R1-A.1: Characterization of Explosives & Precursors

I. PARTICIPANTS

<table>
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<tr>
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<th>Name</th>
<th>Title</th>
<th>Institution</th>
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<tr>
<td>Jimmie Oxley</td>
<td>Co-PI</td>
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<td>Jim Smith</td>
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<tr>
<td>Gerald Kagan</td>
<td>Post-Doc</td>
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<td>URI</td>
<td><a href="mailto:gkagan@chm.uri.edu">gkagan@chm.uri.edu</a></td>
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<td>Austin Brown</td>
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<td>Devon Swanson</td>
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<tr>
<td>Jamie Butalewich</td>
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II. PROJECT DESCRIPTION

A. Project Overview

All new materials require characterization. In the case of explosives, complete characterization is a matter of safety as well as performance. Most homemade explosives (HMEs) are not new, with some having been reported in the late 1800s. However, common handling of these explosives and resulting accidents caused by those involved in the homeland security enterprise (HSE) demand a thorough understanding of their properties. Admittedly, this mission is too big to cover without more researchers, funding, and time. Therefore, we have chosen areas considered most urgent or reachable by our present experience and instrument capabilities. We have examined triacetone triperoxide (TATP) in detail. Presently, we are examining hexamethylene triperoxide diamine (HMTD), erythritol tetranitrate (ETN), and other nitrated sugars and fuel/oxidizer (FOX) mixtures.

Characterization has included a detailed study of the thermal decomposition of ETN. Our work has highlighted a hazardous operation that many in the HSE perform. Because ETN melts at 60°C and appears unchanged to over 100°C (see Fig. 1 on the next page), melt-casing this material is sometimes included in HME training. We examined the thermal decomposition of ETN, both through experimental and computational methods. Our examination revealed that decomposition can occur at its melt. This means cavalier melt-casting may be highly hazardous. In addition to ETN kinetic parameters, decomposition products were examined to elucidate its decomposition pathway. It was found that ETN begins its decomposition sequence by a unimolecular homolytic cleavage of the internal and external O-NO₂ bonds, while the competing HONO elimination reaction is largely suppressed. The global activation energy for decomposition is found to be 104.3 kJ/mol with a pre-exponential factor of \(3.72 \cdot 10^9\) s⁻¹. Despite the ability to exist in a molten state, ETN has a lower thermal...
stability than a similar tetranitrate, PETN, which is not observed to melt [1].

Sugar nitrates contain more than the four nitrate groups that were examined. We have attempted to synthesize both mannitol and sorbitol hexanitrate under a number of nitration conditions. Under no experimental conditions attempted was either sugar totally nitrated. Furthermore, when sitting at room temperature, the amount of hexanitrate in a sample decreased relative to the amount of pentanitrate, suggesting facile decomposition. This information needs to be included in the characterization of these materials.

Development of analytical protocols was necessary to allow us to quantify TATP and HMTD at levels as low as 25 ppm. This work supported Project R1-C.2 allowing quantification of the signature released (from the safe-scent aids) and of the pickup attributable to the enhanced swabs. As part of this work, it was discovered that using acetonitrile (ACN) causes low concentrations of TATP and HMTD to go unobserved. Figure 2 shows the dramatic difference that eliminating ACN made in TATP analysis. This finding was sufficiently important, and has been published [2].

Figure 1: DSC (differential scanning calorimetry) of ETN.

Figure 2: APCI mass spectrum of TATP-NH$_4^+$ (240.1455 m/z) is barely visible in top spectrum with ACN.
Because it has become known that we work with these HMEs, and that we are developing safe-scent training aids, we have been asked about the toxicity of peroxide explosives for bomb-sniffing canines. Nitroarene explosives are toxic and long-term exposure can be fatal to humans. Realizing that no information existed on the toxicity of these compounds, which many in the HSE community are exposed to, we have initiated a study to determine canine and human toxicity, first of TATP and then other peroxide explosives. It is essential we obtain this information on TATP because it is highly volatile. Therefore, anyone working with this material is constantly breathing in its vapors. We must determine to what extent working with TATP is a health threat. Furthermore, we would like to determine if this problem could be used to forensic advantage. In previous studies, we observed that the hair of those handling military explosives is contaminated in as little as 2 hours and the explosive contamination survives in hair overnight, despite showering. We have shown that TATP remains in hair for over two days and is more persistent in hair than the more water-soluble TNT or ethyleneglycol dinitrate (EGDN) (see Fig. 3).

<table>
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<tr>
<th>ug/ g hair Initially</th>
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<th>TNT (648)</th>
<th>PETN (5040)</th>
<th>EGDN (48h)</th>
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<td>77</td>
<td>100</td>
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<td>59</td>
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<td>47</td>
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Percent Retained after Standing in Air for 48 hrs

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<td>Oriental Black</td>
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<tr>
<td>Brown</td>
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Percent Retained after 3 washings with 2% SDS

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Figure 3: Explosive (ug/g) remaining in hair after standing 48 hours or multiple water rinses.

We are now examining whether TATP remains in human body fluids long enough to serve as a new source of forensic evidence of illegal activity. Initial results were extremely erratic, forcing a more detailed examination of aqueous TATP solutions. It was found that even solutions with low concentrations of TATP experience significant loss of TATP by evaporation (see Fig. 4). It became necessary to run control experiments where septum-capped vials were punctured with a syringe every time an aliquot was removed from the septum-capped reaction vial. To obtain loss due to metabolism only, as shown in Figure 5 on the next page, a control concentration was taken as a baseline at each time point.

Figure 4: TATP (50uM) in aqueous solution.
Figure 5 shows the rapid loss of 10 uM TATP under incubation conditions. Since data was adjusted to account for evaporation, the loss of TATP shown is a result of metabolism. In contrast, the solution containing higher concentrations of TATP (50 uM) with NADPH showed no TATP loss other than that which could be accounted for by evaporation. This is the first suggestion that a) TATP may stay in the body, and b) it may be toxic since it appears possible it builds up at higher concentrations.

Only one metabolite has been observed when aqueous TATP solutions are incubated at 37°C with dog liver microsomes: the hydroxyl-TATP (see Fig. 6). Interestingly, we have been unable to observe such intermediate species in previous decomposition studies where we attempted to identify early transformation products of TATP.

Figure 6: NADPH-dependent biotransformation of TATP in dog liver microsomes at 37°C.

B. Year Two (July 2014 through June 2015) Biennial Review Results and Related Actions to Address

There was a request to track the use, in addition to the number of users, of the University of Rhode Island (URI) Explosives Database, an interactive library of analytical data for explosive and energetic compounds. The appropriate code has been added, and monthly usage was reported at the Year 4 Program Review in January 2017.

There was a question as to how we chose what HMEs to study. We continue to study those that are of interest by members of the Department of Homeland Security Science and Technology (DHS S&T) group. This includes studies of FOX mixtures as well as peroxide explosives. This information is shared with other Center projects and especially with other members of R1 thrust.
C. State of the Art and Technical Approach

A major strength of our project is that in many cases we have introduced the best ways to approach these hazardous materials. The instruments used (infrared (IR), Raman, $^1$H and $^{13}$C NMR spectroscopy, and mass spectrometry (MS) and differential scanning calorimetry (DSC)) are commercially available. Thus, we introduced the laboratories serving the HSE to certain safe approaches. This year, for the third time, we offered a one-day hands-on course called “Explosive Analysis.” One participant, who works for the Bureau of Alcohol, Tobacco, Firearms and Explosives (BATFE), wrote the following comment: “I wanted to thank you for hosting that wonderfully informative short course! Every aspect was exactly what I needed to tie up the loose ends of my new field of study [...] This note book now serves as a great tool to catch up the other people who hired on with me [...] Your graduate staff did a great job.” Furthermore, our 2016 paper published in Rapid Communications in Mass Spectrometry [2] received the following comment from David Schiessel (Organic Manager, Babcock Labs) in an email received on November 9, 2016: “[...] alerting LC-MS users about this [ACN] effect, is very applicable to emerging fields; especially to non-targeted analysis where one might completely miss certain suspects/non-targets due to the fact that they poorly ionize under acetonitrile conditions [...] This one’s a keeper in my collection.” In the field of energetics, the results of this task area are often cited [3-7].

D. Major Contributions

- Extensive TATP characterization—safe scent aids, gentle destruction (Years 1-4)
- The limitations of certain oxidizers in terms of terrorist use (Years 1-2)
- Baseline information about HMTD chemical properties and reactivity (Years 1-4)
- Identifying the hazards of humidity to HMTD (Years 2-3)
- Formation mechanism of HMTD initiated (Years 2-3)
- Gentle destruction methods for HMTD (Years 3-4)
- Safe-scent aids for HMTD (Years 3-4)
- Revealing modes by which the peroxide explosive signature can be masked by a solvent (Years 3-4)

In Years 1 and 2, TATP was targeted. We examined factors affecting its synthesis, elucidated mechanisms for its formation and for destruction, investigated hundreds of potential adulterants, and found a gentle method for destruction. In Years 3 and 4, we devised methods of analysis to overcome issues with ACN suppression of the MS signal [2] and issues with evaporation. Both of these problems appear to have gone unrecognized in the HSE and likely have skewed results. TATP was found to evaporate so rapidly from aqueous solution that it required special experimental procedures. In the first years of TATP studies (circa 2000), researchers in the HSE recognized that evidence containing traces of TATP required careful storage. However, it may come as a surprise to many that dilute solutions of TATP drop in concentration even when stored in septum-capped vials, such as those that are routinely used in gas and liquid chromatographic analysis (see Fig. 4).

We began Year 1 of our HMTD studies with the goal of determining the reason for an accident which sent a member of the HSE to the emergency room. We examined the thermal stability and compatibility of HMTD with a number of materials with which it would come in contact. We found and publicized the hazards of humidity when working with HMTD. In Years 2 and 3, we examined HMTD decomposition products and probed the importance of the acid used to catalyze its synthesis. We attempted to identify the composition of its headspace so that realistic dog training aids could be prepared. In Year 4, we have taken a new approach to examining HMTD’s formation mechanism [8]. Understanding the formation mechanisms of various HMEs is essential to discovering ways to block their production. HMTD is formed in front of a Raman laser 785 or 532 nm wavelength. This work has not yet yielded any reportable results. It will continue in Year 5 in addition to our attempts to use isotope-ratio mass spectrometry to examine this problem.
In Years 1 and 2, the physical properties of ETN were examined. Initial work was done to permit MS analysis using a liquid-chromatographic introduction into the MS. In Year 4, the mechanism of its thermal decomposition was thoroughly examined. This work highlighted a hazardous operation that many in the HSE perform. Because ETN melts at 60°C and is visually unchanged, it is mistakenly assumed that this material is safe to handle. Our detailed examination of the stability of ETN revealed that decomposition can occur at melt. This information is just being released [1]. Hopefully, it will caution those in the HSE who have melt-cast this material without rigorous precautions. We have also collaborated with project R1-D.1 to understand ETN decomposition from a theoretical and experimental point of view [1].

In Years 1 and 2, we collected extensive data of thermal properties of FOX mixtures. An enormous study of FOX explosive mixtures was undertaken wherein 11 oxidizers and 13 fuels were examined by differential scanning calorimetry and hot wire initiation. Subsequently, we performed small-scale burn tests (in Year 3) followed by select larger-scale detonations (during Year 4 and continuing) in an initial approach to one of our long-range goals—identifying potential explosivity using only small amounts of material.

Studies of nitrated sugars having more C-OH groups than ETN (mannitol and sorbitol) were initiated in Year 4. We have attempted to synthesize both mannitol and sorbitol hexanitrate by several nitration routes. Under no experimental conditions used were either sugar totally nitrated. Furthermore, while sitting at room temperature, the amount of hexanitrate in the samples decreased relative to the amount of pentanitrate. This information suggests instability, which may be hazardous to members of the HSE working with these materials.

E. Milestones

- A number of HMEs have been examined: ETN, sugar nitrates, FOX explosives, and HMTD.
- Work on FOX resulted in two papers (Years 3-4) and two PhD degrees in Year 3 and three more PhDs in Year 4.
- Examination of ETN reveals surprisingly low thermal stability; it decomposes at an observable rate even at its melting point of 60°C. This finding should warn researchers to demonstrate extreme care when handling it. We have collaborated with Project R1-D.1 to elucidate ETN decomposition kinetics and mechanisms [1].
- Our studies on larger (than ETN) sugar nitrates immediately revealed that complete nitration is extremely difficult to achieve. This is not expected to be a concern to, or even recognized by, terrorists because the incompletely nitrated mixture is likely to be detonable. However, it will provide a forensic signature which can be exploited by the HSE. Year 5 will be devoted to studies of their decomposition and sensitivity.
- To identify limitations or potential of individual FOX mixtures and to attempt to establish general principles for identification of potential explosivity, a survey of the stability and performance of 11 solid oxidizers and 13 fuels was undertaken. Studies in Year 3 included differential scanning calorimetry (DSC), simultaneous differential thermolysis (SDT) (thermal and mass change measured), and hot-wire ignition. Two papers resulted from these studies [9, 10]. In Year 4, we examined FOX mixtures using Parr bomb calorimetry. This technique not only measures heat release, but also the amount and rate of gas production. These same mixtures are being examined in Project R1-B.1 in an attempt to correlate lab-scale tests performed in this project with actual detonability testing. Field trials are expected to continue in Year 5.
- Work on HMTD has focused on determining the first product of its decomposition and improving our limits of detection in laboratory analysis. The amazingly low limit of 25 ng/mL has been achieved by liquid chromatography with mass spectrometric (LC/MS) analysis. This achievement not only aids our own studies on improved swabbing (Project R1-C.2), but the necessary protocols are being distributed via publication to the rest of the HSE community.
• Identifying the extreme volatility of TATP should be of major interest to members of the HSE. Not only does this impact researchers, it impacts those who gather evidence.

• Our newly-launched studies on the possible bio-retention of TATP will point to areas for gathering additional evidence as well as potential safety hazards of which the HSE needs to be aware.

• Our work with ETN and nitrated sugars points out the extreme instability (not necessarily sensitivity) of these materials. In the case of the nitrated sugars, the HSE should be aware that they are unlikely to be working with pure materials.

• Work on FOX explosives has transitioned to Project R1-B.2.

F. Future Plans

Over the years, this project has resulted in well over a dozen papers on HMEs, such as TATP, HMTD, ETN, and other nitrated sugars. This will be a lasting achievement for DHS, as well as the database compendium. There is a myriad of HME-related questions yet to be answered, and each year it is expected that new threats will require in-depth investigation. This project does basic research essential to those in the HSE. However, they may not have the time to pursue this information nor is pursuing it in their job descriptions. The transition for this project is making sure our investigations/information are available in an understandable form to the HSE as well as educating the workforce which will be the future of the HSE. The tasks below are foremost but we are unlikely to finish them in a single year.

• The newly-initiated project on metabolism and bioaccumulation of the peroxide explosives will be continued.

• Laboratory scientists need methods for gentle destruction of HME. We will compile this.

• HMTD formation and transformations will be probed.

• Production of ETN from various starting materials will be examined.

If this project is granted additional time and funding (Year 6), we will examine the threat from azides. Interestingly, both in legitimate industry and in the terrorist community, the use of silver azide risen. To date, characterization of these has been neglected. In addition to updating the explosives database, we will add seminal literature references, as many people use the explosive database as their first resort to learn about materials they are unfamiliar with.

II. RELEVANCE AND TRANSITION

A. Relevance of Research to the DHS Enterprise

Characterization of HME is an ongoing research effort within DHS, including vendors and associated researchers; it impacts the entire HSE. In many cases, our methods of analysis lead the way for other members of the HSE. Our studies on the extreme sensitivity of HMTD to moisture and acidity may have prevented mishandling in a number of laboratories. Many vendors of explosive detection instrumentation have requested access to the explosives database or asked for help in working with various materials characterized in this project. The characterization of these materials is published on our database, which is subscribed to by over 1000 people, 220 of which are from U.S. government agencies and 48 of which are DHS personnel. Furthermore, our work is cited in the DHS HME Safety Protocols Handbook, and we were invited to participate in the DHS Chemical Security Analysis Center & Explosives Division 1st Inter-agency Explosives Terrorism Risk Assessment working group.
B. Potential for Transition

While we are not building detection devices, we provide essential input to those who do, in Government, the security industry and academics. We have worked with numerous companies producing explosive detection instruments. We publish results in the open literature and present at the Trace Explosive Detection (TED) conference annually. Information is also disseminated via regular short courses, and we post results on the URI Explosives Database. A National Institute of Standards & Technology (NIST) senior scientist commented on our database of explosive properties: “It was all we had, in many cases.” This is high praise from the organization that maintains the “Chemistry Webbook.” We have also received such compliments from military labs, both in CONUS (within the U.S.) and OCONUS (without the U.S).

C. Data and/or IP Acquisition Strategy

As data from the program becomes available, it will be provided to the community through DHS, publications, presentations, and the database. We have received requests to license the explosives database.

D. Transition Pathway

Results will primarily be transferred to the user community by publications, presentations and the database.

E. Customer Connections

The connections to DHS (central), the Transportation Security Laboratory (TSL), and the Transportation Security Administration (TSA) are strong. To date, the Federal Bureau of Investigations (FBI) is the major agency outside of DHS that is aware of the details of this project.

IV. PROJECT ACCOMPLISHMENTS AND DOCUMENTATION

A. Education and Workforce Development Activities

1. Course, Seminar, and/or Workshop Development
   a. We have conducted 13 explosives classes on seven different topics ranging from fundamentals of explosives to safety in handling. Over 350 people attended these courses. See details listed in Section IV.H.
   b. There is a new course offering in July 2017 called “Safe Handling of Explosives for Technicians.”
   c. Prof. Oxley has given six invited talks and will be giving two more in Summer 2017.

2. Student Internship, Job, and/or Research Opportunities
   a. We have or will have a number of visitors who come with the express purpose of learning how we handle HMEs and other energetic materials.
      i. We hosted a young man from the Home Office (United Kingdom) for two days in May 2017.
      ii. In July 2017, we hosted two students from the lab of last year’s MSI award recipient (Dr. Bakhtiyarov, New Mexico Tech) for a week.
      iii. Having performed TNT equivalence tests for Advanced Technology Research Centre (ATREC), we were asked to host researchers from ATREC and are invited to send students to their facility in Singapore. No dates have been set for this interaction.
   iv. See also Student Theses in Section IV.G.
3. Interactions and Outreach to K-12, Community College, and/or Minority Serving Institution Students or Faculty
   a. In Summer 2016, we had a visiting professor, Dr. Bakhtiyarov from New Mexico Tech, as well as one graduate and one undergraduate student from his group. He requested to return this summer, but decided to accept a DHS follow-on award instead.
   b. We have done three STEM related magic shows for K-12 students.
   c. In Summer 2016, we initiated a collaborative learning program where high school teachers along with select high school students spent two 2-week sessions solving an analytical chemistry problem.

4. Training to Professionals or Others
   a. See Section IV.H.

5. Other Outcomes that Relate to Educational Improvement or Workforce Development
   a. Three PhD students and two Master’s students graduated this year and are now employed (see Section IV.G. for details).

B. Peer Reviewed Journal Articles


Pending


C. Other Publications

Pending


D. Other Non-ALERT Publications & Conference Proceedings


**E. Other Conference Proceedings**

1. Invited Lecture
   g. Oxley, J.C. “Why Study Explosives?” Wesleyan University, April 29, 2016.

**F. Other Presentations**

1. Seminars - See Section IV.D and E for presentations and invited lectures.
2. Short Courses - Listed under Section IV.H (New and Existing Courses).
3. Interviews and/or News Articles
   f. Brown, Jennings. “Land Mines Kill Thousands A Year, But Can This Drone Help?” *Vocativ*, July 22,
h. Jensen, Alex. *This Morning* (Seoul, South Korea), June 14, 2017, radio interview.

G. **Student Theses or Dissertations Produced from This Project**

1. Swanson, D. “Intermolecular Interactions of Energetic Materials.” PhD dissertation, University of Rhode Island, December 2016. **Devon studied improved swabs, AFM, and co-crystallization of explosives. After receiving his PhD, he took a job with ATK in West Virginia.**

2. Porter, M. “Tools to Fight the Terrorist Threat.” PhD dissertation, University of Rhode Island, May 2017. **Matt studied FOX from small-scale in Project R1-A.1 to large-scale in Project R1-B.2, and also examined HMTD. After receiving his PhD, he took a job with Esterline.**


H. **New and Existing Courses Developed and Student Enrollment**

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I. Technology Transfer/Patents

1. Patent Applications Filed (Including Provisional Patents)
   a. Oxley, J., Smith, J., & Canino, J. "Non-Detonable Explosive or Explosive-Simulant Source" is in the process of being converted from provisional to full patent.

J. Software Developed

1. Databases
   a. The Explosives Properties Database: Over 1000 members are registered for the database, of which 250 are associated with U.S. government agencies. http://expdb.chm.uri.edu/.

K. Requests for Assistance/Advice

1. From DHS
   a. On call for a variety of TSA TSS-E personnel.
   b. Professor Oxley is part of the DHS-formed Inter-Agency Explosive Terrorism Risk Assessment Working Group (IExTRAWG).
   c. Oxley is a member of the NAS committee on “Reducing the Threat of Improvised Explosive Device Attacks by Restricting Access to Chemical Explosive Precursors.” http://dels.nas.edu/Study-In-Progress/Reducing-Threat-Improvised-Explosive/AUTO-7-66-86-I.

2. From Federal/State/Local Government
   a. A TSA explosives specialist emails questions weekly and occasionally calls.
   b. The new URI bomb dog and his trainer rely on our lab for advice and explosives training aids.

V. REFERENCES


[3] Collins, GE; Malito, MP; Tamanaha, CR; Hammond, MH; Giordano, BC; Lubrano, A.L.; Field, C.R.; Rogers,

[4] Hartel, MAC; Klapotke, TM; Stiasny, B; Stierstorfer, J Gas-phase Concentration of TriacetoneTriperoxide (TATP) and Diacetone Diperoxide (DADP); Propellants, Explosives, Pyrotechnics; 2017, 42(6), 623-34; DOI: 10.1002/prep.201700034.


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