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Advanced Energetic Materials for Agent Defeat: Impact-Driven Reactions in Biocidal Reactive Materials for WMD Applications

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Advanced Energetic Materials for Agent Defeat: Impact-Driven Reactions in Biocidal Reactive Materials for Weapons of Mass Destruction (WMD) Applications

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Abstract**—**Dynamic thermite reactions can produce hot iodine gas, which can destroy spore-forming bacteria. Initial experiments have demonstrated kill ratios of up to 0.9999.

1 Objectives

The objectives of the proposed efforts remain as follows: (1) to understand the reaction kinetics of materials that generate biocidal gases, and (2) to understand the way spore-forming bacteria respond when exposed to impact-generated biocidal gases and nanoparticulate metal oxide reaction products.

2 Status

All the apparatus and instrumentation needed for experiments have been developed. All the biological protocols and procedures have been developed. Characterization of combustion-driven iodine pentoxide thermite reactions and silver intermetallic reactions has been completed. It was shown that the spore kill ratios of 0.9999 can be achieved.

3 Accomplishments and Findings

3.1 IAT Effort

The sporicidal effectiveness of iodine pentoxide thermites has been demonstrated in a series of confined burn experiments. *Bacillus subtilis* spores in known quantities were exposed to reaction products under various conditions and for various lengths of times. Kill ratios were determined in subsequent counts of colony-forming units. Under the best conditions, kill fraction was 0.9999. Through use of controls, it was demonstrated that spores were killed by exposure to hot chemicals during the experiments and not by any other process occurring during the exposure and recovery procedures. The relative roles of chemistry and temperature are yet to be resolved. However, cooled deposits from reactions do not possess significant sporicidal effectiveness. Silver intermetallic reactions, on the other hand, were not found to produce sporicidal effects under any conditions.

Time-resolved spectroscopy of iodine pentoxide-aluminum thermite (Figure 1) indicates two events, presumably the thermite reaction followed by a much less intense reaction of iodine with oxygen. Pressure measurements in impact-driven reactions (Figure 2) are consistent with the expected energy release from the iodine pentoxide and neodymium reactants.

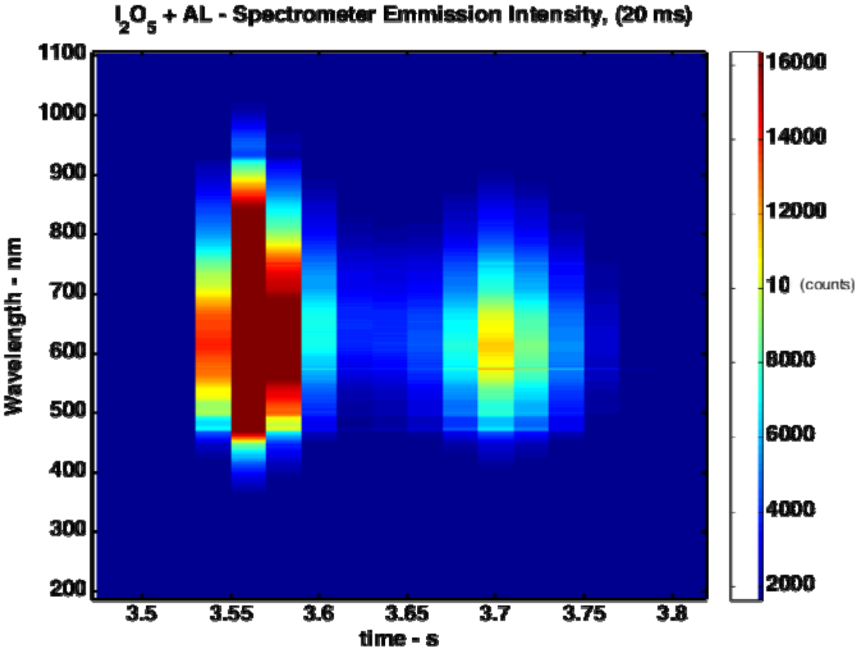

Figure 1. Spectrogram of the iodine pentoxide-aluminum reaction zone.

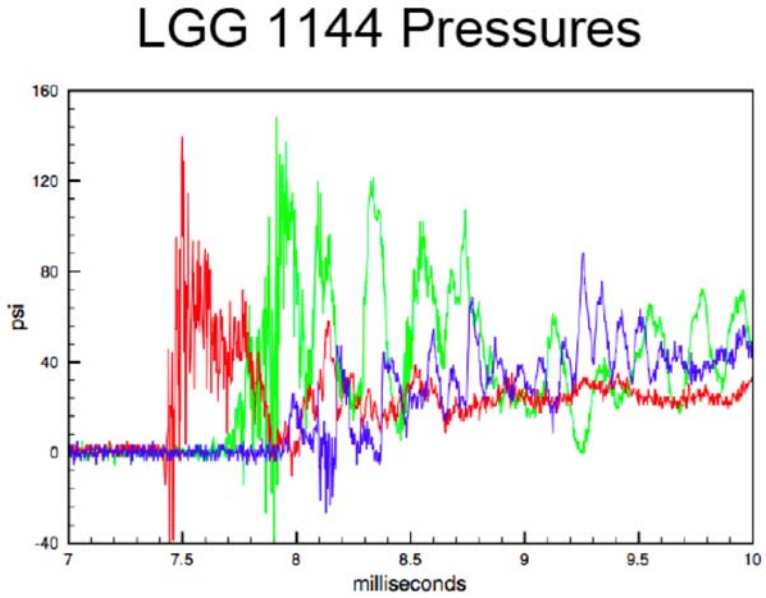

Figure 2. Pressure history: iodine pentoxide + neodymium containing projectile shot through a steel target into a reaction chamber. Vent rates were low enough that the energy release could be computed from pressure data.

Collection and analysis of debris from impact-driven and combustion-driven reactions finds starkly different product morphologies (Figures 3–5). Impact reactions form alumina spheroids and apparently alpha-alumina platelets. Combustion end products are apparently I_2O_4, I_2O_5, and amorphous alumina.

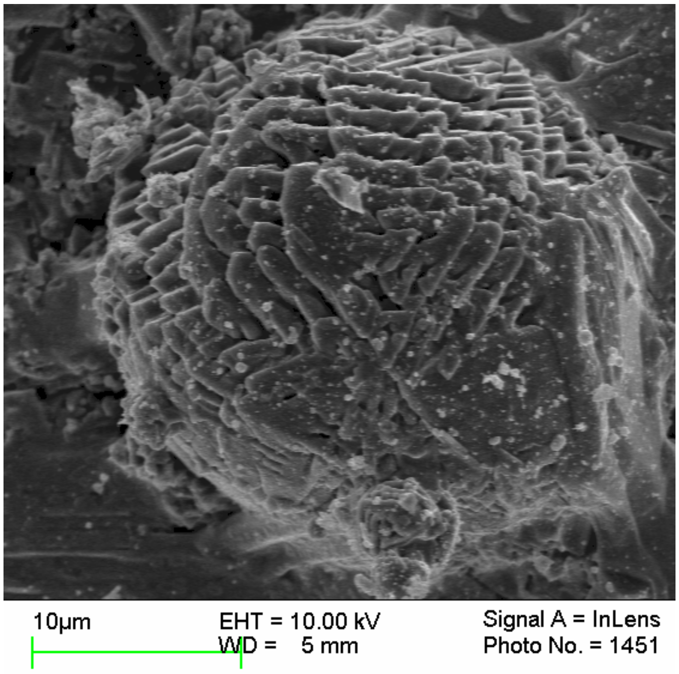

Figure 3. Alumina particle recovered from impact test.

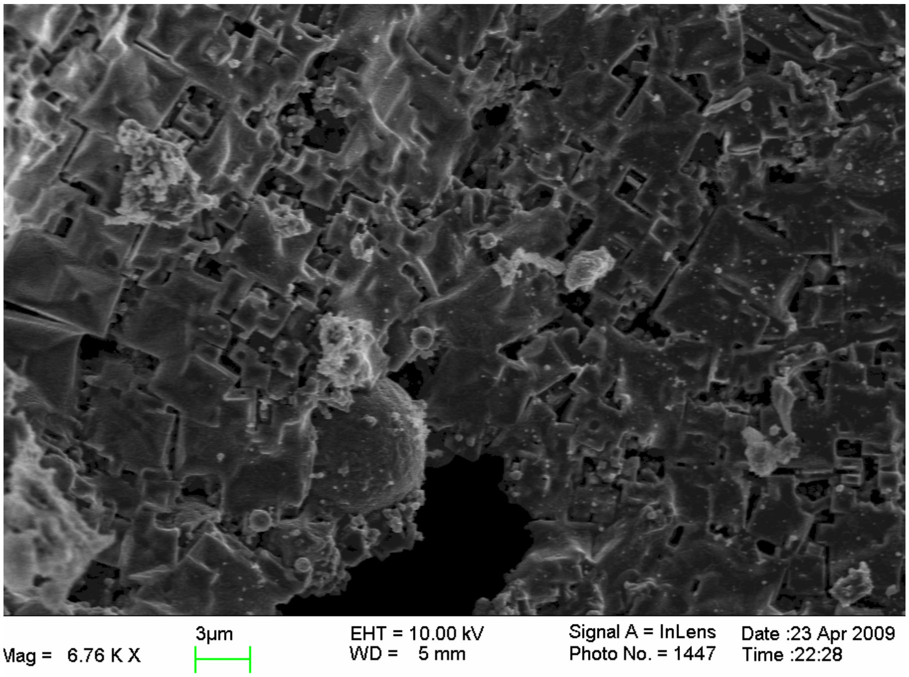

Figure 4. Alpha alumina from impact tests.

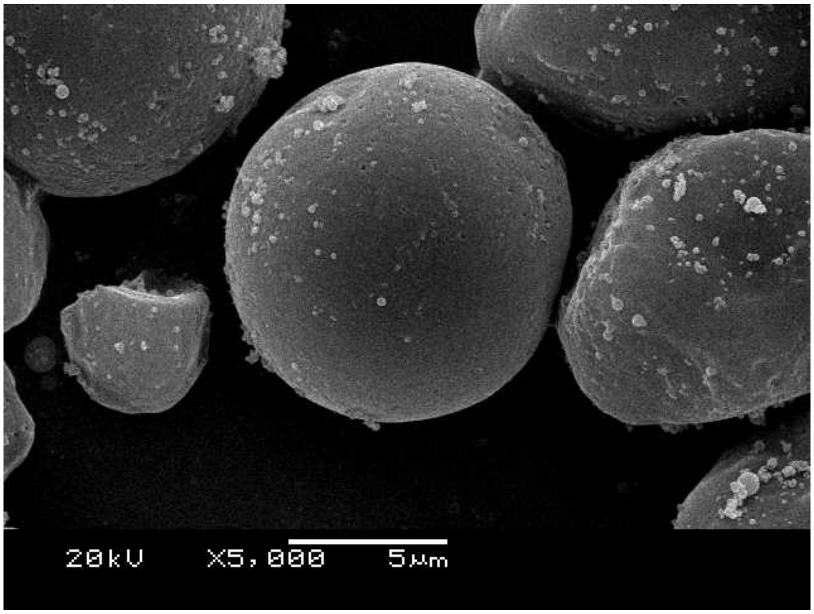

Figure 5. Alumina particles recovered from a combustion experiment.

3.2 Texas Tech University Effort

In order to determine if a chemical kill actually occurs, three thermites with known biocides were compared to a control, nonbiocidal thermite. The possible biocidal oxidizers are iodine pentoxide (I_2O_5), silver oxide (AgO), and silver iodate; the control oxidizer is iron oxide (Fe_2O_3). Each oxidizer was mixed with nano aluminum.

The bacterial substitute used is Bacillus atrophaeus, which is often used as a noninfectious anthrax in testing. The bacteria samples were prepared by transferring 100 µL of the 10^6 spores/µL suspension into plastic test tubes. The aqueous solution was then evaporated out of the tube, leaving the bacteria in the bottom of the tube. These tubes were then placed into holders on the walls of the biocidal reaction chamber (BRC).

Two hundred and fifty mg of thermite was placed in a small petri dish, and the dish was then placed in the bottom of the BRC with the nickel-chromium ignition wires placed in the loose powder. After the reaction was initiated, the bacteria was left in the BRC for an hour of exposure time. The bacteria were then rehydrated, and a pipette was used to transfer the 100 µL of suspension to lysogeny broth (LB) agar plates for incubation. The bacteria were analyzed for growth after 24 and 48 hours; photographs of the agar plates were taken at these intervals as well. The growth rate was then compared to the control sample that was not exposed to the reaction.

The initial kill/no kill tests were performed, and representative data are shown in Figures 6–9. After looking through the photographs of the bacteria growth, the initial findings were that the iodine pentoxide is extremely effective at neutralizing the bacteria after only an hour of exposure time. The silver oxide and iron oxide showed no signs of bacteria neutralization after an hour of exposure time; in fact, the bacteria exposed to the reaction actually grew better than the controls that were not exposed.

Figure 6. Iodine pentoxide + aluminum test five: unexposed control sample after 24 hours of growth.

Figure 7. Iodine pentoxide + aluminum test five, sample 2: exposed for one hour, after 24 hours of growth. This was the only sample of four that showed any signs of growth.

Figure 8. Silver oxide + aluminum test nine: unexposed control sample after 24 hours of growth.

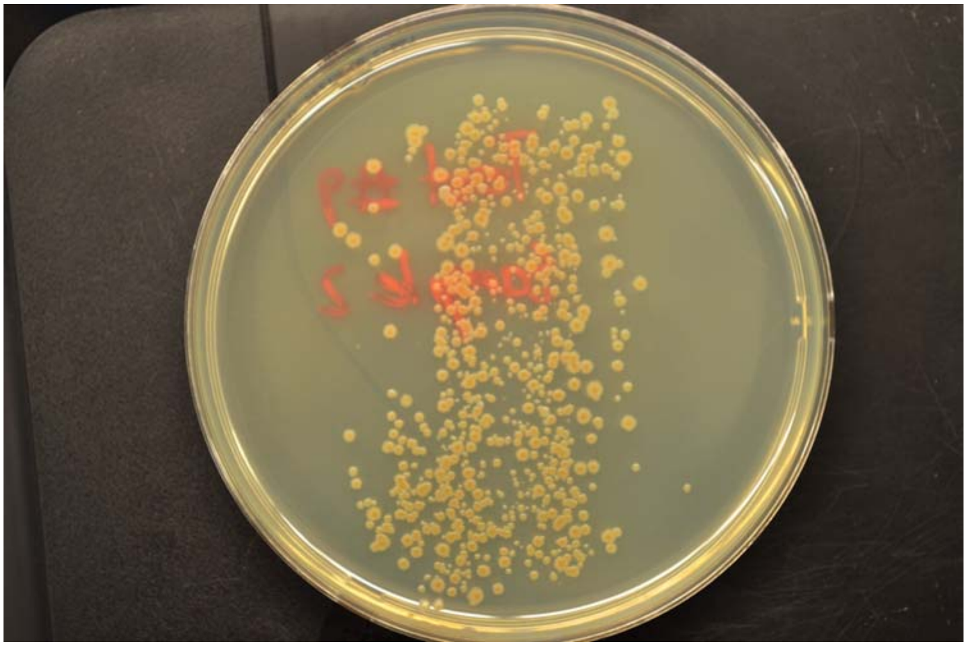

Figure 9. Silver oxide + aluminum test nine, sample 2: exposed for one hour, after 24 hours of growth. All samples showed similar growth rates.

3.2.1 Aluminum-Iodine Pentoxide Kinetics

The aluminum-iodine pentoxide reaction was further analyzed using our differential scanning calorimeter (DSC) and thermogravimentric analyzer (TGA). The DSC component analyzes heat differentials in order to determine if a given reaction gives off or absorbs heat, and the TGA component determines mass changes associated with the reactions. Aluminum and iodine pentoxide were examined individually, as well as in mixtures of varying compositions.

The primary result of this analysis was that the I_2O_5 decomposes into oxygen gas and iodine gas at a relatively low temperature (400^o C). This decomposition means that during a combustion reaction, the aluminum will primarily be reacting with the oxygen gas. The pressure and heat from this reaction will then act as a dispersion mechanism for the iodine gas, allowing it to coat all surfaces within the enclosure. In Figure 10, the endotherm located at 400^o C corresponds to the decomposition of iodine pentoxide.

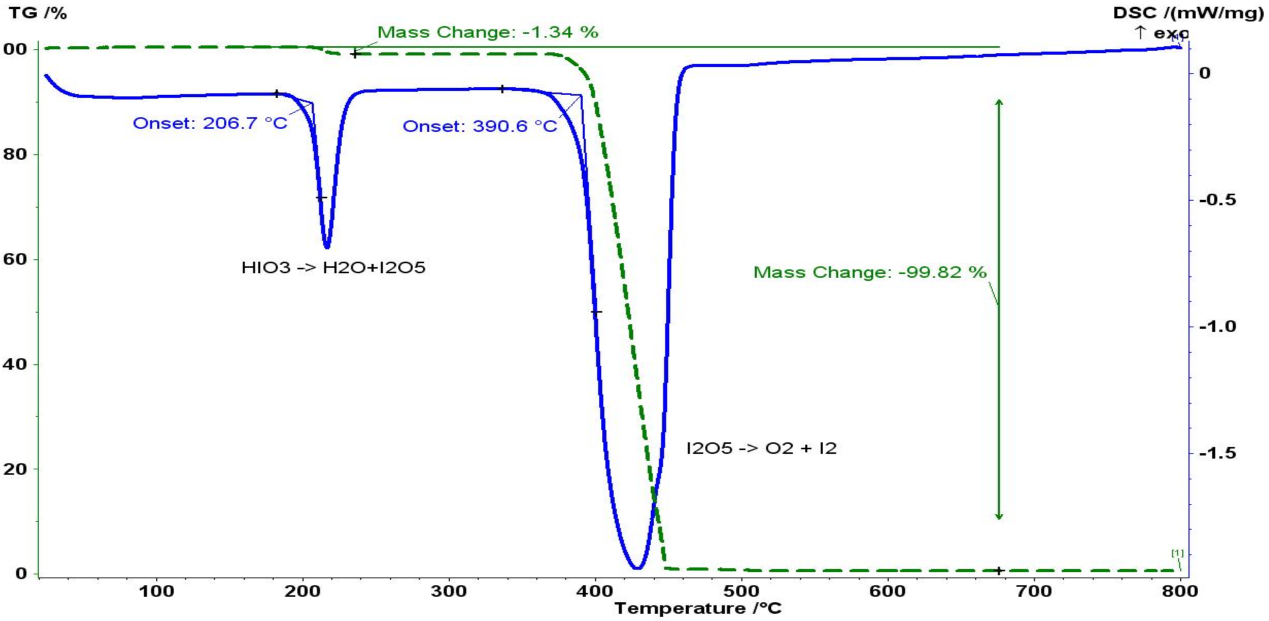

Figure 10. DSC-TGA curve of I_2O_5 in an argon environment. At 200˚ C, iodic acid formed from absorbing atmospheric water decomposes to I_2O_5, and at 400˚ C, I_2O_5 decomposes to iodine and oxygen gas.

Other results of this research are the quantification of the full reaction kinetics between aluminum and iodine pentoxide. Nanometer and micron-scale powders were tested as well as different fuel-to-oxidizer ratios. First, micron powder was found to have no reaction at all in a slow heating environment. In order for micron aluminum to react, the aluminum first has to melt at 660^o C. Under 10^o C per minute heating rates, the oxygen gas produced when I_2O_5 decomposed at 400^o C has 23 minutes to be flushed out of the system by the gases used to protect the sensors within the DSC/TGA. However, nanometer aluminum powder can react in the solid phase at much lower temperatures. A step-by-step analysis of the reactions of aluminum with oxygen gas and the alumina shell and iodine gas was achieved with various experiments. In Figure 11, note the additional exotherms resulting from reaction with aluminum and alumina after the decomposition of I_2O_5. At 200^o C, we have the release of bonded water in the conversion from iodic acid to I_2O_5. At 300^o C, the I_2O_5 begins decomposition and oxygen reacts with the aluminum, while the iodine reacts with the alumina shell. At 550^o C, the iodine is released from the alumina shell and reacts with the aluminum. Finally at 660^{o} C, the unreacted aluminum melts.

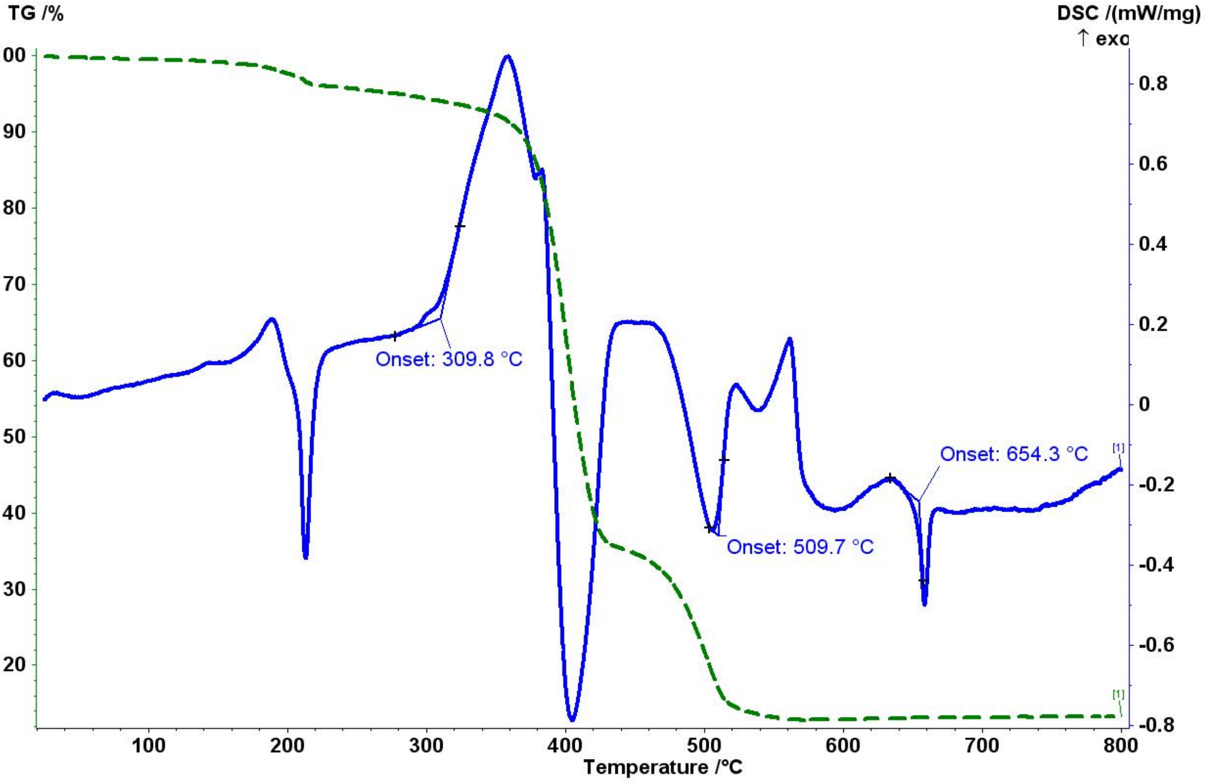

Figure 11. DSC-TGA curve of nano scale Al/I_2O_5 in an argon environment. In addition to the kinetics found in Figure 10, an Al/O_2 reaction causes the exotherm at 390^{o} C, an iodine/alumina reaction causes the step loss of mass at 450^{o} C, and an Al/I_2/O_2 reaction causes the exotherm at 550^{o} C. Note the existence of unreacted Al shown by the endotherm at 660^{o} C, even with a highly fuel-lean mixture ($\varphi = 0.4$).

4 Personnel Supported

At the Institute for Advanced Technology (IAT), this project has supported Dr. Stephan Bless (principal investigator), Mr. Rod Russell (research engineer), Ms. Tiffany Chen (undergraduate student), Dr. Alexandra Blinkova, School of Biological Sciences, The University of Texas at Austin (UT), and technicians in the impact laboratory. At Texas Tech University, the program has supported Dr. Michelle Pantoya (co-principal investigator) and William Clark (graduate student).

5 Publications

There have been no publications during the subject reporting period. There was a presentation at the APS Shock Compression of Condensed Matter Meeting, and a manuscript is in preparation comparing reactions among iodine pentoxide-metal thermite systems.

6 Interactions/Transitions

Because of our success so far in this program and in demonstration tests based on techniques developed in this program, IAT was invited by Energetic Materials and Products, Inc. (EMPI) to provide support for their Phase 3 Defense Threat Reduction Agency (DTRA) Small Business Innovation Research (SBIR) program on defeat of chemical agents.

IAT has been interacting with Prof. Nick Glumac at Purdue University on spectroscopy techniques and with Prof. Karl Christe at the University of Southern California on iodine safety procedures. We have received valuable advice from Prof. James Walker of the UT biology department on design of our experiments. We have an exchange meeting scheduled with Prof. Sergey A. Grishpun at the University of California, Los Angeles, in November.

We have been able to use some devices developed by IAT in support of its university-affiliated research center (UARC) core work on electromagnetic projectile lethality, and likewise that Army program has benefited from access to the safety equipment put in place on the subject contract.

7 Inventions

There were no inventions during the subject reporting period.

8 Honors, Degrees, Etc.

There were no awards or degrees received related to this contract during the subject reporting period.

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