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DEVELOPMENT OF ALTERNATIVE FUELS FROM COAL-DERIVED SYNTHESIS GAS

Draft Topical Report

Demonstration of a One-Step Slurry-Phase Process for the Co-Production of Methanol and Isobutanol

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DEMONSTRATION OF A ONE-STEP SLURRY-PHASE PROCESS FOR THE CO-PRODUCTION OF METHANOL AND ISOBUTANOL

Executive Summary

Liquid phase co-production of methanol and isobutanol (LPIBOH) was demonstrated at DOE's Alternative Fuels Development Unit (AFDU) in LaPorte, Texas. Methanol and isobutanol are key intermediates in a synthesis gas-based route to methyl t-butyl ether (MTBE). The technology was demonstrated in a new 18" slurry bubble-column reactor that was designed to demonstrate higher pressures and temperatures, higher gas superficial velocities, and lower gas hourly space velocities -- all of which are conducive to obtaining optimal isobutanol yield. The integration of the new reactor into the AFDU included the addition of a high-pressure synthesis gas compressor, a high-pressure hydrogen feed source, and a closed-loop methanol-solvent absorption system to remove CO_2 from the unconverted synthesis gas. These modifications were completed in January 1994.

Because this was the first demonstration for the new 2000 psig oxygenates reactor system, it was preceded by an extended checkout and characterization period, including six days of carbonyl burnout and two-phase gas holdup studies, and eight days of LPMEOH operations. The new reactor performed quite well during the LPMEOH shakedown trial, which ran with standard BASF S3-86 methanol catalyst at 40 wt%. Two feed gases were simulated: Texaco gasifier gas and the H_2-rich design feed composition for the Clean Coal III LPMEOH demonstration plant at Kingsport. Reactor productivity met expectations for both gases with a methanol yield of about 11 T/D.

After the close of the Kingsport data period, an additional day was dedicated to testing some of the new equipment. One of the test conditions set a new record for the liquid phase technology program with an inlet gas superficial velocity of 1.17 ft/sec. The new reactor seemed to operate stably during this brief test period.

The LPIBOH run followed after a short turnaround. It employed a cesium-promoted $Cu/ZnO/Al_2O_3$ catalyst developed in Air Products' laboratories and subsequently scaled up to a production-sized batch. Over a thirteen day campaign on simulated Shell gasifier gas, the catalyst and reactor system were tested at a matrix of pressures (750, 1300, 1735 psig) and space velocities (3000, 5000, 8200 sL/kg-hr), representing numerous first-of-a-kind run conditions for the AFDU. Inlet gas superficial velocities spanned an impressive 0.16 to 1.0 ft/sec. Stable reactor performance for a full twelve-hour data period at 1.0 ft/sec was another significant milestone for the liquid phase technology program.

Initial operation at baseline conditions showed slightly superior performance over the laboratory autoclave as measured by the yield of isobutanol and total higher alcohols. Subsequent data periods generally followed laboratory trends, but began to fall short of the laboratory performance results. The catalyst appeared to be deactivating at a faster rate than observed in the autoclave.

Gas holdup and catalyst concentration calculations from nuclear density gauge measurements were generally close to expectations and steady throughout the run.

The last few cases used alcohol injection into the reactor feed to simulate the effect of lower alcohol (methanol, ethanol, propanol) recycle on isobutanol synthesis. Initially alcohol injection caused a nearly threefold increase in isobutanol productivity, which agreed with laboratory observations and results from the chain growth mechanism for production of isobutanol from lower alcohols. However, during the last injection case before the run returned to baseline, the catalyst was poisoned by the inadvertent presence of 1,1,1-trichloroethane in the injected alcohol reactant. This unexpected outcome significantly hindered the investigation of the faster rate of catalyst deactivation observed at the AFDU.

Apart from the catalyst deactivation, the run successfully demonstrated mixed alcohol synthesis in a slurry bubble-column reactor, as well as all of the new equipment installed for the trial. Although the full capabilities of the new oxygenates system will not be tested until future runs, the design objectives for the modifications were met with respect to the LPIBOH run and show every indication of being applicable to other chemistries.

Introduction

The Pittsburgh Energy Technology Center (PETC) sponsors an Indirect Liquefaction program as part of DOE's Coal Liquefaction program. The overall goal of the Coal Liquefaction program is to develop a scientific and engineering knowledge base for the manufacture of synthetic liquid fuels from coal, with which U.S. industry can bring economically competitive and environmentally acceptable technology into the marketplace.

Since the 1990 Clean Air Act Amendments, the market for oxygenated fuels has grown rapidly. MTBE (methyl t-butyl ether) has emerged as the most promising gasoline-blending oxygenate; its production has increased by a factor of 13 in the last 10 years. Currently, the reactants for MTBE production are derived from natural gas and petroleum feedstocks. For national energy security, the production of MTBE from non-petroleum sources is of current domestic interest.

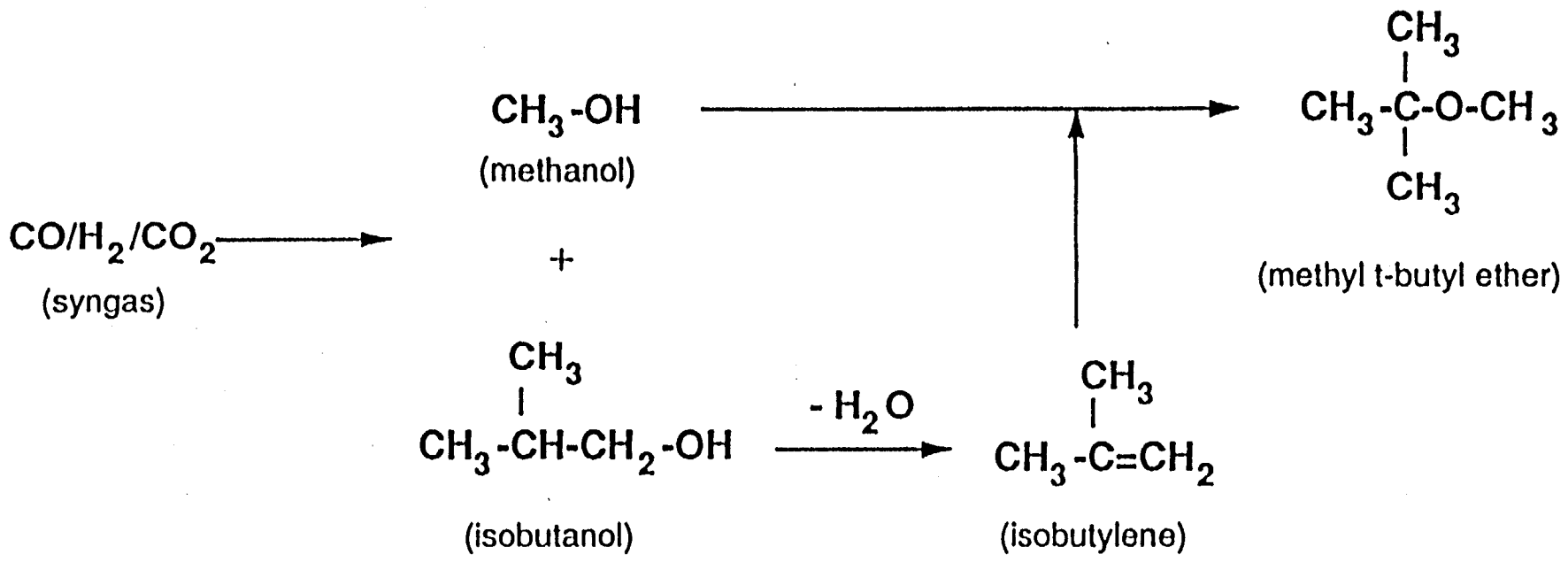
A major thrust of Air Products' ongoing DOE-sponsored Alternative Fuels contract has been the development of a new route to MTBE based on synthesis gas derived from domestic carbonaceous feedstocks, such as coal. The proposed route is shown schematically in Figure 1. Synthesis gas is converted by either a one- or two-stage process to methanol plus isobutanol. In the one-stage process, which is the subject of this work, methanol and isobutanol are co-produced in a single reactor. In the two-stage process, which is the subject of ongoing exploratory laboratory work, methanol is produced selectively from synthesis gas in the first reaction stage, and then part of the methanol is converted to isobutanol in a second reaction stage. The isobutanol produced by either of the two processes is then separated from the co-product methanol and dehydrated to isobutylene. Finally, the product isobutylene is reacted with methanol from the first step, and commercially established technology is used to produce MTBE.

Isobutanol synthesis and isobutanol dehydration are not currently practiced commercially, and thus represent the critical development issues. In previous work done by Air Products under the Alternative Fuels contract, isobutanol dehydration to isobutylene was successfully demonstrated at the pilot scale with high conversion (>90%) and excellent selectivity (>90%) compared with commercially available alumina catalyst (1). The most challenging aspect of the proposed route is the selective production of isobutanol from synthesis gas.

Because the synthesis of alcohols is highly exothermic and removal of this high reaction heat is problematic at high conversion, development has focused on the use of a slurry-phase reactor. This type of reactor has superior heat management capabilities, enabling high levels of catalyst productivity, and is amenable to on-line catalyst addition and withdrawal.

For this slurry-phase process, a cesium-promoted $Cu/ZnO/Al_2O_3$ catalyst was developed in Air Products' laboratories (2). This catalyst is effective at converting synthesis gas to branched primary alcohols, including isobutanol, at relatively low temperatures (540-600 °F) and, therefore, is compatible with slurry-phase operation using standard mineral oil as the liquid medium. Laboratory reaction studies, carried out using 50 ml and 300 ml continuous stirred autoclaves, revealed the best $Cu/ZnO/Al_2O_3$ substrate (BASF S3-86), the optimum cesium loading, and the calcination protocol. In the current study, production of this optimal catalyst was scaled up, and

Figure 1
Reaction Scheme



the conversion of synthesis gas to mixed alcohols was demonstrated at the pilot scale in a slurry bubble-column at DOE's Alternative Fuels Development Unit (AFDU) in LaPorte, Texas.

The AFDU was first commissioned in 1984 as a process development unit for the Liquid Phase Methanol (LPMEOH) process. It is located adjacent to Air Products' hydrogen and carbon monoxide production facility which allows for the demonstration of a wide range of simulated feedstocks. In addition to LPMEOH, the AFDU has been operated to demonstrate slurry-phase DME synthesis, water-gas shift reaction, Fischer-Tropsch synthesis, and isobutanol dehydration to isobutylene (1,3,4). All of these campaigns were carried out in a 22" diameter slurry bubble-column. As part of the current DOE program, the LaPorte AFDU was expanded to add a parallel reactor train, additional synthesis gas compression, and CO_2-removal equipment. The combined effect of these modifications is a more flexible facility to demonstrate and study the scaleup of oxygenated fuels technology.

Objectives

The main objective of this run was to demonstrate liquid phase co-production of methanol and isobutanol at the pilot scale, including scaleup of a proprietary catalyst. To accomplish this objective, the AFDU required significant modifications with the following design objectives:

- parallel reactor train dedicated to oxygenates technology development;
- demonstration at higher pressures and temperatures than existing reactor (2000 psig, 700°F design conditions vs. 1000 psig, 600°F);
- demonstration of higher gas superficial velocities and a wider range of space velocities;
- ability to remove CO_2 and DME from unconverted synthesis gas to maximize recycle and increase reactor throughput;
- ability to increase fresh feed capacity by tie-in to a new Air Products high-pressure hydrogen pipeline.

Engineering and Modifications

The main component of the AFDU expansion is an 18" diameter stainless steel reactor with a design pressure of 2,000 psig at 700°F. The design slurry height is 40 ft, which is twice that of the existing reactor. The internal heat exchanger in the new reactor has more than three times the heat removal capacity of the existing reactor in previous demonstration campaigns. This allows for more aggressive operating conditions with increased synthesis gas conversion. The reactor was designed to demonstrate higher pressures and temperatures, higher gas superficial velocities, and lower gas hourly space velocities--all of which are conducive to obtaining optimal isobutanol yield.

The integration of the new reactor with the existing 22.5" diameter reactor train included the addition of a high-pressure synthesis gas compressor, a high-pressure hydrogen feed source, and a closed-loop methanol-solvent absorption system to remove CO_2 from the unconverted synthesis gas. These modifications were completed in January 1994. The engineering effort is described in detail in another topical report (5).

Process Description

Process flow diagrams for the run are shown in Figures 2 and 3. The operation of the plant is described as follows:

Hydrogen, carbon monoxide, carbon dioxide, and nitrogen are blended and compressed in the 01.10 feed gas compressor. This stream then mixes with recycle gas and additional hydrogen from a high-pressure pipeline to obtain the desired synthesis gas composition and flow. The reactor feed then passes through the 01.15 cooling water exchanger before compressing to approximately 1800 psig in the 01.30 booster compressor. The 01.34 aftercooler is used to control the inlet temperature to the 21.11 feed/product economizer, which preheats the feed against the reactor effluent. The 10.95 high-pressure injection pump may be used to simulate water or alcohol addition to the reactor feed. After injection, the mixed feed is further preheated against high-pressure steam in the 02.63 to ensure vaporization of any injected liquids before the synthesis gas blend is introduced into the bottom of the new 27.20 high-pressure slurry reactor.

The synthesis gas flows upward through the slurry of catalyst and mineral oil as the reaction proceeds. The heat of reaction is absorbed by the slurry and removed through the internal heat exchanger, which also uses mineral oil as its heat transfer fluid. The alcohol products pass through the reactor freeboard with the unconverted synthesis gas, and the gross reactor effluent cools against the feed in the 21.11 economizer. Any traces of slurry oil entrained in the effluent condense and are returned to the bottom of the reactor by the 10.52.02 pumps. The vapor leaving the 21.11 de-pressurizes across a valve to less than 1000 psig; chills against cooling water in the 21.30 hairpin exchangers; and passes into the 22.10 separator where any liquid products (methanol, water, higher alcohols) collect. The liquids flash to near atmospheric pressure in the 22.11 degasser and collect in the 22.15 low-pressure separator before passing on to the 22.16 day tank and eventually a trailer for storage.

To minimize the amount of gas sent to the flare, most of the synthesis gas leaving the 22.10 separator is recycled to the reactor. Since CO_2 is a by-product of much of the oxygenated fuels chemistry, it is often necessary to remove CO_2 from the 22.10 overheads before this stream is recycled. The new closed-loop CO_2-removal system uses methanol to preferentially absorb the CO_2 from the synthesis gas.

The vapor from the 22.10 cools against returning CO_2-lean synthesis gas in the 21.10 gas-gas economizer. It then feeds into the bottom of the 07.10 absorber and contacts against chilled methanol introduced at the top of the column. The CO_2-lean synthesis gas leaves the top of the absorber and rewarms to ambient temperatures in the 21.10 before being recompressed in the 01.20 recycle compressor. A small portion of this gas is purged to flare to prevent the buildup of inerts.

The CO_2-rich liquid collects in the bottom of the 07.10 absorber, de-pressurizes across a valve, and heats up against returning methanol in the 21.45 hairpin exchangers. This liquid then passes into the top of the 07.20 stripper, where it is reboiled to remove the dissolved gases such as CO_2 and DME. The overhead cooling water condenser reduces the amount of methanol solvent lost in the overhead stream, which goes to flare. The liquid from the bottom of the 07.20 cools in the

FIGURE 2

LAPORTE AFDU OXYGENATES MODIFICATIONS - FEED COMPRESSION AND SYNTHESIS

AIR PRODUCTS

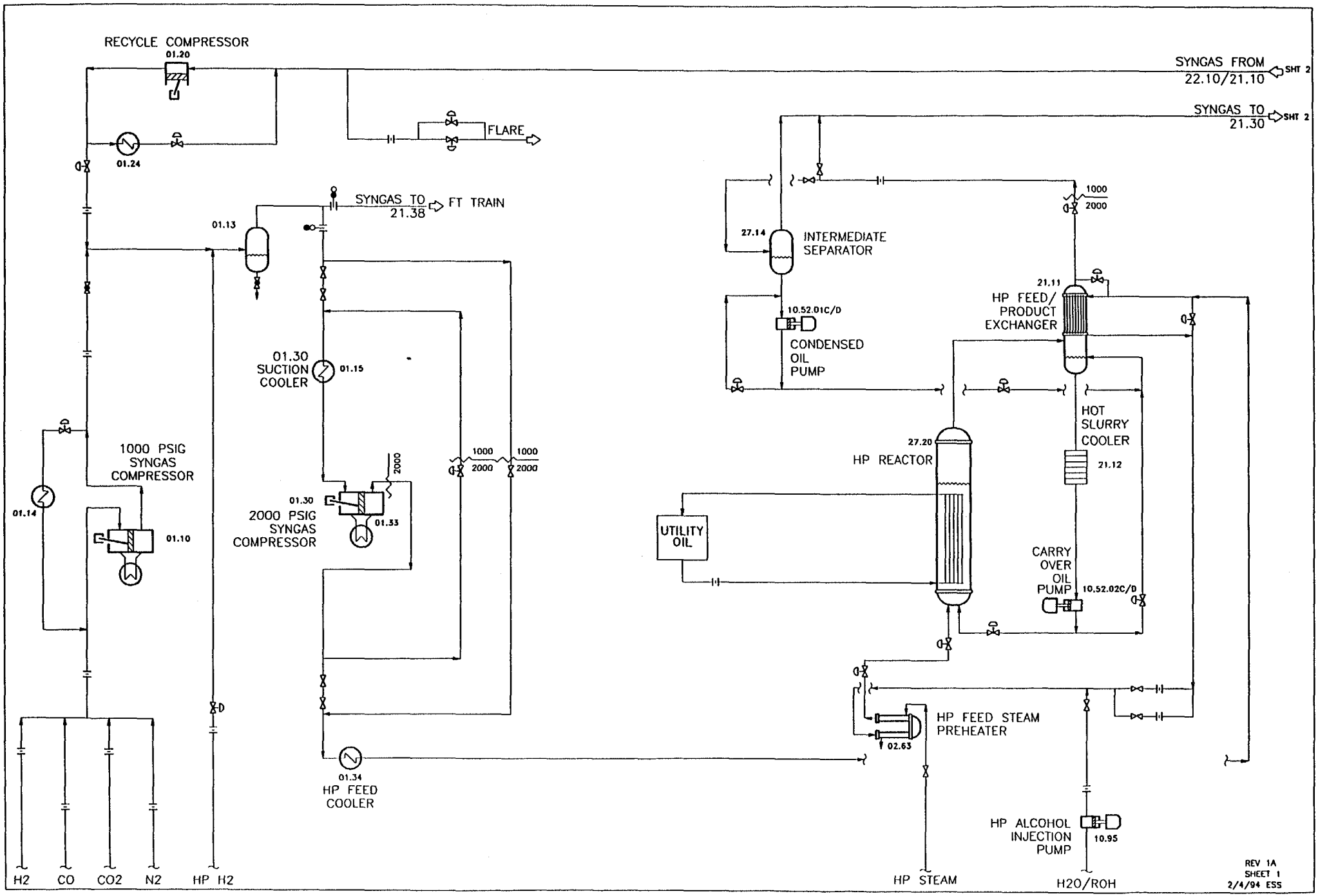
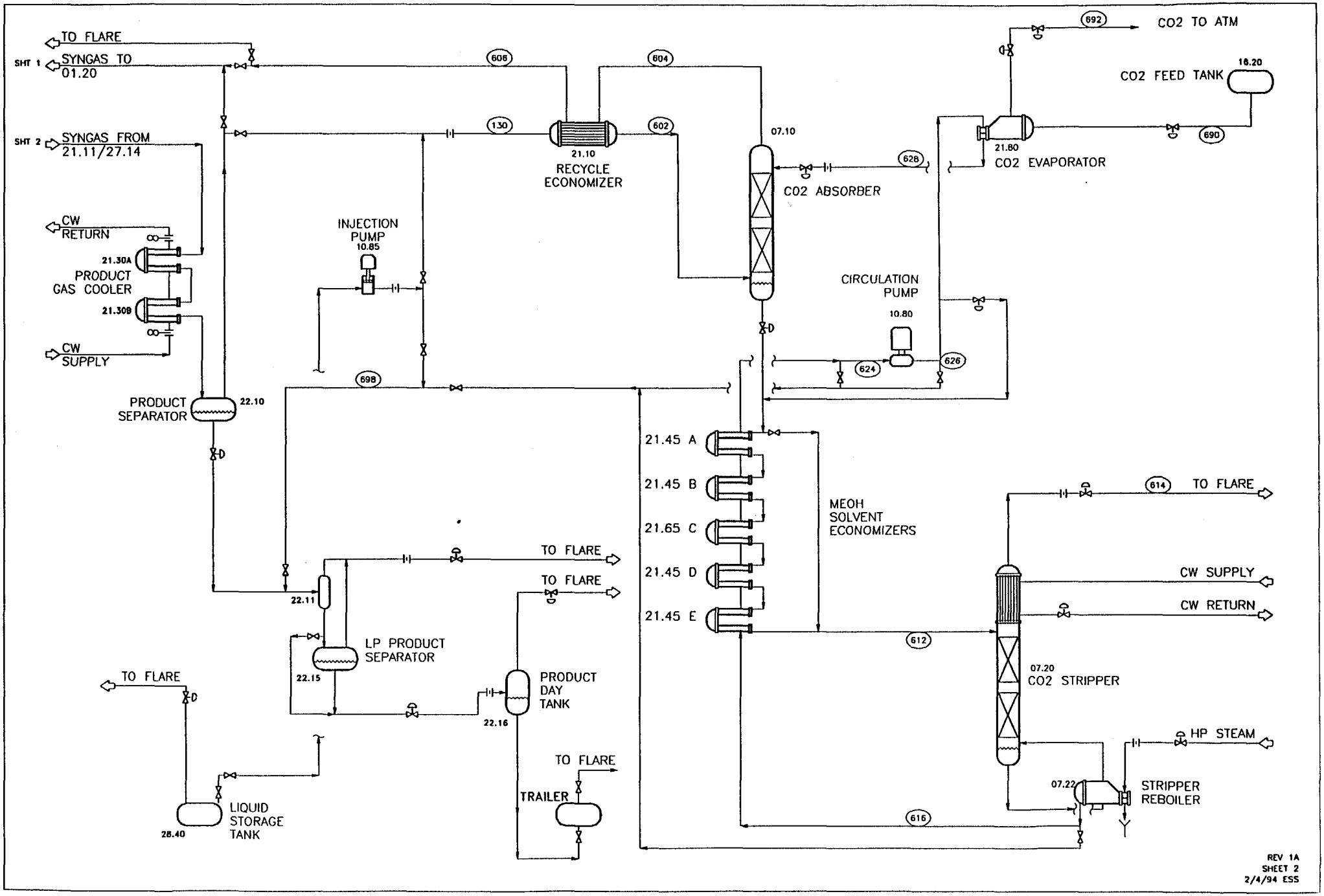


FIGURE 3
LAPORTE AFDU OXYGENATES MODIFICATIONS: PRODUCT COLLECTION & CO2 REMOVAL

AIR PRODUCTS



21.45s prior to recompression in the 10.80 pump. The methanol then chills against liquid CO_2 in the 21.80 kettle evaporator before recycling to the top of the 07.10 absorber.

The synthesis gas from the 22.10 will include equilibrium amounts of methanol, water, and other hydrocarbons, which will build up in the methanol solvent. Methanol is also lost in the 07.20 overheads. As a result, the CO_2-removal system operates in an unsteady state as the composition of the solvent changes. Since this change affects the level of CO_2 removal, the system includes a solvent purge and fresh methanol makeup lines.

Bubble-Column Reactor

The new 27.20 bubble-column reactor for oxygenate synthesis measures 50 ft flange-to-flange and has an 18" inside diameter. Its design slurry level is 40 ft with the remainder being vapor disengagement space. The reactor contains an internal heat exchanger consisting of twelve 3/4" U-tubes occupying 8% of the reactor cross section. In addition, thirteen thermocouples measure the longitudinal temperature profile at 4 ft intervals. A nuclear density gauge, mounted on an external hoist mechanism, spans the space occupied by the internal exchanger to measure slurry level and gas holdup. The design pressure of the reactor is 2000 psig at 700°F.

Analytical Setup

Existing AFDU GCs were modified for on-line analysis of various process gas streams. Two GCs with Flame Ionization Detectors (FIDs) monitored hydrocarbon and alcohol concentrations in the reactor feed and effluent streams. Methods and standards for measuring concentrations of the numerous higher alcohol products were developed during the laboratory studies of this reaction (2). Two other GCs with Thermal Conductivity Detectors (TCDs) measured H_2, N_2, CO, CO_2, H_2O, MeOH, and DME in feed, product, purge, and intermediate streams. A small amount of N_2 (approximately 1 mole%) was added to the reactor feed as an internal standard to verify flow measurements.

Environmental and Safety Issues

The engineering team conducted extensive hazard reviews to evaluate safety issues associated with the plant modifications and LPIBOH operations. These sessions included a preliminary hazards review (PHR), a design hazards review (DHR), and a design verification review (DVR), all held at different stages of the project. In addition to the new sections of the plant, most of the existing sections required reviews because of new high-pressure and -temperature (2000 psig and 700 °F) tie-ins.

The hazard review team performed a "What If" analysis of the pilot plant to identify hazards. Existing safety relief devices were checked for new cases related to high-pressure tie-ins, and in some cases those devices or their piping circuits required modifications. Furthermore, relatively new regulations imposed by OSHA Process Safety Management (PSM) required complete documentation of cases and calculations for all of the existing relief devices, of which there were about 85 in total. In addition, approximately 30 new pressure relief devices were specified to cover new equipment. Subsequent work involved re-evaluating the vent lines and headers for all these devices, as well as checking the main flare header for all of the new flows. This effort led to

a change in scope for the relief header from the new CO_2-removal system to include a new, local flare knockout pot.

Flare radiation effects were also evaluated, and calculations indicated that the existing flare would provide an acceptable radiation heat level to operators at grade or on any of the nearby platforms. Investigations also centered on the possibility of an unconfined vapor cloud explosion (UVCE) should the flare go out and flammables begin to accumulate. Even under this scenario, calculations showed that blast pressure and radiation effects would be acceptable. However, CO toxicity effects of this scenario warranted institution of new procedures for operator response to a flare malfunction condition. The CO_2 vent from the 21.80 evaporator underwent a similar analysis, and the hazard team recommended restricted access to the top platform in a nearby structure to reduce the potential for exposure to CO_2 releases.

The hazard team also conducted a plant Operational Readiness Inspection (ORI) in early February as a final review of the revisions and preparations for the upcoming run. Operator training was completed by process and plant personnel at this time per OSHA PSM standards.

The air emission operating permit exemption filed in 1993 for the LPIBOH run was initially deemed insufficient by the Texas Natural Resource Conservation Commission (TNRCC) in early February 1994. Air Products agreed with the TNRCC to place time limitations on each reaction chemistry demonstrated at the AFDU to satisfy the new requirements of the EPA's Clean Air Act. Based on previous and current operating campaign durations, the accepted time restrictions should not restrict usage of the AFDU so as to inhibit meeting the goals of the Alternative Fuels contract. Additional process calculations and all supporting information were compiled and forwarded to the TNRCC on February 18. Written approval of the exemption for LPIBOH followed on February 28, 1994, in time for start-up.

Catalyst Development

The catalyst used in this work was a commercially available $Cu/ZnO/Al_2O_3$ methanol synthesis catalyst that had been promoted with cesium. Laboratory reaction studies, carried out using 50 ml and 300 ml continuous stirred autoclaves, revealed the best $Cu/ZnO/Al_2O_3$ substrate (BASF S3-86), the optimum cesium loading, and the calcination protocol. Production of this optimal catalyst was then scaled up to a pilot-scale quantity for use in the AFDU. The entire catalyst development effort is described in detail in another topical report (2).

AFDU Shakedown

Preliminary results from this demonstration were reported earlier at DOE's Liquefaction Contractors' Review Conference (6). Because this was the first demonstration for the new 2000 psig oxygenates reactor system, it was preceded by an extended checkout and characterization period, including six days of carbonyl burnout and two-phase gas holdup studies, and eight days of LPMEOH operations.

Two-Phase Gas Holdup Studies

The Nuclear Density Gauge (NDG) for the new 27.20 reactor was calibrated on March 8, 1994. Two-phase gas holdup studies were performed between March 11-16 to investigate the

hydrodynamics of the new reactor. These tests were conducted concurrently with carbonyl burnout and equipment testing. The tests used both N_2 and a CO-rich synthesis gas (nominally 34% H_2, 65% CO, 1% N_2) at operating pressures and temperatures pertinent to the LPMEOH and LPIBOH demonstrations. The results are summarized in Figures 4 - 6.

Figure 4 shows all of the gas holdup data as a function of superficial gas velocity. The data concentrated on pressures of 750 psig and higher. At these pressures, little sensitivity to pressure or temperature was seen, and all the data collapsed toward one basic curve. The results appear to mimic the trends seen during the two-phase gas holdup studies from the LP-III E-5 methanol run (7). However, the current tests included much higher pressures (1750 psig vs. 900 psig) and superficial velocities (0.84 vs. 0.44 ft/sec) than the E-5 tests.

Notably, the gas holdup leveled off above about 0.4 ft/sec at a slightly higher level than in the previous studies (65-70% vs. 60-65%). This result may indicate errors introduced by NDG measurements that are more pronounced in the new reactor. Specifically, the smaller shell diameter and greater area blocked by heat exchanger tubes may contribute to more significant radial variations in the velocity profile. These variations, when weighted according to linear radiation path instead of cross-sectional area, may contribute to inaccuracies in the prediction of average gas holdup from NDG measurements.

In Figure 5, the two-phase gas holdup is plotted as a function of reactor height for various superficial velocities. The curves all exhibit the same general shape, including an interesting, yet repeatable, drop in gas holdup at 63", and again to a lesser degree at 135". The scale is expanded for the higher velocity curves in Figure 6 to magnify this phenomenon. These locations are 6"-8" below the bottom two U-tube supports within the reactor. The supports appear to provide enough of an obstruction to the flow to become manifest in the holdup profile. As the flow becomes more fully developed while moving up through the reactor, the effect decreases until it is no longer evident past the second support.

Carbonyl Burnout

The two-phase gas holdup studies were conducted concurrently with carbonyl "burnout." Because iron and nickel carbonyl, which are known catalyst poisons, can form on new stainless steel, synthesis gas is circulated through the plant to measure background carbonyl levels and passivate the steel if necessary. Initial conditions for carbonyl burnout were 482°F and 750 psig (LPMEOH conditions). Flows varied considerably during the burnout period from once-through to high recycle to accommodate the gas holdup studies. Early on March 14 the temperature was increased to 572°F to simulate conditions for isobutanol synthesis, and later that morning the new 02.63 steam preheater also came on-line. The new 01.30 high-pressure compressor then came on-line late that afternoon for holdup studies at 1300 and 1750 psig. During this period of widely varying operating conditions, $Fe(CO)_5$ readings peaked as high as 400 ppb at the reactor outlet. $Ni(CO)_4$ was never detected at any time.

Early on the morning of March 15, the pressure and temperature were returned to the baseline LPMEOH conditions with the 02.63 still on and the recycle compressor boosting total reactor flow to 78,000 SCFH. At this point, the baseline carbonyl values had leveled off at 62-65 ppb

FIGURE 4

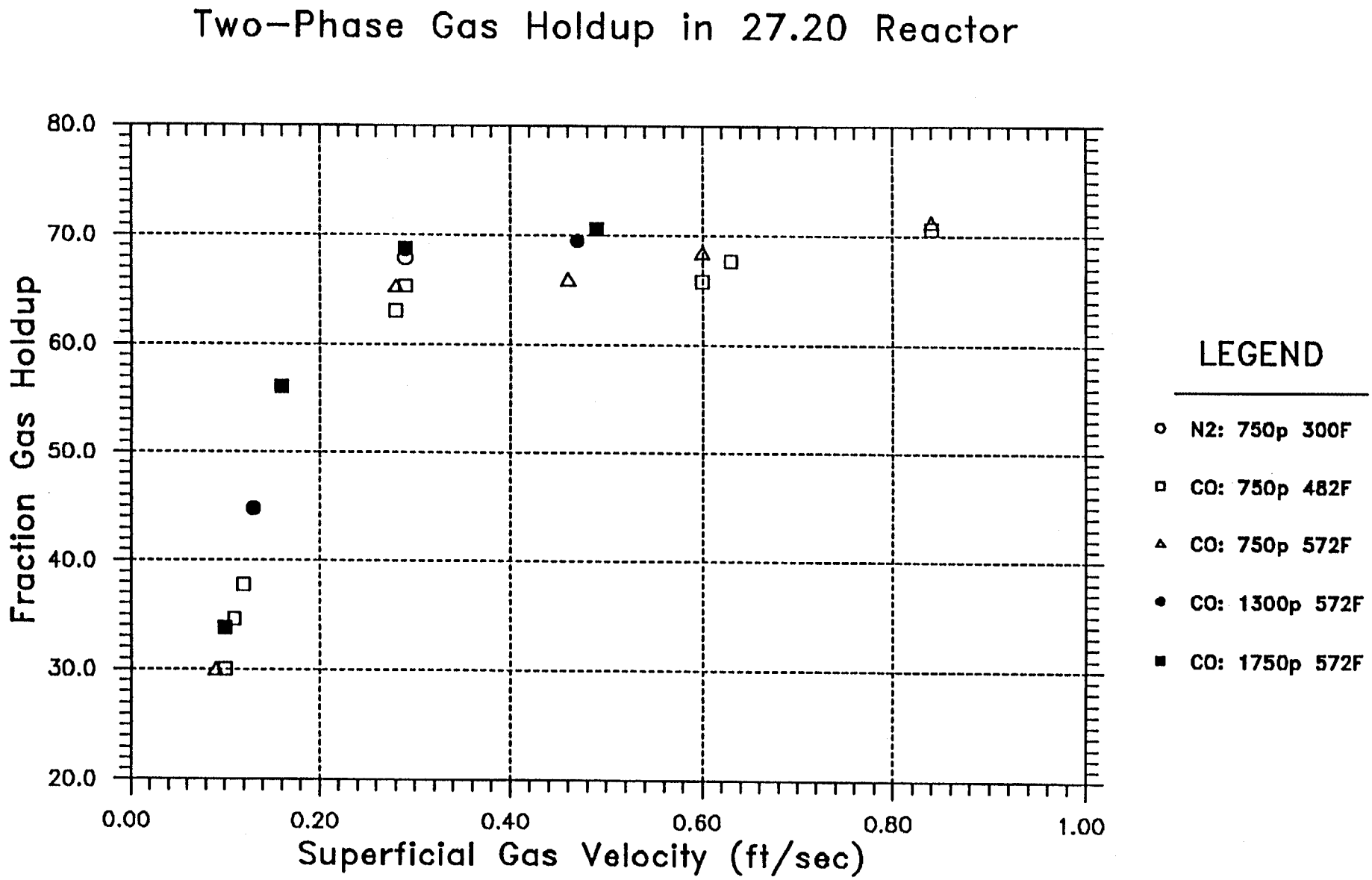

Two-Phase Gas Holdup in 27.20 Reactor
Fraction Gas Holdup
80.0
70.0
60.0
50.0
40.0
30.0
20.0
0.00
0.20
0.40
0.60
0.80
1.00
Superficial Gas Velocity (ft/sec)
LEGEND
N2: 750p 300F
CO: 750p 482F
CO: 750p 572F
CO: 1300p 572F
CO: 1750p 572F

FIGURE 5

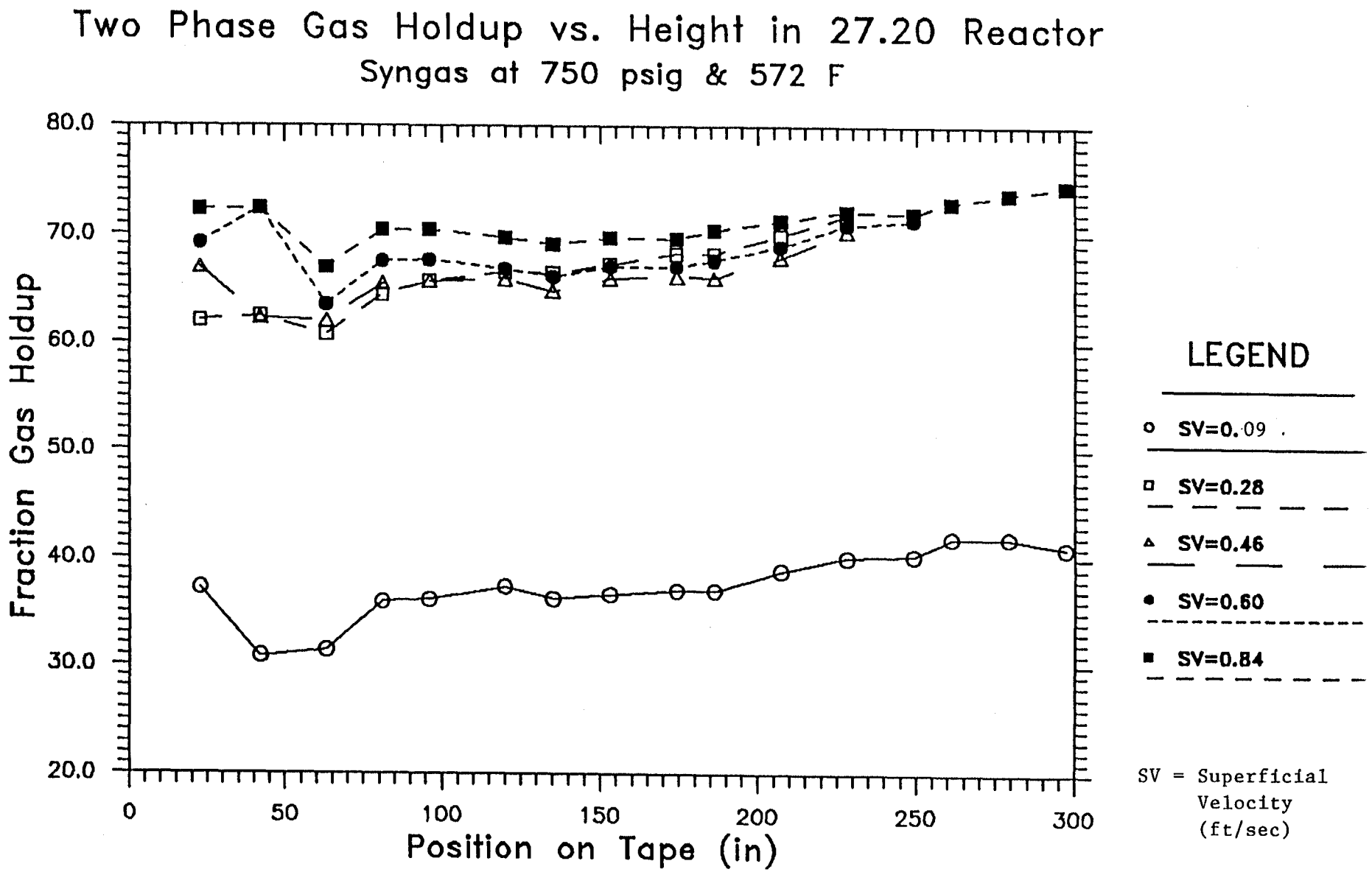
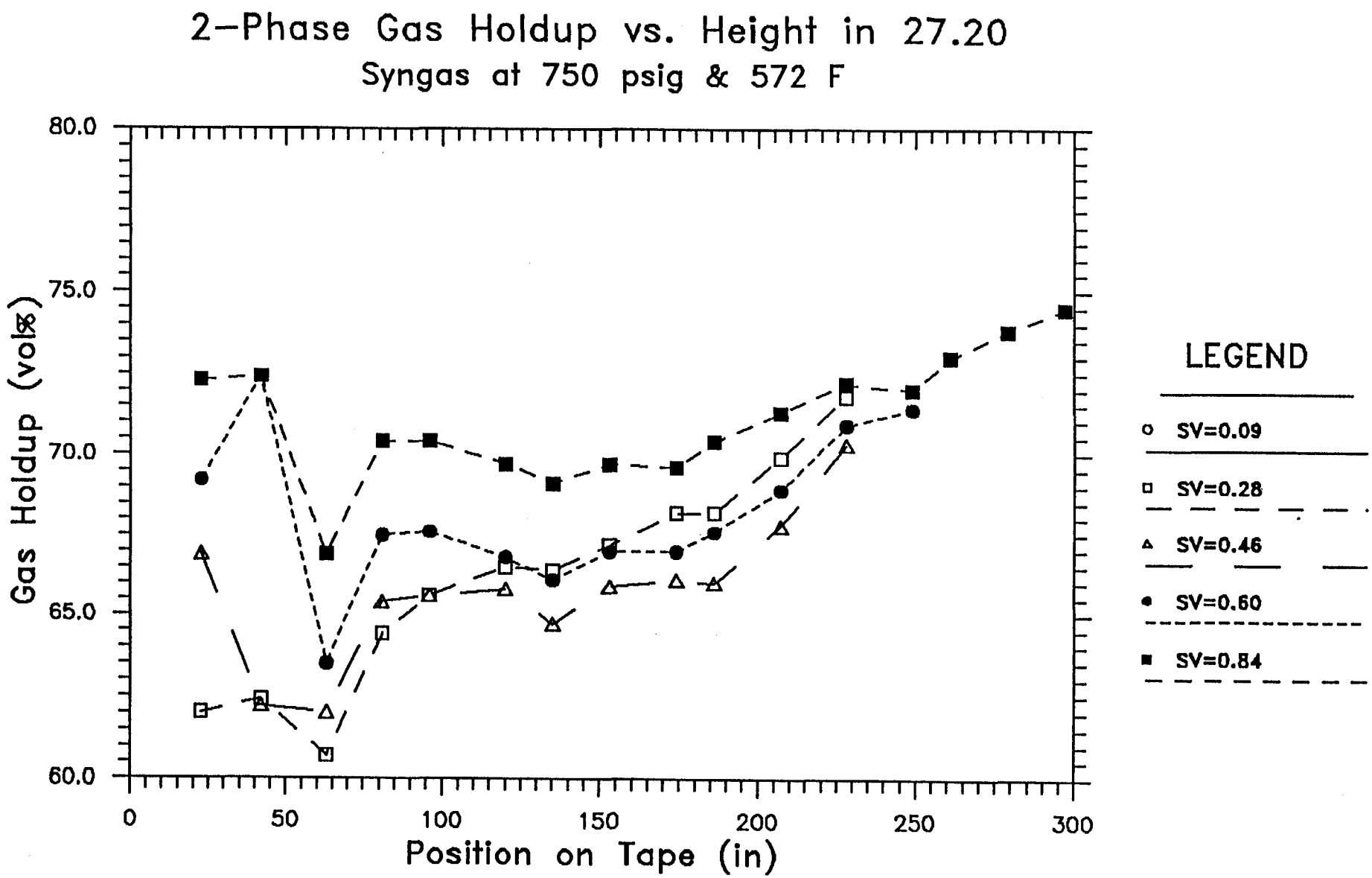

Two Phase Gas Holdup vs. Height in 27.20 Reactor
Syngas at 750 psig & 572 F
Fraction Gas Holdup
80.0
70.0
60.0
50.0
40.0
30.0
20.0
0
50
100
150
200
250
300
Position on Tape (in)
LEGEND
SV=0.09
SV=0.28
SV=0.46
SV=0.60
SV=0.84
SV = Superficial Velocity (ft/sec)

FIGURE 6


2-Phase Gas Holdup vs. Height in 27.20
Syngas at 750 psig & 572 F
Gas Holdup (vol%)
80.0
75.0
70.0
65.0
60.0
0
50
100
150
200
250
300
Position on Tape (in)
LEGEND
SV=0.09
SV=0.28
SV=0.46
SV=0.60
SV=0.84

$Fe(CO)_5$ at the reactor inlet and 68-71 ppb at the reactor outlet. The reactor loop was then cooled and drained. The oil removed from the system contained a visually significant quantity of metallic fines, which apparently contributed to the high level of carbonyl generation.

The system was recharged with fresh oil and brought back up to the baseline LPMEOH conditions, with once-through CO-rich synthesis gas at 20,000 SCFH. Iron carbonyl measurements quickly leveled out at much lower values: 2 ppb at sample point 4 (combined outlet of the 01.10 and 01.20 compressors); 7-9 ppb at sample point 15 (reactor inlet); and 16-17 ppb at sample point 3A (reactor/21.11 outlet). These results seemed to indicate that the particulates flushed out by the first charge of oil had indeed increased the production of iron carbonyls.

The reactor temperature and pressure were then raised to the most extreme conditions for isobutanol synthesis (572°F and 1750 psig) and held for approximately six hours in once-through mode at 20,000 SCFH. Again, no nickel carbonyls were detected, and iron carbonyls in the reactor outlet leveled off at approximately 80 ppb. This higher baseline was not unexpected at these conditions.

To determine if a higher flow rate would decrease the production of carbonyls, the plant was finally run in recycle mode at 58,000 SCFH for four hours. This condition simulated the minimum space velocity case planned for the isobutanol campaign. The $Fe(CO)_5$ measurements were steady at approximately 60 ppb at sample point 4 and 100 ppb at sample point 3A. These results indicate that indeed fewer carbonyls are formed as flow increases, with about 40 ppb $Fe(CO)_5$ produced at this condition, compared to about 80 ppb produced at 20,000 SCFH. Furthermore, it was surmised that additional time on-stream during methanol operations might reduce carbonyl levels even further before the start of isobutanol synthesis. Regardless of that speculation, however, these concentrations were deemed acceptable for the time scale of the operating campaign.

The carbonyl burnout period concluded at 23:55 on March 16, and the plant was cooled and drained in preparation for LPMEOH catalyst loading and reduction.

LPMEOH Catalyst Reduction

The LPMEOH shakedown run for the new reactor commenced on March 17, 1994. Test Authorizations are included in Appendix A, and the overall Run Chronology appears in Appendix B. A 40 wt% oxide catalyst slurry was prepared in the 28.30 catalyst prep tank by mixing 1875 lbs of Drakeol-10 oil with 1250 lbs of standard BASF S3-86 methanol catalyst. The slurry was heated and agitated in the prep tank prior to transfer to the reactor.

Catalyst reduction commenced at 22:00 on March 17. The procedure was carried out at 67 psig and 12,500 SCFH of dilute synthesis gas (nominally 1.4% H_2, 1.9% CO, 96.7% N_2). This flow rate was lower than specified in Test Authorization #37 because of some difficulties in keeping the flare lit with the high N_2 flow. The pressure was lowered accordingly to maintain the appropriate superficial velocity. In addition, the H_2 and CO concentrations were increased

slightly from the target (1.2 and 1.8 mole%, respectively) to increase the flammability of the purge gas going to the flare.

The reduction temperature ramp is shown in Figure 7. It increased at 15°F/hr before entering a 12-hour hold period at 392°F. The temperature ramp then continued at 15°F/hr until the procedure concluded after a one hour hold at 464°F. The slight "stagger" in the curve near 310°F resulted from a temporary hold in the ramp rate to confirm the calculated uptake rates. Gas holdup measurements taken during the process with the NDG were steady at 26%.

Catalyst reduction concluded at 06:42 on March 19. Figures 8 and 9 show uptake vs. time and uptake vs. temperature for the entire reduction period. For these plots, the bottom curve represents H_2, the middle curve represents CO, and the top curve shows the total uptake. The final uptake was 2.34 SCF/lb of catalyst oxide, compared to a theoretical maximum value of 2.82 SCF/lb. This result is comparable to the 1991 LPDME activation, although the uptake rate was faster in that case.

LPMEOH Operations

A slightly H_2-rich synthesis gas blend was introduced to the plant once-through at 10:20 on March 19, 1994. The reactor reached 750 psig at 10:53, and recycle commenced at 11:12. Methanol first appeared in the reactor effluent at 11:22. During the first few hours of operation, significant oil carry-over contaminated the product. Subsequent discovery of an erroneous thermocouple reading controlling the 21.11 feed/product economizer outlet temperature, and operating optimization of its setpoint, markedly decreased the oil carry-over rate. During the balance of the first two days of operation, efforts were directed toward maintaining steady operation with the new equipment; reconciling flows, compositions, and liquid accumulation rates; and identifying other instrumentation (i.e., flowmeters, thermocouples, switches) that needed further calibration and adjustment. In addition, the new high-pressure H_2 tie-in to the fresh feed circuit, and its attendant safety systems, complicated the control of the reactor feed stream and caused several upsets due to swings in source pressure.

Operations were temporarily suspended at 11:52 on March 21, when CO feed flow was interrupted by a power loss at our supplier. An immediate slump test on the reactor slurry confirmed earlier predictions of 40 wt% catalyst concentration. The procedures for attaining the standby condition were executed next, principally cooling the reactor and purging with 1-2% H_2 in N_2. However, the power loss also knocked out cooling water pumps, which, in turn, eventually tripped the feed compressor. As a result, the plant was also depressurized, and the purge flows were re-established in reduction mode at 100 psig. During the outage, the high-pressure H_2 feed location was modified to improve controllability.

Synthesis gas was re-introduced to the plant at 17:51, and steady AF-R9.1 (Texaco gas) operating conditions were achieved by 01:25 on March 22. After restarting, operations remained stable through the balance of the Texaco data period. The addition of a new high-pressure H_2 tie-in at the suction of the 01.20 recycle compressor significantly stabilized the control of the reactor feed composition with respect to fluctuations in H_2 source pressure. The original tie-in point was left undisturbed, so that in future campaigns, for which very high recycle ratios are

FIGURE 7

Reduction Temperature Ramp

LPMEOH

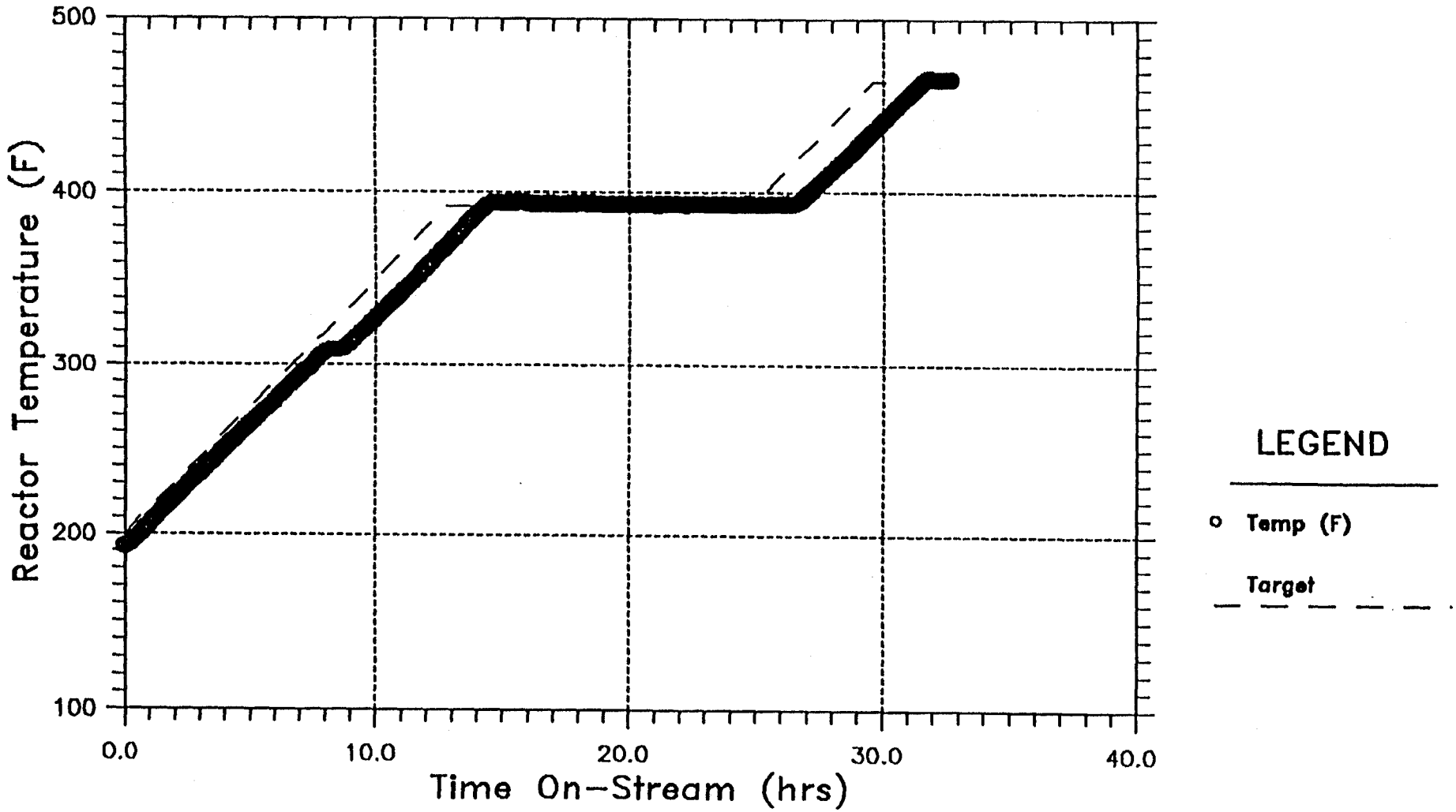


FIGURE 8

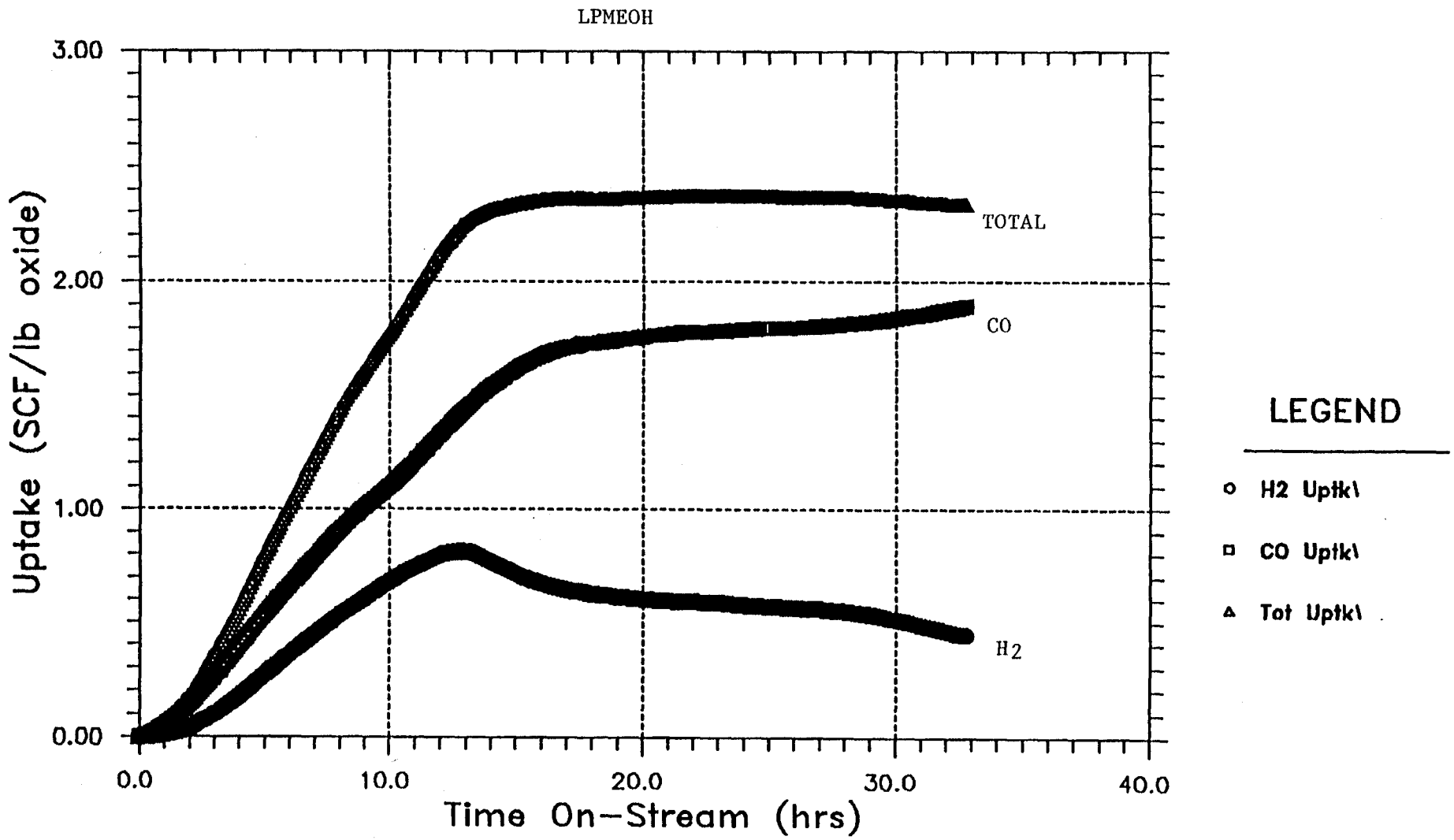
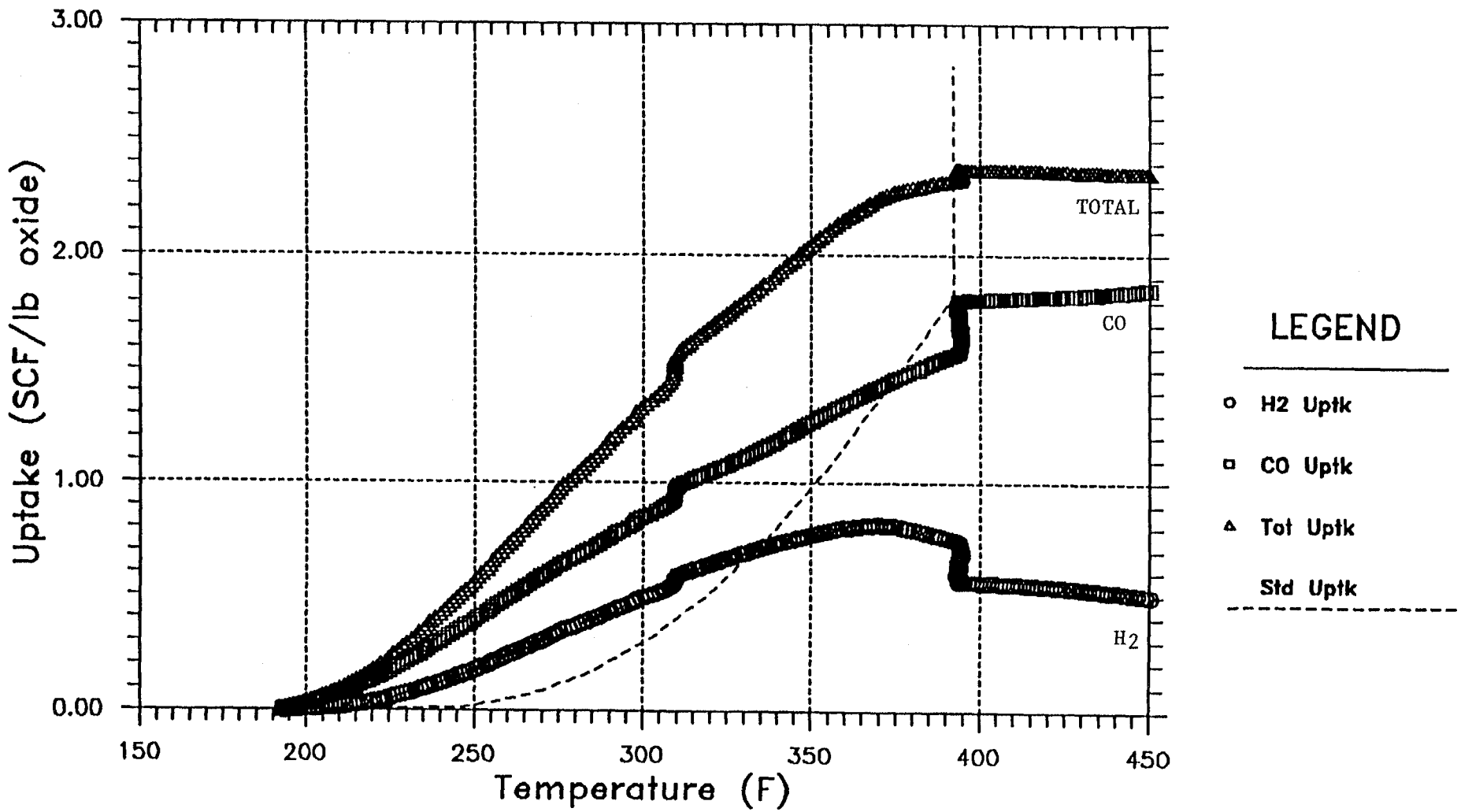

Reduction Gas Uptake vs. Time
LPMEOH
Uptake (SCF/lb oxide)
3.00
2.00
1.00
0.00
0.0
10.0
20.0
30.0
40.0
Time On-Stream (hrs)
TOTAL
CO
H2
LEGEND
H2 Uptk\
CO Uptk\
Tot Uptk\

FIGURE 9

Reduction Gas Uptake vs. Temperature

LPMEOH



necessary, the fresh feed system will not be limited by the capacity of the 01.20 recycle compressor.

The AF-R9.1 data period concluded at 21:00 on March 22. Mass balances for each run condition appear in Appendix C. CO conversion averaged 14.5% per pass for the average conditions of 6832 sL/kg-hr space velocity, 750 psig, and 476°F. Notably, this result exactly matches the predicted conversion for one CSTR at these conditions. The methanol concentration in the reactor effluent averaged 9.1 mole%, and liquid production, measured from levels in the 22.16 day tank, averaged 11.1 tons/day (TPD) of contained methanol. Average oil concentration for the three liquid product samples taken during the data period was 0.243 wt%. NDG measurements showed an average gas holdup of 47.2% and a catalyst concentration of 45.4 wt%. Since predicted values were 42.9% holdup and 42.4 wt% catalyst loading, these results further substantiate the tendency of the NDG to overpredict the extrapolation of previous results to high superficial velocities, as seen in the two-phase holdup studies.

Reactor feed flow and composition swung over to data period AF-R9.2 (Kingsport gas) after 21:00. The transition proceeded very smoothly, and compositions lined out at the H_2-rich reactor feed (nominally 61% H_2, 25% CO, 4% N_2, 10% CO_2) by early morning on March 23. This condition was included in the run plan to obtain data on the proposed operating conditions and feed composition of the Kingsport, Tennessee commercial-scale reactor. The scheme included the collection of bulk liquid product for analysis and testing by Eastman Chemical, and installation of a new line to collect 150 cc high-pressure sample bombs of the methanol product for detailed analysis in Air Products' laboratories. That analysis was intended to quantify the impurity levels generated under these conditions, specifically lighter components such as methyl formate and methyl acetate and higher alcohol by-products such as ethanol. To ensure minimum oil levels in the product, the reactor effluent stream passed through both the 21.11 and the 27.14 separator for the duration of this test.

The AF-R9.2 data period ended at 8:30 on March 24. Overall, operations were smooth except for one significant disturbance in the high-pressure H_2 source pressure. The new tie-in location enabled the plant to stabilize quickly and continue operations at target conditions despite the disturbance.

Reconciling the mass balance required a 20% adjustment to the methanol reading in the reactor effluent stream to match the production recorded in the day tank. Once this adjustment was incorporated (to 17.1 mole%), the balance closed tightly. The standard used for reactor effluent calibration of the GCs contained 6% methanol, which is the maximum level for a gas standard. Measurements of methanol concentrations significantly higher or lower than the calibration value are subject to large errors because of the non-linearity of the calibration. Our on-site analytical expert confirmed that errors of 10-20% could be expected at these high concentrations.

CO conversion averaged 46.5% per pass for the average conditions of 4020 sL/kg-hr space velocity, 739 psig, and 483°F, while the predicted conversion at these conditions is 46.9% for three CSTRs. Liquid production, measured from levels in the product collection area, averaged 11.0 TPD of contained methanol. Average oil concentration for the four liquid product samples

collected during the data period was 0.123 wt%. NDG measurements showed an average gas holdup of 33.5% and a catalyst concentration of 39.4 wt%, which compared well with the predicted value of 34.5% at 40.0 wt% catalyst loading.

The remainder of March 24 was dedicated to testing of some of the new equipment. One of the test conditions required a feed rate of 200,000 SCFH, a point that broke new ground for the LP technology program. The reactor inlet superficial velocity for this case was 1.17 ft/sec. NDG analysis showed a slurry concentration of 44.2 wt% and 48.8% gas holdup. (In order to run this test condition without removing slurry from the reactor, it was necessary to thicken the slurry from the AF-R9.2 run.) The new reactor seemed to operate stably during this brief, two-hour test period.

Shutdown and Turnaround

The plant was shut down at 22:40 on March 24, 1994. The reactor was depressurized, cooled, drained, and flushed on March 25. The LPIBOH catalyst slurry was prepared in the 28.30 prep tank, as described below, and kept agitated, warm, and under N_2 purge. Meanwhile, flush oil was added to the reactor and heated to 300°F overnight to remove the residual methanol slurry in the system. On March 26 the flush oil was drained, and the LPIBOH catalyst slurry was transferred to the reactor.

LPIBOH Demonstration

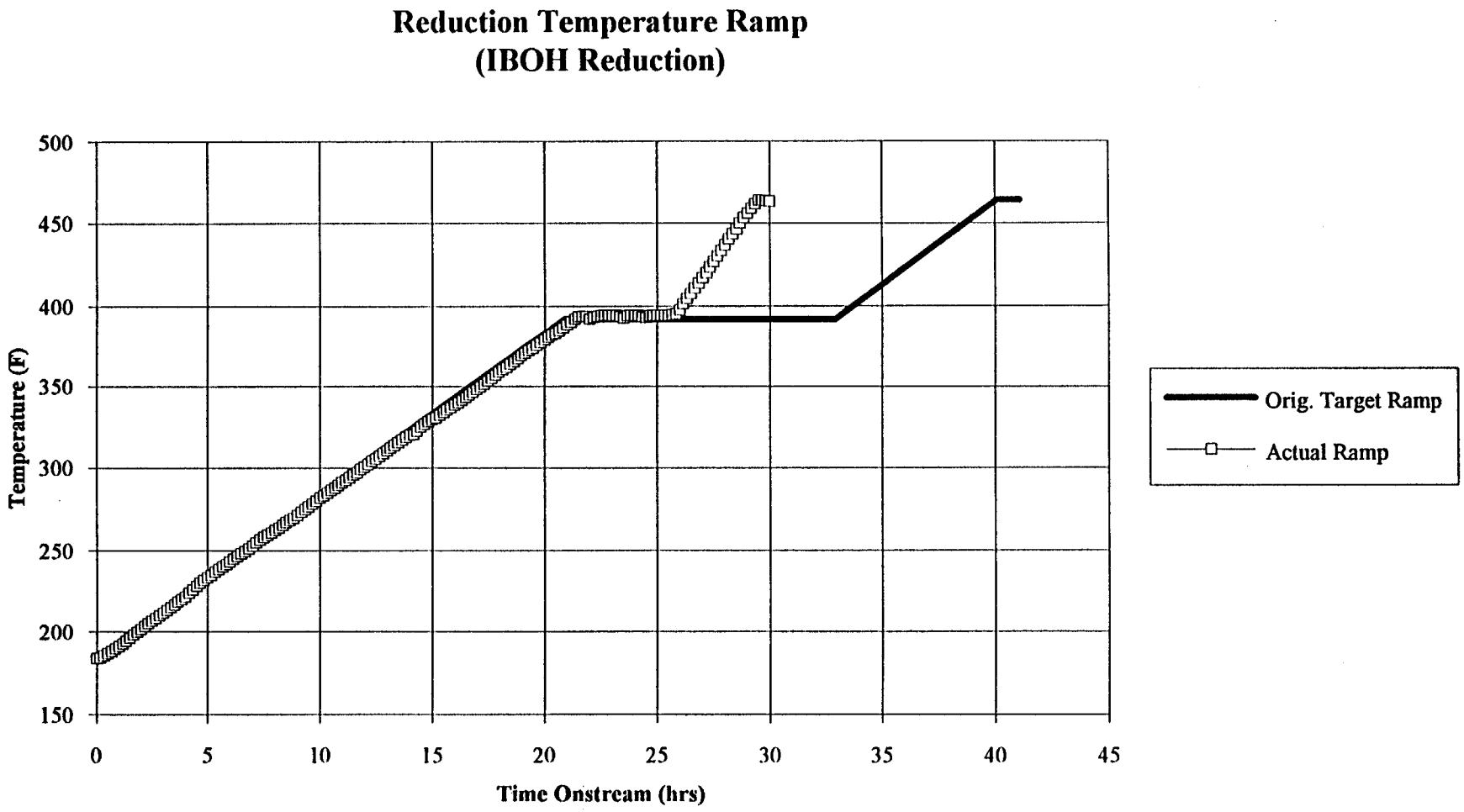
LPIBOH Catalyst Reduction

A 40 wt% oxide catalyst slurry was prepared in the 28.30 catalyst prep tank by mixing 1659 lbs of Drakeol-10 oil with 1106 lbs of LPIBOH catalyst (Cs-promoted BASF S3-86). The slurry was transferred to the reactor, and an additional 283 lbs of flush oil were added to the prep tank to remove any additional catalyst left from the transfer.

Reduction of the Cs-doped catalyst for isobutanol synthesis commenced on March 26 at 14:00, following the temperature ramp shown in Figure 10. Reduction proceeded under dilute synthesis gas (nominally 1.4% H_2, 2.1% CO, 96% N_2, 0.5% CO_2) at 14,000 SCFH and 60 psig. As in the case of LPMEOH, these conditions were decreased from the Test Authorization values to maintain stable flare ignition at the appropriate superficial velocity. The ramp proceeded at 10°F/hr to a hold temperature of 392°F. Notably, no "light-off" period occurred, which is typical of previous in-situ reductions in which the reaction exotherm caused the temperature to exceed the intended ramp rate. This phenomenon also failed to occur during the initial LPMEOH reduction and the subsequent transition to operations on synthesis gas. The apparent improvement in temperature control is attributed to the greater than threefold increase in heat transfer area for the new reactor, compared to the old reactor system in which the previous "light-off" experience occurred.

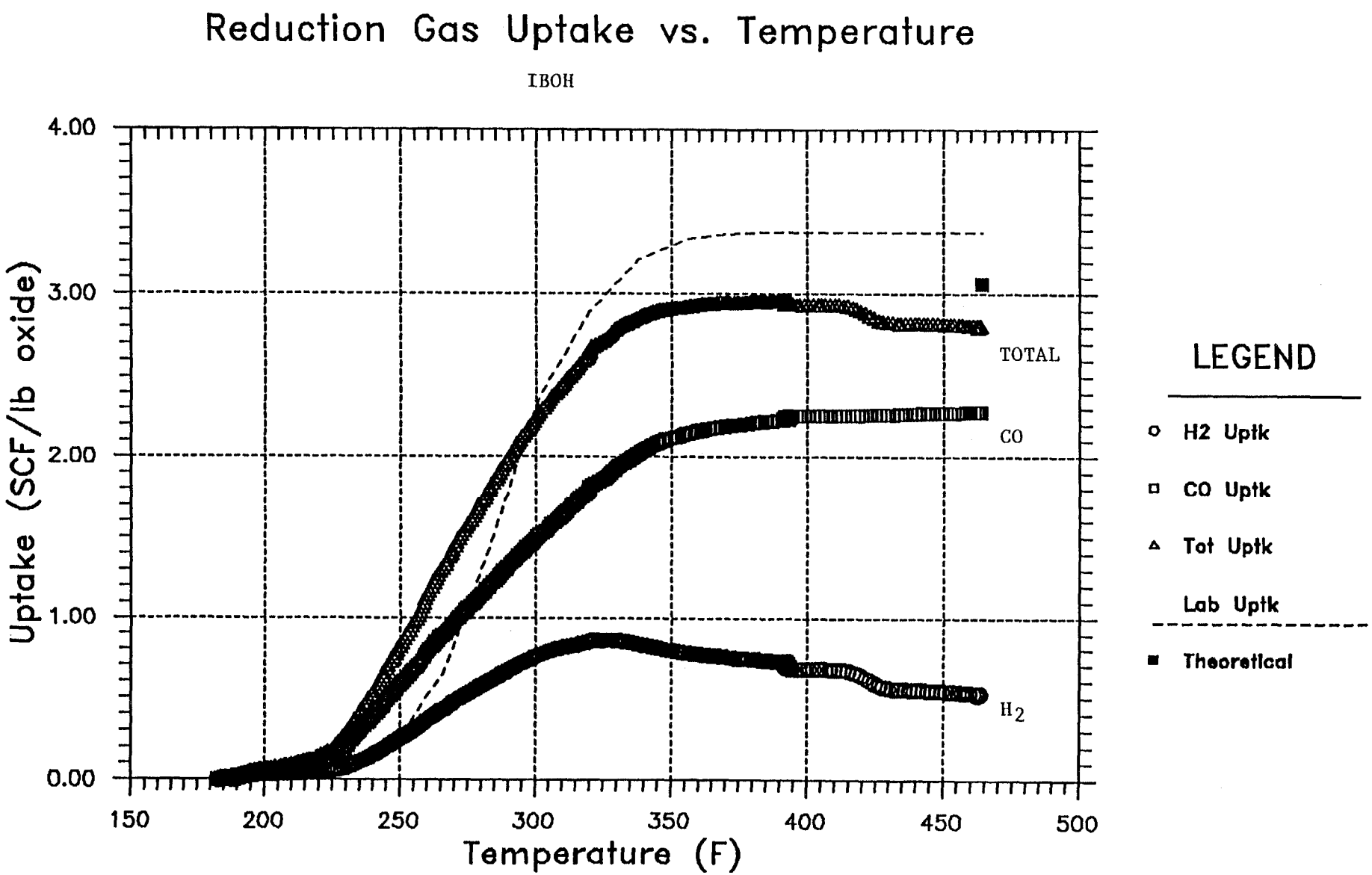
The traditional twelve-hour hold period at 392°F was shortened to four hours to duplicate the laboratory procedure, and the final ramp to 464°F proceeded at 18°F/hr. Both changes were also predicated on the comparison of synthesis gas uptake to the autoclave results, as shown in Figure 11. The final (H_2 + CO) uptake was 2.96 SCF/(lb of catalyst, as oxide). Theoretical maximum

FIGURE 10



3/28/94

FIGURE 11


Reduction Gas Uptake vs. Temperature
IBOH
Uptake (SCF/lb oxide)
4.00
3.00
2.00
1.00
0.00
150
200
250
300
350
400
450
500
Temperature (F)
TOTAL
CO
H2
LEGEND
H2 Uptk
CO Uptk
Tot Uptk
Lab Uptk
Theoretical

uptake for the catalyst is 3.07 SCF/lb oxide, and autoclave results exceeded this value, ranging from 3.38 - 3.59 SCF/lb oxide (on a LaPorte standard basis). This slight shortfall in uptake is typical for other LaPorte vs. autoclave experiments. Reduction concluded at 19:30 on March 27, and a Shell-gas blend (nominally 30% H_2, 66% CO, 1% N_2, 3% CO_2) was introduced a few hours later to begin isobutanol synthesis.

LPIBOH Operations

The run plan for the LPIBOH campaign is summarized in Table 1. Once finalized, this plan was duplicated in the laboratory prior to the run to provide performance and catalyst deactivation expectations. During the actual LaPorte trial, each condition was tested exactly according to plan, except for the durations. Note that 10.1 through 10.3 delineate a space velocity scan at the typical LPMEOH pressure; 10.3 through 10.5 define a pressure scan at the high space velocity condition; and 10.6 and 10.7 fill in some of the intermediate points. Conditions 10.8 and 10.9 complement 10.7 by using alcohol injection to simulate lower alcohol (methanol, ethanol, propanol) recycle to the reactor. Finally, point 10.10 returns to baseline (10.1) to quantify catalyst deactivation. Apart from operation at pressures up to 1750 psig, the campaign also broke new ground with successful demonstration of extended operation at an inlet superficial velocity of 1.0 ft/sec during run 10.3.

After catalyst reduction concluded at 19:30 on March 27, 1994, the "back-end" CO_2-removal system was cooled down, the GCs were recalibrated for isobutanol analysis, and a Shell-gas blend was introduced in once-through mode at 22:05. Plant pressure slowly increased to 750 psig at about 450°F. Once at pressure, recycle flow commenced, and the reactor temperature was increased slowly to the target of 572°F. Methanol first appeared in the reactor effluent at 22:53, and recycle synthesis gas was introduced to the CO_2-removal system at 23:00.

Typical startup problems persisted for the balance of the first two days of operation. Initially, efforts were directed toward troubleshooting instrumentation in the new CO_2-removal section and performing final calibration of the GCs. However. by the time the plant lined out the next morning, a pin-hole leak was discovered in a reactor thermocouple weld, necessitating a complete shutdown for repair. Shortly after the restart, communication problems developed between the GC computer and the DEC data collection system. Then the seals failed on the 10.80 methanol circulation pump, requiring isolation of the "back-end" CO_2-removal area. Finally, the 10.80 pump was returned to service and heatup commenced at 16:30 on March 29. The plant lined out by 21:00 and ran very smoothly at target conditions throughout the AF-R10.1 data period, which closed at 13:00 on March 30.

Initial operation at the baseline conditions showed performance slightly superior to the autoclave, as measured by the production of isobutanol and other higher alcohols (in g/kg oxide-hr) shown in Table 2. Again, mass balances for each run condition appear in Appendix C; the equivalent laboratory data sheets appear in Appendix D. CO conversion averaged 12.7% per pass for the average space velocity of 5044 sL/kg-hr at 750 psig and 573°F, compared to 11.3% in the laboratory. The isobutanol concentration averaged 0.17 mole% in the reactor effluent and 9.0 wt% in the liquid product. NDG measurements showed an average gas holdup of 42.4% and a

TABLE 1
LAPORTE AFDU LPMEOH/LPIBOH RUN PLAN - MARCH 1994

Point	No. Days	Gas Type	Pressure psia	Space Velocity sL/kg-hr	Reactor Feed lbmol/hr	Inlet Sup. Velocity ft/sec	Slurry Wt% oxide
LPMEOH RUN							
AF-R9.1	2	Texaco	765	6700	373	0.84	42.5
AF-R9.2	3	Kingsport	750	4000	223	0.51	40.0
IBOH RUN							
AF-R10.1	2	Shell- Base Case	765	5000	247	0.61	40.3
AF-R10.2	1	Shell	765	3000	148	0.37	39.1
AF-R10.3	2	Shell	765	8200	405	1.00	41.5
AF-R10.4	2	Shell	1315	8200	405	0.58	42.0
AF-R10.5	2	Shell	1750	8200	405	0.44	42.3
AF-R10.6	1	Shell	1750	3000	148	0.16	39.8
AF-R10.7	1	Shell	1315	5000	247	0.36	40.7
AF-R10.8	1	Shell w/ full ROH recycle	1315	5000	247	0.36	40.7
AF-R10.9	1	Shell w/ partial ROH recycle	1315	5000	247	0.36	40.7
AF-R10.10	Optional	Shell- Base Case	765	5000	247	0.61	40.3
	18						

Point 8 is a case with total lower alcohol recycle
Point 9 is a co-product methanol/iboh point where not all of the methanol is recycled.

Table 2

Summary of AFDU vs. Autoclave Results

Run	P (psia)	Space Vel. (sL/kg-hr)	IBOH Productivity Lab (g/kg-hr)	IBOH Productivity AFDU (g/kg-hr)	C2-C6 OH Productivity Lab (g/kg-hr)	C2-C6 OH Productivity AFDU (g/kg-hr)
10.1	765	5000	22.8	25.8	65.1	70.0
10.2	765	3000	19.3	18.7	49.1	45.5
10.3	765	8200	20.6	16.6	69.0	50.5
10.4	1315	8200	24.8	20.8	92.8	86.9
10.5	1750	8200	26.2	23.2	103.0	95.9
10.6	1750	3000	27.7	23.8	71.4	60.6
10.7	1315	5000	23.1	19.2	75.3	64.2

catalyst concentration of 41.1 wt%, compared to predicted values of 40.2% and 40.2 wt%, respectively.

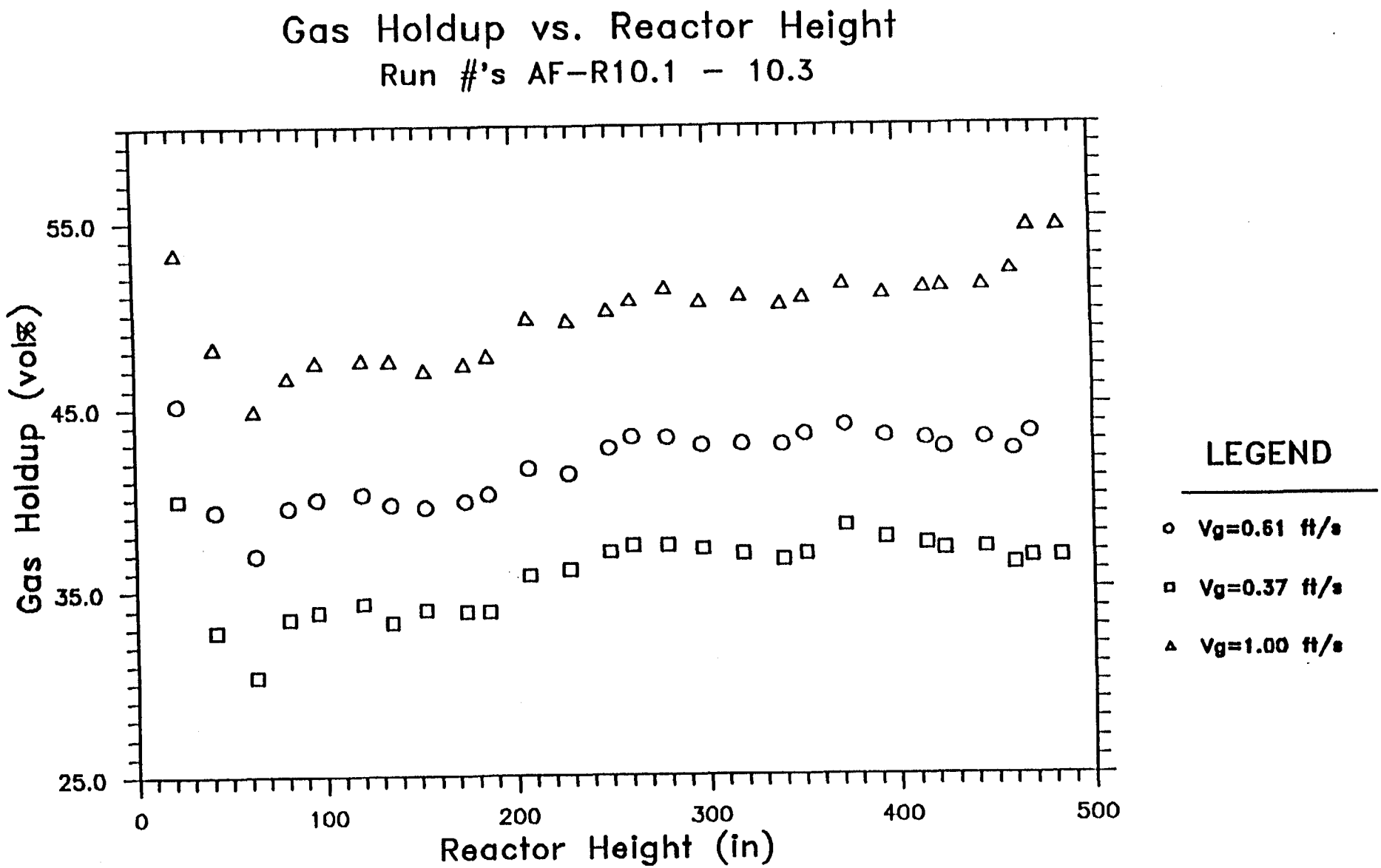
Notably, liquid product analysis was somewhat problematic because of measurable concentrations of numerous higher alcohol isomers. The chromatogram areas totalized well, but identified components typically composed only 90-97 wt% of each liquid product sample. By recommendation of the research staff, the balance was assumed to average C_6-alcohols. Furthermore, the traditional oil analysis by evaporation produced results of roughly 0.5-1.5 wt%, compared to 0.10-0.25 wt% for the LPMEOH cases, despite the observation that the samples were visually free of any oil. Again, the balance of the measured values is presumably other, less-volatile higher alcohols. This hypothesis is supported by the observation that, throughout the run, the samples with greater concentrations of higher alcohols (and lesser methanol concentrations) also exhibited greater concentrations of "oil" by evaporative analysis.

Data period AF-R10.2 ran from midnight to 17:00 on March 31. CO conversion averaged 13.7% per pass for the average space velocity of 3030 sL/kg-hr at 753 psig and 572°F, compared to 13.4% in the laboratory. However, the production of isobutanol and other higher alcohols fell slightly short of autoclave performance, as shown in Table 2. The isobutanol concentration averaged 0.21 mole% in the reactor effluent and 12.8 wt% in the liquid product, but these values declined slightly over the course of the data period. NDG measurements indicated 36.3% average gas holdup and 38.4 wt% slurry concentration, compared to predicted values of 36.4% and 39.1 wt%, respectively.

Condition AF-R10.3, which ran between 04:00 and 16:00 on April 1, provided the first clear evidence that catalyst productivity was decreasing faster in the AFDU than it did when the run plan was duplicated in the laboratory. CO conversion averaged 7.6% per pass for the average space velocity of 8242 sL/kg-hr at 751 psig and 572°F. The laboratory result was 8.5% with a space velocity of 8500 sL/kg-hr. Table 2 also shows that the production of isobutanol and other higher alcohols fell more significantly short of autoclave performance, despite the 3% difference in space velocity. Furthermore, this case upheld another disturbing trend: throughout the course of each data period, liquid samples taken from the product collection area of the plant showed increasing levels of methanol and decreasing levels of isobutanol. The isobutanol concentration averaged 0.07 mole% in the reactor effluent and 5.3 wt% in the liquid product, but this value declined 10% (relative) over a six and a half hour span in the middle of the data period. Thus, not only was the catalyst showing signs of deactivation, but it was also occurring at a faster rate than in the autoclave.

Operationally, the plant achieved long-term, stable hydrodynamic performance with a record inlet gas superficial velocity of 1.0 ft/sec. NDG measurements remained steady throughout the run and indicated 50.1% average gas holdup and 44.8 wt% slurry concentration. Predicted values of 44.3% and 41.5 wt%, respectively, further illustrate the impact of high gas velocity on the accuracy of the NDG. In addition, a reactor slump test at the end of the period indicated 47.5% gas holdup. Three-phase gas holdup data are plotted vs. reactor height for the first three run conditions in Figure 12. As expected, the holdup increases with increasing linear velocity, and the plots show the same characteristic shape as the two-phase data reported earlier.

FIGURE 12


Gas Holdup vs. Reactor Height
Run #'s AF-R10.1 - 10.3
Gas Holdup (vol%)
55.0
45.0
35.0
25.0
0
100
200
300
400
500
Reactor Height (in)
LEGEND
Vg=0.61 ft/s
Vg=0.37 ft/s
Vg=1.00 ft/s

The deactivation trend continued during conditions AF-R10.4 and 10.5, as seen in graphical form in Figure 13. To minimize deactivation, the run plan was accelerated as much as possible, especially during these harsh high pressure, high flow conditions. As a result, by the end of data period 10.5, the actual AFDU run plan was about sixty hours ahead of the autoclave schedule. Thus, in Figure 13, the time plotted along the x-axis corresponds to the AFDU on-stream time, not the laboratory hours on-stream.

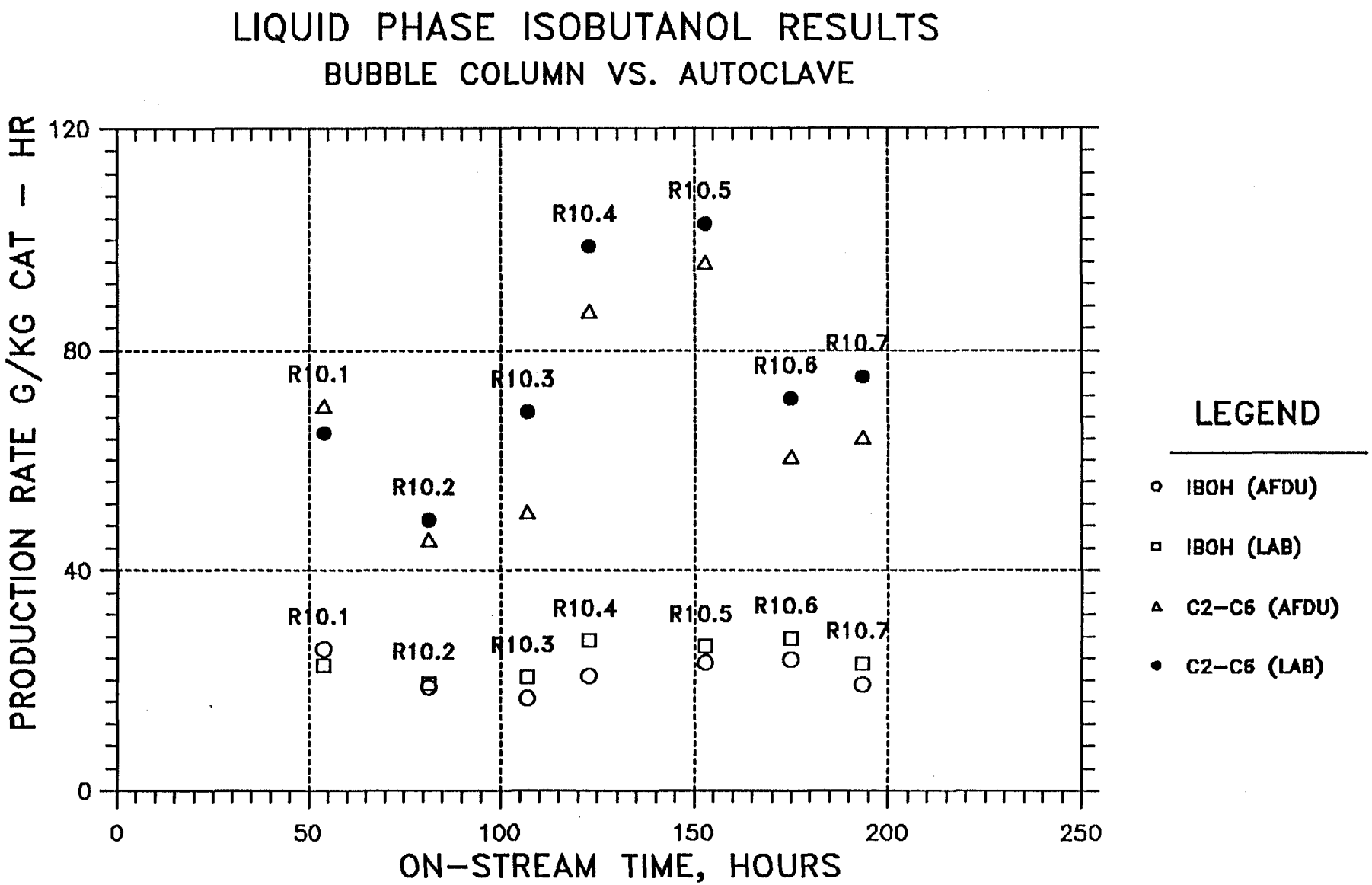
The mass balance period for AF-R10.4 ran from 21:00 on April 1 to 07:00 on April 2. The run ended prematurely when the refrigerant CO_2 supply ran out, forcing a shutdown of the "back-end" scrubbing unit. However, the plant ran very smoothly during the ten-hour data collection period. CO conversion averaged 12.7% per pass for the average space velocity of 8227 sL/kg-hr at 1300 psig and 572°F, compared to 13.3% in the laboratory. Again, productivities of isobutanol and other higher alcohols were significantly lower than those observed in the autoclave, as shown in Table 2. For example, the isobutanol production rate was 20.8 g/kg-hr, compared to 24.8-27.3 g/kg-hr observed in the laboratory at similar conditions. The autoclave results reflect about a 10% decrease in productivity after 24 hours operation at this condition. The AFDU's shorter time at this harsh condition, coupled with the overall shorter on-stream time, contributes to the observation that the AFDU vs. autoclave productivity gap seemed to close somewhat during this data period and the next.

The isobutanol concentration averaged 0.09 mole% in the reactor effluent and 2.6 wt% in the liquid product, but again this value declined slightly over the course of the data period. NDG measurements showed an average gas holdup of 44.0% and a catalyst concentration of 41.9 wt%, compared to predicted values of 46.0% and 42.0 wt%, respectively.

An alcohol spill and fire occurred at 15:20 on Saturday, April 2, when a product trailer was overfilled during a day tank transfer. About 25 gallons of alcohol spilled through the supplemental flare, caught fire, and were extinguished with a nearby fire monitor without damage or injury. Recent installation of this particular fire monitor was an action item from a Hazard Review that identified an alcohol spill in the trailer area as a fire hazard. Another 300 gallons of alcohol spilled near the trailer. The area was covered with an alcohol foam and subsequently cleaned up by EmTech Environmental Services.

Data period AF-R10.5 ran from 02:00 to 14:00 on April 3. CO conversion averaged 15.7% per pass for the average space velocity of 8281 sL/kg-hr at 1735 psig and 573°F, compared to 16.3% in the laboratory. The isobutanol production rate was 23.2 g/kg-hr, compared to 26.2 g/kg-hr in the autoclave, but the laboratory data were obtained at 212 hours on-stream, compared to 153 hours for the AFDU. The isobutanol concentration averaged 0.10 mole% in the reactor effluent and 2.2 wt% in the liquid product, but these values continued to drop through the course of the mass balance period, despite steady operating conditions. NDG measurements indicated 41.8% average gas holdup and 40.9 wt% slurry concentration, compared to predicted values of 46.9% and 42.2 wt%, respectively. These results were the first to show poor agreement between predictions and the NDG at relatively low superficial velocities.

FIGURE 13


LIQUID PHASE ISOBUTANOL RESULTS
BUBBLE COLUMN VS. AUTOCLAVE
PRODUCTION RATE G/KG CAT - HR
120
80
40
0
0
50
100
150
200
250
ON-STREAM TIME, HOURS
R10.1
R10.2
R10.3
R10.4
R10.5
R10.6
R10.7
LEGEND
IBOH (AFDU)
IBOH (LAB)
C2-C6 (AFDU)
C2-C6 (LAB)

Condition AF-R10.6, which ran between midnight and noon on April 4, represented the high-pressure, low space velocity case with the minimum superficial velocity (0.16 ft/sec). CO conversion peaked for the run at 20.9% per pass for the average space velocity of 3026 sL/kg-hr at 1735 psig and 572°F. The laboratory result was 21.8%. Again, Table 2 and Figure 13 indicate a widening productivity gap between AFDU and autoclave performance. For example, the isobutanol production rate was 23.8 g/kg-hr at 175 hours on-stream, compared to 27.7 g/kg-hr observed in the laboratory at similar conditions after 239 hours on-stream. The isobutanol concentration averaged 0.29 mole% in the reactor effluent and 7.2 wt% in the liquid product. NDG measurements remained steady throughout the run and showed an average gas holdup of 27.9% and a catalyst concentration of 35.5 wt%, compared to predicted values of 38.7% and 39.8 wt%, respectively. These data, coupled with the AF-R10.5 results and the expectation that the NDG is accurate at lower gas velocities, indicate that the correlation for predicting gas holdup is probably inadequate at high pressures.

Condition AF-R10.7, the precursor to the alcohol injection cases at intermediate pressure and space velocity, ran from 17:00 on April 4 to 08:00 on April 5. CO conversion averaged 14.2% per pass for the average space velocity of 5070 sL/kg-hr at 1300 psig and 572°F. The laboratory result was 14.6% with a space velocity of 5225 sL/kg-hr. As shown in Table 2, the isobutanol production rate was 19.2 g/kg-hr at 193 hours on-stream, compared to 23.1 g/kg-hr observed in the laboratory at 3% higher space velocity and after 261 hours on-stream. These results continue to show that the catalyst deactivated at a faster rate in the AFDU than in the parallel autoclave run. The isobutanol concentration averaged 0.13 mole% in the reactor effluent and 4.1 wt% in the liquid product. Again, however, the concentration of isobutanol in the liquid product declined 15% (relative) over a five-hour span in the middle of the data period, despite steady operating conditions. At the same time, the methanol concentration rose 3%. NDG measurements indicated 37.5% average gas holdup and 38.9 wt% slurry concentration, compared to predicted values of 41.7% and 40.7 wt%, respectively. Thus, the holdup prediction appears more accurate at the lower pressure, but again, less accurate than at 750 psig where the correlation was developed.

Table 3 summarizes the AFDU performance through all of the cases prior to alcohol injection. Remember that points AF-R10.3 through 10.5 show the pressure effect at the high space velocity condition. As the pressure increased, conversion and productivity increased, but selectivity decreased, as expected. Similarly, runs AFR-10.1 through 10.3 show the space velocity effect at 750 psig, although not in increasing order. As space velocity increased, conversion and selectivity decreased, presumably because of the reduction in residence time. However, productivity was greatest for the intermediate (baseline) condition. Each of these trends was also observed in the laboratory runs.

Run AF-R10.8 duplicates the conditions of AF-R10.7, while also using alcohol injection into the synthesis gas feed to demonstrate the effect of total lower alcohol recycle on higher alcohol synthesis. The injection composition was nominally 84% methanol, 5% ethanol, and 11% 1-propanol at a rate of about 1.8 gpm. During AF-R10.9, the composition was altered to 73% methanol, 9% ethanol, and 18% 1-propanol at a rate of about 0.4 gpm.

Table 3

Summary of AFDU Performance

Run	P (psia)	Space Vel. (sL/kg-hr)	CO Conversion (%)	IBOH Concentration (wt%)	IBOH Productivity (g/kg-hr)	C2-C6 OH Productivity (g/kg-hr)
10.1	765	5000	12.5	9.0	25.8	70.0
10.2	765	3000	13.7	12.8	18.7	45.5
10.3	765	8200	7.6	5.3	16.6	50.5
10.4	1315	8200	12.7	2.6	20.8	86.9
10.5	1750	8200	15.7	2.2	23.2	95.9
10.6	1750	3000	20.9	7.2	23.8	60.6
10.7	1315	5000	14.2	4.1	19.2	64.2

The mass balance period for AF-R10.8 ran from 03:00 to 20:00 on April 6. CO conversion averaged 6.8% per pass for the average space velocity of 5494 sL/kg-hr at 1300 psig and 572°F, compared to 8.3% in the laboratory. As seen in Table 4, the isobutanol and C_2-C_6 alcohol production rates increased nearly threefold over AF-R10.7, which was identical in all other respects. This result supports the chain growth mechanism for production of higher alcohols from lower alcohols. The isobutanol concentration averaged 0.31 mole% in the reactor effluent and 4.8 wt% in the liquid product. NDG measurements indicated 40.6% average gas holdup and 40.1 wt% slurry concentration, compared to predicted values of 43.2% and 41.1 wt%, respectively.

Condition AF-R10.9, using alcohol injection to simulate partial lower-alcohol recycle to the reactor, ran from 16:00 on April 7 to 04:00 on April 8. CO conversion averaged 11.2% per pass for the average space velocity of 5154 sL/kg-hr at 1300 psig and 572°F. The autoclave result was 12.5%. However, throughout the course of this case, catalyst activity began to decline dramatically. The isobutanol concentration in the liquid product dropped from 3.7 wt%, shortly after the start of the data period, to 2.0 wt% nine hours later. Another three hours after that, at the close of the period, the concentration had fallen to 1.6 wt%. NDG measurements remained steady throughout the run and showed an average gas holdup of 37.9% and a catalyst concentration of 39.1 wt%, compared to predicted values of 44.4% and 42.0 wt%, respectively.

Since the AF-R10.9 mass balance indicated that the alcohol injection rate was significantly below target (0.4 gpm vs. 0.7 gpm), the pumping rate was increased with the intent of establishing a new condition (AF-R10.9a) until the alcohol feedstock was exhausted. However, during this period it became increasingly apparent that the catalyst was deactivating very rapidly, and specifically because of this alcohol injection case. Isobutanol and methanol concentrations in the reactor effluent are plotted as a function of on-stream time in Figure 14. The isobutanol concentration shows a rapid decline after 260 hours on-stream, while the methanol concentration begins dropping at about 275 hours. Utility oil inlet and outlet temperatures, as well as the average reactor temperature, are plotted in Figure 15. Apparently, after 237 hours on-stream, the heat of reaction suddenly increased. This coincides with switching the alcohol feed to a different composition at the beginning of AF-R10.9, and is expected because the lower alcohol injection rate results in higher synthesis gas conversion. However, the heat of reaction began declining dramatically after 250 hours on-stream, confirming that the catastrophic deactivation began in the early stages of AF-R10.9. An additional sample of the alcohol feedstock was collected to help determine the reason for the deactivation.

The plant operating conditions were quickly returned to the baseline case, as planned, to check on catalyst activity. However, by the time the reactor had lined out at 19:00 on March 8, the isobutanol had disappeared completely from the effluent, and methanol continued to decline below 1%. As a result, the opportunity to quantify the degree of catalyst deactivation for the duration of the campaign was lost, and was replaced by a search for catalyst poisons. The mass balance from then until 08:00 on March 9 is included with the others in Appendix C. Several liquid samples were collected, but the production rate was too low to flush out the product collection section from the previous run condition. Only after intentionally pushing most of the liquid out of the system was a potentially representative sample collected. NDG scans during the

Table 4

Summary of AFDU Performance

Run	P (psia)	Space Vel. (sL/kg-hr)	CO Conversion (%)	IBOH Concentration (wt%)	IBOH Productivity (g/kg-hr)	C2-C6 OH Productivity (g/kg-hr)
10.1	765	5000	12.5	9.0	25.8	70.0
10.2	765	3000	13.7	12.8	18.7	45.5
10.3	765	8200	7.6	5.3	16.6	50.5
10.4	1315	8200	12.7	2.6	20.8	86.9
10.5	1750	8200	15.7	2.2	23.2	95.9
10.6	1750	3000	20.9	7.2	23.8	60.6
10.7	1315	5000	14.2	4.1	19.2	64.2
10.8 (Al-1)	1315	5000	6.8	4.8	55.9	190.8
10.9 (Al-2)	1315	5000	11.2	3.7	18.4	79.4

FIGURE 14

REACTOR EFFLUENT CONCENTRATIONS DURING LPIBOH

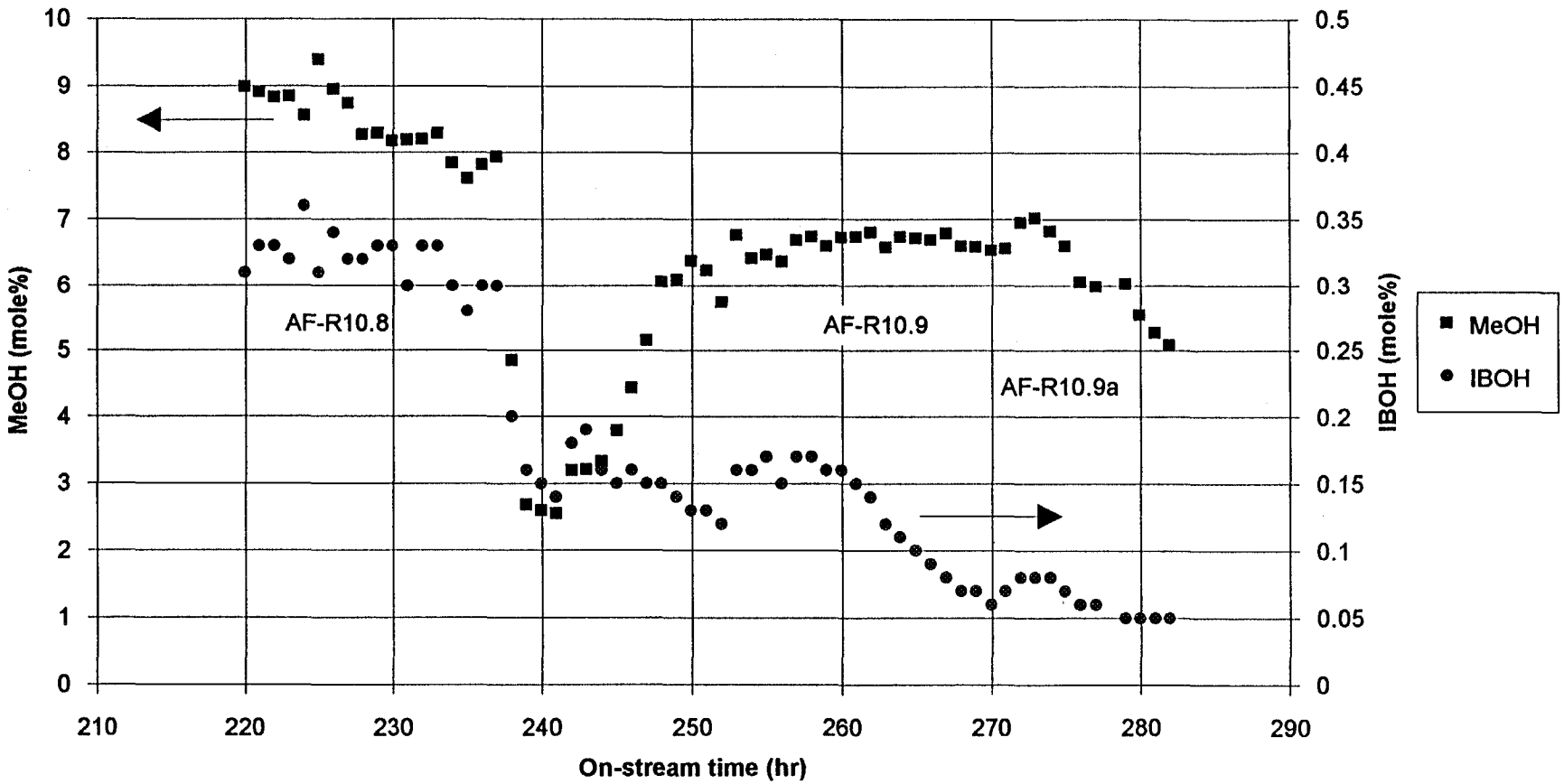
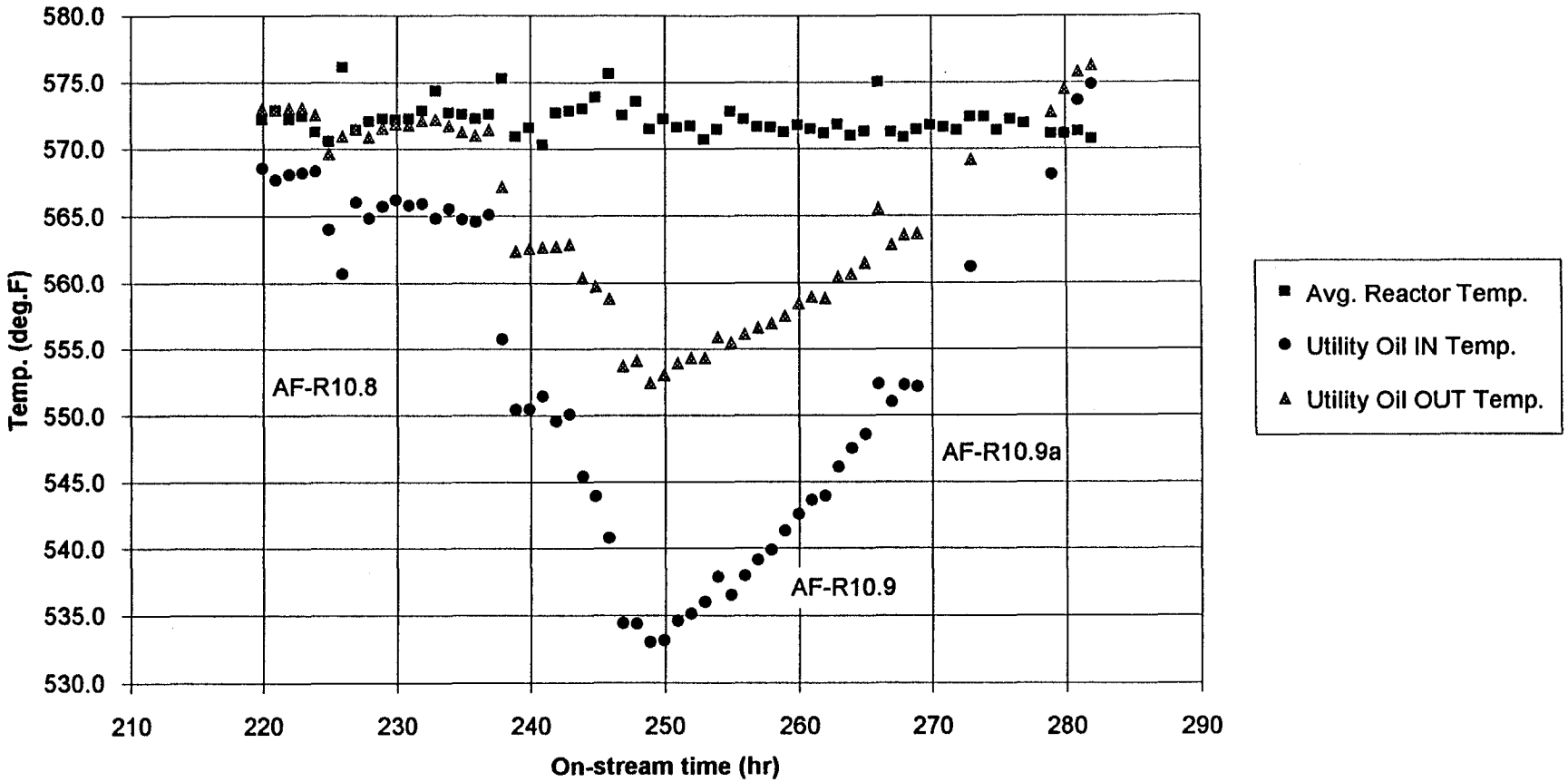


FIGURE 15

TEMPERATURES DURING LPIBOH



period were equivalent to those taken during AF-R10.1 (42.1% gas holdup and 40.8 wt% catalyst concentration).

Post-Run Inspection and Investigation

The post-run carbonyl survey at baseline and high-pressure conditions showed little difference from results prior to the start of the LPMEOH operations. Notably, the 02.63 steam preheater, used to superheat the alcohol feed to the reactor, did not seem to contribute inordinately to carbonyl generation. This unit, with its high skin temperatures, proved to be a significant source of carbonyls during the burnout period and was not in service between then and the alcohol injection cases.

Following the carbonyl tests, cool down commenced at 18:20 on March 9. At 23:00 the synthesis gas feed was withdrawn and replaced with N_2 to purge. Prior to inspection, the reactor was flushed with oil at 275-300°F for 18 hours. During the reactor inspection, less than 5 lbs of catalyst were found in the bottom head, a location where packed, dried catalyst usually collected in the old reactor. The shell and internal heat exchanger were free of catalyst in areas contacted by flush oil, while the freeboard space and top head were still covered with a thin layer of catalyst.

The sample taken from the alcohol injection trailer after AF-R10.9 appeared pale yellow, unlike the samples taken prior to the run, which were clear. Later analysis of the anomalous sample revealed a significant quantity of 1,1,1-trichloroethane, a formerly common cleaning solvent no longer present on the AFDU site. The yellow color may be explained by the presence of trace dissolved iron which was also discovered.

A sample of the spent, end-of-run catalyst was washed to remove the mineral oil and then, along with a sample of the fresh catalyst, was analyzed for elemental composition and crystallite size by XRD. Elemental analysis revealed no iron uptake on the spent catalyst, a good sign that iron carbonyl production was low in the new plant equipment and that the iron present in the injected alcohol did not deposit in a measurable quantity on the catalyst. The only known catalyst poison found was chloride, present at 0.79 wt%, while the fresh catalyst contained no chloride. This level of chloride contamination would certainly cause measurable deactivation of the catalyst.

Interestingly, the Cu crystallite size (via XRD) was very large at 337 ± 13 A. By comparison, the Cu crystallite size determined after the laboratory simulation of the entire LPIBOH run plan was 250 ± 7 A. Though no direct evidence exists, we hypothesize that the formation of copper chloride (CuCl or $CuCl_2$) explains the large Cu crystallite size observed. Since both CuCl and $CuCl_2$ have a much lower melting point than Cu metal, their presence under synthesis conditions may cause increased mobility of copper, resulting in faster sintering.

The evidence indicates that the inadvertent presence of 1,1,1-trichloroethane in the injected alcohol reactant caused the rapid loss in catalytic activity observed during AF-R10.9. The 1,1,1-trichloroethane decomposed on the catalyst surface to produce chloride, which is a known poison for the $Cu/ZnO/Al_2O_3$ substrate. Apparently the alcohol feedstock trailer was cleaned with the solvent and improperly rinsed, leaving trace quantities of the 1,1,1-trichloroethane in the

subsequent alcohol blend. GC analysis of a sample prior to injection focused only on bulk compositional analysis and failed to detect any trace contamination.

The inadvertent poisoning of the catalyst during AF-R10.9 significantly hindered the investigation of the faster rate of catalyst deactivation observed throughout the campaign at the AFDU. Typically, spent (end-of-run) catalyst analysis is used to provide information on catalyst deactivation mechanisms experienced during the run. For example, analysis of Cu crystallite size by XRD could provide clues about the rate of sintering, as it did in this case. Unfortunately, however, it is impossible to distinguish results of this type between pre-10.9 and post-10.9 trends. The cause for the faster deactivation of the catalyst in the early part of the campaign remains unknown.

Conclusions and Future Plans

Apart from the catalyst deactivation, the thirteen-day LPIBOH campaign successfully demonstrated mixed alcohol synthesis in a slurry bubble-column reactor, as well as all of the new equipment installed for the trial. Although the full capabilities of the new system will not be tested until future runs, all of the design objectives for the modifications were met with respect to the isobutanol run, and show every indication of being applicable to other chemistries. The catalyst and reactor systems were tested at a wide range of pressures (750-1735 psig) and space velocities (3000-8200 sL/kg-hr), representing numerous first-of-a-kind run conditions for the AFDU. Inlet gas superficial velocities spanned an impressive 0.16 to 1.0 ft/sec. Stable reactor performance for a full twelve-hour data period at 1.0 ft/sec represented a significant milestone event for the liquid phase technology program. Additionally, the reactor appeared to exhibit stable hydrodynamic performance during a brief test period at 1.17 ft/sec.

Although the catalyst demonstrated in this run was state-of-the-art for isobutanol synthesis, its performance was still short of economic targets for production of MTBE. Work involving collaboration between Air Products and academic researchers will continue on the development of new, improved catalysts.

Acknowledgments

The development work described here was supported in part under a contract from the United States Department of Energy (No. DE-AC22-91PC90018). The author would like to thank Ed Heydorn, Dave Hanauer, and the Air Products operators for efficient operation of the pilot plant. Dean Chin-Fatt and Rob Staskowski provided analytical support, and Elizabeth Schaub, Bharat Bhatt, John Repasky, Kerri Freidl, and Chris Chen provided 24-hour process engineering coverage throughout the operation. Rich Underwood provided laboratory data and other valuable technical assistance during the demonstration.

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APPENDIX A

TEST AUTHORIZATIONS

Memorandum

AIR PRODUCTS

To: Distribution Dept./Loc.:

From: E. S. Schaub/E. C. Heydorn Dept./Ext.: PSG Process/LaPorte AFDU

Date: 3 March 1994

Subject: Test Authorizations # 37,38, 39, & 40 for the LaPorte AFDU Methanol and Isobutanol Runs

Distribution:

W. C. Allen
D. P. Bernhard
B. L. Bhatt/K. G. Freidl/C. Chen
D. M. Brown/W. R. Brown
D. P. Drown
V. E. Stein/J. M. Repasky
R. P. Underwood/B. A. Toseland

Attached for your information are the 4 Test Authorizations for the upcoming Isobutanol/Mixed Alcohols Demonstration and the LPMEOH run which will be used as a shakedown period prior to the Isobutanol run.

If you have any questions, please contact either of us at (713) 479-5485.

Elizabeth Schaub EHeydorn

E. S. Schaub/E. C. Heydorn

TEST AUTHORIZATION # 37

LaPorte Alternative Fuels Development Unit (AFDU)

Date : 03/02/94
By: ESS

RUN NUMBER: AF-A5
APPROX. START DATE: 14 March, 1994

TITLE: IN-SITU METHANOL CATALYST ACTIVATION PRIOR TO SPRING 94 RUN USING DILUTE CO-RICH REDUCTION GAS

OBJECTIVE:
To activate the Liquid-Phase Methanol (LPMEOH) synthesis catalyst.

SUMMARY:
Approximately 1250 lbs of BASF S3-86 oxide is to be slurried with Drakeol-10 oil, transferred to the 27.20 reactor and activated with dilute CO-Rich syngas (3.5% in nitrogen). Approximate run time is 2 days.

TEST DETAILS: See pages 2 to 4 for details.

ANALYTICAL COMMENTS: See page 4.

SAFETY IMPLICATIONS:
Operators should wear protective gear while loading catalyst to protect them from the dust and hot vapor which may be released from the loading nozzle. Protective gear including face shield should be worn during slurry sampling.

This operation will require the venting of unreacted hydrogen and CO. During a previous activation (performed under TEST AUTHORIZATION #29) the off-gas was blended with methane and burned in the flare. Previous calculations (for TA #23) indicated that in the event a combustible mixture could not be maintained, there would be no danger to personnel from venting. The reduction gas flow rates to be used in this run are less than those used in TA #23.

ENVIRONMENTAL IMPLICATIONS:
Minimal, a flame will be maintained at the flare. At 98% destruction efficiency, the CO emission rate would be 0.67 lb/hr.

SPECIAL REMARKS:
Hydrogen and CO concentrations in and out of the reactor must be monitored closely during the reduction. Reactor temperature must be closely monitored and controlled per the attached TEST DETAILS. The utility oil inlet temperature (TI 1244) to the 27.20 internal heat exchanger must not exceed a 200°F difference from the utility oil outlet temperature (TI-1246) or the reactor slurry temperature. These two temperature differentials are measured directly by TDI-1252 & TDI-1237. When adjusting flows or pressure, care should be taken to minimize catalyst carryover (caused by high gas velocity).

AUTHORIZATIONS:

E. C. Heydorn, Plant Mgr

E. S. Schaub, Process Engr

TEST DETAILS:

1. This reduction procedure follows previous methanol catalyst reductions from the LPIII ER-6 reduction (TEST AUTHORIZATION #23), 1991 DME run (#25), and the 1992 LPSHIFT run (#29).

2. Charge the 28.30 prep tank with 1875 lb of oil (265 gallons of Drakeol-10 at 80°F). The oil should be transferred to drums and weighed using the scale for accurate measurement. As an approximation, meter the oil with FQI-334 using a meter correction factor of actual = 1.027 * meter (meter should read 258.2 gal). If the temperature differs from 80°F a corrected oil volume should be used. Heat this oil to 150-200°F.

3. Fill the 27.14 intermediate V/L separator to 25 nuts on LG-358 with approximately 100 gallons of Drakeol-10 oil from storage. Note the FQI-334 readings before and after the addition.

4. When the prep tank oil is at 150-200°F, add 1250 lb of methanol catalyst (BASF S3-86- 2 full drums of Lot 851-1642 and the balance (3+) drums of Lot 553-5072). Add the catalyst very slowly to make a 40 wt% oxide slurry. Keep the slurry well stirred to prevent agglomeration of the catalyst.

5. Heat the slurry to 200°F and continue agitation, under nitrogen, for at least 2 hours to ensure good mixing.

6. When the catalyst and oil have been completely mixed, withdraw a sample of slurry.

7. Establish gas flow through the reactor using nitrogen through V-2627 to prevent slurry back-flow into the distributor. Vent the gas through PV-1261.

8. Pressure transfer the slurry to the reactor and verify operation by noting level with the nuclear density gauge (NDG- estimated level: 25 to 28 ft.)

9. Flush out the prep tank with 283 lb of oil (40 gallons of Drakeol-10 at 80°F). Measure the oil as in step 2 (meter should read approximately 38.9 gal). Pressure transfer the flush oil to the reactor and verify level with the NDG (LI-1242).

10. Close V-645 to prevent utility oil flow back to the prep tank and establish full utility oil flow through the 27.20 internal heat exchanger.

11. Pressurize the reactor loop to 100 psig.

12. Begin heating the slurry to 200°F, following TAVR on the DEC console. Check that the slurry temperatures are in reasonable agreement. Verify that the slurry is well mixed by performing a NDG scan.

13. Establish CO-Rich reduction gas flow at 25,910 SCFH (on FI-126) and vent the flow through PV-170. Establish the following composition:

	Composition	Est. Flows (SCFH)
H2	1.2%	311
CO	1.8	466
CO2	0.5	130
N2	96.5	25,003
	100.0	25,910

MW = 27.8, SCF evaluated at 70°F, 14.7 psia
Target space velocity = 1200 sL/h-kg; Target starting inlet superficial velocity = 0.73 ft/sec

14. When the reactor temperature reaches 200°F, bring reduction gas to the reactor slowly and close the nitrogen purge (V-2627). Establish a final flow to the reactor of 25,910 SCFH. Maintain flow and reducing gas composition as specified in step 13. The temperature-programmed activation consists of the following steps:

    - Heat the slurry at a target rate of 15°F/hr (no more than 18°F/hr, 10°C) until the slurry temperature reaches 392°F (200°C).
    - Hold the slurry temperature at 392°F for 12 hours.
    - Heat the slurry at 15°F/hr until the slurry temperature reaches 464°F (240°C).
    - Hold the slurry temperature at 464°F for 1 hour (or longer if syngas uptake is still apparent).

    H2 and CO concentrations are to be measured continuously for the feed and effluent streams. As long as the cumulative H2 plus CO consumption at a given temperature is equal to or greater than the autoclave reduction data then the activation is proceeding well. Figure 1 shows the consumption profile vs temperature from the labs. If the cumulative consumption curve falls below the autoclave curve, consult the process or research engineer to reduce the heatup rate.

    If the H2+CO concentration in the effluent falls below 0.1 mole %, increase the inlet H2+CO concentration per the instructions of the process or research engineer. The objective here is to prevent reduction gas starvation.

    During the 392°F hold period, it may become necessary to maintain this temperature beyond 12 hours until the difference between inlet and outlet H2+CO concentration falls below 0.05 mole %.

15. The slurry level should be maintained between 90 and 95% of NDG range (approximately 40 ft.) by using LIC 1242 to control the makeup oil rate. Note that as the reactor is heated to 464 F, the slurry will expand. At the same time, some of the oil will be lost in the reactor effluent. If authorized by the process engineer or the plant manager, additional makeup oil can be added to the system via the 27.14 by following the standard procedure; FQI-334 readings and the change in

level of the 27.14 should be recorded before and after each addition. It is important to note that the discharge valve of the 10.52.01 and 02 pumps should be used to throttle to the 100 psig reactor pressure. The pressure in the sump of the 21.11 should be at 150 psig or less.

16. Record any indication of density or viscosity change, such as a change in the pressure drop across the reactor or shaking of the reactor during heatup and reduction.

17. When reduction has been completed, scan the reactor with the NDG. Record levels in the 21.11 and 27.14. Add fresh oil to 27.14 to bring the level up to 25 nuts on LG-358. This charge should be drawn from storage; note the FQI-334 readings before and after addition.

TA #37 is done, consult TEST AUTHORIZATION #38 for the next step.

ANALYTICAL REQUIREMENTS:

1. Catalyst sampling requirements:

   - slurried oxide catalyst from prep tank before reduction, and,
   - from the reactor, slurried reduced catalyst

   Exact quantities to be determined by operations, process, and research.

2. Composition sampling requirements:

   - reactor in and out continuously
   - H_2 and CO are critical
   - CO_2 and N_2 are also required

3. Flow measurement requirements:

   - reactor in at FI-126 and FI-299

REFERENCES:

1. TEST AUTHORIZATION # 23 : Procedure for previous in-situ activation.

RUN PLAN FOR SPRING 1994 IBOH DEMONSTRATION

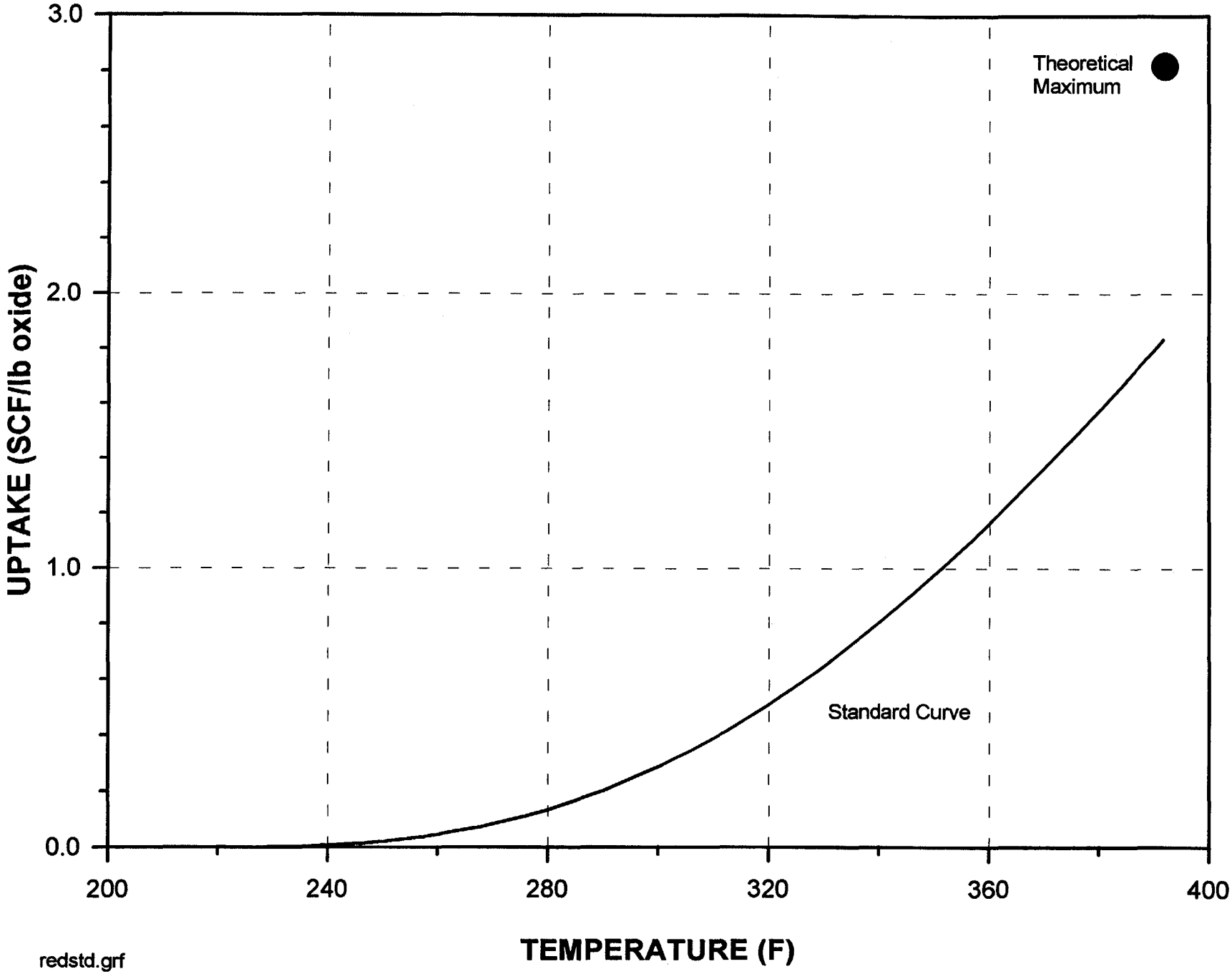
RUN				AF-R9.1	AF-R9.2
Description	---	---	Reduction	MEOH-1	MEOH-2
Duration	---	days	2	2	2
Syngas	---	---	---	TEXACO	KINGSPORT
Inlet Space Velocity	---	sL/kg-hr	1,200	6,700	4,000
Reactor Pressure	PIC-1247	psig	100	750	735
REACTOR					
Pressure	PIC-1247	psig	100	750	735
Temperature	TI-1233	F	---	482	482
Heat Duty		MM BTU/hr	---	1.14	1.21
Inlet Superficial Velocity	---	ft/sec	0.730	0.842	0.514
Outlet Superficial Velocity	---	ft/sec	---	0.644	0.348
Liquid Level	LI-2142	% span	90 - 95%	100%	100%
Catalyst Load	---	lb	1,250	1,250	1,250
Cat Weight Fraction	---	%	40.0%	42.4%	40.0%
Vapor Void Fraction	---	%	23 - 30%	42.9%	34.5%
FEED FLOWS					
LP H2	FIC-101	scfh	311	17,970	20,770
CO	FIC-104	scfh	466	15,581	12,694
CO2	FIC-107	scfh	130	1,192	1,065
N2	FIC-111	scfh	25,003	57	271
01.10 Total Flow	FI-726	scfh	25,910	34,800	34,800
HP H2	FIC-1200	scfh	0	8,665	4,937
01.20 Recycle	FIC-246	scfh	0	101,984	46,578
10.95 PUMP INJECTION					
Total Flow	FI-1221	gpm	zero	zero	zero
MEOH	---	wt%	---	---	---
C2OH	---	wt%	---	---	---
C3OH	---	wt%	---	---	---
REACTOR FEED					
Target Feed Temp	TI-1253	F	---	362.2	390.7
Feed Dewpoint	---	F	---	89.0	78.2
Total Dry Flow	FI-1216	scfh	25,910	145,449	86,316
H2	---	mol%	1.20%	34.71%	60.91%
CO	---	mol%	1.80%	50.57%	24.49%
N2	---	mol%	96.50%	1.00%	3.90%
CO2	---	mol%	0.50%	12.89%	10.03%
MEOH	---	mol%	0.00%	0.71%	0.42%
ETOH	---	mol%	0.00%	0.00%	0.00%
PROH	---	mol%	0.00%	0.00%	0.00%
C1	---	mol%	0.00%	0.03%	0.08%
			100.00%	*99.91%*	*99.83%*

JMRepasky 3/3/94

RUN PLAN FOR SPRING 1994 IBOH DEMONSTRATION

RUN				AF-R9.1	AF-R9.2
Description	---	---	Reduction	MEOH-1	MEOH-2
21.11 Feed/Product Exchanger					
Feed Inlet Temp	TI-1257	F	---	172.2	209.3
Feed Outlet Temp	TI-1263	F	---	402.3	401.6
Total Feed to 02.63 Temp	TI-1216	F	---	362.3	390.7
Reactor Eff. Inlet Temp	TI-1262	F	---	482.0	482.0
Reactor Eff. Outlet Temp	TIC-1260	F	---	280.0	280.0
Reactor Eff. Dew Temp	---	F	---	226.9	267.8
REACTOR EFFLUENT					
Total Flow	FI-196	scfh	---	119,495	62,776
H2	---	mol%	---	20.75%	46.48%
CO	---	mol%	---	50.50%	14.66%
N2	---	mol%	---	1.21%	5.36%
CO2	---	mol%	---	15.85%	14.02%
MEOH	---	mol%	---	11.20%	17.88%
ETOH	---	mol%	---	0.08%	0.24%
PROH	---	mol%	---	0.02%	0.08%
C4OH	---	mol%	---	0.01%	0.03%
IBOH	---	mol%	---	0.00%	0.01%
C5OH+	---	mol%	---	0.01%	0.02%
C1	---	mol%	---	0.05%	0.16%
			---	*99.66%*	*98.95%*
PRODUCT RECOVERY					
Syngas to Backend Flow	FI-682	scfh	---	None	None
22.11 to Flare Flow	FI-237	scfh	---	875	667
Main Flare Flow	FI-245	scfh	---	3,867	3,867
Product Flow	---	gpd	---	3,897	3,527
BACK-END					
MEOH Circulation	FIC-814	gpm	None	None	None
MEOH to 07.10 Temp	TI-814	F	---	---	---
07.10 OH Temp	TI-1275	F	---	---	---
07.20 OH to Flare Flow	FI-7291	scfh	---	---	---
07.20 Reboiler Temp	TIC-7339	F	---	---	---
07.22 Steam Pressure	PIC-7338	psig	---	---	---
07.22 Steam Usage	FI-7338	lb/hr	---	---	---
21.80 CO2 Usage	---	TPD	---	---	---
Total CO2 Usage	Fd+21.80	TPD	0.18	1.63	1.45
RECYCLE FEED					
H2	---	mol%	---	23.38%	57.67%
CO	---	mol%	---	56.85%	18.13%
N2	---	mol%	---	1.36%	6.65%
CO2	---	mol%	---	17.22%	16.30%
MEOH	---	mol%	---	1.01%	0.78%
C1	---	mol%	---	0.04%	0.16%
			---	*99.85%*	*99.69%*

METHANOL CATALYST REDUCTION
Reduction Gas Uptake vs. Temperature



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TEST AUTHORIZATION # 38

LaPorte Alternative Fuels Development Unit (AFDU)

Date : 03/02/94
By: ESS

RUN NUMBER: AF-R9
APPROX. START DATE: 15 March, 1994

TITLE: METHANOL SYNTHESIS WITH BASF S3-86 CATALYST

OBJECTIVE:
To study the performance of S3-86 methanol catalyst in the 27.20 reactor train.

SUMMARY:
Upon completion of the activation step (AF-A5), the reactor feed will be adjusted to a Texaco gas composition (35% H_2, 51% CO, 13% CO_2, 1% N_2). For approximately 2 days, the conditions will be targeted at 750 psig, 482°F, 6,700 sL/kg-hr space velocity, and 40 wt% oxide in oil. After 2 days, the gas composition will be switched to a Kingsport LPMEOH gas composition for three days of operation (60.7% H_2, 24.4% CO, 10.0% CO_2, 3.89% N_2). The objective is to condition the oil, allow the hyper-activity of the catalyst to decline, break-in the new DCS controls, and ultimately line-out at a steady rate of methanol production while collecting data for H_2-rich reactor feed gas.

TEST DETAILS: See page 2.

ANALYTICAL COMMENTS: See page 3.

SAFETY IMPLICATIONS:
Protective gear including face shield should be worn during slurry sampling.

ENVIRONMENTAL IMPLICATIONS:
Minimal.

SPECIAL REMARKS:
The high pressure hydrogen pipe line will be in use during run AF-R9. The CO_2 removal system will not be in operation. Special sample bombs will be used to collect samples of the methanol product produced during case AF-R9.2.

AUTHORIZATIONS:

E. C. Heydorn, Plant Mgr

E. S. Schaub, Process Engr

TEST DETAILS:

1. Upon completion of the catalyst activation (AF-A5), switch from reduction gas to Texaco-type gas by following the standard procedure. The CO2 removal section should NOT be operating during this run (V-2001,V-2003,V-2004,V-2006 shut; V-2000 open). In the event of a premature shut-down, consult TA #20 (RUN E-05) for appropriate standby conditions.

2. Increase the reactor pressure to 750 psig and control the slurry temperature at 482°F. Slowly increase the reactor feed rate to 25,000 SCFH while maintaining slurry level at 95% of NDG span. When the plant has lined out, the reactor feed composition should correspond closely to case AF-R9.1 (refer to Table). Once the compositions are lined out, slowly introduce recycle flow and back off the fresh feed flowrates until they match the targets outlined in the Table for case AF-R9.1. Note that the HP hydrogen pipeline is in service during cases AF-R9.1 and AF-R9.2.

3. When the target feed rate has been achieved, put LIC-1242 in automatic to control slurry level at 95%. Adjust the fresh feed flow to achieve an initial purge flow rate of approximately 3,000 SCFH. Maintain reactor feed flow and reactor temperature and pressure at the case AF-R9.1 values for a nominal 24 hour period.

4. During the first 24 hours, the syngas conversion across the reactor will fall as the catalyst loses it's hyperactivity. The purge flow will increase and the reactor feed composition will be changing during this period. When these rates of change diminish, fine tune the fresh feed flow to reach the desired reactor feed composition as specified for case AF-R9.1. The ultimate purge rate should be around 3,900 SCFH.

5. After the initial break-in period, begin to increase rates to maximize production of methanol. Monitor the air-cooler loading and temperature difference between the utility oil and the slurry and utility oil inlet & outlet using TDI-1237 and TDI-1252. Both of these temperature differences must be below 200°F.

6. The composition of the methanol product is to be monitored every 8 hours. The target oil content of the methanol product should be <=0.2 wt%. If the oil content is higher, lower the 21.11 effluent outlet TIC-1260 setpoint.

7. Maintain conditions for approximately 2 days. After conferring with the process engineer or plant manager, switch to AF-R9.2 run conditions (Kingsport gas). Run this data period for approximately 3 days.

8. Liquid samples of the methanol product will be collected in special sample bombs and shipped to Allentown for detailed analysis during case AF-R9.2. The samples will be collected downstream of the 22.11 separator. Consult with the process engineer and analytical representative for the frequency and manner of taking the samples.

9. When notified by the plant manager that case AF-R9.2 is complete, de-pressurize the plant, and drain the slurry from the 27.20 reactor using the prep tank as an intermediate hold point using the standard shutdown procedures. Drain the 22.10, 22.15 and 22.16. Proceed with TEST AUTHORIZATION #39.

ANALYTICAL COMMENTS:

1. Catalyst sampling requirements:

   - slurried catalyst at end-of-run.

   Exact quantities to be determined by operations, process, and research.

2. Continuous composition sampling requirements (GC):

   - fresh feed,
   - reactor in,
   - reactor out,
   - recycle
   - 22.10 overheads

3. Periodic composition sampling requirements (GC):

   - 22.11 off-gas (frequency to be determined by operations & process)

   Periodic composition sampling requirements (LC):

   - methanol product (every 8 hours during first two days, twice a day thereafter)

4. Flow measurement requirements:

   - fresh feed,
   - reactor in,
   - reactor out,
   - recycle,
   - purge,
   - 22.11 off-gas,
   - methanol product

REFERENCES:

1. TEST AUTHORIZATION #20 - Procedures for reactor standby during shutdown.

2. STANDARD STARTUP PROCEDURES FOR MeOH-ONLY OPERATION

RUN PLAN FOR SPRING 1994 IBOH DEMONSTRATION

RUN				AF-R9.1	AF-R9.2
Description	---	---	Reduction	MEOH-1	MEOH-2
Duration	---	days	2	2	2
Syngas	---	---	---	TEXACO	KINGSPORT
Inlet Space Velocity	---	sL/kg-hr	1,200	6,700	4,000
Reactor Pressure	PIC-1247	psig	100	750	735
REACTOR					
Pressure	PIC-1247	psig	100	750	735
Temperature	TI-1233	F	---	482	482
Heat Duty		MM BTU/hr	---	1.14	1.21
Inlet Superficial Velocity	---	ft/sec	0.730	0.842	0.514
Outlet Superficial Velocity	---	ft/sec	---	0.644	0.348
Liquid Level	LI-2142	% span	90 - 95%	100%	100%
Catalyst Load	---	lb	1,250	1,250	1,250
Cat Weight Fraction	---	%	40.0%	42.4%	40.0%
Vapor Void Fraction	---	%	23 - 30%	42.9%	34.5%
FEED FLOWS					
LP H2	FIC-101	scfh	311	17,970	20,770
CO	FIC-104	scfh	466	15,581	12,694
CO2	FIC-107	scfh	130	1,192	1,065
N2	FIC-111	scfh	25,003	57	271
01.10 Total Flow	FI-726	scfh	25,910	34,800	34,800
HP H2	FIC-1200	scfh	0	8,665	4,937
01.20 Recycle	FIC-246	scfh	0	101,984	46,578
10.95 PUMP INJECTION					
Total Flow	FI-1221	gpm	zero	zero	zero
MEOH	---	wt%	---	---	---
C2OH	---	wt%	---	---	---
C3OH	---	wt%	---	---	---
REACTOR FEED					
Target Feed Temp	TI-1253	F	---	362.2	390.7
Feed Dewpoint	---	F	---	89.0	78.2
Total Dry Flow	FI-1216	scfh	25,910	145,449	86,316
H2	---	mol%	1.20%	34.71%	60.91%
CO	---	mol%	1.80%	50.57%	24.49%
N2	---	mol%	96.50%	1.00%	3.90%
CO2	---	mol%	0.50%	12.89%	10.03%
MEOH	---	mol%	0.00%	0.71%	0.42%
ETOH	---	mol%	0.00%	0.00%	0.00%
PROH	---	mol%	0.00%	0.00%	0.00%
C1	---	mol%	0.00%	0.03%	0.08%
			100.00%	*99.91%*	*99.83%*

JMRepasky 3/3/94

RUN PLAN FOR SPRING 1994 IBOH DEMONSTRATION

RUN				AF-R9.1	AF-R9.2
Description	---	---	Reduction	MEOH-1	MEOH-2
21.11 Feed/Product Exchanger					
Feed Inlet Temp	TI-1257	F	---	172.2	209.3
Feed Outlet Temp	TI-1263	F	---	402.3	401.6
Total Feed to 02.63 Temp	TI-1216	F	---	362.3	390.7
Reactor Eff. Inlet Temp	TI-1262	F	---	482.0	482.0
Reactor Eff. Outlet Temp	TIC-1260	F	---	280.0	280.0
Reactor Eff. Dew Temp	---	F	---	226.9	267.8
REACTOR EFFLUENT					
Total Flow	FI-196	scfh	---	119,495	62,776
H2	---	mol%	---	20.75%	46.48%
CO	---	mol%	---	50.50%	14.66%
N2	---	mol%	---	1.21%	5.36%
CO2	---	mol%	---	15.85%	14.02%
MEOH	---	mol%	---	11.20%	17.88%
ETOH	---	mol%	---	0.08%	0.24%
PROH	---	mol%	---	0.02%	0.08%
C4OH	---	mol%	---	0.01%	0.03%
IBOH	---	mol%	---	0.00%	0.01%
C5OH+	---	mol%	---	0.01%	0.02%
C1	---	mol%	---	0.05%	0.16%
			---	*99.66%*	*98.95%*
PRODUCT RECOVERY					
Syngas to Backend Flow	FI-682	scfh	---	None	None
22.11 to Flare Flow	FI-237	scfh	---	875	667
Main Flare Flow	FI-245	scfh	---	3,867	3,867
Product Flow	---	gpd	---	3,897	3,527
BACK-END					
MEOH Circulation	FIC-814	gpm	None	None	None
MEOH to 07.10 Temp	TI-814	F	---	---	---
07.10 OH Temp	TI-1275	F	---	---	---
07.20 OH to Flare Flow	FI-7291	scfh	---	---	---
07.20 Reboiler Temp	TIC-7339	F	---	---	---
07.22 Steam Pressure	PIC-7338	psig	---	---	---
07.22 Steam Usage	FI-7338	lb/hr	---	---	---
21.80 CO2 Usage	---	TPD	---	---	---
Total CO2 Usage	Fd+21.80	TPD	0.18	1.63	1.45
RECYCLE FEED					
H2	---	mol%	---	23.38%	57.67%
CO	---	mol%	---	56.85%	18.13%
N2	---	mol%	---	1.36%	6.65%
CO2	---	mol%	---	17.22%	16.30%
MEOH	---	mol%	---	1.01%	0.78%
C1	---	mol%	---	0.04%	0.16%
			---	*99.85%*	*99.69%*

JMRepasky 3/3/94

TEST AUTHORIZATION # 39

LaPorte Alternative Fuels Development Unit (AFDU)

Date : 03/02/94
By: ESS

RUN NUMBER: AF-A6
APPROX. START DATE: 24 March, 1994

TITLE: IN-SITU ISOBUTANOL CATALYST ACTIVATION PRIOR TO SPRING 94 RUN USING DILUTE CO-RICH REDUCTION GAS

OBJECTIVE:
To activate the Liquid-Phase Mixed Alcohols synthesis catalyst.

SUMMARY:
Approximately 1106 lbs of CS-doped BASF S3-86 oxide is to be slurried with Drakeol-10 oil, transferred to the 27.20 reactor and activated with dilute CO-Rich syngas (4.0% in nitrogen). Approximate run time is 2 days.

TEST DETAILS: See pages 2 to 4 for details.

ANALYTICAL COMMENTS: See page 4.

SAFETY IMPLICATIONS:
Operators should wear protective gear while loading catalyst to protect them from the dust and hot vapor which may be released from the loading nozzle. Protective gear including face shield should be worn during slurry sampling.

This operation will require the venting of unreacted hydrogen and CO. During the previous similar activation (performed under TEST AUTHORIZATION #29) the off-gas was blended with methane and burned in the flare. Previous calculations (for TA #23) indicated that in the event a combustible mixture could not be maintained, there would be no danger to personnel from venting. The reduction gas flow rates to be used in this run are less than those used in TA #23.

ENVIRONMENTAL IMPLICATIONS:
Minimal, a flame will be maintained at the flare. At 98% destruction efficiency, the CO emission rate would be 0.6 lb/hr.

SPECIAL REMARKS:
Hydrogen and CO concentrations in and out of the reactor must be monitored closely during the reduction. Reactor temperature must be closely monitored and controlled per the attached TEST DETAILS. The utility oil inlet temperature (TI 1244) to the 27.20 internal heat exchanger must not exceed a 200°F difference from the utility oil outlet temperature (TI-1246) or the reactor slurry temperature. These two temperature differentials are measured directly by TDI-1252 & TDI-1237. When adjusting flows or pressure, care should be taken to minimize catalyst carryover (caused by high gas velocity).

AUTHORIZATIONS:

E. C. Heydorn, Plant Mgr

E. S. Schaub, Process Engr

TEST DETAILS:

1. This reduction procedure follows the successful ER-6 reduction (refer to TEST AUTHORIZATION #23)

2. Charge the 28.30 prep tank with 1659 lb of oil (235 gallons of Drakeol-10 at 80°F). The oil should be transferred to drums and weighed using the scale for accurate measurement. As an approximation, meter the oil with FQI-334 using a meter correction factor of actual = 1.027 * meter (meter should read 228.4 gal). If the temperature differs from 80°F a corrected oil volume should be used. Heat this oil to 150-200°F.

3. Fill the 27.14 intermediate V/L separator to 25 nuts on LG-358 with approximately 100 gallons of Drakeol-10 oil from storage. Note the FQI-334 readings before and after the addition.

4. When the prep tank oil is at 150-200°F, add 1106 lb of isobutanol catalyst (CS-BASF S3-86)). Add the catalyst very slowly to make a 40 wt% oxide slurry. Keep the slurry well stirred to prevent agglomeration of the catalyst.

5. Heat the slurry to 200°F and continue agitation, under nitrogen, for at least 2 hours to ensure good mixing.

6. When the catalyst and oil have been completely mixed, withdraw a sample of slurry.

7. Establish gas flow through the reactor using nitrogen through V-2627 to prevent slurry back-flow into the distributor. Vent the gas through PV-1261.

8. Pressure transfer the slurry to the reactor and verify operation by noting level with the nuclear density gauge (NDG estimated level: 22-25 ft.)

9. Flush out the prep tank with 283 lb of oil (40 gallons of Drakeol-10 at 80°F). Measure the oil as in step 2. Meter should read approximately 38.9 gallons. Pressure transfer the flush oil to the reactor and verify level with the NDG.

10. Close V-645 to prevent utility oil flow back to the prep tank and establish full utility oil flow through the 27.20 internal heat exchanger.

11. Pressurize the reactor loop to 100 psig.

12. Begin heating the slurry to 200°F, following TAVR on the DEC console. Check that the slurry temperatures are in reasonable agreement. Verify that the slurry is well mixed by performing a NDG scan.

13. Establish CO-Rich reduction gas flow at 22,920 SCFH (on FI-126) and vent the flow through PV-170. Establish the following composition (Note that this composition is slightly different than those used for typical methanol catalyst reductions.) :

	Composition	Est. Flows (SCFH)
H2	1.4%	321
CO	2.1	481
CO2	0.5	115
N2	96.0	22,003
	100.0	22,920

MW = 27.7, SCF evaluated at 70°F, 14.7 psia
Target space velocity = 1200 sL/h-kg; Target starting inlet superficial velocity = 0.64 ft/sec

14. When the reactor temperature reaches 200°F, bring reduction gas to the reactor slowly and close the nitrogen purge (V-2627). Establish a final flow to the reactor of 22,910 SCFH. Maintain flow and reducing gas composition as specified in step 13. The temperature-programmed activation consists of the following steps:

    - Heat the slurry at a target rate of 10°F/hr (no more than 15°F/hr) until the slurry temperature reaches 392°F (200°C). This ramp rate is slower than methanol reduction procedures. Care should be taken to control this ramp rate at the beginning of the reduction.
    - Hold the slurry temperature at 392°F for 12 hours.
    - Heat the slurry at 10°F/hr until the slurry temperature reaches 464°F (240°C).
    - Hold the slurry temperature at 464°F for 1 hour (or longer if syngas uptake is still apparent).

    H2 and CO concentrations are to be measured continuously for the feed and effluent streams. As long as the cumulative H2 plus CO consumption at a given temperature is equal to or greater than the autoclave reduction data then the activation is proceeding well. Figure 1 shows the consumption profile vs temperature from the labs. If the cumulative consumption curve falls below the autoclave curve, consult the process or research engineer to reduce the heatup rate.

    If the H2+CO concentration in the effluent falls below 0.1 mole %, increase the inlet H2+CO concentration per the instructions of the process or research engineer. The objective here is to prevent reduction gas starvation.

    During the 392°F hold period, it may become necessary to maintain this temperature beyond 12 hours until the difference between inlet and outlet H2+CO concentration falls below 0.05 mole %.

15. The slurry level should be maintained between 80 and 90% of NDG range (estimated 35 ft.) by using LIC 1242 to control the makeup oil rate. Note that as the reactor is heated up to 464 F, the slurry will expand. At the same time, some of the oil will be lost in the reactor effluent. If

authorized by the process engineer or plant manager, additional makeup oil can be added to the system via the 27.14 by following the standard procedure; FQI-334 readings and the change in the level of the 27.14 should be recorded before and after each addition. Note that the discharge valve of the 10.52.01 and 02 pumps should be used to throttle to the 100 psig reactor pressure. The pressure in the sump of the 21.11 should be at 150 psig or less.

16. Record any indication of density or viscosity change, such as a change in the pressure drop across the reactor or shaking of the reactor during heatup and reduction.

17. When reduction has been completed, scan the reactor with the NDG. Record levels in the 21.11 and 27.14. Add fresh oil to 27.14 to bring the level up to 25 nuts on LG-358. This charge should be drawn from storage; note the FQI-334 readings before and after addition.

18. Withdraw a slurry sample.

TA #39 is done, consult TEST AUTHORIZATION #40 for the next step.

ANALYTICAL REQUIREMENTS:

1. Catalyst sampling requirements:

   - slurried oxide catalyst from prep tank before reduction, and,
   - from the reactor, slurried reduced catalyst

   Exact quantities to be determined by operations, process, and research.

2. Composition sampling requirements:

   - reactor in and out continuously
   - H_2 and CO are critical
   - CO_2 and N_2 are also required

3. Flow measurement requirements:

   - reactor in at FI-126 and FI-299

REFERENCES:

1. TEST AUTHORIZATION # 23 : Procedure for previous in-situ activation.

RUN PLAN FOR SPRING 1994 IBOH DEMONSTRATION

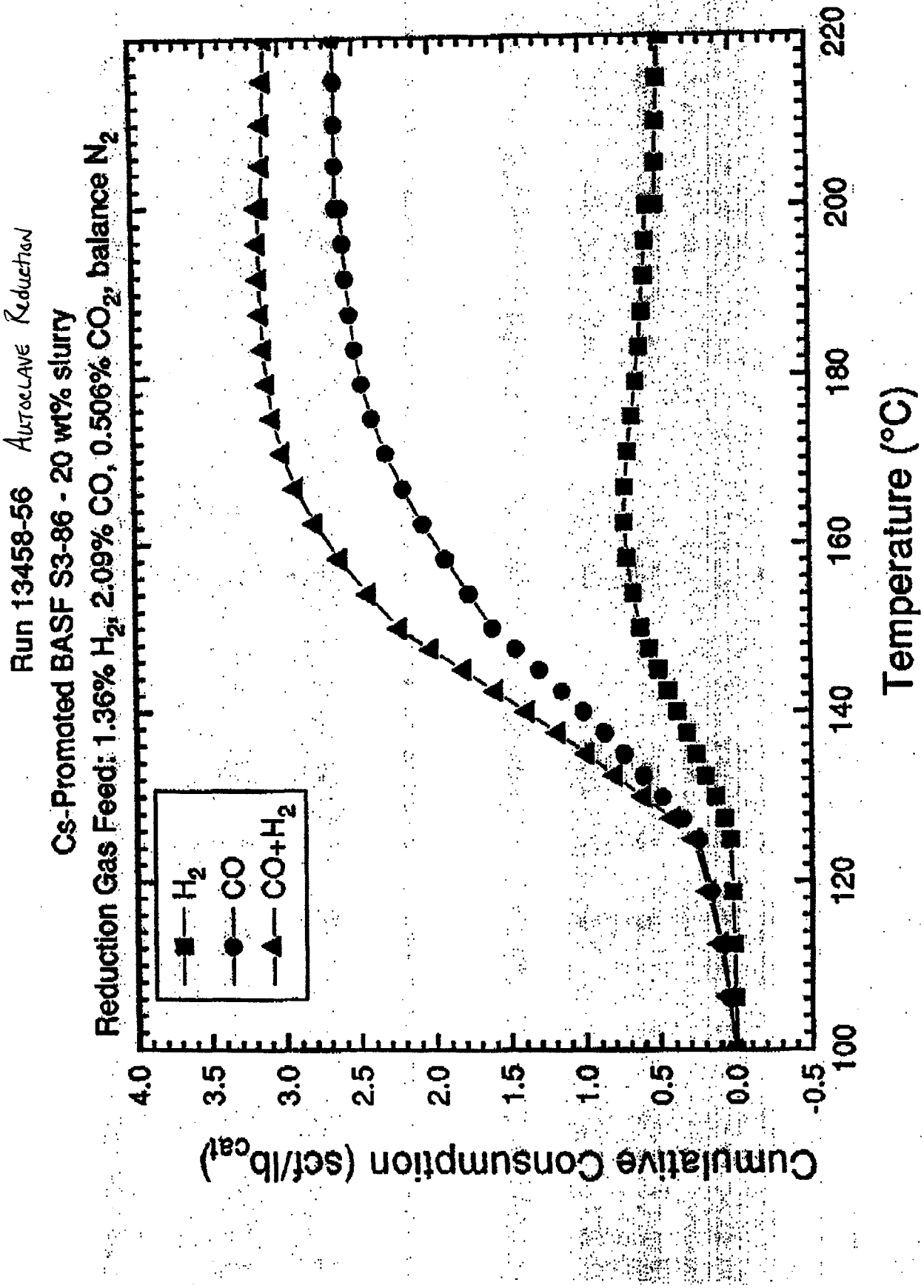
RUN			AF-A6	AF-R10.1	AF-R10.2	AF-R10.3	AF-R10.4	AF-R10.5	AF-R10.6	AF-R10.7	AF-R10.8	AF-R10.9	AF-R10.10	AF-R10.11
Description	---	---	Reduction	IBOH-1	IBOH-2	IBOH-3	IBOH-4	IBOH-5	IBOH-6	IBOH-7	IBOH-8	IBOH-9	IBOH-10	IBOH-11
Duration	---	days	2	2	1	2	2	2	1	1	1	1	1	2
Syngas	---	---	---	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	TEXACO
Inlet Space Velocity	---	sL/kg-hr	1,200	5,000	3,000	8,200	8,200	8,200	3,000	5,000	5,000	5,000	5,000	3,000
Reactor Pressure	PIC-1247	psig	100	750	750	750	1,300	1,735	1,735	1,300	1,300	1,300	750	1,300
REACTOR														
Pressure	PIC-1247	psig	100	750	750	750	1,300	1,735	1,735	1,300	1,300	1,300	750	1,300
Temperature	TI-1233	F	---	572	572	572	572	572	572	572	572	572	572	572
Heat Duty		MM BTU/hr	---	0.53	0.52	0.59	1.48	1.97	1.01	1.12	0.85	1.03	0.53	0.59
Inlet Superficial Velocity	---	ft/sec	0.640	0.611	0.366	1.002	0.583	0.438	0.160	0.355	0.390	0.366	0.611	0.213
Outlet Superficial Velocity	---	ft/sec	---	0.517	0.348	0.859	0.461	0.329	0.115	0.275	0.327	0.291	0.517	0.168
Liquid Level	LI-2142	% span	80 - 90%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%
Catalyst Load	---	lb	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107
Cat Weight Fraction	---	%	40.0%	40.2%	39.1%	41.5%	42.0%	42.2%	39.8%	40.7%	41.1%	42.0%	40.2%	39.6%
Vapor Void Fraction	---	%	25 - 30%	40.2%	36.4%	44.3%	46.0%	46.9%	38.7%	41.7%	43.2%	44.4%	40.2%	38.2%
FEED FLOWS														
LP H2	FIC-101	scfh	321	9,440	7,082	13,518	13,936	9,450	12,104	15,799	9,131	13,932	9,440	10,605
CO	FIC-104	scfh	481	10,016	9,086	12,582	20,790	25,311	13,715	15,670	14,682	15,712	10,016	9,476
CO2	FIC-107	scfh	114	31	0	0	0	0	607	0	0	659	31	0
N2	FIC-111	scfh	22,003	49	54	56	74	73	71	60	66	65	49	75
01.10 Total Flow	FI-726	scfh	22,919	19,536	16,222	26,157	34,800	34,835	26,497	31,529	23,879	30,367	19,536	20,156
HP H2	FIC-1200	scfh	0	0	0	0	10,019	20,322	0	0	0	0	0	0
01.20 Recycle	FIC-246	scfh	0	76,849	40,926	131,169	112,966	102,604	31,305	64,926	72,840	66,495	76,849	38,114
10.95 PUMP INJECTION														
Total Flow	FI-1221	gpm	zero	zero	zero	zero	zero	zero	zero	zero	2.14	0.70	zero	zero
MEOH	---	wt%	---	---	---	---	---	---	---	---	83.00%	69.50%	---	---
C2OH	---	wt%	---	---	---	---	---	---	---	---	5.00%	10.50%	---	---
C3OH	---	wt%	---	---	---	---	---	---	---	---	12.00%	20.00%	---	---
											100.00%	*100.00%*		
REACTOR FEED														
Target Feed Temp	TI-1253	F	---	437.8	443.2	431.7	429.8	429.6	429.5	429.4	400.0	400.0	437.8	431.5
Feed Dewpoint	---	F	---	-6.6	-9.3	-5.2	-9.6	-14.3	-15.1	-9.4	239.5	178.4	-6.6	92.9
Total Dry Flow	FI-1216	scfh	22,920	96,375	57,133	157,308	157,750	157,727	57,770	96,434	96,692	96,859	96,375	58,268
H2	---	mol%	1.40%	29.73%	30.05%	29.87%	29.78%	29.79%	29.72%	29.71%	27.03%	28.73%	29.73%	34.37%
CO	---	mol%	2.10%	65.41%	66.11%	65.70%	65.52%	65.53%	65.38%	65.37%	59.46%	63.21%	65.41%	50.09%
N2	---	mol%	96.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%
CO2	---	mol%	0.50%	2.97%	1.91%	2.99%	2.91%	2.98%	2.97%	2.88%	2.68%	2.87%	2.97%	12.77%
MEOH	---	mol%	0.00%	0.04%	0.03%	0.04%	0.04%	0.03%	0.03%	0.03%	7.89%	2.32%	0.04%	0.55%
ETOH	---	mol%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.33%	0.24%	0.00%	0.01%
PROH	---	mol%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.61%	0.35%	0.00%	0.00%
C1	---	mol%	0.00%	0.84%	0.99%	0.53%	1.04%	0.95%	1.15%	1.15%	1.23%	1.15%	0.84%	0.73%
			100.00%	*99.98%*	*100.09%*	*100.13%*	*100.28%*	*100.28%*	*100.25%*	*100.14%*	*100.22%*	*99.87%*	*99.98%*	*99.53%*

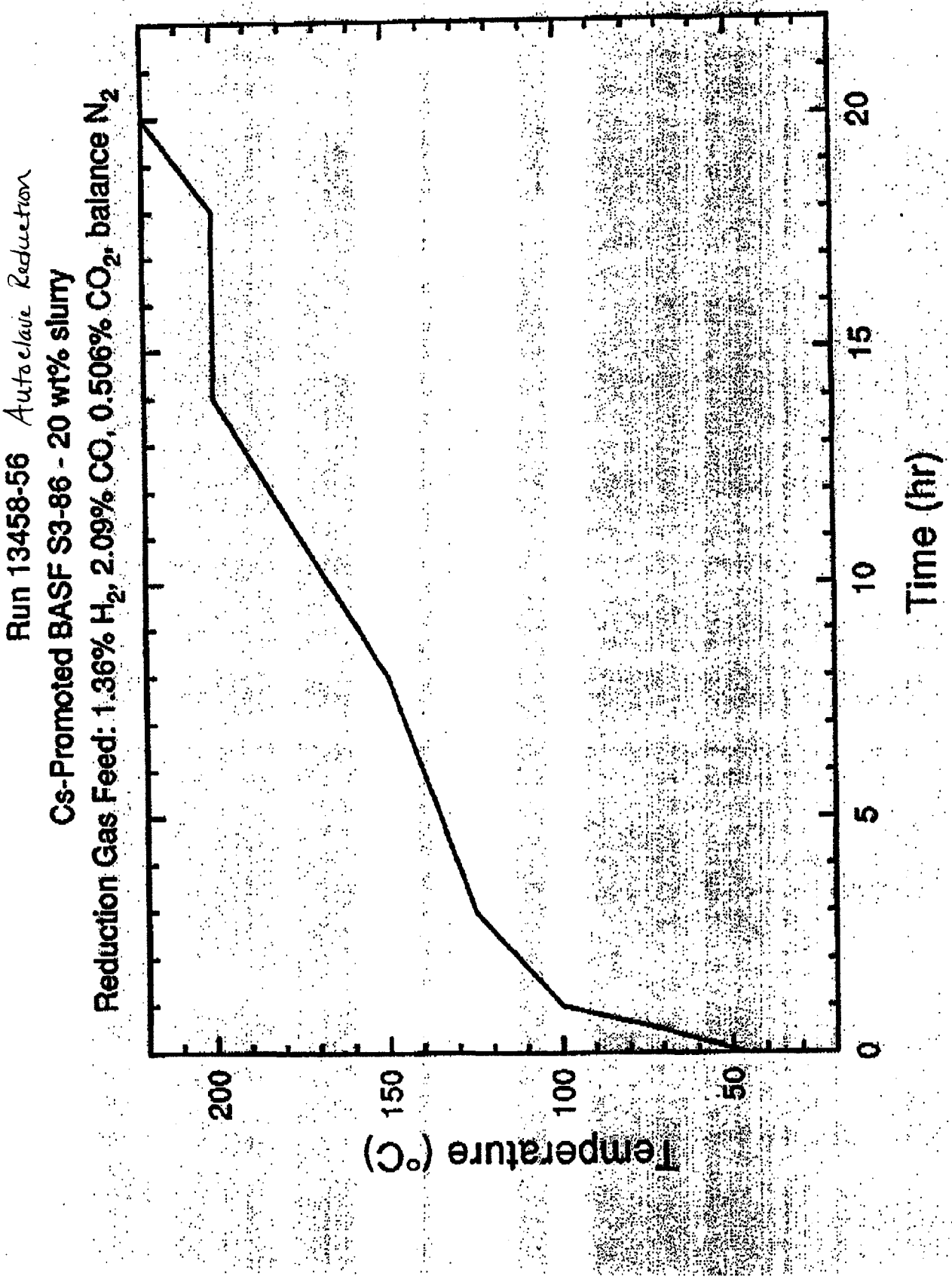
JMRepasky 3/3/94

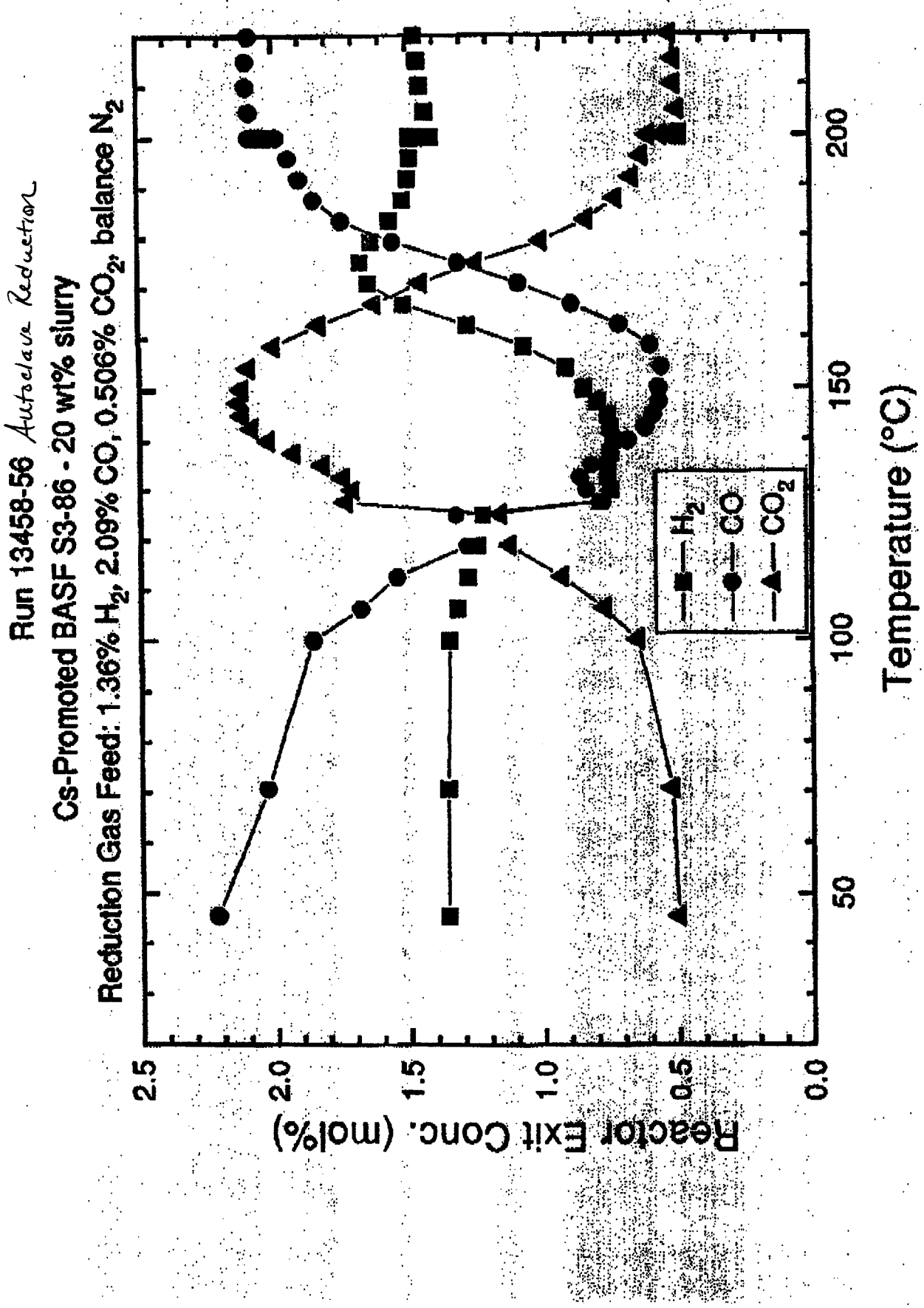
RUN PLAN FOR SPRING 1994 IBOH DEMONSTRATION

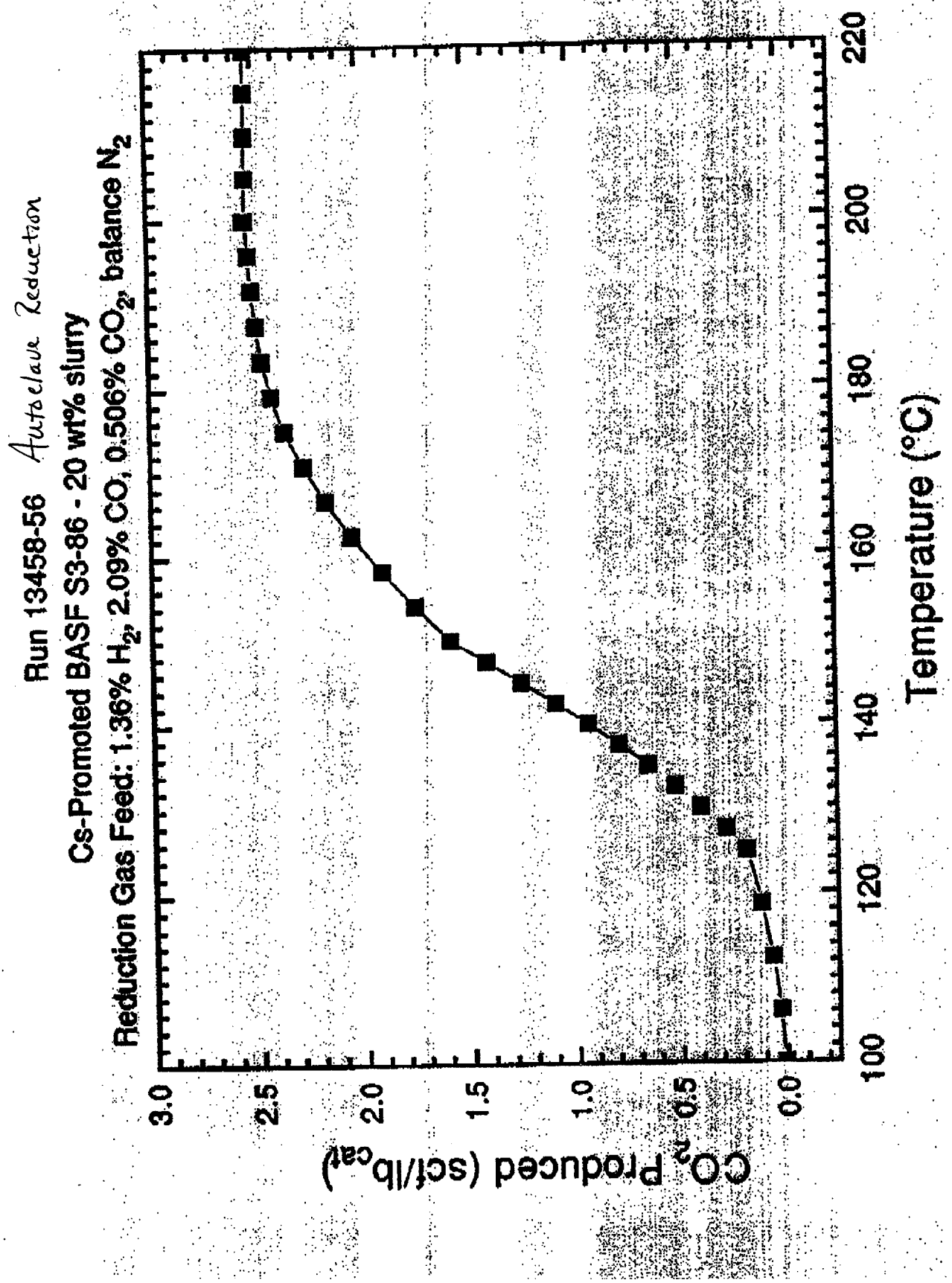
RUN			AF-A6	AF-R10.1	AF-R10.2	AF-R10.3	AF-R10.4	AF-R10.5	AF-R10.6	AF-R10.7	AF-R10.8	AF-R10.9	AF-R10.10	AF-R10.11
Description	---	---	Reduction	IBOH-1	IBOH-2	IBOH-3	IBOH-4	IBOH-5	IBOH-6	IBOH-7	IBOH-8	IBOH-9	IBOH-10	IBOH-11
21.11 Feed/Product Exchanger														
Feed Inlet Temp	TI-1257	F	---	154.1	161.2	147.3	150.0	150.0	150.0	150.0	120.0	120.0	154.1	150.0
Feed Outlet Temp	TI-1263	F	---	492.8	492.0	492.3	492.2	492.2	492.2	492.2	492.0	492.2	492.8	492.2
Total Feed to 02.63 Temp	TI-1216	F	---	437.8	443.2	431.7	429.8	429.6	429.5	429.4	475.8	423.7	437.8	431.5
Reactor Eff. Inlet Temp	TI-1262	F	---	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0
Reactor Eff. Outlet Temp	TIC-1260	F	---	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0
Reactor Eff. Dew Temp	---	F	---	198.9	219.5	186.8	238.7	262.5	275.7	247.3	279.8	263.8	198.9	245.7
REACTOR EFFLUENT														
Total Flow	FI-196	scfh	---	86,318	49,328	143,328	131,379	124,583	43,512	78,551	93,645	83,183	86,318	47,959
H2	---	mol%	---	23.47%	22.60%	24.22%	18.26%	14.48%	13.20%	17.47%	22.24%	18.88%	23.47%	22.64%
CO	---	mol%	---	65.14%	64.82%	65.96%	65.95%	66.18%	63.55%	64.92%	55.69%	61.33%	65.14%	47.28%
N2	---	mol%	---	0.89%	0.82%	0.76%	0.62%	0.67%	0.73%	0.79%	0.66%	1.15%	0.89%	1.19%
CO2	---	mol%	---	5.37%	6.03%	4.54%	6.13%	7.18%	10.72%	7.42%	8.00%	7.89%	5.37%	18.30%
MEOH	---	mol%	---	2.98%	2.79%	3.05%	6.25%	8.33%	6.93%	5.92%	8.60%	6.61%	2.98%	7.24%
ETOH	---	mol%	---	0.13%	0.14%	0.11%	0.26%	0.38%	0.47%	0.31%	0.59%	0.43%	0.13%	0.32%
PROH	---	mol%	---	0.10%	0.14%	0.07%	0.19%	0.28%	0.50%	0.27%	0.89%	0.60%	0.10%	0.21%
C4OH	---	mol%	---	0.03%	0.04%	0.02%	0.05%	0.07%	0.15%	0.07%	0.22%	0.17%	0.03%	0.05%
IBOH	---	mol%	---	0.16%	0.30%	0.10%	0.18%	0.22%	0.46%	0.27%	0.41%	0.37%	0.16%	0.16%
C5OH+	---	mol%	---	0.26%	0.51%	0.16%	0.29%	0.34%	0.70%	0.43%	0.54%	0.52%	0.26%	0.24%
C1	---	mol%	---	1.03%	1.31%	0.65%	1.35%	1.32%	1.82%	1.56%	1.54%	1.53%	1.03%	1.20%
			---	*99.54%*	*99.50%*	*99.62%*	*99.53%*	*99.44%*	*99.22%*	*99.43%*	*99.38%*	*99.46%*	*99.54%*	*98.84%*
PRODUCT RECOVERY														
Syngas to Backend Flow	FI-682	scfh	---	83,669	47,596	139,238	122,473	112,907	39,498	73,105	83,040	76,086	83,669	None
22.11 to Flare Flow	FI-237	scfh	---	94	75	126	360	525	243	252	497	337	94	304
Main Flare Flow	FI-245	scfh	---	3,867	4,004	4,431	4,157	4,253	3,867	3,960	4,292	3,867	3,867	5,605
Product Flow	---	gpd	---	982	725	1,417	3,037	3,941	1,504	1,957	3,884	2,644	982	1,267
BACK-END														
MEOH Circulation	FIC-814	gpm	None	9.17	7.90	11.56	14.66	14.83	8.46	10.43	13.94	13.88	9.17	None
MEOH to 07.10 Temp	TI-814	F	---	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	---
07.10 OH Temp	TI-1275	F	---	-4.6	-7.2	-3.1	-3.8	-2.9	-5.5	-3.5	-4.2	-5.8	-4.6	---
07.20 OH to Flare Flow	FI-7291	scfh	---	2,288	2,377	2,419	4,332	5,046	4,079	3,652	5,356	5,218	2,288	---
07.20 Reboiler Temp	TIC-7339	F	---	296.8	296.5	296.9	296.4	296.3	296.4	296.4	296.5	296.6	296.8	---
07.22 Steam Pressure	PIC-7338	psig	---	235	235	235	235	235	235	235	235	235	235	---
07.22 Steam Usage	FI-7338	lb/hr	---	592.8	592.5	576.8	664.0	692.7	679.2	650.4	710.9	692.0	592.8	---
21.80 CO2 Usage	---	TPD	---	36.18	33.82	36.29	36.50	36.83	35.72	35.49	36.15	33.60	36.18	---
Total CO2 Usage	Fd+21.80	TPD	0.16	36.22	33.82	36.29	36.50	36.83	36.55	35.49	36.15	34.50	36.22	0.00
RECYCLE FEED														
H2	---	mol%	---	25.00%	24.65%	25.51%	20.38%	16.77%	16.18%	19.80%	26.80%	22.14%	25.00%	24.73%
CO	---	mol%	---	68.99%	70.09%	69.20%	73.09%	76.07%	76.84%	72.95%	66.39%	71.17%	68.99%	51.71%
N2	---	mol%	---	0.94%	0.90%	0.80%	0.69%	0.77%	0.89%	0.89%	0.80%	1.34%	0.94%	1.31%
CO2	---	mol%	---	3.69%	2.67%	3.58%	4.06%	4.58%	3.55%	4.27%	3.90%	3.31%	3.69%	19.52%
MEOH	---	mol%	---	0.05%	0.04%	0.05%	0.05%	0.05%	0.05%	0.05%	0.05%	0.04%	0.05%	0.84%
C1	---	mol%	---	1.05%	1.38%	0.64%	1.45%	1.46%	2.12%	1.71%	1.78%	1.72%	1.05%	1.12%
			---	*99.72%*	*99.72%*	*99.78%*	*99.73%*	*99.70%*	*99.62%*	*99.67%*	*99.72%*	*99.73%*	*99.72%*	*99.22%*

JMRepasky 3/3/94


Run 13458-56 Autoclave Reduction
Cs-Promoted BASF S3-86 - 20 wt% slurry
Reduction Gas Feed: 1.36% H2, 2.09% CO, 0.506% CO2, balance N2
H2
CO
CO+H2
Cumulative Consumption (scf/lbcat)
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
100
120
140
160
180
200
220
Temperature (°C)


Run 13458-56 Autoclave Reduction
Cs-Promoted BASF S3-86 - 20 wt% slurry
Reduction Gas Feed: 1.36% H2, 2.09% CO, 0.506% CO2, balance N2
Temperature (°C)
200
150
100
50
0
5
10
15
20
Time (hr)


Run 13458-56 Autoclave Reduction
Cs-Promoted BASF S3-86 - 20 wt% slurry
Reduction Gas Feed: 1.36% H2, 2.09% CO, 0.506% CO2, balance N2
Reactor Exit Conc. (mol%)
2.5
2.0
1.5
1.0
0.5
0.0
50
100
150
200
Temperature (°C)
H2
CO
CO2


Run 13458-56 Autoclave Reduction
Cs-Promoted BASF S3-86 - 20 wt% slurry
Reduction Gas Feed: 1.36% H_2, 2.09% CO, 0.506% CO_2, balance N_2
CO_2 Produced (scf/lb$_{cat}$)
3.0
2.5
2.0
1.5
1.0
0.5
0.0
100
120
140
160
180
200
220
Temperature (°C)

TEST AUTHORIZATION # 40

LaPorte Alternative Fuels Development Unit (AFDU)

Date : 03/02/94
By: ESS

RUN NUMBER: AF-R10
APPROX. START DATE: 25 March, 1994

TITLE: LIQUID-PHASE-ISOBUTANOL PROCESS VARIABLE STUDIES WITH CS-BASF S3-86

OBJECTIVE:
To study the performance of cesium promoted methanol catalyst in a bubble column reactor at different space velocities & pressures and with different feed gas compositions. Two data periods will include mixed alcohol injection to demonstrate increased productivity of isobutanol when lower alcohol products are recycled.

SUMMARY:
Upon completion of the TA #39 (run AF-A6), 11-12 process variable scans, covering two different reactor feed compositions (Texaco and Shell), different space velocities and pressures, will be carried-out. The reactor will be maintained at 572°F throughout the studies.

TEST DETAILS: See pages 2 to 4 for details.

ANALYTICAL COMMENTS: See page 4.

SAFETY IMPLICATIONS:
Protective gear including face shield should be worn during slurry sampling.

ENVIRONMENTAL IMPLICATIONS:
Minimal.

SPECIAL REMARKS:
See Test Details.

AUTHORIZATIONS:

E. C. Heydorn, Plant Mgr

E. S. Schaub, Process Engr

TEST DETAILS:

1. Prior to completion of TA #39 (run AF-A6) the CO2 removal section is charged with approximately 638 gallons of methanol . Follow the procedures set forth by operations. The breakdown of methanol inventory in the back-end is:

07.10	130 gal	(54 gals above normal liquid level of 5 ft)
07.20	207 gal	(61 gals above normal liquid level of 6 ft)
07.22	132 gal	(full)
21.45/21.65	117 gal	(full)
piping	52 gal	

The inventory numbers in the 07.10 and 07.20 include the normal sump liquid level plus the liquid which will be held up in the packing once the 10.80 circulation pump is started. Upon completion of the TA #39 (run AF-A6), the 22.10, 22.15 and 22.16 should be empty, the reactor loop should be de-pressurized, and the CO2 removal section should be blocked-in.

2. Increase the reactor pressure to 750 psig and introduce syngas while the reactor is approximately 430-460 F. Initially control the slurry temperature at 482°F. Slowly increase the reactor feed rate to 25,000 SCFH. The reactor feed composition should correspond to run AF-R10.1 conditions (Shell gas) and the effluent should be directed to flare (once-through operation). Slowly ramp the reactor temperature up to the target of 572 F.

3. Once reactor feed flows have been established, bring the CO2 removal section on-line. When the CO2 removal section is functioning properly, recycle flow may be slowly brought in and the fresh feed flows reduced so that they match the target in the table for AF-R10.1 The process variable scans may begin.

4. PVS RUNS:

Process and control room targets are tabulated in the attached tables. The run descriptors are presented below:

RUN No.	Gas Type	Space Velocity	Pressure (psig)	Estimated No. of Days
AF-R10.1	SHELL	5000	750	2
AF-R10.2	SHELL	3000	750	1
AF-R10.3	SHELL	8200	750	2
AF-R10.4	SHELL	8200	1300	2
AF-R10.5	SHELL	8200	1735	2
AF-R10.6	SHELL	3000	1735	1
AF-R10.7	SHELL	5000	1300	1
AF-R10.8	SHELL with full alcohol injection	5000	1300	1

AF-R10.9	SHELL with partial alcohol injection	5000	1300	1
AF-R10.10	SHELL	5000	750	1
AF-R10.11	TEXACO	3000	1300	2

For each run condition a period of roughly 24 hours of steady operation constitutes a data period. Run 10.10 is optional and will be conducted if the schedule permits.

6. SPECIAL CONSIDERATIONS:

ALCOHOL INJECTION: For cases AF-R10.8 and AF-R10.9, the 10.95 pump will be used to inject alcohols into the reactor syngas feed. Before these runs start, steam flow to the 02.63 heater must be started and TSLL-1253 must be commissioned. Both of these steps will ensure that the liquids which are injected will be vaporized before entering the reactor.

CO2 REMOVAL OPERATION: The CO2 removal system will be operated during cases AF-R10.1-10.10 (Shell). Following AF-R10.10, it will be shut down and blocked in for the final case, AF-R10.11 (Texaco).

WATER BUILD-UP IN CO2 REMOVAL SECTION: Water and higher alcohols enter the back-end of the plant through the vapor off the 22.10 and remain. Additionally, methanol escapes from the back-end with the vapors off the 07.20. As a result, the concentration of water in the circulating methanol increases with time-on-stream. Water is a poor solvent for CO2 and the effectiveness of the circulating liquid will diminish with time. Therefore, it will be necessary to increase 10.80 flow and/or increase plant purge during any given run. The composition of the methanol solvent in the CO2 removal section will be monitored regularly and, if necessary, methanol will be drained out (via the 22.11 and 22.15) and fresh methanol will be added using the 10.85 pump.

WATER FREEZE-OUT IN 21.10 TUBESIDE: Although unlikely, it is possible for the water contained in the 22.10 overhead to plate-out on the tubes of the 21.10. This process, if it occurs at all, will be slow. Indications of freeze-out include: excessive pressure drop, colder temperature at TI-233, and warmer temperature at TI-188. Corrective action is to operate the 10.85 pump to inject methanol into the 21.10 tubeside inlet.

7. Upon completion of the PVSs, this test run is done. De-pressurize the plant and proceed with shut-down.

ANALYTICAL COMMENTS:

1. Catalyst sampling requirements:

   - slurried catalyst at end-of-run.

   Exact quantities to be determined by operations, process, and research.

2. Continuous composition sampling requirements (GC):

   - fresh feed,
   - reactor in, (sample point 4 and 15, when injecting alcohols)
   - reactor out,
   - 22.10 overhead,
   - recycle
   - 07.20 offgas

3. Periodic composition sampling requirements (GC):

   - 22.11 off-gas (frequency to be determined by operations & process)

   Periodic composition sampling requirements (LC):

   - higher alcohol product (every 8 hours)
   - 10.80 suction liquid (once every two days or as required)

4. Flow measurement requirements:

   - fresh feed,
   - 10.95 pump
   - reactor in,
   - reactor out,
   - 22.10 overhead,
   - recycle,
   - purge,
   - 22.11 off-gas,
   - 07.20 off-gas,
   - mixed alcohol product (by level)
   - 10.80 flow,

REFERENCES:

1. TEST AUTHORIZATION #20 - Procedures for reactor standby during shutdown.

2. STANDARD STARTUP PROCEDURES FOR MeOH-ONLY OPERATION

3. STARTUP PROCEDURES FOR OPERATION WITH CO2 REMOVAL.

RUN PLAN FOR SPRING 1994 IBOH DEMONSTRATION

RUN			AF-A6	AF-R10.1	AF-R10.2	AF-R10.3	AF-R10.4	AF-R10.5	AF-R10.6	AF-R10.7	AF-R10.8	AF-R10.9	AF-R10.10	AF-R10.11
Description	---	---	Reduction	IBOH-1	IBOH-2	IBOH-3	IBOH-4	IBOH-5	IBOH-6	IBOH-7	IBOH-8	IBOH-9	IBOH-10	IBOH-11
Duration	---	days	2	2	1	2	2	2	1	1	1	1	1	2
Syngas	---	---	---	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	TEXACO
Inlet Space Velocity	---	sL/kg-hr	1,200	5,000	3,000	8,200	8,200	8,200	3,000	5,000	5,000	5,000	5,000	3,000
Reactor Pressure	PIC-1247	psig	100	750	750	750	1,300	1,735	1,735	1,300	1,300	1,300	750	1,300
REACTOR														
Pressure	PIC-1247	psig	100	750	750	750	1,300	1,735	1,735	1,300	1,300	1,300	750	1,300
Temperature	TI-1233	F	---	572	572	572	572	572	572	572	572	572	572	572
Heat Duty		MM BTU/hr	---	0.53	0.52	0.59	1.48	1.97	1.01	1.12	0.85	1.03	0.53	0.59
Inlet Superficial Velocity	---	ft/sec	0.640	0.611	0.366	1.002	0.583	0.438	0.160	0.355	0.390	0.366	0.611	0.213
Outlet Superficial Velocity	---	ft/sec	---	0.517	0.348	0.859	0.461	0.329	0.115	0.275	0.327	0.291	0.517	0.168
Liquid Level	LI-2142	% span	80 - 90%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%
Catalyst Load	---	lb	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107
Cat Weight Fraction	---	%	40.0%	40.2%	39.1%	41.5%	42.0%	42.2%	39.8%	40.7%	41.1%	42.0%	40.2%	39.6%
Vapor Void Fraction	---	%	25 - 30%	40.2%	36.4%	44.3%	46.0%	46.9%	38.7%	41.7%	43.2%	44.4%	40.2%	38.2%
FEED FLOWS														
LP H2	FIC-101	scfh	321	9,440	7,082	13,518	13,936	9,450	12,104	15,799	9,131	13,932	9,440	10,605
CO	FIC-104	scfh	481	10,016	9,086	12,582	20,790	25,311	13,715	15,670	14,682	15,712	10,016	9,476
CO2	FIC-107	scfh	114	31	0	0	0	0	607	0	0	659	31	0
N2	FIC-111	scfh	22,003	49	54	56	74	73	71	60	66	65	49	75
01.10 Total Flow	FI-726	scfh	22,919	19,536	16,222	26,157	34,800	34,835	26,497	31,529	23,879	30,367	19,536	20,156
HP H2	FIC-1200	scfh	0	0	0	0	10,019	20,322	0	0	0	0	0	0
01.20 Recycle	FIC-246	scfh	0	76,849	40,926	131,169	112,966	102,604	31,305	64,926	72,840	66,495	76,849	38,114
10.95 PUMP INJECTION														
Total Flow	FI-1221	gpm	zero	zero	zero	zero	zero	zero	zero	zero	2.14	0.70	zero	zero
MEOH	---	wt%	---	---	---	---	---	---	---	---	83.00%	69.50%	---	---
C2OH	---	wt%	---	---	---	---	---	---	---	---	5.00%	10.50%	---	---
C3OH	---	wt%	---	---	---	---	---	---	---	---	12.00%	20.00%	---	---
											100.00%	*100.00%*		
REACTOR FEED														
Target Feed Temp	TI-1253	F	---	437.8	443.2	431.7	429.8	429.6	429.5	429.4	400.0	400.0	437.8	431.5
Feed Dewpoint	---	F	---	-6.6	-9.3	-5.2	-9.6	-14.3	-15.1	-9.4	239.5	178.4	-6.6	92.9
Total Dry Flow	FI-1216	scfh	22,920	96,375	57,133	157,308	157,750	157,727	57,770	96,434	96,692	96,859	96,375	58,268
H2	---	mol%	1.40%	29.73%	30.05%	29.87%	29.78%	29.79%	29.72%	29.71%	27.03%	28.73%	29.73%	34.37%
CO	---	mol%	2.10%	65.41%	66.11%	65.70%	65.52%	65.53%	65.38%	65.37%	59.46%	63.21%	65.41%	50.09%
N2	---	mol%	96.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%
CO2	---	mol%	0.50%	2.97%	1.91%	2.99%	2.91%	2.98%	2.97%	2.88%	2.68%	2.87%	2.97%	12.77%
MEOH	---	mol%	0.00%	0.04%	0.03%	0.04%	0.04%	0.03%	0.03%	0.03%	7.89%	2.32%	0.04%	0.55%
ETOH	---	mol%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.33%	0.24%	0.00%	0.01%
PROH	---	mol%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.61%	0.35%	0.00%	0.00%
C1	---	mol%	0.00%	0.84%	0.99%	0.53%	1.04%	0.95%	1.15%	1.15%	1.23%	1.15%	0.84%	0.73%
			100.00%	*99.98%*	*100.09%*	*100.13%*	*100.28%*	*100.28%*	*100.25%*	*100.14%*	*100.22%*	*99.87%*	*99.98%*	*99.53%*

RUN PLAN FOR SPRING 1994 IBOH DEMONSTRATION

RUN			AF-A6	AF-R10.1	AF-R10.2	AF-R10.3	AF-R10.4	AF-R10.5	AF-R10.6	AF-R10.7	AF-R10.8	AF-R10.9	AF-R10.10	AF-R10.11
Description	---	---	Reduction	IBOH-1	IBOH-2	IBOH-3	IBOH-4	IBOH-5	IBOH-6	IBOH-7	IBOH-8	IBOH-9	IBOH-10	IBOH-11
21.11 Feed/Product Exchanger														
Feed Inlet Temp	TI-1257	F	---	154.1	161.2	147.3	150.0	150.0	150.0	150.0	120.0	120.0	154.1	150.0
Feed Outlet Temp	TI-1263	F	---	492.8	492.0	492.3	492.2	492.2	492.2	492.2	492.0	492.2	492.8	492.2
Total Feed to 02.63 Temp	TI-1216	F	---	437.8	443.2	431.7	429.8	429.6	429.5	429.4	475.8	423.7	437.8	431.5
Reactor Eff. Inlet Temp	TI-1262	F	---	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0
Reactor Eff. Outlet Temp	TIC-1260	F	---	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0
Reactor Eff. Dew Temp	---	F	---	198.9	219.5	186.8	238.7	262.5	275.7	247.3	279.8	263.8	198.9	245.7
REACTOR EFFLUENT														
Total Flow	FI-196	scfh	---	86,318	49,328	143,328	131,379	124,583	43,512	78,551	93,645	83,183	86,318	47,959
H2	---	mol%	---	23.47%	22.60%	24.22%	18.26%	14.48%	13.20%	17.47%	22.24%	18.88%	23.47%	22.64%
CO	---	mol%	---	65.14%	64.82%	65.96%	65.95%	66.18%	63.55%	64.92%	55.69%	61.33%	65.14%	47.28%
N2	---	mol%	---	0.89%	0.82%	0.76%	0.62%	0.67%	0.73%	0.79%	0.66%	1.15%	0.89%	1.19%
CO2	---	mol%	---	5.37%	6.03%	4.54%	6.13%	7.18%	10.72%	7.42%	8.00%	7.89%	5.37%	18.30%
MEOH	---	mol%	---	2.98%	2.79%	3.05%	6.25%	8.33%	6.93%	5.92%	8.60%	6.61%	2.98%	7.24%
ETOH	---	mol%	---	0.13%	0.14%	0.11%	0.26%	0.38%	0.47%	0.31%	0.59%	0.43%	0.13%	0.32%
PROH	---	mol%	---	0.10%	0.14%	0.07%	0.19%	0.28%	0.50%	0.27%	0.89%	0.60%	0.10%	0.21%
C4OH	---	mol%	---	0.03%	0.04%	0.02%	0.05%	0.07%	0.15%	0.07%	0.22%	0.17%	0.03%	0.05%
IBOH	---	mol%	---	0.16%	0.30%	0.10%	0.18%	0.22%	0.46%	0.27%	0.41%	0.37%	0.16%	0.16%
C5OH+	---	mol%	---	0.26%	0.51%	0.16%	0.29%	0.34%	0.70%	0.43%	0.54%	0.52%	0.26%	0.24%
C1	---	mol%	---	1.03%	1.31%	0.65%	1.35%	1.32%	1.82%	1.56%	1.54%	1.53%	1.03%	1.20%
			---	*99.54%*	*99.50%*	*99.62%*	*99.53%*	*99.44%*	*99.22%*	*99.43%*	*99.38%*	*99.46%*	*99.54%*	*98.84%*
PRODUCT RECOVERY														
Syngas to Backend Flow	FI-682	scfh	---	83,669	47,596	139,238	122,473	112,907	39,498	73,105	83,040	76,086	83,669	None
22.11 to Flare Flow	FI-237	scfh	---	94	75	126	360	525	243	252	497	337	94	304
Main Flare Flow	FI-245	scfh	---	3,867	4,004	4,431	4,157	4,253	3,867	3,960	4,292	3,867	3,867	5,605
Product Flow	---	gpd	---	982	725	1,417	3,037	3,941	1,504	1,957	3,884	2,644	982	1,267
BACK-END														
MEOH Circulation	FIC-814	gpm	None	9.17	7.90	11.56	14.66	14.83	8.46	10.43	13.94	13.88	9.17	None
MEOH to 07.10 Temp	TI-814	F	---	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	---
07.10 OH Temp	TI-1275	F	---	-4.6	-7.2	-3.1	-3.8	-2.9	-5.5	-3.5	-4.2	-5.8	-4.6	---
07.20 OH to Flare Flow	FI-7291	scfh	---	2,288	2,377	2,419	4,332	5,046	4,079	3,652	5,356	5,218	2,288	---
07.20 Reboiler Temp	TIC-7339	F	---	296.8	296.5	296.9	296.4	296.3	296.4	296.4	296.5	296.6	296.8	---
07.22 Steam Pressure	PIC-7338	psig	---	235	235	235	235	235	235	235	235	235	235	---
07.22 Steam Usage	FI-7338	lb/hr	---	592.8	592.5	576.8	664.0	692.7	679.2	650.4	710.9	692.0	592.8	---
21.80 CO2 Usage	---	TPD	---	36.18	33.82	36.29	36.50	36.83	35.72	35.49	36.15	33.60	36.18	---
Total CO2 Usage	Fd+21.80	TPD	0.16	36.22	33.82	36.29	36.50	36.83	36.55	35.49	36.15	34.50	36.22	0.00
RECYCLE FEED														
H2	---	mol%	---	25.00%	24.65%	25.51%	20.38%	16.77%	16.18%	19.80%	26.80%	22.14%	25.00%	24.73%
CO	---	mol%	---	68.99%	70.09%	69.20%	73.09%	76.07%	76.84%	72.95%	66.39%	71.17%	68.99%	51.71%
N2	---	mol%	---	0.94%	0.90%	0.80%	0.69%	0.77%	0.89%	0.89%	0.80%	1.34%	0.94%	1.31%
CO2	---	mol%	---	3.69%	2.67%	3.58%	4.06%	4.58%	3.55%	4.27%	3.90%	3.31%	3.69%	19.52%
MEOH	---	mol%	---	0.05%	0.04%	0.05%	0.05%	0.05%	0.05%	0.05%	0.05%	0.04%	0.05%	0.84%
C1	---	mol%	---	1.05%	1.38%	0.64%	1.45%	1.46%	2.12%	1.71%	1.78%	1.72%	1.05%	1.12%
			---	*99.72%*	*99.72%*	*99.78%*	*99.73%*	*99.70%*	*99.62%*	*99.67%*	*99.72%*	*99.73%*	*99.72%*	*99.22%*

JMRepasky 3/3/94

APPENDIX B

OVERALL RUN CHRONOLOGY

3/10/94 Thursday — 12:45 Load reactor with ~20 ft3 of Drakeol oil from the prep tank. Level is approximately 123". With no gas flowing, nuke reading in oil phase is 3.6 counts.

Close the shutter and nuke reading drops to 0.000. Therefore, NO background radiation with this source.

Start heating oil with N2. Beginning of carbonyl burnout prep.

3/11/94 Friday — Heatup overnight. Level at 456" at 477 F and 750 psig. N2 gas flowing.

12:15 Bringing syngas into the plant. Reactor at 493 F and 755 psig. Carbonyl analysis starts.

16:29 0.114 ppm Fe at sample point #15 (not seeing any Ni)
16:47 0.085 ppm Fe at sample point #15
17:01 0.059 ppm Fe at sample point #15
17:30 0.052 ppm Fe at sample point #4

3/13/94 Sunday — 12:00 Because of initial startup problems with the 10.52 oil return pumps, steady-state level control in reactor not achieved until now.

13:00 Nuke scan at 20,078 SCFH (once-thru). Level at 151" in 27.20
P = 744 psig; T = 481 F; Composition = 34% H2, 66% CO

14:00 Shutdown test. Level at 86". (Nuke reading at 81" is 8.2 cps/1000)

14:10 Re-start gas flow and start 01.20 to increase flow to 27.20.

15:00 Nuke scan at 52,030 SCFH. Level at 278" in 27.20.
P = 751 psig; T = 481 F

17:00 Nuke scan at 115,268 SCFH. Level at 291" in 27.20.
P = 748 psig; T = 475 F

20:50 Nuke scan at 155,619 SCFH. Level at 291.5" in 27.20.
P = 763 psig; T = 474 F

3/14/94 Monday — 08:30 Nuke scan at 15,598 SCFH. Level at 118" in 27.20.
P = 751 psig; T = 556 F; Composition = 31% H2, 68% CO

09:40 Nuke scan at 47,583 SCFH. Level at 255" in 27.20.
P = 751 psig; T = 558 F; Composition = 31% H2, 68% CO

11:00 Nuke scan at 78,044 SCFH. Level at 246" in 27.20.
P = 755 psig; T = 561 F

12:00 Nuke scan at 101,483 SCFH. Level at 263" in 27.20.
P = 757 psig; T = 565 F

13:35 Nuke scan at 141,073 SCFH. Level at 313" in 27.20.
P = 753 psig; T = 563 F; Composition = 30% H2, 68% CO
Close to 40 psi ΔP in reactor at this flow.

16:50 Nuke scan at 36,190 SCFH. Level at 217" in 27.20.
P = 1305 psig; T = 570 F
This was minimum controllable flow at these conditions
(without going to once-thru).

18:07 Nuke scan at 81,127 SCFH. Level at 343" in 27.20.
P = 1300 psig; T = 567 F

19:28 Nuke scan at 134,719 SCFH. Level at 259" in 27.20.
P = 1303 psig; T = 558 F

20:40 Nuke scan at 108,347 SCFH. Level at 240" in 27.20.
P = 1749 psig; T = 563 F; Composition = 35% H2, 64% CO

22:04 Nuke scan at 185,028 SCFH. Level at 246" in 27.20.
P = 1751 psig; T = 548 F
This was max flow at these conditions.

22:50 Shutdown test. Level at 63". (Nuke reading at 42" is 9.7 cps/1000)

23:51 Nuke scan at 37,307 SCFH. Level at 125" in 27.20.
P = 1753 psig; T = 564 F

3/15/94 Tuesday

07:20 Syngas backed out to 01.10 / 01.20.

07:45 Syngas out of compressors. Flowing with N2 to clean up.

07:50 Cooling plant at 60 F/hour from 482 F.

14:30 Blowdown plant and drain oil. There was evidence of significant fines in the oil, which could be contributing to carbonyl generation! Transferred 200 gallons of fresh oil to prep tank for another pass at carbonyl burnout.

15:45 Transfer fresh oil to reactor. Level at 199" (5.7 cps/1000 at 186") at 104 F and 22 psig.

16:15 Started heatup at 1 F/minute with 02.63 on. Once-thru at 20,000 SCFH.

19:15 GC composition: 33% H2, 65% CO, 2% N2.

19:25 Changed SP1-MW in Bailey to 19.43.

22:00 Utility oil temperature to 495 F and holding. Reactor temperature still increasing.

22:30 Plant lined out at 482 F and 750 psig.

22:52 Nuke scan at 21,977 SCFH (once-thru). Level at 370" in 27.20
P = 752 psig; T = 482 F

3/16/94 Wednesday 06:53 Carbonyl analysis starts. GC composition: 32% H2, 66% CO, 2% N2. 20,000 SCFH (once-thru) at 482 F and 750 psig. 02.63 heater is on.

06:58 0.017 ppm Fe at sample point #3A (not seeing any Ni)
07:12 0.016 ppm Fe at sample point #3A
07:18 0.016 ppm Fe at sample point #3A
07:23 0.007 ppm Fe at sample point #15
07:31 0.009 ppm Fe at sample point #15
07:36 0.009 ppm Fe at sample point #15
07:46 0.002 ppm Fe at sample point #4
07:52 0.002 ppm Fe at sample point #4

12:45 Plant lined out at 1750 psig and 572 F.

Load Drakeol oil to 28.30 prep tank:
Drum #1 = 368 lbs
Drum #2 = 374 lbs
Drum #3 = 380 lbs
Drum #4 = 378 lbs
Drum #5 = 375 lbs
Total = 1875 lbs

18:00 Began recycle to bring plant to 58,000 SCFH.

20:10 Plant lined out at 58,000 SCFH, 1750 psig, and 572 F.

20:25 Nuke scan at 58,683 SCFH. Level at 407" in 27.20.

P = 1751 psig; T = 572 F

23:55 High pressure carbonyl burnout complete. Begin backing out syngas and cooling at 60 F/hour.

3/17/94 Thursday

07:09 Start loading methanol catalyst to prep tank (160 F). Nice sunny day.

Drum #1 = 238 lbs
Drum #2 = 421 lbs
Drum #3 = 411 lbs
Drum #4 = 180 lbs
Total = 1250 lbs

09:00 Catalyst loading complete.

11:48 Prepare flush oil (283 lbs)

12:00 End of transfer to reactor. Level is 291". Pour flush oil into prep tank. Walls very clean, no puddles evident at bottom of tank before adding flush oil.

12:45 Begin transfer of flush oil to reactor. Level ~335".

13:00 Raise reactor pressure toward 50 psig. Level at 355".

14:25 Start bringing gas to 27.20. Starting composition:

1.2% H_2
94.4% N_2
3.2% CO
1.0% CO_2

Average reactor T=183 F (180 F at bottom). PIC-1247 = 94.9 psig.

No GC points reading into the DEC !

14:47 Flare malfunction (high N_2 content in vent gas). Dean rebooting GC computer.

15:10 Flare rupture disk blew. Stopped gas flow to reactor.

17:05 Nuke scan. Level at 309".

19:25 Start bringing reduction gas to 27.20 again. Level at 385".

19:29 Flare malfunction at 22,000 SCFH.

19:30 Flare relit with flow at 20,000 SCFH.

19:32 Starting composition: (G06) 1.04% H2
1.67% CO
96.73% N2
0.71% CO2

Flow at 19,800 SCFH, 83 psig, level at 423". Waiting for another GC scan.

19:40 New composition: (D06) 1.18% H2
1.92% CO
96.17% N2
0.70% CO2

19:48 Starting heatup.

20:15 Average reactor temperature at 190 F. Still no evidence of uptake yet.

20:30 Lost flare again. Flow had crept up to 21,500 SCFH. Backing flow out to try and relight.

21:00 27.20 reactor temperature has levelled off at 194 F.

21:06 Flow stopped to 27.20. FALL-299.

21:20 Flare relit !

21:27 Re-established syngas flow to reactor.

21:45 Syngas flow set at 13,500 SCFH on FI-299.

22:00 Syngas flow up to 14,300 SCFH. Restarted heatup.

New composition: (G06) 1.06% H2
1.67% CO
96.29% N2
0.78% CO2

22:10 False start on the heatup. The heaters had tripped out during the flare malfunction upset. Now they're racked back in.

23:11 G03 lost its CO peak. GC's Gary and Dennis show good agreement otherwise.

24:00 Utility oil temperature at 232 F. Reactor temperature at 222 F. Still evidence of uptake in the outlet concentrations, but temperature hasn't taken off yet.

3/18/94 Friday

02:05 Nuke scan. Level at 413". Holdup=26.4%.

03:45 Noticed that GC Gary hadn't been updating at all for over an hour. Checked the lab, and the unit was out of paper. Replaced paper and got a new scan (still without CO) at 4:05.

06:10 Told Matt to stop temperature ramp because preliminary calculation showed uptake was falling below standard curve.

06:30 Matt restarted temperature ramp. E-beth wasn't calculating uptake right.

07:47 Dean took GC Gary off-line to investigate missing CO peak.

07:51 Nuke scan. Level at 390". Holdup=25.9%.

09:10 GC Gary calibrated and back on-line. GC Dennis off-line.

10:25 GC Dennis back on-line. (10:24 reading was calibration gas.)

12:15 Reactor temperature at 392 F and holding.

15:30 CO inlet concentration has dropped from about 1.6% to about 1.0% in the last hour. Henry adjusted back up.

16:00 Downloaded and plotted cumulative reduction data. Everything still looks OK.

17:25 Nuke scan. Level at 354". Holdup=26.8%.

21:00 Downloaded and plotted another set of cumulative reduction data.

3/19/94 Saturday

00:30 Begin 15 F/hr reactor temperature ramp toward 464 F.

01:30 Matt increased the CO flow slightly and decreased the CO2 in the feed to match desired composition.

03:00 Nuke scan. Level at 328".

03:15 Matt fired up the 02.62 reduction heaters to help warm up the reduction feed gas during the temperature ramp.

03:45 Nuke scan had lots of variability in the slurry region from 318" down. Readings do not settle down but constantly flip values so that the recorded number is +/- 0.5 or so.

05:40 Reach 464 F. Begin hold period.

06:42 Reduction complete. Removing syngas from plant. Reactor will be cooled at 30 F/hr to target 400 F. Low N2 bubbling through reactor.

06:43 TI-1235 (J7 nozzle in reactor) taken off-line for recalibration.

08:00 Oil added to 27.14 (12 nuts).

09:10 Begin blending syngas.

09:22 Stop N2 to 27.20 to take slurry sample. Level at 260" with no gas flow.

10:20 Syngas flow to reactor begins. Plant is pressuring up (PV-201 shut).

10:45 27.20 Avg. T = 408 F, P = 616 psig. Level at 288" and holding. Pressure is ramping linearly, but temperature has leveled off.

10:53 Starting to increase T. We are at pressure (755 psig). Flow has been about 23,700 SCFH since startup.

11:12 Begin recycling a little. Avg. T = 413 F. Level coming up to 305".

11:22 Start seeing MeOH in reactor effluent (SP3A).

11:45 Starting to take oil from 21.11 to reactor. Level at 311". Avg. T = 425 F.

11:49 Start increasing fresh feed (about 20%) and recycle. Fresh feed is 28,600 SCFH. Reactor feed is 72,600 SCFH. Level at 318".

11:57 GC's are now shooting proper sequence.

12:02 More recycle. Reactor feed is 105,000 SCFH. Level at 347".

12:21 Bringing HP H2 into plant.

12:57 PDSHH-1200 on HP H2 tripped.

13:20 27.20 Avg. T = 457 F, P = 749 psig. Level at 373".

13:28 About 150 gallons of liquid in 22.10. Opening LV-220 off 22.10.

14:35 Reactor temperatures cool down to 460 F after U.O. fin fan is turned on.

14:50 TIC-293 on U.O. circuit changed to control off of TI-166 (outlet of fin fan after mixing) instead of TI-293.

15:05 GC Dennis is off-line for recalibration of MeOH peak.

15:49 G05MW is being thrown off by ~3% propane peak which Dean believes is H2O being mistaken as propane. Problem has existed for ~3 1/4 hours. Dean claims to have solved the problem.

16:13 GC Dennis is back on-line. Both GC's should be OK for awhile now.

16:17 Henry pulls product sample from 22.11. Sample appears highly contaminated with oil (about 30% by volume). Yellow (oil) on bottom. 5.99% MeOH on SP3A.

16:32 E-beth calculates 51.4 wt% slurry ! Level down to 317". We will now start bringing level up by pumping oil from 27.14 / 21.11.

16:38 CW shut off to 01.34.

17:15 TI-1235 & TI-1233 were checked and found to be displaying 4 F high in the Bailey (compared to calibrated instrument).

18:57 Level up to 420". Moved setpoint to 438".

19:32 G03 (SP3A) update. MeOH concentration still dropping. 3.68% now, was about 5% on last scan.

20:00 D03 (SP3A) update. MeOH concentration is 2.67%, down from 4%. Level up to 465". Moved setpoint to 480".

20:27 27.20 level at 480" and holding in automatic.

20:40 HP H2 line pressure has dropped 20 psi in the last 4 hours (860 to 840), 10 psi in the last hour alone. This makes feed flow difficult to control.

G03 (SP3A) update came thru. MeOH at 3.79%.

20:55 PDSHH-1200 (HP H2) tripped again, simultaneous with AAL-7351 (LEL monitor) - same as this afternoon at 13:00.

22:05 D03 (SP3A) update. MeOH at 3.86%.

22:31 Nuke scan. Calc's show 42 wt% slurry and 40 vol% gas holdup.

23:25 Matt grabbed a liquid sample from 22.11. About 15% oil by volume. Could not detect a phase separation in the 22.16 sight glass.

23:45 Liquid logs indicate oil loss rate is 138 gal/day (over previous 2 hrs) and liquid product rate is 3312 gal/day. This suggests 4% oil in the product.

3/20/94 00:20 Matt dropping TIC-1260 (21.11 product outlet) setpoint from 260 F to Sunday 250 F to see if that affects the oil in the MeOH problems.

02:00 Liquid logs indicate oil loss rate is 132 gal/day and liquid product rate is 3430 gal/day (very similar to 23:45 results).

02:36 G03 (SP3A) update. MeOH at 3.71%.

03:00 Matt took a liquid sample from 22.11. Oil in bottom similar to the last sample. Reduced TIC-1260 setpoint to 245 F.

03:20 22.16 day tank (117") transfer to trailer.

04:00 Liquid logs indicate oil loss rate is 120 gal/day.

04:30 Nuke scan. Calc's show 41 wt% slurry and 37 vol% gas holdup. Readings relatively steady throughout the liquid.

04:50 PDSHH-1200 and AAL-7351 alarms go on and off continuously.

05:30 PDSHH-1200 and AAL-7351 are back at it again ! It trips and resets itself continuously.

08:30 HP H2 flow rose over the last 2 hours, and feed is now too rich in H2. Henry is decreasing flow.

08:56 GC Gary is off-line for calibration.

09:39 GC Gary back on-line. GC Dennis taken off-line.

10:20 TIC-1260 found to be reading 10-20 F too high in this range. TIC-1260 back in control at 250 F setpoint.

10:38 GC Dennis back on-line.

14:20 22.16 day tank transfer (100 1/8" to 19 5/8"). Begin cleaning oil out of product collection system by pushing all liquids thru to 22.16.

15:46 Moved TIC-1260 setpoint from 250 F to 245 F.

17:03 Henry grabbed a liquid sample from 22.11. Oil still present but significantly reduced.

17:10 D03 shot lost its MeOH peak and CO2 seems abnormally low.

18:06 More PDSHH-1200 alarms. HP H2 line pressure has dropped 35 psig in last 1/2 hour. Matt called the main plant and found out we're losing HP H2 altogether. 01.10 cannot handle full flow with all LP H2, so Matt is backing off on total flow to maintain composition.

18:11 D03 still missing its MeOH peak. D04 isn't totalizing real well either.

18:56 Feed composition: (G04) 33.53% H2
(pretty much on target) 50.10% CO
0.64% N2
14.96% CO2

20:31 Nuke scan. 40 wt% slurry and 35 vol% gas holdup. Level at 478".

3/21/94 Monday

01:00 Henry blew down K.O. pots on GC's. Very little liquid came out. Grabbed a liquid sample from 22.11. There is still an oil layer in the pint jars (~3/8").

01:05 Go to reduce setpoint on TIC-1260 further and discover that the TIC is closed and temperature has been running above setpoint at 251-252 F since the HP H2 tripped out at 18:00. Good news for the 01:00 sample.

01:10 22.16 day tank transfer to trailer (91 1/2" to 20").

02:10 Start CW to 01.34 cooler to bring down TI-1257 (feed inlet to 21.11) from 150 F to ~132 F where it was running before the H2 upset.

03:25 TI-1257 leveled out at 121 F. TIC-1260 reading 238 F. Valve opening up with setpoint at 245 F.

04:30 Nuke scan. 42 wt% slurry and 39 vol% gas holdup. Flow has dropped off to 123,000 SCFH. Start to put HP H2 back into the plant to bring composition and flow in line.

05:15 Plant is swinging.

07:10 GC Dennis is off-line for calibration.

Level is up in the reactor to 510". Plant has been swinging.

10:36 GC Dennis back on-line.

11:52 HYCO plants went down next door, so we lost CO. Shut off all flow to 27.20. CO and H2 blocked in.

Nuke slump test. 40 wt% slurry and 6 vol% gas holdup. Level at 322".

12:40 Cooling down plant at 50 F/hr, down to 300 F. Lining up N2 plus a little LP H2 (to maintain a reducing atmosphere) to feed to 27.20 once-thru at about 10,000 SCFH. Level up to 334".

13:40 Lost CW from HYCO plant, and 01.10 keeps tripping on high discharge temperature. Can't run compressors. We will blow down plant to 100 psig and run in reduction mode under house line pressure.

15:00 Established N2 flow at about 8500 SCFH. D03 is recalibrated for reduction gas and shooting SP-13B continuously.

15:28 Feed composition: (D03) 2.1% H2
(looks good) 0.5% CO
97.8% N2
0.0% CO2

15:50 CO is back. CW pumps should be back on-line by 17:00, so holding temperature here at about 390 F. Maintenance preparing to pressure test new HP H2 tie-in line.

17:30 Reduction circuit to reactor shut off. Pressure test complete on HP H2 line. Will start plant on GC Gary alone until GC Dennis can be recalibrated for regular op's. Reactor level at 338" with no flow.

17:51 Syngas re-introduced to plant once-thru at about 20,000 SCFH.

17:55 Starting heat up. P = 139 psig. Level at 369".

18:20 27.20 Avg. T = 424 F, P = 722 psig. Level at 372". GC Gary is on-line.

18:30 Starting recycle flow from 01.20.

18:40 Compressor trip. High discharge temperature.

19:00 Recycle flow re-established.

19:15 GC Dennis back on-line.

20:05 Flow crossing thru 100,000 SCFH. Reactor level up to 525", so holding flow for a while. Both GC's reading consistent feed compositions.

	38.6%	H2
(D04 - 19:41)	50.9%	CO
	1.3%	N2
	9.2%	CO2

20:30 22.16 day tank transfer to trailer 103" to 19 3/4" (1467.5 gallons).

21:30 Increasing recycle flow again.

3/22/94 Tuesday

01:25 27.20 has lined out at steady operating conditions. Level in auto at 480". T = 481 F, P = 750 psig, F = 145,000 SCFH. MeOH in product at 9.6%!

04:35 Line pressure on HP H2 has been rising steadily. Now up to 784 psig.

04:45 Re-introduced CO2 to thye fresh feed since last 2 SP4 shots have been 10.8% and 9.7%.

05:32 GC Gary 3A analysis lost its MeOH peak (plant has been steady). GC Gary analysis on feed showed 11.6% CO2 - OK.

08:48 22.16 day tank transfer to trailer 121" to 19 7/8" (1782.5 gallons).

10:15 Matt took a liquid sample from 22.11. Slurry sample also taken.

10:30 GC Gary back on-line. GC Dennis taken off-line.

10:45 Nuke scan. Calc's show 45 wt% slurry and 47 vol% gas holdup.

12:18 GC Dennis is back on-line.

14:40 Regression of liquid logs indicates that liquid product rate was 119.5 gal/hr before the restart and 143.8 gal/hr after.

14:45 Nuke scan. Calc's show 45 wt% slurry and 47 vol% gas holdup.

16:15 Liquid logs indicate product accumulation rate equivalent to 14:40 calc. Still no oil loss.

17:00 Took a liquid product sample from 22.11. It looks completely clear.

18:55 22.16 day tank transfer to trailer 101 1/4" to 20" (1432.25 gallons).

19:35 Nuke scan. Calc's show 45 wt% slurry and 47 vol% gas holdup.

20:55 Lined up 27.14 for AF-R9.2. Took a liquid sample from 22.11. It was raining at the time, so water content could be artificially high (?). Again, sample was completely clear.

21:04 Started changing conditions to AF-R9.2

3/23/94 Wednesday 00:05 Still helping Henry line-out on compositions.

00:35 D03 shows 16.4% MeOH in reactor effluent. Wow!

01:30 Still tweaking H2 in reactor feed.

02:20 GC Gary is consistently reading lower (up to 2.5%) than GC Dennis on MeOH in reactor effluent.

03:10 Last 2 GC shots have been good on feed composition.

04:00 Took a liquid product sample from 22.11.

05:05 Nuke scan. Calc's show 39 wt% slurry and 33 vol% gas holdup.

06:45 22.16 day tank transfer to trailer 116" to 20" (1692.2 gallons).

07:54 GC Dennis is off-line for calibration.

Sample points 6 and 8 have had little or no flow for a while. Problem corrected. Also, N2 in fresh feed is 0% often, and since recylce (SP8) is unreliable, it is difficult to close a mass balance. Adding N2 to the 01.10 feed streams. Previously we relied on N2 in the CO stream to be sufficient, but CO stream is now coming from the PSA's and N2 may be different now.

09:30 GC standards have not yet been switched to Kingsport gas standard. We need to let Dean know when we change conditions. Conclusion: data on AF-R9.2 is probably questionable to this point.

10:35 GC Dennis back on-line. GC Gary goes off-line for calibration.

12:15 GC's are back on-line.

12:25 Backed out the fresh N2 in the feed. Latest SP4 reading was 6%.

15:00 GC's differing by 3% on effluent MeOH concentration. Dean says both are very accurate in the 6% range (calibration level) and that the error will be

significant at the concentrations we're running. It is also not unusual to be totalizing >100%.

16:45 HP H2 line pressure dropped from 760 to 680 psig over last 1 3/4 hours. Feed flows and compositions starting to swing some.

16:50 AJ took a liquid sample from 22.11 (both sample bomb and glass jar).

17:11 D03 shows 18.2% MeOH (see entry at 15:00 above).

17:30 Nuke scan. Calc's show 39 wt% slurry and 34 vol% gas holdup.

18:00 Discovered reactor pressure was moved up to 750 psig about 1 3/4 hours ago. Matt returning setpoint to 735 psig.

18:42 22.16 day tank transfer to trailer 111 1/2" to 20" (1612.5 gallons).

22:00 Henry took a liquid sample from 22.11 (both sample bomb and glass jar).

22:25 Dean looked at GC's. Gary and Dennis match pretty well on H2 and CO2 but differ on CO and N2. His comment was that CO and N2 come out very close and GC Dennis is getting poorer separation between the peaks. He's more inclined to believe GC Gary.

22:35 Dean confirmed that GC Dennis is integrating wierdly. He will try to fix it.

Feed composition has been drifting slightly for some time, probably since the HP H2 line pressure problem this afternoon. Henry is making a move to increase the H2 and decrease the CO2.

22:50 Dean thinks he's fixed GC Dennis. First set of points thru looked good.

3/24/94 Thursday

04:00 Took a liquid sample from 22.11 (both sample bomb and glass jar).

06:00 22.16 day tank transfer to trailer (1612.8 gallons).

08:30 Matt took a liquid sample from 22.11 (both sample bomb and glass jar).

08:55 Data period AF-R9.2 ends here. 21.11 testing period begins here.

09:10 Moving to Test A target: Kingsport gas, T = 482 F, P = 750 psig, SV = 6500 (F = 140,400 SCFH).

10:25 Start 21.11 Test A.

16:27 Moving to Test B target: Kingsport gas, T = 482 F, P = 750 psig, SV = 10,000 (F = 215,800 SCFH).

18:35 Moving to Test E target: Kingsport gas, T = 482 F, P = 850 psig, SV = 9000 (F = 194,100 SCFH).

MeOH samples are very cloudy. After settling a few hours, there are oil bubbles in them. Decide to run here until 3-4 AM to let things settle out.

22:39 SD-1 plant trip. High discharge temperature on 01.20 (180 F). Decide to stay shutdown. Start cooling plant.

3/25/94 Friday

10:00 Level at 270" in reactor with no flow. T = ~150 F. Draining slurry.

Load Drakeol oil to 28.30 prep tank:

Drum #1 = 378 lbs
Drum #2 = 395 lbs
Drum #3 = 384 lbs
Drum #4 = 311 lbs
Drum #5 = 191 lbs
Total = 1659 lbs

16:25 Start loading IBOH catalyst to prep tank (152 F).

Drum #1 = 272 lbs
Drum #2 = 273 lbs
Drum #3 = 273 lbs
Drum #4 = 273 lbs
Drum #5 = 15 lbs
Total = 1106 lbs

17:50 Catalyst loading complete. Reactor currently being flushed with oil under N2 flow at about 200 F.

3/26/94 Saturday

07:40 Starting to drain flush oil from reactor.

11:10 Pressure transfer prep tank contents to reactor. Level is 243". Pressure transfer 2 more times. No noticeable change in level.

12:10 Inspect prep tank. Not as clean as MeOH transfer. There is a layer of catalyst on the walls near where liquid level was. Estimate 5-10 lbs of catalyst. Add 283 lbs of flush oil and agitate. Still a crusty layer where the initial slurry level was.

12:25 Pressure transfer flush oil to reactor. Level ~290".

12:40 Withdraw a slurry sample. Lining up reduction gas composition.

13:24 Syngas to the reactor.

13:32 T = 183 F, P = 60 psig, F = 14,500 SCFH. Level at 369".

14:00 Starting composition: (D06) 1.29% H2
1.98% CO
95.68% N2
0.55% CO2

Starting heatup at 10 F/hr target rate.

14:20 GC Gary has not been totalizing well. Dean says there is liquid in the SP13b line (he sees it in the rotameters), and there was liquid in the K.O. pots. They are trying to correct problems with the electric heaters. GC Dennis is OK.

15:15 Increasing H2 in the feed slightly. Last few analyses show 1.1%.

15:40 H2 was a little too high (1.7%).

16:12 GC Gary is going off-line for calibration. Level at 377".

16:58 GC Gary back on-line. During calibration N2 was reading 2% low. There may be some oil in the line which may take a while to purge out.

19:45 Level at 389". H2 and CO uptake is underway. Earlier GC note still holds. GC Gary working well on SP6 but not on SP13b. GC Dennis still working well. Believe GC Dennis.

20:06 GC Gary seems to be improving.

20:20 Uptake appears to be increasing. Uptake vs. time matches MeOH reduction almost identically.

23:55 Level at 384".

3/27/94 Sunday

02:08 Level at 363".

03:05 Bringing the 02.62 reduction heaters online to help warm up the reduction feed gas during the temperature ramp.

04:00 Tried to increase the total flow using N2, since it had dropped to 13,400. However, flare was flickering on one side, so we decided to not risk it.

07:30 Level at 346". Flow had dropped back to about 13,000 SCFH. Matt opened up on the valve throttled at the reactor inlet. Flow is back up to 14,000 SCFH.

11:31 Began 4 hour hold period. Level at 345". T = 393 F. Put nuke in auto control at 352".

12:55 Setpoint on nuke is now 362".

13:30 Flow at 14,800 SCFH. Reduced to approximately 14,000 SCFH.

14:35 Setpoint on nuke is now 376.5".

14:50 Oil added to 27.14 from 6 nuts to 16 nuts.

15:35 Heating up from 394.6 F at 18 F/hr.

20:10 Flow out of the reactor. Level at 295" with no gas. SP13 valved out; 21.11 demister line open. SP3A is now valved in.

20:45 Henry has been trying for a slurry sample for approximately 1 hour. The sample tap (by the DP) is plugged and with 40 psig on it, he can't get a sample. We're giving up.

22:05 Syngas to the reactor. T = 451 F, P = 194 psig, F = 17,250. Level at 315". GC reading on fresh feed: 28% H2, 67% CO, 6% CO2.

22:33 T = 450 F, P = 500 psig, Level at 326".

22:45 T = 450 F, P = 620 psig.

GC Reading (D04): 20.04% H2
51.92% CO 19.57% N2
5.57% CO2

Opening PV-201 to help purge some N2.

22:55 Recycle flow introduced. Level at 328". T = 452 F, P = 700 psig.

23:05 T = 453 F, P = 753 psig. MeOH in effluent = 4.78% (G03 at 22:53).

23:27 T = 464 F, P = 751 psig, F = 73,990 SCFH.

23:56 Showing level in 22.10. T = 489 F, P = 744 psig.

3/28/94 00:05 Feed composition (SP4): 20.70% H2

Monday 60.40% CO 11.85% N2
3.61% CO2

00:25 T = 509 F, P = 754 psig, F = 95,900 SCFH.

01:23 CO2 out of the fresh feed (CO was temporarily shut off).

01:30 T = 541 F, P= 752 psig. Level at 450". TIC-1260 (21.11 outlet) = 265 F.

01:53 Nuke level moved to 480".

02:15 27.20 level at 480". Going to cascade control. T = 565 F.

02:25 H2/CO ratio is coming up as N2 gets backed out. Increased CO from 10.0 to 10.3 MSCFH.

(D04)32.16% H2
58.81% CO
5.57% N2
3.26% CO2

03:05 Pretty well lined out on temperature. Average temp is 568 F, but control is based on TI-1232 which is right at 572 F.

04:30 Nuke scan. Calc's show 39 wt% slurry and 37 vol% gas holdup.
T = 568 F, P = 754 psig, F = 96,400 SCFH.

06:35 Still adding in CO as the N2 concentration drops.

(D04)31.70% H2
63.26% CO
2.23% N2
3.06% CO2

08:40 D04 composition looking good: 30.62% H2
65.48% CO
1.40% N2
2.92% CO2

Flow slipping a little to about 95,500 SCFH.

10:45 GC Dennis off-line for calibration. GC Gary is still on-line.

14:27 Pinhole leak in thermocouple weld. Must cool and depressurize the reactor to fix it. Started cooling at 7 F/5 min.

17:25 T = 353 F, P = 263 psig.

18:30 T = 344 F, P = 2 psig. Thermocouple connection (TI-1235) back-welded. Leak fixed. Blending syngas.

18:45 Syngas composition OK. Heating up.

19:07 T = 383 F, P = 572 psig, F = 23,000 SCFH. Back end on. Starting recycle flow.

22:20 Reactor temperature out of control. We were up to 598 F. The fin fan bypass was 50% open and HIC-166 was 93%. We opened the HIC-166 valves to 85%, and the temperatures finally started to come back.

23:11 Noticed that none of the GC's have updated since 21:38, which is where we had a FROMHP.BAD file that was zero blocks big. All the GC's are analyzing in the lab and printing out the analyses. HP_GC_DRIVER is "hibernating", which is good. Talked with Rob and he doesn't understand why all GC's would be having this problem.

DEC ng_stop and ng_start again.

3/29/94 00:30 Rob was in to look at the GC's. There are no errors on his end, and he Tuesday doesn't want to reboot blindly for fear of losing all his methods. So we go all night with no GC values in to the DEC. Checked printouts in lab to line-out on composition.

03:46 Took out fresh CO2 (was 200 SCFH on set point).

06:12 Nuke scan. Calc's show 41 wt% slurry and 42 vol% gas holdup.
T = 573 F, P = 752 psig, F = 97,000 SCFH.

07:45 10.80 pump seal leaking. Blocking in back-end to fix the pump, while maintaining flow to reactor. Dropping temperature by 50 F in reactor.

Still getting no info from HP computer. We've given up on reconstructing the overnight data. Rob rebooted the HP and we've restopped and restarted nextgen and moved the clocks forward on both computers. (Daylight savings time is this weekend). So 9:59 became 10:59 on the DEC. Also when we first went to work on the DEC, the trend data collection routine had failed around 4:30 in the morning. We cannot access data for that time until probably 11:00 AM on March 29 when it was restarted.

12:25 (Real time) Cloudy MeOH sample from 22.11 (with drops of oil).

16:30 Back-end pressured up. 10.80 pump reinstalled and started. Heating up reactor at 60 F/hr.

17:36 (DEC time) Reactor at 572 F (average) and 750 psig. Reactor PIC on cascade control. Draining liquid from the 21.11.

18:02 F = 93,800. Analyses from GC's:

(D04)	29.98%	H2	(D03)	23.19%	H2
	66.16%	CO		66.19%	CO
	1.00%	N2		1.18%	N2
	2.69%	CO2		6.03%	CO2
				2.28%	MeOH

Matt drained 10 gallons of liquid from the 21.11. There was a large second phase (definitely oils). Raised setpoint of TIC-1260 (21.11 outlet) to 270 F. When reactor heated up, the 21.11 bypass was not in control and the process value was 275 F even though the setpoint was 245 F. Leaving the setpoint at 270 F for now to try and get rid of two phases.

Airco loading the CO2 tank to full.

19:30 GC Gary 3A is disconnected for a while to clean out the rotameter lines with N2. Last bad file from the HP received was at 19:53.

19:35 Reduce reactor setpoint to 574 F to meet average reactor target temperature of 572 F.

20:00 Leaks at trailer during 22.16 transfer. Called in Matlack. Plant running steady. T = 573 F, P = 748 psig, F = 95,390 SCFH. Compositions: IBOH = 0.17 - 0.18%, MeOH = 2.7 - 2.9%.

21:30 Plant running very steady.

23:20 22.16 day tank transfer to trailer 116" to 20" (1692 gallons).
(Note: Bharat's logs are in real time for time period of 20:00 to 23:20. Need to add 1 hour for DEC time.) VES is logging times as DEC-time from here on out. Everyone should follow suit.

3/30/94 03:00 Plant still very steady!
Wednesday

05:25 Henry grabbed a liquid sample from 22.11. Looks good.

07:40 Nuke scan. Calc's show 42 wt% slurry and 43 vol% gas holdup.
T = 573 F, P = 753 psig, F = 96,000 SCFH.

08:48 G03 lost its H2 peak. DEC shows a bad file message from the HP at 08:54. Actual value from lab printout is 24.40. Check into deleting this point from mass balance.

09:30 FI-7291 calculation corrected in the Bailey. G/C forgot to add 14.7 to the pressure and 460 F to the temperature.

11:10 AJ grabbed a liquid sample off the 22.11. Still looks good.

13:30 Changing conditions to AF-R10.2.

AF-R10.1 data period: from 3/29/94 at 21:00 to 3/30/94 at 13:00.

14:00 AF-R10.2 condition reached.

15:45 Rob advised not to use any information from GC Gary between 12:30 and 14:30 DEC-time. Will need to correct for this when doing mass balance for AF-R10.1.

19:00 Bharat and Kerri are investigating how to delete 08:54 bad data point from averaged data. Data point to be deleted is G03001. Last good data point for G03001 was at 07:46. Bad shot was actually at 08:48. Next good shot was at 09:51.

21:00 Noticed DEC is not picking up some HP data. R02 is missing the methanol peak. Closed picture and signal overview and restarted. This did not help the situation. Chris DaCosta will try to work on modem.

22:15 Chris has found the problem. Butane is being sent in component #10 slot which overwrites the methanol #10 slot.

22:20 We continue to lower 10.80 MEOH flow to increase CO2 in the reactor feed to 3%.

23:15 Rob came in and deleted n-butane (#10 slot) and pentane (#11 slot) from GC's Rocco and Bharat.

3/31/94 00:00 Looks like CO2 in the feed is lined out (2.6%).

Thursday

03:40 GC's have not updated since shortly after 02:00. Message screen shows only one bad file received from HP at 02:27. HP printer still working in the lab. Closed signal overview and pictures and did a restart. Signal overview still shows flat compositions.
FROMHP.BAD;815 (most recent) is of size 0 blocks (see 3/28/94 23:11).

04:10 Looks like exactly the same problem as the other night - solution was to reboot the HP. Called Rob. He's coming in.

04:45 Rob is shutting down GC's to reboot.

05:30 Looks like the GC problem is fixed. DEC is starting to get new (.old) files from the HP. The new concentrations coming in are very similar to the old ones that were stuck from 3-4 hours ago. Last point went non-zero just before 07:00. Will exclude 02:00 -07:00 from data.

05:50 Henry grabbed a liquid sample off of 22.11.

07:20 Nuke scan. Calc's show 38 wt% slurry and 36 vol% gas holdup.
T = 572 F, P = 752 psig, F = 57,680 SCFH.

09:15 22.16 day tank transfer to trailer 102 3/4" to 20" (1458.5 gallons).

10:45 Product liquid analysis is showing about 8% "others". Rich Underwood advises that it is reasonable to assume they average to C6-alcohols. Will adjust the hexanol peak.

14:20 Matt grabbed a liquid product sample off of 22.11.

15:50 AJ grabbed another liquid sample from 22.11.

17:04 Changing conditions to AF-R10.3.

AF-R10.2 data period: from 3/31/94 at 00:00 to 3/31/94 at 17:00, excluding 02:00 - 07:00 when GC's were not working correctly.

4/1/94 Friday

02:05 Plant looks steady. Feed composition is still drifting into place, though.

04:05 Still very steady. Data period begins 04:00.

07:04 Nuke scan. Calc's show 45 wt% slurry and 51 vol% gas holdup.
T = 572 F, P = 750 psig, F = 156,570 SCFH.

07:45 AJ grabbed a liquid sample off of 22.11.

10:45 22.16 day tank transfer to trailer 95 5/8" to 20" (1332.6 gallons).

14:15 Took a liquid sample from 22.11.

16:00 AF-R10.3 data period: from 4/1/94 at 04:00 to 4/1/94 at 16:00.

16:05 GC's Rocco, Bharat, and Gary will be taken off line as the plant is down.

16:25 Shutdown test. The liquid dropped from 471" to 263" in reactor.

Method 1: Assuming 0% holdup in liquid:

Gas Holdup = (471 - 263)/471*100 = 44.2 %

Method 2: Assuming calculated 6% holdup in liquid:

Gas Holdup = (471 - 263 * 0.94)/471*100 = 47.5 %

Method 3: Holdup calculated by regular nuke scan = 49.5 %

16:40 Changing conditions to AF-R10.4. 01.30 compressor is on. Increasing pressure to 1300 psig.

16:50 Reactor average temperature overshot to 583 F, coming down now.

17:10 Reactor pressure is now 1317 psig. Another reactor temperature rise observed - peaked at 590 F.

18:10 Put reactor on level control at 480"

4/2/94 00:00 Plant seems steady. GC Gary is totalizing very poorly on SP3 and not Saturday particularly well on SP4.

02:20 22.16 day tank transfer to trailer 103 3/4" to 20" (1475.75 gallons).

03:45 Henry grabbed a liquid sample off 22.11

04:00 Plant still steady. CO_2 consumption at 3750 lb/hr.

05:01 Nuke scan. Calc's show 42 wt% slurry and 44 vol% gas holdup.
T = 573 F, P = 1302 psig, F = 157,000 SCFH.

06:05 Waiting desperately for a CO_2 shipment (more than 2 hours late now).

07:15 CO_2 is gone!!! Closing steam to reboiler, isolating back-end, shutting down 10.80 pump, dropping temperature in reactor at 50F/hr.

AF-R10.4 data period: from 4/1/94 at 21:00 to 4/2/94 at 07:00.

07:35 D04 at 07:20 reads 4.01% CO_2 in feed. Temperaure down to 554 F.

08:35 D04 at 08:22 reads 3.56 % CO_2 in feed. Temperature down to 500 F.

08:40 CO_2 load finally arrived! Starting to bring back-end up again and move to data point AF-R10.5.

11:00 27.20 and 21.11 on level control.

11:45 Blocked in SP15 at root valve. Rob taking GC Gary off-line to fix it.

14:20 22.16 day tank transfer to trailer 123" to 45 1/2" (1366 gallons). During transfer the trailer overfilled, spilling liquid out of trailer and out of flare.

16:45 GC Gary back on line. Seems to be better now.

22.25 CO has been high (67%) in the reactor feed. Have found it difficult to get any attention because operators are busy with more important work. Initial, small moves had little effect, as expected. Finally convinced operators to make larger and faster moves. CO is 67% and dropping right now.

Because of overfill on the trailer, some liquid from the full trailer was transferred to empty trailer (maximum estimate is 500 gallons). We need to keep a running total of trailer inventory. Trailer capacity is 7000 gallons. A rule of thumb would be no more than 4 transfers per trailer.

22:40 22.16 day tank transfer to trailer (second) 102 3/4" to 41 1/2" (1097.5 gallons). Emtec is here doing spill cleanup.

4/3/94 Sunday

00:40 22.16 day tank transfer to trailer 54 1/4" to 20" (603.5 gallons).

03:30 Real time = Dec time! Finally!! Plant is finally steady, although CO_2 is at its upper limit in the feed (about 3%). CO_2 consumption in back-end is about 3100 lbs/hr with 35,000 lbs left in the tank. We should be okay until about 14:00 at this rate, but usage will increase if we start stripping more. Next delivery is scheduled for 10:00. R02 at 03:06 shows nothing but methane and ethane (i.e., we got part-2 but not part-1). Everything looks OK on the HP printout and no bad file messages. Previous point was OK too. Reactor temperature profile has been inverted since we started moving to this condition (more than 15 hours ago). About 580 F on bottom 2 thermocouples (1239 & 1238), 575 F on next 2 (1236 & 1235), then about 567 F thru the rest of the slurry.

04:00 R02 at 03:34 looks good again.

04:53 Nuke scan. Calc's show 41 wt% slurry and 42 vol% gas holdup.
T = 573 F, P = 1735 psig, F = 157,000 SCFH.

06:30 Matt grabbed a liquid product sample off of 22.11.

11:15 22.16 day tank transfer to trailer 117 7/8" to 20" (1723 gallons).

11:15 22.16 day tank transfer to trailer 117 7/8" to 20" (1723 gallons).

12:45 Matt took another liquid sample from 22.11.

14:25 Changing conditions to AF-R10.6.

AF-R10.5 data period: from 4/3/94 at 02:00 to 4/3/94 at 14:00.

17:50 27.14 almost empty. Added oil to go to 9 nuts (approximately 39 gallons).

21:00 Increasing CO, decreasing H2.

22:00 Still trying to increase CO2 in the feed.

4/4/94 Monday 01:30 Plant steady. Start data period at midnight.

02:40 22.16 day tank transfer to trailer 99 3/4" to 20" (1405.5 gallons).

04:50 Henry grabbed a liquid sample off of 22.11.

05:00 Nuke scan. Calc's show 35 wt% slurry and 28 vol% gas holdup.
T = 572 F, P = 1733 psig, F = 57,500 SCFH.

10:45 Matt caught a liquid sample from 22.11.

12:25 Changing conditions to AF-R10.7.

AF-R10.6 data period: from 4/4/94 at 00:00 to 4/4/94 at 12:00.

16:20 Lined out at 16:00, but GC Gary not on-line until now. Data period can start at 17:00.

22:30 22.16 day tank transfer to trailer 91 1/2" to 20" (1260 gallons).

4/5/94 Tuesday 00:35 Henry grabbed a liquid sample off of 22.11.

02:57 Nuke scan. Calc's show 39 wt% slurry and 37 vol% gas holdup.
T = 572 F, P = 1301 psig, F = 96,000 SCFH.
These results are the same as nuke scan at 21:40.

03:30 The plant is very steady - it must be near the end of the run!

05:30 Henry grabbed another liquid sample off 22.11.

08:45 GC's Dennis, Bharat, and Rocco are all off-line for repairs/calibration. Will take a few hours to bring them back up.

09:30 Starting 02.63 to warm it up, and then they will be ready to start alcohol injection case, run AF-R10.8.

10:00 We are hooked up to rear compartment of alcohol injection trailer.

10:40 Started 10.95 pump. Having some problems.

11:15 GC Dennis is back on-line. GC's Rocco and Bharat are still off-line.

13:35 Alcohols finally going in the reactor feed.

14:50 MeOH concentration finally coming up in reactor feed sample (SP15).

16:05 22.16 day tank transfer (#1) to empty trailer 101" to 20" (1428 gallons).

19:30 Reduced 10.95 stroke rate from 100% to 95%. Methanol is currently 8.8% (target methanol = 7.9%). Increased CO from 12.3 to 12.8.

20:45 CO increased to 13.5. 10.95 pump stroke decreased to 90%.

21:15 CO increased to 14.0.

22:40 Thunderstorm and cold front passing through. Temperature dropped as low as 567 F in reactor. Still trying to line out the plant.

4/6/94 Wednesday

00:50 22.16 day tank transfer (#2) to trailer 97" to 20" (1357 gallons). Henry moved 10.95 pump back to 92% stroke from 90%.

01:30 Finally, composition is nearly lined out. Rob is putting GC Gary into its regular sequence.

02:05 Plant pretty well lined out, although still fighting reactor temperature because of the weather change.

03:00 G06 finally updated. All ports in GC Gary now reading new values. Temperature seems to have steadied out, too.

06:14 10.95 pump shutdown while reconnecting hoses to facilitate the transfer from back compartment of trailer to the front.

06:16 10.95 back on-line. Stopped again for a few seconds; then back on again. Reactor temperature spiked briefly to 581 F. Pressure dropped to 1250 psig and overshot back to 1320 psig.

06:20 10.95 stroke increased to 95%.

06:35 MeOH in G02 shot at 06:21 down to 5.95 because of upset. Excluding 06:10 to 07:00 from the data period.

07:15 Increased 10.95 stroke to 100%.

07:45 Matt grabbed a liquid sample off 22.11.

08:48 10.95 shutdown again for a minute or two. . . several times though. Temperature spiked to 587 F. Pressure dropped as low as 1240 psig.

09:40 22.16 day tank transfer (#3) to trailer 103" to 20" (1463 gallons).

12:30 Matt caught another liquid sample off of 22.11.

14:40 GC Dennis was off-line between 13:00 and 14:00. Excluding 13:40 to 15:40 for mass balance.

18:45 22.16 day tank transfer to middle compartment (1000 gallon capacity) of new trailer 93 1/8" to 46" (831 gallons).

20:30 Changing conditions to AF-R10.9.

AF-R10.8 data period: from 4/6/94 at 03:00 to 4/6/94 at 20:00.

While getting ready to change conditions: observed temperature rise in reactor (up to 587 F), and alcohol flow dropped to 0.4 gpm. Looks like the unit is changing conditions by itself! First thought we ran out of feed. Switched tank compartments, but flow is still approximately 0.4 gpm with 100% stroke. Looks like there may be another problem. The previous compartment still shows liquid level, too. Reaction temperature is back in control. Transmission fluid is low in 10.95.

23:20 Henry got 10.95 pump started again. Flow up to 0.65 - 0.70 gpm at 100% stroke.

4/7/94 03:00 10.95 still hanging in there at 0.6 - 0.7 gpm (100 % stroke). We are still Thursday adjusting feed composition (were very CO-rich), but it's slow going at 1 GC shot every half hour or so.

06:00 Finally gettting closer to lining out. A few more tweaks, perhaps.

07:00 Plant is pretty well lined out, although CO2 in the feed is a little low.

08:55 Matt grabbed a liquid product sample off of 22.11.

09:15 Matt added oil to 27.14 from 1 nut to 16 nuts (50 gallons).

11:30 Calculation of alcohol feed rates from compartment #3:
Starting level = 1468.5 gal
Final level = 13 gal
Alcohol consumed = 1455.5 gal over 11.5 hrs
Consumption rate = 2.11 gpm

Average FI-1221 reading during that period = 2.37 gpm. Therefore, the correction factor = 2.37/2.11 = 1.12. To get 0.7 gpm for AF-R10.9, the reading on FI-1221 should be 0.78 gpm.

Improved performance of 10.95 pump. Now set at 0.78 gpm (0.7 gpm actual, per above calculation) on FI-1221.

11:35 22.16 day tank transfer to trailer 116" to 20" (1692 gallons).

15:10 Have been decreasing H2 last 1.5 hours to desired level.

16:00 Data period for AF-R10.9 can begin now.

16:30 Matt grabbed a liquid sample off of 22.11.

19:45 22.16 day tank transfer to trailer 64 1/2" to 20" (784 gallons). Total liquid in trailer is now 6724 gallons (7000 gallon capacity). Trailer is now full.

23:30 Plant running smoothly. CO2 in the feed is low, but no matter how much we crank down the back-end, it keeps drifting down.

4/8/94 Friday

01:20 Henry grabbed a liquid sample off of 22.11.

02:24 Nuke scan. Calc's show 39 wt% slurry and 38 vol% gas holdup.
T = 572 F, P = 1299 psig, F = 97,000 SCFH.

04:15 Henry grabbed another liquid sample off of 22.11.

05:3505:00 mass balance indicated liquid injection rate has been 2/3 of target rate, despite calculation at 11:30 above. Also, level loss on trailer just increased for the first time, meaning that we just recently crossed thru 1/2 full point. Adjusted stroke on pump until FI-1221 read 1.26 gpm (50% increase over reading for duration of the

data period). Henry commented to me that he had decreased the stroke slightly several times during the night to maintain flow near the target. So, AF-R10.9 closes at 05:00. If we choose to run this condition for a while, it can be called AF-R10.9A.

09:30 Matt grabbed a liquid sample from 22.11.

11:30 IBOH is very low (0.06%) in effluent stream. It also looks like the DEC stopped taking data. Stopped and started NEXTGEN. Need to find the time during which the DEC was not taking data so that period can be excluded. In the meantime, the IBOH number appears to be correct. Switched GC Rocco to SP-3A, and Rocco confirms GC Bharat's analysis. We will now calibrate both GC's. We seem to have major deactivation!! Looking at raw GC data, it was gradual over the last 24 hours. Called Rich Underwood, but he could not explain the data.

12:45 22.16 day tank transfer to trailer 108 1/2" to 20" (1559 gallons).

14:20 Looks like we lost data on DEC after 06:00 today, so will not include 06:00 - 14:00 in any data processing.

15:00 Matt grabbed another liquid sample from 22.11.

17:00 Run No. 10.9A complete. Changing conditions to AF-R10.10.

19:00 Took a liquid sample from 22.11.

19:40 Henry grabbed another liquid sample off of 22.11.

20:15 27.14 is bypassed.

22:40 Henry grabbed another liquid sample.

23:15 Nuke scan. Calc's show 41 wt% slurry and 42 vol% gas holdup.
T = 573 F, P = 752 psig, F = 95,600 SCFH.
Looks pretty much like AF-R10.1.

4/9/94 Saturday

00:40 Liquid production rate (from levels in product collection) is very small.

02:40 Henry grabbed another liquid sample off of 22.11.

04:40 Henry added oil into 27.14. 6 1/2 to 8 1/2 nuts (7 gallons). Oil rate appears to be about 2 gph.

05:12 Nuke scan. Calc's show 41 wt% slurry and 42 vol% gas holdup.
T = 572 F, P = 747 psig, F = 96,600 SCFH.

05:40 Henry grabbed one last liquid sample off 22.11. This was the first one that was cloudy, but the previous ones may just be leftover from the last data point.

08:15 Matt lined up carbonyl sample points 3A, 4, 15.

12:40 Fe carbonyl levels: SP4 0.006 ppm
SP3A 0.003 ppm
SP15 0.022 ppm

12:50 Started 01.30 compressor.

14:30 Matt grabbed 22.11 liquid sample.

14:45 Matt grabbed liquid sample from 21.11.

14:50 Matt grabbed sample off center compartment on trailer #SP1738 (alcohol injection source when catalyst died).

15:16 Fe carbonyl levels: SP4 0.006 ppm
SP3A 0.004 ppm
SP15 0.076 ppm

15:25 Dropping flow from 96,000 to 58,000 SCFH.

17:52 Fe carbonyl levels: SP4 0.009 ppm
SP3A 0.006 ppm
SP15 0.069 ppm

18:20 Started shutdown. Ramping temperature down by 60 F/hr.

19:15 01.20 compressor shut down. Pressure dropped to 750 psig.

21:00 Flow switched to go through 27.14. Bypass around 27.14 shut.

22:15 Blocked in back-end. Depressurizing process side and draining 21.80.

23:00 Stopped syngas fresh feed and brought in N2 to purge the plant.

APPENDIX C

LPMEOH AND LPIBOH MASS BALANCES AND LIQUID ANALYSES

RUN NO: **AF-R9.1b** **TITLE: LPMEOH over BASF S3-86 CATALYST with TEXACO GAS : SV=6700**

Balance Period:

Start Date	3/22/94	3:00
End Date	3/22/94	21:00

Time From Start of Run (hr)

Start	62.00
End	80.00

Reaction Conditions

Temperature (°F)	476
Pressure (psig)	750
Space Velocity (sL/kg-hr)	6832
Vg (inlet)	0.85

Slurry Data

Catalyst Weight (lb oxide)	1250
Slurry Concentration (wt %)	45.4
Slurry Level (%)	92
Gas Holdup (vol %)	47.2

Performance Results

CO Conversion to H2 (%)	-0.6
CO Conversion to MeOH (%)	14.5
Theoretical Conversion % (1 CSTR)	14.5
Alcohol Production (Ton/day)	11.1

Atom Balance Closure (% of inlet)

C	0.70
H	-1.31
O	0.59
N	-0.40

Liquid Product Analysis (wt%)

	Sample# 10:15	Sample# 17:00	Sample# 21:02	Sample#	Sample#	Sample#
Methanol	95.877	95.981	96.114			
Ethanol	0.833	0.833	0.783			
1-Propanol	0.334	0.329	0.318			
iso-Propanol	0.021	0.028	0.016			
1-Butanol	0.162	0.163	0.155			
2-Butanol	0.058	0.058	0.053			
iso-Butanol	0.053	0.042	0.044			
2-Methyl-1-Butanol	0.046	0.045	0.044			
1-Pentanol	0.115	0.112	0.107			
2-Methyl-1-Pentanol	0.000	0.000	0.000			
1-Hexanol	0.036	0.043	0.042			
2-Methyl-1-Isobutyrate	0.000	0.000	0.000			
Methyl Acetate	0.218	0.210	0.193			
Ethyl Acetate	0.000	0.000	0.000			
Methyl Formate	0.915	0.870	0.853			
DME	0.085	0.074	0.070			
CO2	0.000	0.000	0.000			
Water	1.026	1.016	1.022			
Oil	0.243	0.261	0.225			
Total	100.022	100.065	100.039			

RUN NO: AF-R9.1b TITLE: LPMEOH over BASF S3-86 CATALYST with TEXACO GAS : SV=6700

		FRESH MAKE-UP	RECYCLE	HP H2 MAKEUP	DRY FEED	ALCOHOL INJECT.	REACT FEED	REACT EFFL	22.10 VAPOR	PURGE 1 PIC-201	PURGE 2 22.11	PURGE 3 07.20	LIQUID PROD
T	F	303.4	140.9	79.8	310.4		295.3	468.9	100.0	85.1	69.2		80.0
P	psig	844.2	871.5	822.4	793.4		793.4	750.1	12.6	622.8	34.8		1.0
Comp	H2	16.23	25.13	100.00	34.49		34.49	21.76	25.14	25.13	30.49		0.00
	CO	77.75	57.50	0.00	52.27		52.27	52.77	57.53	57.50	62.17		0.00
	N2	0.42	1.18	0.00	0.90		0.90	1.08	1.18	1.18	1.59		0.00
	CH4	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.00
	CO2	5.61	16.08	0.00	12.33		12.33	14.75	16.09	16.08	5.74		0.00
	DME	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.05
	MeAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.09
	EtAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.00
	MeFm	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.47
	MeOH	0.00	0.11	0.00	0.00		0.00	9.11	0.00	0.11	0.00		96.59
	H2O	0.00	0.00	0.00	0.00		0.00	0.38	0.06	0.00	0.01		1.83
	Etoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.57
	1-Proh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.18
	iso-Proh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.01
	IBOH	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.02
	1-Buoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.07
	2-Buoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.02
	2-Methyl 1-Buoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.02
	1-Peoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.04
	2-Methyl 1-Peoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.00
	1-hexanol	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.01
	2-Methyl 1-Isobutyrate	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.00
	others	0.00	0.00	0.00	0.00		0.00	0.14	0.00	0.00	0.00		0.03
	TOTAL	100.00	100.00	100.00	100.00		100.00	100.00	100.00	100.00	100.00		100.00
Mole Wt	lb/lb mole	24.689	24.057	2.020	21.018		21.018	25.093	24.044	24.057	21.004		32.191
Flow	SCFH	19568	106283	21470	147321		147321	122930	112202	6317	462		11537
	lb mole/hr	50.61	274.88	55.53	381.02		381.02	317.94	290.19	16.34	1.19		29.84
	lb/hr	1249.5	6612.8	112.2	8008.3		8008.3	7978.1	6977.3	393.0	25.1		960.5

RUN NO: AF-R9.2 **TITLE: LPMEOH over BASF S3-86 CATALYST with KINGSPORT SYNGAS : SV=4000**

Balance Period:		
Start Date	3/23/94	12:00
End Date	3/24/94	9:00

Time From Start of Run (hr)	
Start	95.00
End	116.00

Reaction Conditions	
Temperature (°F)	483
Pressure (psig)	739
Space Velocity (sL/kg-hr)	4020
Vg (inlet)	0.51

Slurry Data	
Catalyst Weight (lb oxide)	1250
Slurry Concentration (wt %)	39.4
Slurry Level (%)	92
Gas Holdup (vol %)	33.5

Performance Results	
CO Conversion to H2 (%)	-4.4
CO Conversion to MeOH (%)	46.5
Theoretical Conversion % (3 CSTR's)	46.9
Alcohol Production (Ton/day)	10.8

Atom Balance Closure (% of inlet)	
C	0.12
H	-1.81
O	0.61
N	-0.29

		Sample# 16:50	Sample# 22:00	Sample# 04:00	Sample# 08:30	Sample#	Sample#
	Methanol	97.511	97.442	97.596	97.624		
	Ethanol	0.275	0.309	0.223	0.224		
	1-Propanol	0.101	0.115	0.099	0.098		
	iso-Propanol	0.014	0.010	0.008	0.029		
	1-Butanol	0.048	0.056	0.039	0.040		
Liquid	2-Butanol	0.034	0.036	0.032	0.030		
Product	iso-Butanol	0.017	0.021	0.015	0.015		
Analysis	2-Methyl-1-Butanol	0.015	0.018	0.016	0.014		
(wt%)	1-Pentanol	0.042	0.049	0.027	0.029		
	2-Methyl-1-Pentanol	0.000	0.000	0.000	0.000		
	1-Hexanol	0.013	0.018	0.011	0.010		
	2-Methyl-1-Isobutyrate	0.000	0.000	0.000	0.000		
	Methyl Acetate	0.047	0.063	0.035	0.044		
	Ethyl Acetate	0.000	0.000	0.000	0.000		
	Methyl Formate	0.470	0.518	0.433	0.432		
	DME	0.069	0.065	0.067	0.069		
	CO2	0.000	0.000	0.000	0.000		
	Water	1.325	1.232	1.401	1.344		
	Oil	0.125	0.141	0.125	0.101		
	Total	100.106	100.093	100.127	100.103		

RUN NO: AF-R9.2 TITLE: LPMEOH over BASF S3-86 CATALYST with KINGSPORT SYNGAS : SV=4000

		FRESH MAKE-UP	RECYCLE	HP H2 MAKEUP	DRY FEED	ALCOHOL INJECT.	REACT FEED	REACT EFFL	22.10 VAPOR	PURGE 1 PIC-201	PURGE 2 22.11	PURGE 3 07.20	LIQUID PROD
T	F	311.7	163.1	88.7	325.3		306.9	462.7	100.0	86.3	77.2		80.0
P	psig	779.7	893.5	681.7	755.2		755.2	738.5	627.1	627.1	34.3		1.0
Comp	H2	16.48	54.45	100.00	58.59		58.59	42.85	54.52	54.45	10.97		0.00
	CO	75.50	23.21	0.00	26.85		26.85	19.54	23.24	23.21	9.84		0.00
	N2	0.54	3.82	0.00	2.35		2.35	3.21	3.82	3.82	1.08		0.00
	CH4	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.00
	CO2	7.48	18.39	0.00	12.21		12.21	15.58	18.41	18.39	72.43		0.00
	DME	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.36		0.05
	MeAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.02
	EtAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.00
	MeFm	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.25
	MeOH	0.00	0.14	0.00	0.00		0.00	17.10	0.00	0.14	5.30		97.03
	H2O	0.00	0.00	0.00	0.00		0.00	1.62	0.00	0.00	0.02		2.34
	Etoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.18
	1-Proh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.05
	iso-Proh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.01
	IBOH	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.01
	1-Buoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.02
	2-Buoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.01
	2-Methyl 1-Buoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.01
	1-Peoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.01
	2-Methyl 1-Peoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.00
	1-hexanol	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.00
	2-Methyl 1-Isobutyrate	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00		0.00
	others	0.00	0.00	0.00	0.00		0.00	0.10	0.00	0.00	0.00		0.01
	TOTAL	100.00	100.00	100.00	100.00		100.00	100.00	100.00	100.00	100.00		100.00
Mole Wt	lb/lb mole	24.922	16.807	2.020	14.738		14.738	19.932	16.786	16.807	37.024		31.873
Flow	SCFH	14719	51488	20474	86682		86682	63344	53234	1972	487		11388
	lb mole/hr	38.07	133.17	52.95	224.19		224.19	163.83	137.68	5.10	1.26		29.45
	lb/hr	948.8	2238.2	107.0	3304.0		3304.0	3265.4	2311.1	85.7	46.6		938.8

RUN NO: **AF-R10.1** **TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS : SV=5000**

Balance Period:

Start Date	3/29/94	21:00
End Date	3/30/94	13:00

Time From Start of Run (hr)

Start	45.92
End	61.92

Reaction Conditions

Temperature (°F)	573
Pressure (psig)	750
Space Velocity (sL/kg-hr)	5044
Vg (inlet)	0.61

Slurry Data

Catalyst Weight (lb oxide)	1100
Slurry Concentration (wt %)	41.1
Slurry Level (%)	91
Gas Holdup (vol %)	42.4

Performance Results

CO Conversion (%)	12.5
H2 Conversion (%)	25.4
Syngas Conversion (%)	16.6
MeOH Production (g/kg-oxide-hr)	183.3
IBOH Production (g/kg oxide-hr)	25.8
C2-C6 OH Production (g/kg oxide-hr)	70.0

Atom Balance Closure (% of inlet)

C	-0.08
H	0.61
O	-0.95
N	-0.95

Liquid Product Analysis (wt%)

	Sample# 5:30 A	Sample# 5:30 B	Sample# 11:10 A	Sample# 11:10 B	Sample#	Sample#
Methanol	62.865	62.839	63.461	63.528		
Ethanol	4.182	4.189	4.213	4.228		
1-Propanol	5.261	5.245	5.253	5.237		
iso-Propanol	0.055	0.055	0.054	0.056		
1-Butanol	1.312	1.321	1.294	1.290		
2-Butanol	0.402	0.407	0.403	0.391		
iso-Butanol	9.104	9.077	8.906	8.868		
2-Methyl-1-Butanol	2.423	2.418	2.367	2.351		
1-Pentanol	0.797	0.786	0.777	0.777		
2-Methyl-1-Pentanol	1.586	1.711	1.646	1.636		
1-Hexanol	0.320	0.320	0.314	0.313		
2-Methyl-1-Isobutyrate	0.718	0.720	0.687	0.688		
Methyl Acetate	0.657	0.660	0.640	0.645		
Ethyl Acetate	0.029	0.027	0.026	0.027		
Methyl Formate	0.000	0.000	0.000	0.000		
DME	0.000	0.000	0.000	0.000		
CO2	0.000	0.000	0.000	0.000		
Water	0.854	0.850	1.179	1.184		
Oil + Others (*)	1.693	1.693	1.693	1.693		
Total	92.258	92.318	92.913	92.912		

The balance of each sample comprises mainly higher alcohol isomers which are not individually analyzed. Assumed to average C6-OH.

(*) Standard oil analysis by evaporation yielded higher than typical results. Actual oil is probably 0.1-0.2 wt%. Balance is presumably other, less volatile higher alcohols.

RUN NO: AF-R10.1 TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS : SV=5000

		FRESH MAKE-UP	RECYCLE	HP H2 MAKEUP	DRY FEED	ALCOHOL INJECT.	REACT FEED	REACT EFFL	22.10 VAPOR	PURGE 1 PIC-201	PURGE 2 22.11	PURGE 3 07.20	LIQUID PROD
T	F	287.4	130.3	63.9	419.3		397.9	559.5	72.8	63.3	61.3	97.4	80.0
P	psig	805.1	880.3	837.7	773.1		773.1	749.5	720.6	698.8	27.7	165.0	1.0
Comp	H2	45.09	26.42	100.00	30.97		30.97	25.81	26.00	26.42	8.23	3.64	0.00
	CO	54.67	69.03	0.00	65.53		65.53	64.04	67.21	69.03	51.64	27.23	0.00
	N2	0.24	0.94	0.00	0.77		0.77	0.86	0.91	0.94	0.50	0.26	0.00
	CH4	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00
	CO2	0.00	3.32	0.00	2.51		2.51	5.60	5.87	3.32	36.01	67.15	0.00
	DME	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.15	0.23	0.00
	MeAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.35
	EtAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.01
	MeFm	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00
	MeOH	0.00	0.00	0.00	0.00		0.00	2.84	0.00	0.00	3.46	1.50	78.74
	H2O	0.00	0.29	0.00	0.22		0.22	0.00	0.01	0.29	0.01	0.00	2.25
	Etoh	0.00	0.00	0.00	0.00		0.00	0.13	0.00	0.00	0.00	0.00	3.64
	1-Proh	0.00	0.00	0.00	0.00		0.00	0.12	0.00	0.00	0.00	0.00	3.49
	iso-Proh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.04
	IBOH	0.00	0.00	0.00	0.00		0.00	0.17	0.00	0.00	0.00	0.00	4.84
	1-Buoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.70
	2-Buoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.22
	2-Methyl 1-Buoh	0.00	0.00	0.00	0.00		0.00	0.03	0.00	0.00	0.00	0.00	1.08
	1-Peoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.36
	2-Methyl 1-Peoh	0.00	0.00	0.00	0.00		0.00	0.02	0.00	0.00	0.00	0.00	0.64
	1-hexanol	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	3.21
	2-Methyl 1-Isobutyrate	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.28
	others	0.00	0.00	0.00	0.00		0.00	0.69	0.00	0.00	0.00	0.00	0.16
	TOTAL	100.00	100.00	100.00	100.00		100.00	100.37	100.00	100.00	100.00	100.00	100.00
Mole Wt	lb/lb mole	16.290	21.645	2.020	20.341		20.342	22.855	22.190	21.645	31.795	37.910	39.571
Flow	SCFH	24000	71712	0	95712		95712	85709	82036	5070	215	3451	3286
	lb mole/hr	62.07	185.47	0.00	247.55		247.55	221.67	212.17	13.11	0.56	8.93	8.50
	lb/hr	1011.2	4014.5	0.0	5035.4		5035.4	5066.5	4708.2	283.8	17.7	338.4	336.3

RUN NO: AF-R10.2 **TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS : SV=3000**

Balance Period:

Start Date	3/31/94	0:00
End Date	3/31/94	17:00

Time From Start of Run (hr)

Start	72.92
End	89.92

Reaction Conditions

Temperature (°F)	572
Pressure (psig)	753
Space Velocity (sL/kg-hr)	3030
Vg (inlet)	0.37

Slurry Data

Catalyst Weight (lb oxide)	1100
Slurry Concentration (wt %)	38.4
Slurry Level (%)	92
Gas Holdup (vol %)	36.3

Performance Results

CO Conversion (%)	13.7
H2 Conversion (%)	25.4
Syngas Conversion (%)	17.3
MeOH Production (g/kg-oxide-hr)	94.5
IBOH Production (g/kg oxide-hr)	18.7
C2-C6 OH Production (g/kg oxide-hr)	45.5

Atom Balance Closure (% of inlet)

C	0.87
H	1.65
O	-0.54
N	-0.45

Liquid Product Analysis (wt%)

	Sample# 5:30 A	Sample# 5:30 B	Sample# 14:20 A	Sample# 14:20 B	Sample# 15:50 A	Sample# 15:50 B
Methanol	55.357	55.845	55.212	55.434	54.809	55.853
Ethanol	3.435	3.441	3.407	3.406	3.352	3.414
1-Propanol	5.380	5.421	5.371	5.378	5.282	5.349
iso-Propanol	0.047	0.052	0.051	0.059	0.053	0.048
1-Butanol	1.322	1.332	1.313	1.324	1.303	1.311
2-Butanol	0.380	0.380	0.377	0.382	0.375	0.377
iso-Butanol	12.835	12.923	12.768	12.771	12.636	12.746
2-Methyl-1-Butanol	3.271	3.094	3.230	3.052	3.190	3.040
1-Pentanol	0.848	0.861	0.844	0.845	0.832	0.842
2-Methyl-1-Pentanol	2.101	1.945	2.341	2.083	2.138	2.306
1-Hexanol	0.321	0.315	0.327	0.329	0.320	0.324
2-Methyl-1-Isobutyrate	1.061	1.072	1.049	1.052	1.035	1.051
Methyl Acetate	0.574	0.583	0.571	0.580	0.565	0.577
Ethyl Acetate	0.024	0.028	0.027	0.029	0.022	0.013
Methyl Formate	0.000	0.000	0.000	0.000	0.000	0.000
DME	0.000	0.000	0.000	0.000	0.000	0.000
CO2	0.000	0.000	0.000	0.000	0.000	0.000
Water	0.897	0.907	1.230	1.243	1.237	1.258
Oil + Others (*)	1.237	1.237	1.687	1.687	1.223	1.223
Total	89.090	89.436	89.805	89.654	88.372	89.732

The balance of each sample comprises mainly higher alcohol isomers which are not individually analyzed. Assumed to average C6-OH.

(*) Standard oil analysis by evaporation yielded higher than typical results. Actual oil is probably 0.1-0.2 wt%. Balance is presumably other, less volatile higher alcohols.

RUN NO: AF-R10.2 TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS : SV=3000

		FRESH MAKE-UP	RECYCLE	HP H2 MAKEUP	DRY FEED	ALCOHOL INJECT.	REACT FEED	REACT EFFL	22.10 VAPOR	PURGE 1 PIC-201	PURGE 2 22.11	PURGE 3 07.20	LIQUID PROD
T	F	274.6	135.5	71.0	343.4		319.1	555.3	68.1	61.5	58.2	70.9	80.0
P	psig	784.5	880.8	841.2	763.4		763.4	752.8	745.6	730.3	27.9	165.0	1.0
Comp	H2	38.34	24.49	100.00	29.31		29.31	24.50	24.19	24.49	7.43	3.30	0.00
	CO	60.69	69.39	0.00	66.32		66.32	64.14	67.10	69.39	48.99	25.92	0.00
	N2	0.88	2.01	0.00	1.63		1.63	1.82	1.92	2.01	1.02	0.51	0.00
	CH4	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00
	CO2	0.09	3.87	0.00	2.58		2.58	6.52	6.78	3.87	39.43	69.26	0.00
	DME	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.15	0.24	0.00
	MeAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.33
	EtAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.01
	MeFm	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00
	MeOH	0.00	0.00	0.00	0.00		0.00	2.45	0.00	0.00	2.95	0.78	73.66
	H2O	0.00	0.25	0.00	0.16		0.16	0.00	0.01	0.25	0.02	0.00	2.67
	Etoh	0.00	0.00	0.00	0.00		0.00	0.10	0.00	0.00	0.00	0.00	3.15
	1-Proh	0.00	0.00	0.00	0.00		0.00	0.11	0.00	0.00	0.00	0.00	3.80
	iso-Proh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.04
	IBOH	0.00	0.00	0.00	0.00		0.00	0.21	0.00	0.00	0.00	0.00	7.34
	1-Buoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.76
	2-Buoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.22
	2-Methyl 1-Buoh	0.00	0.00	0.00	0.00		0.00	0.04	0.00	0.00	0.00	0.00	1.52
	1-Peoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.41
	2-Methyl 1-Peoh	0.00	0.00	0.00	0.00		0.00	0.03	0.00	0.00	0.00	0.00	0.90
	1-hexanol	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	4.57
	2-Methyl 1-Isobutyrate	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.44
	others	0.00	0.00	0.00	0.00		0.00	0.79	0.00	0.00	0.00	0.00	0.20
	TOTAL	100.00	100.00	100.00	100.00		100.00	100.77	100.00	100.00	100.00	100.00	100.00
Mole Wt	lb/lb mole	18.059	22.239	2.020	20.788		20.788	23.436	22.808	22.239	32.533	38.309	42.144
Flow	SCFH	19900	37609	0	57509		57509	51305	48768	8211	198	2174	1791
	lb mole/hr	51.47	97.27	0.00	148.74		148.74	132.69	126.13	21.24	0.51	5.62	4.63
	lb/hr	929.4	2163.2	0.0	3092.0		3092.0	3109.8	2876.8	472.3	16.6	215.4	195.2

RUN NO: **AF-R10.3** **TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS : SV=8200**

Balance Period:

Start Date	4/1/94	4:00
End Date	4/1/94	16:00

Time From Start of Run (hr)

Start	100.92
End	112.92

Reaction Conditions

Temperature (°F)	572
Pressure (psig)	751
Space Velocity (sL/kg-hr)	8242
Vg (inlet)	1.00

Slurry Data

Catalyst Weight (lb oxide)	1100
Slurry Concentration (wt %)	44.8
Slurry Level (%)	90
Gas Holdup (vol %)	50.1

Performance Results

CO Conversion (%)	7.6
H2 Conversion (%)	21.0
Syngas Conversion (%)	11.7
MeOH Production (g/kg-oxide-hr)	274.6
IBOH Production (g/kg oxide-hr)	16.6
C2-C6 OH Production (g/kg oxide-hr)	50.5

Atom Balance Closure (% of inlet)

C	-0.93
H	0.85
O	-1.41
N	0.11

Liquid Product Analysis (wt%)

	Sample# 7:45 A	Sample# 7:45 B	Sample# 14:15 A	Sample# 14:15 B	Sample#	Sample#
Methanol	73.935	74.039	75.423	75.584		
Ethanol	4.142	4.079	4.050	4.050		
1-Propanol	4.416	4.402	4.355	4.318		
iso-Propanol	0.050	0.043	0.049	0.047		
1-Butanol	1.025	1.027	1.002	0.991		
2-Butanol	0.352	0.358	0.352	0.347		
iso-Butanol	5.570	5.628	5.069	5.011		
2-Methyl-1-Butanol	1.408	1.413	1.378	1.370		
1-Pentanol	0.602	0.599	0.580	0.575		
2-Methyl-1-Pentanol	0.990	1.000	0.878	0.899		
1-Hexanol	0.232	0.233	0.226	0.223		
2-Methyl-1-Isobutyrate	0.372	0.372	0.317	0.315		
Methyl Acetate	0.518	0.504	0.476	0.483		
Ethyl Acetate	0.000	0.000	0.000	0.000		
Methyl Formate	0.215	0.226	0.217	0.221		
DME	0.000	0.000	0.000	0.000		
CO2	0.000	0.000	0.000	0.000		
Water	0.711	0.705	0.677	0.673		
Oil + Others (*)	1.540	1.540	2.573	2.573		
Total	96.078	96.168	97.622	97.680		

The balance of each sample comprises mainly higher alcohol isomers which are not individually analyzed. Assumed to average C6-OH.

(*) Standard oil analysis by evaporation yielded higher than typical results. Actual oil is probably 0.1-0.2 wt%. Balance is presumably other, less volatile higher alcohols.

RUN NO: AF-R10.3 TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS : SV=8200

		FRESH MAKE-UP	RECYCLE	HP H2 MAKEUP	DRY FEED	ALCOHOL INJECT.	REACT FEED	REACT EFFL	22.10 VAPOR	PURGE 1 PIC-201	PURGE 2 22.11	PURGE 3 07.20	LIQUID PROD
T	F	296.5	138.0	76.1	431.6		413.8	565.9	79.4	67.7	64.4	80.1	80.0
P	psig	861.4	901.3	816.7	806.0		806.0	751.3	664.7	619.5	30.4	165.1	1.0
Comp	H2	48.18	25.49	100.00	29.46		29.46	25.12	25.13	25.49	9.90	4.34	0.00
(mole%)	CO	50.95	68.97	0.00	65.85		65.85	65.66	68.33	68.97	58.36	33.35	0.00
	N2	0.80	2.23	0.00	1.94		1.94	2.09	2.15	2.23	1.39	0.71	0.00
	CH4	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00
	CO2	0.06	3.31	0.00	2.75		2.75	4.18	4.39	3.31	26.78	60.58	0.00
	DME	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.09	0.24	0.00
	MeAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.25
	EtAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00
	MeFm	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.13
	MeOH	0.00	0.00	0.00	0.00		0.00	2.52	0.00	0.00	3.46	0.77	86.01
	H2O	0.00	0.00	0.00	0.00		0.00	0.00	0.01	0.00	0.01	0.00	1.41
	Etoh	0.00	0.00	0.00	0.00		0.00	0.09	0.00	0.00	0.00	0.00	3.26
	1-Proh	0.00	0.00	0.00	0.00		0.00	0.07	0.00	0.00	0.00	0.00	2.68
	iso-Proh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.03
	IBOH	0.00	0.00	0.00	0.00		0.00	0.07	0.00	0.00	0.00	0.00	2.65
	1-Buoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.50
	2-Buoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.18
	2-Methyl 1-Buoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.58
	1-Peoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.25
	2-Methyl 1-Peoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.34
	1-hexanol	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	1.43
	2-Methyl 1-Isobutyrate	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.12
	others	0.00	0.00	0.00	0.00		0.00	0.37	0.00	0.00	0.00	0.00	0.18
	TOTAL	100.00	100.00	100.00	100.00		100.00	100.18	100.00	100.00	100.00	100.00	100.00
Mole Wt	lb/lb mole	15.497	21.914	2.020	20.793		20.793	22.503	22.180	21.914	29.876	36.650	36.463
Flow	SCFH	27622	128789	0	156411		156411	144910	140643	9580	194	1432	4471
	lb mole/hr	71.44	333.09	0.00	404.53		404.54	374.79	363.75	24.78	0.50	3.70	11.56
	lb/hr	1107.1	7299.5	0.0	8411.6		8411.7	8433.9	8067.9	543.0	15.0	135.8	421.7

RUN NO: **AF-R10.4** **TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS : SV=8200**

Balance Period:

Start Date	4/1/94	21:00
End Date	4/2/94	7:00

Time From Start of Run (hr)

Start	117.92
End	127.92

Reaction Conditions

Temperature (°F)	573
Pressure (psig)	1300
Space Velocity (sL/kg-hr)	8227
Vg (inlet)	0.58

Slurry Data

Catalyst Weight (lb oxide)	1100
Slurry Concentration (wt %)	41.9
Slurry Level (%)	92
Gas Holdup (vol %)	44.0

Performance Results

CO Conversion (%)	12.7
H2 Conversion (%)	34.3
Syngas Conversion (%)	19.6
MeOH Production (g/kg-oxide-hr)	528.0
IBOH Production (g/kg oxide-hr)	20.8
C2-C6 OH Production (g/kg oxide-hr)	86.9

Atom Balance Closure (% of Inlet)

C	0.74
H	-1.26
O	0.07
N	-0.24

Liquid Product Analysis (wt%)

	Sample# 3:45 A	Sample# 3:45 B	Sample# 6:35 A	Sample# 6:35 B	Sample#	Sample#
Methanol	81.365	81.103	81.750	81.772		
Ethanol	3.670	3.698	3.568	3.552		
1-Propanol	3.755	3.773	3.645	3.627		
iso-Propanol	0.042	0.043	0.045	0.042		
1-Butanol	0.830	0.824	0.801	0.796		
2-Butanol	0.262	0.259	0.255	0.254		
iso-Butanol	2.638	2.631	2.551	2.533		
2-Methyl-1-Butanol	0.770	0.776	0.748	0.740		
1-Pentanol	0.442	0.439	0.428	0.423		
2-Methyl-1-Pentanol	0.449	0.451	0.438	0.433		
1-Hexanol	0.178	0.176	0.171	0.173		
2-Methyl-1-Isobutyrate	0.212	0.214	0.206	0.205		
Methyl Acetate	0.873	0.865	0.830	0.839		
Ethyl Acetate	0.000	0.022	0.019	0.009		
Methyl Formate	0.619	0.845	0.851	0.851		
DME	0.000	0.000	0.000	0.000		
CO2	0.000	0.000	0.000	0.000		
Water	0.257	0.273	0.284	0.273		
Oil + Others (*)	0.709	0.709	0.575	0.575		
Total	97.071	97.101	97.165	97.097		

The balance of each sample comprises mainly higher alcohol isomers which are not individually analyzed. Assumed to average C6-OH.

(*) Standard oil analysis by evaporation yielded higher than typical results. Actual oil is probably 0.1-0.2 wt%. Balance is presumably other, less volatile higher alcohols.

RUN NO: AF-R10.4 TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS : SV=8200

		FRESH MAKE-UP	RECYCLE	HP H2 MAKEUP	DRY FEED	ALCOHOL INJECT.	REACT FEED	REACT EFFL	22.10 VAPOR	PURGE 1 PIC-201	PURGE 2 22.11	PURGE 3 07.20	LIQUID PROD
T	F	293.2	118.7	78.4	376.4		360.1	556.9	86.6	73.6	61.8	163.4	80.0
P	psig	782.0	816.1	814.4	1323.3		1323.3	1300.1	696.5	658.0	31.5	165.1	1.0
Comp	H2	6.85	21.48	100.00	30.25		30.25	22.64	22.27	21.48	7.25	3.13	0.00
	CO	91.93	72.56	0.00	65.46		65.46	65.11	70.36	72.56	51.92	30.09	0.00
	N2	1.13	2.11	0.00	1.62		1.62	1.85	2.03	2.11	1.08	0.58	0.00
	CH4	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00
	CO2	0.10	3.85	0.00	2.67		2.67	5.10	5.33	3.85	29.13	59.25	0.00
	DME	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.28	0.43	0.00
	MeAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.41
	EtAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.01
	MeFm	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.47
	MeOH	0.00	0.00	0.00	0.00		0.00	5.12	0.00	0.00	10.34	6.52	90.02
	H2O	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.53
	Etoh	0.00	0.00	0.00	0.00		0.00	0.16	0.00	0.00	0.00	0.00	2.78
	1-Proh	0.00	0.00	0.00	0.00		0.00	0.13	0.00	0.00	0.00	0.00	2.18
	iso-Proh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.03
	IBOH	0.00	0.00	0.00	0.00		0.00	0.09	0.00	0.00	0.00	0.00	1.24
	1-Buoh	0.00	0.00	0.00	0.00		0.00	0.02	0.00	0.00	0.00	0.00	0.39
	2-Buoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.12
	2-Methyl 1-Buoh	0.00	0.00	0.00	0.00		0.00	0.02	0.00	0.00	0.00	0.00	0.30
	1-Peoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.17
	2-Methyl 1-Peoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.15
	1-hexanol	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	1.06
	2-Methyl 1-Isobutyrate	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.07
	others	0.00	0.00	0.00	0.00		0.00	0.54	0.00	0.00	0.00	0.00	0.08
	TOTAL	100.00	100.00	100.00	100.00		100.00	100.79	100.00	100.00	100.00	100.00	100.00
Mole Wt	lb/lb mole	26.246	23.042	2.020	20.576		20.576	23.547	23.075	23.042	31.252	37.018	35.212
Flow	SCFH	25279	108627	22219	156125		156125	136982	124667	12682	220	2909	9056
	lb mole/hr	65.38	280.95	57.47	403.79		403.79	354.28	322.43	32.80	0.57	7.52	23.42
	lb/hr	1716.0	6473.5	116.1	8308.5		8308.6	8342.3	7440.0	755.7	17.8	278.5	824.7

RUN NO: AF-R10.5 **TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS : SV=8200**

Balance Period:

Start Date	4/3/94	2:00
End Date	4/3/94	14:00

Time From Start of Run (hr)

Start	146.92
End	158.92

Reaction Conditions

Temperature (°F)	573
Pressure (psig)	1735
Space Velocity (sL/kg-hr)	8281
Vg (inlet)	0.44

Slurry Data

Catalyst Weight (lb oxide)	1100
Slurry Concentration (wt %)	40.9
Slurry Level (%)	91
Gas Holdup (vol %)	41.8

Performance Results

CO Conversion (%)	15.7
H2 Conversion (%)	47.9
Syngas Conversion (%)	25.7
MeOH Production (g/kg-oxide-hr)	718.8
IBOH Production (g/kg oxide-hr)	23.2
C2-C6 OH Production (g/kg oxide-hr)	95.9

Atom Balance Closure (% of inlet)

C	-0.64
H	1.76
O	-1.03
N	0.14

Liquid Product Analysis (wt%)

	Sample# 6:30 A	Sample# 6:30 B	Sample# 12:45 A	Sample# 12:45 B	Sample#	Sample#
Methanol	82.469	82.474	83.866	83.906		
Ethanol	3.188	3.176	2.990	2.928		
1-Propanol	3.459	3.461	3.196	3.222		
iso-Propanol	0.041	0.040	0.036	0.038		
1-Butanol	0.755	0.756	0.695	0.703		
2-Butanol	0.243	0.244	0.223	0.226		
iso-Butanol	2.352	2.349	2.006	2.024		
2-Methyl-1-Butanol	0.684	0.685	0.597	0.602		
1-Pentanol	0.403	0.405	0.367	0.374		
2-Methyl-1-Pentanol	0.368	0.358	0.298	0.308		
1-Hexanol	0.159	0.166	0.143	0.147		
2-Methyl-1-Isobutyrate	0.203	0.200	0.165	0.167		
Methyl Acetate	0.948	0.946	0.954	0.944		
Ethyl Acetate	0.000	0.018	0.000	0.000		
Methyl Formate	0.952	0.952	1.113	1.098		
DME	0.000	0.000	0.000	0.000		
CO2	0.000	0.000	0.000	0.000		
Water	0.181	0.183	0.144	0.146		
Oil + Others (*)	0.426	0.426	0.432	0.432		
Total	96.831	96.839	97.225	97.265		

The balance of each sample comprises mainly higher alcohol isomers which are not individually analyzed. Assumed to average C6-OH.

(*) Standard oil analysis by evaporation yielded higher than typical results. Actual oil is probably 0.1-0.2 wt%. Balance is presumably other, less volatile higher alcohols.

RUN NO: AF-R10.5 TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS : SV=8200

		FRESH MAKE-UP	RECYCLE	HP H2 MAKEUP	DRY FEED	ALCOHOL INJECT.	REACT FEED	REACT EFFL	22.10 VAPOR	PURGE 1 PIC-201	PURGE 2 22.11	PURGE 3 07.20	LIQUID PROD
T	F	293.8	123.2	85.9	388.7		371.7	546.8	86.8	74.5	68.9	125.2	80.0
P	psig	783.9	831.9	830.8	1748.0		1748.0	1734.8	695.7	661.6	31.9	163.3	1.0
Comp	H2	7.21	17.00	100.00	29.44		29.44	18.28	16.56	17.00	5.62	2.46	0.00
	CO	91.94	75.87	0.00	65.66		65.66	65.90	74.61	75.87	53.94	31.78	0.00
	N2	0.76	2.98	0.00	2.12		2.12	2.52	2.86	2.98	1.51	0.81	0.00
	CH4	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00
	CO2	0.09	4.15	0.00	2.78		2.78	5.52	5.98	4.15	30.09	61.52	0.00
	DME	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.46	0.53	0.00
	MeAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.45
	EtAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00
	MeFm	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.60
	MeOH	0.00	0.00	0.00	0.00		0.00	7.23	0.00	0.00	8.37	2.90	91.13
	H2O	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.32
	Etoh	0.00	0.00	0.00	0.00		0.00	0.20	0.00	0.00	0.00	0.00	2.34
	1-Proh	0.00	0.00	0.00	0.00		0.00	0.16	0.00	0.00	0.00	0.00	1.95
	iso-Proh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.02
	IBOH	0.00	0.00	0.00	0.00		0.00	0.10	0.00	0.00	0.00	0.00	1.03
	1-Buoh	0.00	0.00	0.00	0.00		0.00	0.02	0.00	0.00	0.00	0.00	0.34
	2-Buoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.11
	2-Methyl 1-Buoh	0.00	0.00	0.00	0.00		0.00	0.02	0.00	0.00	0.00	0.00	0.26
	1-Peoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.15
	2-Methyl 1-Peoh	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.11
	1-hexanol	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	1.07
	2-Methyl 1-Isobutyrate	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.06
	others	0.00	0.00	0.00	0.00		0.00	0.68	0.00	0.00	0.00	0.00	0.05
	TOTAL	100.00	100.00	100.00	100.00		100.00	100.65	100.00	100.00	100.00	100.00	100.00
Mole Wt	lb/lb mole	26.152	24.254	2.020	20.804		20.804	24.893	24.664	24.254	31.783	37.426	34.983
Flow	SCFH	24676	105916	26556	157148		157148	131945	116252	5213	257	3898	11948
	lb mole/hr	63.82	273.94	68.68	406.44		406.44	341.26	300.67	13.48	0.67	10.08	30.90
	lb/hr	1669.0	6644.2	138.7	8455.7		8455.7	8494.7	7415.7	327.0	21.1	377.4	1081.1

RUN NO: **AF-R10.6** **TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYGAS**

Balance Period:

Start Date	4/4/94	0:00
End Date	4/4/94	12:00

Time From Start of Run (hr)

Start	168.92
End	180.92

Reaction Conditions

Temperature (°F)	572
Pressure (psig)	1735
Space Velocity (sL/kg-hr)	3026
Vg (inlet)	0.16

Slurry Data

Catalyst Weight (lb oxide)	1100
Slurry Concentration (wt %)	35.5
Slurry Level (%)	91
Gas Holdup (vol %)	27.9

Performance Results

CO Conversion (%)	20.9
H2 Conversion (%)	48.3
Syngas Conversion (%)	29.3
MeOH Production (g/kg-oxide-hr)	214.2
IBOH Production (g/kg oxide-hr)	23.8
C2-C6 OH Production (g/kg oxide-hr)	60.6

Atom Balance Closure (% of inlet)

C	1.55
H	0.75
O	-0.27
N	0.09

Liquid Product Analysis (wt%)

	Sample# 4:50 A	Sample# 4:50 B	Sample# 10:45 A	Sample# 10:45 B	Sample#	Sample#
Methanol	68.455	68.637	68.662	68.799		
Ethanol	2.824	2.833	2.815	2.832		
1-Propanol	5.003	4.995	5.017	5.033		
iso-Propanol	0.041	0.042	0.045	0.041		
1-Butanol	1.002	0.998	1.001	1.001		
2-Butanol	0.265	0.262	0.263	0.259		
iso-Butanol	7.178	7.147	7.155	7.176		
2-Methyl-1-Butanol	1.646	1.615	1.618	1.621		
1-Pentanol	0.581	0.575	0.578	0.576		
2-Methyl-1-Pentanol	0.845	0.832	0.835	0.836		
1-Hexanol	0.218	0.211	0.210	0.213		
2-Methyl-1-Isobutyrate	0.987	0.991	0.981	0.980		
Methyl Acetate	1.295	1.313	1.285	1.277		
Ethyl Acetate	0.032	0.030	0.029	0.039		
Methyl Formate	0.000	0.000	0.000	0.000		
DME	0.000	0.000	0.000	0.000		
CO2	0.000	0.000	0.000	0.000		
Water	1.641	1.676	1.635	1.619		
Oil + Others (*)	1.156	1.156	1.687	1.687		
Total	93.169	93.313	93.816	93.989		

The balance of each sample comprises mainly higher alcohol isomers which are not individually analyzed. Assumed to average C6-OH.

(*) Standard oil analysis by evaporation yielded higher than typical results. Actual oil is probably 0.1-0.2 wt%. Balance is presumably other, less volatile higher alcohols.

RUN NO: AF-R10.6 TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS

		FRESH MAKE-UP	RECYCLE	HP H2 MAKEUP	DRY FEED	ALCOHOL INJECT.	REACT FEED	REACT EFFL	22.10 VAPOR	PURGE 1 PIC-201	PURGE 2 22.11	PURGE 3 07.20	LIQUID PROD
T	F	266.9	131.6	70.0	340.9		318.6	547.3	75.7	68.6	64.8	133.1	80.0
P	psig	720.0	819.9	846.8	1730.9		1730.9	1735.0	733.0	720.0	30.3	158.5	1.0
Comp	H2	42.85	16.53	100.00	29.44		29.44	18.47	17.42	16.53	4.52	1.93	0.00
	CO	56.46	76.66	0.00	66.74		66.74	64.04	72.09	76.66	46.52	24.35	0.00
	N2	0.61	2.04	0.00	1.34		1.34	1.63	1.86	2.04	0.96	0.43	0.00
	CH4	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00
	CO2	0.07	4.78	0.00	2.48		2.48	7.94	8.61	4.78	42.63	68.98	0.00
	DME	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.74	1.20	0.00
	MeAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.67
	EtAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.01
	MeFm	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00
	MeOH	0.00	0.00	0.00	0.00		0.00	6.01	0.00	0.00	4.62	3.12	81.68
	H2O	0.00	0.00	0.00	0.00		0.00	0.00	0.02	0.00	0.02	0.00	3.47
	Etoh	0.00	0.00	0.00	0.00		0.00	0.18	0.00	0.00	0.00	0.00	2.34
	1-Proh	0.00	0.00	0.00	0.00		0.00	0.23	0.00	0.00	0.00	0.00	3.18
	Iso-Proh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.03
	IBOH	0.00	0.00	0.00	0.00		0.00	0.29	0.00	0.00	0.00	0.00	3.68
	1-Buoh	0.00	0.00	0.00	0.00		0.00	0.03	0.00	0.00	0.00	0.00	0.51
	2-Buoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.13
	2-Methyl 1-Buoh	0.00	0.00	0.00	0.00		0.00	0.04	0.00	0.00	0.00	0.00	0.70
	1-Peoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.25
	2-Methyl 1-Peoh	0.00	0.00	0.00	0.00		0.00	0.02	0.00	0.00	0.00	0.00	0.31
	1-hexanol	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	2.48
	2-Methyl 1-Isobutyrate	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.37
	others	0.00	0.00	0.00	0.00		0.00	1.09	0.00	0.00	0.00	0.00	0.18
	TOTAL	100.00	100.00	100.00	100.00		100.00	100.00	100.00	100.00	100.00	100.00	100.00
Mole Wt	lb/lb mole	16.884	24.479	2.020	20.756		20.757	25.563	24.860	24.479	33.972	38.886	37.720
Flow	SCFH	27204	30223	0	57427		57427	47330	41302	7520	236	2575	4002
	lb mole/hr	70.36	78.17	0.00	148.53		148.53	122.41	106.82	19.45	0.61	6.66	10.35
	lb/hr	1187.9	1913.5	0.0	3082.9		3082.9	3129.3	2655.5	476.1	20.8	259.0	390.4

RUN NO: AF-R10.7 **TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS**

Balance Period:

Start Date	4/4/94	17:00
End Date	4/5/94	8:00

Time From Start of Run (hr)

Start	185.92
End	200.92

Reaction Conditions

Temperature (°F)	572
Pressure (psig)	1300
Space Velocity (sL/kg-hr)	5070
Vg (inlet)	0.36

Slurry Data

Catalyst Weight (lb oxide)	1100
Slurry Concentration (wt %)	38.9
Slurry Level (%)	91
Gas Holdup (vol %)	37.5

Performance Results

CO Conversion (%)	14.2
H2 Conversion (%)	39.4
Syngas Conversion (%)	22.1
MeOH Production (g/kg-oxide-hr)	329.8
IBOH Production (g/kg oxide-hr)	19.2
C2-C6 OH Production (g/kg oxide-hr)	64.2

Atom Balance Closure (% of inlet)

C	0.56
H	-0.94
O	-0.13
N	-0.82

Liquid Product Analysis (wt%)

	Sample# 0:35 A	Sample# 0:35 B	Sample# 5:30 A	Sample# 5:30 B	Sample#	Sample#
Methanol	76.516	76.569	78.674	78.542		
Ethanol	3.212	3.242	3.297	3.291		
1-Propanol	4.444	4.474	4.292	4.318		
iso-Propanol	0.040	0.041	0.040	0.040		
1-Butanol	0.893	0.900	0.858	0.863		
2-Butanol	0.885	0.886	0.741	0.745		
iso-Butanol	4.415	4.446	3.763	3.792		
2-Methyl-1-Butanol	1.085	1.109	0.958	0.971		
1-Pentanol	0.490	0.496	0.466	0.465		
2-Methyl-1-Pentanol	0.599	0.603	0.514	0.523		
1-Hexanol	0.193	0.190	0.177	0.179		
2-Methyl-1-Isobutyrate	0.452	0.453	0.321	0.322		
Methyl Acetate	0.940	0.934	0.849	0.833		
Ethyl Acetate	0.024	0.026	0.000	0.000		
Methyl Formate	0.000	0.000	0.000	0.000		
DME	0.000	0.000	0.000	0.000		
CO2	0.000	0.000	0.000	0.000		
Water	0.906	0.890	0.823	0.795		
Oil + Others (*)	1.020	1.020	0.700	0.700		
Total	96.114	96.279	96.473	96.379		

The balance of each sample comprises mainly higher alcohol isomers which are not individually analyzed. Assumed to average C6-OH.

(*) Standard oil analysis by evaporation yielded higher than typical results. Actual oil is probably 0.1-0.2 wt%. Balance is presumably other, less volatile higher alcohols.

RUN NO: AF-R10.7 TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS

		FRESH MAKE-UP	RECYCLE	HP H2 MAKEUP	DRY FEED	ALCOHOL INJECT.	REACT FEED	REACT EFFL	22.10 VAPOR	PURGE 1 PIC-201	PURGE 2 22.11	PURGE 3 07.20	LIQUID PROD
T	F	274.2	141.9	76.6	365.8		345.8	554.1	84.1	74.0	72.0	133.3	80.0
P	psig	756.2	882.4	835.7	1309.9		1309.9	1300.1	718.8	700.5	30.3	150.0	1.0
Comp	H2	51.50	21.04	100.00	29.96		29.96	21.02	21.79	21.04	6.93	3.01	0.00
	CO	47.84	72.45	0.00	65.29		65.29	64.82	69.90	72.45	51.78	29.74	0.00
	N2	0.60	2.77	0.00	2.10		2.10	2.43	2.65	2.77	1.36	0.73	0.00
	CH4	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00
	CO2	0.06	3.75	0.00	2.65		2.65	5.29	5.65	3.75	32.27	63.25	0.00
	DME	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.28	0.62	0.00
	MeAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.43
	EtAc	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.01
	MeFm	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.00
	MeOH	0.00	0.00	0.00	0.00		0.00	5.27	0.00	0.00	7.36	2.64	87.40
	H2O	0.00	0.00	0.00	0.00		0.00	0.00	0.01	0.00	0.01	0.00	1.71
	Etoh	0.00	0.00	0.00	0.00		0.00	0.16	0.00	0.00	0.00	0.00	2.55
	1-Proh	0.00	0.00	0.00	0.00		0.00	0.15	0.00	0.00	0.00	0.00	2.63
	iso-Proh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.02
	IBOH	0.00	0.00	0.00	0.00		0.00	0.13	0.00	0.00	0.00	0.00	2.00
	1-Buoh	0.00	0.00	0.00	0.00		0.00	0.02	0.00	0.00	0.00	0.00	0.43
	2-Buoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.40
	2-Methyl 1-Buoh	0.00	0.00	0.00	0.00		0.00	0.02	0.00	0.00	0.00	0.00	0.42
	1-Peoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.20
	2-Methyl 1-Peoh	0.00	0.00	0.00	0.00		0.00	0.01	0.00	0.00	0.00	0.00	0.20
	1-hexanol	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	1.37
	2-Methyl 1-Isobutyrate	0.00	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00	0.00	0.14
	others	0.00	0.00	0.00	0.00		0.00	0.66	0.00	0.00	0.00	0.00	0.10
	TOTAL	100.00	100.00	100.00	100.00		100.00	100.00	100.00	100.00	100.00	100.00	100.00
Mole Wt	lb/lb mole	14.634	23.140	2.020	20.649		20.649	24.074	23.248	23.140	31.718	37.568	35.858
Flow	SCFH	28789	67430	0	96219		96219	83168	76637	5537	221	2591	5457
	lb mole/hr	74.46	174.40	0.00	248.86		248.86	215.10	198.21	14.32	0.57	6.70	14.11
	lb/hr	1089.6	4035.6	0.0	5138.5		5138.5	5178.4	4608.1	331.4	18.1	251.8	506.1

RUN NO: **AF-R10.8** **TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS**

Balance Period:

Start Date	4/6/94	3:00
End Date	4/6/94	20:00

Time From Start of Run (hr)

Start	219.92
End	236.92

Reaction Conditions

Temperature (°F)	572
Pressure (psig)	1300
Space Velocity (sL/kg-hr)	5494
Vg (inlet)	0.39

Slurry Data

Catalyst Weight (lb oxide)	1100
Slurry Concentration (wt %)	40.1
Slurry Level (%)	91
Gas Holdup (vol %)	40.6

Performance Results

CO Conversion (%)	6.8
H2 Conversion (%)	5.0
Syngas Conversion (%)	6.2
MeOH Production (g/kg-oxide-hr)	634.1
IBOH Production (g/kg oxide-hr)	55.9
C2-C6 OH Production (g/kg oxide-hr)	190.8

Atom Balance Closure (% of inlet)

C	-0.89
H	-6.54
O	-0.89
N	0.66

Liquid Product Analysis (wt%)

	Sample# 07:45 A	Sample# 07:45 B	Sample# 12:30 A	Sample# 12:30 B	Sample#	Sample#
Methanol	75.039	74.974	73.427	73.382		
Ethanol	3.819	3.914	4.113	4.110		
1-Propanol	8.093	8.129	8.613	8.626		
iso-Propanol	0.029	0.028	0.031	0.031		
1-Butanol	0.725	0.724	0.759	0.762		
2-Butanol	0.178	0.183	0.180	0.181		
iso-Butanol	4.504	4.528	5.089	5.116		
2-Methyl-1-Butanol	0.602	0.612	0.519	0.519		
1-Pentanol	0.358	0.358	0.375	0.375		
2-Methyl-1-Pentanol	0.298	0.299	0.281	0.285		
1-Hexanol	0.137	0.140	0.146	0.144		
2-Methyl-1-Isobutyrate	0.313	0.310	0.359	0.358		
Methyl Acetate	1.023	1.012	1.134	1.136		
Ethyl Acetate	0.034	0.039	0.036	0.035		
Methyl Formate	0.000	0.000	0.000	0.000		
DME	0.000	0.000	0.000	0.000		
CO2	0.000	0.000	0.000	0.000		
Water	0.622	0.613	0.624	0.625		
Oil + Others (*)	0.499	0.499	0.443	0.443		
Total	96.273	96.362	96.129	96.128		

The balance of each sample comprises mainly higher alcohol isomers which are not individually analyzed. Assumed to average C6-OH.

(*) Standard oil analysis by evaporation yielded higher than typical results. Actual oil is probably 0.1-0.2 wt%. Balance is presumably other, less volatile higher alcohols.

RUN NO: AF-R10.8 TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS

		FRESH MAKE-UP	RECYCLE	HP H2 MAKEUP	DRY FEED	ALCOHOL INJECT.	REACT FEED	REACT EFFL	22.10 VAPOR	PURGE 1 PIC-201	PURGE 2 22.11	PURGE 3 07.20	LIQUID PROD
T	F	255.2	128.7	63.3	404.9	100.0	439.0	566.6	73.1	62.5	54.3	114.6	80.0
P	psig	753.3	883.1	830.3	1310.2	1310.2	1310.2	1300.0	716.9	691.4	31.6	136.5	1.0
Comp	H2	32.83	29.08	100.00	29.95	0.00	27.61	26.72	28.61	29.08	6.93	4.55	0.00
	CO	66.28	65.74	0.00	65.86	0.00	60.72	57.68	64.84	65.74	51.79	29.55	0.00
	N2	0.81	1.26	0.00	1.15	0.00	1.06	1.08	1.23	1.26	1.36	0.38	0.00
	CH4	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	CO2	0.08	3.92	0.00	3.04	0.00	2.81	4.82	5.32	3.92	32.27	63.74	0.00
	DME	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.28	0.78	0.00
	MeAc	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.53
	EtAc	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01
	MeFm	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	MeOH	0.00	0.00	0.00	0.00	90.20	7.03	8.23	0.00	0.00	7.36	1.00	84.98
	H2O	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.01	0.00	1.26
	Etoh	0.00	0.00	0.00	0.00	3.65	0.28	0.33	0.00	0.00	0.00	0.00	3.18
	1-Proh	0.00	0.00	0.00	0.00	6.12	0.48	0.55	0.00	0.00	0.00	0.00	5.11
	iso-Proh	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02
	IBOH	0.00	0.00	0.00	0.00	0.00	0.00	0.31	0.00	0.00	0.00	0.00	2.38
	1-Buoh	0.00	0.00	0.00	0.00	0.00	0.00	0.03	0.00	0.00	0.00	0.00	0.37
	2-Buoh	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.09
	2-Methyl 1-Buoh	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.23
	1-Peoh	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.15
	2-Methyl 1-Peoh	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.10
	1-hexanol	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	1.41
	2-Methyl 1-Isobutyrate	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.12
	others	0.00	0.00	0.00	0.00	0.04	0.00	0.18	0.00	0.00	0.00	0.00	0.06
	TOTAL	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Mole Wt	lb/lb mole	19.490	21.081	2.020	20.713	34.282	21.771	22.670	21.425	21.081	31.719	37.207	36.554
Flow	SCFH	22572	73550	0	96122	8128	104251	102334	89417	13740	53	2165	10300
	lb mole/hr	58.38	190.23	0.00	248.61	21.02	269.63	264.67	231.26	35.54	0.14	5.60	26.64
	lb/hr	1137.8	4010.1	0.0	5149.5	720.7	5870.2	6000.0	4954.9	749.1	4.4	208.3	973.8

RUN NO: AF-R10.9 **TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS**

Balance Period:			**Time From Start of Run (hr)**	
Start Date	4/7/94	16:00	Start	256.92
End Date	4/8/94	4:00	End	268.92

Reaction Conditions		**Slurry Data**	
Temperature (°F)	572	Catalyst Weight (lb oxide)	1100
Pressure (psig)	1300	Slurry Concentration (wt %)	39.1
Space Velocity (sL/kg-hr)	5154	Slurry Level (%)	91
Vg (inlet)	0.36	Gas Holdup (vol %)	37.9

Performance Results		**Atom Balance Closure (% of inlet)**	
CO Conversion (%)	11.2	C	0.94
H2 Conversion (%)	37.1	H	-0.21
Syngas Conversion (%)	19.5	O	0.69
MeOH Production (g/kg-oxide-hr)	424.5	N	0.07
IBOH Production (g/kg oxide-hr)	18.4		
C2-C6 OH Production (g/kg oxide-hr)	79.4		

Liquid Product Analysis (wt%)

	Sample# 16:30 A	Sample# 16:30 B	Sample# 1:20 A	Sample# 1:20 B	Sample# 4:15 A	Sample# 4:15 B
Methanol	79.413	79.338	83.034	82.999	84.184	84.182
Ethanol	3.522	3.594	4.138	4.134	4.033	4.025
1-Propanol	5.629	5.638	5.936	5.930	6.097	6.106
iso-Propanol	0.026	0.028	0.023	0.021	0.021	0.020
1-Butanol	0.710	0.713	0.498	0.495	0.396	0.396
2-Butanol	0.170	0.169	0.116	0.115	0.104	0.103
iso-Butanol	3.719	3.719	2.031	2.031	1.629	1.628
2-Methyl-1-Butanol	0.591	0.586	0.249	0.246	0.182	0.181
1-Pentanol	0.346	0.345	0.183	0.182	0.140	0.140
2-Methyl-1-Pentanol	0.313	0.315	0.127	0.125	0.091	0.088
1-Hexanol	0.139	0.139	0.097	0.099	0.078	0.076
2-Methyl-1-Isobutyrate	0.240	0.240	0.091	0.092	0.061	0.065
Methyl Acetate	0.878	0.874	0.738	0.745	0.622	0.622
Ethyl Acetate	0.030	0.022	0.022	0.032	0.000	0.000
Methyl Formate	0.000	0.000	0.000	0.000	0.000	0.000
DME	0.000	0.000	0.000	0.000	0.000	0.000
CO2	0.000	0.000	0.000	0.000	0.000	0.000
Water	0.691	0.688	0.650	0.660	0.619	0.622
Oil + Others (*)	0.522	0.522	0.539	0.539	0.532	0.532
Total	96.939	96.930	98.472	98.445	98.789	98.786

The balance of each sample comprises mainly higher alcohol isomers which are not individually analyzed. Assumed to average C6-OH.

(*) Standard oil analysis by evaporation yielded higher than typical results. Actual oil is probably 0.1-0.2 wt%. Balance is presumably other, less volatile higher alcohols.

RUN NO: AF-R10.9 TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS

		FRESH MAKE-UP	RECYCLE	HP H2 MAKEUP	DRY FEED	ALCOHOL INJECT.	REACT FEED	REACT EFFL	22.10 VAPOR	PURGE 1 PIC-201	PURGE 2 22.11	PURGE 3 07.20	LIQUID PROD
T	F	269.6	136.3	68.1	376.3	100.0	461.4	557.0	76.3	66.3	61.2	138.6	80.0
P	psig	759.5	883.0	837.7	1309.6	1309.6	1309.6	1300.0	720.6	699.0	31.3	124.3	1.0
Comp	H2	49.16	23.80	100.00	30.67	0.00	30.18	21.65	23.45	23.80	6.93	4.87	0.00
	CO	50.19	71.76	0.00	65.92	0.00	64.86	65.72	71.25	71.76	51.79	41.72	0.00
	N2	0.58	2.14	0.00	1.72	0.00	1.69	1.93	2.10	2.14	1.36	0.80	0.00
	CH4	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	CO2	0.06	2.30	0.00	1.69	0.00	1.66	2.93	3.20	2.30	32.27	49.28	0.00
	DME	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.28	0.73	0.00
	MeAc	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.35
	EtAc	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01
	MeFm	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	MeOH	0.00	0.00	0.00	0.00	81.76	1.32	6.58	0.00	0.00	7.36	2.59	89.43
	H2O	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.01	0.00	1.27
	Etoh	0.00	0.00	0.00	0.00	7.07	0.11	0.24	0.00	0.00	0.00	0.00	2.96
	1-Proh	0.00	0.00	0.00	0.00	11.14	0.18	0.26	0.00	0.00	0.00	0.00	3.42
	iso-Proh	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01
	IBOH	0.00	0.00	0.00	0.00	0.00	0.00	0.12	0.00	0.00	0.00	0.00	1.16
	1-Buoh	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.25
	2-Buoh	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.06
	2-Methyl 1-Buoh	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.13
	1-Peoh	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.09
	2-Methyl 1-Peoh	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.06
	1-hexanol	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.70
	2-Methyl 1-Isobutyrate	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.04
	others	0.00	0.00	0.00	0.00	0.04	0.00	0.53	0.00	0.00	0.00	0.00	0.06
	TOTAL	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Mole Wt	lb/lb mole	15.243	22.191	2.020	20.308	36.168	20.564	23.515	22.427	22.191	31.719	34.865	34.716
Flow	SCFH	26589	69646	0	96235	1580	97814	85697	78387	6772	80	1452	6786
	lb mole/hr	68.77	180.13	0.00	248.90	4.09	252.98	221.64	202.74	17.52	0.21	3.76	17.55
	lb/hr	1048.2	3997.3	0.0	5054.5	147.8	5202.2	5211.9	4546.8	388.7	6.5	130.9	609.3

RUN NO: AF-R10.10 **TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS**

Balance Period:

Start Date	4/8/94	19:00
End Date	4/9/94	8:00

Time From Start of Run (hr)

Start	283.92
End	296.92

Reaction Conditions

Temperature (°F)	572.50
Pressure (psig)	749.91
Space Velocity (sL/kg-hr)	5068
Vg (inlet)	0.62

Slurry Data

Catalyst Weight (lb oxide)	1100
Slurry Concentration (wt %)	40.8
Slurry Level (%)	91
Gas Holdup (vol %)	42.1

Performance Results

CO Conversion (%)	2.0
H2 Conversion (%)	9.3
Syngas Conversion (%)	4.3
MeOH Production (g/kg-oxide-hr)	76.3
IBOH Production (g/kg oxide-hr)	0.0
C2-C6 OH Production (g/kg oxide-hr)	0.0

Atom Balance Closure (% of inlet)

C	0.30
H	-0.78
O	0.55
N	0.65

Liquid Product Analysis (wt%)

	Sample# 5:40 A	Sample# 5:40 B	Sample#	Sample#	Sample#	Sample#
Methanol	84.932	84.897				
Ethanol	3.674	3.715				
1-Propanol	7.851	7.907				
iso-Propanol	0.060	0.060				
1-Butanol	0.101	0.082				
2-Butanol	0.072	0.071				
iso-Butanol	0.789	0.792				
2-Methyl-1-Butanol	0.088	0.089				
1-Pentanol	0.033	0.032				
2-Methyl-1-Pentanol	0.054	0.041				
1-Hexanol	0.015	0.019				
2-Methyl-1-Isobutyrate	0.025	0.024				
Methyl Acetate	0.257	0.258				
Ethyl Acetate	0.000	0.000				
Methyl Formate	0.000	0.000				
DME	0.000	0.000				
CO2	0.000	0.000				
Water	0.939	0.927				
Oil	0.200	0.200				
Total	99.090	99.114				

The balance of each sample comprises mainly higher alcohol isomers which are not individually analyzed. Assumed to average C6-OH.

RUN NO: AF-R10.10 TITLE: LPIBOH over Cs-Promoted BASF S3-86 CATALYST with SHELL SYNGAS

		FRESH MAKE-UP	RECYCLE	HP H2 MAKEUP	DRY FEED	ALCOHOL INJECT.	REACT FEED	REACT EFFL	22.10 VAPOR	PURGE 1 PIC-201	PURGE 2 22.11	PURGE 3 07.20	LIQUID PROD
T	F	284.8	130.9	74.3	398.3	100.0	473.5	567.2	82.1	71.4	69.0	162.3	80.0
P	psig	805.7	845.0	851.9	777.3	777.3	777.3	749.9	714.8	691.4	29.8	125.1	1.0
Comp	H2	35.01	29.51	100.00	30.40	0.00	30.40	28.46	29.14	29.51	28.32	6.07	0.00
	CO	57.63	67.40	0.00	65.83	0.00	65.83	66.52	66.96	67.40	66.46	39.12	0.00
	N2	0.77	1.04	0.00	0.99	0.00	0.99	1.03	1.03	1.04	1.35	0.41	0.00
	CH4	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	CO2	6.59	2.05	0.00	2.77	0.00	2.77	2.81	2.85	2.05	2.52	47.58	0.00
	DME	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.20	0.00
	MeAc	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.12
	EtAc	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	MeFm	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	MeOH	0.00	0.00	0.00	0.00	0.00	0.00	1.09	0.00	0.00	1.35	6.63	90.07
	H2O	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.01	0.00	1.76
	Etoh	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.72
	1-Proh	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	4.45
	iso-Proh	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.03
	IBOH	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.36
	1-Buoh	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.04
	2-Buoh	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.03
	2-Methyl 1-Buoh	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.03
	1-Peoh	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01
	2-Methyl 1-Peoh	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02
	1-hexanol	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.30
	2-Methyl 1-Isobutyrate	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01
	others	0.00	0.00	0.00	0.00	100.00	0.00	0.10	0.00	0.00	0.00	0.00	0.02
	TOTAL	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Mole Wt	lb/lb mole	19.965	20.668	2.020	20.551	58.883	20.551	21.137	20.891	20.668	21.106	34.347	33.931
Flow	SCFH	15598	80582	0	96180	0	96180	93249	92261	10029	4	1766	1207
	lb mole/hr	40.34	208.41	0.00	248.76	0.00	248.76	241.17	238.62	25.94	0.01	4.57	3.12
	lb/hr	805.4	4307.6	0.0	5112.3	0.0	5112.3	5097.7	4985.1	536.1	0.2	156.9	105.9

APPENDIX D

LABORATORY RESULTS FOR PARALLEL RUN PLAN

300cc #2 AUTOCLAVE RUN SUMMARY

Catalyst: BASF S3-86 CsLaporte Sample13465-26 **Liquid:** Drakeol 10

Run Description: mixed alcohols

Run#:	13458-73B	**Gas Type:**	Shell	**Feed MeOH Rate (ml/hr):**	0
Date:	3/7/94	**Press (psig):**	748	**Feed EtOH Rate (ml/hr):**	0
Hrs:	49	**Temp. (C):**	299.9	**Feed PrOH Rate (ml/hr):**	0
Cat wt (g):	30.01	**Inlet Flow (sccm):**	2596.6	**Syngas GHSV (sl/kg-hr):**	5191.5
Liq wt (g):	120.03	**Exit Flow (sccm):**	2310.7	**Total GHSV (sl/kg-hr):**	5191.5

BULK GASES

Stream	H2(%)	N2(%)	CO(%)	CO2(%)	H2O(%)	Totals (%)
Inlet:	30.583	1.018	65.194	3.063		99.86
Exit:	24.183	1.144	65.004	5.721	0.0497	99.82

ORGANIC PRODUCTS

Product	Conc. (ppm)	Molar Rate (gmole /kg-hr)	Weight Rate (g/kg-hr)	CO2-free Molar Select. (mol%)	CO2-free Weight Select. (wt%)
methanol	30626.830	6.311	202.225	82.420	72.221
ethanol	1075.120	0.222	10.207	2.893	3.645
1-propanol	1009.570	0.208	12.497	2.717	4.463
isobutanol	1494.140	0.308	22.822	4.021	8.151
2-butanol	77.160	0.016	1.179	0.208	0.421
1-butanol	178.140	0.037	2.721	0.479	0.972
2-methyl-1-butanol	340.630	0.070	6.188	0.917	2.210
1-pentanol	88.670	0.018	1.611	0.239	0.575
2-methyl-1-pentanol	308.900	0.064	6.504	0.831	2.323
1-hexanol	67.210	0.014	1.415	0.181	0.505
DME	87.570	0.018	0.831	0.236	0.297
methane	930.470	0.192	3.076	2.504	1.099
ethane	389.640	0.080	2.414	1.049	0.862
propane	89.590	0.018	0.814	0.241	0.291
butane	1.980	0.000	0.024	0.005	0.008
pentane	16.450	0.003	0.245	0.044	0.087
hexane	0.000	0.000	0.000	0.000	0.000
methyl formate	182.000	0.038	2.252	0.490	0.804
methyl acetate	195.580	0.040	2.986	0.526	1.066
methyl isobutyrate	138.220	0.028	2.909	0.372	1.039
ethyl acetate	19.020	0.004	0.345	0.051	0.123
TOTAL	37159.65	7.657	280.009	100.000	100.000
Sum C1-C6 Alcohols	35266.370	7.267	267.367	94.905	95.485
Sum C2-C6 Alcohols	4639.540	0.956	65.142	12.485	23.264
Sum C2-C5 Alcohols	4263.430	0.879	57.223	11.473	20.436
Sum C1-C3 Alcohols	32711.520	6.741	224.928	88.030	80.329
Sum C4-C6 Alcohols	2554.850	0.526	42.439	6.875	15.156
EtOH+PrOH	2084.690	0.430	22.703	5.610	8.108
EtOH+PrOH+iBuOH	3578.830	0.737	45.526	9.631	16.259
Sum C1-C6 HCs	1428.130	0.294	6.573	3.843	2.347
Sum Esters	534.820	0.110	8.492	1.439	3.033

Material balances:
(Element Recoveries - %)

Nitrogen(N):	100.00
Carbon(C):	98.88
Hydrogen(H):	96.10
Oxygen(O):	99.99

Calculations:

CO conv. (%):	11.27
H2 conv. (%):	29.63
CO+H2 conv. (%):	17.13
Net CO2 rate gmol/kg-hr):	4.70

300cc #2 AUTOCLAVE RUN SUMMARY

Catalyst: BASF S3-86 CsLaporte Sample13465-26 **Liquid:** Drakeol 10

Run Description: mixed alcohols

Run#:	13458-73C	**Gas Type:**	Shell	**Feed MeOH Rate (ml/hr):**	0
Date:	3/8/94	**Press (psig):**	750	**Feed EtOH Rate (ml/hr):**	0
Hrs:	69	**Temp. (C):**	300	**Feed PrOH Rate (ml/hr):**	0
Cat wt (g):	30.01	**Inlet Flow (sccm):**	1566.3	**Syngas GHSV (sl/kg-hr):**	3131.6
Liq wt (g):	120.03	**Exit Flow (sccm):**	1370.89	**Total GHSV (sl/kg-hr):**	3131.6

BULK GASES

Stream	H2(%)	N2(%)	CO(%)	CO2(%)	H2O(%)	Totals (%)
Inlet:	30.552	1.017	65.110	3.024		99.70
Exit:	23.723	1.162	64.443	6.518	0.0576	99.77

ORGANIC PRODUCTS

Product	Conc. (ppm)	Molar Rate (gmole /kg-hr)	Weight Rate (g/kg-hr)	CO2-free Molar Select. (mol%)	CO2-free Weight Select. (wt%)
methanol	30418.950	3.719	119.161	78.584	67.089
ethanol	1061.480	0.130	5.978	2.742	3.366
1-propanol	1150.400	0.141	8.448	2.972	4.756
isobutanol	2125.400	0.260	19.260	5.491	10.844
2-butanol	87.300	0.011	0.791	0.226	0.445
1-butanol	199.200	0.024	1.805	0.515	1.016
2-methyl-1-butanol	455.440	0.056	4.908	1.177	2.763
1-pentanol	97.700	0.012	1.053	0.252	0.593
2-methyl-1-pentanol	466.960	0.057	5.833	1.206	3.284
1-hexanol	83.780	0.010	1.047	0.216	0.589
DME	116.780	0.014	0.658	0.302	0.370
methane	1411.840	0.173	2.769	3.647	1.559
ethane	498.820	0.061	1.834	1.289	1.032
propane	126.990	0.016	0.685	0.328	0.385
butane	2.470	0.000	0.018	0.006	0.010
pentane	23.890	0.003	0.211	0.062	0.119
hexane	0.000	0.000	0.000	0.000	0.000
methyl formate	172.890	0.021	1.269	0.447	0.715
methyl acetate	208.570	0.025	1.889	0.539	1.064
methyl isobutyrate	198.700	0.024	2.481	0.513	1.397
ethyl acetate	25.400	0.003	0.274	0.066	0.154
TOTAL	38708.86	4.732	177.618	100.000	100.000
Sum C1-C6 Alcohols	36146.610	4.419	168.286	93.381	94.746
Sum C2-C6 Alcohols	5727.660	0.700	49.124	14.797	27.657
Sum C2-C5 Alcohols	5176.920	0.633	42.245	13.374	23.784
Sum C1-C3 Alcohols	32630.830	3.989	133.588	84.298	75.211
Sum C4-C6 Alcohols	3515.780	0.430	34.698	9.083	19.535
EtOH+PrOH	2211.880	0.270	14.427	5.714	8.122
EtOH+PrOH+iBuOH	4337.280	0.530	33.687	11.205	18.966
Sum C1-C6 HCs	2064.010	0.252	5.516	5.332	3.105
Sum Esters	605.560	0.074	5.913	1.564	3.329

Material balances:
(Element Recoveries - %)

Nitrogen(N):	100.00
Carbon(C):	98.47
Hydrogen(H):	95.41
Oxygen(O):	99.98

Calculations:

CO conv. (%):	13.37
H2 conv. (%):	32.04
CO+H2 conv. (%):	19.34
Net CO2 rate gmol/kg-hr):	3.74

300cc #2 AUTOCLAVE RUN SUMMARY

Catalyst: BASF S3-86 CsLaporte Sample13465-26 **Liquid:** Drakeol 10

Run Description: mixed alcohols

Run#:	13458-73E	**Gas Type:**	Shell	**Feed MeOH Rate (ml/hr):**	0
Date:	3/10/94	**Press (psig):**	750	**Feed EtOH Rate (ml/hr):**	0
Hrs:	116.8	**Temp. (C):**	300.3	**Feed PrOH Rate (ml/hr):**	0
Cat wt (g):	30.01	**Inlet Flow (sccm):**	4252.5	**Syngas GHSV (sl/kg-hr):**	8502.2
Liq wt (g):	120.03	**Exit Flow (sccm):**	3870.23	**Total GHSV (sl/kg-hr):**	8502.2

BULK GASES

Stream	H2(%)	N2(%)	CO(%)	CO2(%)	H2O(%)	Totals (%)
Inlet:	30.461	1.012	64.835	3.011		99.32
Exit:	24.852	1.112	65.191	4.519	0.0411	99.24

ORGANIC PRODUCTS

Product	Conc. (ppm)	Molar Rate (gmole /kg-hr)	Weight Rate (g/kg-hr)	CO2-free Molar Select. (mol%)	CO2-free Weight Select. (wt%)
methanol	30979.230	10.692	342.607	87.948	80.380
ethanol	944.210	0.326	15.014	2.681	3.522
1-propanol	750.320	0.259	15.556	2.130	3.650
isobutanol	804.550	0.278	20.583	2.284	4.829
2-butanol	55.960	0.019	1.432	0.159	0.336
1-butanol	126.800	0.044	3.244	0.360	0.761
2-methyl-1-butanol	182.330	0.063	5.547	0.518	1.301
1-pentanol	64.400	0.022	1.959	0.183	0.460
2-methyl-1-pentanol	121.420	0.042	4.282	0.345	1.005
1-hexanol	39.900	0.014	1.407	0.113	0.330
DME	58.010	0.020	0.922	0.165	0.216
methane	505.450	0.174	2.799	1.435	0.657
ethane	186.430	0.064	1.935	0.529	0.454
propane	40.270	0.014	0.613	0.114	0.144
butane	0.000	0.000	0.000	0.000	0.000
pentane	3.350	0.001	0.083	0.010	0.020
hexane	0.000	0.000	0.000	0.000	0.000
methyl formate	207.850	0.072	4.308	0.590	1.011
methyl acetate	154.170	0.053	3.942	0.438	0.925
methyl isobutyrate	55.920	0.019	1.971	0.159	0.462
ethyl acetate	7.510	0.003	0.228	0.021	0.054
TOTAL	35224.65	12.158	426.234	100.000	100.000
Sum C1-C6 Alcohols	34069.120	11.759	411.631	96.720	96.574
Sum C2-C6 Alcohols	3089.890	1.066	69.024	8.772	16.194
Sum C2-C5 Alcohols	2928.570	1.011	63.335	8.314	14.859
Sum C1-C3 Alcohols	32673.760	11.277	373.177	92.758	87.552
Sum C4-C6 Alcohols	1395.360	0.482	38.455	3.961	9.022
EtOH+PrOH	1694.530	0.585	30.570	4.811	7.172
EtOH+PrOH+iBuOH	2499.080	0.863	51.153	7.095	12.001
Sum C1-C6 HCs	735.500	0.254	5.430	2.088	1.274
Sum Esters	425.450	0.147	10.450	1.208	2.452

Material balances:
(Element Recoveries - %)

Nitrogen(N):	100.00
Carbon(C):	99.36
Hydrogen(H):	97.81
Oxygen(O):	99.89

Calculations:

CO conv. (%):	8.49
H2 conv. (%):	25.75
CO+H2 conv. (%):	14.01
Net CO2 rate gmol/kg-hr):	4.18

300cc #2 AUTOCLAVE RUN SUMMARY

Catalyst: BASF S3-86 CsLaporte Sample13465-26 **Liquid:** Drakeol 10

Run Description: mixed alcohols

Run#:	13458-73G	**Gas Type:**	Shell	**Feed MeOH Rate (ml/hr):**	0
Date:	3/12/94	**Press (psig):**	1301	**Feed EtOH Rate (ml/hr):**	0
Hrs:	165.5	**Temp. (C):**	300.3	**Feed PrOH Rate (ml/hr):**	0
Cat wt (g):	30.01	**Inlet Flow (sccm):**	4090.47	**Syngas GHSV (sl/kg-hr):**	8178.2
Liq wt (g):	120.03	**Exit Flow (sccm):**	3490.83	**Total GHSV (sl/kg-hr):**	8178.2

BULK GASES

Stream	H2(%)	N2(%)	CO(%)	CO2(%)	H2O(%)	Totals (%)
Inlet:	30.830	1.036	66.403	3.105		101.37
Exit:	20.344	1.214	67.450	5.335	0.0460	101.09

ORGANIC PRODUCTS

Product	Conc. (ppm)	Molar Rate (gmole /kg-hr)	Weight Rate (g/kg-hr)	CO2-free Molar Select. (mol%)	CO2-free Weight Select. (wt%)
methanol	59962.700	18.667	598.134	89.478	82.746
ethanol	1592.850	0.496	22.844	2.377	3.160
1-propanol	1258.820	0.392	23.540	1.878	3.257
isobutanol	1074.390	0.334	24.792	1.603	3.430
2-butanol	79.150	0.025	1.826	0.118	0.253
1-butanol	211.600	0.066	4.883	0.316	0.675
2-methyl-1-butanol	219.460	0.068	6.022	0.327	0.833
1-pentanol	91.370	0.028	2.507	0.136	0.347
2-methyl-1-pentanol	140.390	0.044	4.466	0.209	0.618
1-hexanol	60.490	0.019	1.924	0.090	0.266
DME	188.890	0.059	2.709	0.282	0.375
methane	797.660	0.248	3.984	1.190	0.551
ethane	145.210	0.045	1.359	0.217	0.188
propane	34.870	0.011	0.479	0.052	0.066
butane	0.000	0.000	0.000	0.000	0.000
pentane	11.460	0.004	0.257	0.017	0.036
hexane	5.130	0.002	0.138	0.008	0.019
methyl formate	755.030	0.235	14.115	1.127	1.953
methyl acetate	384.750	0.120	8.873	0.574	1.228
methyl isobutyrate	101.400	0.032	3.224	0.151	0.446
ethyl acetate	8.050	0.003	0.221	0.012	0.031
TOTAL	67014.22	20.862	722.854	100.000	100.000
Sum C1-C6 Alcohols	64691.220	20.139	690.940	96.534	95.585
Sum C2-C6 Alcohols	4728.520	1.472	92.806	7.056	12.839
Sum C2-C5 Alcohols	4527.640	1.410	86.416	6.756	11.955
Sum C1-C3 Alcohols	62814.370	19.555	644.519	93.733	89.163
Sum C4-C6 Alcohols	1876.850	0.584	46.421	2.801	6.422
EtOH+PrOH	2851.670	0.888	46.385	4.255	6.417
EtOH+PrOH+iBuOH	3926.060	1.222	71.177	5.859	9.847
Sum C1-C6 HCs	994.330	0.310	6.217	1.484	0.860
Sum Esters	1249.230	0.389	26.433	1.864	3.657

Material balances:
(Element Recoveries - %)

Nitrogen(N):	100.00
Carbon(C):	99.19
Hydrogen(H):	96.82
Oxygen(O):	99.79

Calculations:

CO conv. (%):	13.31
H2 conv. (%):	43.69
CO+H2 conv. (%):	22.94
Net CO2 rate gmol/kg-hr):	5.28

300cc #2 AUTOCLAVE RUN SUMMARY

Catalyst: BASF S3-86 CsLaporte Sample13465-26 **Liquid:** Drakeol 10

Run Description: mixed alcohols

Run#:	13458-73H	**Gas Type:**	Shell	**Feed MeOH Rate (ml/hr):**	0
Date:	3/14/94	**Press (psig):**	1734	**Feed EtOH Rate (ml/hr):**	0
Hrs:	212	**Temp. (C):**	299.7	**Feed PrOH Rate (ml/hr):**	0
Cat wt (g):	30.01	**Inlet Flow (sccm):**	4146.27	**Syngas GHSV (sl/kg-hr):**	8289.8
Liq wt (g):	120.03	**Exit Flow (sccm):**	3391.79	**Total GHSV (sl/kg-hr):**	8289.8

BULK GASES

Stream	H2(%)	N2(%)	CO(%)	CO2(%)	H2O(%)	Totals (%)
Inlet:	30.641	1.016	65.153	3.039		99.85
Exit:	17.046	1.242	66.662	5.597	0.0386	99.21

ORGANIC PRODUCTS

Product	Conc. (ppm)	Molar Rate (gmole /kg-hr)	Weight Rate (g/kg-hr)	CO2-free Molar Select. (mol%)	CO2-free Weight Select. (wt%)
methanol	77421.510	23.419	750.377	89.821	83.271
ethanol	1852.440	0.560	25.814	2.149	2.865
1-propanol	1502.870	0.455	27.307	1.744	3.030
isobutanol	1169.980	0.354	26.232	1.357	2.911
2-butanol	86.850	0.026	1.947	0.101	0.216
1-butanol	255.420	0.077	5.727	0.296	0.636
2-methyl-1-butanol	225.450	0.068	6.011	0.262	0.667
1-pentanol	110.700	0.033	2.952	0.128	0.328
2-methyl-1-pentanol	157.810	0.048	4.877	0.183	0.541
1-hexanol	67.940	0.021	2.100	0.079	0.233
DME	324.790	0.098	4.526	0.377	0.502
methane	957.960	0.290	4.649	1.111	0.516
ethane	117.090	0.035	1.065	0.136	0.118
propane	31.620	0.010	0.422	0.037	0.047
butane	0.000	0.000	0.000	0.000	0.000
pentane	7.200	0.002	0.157	0.008	0.017
hexane	5.920	0.002	0.154	0.007	0.017
methyl formate	1358.440	0.411	24.675	1.576	2.738
methyl acetate	541.370	0.164	12.131	0.628	1.346
methyl isobutyrate	113.240	0.034	3.498	0.131	0.388
ethyl acetate	13.440	0.004	0.358	0.016	0.040
TOTAL	86195.36	26.072	901.122	100.000	100.000
Sum C1-C6 Alcohols	82850.970	25.061	853.343	96.120	94.698
Sum C2-C6 Alcohols	5429.460	1.642	102.966	6.299	11.426
Sum C2-C5 Alcohols	5203.710	1.574	95.989	6.037	10.652
Sum C1-C3 Alcohols	80776.820	24.433	803.497	93.714	89.166
Sum C4-C6 Alcohols	2074.150	0.627	49.846	2.406	5.532
EtOH+PrOH	3355.310	1.015	53.120	3.893	5.895
EtOH+PrOH+iBuOH	4525.290	1.369	79.352	5.250	8.806
Sum C1-C6 HCs	1119.790	0.339	6.447	1.299	0.715
Sum Esters	2026.490	0.613	40.663	2.351	4.512

Material balances:
(Element Recoveries - %)

Nitrogen(N):	100.00
Carbon(C):	98.88
Hydrogen(H):	95.26
Oxygen(O):	99.47

Calculations:

CO conv. (%):	16.30
H2 conv. (%):	54.49
CO+H2 conv. (%):	28.52
Net CO2 rate gmol/kg-hr):	5.69

300cc #2 AUTOCLAVE RUN SUMMARY

Catalyst: BASF S3-86 CsLaporte Sample13465-26 **Liquid:** Drakeol 10

Run Description: mixed alcohols

Run#:	13458-73J	**Gas Type:**	Shell	**Feed MeOH Rate (ml/hr):**	0
Date:	3/15/94	**Press (psig):**	1730	**Feed EtOH Rate (ml/hr):**	0
Hrs:	238.5	**Temp. (C):**	300.3	**Feed PrOH Rate (ml/hr):**	0
Cat wt (g):	30.01	**Inlet Flow (sccm):**	1594.58	**Syngas GHSV (sl/kg-hr):**	3188.1
Liq wt (g):	120.03	**Exit Flow (sccm):**	1248.12	**Total GHSV (sl/kg-hr):**	3188.1

BULK GASES

Stream	H2(%)	N2(%)	CO(%)	CO2(%)	H2O(%)	Totals (%)
Inlet:	30.484	1.005	64.369	3.038		98.90
Exit:	15.634	1.284	64.349	8.265	0.0516	98.19

ORGANIC PRODUCTS

Product	Conc. (ppm)	Molar Rate (gmole /kg-hr)	Weight Rate (g/kg-hr)	CO2-free Molar Select. (mol%)	CO2-free Weight Select. (wt%)
methanol	70909.340	7.893	252.900	82.348	72.389
ethanol	2024.080	0.225	10.379	2.351	2.971
1-propanol	2462.470	0.274	16.464	2.860	4.713
isobutanol	3355.550	0.373	27.685	3.897	7.924
2-butanol	137.470	0.015	1.134	0.160	0.325
1-butanol	381.120	0.042	3.144	0.443	0.900
2-methyl-1-butanol	562.320	0.063	5.517	0.653	1.579
1-pentanol	171.520	0.019	1.683	0.199	0.482
2-methyl-1-pentanol	326.200	0.036	3.710	0.379	1.062
1-hexanol	143.830	0.016	1.636	0.167	0.468
DME	697.970	0.078	3.579	0.811	1.024
methane	2400.050	0.267	4.286	2.787	1.227
ethane	264.840	0.029	0.886	0.308	0.254
propane	85.230	0.009	0.418	0.099	0.120
butane	0.000	0.000	0.000	0.000	0.000
pentane	29.700	0.003	0.239	0.034	0.068
hexane	12.540	0.001	0.120	0.015	0.034
methyl formate	1347.640	0.150	9.008	1.565	2.578
methyl acetate	797.190	0.089	6.573	0.926	1.882
methyl isobutyrate	531.770	0.059	6.045	0.618	1.730
ethyl acetate	0.000	0.000	0.000	0.000	0.000
TOTAL	86109.06	9.585	349.363	100.000	100.000
Sum C1-C6 Alcohols	80473.900	8.957	324.253	93.456	92.813
Sum C2-C6 Alcohols	9564.560	1.065	71.353	11.107	20.424
Sum C2-C5 Alcohols	9094.530	1.012	66.007	10.562	18.894
Sum C1-C3 Alcohols	75395.890	8.392	279.743	87.559	80.073
Sum C4-C6 Alcohols	5078.010	0.565	44.509	5.897	12.740
EtOH+PrOH	4486.550	0.499	26.844	5.210	7.684
EtOH+PrOH+iBuOH	7842.100	0.873	54.528	9.107	15.608
Sum C1-C6 HCs	2792.360	0.311	5.949	3.243	1.703
Sum Esters	2676.600	0.298	21.627	3.108	6.190

Material balances:

(Element Recoveries - %)

Nitrogen(N):	100.00
Carbon(C):	97.89
Hydrogen(H):	91.85
Oxygen(O):	99.54

Calculations:

CO conv. (%):	21.75
H2 conv. (%):	59.86
CO+H2 conv. (%):	34.00
Net CO2 rate gmol/kg-hr):	4.88

300cc #2 AUTOCLAVE RUN SUMMARY

Catalyst: BASF S3-86 CsLaporte Sample13465-26 **Liquid:** Drakeol 10

Run Description: mixed alcohols

Run#:	13458-73K	**Gas Type:**	Shell	**Feed MeOH Rate (ml/hr):**	0
Date:	3/16/94	**Press (psig):**	1302	**Feed EtOH Rate (ml/hr):**	0
Hrs:	260.5	**Temp. (C):**	299.8	**Feed PrOH Rate (ml/hr):**	0
Cat wt (g):	30.01	**Inlet Flow (sccm):**	2613.62	**Syngas GHSV (sl/kg-hr):**	5225.5
Liq wt (g):	120.03	**Exit Flow (sccm):**	2208.48	**Total GHSV (sl/kg-hr):**	5225.5

BULK GASES

Stream	H2(%)	N2(%)	CO(%)	CO2(%)	H2O(%)	Totals (%)
Inlet:	30.348	1.025	65.651	3.001		100.03
Exit:	19.613	1.213	66.349	5.735	0.0407	99.73

ORGANIC PRODUCTS

Product	Conc. (ppm)	Molar Rate (gmole /kg-hr)	Weight Rate (g/kg-hr)	CO2-free Molar Select. (mol%)	CO2-free Weight Select. (wt%)
methanol	59152.540	11.650	373.298	87.211	79.186
ethanol	1603.280	0.316	14.547	2.364	3.086
1-propanol	1554.120	0.306	18.386	2.291	3.900
isobutanol	1578.980	0.311	23.051	2.328	4.890
2-butanol	88.250	0.017	1.288	0.130	0.273
1-butanol	252.650	0.050	3.688	0.372	0.782
2-methyl-1-butanol	298.440	0.059	5.181	0.440	1.099
1-pentanol	117.820	0.023	2.046	0.174	0.434
2-methyl-1-pentanol	276.260	0.054	5.559	0.407	1.179
1-hexanol	78.160	0.015	1.573	0.115	0.334
DME	257.240	0.051	2.334	0.379	0.495
methane	1158.480	0.228	3.660	1.708	0.776
ethane	164.050	0.032	0.972	0.242	0.206
propane	43.470	0.009	0.378	0.064	0.080
butane	0.000	0.000	0.000	0.000	0.000
pentane	12.420	0.002	0.176	0.018	0.037
hexane	6.800	0.001	0.115	0.010	0.024
methyl formate	765.030	0.151	9.048	1.128	1.919
methyl acetate	419.030	0.083	6.114	0.618	1.297
methyl isobutyrate	157.500	0.031	3.168	0.232	0.672
ethyl acetate	12.560	0.002	0.218	0.019	0.046
TOTAL	67827.02	13.359	471.416	100.000	100.000
Sum C1-C6 Alcohols	65000.500	12.802	448.619	95.833	95.164
Sum C2-C6 Alcohols	5847.960	1.152	75.321	8.622	15.978
Sum C2-C5 Alcohols	5493.540	1.082	68.188	8.099	14.465
Sum C1-C3 Alcohols	62309.940	12.272	406.232	91.866	86.173
Sum C4-C6 Alcohols	2690.560	0.530	42.387	3.967	8.991
EtOH+PrOH	3157.400	0.622	32.934	4.655	6.986
EtOH+PrOH+iBuOH	4736.380	0.933	55.985	6.983	11.876
Sum C1-C6 HCs	1385.220	0.273	5.301	2.042	1.125
Sum Esters	1354.120	0.267	18.548	1.996	3.935

Material balances:
(Element Recoveries - %)

Nitrogen(N):	100.00
Carbon(C):	99.16
Hydrogen(H):	96.88
Oxygen(O):	99.83

Calculations:

CO conv. (%):	14.60
H2 conv. (%):	45.39
CO+H2 conv. (%):	24.34
Net CO2 rate gmol/kg-hr):	4.30

300cc #2 AUTOCLAVE RUN SUMMARY

Catalyst: BASF S3-86 CsLaporte Sample13465-26 **Liquid:** Drakeol 10

Run Description: mixed alcohols

Run#:	13458-73L	**Gas Type:**	Shell	**Feed MeOH Rate (ml/hr):**	23.9216
Date:	3/17/94	**Press (psig):**	1299	**Feed EtOH Rate (ml/hr):**	1.448507
Hrs:	289	**Temp. (C):**	300.4	**Feed PrOH Rate (ml/hr):**	3.429895
Cat wt (g):	30.01	**Inlet Flow (sccm):**	2588.86	**Syngas GHSV (sl/kg-hr):**	5176.0
Liq wt (g):	120.03	**Exit Flow (sccm):**	2677.09	**Total GHSV (sl/kg-hr):**	5670.0

BULK GASES

Stream	H2(%)	N2(%)	CO(%)	CO2(%)	H2O(%)	Totals (%)
Inlet:	30.218	1.031	66.094	2.987		100.33
Exit:	24.733	0.997	58.610	5.098	0.0589	100.41

ORGANIC PRODUCTS

Product	Conc. (ppm)	Molar Rate (gmole /kg-hr)	Weight Rate (g/kg-hr)	CO2-free Molar Select. (mol%)	CO2-free Weight Select. (wt%)
methanol	90743.700	21.664	694.174	83.166	72.902
ethanol	3683.520	0.879	40.514	3.376	4.255
1-propanol	6328.990	1.511	90.765	5.801	9.532
isobutanol	3451.600	0.824	61.081	3.163	6.415
2-butanol	104.480	0.025	1.849	0.096	0.194
1-butanol	362.680	0.087	6.418	0.332	0.674
2-methyl-1-butanol	250.870	0.060	5.280	0.230	0.554
1-pentanol	149.670	0.036	3.150	0.137	0.331
2-methyl-1-pentanol	230.560	0.055	5.624	0.211	0.591
1-hexanol	97.530	0.023	2.379	0.089	0.250
DME	277.820	0.066	3.056	0.255	0.321
methane	1187.110	0.283	4.547	1.088	0.478
ethane	208.440	0.050	1.496	0.191	0.157
propane	70.840	0.017	0.746	0.065	0.078
butane	0.000	0.000	0.000	0.000	0.000
pentane	11.190	0.003	0.193	0.010	0.020
hexane	11.310	0.003	0.233	0.010	0.024
methyl formate	1082.760	0.259	15.524	0.992	1.630
methyl acetate	857.830	0.205	15.172	0.786	1.593
methyl isobutyrate	253.650	0.061	6.185	0.232	0.650
ethyl acetate	42.690	0.010	0.898	0.039	0.094
TOTAL	109110.9	26.050	952.198	100.000	100.000
Sum C1-C6 Alcohols	105403.600	25.164	911.233	96.602	95.698
Sum C2-C6 Alcohols	14659.900	3.500	217.059	13.436	22.796
Sum C2-C5 Alcohols	14331.810	3.422	209.056	13.135	21.955
Sum C1-C3 Alcohols	100756.210	24.055	825.452	92.343	86.689
Sum C4-C6 Alcohols	4647.390	1.110	85.781	4.259	9.009
EtOH+PrOH	10012.510	2.390	131.279	9.176	13.787
EtOH+PrOH+iBuOH	13464.110	3.214	192.359	12.340	20.202
Sum C1-C6 HCs	1488.890	0.355	7.214	1.365	0.758
Sum Esters	2236.930	0.534	37.778	2.050	3.967

Material balances:
(Element Recoveries - %)

Nitrogen(N):	100.00
Carbon(C):	100.76
Hydrogen(H):	101.51
Oxygen(O):	101.21

Calculations:

CO conv. (%):	8.30
H2 conv. (%):	15.36
CO+H2 conv. (%):	10.52
Net CO2 rate gmol/kg-hr):	5.27

300cc #2 AUTOCLAVE RUN SUMMARY

Catalyst: BASF S3-86 CsLaporte Sample13465-26 **Liquid:** Drakeol 10

Run Description: mixed alcohols

Run#:	13458-73M	**Gas Type:**	Shell	**Feed MeOH Rate (ml/hr):**	6.553796
Date:	3/18/94	**Press (psig):**	1299	**Feed EtOH Rate (ml/hr):**	0.986254
Hrs:	312.25	**Temp. (C):**	300	**Feed PrOH Rate (ml/hr):**	1.85995
Cat wt (g):	30.01	**Inlet Flow (sccm):**	2629.12	**Syngas GHSV (sl/kg-hr):**	5256.5
Liq wt (g):	120.03	**Exit Flow (sccm):**	2371.09	**Total GHSV (sl/kg-hr):**	5408.6

BULK GASES

Stream	H2(%)	N2(%)	CO(%)	CO2(%)	H2O(%)	Totals (%)
Inlet:	30.257	1.020	65.343	3.006		99.63
Exit:	21.471	1.131	63.413	5.410	0.0466	99.67

ORGANIC PRODUCTS

Product	Conc. (ppm)	Molar Rate (gmole /kg-hr)	Weight Rate (g/kg-hr)	CO2-free Molar Select. (mol%)	CO2-free Weight Select. (wt%)
methanol	67990.530	14.377	460.665	82.902	72.478
ethanol	2702.880	0.572	26.330	3.296	4.143
1-propanol	4183.910	0.885	53.143	5.102	8.361
isobutanol	2899.830	0.613	45.451	3.536	7.151
2-butanol	92.810	0.020	1.455	0.113	0.229
1-butanol	299.960	0.063	4.701	0.366	0.740
2-methyl-1-butanol	267.640	0.057	4.989	0.326	0.785
1-pentanol	132.180	0.028	2.464	0.161	0.388
2-methyl-1-pentanol	251.130	0.053	5.426	0.306	0.854
1-hexanol	90.370	0.019	1.953	0.110	0.307
DME	227.750	0.048	2.219	0.278	0.349
methane	1103.780	0.233	3.744	1.346	0.589
ethane	189.930	0.040	1.208	0.232	0.190
propane	62.350	0.013	0.581	0.076	0.091
butane	0.000	0.000	0.000	0.000	0.000
pentane	9.990	0.002	0.152	0.012	0.024
hexane	9.260	0.002	0.169	0.011	0.027
methyl formate	853.950	0.181	10.844	1.041	1.706
methyl acetate	644.920	0.136	10.102	0.786	1.589
methyl isobutyrate	235.910	0.050	5.095	0.288	0.802
ethyl acetate	30.320	0.006	0.565	0.037	0.089
TOTAL	82013.17	17.342	635.596	100.000	100.000
Sum C1-C6 Alcohols	78911.240	16.686	606.576	96.218	95.434
Sum C2-C6 Alcohols	10920.710	2.309	145.912	13.316	22.957
Sum C2-C5 Alcohols	10579.210	2.237	138.533	12.899	21.796
Sum C1-C3 Alcohols	74877.320	15.833	540.138	91.299	84.981
Sum C4-C6 Alcohols	4033.920	0.853	66.438	4.919	10.453
EtOH+PrOH	6886.790	1.456	79.474	8.397	12.504
EtOH+PrOH+iBuOH	9786.620	2.069	124.924	11.933	19.655
Sum C1-C6 HCs	1375.310	0.291	5.855	1.677	0.921
Sum Esters	1765.100	0.373	26.606	2.152	4.186

Material balances:
(Element Recoveries - %)

Nitrogen(N):	100.00
Carbon(C):	99.76
Hydrogen(H):	98.99
Oxygen(O):	100.27

Calculations:

CO conv. (%):	12.48
H2 conv. (%):	36.00
CO+H2 conv. (%):	19.92
Net CO2 rate gmol/kg-hr):	4.39