# IV. DETAILED DESCRIPTION OF TECHNICAL PROGRESS

#### A. Pilot Plant Installation

## 1. Completion of Pilot Plant Construction

Before the Liquid Phase Methanation (LPM) Pilot Plant could be shipped to its demonstration site, several items remained to be completed under phase III of Contract No. E(49-18)-1505. The most crucial was the repair of 213 welds which failed to meet 100 percent radiography standards. At the beginning of July, 1976, 151 of these defective welds had been repaired, 101 repaired welds had been radiographed, and 73 weld repairs were determined to be acceptable. The remaining 28 repaired welds required a second round of repairs.

The weld repair program proceeded slowly during July, 1976 due to a one-week closure for annual vacation at the fabricator shop (ICI Corp., Texas City, Texas), some inclement weather, which prevented outdoor work on the LPM skid, and lack of sufficient welders to speed up the repair program. Also, repair of the 4" welds, which was done "in place" on the LPM skid, was hampered by excessive rust within the piping, causing numerous repaired welds to fail to meet the 100 percent radiography inspection standards. This rust resulted from the long project delay and failure of ICI Corp. to dry out the piping after the hydrostatic test of September, 1975. An adequate cleaning and welding procedure was devised to overcome this problem, and, by the end of July, 1976, all but five of the welds had been repaired and passed radiographic inspection. Sections of 2" pipe, which had been removed earlier for easier weld repair within the fabricator ship, were reinstalled on the pilot plant skid.

Chem Systems personnel visited the ICI Corp. fabrication shop on July 14 and 15, 1976, to determine the progress in weld repair and to inspect the LPM skid to see whether deficiencies found during earlier inspection

trips had been corrected. Most items from the earlier checkout lists were found corrected and a list of deficiencies still requiring work was left with the engineering contractor, Davy Powergas.

Weld repair to 100 percent radiography standards was completed early in August, 1976. Following this, all rails and gratings were installed on the pilot plant skid and the unit prepared for hydrostatic testing by partitioning the piping into five sections. The hydrostatic test was performed on August 10, 1976 and witnessed by personnel from Chem Systems and Davy Powergas. Each piping section was submitted to a hydrostatic test pressure of 150 percent of the normal operating pressure for a period ranging from 1.5 to 2 hours. The five test sections performed well with only very minor leakage at flanged joints.

At the end of the hydrostatic test, all vessels and piping were drained, all piping blanks were removed and the piping was reconnected. The system was not dried by air blowing at that time, however, because the air compression system at ICI Corp. was malfunctioning. This problem was left with ICI Corp. and Davy Powergas to solve.

Chem Systems personnel inspected the pilot plant, exclusive of the control room, upon completion of hydrostatic testing on August 11, 1976. A number of discrepancies and omissions were noted and transmitted to Davy Powergas personnel for immediate rectification. Included in this list were replacement of screwed fittings in the pump seal flush system with socket welded fittings, rebuilding the reactor level detector lift mechanism with a workable design, revising all process flow diagrams to reflect the "as-built" condition of the pilot plant, adding all missing sections to the equipment data books, and other miscellaneous items.

Re-insulation of the process piping, originally exposed for X-ray testing and weld repair, was commenced on August 16, 1976. By the end of the month, this work had been completed and the paint retouched where necessary.

The construction phase of the Liquid Phase Methanation (LPM) Pilot Plant was completed and a thorough inspection was carried out by Chem Systems personnel on September 9-10, 1976. Other than some minor details which were to be corrected immediately, two items were found incomplete and were to be field installed, namely:

- Reactor level detector lift mechanism parts shipped with pilot plant.
- 2. Reactor oil pump seal flush piping system parts shipped with pilot plant and welded at demonstration site.

Disassembly of the LPM Pilot Plant in preparation for shipment to the designated demonstration site was begun on September 15, 1976. By September 20, ICI Corp. had finished and this task was completed.

### 2. Pilot Plant Relocation

Several vendors were interviewed during the July 14, 1976 visit to the ICI Corp. shop with respect to their interest in supplying services for preparing the pilot plant sections for shipment, rigging, truck shipment from Texas City to the designated demonstration site and chemical cleaning. The vendors were all recommended by Davy Powergas. Each vendor was requested to thoroughly examine the LPM skid assembly and submit copies of detailed bid proposals to Chem Systems by August 6, 1976. The proposals were to thoroughly describe services, materials and equipment to be supplied, to describe what the vendors required in the way of site cooperation, and to clearly state prices, terms of payment, insurance coverages and job references for similar work.

In early August, 1976, quotations for crating, rigging and truck shipment of the LPM Pilot Plant to one of the proposed demonstration sites were received by Chem Systems and vendors selected for preparing the skids for shipment. loading onto low-boys, and actual shipment.

Five low-boy trailers were delivered to ICI Corp., Texas City, on September 20, 1976. The rigger (Westheimer Rigging and Heavy Hauling Company) began work on the same day, and by September 22, all skid sections and the control house had been loaded on trailers. The crater (First Class Export Crating, Inc., Houston, Texas) began meaningful crating activities on September 21. By September 28, this work was completed. With Chem Systems personnel present on September 29, C & H Transportation Company truckers removed the five trailers from the ICI Corp. yard and the pilot plant was on its way to Institute of Gas Technology's HYGAS plant in Chicago, Illinois, selected by ERDA and AGA as the first demonstration site.

The four pilot plant skid sections and control room were delivered to the IGT plant during the first week in October, 1976. They were unloaded from the low-boy trailers in the IGT parking lot and moved to their specified location at a later date.

During the visit by Chem Systems personnel on July 14 and 15, 1976, the spare parts situation was reviewed at the Davy Powergas offices. A list of recommended spare parts was prepared upon the recommendation of the vendors of the various process items in the pilot plant and reviewed by various concerned DPG personnel. With the selection of IGT HYGAS plant as the first site, requests for quotations on all essential pilot plant spare parts were sent out to the appropriate vendors.

### 3. Pilot Plant Reassembly and Installation

Meetings were held at the HYGAS facility with Chem Systems, IGT and ERDA personnel on October 6 and 19, 1976 to discuss reassembly, installation and operation of the LPM Pilot Plant. A summary of these discussions and the decisions reached are presented in the paragraphs below.

Chem Systems was to provide technical supervision for reassembly and operation, including 24-hour per day coverage during the initial start-up, and then one man as resident for daily contact. The mechanical, electrical and instrument contractors at IGT were to do the reassembly work under IGT supervision. IGT also was to provide four operators for continuous coverage during initial start-up and operation.

The pilot plant was to be shaken down and operated with hydrogen from the HYGAS plant reformer except when gasifier product was available. The hydrogen stream, which by-passed the shift conversion system, can deliver 2MM SCFD at 1,000 psig and has a 6 to 1 hydrogen/carbon monoxide mole ratio. This gas was to be tested in the bench scale unit to check the kinetic model and determine the type of provess variable scans that can be performed. Chem Systems was to review its program for pilot plant operation and revise the test program to reflect the use of the 6  $\rm H_2/CO$  stream.

A detailed list of spare parts was to be provided to IGT personnel for their review. It was decided to order all items suggested by Chem Systems and these orders were subsequently placed.

Prior to assembly, major vessels were opened and inspected for proper placement of internals and corrosion. Since rusting was found to be minimal, it was decided to eliminate chemical cleaning from the schedule of events to take place during installation and shakedown.

It was decided to have two people from IGT visit Chem Systems in Hackensack to observe the PDU in operation in order to familiarize them with the LPM process.

The HYGAS site was prepared for the LPM skid and control room during October, 1976. Texas Nuclear was notified to ship the level detector nuclear source to IGT.

Chem Systems was to have its own analytical system in the control room, which includes a sampling system, chromatograph and integrator. IGT's analytical service was to be used as a back-up and additional check on analytical results. All equipment for the analytical system was ordered.

The following are some miscellaneous items discussed in the October, 1976 meetings:

- 1. The skids were to be anchored to the ground.
- 2. Gas feed to the LPM skid would be delivered at  $500^{\circ}F$  from zinc oxide beds. The LPM feed gas line is ASTM-A-106B seamless pipe, rated at 1,100 psig and  $450^{\circ}F$ . IGT's metallurgist checked suitability of this piping material.
- IGT was to modify the process oil filter lift system for easily removing the vessel heads so that filter elements can be changed rapidly.
- 4. Carbon steel bolts found in the LPM reactor sparger are to be replaced with stainless steel.

Early in November, 1976, the LPM skid and control room were moved into place on the prepared site and anchored. Work began immediately on connecting all interfacing lines, instrument systems and electrical

systems. By the end of November, most of this work was completed with the exception of the seal flush piping revisions, flanges on the oil inlet line, level detector lift mechanism, miscellaneous pneumatic hookups on the skid, main power supply to the control room and installation of the control room pressurization system.

A checklist of items requiring attention before the pilot plant could achieve full operation was prepared and kept up to date. Included in this list were a number of revisions considered necessary for reliable operation of the pilot plant. Most important of these was the addition of steam tracing and insulation to a number of lines and vessels to prevent freeze-up shutdowns.

By the end of December, 1976, some of the spare parts had arrived and the remainder were expedited. The nuclear source for the level detector arrived at IGT and was stored until needed. Instruments for the analytical system to be located in the control room began arriving at Chem Systems' Hackensack Laboratories. The analytical system was fully checked out in the laboratory before being shipped to IGT.

A revised experimental program for pilot plant operation was prepared and transmitted to ERDA and AGA. This program detailed the experiments to be performed utilizing the synthesis gas stream ( $H_2/CO = 6/1$ ) from the IGT reformer and incorporated runs with HYGAS product when available.

Work began on revising the LPM Pilot Plant operating instructions to IGT format. Concurrently, line and valve lists were started, and tagging of all lines and valves on the skid were initiated.

Weekly progress meetings for IGT, CSI and ERDA personnel were initiated on November 16, 1976. All facets of the project were discussed including current progress, scheduling, and outstanding items requiring attention.

A detailed checklist, including priorities, was begun to keep everyone informed of critical items requiring repair, installation or revision. This list was updated at all subsequent meetings.

Most of the major mechanical, electrical and instrument installations were completed early in December, 1976. With electric power brought to the control room, all pumps on the skid were bumped to check rotation and electrical hookups. A number of problems were discovered and remedied. The control room pressurization units and combustible gas alarm were installed and made operational. The alarm and shutdown systems were checked and found to require debugging.

The nuclear level detector and its lift mechanism were thoroughly examined for practicality of operation. It was decided to motorize the lift mechanism, which was installed according to Davy Powergas specifications. This was to make use of the detector more convenient. The unit was to be controlled from the second level, north of the reactor. Equipment for motorizing the lift mechanism was ordered.

Meetings were held with IGT personnel to coordinate efforts regarding analytical requirements, responsibilities and equipment for the LPM program. IGT was to sample the feed and product gases, and these two streams plus reduction hydrogen and degasser vapors were to be analyzed in the pilot plant control room. A list of all necessary equipment was drawn up and ordered. Also, a heated shed for standard gas bottles was designed and constructed.

By the end of December, 1976, the LPM Pilot Plant operating instructions had been revised to IGT format.

Winterizing the pilot plant to withstand the cold Chicago weather was the main focus of mechanical work during the month of January, 1977. All additional steam tracing was completed, but the steam was not turned on until early in February, 1977 due to cold weather experienced at the end of January.

Other than winterizing, mechanical, electrical and instrumentation work were mainly concerned with solving the problems encountered with various systems during shakedown. In the control room, the relay panel was integrated into the electrical circuits. One by one, the relays, as well as all alarms and shutdowns, were checked out and corrected where necessary. The digital thermocouple readout was made operational, as well as the Honeywell temperature recorder. The oil make-up pump was taken out for insertion of new packing and then reinstalled. Piping around this pump was rebuilt when it was found to be mis-aligned, causing stress on the pump head. Mechanical seals were ordered for the BFW pumps based upon experience at IGT for this service. Meanwhile, both BFW pumps were taken out for inspection and repacking. One was reinstalled. One of the condensed oil return pumps was drawing excessive current and was disassembled to find the problem. Upon reassembly, the pump worked properly as did all other pumps in the pilot plant. The seal flush system on the main circulating oil pumps was found to be connected improperly and was corrected.

Double block and bleed valves were installed on the hydrogen return line from the reactor to the hydrogen exchanger. The gas cylinder storage shed was finished and sample lines from the skid to the control room were installed. Flow element FE-106 was found to be improperly sized and a new element was ordered. Oil heater, H-101, was checked out thoroughly and it was found that the SCR had shorted out. After replacement, the heater worked properly. FIC-102 had its action reversed at the controller and was corrected. Ice blockages were found in FI-100, FI-107, FE-109 and PV-104. Alarms for the gas heater, H-102, were

debugged. The Honeywell recorder had to be repaired for the second time.

During the second week in February, 1977, the flue on the control room pressurization unit was installed and the air conditioner placed in position. Instruments FIC-105 and PIC-102 were properly aligned. It was found that TT-117, which controls the circulating oil temperature, had the wrong range and a replacement was ordered. LIC-102 on the product gas separator was made operational as was LIC-106 on the BFW system and LT-102 on the circulating oil cooler. However, the latter had a leaking flange which was repaired.

All leaks found from the first shakedown run were fixed during the third week in February, 1977. In addition, a section of misaligned pipe near the hydrogen exchanger was cut out and repiped so that a leaking gasket on the head of the exchanger could be fixed. After reconnecting the hydrogen exchanger, it was still leaking at the connecting flanges. Therefore, it was decided to pull the exchanger out and machine the flaring faces where they had been badly gouged by improper assembly during construction of the pilot plant in Texas City. One of these flanges had been bent out of alignment in order to connect it to external piping. When the manufacturer of the exchanger, R. W. Holland Co. was consulted about this repair, they recommended sending the unit back to them since they considered information about the flange connections proprietary. Due to time limitations, an attempt was made to machine these surfaces at IGT. After many attempts to repair the flange, the entire exchanger was taken out of the system and returned to R. W. Holland Co. for repair.

The control room pressurization unit was debugged and panel mounted stop-start buttons were rewired according to standard convention. The control room air conditioner was hooked up and tested. Local start-stop stations were replaced with the lockout type on pumps for safety purposes. A new reactor high temperature alarm and shutdown switch was

installed so that this operation would not be controlled by the faulty Honeywell recorder. Surface thermocouples were ordered and placed in various locations in the pilot plant to increase operating reliability.

The oil level transmitter on the product gas separator was removed, repaired and reinstalled. A reflex type level gauge, intended for measurement of the oil-water interface in the product gas separator, was replaced with a clear-type gauge. The piping on the product gas separator oil-water interface level gauge was redone so that it would operate properly.

Details for motorizing and installing the reactor level detector lift mechanism were completed and a lift motor was installed on the reactor level detector. Power lines were run from the lift motor to the motor control center and a local up-down station was installed. The detector was run completely up and down. Then, the detector was connected to the amplifier in the control room and to the locally mounted readout device. Two sprockets, which were not firmly attached, fell off during operation of the lift mechanism. A better method of attachment was developed. The detector heater was turned on and the unit calibrated by Texas Nuclear personnel.

All the analytical equipment, including chromatograph, integrator, etc., were brought to Chicago and the system installed, tested and prepared for operation. Both of the sample gas conditioning hot boxes were fabricated and installed on the skid. The steam traced sample lines were also completed. All gas cylinders required for chromatographic analysis arrived. The GC, integrator and most of the analytical system were set up. Procedures for operation of the analytical system were written.

Revised orifice calibrations were completed for all D/P cells to account for the Freezene-100 oil and 10  $\rm H_2/CO$  feed gas expected from IGT's steam-methane reformer.

New demister internals were received and installed during the next shutdown. The orifice taps on the seal flush return line were modified. Spare parts were organized and inventoried. An inventory list was transmitted to IGT, since it was their responsibility to reorder items as they are used.

Once all steam tracing was completed, this task was considered completed.

### 4. Start-up and Pre-operational Testing

A schedule for shakedown operations was worked out and transmitted to ERDA in November, 1976. Included in this schedule were the following operations:

- Nitrogen pressurization and leak testing.
- Cold oil flow tests.
  - Gas feed tests
  - Preparation for experimental program.

The objective of the cold oil flow tests was to fill all vessels with the process liquid to be actually used during operations, check pump operation, and commission all liquid controlling instrumentation. Also, the reactor level detector was to be calibrated during these tests. The gas feed tests would accomplish the same for the gas systems. Preparation for the experimental program included commissioning the analytical system, filling the reactor with catalyst and inserts and cleaning out the filter units. Shakedown was to be completed by mid-January, 1977. Leak testing of various segments of the pilot plant with low pressure nitrogen began on December 6, 1976. The first vessel checked was the oil make-up drum because a 1,500-gallon shipment of Freezene-100 was expected

on December 9. Frozen lines and valves were encountered in numerous places around the oil make-up drum. These problems were overcome and leak testing proceeded to the hydrogen reduction system, the inlet feed gas lines and the reactor. Meanwhile, the 1,500-gallon oil shipment arrived and was successfully pumped into the make-up drum.

Frozen lines were encountered in the hydrogen reduction system and steamed to free the blockages. With warmer weather, it was possible to free most of the pilot plant of ice blockages and blow the lines clear. All valves on major lines were worked over, greased and repacked where necessary. All broken pressure gauges were replaced. During pressurization, cracked lines were found in the nitrogen inlet and on the gas separator causing delays in completing the leak testing.

By the end of December, 1976, all systems up to and including the reactor checked out okay. However, some elusive leaks in the reactor separator to product gas separator systems prevented completion of leak testing. Also, valves isolating the water degasser were not found functioning properly. Meanwhile, the process oil filter vessels were loaded with cartridges and prepared for operation.

Cold oil circulation was initiated on December 30, 1976 with the start-up of the oil make-up pump. Oil was circulated in a closed loop from the make-up drum through the pump and back to the drum. However, the pump was found to be leaking excessively through the packing. It was taken apart and found to contain no packing. The pump was taken our for inspection, cleaning and repacking. With the oil make-up pump operational, oil was pumped into the product gas separator to a level of 20 inches. During this operation, it was discovered that flow transmitters and level control devices were not functioning due to the extremely cold weather. Each instrument in the entire pilot plant had to be checked. Some were frozen and some were incorrectly connected. Within a week, all instruments were sending signals to the control panel and some were responding properly to flow and level conditions in the pilot plant.

Nitrogen leak testing was completed during January, 1977. The segment of the system which includes the reactor separator, circulating oil cooler, process oil filters, product gas separator and water degasser was losing pressure too rapidly. The lines connecting the product gas separator with the water degasser was found blocked and were steamed and cleaned out. A faulty globe valve was cut out and replaced, allowing isolation of the degasser which checked out okay. In the remainder of this system, leaky grease fittings were discovered on 4" valves and an orifice plate was found missing. Major leaks were discovered on the circulating oil pumps and were corrected.

In the boiler feedwater system, many of the lines were frozen solid. Valves were repacked and lines steamed. The BFW pumps were also frozen and removed for inspection and repacking. Finally, the BFW system was cleared of blockages and it held pressure with less than a 5 psi drop per hour, as did the entire pilot plant, thus completing leak testing.

Cold oil flow tests continued in January, 1977, with the filling of lines to pumps P-102 A&B with oil from the product gas separator. The two pumps were started and oil flowed through the by-pass back to the product Local flow indicators on these lines were not functioning. While trying to pump from P-102 to filters F-102 A&B, it was discovered that the check valves in these lines were rusted shut. They were forcibly removed and cleaned. Then oil was pumped to the reactor separator, the F-102 filters and the seal flush and wear ring lines at pumps P-101 A&B. Local flow indicators on the wear ring flush lines were taken out and cleaned. The main circulating oil pumps were started for short periods after correcting some electrical problems on one of them. The two check valves on the discharge of these pumps were frozen shut, but the discharge pressure from the pumps forced them to open. Oil was pumped to the main filters and the circulating oil cooler, but ice blockages prevented the use of the filters and the filter by-pass had to be used instead. It was also found that no oil would pass through the seal flush on P-101 A&B and the tandem seal piping had to be repiped. Meanwhile, the entire system was loaded with oil, with the exception of the reactor.

The following week, the seal flush system was turned on and it worked. The main circulating oil pumps were started up and, with the reactor separator level control on automatic, the entire oil system operated properly at atmospheric pressure. Some time was spent trying to get all the flow and level controllers in the oil system to function properly. This was not successful in all cases due to extremely cold weather and high oil viscosity. The oil heater was turned on for the first time after debugging the alarm and shutdown circuits connected with oil flow rate. The oil was heated to 150°F, but due to a burned out SCR, the heater controller was not working automatically. The unit was shut down to make repairs to the heater circuits.

During the first week in February, 1977, cold oil was circulated and then heated to 200°F. Gas flow was initiated through the hydrogen reduction system. The gas heater was used to raise the reactor temperature to 50°F. The nitrogen flow was transferred downstream to test the pilot plant pressure controllers and gas heating was continued. Gas feed through the main feed gas line was also tested successfully. The system was then ready for an integrated gas-oil run without catalyst, requiring round-the-clock operation.

A continuous run was initiated during the second week in February, 1977 to test integrated operation with oil and gas flow. Shift coverage with IGT operators was started on a 5-day per week basis. The first day, oil flow was started and heated to  $200^{\circ}$ F. The reactor was filled with oil for the first time and the by-pass closed. The cooling water system was turned on. Nitrogen was fed through heater H-102 and into the main feed gas line through a hose to check operation of the flow control valve.

System pressure was maintained at 100 psig. After integrated gas and oil flow was stabilized, nitrogen feed gas was switched to the direct line from H-102 to the reactor.

With the system holding steady, filter F-101A was steamed for eight hours to break through an ice blockage which was preventing its use. Oil broke through the ice finally and the filter pressure drop fell from 15 to 5 psi. During the second evening, the oil was heated to  $400^{\circ}$ F and the inlet gas to  $200^{\circ}$ F.

The second filter, F-101B, was steamed to break up its ice blockage. When the hot oil broke through this filter, the entire system was upset by the pressure surge and it took a half-hour to bring everything under control. The BFW system was started up for the first time using pump P-103A to fill E-103. Steam was generated in the circulating oil cooler lowering the oil temperature to 355°F temporarily.

By the fourth day, steam pressure in E-103 had risen to 250 psi and the entire BFW system was placed on automatic control with the exception of the steam vent which had to be controlled manually pending replacement of TI-117. Venting 250 psi steam did not produce any noise, so the need for a silencer was eliminated.

On the fourth night, the gas temperature was increased to  $250^{\rm OF}$  and while trying to heat the oil to  $500^{\rm OF}$ , it was noticed that the operating limit of oil heater H-101 was  $420^{\rm OF}$ . Adjustments to seal flush and circulating oil flows raised this temperature to  $438^{\rm OF}$ , but this was still insufficient. The unit was shut down on the fifth night. A considerable amount of oil was found in the flare header, which was attributed to the fact that the demister internals had not been installed at the skid fabricator's shop.

The circulating oil heater was thoroughly checked out and found to be operating properly with a total output of 135 KW, which is 10 percent below nameplate value. Therefore, the causes for insufficient oil heating were heat losses through exposed flanges on the major vessels and an excessive wear ring flush flow rate to the circulating oil pumps. IGT was notified to box and insulate all exposed flanges as requested earlier by CSI personnel.

A second continuous run was started almost immediately with the reactor blocked out, most exposed flanges in the circulating oil system covered with temporary insulation and the wear ring flush shut off. The oil quickly reached 560°F while the system was maintained at 100 psig with a low flow of LP nitrogen. A high pressure nitrogen line was connected to the process feed gas line and the system pressure was increased to 300 psig. The unit operated smoothly at these conditions, however, numerous leaks were detected at the flanges, valves and other fittings.

It was decided not to increase pressure further until the flanges and other major leaks had been tightened. Heaters were turned off and in less than 24 hours, the unit was completely shut down.

During operation at 300 psig, with the product gas separator at 120°F, it was noticed that the product gas continued to contain a large amount of oil mist. The gas demister was, therefore opened for inspection and was found to contain no internals at all. Conversations with Davy Powergas revealed that Brink, the manufacturer, shipped the internals separately to Industrial Contractors, the fabrication shop. An effort was made to locate this item, but it could not found by DPG at the fabricator shop in Texas City although it was definitely shipped there. A replacement was ordered from Brink which took four weeks for delivery.

During this continuous run, the water degasser was started up for the first time. Initially, a blockage was found in the feed line to this unit, but, once it was freed, the unit was partly filled with water and oil drained from the product gas separator. All instruments on the degasser functioned properly. However, it was not possible to drain the liquid into the IGT incinerator line because the degasser had insufficient backpressure built up.

A two-day run was made to check the unit for leaks at high pressure during the last week in February, 1977. Before starting up, the product gas separator was partially loaded with water to check operation of the oil-water interface control devices. The pilot plant was brought to  $460^{\circ}$ F and 600 psig with the reactor on-stream. It was found that the oil-water interface transmitter was still not working properly and that the corresponding level sight glass needed some repiping. Several new leaks were discovered at the higher pressure.

During the first week in March, 1977, the pilot plant was prepared for the first experimental run. The reactor level detector was calibrated with the assistance of a representative from Texas Nuclear. Hexane and a glycol-water mixture were pumped into the reactor to simulate the density range expected during the operation. The interface between these two liquids was easily distinguished using the level detector.

with the calibration completed, the reactor was drained and dried out with nitrogen. Inert material was added to the reactor to a height of 2.8 feet above the bottom flange. This consisted of 1,100 lbs. of 3/4" carborundum cylinders followed by 900 lbs. of 1/2" carborundum cylinders. Then, 496 lbs. of Calsicat Ni-230-S catalyst spheres were charged, bringing the catalyst level to 6.0 feet above the bottom flange. Thus, the catalyst bed height was 3.2 feet as measured with the reactor level detector. The catalyst bed was maintained under nitrogen blanketing until start-up.

IGT analyzed their hydrogen stream and found it to contain a  $\rm H_2$  to CO ratio of approximately 10/1. This differed from the 6/1  $\rm H_2/CO$  gas originally anticipated in October, 1976 by IGT. However, no problems were anticipated regarding use of this gas for LPM process variable scans.

Start-up and pre-operational testing of the LPM Pilot Plant was, thus, successfully finished and the unit readied for the experimental program.