LAWRENCE
LIVERMORE
NATIONAL
LABORATORY

LLNL-CONF-864170

Development of a Pilot Plant for High Explosives Synthesis

S. Hurrey, N. Zuckerman, A. Cielinski

May 8, 2024

2024 IPS Seminar
Albuquerque, NM, United States
July 14, 2024 through July 18, 2024

Disclaimer

Development of a Pilot Plant for HE Synthesis

Suzanne Hurrey*, Nate Zuckerman, and Alec Cielinski
Energetic Materials Center
Lawrence Livermore National Laboratory
hurrey1@llnl.gov

Introduction

Lawrence Livermore National Laboratory (LLNL) has been developing an agile Pilot Plant for High Explosive (HE) Synthesis to complement and inform scale-up from in-house small scale lab synthesis and transition to larger scale production in support of LLNL's stockpile stewardship mission. This paper will focus on the development of this intermediate scale synthesis process with full modern processes and automation.

Background

LLNL's Energetic Materials Center (EMC) specializes in the modeling and experimentation surrounding the development, characterization, effectiveness, and surety of high-explosive materials. To improve the safety, performance, and understanding of energetic materials and investigate advanced manufacturing techniques and new materials development, EMC explores the energy released during energetic chemical reactions, the mechanical response of those materials, and their long-term aging and chemical compatibility.

The LLNL site provides flexibility and resources for research within the NNSA Complex. High Explosives (HE) synthesis capability on a pilot plant scale is a core competency needed at LLNL to enable a more responsive NNSA Complex through scientific understanding that promotes innovation, informs predictive modeling, and reduces development time.

The transition from laboratory scale to production scale while keeping the same quality and throughput can be difficult. Viable new HE materials must have a scalable and cost effective synthetic and purification process, and determination of viability requires significant material for testing. Sub-100 g quantities are required for initial handling safety, chemical and physical properties, and small-scale performance testing. Multi-kilo quantities are needed for formulation, pressing, and large-scale performance and safety studies.

Pilot Plant Design

In 2015, LLNL began designing a new Pilot Plant as a safety driven effort to modernize synthesis and away from manual/contact operations in 50-year-old glass vessels with little instrumentation or process control. The vision for the new Pilot Plant was that it would be a state-of-the-art, safety-focused agile facility with capabilities to allow for Research and Development (R&D) for a variety of HE molecules. Due to the hazardous nature of the chemicals and reactions, the capability to be largely operated remotely to provide maximum safety protections was required. Dedicated to R&D, it will allow synthesis at the 1-2 kg scale, with additional small-scale reaction capabilities initially included in the design. Scope included provisions for future equipment to ensure the design and utilities would be able to accommodate their installation, allowing for new capabilities to be added in order to accommodate future innovations with minimal impact.

As the Pilot Plant provides a unique capability within LLNL, LLNL partnered with a full service outside engineering firm, Hart Design Group, who has a demonstrated record with similar systems in industry, to aid in the design of the new chemical process. The design was an iterative process of refining the lab's vision of the Pilot Plant capabilities into equipment specifications. Industry and DOE standards for process and equipment design had to be met for pressure safety, electrical design, etc.

Equipment Functional Requirements

Eight chemical processes were considered initially in the Pilot Plant design to encompass the entire range of anticipated synthesis schemes, including nitration, amination, and oxidation. To provide agility, the design provided modular equipment that could serve multiple purposes and cover the ranges of compatibility and process conditions. Where design elements could not be compromised, various options were provided, such as equipment of different sizes (reactors of different volumes), different capabilities (pumps that provide low or high flow rates), and different materials of construction (glass-lined, Hastelloy C, tantalum, and PTFE). These modular components are interconnected by manifolds equipped with flexible hoses to allow the configuration to be changed easily. The facility can be operated with up to 200 L material, with operating temperatures ranging from -10 to 150 deg C, and pressures of full vacuum to 120 psig.

From a high level, the Pilot Plant equipment functional requirements include:

- Ability to stage raw materials, including flammable and toxic liquids, powders, solids, and gases.
- Ability to transfer staged raw materials to several potential reaction vessels at either a fast rate or at slow, carefully controlled, rates.
- Agitated reaction vessels with overhead condensers and ability to control process variables such as temperatures, pressure, and agitation.
- In-situ reaction and process monitoring through process analytical technologies.
- Ability to crystallize materials and filter using several forms of filtration and cleaning methods.
- Ability to clean the system fully to prevent contamination between campaigns.
- Ability to view inside vessels and rooms when operating in remote mode (cameras).

Utilities required to enable the functionality include:

- Vacuum and Nitrogen systems
- Cooling water and various independent temperature control units
- Electrical Substation
- Room, hood, and process ventilation
- Relief systems

Facility Design

The new Pilot Plant needed to be built within the existing Pilot Plant building, which was designed and constructed to contain explosive materials with earthen berms on 3 sides of the structure. Before installation of new programmatic equipment could begin, the building was retrofitted, with infrastructure and utilities upgraded to accommodate the new demands. The building's existing rooms were converted to allow raw material staging within a walk-in hood in a separate space from the synthesis equipment. The electrical classification of the space containing synthesis equipment was designated Class I Div 1 while utilities were located in general use areas.

Due to its remote location and sensitive environment, performing construction work at LLNL S300 is expensive and subject to extensive environmental controls. To minimize on-site construction work, the synthesis equipment was modularized and fabricated on skid system off-site for testing and reduction of installation at S300. The equipment layout on the skids was challenged by the limitations of the existing building. As much as possible, gravity is used to aid in material transfers, so the flow of material is from the top down. Adding all the desired capabilities to the reactors required that common ports and common headers be used, including the use of double-walled piping.

Figure 1: Pilot Plant Skid 3D Model

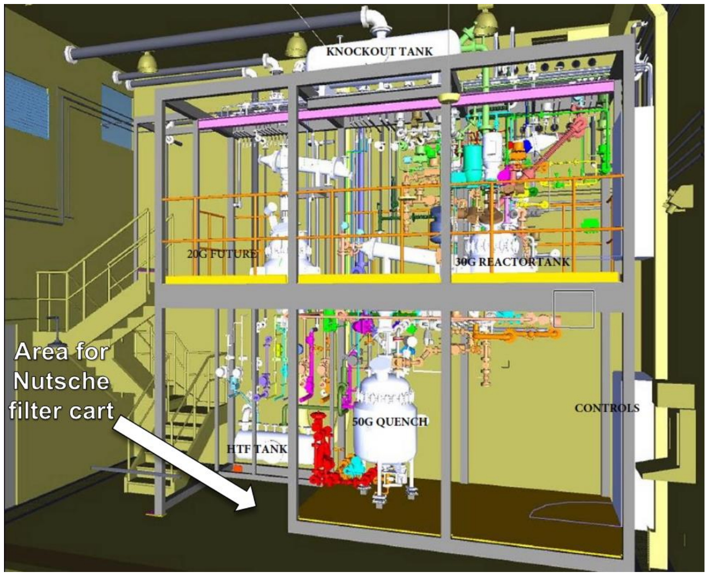


Figure 2: Pilot Plant Skids at fabrication facility and Pilot Plant Skids installed at Site 300

Safety Reviews

A collaborative effort with Hart and LLNL evaluated potential safety considerations from the various chemicals, reactions, operating conditions, and HE molecules that the facility might use in synthesis in order to determine appropriate rigor in the design of equipment and controls. A multidisciplinary team, including LLNL synthesis Subject Matter Experts (SMEs), performed a Failure Modes and Effects Analysis (FMEA) to identify risks associated with each part of the facility. When a risk was found to be too high, due to consequence and likelihood, the team identified appropriate controls to include in the system to reduce the risk. Controls included ensuring adequately sized pressure relief devices, fire protection and containment considerations, redundant temperature instruments, backup agitation, and interlocks. Due to high heats of reaction associated with the synthesis routes intended in the Pilot Plant, and known decomposition at elevated temperatures, runaway scenarios were a particular focus of the FMEA. The design included capabilities for an emergency quench to stop runaway reaction scenarios.

In addition to various safety actions programmed within the process control software, an independent Safety Interlock System (SIS), standardly used at other LLNL facilities to provide “run-safe” capability, was designed to provide additional layer of safety, putting the equipment in a safe-state unless certain safety permissives are met. Permissives include ensuring process temperatures are below upper safety limits, and that emergency-stop buttons, building door positioners, seismic sensors, and loss of power detectors indicators are not activated.

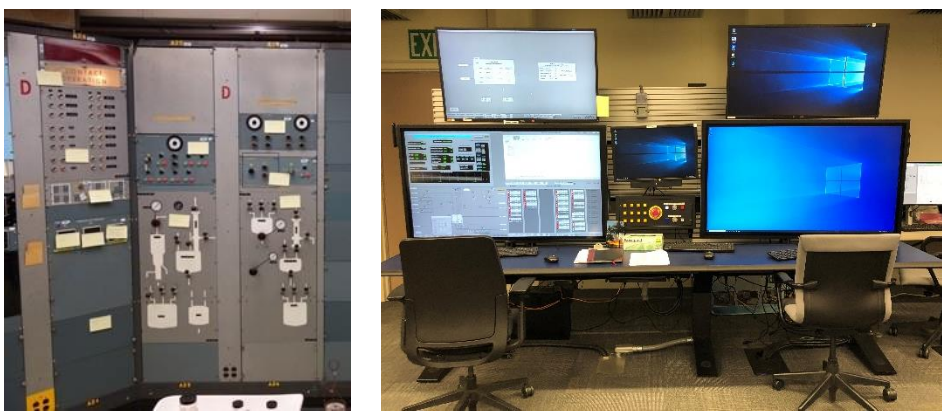
Each specific synthesis route requires another FMEA to be performed, specific to the risks associated with the chemicals and procedure. Hazards associated with working with high hazardous materials drive controls such as independent verification of equipment, PPE requirements, and even potentially remote operations.

Control System Design

As the intent was to convert hands-on processing to majority remote, the process control system design was a significant project deliverable. An existing administrative building located ~150 ft away was upgraded to serve as the Control Room for remote operations.

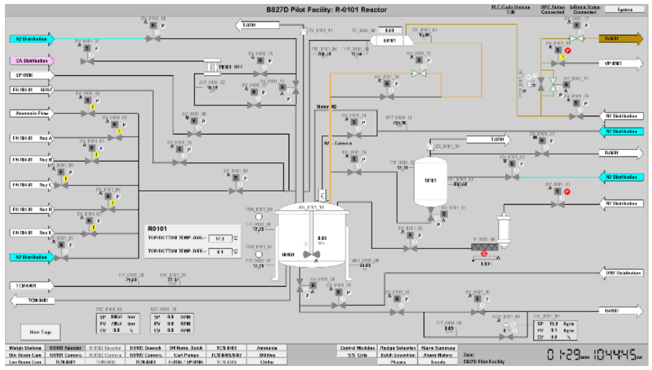
A completely remote system was extremely cost-prohibitive and not seen as necessary for all steps in a synthesis process. But even for a majority remote process, the functionality, interface, and operability of the control system had to be customized for the Pilot Plant equipment. The complexity of providing agile yet safety-focused controls added an order of magnitude to the cost and schedule in order to develop, integrate with equipment, and to verify and validate functionality. Though used widely in industry and commercially available, the chosen control system platform, Wonderware®, was new to the Chemistry Area and required upgrades to the facilities related information technology (IT) infrastructure and new skills to be acquired by the small operations staff (3) in addition to their other responsibilities.

Figure 3: Operator Interface Systems in the Control Room: Before and After



To provide a safer and more repeatable process for the operator, the control system had to be designed to handle the complexity of operations without adding additional burdens. 20 different operator interface screens were created to allow the operators to navigate through the entire system while providing information on the process, such as status of equipment (on/off or open/closed) and modes (manual or automatic), as well as the current values for temperature, pressure, weight, agitation, and flow.

Figure 4: Operator Interface Screen for 100-L Reactor

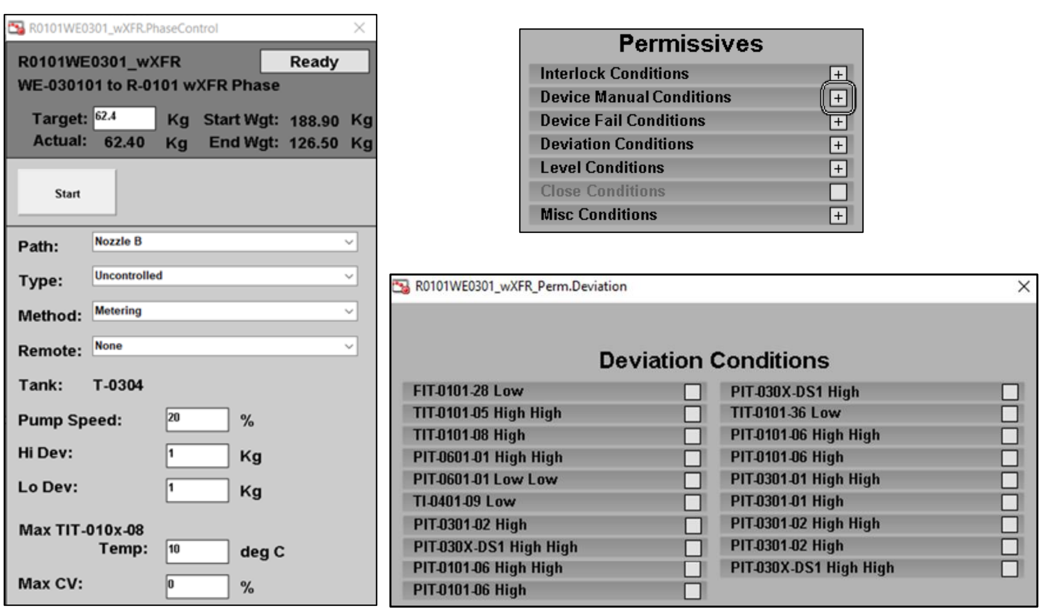


Each process variable can have operating ranges assigned to them so that an alarm is created and the operator notified if their value exceeds High, High High, Low, Low Low alarm points. Automatic actions can also be assigned to these alarm conditions. For example, on High High process temperature, full cooling is applied to the system. Devices can also include permissives, so that inadvertent actions are prevented. For example, a pump cannot be started if the downstream valve is not open.

Equipment can be controlled manually via the operator interface or automatically through phases. Phases allow the operator to perform a sequence of actions with multiple options and parameters that can be adjusted by the operator to perform specific functions. As this is an R&D facility, flexibility is needed to make small changes to how the batch will be run, and phases make this easier to perform.

An example of a transfer phase is: while in remote mode, transfer a raw material from vessel #1 located on scale #2, through pump #3, using hoses connected to manifold nozzle B, at a rate of ##.# kg/min to Reactor #4, until ##.# kg have been transferred, while keeping the pump rate less than ##.# %, pausing the transfer if the temperature in Reactor #4 is above ##.# deg C. When the transfer is completed, purge the line with nitrogen from the source vessel to the destination before closing all valves. If during the transfer a permissive is no longer true (such as a valve that must be in auto is changed to manual, or the weight or pressure in Reactor #4 exceeds their High alarm point), hold the phase until the condition is resolved and the operator acknowledges and resumes the phase.

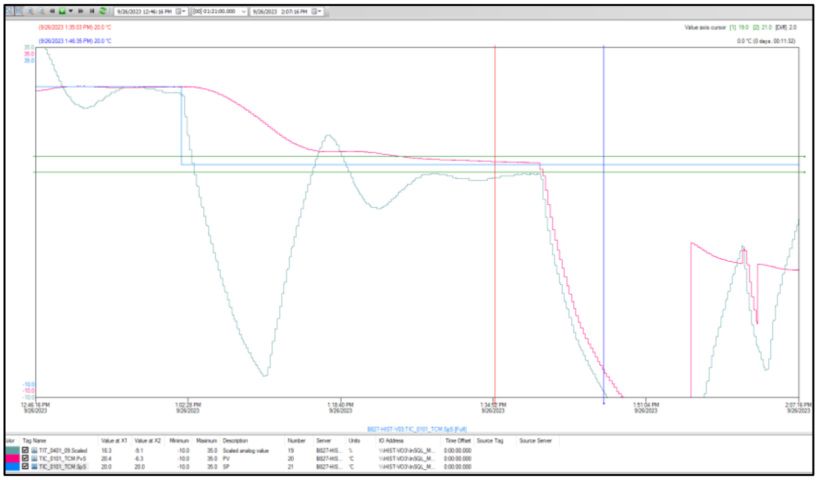
Figure 5: Phase Options within a Transfer Phase and Examples of Permissives (conditions must not be true)



As an additional safety measure, no transfers are started simply by pressing start in a phase. "Deliberate operations" is built into each phase, requiring the operator to confirm the flow path is correct and performing a phase release on a key device in the flow path before a transfer will begin.

The information on each device and instrument is continuously captured via a historian system, which enables the operator to build dynamic trends and monitor key process variables over time. This includes setpoints, values, alarm points, and device state.

Figure 6: Process Variable Trending in Historian



To provide the desired flexibility and safety, the control system includes over 1180 alarms, 800 I/O points, 100 controlled valves, 110 software interlocks, and over 160 phases.

While overall the system appears complicated due to the need for the broad potential synthesis requirements, the functionality becomes simpler when focused on a single synthesis path. In fact, after construction of the process was completed and basic commissioning activities were done, ensuring that process equipment functioned as intended, the LLNL team narrowed the system control testing to focus on 1 chemical synthesis route to demonstrate the system, rather than include all the various capabilities that were provided though not yet required. This approach defers the time to bring these capabilities online to the future, and had to be weighed against the costs and benefits of performing it during startup; provide full system capability initially at the expense of a longer facility startup, or reducing the scope for the facility start up at the expense of potentially impacting future agility due to need to perform additional testing.

Construction, Commissioning, and Startup

As with virtually every project, the HE Synthesis Pilot Plant project had both inevitable and unforeseen challenges. Though agile Pilot Plant designs are not atypical in the chemical manufacturing industry, many of the lessons learned in this project stemmed from the unique nature of the facility within the context of LLNL. The complexity of designing, installing, and commissioning a state-of-the-art, agile, and safety-focused Chemical Synthesis pilot scale system in a facility that previously used 50-year-old hand-operated glass equipment in an R&D capacity, within the lab with no similar systems, was underestimated in many ways. LLNL learned much from this project to improve projects throughout the enterprise.

Commissioning of the system was challenged when the installation of piping, instruments, and equipment became decoupled from controls system integration and further complicated by scope changes. This led to incomplete documentation and left issues resolution to LLNL, in spite of limited internal resources.

The Pilot Plant commissioning process was iterative; installation verifications, pre-functional tests, and then functional tests utilizing water were performed. When issues were encountered during this process, the operations team needed to determine if the issues were real or just due to documentation not having been updated to reflect the as-built condition. If the issue was real, the team would evaluate the impact of the issue to determine what actions might address it, weighing the costs and benefits of each. Many times, the issue could not be accepted as-is and had to be resolved somehow. For example, the control code had originally been written to include equipment that was intended to be installed in the future. As that equipment was not installed, the code caused errors, alarms, and prevented phases from running. The decision was made to not "delete" this extensive code, but instead to "deactivate" it to allow the process to work as desired while minimizing time to implement the change. Each new issue and subsequent change required documentation updates and additional testing before commissioning activities could resume.

Recall the transfer phase described in figure 5; validating that this phase works as intended, with many potential options provided, requires testing each option against all the other options. Thus, due to the complexity of the control system, specifically capabilities provided by the design not needed yet, the focus of the commissioning activities was narrowed to only cover the intended operations needed for the first synthesis route. Unused, thus untested and unvalidated, code was "deactivated" to be inaccessible but easily recovered for testing in the future.

Once the required code functioned as needed for the initial synthesis route, batch procedures were finalized in conjunction with performing water batching to simulate all the material movements, functionality testing the system to the best of our ability prior to adding hazardous material. This also enabled the operators to become comfortable with the control system operation while being involved in developing the required supporting documents (procedures and training).

Conclusion

LLNL's investment in the new HE Synthesis Pilot Plant at Site 300 allows for R&D for a variety of new and conventional HE molecules to complement and inform scale-up and larger scale production. Designing a pilot plant that is safety-focused, agile, state-of-the-art, and fully automated was challenging as these goals create constraints on each other; cost and benefits had to be evaluated against the end goal and compromises made. Despite the efforts during design and commissioning to create the best possible system, it is expected additional improvements and scope will be identified to continuously improve the performance, capability, safety, efficiency, and operability of the system to meet future challenges. The capabilities installed in the modern facility, equivalent to many industrial facilities, and the flexibility for future scope, make the LLNL Pilot Plant capable of rising to future challenges.

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