Modernization and Capabilities of the Lawrence Livermore National Laboratory Pilot Facility for Remotely Controlled Energetic Materials Synthesis

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LLNL Energetic Materials Synthesis Group

- Six PhD synthesis chemists with primary residence in the LLNL High Explosives Applications Facility (HEAF, Site 200)
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- **Focus**: Synthesis of new insensitive high explosives (IHE) and conventional high explosives (CHE)

- Synthesis group supports:
  - Basic explosives R&D
  - Stockpile stewardship
  - Counterterrorism
  - Development of HE for military and commercial use
LLNL HE Synthesis Developmental Process

- Weapon systems
- Material qualification
- Performance tests
- Safety characterization
- Modeling
- Formulations
- Materials

Decades

Propose Compounds

- HE Synthesis
- Chemists and Modelers

Performance Characterization

Scale-up

Small-Scale Safety Tests

Performance Predictions

Synthesis

Measured Properties

Density & Heat of formation

Site 300 RC1e Reaction
Calorimeter and Pilot Scale Facility
**LLNL HE Synthesis Developmental Process**

- **Small-scale R&D:** Aim to provide enough information on new HE to “put it on the shelf” and make it available as needs arise from customers.
  - Safety testing: Friction, spark, impact, thermal stability.
  - Heat of formation, density and small scale performance

- **Down-select and scale-up:** Perform small scale reaction calorimetry and synthesize multi-gram quantities.
  - Formulation and pressing studies, ODTX, detonation calorimetry

- **Pilot Scale Process:** Full safety analysis of a synthetic process before pilot scale synthesis conducted. Aim to make 1-2 kg scale before transitioning process outside LLNL.

LLNL working toward enhancing efficiency of new material qualification.
LLNL and Site 300 HE Synthesis Capability

- **HEAF synthesis capabilities:** 1 kg, typically < 95 g.
  - Small-scale safety testing, calorimetry, and performance testing.

- **Livermore’s Experimental Test Site, Site 300 (S300):**
  Provides pilot scale synthesis and formulation capabilities for several kilogram quantities of HE (827D).

- **S300 synthesis capabilities have not been utilized since 2013.**

HEAF Bench Scale
(A) Round bottom flask, (B) Automated synthesis work station, (C) Modular continuous flow reactor
Need for Pilot Scale HE Synthesis at LLNL

- Viable new HE materials must have a scalable and cost effective synthetic and purification process.
- Determination of viability requires significant material for testing.
  - *Sub-100 g quantities*: initial handling safety, chemical and physical properties, and small-scale performance testing.
  - *Multi-kilo quantities*: required for formulation, pressing, and large scale performance and safety studies.
- Design and planning for the modernization and renovation of 827D began in early 2016 with subcontractor Hart Design Group (Cumberland, Rhode Island).
- **LLNL HE chemists strive to transition a fully scalable synthesis and purification process for new materials.**

LLNL is dedicated to R&D and is not a production facility.
San Francisco Bay area and Site 300

- Napa
- San José

Google Maps
Site 300: 7,000 acres rural foothills
HE Synthesis: 827D First Floor Layout and Former Utilization of Space

Class I/Div I HE Work Area
HE Handling

Mechanical Room
Two Floors

Storage

Class I/Div I HE Work Area
Pilot Reactors

827A Control Room about 150 yards away
Former Glass Pilot Scale Reactors

Out of commission since 2013 and demoed in 2015.
Pilot Facility Operations in Recent Past

- Pilot operations were hands on, contact operations: “bucket” chemistry.
- Heavily reliant on operator monitoring of processes and manual transfers of hazardous chemicals.
- Capability for remote operations were very limited.

Safety driven renovation and modernization began in 2015.
Modernization Project Focused on Remote Capabilities and an Agile Facility

- As an R&D facility, flexibility in synthesis is required.
  - Very difficult (and expensive) to meet the demands of all chemistry in one system.

- Desire to convert hands-on processes to majority remote processes adds difficulty and cost.
  - Specialized custom software required.
  - In-situ reaction and process monitoring required.

- A completely remote system is extremely cost-prohibitive and not necessary for all steps in a process.

- Agility can be added by having modular reactors and separate scalable capabilities: continuous flow processes, high pressure reactors, and pilot vessels of varied materials of construction.
Recipe for an Agile Pilot Facility

- 8 chemical processes were considered that encompass the entire range of anticipated synthesis schemes to develop the list below:

**Pilot Reactors**
- 50L Pfaudler w/sample loop (future)
- 100L Pfaudler w/sample loop
- 200L Pfaudler

**Process Analytical Technology (PAT)**
- Raman spectrometer
- pH Meter
- Canty particle sizer
- Reaction monitoring camera

**Ancillary Equipment**
- Solids addition conveyor
- Molten addition funnel
- Remote liquid reagent addition
  - Air operated diaphragm pumps
  - Piston metering pump
- Ammonia gas cabinet
- Nutsche filter
- Bag filter
- Tantalum condensers
- RC1e reaction calorimeter

Fully integrated industrial control software.
Renovated 827D First Floor Layout

- HE Powder Processing
- Remote Reagent Delivery
- Pilot Skid
- Contact Synthesis Area
- Mechanical Room
Pilot Skids, Reagent Delivery, and General Purpose Scale-up area
3D Model of Pilot Reactor Skid

Area for Nutsche filter cart
Constructed Pilot Skid

Pilot Skids at Fabrication: Hart, Cumberland, RI

Pilot Skid Critical Lift and Installation: B827D, Site 300
Typical Synthetic Process in Pilot Skid: Nitration

1. Load liquid and solid reagents

Contact operation conducted in a fume hood.
Typical Synthetic Process in Pilot Skid: Nitration

1. Load liquid and solid reagents
2. Charge solvent (sulfuric acid)

Remote operation conducted from any of the HMI stations.
Typical Synthetic Process in Pilot Skid: Nitration

1. Load liquid and solid reagents
2. Charge solvent (sulfuric acid)
3. Dose/dissolve starting material.

Remote operation from HMI.
Typical Synthetic Process in Pilot Skid: Nitration

1. Load liquid and solid reagents
2. Charge solvent (sulfuric acid)
3. Dose/dissolve starting material.
4. Dose nitric acid.

Remote operation from Control Room.
Typical Synthetic Process in Pilot Skid: Nitration

1. Load liquid and solid reagents
2. Charge solvent (sulfuric acid)
3. Dose/dissolve starting material.
4. Dose nitric acid.
5. Quench Reaction

Remote operation from control room.

Class I Div 1 Boundary
Typical Synthetic Process in Pilot Skid: Nitration

1. Load liquid and solid reagents
2. Charge solvent (sulfuric acid)
3. Dose/dissolve starting material.
4. Dose nitric acid.
5. Quench Reaction
6. Filter Reaction

Remote and potentially contact from HMI.
Typical Synthetic Process in Pilot Skid: Nitration

1. Load liquid and solid reagents
2. Charge solvent (sulfuric acid)
3. Dose/dissolve starting material.
4. Dose nitric acid.
5. Quench Reaction
6. Filter Reaction
7. Collect Product.

Contact operation

Remove HE to process

Class I Div 1 Boundary
Typical Synthetic Process in Pilot Skid: Nitration

1. Load liquid and solid reagents
2. Charge solvent (sulfuric acid)
3. Dose/dissolve starting material.
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5. Quench Reaction
6. Filter Reaction
7. Collect Product.

Timing of valve sequence actuation, alarm parameters, emergency stops, and PID control managed through integrated control system (ICS)
Industrial Control System (CS)

- State of the art Modicon system for remote operation
  - CS infrastructure will be expandable to future projects.

- Controls System Integration (CSI) Subcontract (Avanceon, Exton, PA)
  - Offers engineering controls while delivering batch process flexibility and data collection capability using Schneider Electric’s Wonderware software platform.
  - COTS hardware & software with industry standards-based configurable batch control software (Wonderware InBatch)

Modern, safe, capable, remotely operable controls
Control System Summary

- The CS has been developed by LLNL and Avanceon (Exton, PA)
- 1. 1180+ Alarms
- 2. 800+ I/O Points
- 3. 100+ Control Valves
- 4. 160 Phases
- 5. 111+ Interlocks
- 6. 415+ Page Functional Specification

- There are three operator interface terminals (OIT) for the pilot plant:
  - 827A – Control Room
  - 827D Chemical Addition Room
  - 827D Pilot Skid

Special system designed to prevent control room operation while operator is in the pilot facility.
Capabilities of Integrated Control Software

- Pre-coded operational functions built into custom Wonderware InBatch software (phases).
  - *For example:* Purging the vessels with inert gas will not require the operator to manually open all valves in sequence. The system will open all appropriate valves on a command to “purge” a vessel.
  - InBatch provides a traceable, reliable, and structured “recipe” based process for running a synthetic process from start to finish. The capability to exit the recipe and proceed manually is still available.

- In situ vessel cameras and Raman spectrometer allow for real time visualization of the physical and chemical reaction processes.
  - Ability to tie a spectral result to a control system response is possible. For example, add more reagent until a spectral peak disappears.

- Custom alarms set on instruments (pressure, temperature, etc.) that elicit varied levels of response.
  - Exceed desired process temperature can stop a dose, or initiate maximum cooling of the TCM depending on a High or High-High alarm.
P&ID for 100 L Glass Lined Reactor
Represented not as an Operator Friendly representation.
Control Screen for 100 L Glass Lined Reactor

Operator friendly and fully interactive screens developed by Avanceon.
Summary of Pilot Skid Capabilities

- New facility provides a safe remotely controlled skid for a variety of synthetic processes:
  - Rated for operating pressures of up to 135 psi.
  - MoC: glass-lined carbon steel vessels, hastelloy, tantalum, and PTFE for wetted materials.
  - Reaction temperatures of -10 to 150 ºC.
  - Ammonia charging capability (vaporizer) for both 100L and 200L vessels.
  - Hydrogen peroxide dosing.
  - Metered solids addition via conveyor.
  - Remote reagent addition via metering pump, diaphragm pump, vacuum, or pressure.
  - Ability to phase separate, distill, and recirculate between vessels.
  - Sample loop on 100L vessel for three inputs: currently Raman and pH.
  - Ability to conduct CIP with InBatch automated recipe.
  - Nutche and bag filtration.
Additional Pilot Facility Capabilities

- General purpose electrical area housing RC1e calorimeter.
- Space for future general purpose equipment: wish lists include continuous flow reactor, 5 gallon Parr reactor, and Buchi glass plants.
  - General purpose electrical room rating allows for HE synthesis and processing in fume hoods utilizing non-rated commercial equipment.
- Identically sized room on opposite side of building from pilot skid houses fume hood for powder handling and drying oven.
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