

III. LAPORTE LPMEOH PDU DESCRIPTION

A. Background

The primary function of the LaPorte LPMEOH PDU is to acquire necessary plant data at a small but representative engineering scale for testing the feasibility of the LPMEOH process. The feasibility determination includes analyzing the sensitivity of major process variables, and evaluating the process design. The PDU was designed with the capability of generating and collecting plant data over a wide range of potential operating conditions for the LPMEOH process.

Two types of reactor configurations have been developed for the LPMEOH process. The first, termed the liquid-fluidized catalyst reactor, employs an ebullated bed of 3-6 mm diameter particles where the upward flow of inert hydrocarbon liquid and synthesis gas results in the fluidization of the catalyst to a desired bed expansion. Phase separation between solid and liquid/gas occurs at the top of the reactor. Thus the catalyst bed remains within the fixed boundaries of the reactor. The second configuration is termed the liquid-entrained catalyst reactor. In this system, small catalyst particles (micron size) are suspended or slurried in the inert liquid and circulated through the reactor. Contact with the synthesis gas is made by feeding the gas concurrently with the upward flow of liquid.

The LaPorte LPMEOH PDU was originally intended to be a virtual reuse of the Liquid Phase Methanation (LPM) Pilot Plant operated from 1977 to 1978 at the IGT HYGAS facility in Chicago, IL. Relocation of this equipment to LaPorte, TX and some renovation work was anticipated to allow operation in the liquid-fluidized mode. At a later date, a skidded train of equipment would be designed and tie in alongside the first unit to allow liquid-entrained operation.

Past experience with PDU design and operation showed, however, that full consideration of all modes of operation during the initial design stage would decrease the chance of extensive modifications later in the program that would not be identified otherwise. In recognition of this, a single "unified design" approach was adopted for the design of this PDU. Equipment, instrumentation, and valving specifications included the consideration of both modes of operation from the start of the design effort. Under this premise, a process flowsheet of the unified design was developed jointly by Air Products and Chem Systems. The flowsheet incorporated a high degree of operational flexibility, and the resultant PDU design permitted switching operations from the liquid-fluidized to the liquid-entrained mode without equipment or piping alterations. A common reactor was designed to accommodate both modes.

As a result of differences between the LPM and LPMEOH processes, and extensive re-engineering of the Chicago unit to provide flexibility and safety, a limited amount of the original equipment assembly from the Chicago LPM Pilot Plant was able to be reused. During development of the LaPorte LPMEOH PDU design, equipment components from the DOE Synthoil facility located at the Pittsburgh Energy Technology Center (PETC) in Bruceton, PA were made available by DOE. A number of these key process equipment items and electronic instrumentation devices were incorporated into the LaPorte LPMEOH PDU design. A summary of the major LaPorte process equipment items identified by source is presented in Table III-1. A brief review of this tabulation indicates the extent to which the LaPorte LPMEOH PDU design departed from the original LPM Pilot Plant.

B. Process Design Basis

High operating pressure favors the equilibrium of the methanol synthesis reaction, and it was desirable to design the PDU to achieve a reasonably high pressure. Ideally, this could have been as high as 100 atm (1500 psi). The reuse of existing equipment,

TABLE III-1
LAPORTE LPMEOH PDU MAJOR PROCESS EQUIPMENT LIST BY SOURCE

<u>Chicago, IL</u> <u>LPM Equipment</u>	<u>Bruceton, PA</u> <u>Synthoil Equipment</u>	<u>New</u> <u>Purchased Equipment</u>
27.10 Reactor	10.52 Oil Condensate Pumps	01.10 Feed Compressor 01.20 Recycle Compressor
27.14 Intermediate V/L Separator	10.56 Low Pressure Oil Return Pumps	01.13 Feed Surge Tank
21.30 Product Cooler	28.10 Methanol Storage Tank	10.50 Slurry Circulation Pump
22.10 Product Separator	10.53 Utility Oil Pumps	21.10 Feed/Product Exchanger
22.11 Degasser		21.20 Slurry Exchanger
22.12 Demister		27.13 Primary V/L Separator
22.51 Condensed Oil Filters		22.15 LP Liquid-Liquid Separator
15.40 Utility Oil Heater		29.30 Slurry Prep Tank
*02.83 Booster Utility Oil Heater		*02.61 Reduction Steam Heater
01.12 Feed Inlet Filter		*02.62 Reduction Electric Heater
01.22 Recycle Inlet Filter		*02.60 Carbonyl Guard Beds
		21.40 Utility Oil Cooler
		28.53 Utility Oil Expansion Tank
		01.14 Feed Compressor Recycle Cooler
		01.24 Recycle Compressor Recycle Cooler

* During shakedown the arrangement of several heaters was modified. This listing provides the equipment names as changed.

however, limited the maximum operating pressure to 62 atm (900 psig). This was considered acceptable, since 62 atm is close to the optimum pressure in many commercial methanol plants, and a wide operating pressure range remained available (35-62 atm, 500-900 psig). The ranges of other operating variables were chosen to encompass operating conditions of interest (Table III-2).

Two principle reactor feed compositions were identified for the design basis (Table III-3):

1. Balanced Type - representative of synthesis gas from a Texaco coal gasifier with shift and CO₂ removal, suitable for total conversion to methanol via recycle of unreacted gases. This is the so-called "all-methanol" application.
2. CO-rich (Unbalanced) Type - representative of synthesis gas from a Texaco gasifier without shift and CO₂ removal, suitable for single-pass methanol production with a resulting CO-rich fuel gas. This is "single pass, coproduct" application which is synergistic with the Integrated Gasification Combined-Cycle process to make electric power.

These different reactor feed compositions are blended from hydrogen, carbon monoxide, nitrogen and methane supplied by the adjacent Air Products' syngas facility. Carbon dioxide is trucked into the plant as liquid and stored on-site. Since only a portion of the reactor feed is converted per pass, the unconverted syngas is recycled and mixed with fresh make-up gas. The make-up gas is blended according to recycle flow and composition so that the reactor feed (make-up plus recycle) simulates either the balanced or CO-rich gas type. Recycling the unconverted syngas reduces gas consumption by about 70%.

TABLE III-2
RANGE OF OPERATING VARIABLES FOR LAPORTE LPMEOH PDU

	<u>Minimum</u>	<u>"Normal"</u>	<u>Maximum</u>
Reactor Pressure, kPa (psig)	3,500 (500)	5,300 (750)	6,300 (900)
Reactor Temperature, °C (°F)	220 (428)	250 (482)	270 (518)
Liquid-Fluidized Space Velocity, l/hr-kg cat	1,000	2,500	4,000
Liquid-Entrained Space Velocity, l/hr-kg cat	2,000	6,000	15,000

TABLE III-3
LAPORTE LPMEOH PDU FEED GAS COMPOSITIONS
 (Based on Texaco Gasifier)

	<u>Balanced</u>	<u>CO-rich</u>
H ₂	55%	35%
CO	19	51
CO ₂	5	13
N ₂	<u>21</u>	<u>1</u>
	100%	100%
H ₂ /CO Ratio	2.89	0.69
Balance Ratio, H ₂ /(CO + 1.5 CO ₂)	2.08	0.50

C. Process Description

A simplified process flowsheet for the LaPorte LPMEOH PDU is shown in Figure III-1. The make-up synthesis gas is compressed from 1,000 kPa (135 psig) to the reactor pressure (between 3,500 and 6,300 kPa, 500-900 psig) by the 01.10 feed compressor. This compressor is a two-throw, balanced-opposed machine, which functions as two separate services (one single stage, one 2-stage) sharing a common frame and lubrication system. The compressed feed is mixed with recycle gas from the 01.20 compressor and the combined flow is heated through the 21.10 feed/product exchanger. The feed subsequently flows through two empty guard bed vessels originally designed for the removal of metal carbonyls in the feed gas.

The original carbonyl removal system included an economizer, an electric heater and two alumina-loaded guard beds. The guard beds were to operate at 350°C (662°F) to remove any iron carbonyl formed in the piping and equipment under high carbon monoxide partial pressure. However, in order to eliminate an exotherm encountered during commissioning (see Section IV), the PDU was modified to limit the feed gas temperature below 200°C (392°F). This modification eliminated the economizer and electric heater and made the alumina ineffective as a guard bed material for iron carbonyl removal. A carbonyl survey conducted during the initial PDU shakedown showed an iron carbonyl concentration of less than 10 ppbv in the reactor feed. This low iron carbonyl concentration was judged to be acceptable for the initial, short-term PDU operations and the guard beds were left empty during the shakedown run.

The heated feed gas is introduced in the reactor bottom and mixed with the incoming inert oil or catalyst/oil slurry in a distributor/plenum zone. The 27.10 reactor is a cylindrical vessel constructed of low alloy steel (1/2% Mo) and sprayed internally with copper to prevent the formation of iron carbonyl (see Figure III-2). The bubble cap tray distributor, designed principally for liquid-fluidized operation, is capable of handling slurry in liquid-entrained operation.

FIGURE III-1
SIMPLIFIED PROCESS FLOWSHEET FOR
LAPORTE LPMEOH PDU

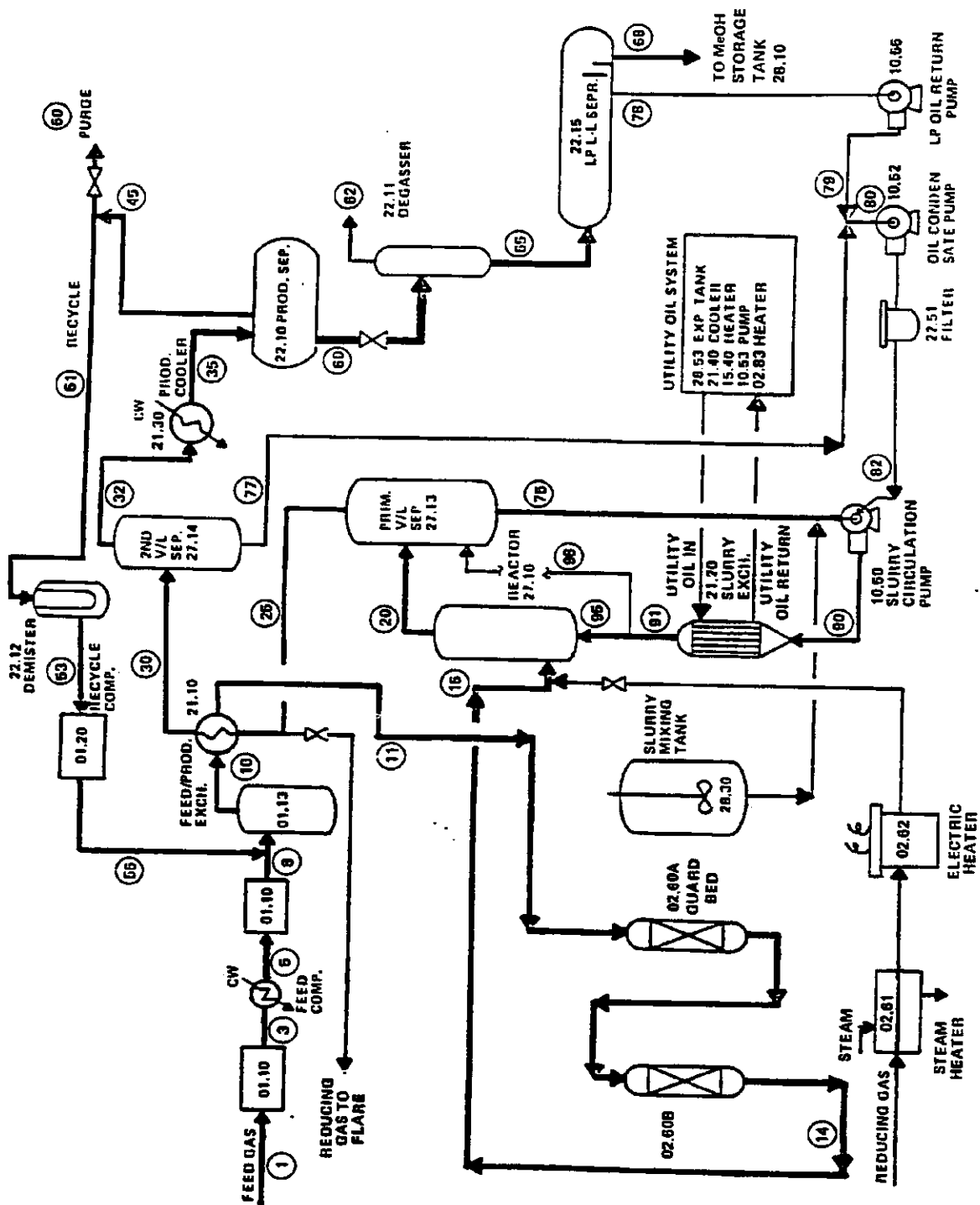
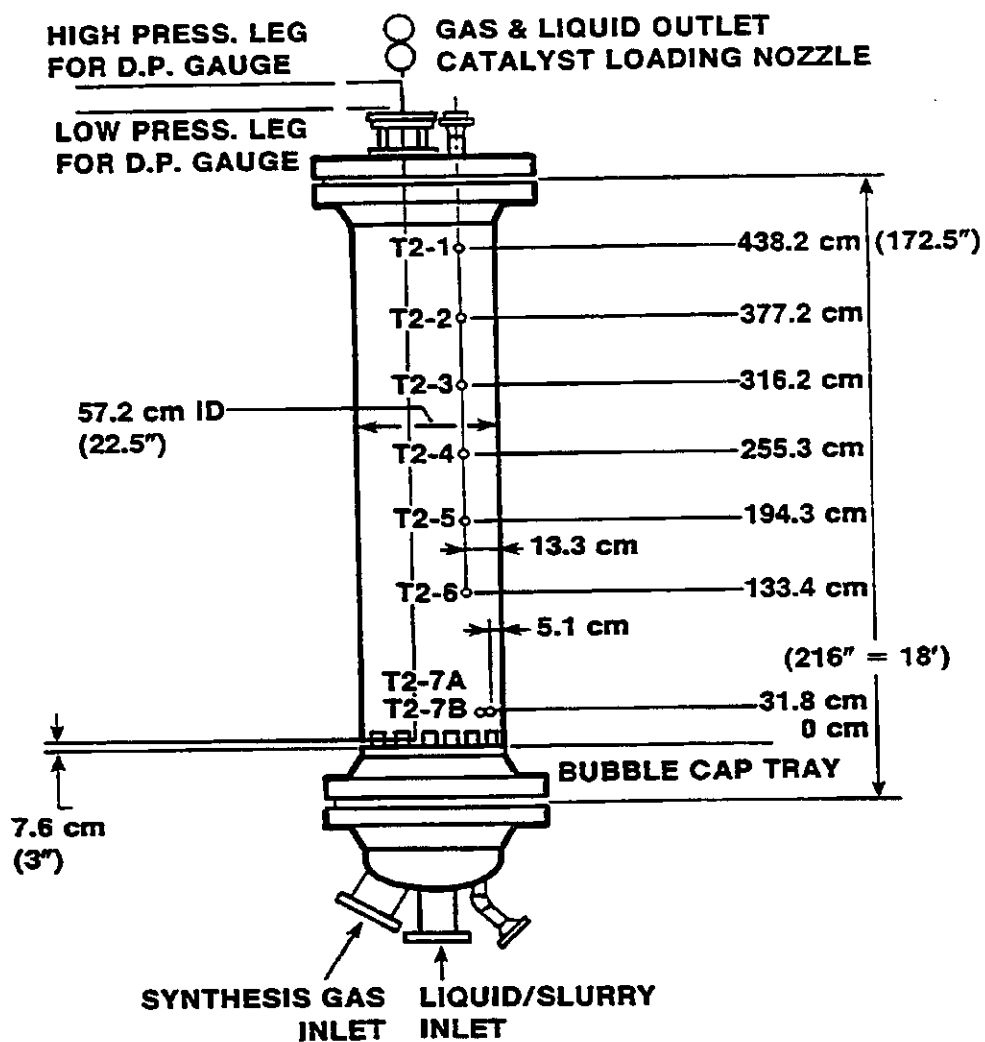


FIGURE III-2 LAPORTE LPMEOH PDU REACTOR



The inert hydrocarbon liquid or slurry that circulates through the reactor is separated from the methanol product and unconverted synthesis gas in the 27.13 primary V/L separator. It is recirculated to the bottom of the reactor via the 21.20 slurry heat exchanger by the 10.50 slurry circulation pump. The circulating liquid can be heated or cooled in the slurry heat exchanger to maintain a constant reactor temperature, depending on the CO conversion in the reactor, the heat loss of the system, and the rate of cold seal flush oil required by the slurry circulation pump. A utility oil system, which includes a utility oil expansion tank (28.53), utility oil circulation pumps (10.53), a utility oil cooler (21.40), and utility oil heaters (02.83, 15.40), is designed to provide indirect heating or cooling of the circulating liquid and to eliminate the possibility of overheating the catalyst. The slurry circulation pump is a centrifugal pump driven by a variable speed electric motor and provided with a specially designed double mechanical seal. The seal is provided with seal flush and a circulating barrier fluid to eliminate the possibility of leaks of the slurry to the atmosphere. The slurry circulation system is described further below in Design Features.

The methanol product and unconverted synthesis gas exiting the top of the primary V/L separator are cooled against the incoming feed gas in the feed/product exchanger to 150°C (302°F), condensing the bulk of the vaporized hydrocarbon liquid into the 27.14 intermediate V/L separator. The liquid mixes with oil condensate recovered further downstream and is returned to the process as seal flush for the slurry circulation pump via the 10.52 oil condensate pumps. The uncondensed vapor is further cooled to about 40°C (104°F) by cooling tower water in the 21.30 product gas cooler. Condensed methanol product and a small quantity of inert hydrocarbons flow from the 22.10 product separator, are reduced in pressure, and the flashed gas is vented at the top of the 22.11 degasser to the plant flare header. The methanol-hydrocarbon stream passes through the 22.15 low pressure liquid-liquid separator, where the hydrocarbon oil separates, and is returned to the process via the 10.56 oil

return pumps. The methanol product is sent to the 28.10 product storage tank.

The unconverted synthesis gas leaving the product separator is compressed and recycled to the reactor. A small purge stream is withdrawn to prevent the buildup of inerts in the reactor loop.

The oxide form of the catalyst is reduced with a mixture of hydrogen and nitrogen which is heated with the 02.61 reduction steam heater and the 02.62 reduction electric heater prior to its introduction to the reactor. In the liquid-fluidized operating mode, the catalyst reduction is done in the reactor prior to the injection of the synthesis gas and hot hydrocarbon liquid. In the liquid-entrained mode, the catalyst reduction takes place during slurry circulation in the slurry loop, also before the synthesis gas is introduced.

D. Design Features

Slurry Circulation System

One of the unique features of the LaPorte LPMEOH PDU is the slurry circulation system. The system consists of the reactor, primary V/L separator, slurry circulation pump, and slurry/oil heat exchanger. Considerable engineering effort was employed in the design of the slurry circulation system. The important design objectives were reliability, proper catalyst slurry suspension, minimum erosion, and safety. Careful selection of the slurry pump and associated flow control equipment was made. Special configurations for the separators and heat exchanger, and exacting specifications for piping layout, were used. These special design considerations given to the slurry circulation system are summarized below:

- Slurry Pump

A single centrifugal pump is used in this high temperature, high suction pressure, slurry application. The pump design selected (Lawrence Pumps Inc.) for this application was the product of much development for synfuel technologies during the 1970's. However, the temperature and pressure conditions of this LPMEOH application represented an extension of the state-of-the-art for this design.

This unit incorporated a removable inner liner of specially hardened material inside a pressure containment casing. The impeller was also fabricated in this high chrome alloy for hardness. A specially designed double mechanical seal was a key to success in this application, since experience has shown that leakage of process fluid to atmosphere represents a significant risk with respect to fire hazards and plant reliability. Several alternate seal designs were reviewed and the two most promising alternates (Durametallic Corp., Stein Seal Co.) were incorporated into the interchangeable cartridge assemblies within the pump. In order to meet the needs for flexibility in servicing these alternate seal designs and provide responsiveness to the varying system pressure, APCI developed and assembled in-house a specially designed barrier fluid system.

- Slurry Flow Measurement and Control

In order to meet the needs of variable operating conditions planned for this PDU, as well as uncertainty in the viscosity that the slurry would exhibit, it was necessary to provide a means of slurry flow measurement and control. Much consideration was given to plugging and/or erosion, problems which standard in-line devices would be expected to experience in this slurry service. Most of the state-of-the-art devices

for slurry services utilized in the synfuels technologies were still inordinately expensive in relation to the LaPorte PDU need. As a more attractive alternate, it was elected to control the slurry flow by adjusting the speed of the centrifugal slurry pump. A frequency controller was incorporated into the electrical motor control for this pump. Flow measurement is obtained from a relatively inexpensive wedge meter with isolated differential pressure taps. Even though the reliability of the differential pressure reading would not be high in this service, rough calibration of flow-to-pump amperage and speed could serve as a back-up to this flow reading.

- Slurry Piping, Valves and Expansion Joints

There were several challenges in developing a reliable and safe piping system for the slurry circulation loop. Considerable attention was given to the valves incorporated into this design. Due to their considerable expense, the need for each valve in the slurry loop was critically evaluated as the flowsheet was developed. Essentially three different services were identified for valves that would experience slurry flow.

Most valving in this slurry system is utilized in drain service. These valves are normally closed and cycled on an infrequent basis for start-up and shutdown operations. For this service, requiring a tight shutoff, but allowing a compromise on area for flow cross section, a basic plug valve design was utilized. This particular valve design has decades of operational history on cold, water-based mining slurries. With recent embellishments, such as selectability in materials of construction and stellite of body and plug, this valve design has proven worthy during synfuels technology development.

For two of the valve applications, reliable isolation was required on a normally flowing stream. Since this was a requirement stemming from hazards analysis, cost was relegated

to a lesser priority, and a full ported, metal-to-metal ball valve of a more recent and specialized design was identified. This design has demonstrated the cycling reliability required through synfuels technology development in the late 1970's.

One application called for a less reliable isolation service for diverting process flow infrequently during start-up operation, but normally experiencing full process flow. The valve used here was a moderate upgrade of a design utilized for decades in boiler blowdown service. This particular valve performed acceptably at LaPorte.

Particular attention was given to routing of slurry piping in a smooth and direct manner. Where bends in piping were required, double radius elbows were specified. Specification of the piping thickness was adjusted to provide a cross-sectional area that would maintain the linear slurry velocity within a range which would minimize risks of plugging and erosive wear. It was found that a conflict in requirements for release of thermal expansion stresses and the desire for simple direct pipe routing occurred on the suction and discharge of the slurry pump. In resolution of this dilemma, an expansion joint design with sufficient internal protection against potential slurry erosion and plugging was incorporated since it had an overall integrity equivalent to flanged piping connections.

- Reactor Internals

The reactor internal devices for distribution and contacting of the three phases in the liquid-fluidized mode were given careful consideration and review throughout the first year of PDU design activity. Ultimately, a subcontract was awarded to Hydrocarbon

Research, Inc. to conduct cold flow studies and contribute their proprietary expertise to the design of liquid and gas spargers and a bubble cap distributor tray specifically for the liquid-fluidized mode of LaPorte LPMEOH PDU operation. In addition, during the design, consideration was given to slurry in the reactor loop due to catalyst attrition. This would assure continuing operation of the PDU in the liquid-fluidized mode even when a significant quantity of catalyst bed extrudates is attrited to fines.

In parallel with this distributor design, alternative internal devices were designed in-house specifically for the liquid-entrained mode of operation.

- Nuclear Density Gauge

A nuclear density gauge (Texas Nuclear Corporation), which consists of a traversing gamma-ray radiation source and a detector, provides a virtual window for observation of hydrodynamic events inside the 3/4"-thick steel walls of the reactor. Originally intended to measure expanded catalyst bed height in the liquid-fluidized mode, the analog signal coming from this nuclear detector, along with a careful calibration, could be used to calculate important gas hold-up and slurry concentration information in the liquid-fluidized and liquid-entrained systems.

E. Data Acquisition System

The primary objective of the LaPorte program is to demonstrate the feasibility of the LPMEOH process at the PDU scale and to gather process design information. Therefore, the data pertinent for process design and the catalyst performance are the most important "products" from the LaPorte LPMEOH PDU operation. To achieve the

objective, a data acquisition system (DAS) was installed to collect, store, and report the process and the analytical data generated from the PDU. An illustration of the integrated computer/analytical hardware configuration is given by Figure III-3.

The data acquisition system includes a Digital VT103 microcomputer with 128K bytes of memory. A process monitoring/process control software package, consisting mainly of a set of FORTRAN programs structured around an interactive data base, updates all the relevant process variables on a regular basis according to a preset scanning scale.

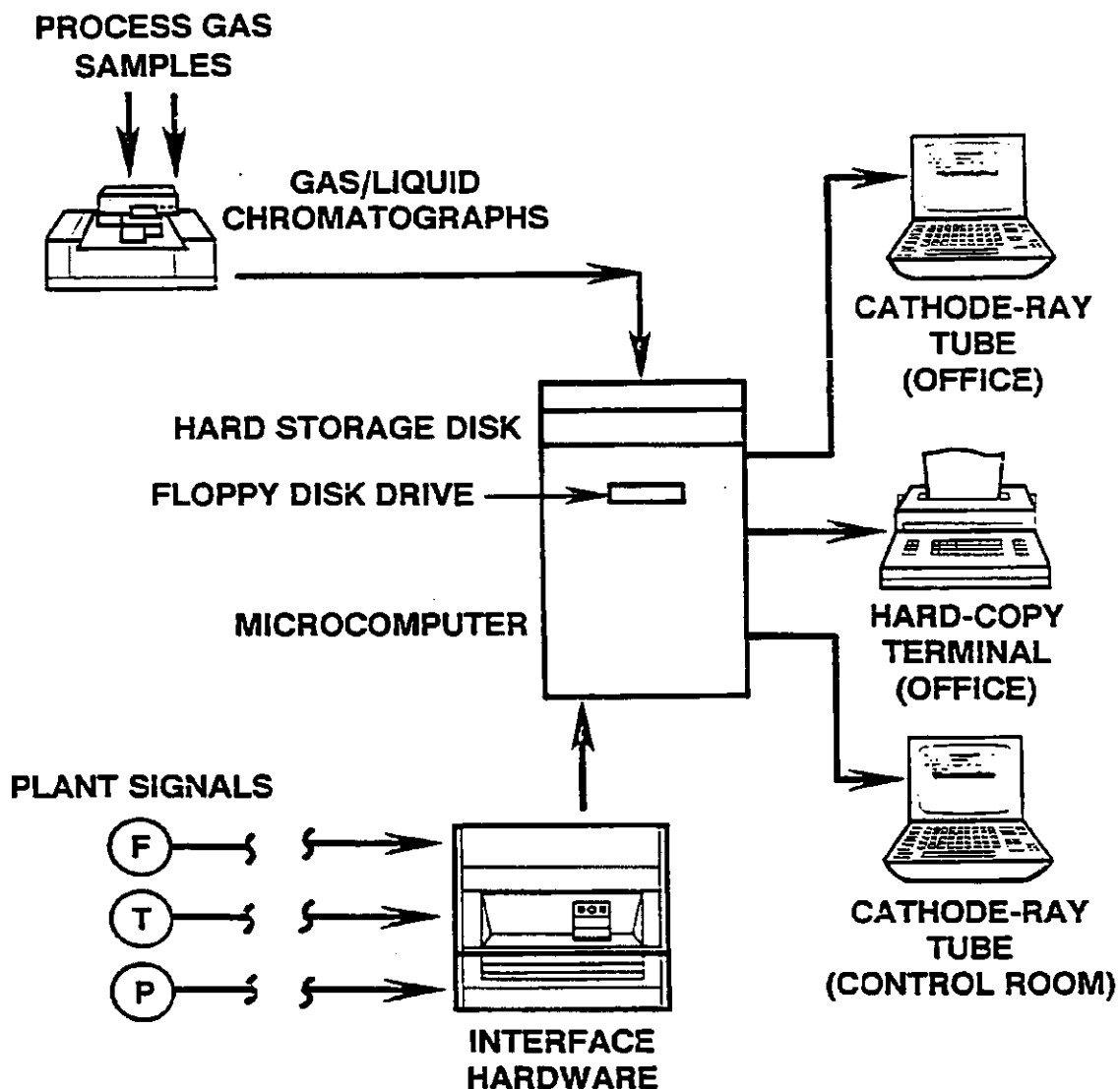
The current values of those variables are supplied to the data base in one of the following manners:

- Manually, through keyboard inputs.
- Directly, through the interface gear terminal, which is hard wired to the plant monitoring points.
- Directly, through a port, which is hard wired to a gas chromatograph microprocessor.
- Internally, through special calculation routines stored in memory.

There are 396 variables in the data base. For each of these variables, the following information is defined:

- Variable name and number
- Signal name and interface channel number
- Averaging factor and scan rate
- Engineering units and range
- Instrument units and range
- Type of sensor (in the case of thermocouples)
- Type of calculation to be performed on the variable

FIGURE III-3
LAPORTE LPMEOH PDU DATA
ACQUISITION SYSTEM:
INTEGRATED COMPUTER/ANALYTICAL
HARDWARE CONFIGURATION



The data base system interacts with programs so that the required data are received, manipulated, and stored. These programs are as follows:

- An analytical data program reads the on-line analytical reports supplied by the gas chromatograph (GC) integrator (Spectra-Physics SP-4000) and sends the relevant component concentration data are sent to their proper variable locations in the data base.
- Interface programs read the on-line signals coming through the interface terminals. The programs convert these analog signals to their proper units based on special calibration tables and user-defined engineering units. The calculated values are then sent to their proper variable locations in the data base.
- A user input program permits the manual input of process variables such as time and date.

From the information stored in the data base, a set of 75 variables is retrieved every five minutes and stored in a historian file. A set of FORTRAN programs then interacts with these data and computes an hourly average of the 12 five-minute averages and creates a file to store 207 selected process and analytical variables. Process data stored in the data base are displayed on the two cathode ray tubes located in the control room and laboratory so that operators and engineers on site can monitor the performance of the PDU. A hard-copy terminal is also available.

Non-routine process data that cannot be taken by the DAS automatically, such as catalyst bed density and bed heights, are recorded by the plant operators using the nuclear density gauge positioned at the PDU reactor.

For the analytical portion of the system, gas analysis is performed using two on-line Carle Instruments gas chromatographs. The gas sampling system, which includes a multi-screen sample rotameter panel and automatic stream selector station, is provided with timers and automatic switches so that these analyses can proceed virtually unattended. Liquid samples of oil and product methanol are analyzed using two liquid sample chromatographs supplied by Shimadzu Scientific Instruments. A Spectra-Physics 4000 electronic integrator with three DIM (Data Interface Module) units is also provided to interface the chromatographic analyses with the data acquisition microcomputer. An Epson MX-80 hardcopy printer is provided for reporting GC analyses. In addition, each DIM on the Spectra-Physics 4000 has a separate strip chart recorder for GC analyses. Finally, a Karl Fisher apparatus and analytical balance are available for water determination and sample weighing.

The two gas chromatographs routinely analyze the following six gas streams:

- Make-up feed gas
- Recycle and purge gas
- Guard bed feed gas
- Reactor feed gas
- Primary V/L separator gas (reactor effluent)
- Flashed gas

The overall cycle time for this gas phase analysis is one hour with each unit sampling three points plus a standard over individual 15 minute detection cycles. Both thermal conductivity and flame ionization detection are utilized to identify the major components for material balance purposes. The concentration range (mole %) and accuracy of measurement for each gas component are tabulated in Table III-4.

TABLE III-4
LAPORTE GAS CHROMATOGRAPH ANALYSES: COMPONENTS, RANGE, AND ACCURACY

<u>Component</u>	<u>Range and Accuracy</u>
H ₂	5-70 mol% $\pm 2\%$ Relative
CO	5-70 mol% $\pm 2\%$ Relative
CO ₂	1-50 mol% $\pm 2\%$ Relative
N ₂	0.2-25 mol% $\pm 4\%$ Relative
CH ₄	0.5-5 mol% $\pm 3\%$ Relative
H ₂ O	0.1-5 mol% $\pm 5\%$ Relative
CH ₃ OH	0.5-40 mol% $\pm 3\%$ Relative
CH ₃ OCH ₃	0.5-40 mol% \pm *

* Accuracy a function of methanol concentration

Non-routine analyses are performed to obtain methanol product composition, circulating oil/slurry properties, impurities in the make-up feed gas and reactor feed gas, and catalyst state.

The methanol product is collected periodically and analyzed by the liquid chromatograph for the components (concentrations in wt%) given in Table III-5.

Analyses to be performed on the oil sample taken from the circulation loop are the catalyst fines concentration, light hydrocarbons in oil, and boiling point curve.

Trace contaminants, such as S, NO_x , NH_3 , C_2H_2 , CN^- , Cl^- , $\text{Fe}(\text{CO})_5$, and $\text{Ni}(\text{CO})_4$, are poisons to methanol synthesis catalysts. It is important that the concentration of these components in the make-up feed gas and the reactor feed gas be checked periodically to prevent compromise of the PDU data. The gas streams of interest, the impurities of concern, and the nominal frequency of sampling are given in Table III-6.

Catalyst samples removed from the oil circulation loop during the run are subjected to extensive physical and chemical analyses to determine the change in catalyst properties. These analyses have proven useful/critical for data interpretation. The properties of the fresh and the spent catalyst are analyzed for comparison and are given in Table III-7.

TABLE III-5
LAPORTE LIQUID CHROMATOGRAPH METHANOL
PRODUCT ANALYSES: COMPONENTS, RANGE, AND ACCURACY

<u>Component</u>	<u>Range and Accuracy</u>
Methanol	By difference $\pm 2\%$ Relative
Dimethyl Ether	0.1-5 wt% \pm *
Water	0.1-10 wt% $\pm 2\%$ Relative
Ethanol	0.1-5 wt% $\pm 3\%$ Relative
Propanols	0.01-1 wt% $\pm 5\%$ Relative
Butanols	0.01-1 wt% $\pm 5\%$ Relative
Pentanols	0.01-1 wt% $\pm 5\%$ Relative
Alkanes/Alkenes	0.01-0.5 wt% $\pm 5\%$ Relative
Esters	0.02-1 wt% $\pm 5\%$ Relative
Aldehydes	0.01-0.5 wt% $\pm 5\%$ Relative
Oil	0.5-2 wt% $\pm 10\%$ Relative

* Accuracy a function of methanol concentration.

TABLE III-6
LAPORTE TRACE COMPONENT GAS ANALYSES

CO₂ Feed:

<u>Possible Impurities</u>	<u>Nominal Frequency of Sampling</u>	<u>Analytical Method Used</u> (All NIOSH)	<u>Detection Limit</u>
S	One/batch	P&CAM 146	Set in Procedure
NO	One/batch	S321	
NH ₃	One/batch	S347 or P&CAM 205	
C ₂ H ₂	One/batch		
CN ⁻	One/batch	P&CAM 116	
Cl ⁻	One/batch	P&CAM 155	
NO ₂	One/batch	S320 or P&CAM 108	

Make-up Feed:

<u>Possible Impurities</u>	<u>Nominal Frequency of Sampling</u>	<u>Analytical Method Used</u>	<u>Detection Limit</u>
S	Once initially, then one/month	Same as above; sample time may be 10 times longer.	Set in Procedure
NO _x			
NH ₃			
C ₂ H ₂			
CN ⁻			
Cl ⁻			
NO ₂			
Fe(CO) ₅		Scrub with K ₂ Cr ₂ O ₇ & HNO ₃ and Atomic Absorption	
Ni(CO) ₄			

Reactor Feed:

<u>Possible Impurities</u>	<u>Nominal Frequency of Sampling</u>	<u>Analytical Method Used</u>	<u>Detection Limit</u>
Fe(CO) ₅	Frequency determined by on-site technical staff	Scrub with K ₂ Cr ₂ O ₇ & HNO ₃ and Atomic Absorption	0.01 ppmv
Ni(CO) ₄			

TABLE III-7
LAPORTE CATALYST ANALYSES

<u>Catalyst (Extrudates and Circ. Fines) Properties of Interest</u>	<u>Analytical Method Used</u>	<u>Detection Limit</u>
Catalyst Inventory	Soxhlet Extraction	
Circ. Fines Conc., wt%		
Catalyst Particle Dimensions	Manually	
Catalyst Density, g/cm ³	Manually	
Side Crush Strength, kg	Manually	
Size Distribution	Manually	
Total Surface Area, m ² /g	B.E.T. Method	
Pore Volume, cm ³ /g	(N ₂ Displacement)	
Average Pore Radius, Å	(N ₂ Displacement)	
Copper as CuO, wt%	AA	+2% Relative
Zinc as ZnO, wt%	AA	+2% Relative
Iron, ppm	AA	+2% Relative
Nickel, ppm	AA	+2% Relative
Carbon, wt%	Elemental Analysis	
Alumina, wt%	AA	+2% Relative
Surface Valence States of Cu, Zn	ESCA/Auger	
Crystallite Size of Cu, Zn	XRD	