Title: Isobutanol Dehydration: A Key Step in Producing MTBE from Syngas
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Organization: Air Products and Chemicals, Inc.
Contract Number: DE-AC22-91PC90018
Period of Performance: September, 1992 to September, 1993
Objective: Development of a process for the dehydration of isobutanol to isobutylene, one of the key steps in a process for the conversion of syngas to MTBE

Accomplishments & Conclusions:

A process for the dehydration of isobutanol to isobutylene has been demonstrated in DOE's LaPorte, Texas, AFDU (Slurry Phase Alternative Fuels Development Unit). Preliminary results from the August plant trial are described. The laboratory development of the process including details of the kinetics of the dehydration are reported.

Laboratory operations in a 300 cc CSTR (continuous stirred tank reactor) demonstrated that the process conditions were suitable for the AFDU. Isobutylene was obtained with high selectivity (>92 mole %) at high conversions (>90%) by dehydration over a γ-alumina catalyst. Feed rates of 200-400 standard liters/ hr.-Kg. of catalyst were achieved at an operating temperature of 310 °C. Extended life studies showed an initial exponential decay in activity for the first 50-60 hours followed by a slow, linear decay such that the conversion fell from 92% to 82% after continuous operation for 250 hours. A power law kinetic expression has been fit to the data.

Liquid Phase Isobutylene (LPIBUE) technology was demonstrated in the bubble column reactor at the LaPorte AFDU. The 10-day run demonstrated the technology at the pilot scale (6 T/D isobutylene). This run demonstrated the ability to run endothermic reactions with expanding gas flow in the AFDU. The plant ran well for the entire operation. The temperature of 600°F is the highest ever run in the plant.

- Hydrodynamic behavior was stable. Uniform temperatures profiles were obtained. Superficial velocities ranged from 0.7 to 0.12 ft/sec. Catalyst concentration was 34 wt. %. Reactor gas hold up ranged from 11% to 32% within the reactor with average holdups varying from 14% to 25 %.
- Alcohol conversion as high as 98% with an isobutylene selectivity of 92 mole %, meeting our objective of 90% conversion.
- The conversion was consistently higher in the bubble column than the laboratory CSTR as expected from previous runs. Excellent selectivity was achieved in the demonstration unit as well as the laboratory.
- The catalyst activity was stable after the initial aging. The isobutanol conversion dropped 2% initially in one day, but only dropped about 1% over next 7 days.

Introduction

The market for fuel oxygenates has grown rapidly as a response to the 1990 Clean Air Act Amendments. MTBE(methyl *t*-butyl ether) has emerged as the most promising gasoline-blending oxygenate. MTBE production has increased by a factor of 13 in the last 10 years. Currently, raw materials for MTBE production are derived from natural gas and petroleum feed stocks. Methanol, produced via synthesis gas, reacts with isobutylene to produce MTBE. Isobutylene, which is a byproduct of catalytic cracking or dehydrogenation of butane, is petroleum derived. Future demand for isobutylene may well exceed the supply of petroleum-derived sources.

This project is part of an overall plan to develop technology to produce MTBE from alternate feed stocks. It is part of PETC's (the Pittsburgh Energy Technology Center) Indirect Liquefaction program which is part of the DOE' s Coal Liquefaction program. Feed stocks such as coal or petroleum coke can be converted into synthesis gas. Syngas is the raw material for the MTBE process.

Figure 1 diagrams the proposed scheme for production of MTBE from synthesis gas. The first step co-produces methanol and isobutanol selectively. (Demonstration of this technology is scheduled for March of 1994.) The second reaction step produces isobutylene by dehydration of the isobutanol which has been separated from the mixed alcohols product stream. Finally, using well-established technology, the isobutylene reacts with the methanol, produced in the first step, to produce MTBE . This paper discusses the technology developed over the last year to produce isobutylene from isobutanol. Figure 2 gives an outline of today's talk. Both the laboratory work to define the process and a preliminary look at the results of the highly successful August demonstration in the Air Products-operated Alternate Fuels Demonstration Unit will be discussed.

Laboratory Work

Chemistry

As shown in Figure 3, isobutanol reacts to produce isobutylene and water. In contrast to all reactions studied previously in the LaPorte facility, the volume of gas increases through the reactor since this reaction produces 2 moles of product for each mole of feed. The reaction occurs through two possible parallel paths. Dehydration can occur directly through an acid-catalyzed reaction or indirectly through a bimolecular reaction to produce the ether which, in turn, reacts to form isobutylene through one of two paths. This later reaction path is favored at low temperatures. At the higher temperatures (>250°C) and high conversions used in the present process,

only a small amount of diisobutyl ether is formed. The only significant byproducts seen in our study come from isomerization of the isobutylene. Typical product purity is shown by the GC trace of Figure 4. Selectivities to isobutylene and total butenes are shown as a function of conversion in Figure 5. At low conversions, some diisobutyl ether is formed so that the selectivity to total butenes is relatively low. Most of the butenes formed are isobutylene since isomerization is low at the short contact times of the low conversion process. As conversion increases, the diisobutyl ether reacts to form isobutylene such that the total butenes produced increase. Isomerization increases only slightly so that the selectivity to isobutylene remains high. Finally, at the long residence time required to achieve very high conversions, isomerization becomes much more important, and the selectivity to isobutylene decreases while the selectivity to total butenes is very high since all of the diisobutyl ether is converted at long reaction times, and dehydration is the only reaction occurring to a significant extent.

Reactor Modifications

All our laboratory development efforts were carried out in a CSTR (continuous stirred-tank reactor) similar to that used in previous process development work. Kinetics and laboratory reactions are carried out in this "perfectly mixed" single stage reactor. We rely on our engineering ability and pervious experience at the AFDU to translate these results successfully to the slurry bubble-column.

Figure 6 is a diagram of the laboratory reactor system. As you might recall from previous talks, the reactor contains the catalyst slurried with a mineral oil. In this case Drakeol-10, the same oil as used in the demonstration unit, was used. Gas or vapor is fed to the reactor. All products are withdrawn as a vapor. The reactor system was modified from our previous work so that we could vaporize the liquid, isobutanol, before entering the reactor. In addition, since the product stream has a dew point as high a 113°C, as calculated from our in-house thermodynamic routines, the outlet was modified to prevent condensation. Extensive modifications to the analytical scheme were also made to accommodate the almost 50 mole % water concentration in the exit scheme.

Since the features of vapor feed, complete feed conversion and gas expansion through the bed are all significantly different from previous runs, the reactor was checked to determine that it was not mass transfer limited. The results of these tests are shown in Figure 7. Normal operating speed for the impeller is 1400 rpm. This is on the flat part of the curve beyond the mass transfer limit. The amount of catalyst was increased from 20 to 30 grams. The rate increased proportionally to the amount of catalyst. This is a clear indication that the reactor is in the kinetic controlled and not mass transfer controlled region.

Reaction Results

Previous laboratory studies, carried out by Fran Waller in our Corporate research labs using a gas phase tubular reactor, identified γ-alumina as the most active acid catalyst for dehydration. Typical reaction results from our CSTR slurry reactor using γ-alumina catalyst are shown in Figure 8. Conversion decreases with time on stream. (Catalyst life is discussed below.) The selectivity to isobutylene is high and increases slightly as the conversion decreases as is expected from the chemistry discussed above. As a consequence of the decreasing rate and increasing selectivity, the rate of production remains about constant.

One major question which must be answered is "Is there a commercially available catalyst which has the sufficient activity?" Since the separation of water and isobutanol is difficult, recycle of isobutanol should be avoided for an economic process. This means high conversion in a single pass was desired. Previous plant runs, e.g., LPMEOH, can be used to identify the operating limits of the AFDU. The combined requirements of high conversion and plant constraints determine the operating window for the plant. Figure 9 shows this operating window superimposed on reaction results for four γ-alumina catalysts. As shown we have identified two catalysts which have the required activity.

Catalyst Aging

The relatively rapid aging in the first 20 hours of operation as shown above was the only cause of concern in this data. Therefore, aging of the catalyst was studied in some detail. The results for a longer life study are shown in Figure 10. The rate of reaction decreases exponentially, i.e., aging is rapid initially and levels out to a very low rate after about 40 hours. This aging pattern is acceptable for long term running. Figure 10 shows data for both commercial grade isobutanol and reagent grade. The results using reagent grade feed show the same trends as the commercial feed. Initial aging is more rapid for the reagent grade feed, but the catalyst activity levels out to about the same value for the longer times.

TGA (thermo-gravimetric analysis) of aged catalyst samples show a significant weight loss above 400°C in air. Weight loss in this region is typically attributed to the burning of coke. Coke formation has been reported as the reason for activity loss in the dehydration of *t*-butyl alcohol (Grane, et al., U. S. Patent 3,665,048 [1992].) Surface area only decreased slightly (~4%) for the same catalyst. Thus, coke formation is the most likely reason for loss of catalyst activity. In addition, the rapid loss of life followed by a stabilizing is consistent with a distribution of activities for catalytic sites.

Kinetics

Although the reaction is clean and appears simple, the kinetics of alcohol dehydration are complicated. Many differing mechanisms have been proposed. (For example see Knozinger, H., "Dehydration of Alcohols on Aluminum Oxide", Angew. Chem. Internat. Edit., Vol. 7, No. 10, (1968)) Since equimolar amounts of water and isobutylene are produced, the contribution of water and isobutylene on the kinetics can only be separated by series of experiments adding water and isobutylene to the feed. We have just finished such a series of experiments and will report on these results at a later date.

Thus far, several models from the literature as well as power law kinetics have been used. These expressions are shown in Table 1. As has been our custom, we report the rate as a function of the fugacities of the gas phase. As noted only the power law kinetic model fits the data well. Water acts to retard the rate of reaction strongly. This might be expected since water is strongly adsorbed by alumina catalysts.

The fit of the power law model to the data is also shown in Figure 11. The fit is good, and the expression can be used for design purposes. However, additional work is needed to get a better mechanistic interpretation. This work is planned at the end of the additional kinetic laboratory runs.

AFDU Demonstration

Run Objectives

The main objective of the this run was to demonstrate the liquid phase isobutylene technology at a pilot scale (6 T/D isobutylene) in a bubble column reactor and address scale up issues such as:

- Catalyst performance
- Exothermic Reaction
- Low Pressure
- Hydrodynamics (Expanding Flow)

The criteria of success included:

- Good catalyst activity - achieve 90% isobutanol conversion
- Stable catalyst performance
- Good correlation between the bubble column and the stirred autoclave.
- Isothermal reactor operation and expected hydrodynamic behavior

Plant Modifications

The modifications to the AFDU for the isobutylene demonstration involved the isobutanol feed system and the utility oil system. The modifications were needed to vaporize the liquid feed as well as provide the heat of reaction for the endothermic reaction.

Figure 12 is a simplified process flow diagram for the isobutylene run. The isobutanol is pumped from a trailer using 10.95 pump, vaporized in the 02.61 Steam Heater at 400°F and then superheated in the 02.62 Electric Heater to 600°F before its introduction to the reactor. The oil vaporized from the 27.10 Reactor is condensed in the 22.14 Vapor-Liquid Separator at about 300°F and pumped back to the reactor. Most of the unconverted isobutanol and the water produced is condensed in the 22.18 Vapor-Liquid Separator at about 100°F and collected in the 22.16 Day Tank. The isobutylene along with other minor products are sent to flare.

The utility oil system needed to be operated at much higher temperature (660°F average) than previous operations (< 500°F). This is due to a higher reactor temperature (up to 600°F) and a need to supply the heat of reaction. The insulation on the utility oil system was upgraded from the Personnel Protection specifications to the Heat Conservation specifications to reduce heat losses. Also, the entire utility system was rated to 700°F. This required changing some seals and expansion joints. Typically used Drakeol-10 utility oil was expected to have significant vaporization at the higher temperatures. Hence, a heavier oil, Drakeol-34, was evaluated and used in the utility oil system.

Run Plan

A 10-day run plan (Table 2) included operations at six different process conditions and a hydrodynamic study using radioactive tracers. Due to feedstock (isobutanol) costs, the run duration was kept minimum. However, the plan represented aggressive process conditions with a wide range of process variables which allowed us to demonstrate the process.

Effects of space velocity (linear velocity), pressure and temperature were studied during the process variable study (Run AF-R7). The range of the operating conditions were: 30-34 wt. % alumina concentration, 572-600°F, 25-40 psig, 115-400 sl/hr-kg space velocity, 0.12-0.40 ft/sec linear inlet velocity and 0.22-0.70 ft/sec linear outlet velocity. The initial baseline run was repeated at the end to check for the catalyst stability. The tracer study was added since the operation involved an endothermic reaction with molar expansion. All the past LaPorte operations involved exothermic reactions with molar contraction.

Preliminary Results

Reaction Results

The isobutylene demonstration run started on 2 August 1993. The reactor was charged with 827 lb. of Engelhard Gamma Alumina # AL-3919P and 1815 lb. of Penreco Drakeol-10 oil to obtain about 30 wt.% catalyst concentration on a moisture free basis. The catalyst was heated under nitrogen with a temperature ramp to 572°F. The initial isobutanol conversion was 96.7 mole %, 3% higher than observed in the laboratory autoclave. The isobutylene selectivity was 93.9 mole %, about 1% higher than the autoclave.

The conversion and selectivity at various conditions are plotted as a function of time on-stream in Figures 13 and 14. Data from the autoclave are also included for comparison. The conversion was consistently higher in the bubble column. This higher conversion was expected based on previous runs that show the slurry bubble column performance is best simulated by two to three stirred tanks in series which give higher conversion than the single stirred tank in the laboratory. (A detailed analysis of the number of stirred tank equivalents will be performed when the final kinetic expression is developed in the laboratory.) The selectivity in the two systems were comparable.

The isobutanol conversion dropped about 2% initially from Day 1 (Run 7.1A) to Day 2 (Run 7.1B). When the operating condition was repeated at the end of the process variable study (Day 8, Run 7.7), the conversion had decreased only additional 1%. Thus, the catalyst aging pattern was similar to that observed in the autoclave. The catalyst activity was relatively stable after the initial aging period.

Figures 15 and 16 show the effect of space velocity. The conversion decreased with increase in space velocity. The selectivity to total butenes decreases slightly with increasing space velocity due to the formation of the diisobutyl ether just as we have seen in the laboratory. Comparison of the ratio of the isobutylene to total butenes shows that the selectivity increases with increasing space velocity. This effect is attributed to a reduction in isomerization at the lower residence time, again just as seen in the laboratory reactor. The isobutylene productivity increased with increase in space velocity (Figure 16). This behavior is typical for stirred tanks in series.

The effect of temperature is shown in Figure 17. The conversion increased from 94 mole % at 573°F to 98 mole % at 601°F. The isobutylene selectivity

showed a small decrease as the isomerization increased while the total butene selectivity remained constant at a very high level.

The effect of pressure was studied in Run AF-R7.5 and is summarized in Table 3. The pressure was increased to 40 psig at 600°F and 230 sl/hr-kg. The isobutanol conversion was 94.9%, which is significantly higher than the 88.4% observed in the autoclave. Water retards reaction rate less in the bubble column than the autoclave. The most probable explanation for this is the tanks-in-series nature of the slurry bubble column. In the "perfectly mixed" laboratory reactor, the effect of higher water partial pressure at high pressure is felt equally through the entire reaction mass; while for the tanks-in-series of the slurry bubble column the effect of higher water partial pressure is felt gradually as the water of reaction builds in the reactor. The advantage of the bubble column over the autoclave would increase at higher pressure, as water partial pressure increases. The isobutylene selectivity was about 93 mole %, which is comparable to the autoclave.

Hydrodynamic Results

Reactor gas hold-up was measured by nuclear density measurements conducted periodically throughout the run . Figure 18 shows the gas hold up for the various trial conditions. The gas hold up remained stable during the run. For example, at the baseline condition (Run AF-R7.1A, R7.1B and R7.7), the gas hold up remained around 13.5 vol %. The effect of gas velocity on the hold up is shown in Figure 19. As expected, the hold up increased with increase in velocity. However, the holdup was higher than predicted from previous correlations at the higher velocity. This may be due to the significant change in pressure with reactor height at the relatively low pressures used in this run. Gas hold up increased significantly with the reactor height at all the conditions. This appears to be a result of molar expansion in the reactor, increasing the gas velocity. These data have not been corrected for the reactor pressure drop.

The catalyst concentration profile was checked, and the gas hold up measurements were confirmed by periodically shutting down the feed to the reactor for about 10 minutes. Nuclear density measurements were taken rapidly along the length of the reactor for the ungassed condition. The uniformity of the catalyst profile was inferred from the constant readings along the length of the reactor. These readings were obtained before significant catalyst settling occurred. The height of the ungassed liquid was used to confirm the accuracy of the gas hold up estimates made from the nuclear density scans during a run condition.

The reactor operated essentially isothermal throughout the run, with the temperature profile showing only a 2°F temperature variation along the height of the reactor.

After completing the process variable study, a tracer study was conducted to evaluate the mixing in both gas and liquid phases. A vapor residence time distribution study was performed by injecting Argon-41 into the inlet gas line and monitoring its progress through the reactor by several detectors. Four different gas velocities were tested over two days. Throughout the study, excellent pulse shapes were obtained at the inlet. Sharp responses were obtained at other four locations along the height of the reactor as well as at the reactor exit. Detailed data analysis is needed before any conclusion can be reached. Liquid phase mixing measurements were made by injecting of radioactive Mn_2O_3 at two different heights of the reactor on each day to study . The data showed interesting trends; portions of the tracer showed flow in both upward as well as downward direction. The data will be analyzed in depth to characterize the mixing in the reactor.

At the completion of the tracer study, the plant was switched to nitrogen feed on 8/12/93 in preparation for shutdown. A final inspection of the equipment items after the shut down showed the reactor, slurry prep tank, and feed/product heat exchanger to be relatively clean with no evidence of solids coagulating in the sparger.

Summary

The Liquid Phase Isobutylene (LPIBUE) technology has been demonstrated in the bubble column reactor at the LaPorte AFDU. The 10-day run demonstrated the technology at the pilot scale (6 T/D isobutylene). This run demonstrated the ability to run endothermic reactions with expanding gas flow in the AFDU for the first time. The plant was operated at aggressive conditions (600°F, 0.7 ft/sec outlet velocity, 0.12 ft/sec inlet velocity and 34 wt. % alumina concentration) and performed exceedingly well during the entire demonstration. The reaction temperature was the highest ever run in the plant.

- Hydrodynamic behavior was stable. Uniform temperatures were obtained along the height of the reactor. Reactor gas hold up ranged from 11% to 32% within the reactor with average holdups varying from 14% to 25 %.
- Alcohol conversion as high as 98% with an isobutylene selectivity of 92 mole %, meeting our objective of 90% conversion.
- The conversion was consistently higher in the bubble column than the laboratory CSTR. Apparently, the slurry reactor has some plug flow character under expanding gas conditions. This same behavior has been seen previously for shrinking gas flows, e.g. MeOH. The selectivity in the

slurry column was excellent and essentially the same as seen in the laboratory.

- The catalyst activity was stable after the initial aging. The isobutanol conversion dropped 2% initially in one day, but only dropped about 1% over next 7 days.

Future Work

The results presented here are preliminary, since the demonstration was completed only quite recently.

- The plans are to complete the process variable results, and analyze the tracer study results in detail. The kinetic modeling study will be completed. a model developed and used as a tool to understand the plant results.
- Process flow sheets of an integrated process for making MTBE from Syngas will be developed.
- The economics of the integrated plant will be compared to standard technologies.

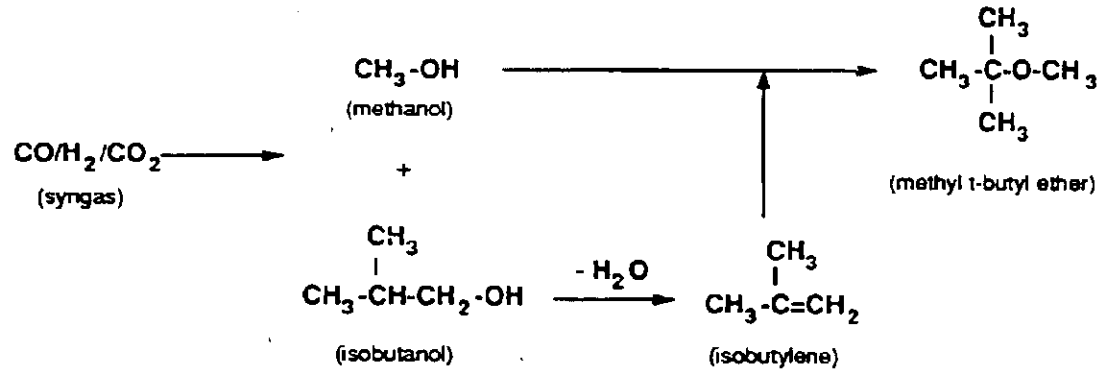


Figure 1
Reaction Scheme

Figure 2
Outline
Contractors Meeting Sept., 1993

Introduction

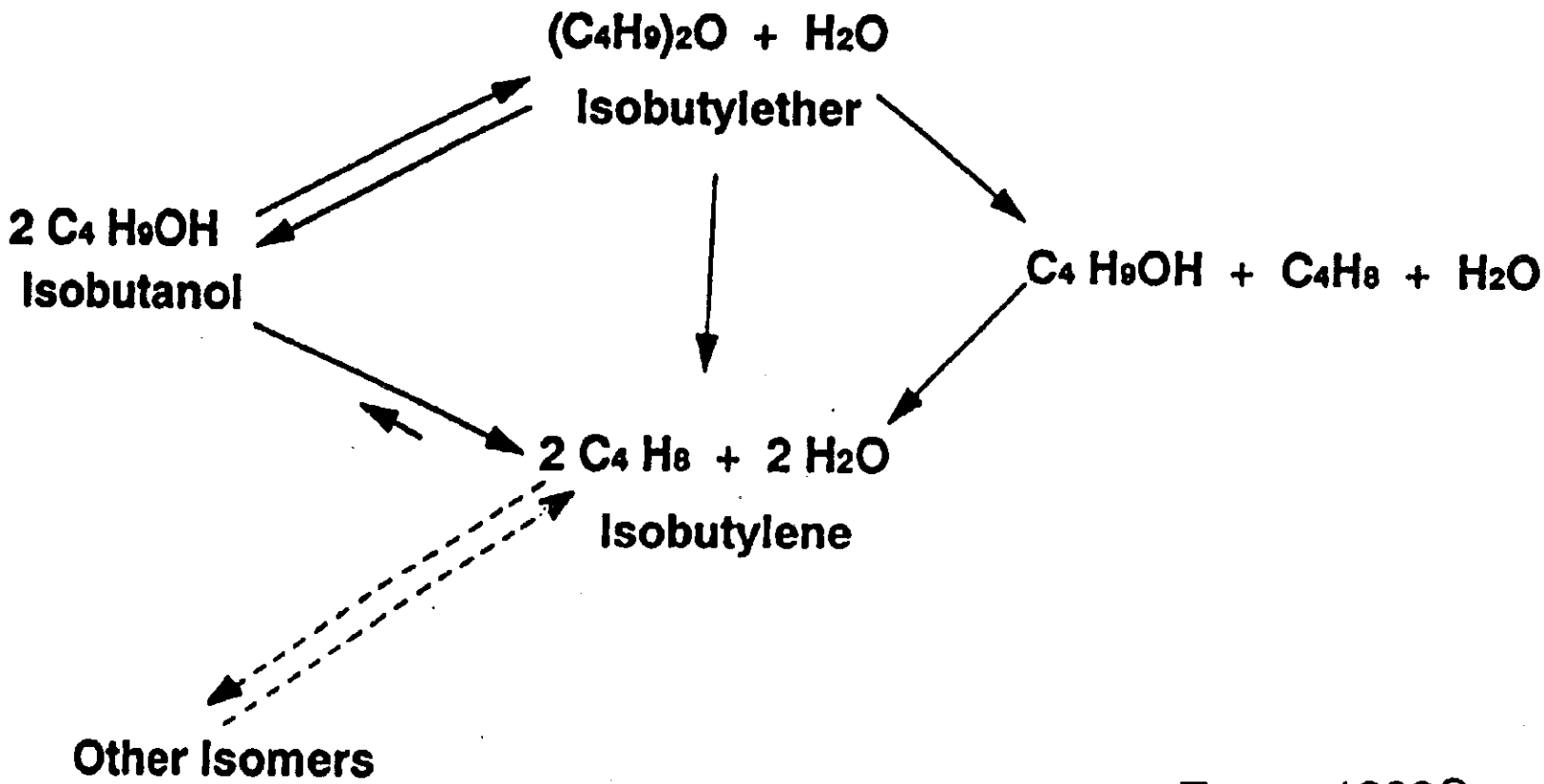
Laboratory Process Development
- Chemistry
- Reactor Modifications
- Reactor Results
- Kinetics

AFDU Demonstration
- Plant Modifications
- Run Objectives
- Results
  - -Chemistry
  - - Hydrodynamics

Summary and Future Plans

Figure 3

CHEMISTRY

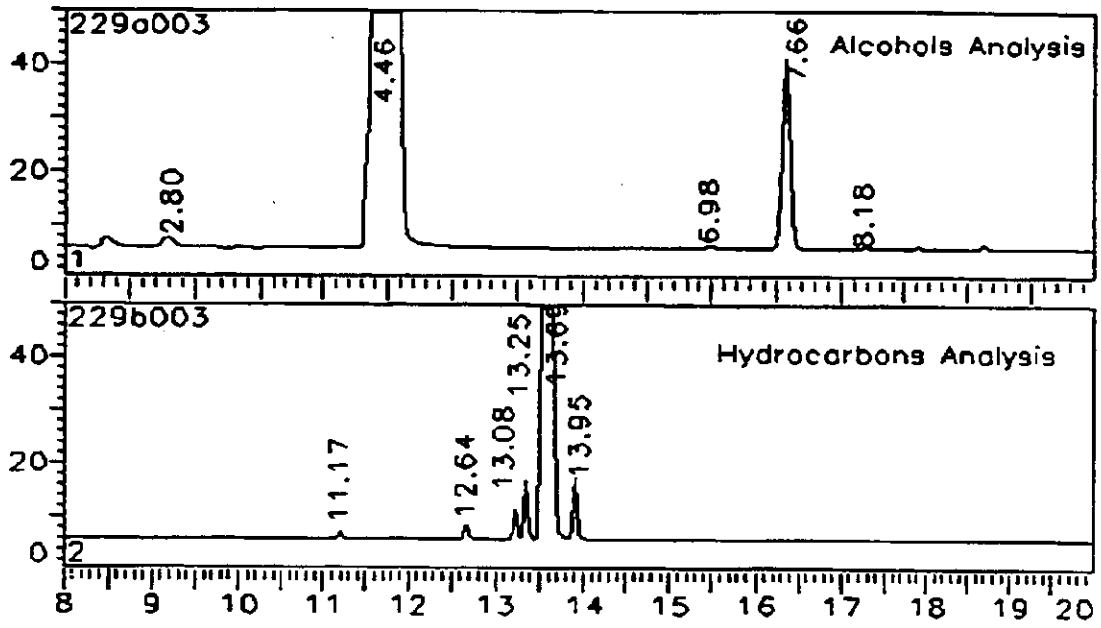


ΔTadb=-130°C

Figure 4
Typical Exit Stream Composition
(Conditions: 300° C, GHSV = 221 Std. L kg-cat.$^{-1}$ h^{-1})

Compound	Composition
Isobutanol	13.4 (Mole. %)
Water (calc'd)	43.02
Isobutene	40.48
c-2-Butene	1.15
1-Butene	0.80
Diisobutyl Ether	0.60
t-2-Butene	0.41
1-Propanol	0.09
Isobutane	0.05
1-Hexanol	0.01

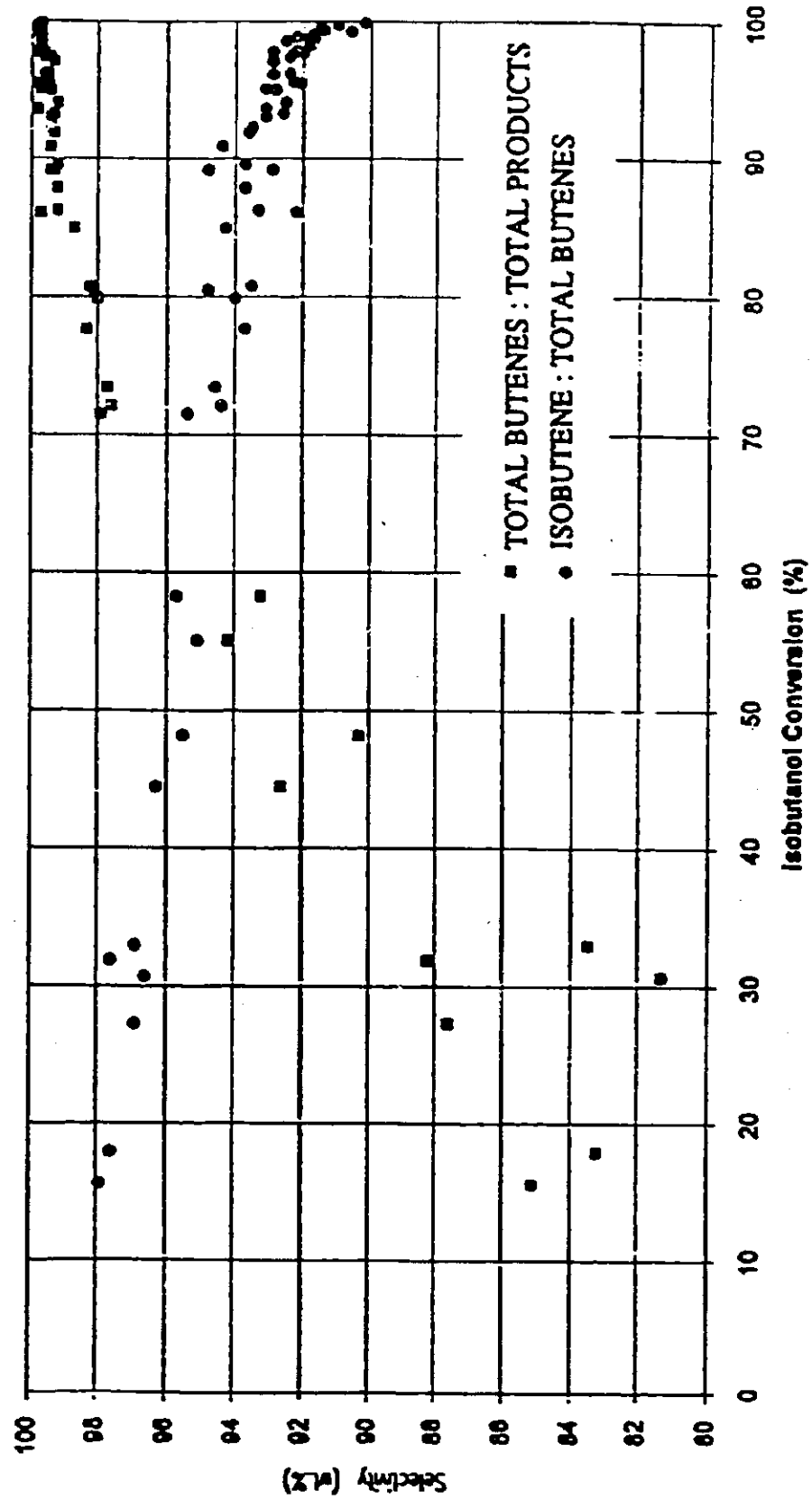
Typical GC Traces Showing Trace Compounds

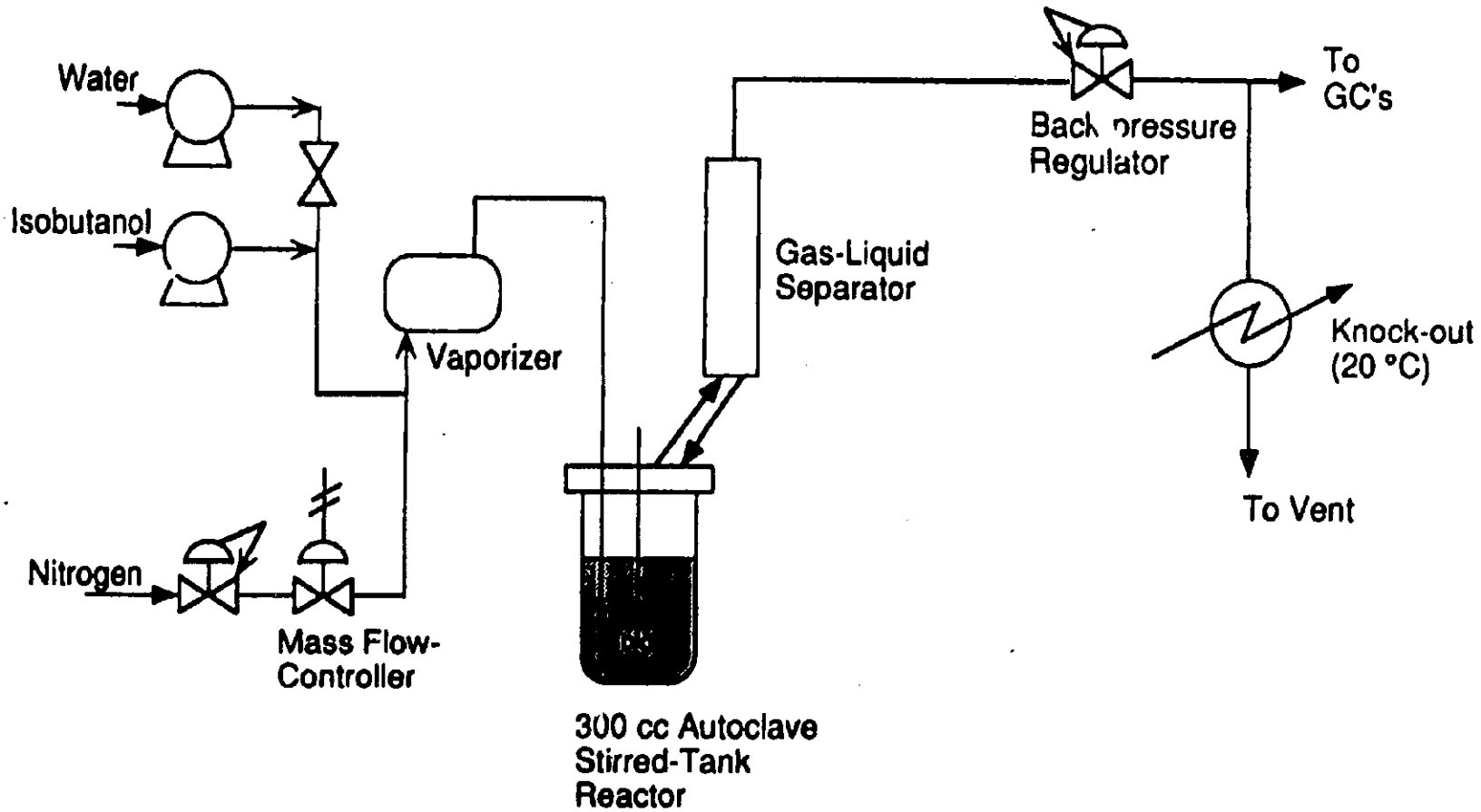


Retention Time (min.)

Figure 5

Selectivity as a Function of Isobutanol Conversion





Bench Scale Slurry-phase Reactor for Reaction Rate Measurements

Figure 6

Figure 7
Isobutanol Conversion Vs. Stirrer Speed
(30 Wt. % Slurry, 400 Std. L/kg h, 310 C, 30 psig)

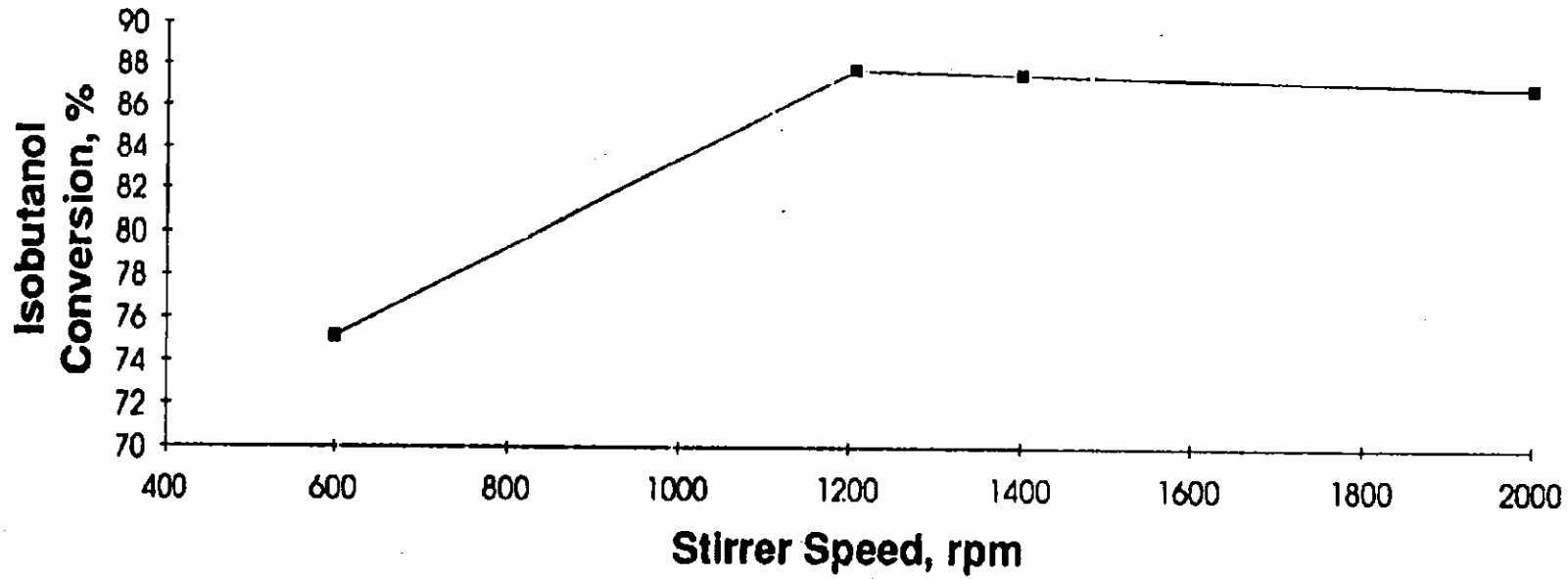
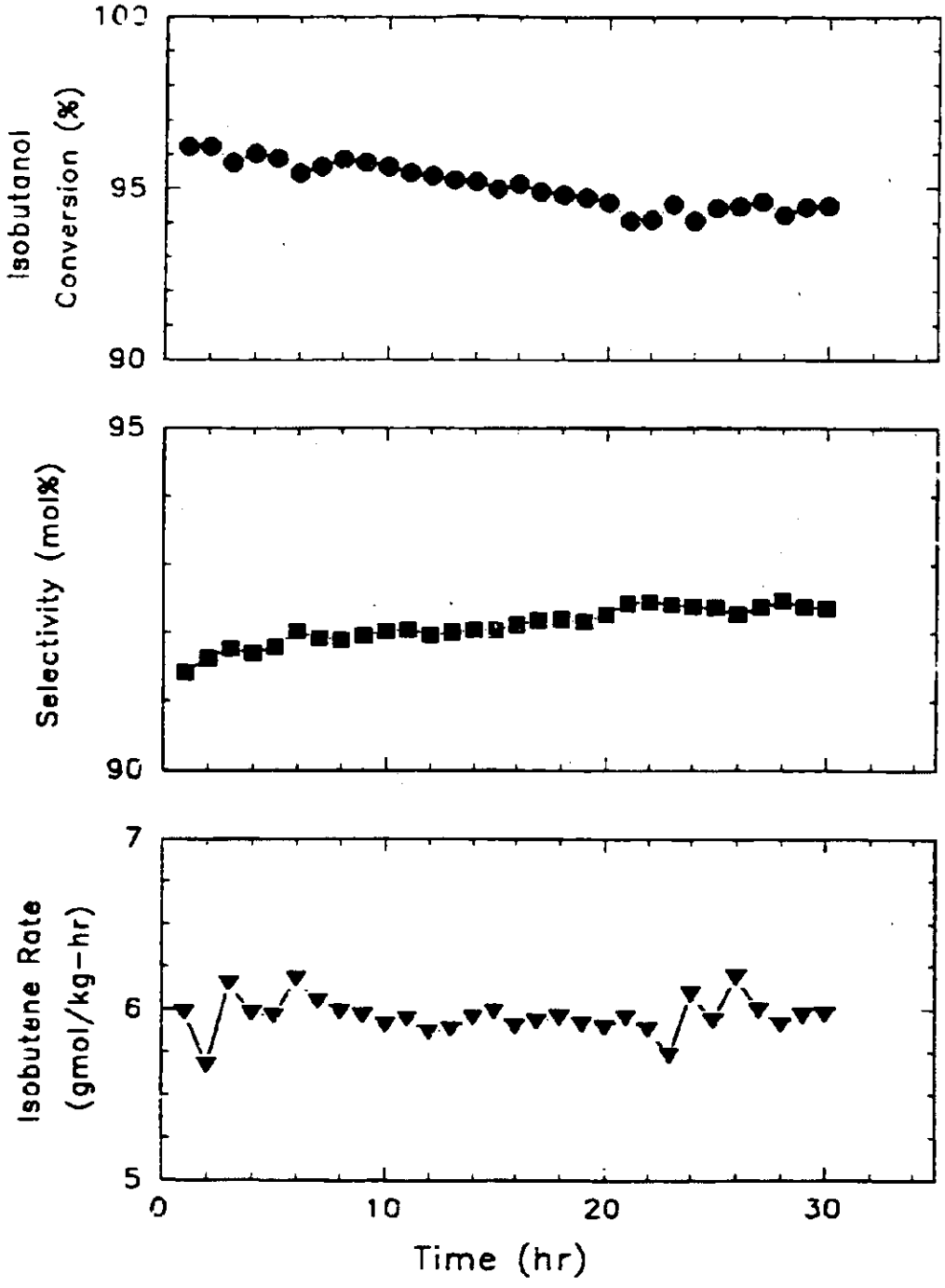


Figure 8



BATDOE3.SPG

Figure 9
Isobutanol Conversion vs. Normalized Feed Rate

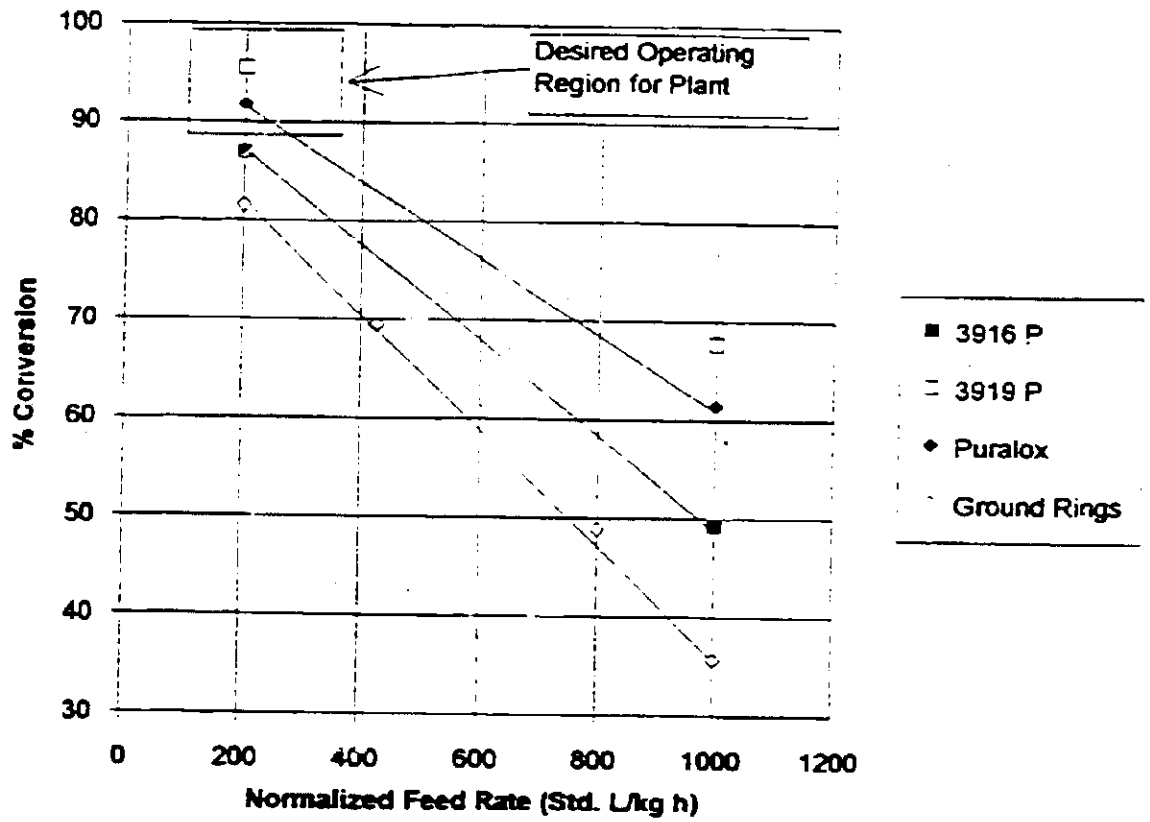


Figure 10
Isobutanol Conversion vs. Time on Stream
Research and Commercial Grade Feeds

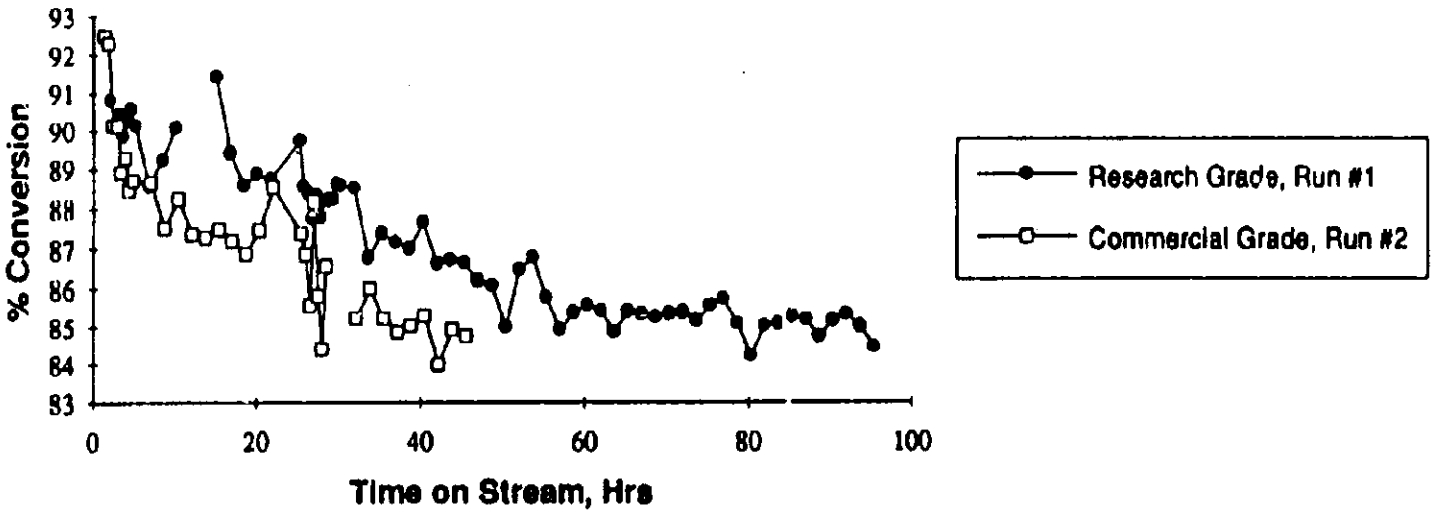


Table 1
Simple rate-law expressions for dehydration reactions

A. **Power Law**

$$-r = k\,(p_a)^{0.494}\,(p_w)^{-1.22}$$

B. Knözinger's[1] (isobutanol dehydration over g-Al_2O_3 at low T, P)

$$-r = k\,p_a^{1/2} / (\,p_a^{1/2} + p_w)$$

C. Similar to Gottifredi, et al.[2] (2-propanol dehydration at low T)

$$-r = k\,p_a / (1 + K_a\,p_a + K_w\,p_w)^2$$

D. from Miller and Kirk[3] (n-butanol dehydration)

$$-r = k\,(\,p_a - p_w^2/K\,) / (1 + K_a\,p_a + K_w\,p_w)$$

ONLY THE POWER LAW MODEL FITS THE DATA.

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1. H. Knözinger, *J. Catal.*, **12** (1968) 121-128.
2. Gottifredi, J.C., Yeramin, A.A., and R.E. Cunningham, *J. Catal.*, **12** (1968) 245.
3. Miller, D.N. and R.S. Kirk, *AIChE J.*, **8** (1962) 183.

Figure 11
Predicted Power Law Rate vs. Actual Rate

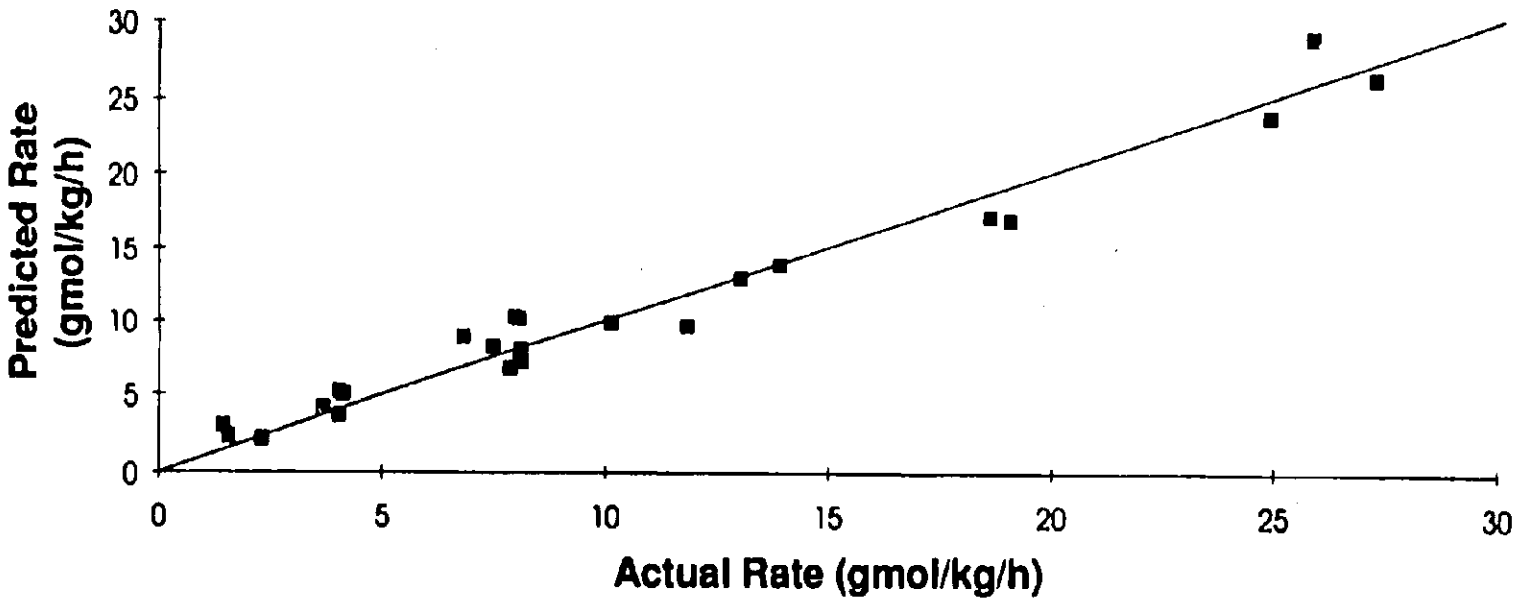

FIGURE 12

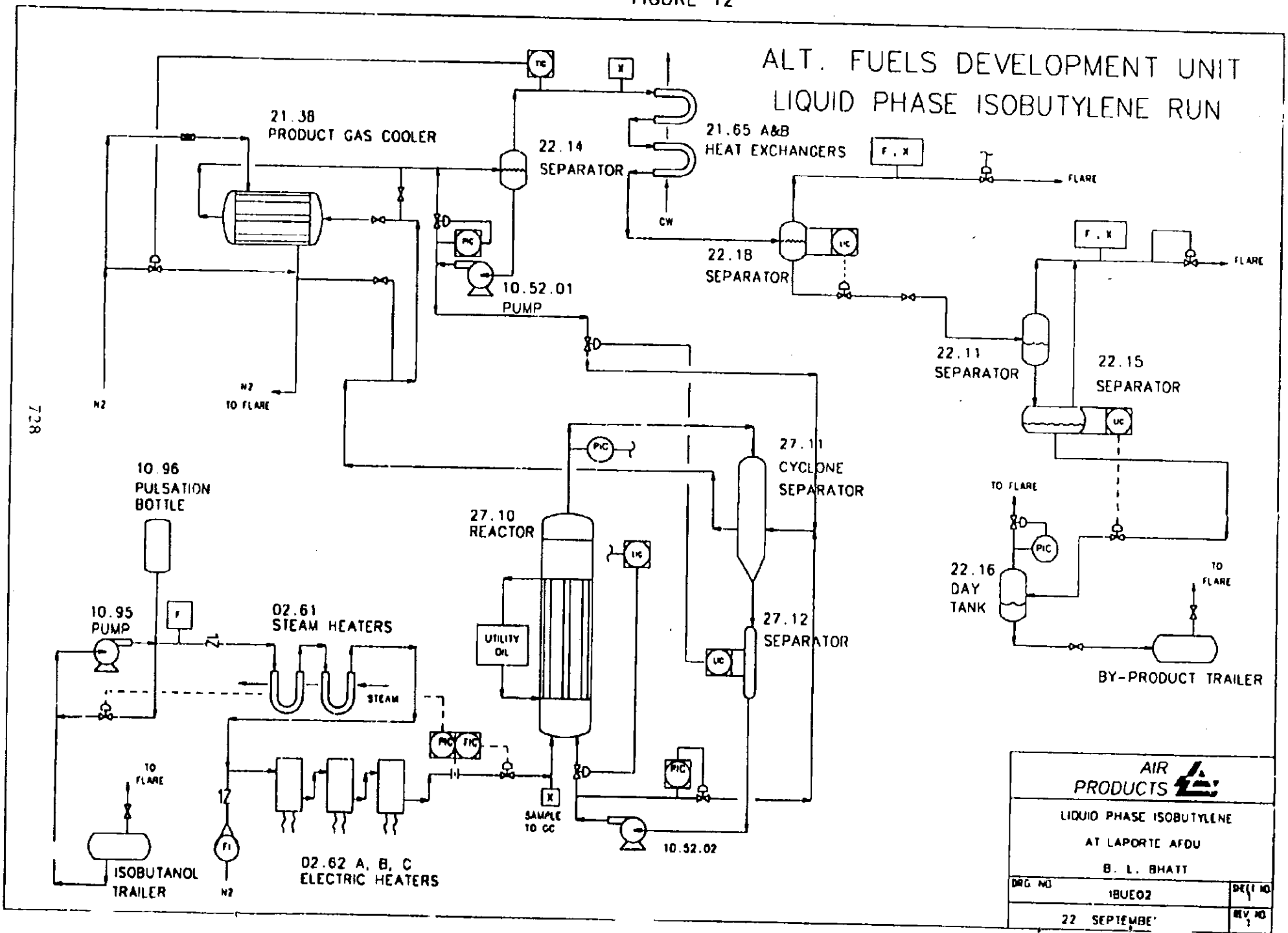
ALT. FUELS DEVELOPMENT UNIT
LIQUID PHASE ISOBUTYLENE RUN
21.38
PRODUCT GAS COOLER
22.14
SEPARATOR
21.65 A&B
HEAT EXCHANGERS
CW
FLARE
22.18
SEPARATOR
10.52.01
PUMP
N2
N2
TO FLARE
22.11
SEPARATOR
22.15
SEPARATOR
10.96
PULSATION
BOTTLE
27.11
CYCLONE
SEPARATOR
27.10
REACTOR
UTILITY
OIL
TO FLARE
22.16
DAY
TANK
TO
FLARE
10.95
PUMP
02.61
STEAM HEATERS
STEAM
27.12
SEPARATOR
BY-PRODUCT TRAILER
TO
FLARE
SAMPLE
TO GC
10.52.02
ISOBUTANOL
TRAILER
N2
02.62 A, B, C
ELECTRIC HEATERS
AIR
PRODUCTS
LIQUID PHASE ISOBUTYLENE
AT LAPORTE AFDU
B. L. BHATT
DRG NO
1BUE02
22 SEPTEMBE
REV NO
1

TABLE 2

LIQUID PHASE ISOBUTYLENE DEMONSTRATION RUN

RUN NO.	DESCRIPTION	NO. OF DAYS ON-STREAM	TEMPERATURE DEG F	PRESSURE PSIG	SPACE VEL SL/HR - KG	LIN. GAS VEL (IN) FT/SEC
					N2	
AF-A4	REACTOR SLURRY HEAT UP	1	200 - 572	30	270	0.16 - 0.25
	PROCESS VARIABLE STUDY				ISOBUTANOL	
AF-R7.1	BASELINE	1	572	25	115	0.12
AF-R7.2	EFFECT OF SPACE VEL	1	572	25	230	0.23
AF-R7.3	EFFECT OF SPACE VEL	1	572	25	350	0.35
AF-R7.4	EFFECT OF TEMP	1	600	25	115	0.12
AF-R7.5	EFFECT OF PRESSURE	1	600	40	230	0.17
AF-R7.6	EFFECT SPACE VEL	1	572	25	400	0.40
AF-R7.7	RETURN TO BASELINE	1	572	25	115	0.12
	HYDRODYNAMIC TRACER STUDY					
	INJECTIONS				ISOBUTANOL	
AF-R8.1	GAS-INLET (2)	1/4	572	25	115	0.12
AF-R8.2	GAS-INLET (2)	1/4	572	25	230	0.23
AF-R8.3	LIQUID-TOP (1)	1/4	572	25	230	0.23
AF-R8.4	LIQUID-BOTTOM (1)	1/4	572	25	230	0.23
AF-R8.5	GAS-INLET (2)	1/4	572	25	315	0.32
AF-R8.6	GAS-INLET (2)	1/4	572	25	400	0.40
AF-R8.7	LIQUID-TOP (1)	1/4	572	25	400	0.40
AF-R8.8	LIQUID-BOTTOM (1)	1/4	572	25	400	0.40

FIGURE 13

LIQUID PHASE ISOBUTYLENE DEMONSTRATION

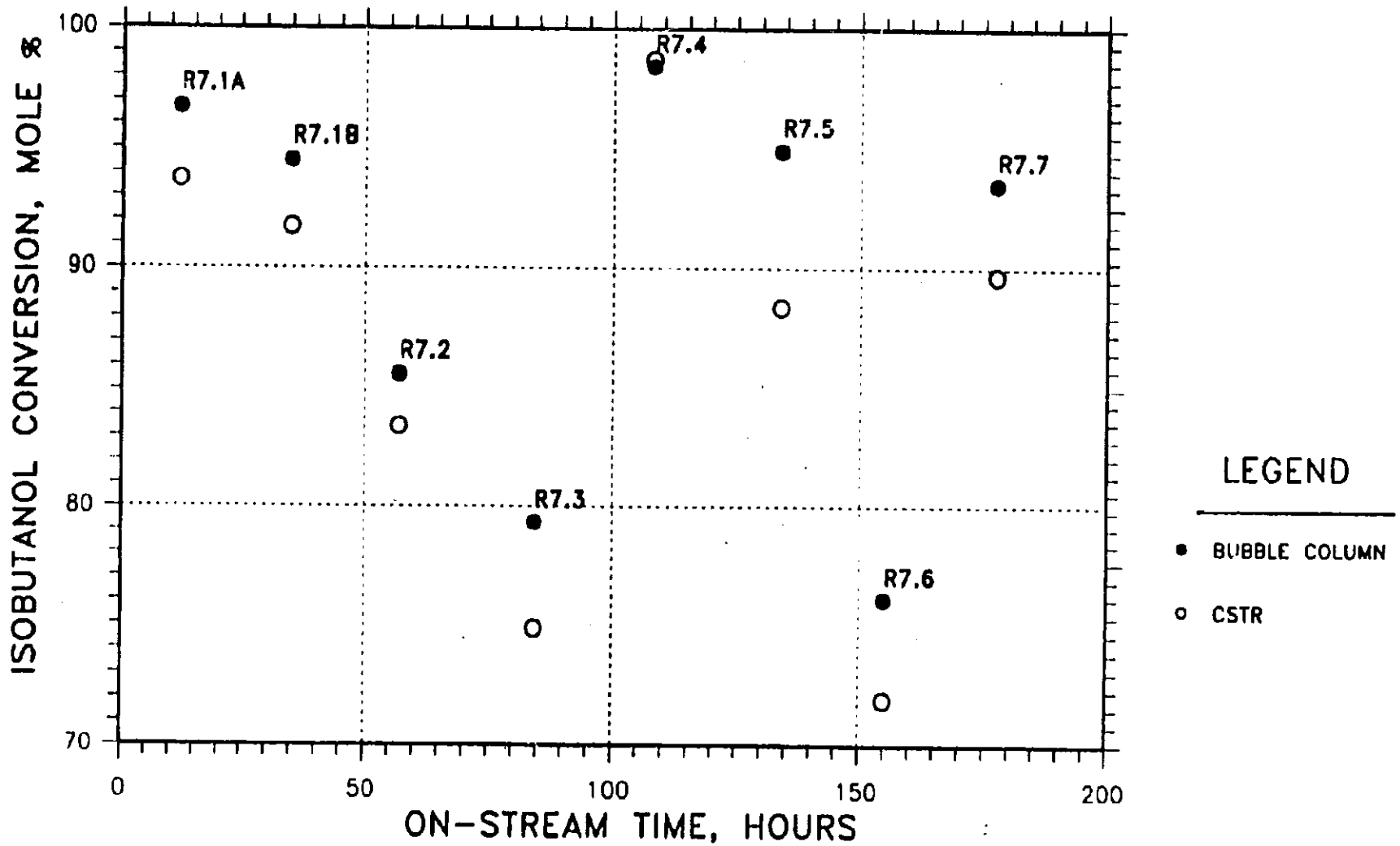

FIGURE 14

LIQUID PHASE ISOBUTYLENE DEMONSTRATION

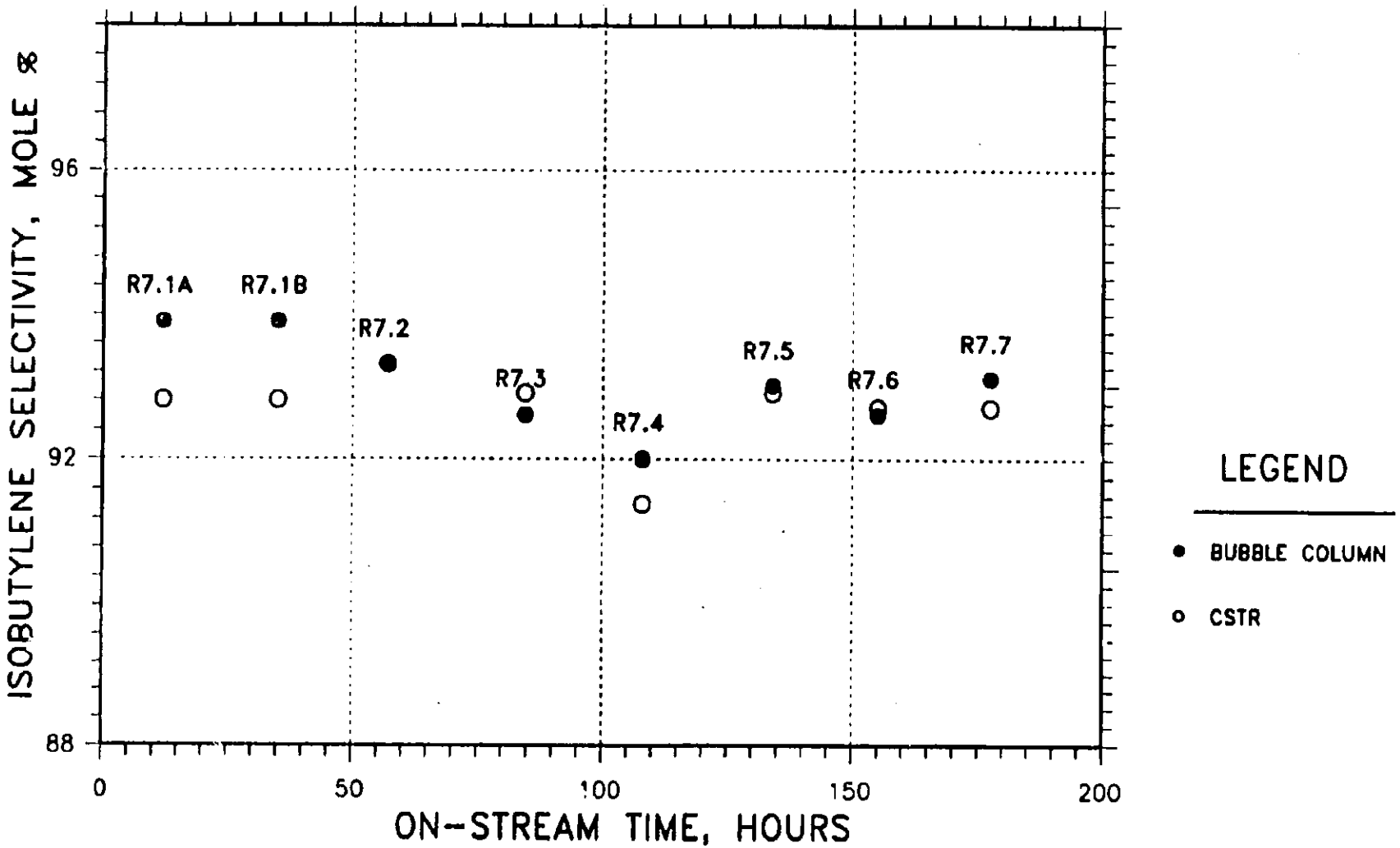

FIGURE 15

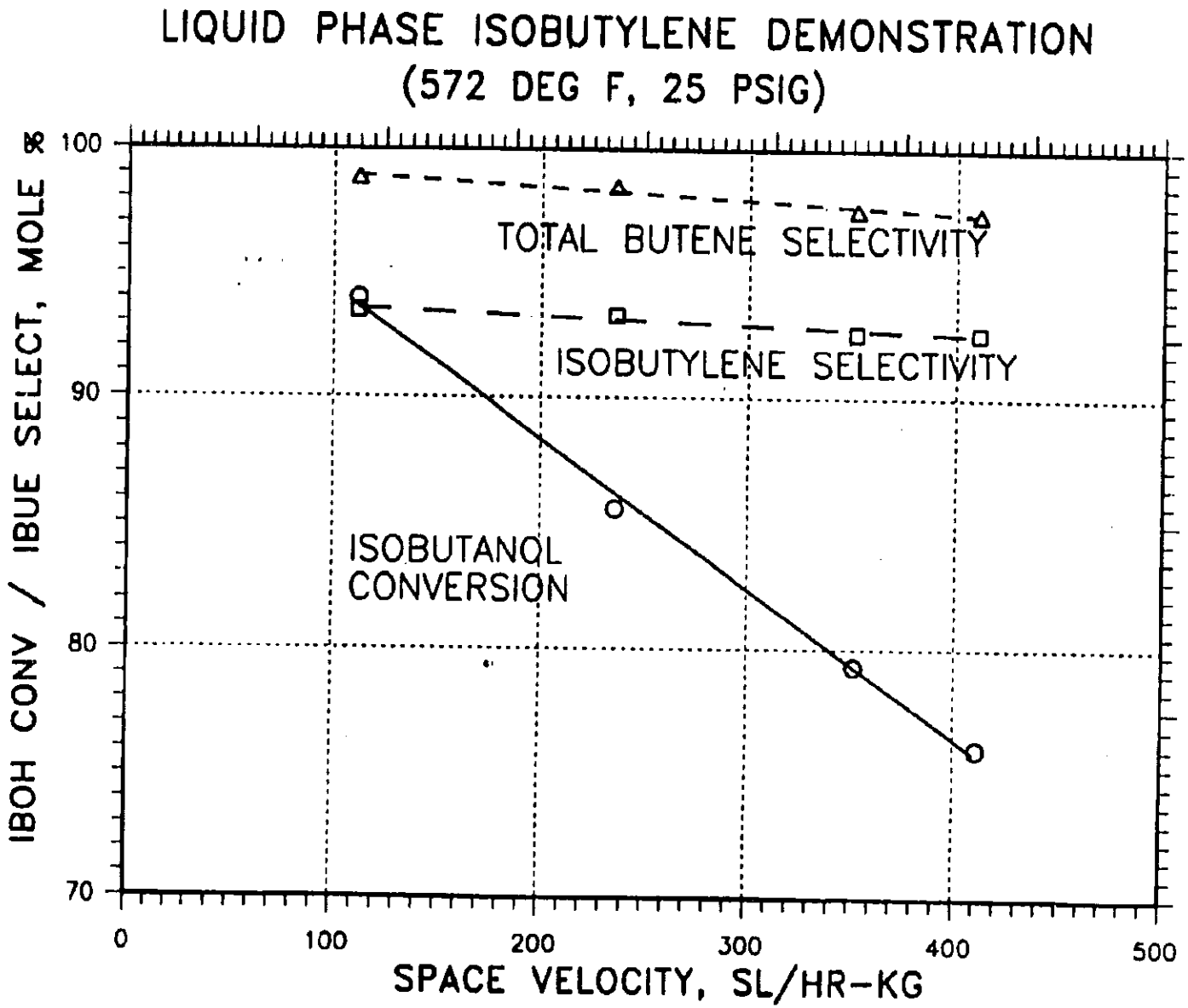
LIQUID PHASE ISOBUTYLENE DEMONSTRATION
(572 DEG F, 25 PSIG)
IBOH CONV / IBUE SELECT, MOLE %
100
90
80
70
0
100
200
300
400
500
SPACE VELOCITY, SL/HR-KG
TOTAL BUTENE SELECTIVITY
ISOBUTYLENE SELECTIVITY
ISOBUTANOL
CONVERSION

FIGURE 16

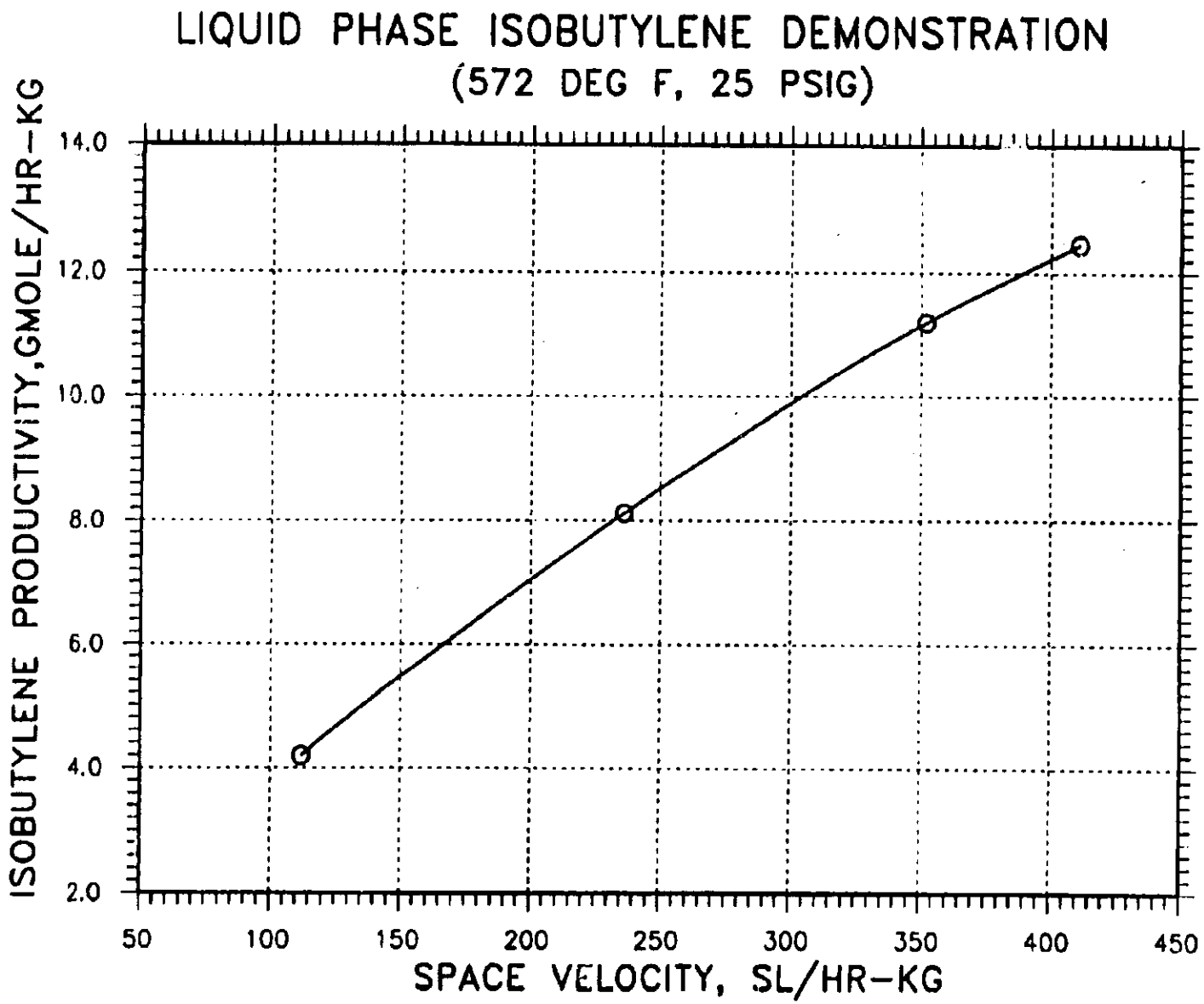
LIQUID PHASE ISOBUTYLENE DEMONSTRATION
(572 DEG F, 25 PSIG)
ISOBUTYLENE PRODUCTIVITY,GMOLE/HR-KG
14.0
12.0
10.0
8.0
6.0
4.0
2.0
50
100
150
200
250
300
350
400
450
SPACE VELOCITY, SL/HR-KG

FIGURE 17

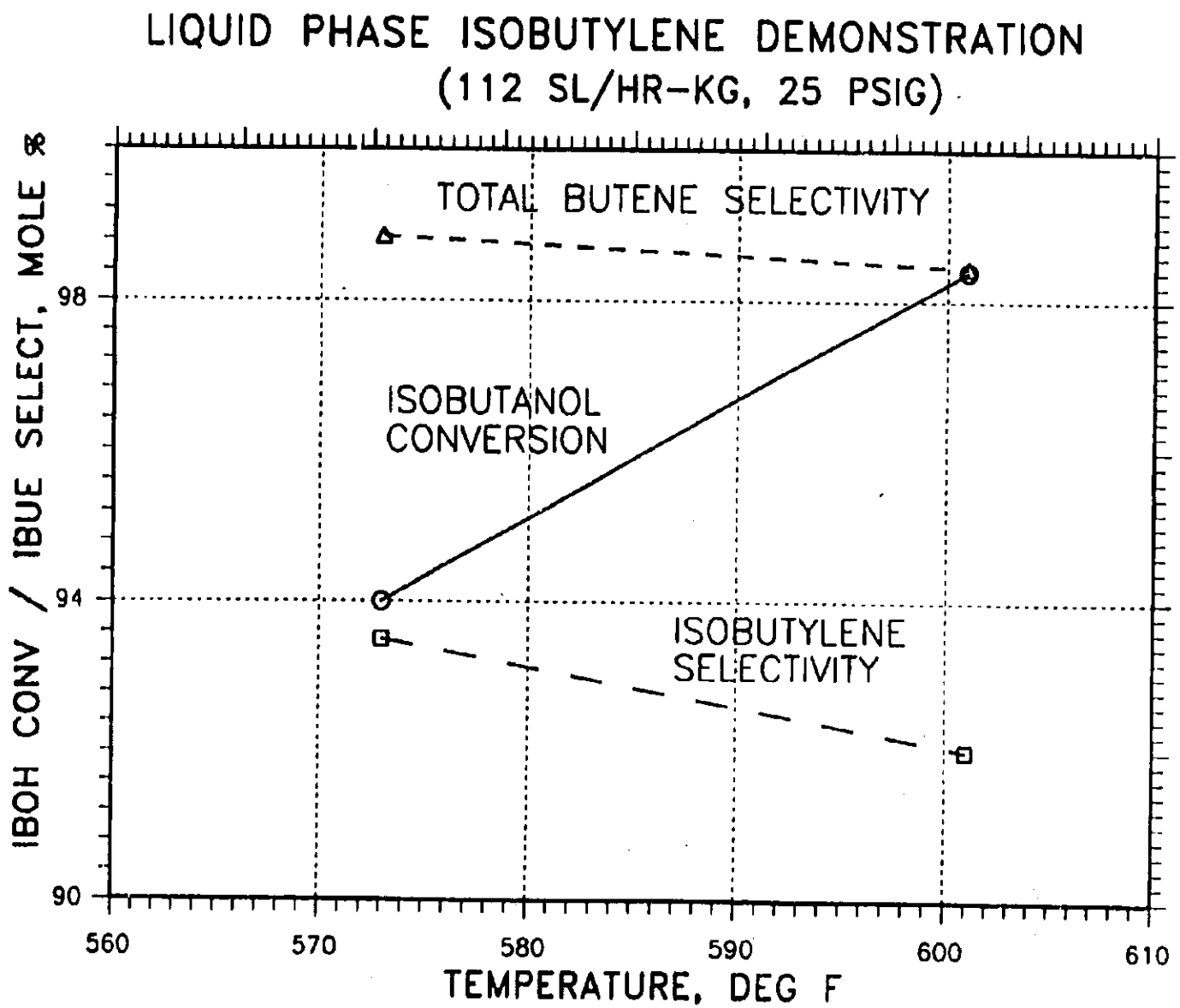
LIQUID PHASE ISOBUTYLENE DEMONSTRATION
(112 SL/HR-KG, 25 PSIG)
TOTAL BUTENE SELECTIVITY
ISOBUTANOL
CONVERSION
ISOBUTYLENE
SELECTIVITY
IBOH CONV / IBUE SELECT, MOLE %
98
94
90
560
570
580
590
600
610
TEMPERATURE, DEG F

Table 3
Effect of Pressure
$-r=k(P_{IBOH})^{0.494}(P_{H2O})^{-1.22}$

Conditions
(GHSV: 230 L $kg^{-1}h^{-1}$; Temp: 600 ^oF; Press: 40 psig)

	AFDU	Lab
Conversion	**94.9**	**88.4**
Selectivity	**93**	**93**

FIGURE 18

LIQUID PHASE ISOBUTYLENE DEMONSTRATION

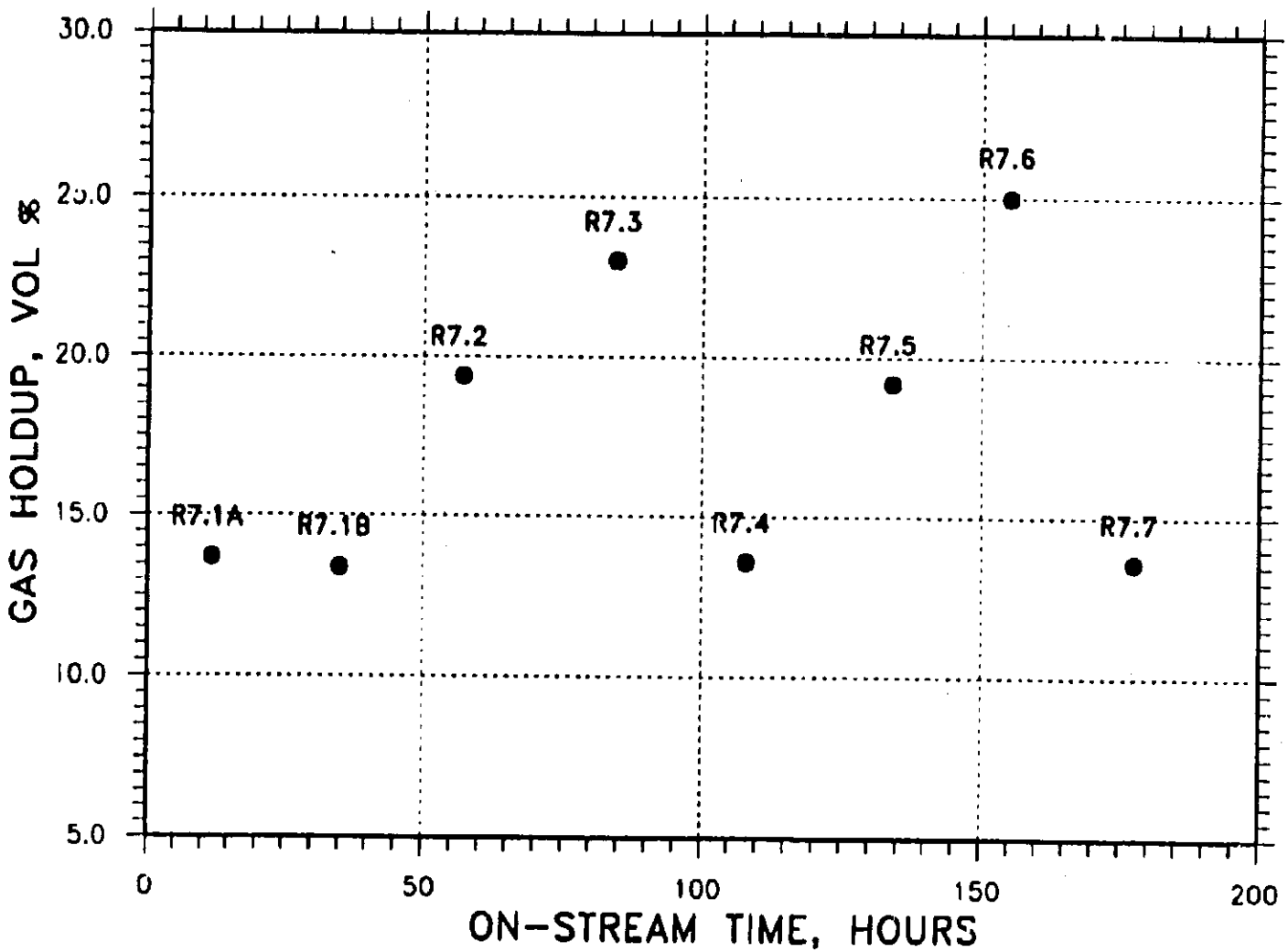

FIGURE 19: GAS HOLDUP VERSUS REACTOR HEIGHT (572 F, 25 psig)