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LLNL-TR-738390

Synthesis of a Novel Energetic Heterocyclic Oxidizer with Higher Energy and Lower Sensitivity (Phase 2) Final Report CRADA No. TC02125.0

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September 12, 2017

Disclaimer

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

Synthesis of a Novel Energetic Heterocyclic Oxidizer with Higher Energy and Lower Sensitivity (Phase 2)

Final Report
CRADA No. TC02125.0
Date Technical Work Ended: December 22, 2008

Date: March 25, 2009 Revision: 3

A. Parties

This project was a relationship between Lawrence Livermore National Laboratory (LLNL) and Physical Sciences Inc. (PSI).

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B. Project Scope

This was a collaborative effort between Lawrence Livermore National Security, LLC as manager and operator of Lawrence Livermore National Laboratory (LLNL), Physical Sciences Inc. (PSI), and Aerojet, Inc. (Aerojet) to develop a new synthesis of a Novel Energetic Heterocyclic Oxidizer with Higher Energy and Lower Sensitivity (Phase 2).

This project was a continuation of work originally performed under a Phase 1 of the Small Business Technology Transfer (STTR). The success of the Phase 1 led to the award of a Phase 2 of the STTR. In Phase 1 of the STTR, the target energetic compound, 3,4-bis(4-nitro-1,2,5-oxadiazol-3yl)-1,2,5-oxadiazole-1-oxide (DNTF), was synthesized at the 5g scale and small-scale safety tests were performed. DNTF showed promising performance and safety properties. DNTF was shown to be relatively insensitive while performing better than the current industry standard, HMX, in solid propellant formulations. Because of the successful research and development project involving PSI, LLNL and Aerojet in Phase 1 of the STTR, the sponsors wanted to obtain

larger quantities of DNTF for further testing. This project to synthesize 100g of DNTF consisted of the following four (4) major tasks and deliverables:

Task 1:
PSI will synthesize 150-200g of 3,4-diaminofurazanfuroxan (DATF)
(Months 1-2). The material will be shipped to LLNL for conversion to DNTF.

Task 2:
LLNL will synthesize 100g of 3,4-dinitrofurazanfuroxan (DNTF) and ship DNTF sample(s) to Aerojet (Months 2-6)

The synthesis of DNTF by LLNL will be performed at the Energetic Materials Center at LLNL. The DNTF will be shipped to Aerojet using approved DOT shipping containers.

Task 3:
Both LLNL and Aerojet will perform small-scale safety and thermal stability tests (Months 5-7). In addition, Aerojet will conduct performance testing at its location.

Task 4:
Management and Reporting (Month 7)
This task covers the documentation and reporting for this CRADA by both PSI and LLNL. The reporting will be done via progress reports, lab notebooks, e-mail, phone calls, a comprehensive final report, and a face-to-face visit at the program's completion. PSI will act as a focal point for gathering and reporting the information necessary for the project's reports to the sponsor.

Deliverables:

1. PSI will synthesize and ship DATF to LLNL - due Month 2.
2. LLNL will synthesize, test and ship DNTF to Aerojet [The DNTF will be shipped to Scott Dawley at the Aerojet address specified in Section D] - due Month 6.
3. Aerojet will test DNTF and report to PSI - due Month 7.
4. LLNL and PSI will write reports for DOE and the sponsor – due Month 7-Month 9.

This CRADA was designated as a nine (9) month project. All tasks and deliverables were successfully completed on time.

C. Technical Accomplishments

The specific technical accomplishments of this project were the synthesis and scale-up of DNTF and its precursors, leading to the production of 100g of purified DNTF. In addition two new compounds, DATF-1 and DNTF-1 were synthesized and characterized. The use of DNTF in high-energy rocket propellants was demonstrated.

D. Expected Economic Impact

The high-energy rocket propellants may be useful for certain DOD applications that require improved performance.

D.1 Specific Benefits

Benefits to DOE

This CRADA benefits DOE by increasing LLNL's expertise in energetic materials. The project yielded two new energetic compounds, DATF-1 and DNTF-1. DATF-1 may have applications as an insensitive compound. New insensitive compounds are of interest to the DOE. In addition the expertise gained in the project by our synthesis chemists will be beneficial to future DOE projects. This project also allowed additional training for a new explosives chemist at LLNL. There is a need in the DOE for training the new scientists.

Benefits to Industry

It was demonstrated in this project that DNTF is a viable candidate for future propellants formulations with enhanced performance. The addition of DNTF to test formulations performed better than the baseline formulations. In the future DNTF may be used in new, higher energy formulations that could lead to increased range, weapon miniaturization or increased Isp. These formulations would surpass the state-of-the art formulations of today.

E. Partner Contribution

PSI delivered on the synthesis of approximately 200g of DATF. They also improved the synthesis of DATF with respect to yield and purity. PSI was instrumental in the development of two new compounds developed under this CRADA, DATF-1 and DNTF-1. Aerojet completed the small-scale propellant testing of DNTF, along with small-scale safety analysis. Their testing demonstrated the potential for DNTF as a high-energy propellant additive. All the deliverables were met and the project was completed to the sponsor satisfaction.

The inventions under this CRADA include the synthesis of two new compounds, DATF-1 and DNTF-1 and the use of DNTF in high-energy rocket propellants. These are described in a record of invention. (See Section F, Subject Inventions.)

The Industrial partners are attempting to obtain funding for a Phase III on this project. The Phase II would identify a commercial partner that would scale-up the synthesis of DNTF to larger quantities for further testing by our industrial partner, Aerojet.

F. Documents/Reference List

Reports

No published reports were generated at this time. Annual reports to the sponsor were completed for the Phase I and Phase II of the CRADA. A patent or journal article may be published in the future.

Copyright Activity

No software was developed under this CRADA.

Subject Inventions

The following joint Subject Invention was disclosed for this CRADA project:

LLNL Case No.: IL-12070 Patent pending

The Industrial Participant, Physical Sciences Inc., has not expressed an interested in licensing this Subject Invention.

Background Intellectual Property

No Background Intellectual Property was disclosed by either party.

G. Acknowledgement

Industrial Participant's signature of the final report indicates the following:

1) The Participant has reviewed the final report and concurs with the statements made therein.
2) The Participant agrees that any modifications or changes from the initial proposal were discussed and agreed to during the term of the project.
3) The Participant certifies that all reports either completed or in process are listed and all subject inventions and the associated intellectual property protection measures generated by his/her respective company and attributable to the project have been disclosed and included in Section E or are included on a list attached to this report.
4) The Participant certifies that if tangible personal property was exchanged during the agreement, all has either been returned to the initial custodian or transferred permanently.
5) The Participant certifies that proprietary information has been returned or destroyed by LLNL.

Ana Racoveanu 04/29/09

Ana Racoveanu — Date
Physical Sciences Inc.

Philip F Pagoria 5/14/09

Philip F. Pagoria, LLNL Principal Investigator — Date
Lawrence Livermore National Laboratory

for Veronica Lopia 5/27/09

Erik J. Stenehjem, Industrial Partnerships Director — Date
Lawrence Livermore National Laboratory

Attachment I – Final Abstract

Synthesis of a Novel Energetic Heterocyclic Oxidizer with Higher Energy and Lower Sensitivity (Phase 2)

Final Abstract (Attachment I)
CRADA No. TC02125.0
Date Technical Work Ended: December 22, 2008

Date: March 16, 2009 Revision: 2

A. Parties

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B. Purpose and Description

The purpose of this CRADA was to synthesize a new oxidizer, DNTF, for use in high-energy propellants and test the material with respect to its sensitivity, performance and stability. A total of 100g of pure DNTF was synthesized and tested in various propellant formulations and compared to known base-line formulations. The addition of DNTF increased the performance over the baseline formulations while keeping the sensitivity level relatively constant. Therefore, DNTF was found to be a viable candidate oxidizer for new high-energy formulations of the future. In this project two new energetic compounds, DATF-1 and DNTF-1, were also synthesized, with structures similar to DNTF. Although not tested in high-energy formulations these two compounds have some interesting properties as energetic compounds. Only small amounts of these new compounds were synthesized. All the deliverables for this project were met.

C. Benefit to Industry

It was demonstrated in this project that DNTF is a viable candidate for future propellants formulations with enhanced performance. The addition of DNTF to test formulations performed better than the baseline formulations. In the future DNTF may be used in new, higher energy formulations that could lead to increased range, weapon miniaturization or increased Isp. These formulations would surpass the state-of-the art formulations of today.

D. Benefit to DOE/LLNL

The project yielded two new energetic compounds, DATF-1 and DNTF-1. DATF-1 may have applications as an insensitive compound. New insensitive compounds are of interest to the DOE. In addition the expertise gained in the project by our synthesis chemists will be beneficial to future DOE projects. This project also allowed additional training for a new explosives chemist at LLNL. There is a need in the DOE for training the new scientists.

E. Project Dates

April 16, 2008 to December 22, 2008.