SUBTASK 3.9 – DIRECT COAL LIQUEFACTION PROCESS DEVELOPMENT

Final Report

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SUBTASK 3.9 – DIRECT COAL LIQUEFACTION PROCESS DEVELOPMENT

ABSTRACT

The Energy and Environmental Research Center (EERC), in partnership with the U.S. Department of Energy (DOE) and Accelergy Corporation, an advanced fuels developer with technologies exclusively licensed from ExxonMobil, undertook Subtask 3.9 to design, build, and preliminarily operate a bench-scale direct coal liquefaction (DCL) system capable of converting 45 pounds/hour of pulverized, dried coal to a liquid suitable for upgrading to fuels and/or chemicals. Fabrication and installation of the DCL system and an accompanying distillation system for off-line fractionation of raw coal liquids into 1) a naphtha–middle distillate stream for upgrading and 2) a recycle stream was completed in May 2012. Shakedown of the system was initiated in July 2012. In addition to completing fabrication of the DCL system, the project also produced a 500-milliliter sample of jet fuel derived in part from direct liquefaction of Illinois No. 6 coal, and submitted the sample to the Air Force Research Laboratory (AFRL) at Wright–Patterson Air Force Base, Dayton, Ohio, for evaluation. The sample was confirmed by AFRL to be in compliance with all U.S. Air Force-prescribed alternative aviation fuel initial screening criteria.

This subtask was funded through the EERC–DOE Joint Program on Research and Development for Fossil Energy-Related Resources Cooperative Agreement No. DE-FC26-08NT43291. Nonfederal funding was provided by Accelergy Corporation.

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EXECUTIVE SUMMARY

The Energy and Environmental Research Center (EERC), in partnership with the U.S. Department of Energy (DOE) and Accelergy Corporation, an advanced fuels developer with technologies exclusively licensed from ExxonMobil, undertook Subtask 3.9 to design, build, and preliminarily operate a bench-scale direct coal liquefaction (DCL) system capable of converting pulverized, dried coal to a liquid suitable for upgrading to fuels and/or chemicals. The primary objectives of Subtask 3.9 were to:

- Finalize the design of a continuous-mode bench-scale DCL reactor system based on a preliminary design developed by the EERC and Accelergy.
- Construct and shake down the DCL reactor system.
- Use the DCL system to initiate production of a jet fuel derived from Illinois No. 6 coal, with the subsequent objective of submitting a sample of the jet fuel to the U.S. Air Force Research Laboratory (AFRL) at Wright–Patterson Air Force Base for analysis to assess compliance with key selected fuel property requirements cited in MIL-DTL-83133F, the U.S. military specification for JP-8 jet fuel.

Over the course of the approximate 30-month project, EERC design and engineering staff worked in partnership with Accelergy-provided design consultants on:

- Improving the overall DCL system design.
- Integrating the design into existing and project-developed EERC facilities and infrastructure.
- Assessing and developing strategies for mitigating the operational risks associated with the DCL system, which—because system operation necessitates the use of high pressure, high temperature, and hydrogen—were of critical importance to maximizing the safety of the operational staff.

At project initiation, the DCL system was slated for installation in an EERC building anticipated to be an ideal location based on a preliminary engineering estimate of the infrastructure improvements needed to support safe and efficient operation of the DCL system. Key needed improvements included ventilation, fire alarm, fire suppression, and gaseous exhaust combustion/flare systems. Because the actual total cost of making these improvements (based on qualified contractor bids received) was higher than the project budget could support, a new site was needed. Several options were evaluated, including skid-mounting the DCL system and temporarily installing it in a semi-outdoor location that would enable seasonal operation only, but none of the evaluated options offered the possibility of meeting fuel production deliverable objectives in accordance with project cosponsor Accelergy Corporation expectations. At the

approximate 12-month point of the project, developments in nonrelated EERC projects led to an opening in the EERC National Center for Hydrogen Technology building, which is equipped with all necessary DCL project-required infrastructure, and tailoring of the DCL unit layout to accommodate this new space was initiated.

Although the site-selection problem resulted in the need to request a 10-month no-cost project extension, activities associated with developing equipment specifications, soliciting bids for major equipment delivery, ordering system fabrication supplies and equipment, and fabrication of unit operations were ongoing and enabled maintaining project progress and focus throughout the time the DCL system was without a permanent installation site. Fabrication and installation of the DCL system and an accompanying distillation system (for off-line fractionation of raw coal liquids into a naphtha–middle distillate stream for upgrading and a recycle stream) was completed in May 2012. Shakedown of the system was initiated in July 2012. In addition to completing fabrication of the DCL system, the project also produced a 500-milliliter sample of jet fuel derived in part from direct liquefaction of Illinois No. 6 coal, and submitted the sample to AFRL for evaluation. The sample was confirmed by AFRL to be in compliance with all U.S. Air Force-prescribed MIL-DTL-83133F-derived alternative aviation fuel initial screening criteria.

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SUBTASK 3.9 – DIRECT COAL LIQUEFACTION PROCESS DEVELOPMENT

INTRODUCTION

The Energy and Environmental Research Center (EERC), in partnership with the U.S. Department of Energy (DOE) and Accelergy Corporation, an advanced fuels developer with technologies exclusively licensed from ExxonMobil, undertook Subtask 3.9 to design, build, and preliminarily operate a bench-scale direct coal liquefaction (DCL) system capable of converting pulverized, dried coal to a liquid suitable for upgrading to fuels and/or chemicals. The primary objectives of Subtask 3.9 were to:

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DCL PROCESS OVERVIEW

DCL refers to a process by which coal is directly converted—via an essentially one-step process—to a liquid. Subsequent distillation and hydroprocessing is typically required to fractionate the raw liquid and convert its “naphtha–middle distillate” fraction to fuel and/or chemical products. DCL processes typically utilize ground (to about −100 mesh) and dried (to about 2% moisture content) coal and operate at temperatures and pressures of about 400°–500°C and 2000–3000 psi, respectively, in the presence of hydrogen, solvent, and a liquefaction catalyst. A “start-up” solvent (typically a coal-derived creosote liquid or anthracene oil) is needed to initiate liquefaction. DCL process outputs include a gas product comprising about 10% of dried coal input and a raw liquid product comprising about 90% of dried coal input. The raw liquid product is distilled to yield naphtha and middle distillate products for hydroprocessing-based upgrading, a heavy oil for recycle, and a tar-like “vacuum bottoms” material (which includes unreacted and partially reacted coal, ash, and catalyst) for recycle.

DCL PROCESS AND SYSTEM DESCRIPTION

The EERC–Accelergy DCL system is designed to process 45 pounds/day of coal. The system is installed on the first and second floors of the EERC National Center for Hydrogen Technology (NCHT) building, which is equipped with infrastructure necessary for safe and efficient operation including fire alarm and suppression systems and a hazardous gas alarm, ventilation system, gaseous emissions flare/combustion system, and nitrogen compressor. The intended uses of the DCL system are to 1) generally validate results of bench-scale batch-process DCL tests conducted to convert coal to liquids suitable for upgrading to jet and other transportation fuels, and 2) produce raw coal liquids for upgrading to large (up to 25-gallon) fuel samples for evaluation by AFRL. The DCL system is not intended to generate material balance or process scale-up data. The system will operate in a semicontinuous mode and comprises two primary subsystems:

1. The reactor system, comprising the primary process equipment needed to conduct liquefaction operations.

2. The off-line distillation system, installed in a large walk-in hood located in a laboratory near the reactor system. The distillation system is used to fractionally distill the raw coal liquids into a naphtha–middle distillate stream for upgrading (to finished fuel and/or chemical products) and a vacuum gas oil (VGO) solvent for recycle to the reactor system.

Figure 1 illustrates the key unit operations and input/output streams of the DCL process. In using the DCL system to produce a raw coal liquid sample from a target coal, initial processing is needed to prepare target coal-derived VGO and vacuum bottoms materials.

Vacuum bottoms material (referred to in Figure 1 as “bottoms”) is generated in the main reactor system and VGO (referred to in Figure 1 as “recycle solvent”) is recovered using the off-line distillation system. For process initiation, a start-up slurry consisting of target coal, catalyst, and VGO derived from distillation of a procured coal tar will be premixed in a slurry mixing tank and then transferred to a slurry feed tank. The start-up slurry will then be fed into the tubular reactor at a predetermined rate. The resulting slurry product will be stripped to remove volatile products. The volatile products will be condensed to form liquid using a series of condensers, and remaining slurry (vacuum bottoms) will be collected and transferred to a freezer for storage. The condensed raw coal liquids that are collected (on a 6-hour basis) will be transferred to the off-line distillation system and distilled to yield about 4.2 liters of VGO and 3 liters of distillate comprising naphtha, water, and a “middle distillate cut” (which will necessarily contain a significant amount of start-up slurry-derived coal tar distillate). The VGO obtained from distillation (along with the previously collected vacuum bottoms) will be used for recycle. The purpose of the initial processing is to generate enough target coal-derived vacuum bottoms and VGO to start the liquefaction process with 100% target coal-derived inputs, after which the process will be self-sustaining in production of 100% target coal-derived raw liquids. After sufficient amounts of vacuum bottoms and VGO needed to initiate the liquefaction process are produced, further processing will be conducted to produce target coal-derived middle distillate for upgrading to jet fuel or other fuels. Middle distillate upgrading will be conducted in a separate EERC hydroprocessing facility.

The liquefaction process will utilize predried (1%–2% moisture) coal and vacuum bottoms, both ground to −100-mesh-size particles. During sample production liquefaction operations, coal, vacuum bottoms, VGO, and catalyst will be fed to the slurry tank. The overall process involves feeding slurry consisting of an approximate 1/1/1 mixture of coal (2 pounds [lb]), coal-

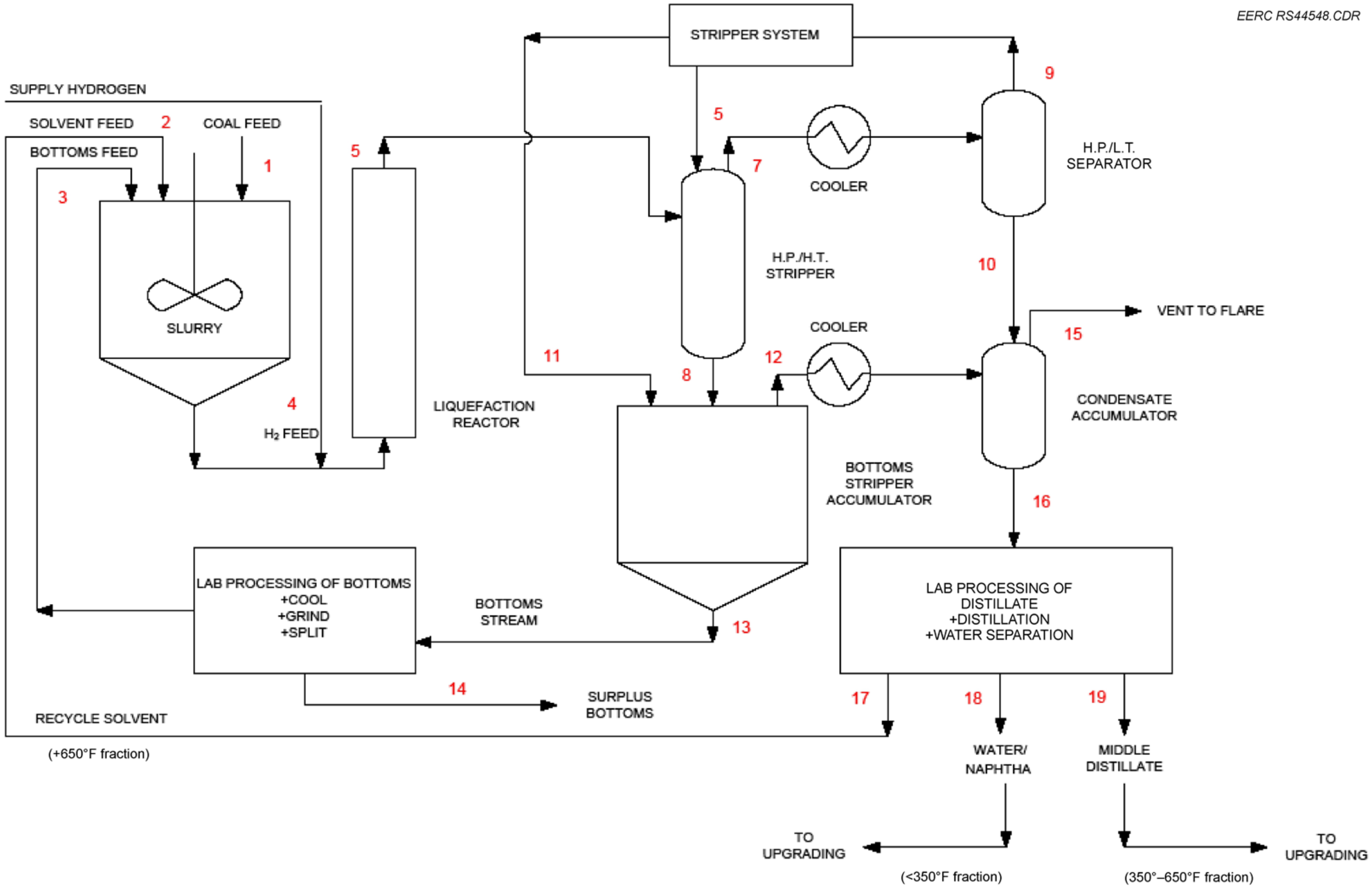

Figure 1. DCL process schematic illustrating unit operations and process streams.

derived VGO solvent (1.8 lb), vacuum bottoms (1.8 lb) and 100–300 parts per million (ppm) catalyst per hour through a tubular reactor system heated in a fluidized sand bath. A constant pressure of 2500 psi will be maintained through use of a pressure control valve and constant flow of hydrogen. Standard DCL system operating conditions are:

- Temperature – 450°C
- Pressure – 2500 psi
- Residence time – 60 minutes

These operating conditions can be varied depending on the composition and reactivity of the material that will be processed. However, the maximum allowable temperature and pressure for the system are 500°C and 2500 psig, respectively. The desired operating temperature can be maintained with automated controls. The 60-minute residence time is based on a 2-liter/hour (L/hr) flow rate with six 43-inch-long reactor tubes installed. This residence time can be changed by either adding or subtracting tubes or by changing the overall flow rate of the system. The pressure of the system is also variable if needed and could be increased to up to 2750 psig. The DCL process will use 2 lb of predried coal an hour. The solvent VGO and vacuum bottoms will be recycled. The amounts and compositions of the products will be dependent on the coal used. The DCL system will produce the following products:

1. Gases: The gas yield will be about 0.15 lb/hr or 8 wt% of dry ash-free (daf) coal. The gases produced will include H_2, H_2S, NH_3, CO, CO_2, methane, ethane, propane, and butane. The gases will be passed through a scrubber to remove corrosives, and the scrubbed gases will be routed to a flare system for destruction.

2. Liquids: Two types of liquids—primarily aqueous and organic (oils) will be produced by the DCL system:

    - Aqueous products (0.18 lb/hr or 10 wt% of daf coal added): This fraction will contain small amounts of alcohols, ketones, phenols, mercaptans, and hydrocarbons dissolved in water. Therefore, wastewaters are potential hazards and need to be disposed of according to U.S. Environmental Protection Agency guidelines.

    - Oils: Oils will be taken to a semiautomatic off-line distillation system for fractional distillation to produce the following fractions:

        - Naphtha (room temperature to 350°F; 0.15 lb/hr or 8 wt% of daf coal used) to be reformed or upgraded to produce a gasoline blendstock or chemical feedstock.

        - Middle distillate (350°–650°F; 0.78 lb/hr or 44 wt% of the daf coal added) to be upgraded to produce jet and/or diesel fuels.

        - Heavy oil (>650°F) to be recycled.

Oil distillates will be stored in a freezer until they are ready for upgrading to finished fuels. The finished fuels will be packaged under nitrogen and stored in a freezer.

- Solids (30 wt% of the daf coal added): The amount of vacuum bottoms produced is expected to be 0.54 lb/hr. Since the ash contained in the coal will also appear in the vacuum bottoms, the actual total amount of bottoms is expected to be 0.75 pounds an hour. The tarlike vacuum bottoms product, which comprises the residue remaining after stripping of the liquefaction slurry (with a boiling point of >1000°F), will be recycled. The solids will be stored in a freezer until recycled.

In summary, the liquefaction slurry produced by the DCL unit will be continuously stripped to drive off most of the volatiles. The remaining slurry will be continuously siphoned off as tar and collected in collection vessels and stored in a freezer. The frozen bottoms will be pulverized to –100-mesh size and transferred into the mixing tank for recycle. The volatile products will be carried through a series of condensers and the condensate (distillate) will be collected in collection vessels for transfer to the off-line distillation system. The condensate will be periodically batch-distilled to recover the VGO for transfer to the slurry mix tank and recycle. Naphtha and middle distillate will be stored in a freezer prior to undergoing off-line upgrading and/or other processing. The uncondensed gases will be sent to a flare system for combustive destruction. The gaseous, liquid, and solid products formed during liquefaction are potentially hazardous and must be handled, stored, and disposed of according to published city and state regulations. All personnel designated to handle these products will be appropriately trained. The DCL system will be equipped with an emergency medical kit, eye wash stations, and a safety shower.

DCL SYSTEM DESIGN/FABRICATION TIME LINE

The EERC DCL system concept and initial design (comprising a preliminary process flow diagram, process stream descriptions, and overall material balance) were developed in collaboration with Accelergy prior to initiation of Subtask 3.9. Initial Subtask 3.9 activities focused on DCL design completion and improvement, and definitively establishing the magnitude and cost of EERC infrastructure improvements required to ensure safe and efficient DCL system operation. As the subtask progressed, work emphasis shifted to procurement of equipment and supplies and fabrication of individual unit operations in preparation for their insertion into an integrated system. Although the subtask experienced an unanticipated and schedule-impacting change in installation location because of higher-than-projected infrastructure improvement costs for the originally selected location, the ability to maintain unit operation design and fabrication activities throughout this challenge enabled a minimal impact on project cost, and fabrication and installation of the DCL system (and accompanying off-line distillation system) were completed within budget. Subtask activities were carried out over the period of April 2010 to May 2012. Key milestones achieved during each quarter of the 26-month effort are described below.

April–June 2010

- The DCL system design was significantly improved by incorporation of a unit operation that enables online stripping of volatiles from raw DCL coal liquids, thereby reducing off-line distillation requirements.

- Size and performance specifications and bid requests for major DCL equipment pieces were developed.

July–September 2010

- All major equipment pieces and fabrication supplies for the DCL reactor and ancillary systems were ordered.

- Based on a detailed engineering assessment conducted in collaboration with a qualified and highly recommended commercial architectural and engineering firm, specifications were developed for the infrastructure improvements needed to support DCL system operation in the originally selected EERC location, and bid requests for making the improvements were provided to qualified contractors.

October–December 2010

- Contractor bids for making the structural, mechanical, and electrical improvements required for installation of the DCL reactor system in the originally selected EERC location were received and reviewed. The bids showed that the total cost of making the necessary improvements was significantly higher than estimated. Because the cost was also significantly beyond what the project budget could support, a decision was made to evaluate alternative locations for installation of the DCL system.

January–March 2011

- Based on an evaluation of alternative locations for installing the DCL system, a decision was made to skid-mount the system, with the goal of eventually moving it into a new building being constructed at the EERC. Skid-mounting was deemed the best solution at this time, since it would enable completion of system fabrication and possible seasonal (May–September) operation of the system in a semioutdoor location, if operation was needed prior to completion of the new building.

- An improved set of DCL system process and instrumentation drawings (P&IDs) was prepared based on a breakdown of the overall system into the four major process areas described below:

  - Area 100 – gas supply and delivery
  - Area 200 – feed slurry and solvent preparation and delivery
  - Area 300 – preheaters, sand bath heater, and reactors
  - Area 400 – product separation and collection vessels and product gas cleanup and recycle system

April–June 2011

- Because of unexpected developments in projects ongoing in the NCHT building, a two-level site in the NCHT building was secured for permanent installation of the DCL reactor system. The NCHT site is equipped with the fire alarm, fire suppression, ventilation, and gaseous emission combustion/flare systems needed for safe and efficient DCL system installation and operation.

- A two-level layout for the DCL system was developed, and system installation in the NCHT building was initiated.

- The EERC worked closely with an Accelergy-retained consultant team to help expedite the transition of the DCL system design from a skid-mounted to a permanent installation design and to help ensure that the design will translate to a safe, efficient, and easily operable and maintainable DCL system. Key activities included detailed review and modification—as recommended by the Accelergy team—of P&IDs, equipment specification lists, and process flow diagrams. Because of the design and layout adjustment required in transitioning from a skid-mounted to a permanently installed DCL system configuration, a no-cost project schedule extension request was made to the DOE. The request was graciously granted.

July–September 2011

- The 2-level DCL system layout for installation in the NCHT building was finalized, as were a complete set of P&IDs based on the new system layout.

- With on-site assistance from Accelergy-retained consultants, a comprehensive hazardous operations (HAZOP) assessment of the overall DCL reactor system was conducted. The assessment included the off-line batch distillation system that will be used to separate raw coal liquids into product and recycle streams. Nine EERC staff and two Accelergy-retained consultants participated in the assessment. Many DCL system safety and operability concerns were raised and discussed. Appendix A comprises a list of action items generated as a result of the HAZOP assessment and includes a description of steps taken to address safety and operability issues. Appendix A also contains a list of valve failure response actions. Two major concerns that came out of the HAZOP are described below:

    1. The DCL system hydrogen compressor was originally sited almost directly below the reactor system, which represented an unacceptably high risk of ignition/explosion of hydrogen (from a possible compressor leak) by its potential contact with hot reactor system surfaces. This issue was resolved by eliminating the hydrogen compressor and replacing it with a remotely sited high-pressure hydrogen supply.

    2. A thermal oxidizer (utilized by several EERC gasification projects ongoing in the NCHT building) was sited sufficiently close to the DCL reactor system to represent an unacceptably high hydrogen ignition/explosion risk because of the potential for possibly leaking hydrogen (from the reactor system) to contact an exposed hot surface of the thermal oxidizer. This issue was resolved by instituting a policy that calls for turning off the thermal oxidizer and cutting off (and locking out access to) its natural gas fuel supply during all DCL system runs.

October–December 2011

- The bid process for selecting the automated off-line distillation system needed for raw coal liquids fractionation and the upgrading of product fractions was completed, and a system was selected and ordered.

- Arrangements were made with the Stella-Jones Corporation to deliver to the EERC (by February 2012) a coal-derived solvent, RT-12, for use as a DCL system start-up solvent.

- Preparation of a comprehensive DCL system operations manual and a procedural manual for conducting the fractional distillation of raw coal liquids into recycle and product streams was initiated.

January–March 2012

- A DCL system operations manual and a distillation procedures manual were completed; copies are provided as Appendixes B and C, respectively.

April–June 2012

- Fabrication and installation of the DCL reactor system was completed. Figures 2–7 are photographs of the installed system.

- Fabrication and installation of the off-line distillation system was completed. Figure 8 is a photograph of the installed system.

SHAKEDOWN OF OFF-LINE DISTILLATION SYSTEM

Although the DCL system is designed to enable continuous-mode operation, it is not equipped with an online distillation capability for real-time fractionation of raw coal liquids into product and recycle streams. This means that the raw coal liquids must be recovered and removed from the DCL, manually transferred to an off-line batch distillation system, and fractionated into product and recycle streams, after which the product stream (naphtha plus middle distillate) is transferred to an upgrading process for conversion to fuel and/or chemical products, and the recycle fraction VGO is transferred back to the DCL system for recycle. It is anticipated that approximately 1 liter of VGO an hour will be needed to sustain continuous operation of the DCL system. Shakedown of the newly installed distillation system was performed using two surrogate solutions: one representative of raw coal liquids and one representative of a middle distillate upgrading product from which diesel, jet fuel, and naphtha products could be distilled. The objective of the shakedown was to optimize the heat input rate needed to attain the shortest-duration distillation time required to fractionate the raw coal liquid or fuel-grade mixture into the desired distillation cuts. Details of the shakedown are as follows:

1. Shakedown with surrogate raw coal liquids – A 6-liter sample of raw coal liquids surrogate was prepared by mixing 8 vol% water with 8, 30, and 54 vol% of previously produced coal liquids boiling in the naphtha (<300°F), middle distillate (300°–600°F) and vacuum gas oil (600°–1000°F) ranges, respectively. The distillation was repeated

several times by using spinning band and packed columns to define the operating conditions that will yield the following fractions/distillation cuts:

- Light (naphtha) fraction (<300°F)
- Middle distillate fraction (300°–600°F)
- Heavy VGO fraction (>600°F)

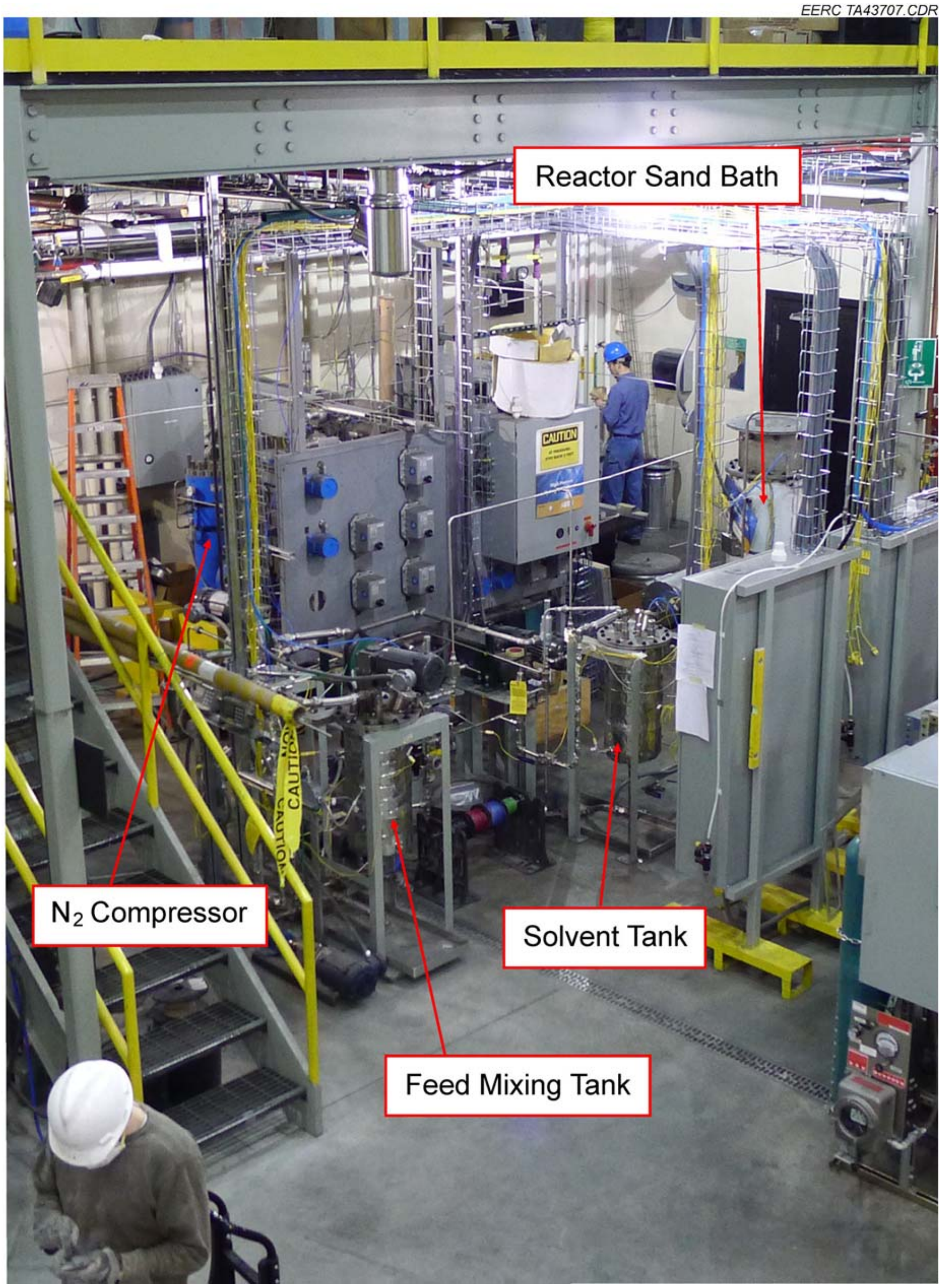

Figure 2. First-floor view of DCL reactor system.

EERC TA43708.CDR

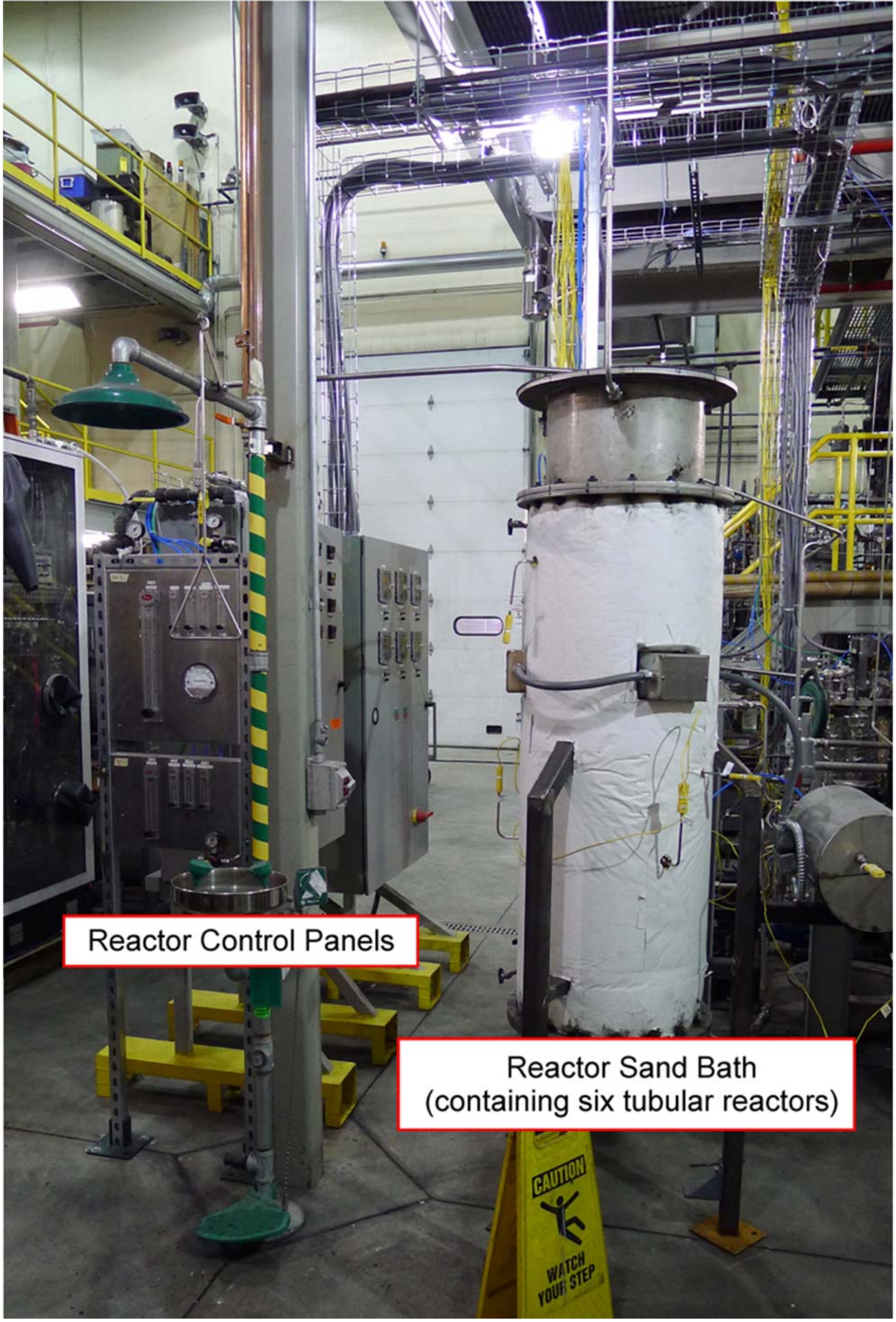

Figure 3. Close-up of DCL reactor sand bath.

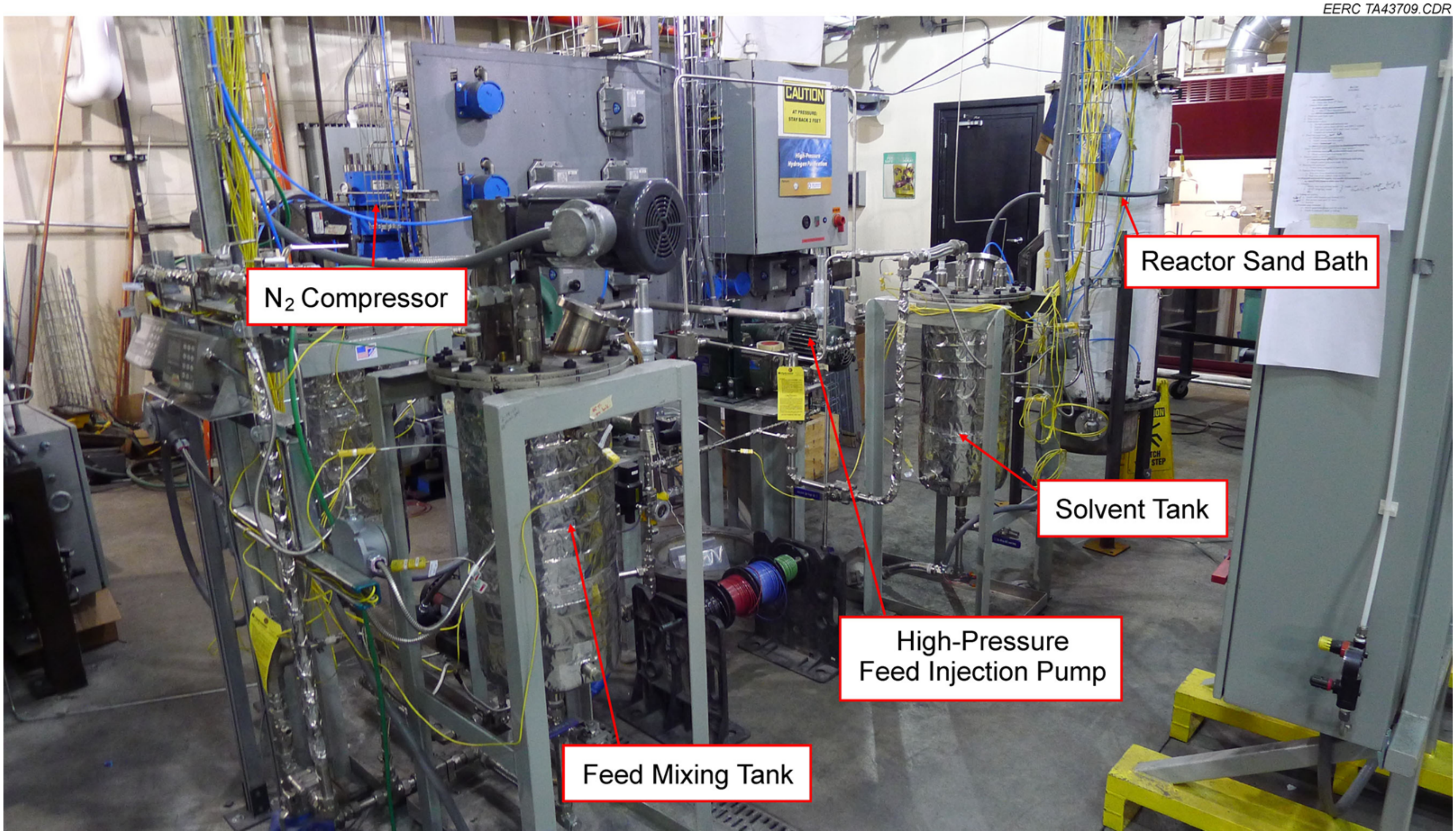

Figure 4. Close-up of prereactor feed preparation and injection systems.

Figure 5. Nitrogen compressor system.

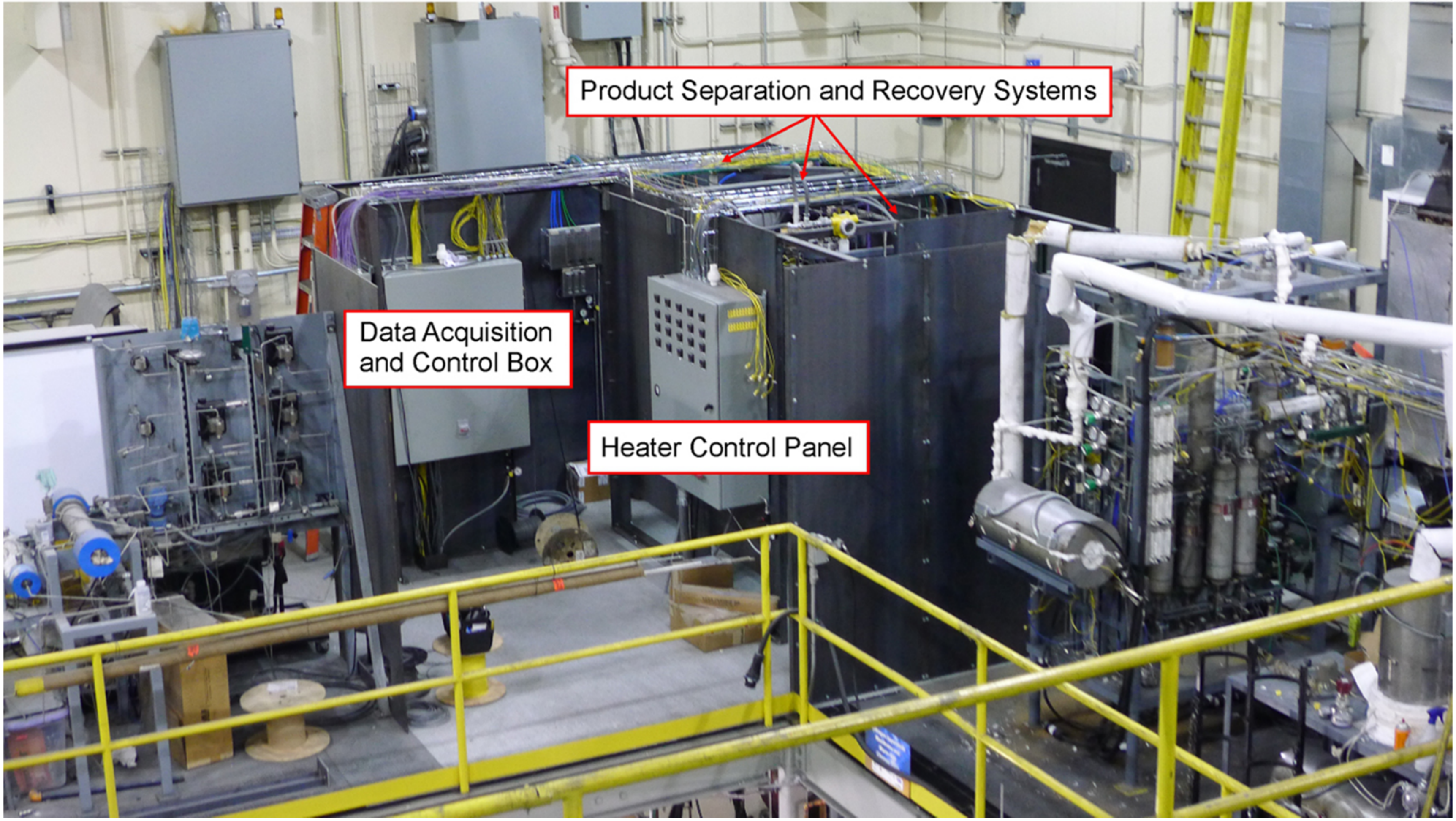
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Figure 6. Second-floor view of DCL reactor system.

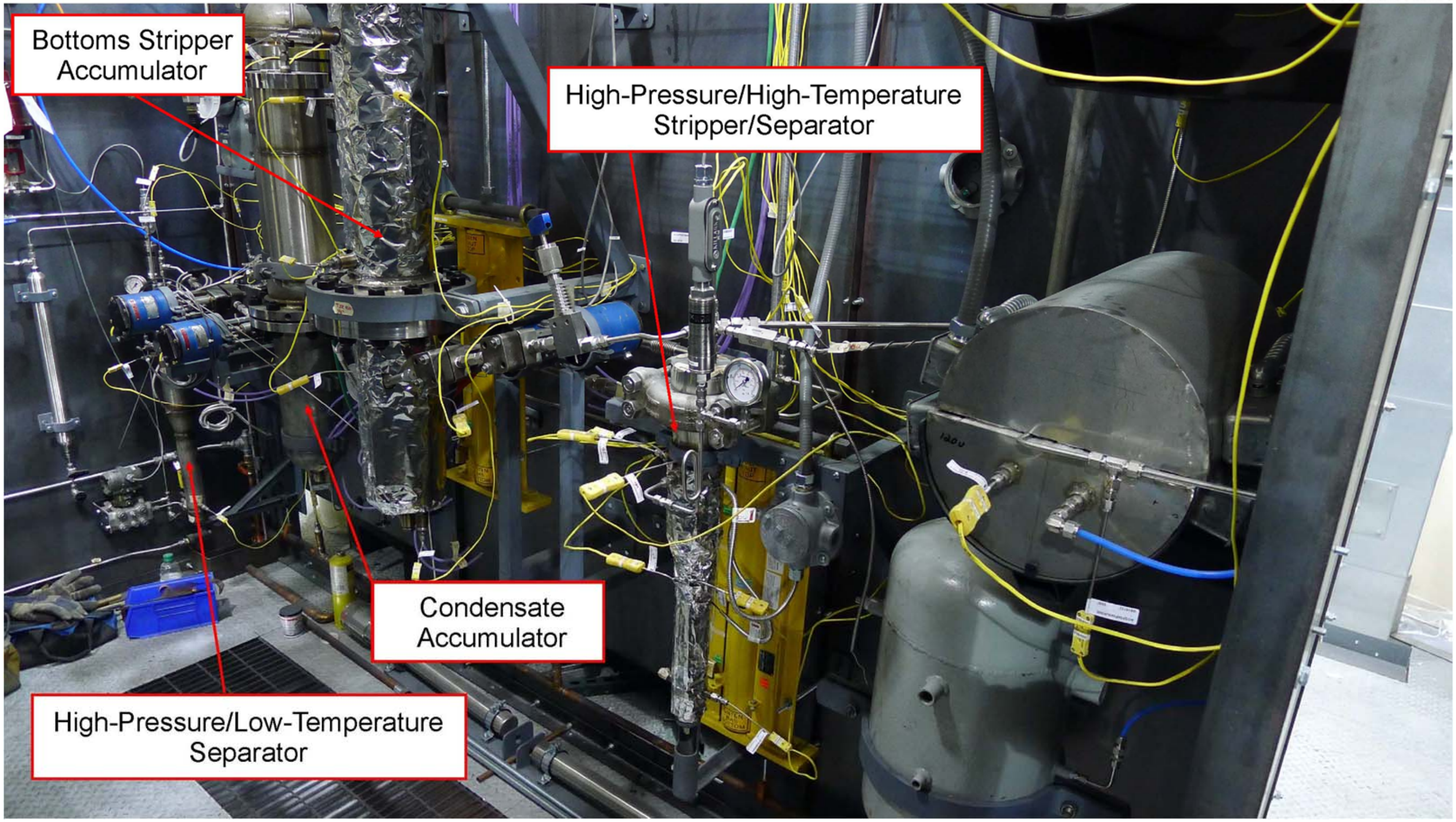

Figure 7. Close-up of product separation and accumulation systems.

EERC TA44554.CDR

Figure 8. Off-line distillation system.

2. Shakedown with surrogate middle distillate upgrading product – A 6-liter mixture of normal paraffins, isoparaffins, cycloparaffins, and aromatics representative of a likely middle distillate upgrading product was prepared. The mixture comprised about 2 vol% water, 5 vol% naphtha, 63 vol% commercial jet fuel, and 30% diesel fuel. As before, the distillation was repeated several times by using spinning band and packed columns to define the operating conditions that will yield the following distillation cuts:

    - Naphtha fraction (<300°F)
    - Jet fraction (300°–500°F)
    - Diesel fraction (>500°F)

JET FUEL SAMPLE PRODUCTION

In order to provide a 500-milliliter coal-derived jet fuel sample to AFRL for evaluation prior to completion of DCL system fabrication, a batch autoclave coal liquefaction system was utilized to convert Illinois No. 6 coal to a raw coal liquid, which was then distilled to yield a middle distillate cut that was upgraded (via hydroprocessing) to a jet fuel blendstock. Figures 9 and 10 are a schematic and photo, respectively, of the batch autoclave system used to produce the raw coal liquids. Appendix D describes the procedures used to produce the coal-derived jet fuel blendstock. Because the U.S. Air Force has been directed to evaluate options for accessing petroleum-alternative fuels with carbon footprints that are equal to or smaller than those of petroleum-derived fuels, a decision was made to mix the coal-derived cycloparaffin-rich and aromatics-containing jet fuel blendstock with an isoparaffin-rich and normal paraffin-containing jet fuel blendstock generated via hydroprocessing of canola oil, at a blend ratio of 60% coal-derived material–40% renewable oil-derived material. The resulting sample was delivered to AFRL, where it was analyzed and found to comply with all AFRL JP-8 jet fuel screening criteria.

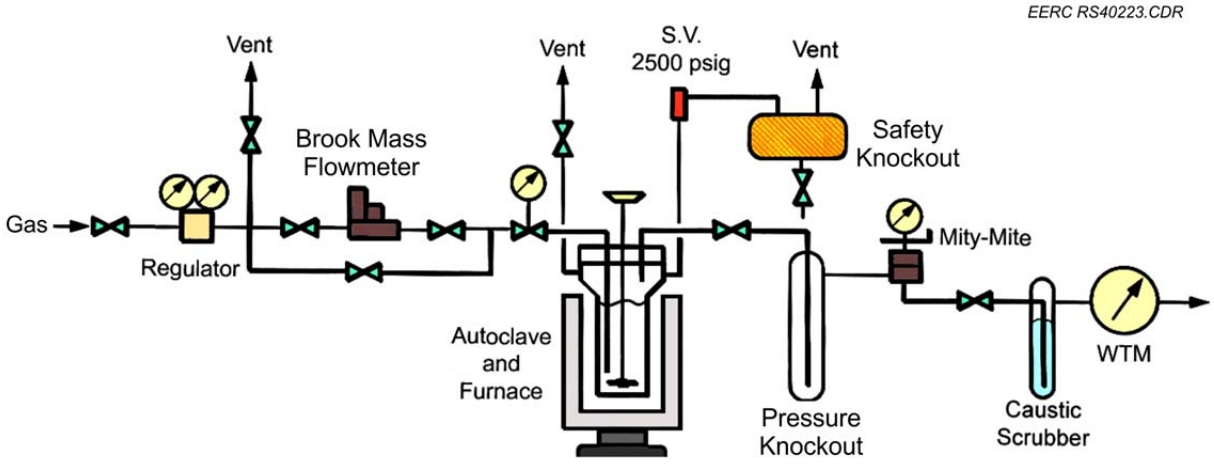

Figure 9. Schematic of 2-gallon batch autoclave system.

Figure 10. Photo of 2-gallon batch autoclave system.

PLANS FOR THE DCL SYSTEM

DCL system shakedown activities were initiated in the second half of July 2012 (see Appendix E for an overview of shakedown procedures), after which plans call for validation of DCL system performance by using the system to generate at least 1 liter of steady-state-produced raw coal liquids from Illinois No. 6 coal. System performance will be validated if the coal throughput achieved and the chemical and physical properties of the raw coal liquids generated match those achieved and generated in Exxon-conducted DCL tests with Illinois No. 6 coal (the data for which are in the possession of Accelergy). Following validation with Illinois No. 6, the DCL system will be used to generate raw coal liquids from a Montana subbituminous coal in sufficient quantity to yield 2 liters of middle distillate for upgrading to a jet fuel sample, which will be delivered to AFRL for evaluation. Plans then call for use of the DCL system to produce several gallons of lignite coal-derived raw coal liquids, which will be delivered to an oil refinery for evaluation as a refinery input. The order of these three process operations (bituminous coal first, followed by subbituminous, then lignite) is necessary to ensure the best potential for successful operation with each coal, because Exxon/Accelergy experience has shown that bituminous coals are easiest to process, subbituminous coals are somewhat more challenging, and lignites are most difficult.

APPENDIX A

DCL SYSTEM HAZOP REVIEW STATUS AS OF 25 JULY 2012

NOMENCLATURE

ALARA	as low as reasonably achievable
ATC	air-to-close
ATO	air-to-open
AV	automated valve
BSA	bottoms stripper accumulator
CA	condensate accumulator
CV	check valve
DAQ	data acquisition
DCL	direct coal liquefaction
DOT	Department of Transportation
EERC	Energy & Environmental Research Center
ESA	electrical swing adsorption
FCV	fuel cell vehicle
FB	fluid-bed
FIC	flow indicator/controller
HP	high-pressure
HP/HT	high-pressure/high-temperature
HP/LT	high-pressure/low-temperature
HV	hand valve
HX	heat exchanger
LIC	level indicator/controller
LP	low pressure
MAWP	maximum allowable working pressure
MFC	mass flow controller
MOP	maximum operating pressure
MSDS	material safety data sheet
MV	manual valve
MWP	maximum working pressure
NCHT	National Center for Hydrogen Technology
OD	optical density
PC	pulverized coal
P&ID	piping and instrumentation diagrams
PIR/PI	pressure indicator recorder/pressure indicator
PPE	personal protection equipment
PSV	pressure safety valve
PVC	pressure valve control
RV	relief valve
SOP	standard operating procedure
SP	set point
TC	thermocouple
TEFC	totally enclosed fan cooled
UND	University of North Dakota
VFD	variable frequency drives
VFO	variable frequency oscillator

DCL SYSTEM HAZOP REVIEW STATUS AS OF 25 JULY 2012

9/13/11 Action Items Area 100H

- Disconnect gas analyzer from H_2 comp.
- Drain on bottom of vent line – stack pipe in pipe.
- Decide on H_2 accumulator location.
- Determine location of H_2 six-pack – review criteria.
- Label exactly how H_2 connects to DCL system – verify safety valves RV-6 RV-7.
- Add relief pressures on P&IDs.
- Shutdown/start-up and operating logic to P&ID.
- Possibly add all details on compressor such as manufacturer maintenance procedure.
- Volume release of H_2 and/or H_2 accumulation.

9/13/11 Area 100N Action Items

- Verify all electrical classification.
- Hydro test accumulators or pressure test according to DOT testing.
- Verify how we do all of our reliefs; can't use thermal oxidizer as a safety device.

9/13/11 Area 100 Action Items

- Develop drawing showing how MFC will be implemented.
- SOP needs to address what we will do in case of loss of house N_2 to our purge for control boxes.
- Delete burst pressure from drawing legend; change MWP to MOP or MAWP.
- Add another check valve after CV-114.
- Decide whether or not we need to double valve or add a bleed line on PIR/PI isolation valves.
- Possibly put seal on all valves to purged vent valve safety valves – PSV-104 and PSV-112.
- Evaluate possible N_2 leak and raising N_2 level in vicinity.

9/13/11 Area 200

- Change slurry mix tank temperature to 300°F.
- Determine operating pressure of product tanks – would be advisable to be 50 psi.
- Add N_2 purge to all tank vents lines/blanket tanks.
- Add that the Viking pumps have VFDs.
- Check to see if we can get Group B motors that are VFO rated.
- Verify that the motors have no arcing or sparking.
- Possibly provide a flush option for Viking pumps.
- Add flow direction on cooling water on each tank condenser (change valves to rotometers).
- Consider adding external relief on the three Viking gear pumps or see if we can remove and check the internal reliefs.
- If possible, display more detail such as flex connection, show scales.

- Add all design temps and press on P&IDs.
- Address coal moisture in the process.

9/13/11 Area 300

- Slurry preheater – need to edit and change description.
- Change where we mix the H_2 with the slurry before the sand bath– possibly before preheat.
- Possibly move or eliminate PSV302 because of the change in the H_2 addition – need to discuss this design change – all reactor components designed higher than everything else.
- Determine the appropriate location for the sand bathheater (add TC) in exit pipe/determine proper size of exit.
- Put air usage of sand bath on P&ID.
- Change sand bath to N_2 instead of air for fluidization.
- Low-flow switch that will turn off heaters in sandbath.
- Verify proper labeling of MV-304 and FIC-302.
- Check all check valve symbols and make sure they are correct.
- Consider changing our manifold design using sintered metal thimbles.

9/13/11 Area 400 High Pressure/400 Low Pressure Combined

- Review need for reliefs on vessels in 400 HP P&ID.
- Heat exchangers – sublimation of ammonia and chlorides need to have a capability for water injection at the front of HX-406.
- Evaluate potential for H_2 embrittlement in HX-406 and HX-408.
- Consider switching our sulfur scrubbers to 400 low pressure after condensate accumulator before thermal oxidizer.
- Develop a procedure for operating with our nuclear sources open.
- Wayne suggests we eliminate Valve 406 on 400LP and put AV-410 as close as possible to bottom of bottoms stripper accumulator.
- Look at process of grinding bottoms.

9/14/11 Risk Assessment Process

- Comments by Wayne and Dick (Accelergy consultants) about layout confined space – operators in the immediate area layout – is there enough room for maintenance and operator routine activities.
- H_2 compressor – they would like it moved.
- Dick: Move the compressor – concerned with the location of the thermal oxidizer, concerned with its system operations – he is also concerned with our electrical equip. classification.
- Evaluate location of compressor.
- Evaluate partitions – work space.
- Adequate ventilation?
- Reevaluate flame jet ignition – flow restrictions.
- Add drip-pans and liquid containment.

- Evaluate potential of light gases being trapped under solid floor decking.
- Evaluate benefits of using H_2 six-packs only and no H_2 compressor.

9/14/11 Risk Assessment Cont. New Action Items Area 100

- Dick's comments:
  - 100 – Add valve failure action as a component, such as what happens with loss of air pressure.
  - Evaluate whether we should add an E-stop or use a surge flow device to control automated shutoff.
  - Shutdown matrix – needs to be developed.
  - Wayne would like us to add a tubing fitting failure section.
  - Dick also thinks fitting failure also needs to be addressed for possible under pressure for large leak: how do we address it – safety valve – flow restriction; use check valves in every possible situation that we can.

9/14/11 Area 200 Comments Action Items

- Could add a rupture disc before safety relief valve on HP pumps.

Area 300 Comments Action Items

- Mike Collings concerned about our temp of H_2 preheaters – incorporate a low-flow cut off on the heaters (preheaters).
- How we will deal with plugged lines.
- Separate stack for sandbath.

9/14/11 Area 400 Comments Action Items

- Reposition CV-412 as close to vessel as possible.
- Consider putting a check valve between HP/HT separator/stripper and HP/LT separator.
- Remember to change length of dip tube in HP/LT separator.
- Eliminate PSV-406; PSV-402; PSV-404 in 400 HP area drawing.

9/14/11 Reviewing Dick's HAZOP Outline "Action Items"

- Establish program to annually check safety valves.
- Good housekeeping.
- Identify egress – keep clear.
- Proper PPE, MSDS – "Be specific in PPE."
- Concerns with explosive hazards – hot surfaces.
- H_2S exposure and how to detect.
- Safety showers and eye washes – locate with thought.
- Stress corrosion and cracking – corrosive materials.
- Restriction on flex hoses – stainless braided type.

- Radiation sources – controlling and training procedures for working around – signage.
- Second-floor eye wash/shower station.
- Helium sniffer – for leak checking.
- Air quality control permit – SO_2, H_2S, CO_2, NO_2.
- Hazardous waste – types and quantities.
- Control in crushing bottoms, keep dust and contamination at a minimum.
- Proper training of personnel before opening any valve or breaking apart fittings – use of proper PPE – gloves, face shield, respirator.

P&ID ACTION ITEMS

Area	Action Item	Person Responsible	Action Taken	Date
100 H	Drain on bottom of vent line – stack pipe in pipe	Dusty/Mark	There is a drain on the bottom of the stack going out the roof. An additional drain valve has been installed at the bottom of the vent line section running from the first up to the second floor.	11/1/2011
100 H	Decide on H_2 accumulator location	Mark/Dusty	There will be no storage of H_2 in pressurized cylinders or vessels within the immediate vicinity of the Accelergy system or below heated vessels on the second deck.	5/28/2012
100 H	Determine location of H_2 six-pack – review criteria	Mark	A location for permanent placement of six-packs is being assessed. Preliminary plans for temporary storage include placement of up to 2 six-packs east of the NCHT building. Stainless steel tubing (1/4-inch OD) will connect the six-packs to the Accelergy process through a distance of approximately 60 feet. The H_2 pressure will be regulated at the six-packs to the pressure required for the Accelergy process.	5/28/2012
100 H	Label exactly how H_2 connects to DCL system	Mark	Changed on drawing Accelergy-v11.vsd.	5/28/2012
100 H	Verify safety valves RV-6 RV-7 and determine if can be reset for lower pressure	Dusty/Mark	Can only be factory set (Autoclave Engineers), currently set to 14,500 psi; a new safety relief valve (PSV-116) with a 6000 psi relief set point has been installed to protect against over pressurization of the H_2 accumulators.	3/1/2012
100 H	Add relief pressures on P&IDs	Dusty	Relief pressures to be updated on Accelergy-v7.vsd (RV1 = 1500 psi, RV2 = 400 psi, RV3 = 14,500 psi, RV6 = 14500 psi, RV160 = 600 psi, RV130 = 600 psi).	10/17/2011
100 H	Shutdown/start-up and operating logic to P&ID	Dusty	N_2 compressor start-up can be taken from ESA start-up procedure. P&ID can include two manual valves for the compressor oil bypass that are used for start-up, along with a valve for water supply.	10/7/2011
100 H	Add all details on N_2 compressor such as manufacturer maintenance procedure	Dusty	N_2 compressor is a Fluitron Diaphragm with leak detection. The oil must be changed every 500 hours or 6 months, and O-rings and/or metal diaphragms should be changed if oil is seen in the leak detection piping. It should be used at least every 6 months to keep lubrication. See compressor user guide for further operation.	10/7/2011
100 H	Volume release of H_2 and/or H_2 accumulation	Mark/Dusty	The equipment located in the upstairs barricade is of such a small volume, less than 8-scfh per vessel, that a localized flammable formation will be short-lived. The barricades provide protection from a jet flame for those outside the barricade, and multiple egress locations allow workers inside to vacate. The key concern in this location is ventilation, and it is proposed that an “elephant trunk” will be used to add local ventilation during sample taking to limit vapors and flammables gases in the region. The south barricade wall will also be removed to allow for better access and air flow, as the hydrogen equipment will be located near the north wall barricade. The flooring directly under the hydrogen filled equipment will be replaced with grating and a liquid trap installed further underneath, allowing airflow from 1st floor up and past the equipment while still protecting from liquid spills. The placement behind the barricade should allow adequate ventilation without enough confinement for an explosion hazard, but still keep the risk of the system within range as determined in the risk assessment. Workers inside the barricade will be exposed to a higher risk but is taken upon by project employees familiar with the system rather than the general worker population, and flame and gas detection will be installed to provide hazard notification.	9/23/2011

Continued . . .

P&ID ACTION ITEMS (continued)

Area	Action Item	Person Responsible	Action Taken	Date
100 N	Verify all electrical classification	Mark/Jim	Motors for N_2 compressor and high-pressure pumps were cleared for use by electrical inspector; thermostat (potential arcing device) in gear pump motors has been disconnected	11/1/2011
100 N	Hydrotest accumulators or pressure test according to DOT testing	Jim	These are certified tanks with a DOT pressure rating of 10,000 psi.	10/4/2011
100 N	Verify how we do all of our reliefs; can't use thermal oxidizer as a safety device	Mark	Clean, dry reliefs from H_2 and N_2 supply will be directed to the existing N_2-purged 1½-inch relief/vent line on the ESA system; 1-inch CS vent lines independently connect Knock-Out 1 and 2 to a new 2-inch dirty vent line; Knock-Out 1 serves the reliefs for the high-pressure slurry and solvent flush pumps; Knock-Out 2 serves the reliefs for the bottoms stripper accumulator and condensate accumulator.	3/1/2012
100	Develop drawing showing how MFC will be implemented	Mark	Added on drawing Accelergy-v7.vsd; mass flow controllers are contained in two N_2 purged Hoffman-type boxes; H_2 FCV's 102 and 104 are in one box and N_2 FCV 106 and a spare H_2 FCV are in a second N_2 purged box; N_2 purge flow to each box will be controlled through a 0 to 5 psig regulator, a rotameter on an outlet port will show positive flow out of the box; this method requires good sealing around penetrations and doors to maintain positive flow to the outlet rotameter.	9/23/2011
100	SOP needs to address what we will do in case of loss of House N_2/air to our purge for control boxes	Mark	Purges from house N_2 and house air will be used on DAQ, heater controller, VFD, and mass flow controller boxes; loss of purge will be signaled on annunciator box in control room; loss of purge will not shut down operation but will signal operator to investigate loss of purge.	9/19/2011
100	Delete burst pressure from drawing legend change MWP to MOP or MAWP	Mark	Changed on drawing Accelergy-v6.vsd; MOP set at 3000 psig, reference to burst pressure removed.	9/15/2011
100	Add another check valve after CV-114	Mark	Changed on drawing Accelergy-v6.vsd; CV-116 added after CV-114.	9/15/2011
100	Decide whether or not we need to double valve or add a bleed line on PIR/PI isolation valves	Jim	No PIR/PI process isolation valves are being used.	5/10/2011
100	Safety sealed all valves to purged vent; safety valves – PSV-104 and PSV-112 were sealed	Mark	Pressure safety valves 104 and 112 have been factory set at 3300 psig and are lead tag sealed to prevent tampering.	3/1/2012
100	Evaluate possible N_2 leak and raising N_2 level in vicinity	Mark/Dusty	The nitrogen compressor could create a localized area of low oxygen and become an asphyxiation risk, but the required leak size to allow this to occur would be large enough to be noticeable by any employee in the vicinity. Once again, ruptures are unlikely, and a leak letting enough nitrogen into the atmosphere is easily heard by workers, allowing them to take action. Because the gas is nonflammable and easily detected, it is recommended that the nitrogen compressor can remain indoors.	9/23/2011
200	Change slurry mix tank temp to 300°F	Mark	Changed on drawing Accelergy-v6.vsd; temperature changed from 400°F down to 300°F for slurry mix and slurry feed tanks.	9/15/2011
200	Determine operating pressure of product tanks – would be advisable to be 50 psi (if venting to thermal oxidizer)	Jim	The design drawings were reviewed by the EERC engineering team, and it was determined that the tanks were capable of withstanding 50 psi pressure.	3/15/2012

Continued . . .

P&ID ACTION ITEMS (continued)

Area	Action Item	Person Responsible	Action Taken	Date
200	Add N_2 purge to all tank vents lines/blanket tanks	Mark	Changed on drawing Accelergy-v6.vsd; N_2 purges added for slurry mix, slurry feed, and solvent flush tanks.	9/15/2011
200	Add that the Viking pumps have VFDs	Mark	Changed on drawing Accelergy-v6.vsd; VFD reference added to motor description	9/20/2011
200	Check to see if we can get Group B motors that are VFD rated	Mark	WEG offers Class 1, Div 2, Group B, C, D three-phase TEFC motor with Class F insulation, VFD rated.	9/20/2011
200	Verify that the motors have no arcing or sparking	Jim	Motors for the slurry/solvent gear pumps are Class 1 Div 1 Group C&D explosion proof motors with T3C (160 C) max surface temperature rating, the manufacturer indicated motors have thermostats that can be arcing but no brushes or switches, the thermostats have been disconnected; motors for the mixers are Class 1 Div 1 Group C&D hazardous location motors with T3C (160 C) max surface temperature rating, the manufacturer indicated that the motor is nonarcing or sparking.	11/1/2011
200	Provide a flush option for Viking pumps	Mark	Flush line shown as a future option; no flush supply pump currently available.	5/4/2012
200	Add flow direction on cooling water on each tank condenser (change valves to rotometers)	Mark	Changed on drawing Accelergy-v6.vsd for slurry mix tank; condensers removed from slurry feed and solvent flush tanks.	9/15/2011
200	Consider adding external relief on the 3 Viking gear pumps or see if we can remove and check the internal reliefs	Mark	Changed on drawing Accelergy-v6.vsd; external pressure safety valves PSV-206, -208, and –210 added for slurry mix, slurry feed, and solvent flush tank circulation pumps, respectively; relief set points are 100 psig for each valve.	9/15/2011
200	Display more detail such as flex connection, show scales	Mark	Changed on drawing Accelergy-v6.vsd; Flex Lines FL-202, -204, and -206 added to slurry mix tank and Flex Lines -208, -210, -212, and -214 added to slurry feed tank at gear pump inlets, recycle lines, and N_2 vent lines.	9/20/2011
200	Add all design temps and press on P&IDs	Mark	This is in progress and is expected to be complete before the system is operated.	Ongoing
200	Address coal moisture in the process	Ramesh	All input coals will be dried to 2%–3% moisture prior to charging, and the coal percentage in feed slurry will be based on 2%–3% moisture.	10/4/2011
300	Slurry preheater need to edit and change description	Mark	Slurry will be preheated (from 300°F up to 570°F) within the electrically heat-traced ½-inch OD, 0.083-inch wall tubing supplying slurry from the outlet of the HP slurry pump to the reactor inlet.	5/4/2012
300	Change where the H_2 is mixed with the slurry to before the sand bath– possibly before preheat	Mark	Changed on drawing Accelergy-v6.vsd; H_2 added to slurry just before entering sand bath vessel; will operate slurry and H_2 preheaters at 570°F.	9/16/2011
300	Move or eliminate PSV302 because of the change in the H_2 addition – need to discuss this design change – all reactor components designed higher than everything else	Mark	Changed on drawing Accelergy-v6.vsd; removed PSV-302; vessel and piping components have 3500 psig MAWP which is above maximum gas and slurry supply pressure.	9/16/2011
300	Determine the appropriate location for the sand bath heater (add TC) in exit pipe/determine proper size of exit	Jim	Sand bath heater will be located on the first (main) level.	
300	Put N_2 usage of sand bath on P&ID	Mark	Changed on drawing Accelergy-v6.vsd; N_2 usage approximately 360 scfh.	9/15/2011
300	Change sand bath to N_2 instead of air for fluidization	Mark	Changed on drawing Accelergy-v6.vsd; N_2 replacing air as fluidizing gas.	9/15/2011

Continued . . .

P&ID ACTION ITEMS (continued)

Area	Action Item	Person Responsible	Action Taken	Date
300	Low-flow switch that will turn off heaters in sand bath	Mark	Pressure transducer PIR-306 (placed upstream of the sand bath gas distributor) will function to indicate loss/reduced N_2 flow to the sand bath; a visual alarm on the computer control screen will alert the operator to turn heaters SPs down or off; future PC control scheme could have automated shutdown of heaters.	5/4/2012
300	Verify proper labeling of MV-304 and FIC-302	Mark	Changed on drawing Accelergy-v6.vsd; MV-304 removed; FIC-302 functions as flow control and rate display.	9/15/2011
300	Check all check valve symbols, and make sure they are correct	Mark	Changed on drawing Accelergy-v6.vsd; flow direction of check valve CV-304 corrected.	9/15/2011
300	Consider changing our manifold design using 1) sintered metal thimbles or 2) sintered metal grid above support plate with properly sized holes	Jim	The drain is on the bottom of the fluid-bed sand bath. Adding a sintered metal or similar screen will not allow bed sand to be drained. We will assess after short-term runs if penetration of sand into the manifold is an issue and if re-design is required.	5/10/2012
400	Review need for reliefs on vessels in 400 HP P&ID	Mark	Changed on drawing Accelergy-v6.vsd; removed pressure safety valves PSV-402 and -406; vessel and piping components have 3500 psig MAWP which is above maximum gas and slurry supply pressure.	9/16/2011
400	Heat exchangers – sublimation of ammonia and chlorides need to have a capability for water injection at the front of HX-406	Jim	This modification was done by adding a tee and high-pressure valve to give the capability for water injection.	11/23/2011
400	Evaluate potential for H_2 embrittlement in HX-406 and HX-408	Jim	We will do periodic checking of the critical areas of the system to ensure that no embrittlement is taking place. The materials used in the construction of the DCL are of the highest quality that was available to us. Also, our past experience with these materials in a H_2 atmosphere has shown them to be capable of the service.	Ongoing
400	Consider switching our sulfur scrubbers to 400 low pressure after condensate accumulator before thermal oxidizer	Mark	Changed on drawing Accelergy-v8.vsd; removed sulfur scrubber pressure vessels; current plan is destruction of process effluent gas in an existing flare.	5/4/2012
400	Develop a procedure for operating with our nuclear sources open	Mark	A radiation survey has been completed for closed and opened sources; operating personnel will be instructed in the location of sources and principle of ALARA for minimizing exposure while performing maintenance, taking samples, or doing field readings; area signage indicates the presence and locations of nuclear sources; the process area with the sources will also have markings on the decking to show areas where sustained work activities may require sources to be closed; discussion of operation with open nuclear sources will be included in the operations manual.	Ongoing
400	Eliminate Valve 406 on 400LP and put AV-410 as close as possible to bottom of bottoms stripper accumulator	Mark	Changed on drawing Accelergy-v6.vsd; moved automated Valves AV-410 and -412 to be close-coupled to bottom of the bottoms stripper accumulator.	9/15/2011
400	Look at process of grinding bottoms	Jim	Preliminary laboratory trials have shown that freezing the bottoms followed by grinding in a ball-media tumbling mill can produce the desired recycle feedstock.	Ongoing

Continued…. . .

RISK ASSESSMENT ACTION ITEMS

Area	Action Item	Person Responsible	Action Taken	Date
	Evaluate second-floor layout, and determine how to minimize confined space for operator safety and access for maintenance	Jim	Removed the south wall of the second-floor barricade and relocated all of the mix tanks and related pumps to the 1st level. Removed a section of the solid flooring below the second-floor pressure vessels to allow better air circulation and ventilation.	10/3/2011
100 H	Evaluate the possibility of relocating H_2 compressor	Dusty	The ESA compressor will be used for compression of N_2 or N_2/He blends only. This compressor will not be used for H_2 compression. H_2 will be supplied from six-packs that will be placed external to the NCHT building. There will be no internal H_2 storage within proximity to the Accelergy system or below the hot vessels located on the second deck.	5/28/2012
	Evaluate location and safe operability of the thermal oxidizer	Jim	The DCL system will utilize an existing flare for destruction of the product gas; the thermal oxidizer will be shut off and its fuel supply locked out during DCL system operation.	Ongoing
	Evaluate existing electrical equipment relative to the area classification	Jim	All of the electrical motors were checked, and they all meet the electrical requirement for the NCHT facility. We are only using heating cables and clam-shell-like heaters that meet the electrical code requirements. Any other electrical devices that will be added in the future will also be evaluated to meet the code requirements.	2/3/2012
	Evaluate adequacy of ventilation and possible need for ducting in/around second-floor equipment	Ken/Christin	Trunk lines will be installed to address additional ventilation requirements after system fabrication, pressure testing, and shakedown have been completed.	Ongoing
100 H	Reevaluate need for barricading with respect to predicted flame jet ignition	Dusty/Mark/Jim	Some barricading has been eliminated; the remaining barricading functions to protect personnel entering the pilot area from the second-floor lobby and also to minimize possible flame projection to adjacent equipment; the barricading also functions to provide equipment support.	1/10/2012
200	Add drip pans and liquid containment around slurry and solvent tanks	Mark	All slurry and solvent tanks and associated pumps have been moved to the first floor; each tank has a spill tray in case of leak; need to look at method for blocking off floor drain.	Ongoing
100 H	Evaluate potential of light gases being trapped under solid floor decking	Ken/Christin	This has been addressed with the removal of the section of solid decking on the second level.	10/3/2011
100 H	Evaluate possibility of using H_2 six-packs and eliminating need for electric H_2 compressor	Mark/Jim/Ken	A location for permanent placement of six-packs is being assessed. Preliminary plans for temporary storage include placement of up to 2 six-packs east of the NCHT building. Stainless steel tubing (¼-inch OD) will connect the six-packs to the Accelergy process through a distance of approximately 60 feet. The H_2 pressure will be regulated at the six-packs to the pressure required for the Accelergy process. The DCL system H_2 utilization rate is ~75 scfh, six-pack volume is 2550 scf at 6000 psig. Utilization to 3000 psig or ½ original volume would give 17 hours of operation, and a 100-hour run would require 5.88 six-packs.	5/28/2012
100 H	Evaluate possible use of air-driven booster for six-pack scavenging	Mark	The use of a scavenging compressor would facilitate the need for limited additional H_2 storage in the event that the booster had mechanical issues. This would give time to switch out to another six-pack.	5/28/2012
All	Perform valve failure action assessment	Mark	1st iteration completed; will incorporate necessary action responses into SOP and computer control logic.	Ongoing

Continued. . .

RISK ASSESSMENT ACTION ITEMS (continued)

Area	Action Item	Person Responsible	Action Taken	Date
All	Assess impact of possible loss of house air pressure	Mark	House air is needed for operation of blocking and control valves; pressure transducer PIR-002 has been added to monitor gas supply pressure; programmable low-pressure alarm limits on the control computer will alert operations staff to a low-pressure condition; regulated N_2 at slightly lower pressure than house air pressure will provide a pneumatic pressure source in the instance that house air pressure is lost; check valves in both the air and N_2 supply lines prevent backflow of the high-pressure gas into the lower-pressure source line; response actions have been included in the SOP.	4/1/2012
100 H, N	Evaluate need for surge flow devices (lines entering building, flow taken off supply header) to limit gas release in case of fitting failure	Dusty/Mark	Surge devices should be used on any large-volume section of piping, specifically on H_2 six-packs. High-pressure tubing has been used between the N_2 compressor, N_2 accumulators, and high-pressure N_2 regulator to reduce the orifice size of any leaks in case of regulator failure.	10/7/2011
400	Evaluate need for check valves (between process vessels) to limit gas release in case of fitting failure	Mark	A potential location for a check valve is between the HP/HT and HP/LT vessels; upstream of the heat exchangers may present problems with possible plugging from condensed heavy organics or coal solids. Downstream of the heat exchangers provides a low-temperature location but with two-phase flow. The overall system volume is small, and presently, there are no plans to incorporate a check valve between the vessels.	9/20/2011
All	Evaluate need for E-stop(s) for controlled automated shutdown in various emergency situations	Mark	E-stops are presently not incorporated in the current PC control program; near-term action responses in the SOP will incorporate manual intervention; E-stop concept(s) will be outlined and programmed at a later date.	Ongoing
All	Develop shutdown matrix	Mark	The standard operating procedure contains instructions for a normal, controlled shutdown via manual interface with the PC control/data acquisition program; the first iteration of an emergency shutdown procedure has been developed that utilizes manual interface with the computer; future versions of the computer control program will contain an E-stop emergency shutdown.	Ongoing
200	Add rupture disc before safety relief valve on HP pumps	Mark	Rupture disks will not be incorporated; evaluating use of HP pump internal hydraulic relief as more reliable than rupture disks.	5/4/2012
200, 300, 400	Incorporate low/no-flow cut-off procedure for vessel and coil heaters	Mark	Over-temperature thermocouples have been added to electrically heated high-pressure tubing and vessels; the data acquisition and control program will allow user-defined temperature limits for the heater controllers and over-temperature thermocouples; the operator will take corrective action to turn heaters down or off.	5/1/2012
200, 300, 400	Implement protocol/procedures for dealing with plugs	Jim	This has been discussed, and we are still developing the proper procedure to prevent any equipment or personnel injury.	
300	Evaluate separate stack for sand bath hot N_2 venting	Jim	A separate stack for the sand bath fluidizing N_2 vent has been installed; this stack incorporates a drain valve and a water-cooled heat exchanger to reduce the N_2 temperature before venting to the NCHT roof.	3/1/2012

Continued. . .

RISK ASSESSMENT ACTION ITEMS (continued)

Area	Action Item	Person Responsible	Action Taken	Date
400	Reposition CV-412 as close to vessel as possible	Jim	CV-412 has been placed as close as possible to the inlet of the HP/HT stripper/separator in the stripping line; similarly CV-304 has been placed as close as possible to the point where gas mixes with the slurry and the inlet of the slurry reactor.	9/22/2011
400	Consider putting a check valve between HP/HT separator/stripper and HP/LT separator	Mark	A potential location for a check valve is between the HP/HT and HP/LT vessels; upstream of the heat exchangers may present problems with possible plugging from condensed heavy organics or coal solids. Downstream of the heat exchangers provides a low-temperature location but with two-phase flow. The overall system volume is small, and presently, there are no plans to incorporate a check valve between the vessels.	9/20/2011
400	Change length of dip tube in HP/LT separator	Mark	Changed on drawing Accelergy-v6.vsd; dip tube in HP/LT separator should end below concentric reducer; this was a drawing error not a construction error.	9/15/2011
400	Eliminate PSV-406; PSV-402; PSV-404 in 400 high-pressure drawing	Mark	Changed on drawing Accelergy-v6.vsd; pressure safety valves PSV-402, -404, and -406 removed; tubing and vessels have 3500 psig MAWP, which is higher than maximum gas and slurry supply pressure.	9/15/2011
	Establish program to annually check safety valves	Jim	This has been discussed, and we are still developing the proper procedure to prevent any equipment or personnel injury.	Ongoing
	Establish and maintain good housekeeping	Jim	Good housekeeping will be continuously emphasized, especially with respect to preventing trip or other injury hazards.	Ongoing
	Identify egress – keep clear	Jim	Egress signage has been ordered and received but not yet mounted.	Ongoing
	Proper PPE, MSDS – “Be specific in PPE”	Christin		
	Concerns with explosive hazards – hot surfaces	Dusty	Hot surfaces are an ignition concern. These should be limited by encasing the hot surface in insulation to ensure that gas will not likely reach autoignition temperature except if it is leaking from the hot surface process. Increased ventilation around hot surfaces will also help lower ignition probability from a hot surface.	10/7/2011
	H_2S exposure and how to detect	Ramesh	Portable H_2S monitor is currently being used, and will be used during operation.	
	Safety showers and eye wash locations	Ken/Christin	EERC Safety Office is looking at the need for a shower/eye wash station on the second-floor process area.	Ongoing
	Stress corrosion and cracking – corrosive materials	Doug	Prior to purchasing all materials and equipment, every effort was made to address this possibility. Will monitor all equipment and vessels closely after operation commences for any such occurrences.	1/3/2012
	Restriction on flex hoses – stainless braided type	Jim	All flexible hoses are constructed of a ribbed tube enclosed within a braided protective exterior. The ribbed tube and exterior braiding are made of 316SS. The maximum service temperature is 1250°F. The 1-inch hoses used on the mix, feed, and flush solvent tanks are rated at 718 psi at 72°F. Maximum expected temperature is 300°F. The hoses are designed to withstand vibration and misalignment. The safety reliefs on the gear pumps are set at 100 psig so the maximum pressure rating of the hoses will not be exceeded.	5/10/2012

Continued. . .

RISK ASSESSMENT ACTION ITEMS (continued)

Area	Action Item	Person Responsible	Action Taken	Date
	Radiation sources – controlling and training procedures for working around – signage	Ken/Christin	Mark and Jim have taken training from UND; before operation commences, other personnel that will be involved with the opening or closing of the sources will take the training at UND.	Ongoing
	Second-floor eye wash/shower station	Ken/Christin		
	Helium sniffer – for leak-checking	Mark	A He/H_2 sniffer has been purchased.	4/26/2012
	Air quality control permit – SO_2, H_2S, CO_2, NO_2	Christin	Data have been provided to EERC Safety Office Manager Ken Grohs regarding potential maximum production of H_2S (and/or SO_2).	9/19/2011
	Hazardous waste – types and quantities	Ramesh	Still compiling complete list of all waste materials that will be generated during DCL system operation. Currently have an existing EERC protocol on storage and disposal of hazardous materials.	Ongoing
	Control in crushing bottoms, keep dust and contamination at a minimum	Jim	Will be completed when exact method for bottoms crushing has been established.	Ongoing
	Proper training of personnel before opening any valve or breaking apart fittings – use of proper PPE – gloves, face shield, respirator	Jim	Proper training will need to be performed for all personnel working on the system. Ventilation trunks will be required any time the process is opened to the environment if flammable gases or toxic vapors could be released. Processes should be purged of gases before opening, as the classification of a Division II is that vapors and gases are not expected to be present except through failure. Training is initiated during construction and installation of various system components and will continue as equipment additions and modifications made.	Ongoing

RESPONSIBLE PERSONNEL

ID	Full Name	Position/Function
Ted	Ted R. Aulich	Project Manager
Greg	Gregory T. Dvorak	Programming
Ken	Ken L. Grohs	Manager, Facilities and Safety
Doug	Douglas J. Hajicek	Senior Design Engineer
Bob	Robert R. Jensen	Programming
Dusty	Dustin P. McNally	Hydrogen Safety/Risk Assessment
Mark	Mark A. Musich	Project Engineer
Christin	Christin R. McWaters	Facilities and Safety
Ramesh	Ramesh K. Sharma	Lead Chemist, Process Technologist

AUTOMATED VALVE LIST

Area	Tag No.	Name	Actuation
100	AV 102	Hydrogen six-pack isolation	ATO
100	AV 104	Hydrogen accumulator/MFC isolation	ATO
100	AV 106	Nitrogen accumulator/MFC isolation	ATC
100	AV 110	Reaction hydrogen MFC – blocking	ATO
100	AV 112	Purge nitrogen MFC – blocking	ATC
100	AV 114	Hydrogen/nitrogen isolation	ATO
200	AV 202	Slurry cutoff	ATO
200	AV 204	Solvent flush cutoff	ATC
400	AV 402	HP/HT separator/stripper – top lock valve	ATO
400	AV 404	HP/HT separator/stripper – bottom lock valve	ATO
400	AV 406	HP/LT separator – top lock valve	ATO
400	AV 408	HP/LT separator – bottom lock valve	ATO
400	AV 410	Bottoms stripper accumulator – top lock	ATO
400	AV 412	Bottoms stripper accumulator – bottom lock	ATO
400	AV 414	Condensate accumulator – top lock valve	ATO
400	AV 416	Condensate accumulator – bottom lock valve	ATO
400	PCV 418	Pressure Control Valve 418	ATC
400	PCV 420	Pressure Control Valve 420	ATC

LOSS OF SUPPLY AIR

Step No.	Action		Computer/Field
1	Toggle all H_2 AV valves to closed position.	Manual/automatic	Computer
2	Input 0% flow on H_2 MFCs.	Manual/automatic	Computer
3	Toggle all N_2 AV valves to open position.	Manual/automatic	Computer
4	Toggle flush solvent supply blocking valve to open position.	Manual/automatic	Computer
5	Toggle slurry supply blocking valve to closed position.	Manual/automatic	Computer
6	Reduce preheater, reactor, and pressure vessel temperature S's.	Manual/automatic	Computer
7	Toggle all top lock and bottom lock valves from automatic to manual mode (valves will default to closed state).	Manual	Computer
8	Close HV upstream of PCV.	Manual	Field
9	Assess cause of failure and time required for repair – for repairs lasting longer than 5 minutes, proceed to Step 18.		
10	Reset valve position and/or operating mode to normal on computer.	Manual	Computer
11	Verify cycling of top lock and bottom lock valves for all separators/accumulators.	Manual	Computer
12	Open HV upstream of PCV.	Manual	Field
13	Demonstrate control of system pressure through manual cycling of PCV.	Manual	Computer
14	Establish normal system pressure and toggle to automatic pressure control.	Manual	Computer
15	Toggle all H_2 AV valves to open position.	Manual	Computer
16	Slowly establish normal flows on H_2 MF's.	Manual	Computer
17	Proceed to Step 31.		
18	Establish N_2 flow through N_2 MFC.	Manual	Computer
19	Control system pressure using HVs on manual bypass.	Manual	Field
20	Monitor level in HP/HT stripper/separator.	Manual	Computer/Field
21	Reset to normal operating mode on computer.	Manual	Computer
22	Verify cycling of top lock and bottom lock valves for all separators/accumulators.	Manual	Computer
23	Open HV upstream of PCV.	Manual	Field
24	Demonstrate control of system pressure through manual cycling of PCV.	Manual	Computer
25	Slowly close manual bypass valves.	Manual	Field
26	Establish normal system pressure and toggle to automatic pressure control.	Manual	Computer
27	Toggle all H_2 AV valves to open position.	Manual	Computer
28	Slowly establish flows on H_2 MFCs.	Manual	Computer
29	Slowly reduce flow on N_2 MFC.	Manual	Computer
30	Toggle all N_2 AV valves to closed position.	Automatic	Computer
31	Increase preheater, reactor, and pressure vessel temperature SPs to original values.	Manual	Computer
32	Toggle slurry supply blocking valve to open position.	Automatic	Computer
33	Toggle flush solvent supply blocking valve to closed position.	Automatic	Computer
34	Resume normal operation.		

VALVE FAILURE RESPONSE

Area	Tag No.	Name	Actuation	Failure	Consequences	First Action	Second Action	Third Action	Fourth Action	Fifth Action	Sixth Action	Seventh Action			Comment	Other Possible Action
100	AV 104	Hydrogen accumulator/MFC isolation	ATO	Closes/won't open/leaks	Loss of system H_2 flow; reduction of system pressure	Replace slurry flow with flush solvent flow	Replace H_2 flow with N_2 purge at stripping H_2 FCV	Replace H_2 flow with N_2 purge at reaction H_2 FCV	Isolate blocking valve and vent off H_2	Repair, replace (with spare), or remove blocking valve	Begin transition back to slurry and H_2 flow					Eliminate valve
100	AV 106	Nitrogen accumulator/MFC isolation	ATC	Leaks	Loss of ability to purge system with N_2 during start-up or emergency flush	Replace slurry flow with flush solvent flow	Reduce preheater, vessel, and reactor heater SPs	Isolate blocking valve and vent off N_2	Repair, replace (with spare), or remove blocking valve	Begin transition to solvent and N_2 flow	Continue following start-up procedure				To be followed when failure during start-up or purge	Eliminate valve
100	AV 110	Reaction hydrogen MFC – blocking	ATO	Closes/won't open/leaks	Loss of H_2 flow to reactor	Replace slurry flow with flush solvent flow	Replace stripping H_2 flow with N_2 purge flow	Replace reaction H_2 flow with N_2 purge flow	Close manual valve downstream of blocking valve	Assess that manual valve is sealed before removal of blocking valve	Vent off H_2	Repair, replace (with spare), or remove blocking valve	Begin transition back to slurry and stripping H_2 flow			Eliminate valve
100	AV 112	Purge nitrogen MFC – blocking	ATC	Leaks	Loss of ability to purge reactor through N_2 FCV during emergency flush	Replace slurry flow with flush solvent flow	Replace stripping H_2 flow with N_2 purge flow	Replace reaction H_2 flow with N_2 purge flow	Close manual valve downstream of blocking valve	Assess that manual valve is sealed before removal of blocking valve	Vent off N_2	Repair, replace (with spare), or remove blocking valve	Begin transition to solvent and N_2 flow	Continue following start-up procedure	To be followed when failure during start-up or purge	Eliminate valve
100	AV 114	Hydrogen/nitrogen isolation	ATO	Closes/won't open/leaks	Loss of ability to purge through H_2 FCVs during start-up or emergency flush	Replace slurry flow with flush solvent flow	Reduce preheater, vessel, and reactor heater SPs	Initiate N_2 flow through N_2 purge FCV	Assess status of test	If conditions allow, isolate blocking valve and vent off N_2	Repair, replace (with spare), or remove blocking valve	Begin transition to solvent and N_2 flow	Continue following start-up procedure			Eliminate valve
200	AV 202	Slurry cutoff	ATO	Closes/won't open/leaks	Loss of slurry flow to system	Open solvent flush cutoff valve to flush HP slurry pump	Replace H_2 flow with N_2 purge at stripping H_2 FCV	Replace H_2 flow with N_2 purge at reaction H_2 FCV	Transition to HP solvent flush pump operation	Close HP slurry pump outlet valve	Close manual valve upstream of blocking valve	Repair, replace (with spare), or remove blocking valve	Begin transition to solvent and N_2 flow	Continue following start-up procedure		
200	AV 204	Solvent flush cutoff	ATC	Leaks	Loss of ability to flush HP slurry pump with solvent during emergency	Initiate solvent flow through HP flush solvent pump	Replace H_2 flow with N_2 purge at stripping H_2 FCV	Replace H_2 flow with N_2 purge at reaction H_2 FCV	Close HP slurry pump outlet valve	Close manual valve upstream of blocking valve	Repair, replace (with spare), or remove blocking valve	Begin transition to solvent and N_2 flow	Continue following start-up procedure			
200	AV 206	Solvent flush cutoff	ATC	Opens while feeding slurry	Possible replacement of slurry with solvent	Close manual hand valve upstream of automated valve										
400	AV 410	Bottoms stripper accumulator – top lock	ATO	Closes/won't open/leaks	Loss of ability to remove bottoms from BSA vessel	Close manual valve upstream of BSA vessel letdown valves	Assess need to replace slurry with solvent flush (large volume of BSA may allow longer time for assessment and repair)	Complete repair or replacement of valve if assessed to require short time period and continue with operation	If shutdown required, follow normal shutdown including solvent flush to remove solids from reactor and HP/HT vessel	Recover warm liquids from bottom of BSA vessel after system depressurization						
400	AV 412	Bottoms stripper accumulator – bottom lock	ATO	Closes/won't open/leaks												
400	AV 414	Condensate accumulator – top lock valve	ATO	Closes/won't open/leaks	Loss of ability to remove liquids from condensate accumulator vessel	Close manual valve upstream of condensate accumulator vessel letdown valves	Assess need to replace slurry with solvent flush (large volume of accumulator may allow longer time for assessment and repair)	Complete repair or replacement of valve if assessed to require short time period and continue with operation	If shutdown required, follow normal shutdown including solvent flush to remove solids from reactor and HP/HT vessel	Recover liquids from bottom of condensate accumulator after system depressurization						
400	AV 416	Condensate accumulator – bottom lock valve	ATO	Closes/won't open/leaks												
400	PCV 418	Pressure Control Valve 418	ATC	Opens/leaks	Drop in system pressure upstream of pressure control valves and increase in downstream system pressure (downstream vessels are protected with safety reliefs)	Close manual valves upstream and downstream of affected control valve	Open upstream and/or downstream manual valves on spare control valve	Continue with system operation with spare control valve	Assess ability to make repairs to affected valve	If valve not repairable, assess continuing operation without backup pressure control						
400	PCV 420	Pressure Control Valve 420	ATC	Opens/leaks												

APPENDIX B

DCL PROCESS DEVELOPMENT UNIT
STANDARD OPERATING PROCEDURE
AS OF 25 JULY 2012

DIRECT COAL LIQUEFACTION PROCESS DEVELOPMENT UNIT

Standard Operating Procedure (SOP)
Version 3.2

Prepared by:

Mark A. Musich

Energy & Environmental Research Center
University of North Dakota
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Grand Forks, ND 58202-9018

25 July 2012

TABLE OF CONTENTS

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Ted Aulich	Sr. Research Manager	701-777-2982
Malhar Khambete	Research Engineer	701-777-5007
Mark Musich	Research Engineer	701-777-5263
Jim Tibbetts	Research Engineer	701-777-5282
EERC Reception Desk		701-777-5000
Facilities Maintenance		701-777-5199
Instrumentation Shop		701-777-5110
Machine Shop		701-777-5140
National Center for Hydrogen Technology Control Room		701-777-5388

INTRODUCTION

With guidance from Accelergy, the Energy & Environmental Research Center (EERC) designed, fabricated and installed a 50-pound/day (dry coal input basis) direct coal liquefaction (DCL) reactor system at EERC. The system is installed on two floors of the EERC National Center for Hydrogen Technology (NCHT) building. This building is equipped with necessary infrastructure including fire alarm and suppression systems, hazardous gas alarm, ventilation system, process gas flare, air and nitrogen (high and low pressure).

The intended uses of the DCL system are to 1) generally validate results of bench-scale DCL tests conducted to convert coal to liquids suitable for upgrading to jet and other transportation fuels, and 2) produce coal liquids for upgrading to large (up to 25-gallon) fuel samples for evaluation by the U.S. Air Force Research Laboratory (AFRL) at Wright Patterson Air Force Base, Dayton, Ohio. The DCL system is not intended to generate material balance or scalable data.

The purpose of this manual is to familiarize operators with the overall DCL process, process components, and process operation methodology. The various steps involved in the overall DCL process are shown in Figure 1. The DCL system will operate in a semicontinuous mode and comprises two primary subsystems:

1 The reactor system (Figures 2–7), comprising the primary process equipment needed to convert coal to a raw coal liquids product.

2 The distillation system (Figure 8), which will be used to separate the raw coal liquids product into vacuum gas oil (VGO), naphtha, water, and “middle distillate” fractions. The middle distillate fraction will be upgraded (in a separate facility), the naphtha fraction will be upgraded and/or otherwise utilized, and the VGO fraction will be recycled. The distillation will be installed in a large hood in the NCHT building near the DCL reactor system.

The DCL system comprises the following “process areas”:

- Area 100 – hydrogen/nitrogen supply and delivery
- Area 200 – feed slurry/solvent preparation and delivery
- Area 300 – preheaters, sand-bath heater, and reactors
- Area 400 high pressure (HP) – product stripper and condensate separator
- Area 400 low pressure (LP) – bottoms and condensate recovery and collection

Appendix A contains computer screen shots of the PC-based process control and data-logging system. Appendix B presents the valve start-up positions.

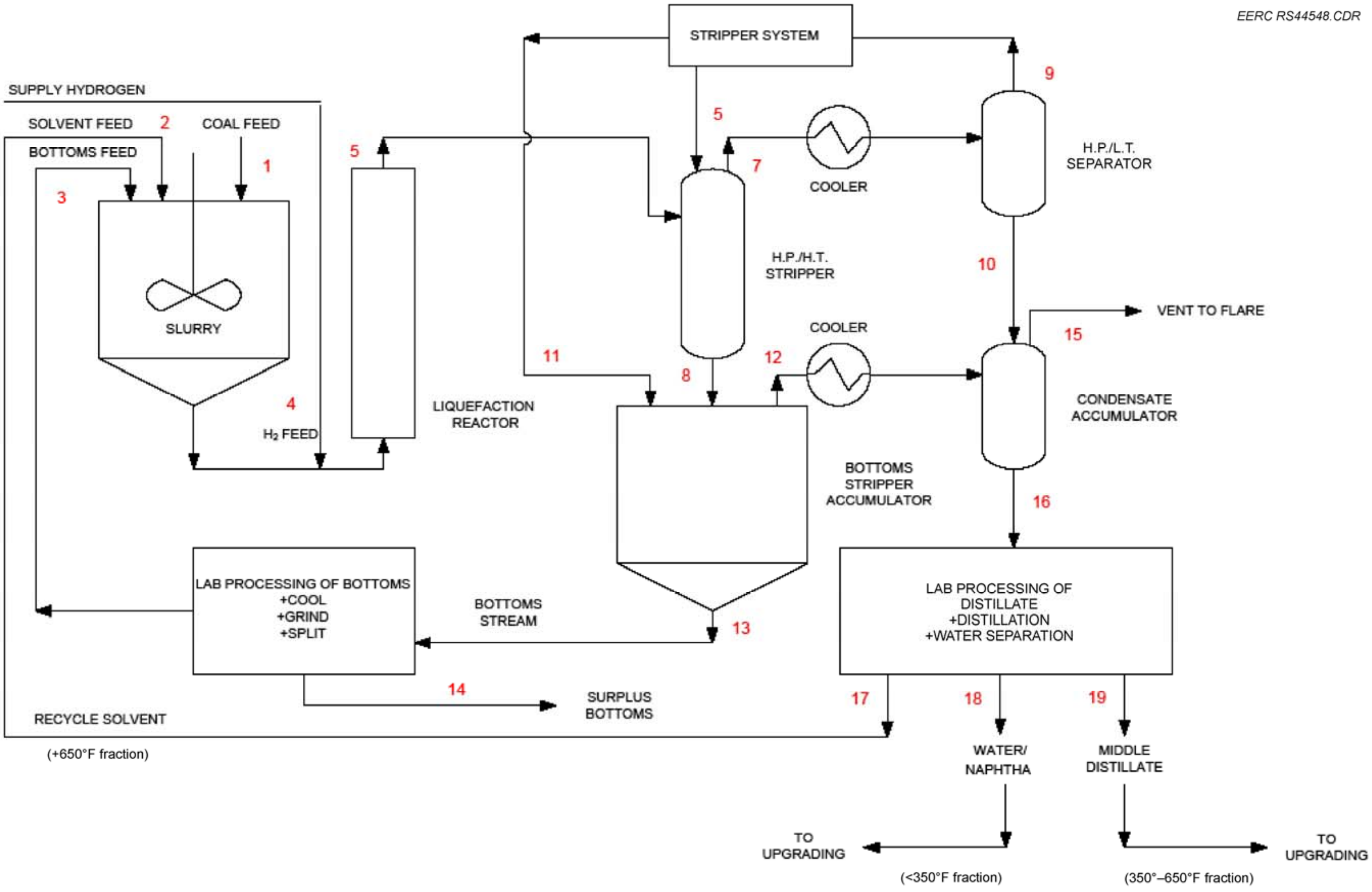


Figure 1. DCL process flow diagram.

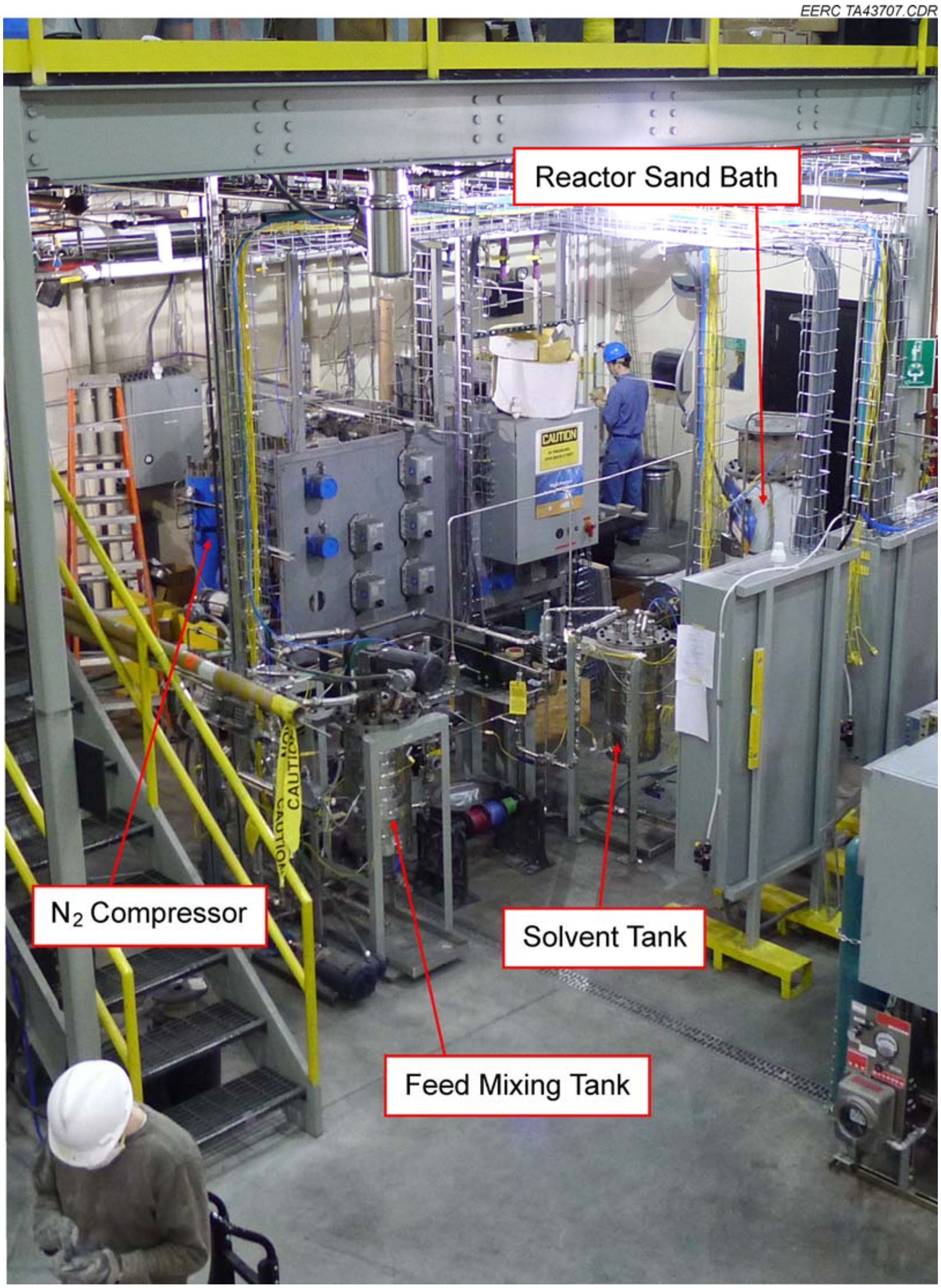


Figure 2. First-floor view of DCL reactor system.

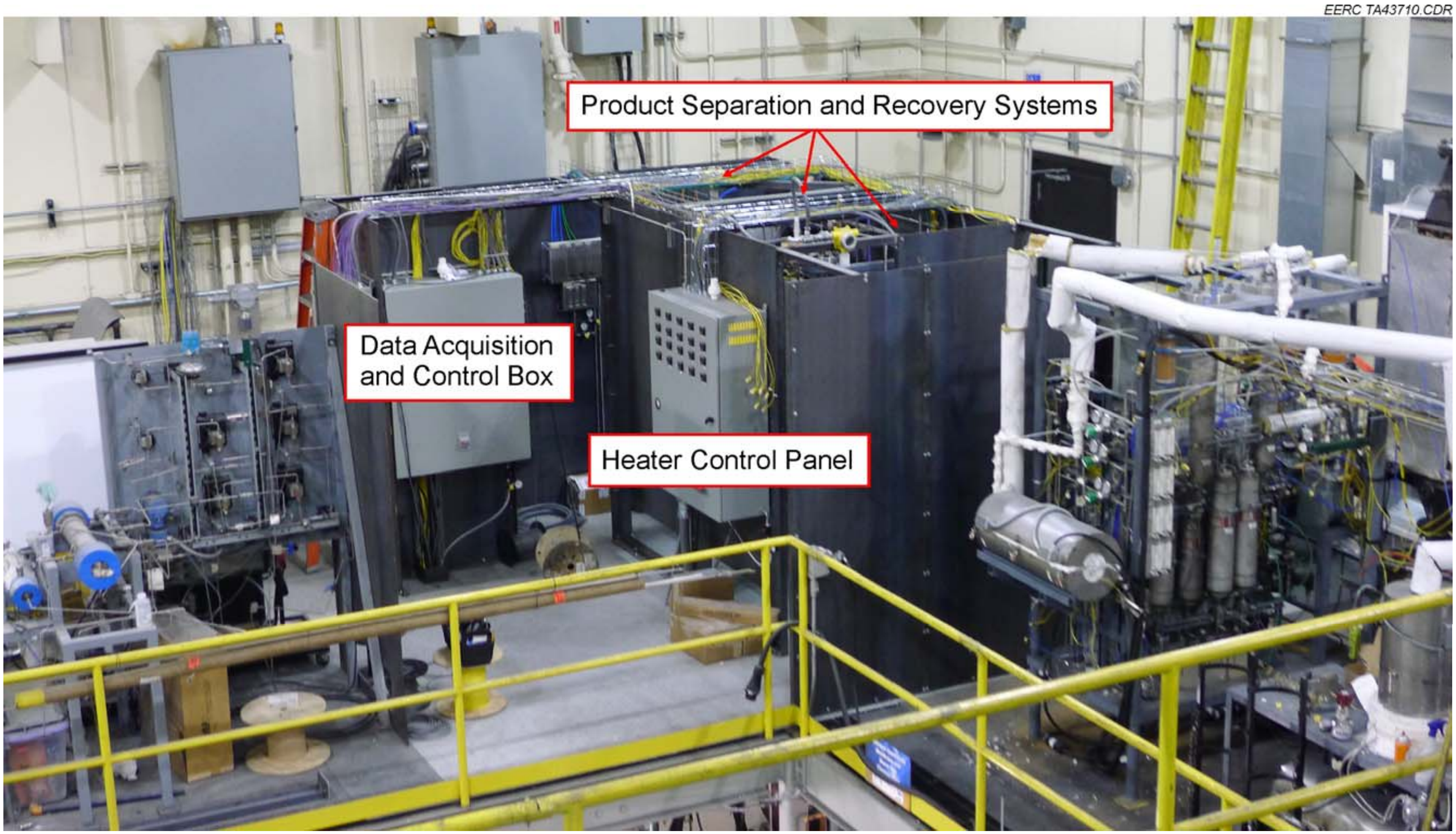


Figure 3. Second-floor view of DCL reactor system.

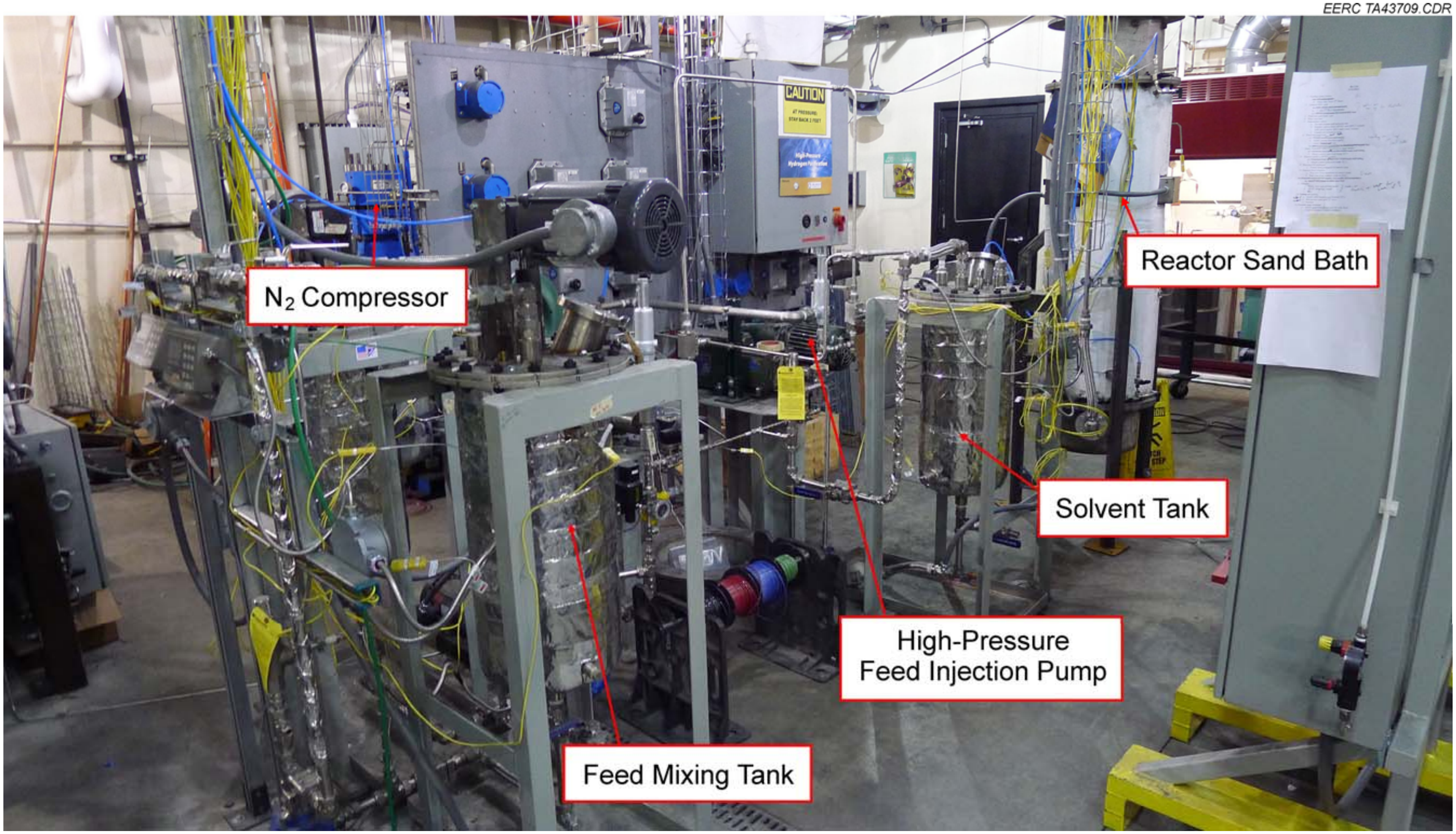


Figure 4. Close-up of prereactor feed preparation and injection systems.

EERC TA43708.CDR

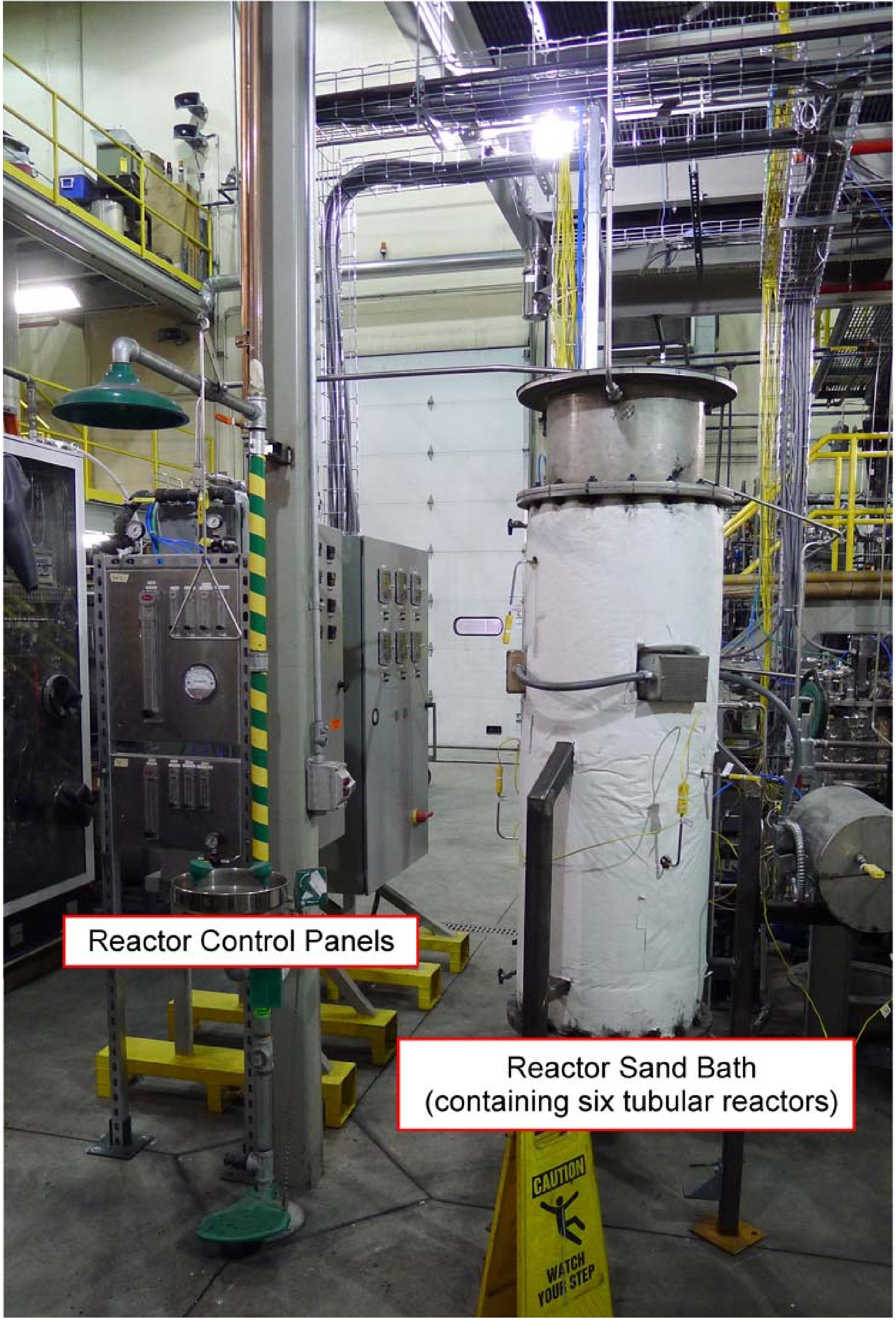


Figure 5. Close-up of DCL reactor sandbath.

EERC TA43711.CDR

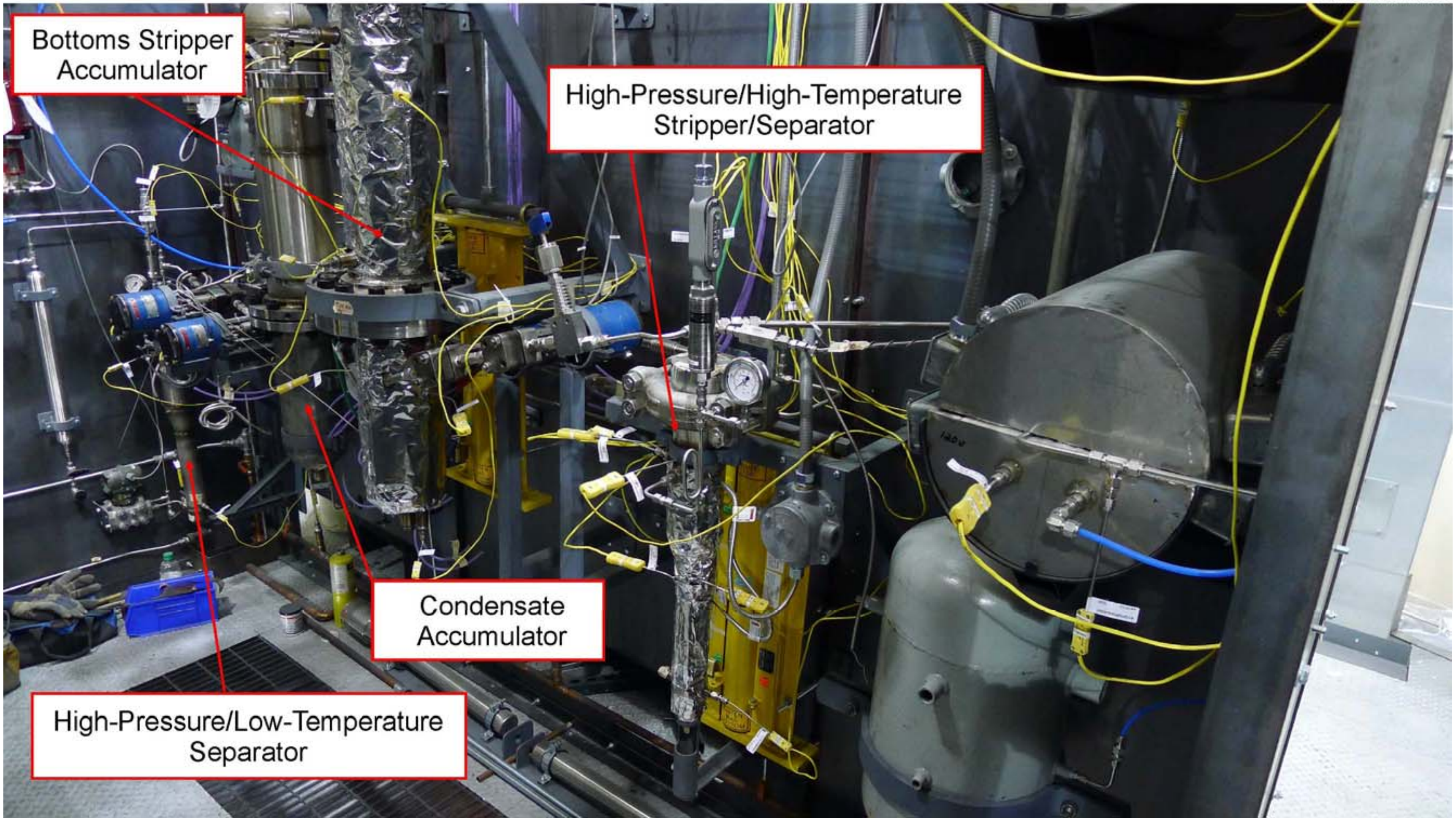


Figure 6. Close-up of product separation and accumulation systems.

Figure 7. Nitrogen compressor system.

Figure 8. P9600 Spinning band distillation system.

PROCESS SUMMARY

Generating raw coal liquids from a target coal is a three-step procedure comprising 1) target coal preparation, 2) preparation of target coal-derived VGO and vacuum bottoms in sufficient quantities (50 pounds each) to initiate a liquefaction run, and 3) liquefaction for production of target coal raw coal liquids. Procedures for the three steps are described below.

Step 1 – Target Coal Preparation

It is important that as-received and prepped coals are stored under nitrogen in airtight containers. Since the as-received coal is in the form of large lumps and contains a significant amount of water (5%–40%) depending on the coal type, it is necessary to predry the coal to save energy needed to heat water in the coal. The lump coal will be floor-dried to remove any surface moisture and any easily volatilized inherent moisture. Also, to achieve a high degree of conversion, the coal needs to be pulverized to enable optimal coal–solvent–catalyst contact during liquefaction. Thus, the as-received coal is pulverized and sized using a 100-mesh screen in an automated classifier, collecting all coal passing through the screen. The pulverized coal will then be fed into a rotary kiln at 110°C under nitrogen flow until the water content of the coal is about 1%. The dried coal is then collected and stored under nitrogen in airtight containers prior to undergoing liquefaction.

Step 2 – Preparation of Vacuum Gas Oil (VGO)

Initial processing is designed to generate test coal-derived VGO and vacuum bottoms. The bottoms will be generated in the main reactor system and the VGO (recycle solvent) will be generated in the distillation unit. Initially, a start-up slurry consisting of coal, catalyst (100 ppm in coal–catalyst mix), and hydrogenated anthracene oil (1.6 solvent/coal mass ratio) will be premixed in a slurry mixing tank (feed mixing tank, Figure 4), and then transferred to a slurry feed tank with the help of a pump. The start-up slurry will then be fed into the tubular reactor housed in a fluidized bed-sand bath at a predetermined rate using a high-pressure pump (Figure 4). The volatile products will be condensed to form a liquid using a series of condensers, and remaining slurry (vacuum bottoms) will be collected and transferred to a freezer for storage. The frozen slurry will be pulverized to –100-mesh size and transferred into the mixing tank for recycle. The uncondensed gases will pass through a scrubber for removal of acidic gases, and the remaining gas will be sent to the flare system. The condensed liquid will be collected every 6 hours, transferred to a distillation unit located near the DCL system, and batch distilled to yield about 4.2 liters of VGO and 3 liters of distillate comprising naphtha, water, and a “middle distillate cut” (which will necessarily contain a significant amount of start-up slurry-derived coal tar distillate). The VGO obtained from distillation (along with the previously collected vacuum bottoms) will be used for recycle.

The purpose of the initial processing is to generate enough test coal-derived vacuum bottoms and VGO to start the liquefaction process, and thereafter the process will be self-sustaining and generate bottoms and VGO on a continuous basis. After sufficient amounts of vacuum bottoms and VGO needed to initiate the liquefaction process are produced, further processing will be conducted to produce test coal-derived raw middle distillate for upgrading to jet fuel or other fuel. Middle distillate upgrading will be conducted in a separate EERC hydroprocessing facility.

The gaseous, liquid, and solid products formed during liquefaction are potentially hazardous and must be handled, stored, and disposed of using care and appropriate skin and eye protection.

Step 3 – Liquefaction (production of middle distillate)

After generating sufficient quantities of test coal-derived solvent VGO and vacuum bottoms, the liquefaction of test coal will be conducted to generate distillate fuel referred to as middle distillate for upgrading to produce synthetic fuels. The standard operating conditions that will be frequently used are as follows:

- Temperature = 450°C
- Residence time = 60 minutes
- Pressure = 2500 psi

During liquefaction processing of test coal, vacuum bottoms ground to –100 mesh, solvent/VGO, and catalyst will be transferred to the slurry tank. The overall process involves feeding slurry consisting of an approximate 1/1/1 mixture of dried coal (2 pounds), coal-derived solvent/VGO (1.8 pounds), vacuum bottoms (1.8 pounds) and 100–300 ppm catalyst per hour to the reactor. A constant pressure of 2500 psig will be maintained through use of a pressure control valve and constant flow of hydrogen. Operating conditions can be varied depending on the composition and reactivity of the material that will be processed. The temperature can be lowered or raised to 500°C with automated controls. The 60-minute residence time is based on a 2-pound/hour dry coal input rate with six 39-inch reactor tubes installed. This residence time can be changed by either adding or subtracting tubes or by changing the overall flow rate of the system.

Liquefaction will utilize the same procedures described above for Step 2 – Preparation of VGO, with the exception that the initial solvent charge to be utilized will comprise Step 1—the prepared VGO material rather than the hydrogenated anthracene oil.

DCL System Products

Appendix C describes a theoretical material balance around the DCL system operating with an input of 2 pounds of air-dried coal an hour. The solvent (VGO) and vacuum bottoms will be recycled. The amounts and composition of the products will be dependent on the test coal used. Overall, the DCL system will produce following products:

1. Gases: The gas yield will be about 0.13 pounds/hour or 8 wt% of dry ash-free (daf) coal. The gases produced will include H_2, H_2S, NH_3, CO, CO_2, methane, ethane, propane, and butane. The gas will be routed to a flare system for destruction.

2. Liquids: Two types of liquids, primarily aqueous and oils will be produced by the DCL system.

    - Aqueous product yield will be about 0.18 pounds/hour or 10 wt% of daf coal added. This fraction will contain small amounts of alcohols, ketones, phenols, mercaptans, and hydrocarbons dissolved in water. Therefore, wastewaters are potential hazards and need to be disposed of according to U.S. Environmental Protection Agency guidelines.

    - Oils will be taken to a semiautomatic distillation unit for fractional distillation (refer to distillation manual) to produce the following fractions:

        1) Naphtha (room temperature–350°F) (0.14 pounds/hour or 8.0 wt% of daf coal used) to be reformed or upgraded to produce a gasoline blendstock.

        2) Middle distillate (350°–650°F) (0.78 pounds/hour or 44 wt% of daf coal added) to be upgraded to produce fuels.

        3) Heavy oil (>650°F) to be recycled.

3. Solids (vacuum bottoms, including ash) yield is expected to be 0.60 pounds/hour, and comprise about 0.36 pounds/hour (30 wt% of daf coal) organic material and 0.24 pounds/hour ash. The tarlike vacuum bottoms product, which comprises the residue remaining after distillation of the liquefaction slurry (with a boiling point of >1000°F) will be recycled.

The distillate oils will be stored in a freezer until they are ready for upgrading to produce finished fuels. The finished fuels will be packaged under nitrogen and stored in a freezer.

The VGO and vacuum bottom fractions will be stored in a freezer until recycled. After the test has been completed, the system will be flushed with solvent to clean the unit. The flushing of the system is needed to prevent the residual slurry from solidifying and plugging the unit. After the system is cleaned, shut down will follow the procedure described below.

PRERUN CHECKLIST

1. Material safety data sheets are readily available for all materials.

2. Maintenance items have been completed and heat-tracing and insulation are in place.

3. Heater power cords are connected and thermocouple (TC) wires are plugged in.

4. Lock-out/tag-outs have been removed.

5. Areas around equipment are clear of tools, air hoses, combustible materials and other potential trip hazards.

6. Safety eye wash and shower stations are operational and flushed.

7. Personal protective equipment (PPE) are in their proper locations.

8. Two-way radios are operational, and spare batteries have been charged.

9. All raw materials are in place and of adequate supply:
   a. Hydrogen six-packs
   b. Start-up oil – creosote oil or equivalent
   c. Coal
   d. Flush solvent

10. Portable area monitors are charged and operational.

11. Purges are on to electrical boxes for adjacent systems.

12. Product gas analyzer is operational.

13. Area monitors for hydrogen and combustibles are operational.

14. Verify that the building ventilation is operational.

15. Spill cleanup kits are in place and properly stocked.

ANCILLIARY SYSTEM SETUP

1. Start up the system control/data logging computer, and create a run file with the name shown in the Test Plan (provided by the Project Manager).

2. Turn on flashing lights to alert area personnel to system operation.

3. Turn on hydrogen and combustible gas area monitors; calibrate as necessary.

4. Turn on instrumentation air (and back up N_2) supply to solenoids and I/P converters.

5. Set purge nitrogen flows to flow control valve electrical boxes.

6. Set purge nitrogen flows to vent lines and knock-out pots.

7. Verify in the field that all automated valves are in the closed position.

8. Verify in the field that all manual valves are in the start-up positions.

9. Verify that all process variables are being displayed properly and that trending screens are operational.

10. Investigate and satisfy all alarm conditions.

11. Turn on cooling water supply and booster pump (as necessary).

12. Turn on product gas analyzer and calibrate. Verify operation at control computer.

13. Start-up gasifier flare according to instructions on wall by ignition control system. Monitor temperatures on the laptop control computer.

14. Set up nuclear-level monitors (level indicator/control [LIC]-402 and LIC-406).
    a. Unlock and open source shutter.
    b. Power up master evaluation units and verify operation.

15. Check area to verify that nonelectrically rated systems are deenergized.

16. Check indicator board in control room to verify that purges are active for all systems in the pilot plant area.

SYSTEM LEAK-CHECK WITH HELIUM/NITROGEN BLEND

1. Set up pressure control system:
    a. On Area 100 control screen, toggle pressure control valves (PCV) PCV-418 and PCV-420 to manual operation.
    b. Stroke PCV-418 and PCV-420 from 0% open to 100% open several times; verify response in field.
    c. Set PCV-418 and PCV-420 to 0% open (100% closed).
    d. Verify that hand valve (HV) HV-420 (before PCV-418) and HV-422 (before PCV-420) are closed.

2. Start-up compressor with helium/nitrogen blend:
    a. Turn on cooling water to compressor.
    b. Verify that all compressor alarms are satisfied.
    c. Open valve on helium cylinder to bring helium to the helium mass flow control valve.
    d. Open house nitrogen supply valve to bring nitrogen to the nitrogen mass flow control valve.
    e. Input helium and nitrogen set points (SPs) at their respective mass flow controllers.
    f. Monitor helium pressure on pressure indicator (PI) PI-102 and pressure indicator recorder (PIR) PIR-102.
    g. Start compressor and verify automatic start/stop cycling.

3. Set helium/nitrogen blend pressure:
    a. Open HV-102 downstream of gas accumulator.
    b. Using PIR-106 and PI-106, set regulated helium pressure to 2950 psig using PRV-104.

4. Perform leak-check of reactor hydrogen line and components:
    a. Open automated valve (AV) AV-104.
    b. Open AV-110.
    c. Slowly open HV-110 to bring helium/nitrogen to flow control valve (FCV) FCV-104.
    d. Input a low-flow SP (<10 scfh) on FCV-104.
    e. Allow PIR-110 and PI-110 to increase to 2950 psig.
    f. Close AV-104.
    g. Observe PIR-110 and PI-110. Look for leaks between AV-104, HV-106, HV-112, and HV-118.
    h. Utilize the LACO Gascheck G3 helium/hydrogen sniffer as necessary.
    i. Input a SP of 0 scfh for FCV-104.
    j. Close HV-110 and AV-110.

5. Perform leak-check on downstream systems:
    a. Open AV-104.
    b. Open HV-110.
    c. Input a low-flow SP (<10 scfh) on FCV-104.
    d. Allow PIR-110 and PI-110 to increase to 2950 psig.
    e. Open AV-110.

   f. Slowly open HV-112 to initiate helium/nitrogen flow to the rest of the system.
   g. Look for and seal gross leaks.
   h. Close HV-102.
   i. Observe PIR-106/PI-106, PIR-110/PI-110, PIR-302/PI-302, PIR-304/PI-304, PIR-402/PI-402, and PIR-404/PI-404 for a period of 15 minutes. Look for leaks between HV-102 and HV-420/HV-422.
   j. Utilize the LACO Gascheck G3 helium/hydrogen sniffer to find leaks.

6. Vent helium/nitrogen blend from system:
   a. Shut down the compressor.
   b. Close valve on helium supply cylinder.
   c. Close the nitrogen supply valve.
   d. Isolate the compressor discharge accumulators.
   e. Open HV-462.
   f. Slowly bleed system pressure through HV-464.
   g. Verify that system pressure is decreasing by observing PIR-106/PI-106, PIR-110/PI-110, PIR-302/PI-302, PIR-304/PI-304, PIR-402/PI-402, and PIR-404/PI-404).

7. Put manual and automated valves in start-up mode:
   a. Input a SP of 0 scfh for FCV-104.
   b. Close HV-108 and HV-112.
   c. Close AV-108 and AV-110.
   d. Close HV-106 and HV-110.
   e. Close PCV-418 and PCV-420.
   f. Close AV-104.
   g. Close HV-102.
   h. Open PRV-104.

SYSTEM PRESSURIZATION

1. Set up system pressure control loop:
   a. On the Area 400HP control screen, input a pressure control SP of 2500 psig.
   b. Toggle to PCV-418 to automatic pressure control.
   c. Leave PCV-420 in manual mode with SP of 0% open/100% closed.

2. Start-up the compressor:
   a. Turn on cooling water to compressor.
   b. Open compressor discharge accumulator cylinder valves.
   c. Open HV-132 and HV-134 to bring house nitrogen to the compressor.
   d. Monitor house nitrogen pressure on PI-112 and PIR-112.
   e. Verify that all alarm lights at compressor start/stop panel are satisfied.
   f. Verify low pressure (restart) and HP (stop) SPs at compressor.
   g. Start compressor and verify automatic start/stop cycling.
   h. Monitor the compressor discharge pressure on PI-114 and PIR-114.

3. Set nitrogen source pressure:
   a. Open HV-104.
   b. Using PIR-116 and PI-116, set regulated nitrogen pressure to 2950 psig using PRV-106.
   c. Open AV-106 and then AV-114.
   d. Slowly open HV-118.

4. Start nitrogen flow to reactor:
   a. Open HV-110 to bring nitrogen to FCV-104.
   b. Input a low-flow SP (<10 scfh) on FCV-104.
   c. Verify flow of nitrogen by watching pressure increase on PIR-110 and PI-110 and flow value on control screen for FCV-104.
   d. After indicated flow drops to 0, and PIR-110 and PI-110 pressures have stabilized, open AV-110.
   e. Slowly open HV-112 to initiate nitrogen flow to the rest of the system.
   f. Slowly increase FCV-104 flow SP until the Test Plan value is attained for reaction hydrogen flow rate.
   g. Continue to monitor system pressure and response of PCV-418 (and/or PCV-420).

5. Bring system to operating pressure:
   a. Monitor system pressure at PIR-404.
   b. Adjust PID values for PCV-418 to obtain the desired pressure control response.
   c. Utilize PCV-420 in manual mode as necessary to control system pressure.
   d. Open HV-470 and HV-472.
   e. Slowly close HV-474 (product gas meter bypass).
   f. Monitor product gas flow rate (flow indicator [FI] FI-402) on control screen.
   g. Compare nitrogen flow rate values from the FCVs to the calculated product gas rate displayed on control screen.

6. Start sampling process gas:
    a. Open HV-410 in the sample gas line.
    b. Verify in the field that the product gas analyzer is getting sufficient sample flow.
    c. Verify that the product gas composition is trending toward 100% nitrogen.

7. Continue to monitor critical system parameters:
    a. Nitrogen flow.
    b. Product gas flow.
    c. System pressure.

FLUSH SOLVENT PREPARATION

1. Verify personnel are wearing proper PPE:

2. Load flush solvent to the solvent tank T-206.
    a. Open HV-xxx on the flush solvent tank.
    b. Transfer flush solvent from the storage vessel to the tank.
    c. Close HV-xxx.

3. On the Heat-trace control screen input control SPs for the Area 200 heat-trace lines associated with the flush solvent tank.

4. Initiate recirculation and mixing of flush solvent:
    a. On the Area 200 control screen, verify that the input rpm for gear pump P-206 is 0.
    b. Turn on P-206 at the control screen.
    c. Initiate solvent circulation by slowly increasing pump speed; do not exceed 600 rpm.

5. Initiate heating of flush solvent:
    a. On the Administration screen, input low and high alarm SPs for the heater controllers and the over-temperature TCs.
    b. On the Area 200 control screen, input the ramp rate for flush solvent tank heaters and recycle piping heat-trace.
    c. Toggle the heater controllers from manual to auto, and input a control SP in accordance with the Test Plan.
    d. Monitor T-206 temperatures and make necessary adjustments to the tank heaters and heat-trace until the solvent temperature attains that prescribed in the Test Plan.

START-UP SOLVENT HEATUP

1. Turn on cooling water to the slurry mix tank overhead condenser (HX-202).

2. Verify personnel are wearing proper PPE.

3. Load start-up solvent to the slurry mix tank T-202:
    a. Tare the scale for the slurry mix tank.
    b. Open HV-xxx on the slurry mix tank.
    c. Transfer solvent from the storage vessel to the slurry mix tank until the mass prescribed in the Test Plan has been added.
    d. Close HV-xxx.

4. Initiate recirculation and mixing of slurry mix tank solvent:
    a. On the Area 200 control screen, verify that the input rpm for gear pump P-202 is 0.
    b. Turn on P-202 at the control screen.
    c. Initiate solvent circulation by slowly increasing pump speed; do not exceed 600 rpm.
    d. On the control screen, verify the input rpm for mixer M-202 is 0.
    e. Turn on M-202 at the control screen.
    f. Initiate solvent mixing by slowly increasing mixer speed; max speed is 345 rpm.

5. Initiate heating of start-up solvent:
    a. On the Administration screen, input low and high alarm SPs for the heater controllers and the over-temperature TCs.
    b. On the Area 200 control screen, input the ramp rate for slurry mix tank heaters and recycle piping heat-trace.
    c. Toggle the heater controller from manual to auto, and input a control SP in accordance with the Test Plan.
    d. Monitor T-202 temperatures and make necessary adjustments to the tank heaters and heat-trace until the solvent temperature attains that prescribed in the Test Plan.

6. Transfer solvent to slurry feed tank T-204:
    a. Tare the scale for the slurry feed tank.
    b. Open HV-208 on the transfer line to the slurry feed tank.
    c. Observe the scale weight on the slurry feed tank to verify the transfer of solvent; partially or fully close HV-206 on the slurry mix tank recycle line to assist flow to the feed tank.
    d. Transfer solvent from the mix tank to the feed tank until the mass prescribed in the Test Plan has been added.
    e. Open HV-206 and close HV-208.

7. Initiate recirculation and mixing of slurry feed tank solvent:
    a. On the Area 200 control screen, verify that the input rpm for gear pump P-204 is 0.
    b. Turn on P-204 at the control screen.
    c. Initiate solvent circulation by slowly increasing pump speed; do not exceed 600 rpm.

    d. On the control screen, verify the input rpm for mixer M-204 is 0.
    e. Turn on M-204 at the control screen.
    f. Initiate solvent mixing by slowly increasing mixer speed; maximum speed is 345 rpm.

8. Initiate heating of slurry feed tank solvent:
    a. On the Administration screen, input low and high alarm SPs for the heater controllers and the over-temperature TCs.
    b. On the Area 200 control screen, input the ramp rate for slurry mix tank heaters and recycle piping heat-trace.
    c. Toggle the heater controller from manual to auto, and input a control SP in accordance with the Test Plan.
    d. Monitor T-204 temperatures and make necessary adjustments to the tank heaters and heat-trace until the solvent temperature attains that prescribed in the Test Plan.

9. Continue to monitor critical system parameters:
    a. Nitrogen flow.
    b. Product gas flow.
    c. System pressure.
    d. Solvent temperature.
    e. Heater output.
    f. Gear pump performance.
    g. Condenser temperatures and coolant flows.

AREAS 300 AND 400 START-UP

1. Turn on cooling N_2 to HP/high temperature (HT) stripper/separator overheads cooler (HX-406):
    a. Open HV-442.
    b. Control flow with flow indicator/control (FIC) FIC-402 rotameter.

2. Turn on cooling water to the HP/HT stripper/separator overheads cooler (HX-408):
    a. Open HV-444.
    b. Control flow with FIC-404 rotameter.

3. Turn on cooling water to the bottoms stripper accumulator (BSA) overhead cooler (HX-410):
    a. Open HV-458.
    b. Control flow with FIC-406 rotameter.

4. Initiate heating of process vessels and heat-trace lines:
    a. On the Administration screen, input low and high alarm SPs for the heater controllers and the over-temperature TCs.
    b. On the Area 300 control screen, input the ramp rates for slurry and solvent line heat-traces.
    c. On the Area 400 control screen, input the ramp rates for the HP/HT stripper/separator and BSA process vessels and slurry line heat-trace.
    d. Toggle the heater controllers from manual to auto, and input a control SP in accordance with the Test Plan.
    e. Monitor temperatures and make necessary adjustments to the vessel heaters and heat-trace until the temperatures prescribed in the Test Plan are attained.

5. Start-up fluidizing N_2 to sand bath (T-302):
    a. Open HV-302 on fluidizing N_2 supply line.
    b. Set the N_2 pressure to ~15 psig using pressure relief valve (PRV) PRV-302.
    c. Initiate N_2 flow to the sand bath through FIC-302.
    d. Monitor the bed differential pressure on dPI-308.
    e. Adjust FIC-302 to attain a flow rate of ~6 scfm
    f. Monitor the source N_2 pressure on PIR-306/PI-306.

6. Turn on the sand bath heaters:
    a. On the Area 300 control screen, verify that the PIR-306 alarm condition is satisfied.
    b. On the Administration screen, input low and high alarm SPs for the heater controllers, sand bath temperatures, and the reactor outlet temperatures.
    c. On the Area 300 control screen, input the ramp rates for the sand bath heaters.
    d. Toggle the heater controllers from manual to auto, and input control SPs.
    e. Monitor T-302 temperatures temperature indicator/reactor (TIR) (TIR-322, -324, -326), and make necessary adjustments to the sand bath heaters until the bed temperature attains the start-up value prescribed in the Test Plan.

7. Turn on the reaction hydrogen preheater:
    a. On the Administration screen, input low and high alarm SPs for the heater controller and over-temperature TC.
    b. On the Area 300 control screen, input the ramp rate for the electric heater.
    c. Toggle the heater controllers from manual to auto, and input a control SP.
    d. Monitor the outlet temperature (TIR-304), and make necessary adjustments to the reaction hydrogen preheater until the outlet temperature attains that prescribed in the Test Plan.

8. Continue to monitor critical system parameters:
    a. Nitrogen flow.
    b. Product gas flow.
    c. System pressure.
    d. Solvent temperature.
    e. Heater output.
    f. Sand bath performance.
    g. Gear pump performance.
    h. Coolant flow and temperature.

SLURRY PREPARATION

1. If necessary add more solvent to slurry mix tank T-202.

2. Add recycle bottoms (heavies) to heated start-up solvent:
    a. Depressurize feed hopper on solids feeder.
    b. Add pulverized recycle bottoms to the feed hopper.
    c. Pressure the feed hopper with nitrogen.
    d. Slowly open the valve between the solids feeder and the mix tank.
    e. Slowly feed bottoms to the mix tank to allow the bottoms to melt and become dispersed in the start-up solvent.
    f. Allow bottoms/solvent slurry to mix and circulate until temperatures have stabilized at desired levels.
    g. Close the valve isolating the solids feeder and the mix tank.
    h. Depressurize the feed hopper.

3. Add coal to heated start-up solvent:
    a. Add pulverized coal to the feed hopper.
    b. Pressure the feed hopper with nitrogen.
    c. Slowly open the valve between the solids feeder and the mix tank.
    d. Slowly feed coal to the mix tank to allow the coal to become evenly dispersed in the start-up solvent.
    e. Allow the coal/bottoms/solvent slurry to mix and circulate until temperatures have stabilized at desired levels.
    f. Close the valve isolating the solids feeder and the mix tank
    g. Depressurize the feed hopper.

4. Continue to monitor critical system parameters:
    a. Nitrogen flow.
    b. Product gas flow.
    c. System pressure.
    d. Solvent and slurry temperatures.
    e. Heater output.
    f. Sand bath performance.
    g. Gear pump performance.
    h. Coolant flow and temperature.

START-UP SOLVENT PROCESSING

1. On the Area 200 control screen, verify that the HP pump (P-208) inlet and outlet heat-trace are functional and at temperature.

2. Start pumping start-up solvent (from T-204):
   a. Verify that the HP slurry pump is adjusted to a low-stroke length.
   b. Open AV-202.
   c. Open HV-258.
   d. Start HP slurry pump at field start/stop station.
   e. Verify solvent pressurization on PI-216/PIR-216.
   f. When PI-216/PIR-216 pressure reaches PIR-304/PI-304 pressure, open HV-220.
   g. Verify solvent pumping:
      – Slurry heat-trace and sand bath temperatures should drop and then rebound as the heaters activate.
      – System pressure may become more cyclical because of pump pulsing.
   h. Adjust the stroke rate on the HP pump to attain the desired volumetric feed rate as determined from calibration.

3. Monitor liquid levels in the Area 400 HP process vessels:
   a. HP/HT stripper/separator pressure valve (PV) (PV-402) level shown by LIC-402.
   b. HP/low temperature (LT) separator (PV-404) level shown by LIC-404.

4. Monitor overhead heat exchanger temperatures, and make changes to coolant flows as necessary.

5. Initiate letdown in the HP/HT stripper/separator (PV-402) when the level reaches the SP prescribed in the Test Plan:
   a. On the Area 400HP control screen, toggle AV-402 and AV-404 to manual mode.
   b. Verify in the field that AV-402 and AV-404 are closed.
   c. Slowly open HV-402.
   d. Open, then close AV-402 to draw pressurized solvent into the lock.
   e. Close HV-402.
   f. Open, then close AV-404 to flash solvent from the lock.
   g. Toggle AV-402 and AV-404 into automatic mode.
   h. Open HV-402 when AV-402 and AV-404 are both closed.
   i. Verify solvent letdown/discharge:
      – The level trend indicated by LIC-402 should exhibit a saw tooth cycle.
      – A slight and cyclic temperature increase may be seen on TIR-416.
   j. Monitor solvent level in the BSA by LIC-406.

6. Initiate letdown in the HP/LT separator (PV-404) when the level reaches the SP prescribed in the Test Plan:
   a. On the Area 400 HP control screen, toggle AV-406 and AV-408 to manual mode.
   b. Verify in the field that AV-406 and AV-408 are closed.

   c. Slowly open HV-404.
   d. Open, then close AV-406 to draw pressurized distillate into the lock.
   e. Close HV-404.
   f. Open, then close AV-408 to flash distillate from the lock.
   g. Toggle AV-406 and AV-408 into automatic mode.
   h. Open HV-404 when AV-406 and AV-408 are both closed.
   i. Verify distillate letdown/discharge:
      – The level trend indicated by LIC-404 should exhibit a saw tooth cycle.
      – A slight, cyclic temperature may be seen on TIR-436.
   j. Monitor solvent level in the BSA by LIC-406.

7. Monitor liquid levels in the Area 400LP process vessels:
   a. BSA (PV-406) level shown by LIC-406.
   b. Condensate accumulator (CA) (PV-408) level shown by LIC-408.

8. Continue pumping start-up solvent until the level in the slurry feed tank is below xx% of capacity and the BSA begins to fill.

HYDROGEN SYSTEM OPERATION

1. Set hydrogen source pressure:
   a. Open HV-102.
   b. Using PIR-106 and PI-106, set regulated nitrogen pressure to 2950 psig using PRV-104.

2. Initiate hydrogen feeding:
   a. From the control screen, open AV-106.
   b. Close HV-124 shortly after.
   c. Verify feeding of hydrogen:
      - No permanent change in FCV-104 output.
      - No permanent change in system pressures.
      - Output on reaction hydrogen preheater increases (due to higher heat content gas relative to nitrogen).
   d. Make adjustments to hydrogen flow rates at FCV-104 in accordance with the Test Plan.
   e. Monitor the product gas analyzer for the appearance of hydrogen.
   f. Monitor area monitors for the possible presence of hydrogen.
   g. Continue to make adjustments to the flare as the product gas hydrogen content increases.

SLURRY PROCESSING

1. Transfer coal/bottoms/solvent slurry to the slurry feed tank:
   a. Open HV-208 on the transfer line to the slurry feed tank.
   b. Observe the scale weight on the slurry feed tank to verify the transfer of slurry; partially or fully close HV-206 on the slurry mix tank recycle line to assist flow to the feed tank.
   c. Transfer slurry from the mix tank to the feed tank until the level in the slurry mix tank is below xx % of capacity.
   d. Open HV-206 and close HV-208.
   e. Verify slurry pumping:
      – No permanent change in system pressures
      – Modest changes in slurry preheater output (due to change in heat content relative to solvent only)

2. Allow reactor temperatures to stabilize and slowly increase the fluid-bed (FB) sand bath temperature to attain the reactor conditions prescribed in the Test Plan.

3. Initiate letdown in the BSA (PV-406) when the level reaches the SP prescribed in the Test Plan:
   a. On the Area 400LP control screen, toggle AV-410 and AV-412 to manual mode.
   b. Verify in the field that AV-410 and AV-412 are closed.
   c. Slowly open HV-406.
   d. Open, then close AV-410 to draw pressurized bottoms into the lock.
   e. Close HV-406.
   f. Open, then close AV-412 to flash bottoms from the lock.
   g. Toggle AV-410 and AV-412 into automatic mode.
   h. Open HV-406 when AV-410 and AV-412 are both closed.
   i. Verify bottoms letdown/discharge:
      – The level trend indicated by LIC-406 should exhibit a saw tooth cycle.
      – A change should be observed in the coolant return temperature on the circulating chiller.
   j. Open the bottoms collection pot periodically to verify product recovery.

4. Initiate letdown in the CA (PV-408) when the level reaches the SP prescribed in the Test Plan:
   a. On the Area 400 LP control screen, toggle AV-414 and AV-416 to manual mode.
   b. Verify in the field that AV-414 and AV-416 are closed.
   c. Slowly open HV-408.
   d. Open, then close AV-414 to draw pressurized distillate into the lock.
   e. Close HV-408.
   f. Open, then close AV-416 to flash distillate from the lock.
   g. Toggle AV-414 and AV-416 into automatic mode.
   h. Open HV-408 when AV-414 and AV-416 are both closed.
   i. Verify bottoms letdown/discharge:
      – The level trend indicated by LIC-408 should exhibit a saw tooth cycle.

    j. Open the distillate collection pot periodically to verify product recovery.

5. Continue to monitor critical system parameters:
    a. Hydrogen flow and source pressure.
    b. Product gas flow and composition.
    c. System pressure.
    d. Slurry temperature.
    e. Heater output.
    f. Sand bath performance.
    g. Gear pump and HP pump performance.
    h. Coolant flow and temperature.
    i. Liquid levels.
    j. Letdown valve operation.
    k. Product collection.

5. After system operation and performance has stabilized, prepare another batch of slurry in the slurry feed tank.

NORMAL SHUTDOWN

1. Initiate routine for final product collection.

2. Replace hydrogen flow with nitrogen flow:
   a. Open HV-124 and close AV-104.
   b. Reduce nitrogen flow rates to start-up levels.

3. Monitor product gas composition.

4. Replace slurry flow with flush solvent flow:
   a. Open AV-204.
   b. Close AV-202.
   c. Increase the pumping rate of HP pump P-208 as necessary; do not exceed the letdown limits of the HP separators.

5. After 60 minutes of flush, turn off the FB sand bath heaters. Leave the fluidizing air on.

6. Shut down hydrogen compressor:
   a. Isolate hydrogen compressor discharge accumulator.
   b. Isolate hydrogen six-pack.
   c. Bleed to vent supply and discharge lines from hydrogen compressor.

7. Continue processing with flush solvent for xx minutes.

8. Turn off remaining Area 300 and Area 400 heaters and heat-tracing.

9. Continue processing with flush solvent until system temperatures drop below xxx F.

10. Turn off coolant flows to Area 400 heat exchangers.

11. Shut off cooling water to BSA product collection pot.

12. Depressurize system:
    a. Shut off nitrogen flow at FCVs.
    b. Put pressure control valves (PCVs) in manual.
    c. Gradually open PCVs to bring system pressure down to 0 psig.

13. Drain residual slurry from mix tank T-202:
    a. Turn of tank heaters and heat-trace associated with T-202.
    b. Shut down recirculation pump.
    c. Shut down tank mixer.
    d. Drain residual slurry into proper salvage container through HV-256.

14. Drain residual slurry from feed tank T-204:

    a. Turn of tank heaters and heat-trace associated with T-204.
    b. Shut down recirculation pump P-204.
    c. Shut down tank mixer M-204.
    d. Drain residual slurry into proper salvage container through HV-258.

15. Load start-up solvent to the slurry mix tank T-202.

16. Turn on tank heater and heat-trace lines associated with T-202.

17. Initiate recirculation and mixing of slurry mix tank solvent.

18. Transfer solvent to slurry feed tank T-204.

19. Turn on tank heater and heat-trace lines associated with T-204.

20. Initiate recirculation and mixing of slurry feed tank solvent.

21. Repeat Steps 13 through 20 using flush solvent.

EMERGENCY REACTOR FLUSH

1. Replace reaction H_2 flow with purge N_2 flow:
   a. Set FCV-104 to 0 scfh.
   b. Close AV-110.
   c. Set FCV-106 to 5 scfh to help keep gas line free of solids.

2. Reduce slurry feed line heat-trace heater SP.

3. Reduce FB sand-bath heater SPs.

4. Replace slurry feed to HP slurry pump with flush solvent:
   a. Open AV-204.
   b. Open HV-260 to allow flush solvent to HP slurry pump inlet.
   c. Close AV-202.
   d. Continue pumping for several minutes to clear the HP pump check valves of solids.

5. Initiate pumping flush solvent through HP flush solvent pump:
   a. Open HV-224.
   b. Initiate HP pump stroke.
   c. Verify pumping of solvent via TIR-332 (HP pump outlet temperature), pulsations in reactor inlet and outlet pressures (PIR-302, -304), and reactor temperatures (TIR-314. -316, -318).

6. Suspend pumping flush solvent through HP slurry pump.
   a. Shut off HP slurry pump
   b. Close AV-204
   c. Close HV-260

7. Increase the flush solvent pumping rate.

8. Continue to monitor the reactor inlet and outlet pressures to observe the pressure differential across the reactor sections.

9. Assess whether operation of the system can be continued.

10. If operation can be sustained, continue with a modified start-up:
    a. Return heater SPs to normal conditions.
    b. Initiate solvent pumping through the HP slurry pump.
    c. Suspend solvent pumping through the HP flush solvent pump.
    d. Replace flush solvent with slurry through the HP slurry pump.
    e. Replace N_2 purge flow with reaction H_2 flow and bring to normal flow.

EMERGENCY SHUTDOWN

1. Activate all audible and visual alarms if not already tripped.
2. Clear all operations staff (those on the project and others working on adjacent systems) from the pilot plant.
3. Notify Ken Grohs and give current assessment of emergency.
4. Shut off all gas flows (H_2 and N_2) at control computer.
5. Shut off H_2 compressor at electrical swing adsorption control computer.
6. Close all gas-blocking valves.
7. Suspend product letdown sequences.
8. Shut off all heaters.
9. Shut off all mixers and gear pumps.
10. Continue to monitor process from the control computer.

LOSS OF SOLENOID ACTIVATION GAS PRESSURE

(failure of house air supply)

NOTE:

- The house air supply pressure for solenoids that activate control and blocking valves is measured by PIR-002. Normal house air pressure is ~88 psig.

- Nitrogen (N_2) regulated to ~5 psig lower than the house air pressure is plumbed into the solenoid air supply line. In the instance that the air pressure drops below ~83 psig, N_2 flow will commence to replace air.

- Check valves to prevent reverse flow of the HP gas source into the supply line of the lower-pressure gas.

- Lo and LoLo limit alarms will be programmed for PIR-002 to signal a drop in gas supply pressure.

- The valve positions after loss of air pressure are:
  - Hydrogen supply valves (AV-104, -108, -110) – fail closed.
  - Nitrogen supply valves (AV-106, -112) – fail open.
  - Slurry supply valve (AV-202) – fail closed.
  - Solvent flush supply valve (AV-204) – fail open.
  - PCVs (PCV-418, -420) – fail open.

- The consequences of the valve position changes are:
  - Loss of hydrogen supply.
  - Loss of slurry supply.
  - System depressurization up to the PCV(s).
  - Possible pressurization of vessels downstream of the PCV(s).

- The response to the loss of solenoid activation gas pressure is listed below. The responses requiring input at the computer will be performed manually for the near future. Steps 1 through 6 can be performed by the computer (future) if programmed:

  1. Toggle automated H_2 valves (AV-104, -108, and -110) to the closed position.

  2. Input 0 scfh flow SP on H_2 mass flow controller FCV-104.

  3. Toggle automated N_2 valves (AV-106 and AV-112) to the open position.

  4. Toggle flush solvent supply blocking valve AV-204 to the open position.

  5. Toggle slurry supply blocking valve AV-202 to the closed position.

6. Reduce heater controller SPs (controllers will follow programmed ramp-down rates):
    a. Preheaters electrical heater (EH) EH-342 and -344.
    b. Sand bath heaters EH-352, -354, -356.
    c. Vessel heaters EH-484, -486, -490, -492.

7. Toggle all top lock and bottom lock valve sets from automatic to manual mode (valves default to closed state):
    a. AV-402, -404.
    b. AV-406, -408.
    c. AV-410, -412.
    d. AV-414, -416.

8. Close HV-420 upstream of PCV-418 and/or HV-422 upstream of PCV-420.

9. Quickly assess cause of failure and the time required for repair; for repairs lasting longer than 5 minutes, proceed to Step 18.

10. After repair is complete, reset the computer interlock to a normal operating mode (future).

11. Cycle top lock and bottom lock valves (in manual mode) for all separator and accumulator vessels; verify in the field.

12. Open HV-420 (upstream of PCV-418) and/or HV-422 (upstream of PCV-420).

13. Demonstrate control of system pressure through manual cycling of PCV-418 and/or PCV-420.

14. Establish normal system pressure, and toggle over to automatic pressure control.

15. Toggle automated H_2 valves (AV-104, -108, and -110) to the open position; verify in the field.

16. Slowly establish normal flows on FCV-104.

17. Proceed to Step 31.

18. Establish no more than 25 scfh N_2 flow through FCV-106.

19. Control system pressure using HV-284 and HV-285, venting gas to the thermal oxidizer.

20. Continue to monitor LIC-402 on the HP/HT stripper/separator.

21. After repair is complete, reset the computer interlock to a normal operating mode.

22. Cycle top-lock and bottom-lock valves (in manual mode) for all separator and accumulator vessels; verify in the field.

23. Open HV-420 (upstream of PCV-418) and/or HV-422 (upstream of PCV-420).

24. Demonstrate control of system pressure through manual cycling of PCV-418 and/or PCV-420.

25. Slowly close HV-284 and HV-285.

26. Establish normal system pressure and toggle over to automatic pressure control.

27. Toggle automated H_2 valves (AV-104, -108, and -110) to the open position; verify in the field.

28. Slowly establish normal flows on FCV-104.

29. Slowly reduce N_2 flow through FCV-106 to 0 scfh.

30. Toggle automated N_2 valves (AV-106 and AV-112) to the closed position; verify in the field.

31. Increase preheater, reactor, and vessel heater controller SPs to normal operating condition (controllers will follow programmed ramp-up rates).

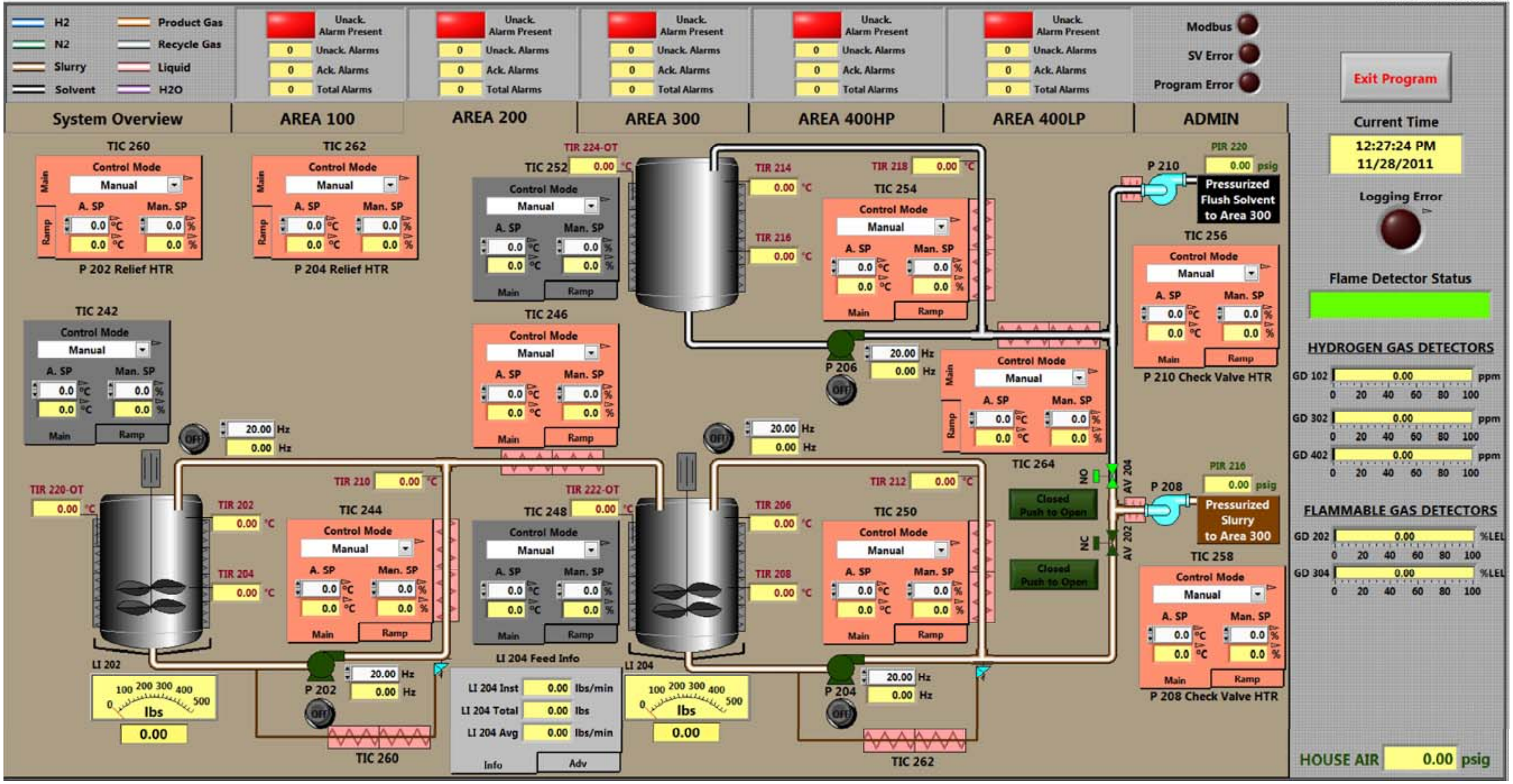
32. Toggle the slurry blocking valve (AV-202) to the open position; verify in the field.

33. Toggle the flush solvent blocking valve (AV-204) to the closed position; verify in the field.

34. Resume normal operation.

APPENDIX A

PROCESS CONTROL COMPUTER SCREEN SHOTS



Current Time
12:27:24 PM
11/28/2011

HOUSE AIR 0.00 psig

Figure A-1. Area 200 screen.

EERC TA44581.CDR

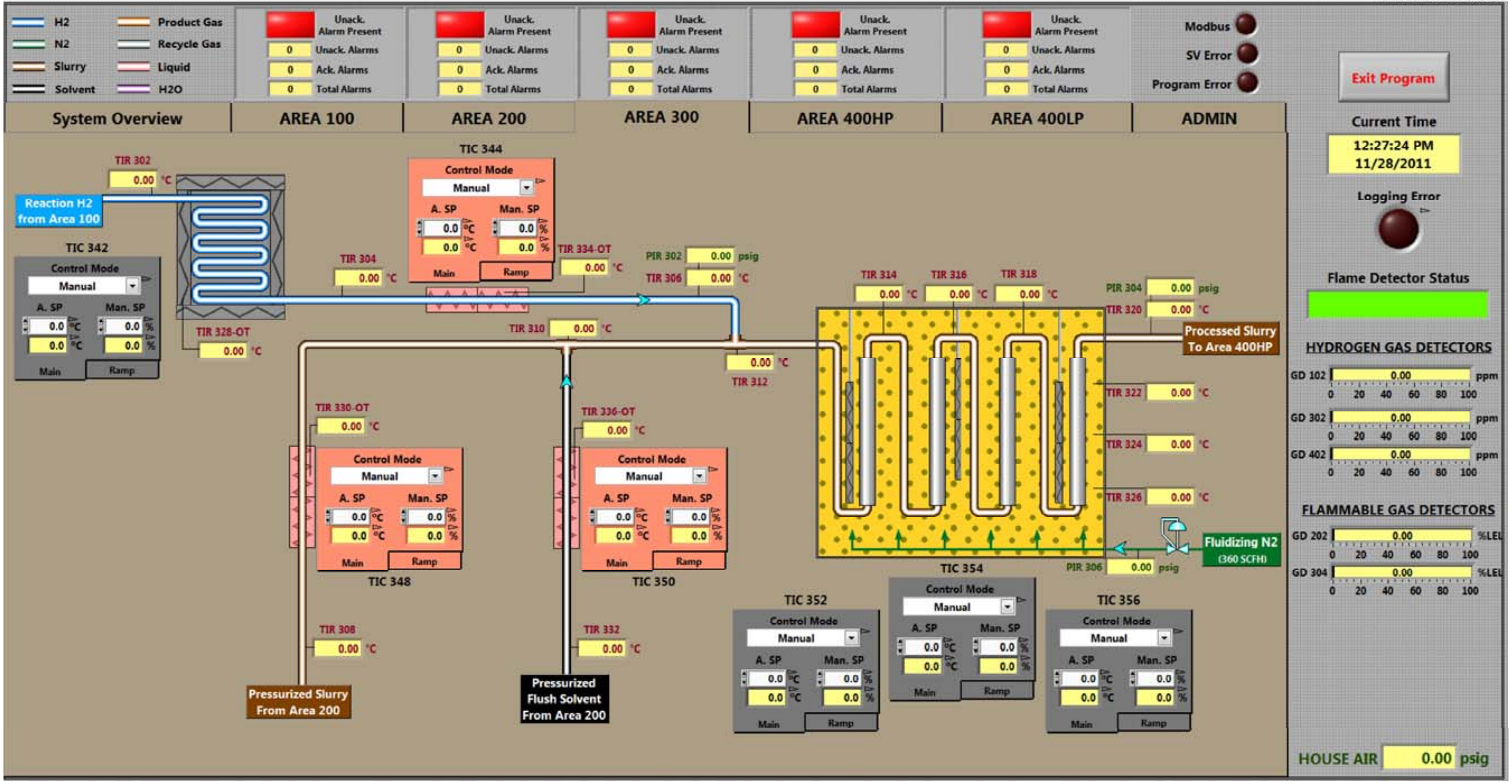
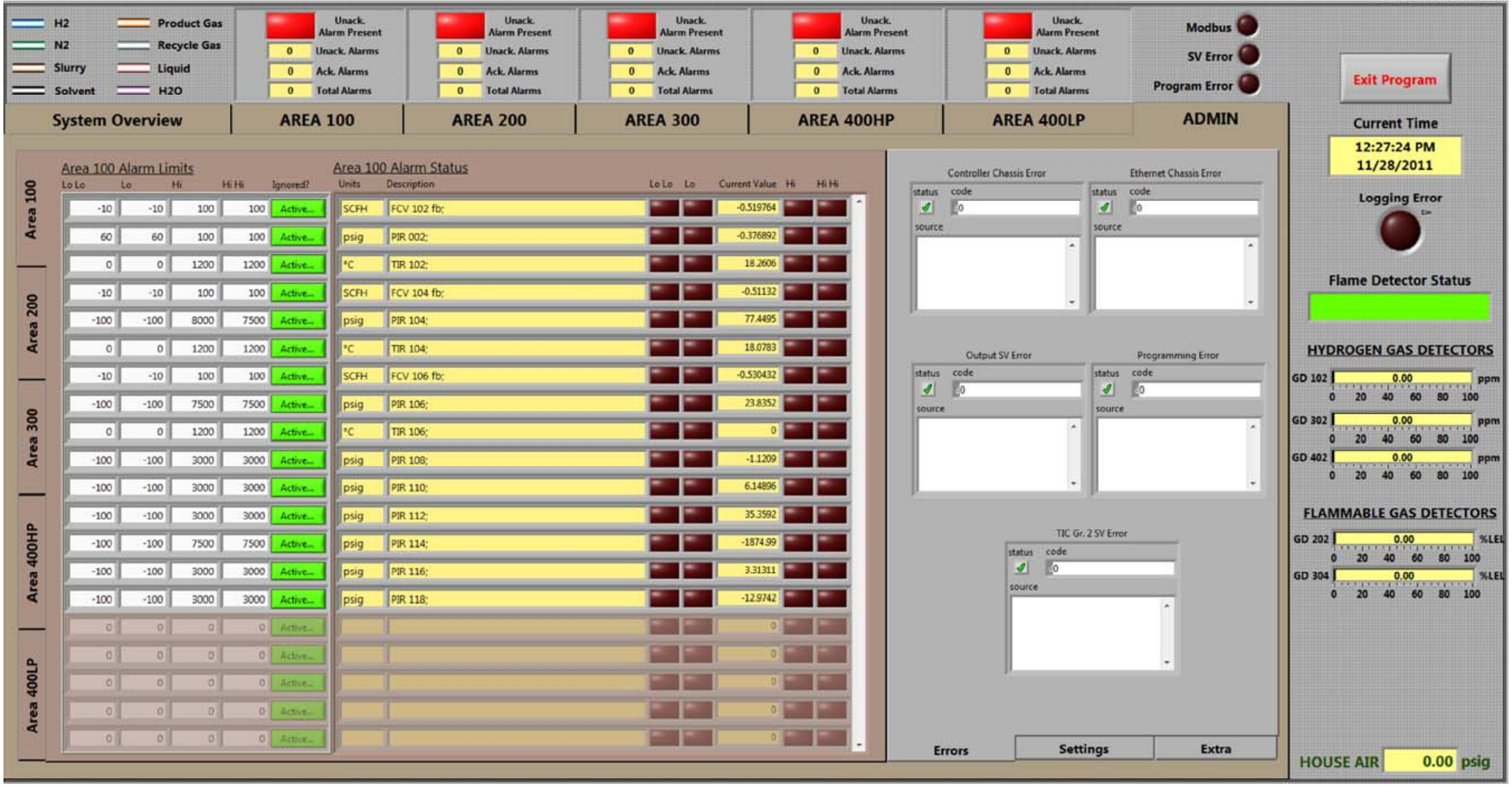


Figure A-2. Area 300 screen.

EERC TA44580.CDR



	AREA 100	AREA 200	AREA 300	AREA 400HP	AREA 400LP
Unack. Alarm Present	🟥	🟥	🟥	🟥	🟥
Unack. Alarms	0	0	0	0	0
Ack. Alarms	0	0	0	0	0
Total Alarms	0	0	0	0	0

Area 100 Alarm Limits

Lo Lo	Lo	Hi	Hi Hi	Ignored?
-10	-10	100	100	Active...
60	60	100	100	Active...
0	0	1200	1200	Active...
-10	-10	100	100	Active...
-100	-100	8000	7500	Active...
0	0	1200	1200	Active...
-10	-10	100	100	Active...
-100	-100	7500	7500	Active...
0	0	1200	1200	Active...
-100	-100	3000	3000	Active...
-100	-100	3000	3000	Active...
-100	-100	3000	3000	Active...
-100	-100	7500	7500	Active...
-100	-100	3000	3000	Active...
-100	-100	3000	3000	Active...
0	0	0	0	Active...
0	0	0	0	Active...
0	0	0	0	Active...
0	0	0	0	Active...
0	0	0	0	Active...

Area 100 Alarm Status

Units	Description	Lo Lo	Lo	Current Value	Hi	Hi Hi
SCFH	FCV 102 fb;	●	●	-0.519764	●	●
psig	PIR 002;	●	●	-0.376892	●	●
°C	TIR 102;	●	●	18.2606	●	●
SCFH	FCV 104 fb;	●	●	-0.51132	●	●
psig	PIR 104;	●	●	77.4495	●	●
°C	TIR 104;	●	●	18.0783	●	●
SCFH	FCV 106 fb;	●	●	-0.530432	●	●
psig	PIR 106;	●	●	23.8352	●	●
°C	TIR 106;	●	●	0	●	●
psig	PIR 108;	●	●	-1.1209	●	●
psig	PIR 110;	●	●	6.14896	●	●
psig	PIR 112;	●	●	35.3592	●	●
psig	PIR 114;	●	●	-1874.99	●	●
psig	PIR 116;	●	●	3.31311	●	●
psig	PIR 118;	●	●	-12.9742	●	●
		●	●	0	●	●
		●	●	0	●	●
		●	●	0	●	●
		●	●	0	●	●
		●	●	0	●	●

Controller Chassis Error
status ✓ code 0
source

Ethernet Chassis Error
status ✓ code 0
source

Output SV Error
status ✓ code 0
source

Programming Error
status ✓ code 0
source

TIC Gr. 2 SV Error
status ✓ code 0
source

Current Time
12:27:24 PM
11/28/2011

HYDROGEN GAS DETECTORS

GD 102 0.00 ppm
GD 302 0.00 ppm
GD 402 0.00 ppm

FLAMMABLE GAS DETECTORS

GD 202 0.00 %LEL
GD 304 0.00 %LEL

HOUSE AIR 0.00 psig

Figure A-3. Administration screen.

APPENDIX B

START-UP VALVE POSITIONS

NOMENCLATURE

AV	automated valve
BSA	bottoms stripper accumulator
CA	condensate accumulator
FB	fluid-bed
FCV	flow control valve
HP	high-pressure
HP/HT	high-pressure/high-temperature
HP/LT	high-pressure/low-temperature
HV	hand valve
LIC	level indicator/controller
LP	low pressure
PCV	pressure control valve
PI	pressure indicator
PIR	pressure indicator recorder
PVC	pressure valve control
TO	thermal oxidizer

Area	Tag No.	Valve Name	Start-Up Position
Purged Vent Line	HV 002	First-floor drain valve	C
Purged Vent Line	HV 004	Second-floor drain valve	C
Dirty Vent Line	HV 020	Knock-Out Pot 1 drain	C
Dirty Vent Line	HV 022	Knock-Out Pot 2 drain	C
Dirty Vent Line	HV 024	2-inch dirty vent stack drain	C
100	HV 102	Hydrogen supply isolation	C
100	HV 104	Nitrogen supply isolation	C
100	HV 106		C
100	HV 108		C
100	HV 110	Reaction hydrogen FCV isolation – inlet	C
100	HV 112	Reaction hydrogen FCV isolation – outlet	C
100	HV 114	Purge nitrogen FCV isolation – inlet	C
100	HV 116	Purge nitrogen FCV isolation – outlet	C
100	HV 118	Hydrogen/nitrogen isolation	C
100	HV 124	H_2 supply vent	C
100 N	HV 132	Nitrogen supply isolation	C
100 N	HV 134	Nitrogen supply isolation	C
100 N	HV 136	PIR 114 isolation	O
100 N	HV 138	Nitrogen cylinder valve	C
100 N	HV 140	Nitrogen cylinder valve	C
100 N	HV 142	PI isolation	O
100 N	HV 144	Nitrogen accumulator vent	C
100 N	HV 146	Blocking to Area 100	C
100 H	HV 148	Hydrogen cylinder valve	C
100 H	HV 150	Hydrogen cylinder valve	C
100 H	HV 152	Hydrogen cylinder bleed valve	C
100	AV 104	Hydrogen accumulator/FCV isolation	C
100	AV 106	Nitrogen accumulator/FCV isolation	C
100	AV 108		C
100	AV 110	Reaction hydrogen FCV blocking	C
100	AV 112	Purge nitrogen FCV blocking	C
100	AV 114	Hydrogen/nitrogen isolation	C
200	HV 202	Slurry mix tank P-202 inlet	O
200	HV 204	Slurry mix tank P-202 outlet	O
200	HV 206	Slurry mix tank recycle	O
200	HV 208	Slurry feed tank inlet	C
200	HV 210	Slurry feed tank P-204 inlet	O
200	HV 212	Slurry feed tank P-204 outlet	O
200	HV 214	Slurry feed tank recycle	O
200	HV 216	Solvent tank P-206 inlet	O
200	HV 218	Solvent tank P-206 outlet	O
200	HV 220	Slurry line P-208 outlet	C
200	HV 224	Solvent line P-210 inlet	O
200	HV 226	Solvent line P-210 outlet	C
200	HV 250	Slurry mix tank drain	C
200	HV 252	Slurry feed tank drain	C
200	HV 254	Solvent tank drain	C

Continued. . .

(continued)

Area	Tag No.	Valve Name	Start-Up Position
200	HV 256	Quench pot drain	C
200	HV 258	HP slurry pump – slurry inlet isolation	C
200	HV 260	HP slurry pump – solvent inlet isolation	O
200	AV 202	Slurry cutoff	C
200	AV 204	Flush solvent cutoff	C
300	HV 302	Fluidizing N_2 isolation	C
300	HV 306	Blow out – HP dP line	C
300	HV 308	HP leg dP isolation	O
300	HV 310	LP leg dP isolation	O
300	HV 312	Blow out – low pressure dP line	C
300	HV 314	FB fill	C
300	HV 316	FB drain	C
300	HV 318	FB vent stack HX drain	C
400	HV 402	HP/HT separator/stripper – bottoms isolation	O
400	HV 404	HP/LT Separator – bottoms isolation	O
400	HV 406	BSA – bottoms isolation	O
400	HV 408	CA – bottoms isolation	O
400	HV 410	Sample gas line	C
400	HV 420	PCV 418 inlet isolation	O
400	HV 422	PCV 420 inlet isolation	O
400	HV 424	PCV 418 outlet isolation	C
400	HV 426	PCV 420 outlet isolation	C
400	HV 430	HP/HT separator/Stripper drain	C
400	HV 432	HP/LT separator drain	C
400	HV 438	LIC 404 lo-side isolation	O
400	HV 440	LIC 404 hi-side isolation	O
400	HV 444	LIC 404 lo-side drain	C
400	HV 446	BSA drain	C
400	HV 448	CA drain	C
400	HV 454	LIC 408 lo-side isolation	O
400	HV 456	LIC 408 hi-side isolation	O
400	HV 460	LIC 408 lo-side drain	C
400	HV 462	PCV manual bypass to TO	C
400	HV 464	PCV manual bypass to TO	C
400	HV 466	Liquid knock-out drain	C
400	HV 468	Liquid knock-out drain	C
400	HV 470	Gas meter inlet isolation	C
400	HV 472	Gas meter outlet isolation	C
400	HV 474	Gas meter bypass	O
400	HV 476	BSA product collection pot vent isolation	O
400	HV 478	CA product collection pot vent isola.	O
400	AV 402	HP/HT separator/stripper – top lock valve	C
400	AV 404	HP/HT separator/stripper – bottom lock valve	C
400	AV 406	HP/LT separator – top lock valve	C
400	AV 408	HP/LT separator – bottom lock valve	C
400	AV 410	BSA – top lock valve	C

Continued. . .

(continued)

Area	Tag No.	Valve Name	Start-Up Position
400	AV 412	BSA – bottom lock valve	C
400	AV 414	CA – top lock valve	C
400	AV 416	CA – bottom lock valve	C
400	PCV 418	PCV	C
400	PCV 420	PCV	C

APPENDIX C

COAL LIQUIDS DISTILLATION PROCEDURES
AS OF 25 JULY 2012

COAL LIQUIDS DISTILLATION PROCEDURES AS OF 25 JULY 2012

Ramesh K. Sharma

A semiautomatic distillation system was installed at the Energy & Environmental Research Center (EERC) to support direct coal liquefaction (DCL) process development activities. The system was designed to enable performance of relatively simple as well as complex ASTM International methods such as D2892 and D5236. Since the system was designed for the distillation of crude oil and petroleum products, its use in coal liquids distillation requires some tailoring of standard methods for efficient and safe operation. This system's automation minimizes the operator time needed to perform the test. The equipment design makes the test straightforward and easier to perform. This system is installed in a laboratory adjacent to the DCL system. It is in a large hood for good ventilation and to make it easier and safer for the technicians to operate and do maintenance and cleaning.

OBJECTIVES

1. **Production of recycle solvent:** The primary objective of the unit is to separate the coal liquids produced by the DCL unit into light distillate (<600°F) and heavy distillate (600°–1000°F). The light distillate will be sent to an upgrading facility to convert the coal liquids into usable fuels, and heavy liquid will be used as a recycle solvent for the continuous operation of the DCL unit.

2. **Production of fuel samples:** The liquid distillate will be upgraded by hydroprocessing comprising hydrotreating and hydrogenation to produce clean fuels. The hydroprocessed distillate will be fractionally distilled in this distillation system according to ASTM standards D86 or D2892 to produce transportation fuels such as naphtha, aviation fuel, and diesel.

This semiautomatic distillation system is installed next to the DCL unit that will be used to distill coal liquids produced from the direct liquefaction of coals to produce recycle solvent or fuels from catalytic upgrading of light distillate. The equipment design makes the test straightforward and easier to perform. The fully assembled distillation unit is shown in Figure C-1.

Distillation Column Configuration

The packed column configuration will be used to distill the crude oil up to a maximum 450°C atmospheric-equivalent vapor temperature. It is sometimes called true boiling point distillation or D2892. This distillation column has 15 theoretical plates and complies fully with ASTM D2892. Although a variety of column packing types are available, including Propak, Helipak, and structured packing, this system has Propak columns. Other packing types are also available. It will have a 12-L pot. Pot sizes of 2 to 100 L are also available to be used if needed.

EERC TA44554.CDR

Figure C-1. Off-line distillation system

A spinning band distillation column that can accommodate pot sizes from 10 L to 12 L will be installed in addition to traditional packed columns and vacuum pot stills. The vacuum pot still configuration is typically used to distill the residual material from the D2892 distillation out to atmospheric-equivalent vapor temperatures up to 565°C or higher. The vacuum pot still complies fully with ASTM D5236. Pot sizes of 1 to 22 liters are available. Because of the quicker turnaround time, this column will be used for distillation to produce recycle solvent to support continuous operation of the DCL unit. Usually, one distillation column is the D2892 packed column (true boiling point) and the other is the D5236 vacuum pot still column. Only one distillation column will be operated at any time. Both distillation columns will share the same controller, fraction collector, vacuum control system, and recirculating bath.

Automation and Accessories

The crude oil distillation equipment will operate in a semiautomatic mode and will require limited attention, including that of changing the receiver flasks. The option to buy this semiautomatic distillation unit was based on the ease of operation and the lower cost of a fully automatic unit. Although this crude oil distillation is semiautomatic, to some extent, it will make the crude oil distillation process easy and efficient from beginning to end.

Microprocessor Controller

The microprocessor will minimize the amount of operator time required for each crude oil distillation. It can call up your stored programs from one of 19 available memory locations and let the microprocessor perform the crude oil distillation. Each fraction has programmable open and closed temperature cut points, heat rates, and reflux ratio. There is a programmable shutdown at the end of the distillation run. The microprocessor allows easy editing of each of the parameters while the crude oil distillation is in progress.

Fraction Collection

The fraction collector can automatically collect crude oil fractions based on temperature cut points at atmospheric pressure or under vacuum. Receivers are graduated for easy observation of the cut volume by the operator. The fraction collector that was chosen for this system is an eight-place unit.

Vacuum System

Distillation of crude oil above 200°C must be performed at reduced pressures. The system includes a 2-stage mechanical vacuum pump, cold trap, pressure sensor, and proportional control valve. The operator can input a vacuum set point from 100 mm of Hg to 0.1 mm of Hg, and the vacuum system will automatically control vacuum to the set point level.

Recirculating Bath

The recirculating bath controls the condenser temperature in the range of –20°C to 100°C. The operator inputs the desired temperature, and the bath automatically controls the temperature to that set point.

Distillation Procedure

The distillation system consists of two columns, namely a spinning band column and a packed column. The spinning band column will be used when a simple distillation is desired to fractionally distill the coal liquids to generate only light and heavy distillates, and the packed column will be used for more precise ASTM D86 and D2892 distillations to generate fuel samples. Both of these columns will be attached to the distillation flask via a high-vacuum stopcock to provide easy switching between these two types of distillations as needed.

1. Since the coal-derived liquids have a strong odor and are potential health hazards, all operations must be performed in the hood. Also, extreme care should be taken during collection and transfer of these liquids from the DCL unit to the distillation assembly. If there is a spill, then the area should be vacated, and only skilled operators should clean up the spill

with disposable wipes and acetone and dispose of the cleanup material as a hazardous substance.

2. Personnel should become familiar with the various components of the distillation system unit and get comfortable with the unit operation. The major components of the distillation unit are shown toward the end of the “Procedure” section.

PROCEDURE

1. Fill the distillation flask containing a 1-inch egg (or octagonal) stir bar with 6–8 liters of coal liquids. Please make sure the flask is never filled to more than 60% of its capacity. The flask is placed in the heating mantle, and the heating mantle rests on a stirrer.

2. Attach the appropriate column to the flask, and then complete the rest of the assembly as shown in Figure C-1. In order to have a leak-free assembly, make sure to apply vacuum grease to all ground-glass joints. If there is still a leak in the system, you will be able to quickly know if you are not reaching near-full vacuum each time. If that happens, then make sure to tighten the vacuum tubing with clamps.

3. During this run, you are going to have to insulate your fractionating column. Glass wool is the best heat-resistant insulator. If you find it at your hardware store, make use of it. Fleece also works incredibly well but will not tolerate temperatures above 150°C. If you expect your oil to come across above that temperature and cannot find glass wool, cotton cloth (such as pieces of a towel or sock) wrapped around the column will suffice. Use wire to keep the insulating material on. If you are distilling your oil at a higher temperature, you will need insulation; otherwise, too much oil will fall back into the distillation pot as reflux. If you have a good vacuum (<10 mm Hg) or your column is silvered and vacuum-jacketed, you likely can get away with no insulation.

4. Next, place the three-way adapter on top, then the condenser, then the vacuum adapter, and attach round bottomed flasks to the condenser as the receiving flasks. You should have placed a clamp at the fractionating column and at the receiving flask. If desired, you can optionally place another clamp on the condenser. Last, fit your thermometer to your thermometer adapter (if you are using one) and place your thermometer into the three-way adapter. Get your vacuum ready, but do not connect it or turn it on.

Now, set the stirrer to spin at a high rate, and heat the mantle at a rate that matches the distillation to the temperature at which you expect the coal liquid to come over. The temperature of the heating mantle and the liquid in the distillation pot will be constantly displayed by the digital controller and recorded on the PC. Keep temperature constant. Make sure to control the heating rate to match the distillation of the oil as indicated by the product dripping into the receiver flask. In general, the product should distill in drops (1 to 2 drops/second) rather than stream. If the heating rate is not controlled, then you run the risk of vastly overshooting the point at which the oil is going to come over and it will come over too quickly into the receiver flask. The quick heating may lead to bumping the coal liquid into the receiver flask without any fractionation. If this happens, then you have to start all over again. Since the liquefaction product contains water, naphtha, middle distillate, and heavy oil, then it is absolutely necessary to start the initial distillation at ambient pressure. If you are using a spinning band configuration to produce the recycle solvent, then the fractions of interest are as follows:

a. Naptha (<300°F)
b. Middle distillate (350°–3650°F)
c. Heavy distillate or recycle solvent (>650°F)

If an upgraded coal liquid is distilled to produce finished fuel, then the distillation will be performed using a packed column. In this case, the fractions of interest are described below:

a. Naptha (<160°F)
b. Naphtha (160°–300°F)
c. Jet fraction (300°–500°F)
d. Diesel or heavy oil (>500°F)

5. A quick indicator of the changes from one to the other fraction is that no more oil comes out. At this stage, you will have to increase the temperature or increase the vacuum or both so that the next fraction starts to come over. At that point, you will see the temperature on your thermometer climb quickly and hit the temperature at which the send fraction will begin to collect. A good visual indicator of a different fraction starting to come over is that there will be little “swirls” in the receiving flask as the new liquid drips in. This is because the index of refraction of the next fraction is different.

6. Please record the weight of each fraction as well as the residual liquid and pot.

After completion of distillation, make sure the unit is clean for the next distillation. The best way to clean is to use acetone to remove all of the oils, then wash the unit with soapy water (if you feel it necessary). If there is any charring in your distilling flask, rinse with acetone, then let it soak in household ammonia or 10% sodium hydroxide for a few hours. Then use a brush to easily scrub it out.

Please perform this simple distillation to familiarize yourself with it before using the more complex semiautomatic distillation. This practice is necessary to avoid any damage to the rather expensive unit.

APPENDIX D

PROCEDURES FOR BATCH DCL-BASED JET FUEL PRODUCTION

PROCEDURES FOR BATCH DIRECT COAL LIQUEFACTION-BASED JET FUEL PRODUCTION

INITIAL LIQUEFACTION FOR VACUUM GAS OIL (VGO) PRODUCTION

Initially, the liquefaction tests of a mixture of predried Illinois No. 6 coal and creosote oil were carried out using sulfided Co-Mo catalyst to produce Illinois No. 6 coal-derived solvent vacuum gas oil (VGO) for subsequent testing of the coal to produce a middle distillate. These tests used solvent to coal ratio of 1.6 bottom mixed with 100 ppm catalyst.

In a typical once-through run, a slurry consisting of approximately 700 g of dry coal, 998 g of the solvent VGO, and 1.1337 g of catalyst was placed in the 2-gallon autoclave equipped with mechanical stirring and hydrogen flow-through capability. The reactor was charged with 1300 psig hydrogen pressure and placed in the heating jacket. The reactor was then heated to the desired temperature; heating continued for 1 hour in a hydrogen flow mode. At the end of the run, the reactor was cooled to room temperature. The gas was discharged through a sodium hydroxide trap (aqueous 10% NaOH) and collected in a gas bag using a wet-test meter. The reactor was opened, and the contents of the reactor were distilled under high vacuum (Figure D-1) to obtain the following fractions:

- Gas fraction (up to C4)
- Naphtha (C5 – 160°F)
- Naphtha (160°–350°F)
- Distillate (350°–650°F)
- VGO (650°–1000°F)
- Vacuum bottoms (>1000°F)

Liquefaction for Middle Distillate Production

Once the desired quantities of Illinois No. 6 coal-derived solvent were prepared, further testing was carried out to generate a 1-liter sample of middle distillate for further hydroprocessing/product quality testing to produce a 500-mL jet fuel sample. The tests for producing middle distillate were carried out in a 2-gallon autoclave with flow-through capability.

In a typical bottom-recycle run, a slurry consisting of approximately 900 g of the dry coal, 802 g of vacuum bottoms obtained from initial testing, 802 g of the solvent VGO, and 0.60 g of catalyst was placed in the 2-gallon autoclave equipped with mechanical stirring and hydrogen flow-through capability. The reactor was charged with 1300 psig hydrogen pressure and placed in the heating jacket. The reactor was then heated to the desired temperature, and heating continued for 1 hour in a hydrogen flow mode. At the end of the run, the reactor was cooled to room temperature. The gas was discharged through a sodium hydroxide trap (aqueous 10% NaOH) and collected in a gas bag using a wet-test meter. The reactor was opened, and the contents of the reactor were distilled under high vacuum to obtain the following fractions:

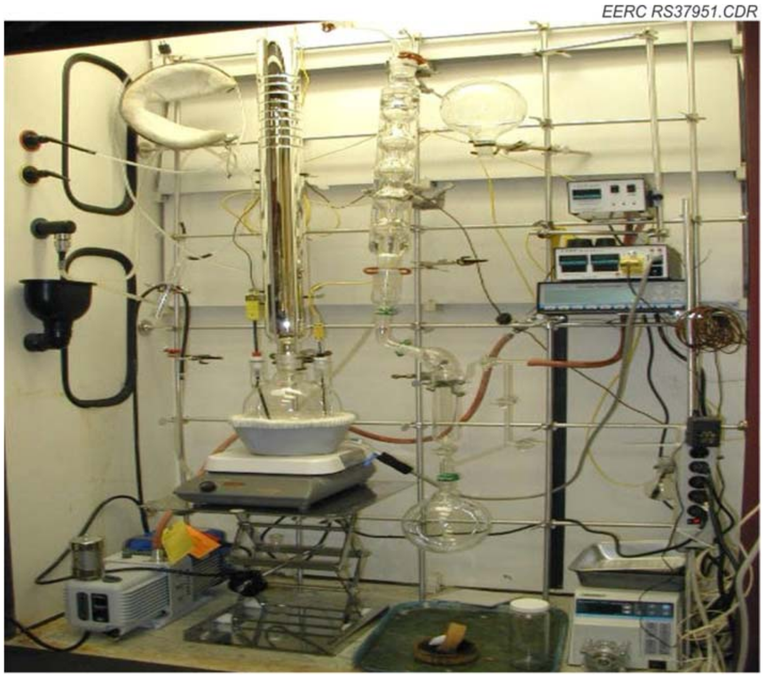


Figure D-1. Bench-scale fractional distillation system.

- Gas fraction (up to C4)
- Naphtha (C5 – 160°F)
- Naphtha (160°–350°F)
- Distillate (350°–650°F)
- VGO (650°–1000°F)
- Vacuum bottoms (>1000°F)

UPGRADING OF COAL DISTILLATE

The middle distillate produced from the direct liquefaction of coal was upgraded by a two-stage catalytic hydrotreatment and saturation to produce naphthenic fuel derived from coal.

Hydrotreating

The catalytic hydrotreatment of the middle distillate (350°–650°F) using KF-841 catalyst was carried out in a small continuous reactor (SCR) described in Figure D-2 to produce JP-8-compliant jet fuel. Hydrotreatment of the middle distillate was carried out to remove heteroatoms such as nitrogen, sulfur, and oxygen along with hydrocracking and hydrogenation. Initial tests were conducted using middle distillate produced from once-through and first bottom-recycle runs (slop) to validate the catalysts and reactor system and optimize the conditions. The hydrotreatment was carried out in a tubular reactor packed with KF-841 (Ni–Mo) catalyst supplied by Albemarle. The catalyst was diluted with an equal volume of glass beads. The catalyst packing, presulfidation, and operating conditions are described below:

EERC RS37934.CDR

Figure D-2. SCR system.

Procedure

Note: Requires 24-hr continuous unit operation coverage.

1. Install reactor in upflow mode of operation.

2. Place feed pump in Plexiglas box (or equivalent) to maintain a constant feed temperature (i.e., constant density).

3. Place two liquid drain pots (accumulators) in service for alternating product collection from the condenser.

4. Suggest installing double-block values (if not already) on both product drain (accumulator) pots.

5. If available, install online analyzer to monitor H_2S breakthrough during catalyst sulfiding step.

6. If analyzer is not available, provide a port to use a Dräger pump and tube for H_2S detection.

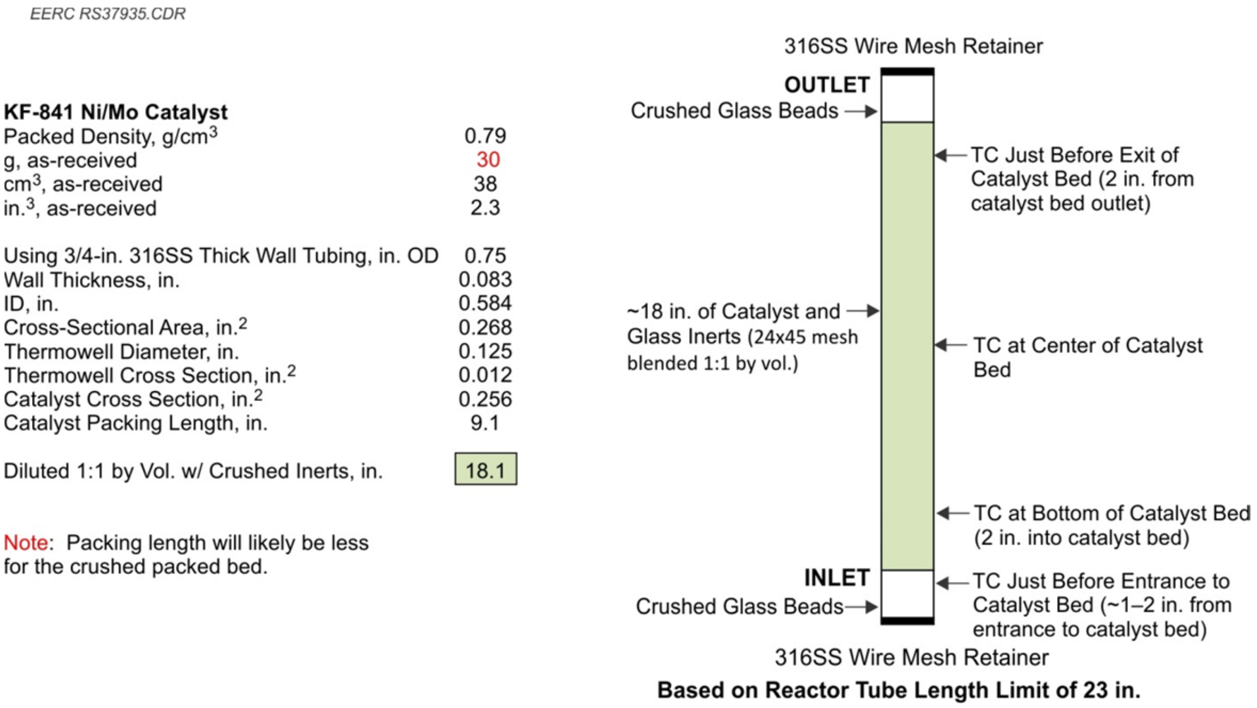
Hydrotreating Run (Stage 1) Is as Follows:

1. Unit preparation
   a. Rinse out unit without reactor inline.
   b. Pressure test: cold with N_2 to 2500 psig.

c. Pressure test: cold with H_2 to 2500 psig.
d. Check H_2/distillate feed flow calibrations.
e. Load catalyst in reactor (24 × 45 mesh crushed, 1:1 crushed inert dilution; Figure D-3).
f. Install reactor and pressure-test: cold with H_2 to 2500 psig.

2. Unit operations
   a. Dry out catalyst in reactor (~8-hr heatup and hold at 300°F).
   b. Wetting catalyst with surrogate distillate (~4 hr at 250°F).
   c. Heatup and two-step catalyst activations (dimethyldisulfide [DMDS] in surrogate distillate) ~16 hr.
   d. Line-out and activity check with “off-spec.” distillate.
   e. Switch to “on-spec.” distillate.
   f. Off-spec. hydrotreated (HT) product collection (for Stage 2 break-in feed).
   g. Switch to on-spec. HT product collection.

3. Distillate inspection
   a. Product distillation, if deemed necessary (save 350°F)
   b. Product analysis (H_2S and NH_3 stripped, if no distillation performed)
   c. Product evaluation (for confirming/adjusting Stage 2 conditions)


EERC RS37935.CDR

KF-841 Ni/Mo Catalyst	
Packed Density, g/cm^3	0.79
g, as-received	30
cm^3, as-received	38
in.^{3}, as-received	2.3
Using 3/4-in. 316SS Thick Wall Tubing, in. OD	0.75
Wall Thickness, in.	0.083
ID, in.	0.584
Cross-Sectional Area, in.^{2}	0.268
Thermowell Diameter, in.	0.125
Thermowell Cross Section, in.^{2}	0.012
Catalyst Cross Section, in.^{2}	0.256
Catalyst Packing Length, in.	9.1
Diluted 1:1 by Vol. w/ Crushed Inerts, in.	18.1

Note: Packing length will likely be less for the crushed packed bed.

Figure D-3. Packed-bed tubular reactor for hydrotreating of middle distillate (TC is thermocouple).

Start-Up Procedure

1. Water-wash the start-up distillate to remove Cl (use a separation funnel with at least three washes, until Cl <20 ppm by x-ray fluorescence).
2. Dry catalyst in situ.
3. Purge unit with inert gas (N_2) after pressure testing.
4. Pressure H_2 to 300 psig.
5. Set H_2 rate to 270 cm^3.
6. Heat to 300°F at 50°F/hr rate.
7. Check knock-out (KO) pot for water and drain.
8. Maintain at 300°F for at least 1 hr.
9. Check KO pot for water and drain.
10. When little or no water is in pot, lower temperature to 250°F.
11. Introduce distillate feed at 15 cm^3/hr to wet the catalyst.
12. Continue wetting for 4 hr (~60 cm^3 of distillate).
13. Collect and save the wetting distillate to reuse for catalyst break-in use.
14. Blend sulfiding distillate one pump bath at a time to minimize distillate use for sulfiding step.
15. Add 1.5% sulfur as DMDS to water-washed start-up distillate (2.25 g DMDS per 100 g distillate).
16. Increase reactor temperature to 450°F (take care not to overshoot by more than a few degrees).
17. Replace break-in distillate with sulfiding distillate feed at 15 cm^3/hr when close to 450°F.
18. Hold temperature at 450°F (take care not to allow any exotherm to exceed 475°F, which can promote coking and metal oxide reduction).
19. Begin to continuously monitor product gas for H_2S breakthrough with online laser analyzer or with a H_2S Dräger tube (caution – because of high H_2S concentrations, use a proper face mask and vacuum snorkel trunk).

20. Continue to sulfide until a steady, copious breakthrough of H_2S is observed (~3000 ppm).

21. Empty KO pot as needed and save the product (caution – because of high H_2S concentrations, use a proper face mask and vacuum snorkel trunk).

22. Once H_2S breakthrough is observed, increase temperature to 650°F at 25°F/hr (~8-hr heatup time).

23. Hold until H_2S breakthrough is steady at >1000 ppm in the product gas.

24. Replace sulfiding feed with break-in feed.

25. Adjust process conditions to program settings.

Procedure

1 Adjust process conditions to program run conditions.

2 Allow 4 hr of initial line-out with break-in distillate feed (~60 cm^3).

3 Switch to alternate product accumulator pot.

4 Drain sample from first pot into glass bottle and label with time and date (caution – this sample will contain H_2S).

5 Purge product to remove H_2S and NH_3 (~30 min of mild N_2 bubbling) in enclosed lab hood (repeat this procedure with each subsequent sample).

6 Continue to collect a product sample every 4 hr (~10 samples total over 2 days of line-out).

7 During the line-out period, adjust reactor temperature in accordance with instructions to meet product targets.

8 When line-out feed is consumed, switch to “on-spec.” program distillate feed (~5–6 days of operation).

9 Continue with above sampling procedure every 4 hr until instructed otherwise.

10 Adjust reactor temperature as needed to meet product targets.

11 Sampling intervals may be extended based on analytical test feedback.

12 Combine distillate product glass bottle samples from line-out operations as instructed.

13 Combine distillate product glass bottle samples from program operations as instructed.

14 Submit both products for final analytical tests indicated in the “Analytical Targets” sheet.

15 Note: Save all liquid samples, including the distillate from sulfiding operation after stripping out H_2S.

After the desired amount of middle distillate was hydrotreated, a gentle stream of nitrogen was bubbled through the sample to remove the dissolved ammonia and hydrogen sulfide produced during the process. The sample was analyzed by simulated gas chromatography distillation (GCD) and gas chromatography–mass spectroscopy (GC–MS), elemental analysis, and density measurement.

Saturation

The product from Stage 1 hydrotreatment contained almost 30% aromatic components. In order to produce a fully naphthenic jet fuel, the hydrotreated product was further hydrogenated using a saturation catalyst, Criterion 654 catalyst. The hydrogenation was carried out in a SCR described above. The catalyst diluted with an equal volume of glass beads was packed in the reactor as shown in Figure D-4. The reaction procedure is described below.

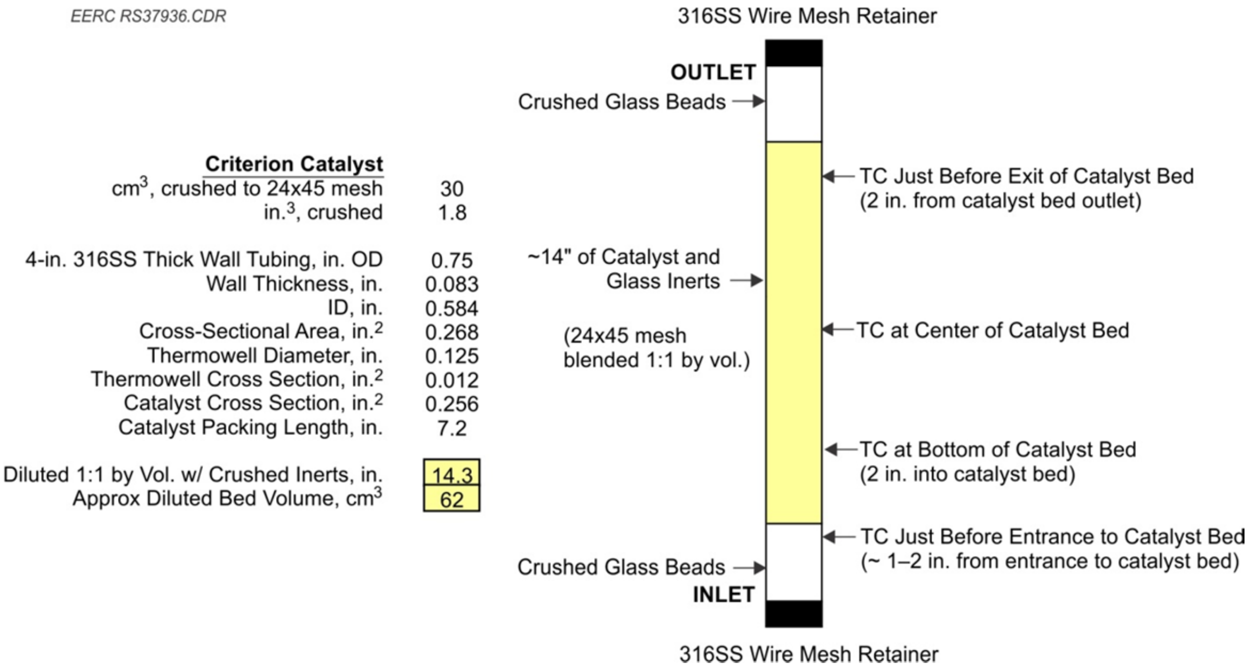


Figure D-4. Packed-bed tubular reactor for saturation of hydrotreated middle distillate.

Saturation Run (Stage 2 upgrading) Procedure

Catalyst preparation:

1. Measure out and record weight of 30 cm^3 of “as-received” catalyst.

2. Crush catalyst to 24 × 45 mesh size.

3. Crush enough catalyst with an equal volume of 24 × 45 mesh crushed Pyrex beads.

4. Load the 1:1 by volume mixture into the reactor as indicated in the reactor diagram.

Catalyst activation of criterion’s recommendation procedure:

1. Install reactor after pressure testing unit.

2. Start nitrogen flow through unit at ambient pressure.

3. Heat reactor to 500°F (260°C) and hold for 1 hr – caution do not exceed 545°F (285°C) in the reactor.

4. After 1 hr at 500°F, cool to 300°C (150°C) with nitrogen still flowing.

5. At 300°F, switch from nitrogen to hydrogen flow.

6. Adjust unit conditions to 1500 psig and 785 cm^3 hydrogen feed rate.

7. Hold at 300°F (150°C) for 4 hr.

8. After 4 hr at 300°F, increase temperature to 525°F (275°C) at 50°F/hr (28°C/hr).

9. Hold at 525°F (275°C) for at least 2 hr.

10. Cool to 300°F (150°C).

11. At 300°F (150°C), start the distillate feed at 33 cm^3/hr.

AROMATIC SATURATION CONDITIONS

- Temperature: 300°F
- Pressure: 1500 psig
- Hydrogen feed: 785 cm^3
- Liquid flow: 33 cm^3/hr
- Liquid hourly space velocity: 0.532

After the desired amount of middle distillate has been saturated, a gentle stream of nitrogen was bubbled through the sample to remove any dissolved ammonia and hydrogen sulfide produced during the process. The water was separated from the saturated product. The sample was analyzed by simulated GCD and GC–MS, elemental analysis, and density measurement.

Preparation of Jet Fuel Sample

The product obtained from two-stage hydrotreatment of middle distillate was fractionated, and the jet fraction was analyzed by various American Society for Testing and Materials standards to evaluate compatibility with JP-8 specifications. After a series of fractional distillations and analysis, the fraction distilling in 250°–475°F was found to meet most of the JP-8 specifications tested so far. This sample was mixed with a renewable (canola) oil-derived isoparaffin-rich jet fuel blendstock at a blend ratio of about 60% coal-derived material–40% renewable oil-derived material to produce a composite fuel. This fuel was tested in-house to evaluate target properties of petroleum-derived jet fuel (JP-8). After confirming that the sample met the target specifications for JP-8, the sample was submitted to Air Force Research Laboratory for evaluation.

APPENDIX E

DCL SYSTEM SHAKEDOWN PLANS

DCL SYSTEM SHAKEDOWN PLANS

SYSTEM SHAKEDOWN

The purpose of the system shakedown is to make sure that all components of the unit are working as expected. The system should be able to perform at high temperature and high pressure for extended periods of time. The shakedown is a five-part operation:

1. An initial direct coal liquefaction (DCL) system leak check will be performed using nitrogen, with insulation off the fittings, tubing, and vessels so that each connection and joint can be checked. Initial leak checks will be performed with nitrogen at ambient temperature at pressures of 500, 1000, 1500, 2000, and, finally, at 2750 psig. Leaks will be located using a combination of an ultrasonic leak detector and bubble-producing leak-detecting solution. Follow-up pressure checks will be conducted at 2750 psig using a blend of nitrogen and helium. Helium will act as a surrogate for hydrogen. A LACO GasCheck G3 hand-held thermal conductivity helium/hydrogen sniffer will be used to detect leaks. A final leak check will be performed at 2500 psig using hydrogen.

2. After verifying that the system can hold pressure, functionality of the level-monitoring systems on the product collection vessels as well as functionality of the liquid letdown valves and system pressure control valves will be assessed. These tests will be performed at ambient temperature and at pressures of ambient to 2750 psig under a nitrogen atmosphere. Hydrogenated anthracene oil will be used as the test liquid at a nominal pumping rate of 2 liter/hour (34 cm^3/min). (This rate is determined on the basis of a 60-minute residence time for the oil in the reactors).

3. Similar tests to those of Step 2 will then be performed at operating temperature and pressure. The system will be heated to 450°C with nitrogen at 2750 psig, and another series of leak checks will be performed. The system will then be cooled down to 200°C, after which the nitrogen will be replaced with hydrogen and the pressure reduced to 2500 psig. Pumping of hydrogenated anthracene oil will then be initiated at a nominal 2-liter/hour flow rate. The temperature of the reactor system will be increased to 312°C at a heat up rate of 56°C/hr and then to a final temperature of 450°C at 28°C/hr. Testing with anthracene oil will be conducted for a minimum of 2 hours, allowing the desired liquid levels in the separators to be achieved and steady level and pressure control attained.

4. Subsequent shakedown testing will be performed using a slurry consisting of solvent and coal (solvent/coal ratio = 3.2), again at 2 liter/hour (34 cm^3/min). As satisfactory system performance is attained, the solvent/coal ratio will be incrementally reduced until a ratio of 1.6 (solvent/coal) is attained.

5. If the shakedown using a solvent/coal mixture is successful, the slurry mixture will be modified by adding vacuum bottoms. The initial slurry will consist of a solvent/coal/

bottoms ratio of 1/1/0.5 with the ratio increased to 1/1/1. All tests will be conducted for at least 2 hours at a 2-liter/hour (34-cm^3/min) slurry flow rate.

In addition to monitoring all system pressures, temperatures, and liquid and gas flow rates, the effectiveness of the stripping hydrogen and stripping recycle gas for effecting the proper product separations will be assessed. The bottoms and condensate removed during Steps 4 and 5 will be processed (see distillation manual) to allow for recycle and product evaluation.

PRODUCTION OF MIDDLE DISTILLATE

Once a successful shakedown is completed, the system is ready for production of distillate fuel. Initially a “once-through operation” will be performed using a slurry consisting of solvent/coal (ratio = 1.6) and a 100-ppm catalyst. The slurry will be fed to the system for 24 hours to generate 50 pounds of vacuum gas oil and bottoms each for use in the “bottom recycle run.” For the bottom recycle run, a slurry consisting of solvent/coal/bottom (ratio = 1/1/1) and a 100-ppm catalyst will be pumped for 10 hours to generate enough condensate to produce approximately 3 L of middle distillate upon distillation of the condensate.