Center for Fire & Explosives, Forensic Investigations, Training & Research (CENFEX)

Proficiency Testing for the Thermal Sensitivity of Pentaerythritol Tetranitrate (PETN) and Cyclotrimethylenetrinitramine (RDX) utilizing Differential Scanning Calorimetry (DSC)

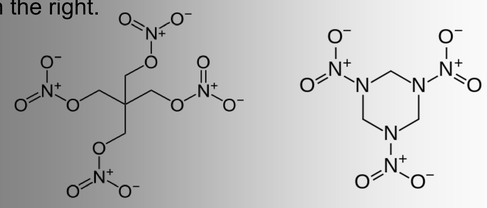
Matthew K. Green, Ph.D. – School of Forensic Sciences, CENFEX Fire and Explosives Laboratory

Julianne Green – School of Forensic Sciences, Undergraduate Laboratory Intern

INTRODUCTION

DSC, or Differential Scanning Calorimetry, can be used as a method of testing the thermal sensitivity of energetic materials. DSC is the most commonly used thermal analysis technique due to its ease of operation and rapid analysis. Furthermore, this instrumentation provides precise, reproducible heat flow measurements.[1] Thermal sensitivity testing determines the potential hazards of energetic materials when exposed to increasing levels of heat. Heat capacity measurements are particularly relevant when mixing, drying, transferring, and/or storing energetic materials.[1]

Two commonly used explosives are PETN and RDX. PETN, or pentaerythritol tetranitrate, is a high explosive that is used in military detonating cord and blasting caps.[2] RDX, or cyclotrimethylenetrinitramine, is used extensively in military munitions.[3] The chemical structures are shown below, PETN on the left and RDX on the right.



As a means to increase the validity of thermal sensitivity testing, proficiency tests can be performed to improve protocols and standardization. The Explosives Testing Users Group (ETUG), organized by Safety Management Services, Inc., developed a "Round Robin" testing project, and selected thermal analysis of PETN and RDX. For this research, a TA Instruments, Inc. SDT 650 was used to test the thermal sensitivity of the explosive standards. This research demonstrates the DSC proficiency testing performed at the CENFEX Fire and Explosives Research Laboratory. Results from the laboratory were compared to federal, national, and international laboratories participating in the Round Robin testing project. Statistical analyses demonstrated similarities among participating laboratories and identified potential protocol and standardization improvements.

MATERIALS

Instrument:

- SDT 650 – TA Instruments, Inc.

Supplies:

- Tzero pans and lids – TA Instruments, Inc.
- Tin metal calibrant – TA Instruments, Inc.
- RDX – Omni Distribution, Inc.
- PETN – Omni Distribution, Inc.

PROFICIENCY TESTING

Procedure:

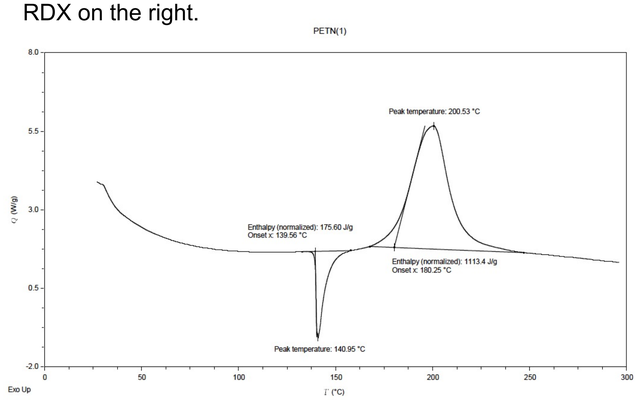
1. DSC Calibration Verification
2. PETN and RDX Analyses
3. Result Comparison and Statistical Analysis

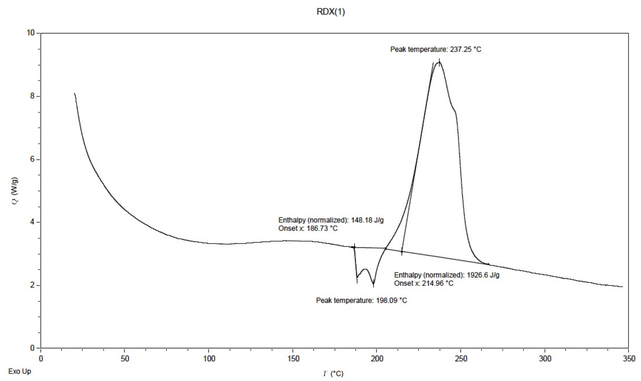
Step 1:

- Three separate tin calibrant samples were analyzed using the SDT 650 DSC/TGA Instrument from TA Instruments, Inc.
- The melting point of tin was determined for each calibrant sample, and the average was calculated to be 232.14°C
- The acceptable range listed in the ETUG DSC Round Robin Procedure[4] is 230.9°C to 232.9°C
- Observed results demonstrated successful DSC calibration

Step 2:

For PETN and RDX, three proficiency tests were completed to determine both the average melting temperature and average decomposition onset temperature. Examples of observed thermograms are shown below, PETN on the left and RDX on the right.

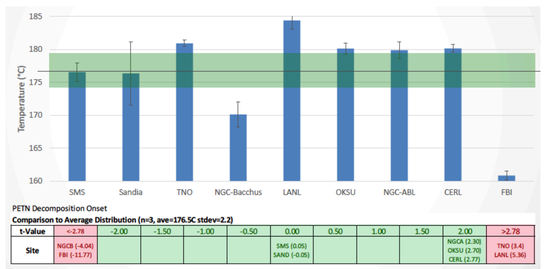


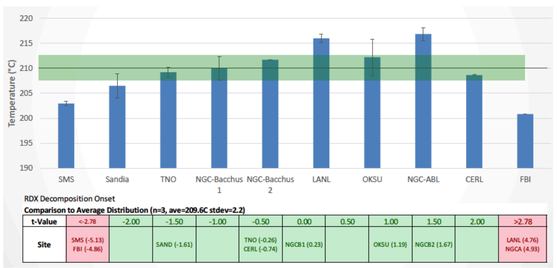


Results: Average melting and decomposition temperatures for PETN – 139.59°C and 180.35°C
Average melting and decomposition temperatures for RDX – 186.74°C and 212.00°C

Step 3:

At the Explosives Testing Users Group 2018 Meeting in Park City, Utah, results from each participating laboratory were presented and discussed. Reported values were compared via statistical analysis. Result comparison graphs[5] for decomposition onset temperatures are listed below, PETN on the left and RDX on the right. Each bar represents a participating laboratory, with "OKSU" showing results from the CENFEX Fire and Explosives Research Laboratory at OSU-CHS School of Forensic Sciences.





RESULTS SUMMARY

- Thermal analysis is a crucial component of characterizing explosive materials
- Differential Scanning Calorimetry (DSC) analysis is the most common and understood method of thermal characterization
- Explosives Testing Users Group (ETUG) Round Robin completed to improve standardization within the field of energetics characterization
- Results obtained at the CENFEX Fire and Explosive Research Laboratory reflect consistency, for tin metal and both selected energetic materials, PETN and RDX
- 9 national and international laboratories participated in the proficiency testing exercise
- CENFEX Fire and Explosives Research Laboratory results matched with the group average temperatures for:
  - Tin melting onset
  - PETN melting onset
  - PETN decomposition
  - RDX decomposition
- Substantial deviation from the group mean was observed for RDX melting onset
- Following procedural adjustments, average values for PETN and RDX temperatures can potentially be established for certification by the Explosives Testing Users Group

REFERENCES

1. Conkling and Mocella, Chemistry of Pyrotechnics: Basic Principles and Theory, 2nd Edition, 2011.
2. Beveridge, Forensic Investigation of Explosions, 2nd Edition, 2012
3. United States Environmental Protection Agency, Technical Fact Sheet – RDX, 2014
4. Explosive Testing User's Group Round Robing Testing Procedure, Safety Management Services, 2018.
5. Guymon and Snow, ETUG DSC Summary of Results, 10th Annual Explosives Testing Users Group Meeting, 2018

ACKNOWLEDGEMENTS

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