Pacific Northwest
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PNNL- 30587

Collaborative Research and Development Program on Explosive Detection Technology

FY 2014 through FY 2020

October 2020

Robert G. Ewing
David A. Atkinson

U.S. DEPARTMENT OF ENERGY

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

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Summary

In September 2013, at the 6th Permanent Coordinating Group Meeting between the U.S Department of Energy (DOE) and the French Institut de Radioprotection et de Sureté Nucléaire (IRSN), France expressed an interest in bilateral cooperation with the United States because its newly revised regulations that require enhanced explosives detection capabilities at nuclear and radiological facilities.

In the ensuing years, PNNL (DOE/NNSA) and IRSN sought to identify an area of collaboration within explosives detection that would leverage the specific technical strengths of each organization. Based upon awareness of each other's technical acumen gleaned from the scientific literature on explosives detection, it was clear that specific organizations within each nation could provide the needed expertise to enable enhancement of explosives detection through a collaborative development effort. The French Institut Saint-Louis was determined to be an optimal partner for IRSN to develop a collaboration with DOE/NNSA using PNNL's detection team in this effort. Thus, the dialog was started between the technical experts at each organization to define where complementary expertise in explosives detection could be best leveraged. The technical plans and objectives of this project were sound with promising results. In the end, the joint action sheet was not implemented. The challenge with executing the project was in the complexity of getting a signed agreement between DOE, IRSN and ISL. Most of the obstacles surrounded the ability to protect intellectual property and obtain an agreement which included all of the parties.

At a high level, this report documents the interactions and attempt to develop a cooperative framework for explosives detection development from FY 2014 through FY 2020.

Acronyms and Abbreviations

AFT-MS	Atmospheric Flow Tube-Mass Spectrometry
DOE	U. S. Department of Energy
IRSN	Institut de Radioprotection et de Sureté Nucléaire
ISL	Institut Saint-Louis
NNSA	National Nuclear Security Administration
ORNL	Oak Ridge National Laboratory
PCG	Permanent Coordinating Group Meeting (between DOE and IRSN)
PNNL	Pacific Northwest National Laboratory

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1.0 Introduction

In September 2013, at the 6th Permanent Coordinating Group (PCG) Meeting between the U.S Department of Energy (DOE) and the French Institut de Radioprotection et de Sureté Nucléaire (IRSN), France expressed an interest in bilateral cooperation with the United States in response to newly revised French regulations requiring enhanced explosives detection capabilities at nuclear and radiological facilities.

Explosive detection is a key element of a robust nuclear security enterprise and a critical component of a physical protection system for fixed site nuclear facilities and transportation of nuclear material. Explosives remain a constantly evolving threat, thus it is necessary to continually reassess resources dedicated to detecting different types of explosives. In recent years, research and development related to explosives detection has been primarily focused on enhancing detection capabilities for aviation security. While this area of security is important, there is also a need to enhance explosives detection capabilities for a broader range of security applications including nuclear security. Explosive detection technology has advanced significantly in recent years, and it appeared useful to cultivate IRSN's and NNSA's individual experience and knowledge into a collaborative effort between France and the United States.

At the 6th PCG held in Richland, Washington (September 2013), PNNL provided a demonstration of its recently developednext generation explosives detection technology. This PNNL technology provides enhanced, real-time vapor detection of common explosives under ambient conditions, rather than the typical method of contact based collection of explosive particles with subsequent heating and desorption of the sample.

Through discussions between DOE and IRSN in several PCG meetings, the Institut Saint-Louis (ISL), a French-German research institute, was identified as an optimal collaborator by partnering with IRSN. The Institut Saint-Louis has a long history of explosives research including detection capabilities using novel chemical sensors with micro-electromechanical machines.

Pursuant to Article 2 and consistent with Article 4 of the Agreement between the Department of Energy of the United States of America and the Institut de Radioprotection et de Sûreté Nucléaire of France for Cooperation in Research and Development in the Physical Protection of Nuclear Material and Facilities and in Nuclear Material Safeguards Technologies signed on 20 June 2006 (the Agreement), the United States Department of Energy (DOE), through its National Nuclear Security Administration (NNSA) and the Institut de Radioprotection et de Sûreté Nucléaire (IRSN), intended to work on enhancing Explosive Detection Technology.

The overall objective of this project was to combine the expertise of PNNL and ISL for advancements in the ultra-trace detection of explosive compounds. Specific objectives included:

- Establish collaborations between PNNL/DOE, IRSN and ISL
- Share and develop bibliography on relevant explosives detection topics
- Coordinate and conduct collaborative experimental work on explosive detection
- Capture the progress in a final report and investigate potential further collaborations

2.0 Explosive detection capabilities at PNNL and ISL

Both PNNL and ISL have published expertise in trace explosives detection. The approaches and expertise are somewhat different, but potentially complementary. The combination of the approaches develop by each organization could lead to overall improvements in detection capability, such as in sensitivity and selectivity. PNNL has developed a new ionization source that when interfaced to a mass spectrometer has enabled vapor detection of explosives at parts-per-quadrillion levels. ISL has worked with nanostructured sensors and vapor generators for explosive compounds. Based upon the combined expertise of PNNL and ISL, areas considered for collaboration included:

1) Investigate vapor pressures of new nano-structured explosive mixtures. Specifically, determination if vapor pressures of nanometer sized crystals of two different explosives differ from the vapor pressures of the individual bulk materials.

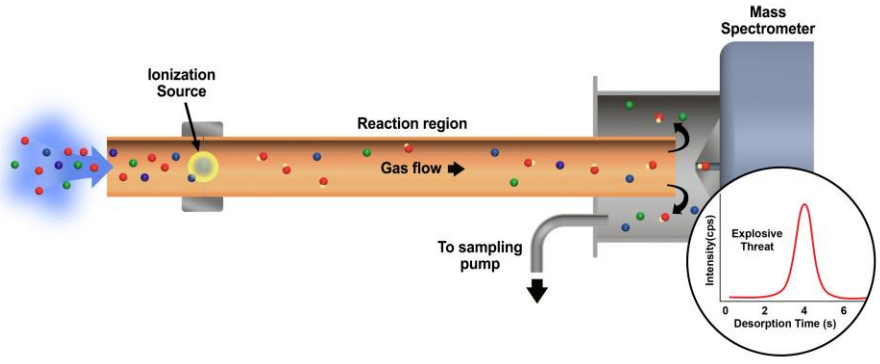
2) Determine ways to improve detection performance and how it could be applied to both facility and transportation security.

3) Explore optimal sample collection and desorption techniques that can be integrated into the vapor detection technology.

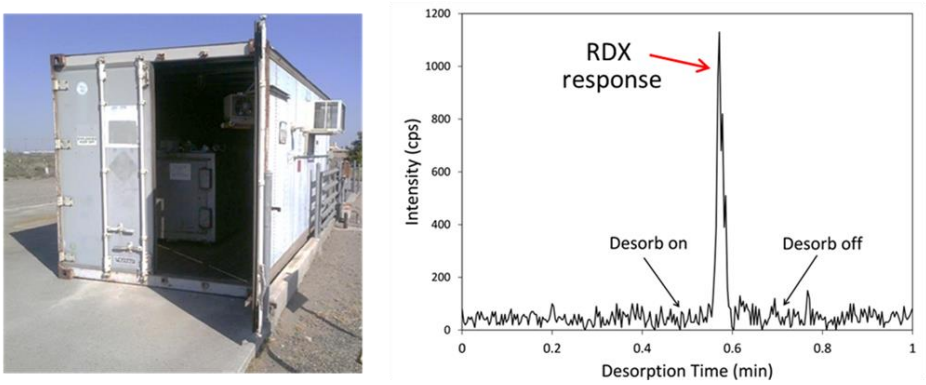
4) Consider comparing ISL micro cantilevers with PNNL vapor detection technology for combined selectivity enhancement.

2.1 PNNL capabilities

PNNL has broad explosives detection expertise that includes chemical-based methods (such as ion mobility spectrometry and mass spectrometry) as well as physical anomaly-based detection systems (such as millimeter wave and x-ray imaging techniques). The sensitive chemical-based detection system with mass spectrometry provides the broadest range of operational capability, as it can be used to detect threats on people, as well as in cargo and other items when operationally configured to do so. PNNL has recently developed a highly sensitive (parts per quadrillion for vapors) and exceedingly selective technology know as Atmospheric Flow Tube-Mass Spectrometry (AFT-MS) for greatly enhanced chemical-based explosives detection. The AFT-MS (shown right) enables the direct, real time vapor detection of low volatility explosives, and was an optimal choice for this joint project. The AFT-MS consists of an ionization source, an extended reaction region and a mass spectrometer.

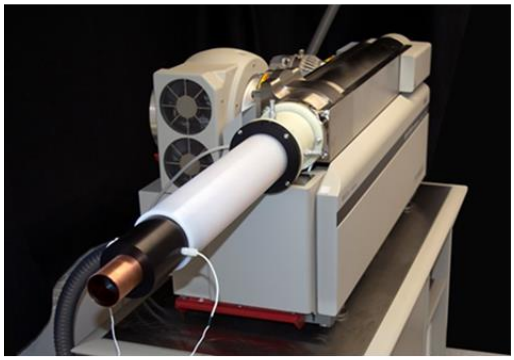


With the low parts-per-quadrillion detection levels, PNNL has demonstrated the detection of RDX vapor in a cargo container after a 2-minute collection time followed by sample desorption (shown below).

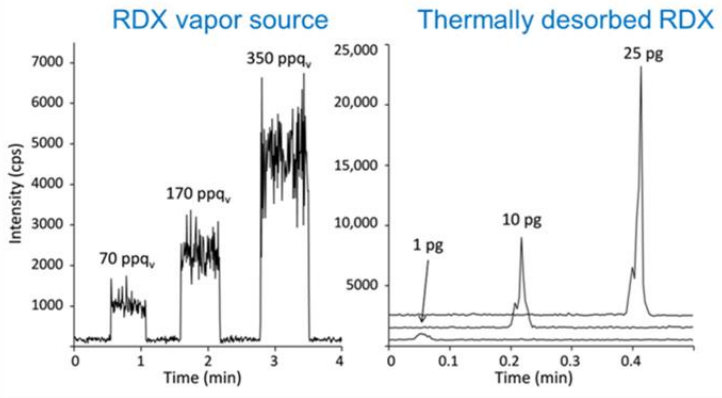


Vapor Detection of RDX in a Cargo Container

Current vapor detection levels are ~ 10 parts per quadrillion for real-time continuous detection and sub-picogram detection levels from collected and desorbed samples. Example RDX detection is shown below.



AFT-MS at PNNL



This new technology, AFT-MS, developed at PNNL has resulted in 3 patents, an R&D 100 Gold Medal award as a market disrupter (2019), and several peer-reviewed publications (4 relevant to explosives detection provided in the following section).

2.2 PNNL references

1) Ewing RG, Atkinson DA, Clowers BH. “Direct Real-Time Detection of RDX Vapors Under Ambient Conditions” *Analytical Chemistry*. 2013; 85(1):389-397

2) Ewing RG, Clowers BH, Atkinson DA. “Direct Real-Time Detection of Vapors from Explosive Compounds” *Analytical Chemistry*. 2013; 85(22):10977-10983

3) R.G. Ewing, A. Heredia-Langner, M. G. Warner (2014) "Optimizing detection of RDX vapors using designed experiments for remote sensing" *Analyst. 2014;* 139(10):2440-2448.

4) R.G. Ewing, B.R. Valenzuela, D.A. Atkinson, E.D. Wilcox Freeburg (2018) “Detection of Inorganic Salt based Home Made Explosives (HME) by atmospheric flow tube – mass spectrometry (AFT-MS)” *Analytical Chemistry, 90 (13), pp 8086–8092*

2.3 ISL capabilities

The Institut Saint-Louis has developed assembled chemical detectors made from micro-electromechanical machines for vapor detection, provided characterization of explosives traces by the nanocalorimetry and used nanostructured sensors for the detection of ultralow concentrations of Explosives. ISL has also developed a tool for generating trace vapor levels of energetic compounds.

2.4 ISL references

1) Spitzer D, Cottineau T, Piazzon N, Josset S, Schnell F, Pronkin SN, Savinova ER, Keller V. “Bio-inspired nanostructured sensor for the detection of ultralow concentrations of explosives” *Angewandte Chemie International Edition*. 2012 May 29;51(22):5334-8.

2) Piazzon N, Rosenthal M, Bondar A, Spitzer D, Ivanov DA. “Characterization of explosives traces by the nanocalorimetry” *Journal of Physics and Chemistry of Solids*. 2010 Feb 1;71(2):114-8.

3) Comet M, Siegert B, Pichot V, Gibot P, Spitzer D. “Preparation of explosive nanoparticles in a porous chromium (III) oxide matrix: a first attempt to control the reactivity of explosives” *Nanotechnology*. 2008 Jun 3;19(28):285716.

4) Bonnot K, Bernhardt P, Hassler D, Baras C, Comet M, Keller V, Spitzer D. “Tunable generation and adsorption of energetic compounds in the vapor phase at trace levels: a tool for testing and developing sensitive and selective substrates for explosive detection” *Analytical chemistry*. 2010 Apr 15;82(8):3389-93.

5) Comet M, Martin C, Schnell F, Spitzer D. “Energetic Nanoparticles and Nanomaterials for Future Defense Applications”. *Human Factors and Mechanical Engineering for Defense and Safety*. 2019 Mar 1;3(1):1.

3.0 Timeline

Most of this project focused around development and discussion of joint objectives, sharing information, drafting an action sheet and setting up collaborations with ISL. These interactions occurred via in-person meetings followed up with emails and conference calls. A majority of these discussions were part of the PCG meetings that occurred annually. Progress was tracked through meeting minutes and action items. Collaborative work between DOE and IRSN outlined in signed action sheets. For this work the draft action sheet, PP04 "Collaborative Research and Development Program on Explosive Detection Technology", was created. Table 1 (below) provides a timeline of the meetings between the United States and France where the joint project on explosives detection was discussed. This table lists the meetings, dates, locations and brief purposes. Following the Table 1, a short description of each event is provided.

Table 1. Timeline of Interactions between the United States and France on the topic of Explosives Detection

Event	Date	Location	Purpose
6th PCG Meeting	September 2013	PNNL, Richland, Washington	Laboratory demonstration of PNNL explosive vapor detection technology
7th PCG Meeting	September 24-25, 2014	IRSN, Fontenay aux Roses, France	Discuss joint work on explosives detection
8th PCG Meeting	September 2017	SNL, Albuquerque, NM	PNNL did not attend. Some discussion on explosives detection occurred
9th PCG Meeting	September 15-16, 2016	IRSN, Fontenay-aux-Roses, France	Draft action sheet prepared with collaboration ideas to discuss during visit to ISL on Sept. 19.
Visit to ISL	September 19, 2016	ISL, Saint-Louis, France	Visited Institut Saint-Louis
10th PCG Meeting	August 29-30, 2017	DOE/NNSA, Washington D.C., USA	Provided update on interactions with ISL and detection technology
Meeting with IRSN and ISL	May 24-25, 2018	IRSN, Fontenay-aux-Roses, France	Discussed proposed PNNL and ISL collaboration ideas
11th PCG Meeting	September 10-12, 2018	IRSN, Fontenay-aux-Roses, France	Action Sheet PPO4 signed by United States (DOE/NNSA)
Meeting with IRSN and ISL	April 25-26, 2019	IRSN, Fontenay-aux-Roses, France	Discuss draft action sheet PP04

12th PCG Meeting	September 4-6, 2019	ORNL, Oakridge Tennessee	Discussed challenges of collaboration with ISL

DOE = U. S. Department of Energy; IRSN = Institut de Radioprotection et de Sureté Nucléaire; ISL = Institut Saint-Louis; NNSA =National Nuclear Security Administration; PCG = Permanent Coordinating Group Meeting; PNNL = Pacific Northwest National Laboratory, ORNL = Oak Ridge National Laboratory

3.1 6th Annual PCG Meeting between the United States (DOE-NNSA) and France (IRSN)

During the 6th PCG meeting in 2013, IRSN expressed interest in bilateral cooperation with the U.S. on explosives detection and agreed to contact the Institut Saint-Louis as the lead laboratory of the research in this area in France. Further clarification on the needs and objectives of cooperation in this area by IRSN and/or Institut Saint-Louis was requested by DOE/NNSA.

PNNL provided a laboratory demonstration of the explosive vapor detection technology.

3.2 7th Annual PCG Meeting between the United States (DOE-NNSA) and France (IRSN)

Robert Ewing provided an overview of PNNL's detection capabilities using vapor pressure and Atmospheric Flow Tube – Mass Spectrometry (AFT-MS). AFT-MS can do simultaneous monitoring of PETN, NG and RDX. Recent work has focused on explosives detection in cargo containers. PNNL noted that two new reports were published since the 6th PCG and current efforts have involved working to reduce the size of the AFT-MS detector.

IRSN is still in the process of trying to identify the right POC at Institut Saint Louis. While a collaborative effort between IRSN and DOE/NNSA is unlikely in 2015 due to timing and because IRSN does not currently have explosives expertise, cooperation with Institut Saint-Louis might be possible to facilitate a project. IRSN agreed to contact the Institut to define capabilities and needs. Once the objective of an explosives detection effort is clarified, IRSN will facilitate contact between DOE/NNSA, PNNL, and Institut Saint-Louis to potentially develop the scope of a project and an Action Sheet.

3.3 8th Annual PCG Meeting between the United States (DOE-NNSA) and France (IRSN)

PNNL did not attend the 8th PCG in 2015, however the explosives collaboration project was discussed. IRSN indicated it had been in touch with ISL and that they are interested in this type of cooperation, though approval would be required from both the French and German sides.

DOE/NNSA has a separate intergovernmental agreement with Germany on physical protection and nuclear safeguards that could potentially help facilitate a trilateral effort.

3.4 9th Annual PCG Meeting between the United States (DOE-NNSA) and France (IRSN)

At the 9th PCG in 2016 held at IRSN, Fontenay-aux-Roses, France, PNNL provided updates on their development of the explosive vapor detection technology. Further collaborations with ISL were discussed.

3.5 Visit to Institut Saint-Louis

Following the PCG representative from NNSA, PNNL, SNL and IRSN visited with staff at ISL held at Saint-Louis, France. Presentation were given by ISL and PNNL. Following the presentation, a tour of the facilities at ISL was provided. At this meeting a tentative schedule for collaborations, listed in Table 2, was developed. This schedule was pending authorization by a signed action sheet.

Table 2. Draft Action Sheet Tasks

Participant	Task Description	Complete by
NNSA/ DOE/PNNL, IRSN/ISL	Task 1.1: Organization of an ISL-PNNL bilateral meeting the United States (at PNNL)	February 2017
NNSA/ DOE/PNNL, IRSN/ISL	Task 1.2: Organization of an ISL-PNNL bilateral meeting in France (at ISL)	May 2017
NNSA/ DOE/PNNL, IRSN/ISL	Task 2.0: Share/develop bibliography	December 2016
NNSA/ DOE/PNNL, IRSN/ISL	Task 3.0 Coordinate experimental work and collaboration	June 2017
NNSA/ DOE/PNNL, IRSN/ISL	Task 4.0 Final report	September 2017

3.6 10th Annual PCG Meeting between the United States (DOE-NNSA) and France (IRSN)

At this meeting, Draft Action Sheet PPO4 was discussed. Robert Ewing led this discussion, beginning with the history and background of the project. It began at the 2013 PCG meeting in Richland, Washington, around the time of the development of the technology to detect explosive vapors at PNNL. France had done similar work in explosives detection at Institut Saint-Louis (ISL). In the previous year, DOE

representatives visited Denis Spitzer's laboratory at ISL, and started the integration process.

Pierre Funk from IRSN explained that he thought it would have taken less time for IRSN to finalize feedback from ISL, but ISL was unexpectedly busy. He had asked about whether there was interest on the part of ISL and received a strong affirmative response. At last contact, ISL was in the review process. He expected feedback in mid-September, anticipating that it would not differ much from expectations. If IRSN is satisfied with the content upon receipt, then the signature process can begin. From the French perspective, IRSN would be happy to move forward, but ISL is a joint French-German institution, so handling the signature process may not be straightforward.

3.7 Meeting with IRSN and ISL at Fontenay aux Roses, France

On May 25, 2018, DOE (NNSA, PNNL and SNL) representatives meet with Representatives of the Institute of Radiological and Nuclear Security (IRSN) and ISL to discuss action sheet PP04, advanced application of explosive detection technology in Nuclear fixed site and transport security. Topics in the action sheet were finalized in preparation for signatures.

3.8 11th Annual PCG Meeting

At the 11th PCG, Action Sheet PPO4 was signed by United States (DOE/NNSA) and delivered to IRSN for signatures. An ISL technical representative (Guillaume Thomas) attended this meeting and contributed to the technical discussions with PNNL and IRSN. The action sheet, PP04 was also discussed.

3.9 Meeting with IRSN and ISL at Fontenay aux Roses, France

On April 25-26, 2019, NNSA/SNL/PNNL (M. Snell, J. Pelletier, R. Ewing) met with ISL (L. Shlur) and IRSN (F. Braina, O. Fichot, C. Mano, P. Funk). Draft action sheet PP04 was reviewed with discussions on adding ISL to the action sheet. Technical updates were provided by both PNNL and ISL and the next steps were discussed.

3.10 12th Annual PCG Meeting

At the 12th PCG, IRSN agreed to check with ISL and let ISL know that the 12th PCG contingent acknowledges the difficulty involved in signing the action sheet due to proprietary items. ISL was invited to propose some alternatives that will allow them to sign an agreement. IRSN will also investigate other potential technical partners in France.

4.0 Conclusions and Opportunities

The technical plans and objectives of this project were sound with promising results. In the end, the joint action sheet was not implemented. The challenge with executing the project was in the complexity of getting a signed agreement between DOE, IRSN and ISL. Most of the obstacles surrounded the ability to protect intellectual property and obtain an agreement which included all of the parties. The obstacles encountered involving the handling of proprietary items and intellectual property were unable to be resolved. This issue prevented the development of a collaborative technical project between DOE/NNSA, PNNL, IRSN and ISL related to explosives detection.

PNNL will continue the development of vapor detection with other sponsors. If an appropriate collaboration becomes feasible in the future joint explosives detection development can be readdressed between PNNL and IRSN.

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