

THERMAL SAFETY CHARACTERIZATION ON PETN, PBX-9407, LX-10-2, LX-17-1 AND DETONATOR IN THE LLNL'S P-ODTX SYSTEM

P. C. Hsu, S. Strout, J. G. Reynolds, E. Kahl, F. Ellsworth, T. Healy

September 28, 2017

Disclaimer

This document was prepared as an account of work sponsored by an agency of the United States government. Neither the United States government nor Lawrence Livermore National Security, LLC, nor any of their employees makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States government or Lawrence Livermore National Security, LLC. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States government or Lawrence Livermore National Security, LLC, and shall not be used for advertising or product endorsement purposes.

This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

Thermal Safety Characterization of PETN, PBX-9407, LX-10-2, LX-17-1 and Detonators by P-ODTX

Peter. C. Hsu, Steve A. Strout, John G. Reynolds, Evan M. Kahl, G. Fred Ellsworth, and Thomas E. Healy

September 28, 2017

Lawrence Livermore National Laboratory

Abstract. Incidents caused by fire and other thermal events can heat energetic materials that may lead to thermal explosion and result in structural damage and casualty. Thus, it is important to understand the response of energetic materials to thermal insults. The One-Dimensional-Time to Explosion (ODTX) system at the Lawrence Livermore National Laboratory (LLNL) has been used for decades to characterize thermal safety of energetic materials. In this study, an integration of a pressure monitoring element has been added into the ODTX system (P-ODTX) to perform thermal explosion (cook-off) experiments (thermal runaway) on PETN powder, PBX-9407, LX-10-2, LX-17-1, and detonator samples (cup tests). The P-ODTX testing generates useful data (thermal explosion temperature, thermal explosion time, and gas pressures) to assist with the thermal safety assessment of relevant energetic materials and components. This report summarizes the results of P-ODTX experiments that were performed from May 2015 to July 2017. Recent upgrades to the data acquisition system allows for rapid pressure monitoring in microsecond intervals during thermal explosion. These pressure data are also included in the report.

1.0 Introduction

Over the last few decades, there has been a considerable research effort on the thermal decomposition and thermal explosion violence of energetic materials at elevated temperatures in different sample geometries and confinements [1-3]. Thermal explosion studies on various energetic materials in two-dimensional geometry such as the Scaled-Thermal-Explosion-Experiment (STEX) system [4] and the Sandia-Instrumented-Thermal-Ignition (SITI) system have been reported [5]. The One-Dimensional Time to Explosion (ODTX) system at LLNL has been used since 1970s for thermal explosion studies [6-10]. This method is attractive because of the one-dimensional geometry and minimal sample requirement (up to 2 g for each test). With the recent integration of a pressure monitoring element, the system can study pressure behavior of materials subjected to and during thermal exposure. Rapid pressure monitoring in usec intervals allow for enhanced pressure determination in the time right before thermal explosion. The test data can be used for the validation of existing thermal models, particularly for introducing pressure terms. This report summarizes the efforts in developing P-ODTX and examines some of the results of studies on PETN, PBX-9407, LX-10-2, LX-17-1 and selected detonator samples.

2.0 ODTX Summary

2.1 System Description

Figure 1 on the left shows the ODTX system, operated remotely in a test cell. The testing involves heating a 1.27-cm diameter spherical sample in a spherical cavity between two aluminum anvils. The sample is remotely delivered to the anvil cavity via the sample delivery system when the anvils reach a predetermined temperature. A microphone sensor measures a sound signal, which indicates the time at which a thermal explosion occurs. The detail description of the LLNL-ODTX system can be found elsewhere [11].

Figure 1 on the right shows a drawing of a close-up of the sample area. To maintain pressure on the sample, a hydraulic piston applies pressure on the anvils. To seal the sample cavity, a copper gasket is used around the Al sphere holding the sample with Al knife edges to complete the seal.

Samples of various configurations (pressed parts, cast parts, powders, pastes, and liquids) can be tested in the ODTX system. Pressed and cast samples are loaded into the cavity directly without secondary containment. An Al shell is used as a secondary containment to hold powder samples, pasty samples, or liquid samples before loading to the system. About 10 ODTX tests are performed on a material at different temperatures to obtain times to thermal explosion. Reproducibility of the testing is within \pm 5%. These tests require a total of 20 g of material.

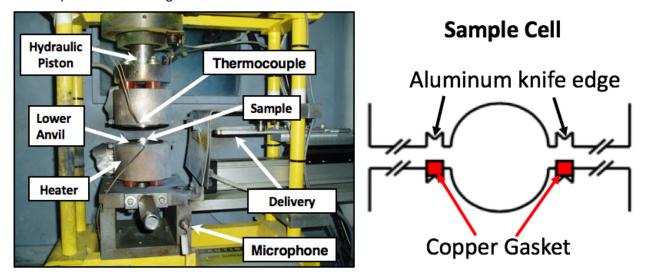


Figure 1. LLNL ODTX system (left) and close-up diagram of sample cavity (right)

2.2 System Experiments

Understanding the response of energetic materials to thermal incidents is very important for the handling, storage and transportation of energetic materials. ODTX is unique because of the capability to generate data that addresses three important technical topics:

- (1) lowest temperature at which thermal explosion would occur (T_{ii}),
- (2) times to thermal explosion at temperatures above T_{li} for deriving activation energies and frequency factors for thermal decomposition kinetics,
- (3) thermal ignition/explosion violence.

Lowest Temperature for Thermal Explosion, (T_{li})

Knowing T_{li} for each energetic material is very important for safe storage and transportation to avoid incidental detonation. Two possible scenarios for causing incidental thermal explosions are:

- Energetic materials may be kept and stored in closed containers that are exposed to hot climates.
 During the summer in some desert areas, outdoor temperature may exceed 120 °F while the
 surface temperature of metallic storage containers exposed to the sun may exceed 170 °F (77 °C).
 Given enough time, some energetic materials may ignite and explode.
- 2. If containers storing energetic materials are kept inside a parked van or truck with windows closed for an extended period of time, the air inside the van may exceed 170 °F. Given enough time, some energetic materials in the containers may ignite and explode.

Time to Explosion Data, Activation Energy, and Frequency Factor

Times to thermal explosion at temperatures above T_{ii} are used for the calculation of activation energy and frequency factor as well as the decomposition kinetics parameters represented by a single-step Prout-Tompkins (Arrhenius) model.

Thermal Explosion Violence

Violence from thermal explosion is an important parameter for predicting degree of damage. After ODTX testing (heating system to explosion), each anvil can be scanned with a surface profilometer to determine the cavity volume increase. Figure 2 shows anvils before and after thermal explosion from the ODTX testing. The violent thermal explosion discolored the anvils and created craters in the anvils.

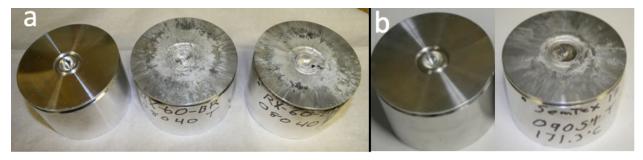


Figure 2. ODTX anvils before and after thermal explosion of: a) hydrogen peroxide-glycerol mixture, left is a pristine anvil, middle is the top anvil and right is the bottom after the thermal explosion [13]; b) Semtex, left pristine anvil, right, bottom anvil after explosion [9]

3.0 P-ODTX—Pressure Measurement Capability on ODTX

Gas evolution of energetic materials in confinement at high temperatures is important to understand the thermal decomposition kinetics. The pressure data also offers the opportunity to validate the existing thermal models and helps to determine whether systems or devices would disassemble when exposed to thermal insults (to avoid further structure damage and casualty) before thermal explosion would occur. To enhance the understanding the gas evolution, the ODTX system has been modified with pressure transducer to do on-line real-time monitoring during thermal experiments. Figure 3 shows the system modification. The top anvil of the system has been drilled and tapped to go through to the sample cavity. To this, a pressure transducer is installed with leads connecting to a data recording system. To accommodate the rapid pressure change occurring near explosion, the system data acquisition was modified in 2016 for rapid monitoring that records data on a µsec-time scale. Each pressure transducer used in the P-ODTX is calibrated for thermal drift at elevated temperatures and leak-tested before use.



Figure 3. Modification of the top anvil incorporating pressure transducer for P-ODTX: a) top of anvil with bulk head, b) top with pressure transducer inserted, and c) close-up of pressure transducer

3.1 P-ODTX Test Data on PETN powder, PBX-9407, LX-10-2, LX-17-1, and Detonator Samples (Cup Test)

P-ODTX tests were performed on PETN, PBX-9407, LX-10-2, LX-17-1, and detonators at different heating rates with gas pressures and thermal explosion temperatures recorded. Each sample was placed inside the anvil cavity in confinement and heated up at a prescribed heating rate until thermal explosion occurred. Figure 4 shows thermal explosion (cook-off) temperature vs. heating rates for various energetic materials.

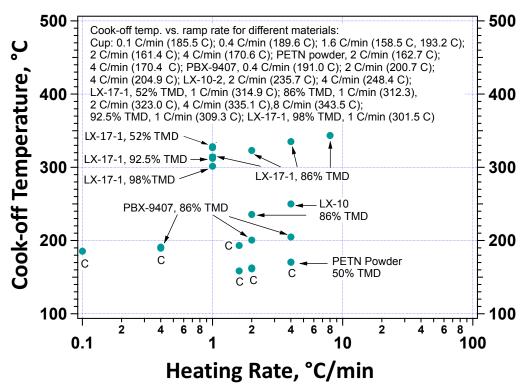


Figure 4. Thermal explosion (cook-off) temperatures at different heating rates and sample densities; average thermal explosion temperature was used for materials tested for reproducibility; C stands for detonator samples.

Faster heating results in higher thermal explosion temperatures and shorter times to explosion. Table 1 lists the materials and test data in Figure 4. Some tests were repeated at identical condition for reproducibility studies. Fast data recording was implemented in mid-2016 that made the pressure measurements in the μ sec-time frame possible. This unique capability captures high-resolution pressure data right before thermal explosion. P-ODTX system has been used extensively to determine thermal explosion data and gas pressure at 1 °C/min heating rate on various batches of LLM-105 and their formulations for the scale-up production process [12].

Thermal explosion (cook-off) temperature is a function of material, heating rate, density, specific surface area, and geometry and can be as expressed by the general equation (1):

$$T_{thx} = A + B \Omega^{x} \rho^{y} S^{z} G^{w}$$
 (1)

Where T_{thx} is the thermal explosion temperature; A and B are constants, characteristic of material; Ω is heating rate; ρ is sample pressed or packed density; S is specific surface area of test sample; G is geometry factor; w, x, y and z are factors that are characteristic of material.

P-ODTX data for PETN (packed powder at 50% TMD), PBX-9407 (pressed part at 86% TMD), and LX-10-2 (pressed part at 86% TMD) are described in Section 3.2, Section 3.3, and Section 3.4, respectively. Experimental results on LX-17-1 and detonators are described in Section 3.5 and Section 3.6, respectively.

Table 1. Thermal explosion data at different heating rates and sample densities for selected materials; average value of temperature of explosion was used for material tested for reproducibility

Sample	Density, % TMD	Heat rate, °C/min	t _{explosion} , h ¹	T _{explosion} , °C ²	HR Data ³
PETN ⁴	50	2.0	1.20	162.7	N
PETN ⁴	50	4.0	0.65	170.4	N
PBX-9407	86	0.4	7.10	191.0	Υ
PBX-9407	86	2.0	1.52	200.7	N
PBX-9407	86	4.0	0.80	204.9	N
LX-10-2	86	2.0	1.81	235.7	N
LX-10-2	86	4.0	0.99	248.4	N
LX-17-1 ⁵	7.2 ⁶	1.0	5.11	325.3	Υ
LX-17-1 ⁵	12.3 ⁶	1.0	5.08	323.3	Υ
LX-17-1 ⁵	52 ⁶	1.0	4.89	314.9	Υ
LX-17-1 ⁵	86	1.0	4.85	311.2	Υ
LX-17-1 ⁵	86	1.0	4.84	312.0	Υ
LX-17-1 ⁵	86	1.0	4.86	313.7	Υ
LX-17-1	86	2.0	2.55	323.0	N
LX-17-1	86	4.0	1.38	335.7	N
LX-17-1	86	4.0	1.34	334.4	Υ
LX-17-1 ⁵	86	8.0	0.71	343.5	Υ
LX-17-1 ⁵	92.5	1.0	4.79	309.3	Υ
LX-17-1 ⁵	98	1.0	4.66	302.6	Υ
LX-17-1 ⁵	98	1.0	4.65	300.7	Υ
LX-17-1 ⁵	98	1.0	4.65	301.2	Υ

^{1.} Time to explosion; 2. Temperature of explosion (cook-off); 3. Fast data capability; 4. Powder; 5. Leveraged from another project; 6. Powder LX-17-1, density calculated from overall sample volume

6. Powder LX-17-1, density calculated from overall sample volume

3.2 P-ODTX Test Results on PETN Powder

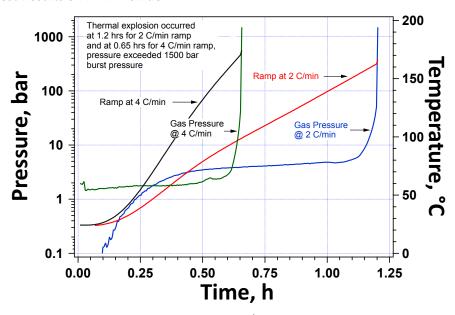


Figure 5. P-ODTX of PETN powder at 50% TMD at 4 and 2 °C/min heating rates, pressure as a function of time

Figures 5 and 6 show the pressure and temperature profiles for 50% TMD PETN powder at 2 and 4 °C/min heating rates. Thermal explosions occurred after heating for 0.65 h and 1.2 h at the 4 °C/min and 2 °C/min heating rates, respectively. Gas pressure did not appreciably increase until 150 °C, a few minutes before thermal explosion. Gas pressure increased very rapidly (> 1500 bar) when thermal explosion occurred. Faster heating resulted in shorter time and higher temperature to thermal explosion. No fast data recording capability was available at the time when PETN powder was tested in the P-ODTX system.

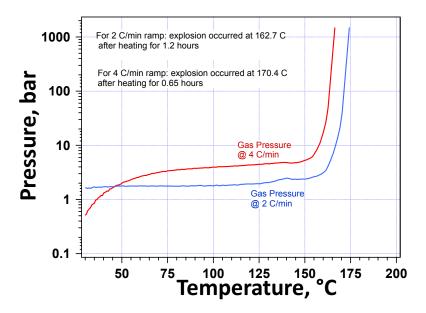


Figure 6. P-ODTX of PETN powder at 50% TMD at 4 and 2 °C/min heating rates, pressure as a function of temperature 3.3 P-ODTX Test Results on PBX-9407

Thermal explosion occurred Thermal explosion occurred 1000 at 1.52 hrs for 2 C/min ramp at 0.8 hrs for 4 C/min ramp 200 Ramp at 2 C/min Ramp at 4 C/min 100 Gas Pressure @ 4 C/min Gas Pressure @ 2 C/min 10 1

Pressure, bar 0.1 0.75 1.00 0.50 1.25 0.00 0.25 1.50 Time, h

Figure 7. P-ODTX of PBX-9407 at 86% TMD at 4 and 2 °C/min heating rates; pressure and temperature as a function of time (pressure fluctuation around 1 bar was due to the noise of the pressure transducers)

Figures 7 and 8 show the pressure and temperature profiles for 86% TMD PBX-9407 at 2 °and 4 °C/min heating rates. Thermal explosions occurred at 204.9 °C after heating for 0.80 h at 4 °C/min and at 200.7 °C after heating for 1.52 h at 2 °C/min heating rate. Gas pressure did not appreciably increase until 180 °C, a few minutes before thermal explosion. Gas pressure increased very rapidly (> 1500 bar) when thermal explosion occurred. Both tests were performed prior to the completion of upgrade to the fast pressure data acquisition system. After the update, a third P-ODTX test was done at 0.4 °C/min heating rate yielding a thermal explosion at 191.0 °C at 7.10 h.

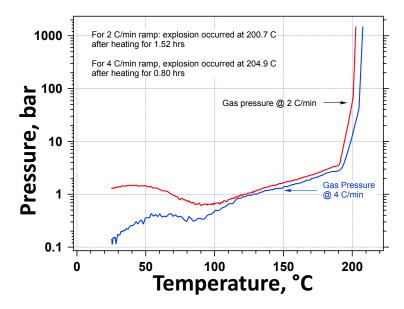


Figure 8. P-ODTX of PBX-9407 at 86% TMD at 4 and 2 °C/min heating rates; pressure as a function of temperature (pressure fluctuation around 1 bar was due to the noise of the pressure transducers)

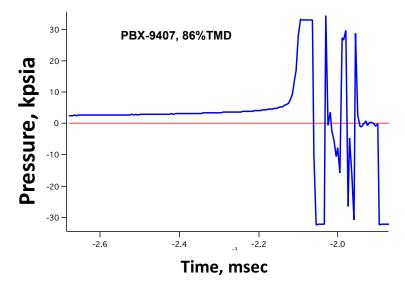


Figure 9. Close-up of gas pressure recording for 86% TMD PBX-9407 at 0.4 $^{\circ}$ C/min heating rate using the fast response instrumentation (for full screen shot see Appendix A)

Figure 9 shows the pressure profile using the fast pressure data acquisition system for the a few msec before thermal explosion for PBX-9407 at 86% TMD at the 0.4 °C/min heating rate. The pressure increased from 5,000 psia to over 30,000 psia in 0.1 msec; the scope was set to record data every 5 μ sec. Appendix A shows a screen shot of the full pressure profiles.

3.4 P-ODTX Test Results on LX-10-2

Figures 10 and 11 show the pressure and temperature profiles for 86% TMD LX-10-2 at 2 and 4 °C/min heating rates. Thermal explosions occurred at 248.4 °C after heating for 0.99 h at 4 °C/min and at 235.7 °C

after heating for 1.81 h at 2 °C/min. The gas pressure did not appreciably increase until 200 °C, a few minutes before thermal explosion. Gas pressure then increased very rapidly (> 1500 bar) when thermal explosion occurred. No fast data recording capability was available at the time when LX-10-2 was tested in the P-ODTX system. It may be desirable to perform more P-ODTX tests on LX-10-2 at slower heating rates 1 °C/min and 0.1 °C/min.

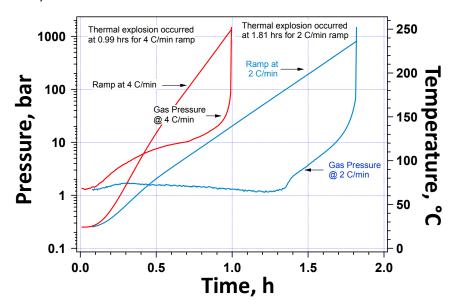


Figure 10. P-ODTX of LX-10 at 86% TMD at 4 and 2 °C/min heating rates; pressure as a function of time (pressure fluctuation around 1 bar was due to the noise of the pressure transducers)

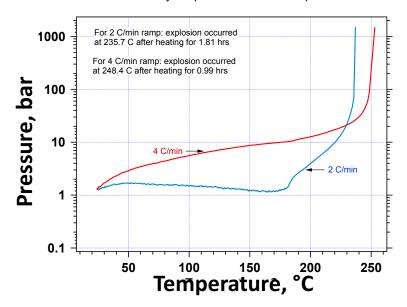


Figure 11. P-ODTX of LX-10 at 86% TMD at 4 and 2 °C/min heating rates; pressure as a function of temperature (pressure fluctuation around 1 bar was due to the noise of the pressure transducers)

3.5 P-ODTX Test Results on LX-17-1

Testing at Different Heating Rates and Densities

Table 1 shows the data for 14 P-ODTX tests that have been conducted on the LX-17-1 at different heating rates (1 to 8 °C/min) and different sample density (52% TMD to 98% TMD) from May 2015 to September 2017. Some were tested more than once at identical condition for reproducibility. The average thermal explosion temperature for the 3 tests performed for the 86% TMD sample at 1 °C/min heating rate was 312.3 ± 1.0 °C. Three tests were also conducted on the 98% TMD sample at 1 °C/min heating rate, with averaged thermal explosion temperature of 301.5 ± 0.8 °C. Two tests were conducted on the 86% TMD sample at the higher heating rate of 4 °C/min, and the average thermal explosion temperature was 335.1 ± 0.65 °C. The table shows that the reproducibility of the P-ODTX test for thermal explosion temperature measurement is within $\pm 0.5\%$, validating that the P-ODTX system is a useful tool for measuring thermal explosion temperature of LX-17-1 in confinement at different heating rates.

Figure 12 shows a close-up of the LX-17-1 thermal explosion data shown in Figure 4, including the 7.2% TMD and 12.3% TMD samples. These two samples are formed by varying the amount of LX-17-1 powder in the sample cavity. The effect of density and heating rate on the thermal explosion properties are discussed below, but it is clear from the figure, that the relationships are closely linear. Testing at 0.1 and 100 °C/min heating rates on various sample density is being planned and the data will be used to support a revised view of thermal behavior based on equation (1).

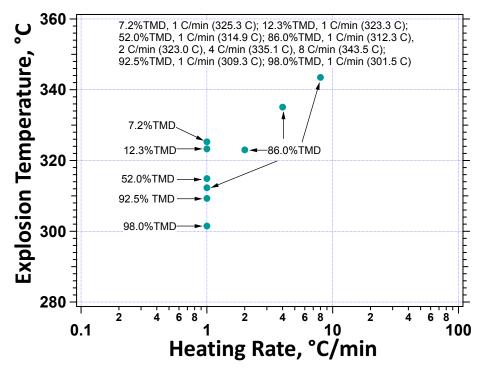


Figure 12. Thermal explosion (cook-off) temperatures at different heating rates and sample densities for LX-17-1; average thermal explosion temperature was used for materials tested for reproducibility

P-ODTX Test Results on 86% TMD Samples

Two P-ODTX tests were performed on 86% TMD LX-17-1 samples at 2 and 4 °C/min heating rates in 2015 before the upgrade to the data acquisition system. Figures 13 and 14 show the pressure as a function of time and temperature. Thermal explosions occurred at 335.7 °C after heating for 1.38 h at 4 °C/min and at 323.0 °C after heating for 2.55 h at 2 °C/min. Gas pressure increased slowly when the heating started, accelerated at 250 °C, and increased very rapidly above 300 °C until thermal explosion occurred. Gas pressure exceeded the pressure limit (210 bar) of the pressure transducer used for the experiment. After

the upgrade to fast pressure recording, P-ODTX test on the 86% TMD sample at 4 °C/min heating rate was repeated in 2017.

Figure 15 shows the gas pressure right before thermal explosion. Pressure increased from 5000 psia to over 30,000 psia in 1.5 msec. The gas pressure increases of PBX-9407 was much faster (see Figures 7-9) right before thermal explosion than that of LX-17-1. This is indicative of deflagration rate of the energetic material. For the gas pressure at a few msec before thermal explosion, the pressure increased from 5,000 psia to over 30,000 psia in 1 msec; the scope was set to record data every 20 μ sec.

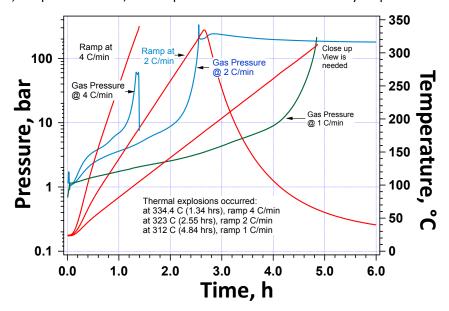


Figure 13. P-ODTX of LX-17-1 at 86% TMD at 4, 2, and 1 °C/min heating rates; pressure as a function of time

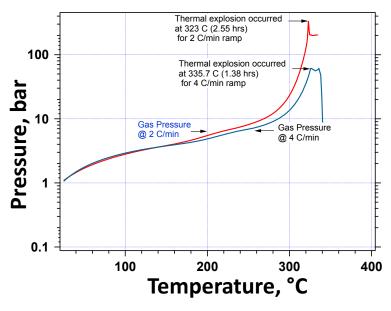


Figure 14. P-ODTX of LX-17-1 at 86% TMD at 4 and 2 °C/min heating rates; pressure as a function of temperature

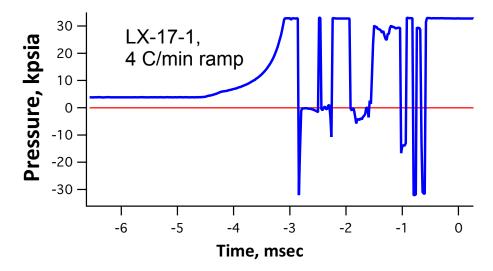


Figure 15. Close-up of gas pressure recording for LX-17 at 86% TMD at 04 °C/min heating rate using the fast response instrumentation (for full screen shot see Appendix B)

Effect of Sample Density on Gas Pressure Profiles at 1 °C/min Heating Rates

Figure 16 shows the gas pressure profiles from screen shots when heating at 1 °C/min for LX-17-1 samples of different density: 52%, 86%, 92.5%, and 98% TMD. The pressure profile for the 98% TMD sample showed a sudden jump after temperature reached 295 °C, possibly due to the sample cracking and breaking apart. These samples have virtually no free space, so this also could be due to thermal-mechanical effects. Pressure curves for samples of 52%, 86%, and 92.5% TMD do not exhibit this pressure behavior. This behavior has been seen in SITI experiments as an effect of density, also [5]. Full screen shots of all these tests are listed in Appendix B.

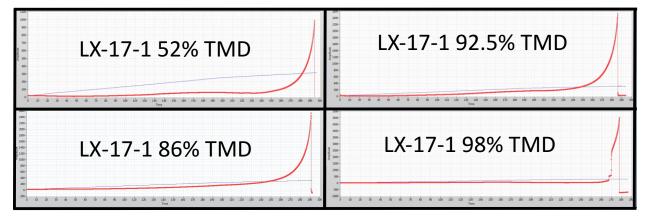


Figure 16. Screen shot of gas pressure recording for 52, 86, 92.5 and 98% TMD LX-17 at a 1 $^{\circ}$ C/min heating rate; all x-axis units are relative time from start and y-axis units are amplitude that is calibrated to pressure

P-ODTX Testing at Slow and Fast Heating

Figure 12 shows a significant effect of heating rate on thermal explosion temperatures when other factors are kept constant. Holding the density constant, LX-17-1, PBX-9407, LX-10, and PETN exhibit an increase in thermal explosion temperature with increasing heating rate. P-ODTX work is being planned to increase the heating rate range from a very slow 0.1 °C/min to a very fast 100 °C/min on samples of 86% and 98% TMD.

3.6 P-ODTX Test Results on Detonators (Cup Tests)

Figure 17 shows the P-ODTX configuration for testing detonators—the cup test. The system has been modified from the standard Al sphere configuration to accommodate the different geometry of the detonator design. Seven tests were performed at 0.1, 0.4, 1.6 (repeated), 2 and 4 °C/min heating rates. Table 2 shows times to thermal explosion, temperature at thermal explosion and comments on gas pressures.

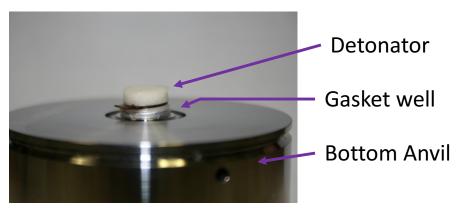


Figure 17. Detonator Cup Test configuration on bottom anvil of P-ODTX

Table 2. P-ODTX results for detonator samples in Cup Test

Detonator	Heat rate, °C/min	t _{explosion} , h ¹	T _{explosion} , °C ²	Notes
Det-1 ³	0.1	27.25	185	
Det-2 ³	0.4	7.01	189.6	Similar to PBX-9407, 191.0 °C
Det-3 ³	1.6	1.41	158.5	Similar to PETN
Det-4 ³	1.6	1.78	193.2	Similar to PBX-9407
Det-5	2	1.18	161.4	Similar to PETN powder, 162.7 °C
Det-6	4	0.66	170.6	Similar to PETN powder, 170.4 °C
Det-7	2	Soaked at 115 °C and	N/A ⁴	Low pressure, 68 psia at 115 °C;
		130 °C; no explosion		higher pressure, 760 psia at 130 °C

^{1.} Time to explosion; 2. Temperature of explosion; 3. Fast data capability; 4. Not applicable

Table 2 indicates that Detonator samples 5 and 6 had similar times to thermal explosion and thermal explosion temperatures to those of PETN powder. Hence, when heating at 2 and 4 °C/min, the detonators exhibited same thermal response as PETN powder. When heating at 0.4 °C/min, Detonator sample 2 showed same thermal response as PBX-9407 as their temperatures and times of thermal explosion were similar: Detonator sample 2, 189.6 °C and 7.01 h; PBX 9407, 191.0 °C and 7.10 h. Tests at 1.6 °C/min heating rate was performed in duplicate with different results. Detonator sample 3 exhibited similar thermal response to PETN, while Detonator sample 4 showed similar thermal response to PBX-9407. It could be possible, when heating at 1.6 °C/min, the thermal response falls in the transition zone of PETN and PBX-9407. More P-ODTX experiments are necessary on PETN powder and PBX-9407 at 0.1 and 1.6 °C/min heating rates for more direct comparison with detonators.

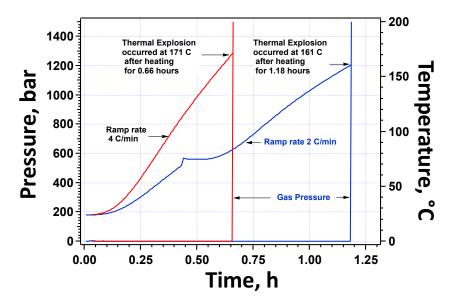


Figure 18. P-ODTX of Detonator sample 6 at 4 °C/min and Detonator sample 5 at 2 °C/min heating rates; pressure as a function of time

Figures 18 and 19 show the comparison of pressure and temperature profiles for the Detonator samples 6 and 5 at 2 and 4 °C/min heating rates, respectively. The left side shows thermal explosion occurred after heating for 0.66 h at 4 °C/min. This is the same as PETN powder. The right side shows gas pressure increases were negligible, probably due to the presence of thin film and foam that blocked the gas release in a short period of time before thermal explosion occurred.

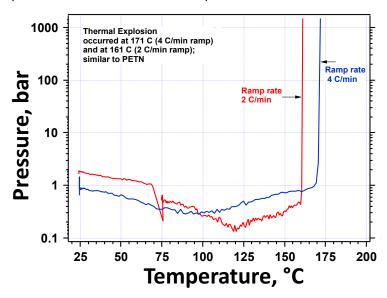


Figure 19. P-ODTX of Detonator sample 6 at 4 °C/min and Detonator sample 5 at 2 °C/min heating rates; pressure as a function of temperature (pressure fluctuation around 1 bar was due to the noise of the pressure transducers)

To understand this pressure behavior, Detonator sample 7 was heated at 2 °C/min to 115 °C and soaked overnight to allow enough time for gas molecules to permeate through this thin film and foam (if occurring). The system was then cooled to ambient temperature, then reheated to 130 °C and soaked overnight. Figure 20 shows the pressure and temperature behavior under these conditions. Gas

pressurization of 4.5 bar was observed with heating at 115 °C and 52 bar was observed at 130 °C. No thermal explosion occurred both hold temperature.

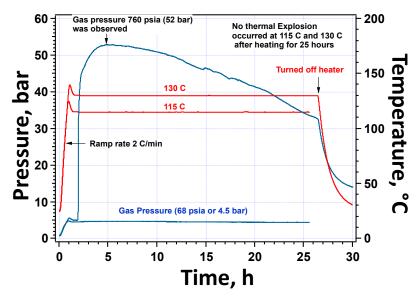


Figure 20. P-ODTX of Detonator sample 7, first heated at 2 °C/min to 115 °C, let to soak overnight, then cooled to room temperature; second heated at 2 °C/min to 130 °C, let to soak overnight then cooled to room temperature

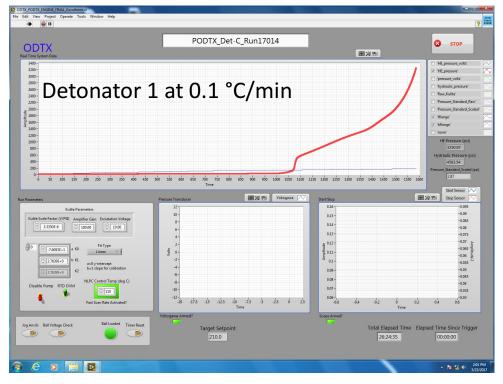


Figure 21. Screen shot of pressure monitoring for P-ODTX for Detonator sample 1 at 0.1 °C/min heating rate

Figure 21 left side shows the screenshot of pressure and temperature profiles on P-ODTX test of Detonator sample 1 at $0.1\,^{\circ}$ C/min heating rate; a sudden jump in gas pressure indicating melting of PETN that created passage for gas molecules. The pressure profile of Detonator sample 2 shows a similar pressure jump at the $0.4\,^{\circ}$ C/min heating rate. The sudden jump in gas pressure in both profiles indicates the melting of PETN

created passage for gas molecules. Full screen shot of Detonator sample 2 at 0.4 °C/min heating rate is shown in Appendix C.

4.0 Summary

The thermal behavior of PETN, PBX-9407, LX-10-2, LX-17-1, and detonator samples have been characterized in the P-ODTX system. The effects of heating rate and sample density on the thermal explosion temperature and time were significant. Faster heating resulted in higher thermal explosion temperatures. Also, thermal explosion temperatures and times for a dense LX-17-1 part at 98% TMD are higher than those of correspondingly less dense parts. Thermal response of the detonator samples to different heating rates exhibited a unique characteristic. When heating at 2 and 4 °C/min, the thermal explosion temperatures are very similar to those of PETN powder. When the detonator samples were exposed to slow heating of 0.1 °C/min and 0.4 °C/min, the thermal explosion temperatures were close to those of PBX-9407. For LX-17-1, the pressure profile for the 98% TMD sample showed a sudden jump after temperature reached 295 °C, probably due to the sample compression and breaking apart. This study also demonstrated, for the first-time, direct gas-pressure measurements in µsec intervals during a thermal explosion of LX-17-1, PBX-9407 and detonators. Rate of gas pressure increase right before thermal explosion is indicative of deflagration rate. More thermal experiments on LX-17-1 are being planned to address the slow heating (0.1 °C/min) to fast heating (100 °C/min) range.

ACKNOWLEDGMENTS

Funding from W Program is greatly appreciated. This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. LLNL-TR-739143.

References

- [1] Catalano E, McGuire R, Lee E, Wrenn D, Ornellas D and Walton J 1976 The Thermal Decomposition and Reaction of Confined Explosives", 6th International Detonation Symposium.
- [2] Chidester S K, Tarver C M, Green L G and Urtiew P A 1997 On the Violence of Thermal Explosion in Solid Explosives *Combustion and Flame* **110** *264-280*.
- [3] Tarver C M and Tri T 2004 Thermal Decomposition Models for HMX-based plastic bonded explosives *Combustion and Flame* **137** 50-62.
- [4] Wardell J F and Maienschein J L 2002 The Scaled Thermal Explosion Experiment 12th International Detonation Symposium, pp 384-393, San Diego August.
- Dodd A B and Kaneshige M J 2006 Cook-off Model Development and Analysis of Energetic Materials Using Instrumented Thermal Ignition (SITI) Experimental Data 13th International Detonation Symposium, pp 516-526, Norfolk July.
- [6] Williams M R and Matei M V 2006 The Decomposition of Some RDX and HMX Based Materials in the One-Dimensional Time to Explosion Apparatus Part 1 Time to Explosion and apparent Activation Energy *Propellats Explosive Pyrotechnics* **31** 435-441.
- [7] Koerner J, Maienschein, J L, Burnham A and Wemhoff A 2007 ODTX Measurements and Simulations on Ultra Fine TATB and PBX 9502, UCRL-Conf-232590, LLNL.
- [8] Maienschein J L, Garza R G, Nichols A L and Chandler J B 1996 Kinetics of Thermal Ignition of AP/AI/HTPB Propellants, JANNAF Meeting, Monterey.

Thermal Safety Characterization by P-ODTX

- [9] Hsu P C, Hust G, Howard M, Chidester S K, Springer, H K and Maienschein J L 2010 The ODTX System for Thermal Ignition and Thermal Safety Sudy of Energetic Materials, 14th International Detonation Symposium, Coeur d'A;eme, Idaho, April 11-16, 984-990.
- [10] Hsu P C, Hust G, Howard M, Chidester SK, Springer H K and Maienschein J L 2011 Study of thermal Thermal Sensitivity and Thermal Explosion Violence of Energetic Material s in the LLNL ODTX System, APS Conference, Chicago, July.
- [11] Hsu P C 2009 LLNL OD TX System for Thermal Safety Determination of Energetic Materials, LLNL-BR-411732.
- [12] Hsu P C et al. Recent Advanced on Thermla Safety Characterization of Energetic Materials, APS Shock Compressional Meeting on Condensed Matters, St. Louis, MO, 2017
- [13] P C Hsu, G Hust, M X Zhang, T K Lorenz, J G Reynolds, L Fried, H K Springer and J L Maienschein, Study of thermal sensitivity and thermal explosion violence of energetic materials in the LLNL ODTX system, J. Physics, Conference Series, 500 (2014), 052019.

Appendix A. Full screen shot of temperature and pressure profiles on P-ODTX of PBX-9407 at 86% TMD at 0.4 °C heating rate.

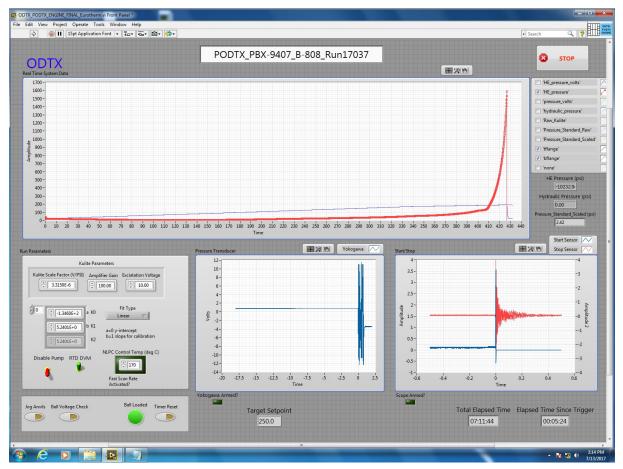
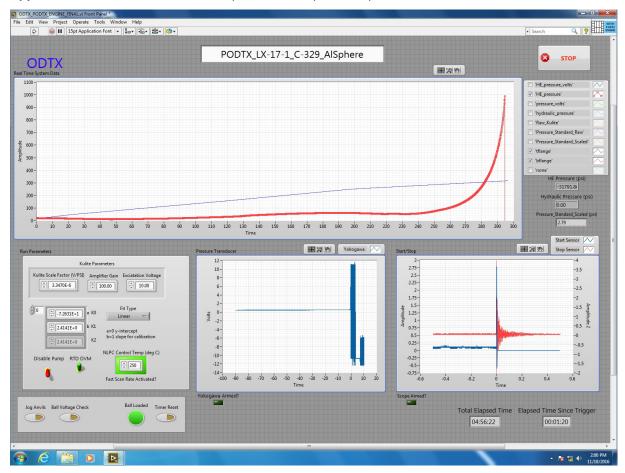


Figure A-1. Full screen shot recording for 86% TMD PBX-9407 at 0.4 °C/min heating rate; top profile is normal pressure monitoring (amplitude vs. time) which is calibrated to pressure vs. time as in Figures 7 and 8; lower left profile is meter output using fast scanning (volts vs. time) which is calibrated to pressure vs. time as in Figure 9



Appendix B. Full screen shots of temperature and pressure profiles on P-ODTX of LX-17-1

Figure B-1. Full screen shot recording for 52% TMD LX-17-1 at 4 °C/min heating rate; top profile is normal pressure monitoring (amplitude vs. time) which is calibrated to pressure vs. time; lower left profile is meter output using fast scanning (volts vs. time) which is calibrated to pressure vs. time

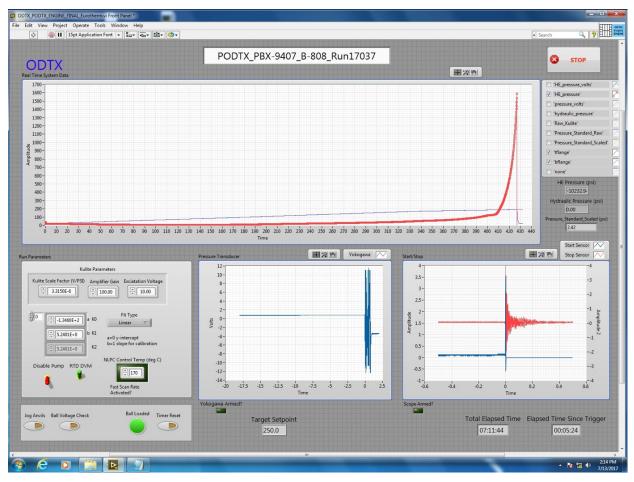


Figure B-2. Full screen shot recording for 86% TMD LX-17-1 at 4 °C/min heating rate; top profile is normal pressure monitoring (amplitude vs. time) which is calibrated to pressure vs. time as in Figures 13 and 14; lower left profile is meter output using fast scanning (volts vs. time) which is calibrated to pressure vs. time as in Figure 15

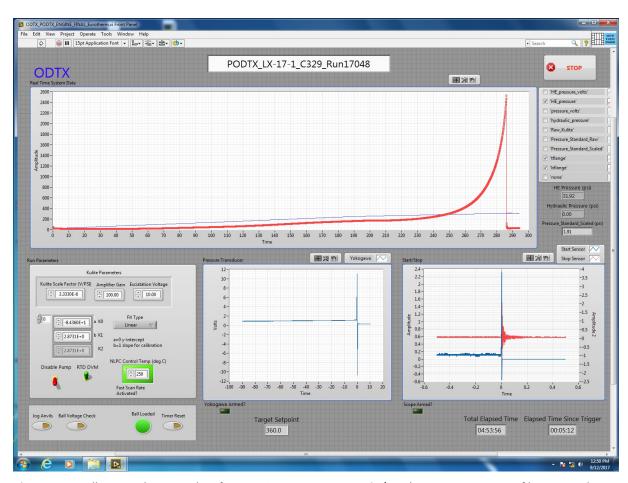


Figure B-3. Full screen shot recording for 92.5% TMD LX-17-1 at 4 °C/min heating rate; top profile is normal pressure monitoring (amplitude vs. time) which is calibrated to pressure vs. time; lower left profile is meter output using fast scanning (volts vs. time) which is calibrated to pressure vs. time

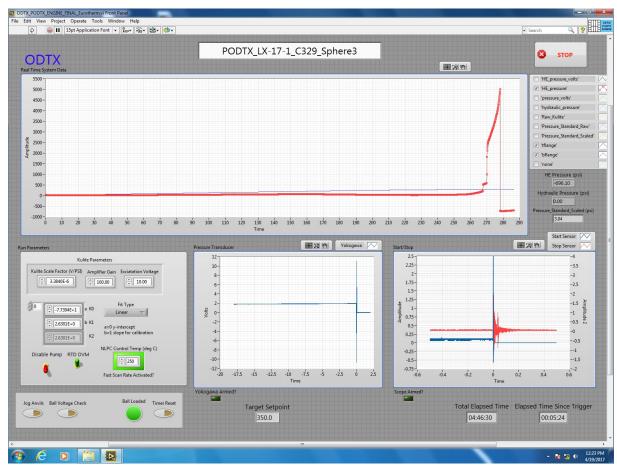


Figure B-4. Full screen shot recording for 98% TMD LX-17-1 at 4 °C/min heating rate; top profile is normal pressure monitoring (amplitude vs. time) which is calibrated to pressure vs. time; lower left profile is meter output using fast scanning (volts vs. time) which is calibrated to pressure vs. time

Appendix 3: Screenshot of pressure and temperature profiles on P-ODTX test of Detonator sample 2 at 0.4 C/min heating rate

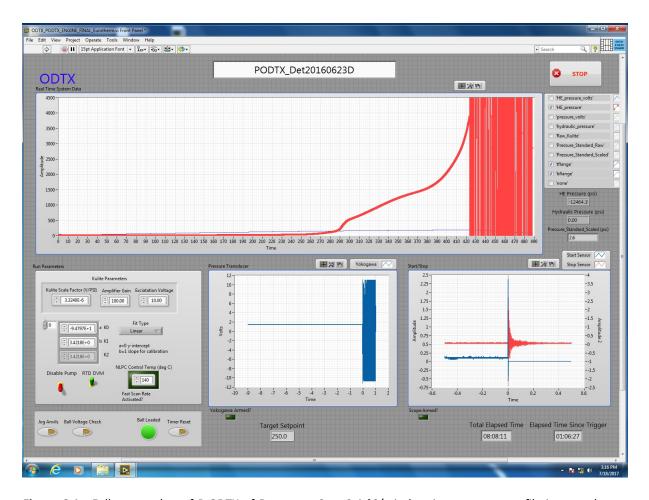


Figure C-1. Full screen shot of P-ODTX of Detonator 2 at 0.4 °C/min heating rate; top profile is normal pressure monitoring (amplitude vs. time) which is calibrated to pressure vs. time; lower left profile is meter output using fast scanning (volts vs. time) which is calibrated to pressure vs. time; a sudden jump in gas pressure indicating melting of PETN that created passage for gas molecules.