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

Insensitive Munitions and Energetic Materials Technology Symposium, Portland, Oregon

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Lawrence Livermore National Laboratory

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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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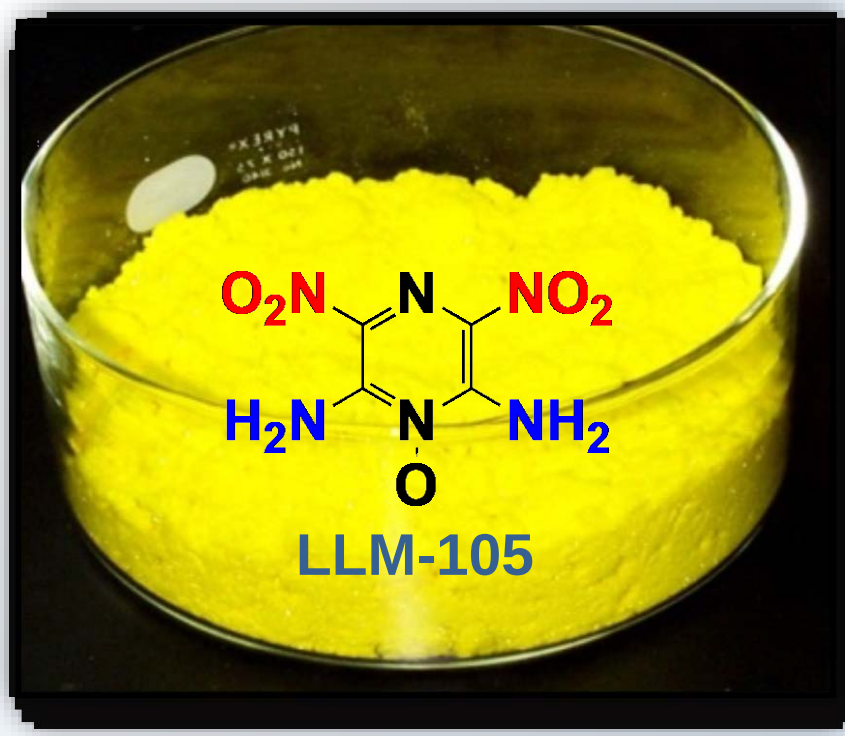
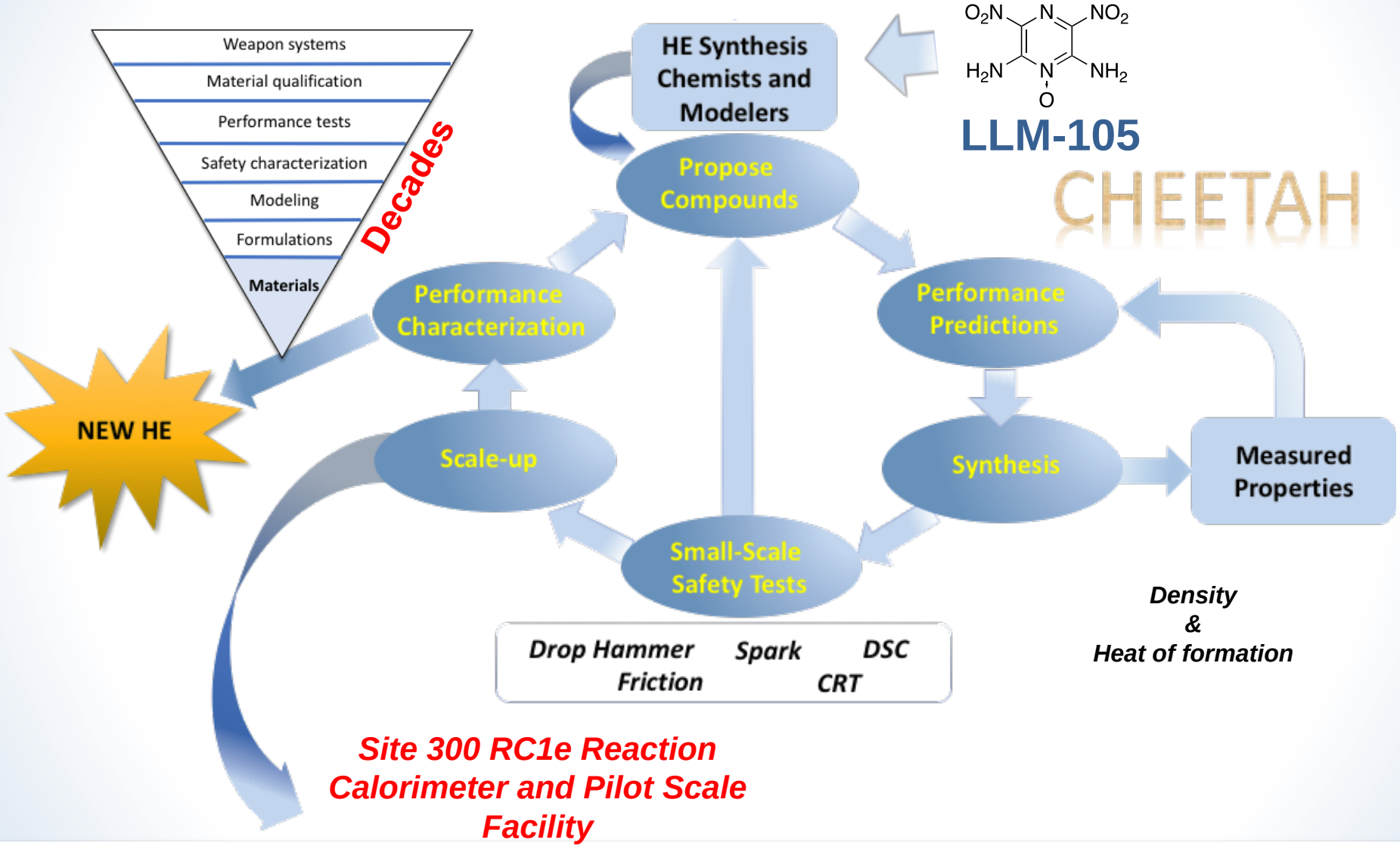
- **Focus:** Synthesis of new insensitive high explosives (IHE) and conventional high explosives (CHE)
- Synthesis group supports :
  - Basic explosives R&D
  - Stockpile stewardship
  - Counterterrorism
  - Development of HE for military and commercial use



LLNL HE Synthesis Developmental Process



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

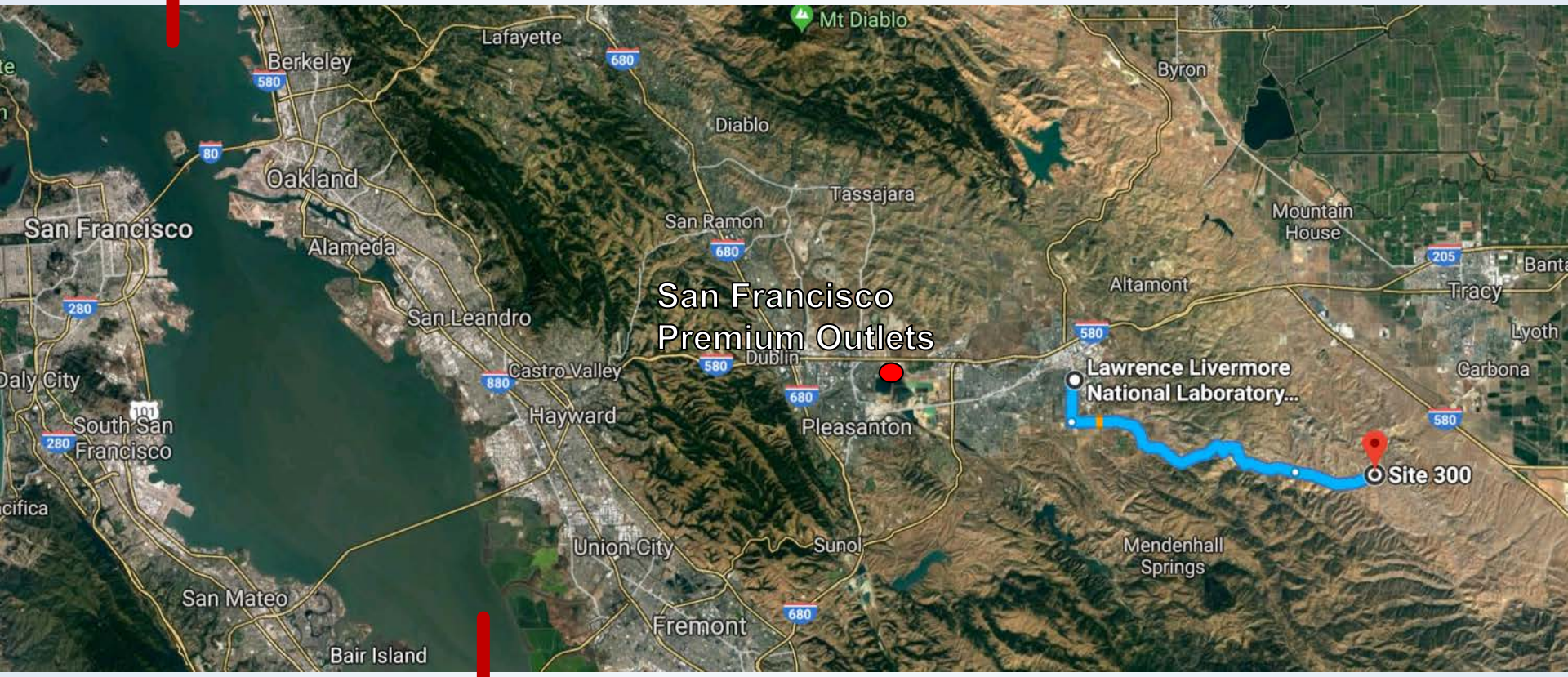
Need for Pilot Scale HE Synthesis at LLNL

- 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.

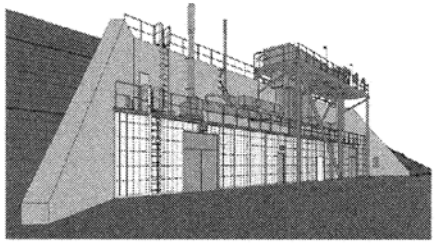
San Francisco Bay area and Site 300

Napa



Google Maps

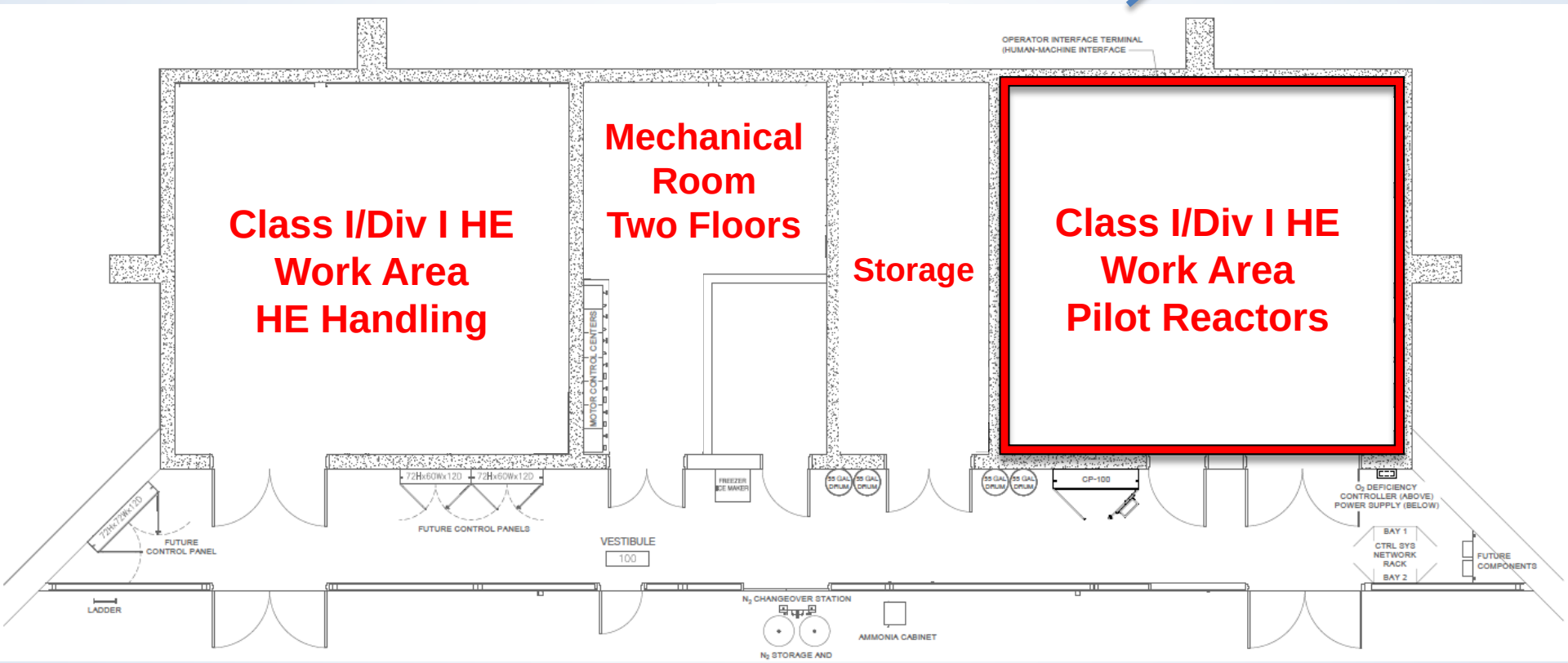
San José



Site 300: 7,000 acres rural foothills

HE Synthesis: 827D First Floor Layout and Former Utilization of Space

827A Control Room



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.

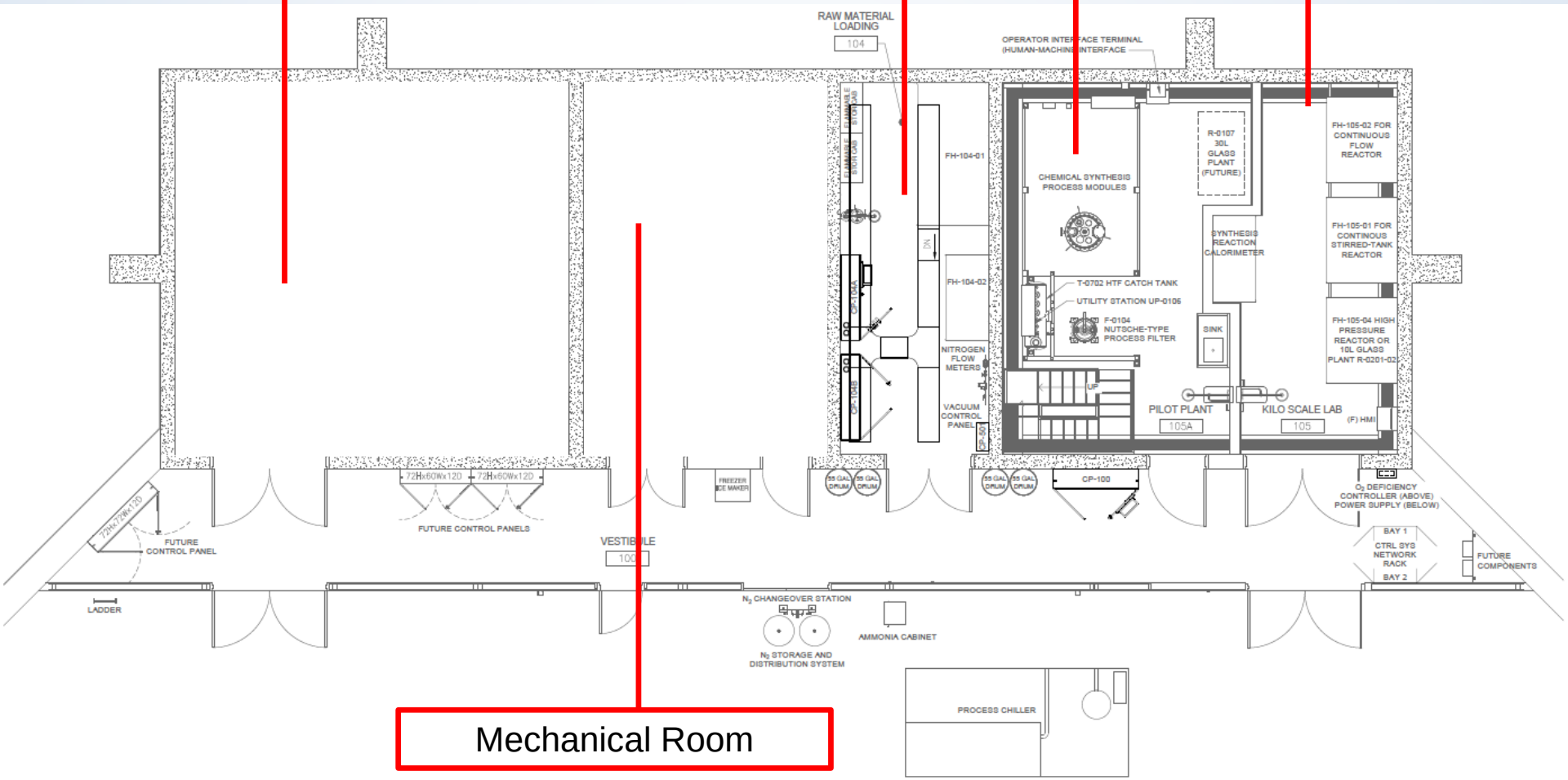
Renovated 827D First Floor Layout

HE Powder Processing

Remote Reagent Delivery

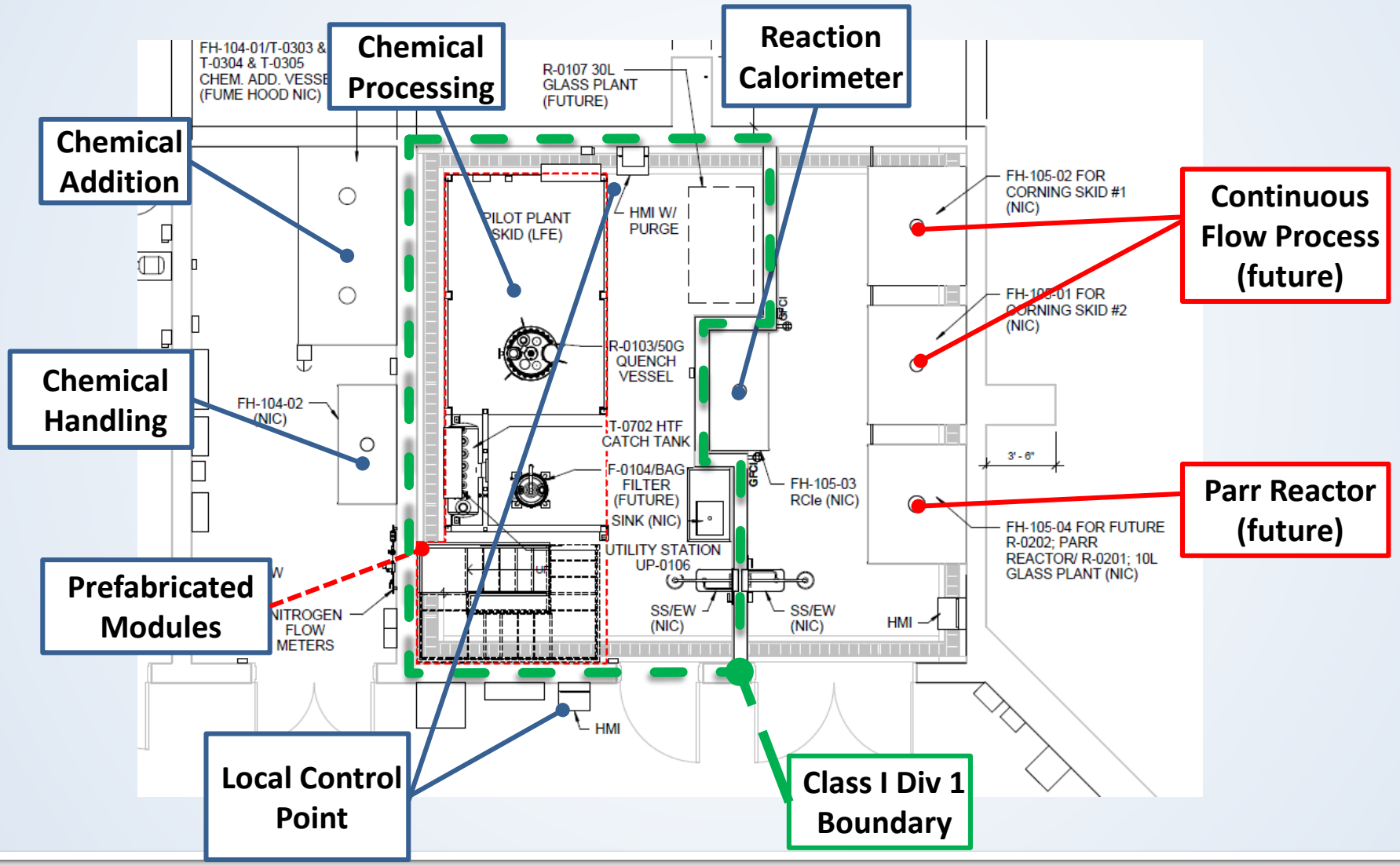
Pilot Skid

Contact Synthesis Area

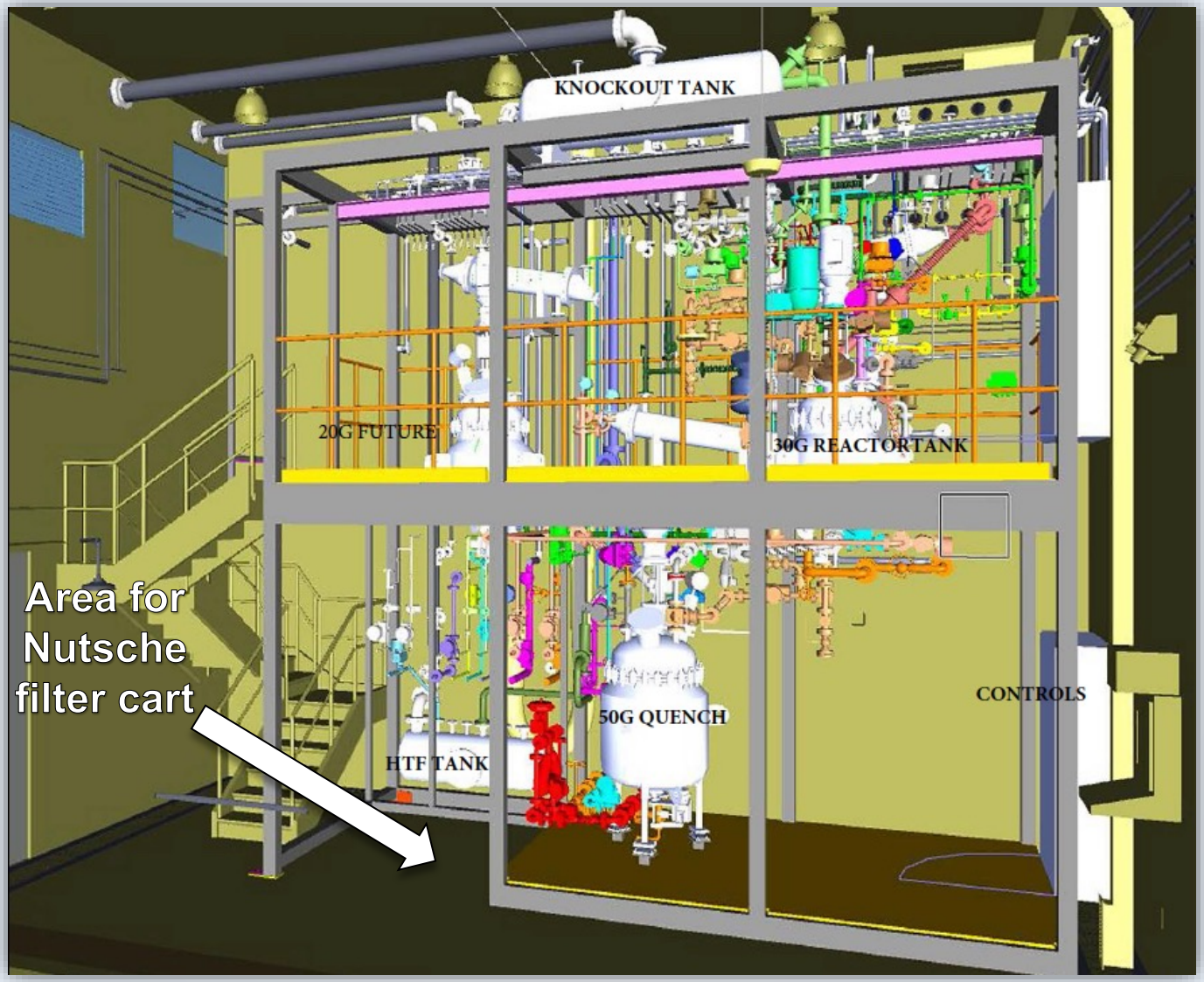


Mechanical Room

Pilot Skids, Reagent Delivery, and General Purpose Scale-up area



3D Model of Pilot Reactor Skid



Constructed Pilot Skid

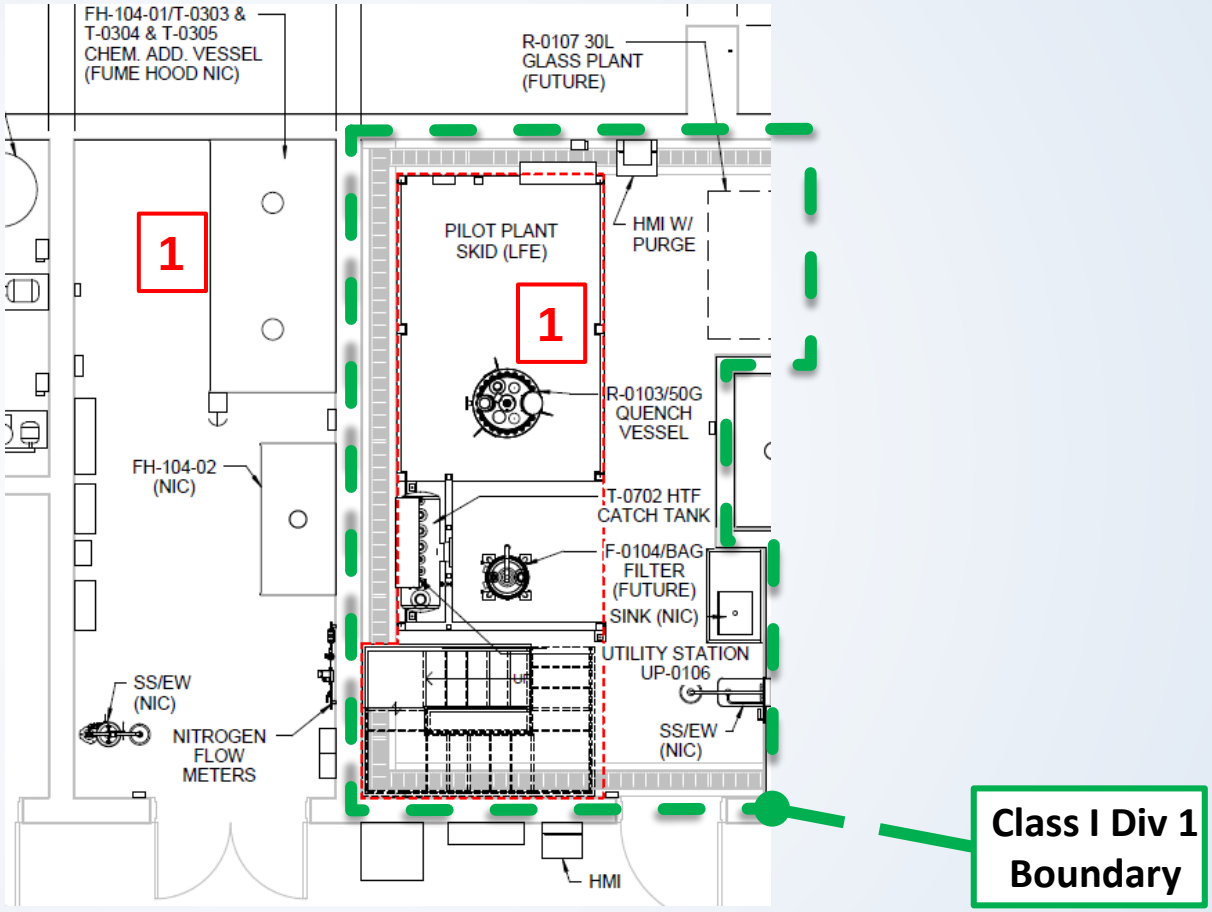
Pilot Skids at Fabrication: Hart, Cumberland, RI

Pilot Skid Critical Lift and Installation: B827D, Site 300

Typical Synthetic Process in Pilot Skid: Nitration

1. Load liquid and solid reagents

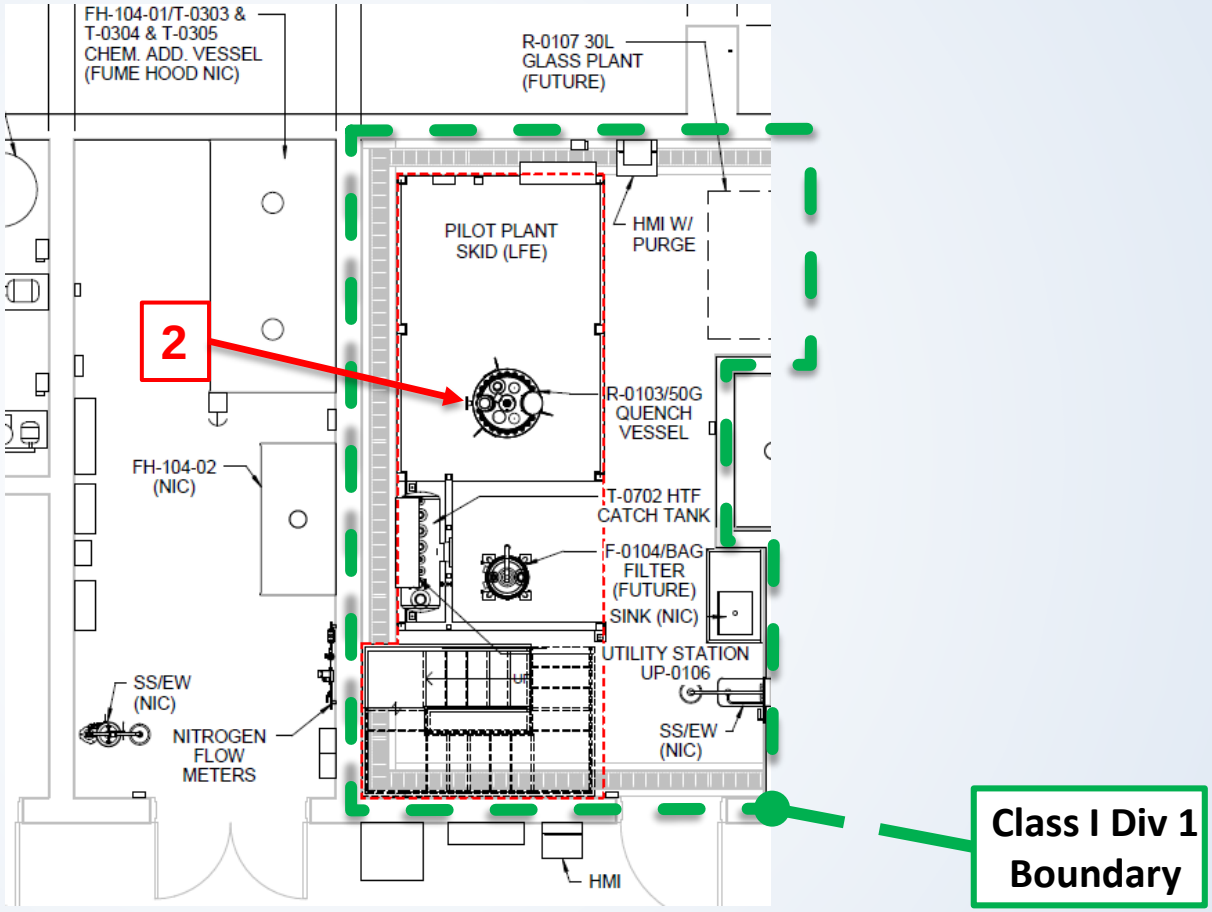
Contact operation conducted in a fume hood.



Typical Synthetic Process in Pilot Skid: Nitration

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

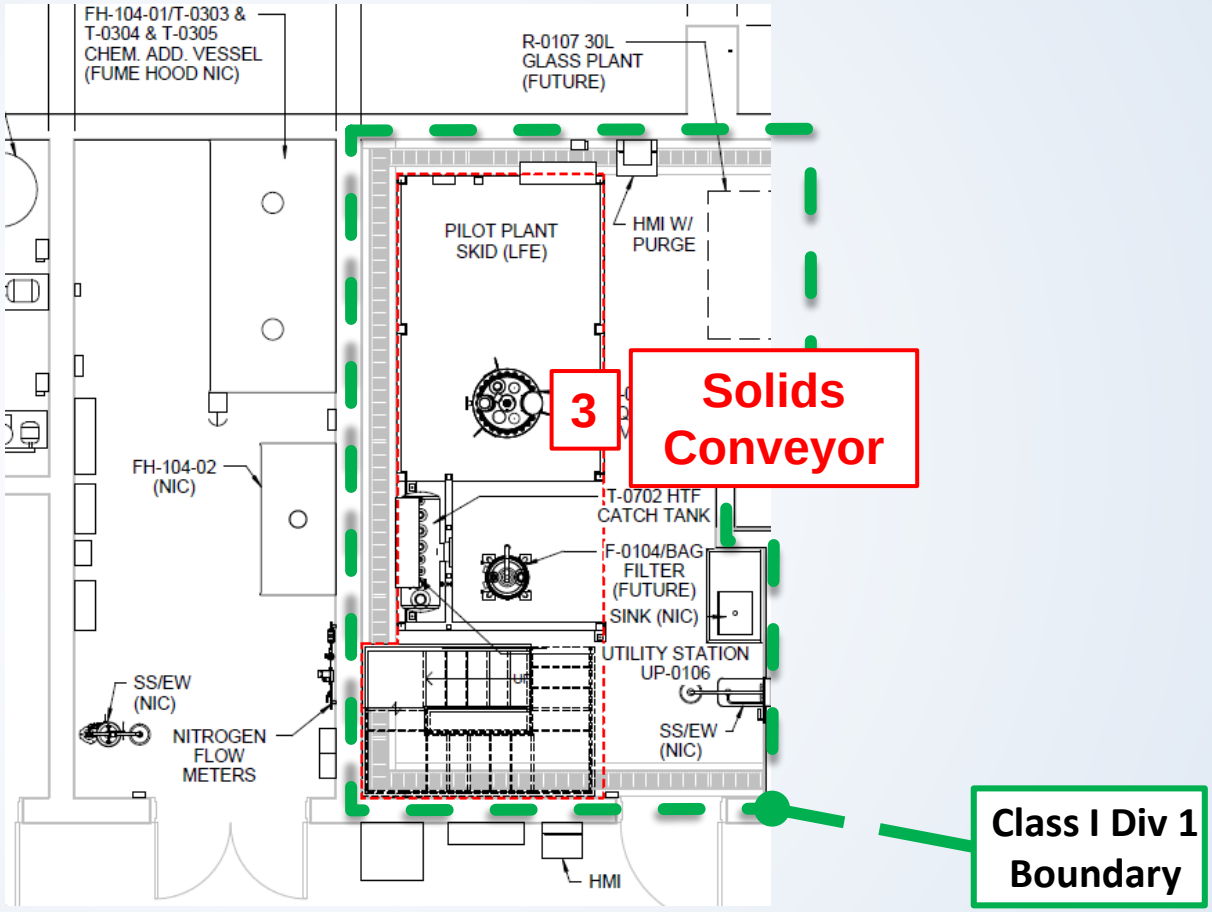
Remote operation conducted from any of the HMI stations.



Typical Synthetic Process in Pilot Skid: Nitration

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

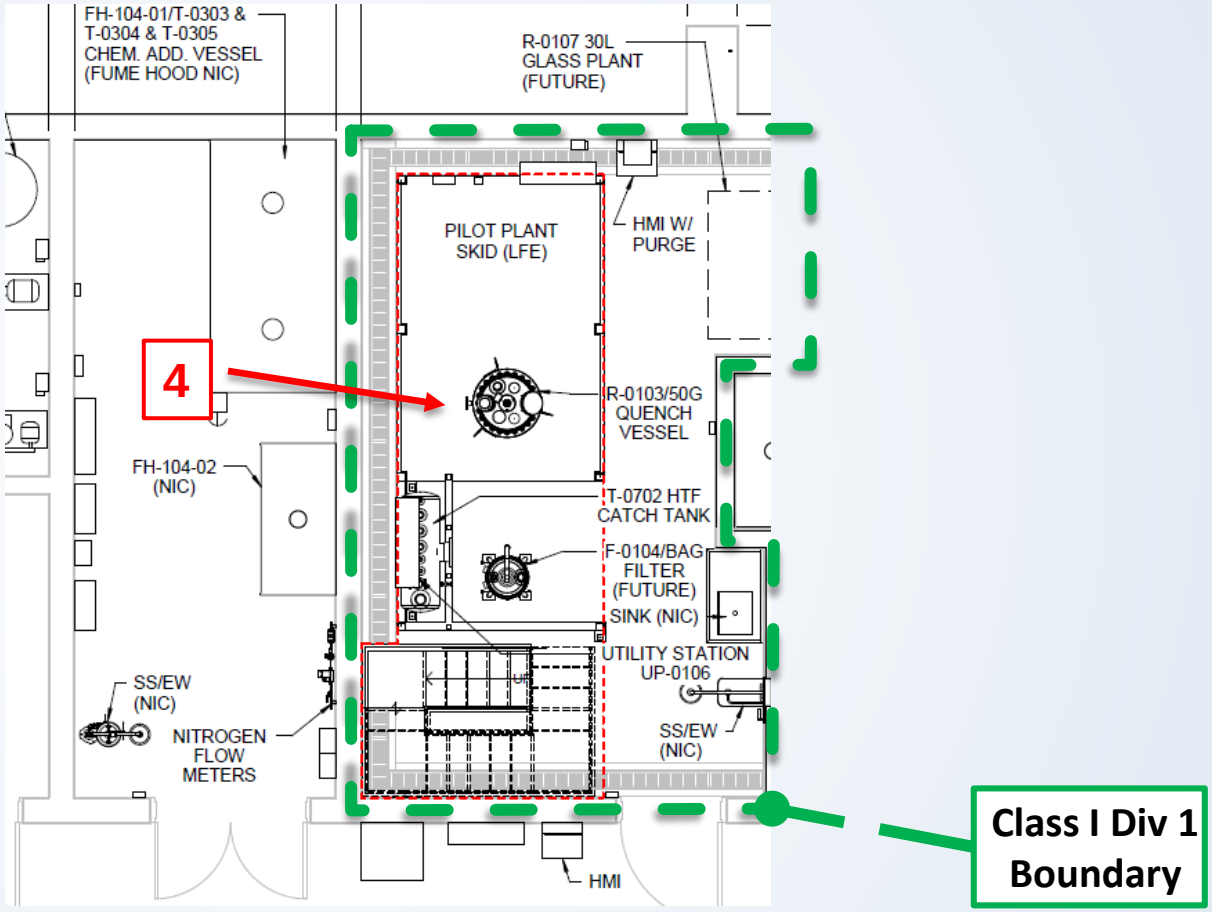
Remote operation from HMI.



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.

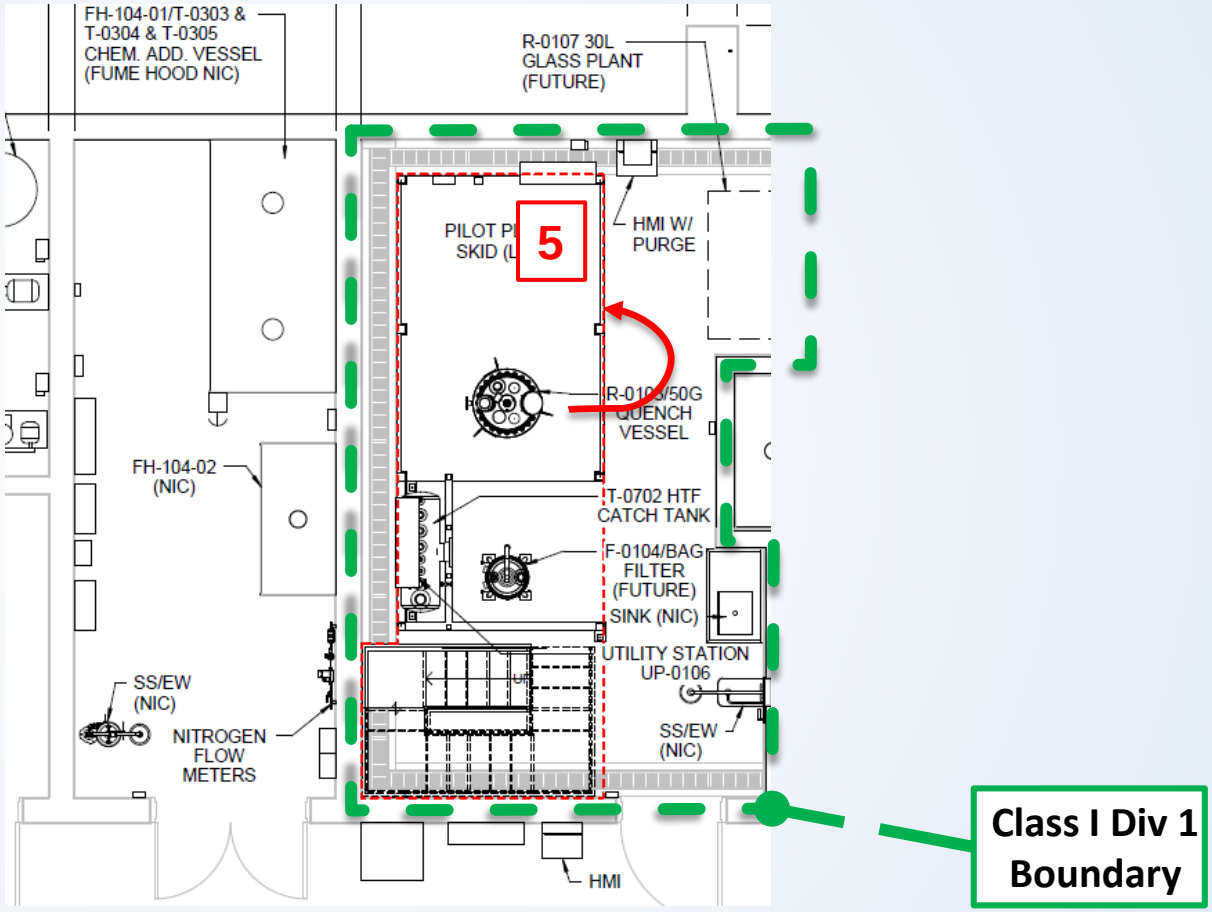
Remote operation from Control Room.



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

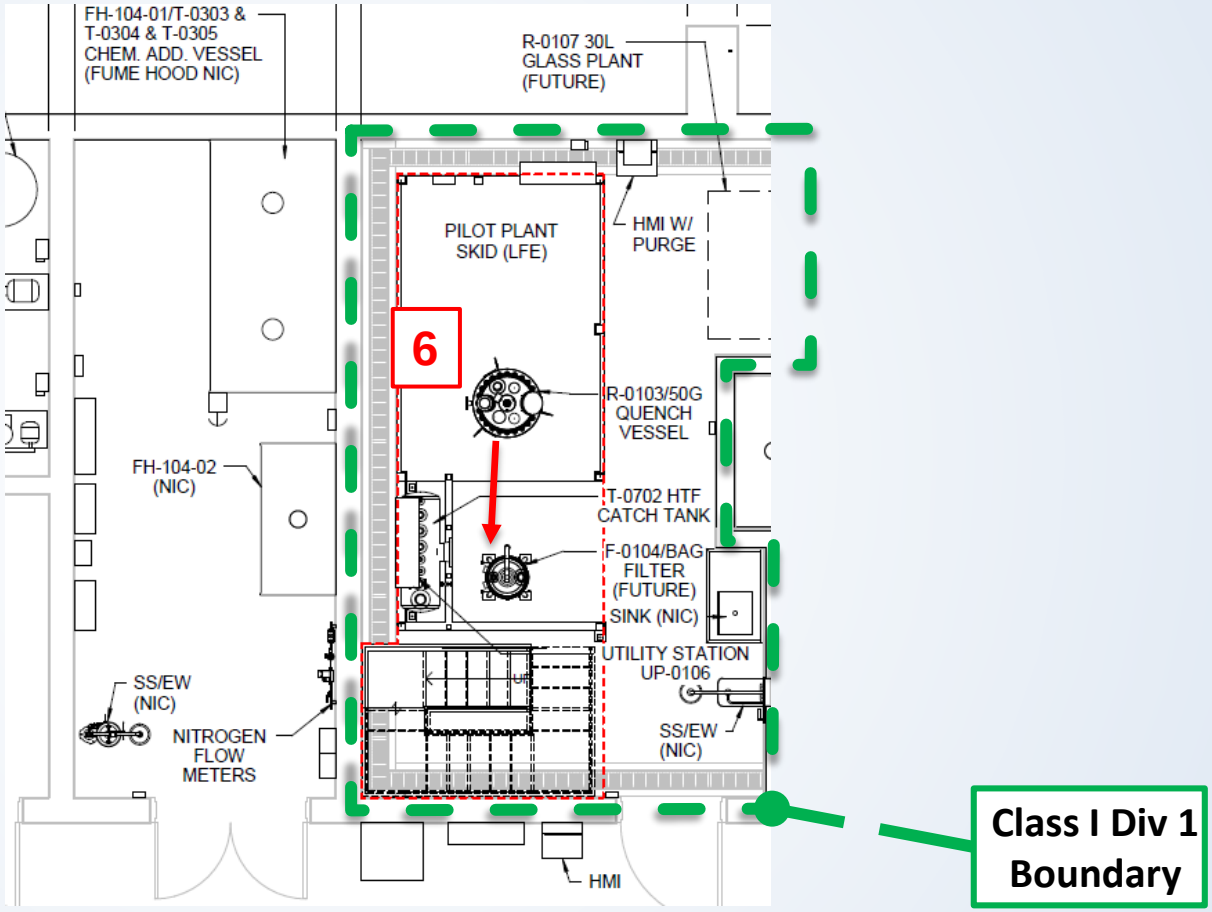
Remote operation from control room.



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.



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1. Load liquid and solid reagents
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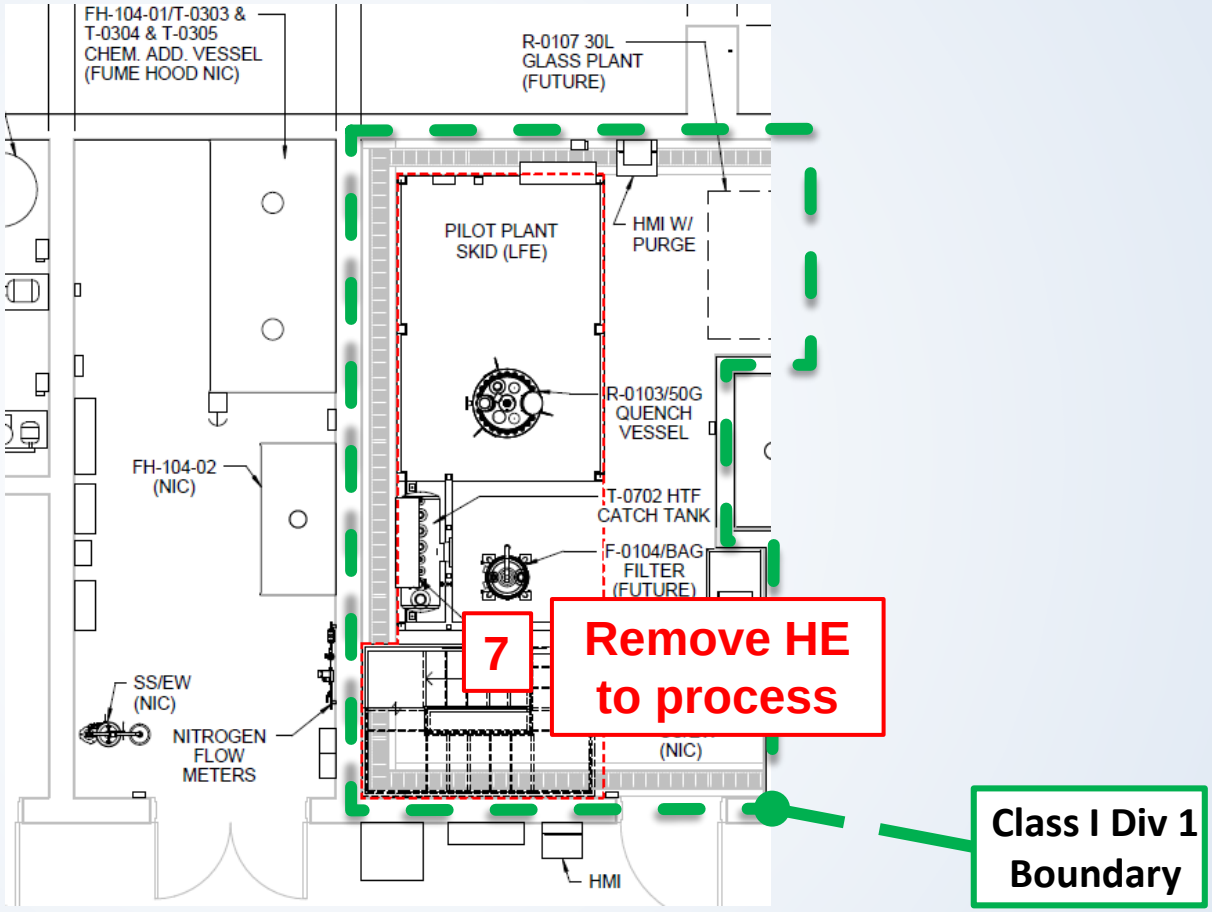
5. Quench Reaction
6. Filter Reaction
7. Collect Product.

Contact operation



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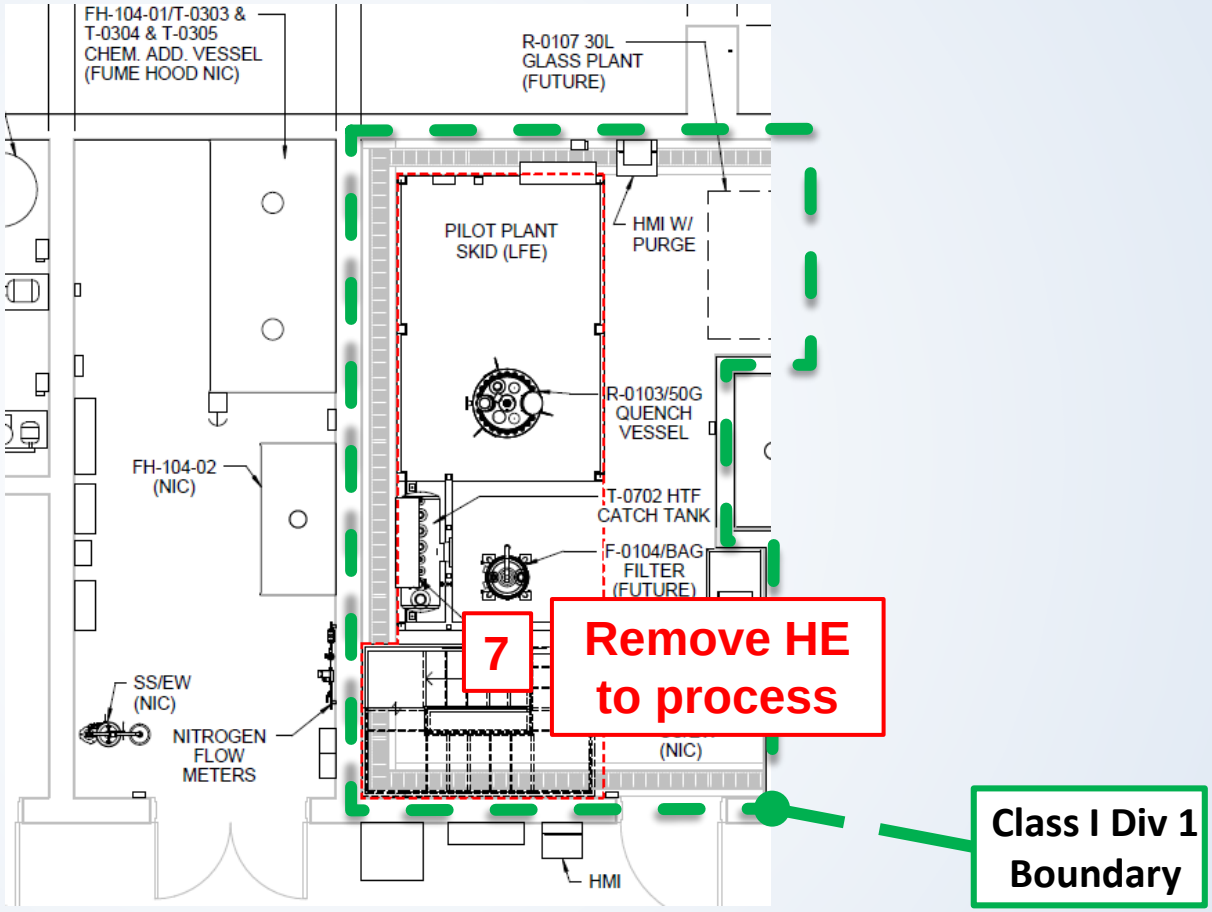


Timing of valve sequence actuation, alarm parameters, emergency stops, and PID control managed through integrated control system (ICS)

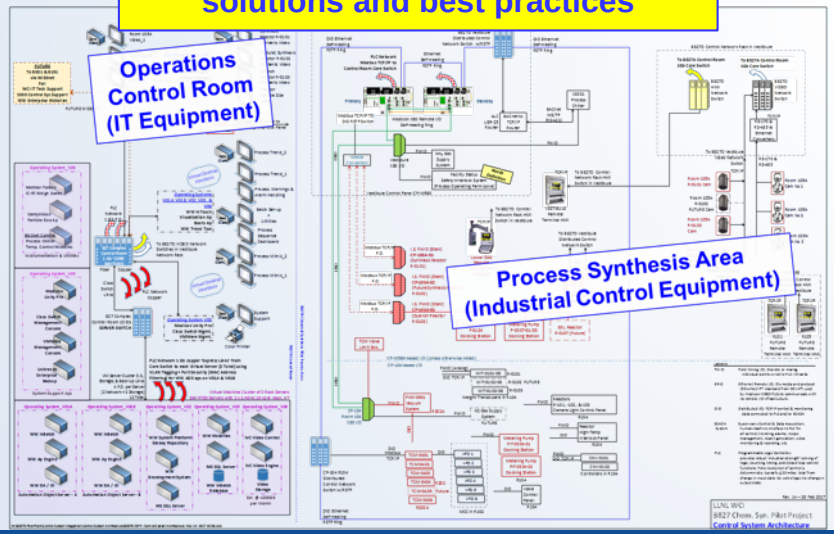
Industrial Control System (CS)

1965 → 2018

Synthesis Controls BEFORE

Synthesis Controls AFTER

CS Architecture uses contemporary industrial process control systems solutions and best practices



- 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:

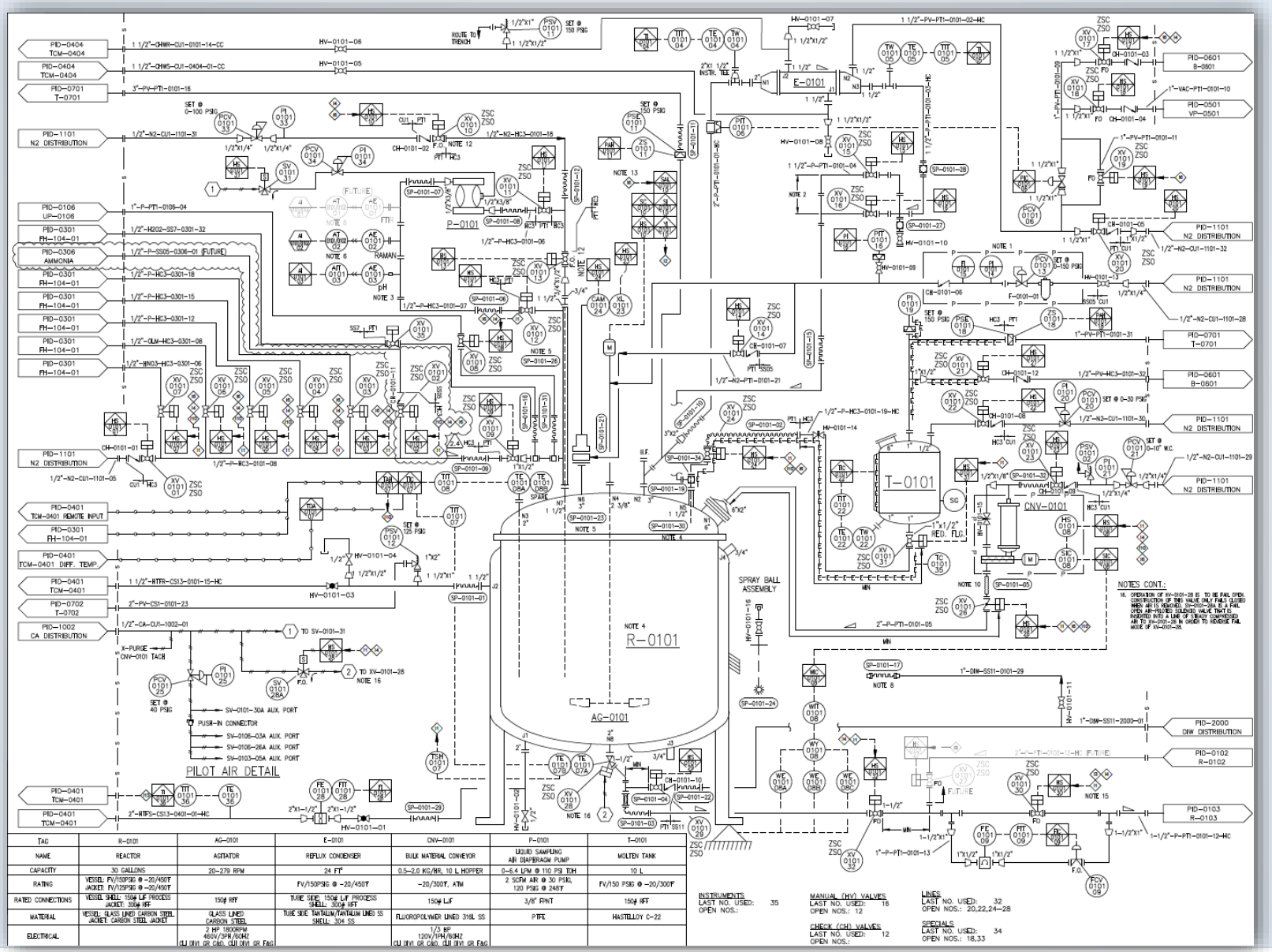
  827A – Control Room, 827D Chemical Addition Room, and 827D Pilot Skid

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.

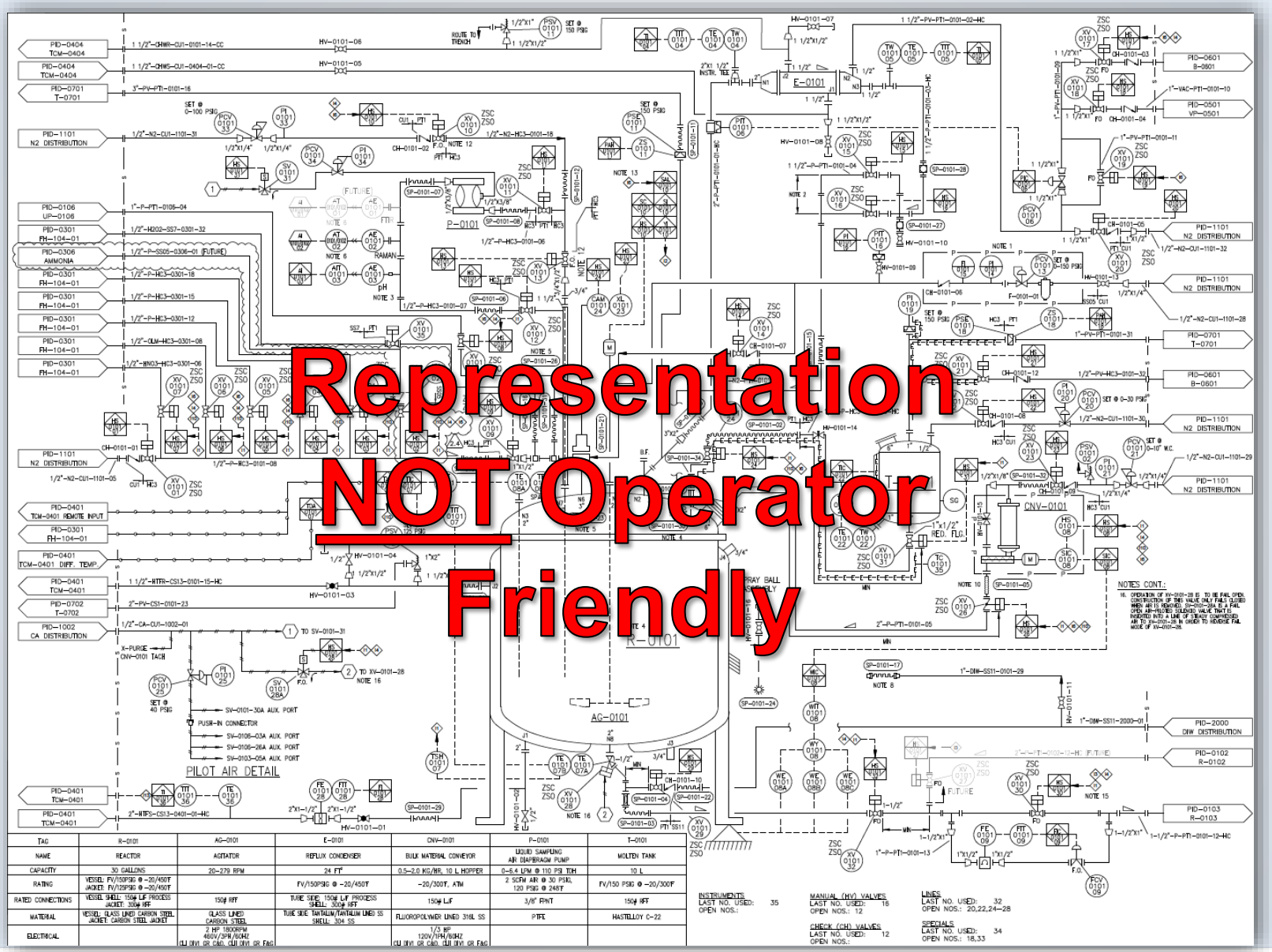
P&ID for 100 L Glass Lined Reactor



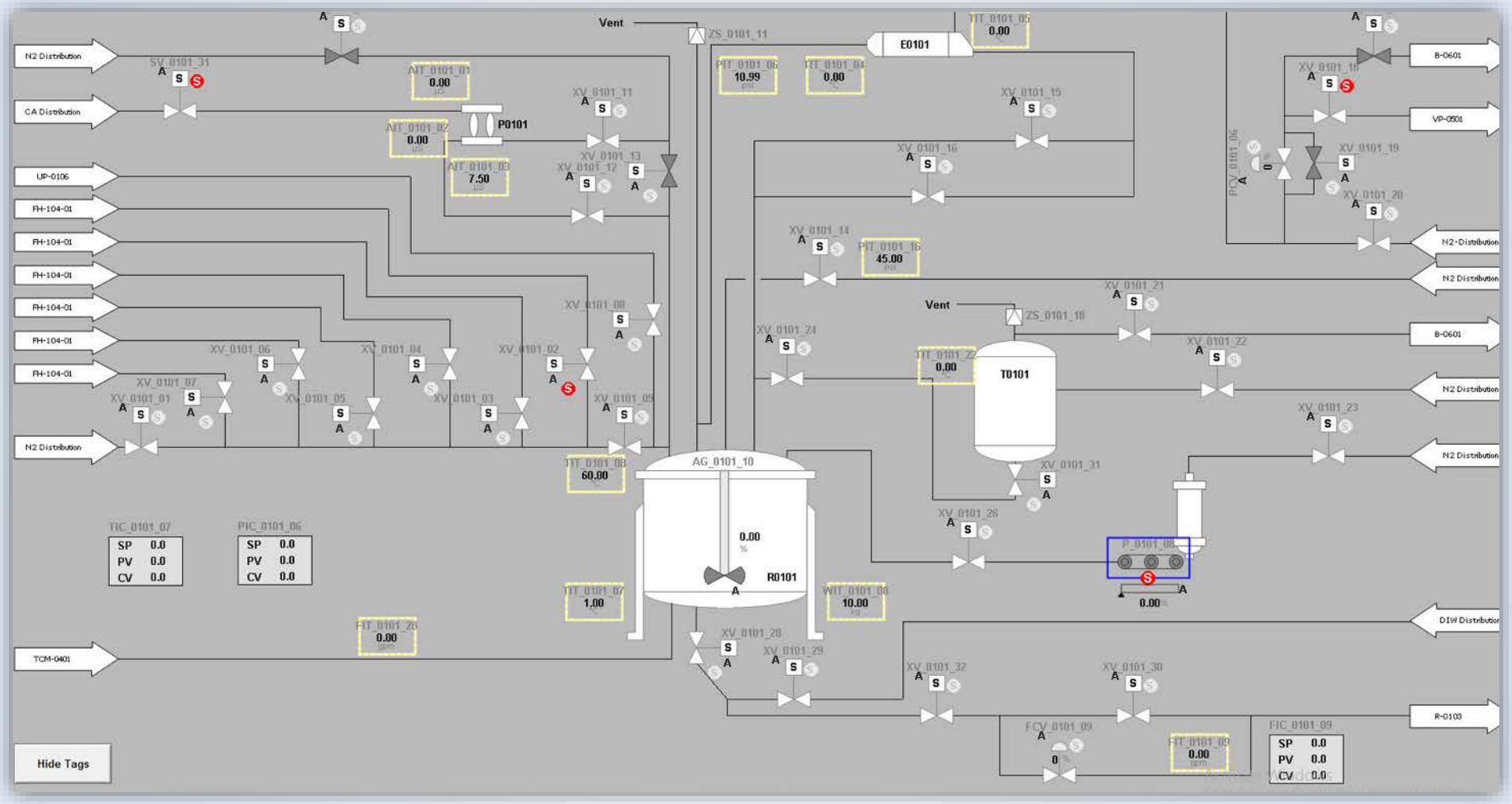
TAG	R-0101	AG-0101	E-0101	CNV-0101	P-0101	T-0101
NAME	REACTOR	AGITATOR	REFLUX CONDENSER	BULK MATERIAL CONVEYOR	LIQUID SAMPLING AIR DIAPHRAGM PUMP	MOLTEN TANK
CAPACITY	30 GALLONS	20–279 RPM	24 FT²	0.5–2.0 KG/HR, 10 L HOPPER	0–6.4 LPM @ 110 PSI TDH	10 L
RATING	VESSEL: FV/150PSIG @ −20/450°F JACKET: FV/125PSIG @ −20/450°F		FV/150PSIG @ −20/450°F	−20/300°F, ATM	2 SCFM AIR @ 30 PSIG, 120 PSIG @ 248°F	FV/150 PSIG @ −20/300°F
RATED CONNECTIONS	VESSEL: SHELL: 150# LJF PROCESS JACKET: 300# RFF	150# RFF	TUBE SIDE: 150# LJF PROCESS SHELL: 300# RFF	150# LJF	3/8" FPNT	150# RFF
MATERIAL	VESSEL: GLASS LINED CARBON STEEL JACKET: CARBON STEEL JACKET	GLASS LINED CARBON STEEL	TUBE SIDE TANTALUM/TANTALUM LINED SS SHELL: 304 SS	FLUOROPOLYMER LINED 316L SS	PTFE	HASTELLOY C-22
ELECTRICAL		2 HP 1800RPM 460V/3PH/60HZ CU DIVI OR C&D, CU DIVI OR FAG		1/3 HP 120V/1PH/60HZ CU DIVI OR C&D, CU DIVI OR FAG		

INSTRUMENTS	MANUAL (HV) VALVES	LINES
LAST NO. USED: 35	LAST NO. USED: 18	LAST NO. USED: 32
OPEN NOS.:	OPEN NOS.: 12	OPEN NOS.: 20,22,24–28
	CHECK (CH) VALVES	SPECIALS
	LAST NO. USED: 12	LAST NO. USED: 34
	OPEN NOS.:	OPEN NOS.: 18,33

P&ID for 100 L Glass Lined Reactor



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.
  - Nutsche 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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