearest 0.001 g in the CRT lab notebook along with detailed information about each sample.

Reactor vessel preparation:

Each weighed sample aliquot (single substance or mixture) is weighed into a stainless steel crucible as outlined above. A small amount of glass wool is placed above the sample in the crucible to keep the sample contained. A stainless steel reactor vessel, shown in Figure 1, is assembled in the following order: Spacer 1, crucible with sample, spacer 2, and the weight followed by a new Swagelok 5/8 NI-10-VCR-2 gasket <http://www.swagelok.com>.



***Figure 1. CRT Reactor vessel
Loading samples on the manifold***

The custom reactor vessel is made from 304 series stainless steel for resistance to corrosion and for controlled heating of the sealed sample during the 22 h heating cycle. All of the other components are made from 316 stainless steel and welded to fit together. The internal volume of the reactor vessel is 17 cc. The reactor vessel is connected to the

manifold, shown in Figure 2, for evacuation to <50 mTorr and for backfilling with He to 14.72 psi (1 atm.). The reactor vessel can be assembled and disassembled for multiple tests. The disassembled vessels are cleaned, washed and dried for future use.

After the samples are weighed and assembled into the reactor vessels, they are connected to the sample inlet port on the sample prep manifold, shown in Figure 2, using a new Swagelog 1/4 NI-4-VCR-2 gasket. Atmospheric gas is then evacuated from the reactor vessel to <50 mTorr and back filled with 14.7 psi of He (1 atm.). The system is flushed with He gas and then evacuated a few times to rid any air in the reactor vessel.

As mandated by HEAF FSP, a 5- μ m primary filter and 20- μ m secondary filter is attached on the vacuum line to prevent explosive particulates and other residues from entering the vacuum pump.

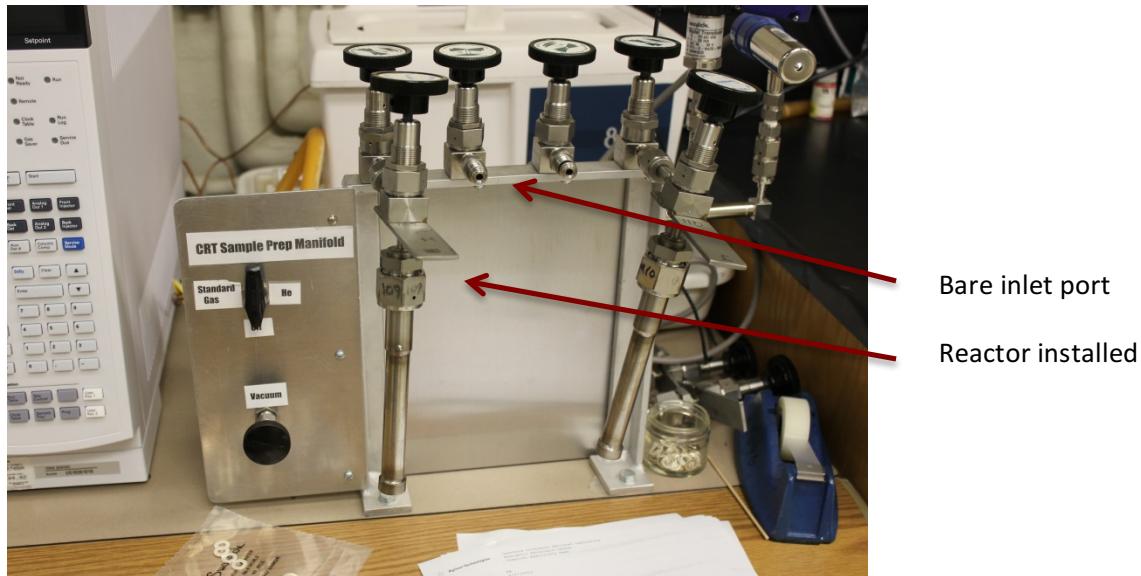


Figure 2. Sample prep Manifold

Sample preparation method for epoxies

When using epoxy, the following pre-mixing test (also called coupon test) is performed: Mix a small amount, about 10 mg, of HE and alien epoxy material and visually observe for evidence of a reaction. If there is no evidence of a reaction by a color change or an outgas as the mixed materials cure, the materials can be weighed and allowed to cure in the crucibles.

Cured epoxy method

After weighing either the epoxy alone or as a mixture into the crucible, it is allowed to cure prior to placing the sample in the reactor vessel.

Uncured epoxy method

This method is used for curing epoxies during the sample evacuating and heating process. The following procedure is used to prevent air from being entrapped in the epoxy as it cures and splattering uncured material inside the reactor vessel during the evacuation:

1. Attach the reactor vessel to the inlet on the manifold with the reactor vessel's valve in the closed position.
2. Evacuate the connector of the reactor vessel to <30 mTorr.
3. Next, close the vacuum pump valve on the manifold (static vacuum), and slowly open the reactor vessel valve which contains the curing material.
4. Fill the reactor vessel with He to 14.7 psi.
5. Slowly open the vacuum valve on the manifold and evacuate (dynamic vacuum) the reactor vessel with the curing epoxy to 12.0 psi.
6. Again, back fill the vessel to 14.7 psi.

Repeat the process by using the protocol listed in Table 1. This process eliminates atmospheric and epoxy gas from the vessel as the epoxy cures, and does not disturb (bump) the sample. When the last cycle is complete, back fill the vessel with 14.7 psi He and remove the reactor vessel from the manifold then transfer to the appropriate heating bath. In the CRT lab notebook, record the time the samples are placed into the oil bath as well as the bath temperature.

Table 1. Back-fill sequence for epoxy material curing during the evacuation process.

Back-fill with He (psi)	Evacuate (psi)	Cycles
14.7	12.0	3
14.7	7.0	3
14.7	3.0	3
14.7	1.0	3

Sample heating

After backfilling with 14.7 psi He, the sample reactor vessels are then transferred to the oil baths and heated isothermally at a specified temperature for 22 h. The temperature, reactor vessel identification number, and the sample material are recorded in the CRT lab book. Two silicon oil baths (in the CRT lab) are used to heat the samples. The oil bath, temperatures are set for the specific temperatures of 80°C or 120°C, see Figure 3.

Depending on the circumstances for selecting a particular temperature, a table showing effects of temperature is provided in Part 3, Table 3. Each tank has a mechanical stirrer that helps maintain each bath at their set temperature and ensure to thermal equilibrium throughout the tank (isothermal heating). A temperature control unit is used to maintain and stabilize the baths. A secondary, fail-safe temperature control is used to prevent thermal runaway of the oil bath temperature. The temperature control unit is capable of maintaining temperatures that are within $\pm 1^\circ\text{C}$ of the set temperature.

At the end of the 22 h. heating cycle, the reactor vessels are removed from the silicon oil bath and allowed to return to ambient temperature before GC analysis as described in Part 2. If the pressure and the ambient temperature in the reactor vessel remain constant, an indefinite delay in GC analysis is possible; however samples are normally analyzed on the same day.



Figure 3. Silicone oil heat bath

Reactor vessel cleaning

After the CRT analysis is complete, the samples are removed from the reactor vessels. Open the reactor vessel and inspect the sample, noting discoloration or a texture change. If the sample cannot be removed easily from the stainless steel crucible, the crucible becomes contaminated-HE waste. These crucibles or other affected parts are soaked in or cleaned with iso-propyl alcohol (IPA), acetone or any other solvent. The sample crucibles are then washed with deionized (DI) water and subsequently put in a concentrated nitric acid in an Erlenmeyer flask and left capped for a few days in the hood. These parts are then removed from the nitric acid flask and rinsed with DI water and then combined with other parts in the ultrasonic bath.



Figure 4. Branson Ultrasonic bath 3800

The reactor vessel assemblies/components are washed with DI water and then put in the ultrasonic bath for one hour. The parts are then rinsed again with DI water and put in the drying oven, shown in Figure 4, and dried for 24 h.



Figure 4. Vacuum Drying Oven

Handling of spent HE after analysis

The content of the sample crucible is emptied into a small plastic polypropylene Ziploc bag. A HE label with all the samples information is placed on the bag. If it was compatibility test, write the type of alien material (s) on the bag with a permanent marker. All samples that undergo CRT analysis must have their storage compatibility (SC) criteria changed from the SC category they held prior to the analysis. Typical HE samples for CRT analysis contain a SC of (A, D, or L). After CRT analysis, all HE materials are changed to the SC L. Category L HE may not be stored with the other SC groups. These waste sample bags are stored in a locked HE can then given to the HE explosive waste management person in charge. Storage Compatibility information can be found in “Explosives Hazards Classification” in the HEAF FSP. Handling Waste can be found under the “Waste Management and Procedures” in the HEAF FSP.

Part 2: Gas Chromatography

The gas yields are measured using a GC with a TCD, shown in Figure 5. The method is based on expanding the gas from the pressurized reactor vessel into a fixed volume that includes the GC injector sample loop, shown in Figure 6. This causes a fixed fraction of the gas in the sample reactor to be injected into the GC when the injector loop is then switched into the GC gas line. The gas sample components in the injector loop are then forced under pressure to flow into a packed SS C-5000, Porapak Q 50/80 mesh polar molecular sieve; 20' by 1/8" outside diameter (OD) by 0.085" inside diameter (ID) column from Grace Discovery Science.

The TCD utilizes the He carrier and reference gas flows, and then an integrator and computer (PC) provide the chromatogram results. Liquid nitrogen is used to bring the oven to sub-ambient temperatures (-60°C) and Matheson Tri Gas standard gas cylinder tanks are used for the calibration and check standard. Calibration procedures are given in

Appendix 1A. Pressure regulators are utilized to control the flow. The He pressure cylinder must be at 60 psi. The reference flow is set at about 2 times the rate of the He carrier flow in the column, 25 mL/min, plus the makeup gas, 3 mL/min. The total He flow is set at 28 mL/min, 25+3 mL/min. Reference He flow is set at 53 mL/min thus 53/28 is 1.9.

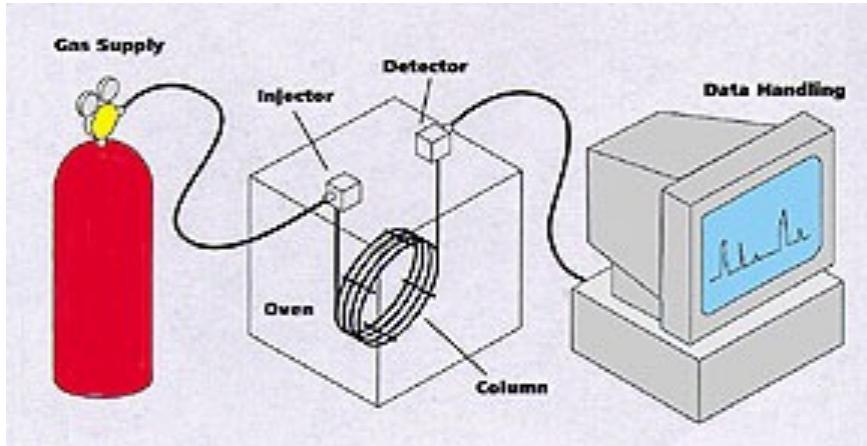


Figure 6. Agilent Model 7890A gas chromatograph

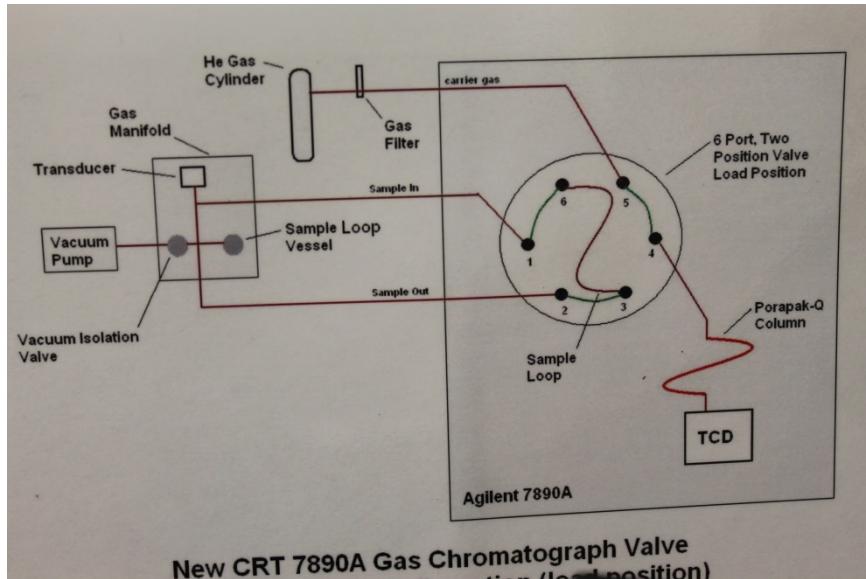


Figure 7. Schematic of sample loop injection valve

Operation of the GC

To warm up GC, press the following keys on the front panel:

Oven + ON; on the keypad of the instrument type 100°C for oven temperature to bake out the column until the baseline comes back to where it was before. Samples are run from -60 to 60°C. The TCD should never be in the on position without He flow.

Sample data entry

On the PC screen select RUN CONTROL, SAMPLE INFORMATION

Enter the following information: Operator name, Date, year, month, day, file number, type of run: He baseline check, standard gas check, sample X- PBX 9404 standard check.

In the comment section include, sample name, requestor information, the reactor vessel number, and other pertinent sample information as detailed as possible.

Operation

The previously heated sample reactor vessel is connected to the injection port on the sample manifold system. To remove atmospheric gas from the line leading to the GC and the injector loop, the vacuum valve is opened until the vacuum reaches <50 mTorr. The Liquid nitrogen cylinder is opened to cool the temperature in the GC oven to -60°C.

When the system has stabilized, a green ready light will appear on the PC. When the GC has reached <50 mTorr, isolate the GC from the manifold vacuum system by closing the vacuum valve. Open the valve on the sample reactor. Next, press the Run method key on the PC screen. The sample will be injected into the GC. Two minutes into the run, the sample reactor vessel can be removed and a new reactor vessel replaced. It will take approximately 17 min to complete the chromatographic run. At the end of each run, there is a 4 min post run at 100°C to remove anything that may still be present in the column. A baseline is performed by filling a reactor vessel with He at 14.7 psi and running this as a sample. The Standard Gas at 7.36 psi and PBX 9404 are samples used for Quality Control.

Part 3. Data Analysis

Thermal Stability and Compatibility Data Analysis

The *thermal stability* of an HE is determined by the sum of the volumes of generated gases compared to the standard volume of gases generated from a sample of PBX 9404 (1.5 cc- 2 cc/g when heated for 22 h at 120°C). A HE sample that generates more than twice this amount, (4 cc/g HE) is considered thermally unstable.

Chemical Compatibility is determined by calculating the incremental (excess) gas evolution:

$$G_{inc} = [(M_{HE})(G_{tot}/(M_{HE}) - (HE_{wt})(G_{tot,HE}) + (A_{wt})(G_{tot,A})]/M_{HE}$$

G_{inc} is the Incremental or excess gas of the mixtures.

M_{HE} is the weight of the HE material in grams contained in the mixture.

G_{tot} the total of the gas components in cc

M is the mixture (HE + alien)

A alien material (not HE)

Materials are considered incompatible if the incremental gas generation is greater than 1.5 cc/g of energetic material. (5) Due to uncertainties in both the criterion and the measurement itself, incremental gas generation between 0.75 and 1.5 cc/g is considered a questionable compatibility, and additional consideration is required to conclude that the materials are compatible for the application in question.

Table 2. Thermal Stability and Compatibility data

Thermal Stability <4.0 cc/g Y or N ?			OK	No reaction observed											
			?	minor reaction, questionable compatibility (Excess gas > 0.75 cc/g HE)											
			#	Compatibility No Good (Excess gas > 1.5 cc/g HE) (Excess gas rounded to whole number in g/cc)											

Category	Explosive	Alien Material	DATE	Wt. EX (g)	Wt. Alien (g)	Temp (°C)	N ₂ (cc)	O ₂ (cc)	CO (cc)	NO (cc)	CO ₂ (cc)	N ₂ O (cc)	Total (cc)	cc/g EX	Compat? Stable?	Explosive Details/ stdev
Aliens	Ave.	615 Hysol	2/27/07	0.250	120	0.01	0.00	0.00	0.00	0.04	0.01	0.05	0.18	Y	Lot#6AC1294C	
RDX Hi/Misc.	Ave.	PBX 9407	2/27/07	0.250	120	0.06	0.00	0.00	0.00	0.03	0.02	0.11	0.42	Y	B 808 Powder	
TATB, hi %	Ave.	LX-17	2/27/07	0.250	120	0.03	0.00	0.00	0.00	0.02	0.00	0.05	0.20	Y	C-063	
RDX Hi/Misc.	Ave.	PBX 9407	2/27/07	0.250	0.250	120	0.18	0.00	0.01	0.05	0.13	0.70	1.07	4.26	4.00	Lot#6AC1294C B808 Powder
TATB, hi %	Ave.	LX-17	2/27/07	0.250	0.250	120	0.00	0.00	0.00	0.00	0.02	0.00	0.03	0.13	ok	Lot#6AC1294C C063

The results in Table 2 show that PBX-9407, LX-17 and 615 Hysol Adhesive are individually thermally stable; PBX-9407/615 Hysol Adhesive are not compatible and LX-17/615 Hysol Adhesive are compatible.

Correcting Sample Results for Air Leaks.

Results from test samples that contain values for N₂, O₂ and Ar in a similar proportion as atmospheric air (N₂ 78.084%, O₂ 20.946%, a 3.73:1 ratio and Ar 1%) are indicative of air leakage. Oxygen and argon are measured and used to detect and correct for air leaks.

The presence of Ar is a primary indicator of air leakage. If air leakage is detected, it is assumed that the entire O₂ result is from leakage and the O₂ result is then subtracted from

the total gas evolved. A value equal to the proportion of N₂ leakage (3.73 times the O₂ result) is then subtracted from the N₂ sample result and total gas evolved. If argon is present, it is noted in the analysis report, but is not included in the total gas evolved.

Baseline Correction for CO₂ Contamination

As CO₂ is one of the more common evolved gases of interest. Background levels of CO₂ are assessed by measurement of a Helium blank and a correction is then applied to sample results. Blanks are prepared by filling a clean, empty 17 cc reactor vessel with 14.7 psi He from the He carrier gas stream. The reactor is then connected to the GC and baseline contamination is determined by measuring CO₂ levels. The CO₂ amount is then subtracted from any sample result that contains a CO₂ component. Our current practice is to monitor the baseline periodically and compare the results with our historical record to monitor potential problems with the GC column fittings.

Modified Arrhenius Equation for CRT

The following equation describes the amount of gas evolved from PBX-9404 (94% HMX, 3% Nitrocellulose, 3% trichloro ethyl phosphate, CET, and 1% diphenyl amine, DPA) as a function of temperature based on the amounts measured by the CRT at 80°C and 120°C. The analysis demonstrates that the CRT performed at 80°C is at the very low end of the energy activation of PBX-9404.

For PBX 9404, the CRT gives 0.2 cc/g out-gassing at 80°C (353.16 K) and 1.6 cc/g at 120°C (393.16 K). The modified Arrhenius equation is

$$g = g_0 \exp\left(-\frac{T_0}{T}\right)$$

where g is cc/g at temperature T and g₀ and T₀ are constants.

Using the equations:

$$0.2 = g_0 \exp\left(-\frac{T_0}{353.16}\right)$$

$$1.6 = g_0 \exp\left(-\frac{T_0}{393.16}\right)$$

T₀ and g₀ are solved to get

$$g = 1.504e8 \exp\left(-\frac{7218}{T}\right).$$

Table 3 provides extrapolation results for PBX-9404 at elevated temperatures.

Table 3. Outgas Increase with Elevated Temperature

Temp (°C)	Temp (K)	g (cc/g)
80	353.16	0.20
120	393.16	1.59
160	433.16	8.68
200	473.16	35.51
240	513.16	116.64

These results indicate that the out-gassing increases rapidly with temperatures above 120°C. Assuming that all other explosives will out-gas, the results of PBX-9404 show

that the CRT should be performed at 120°C. If we only run at 80°C, the evolved gas is too low to provide a result that can be compared directly to other materials tested at 120°C. However, safety concerns and special experiments with other explosives may require that some samples be run at 80°C. Even so, it should be remembered in these cases that additional care is needed to ensure that these materials are not used in a situation that could lead to thermal runaway.

Appendix 1A. Calibration

To assure the accuracy of the CRT analysis, a 4 level linear calibration is performed from the concentrations listed in Table A1. The equation for the linear curve fit is: $y = mx+b$

Where

x = concentration

y = response by area

m = slope of the line

b = y-intercept

The line is forced through the origin ($b=0$) to assure that samples of low concentrations are detected. Seven gases (N_2O , Ar, CO, NO, CO_2 , and O_2) are contained in the calibration curve. Gas bottles containing known concentrations from Matheson Tri Gas are used for the calibration and to determine retention times. The results from the calibration are used to compare the results from unknown samples, which contain the same retention time, and by using the calibration curve, concentration can be determined. NO is in a separate bottle due to stability issues and it is stored in a hood for safety reasons.

A calibration is performed using four volumes of the standard gas at 1.84 psi, 3.68 psi, 7.36 psi and 14.7 psi. The internal volume of the reactor vessel is 17 cc. To perform the calibration, the standard gas bottle, which contains different percentages of each gas, is filled into the 17 cc standard gas reactor vessel. The table below illustrates how the calibration concentrations are entered into the calibration method on the GC.

Table A1. Calibration Method

		14.7 psi	7.36 psi	3.68 psi	1.84 psi
	%	cc ^a	cc	cc	cc
N_2	4	0.680	0.340	0.170	0.085
O_2	2	0.340	0.170	0.085	0.043
Ar	1	0.170	0.085	0.042	0.021
CO	1	0.170	0.085	0.042	0.021
CO_2	4	0.680	0.340	0.170	0.085
N_2O	4	0.680	0.340	0.170	0.085
NO ^b	2	0.340	0.170	0.085	0.043

^a Note- the "cc" values in this table represent the relative amounts of gas components in the fixed volume reactor vessel.

^b Nitric oxide gas is contained in a separate gas cylinder and its calibration is performed separately and follows Hazard Assessment and Control Form (HAC) HAC 191-1404-2006.

Appendix B, Quality Control

The standard gas mixture used to calibrate the GC is shown in Figure B1.

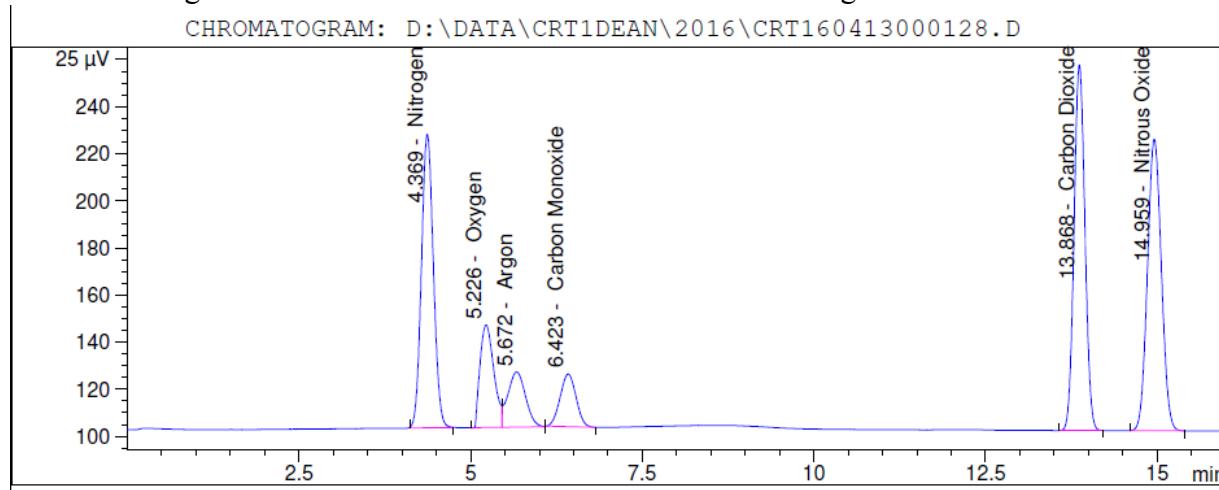
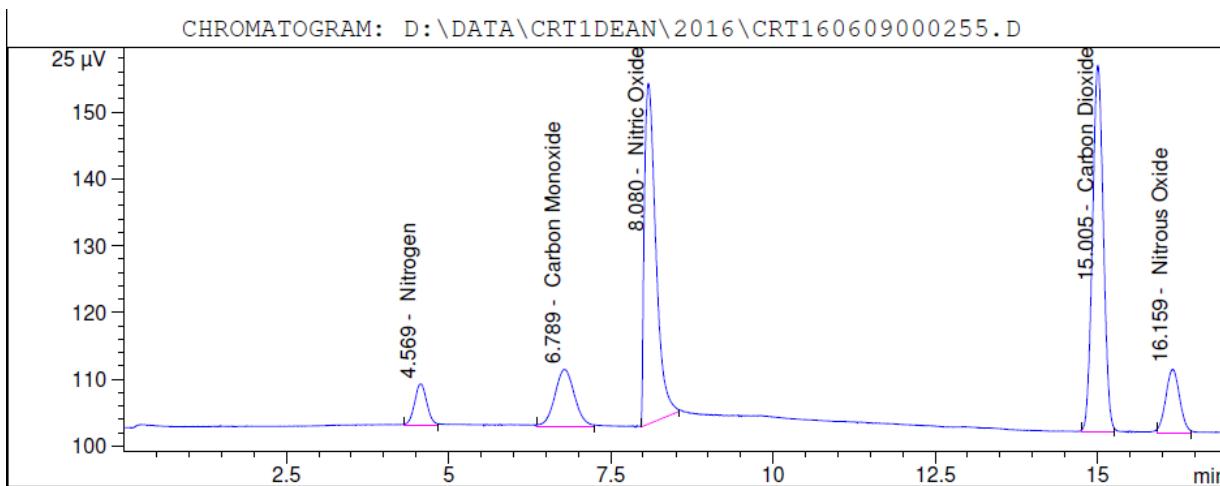


Figure B1. Chromatogram 7.36 psi Standard Gas

The initial continuing calibration verification (ICCV) using the standard gas at 7.36 psi is run at the beginning of a set of sample runs. The acceptance criteria for the ICCV run is plus or minus 3 standard deviations see. If an ICCV falls outside the control limits, a new calibration must be performed.

PBX 9404 is used as a calibration check standard. A chromatogram of PBX 9404 after heating for 22 hours at 120 °C is shown in Figure B2.



RESULTS FOR RUN ID: D:\DATA\CRT1DEAN\2016\CRT160609000255.D

Compound Name	Exp. Ret (min)	Meas. Ret (min)	Height (25 μ V*s)	Area (25 μ V*s)	Amount (cc)
Nitrogen	4.600	4.569	6.19	81.197	0.01828
Oxygen	5.510	0.000	0.00	0.000	0.00000
Argon	6.200	0.000	0.00	0.000	0.00000
Carbon Monoxide	6.800	6.789	8.49	178.088	0.04030
Nitric Oxide	8.200	8.080	51.05	628.710	0.55442
Carbon Dioxide	15.000	15.005	54.78	638.395	0.12600
Nitrous Oxide	16.160	16.159	9.59	144.540	0.02811
Totals:					0.76711

Figure B2. Chromatograph and results of gases released from 0.25 g of PBX 9404 Lot B-336.

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