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July 1987
ENGLISH

LIQUEFACTION OF COAL

DP/POL/82/002

Technical Report

Mission 14 June to 27 June 1987

Prepared for the government of the Polish People's Republic
by the United Nations Industrial Development Organization,
acting as executing agency for United Nations Development Programme

Based on the work of Joe E. Kimes,
Operations Expert

United Nations Industrial Development Organization
Vienna

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6.?

ABSTRACT

Project DP/POL/82/002

This report covers a trip to Katowice, Poland and, in particular, an operational review of the 30.0 kg/hr process developmental unit in the Institute of Carbochemistry in Tychy-Wyry. The period of this report was 14 June through 27 June 1987 inclusive. Although the unit was not in operation, I reviewed with Dr. Walter Matula and his staff the procedure for plant startup and operation, operational problems observed during their two (2) operating periods, and the operational data collected from these operating periods.

From this review, I recommend that (1) the directional flow on the high pressure letdown valves be reversed, (2) the gas compressor section be modified (3) the ability to measure different flows be improved, (4) the pasting solvent be evaluated using a micro-autoclave, (5) the coal concentration in the coal slurry be increased to improve coal conversion, (6) the number of flanges on the reactor system be reduced as improvements to the system are completed, and (7) the mechanism for the exchange of technical information with the Wilsonville, USA facility be established.

The focus of this report is on the recommendations as outlined above. These recommendations are based upon the daily meeting held with the different departmental heads within the Institute of Carbochemistry. This exchange of information through meeting was made necessary since the plant was not in operation during my entire visit.

INTRODUCTION

The period covered in this report was 14 June through 27 June 1987 inclusive. The job description for this project was DP/POL/82/002/11-05/32.1.I. The objective of this project was to advise on the basic technical problems of experimental units for direct coal hydrogenation, in particular: the evaluation of operating ability of individual sections of the modernized unit, discussion on startup schedule with particular attention to the proper order of starting up works, evaluation of availability of unit integrated mode of operation, discussion of selection of optimal variant of computer system for collecting data from the unit.

Since both the 30.0 kg/hr and the 5.0 kg/hr units were not operating, I met with the different departments within the Institute of Carbochemistry in Tychy-Wyry. Although I did tour both the 30.0 kg/hr and the 5.0 kg/hr units, the majority of the information for this report was received through a discussion with key personnel within these different departments.

Using this information from both the plant tour and these discussions, I have then made the enclosed recommendations to resolve the different observed operational problems with particular focus on the 80.0 kg/hr PDU facility. In this exchange of information, I outlined the evolution of the technological developments in coal liquefaction at the Wilsonville, USA facility. It was the operational experience gained from the Wilsonville facility that is contained in this report. The recommendations are therefore, based upon similar experiences from the fourteen (14) years of operation at the Wilsonville facility.

Once the period for observation and discussion was completed, I expressed my gratitude to Dr. Jozef Matoszewski's staff and to Dr. Walter Matula and his staff for a most informative visit to the Central Mining Institute and to the Institute for Carbochemistry in Tychy-Wyry. I then traveled to Vienna for a debriefing with Dr. M. Derrough and the UNIDO staff involved with this project. The efforts of all were appreciated since this trip proved to be very successful.

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APPENDIX 20-

RECOMMENDATIONS

This section of the report outlines the objectives of my trip to Poland. As an expert in the operation of a coal liquefaction facility, the focus of my trip was to work with the staff at the Institute of Carbochemistry in Tychy-Wyry, to identify possible operating problems, and recommend possible solutions. As a basis for this discussion, I have included in the Appendix Figure 1, a basic flow diagram of the Tychy-Wyry facility. The following list are my recommendations. The format for this sections is that the identification of a problem is followed by my recommendation to resolve each problem. These recommendations are arranged in order of their importance, with those conditions affecting the safety of the plant listed first.

1. The directional flow on the high pressure letdown vales should be reversed.

The observed problem with the letdown was two (2) fold. First the plant experienced very short operational life of the high pressure letdown valves, and second, the top section of the valve bodies showed signs of excessive erosion.

The flow pattern on the letdown valves, although standard, is backward to the Wilsonville facility. This was listed first because of its serious nature. Currently, the flashed material is being depressurized within the valve body itself. It was demonstrated at Wilsonville that this flow pattern was unsafe and detrimental to long valve life. It is unsafe because the valve body is currently exposed to a very erosive material which can erode away the valve body and begin to leak externally. This leak because of the chemicals involved then becomes an extreme fire hazard.

I have therefore recommended that the flow through this valve be reversed. This allows the flashed material to be depressurized away from the working of the valve. As part of this recommendation, I would also add an additional small vessel similar to the Wilsonville facility. A drawing of both the valve and the vessel are shown in the Appendix, Figures 2 and 3. With this design, the supersonic velocities are focused away the internals of the valve and into this small vessel, which was designed for such service. With this design, the Wilsonville was able to operate a single letdown valve for periods up to three thousand (3000) hours.

The level control on the high pressure separator should also be modified. These modifications should improve the ability to control the level within the high pressure separator and as such reduce the amount of gas being discharged via the high pressure letdown valves. This will again help to reduce the velocities at the letdown valves and lengthen the life of these valves. Currently this control system is periodically pumped clear with oil. The level control should be purged with hydrogen gas for two reasons. First, the gas purge should provide for better level control. And secondly, the liquid material in the high pressure separator is maintained at temperatures sufficiently high for chemical reaction to take place and currently is essentially void of hydrogen. Such conditions at Wilsonville produced a coke-like-material in the high pressure separator. The conclusion was that this coke-like-material was formed as a retrograde reaction caused by high temperature and the lack of hydrogen. The hydrogen purge will provide a small but adequate hydrogen flow to greatly reduce the formation of these coke-like-material. The high pressure vessels operating volume should also be operated at a minimum to reduce the possibility of such retrograde reactions.

2. The gas compressor section should be modified.

The observed problem is the gas compressor was damaged during the last coal feed run and is currently down for repairs. The recycle gas compressor was damaged during operation. All parties agreed that the most likely cause of this damage was liquid carry-over from the reactor system.

The solution to this problem has two parts. First, the overhead flows within the reactor system were much higher than expected. As a result the design of the cooling system for this flow was too small. With the current operational data, a adequate cooling system can now be designed and installed. This installation should be designed with sufficient capacity to handle any expected increases to this flow in the future. Also, as the amount of solvent added to the coal slurry is reduced, the amount of flashed liquids should be reduced. This part of the recommendation is discussed in greater details in the fifth recommendation since the reduction of solvent in the coal slurry may also improve coal conversion.

Second. the suction vessels on the recycle compressor is too small. The Wilsonville facility experienced similar liquid carry-over during upset conditions. To protect the compressor section in Wilsonville, a one hundred (100) gallon high pressure vessel. that no longer was in service, was installed on the compressor suction system. A vessel of this size is expensive to construct. Since a suitable vessel was not readily available. the design and installation of a cost effective sized vessel needs to be completed.

Although not entirely related to the above problem. I would recommend two additional modifications to the compressor system. First. I would install a second vessel on the discharge of the compressor section. The purpose of this vessel is to (1) provide a means to collect and drain liquid that has condensed at the higher discharge pressure and (2) to use the volume in this vessel as a pulsation damper to reduce flow surges caused by the compressor strokes. The second modification would be to install a pressure control system on the discharge of each compressor. This installation is standard on most compressor system within the USA and is illustrated in the Appendix, Figure 4. The purpose of this system would be to provide better flow control of the gas flows from each compressor. A second benefit is that the gas flow from the compressor can be stopped and the compressor will automatically recycle itself back to the compressor suction. With this design, the compressor flow can be stopped and started with the compressor safely operating during the entire period. This would permit the plant personnel to direct their attentions to other areas of the plant with the knowledge that the compressor section is on automatic recycle until needed.

3. The ability to measure flows needed to be improved.

In general terms three types of problems were observed. Since the recommendations are different for each problem, each problem is discussed separately. The first problem is the measurement of the overhead stream since the water and oil products form an emulsion that prevents the accurate measurement of either the water or oil component within this stream.

This problem was observed also at the Wilsonville facility. The cause of the problem is that the specific gravity of both components are too close for adequate separation. The solution is simply to separate the mixture into three streams by means of a simple flash. The simple flash system is illustrated in the Appendix, Figure 5. Briefly, the mixture is heated to approximately 300° F. and the heavier liquid material is then collected. The flashed vapor is then cooled to condense all liquid. This liquid contains two components, the water and the lighter oils. In a second vessel these two components are separated by means of a weir within the vessel. Because the heavier products were removed through the flash procedure, these two products now separate easily since their specific gravities are no longer similar.

The second observed problem concerned the ability to measure gas flows accurately. The recommendations already outlined to the gas compressor section will also improve the flow measurements. The reduction of liquid in the gas stream and compressor surges will reduce the spikes currently observed in this flow measurement. The next step is to install purchased calibrated flow orifices with electronic flow measurement devices (Dp cells). Operational experience has shown that small amounts of light liquids can still condense within the Dp cell, itself, and cause flow measurement errors. As a result, these Dp cells need to be mounted as high above the orifices as possible to reduce the collection of liquids within the cells themselves. And to further reduce the collection of liquids within these flow cells, the cells should be installed so that they would freely drain of any liquid. One difficulty mentioned was such calibrated instrument systems are difficult to obtain in Poland.

The third observed problem concerned the ability to measure various liquid streams which contained heavy solid-like materials. Our discussion focused on several streams from the coal slurry measurement to the measurement of various product streams. In this discussion, we evaluated the different methods of measuring coal slurry at Wilsonville. The most accurate method employed is the use of vessels with weigh scales. Both the coal slurry blend vessel and the feed vessel are installed on weigh scales. Because such installations require piping modifications to be accurate, this expense has limited this application to only the feed tanks at Wilsonville. It is from these measurements the Wilsonville facility based its material balance data. As a flow indication, the wilsonville facility uses the U-tube flow meters manufactured by Micromotion. To date this instrument is the only device which has proven to provide repeatable flow indication in streams similar to the slurry feed.

Since the use of weigh scales are very expensive, wilsonville has employed the use of a small vessel that batches the continues flow to reduce the cost. In this manner, the weights of various product streams are obtained. These vessels have two control valves; one on the inlet and the other on the outlet. Both are controlled by the level within the vessel. This level is measured by a gas purged differential pressure cell which has been calibrated for the specific product. As the vessel fills, the bottom discharge valve is closed, and the inlet valve is open. When the level reaches a set "high level", the inlet valve closes and the bottom discharge valve opens. The material in the vessel drains to a set "low level" and then the two valves are again reversed to fill the vessel. Since the weight of one cycle of this batch system has been determined, the flow measurement consists of simply counting the number of cycles the vessels dumps and multiplying by the calibrated weight for each dump.

4. The pasting solvent should be evaluated using a micro-autoclave.

Since the pasting solvent is void of any non-distillables fractions, there is a concern that the feed preheater might coke. This is of particular concern during the initial startup of the unit.

I recommend that a test using a micro-autoclave be used to evaluate the pasting solvent. This test was used daily at Wilsonville until the recycle of heavier non-distillable fractions were returned in the pasting solvent. This procedure was so important to the research efforts at Wilsonville that the Topical Report No. 8 was issued outlining this procedure, the equipment used, and the data from different operating periods at Wilsonville. Because this report goes into such detail, I left one report with the staff at the Institute of Carbochemistry.

and have enclosed one in the Appendix Figure 6.

The continual use of a micro-autoclave at the Institute of Carbochemistry is most likely not entirely necessary. Since after startup the high pressure separator bottom product is reclyced, the resid in this stream should reduce the formation of coke in the preheater. However, this assumption should be verified before such test should be considered no longer necessary. During this evaluation period, the collection of this data from the micro-autoclave and the data collected from the NMR should be correlated. Most-likely the NMR data will prove to be superior in the long run, but since such data was not collected at Wilsonville the final analysis has to be determined through the above outlined comparison test.

5. The coal conversion in the reactor systems should be increased.

The measured coal conversion to date has been much lower than the similar operating data from Wilsonville. Currently the coal conversion has been eighty-five (85) percent as compared to ninety-one (91) percent at Wilsonville. Both conversions are based upon a laboratory test using THF.

To improve coal conversion, I recommend three different steps. Each of these steps should be taken after the unit has demonstrated the ability to operate smoothly for a period of time. The reason for the delay is that these recommendations will cause the viscosity of the coal slurry and the high pressure recycle loop material to increase. To minimize additional operational problems, all other operational problems should either have been resolved or at least made manageable before embarking on these new run conditions.

The first step is to gradually decrease the amount of solvent in the pasting solvent. Currently the operations has been with thirty-eight (38) percent coal concentration in the slurry feed. The design maximum for this system was a fifty-five (55) percent coal concentration in the slurry feed. As this concentration is increased, the amount of solvent through out the system will be decreased. One immediate benefit would be the reduction of flow to the already overloaded overhead product coolers. (This was mentioned in the recommendation 2.) Another affect will be on the coal concentration in the reactor. If this reaction system response similar to the Wilsonville facility, then this should improve coal conversion. Data from the laboratory test at the Institute of Carbochemistry would support this recommendation also. Our discussion indicated that from their data this could improve coal conversion by an additional two to three percentage points.

The second recommendation would be to reduce the solvent in the high pressure separator recylce loop. The reason this recommendation should follow and not proceed the first recommendation, is simply the solvent in this loop will naturally drop as the solvent is reduced in the slurry feed. Therefore, to prevent the viscosity in this loop from getting to high, drop the solvent in this loop secondly. Currently the solvent in this loop is averaging fifty-five (55) percent. It should be reduced to the minimum design of thirty (30) percent. Again, data from the laboratory test at the Institute of Carbochemistry would support this recommendation. Our discussions indicated that this could improve coal conversion by an additional one to two percentage points.

Once the above two steps have been completed, the third step is then to determine what reactor temperature gives the optimum coal conversion while at the same time not greatly increasing the C1-C4 gas make. The data from the operation of the thermal reactor at Wilsonville indicated that beyond the optimum reactor temperature, coal conversion dropped and the C1-C4 gas make increased greatly. This data was also very depended upon the quality of the pasting solvent. But with the analytical tools available to the Institute of Carbochemistry, such test should be developed in the smaller units and then verified in the 80 kg/hr PDU facility.

One mechanical limitation to these recommendation may be the feed pumps. Currently these pumps are variable stroke. As such, at times, these pumps have areas within the pumping chamber that can fill with coal solids. As these solids accumulate, they can plug the pump or break loose and flow into the system as large chunks and plug smaller opening within the reactor system. The solution in this case is to change the pumps to variable speed. Similar to Wilsonville, then the feed pumps would have a full stroke which prevents the accumulation of coal material within the pumping chamber: and the pump revolutions varied to provide the desired coal slurry feed rate.

6. The number of flanges on the reactor system should be reduced as improvements to the system are completed.

The observed problem is the significant increase in the number of flanges within the reactor system. The experience at Wilsonville has shown that each additional flange increases the chance of a leak. With each leak comes the possibility of a serious fire. At Wilsonville, the forty (40) foot reactor has only two (2) flanges-one at the top and one at the bottom of the reactor. At the 30 kg/hr PDU facility, a similar reactor has ten (10) flanges.

I recommend that as modifications are completed on the reactor system a systematic procedure is developed to reduce the overall number of flanges on the this high pressure system. The difference in the number of flanges also reflects a major difference between the facility at Tychy-Wyry and the facility at Wilsonville. Within the maintenance department at Wilsonville, there are a minimum of five (5) men who are qualified to weld on the stainless steel reactor piping system.

Also, within the facility at Wilsonville, an inventory of the necessary types of pipe and the equipment to perform such procedures is constantly maintained. At the Tychy-Wyry facility, there is neither the equipment or the personnel to perform such routine work. This limitation has been reflected in the design of the reactor system-additional flanges. Hopefully, as the operating staff develops operating experience, they will know which flanges that were installed in case of a plug, etc. can be eliminated.

7. A mechanism should be developed so that non-proprietary data can be exchanged.

The obvious problem is that two independent groups are developing coal liquefaction technology with limited funds. In particular, the Institute of Carbochemistry could benefit a great deal from the historic data collected and reported from Wilsonville.

I recommended that all parties involved within the two facilities explore the possibility of such an exchange. Neither facility has the financial resources to duplicate experimental data that already exists. Although, the engineering and design staff at Tychy-Wyry have a great need for the operational and design information from the Wilsonville facility; this exchange need not be totally one-sided. The research facility at Tychy-Wyry has at their laboratory such equipment as the NMR and the electron microscope. Data from such developmental work could improve the current modeling effort being developed at Wilsonville.

This exchange can be as simple as arranging for the publish reports from each unit to be sent to the other party. If this informational exchange pro ed beneficial, then a full technical exchange in meeting form could be employed. Since operating funds are limited for both facilities, such exchanges could possible reduce expensive duplication of research for both parties.

To begin exploring the possibility of such an exchange, I met with Gary Styles, Project Manager for Southern Company Service on Tuesday, 30 June. He indicated that such an exchange would have to be approved by all the current sponsors at Wilsonville, and that he would begin exploring this possibility with the sponsors in the near future.

CONCLUSIONS

The focus of the recommendations were on the liquefaction section of the 80 kg/hr PDU facility at Tychy-Wyry. This is illustrated in the top portion of the basic flow diagram. Although, the research staff had completed some bench scale developmental work on the coking section, that section of the plant had not been operated. And the general feeling at the facility was, at the time of my visit, that section would not be operated because of limited operating funds. Because of the this information, I focus my attention on the part of the facility which (1) had been operating and (2) would be operating in the future.

The other area of the general objectives which I did not spend much time on was a computer system for the collection of plant data. I outlined how the system in Wilsonville operated. But I did not dedicate much review time for this topic since the engineering staff indicated that, even if a computer was available, the instruments needed to relay the operating data to the computer from the reactor system were difficult to obtain. Although a computer system has enabled the Wilsonville facility to greatly increase the amount of data evaluated, Wilsonville did operate for approximately half of it's history without such a system. It simply requires more people to evaluate the data and this evaluation requires more time.

In general, however, the facility at Tychy-Wyry has some analytical equipment that far exceeds the ability of the Wilsonville facility. Along with such equipment as the NMR and the electron microscope, the research staff at Tychy-Wyry are of greater numbers. As such they can provide some analytical data for their reactor system that is almost impossible to obtain at Wilsonville. Another strong point for this facility is the ability to evaluate various run conditions in the smaller 5.0 kg/hr reactor system.

Most of the recommendations I have presented are natural for a new facility. Each time a new facility is started, each individual piece of equipment has to be evaluated from an operational point of view. With the experience of the staff at the Tychy-Wyry facility, they will go through a learning curve as we all have to do. The one factor that concerned me was the general level of the plant operating staff. The very nature of this type of research developmental work requires a specially trained operating work force. The Wilsonville facility has generally understood this principle and has always strived to maintain a highly skilled pool of plant operators. To be able to train and keep such operator, a competitive pay scale has been offered. The Tychy-Wyry facility perhaps understands this principle also. Unfortunately, their facility does not compete as well with the other employers in the area. Therefore, their ability to attract a top operating staff has been affected. The evaluation of this segment of their facility was not possible since it was not in operation during my visit.

I again want to thank Dr. Jozef Matoszewski's staff and Dr. Walter Matula and his staff for a most informative visit. Since the plant was not operating, most of my observations were made during the daily discussion with the different departmental staff members. It was through their openness during these discussion that I was able to complete this assignment.

DAILY JOURNAL

14-15 June: Spent these two days traveling from Chelsea, Alabama to Katowice, Poland

16-17 June: Spent these two days reviewing the research developed for this PDU facility, learning the basic flows as outlined in the flow schematic in the Appendix Figure 1.

18 June: This was a holiday so no visit was made to the research facility at Tychy-Wyry.

19 June: Spent this day in reviewing startup of the facility, discussing plant operational problems and reviewing operational data from the two periods of operation.

20-21 June: Marion Krzyminski,a staff engineer who translated for me during the discussion, acted as host and showed me some of the local sites.

22 June: Spent the day discussing plant operation with the engineers concerned with the mechanical design of the PDU facility.

23 June: Spent this final day reviewing plant operating data with both the research group and the engineering staff responsible for plant modeling.

24 June: Spent this day travelling to Vienna, Austria from Katowice, Poland.

25 June: Met with the UNIDO staff and held a debriefing meeting with Dr. M. Derrough.

26 June: Spent this long day travelling to Chelsea, Alabama from Vienna, Austria.

APPENDIX

FLOW DIAGRAM FOR THE 80 KG/HR PDU. . . . . FIGURE 1.

INTERNAL DESIGN HIGH PRESSURE LET DOWN VALVE . . . . . FIGURE 2.

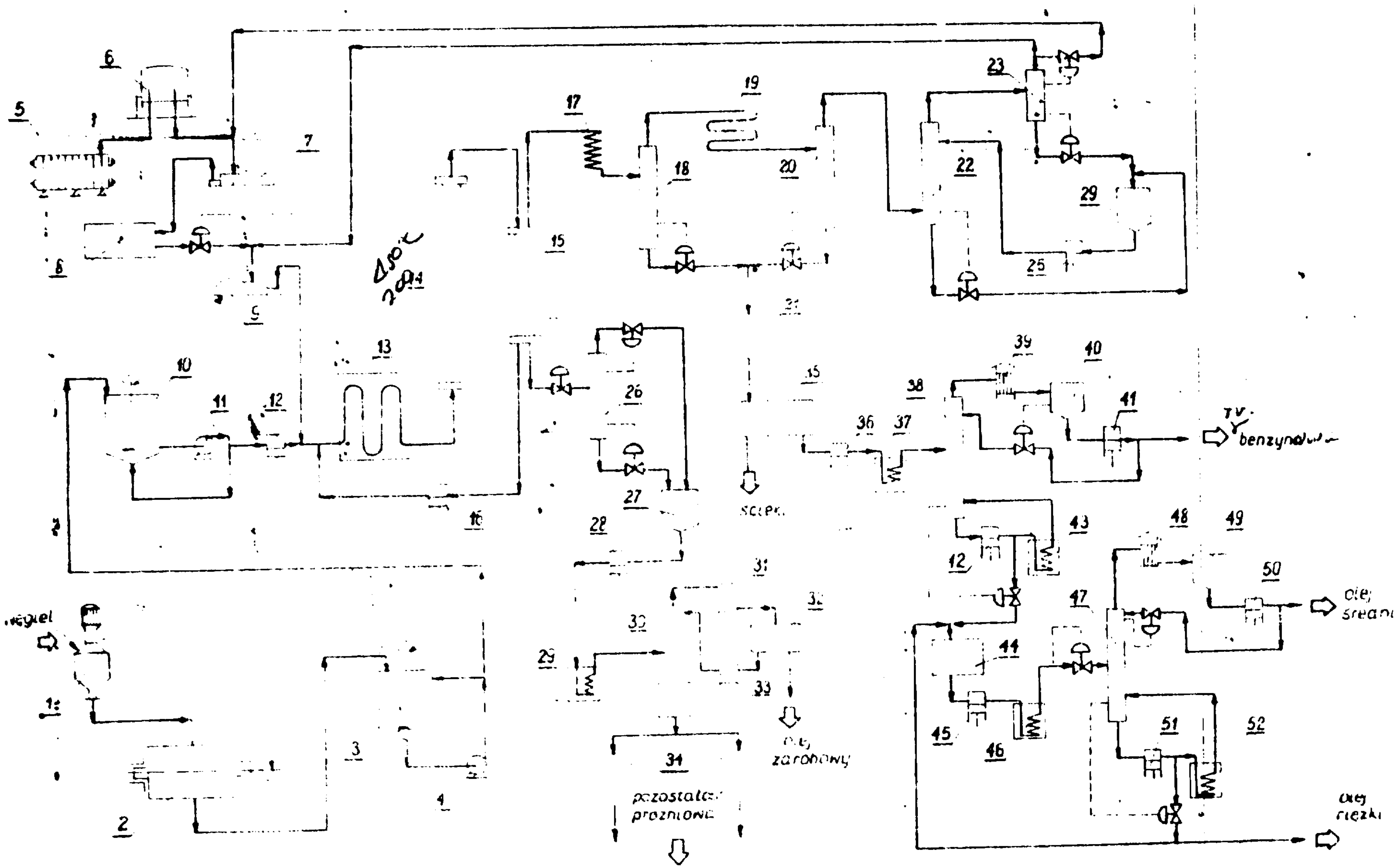
DESIGN OF THE HIGH PRESSURE LET DOWN VESSEL. . . . . . FIGURE 3.

PRESSURE CONTROL SCHEMATIC FOR GAS COMPRESSOR. . . . . FIGURE 4.

PRODUCT SEPARATION VIA FLASH SYSTEM. . . . . FIGURE 5.

WILSONVILLE TECHNICAL REPORT NO. 8 . . . . FIGURE 6.

FIGURE 1



Rys. 1 Schemat technologiczny instalacji

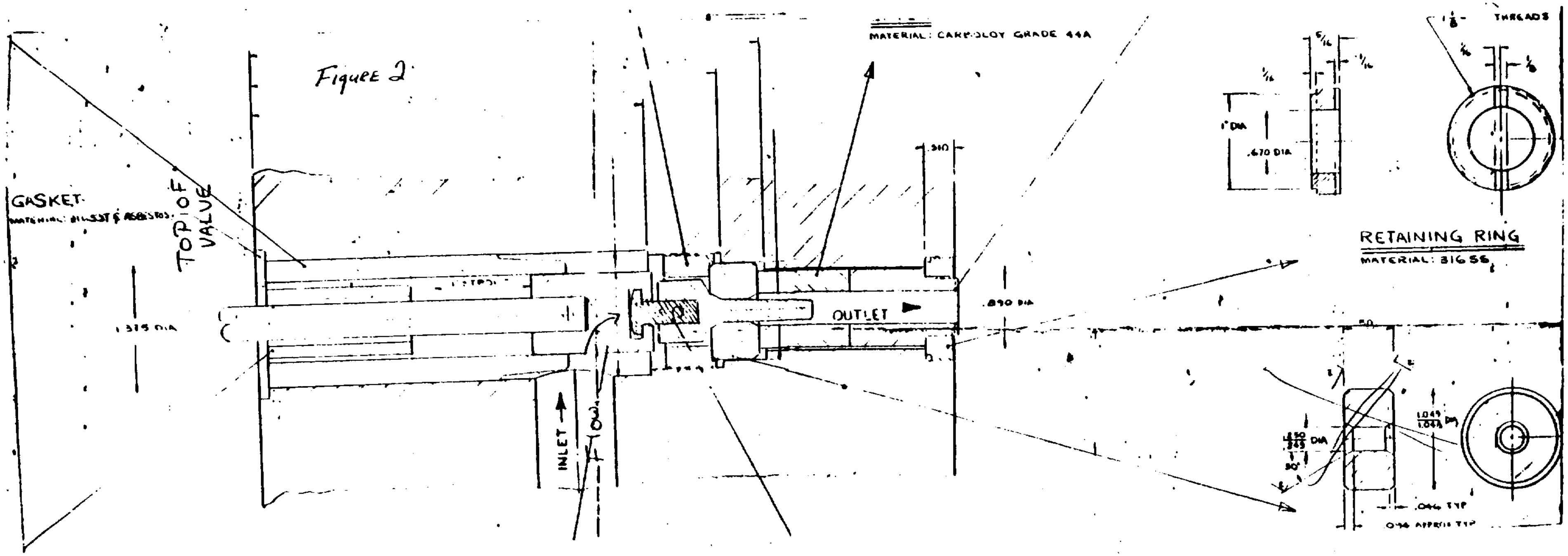


Figure 2

GASKET
MATERIAL: 316 SST & ASBESTOS
TOP OF VALVE
1.375 DIA
INLET
OUTLET
.310
.890 DIA
MATERIAL: CARBOLOY GRADE 44A
1" DIA
.670 DIA
5/16
1/16
1/8
THREADS
RETAINING RING
MATERIAL: 316 SS
1.049
1.048 DIA
DIA
30°
.046 TYP
.046 APPROX TYP

Figque 3

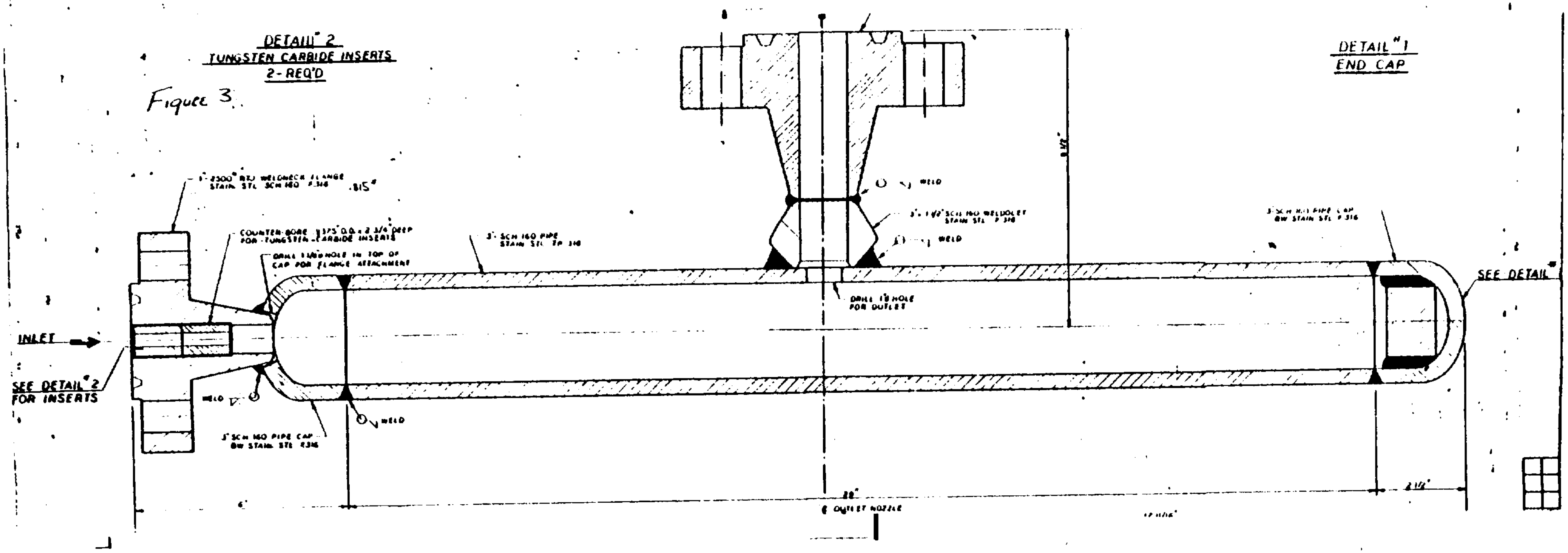
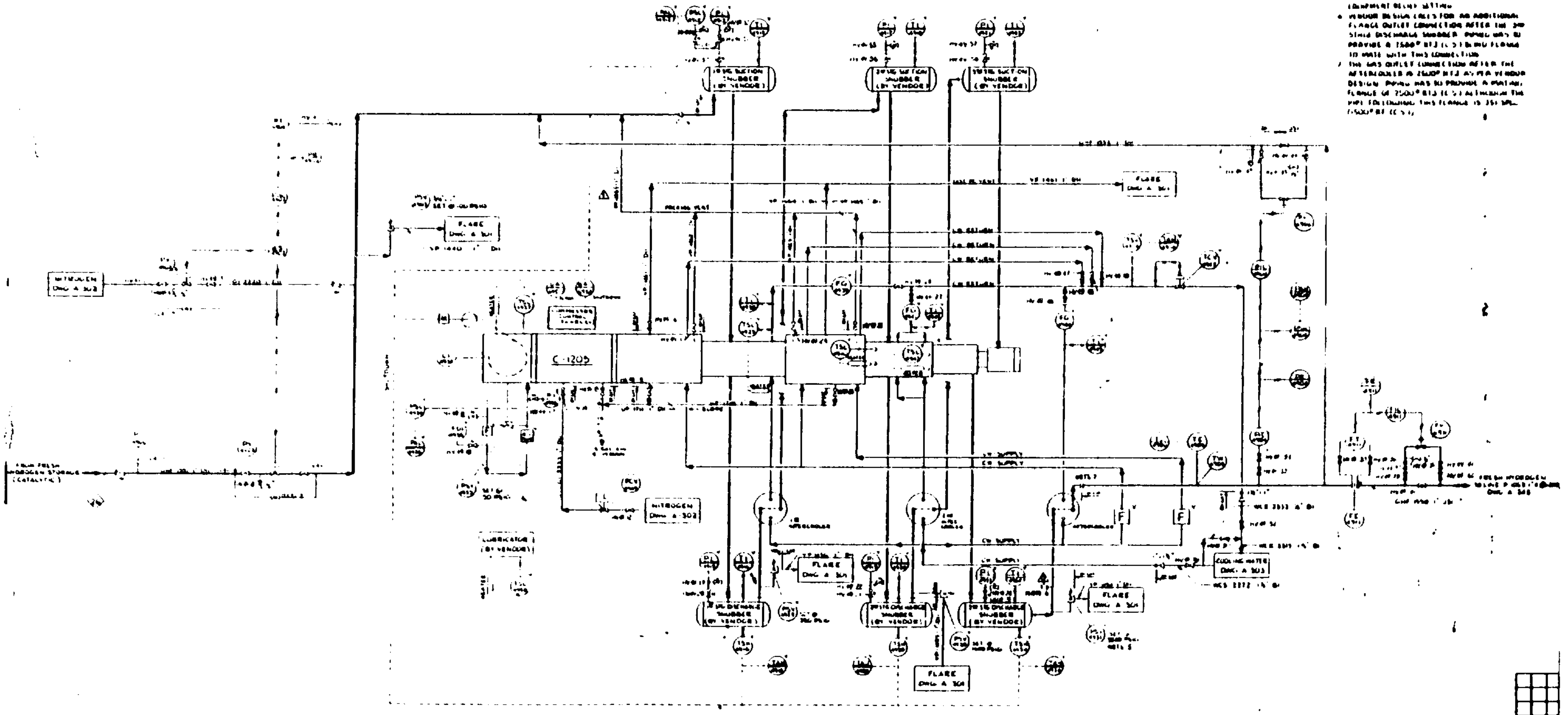

DETAIL #2
TUNGSTEN CARBIDE INSERTS
2-REQ'D
DETAIL #1
END CAP
1"-2500# RTJ WELDNECK FLANGE STAIN STL SCH 160 F316
.815"
COUNTER-BORE .1375" O.D. x 2 3/4" DEEP FOR TUNGSTEN CARBIDE INSERTS
DRILL 1 1/8" HOLE IN TOP OF CAP FOR FLANGE ATTACHMENT
3"-SCH 160 PIPE STAIN STL TP 316
3"x 1 1/2" SCH 160 WELDOLET STAIN STL F 316
WELD
WELD
WELD
WELD
DRILL 1 1/8" HOLE FOR OUTLET
8 1/2"
3" SCH 160 PIPE CAP BW STAIN STL F316
3" SCH 160 PIPE CAP BW STAIN STL F 316
SEE DETAIL #1
INLET
SEE DETAIL #2 FOR INSERTS
6"
28"
℄ OUTLET NOZZLE
2 1/2"
49 11/16"

Figure 4



NOTES

1. FOR GENERAL NOTES REF DWG'S LEGEND ETC. SEE DWG. A 336
2. LINE MARKED WITH SYMBOL REPRESENTS DIRECT TIE-IN WITH EXISTING [illegible] SERVICE LINE
3. ALL INTERNAL PIPING & INSTRUMENTATION FOR OIL LUBRICATING PROCESS & COOLING WATER SYSTEMS ARE SUPPLIED BY VENDOR
4. ALL PIPING & INSTRUMENTATION FOR NITROGEN VENTS & BLOWDOWN ARE BY CATALYTIC
5. VENDOR SUPPLIED PSV [illegible] IS INITIALLY SET AT 2500 PSIG. [illegible] MUST RESET THIS SAFETY VALVE TO [illegible] PSIG PRIOR TO START UP IN ORDER TO CONFORM TO DOWNSTREAM EQUIPMENT DESIGN SETTING
6. VENDOR DESIGN CALLS FOR AN ADDITIONAL FLANGE OUTLET CONNECTION AFTER THE 3RD STAGE DISCHARGE SNUBBER. PIPING HAS TO PROVIDE A 1500# RTJ (C.S.) BLIND FLANGE TO MATE WITH THIS CONNECTION
7. THE GAS OUTLET CONNECTION AFTER THE AFTERCOOLER IS 2500# RTJ AS PER VENDOR DESIGN. PIPING HAS TO PROVIDE A MATING FLANGE OF 2500# RTJ (C.S.) ALTHOUGH THE PIPE FOLLOWING THIS FLANGE IS [illegible] 1500# RF (C.S.)

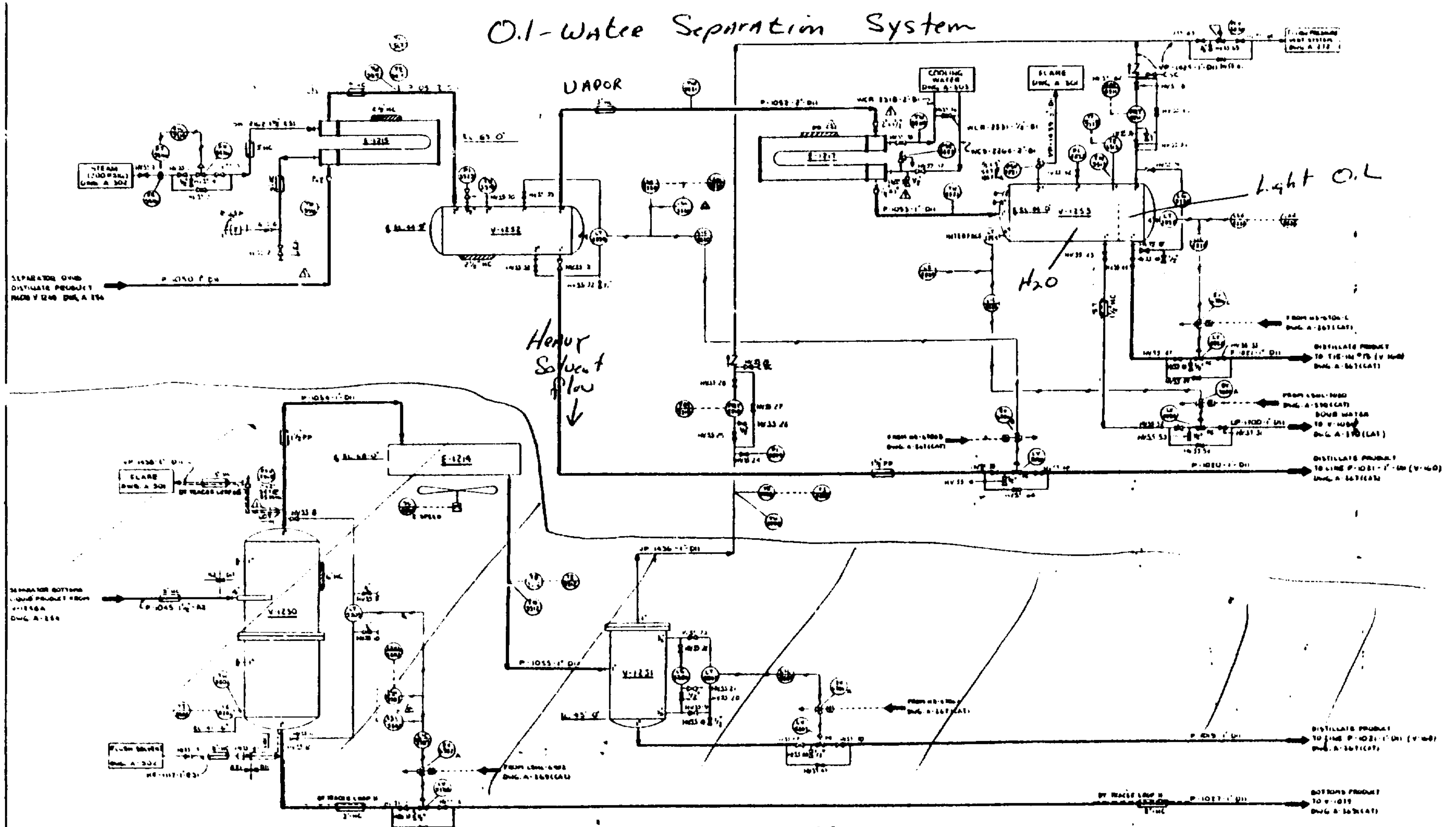

Oil-Water Separation System

FIGURE 5

Figure 6

Document No. DOE/PC/50041-18
Distribution Category UC-90d

THE WILSONVILLE
ADVANCED COAL LIQUEFACTION
RESEARCH AND DEVELOPMENT FACILITY

Wilsonville, Alabama

TOPICAL REPORT NO. 8

SOLVENT ACTIVITY STUDIES

Published: July 1983

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ABSTRACT

This topical report presents data and results relative to the role of recycle solvent in the SRC process at the Advanced Coal Liquefaction R & D facility at Wilsonville, Alabama. Data gathered over a period of two years is presented in a convenient format and conclusions based upon this data are summarized. Among the various aspects of the topic discussed in this report are the following: impact of solvent on process performance, achievement of steady state over extended recycle operation, control of solvent activity and the interrelationships among various solvent properties. Finally, a general discussion on the current perception, vis-a-vis the role of solvent in coal liquefaction reaction, has been included.

I. INTRODUCTION

Direct coal liquefaction is the conversion of coal to lower molecular weight products and occurs when coal and hydrogen are reacted at an elevated pressure and temperature in the presence of a hydrogen donor (H-donor) solvent. The ultimate goals of all liquefaction processes are to increase the H/C atomic ratio and remove heteroatoms and inorganic mineral matter(Ref. 1).

It is now generally accepted that the hydrogen donor activity of the solvent plays a very important role in the process of coal conversion. Coal conversion can be envisioned to occur in three stages, i.e., dissolution, hydrogen transfer, and rehydrogenation. In each of these steps, the nature of the solvent can affect the rate of reaction, the type of reaction, and the selectivity of the products(Ref. 2).

This report presents data and results relative to the role of recycle solvent in the SRC process gathered at the Wilsonville Advanced Coal Liquefaction R & D facility. During a two year period, Kentucky 9 and Illinois 6 coals were processed at the Wilsonville facility and the solvent was analyzed. The objective of this report is to present all available data in a convenient format and to summarize findings and conclusions.

II. PROCESS DESCRIPTION

In the solvent refined coal (SRC-I) process, pulverized coal is slurried with process-derived solvent. Feed gas is added to the slurry and the mixture is heated and fed to the dissolver. Effluent from the dissolver is flashed and the gases are separated and scrubbed to remove hydrogen sulfide and carbon dioxide. The scrubbed recycle gas is mixed with fresh makeup hydrogen to maintain 85 mole percent hydrogen in the feed gas.

The flashed dissolver effluent underflow, containing ash and undissolved coal solids, is then processed by Critical Solvent Deashing (CSD) to remove the ash and unreacted coal.

Before being fed to the CSD unit, the SRC slurry is distilled to separate the solvent. The slurry is then extracted with a deashing solvent (DAS) at conditions close to the critical temperature and pressure of that solvent. Heavy ash concentrate, SRC, and light SRC (LSRC) are separated and collected, and the DAS is recovered for recycle. The LSRC can be recycled to the front end as a component of the process solvent feed in proportions varying from 5% to 30%.

At the SRC unit, the recovered liquid is separated by fractional distillation. The liquid fraction boiling above 450°F is termed "distillate" solvent. This solvent is the major component of the recycled process solvent that is used as feed to the SRC unit. The other component of the process solvent is LSRC (5% to 30%).

In 1981, a catalytic hydrotreater began operation at Wilsonville to allow further SRC upgrading. Following the successful start-up of the unit, the facility was operated in a non-integrated two-stage liquefaction (NTSL) mode. In this mode, the thermal coal dissolver (SRC unit), the Critical Solvent Deashing (CSD unit), and the catalytic hydrotreater (HTR unit) are combined sequentially without any recycle of the hydrotreater products to the first stage (SRC and CSD units).

Figure 1 shows the NTSL process in a schematic form. Figures 2, 3, and 4 are simplified process flow diagrams for the three units.

In this report, the role of process solvent relative to the operation and performance of the SRC and CSD units is analyzed.

III. THE ROLE OF PROCESS SOLVENT - CURRENT PERCEPTION

The activity of the process solvent is known to have a significant impact on the coal liquefaction process. However, in spite of extensive research in recent years, the precise role of solvent in coal liquefaction reaction mechanism is not yet fully understood. This is not surprising since the reactions involved are numerous, simultaneous, and quite complex. Nevertheless, in recent years, a broad conceptual picture has emerged.

Coal is a heterogeneous organic rock interspersed with inorganic minerals. The organic portion of the coal is a mixture of distinct rock types known as macerals. The most predominant maceral is vitrinite. The vitrinite portion of coal is a highly cross-linked polymer consisting of a number of stable cluster units tied together by a network of weak links. These links include aliphatic carbon, ethers, weak hydrogen bonds, and charge-transfer complexes. During the dissolution step, the thermal energy at reaction temperatures (600°F to 850°F) is sufficient to rupture these links and break up the large macromolecule into a number of smaller, but highly reactive, free radicals. This transition occurs within the preheater tubes in a very short period of time. The free radicals that are formed have a very short half-life and they tend to get stabilized by various reactions. If donatable hydrogen is available in sufficient quantity, these free radicals are "capped" or stabilized by hydrogen and stable, low molecular weight, species are formed. If there is a deficiency of donatable hydrogen, then the probability of free radical stabilization via condensation reactions (autostabilization) increases. This results in high molecular weight species also known as char or coke. These free radical reactions take place in liquid phase. Thus the solvent, which is in liquid phase and which contains chemical species with donatable hydrogen, dictates the reaction path taken by the reactive free radicals.(Ref. 1)

It is now generally accepted(Ref. 1, 2) that there are four groups of chemical species in the solvent that affect the overall liquefaction process. These are: H-donors, H-shuttlers, phenols (heavy and light), and H-abstractors. The activity or "effectiveness" of a particular solvent depends on the relative and absolute concentration of these chemical groups. In the following sections, the role of each group is reviewed.

- H-Donors: (Ref. 1, 2)

  In general, almost all of the chemical species in solvent are capable of donating hydrogen under the appropriate set of reaction conditions. However, some do so more readily and without adverse effects. It is these species that are normally referred to as H-Donors.

The primary effect of thermal fragmentation in the preheater is the creation of hydrogen demand by reactive free radicals. This demand has to be satisfied and the free radicals will tend to pick up hydrogen from any available source. The source for hydrogen is a function of the demand, and generally follows a hierarchial mechanism with the most active H-donors receiving preference. Conventional hydroaromatic compounds such as dihydrophenanthrene and tetralin are the first to provide hydrogen. This mechanism is the preferred one since the resulting aromatic compounds can be rehydrogenated - thus preventing solvent degradation. However, if the hydroaromatics cannot fully satisfy the hydrogen demand, then relatively poor H-donors, such as, p-cresol become donors. In the case of these compounds, they too become reactive and tend to get incorporated into the product SRC matrix. (This increases the product oxygen content and produces solvent deficiency.) It is now well established that a high concentration of certain hydroaromatic compounds not only enhances conversion, both at short and long contact times, but also improve the product distribution and hydrogen utilization efficiencies(Ref. 2). The absence of hydroaromatic concentration does not preclude coal conversion, but the products may contain repolymerized solids or coke and the reaction can lead to increasing incorporation of solvent species into SRC products.

- H-Shuttlers:(Ref. 1, 2)

Although H-donors are a major controlling species in coal liquefaction, they are not the sole factor. Coal and SRC can also be a source of hydrogen. Bench-scale experiments have proven that significant levels of coal can be converted to pyridine soluble products in total absence of hydrogen gas or hydroaromatic compounds, but in the presence of non-donor solvents such as naphthalene and pyrene(Ref. 3). The apparent mechanism for this is an H-shuttling phenomenon whereby hydrogen atoms from hydrogen rich portions of the coal structure are redistributed to free radicals via the reversible hydrogenation/dehydrogenation of the H-shuttlers. This process has its limitations because the small quantity of available hydrogen within coal is rapidly consumed. This results in hydrogen transfer occurring at the expense of coke formation. Several studies have shown that hydrogen shuttling is more efficient for high-boiling polycondensed aromatic ring systems (Ref. 2). In situations where the concentration of H-Donors in the solvent is limited, H-donors and H-shuttlers can act synergistically to yield an effective solvent. This synergism, demonstrated on a bench-scale, may be important for low-severity runs with high LSRC recycle.

- Phenols: (Ref. 2)

  Phenolic compounds constitute a major portion of the steady-state recycle solvent in SRC process (20 to 30%) (Ref. 2). Both light and heavy phenols contribute substantially to the physical solvation properties of the solvent. Even highly polar coal products can, therefore, dissolve in the liquid phase. Phenols can also donate hydrogen through alkylation reactions especially if the hydroaromatic concentration is low. However, in this case, phenols get consumed by becoming incorporated in the SRC product. Reactions with heavy and light phenols are similar. However, in the case of heavy phenols, hydrogen can be donated via dehydrogenation in addition to alkylation. Some reactions involving heavy phenols can lead to coke formation (Ref. 2).

- H-Abstractors: (Ref. 1, 2)

  High concentrations of H-donors generally produce higher quality of SRC (with higher hydrogen, lower oxygen, sulfur, and aromatic carbon). However, if some solvent components consume hydrogen, then the overall amount of hydrogen available for reaction with coal is reduced. Benzophenone is such a component. Since coal or SRC can function as H-donors, any H-abstractor in solvent can lower the quality of the SRC. In addition, H-abstractors promote coke formation from SRC components or even solvent components, and lower the observed conversion.

One major problem of all liquefaction processes employing hydrogen donor solvents is the recycling of the solvent. In order to achieve a stable steady-state, it is necessary to provide a means of replenishing the H-donor capacity of the process solvent. During the reaction, solvent activity declines due to conversion of hydroaromatics to condensed aromatics. If these hydroaromatics do not get replenished, deleterious side reactions such as phenol condensation, dimerization, and coke formation occur. Stable solvent composition is achieved by hydrogen transfer from the gas phase to condensed aromatics and the subsequent production of new hydroaromatics from coal. Rehydrogenation reactions are generally slow and are presumably helped by catalytic action of coal minerals (such as pyrites) and hydrogen partial pressure. In addition to the desired rehydrogenation reactions, solvent is subject to several deleterious side reactions such as isomerization, cracking, polymerization, and adduction(Ref. 9). These reactions can cause an irreversible reduction in solvent activity.

From the above discussion on the role of process solvent in coal liquefaction processes, it becomes obvious that monitoring and controlling the solvent activity is crucial for process stability. It is also clear that a meaningful characterization of solvent activity can be very difficult and involves time consuming and sophisticated analytical methods. At Wilsonville, solvent activity is monitored by microautoclave tests (described in Section IV). In addition, several physical properties such as specific gravity, viscosity, boiling ranges, and elemental analysis are routinely monitored. These tests have been useful in monitoring the process operations. However, for the purposes of data correlation and quantitative analysis, more comprehensive testing procedures may have to be developed.

IV. DISCUSSION OF RESULTS

This report analyzes solvent property data monitored at Wilsonville from 1 January 1980 (Run 195) to 6 March 1982 (Run 236). During most of this period (1980 and 1981), high sulfur Kentucky 9 coal from Fies mine was processed. During 1982, the feed coal was changed to Illinois 6 coal from the Burning Star mine. Tables 1 and 2 summarize the reported distillate and process solvent properties for all material balance runs during the report period. The table contains the following data:

- LSRC concentration in Process Solvent
- Short and long microautoclave conversion data
- Specific gravity
- Elemental analysis
- Boiling ranges

Distillate solvent is a process-derived fraction with a boiling range between 450°F at 760 mm Hg and 600°F at 0.1 mm Hg. The distillate solvent is blended with varying quantities (from 5 to 30%) of LSRC to form the recycle process solvent that is actually mixed with the feed coal.

All runs listed in Tables 1 and 2 were conducted in either the single stage or non-integrated two-stage mode. The single stage mode involves the conversion of coal in the thermal dissolver followed by the separation of ash in the CSD unit. The non-integrated two-stage mode involves further conversion of the deashed SRC in the catalytic HTR unit. In both these modes, hydrotreated product concentration in the process solvent is zero. It must be pointed out that for runs with a low concentration of LSRC in the process solvent, the physical properties of the distillate and process solvents are very similar. However, for runs with high concentration of LSRC (30%), distillate and process solvents are quite dissimilar. Generally, low severity operations, with dissolver at 2,000 psig and between 750 and 785°F, use process solvents with a high LSRC concentration. High severity operations, with dissolver at 2,100 psig and between 825 and 840°F, use process solvents with a low LSRC concentration. (See Section 3 below.) Details of the operating conditions and results for each material balance and the analyses of the feed coal are summarized in Tables 3 and 4, respectively.

Figures 5, 6, and 7 plot the variations in solvent activity, LSRC addition rate, hydrogen content, and specific gravity over the report period. Pertinent run conditions and instances of anthracene oil additions are also shown on the plots. For periods of high LSRC addition, distillate solvent properties were used in the plots. During periods of low LSRC addition, properties of process solvent were sometimes used in lieu of distillate solvent properties if the former was unavailable. As mentioned earlier, during periods of low LSRC recycle (≃5%), the properties of both distillate and process solvent are similar and can be used interchangeably.

1. Solvent Activity Measurement

Because of the very complex nature of interaction between solvent and coal at reaction conditions, effective characterization of solvent activity can involve a combination of several sophisticated analytical tests. At Wilsonville, a relatively simple test, using a microautoclave, has been developed to monitor solvent activity. It is recognized that this empirical test, conducted in the absence of hydrogen gas, does not completely define the solvent characteristics. Nevertheless, it has been found to be useful for routine process monitoring. In this test, a "standard coal" is reacted with the solvent sample at standard conditions in a 30 cc laboratory microautoclave.

The microautoclave apparatus is illustrated in Figures 8 and 9. Equipment specifications are as follows:

Reactor dimensions	
Volume, ml	30
Inside diameter, in.	5/8
Outside diameter, in.	7/8
Height, in.	6 (working)
Material	316 SS
Mixing ball in reactor	
Number	1
Size, in.	3/8
Material	316 SS
Reactor orientation	Vertical
Agitation, strokes/minute	1,000
Stroke length, in.	1½
Standard coal	Indiana V (Old Ben), supplied by Conoco

The tetrahydrofuran (THF) conversion achieved in this test is assumed to be an indication of solvent activity. Standard conditions for the test are given below:

Test	Temp, °F	Solvent to coal ratio	Reaction time, min.
Short (kinetic)	750	8:1	10
Long (Equilibrium)	750	2:1	30

The short (kinetic) test gives an indication of the hydrogen transfer rate and hydrogen shuttling ability of the solvent. The long (equilibrium) test gives an indication of the concentration of donatable hydrogen in solvent. For the SRC process or distillate solvent, short test conversions are usually higher than long test conversion. This is due to either the higher solvent ratio used for the short test or repolymerization of free radicals in the hydrogen-donor limiting environment of the long test. It is interesting to note that for hydrotreated solvent, which presumably has a higher concentration of donor hydrogen, the long test conversions are consistently higher than the short test conversions.

A variant of the above test can be used to measure feed coal reactivity. In this test, a standard (synthetic) solvent composed of 25% tetralin in 1-methylnaphthalene is used to measure the coal conversion at standard reactor conditions. This is useful in monitoring the relative activity of the feed coal.

One problem associated with the microautoclave tests is the use of a "standard" coal. Coal is known to be heterogeneous and can vary in composition within a given mine. This means that the reactivity of the "standard" coal can vary significantly from batch to batch. To avoid this problem, Wilsonville ordered a fairly large stock of standard coal during 1978 with the hope that the same batch of coal would be available for many years. The Indiana V standard coal was packaged in two pound quantities in sealed plastic containers. These were stored in a metal container under ambient conditions. This strategy gave rise to its own problems. Once a sample of coal is mined, crushed, and stored, its properties can change with time - presumably by air oxidation and/or ambient temperature cycling. The following table illustrates the change in reactivity of standard coal with time.

Date	Storage duration, months	Standard coal reactivity(a) Short(b)	Standard coal reactivity(a) Long(c)
June 1978	0	70	78
Nov 1978	6	68	75
May 1982	48	62	69

(a) % THF conversion in microautoclave using 25% tetralin in 1-methylnaphthalene.
(b) Solvent/coal ratio - 8/1; reaction time = 10 minutes.
(c) Solvent/coal ratio - 2/1; reaction time = 30 minutes.

The data is plotted in Figure 10. This figure shows that standard coal activity by both the short and long tests declined steadily. During the period from January 1980 to March 1982 (Runs 195 to 236), there was a 3.5% and 2.5% drop in coal reactivity by the short and long tests, respectively. This means that, for any long term trend analysis, a correction of 1.0 to 1.5% per year must be considered. This steady drop in activity of the standard coal does not affect any short term trend conclusions since the order of magnitude of experimental error is ±2%.

2. Coking Reactions and Solvent Activity

Coking in liquefaction plants has been a matter of concern in existing pilot facilities as well as in the design of commercial size units. As explained in Section III, the fundamental reasons for coke formation are: (a) excessive demand for hydrogen by free radical coal fragments and/or (b) inability of the solvent to donate sufficient quantity of hydrogen to satisfy the demand. Factors that control the basic demand for hydrogen are: heat flux in the preheater tubes, reaction temperature, and the type of coal. In general, higher fluxes and reaction temperatures increase the rate of thermal decomposition which, in turn, increases the demand for hydrogen. Different types of coal have reactive macerals with different chemical structure. Hence, one can expect that different coals will produce different levels of hydrogen demand even if the preheater flux and the dissolver temperature are held constant. Factors that control the availability of hydrogen to satisfy this demand are: solvent activity (or effectiveness), hydrogen partial pressure, total reactor pressure, and the solvent to coal (S/C) ratio. Hence, adverse coking reactions can be eliminated or, at least, minimized by:

Controlling the hydrogen demand by

- reducing the preheater flux,
- reducing the reactor severity, and
- proper selection of feed coals.

Improving the availability of hydrogen by

- increasing the total pressure,
- increasing the hydrogen partial pressure,
- improving the solvent activity.

The SRC liquefaction process has been undergoing a gradual evolution over a period of eight years at Wilsonville. It is fair to say that, in recent years, preheater and dissolver coking has been less of a problem than it was during the earlier operating periods. In fact, during the period from 1 January 1980 (Run 195) to 30 March 1982 (Run 236), there was no instance of an involuntary plant shutdown solely due to coke formation in the preheater or dissolver. Operations at lower dissolver severity, higher dissolver pressures (2,000 psi instead of 1,400 to 1,700 psi), use of dissolver solids withdrawal system, limited use of solvent activity control, use of coals that are less susceptible to coking, such as Kentucky 9 and Illinois 6 (Burning Star), and generally low heat flux in the fired heater (2,500 to 3,00 Btu/hr-ft^2) could have contributed to this excellent record. Notwithstanding this recent performance record, coking phenomenon is still considered a critical issue in eventual design of a commercial plant.

It has been known for some time that the economic feasibility of the process on a commercial scale depends, to a large extent, on an optimal heat recovery system and equipment sizes. There is a clear economic incentive to increase the preheater flux (7,000 to 10,000 Btu/hr-ft^2) and reduce the quantity of recycle gas at the heater inlet. Both improvements in design imply higher probability of coke formation. This tendency to form coke can be alleviated by maintaining high solvent activity. In this section, Wilsonville data related to solvent activity and coke formation is presented.

During early operating period (Runs 135 to 156) when Indiana V and Kentucky 6 and 11 coals were processed, a definite correlation could be seen between coke formation and solvent activity. The following data summarizes pertinent data from these runs(Ref. 4).

Coal	Indiana V			Kentucky 6 and 11		
Run	135	137	140	150/151	155	156
Dissolver conditions						
Space rate, lb/hr-ft^3	47	52	47	47	50	25
H_2 partial press, psi	1,440	730	1,390	1,140/1,450	950	1,200
Solvent activity						
Short, %	76	72	77	64	63	67
Long, %	76	67	71	63	62	69
Coke deposition						
Preheater	No	Yes	No	Yes	Yes	No
Dissolver distributor, lb	<1	15	6	15	15	<1

From the foregoing table it can be seen that when the solvent activity was low (less than 65) and/or the hydrogen partial pressure was low (less than 1,000 psi), coke was deposited in both the preheater tube and the dissolver distributor.

During the above-mentioned period, there were several runs with decreasing solvent activity, but no apparent coke deposition. These are: Runs 129, 136, 143 (Indiana V), and Run 149 (Kentucky 6 and 11). Four batches of representative distillate solvent from these runs were sent to Conoco for use in their Short Residence Time process development program. These batches are identified along with sampling dates in the following table(Ref. 5).

Solvent batch	Date sampled	Coal type	Run no.
I	September, 1977	Indiana V	129
III	April, 1978	Indiana V	136
IV	August, 1978	Indiana V	143
VI	October, 1978	Kentucky 6 and 11	149

All the samples were fairly large (approximately 5 drums) and were taken from the solvent inventory. During the period over which these samples were taken, no anthracene oil was added to the system. Data from these samples provide an excellent basis for describing "good" and "bad" solvents (Ref. 5).

The properties of these solvents were determined by Conoco (Ref. 5) and are summarized in the table below. Note that the solvent microautoclave activity as measured by Conoco cannot be directly compared with the solvent microautoclave activity measured at Wilsonville.

Solvent batch	H, %	Solvent activity, %		Aromatics, %		
		Short	Long	Condensed	Uncondensed	R_{AR} (a)
I	8.0	81.4	76.5	2.15	0.9	2.39
III	9.0	76.5	74.4	1.31	0.95	1.37
IV	8.8	77.4	73.7	1.27	1.09	1.17
VI	8.9	75.4	67.4	1.21	1.09	1.11

(a) $R_{AR} = \frac{\text{Condensed aromatics}}{\text{Uncondense aromatics}}$

The above table shows that in terms of microautoclave tests, Batch 1 solvent was significantly more active than Batch VI solvent. The microautoclave conversion data was reported to correlate well with the ratio of condensed aromatic to uncondensed aromatic. The condensed aromatics concentration seems to improve the hydrogen donor capacity(Ref. 5).

All the solvent batches were used by Conoco in a bench-scale Short Residence Time (SRT) extraction unit. They found a strong relationship between solvent activity, as determined by microautoclave, and process operability. "Batch I solvents (good activity) were all operable in fourteen runs in the SRC unit, in the sense that no problems with reactor plugging (coking) caused premature termination of any run. Four attempts were made to operate the SRT unit with Batch III solvent, but all failed because of plugging..."(Ref. 5) They could operate the unit with Batch III and Batch IV solvent only after addition of hydrogen gas (increasing hydrogen partial pressure) or by prehydrogenation of the solvent over catalyst (improving solvent activity).

Another instance of process instability and coking due to "poor" solvent was experienced during the early days of SRC process development (1974/1975) with Illinois 6 (Monterey) coal. From December 6, 1974, to December 23, 1974, (Runs 16, 17, and 18), the plant operated relatively smoothly with coal conversions of 90 to 93%. However, during April 13 to May 5, 1975, the plant performance was very "erratic" with coal conversions fluctuating between 75% to 85%. Representative samples of distillate solvent from these runs were sent to Mobil Research and Development Co. for analysis. During this period, microautoclave method for characterizing solvent activity was not yet developed at Wilsonville. The following table summarizes the results of Mobil's analysis(Ref. 2):

	Estimated available hydrogen content (gH/100 g solvent)				
Run	Hydroaromatics	Heavy phenols	Total	Reported conversion	Comments
17	0.24	0.27	0.51	95	Stable process performance
27	0.13	0.40	0.53	75-85	Erratic process performance

The table shows that the total hydrogen donor concentrations were similar for both the runs, but the "good" solvent contained significantly more hydroaromatics and the "poor" solvent contained more heavy phenols. The Mobil data indicated that the low apparent conversion (and erratic process performance) was due to solvent coking which resulted from H-donation from heavy phenols. The chemistry of the heavy phenolic component of recycle solvent is not well understood. Heavy phenols having more than 9 carbons have the potential of hydroaromatic structure and could thus function as hydrogen donors. The more functional heavy phenols can also form coke via condensation reactions. Since coke formed during the reaction cannot be distinguished from unconverted carbon, low apparent conversion can be the result.

From the above discussion, one can conclude that there are several factors that can lead to coke formation in the liquefaction reaction. Coke formation can be minimized by maintaining high process solvent activity. This fact becomes more critical if other design and operating features tend to increase the likelihood of coke formation. For optimal design of commercial units, maintenance of high solvent activity can be very important.

3. Solvent Activity and Process Performance

Most of the runs during the report period can be classified into "high" and "low" severity runs.

The high severity runs were carried out at dissolver temperatures of 825 to 840°F and 2,100 psig pressure with process solvent containing about 5% LSRC. This process solvent has properties similar to the distillate solvent since the LSRC concentration is quite low.

The low severity runs were carried out at 750 to 785°F and 2,000 psig pressure with process solvent containing as much as 30% LSRC. In this case, the process solvent is significantly heavier and more active by the kinetic test.

The available material balance data on high and low severity runs at relatively constant dissolver conditions and constant feed coal are summarized below:

	High severity	Low severity
Coal type	Ky 9 (Fies)	Ky 9 (Fies)/Ill. 6[a]
Reactor temperature, °F	840	785
Reactor pressure, psig	2,100	2,000
Coal space rate, lb/hr-ft^3	40	35/18[b]
Process solvent/coal ratio	1.6/1	2/1
LSRC rate, % process solvent	5	30
Runs	209, 210, 219, 220, 225, 227, and 235	217, 221, 222, 232, 233, 234, and 236
No. of Material Balance data points	17	14

(a) Illinois 6 (Burning Star) coal was used for Run 236.
(b) Space rate for Runs 221 and 222 was 18 lb/hr-ft^3.

Details of solvent properties, coal properties, run conditions, and results for all of the above listed runs are given in Tables 1 through 4.

The low severity runs using a high LSRC recycle rate were based in large part on the bench work done by Conoco(Ref. 5). This work clearly showed that presence of LSRC in process solvent markedly changed the product distribution, favoring significantly higher distillate yields than without LSRC in recycle solvents. Liquefaction temperatures of 750 to 800°F were normally sufficient to generate enough distillate and LSRC for the plant to be in balance. This was not the case when only distillate solvent was recycled at the same reactor conditions(Ref. 6). These experiments at Conoco clearly demonstrated that the nature of the solvent was crucial in determining the yield structure (and, consequently, the efficiency of hydrogen utilization). Subsequent tests at

Wilsonville confirmed the Conoco findings. The following table compares the results for Run 210AB and Run 217AB along with the pertinent coal properties.

Run no.	210AB	217AB
Severity	High	Low
Solvent activity (kin), %		
Distillate	69	79
Process	~69	82
Yields, % MAF coal		
C_1-C_5	7	3
Distillate	26.1	28.9
SRC	52	57
Hydrogen consumption, % MAF coal	2.3	2.0
Coal analysis, %		
Carbon	71.99	72.42
Hydrogen	5.03	4.99
Total sulfur	3.18	3.29
Pyritic sulfur	1.14	1.42
Sulfide sulfur	0.02	<0.01
Sulfate sulfur	0.06	0.01
Ash	8.4	8.9
Coal reactivity, %	81.0	75.7

Run 210AB was a high severity run with relatively low process/distillate solvent activity. Run 217AB was a low severity run with relatively high process and distillate solvent activity. The high activity of solvent in Run 217AB was due to LSRC recycle and the addition of anthracene oil during previous Run 214. The above data indicate that it is possible to increase or maintain distillate yield concurrent with a significant reduction of C_1-C_5 gas yield simply by manipulating the nature of the process solvent (LSRC addition) and lowering the dissolver temperature.

Another run which illustrates the effects of LSRC recycle on yield structure is Run 228. This was a high severity run with dissolver temperature of 825°F. When LSRC rate was reduced from 0.26 lb/lb MAF coal (20 to 25 May) to 0.12 lb/lb MAF coal (27 to 31 May), the distillate solvent yield decreased from 24.0 to 14.5% MAF coal. The results are summarized on the following page along with sulfur and ash analysis in coal.

Run no.	228	
R101 Dissolver		
Temperature, °F	825	
Pressure, psig	2,100	
Volume used, %	75	
Period, 1981	20 to 25 May	27 to 31 May
LSRC rate, lb/lb MAF coal	0.26	0.12
Distillate solvent activity (kin.), %	77	76
Feed coal properties, %		
Sulfur	2.89	3.15
Ash	8.00	7.95
Yield, % MAF coal		
Total gas	7.9	11.4[a]
Distillate solvent	24.0	14.5[a]

[a] From 228AB material balance.

Although the above data illustrate the improvement in yield structure that can be achieved by recycling varying amounts of LSRC, there is enough evidence to show that, at constant dissolver conditions and LSRC rate, solvent activity may be playing some role in determining the process performance.

- High Severity Runs

  After completion of Run 217, which attained a high distillate solvent activity, the plant was operated (Run 219) at high severity conditions duplicating Run 210. The only difference between Run 219 and Run 210 was the distillate solvent quality. It was "high" for Run 219 (78%) and "low" for Run 210 (69%). Pertinent properties of coal and the results of these two runs are summarized on the following page for comparison:

Run	210AB	219AB
Severity	High	High
Solvent activity (kin), %		
Distillate	-	-
Process	69	78
Yields, % MAF coal		
C_1-C_5	6.7	4.0
Distillate	26.1	28.2
SRC	51.8	58.6
Hydrogen consumption, % MAF coal	2.3	2.6
SRC analysis, %		
Oils	10.3	25.3
Asphaltene	43.5	55.6
Preasphaltene	46.2	19.1
$\frac{\text{Oils + Asphaltene}}{\text{Preasphaltene}}$	1.2	4.2
SRC recovery, % cresol solubles in T102 bottoms	80.0	86.0
Energy rejected to ash concentrate, % of feed coal energy	22.6	17.6
Coal analysis, %		
Carbon	71.99	71.30
Hydrogen	5.03	4.66
Total sulfur	3.18	3.38
Pyritic sulfur	1.14	1.37
Sulfide sulfur	0.02	0.01
Sulfate sulfur	0.06	0.06
Ash	8.40	8.73
Coal reactivity, %	81.0	82.4

Among the key differences in results from Runs 210 and 219, which differ primarily in the activity of the distillate solvent were the higher (oil + asphaltene)/preasphaltene ratio in SRC from the SRC unit for Run 219. This presumably improves the performance of the CSD unit as shown by the increased SRC recovery and reduced energy rejection.

Another set of runs which can be used to evaluate the impact of distillate solvent activity on process performance are Runs 225 and 235. Prior to Run 225, th entire solvent inventory was replaced by anthracene oil. The oil was prehydrogenated under mild conditions which caused the distillate solvent activity to be high for Run 225 (82%). This activity declined steadily over 8 to 9 months until it was 70% for Run 235. It is strongly suspected that the deteriorating coal properties (Table 4), as shown by increased sulfate content of feed coal during Runs 229 to 234, played a key role in the solvent degradation. (See Section 4.) Dissolver conditions for the two runs were almost identical. The following table compares the results from Run 225 and Run 235.

Run	225BC	235C
Severity	High	High
Solvent activity (kin), %		
Distillate	82	71
Process	-	-
Yields, % MAF coal		
C_1-C_5	4.2	6.9
Distillate	30.4	26.5
SRC	55	55
Hydrogen consumption, % MAF coal	1.7	2.1
SRC analyses, %		
Asphaltene + oil	76	66
Preasphaltene	24	34
Energy rejected to ash concentrate, % of feed coal energy	18.5	22.3
Coal analyses, %		
Carbon	72.80	74.77
Hydrogen	4.98	5.15
Total sulfur	3.35	3.07
Pyritic sulfur	1.37	1.20
Sulfide sulfur	<0.01	0.01
Sulfate sulfur	0.04	<0.01
Ash	8.50	7.68
Coal reactivity, %	80.1	78.4

Comparing the yield structure for Run 225BC and 235C indicates that Run 235C had a slightly higher hydrocarbon gas make, and somewhat higher energy rejection to ash concentrate at the CSD unit. Although these differences, between Runs 210 and 219 or between 225 and 235 are not dramatic, they are all consistent and point toward a somewhat better process performance with higher distillate solvent activity.

- Low Severity Runs

Three sets of low severity runs at a constant coal space rate indicated the effect of solvent activity on process performance during runs with a high LSRC recycle rate (30% of process solvent). These runs are summarized on the following page.

Runs	216/217	233/234	236
Coal type	Ky 9 (Fies)	Ky 9 (Fies)	Ill. 6 (Burning Star)
Coal space rate, lb/hr-ft^3	35	30	30
Coal properties, % MF			
Ash	8.3	7.7	10.8
Total sulfur	3.3	2.7	3.4
Pyritic sulfur	1.4	0.8	1.2
Sulfate sulfur	0.06	0.2	<0.01
Reactivity, %	--	74	77
Solvent activity (kin), %			
Process	83	76(a)	80
Distillate	80	66(b)	73
LSRC in process solvent, %	29	31	30

(a) Average of two data points (76.3 and 75.3).
(b) MB average.

The typical yield structure for corresponding material balances are given below:

	217AB	234C	236A
Yields, % of MAF coal			
SRC	57	72	56
Distillate solvent	24	7	20
Total distillates	29	11	26
Total gas	5.6	7.1(a)	7.8(a)
Hydrogen consumption, % MAF coal	2.0	2.0(a)	1.6(a)

(a) By ethylene injection method.

Comparison of Runs 217 and 234 indicated a significantly higher distillate production for Run 217. This higher distillate make for Run 217 cannot be fully attributed to higher solvent activity for Run 217 since the properties of coal were different. Kentucky 9 Fies Coal for Run 217 had a low sulfate and a high pyrite sulfur content compared to the coal for Run 243 even though they came from the same mine. These differences could have been caused by aging of coal during storage. Feed coal for Run 243 had been stored for approximately six months at ambient conditions before it was processed in the plant. In contrast, the feed coal processed during Run 217 was stored at ambient conditions for only one month prior to being processed. The higher oxidation level of the feed coal during Run 243 is reflected by the higher level of sulfate content and lower level of pyrite content. It has been reported that even slight oxidation can decrease the coal reactivity. The oxidation process which involves cross-linking reactions causes a reduction in the plastic properties of coal (Ref. 7, 8). Aging of coal on air exposure has an effect on its conversion. Davies and his co-workers (Ref. 9) have reported that coal conversion of aged coal to THF solubles is significantly less than that from fresh coal. The mineral contents of coal and their effect on solvent activity is discussed in detail in Section 5 under "Feed Coal Specifications".

Comparison of Run 234C with Run 236A is quite interesting. Run 236A used a different coal (Illinois 6 Burning Star mine). Nevertheless, it was more reactive than the Ky 9 coal used during Run 234. The pyritic sulfur was quite high and sulfate content was low. However, distillate solvent activity during Run 236 was somewhat higher than that for Run 234. Run 236 had a distillate yield considerably higher than that of Run 234 even though distillate solvent balance was still maintained during Run 234. Apparently, interaction of LSRC with a reactive coal played a key role in the conversion to distillate range products at low severity conditions.

The performance of the CSD unit seemed to indicate that more reactive preasphaltenes were present in the CSD feed during Run 236 when compared to Runs 217 or 234. Distillate solvent activity appeared to be important not only for the interaction of LSRC with reactive coal, but also for the stabilization of reactive preasphaltenes downstream of the reactor in the absence of hydrogen gas.

4. Steady State Solvent

Figure 11 is a plot of distillate solvent activity as a function of on-stream time for three high severity runs (Runs 219/220; Run 225, and Run 235). This plot depicts the achievement of steady state properties of distillate solvent after continuous recycle over 30-60 days. Run 219/220 and Run 225 started with relatively active solvents. Run 219/220 followed Run 217 when the solvent activity was high due to LSRC recycle and prior addition of anthracene oil. Run 225 started with a mildly hydrogenated anthracene oil. In contrast to Runs 219/220 and 225, Run 235 started with a solvent of lower activity. This run followed Run 234 when the solvent activity declined steadily over a forty-day period. For all the three runs, steady state was achieved over a 30-60 day period. However, the steady state activity for Run 219/220 and Run 225 was higher (78%) than the steady state activity for Run 235 (71%). Since coal properties were approximately equivalent, the different steady states were apparently related to the nature of the starting solvent (Ref. 10).

Figure 12 is a plot of distillate and process solvent activities as a function of on-stream time for two low severity runs (Run 217 and Runs 230-234). Run 217 had high process and distillate solvent activities, but the duration of the run was not sufficient to know whether steady state was achieved. Runs 230-234 were approximately 70 days long. For the first

30 days, the solvent appeared to have reached a steady state activity (73%). However, after this initial period, a sharp decline was observed over the entire remaining run period. As mentioned earlier, the feed coal during this period had low pyrite and high sulfate content. This deterioration of the feed coal (presumably by air oxidation) probably caused the observed activity trend(Ref. 10).

5. Solvent Activity Control

From the discussion in the previous section, it is obvious that maintenance of adequate solvent activity is critical for good performance and stability. Although it is possible to start up the process with a relatively active solvent (such as mildly hydrogenated anthracene oil), there is no certainty that solvent activity can be maintained over an extended period of time. All indications point out that initial high solvent activity declines with time, presumably to some steady-state value. This value depends on the operating conditions, the type of coal and the nature of the starting solvent. At this time, available data is insufficient to predict the steady-state activity. In any case, it has been demonstrated that the activity can fall to levels where process performance and stability is adversely affected. It is inconceivable that external, high activity solvent can be continuously added to maintain the solvent activity for a large commercial plant. It is, therefore, very important to be able to maintain an adequate level of solvent activity by manipulating the process flow scheme and/or operating conditions of the various units within the process.

Prior to the installation of the CSD and HTR units, anthracene oil (Allied Chemical 24CB) was added to the system fairly often. Although this was done primarily to maintain adequate inventory, the activity of the solvent was improved after every addition. This method of improving solvent activity, although quite effective on a small scale, is not practical for commercial units. The installation of the CSD and the HTR units added a new dimension to the Wilsonville process. Streams external to the SRC unit, such as LSRC, hydrotreated SRC, and hydrotreated solvent are now available for control of process solvent activity in the thermal dissolver.

In this section, various techniques used at Wilsonville to control solvent activity will be discussed.

- Anthracene Oil Addition

  Anthracene oil is a highly aromatic coal derived start-up solvent used at Wilsonville. Table 5 lists some pertinent properties of this oil. Fresh anthracene oil is a poor hydrogen donor. But, upon mild hydrogenation in the presence of coal, this oil becomes an excellent hydrogen donor. During the initial hydrogenation of this oil, the aromatic components in the oil presumably get hydrogenated and become hydroaromatics.

During the period covered in this report (January 1980 to March 1982), anthracene oil was added to the system on four different occasions. This does not include an occasion when the entire inventory was replaced with anthracene oil (6 January 1981). On two of the occasions, the amount this oil added was very small in comparison to the total system inventory (31 March 1981 to 10 April 1981). The instances of anthracene oil addition and the quantity added are noted on the Figures 5 and 6 trend charts.

On 24 June 1980, 8,600 lb of anthracene oil was added to the system. This represented a 25.6% increase in inventory of distillate solvent. During this period, LSRC addition rate was held at 40 to 50 lb/hr - constituting less than 7% of the process solvent. This process solvent has properties similar to the distillate solvent since the LSRC concentration is quite low. Figure 13 shows the trend in process solvent activity from 22 to 26 June 1980. It shows that the solvent activity increased from 73% to 76% in about 16 days. This increase in activity was sustained for about three weeks. It is interesting to note the temporary decline in solvent activity (to 71%) immediately after the addition of anthracene oil. This dip is due to the fact that fresh anthracene oil, prior to hydrogenation, is in fact, a poor solvent.

- LSRC Addition

Addition of LSRC has a direct effect on the composition of the process solvent. Since LSRC is basically a light fraction of residual material, most of its components boil above 650°F. Hence, addition of LSRC makes the process solvent substantially heavier and more active. Since the residual material is separated by the vacuum column, the distillate solvent from runs with high LSRC addition are not significantly heavier, and are marginally more active.

Two different periods during 1980 to 1982 are used to illustrate the effect of LSRC addition. These periods are summarized below:

Period	Runs	LSRC, % process solvent (Run)		Net increase in solvent activity, (kinetic)	
		Before	After	Process solvent	Distillate solvent
Aug/Sept 1980	213-217[a]	0(214/215)	30(216)	11	7
Nov/Dec 1980	220-221[b]	6 (220)	26(221)	10	4

(a) Anthracene oil was added during Run 214. Dissolver severity was also reduced.
(b) Dissolver severity was also reduced.

Runs 213-217:

This period illustrates the combined effect of LSRC and anthracene oil addition. A trend chart for this period on an expanded scale is given in Figure 14. On 10 August 1980, 14,800 lb of anthracene oil (or 43.8% of inventory) was added to the system. After 15 days of operation, the plant was shut down from 26 August through 4 September 1980. The plant resumed operations on 4 September with Run 216. The dissolver severity was reduced and the LSRC rate was increased. The solvent activity during Runs 216 and 217 was influenced by both anthracene oil addition and by increased rate of LSRC recycle.

Prior to 10 August, the distillate solvent activity was about 73%. However, during Run 216, it was almost 80%. This substantial increase is attributed mainly to the anthracene oil. Process solvent activity during Runs 216 and 217 was about 84%. This represents a 11% increase over the distillate solvent activity prior to 10 August. The net increase in process solvent activity represents a combined effect of LSRC and anthracene oil additions.

Runs 220-221:

During Run 220 (high severity conditions), the average process (or distillate) solvent activity was 74% and the LSRC recycle rate was only 60 lb/hr. For Run 221 (low severity conditions), the LSRC recycle rate was increased to 200 lb/hr. During this period, and during more than two months prior to this period, no anthracene oil was added to the system. The increase in LSRC recycle caused the distillate solvent activity to increase to 78% during Run 221. The process solvent activity increased to 84%. A trend chart for this period is given in Figure 15.

From the above data, it can be concluded that, during low severity operation, addition of LSRC in sufficient quantity (up to 30%) significantly improves the process solvent activity and marginally improves the distillate solvent activity. The changes in both distillate and process solvent activities are almost immediate.

- Solvent Activity Control By Distillation

It has been observed that, for any given distillate solvent, the heavier fractions are more active than the lighter fractions. Data supporting this observation are discussed in the next section. Therefore, it should be possible to improve the quality of the solvent by fractional distillation. This method of controlling the solvent activity is quite practical for a commercial plant where fractionators can be installed to split the solvent into many cuts. These cuts, in turn, could be blended to meet a desired specification. This method is not practical at Wilsonville since the available vacuum tower (T102) was not designed to split the solvent into sharp fractions. At Wilsonville, the distillate solvent boiling range can be modified by withdrawing a portion of the liquid from a point (tray-3) above the main draw off point (tray-8). This causes the tray-8 material to become heavier. However, the stream from tray-3 still contains a substantial quantity of +650°F material. This inefficient draw off, over a period of time, can cause the system distillate solvent inventory to drop below levels necessary for plant operation - unless process conditions are such that significant excess amount of solvent is continuously produced. Hence, solvent activity control by withdrawing tray-3 material is not routinely employed.

During Run 236, tray-3 liquids were withdrawn from 12 through 17 February 1982. The total quantity of the withdrawn liquid amounted to 23% of the distillate solvent inventory. The effects of this action are illustrated in Figure 16. Prior to 12 February, distillate solvent contained 41% of the +650°F fraction and its activity was declining and averaged 70%. Process solvent during this period contained 55% of the +650°F fraction and its activity was 79%. After removal of the tray-3 liquids, distillate solvent activity increased to 74%. Tray-3 liquids were withdrawn for four days (12, 13, 15, and 17 February). Approximately two weeks after the last withdrawal, the distillate and process solvent activities declined to 72% and 77%, respectively.

- Addition of Hydrotreated SRC

Recent work at the University of Wyoming(Ref. 11) indicated that the presence of mildly hydrotreated

SRC in process solvent (up to 50%) significantly improved the yield structure in a batch reactor. These results have formed the basis of integrating the thermal SRC unit and the catalytic hydrotreating unit. Microautoclave tests at Wilsonville have shown that solvents of improved activity can be obtained with addition of hydrotreated SRC.

Two sets of tests were conducted at Wilsonville to evaluate the effects of hydrotreated SRC addition to distillate solvent from the thermal reactor. The hydrotreated SRC and distillate solvent are from NTSL mode of operation. In the first test, hydrotreated SRC from a given batch was added to distillate solvent in varying amounts. The hydrotreated SRC was produced at a reactor temperature of 755°F. The resulting mixture was tested with the microautoclave (in the absence of hydrogen gas) to determine the solvent activity (kinetic). The results of these tests are plotted in Figure 17. This figure indicates that an increase in the concentration of hydrotreated SRC increases the solvent activity. A 20% increase in activity (kinetic) was achieved at a 50% HTR SRC concentration.

A second test was made using hydrotreated SRC from different batches produced at varying HTR reactor temperatures and catalyst ages. The concentration of the blend was maintained at 30%. Figure 18 is a plot of hydrogen content of the residual material as a function of the percentage increase in solvent activity. This plot includes two data points where SRC and LSRC were used in lieu of the hydrotreated SRC. Figure 18 shows that increasing the hydrogen content of the residue (HTR SRC, SRC, or LSRC) increases the solvent activity. A 25% increase in solvent activity is possible if the hydrotreated SRC has 7.25% hydrogen. In general, high HTR reactor temperatures and high catalyst age reduce the hydrogen content of the SRC. Hence, these tests indicate that SRC, hydrotreated under mild conditions and in the presence of relatively fresh catalyst, may be effective in controlling the process solvent activity in the thermal stage. These tests do not reveal any long-term effects on the process due to extended recycle operation in a continuous mode. This subject will be further discussed in a separate report dealing with the integrated runs.

- Feed Coal Specifications

Since the solvent used in all liquefaction processes is basically derived from the feed coal, it stands to reason that the nature of the coal itself has some effect on solvent activity. Although the feed coal is a heterogeneous rock whose composition can vary widely from place to place within the same mine, some control over its composition is possible by writing proper specifications and purchase contracts.

Data relative to solvent activity and coal properties is not very conclusive primarily because solvent activity is also a function of several other variables such as reaction conditions, anthracene oil additions, LSRC recycle, etc. A least-square correlation between the distillate solvent activity and the mineral content of feed coal yielded the following results:

Independent Variable	Correlation Coefficients	
	High Severity	Low Severity
Pyrite sulfur	+0.32	+0.76
Ash	+0.59	+0.86

The above table seems to imply a strong correlation at low severity conditions and a weaker correlation at high severity conditions. However, an examination of the run history for low severity conditions reveals that, for Runs 217, 221, and 222, addition of anthracene oil during Run 214 was an important factor in producing high solvent activities. Lack of strong correlation at high severity conditions can be explained by the fact that, for these runs, the range of variation in coal properties was quite narrow.

During an earlier period (Runs 160 to 212), Kentucky 9 coals from various mines (Pyro, Lafayette, Dotiki, and Fies) were processed at constant dissolver conditions (1,700 psig, 825°F). The average coal conversion was significantly higher (94.3%) when processing coal from Fies mine and was significantly lower (92.2%) when processing coal from Lafayette mine. A total of ten material balances were made. Figure 19 is a plot of average coal conversion versus solvent activity. A fairly good correlation between solvent activity and coal conversion is indicated. Lower conversion of Lafayette coal corresponds to lower solvent activity and higher conversion of Fies coal corresponds to higher solvent activity. If the dissolver is viewed as isolated from the rest of the plant, then solvent activity can be considered an independent variable. With this assumption, the above correlation agrees very well with the observations made by Ruberto at Gulf Research and Development that: "conversion levels depend primarily on the level of transferable hydrogen in the solvent"(Ref. 12). However, the process solvent used at the pilot plant is a recycle stream, and, therefore, not strictly an independent variable. It is, therefore, difficult to determine whether the low conversion of Lafayette coal and high conversion of Fies coal are due to independent variations in solvent activity or whether the variations of both solvent activity and coal conversion are due to differences in chemical composition of the coals. It is interesting to note that Lafayette coal had the lowest pyritic sulfur content. The observations made at Wilsonville seem to imply that mineral content of coal may have a beneficial effect on liquefaction reaction, although conclusive data does not exist. At least, the data is not inconsistent with what has been reported in the literature. For example, conversion of coal to liquid products has been found to increase as the mineral matter content and the concentration of the iron and titanium in the coal increase(Ref. 13). The catalysis by iron compounds in coal liquefaction reaction has been used by the Germans to improve liquefaction results(Ref. 14). Wright and Severson reported that addition of iron contained in the residues from coal liquefaction increased the hydrogen transfer capacity of anthracene oil(Ref. 15).

6. Solvent Properties

- Specific Gravity

The specific gravity of distillate solvent during the period of this report varied from 1.007 to 1.053. For periods of high LSRC recycle (low severity runs), the specific gravity of process solvent varied from 1.05 to 1.07.

Figures 5, 6, and 7 show the specific gravity trends of distillate solvent (or process solvent with less than 7% LSRC) from January 1980 through March 1982. The average specific gravity for 1980 was 1.023 and for 1981 it was 1.034. This increase in 1981 can be attributed to the replacement of the entire solvent inventory with heavier anthracene oil in January 1981. The plot shows a sharp increase in specific gravity at each instance of significant anthracene oil addition. However, these increases were short-lived. In each case, the gravity dropped to some steady-state value.

Tables 1 and 2 summarize the specific gravities of both process and distillate solvents for all MB runs during this report period. This table also lists the corresponding solvent activities. In general, heavier solvents seem to be more active. Addition of 20 to 30% LSRC significantly increases both the density and the activity of the process solvent. Figure 20 plots solvent activity (kinetic) versus solvent density for both distillate and process solvents. The following least squares straight lines were obtained:

Equation	Correlation coefficient	Remarks
SA = 286.7 (sp. gr.)-221.7	0.72	Data for process solvent with high LSRC recycle excluded
SA = 266.6 (sp. gr.)-201.1	0.81	All points included

The first equation is a least-squares fit of all material balance data related to distillate solvent and process solvent containing less than 7% LSRC. The second equation includes data for all low severity process solvent data which can contain up to 30% LSRC. As can be seen, one line can be used for all the points.

The strong correlation indicated by the results implies that continuous monitoring of the solvent specific gravity can be a useful method for predicting trends in solvent activity. However, wide scatter in the daily data is indicative of other factors affecting solvent activity. Hence, a direct correspondence between solvent activity and its specific gravity cannot be assumed. Figure 21 shows the trends in solvent activity as measured by microautoclave (solid line) superimposed on trends predicted by specific gravity measurements (dotted line) for two typical periods. As can be seen, the two lines agree with one another reasonably. The specific gravity lines show less day to day variation and are smoother.

- Hydrogen Content

Of all of the physical and chemical properties, one would expect the solvent hydrogen content to be most closely related to the solvent activity. After all, the kinetic solvent activity is an indication of hydrogen donor capability of the solvent. However, this is not the case. Hydrogen in solvent, as measured by an elemental analyzer gives the total hydrogen content. Not all of this hydrogen is available for donation. The readily transferable hydrogen attached to hydroaromatic compounds and heavy phenols are a small fraction of the total hydrogen content. Hence, one can expect that measurement of the transferable hydrogen, with instruments using nuclear magnetic resonance (NMR), would be more useful for correlation with solvent activity than measurement of total hydrogen with instruments such as an elemental (CHN) analyzer. Due to lack of analytical equipment at Wilsonville, only total hydrogen is routinely measured.

During January 1981, the entire solvent inventory was replaced with anthracene oil. This oil is rich in aromatic compounds and has a very poor hydrogen donor activity (59.9%) and a relatively low total hydrogen content (6.35%). However, after a few days of hydrogenation, the aromatic compounds in anthracene oil are converted to hydroaromatics and the solvent activity increases sharply to 84%. Because of this solvent changeover, Run 225 (January/February 1981) had a high average solvent activity (79.4%). However, as the plant continued to operate without any new addition of anthracene oil, solvent activity steadily declined until it averaged 65.8% for Run 234. This accelerated decline of solvent activity may have been precipitated by the relatively poorer liquefaction properties of the feed coal. Increased rates of deleterious side reactions probably occurred due to a decreased demand for hydrogen by the feed coal. (Hydrogen, "H", content of solvent was at "unsteady state".) The following table compares the properties of the solvent from Runs 225 and 234 as determined by Conoco using NMR(Ref. 7):

Run no.	Date	Days on Recycle	% Total hydrogen	Solvent(a) activity, (kinetic)	Distribution Ratios		
					Condensed aromatics / uncondensed	Cyclic aliphatics / alkyl	% Aromatic
225	15 Feb 1981	32	8.1	79.4	3.83	0.651	38.6
234	9 Sept 1981	176	9.2	65.8	1.75	0.533	25.3

(a) Solvent activity (kinetic) microautoclave tests were done at Wilsonville.

Conoco determined that the ratio of condensed to uncondensed aromatic hydrogen correlates well with the solvent kinetic activity test conversion, and the ratio of cyclic to alkyl aliphatic hydrogen correlates well with the solvent equilibrium activity test conversion. Hence, the above table shows that NMR analysis can be directly related to both kinetic and equilibrium solvent activity. Whereas, the total hydrogen seems to increase with decreases in solvent activity (kinetic).

Figures 5, 6, and 7 show the trends in process solvent hydrogen content and activity for the entire report period. A comparison of the two trend lines reveals that, for the most part, an increase in total hydrogen content decreases the solvent activity. This rule, however, is not applicable to relatively short period immediately after the addition of a significant quantity of anthracene oil. During these periods, presumably when the aromatics in the anthracene oil are being hydrogenated to hydroaromatics, an increase in hydrogen content seems to increase the solvent activity.

Figure 22 is a plot of hydrogen content of distillate solvent, and/or process solvent with less than 7% LSRC, versus solvent activity. Although there seems to be an overall correlation, the scatter is quite high. The correlation coefficient for all of the points was 0.52.

- Effects of +650°F Liquid Fractions

Previous studies have indicated that higher concentrations of +650°F fractions in process solvent have resulted in better solvent activity(Ref. 16). Addition of LSRC is one method for increasing this fraction(Ref. 5). The following table summarizes the results of fractionation studies conducted at Wilsonville in 1979. In these tests, a sample of Wilsonville distillate solvent was fractionated into various cuts in a laboratory column and each cut was tested for solvent activity (kinetic and equilibrium).

Boiling range, °F	Wt, %	Avg. boiling point, °F	Solvent activity,	
			Kinetic	Equilibrium
477-536	27.9	507	61.0	56.3
552-619	23.8	586	65.1	61.1
637-756	28.1	697	69.8	64.0
756-EP	13.9	756	69.6	69.6
Composite	100.0	-	65.1	-

Figure 23 is a plot of this data which clearly shows that heavier boiling fractions are more effective solvents. This confirms similar observations made independently by Sandia Laboratories(Ref. 16).

The possible reasons for the apparent positive influences of heavy boilers on solvent quality are numerous. Higher boiling fractions have greater solubility for both hydrogen and the initial reaction products. It has also been demonstrated that hydrogen transfer from partially hydrogenated higher boiling three-ring compounds is faster than the transfer from similar boiling two-ring compounds.

This would suggest that greater concentrations of the high boiling liquids, with proper ring structure, should aid in improving solvent activity. Solvent activity data from Wilsonville generally supports this theory(Ref. 6).

Figure 24 is a plot of solvent activity (kinetic) versus weight fraction of +650°F material. The plot includes distillate and process solvent data for both high and low severity runs. This figure does reveal a general trend of high solvent activity with high +650°F fraction although the correlation coefficient is only 0.63. Figure 25 is a similar plot for Run 236 which was a low severity run using Illinois #6 (Burning Star) coal. Correlation coefficient for this plot is 0.78.

V. CONCLUSIONS

The following conclusions were drawn from the analyses of data presented in this topical report.

Process Performance

- Coking reactions in coal liquefaction systems occur primarily because of excessive hydrogen demand by free radical coal fragments combined with the inability of the solvent to satisfy this demand. This situation can lead to plugging of lines, coke buildup in dissolvers, erratic letdowns, and low apparent conversion.
- Several process design and operating variables, including solvent activity, influence the extent of coke formation. Maintenance of adequate solvent activity is one way to minimize this deleterious side reaction.
- Results of Wilsonville low severity runs confirm the anticipated benefits of recycling LSRC as a component of process solvent. A 30% blend of LSRC and distillate solvent substantially improves the process solvent activity and makes it possible to operate at solvent balance conditions even at a relatively low dissolver temperature of 780°F.
- At high severity conditions (dissolver tempera- greater than 800°F), increasing the LSRC concentration in process solvent from 5% to 10% substantially increases the distillate solvent yields.
- Interaction of LSRC with a reactive coal is important for the production of distillates at low severity conditions.
- Distillate solvent activity is important to the LSRC interaction and to the stabilization of reactive coke precursors in absence of gaseous hydrogen.
- For a given set of operating conditions and feed coal, the distillate solvent activity achieves a steady state value after about 30-60 days of recycle. This steady state value appears to be a function of starting solvent properties.

Solvent Activity Control

- Solvent activity can be improved by addition of LSRC or anthracene oil. The response time is shorter with LSRC addition.

- Control of distillate solvent boiling range by fractionation is a feasible technique for maintaining a desired level of solvent activity.

- Based upon laboratory studies at Wilsonville, addition of mildly hydrotreated SRC seems to be an effective method for controlling the process solvent activity in the thermal stage. Demonstration of this technique on a continuous integrated basis is required before arriving at any conclusion.

- Feed coal properties seem to influence the steady state solvent activity achieved after extended recycle operations. Oxidized feed coal with low reactivity may cause a sharp decline in solvent activity.

Solvent Properties

- For the range studied, a decrease in solvent activity can be correlated with an increase in its hydrogen content and a decrease in its specific gravity.

- An increase in +650°F liquid fraction in recycle solvent generally resulted in better solvent activity.

REFERENCES

1. Shah, Y. T., "Reaction Engineering in Direct Coal Liquefaction", Addison-Wesley Publishing Co. (1981).

2. Whitehurst, D. D., et. al., "The Nature and Origin of Asphaltenes in Processed Coal", EPRI Final Report AF-1298, Project 410 (1979).

3. Davies, G. O., Derbyshire, F. J., and Price, R. J., J. Inst. Fuels, Vol. 50, p 121 (1977).

4. Weber, W. H., and Styles, G. A., "Effects of Solvent Properties in the SRC Process", Third Annual EPRI Contractors' Conference on Coal Liquefaction, Palo Alto, California (1979).

5. Kleinpeter, J. A., et. al., "Process Development for Improved SRC Options: Short Residence Time Studies", EPRI Final Report AP-1425, Project 1134-1 (1980).

6. Wolk, R. H., "Evolutionary Changes in the Perception of the Role of the Recycle Solvent in the Direct Hydroliquefaction of Coal", AIChE Spring 1981 National Meeting, Houston, Texas (1981).

7. Neavel, R. C., Proceedings of the Coal Agglomeration and Conversion Symposium, DOE, Morgantown Energy Technology Center, Morgantown, West Virginia (April 1976).

8. Lowry, H. H., Editor, "Chemistry of Coal Utilization," John Wiley and Sons, Inc., New York (1976).

9. Davies, G. O., Derbyshire, F. J., and Kimber, G. M., E. C. E. Symposium on Gasification and Liquefaction of Coal, Dusseldorf, West Germany (January 1976).

10. Cantrell, C. E., Lee, J. M., and Johnson, T. W., "Performance of Coal Liquefaction Solvents in Selective Recycle Operation", Seventh Annual EPRI Contractors' Conference on Coal Liquefaction, Palo Alto, California (1982).

11. Silver, H., F., and Hurtubise, R. J., "The Effect of Solvent Characteristics on Coal Liquefaction", Quarterly Technical Progress Report, Dec 1981-Feb 1982, DOE Contract No. DE-AC-22-79ET14874, (1982).

12. Ruberto, R. G., "Fate of Solvents in Non-Catalytic Depolymerization of Kentucky Bituminous Coals", Fuels Proc. Tech. Elsevier Scientific Publishing Co., Amsterdam, Vol. 3, p 7-24 (1980).

13. Mukhurjee, D. K., and Choudhury, P. B., "Catalytic Effect of Mineral Matter in a N. Assam Coal on Hydrogenation", Fuel, Vol. 55, No. 1, p 4-8 (1976).

14. Wu, W. R. K., and Storch, H. H., "Hydrogenation of Coal Tar", USDI, Bureau of Mines Bulletin 633, (1968).

15. Wright, C. H., and Severson, D. E., Am. Chem. Soc. Div. Fuel Chem. Prepr., Vol. 16, p 68 (1972).

16. Bickel, T. C., et. al., "Coal Liquefaction Process Research", Sandia National Laboratories Energy Report, Sand-80-2793,Albuquerque, N. M., Quarterly Report, April-June (1980).

Table 1

Physical and Chemical Properties of Distillate Solvent

MB Run	Activity % THF conversion Short	Activity % THF conversion Long	Specific gravity, at 60°F	Elemental analysis, wt % C	Elemental analysis, wt % H	Elemental analysis, wt % N	Elemental analysis, wt % S	Elemental analysis, wt % O	Composition by boiling point range (°F), wt % TBP-350	Composition by boiling point range (°F), wt % 350-450	Composition by boiling point range (°F), wt % 450-550	Composition by boiling point range (°F), wt % 550-650	Composition by boiling point range (°F), wt % 650-EP	Residue
201ABC														
202A														
203DE														
204C														
206A														
206B														
208AB														
209AB														
210AB														
211B														
212AB														
217AB	79.3			87.32	8.72	0.80	0.46	2.70						
217CD														
219AB														
220AB														
220DE														
221B	80.4	69.1	1.020	86.27	9.14	0.53	0.36	3.70	0.0	1.23	28.07	26.22	44.48	3.0
221G	75.1	68.4	1.021	87.84	8.97	1.00	0.23	1.96	0.0	1.40	25.70	24.74	48.16	3.0
222ABC	74.8	69.7	1.025	85.78	9.20	0.65	0.61	3.76	0.0	0.44	23.58	23.84	52.14	3.0
225BC	82.2	65.0	1.053	87.78	7.87	0.71	0.58	3.06	0.02	3.17	27.80	26.65	42.36	3.0
225F	78.5	65.5	1.046	87.04	8.28	0.70	0.35	3.63	0.0	2.52	28.06	25.58	43.84	3.0
225G	81.2	64.9	1.044	86.42	8.17	0.60	0.48	4.33	0.01	2.73	28.20	25.77	43.28	3.0
225I	75.6	61.5	1.047	87.74	8.10	0.64	0.23	3.29	0.0	3.11	24.23	26.63	46.04	3.0
227A	70.6	63.1	1.044	86.23	8.23	0.64	0.41	4.49	0.0	3.19	31.20	27.11	48.50	3.0
227CD	73.6	62.7	1.044	85.78	7.94	0.61	0.37	5.30	0.0	3.23	33.42	27.60	35.75	3.0
227E	78.9	67.6	1.046	87.51	7.97	0.93	0.31	3.28	0.0	2.76	26.34	27.74	43.17	3.0
227G	80.1	66.8	1.046	86.38	7.83	0.77	0.28	4.74	0.01	3.05	27.07	30.29	39.57	3.0
228AB	69.4	62.7	1.033	87.83	8.36	0.48	0.29	3.04	0.03	5.07	33.85	24.35	36.71	3.0
229A	69.4	63.3	1.025	87.78	8.84	0.67	0.30	2.41	0.0	4.85	33.48	24.80	36.88	3.0
229B	65.6	61.4	1.026	86.79	8.80	1.36	0.35	2.70	0.01	5.88	33.03	24.05	37.05	3.0
232A	71.3	65.9	1.024	86.61	9.02	0.45	0.54	3.38	0.0	4.49	27.86	23.54	44.11	3.0
233B	67.3	63.5	1.022	86.36	9.30	0.64	0.44	3.26	0.0	4.05	27.82	23.64	44.50	3.0
234A	69.8	64.2	1.014	86.42	9.02	0.75	0.39	3.42	0.0	3.65	29.04	24.64	42.66	3.0
234B	63.1	62.4	1.013	85.42	9.31	1.02	0.36	3.89	0.0	3.51	29.65	23.04	43.80	3.0
234C	62.6	61.2	1.007	85.67	9.09	1.67	0.52	3.05	0.0	3.57	29.76	26.45	40.21	3.0
234D	67.8	62.5	1.008	85.61	9.31	0.66	0.43	3.99	0.0	3.71	31.54	26.40	38.34	3.0
235AB	74.3	61.7	1.018	86.56	8.73	0.64	0.20	3.87	0.0	3.08	33.06	21.08	39.79	3.0
235C	70.5	64.2	1.018	85.46	8.90	0.96	0.17	4.51	0.0	2.95	35.02	19.78	42.26	3.0
235D	72.3	60.7	1.022	86.38	8.70	1.06	0.28	3.58	0.0	2.25	35.58	24.88	37.29	3.0
235E	76.4	60.6	1.021	84.90	8.66	0.56	0.22	5.66	0.0	4.18	36.35	25.56	33.90	3.0
236A	74.7	61.5	1.036	85.09	8 60	1.08	0.45	4.78	0.0	0.54	20.59	23.99	54.88	3.0
236B	71.4	62.5	1.028	84.91	8.90	0.62	0.53	5.04	0.0	1.19	25.25	25.03	48 52	3.0

Table 2
Physical and Chemical Properties of Process Solvent

MB Run	LSRC in solvent, wt %	Activity % THF conversion		Specific gravity, at 60°F	Elemental analysis, wt %					Composition by boiling point range (°F), wt %					
		Short	Long		C	H	N	S	O	IBP-350	350-450	450-550	550-650	650-EP	Residue
201ABC	0.0	68.3	60.0	1.019	87.77	8.39	0.39	0.60	2.85	0.0	0.24	47.97	23.88	27.90	0.82
202A	3.0	64.2	60.4	1.013	87.40	8.70	0.49	0.38	3.03	0.15	10.21	46.04	21.41	22.19	0.0
203DE	3.1	69.0	64.2	1.012	87.27	8.77	0.63	0.39	2.94	0.0	1.56	46.50	24.09	27.85	0.30
204C	2.9	70.4	61.2	1.016	87.31	8.84	0.52	0.25	3.08	0.09	1.28	43.03	25.44	29.17	0.27
206A	0.0	66.7	60.3	1.007	87.73	9.04	0.21	0.23	2.79	0.10	0.43	43.98	24.04	31.45	0.63
206B	0.0	66.8	59.9	1.010	87.53	8.87	0.48	0.10	3.02	0.0	0.25	42.10	23.83	33.82	4.16
208AB	5.1	69.6	61.4	1.013	86.95	8.72	0.84	0.40	3.09	0.02	1.57	42.03	24.41	31.97	0.11
209AB	5.0	71.6	65.0	1.025	87.71	8.78	0.39	0.49	2.63	0.0	3.11	33.87	24.43	38.59	3.0
210AB	5.1	69.1	64.0	1.025	87.45	8.71	0.28	0.27	3.29	0.0	2.67	38.01	24.67	34.66	0.33
211B	5.1	77.4	67.3	1.021	88.35	8.82	0.38	0.50	1.95	0.06	3.85	38.38	22.98	34.72	3.0
212AB	5.1	70.6	64.7	1.014	88.89	9.10	0.80	0.40	0.81	0.0	3.80	36.71	25.24	34.26	0.77
217AB	29.4	81.7	61.5	1.066	86.98	8.14	0.88	0.69	3.31	0.47	4.48	21.64	19.25	54.15	16.11
217CD	29.2	82.6	65.3	1.068	85.74	8.24	0.96	0.66	4.40	0.28	4.27	18.43	17.65	59.39	21.10
219AB	4.9	76.0	67.6	1.029	85.83	8.46	0.80	0.37	4.54	0.09	3.70	32.37	27.59	36.25	4.43
220AB	6.1	78.1		1.035	86.98	8.63	0.91	0.38	3.10	0.09	3.77	29.17	26.56	40.41	5.06
220DE	6.1	81.1	66.6	1.046	86.38	8.76	0.73	0.41	3.72	0.13	3.07	21.37	23.25	52.23	3.97
221B	26.3			1.070	87.33	8.61	0.92	0.55	2.59	0.18	1.56	26.28	24.31	47.66	3.0
221G	26.0			1.064	86.67	8.48	0.86	0.92	3.07	0.01	1.11	26.96	24.73	47.19	3.0
222ABC	25.3			1.064	86.67	8.43	0.84	0.58	3.48	0.22	0.81	22.89	23.29	52.79	3.0
225BC	6.1			1.062	87.94	8.00	0.80	0.58	2.68	0.11	2.97	27.43	26.88	42.61	3.0
225F	5.7			1.050	87.33	8.25	0.58	0.48	3.36	0.11	2.63	27.82	25.66	43.79	3.0
225G	6.3			1.053	88.56	8.11	0.82	0.42	2.09	0.15	2.38	28.33	26.47	42.06	3.0
225I	6.4			1.055	87.11	8.13	0.79	0.45	3.52	0.0	2.37	28.27	26.04	43.32	3.0
227A	6.8			1.050	85.36	8.02	0.69	0.39	5.54	0.07	2.97	28.14	27.56	41.25	3.0
227CD	6.6			1.049	85.93	7.86	0.71	0.39	5.11	0.11	3.41	31.27	27.19	38.03	3.0
227E	7.0			1.049	88.22	8.12	0.96	0.38	2.32	0.58	3.38	31.74	29.15	35.16	3.0
227G	6.8			1.047	86.80	7.92	0.69	0.35	4.24	0.13	4.06	31.67	28.88	35.26	3.0
228AB	6.7			1.035	87.74	8.62	1.12	0.29	2.23	0.52	4.68	33.27	24.31	37.21	3.0
229A	6.9			1.033	87.48	8.95	0.79	0.40	2.38	0.07	3.98	31.69	24.99	39.28	3.0
229B	6.6			1.029	85.73	8.88	0.70	0.26	4.43	0.11	5.14	31.89	24.78	38.08	3.0
232A	29.6			1.063	86.59	8.24	0.61	0.56	4.00	0.56	3.02	18.67	15.70	62.04	22.80
233B	29.6				86.43	8.84	1.01	0.64	3.08	0.37	2.82	28.00	22.75	46.07	3.0
234A	29.5				85.85	8.62	0.92	0.60	4.01	0.40	2.93	27.23	25.18	44.27	3.0
234B	32.3				86.05	8.58	0.91	0.59	3.87	0.0	2.74	26.15	26.37	44.74	3.0
234C	31.3				85.74	8.63	0.74	0.63	4.26	0.12	2.52	22.05	20.93	54.38	3.0
234D	31.4				86.96	8.42	1.62	0.66	2.34	0.0	2.67	23.87	22.14	51.32	3.0
235AB	5.2				87.36	8.72	0.58	0.33	3.01	0.0	1.91	33.06	25.66	39.36	3.0
235C	5.6				86.19	8.92	0.82	0.35	3.72	0.0	4.02	38.58	26.70	30.70	3.0
235D	5.5				86.35	8.55	0.96	0.21	3.93	0.0	3.21	34.88	26.14	35.78	3.0
235E	5.7				85.31	8.56	0.68	0.30	5.15	0.0	3.72	36.37	26.09	33.81	3.0
236A	29.5	83.4	54.5	1.122	85.12	8.02	0.79	0.62	5.45	0.35	0.53	14.96	18.64	65.52	24.89
236B	29.6	81.5	59.4	1.050	85.57	8.31	1.18	0.57	4.37	0.04	0.89	18.72	19.32	62.03	23.98

Table 3
Operating Conditions and Yields - Material Balance Data
SRC Unit

MB Run	Run starting date	Duration, days	Coal[(a)]	Coal feed, MF lb/hr	Coal ash, wt %	Slurry conc., % MF coal	Preheater inlet press., psig	Reactor press., psig
201ABC	2/11/80	16	L	518	9.41	37.3	1,731	1,700
202A	2/26/80	11	D	525	9.04	37.9	1,740	1,700
203DE	3/7/80	10	D	505	9.30	36.9	1,742	1,700
204C	3/16/80	17	D	537	9.10	37.5	1,860	1,800
206A	4/17/80	11	D	535	10.13	37.9	1,840	1,800
206B	4/17/80	11	D	523	9.46	38.1	1,840	1,800
208AB	5/4/80	6	D	556	9.05	39.7	1,850	1,800
209AB	5/25/80	13	F	520	7.16	37.7	2,140	2,100
210AB	6/7/80	10	F	535	8.40	39.1	2,150	2,100
211B	6/20/80	25	F	543	8.12	39.3	1,710	1,700
212AB	7/14/80	16	F	527	8.20	38.2	1,745	1,700
217AB	9/17/80	12	F	313	8.87	33.4	2,059	2,000
217CD	9/17/80	12	F	304	8.63	32.7	2,057	2,000
219AB	10/11/80	13	F	535	8.73	36.5	2,195	2,100
220AB	10/23/80	17	F	544	8.52	36.6	2,215	2,100
220DE	10/23/80	17	F	540	8.66	36.2	2,203	2,100
221B	12/9/80	20	F	311	8.65	30.8	2,043	2,000
221G	12/9/80	20	F	333	8.57	30.1	2,032	2,000
222ABC	12/29/80	7	F	332	8.44	29.7	2,039	2,000
225BC	1/11/81	38	F	564	8.50	36.3	2,188	2,100
225F	1/11/81	38	F	548	8.39	35.5	2,180	2,100
225G	1/11/81	38	F	556	8.61	37.2	2,195	2,100
225I	1/11/81	38	F	556	8.77	37.4	2,177	2,100
227A	3/21/81	58	F	575	7.85	39.0	2,141	2,100
227CD	3/21/81	58	F	562	8.00	38.8	2,147	2,100
227E	3/21/81	58	F	573	8.29	39.4	2,145	2,100
227G	3/21/81	58	F	564	7.98	38.7	2,140	2,100
228AB	5/18/81	25	F	573	8.52	38.8	2,137	2,100
229A	6/1/81	21	F	552	7.86	38.7	2,326	2,300
229B	6/1/81	21	F	560	7.95	38.4	2,329	2,300
232A	7/13/81	9	F	476	7.86	33.6	2,005	2,000
233B	7/21/81	25	F	430	7.88	32.9	2,000	2,000
234A	8/14/81	30	F	413	7.54	33.1	2,011	2,000
234B	8/14/81	30	F	396	7.38	32.7	2,023	2,000
234C	8/14/81	30	F	431	7.81	33.8	2,011	2,000
234D	8/14/81	30	F	416	7.64	33.8	2,027	2,000
235AB	10/17/81	66	F	531	7.58	37.6	2,142	2,100
235C	10/17/81	66	F	520	6.87	38.4	2,145	2,100
235D	10/17/81	66	F	534	7.61	37.6	2,144	2,100
235E	10/17/81	66	F	546	7.67	39.3	2,148	2,100
236A	1/5/82	62	B	410	10.57	33.0	2,035	2,000
236B	1/5/82	62	B	403	10.98	33.5	2,044	2,000

(a) L - Lafayette, Ky 9; D - Dotiki, Ky 9; F - Fies, Ky 9; B - Burning Star, Ill. 6.

(Table continued)

Table 3 (continued)
Operating Conditions and Yields - Material Balance Data
SRC Unit

MB Run	Temperature, °F			R101 volume in use, %	Coal space rate, MF lb/hr-ft³		Hydrogen consumption, % MAF coal
	Preheater outlet	R101 Dissolver					
		Bottom	Outlet		Cumulative	R101	
201ABC	797	818	824	75	31.1	38.2	2.2
202A	794	819	826	75	31.5	38.7	1.4
203DE	789	819	826	75	30.3	37.2	2.2
204C	802	834	841	75	32.2	39.5	2.5
206A	804	833	840	75	32.1	39.4	1.5
206B	814	834	840	75	31.4	38.5	2.3
208AB	804	834	843	75	33.3	40.9	0.9
209AB	793	825	839	75	31.2	38.3	1.1
210AB	-	-	835	75	32.1	39.4	2.5
211B	760	816	825	75	32.5	40.0	1.9
212AB	760	816	825	75	31.6	38.8	2.3
217AB	794	782	785	50	25.5	34.0	2.0
217CD	793	780	784	50	24.7	33.1	1.6
219AB	788	812	840	75	32.1	39.4	2.6
220AB	787	804	840	75	32.6	40.1	2.2*
220DE	796	806	840	75	32.4	39.8	2.2*
221B	781	745	765	100	14.5	16.9	1.7*
221G	760	745	770	100	15.5	18.1	1.6
222ABC	758	735	754	100	15.5	18.1	1.3
225BC	770	812	842	75	33.8	41.5	1.7*
225F	775	813	841	75	32.9	40.4	1.9
225G	772	812	841	75	33.3	40.9	2.3*
225I	771	812	842	75	33.3	40.9	1.8
227A	792	795	841	75	34.5	42.3	2.6*
227CD	792	793	841	75	33.7	41.4	2.7*
227E	791	797	841	75	34.4	42.2	2.8*
227G	792	802	839	75	33.8	41.5	2.7*
228AB	765	800	825	75	34.4	42.2	2.3*
229A	773	798	823	75	33.1	40.6	2.5
229B	776	796	824	75	33.6	41.2	2.2
232A	752	766	785	75	28.6	35.0	2.0*
233B	763	766	786	75	23.8	29.1	2.1*
234A	765	769	786	75	24.8	30.4	2.1*
234B	757	764	787	75	28.6	29.1	2.3*
234C	769	768	785	75	25.9	31.7	2.0*
234D	763	766	786	75	25.0	30.6	2.2*
235AB	750	797	837	75	31.9	39.1	2.2*
235C	793	804	833	75	31.2	38.3	2.1*
235D	792	804	834	75	32.0	39.3	2.6*
235E	787	803	836	75	32.8	40.2	2.1*
236A	772	769	785	75	24.6	30.2	1.6*
236B	765	768	785	75	24.2	29.6	1.7*

*By ethylene injection method.

(Table continued)

Table 3 (continued)
Operating Conditions and Yields - Material Balance Data
SRC Unit

MB Run	Hydrogen in feed gas, mole %	Hydrogen partial press., B102 inlet, psi	SRC yield, % MAF coal	Distillate solv. yield, 450-EP (°F), % MAF coal	Naphtha yield, IBP-350 (°F), % MAF coal	Mid. dist. yield, 350-450 (°F), % MAF coal	Total dist. yield, % MAF coal	Total gas yield, % MAF coal	Unreacted coal, wt %
201ABC	85.0	1,484	69.7	10.1	2.6	4.4	17.1	7.1	7.5
202A	82.8	1,453	55.9	22.9	2.2	3.6	28.7	7.6	6.2
203DE	81.3	1,426	59.6	14.7	4.9	4.0	23.6	8.1	7.0
204C	82.5	1,547	55.5	17.0	6.4	7.0	30.4	7.9	6.4
206A	82.5	1.530	55.9	16.5	5.8	7.0	29.3	8.4	7.3
206B	84.6	1,569	60.6	13.7	5.8	5.7	25.2	7.8	8.5
208AB	84.0	1,567	59.2	15.6	4.2	6.1	25.9	9.5	7.6
209AB	80.3	1,731	54.8	17.4	2.3	6.2	26.1	11.9	6.2
210AB	82.2	1,780	51.8	16.0	4.4	5.7	26.1	10.3	5.8
211B	84.5	1,458	60.3	20.3	0.2	4.2	24.7	5.3	6.0
212AB	83.0	1,461	57.9	23.8	1.9	3.6	29.3	7.2	5.7
217AB	92.1	1,902	57.1	24.2	1.9	2.8	28.9	5.6	6.6
217CD	91.8	1,902	60.2	18.4	4.4	3.8	26.[illegible]	5.5	6.9
219AB	82.3	1,819	58.6	17.4	4.8	6.0	28.2	8.2	6.4
220AB	82.3	1,823	53.6	22.8	4.7	5.3	32.9	11.3	5.6
220DE	84.5	1,874	54.3	20.8	4.0	4.8	29.6	8.3	6.4
221B	86.2	1,774	59.6	23.4	2.5	2.5	28.4	5.6	6.6
221G	82.8	1,695	64.0	12.6	5.6	2.0	21.2	7.5	6.3
222ABC	85.3	1,752	63.7	15.5	3.2	1.9	20.6	5.8	7.6
225BC	83.6	1,842	55.2	21.7	4.2	4.5	30.4	8.1	6.3
225F	77.4	1,699	55.8	17.4	4.8	6.6	28.8	8.1	7.3
225G	82.2	1,817	56.4	19.0	4.4	5.2	28.6	9.2	6.9
225I	83.9	1,839	58.[illegible]	17.1	3.9	4.1	25.1	8.7	7.3
227A	79.6	1,716	59.4	15.6	3.7	6.1	25.4	12.4	7.[illegible]
227CD	82.1	1,775	53.6	21.1	4.5	6.8	32.4	10.1	6.[illegible]
227E	84.1	1,817	57.2	12.5	5.9	7.2	25.6	8.3	8.1
227G	84.5	1,821	54.7	18.3	5.0	8.8	32.1	9.1	7.5
228AB	87.0	1,872	58.8	14.5	4.3	6.0	24.8	11.4	7.7
229A	83.6	1,957	60.3	15.1	4.6	5.2	24.9	8.3	6.3
229B	84.2	1,974	57.1	17.9	4.6	4.6	27.3	7.5	8.1
232A	83.3	1,690	67.7	10.4	3.0	3.2	16.6	7.0	7.7
233B	81.9	1,650	71.1	8.4	2.9	3.4	14.7	-	7.9
234A	85.0	1,720	71.1	8.7	2.5	2.1	13.3	6.2*	7.9
234B	85.4	1,740	67.4	10.6	2.0	1.9	14.5	6.5*	8.3
234C	82.2	1,670	72.1	6.8	2.4	1.9	11.1	7.1*	8.6
234D	83.2	1,700	67.0	10.7	3.0	2.9	16.6	6.6*	8.6
235AB	85.4	1,840	58.7	12.7	3.4	5.1	21.2	10.9*	7.3
235C	84.5	1,830	55.2	17.2	3.2	6.1	26.5	10.1*	6.5
235D	84.7	1,830	57.1	14.5	3.1	5.8	23.4	10.2*	8.9
235E	87.8	1,900	56.0	18.9	3.9	5.0	27.8	9.0*	6.7
236A	84.8	1,730	55.7	19.6	1.8	4.7	26.1	7.8*	8.1
236B	86.5	1,781	55.0	20.0	2.7	4.7	27.4	7.8*	8.8

*By ethylene injection method.

Table 4
Properties of Feed Coal

MB Run	Coal[(a)]	Activity[(b)]	Conversion to cresol solubles % MAF coal	Ultimate analysis, % C	H	N	Cl	S	Ash	O	Moisture, %
201ABC	L	78.2	92.5	73.04	4.39	0.49	-	2.44	9.41	10.23	1.23
202A	D	80.2	93.8	69.81	4.22	1.18	0.27	3.25	9.04	12.23	0.95
203DE	D	80.2	93.0	73.71	4.75	1.31	0.28	3.58	9.30	7.57	1.40
204C	D	79.7	93.6	73.76	4.92	1.08	0.48	3.26	9.10	7.40	1.19
206A	D	79.6	92.8	73.22	5.01	0.55	0.22	3.41	10.13	7.46	1.09
206B	D	79.7	92.3	74.72	4.72	1.01	0.31	2.90	9.46	8.88	0.91
208AB	D	79.5	92.4	74.21	5.10	0.75	0.26	2.67	9.05	7.96	0.94
209AB	F	80.5	93.8	71.59	4.83	0.79	0.07	2.32	7.16	12.24	2.43
210AB	F	81.0	94.7	71.99	5.03	0.84	0.10	3.18	8.40	10.46	1.92
211B	F	83.5	94.2	73.15	4.91	1.09	0.09	3.07	8.12	9.57	1.95
212AB	F	80.6	94.3	70.73	4.81	1.38	0.14	3.14	8.20	11.60	1.68
217AB	F	75.7	93.0	72.42	4.99	1.53	0.07	3.29	8.87	8.83	1.93
217CD	F	78.0	93.2	71.11	5.02	1.47	0.06	3.28	8.63	10.43	0.86
219AB	F	82.4	92.5	71.30	4.66	1.18	0.07	3.38	8.73	10.68	1.53
220AB	F	76.2	93.6	69.71	4.80	1.12	0.07	3.69	8.52	12.09	1.44
220DE	F	77.9	93.5	70.86	5.06	1.48	0.07	3.42	8.66	10.45	0.86
221B	F	79.0	92.8	72.04	4.72	1.51	0.09	2.87	8.65	10.12	4.68
221G	F	82.4	93.7	72.06	4.81	1.40	0.10	3.25	8.57	9.81	1.53
222ABC	F	79.3	91.6	71.02	4.79	1.24	0.02	3.00	8.44	11.49	1.81
225BC	F	80.1	93.8	72.80	4.98	1.36	0.08	3.35	8.50	8.93	2.03
225F	F	76.4	93.5	74.31	4.87	1.15	0.11	3.19	8.39	7.98	1.35
225G	F	79.9	93.2	72.87	4.86	1.00	0.15	3.15	8.61	9.36	1.93
225I	F	76.2	93.7	71.42	4.80	1.13	0.06	3.10	8.77	10.72	1.58
227A	F	80.3	94.0	71.40	4.82	1.12	0.10	3.01	7.85	11.70	1.83
227CD	F	82.1	93.8	72.84	4.58	1.39	0.06	3.22	8.00	9.91	1.82
227E	F	82.4	93.4	71.18	4.65	1.42	0.09	3.14	8.20	11.32	0.93
227G	F	83.3	94.0	71.72	4.58	1.39	0.08	3.29	7.98	10.98	1.54
228AB	F	79.9	94.3	72.14	4.80	1.24	0.10	3.45	8.52	9.75	2.20
229A	F	76.1	94.7	72.74	4.80	1.40	0.11	2.85	7.86	10.24	1.37
229B	F	79.5	92.8	71.24	4.92	1.48	0.12	2.98	7.95	11.31	1.75
232A	F	71.6	93.4	71.27	4.77	1.06	0.10	3.17	8.00	11.63	1.92
233B	F	75.0	93.4	72.31	4.77	1.46	0.10	2.75	7.88	10.73	2.02
234A	F	74.2	92.2	72.22	4.58	1.05	0.09	2.81	7.54	11.71	2.28
234B	F	73.7	92.8	71.54	4.72	1.58	0.09	2.66	7.54	11.87	2.18
234C	F	77.2	93.1	72.07	4.86	1.35	0.08	2.91	7.81	10.92	-
234D	F	73.9	92.5	72.42	4.82	1.46	0.09	3.19	7.64	10.38	-
235AB	F	78.4	93.6	74.77	5.15	1.34	0.07	3.07	7.68	7.92	1.47
235C	F	77.6	94.5	73.18	4.92	1.75	0.05	3.22	6.87	10.01	1.61
235D	F	78.5	93.7	73.12	4.88	2.00	0.09	2.79	7.61	9.51	1.70
235E	F	80.5	94.0	72.54	5.00	1.56	0.05	2.84	7.67	10.34	2.29
236A	B	78.2	92.4	68.68	4.61	0.88	-	3.49	10.57	11.77	2.22
236B	B	74.3	92.4	68.66	4.66	1.16	0.09	3.27	10.98	11.18	2.52

(a) L - Lafayette, Ky 9; D - Dotiki, Ky 9; F - Fies, Ky 9; B - Burning Star, Ill. 6.
(b) Microautoclave conversion, long run.

(Table continued)

Table 4 (continued)
Properties of Feed Coal

MB Run	Ash content, %					Sulfur forms, %			
	SiO_2	TiO_2	Fe_2O_3	Al_2O_3	K_2O	Pyritic	Sulfate	Sulfide	Organic
201ABC	59.70	1.56	13.06	20.39	2.14	0.66	0.01	<0.01	1.77
202A	49.35	1.34	20.85	18.99	2.33	1.18	0.03	<0.01	2.04
203DE	50.53	1.35	20.13	19.03	2.45	1.34	0.03	<0.01	2.21
204C	51.08	0.54	18.90	18.63	2.01	1.20	<0.01	<0.01	2.06
206A	46.67	0.66	18.94	18.53	2.52	1.14	<0.01	<0.01	2.27
206B	48.18	0.65	19.07	18.73	2.54	1.03	<0.01	<0.01	1.87
208AB	47.85	0.73	18.83	19.67	2.44	1.08	<0.01	<0.01	1.59
209AB	46.50	1.07	23.96	18.69	1.88	1.18	<0.01	<0.01	2.14
210AB	47.82	0.87	22.05	17.92	2.24	1.14	0.06	0.02	1.96
211B	45.83	0.79	22.92	18.07	1.95	1.11	0.13	0.06	1.77
212AB	46.99	1.20	23.60	18.41	1.82	1.11	0.01	0.16	1.86
217AB	47.86	0.91	23.64	18.93	1.86	1.42	0.01	<0.01	1.86
217CD	49.03	0.84	23.20	17.03	1.87	1.42	0.10	<0.01	1.86
219AB	47.11	0.89	23.90	18.26	2.03	1.37	0.06	0.01	1.94
220AB	47.90	1.05	23.51	17.29	1.93	1.59	0.04	<0.01	2.06
220DE	49.74	0.78	23.17	18.07	1.88	1.15	0.04	0.21	2.02
221B	48.26	0.82	21.15	18.10	2.33	1.07	0.08	<0.01	1.72
221G	48.36	0.87	23.61	17.65	2.01	1.36	0.04	<0.01	1.85
222ABC	50.70	1.38	21.04	19.01	1.34	1.10	0.10	0.02	1.78
225BC	50.09	0.79	20.62	20.24	1.47	1.37	0.04	<0.01	1.94
225F	47.92	1.27	20.93	19.61	2.09	1.11	0.09	<0.01	1.99
225G	51.16	0.90	19.13	18.95	1.95	1.10	0.05	0.02	1.98
225I	51.07	0.78	19.18	19.62	1.70	1.10	0.06	<0.01	1.94
227A	49.88	0.92	20.68	19.06	1.00	1.14	<0.01	<0.01	1.87
227CD	48.43	0.88	20.73	18.81	1.05	1.22	0.04	0.03	1.93
227E	48.61	1.06	20.63	18.22	2.20	1.17	<0.01	0.03	1.94
227G	47.90	1.15	20.70	18.11	2.24	1.27	<0.01	<0.01	2.02
228AB	50.93	1.43	23.10	16.54	1.27	1.48	<0.01	<0.01	1.97
229A	47.86	0.92	22.67	18.78	1.33	1.01	0.13	0.07	1.64
229B	47.46	0.92	22.92	18.87	1.55	1.06	0.09	<0.01	1.63
232A	48.50	1.24	22.58	17.88	1.53	1.03	0.26	0.01	1.87
233B	49.98	1.40	21.14	17.10	1.36	0.72	0.23	<0.01	1.80
234A	48.53	1.24	22.56	19.04	0.25	0.70	0.28	0.03	1.80
234B	48.38	1.27	22.77	18.94	1.84	0.66	0.25	0.02	1.73
234C	47.95	1.16	21.72	18.04	1.32	1.00	0.06	0.02	1.53
234D	50.44	1.17	20.83	17.74	1.32	1.09	0.11	0.04	1.95
235AB	49.53	1.30	21.56	19.00	1.57	1.20	0.06	0.01	1.80
235C	49.03	1.05	20.59	18.77	1.10	1.04	< 0.01	< 0.01	2.18
235D	51.86	1.05	19.54	19.32	1.66	1.18	0.03	< 0.01	1.80
235E	49.06	0.97	19.88	20.03	1.84	1.13	< 0.01	< 0.01	1.94
236A	48.36	0.92	16.34	18.14	1.38	1.33	< 0.01	0.01	2.03
236B	49.46	0.92	17.41	19.14	1.45[1]	1.13	< 0.01	0.02	2.21

Table 5
Properties of Anthracene Oil

Specific gravity at 38/15.5°C	1.06
Water, %	1.0
Benzene insolubles, wt %	0.50

GC Simulated Distillation

Boiling point range, °F	Weight %
IBP-350	0.05
350-450	6.94
450-500	20.92
500-550	13.50
550-650	22.72
650-EP	35.87
Residue	0.00

Elemental analysis, %

Carbon - 90.76
Hydrogen - 6.35
Nitrogen - 1.92
Chlorine - 0.13
Sulfur - 0.13
Ash - 0.30
Oxygen - 0.41

Activity (kinetic) % THF conversion	59.9

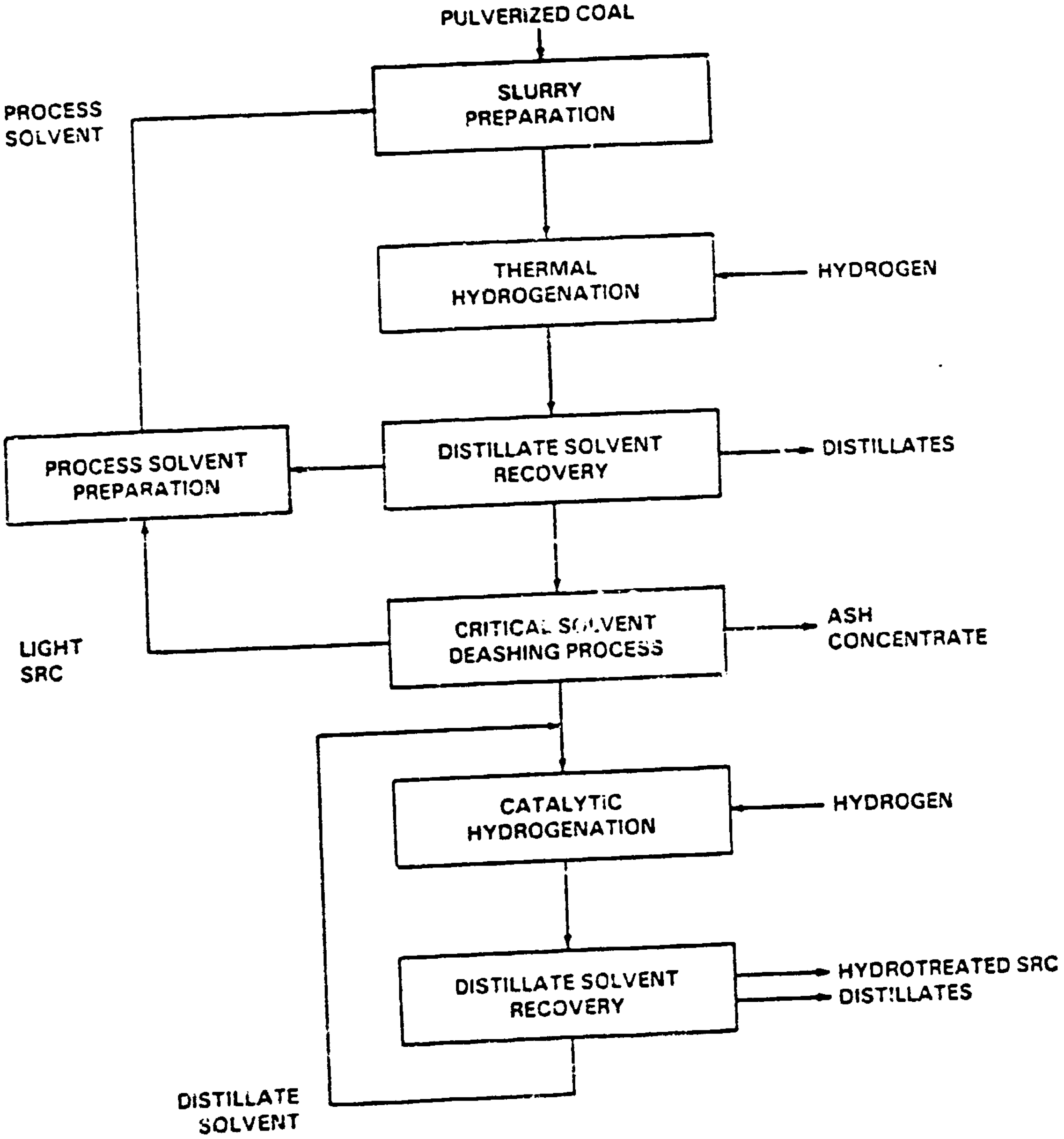


FIGURE 1. TWO STAGE LIQUEFACTION (NON INTERGRATED MODE)

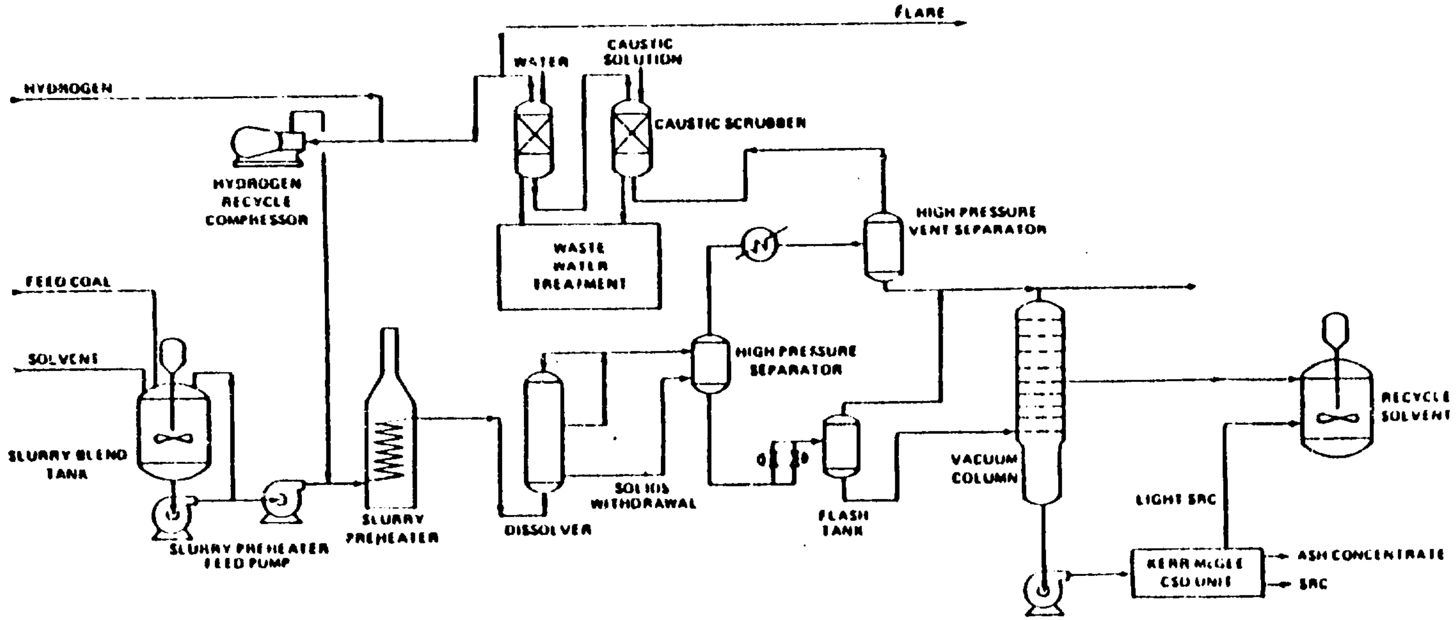


FIGURE 2. SOLVENT REFINED COAL PROCESS

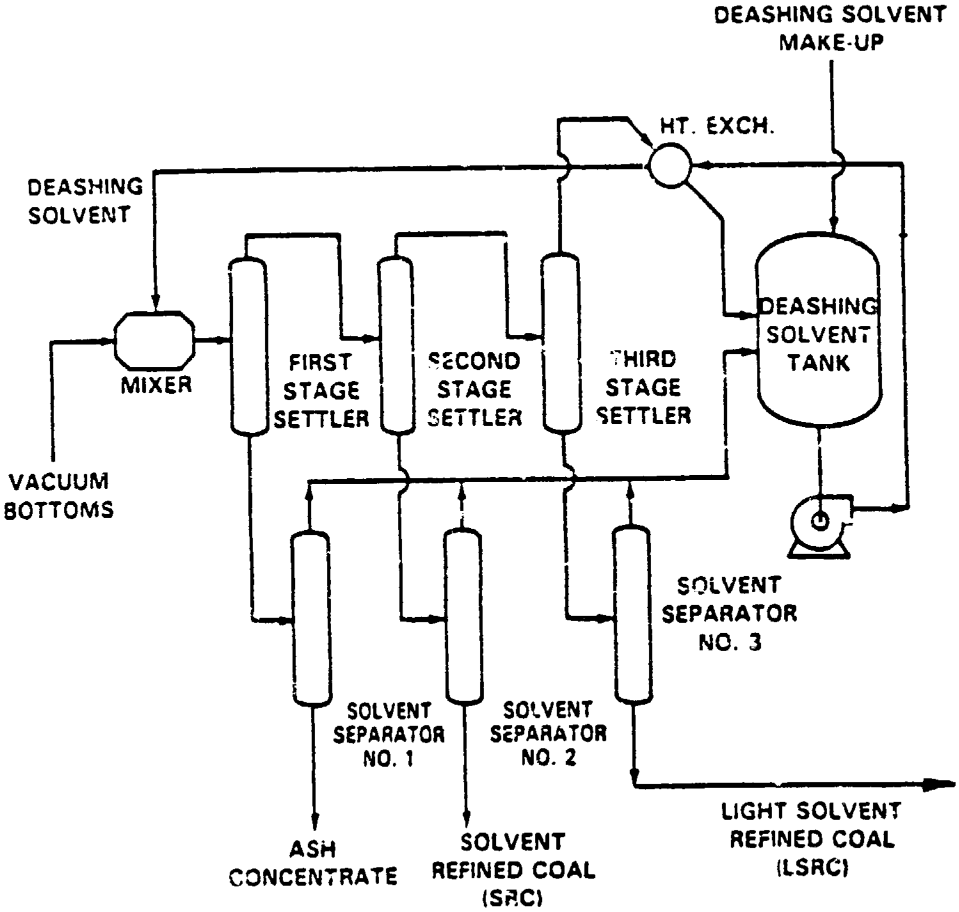


FIGURE 3. CRITICAL SOLVENT DEASHING PROCESS

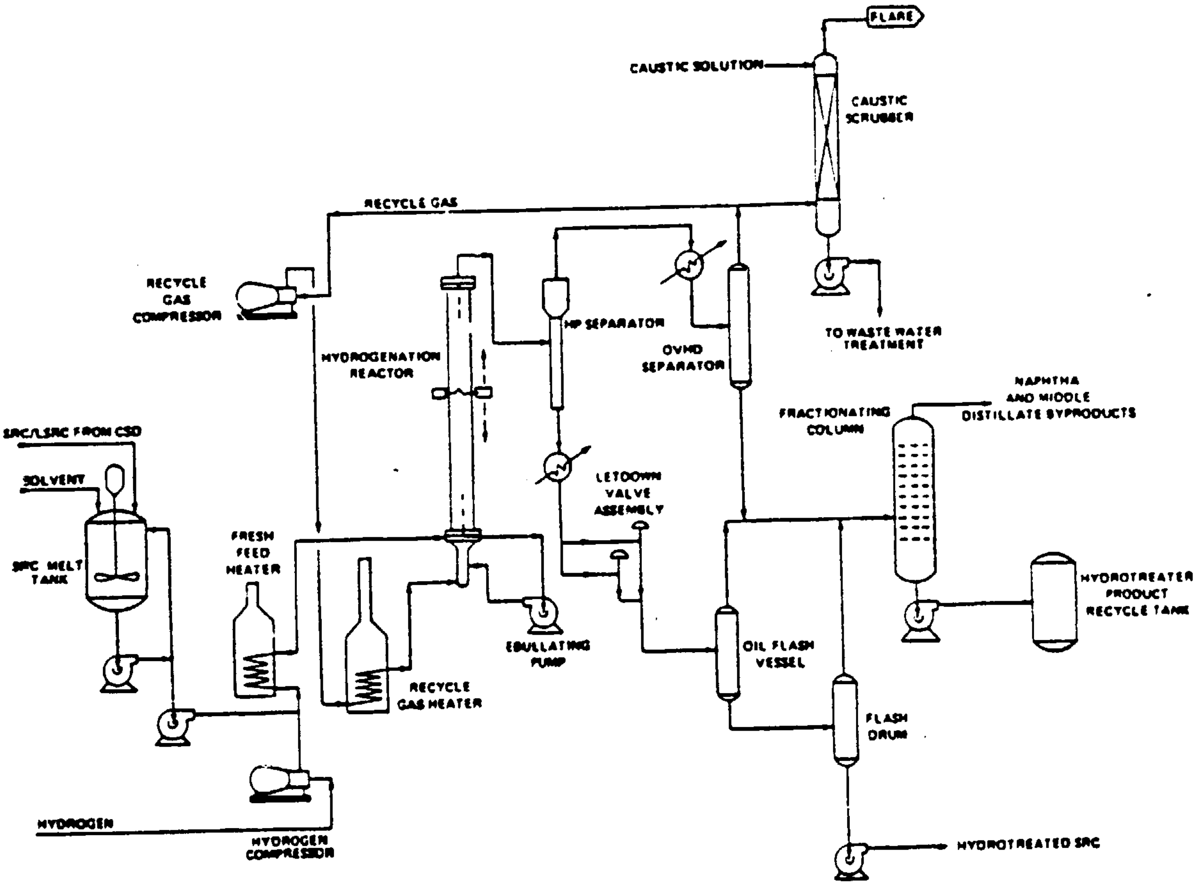


FIGURE 4. EBULLATED BED HYDROTREATER PROCESS

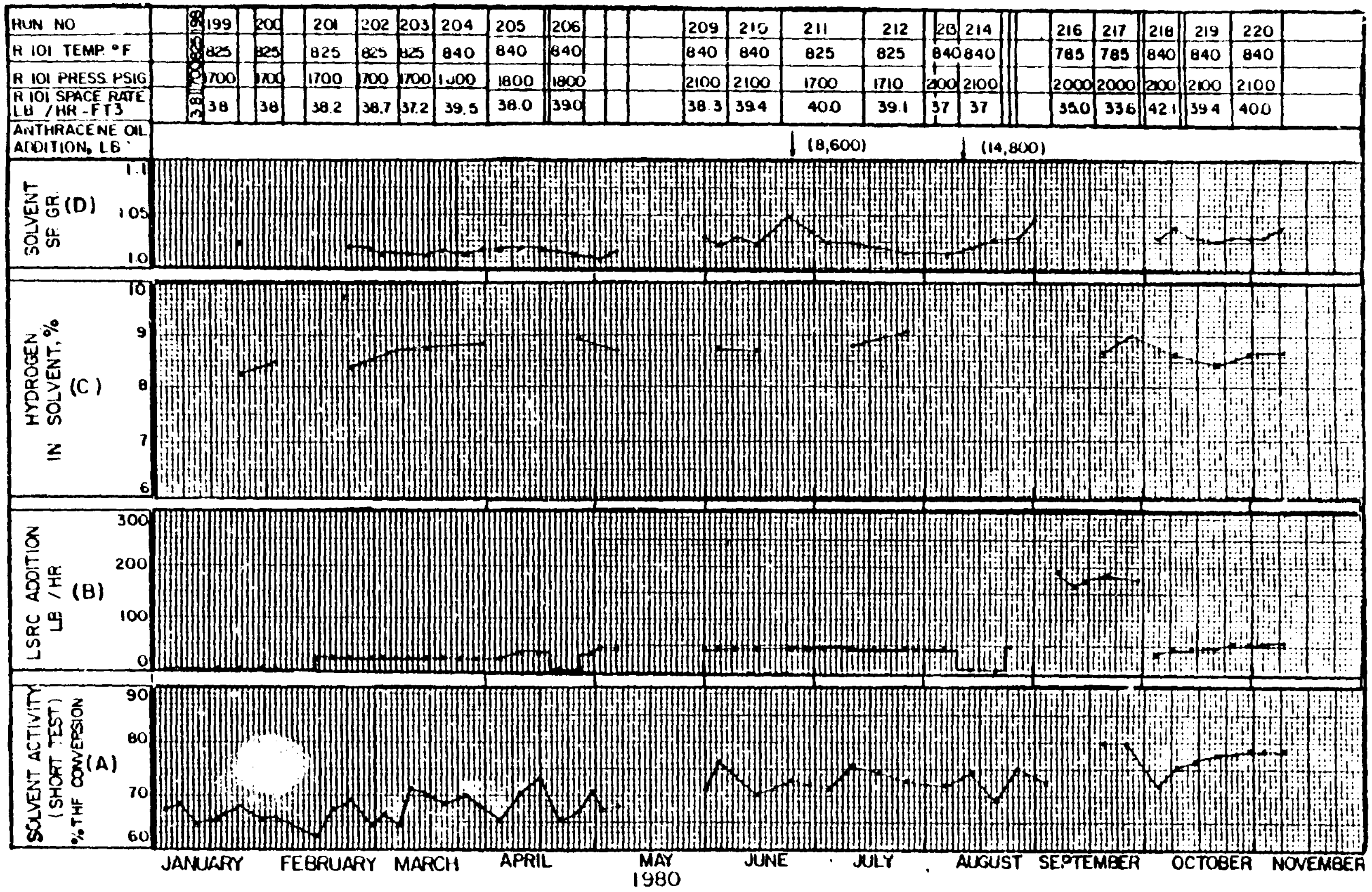


FIGURE 5. TIME PLOTS FOR DISTILLATE SOLVENT ACTIVITY, HYDROGEN CONTENT, SPECIFIC GRAVITY AND LSRC ADDITION (JANUARY THROUGH NOVEMBER 1980)

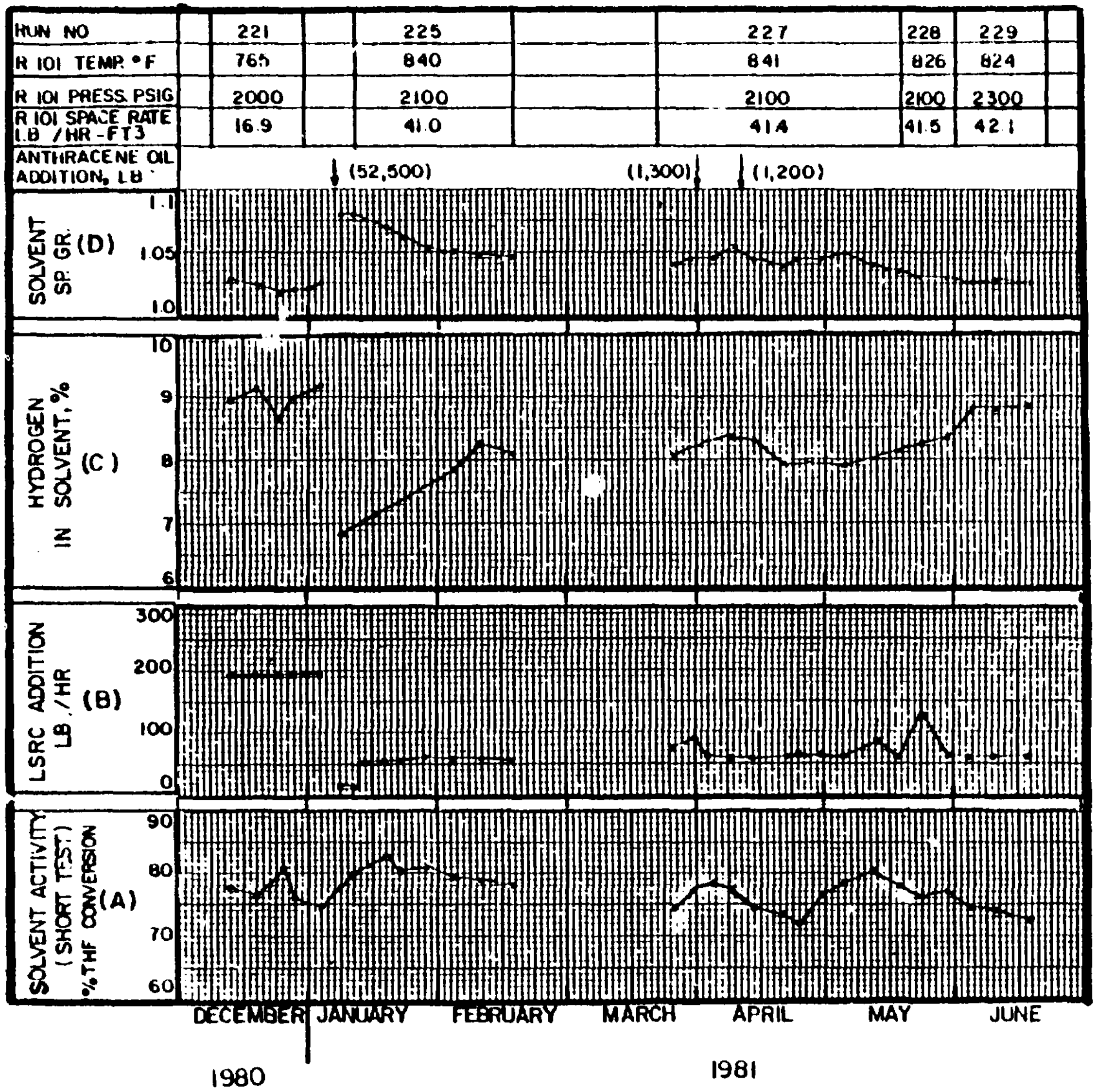


FIGURE 6. TIME PLOTS FOR DISTILLATE SOLVENT ACTIVITY, HYDROGEN CONTENT, SPECIFIC GRAVITY AND LSRC ADDITION (DECEMBER 1980 THROUGH JUNE 1981)

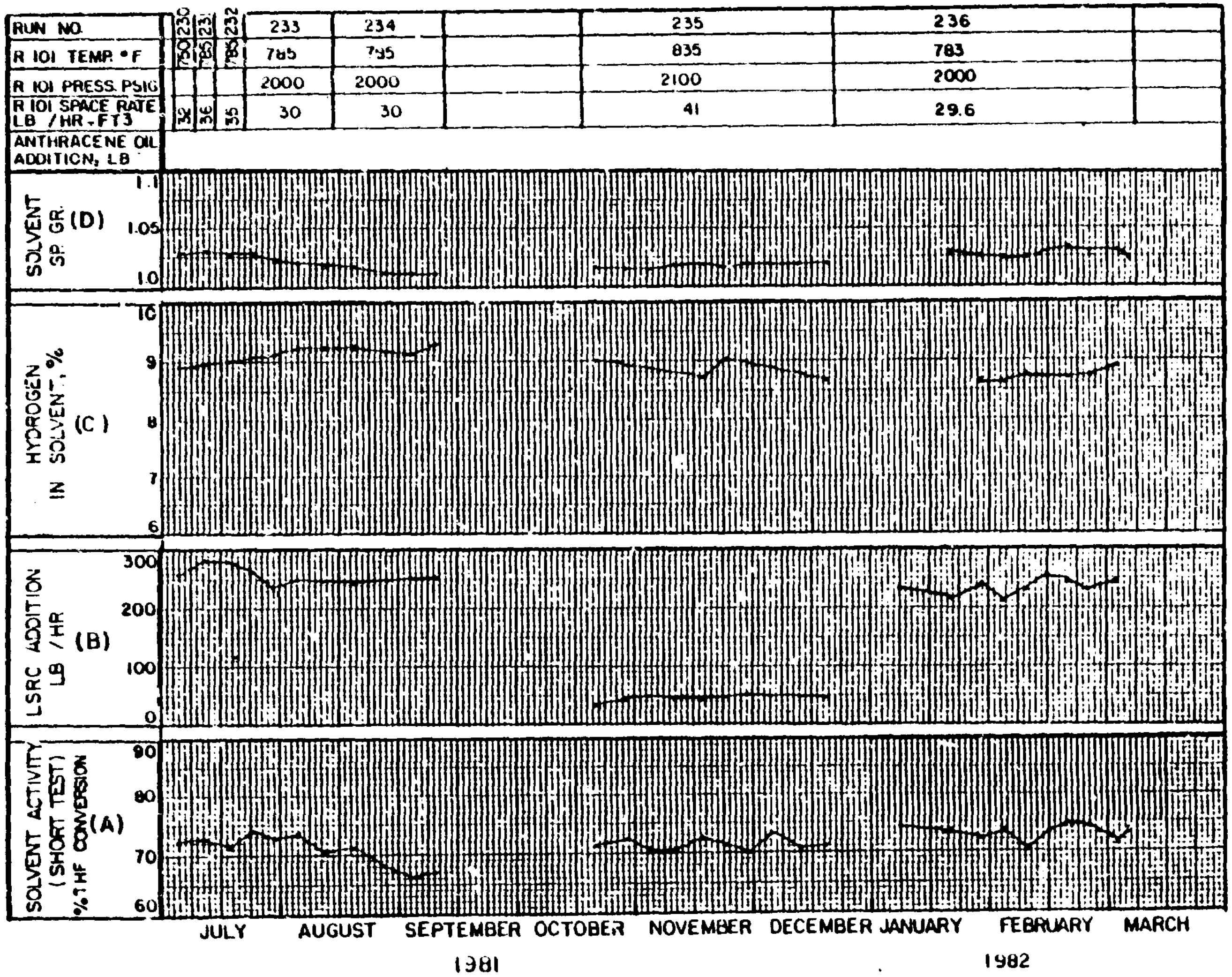
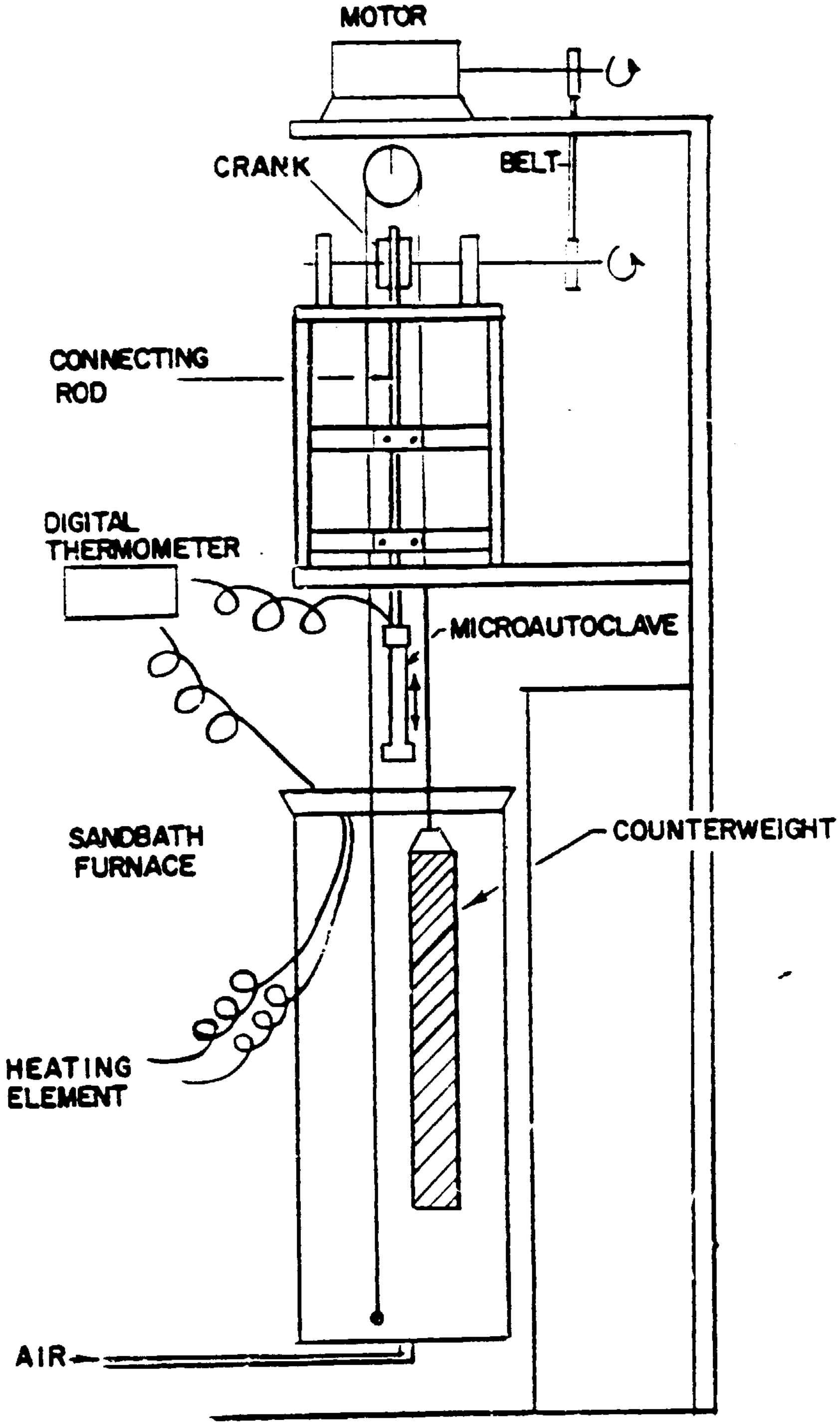


FIGURE 7. TIME PLOTS FOR DISTILLATE SOLVENT ACTIVITY, HYDROGEN CONTENT, SPECIFIC GRAVITY AND LSRC ADDITION (JULY 1981 THROUGH MARCH 1982)



SANDBATH DIMENSIONS: LENGTH= 31", I.D.= 6", O.D.= 9"

FIGURE 8. MICROAUTOCLAVE UNIT

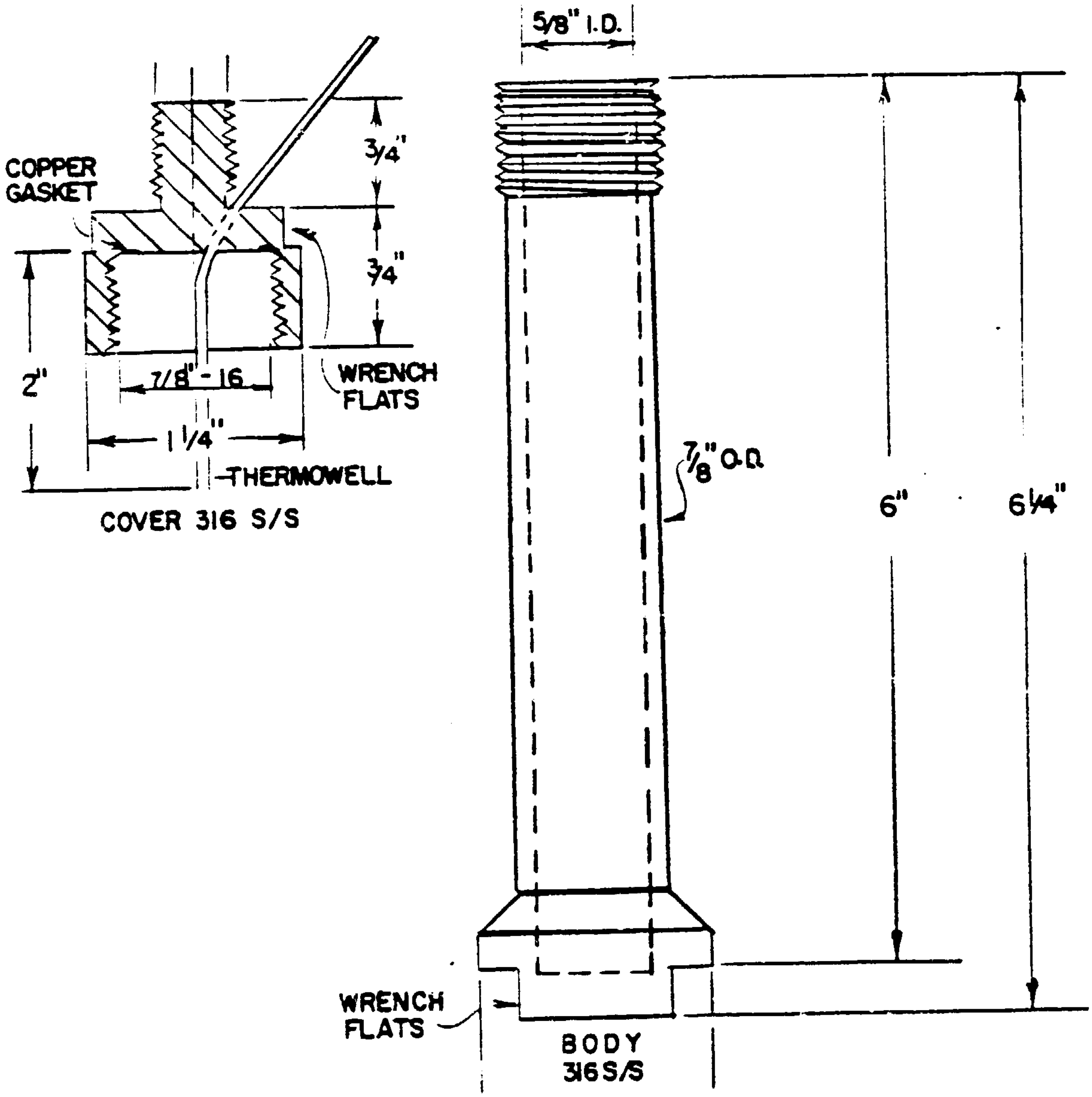


FIGURE 9. MICROAUTOCLAVE REACTOR

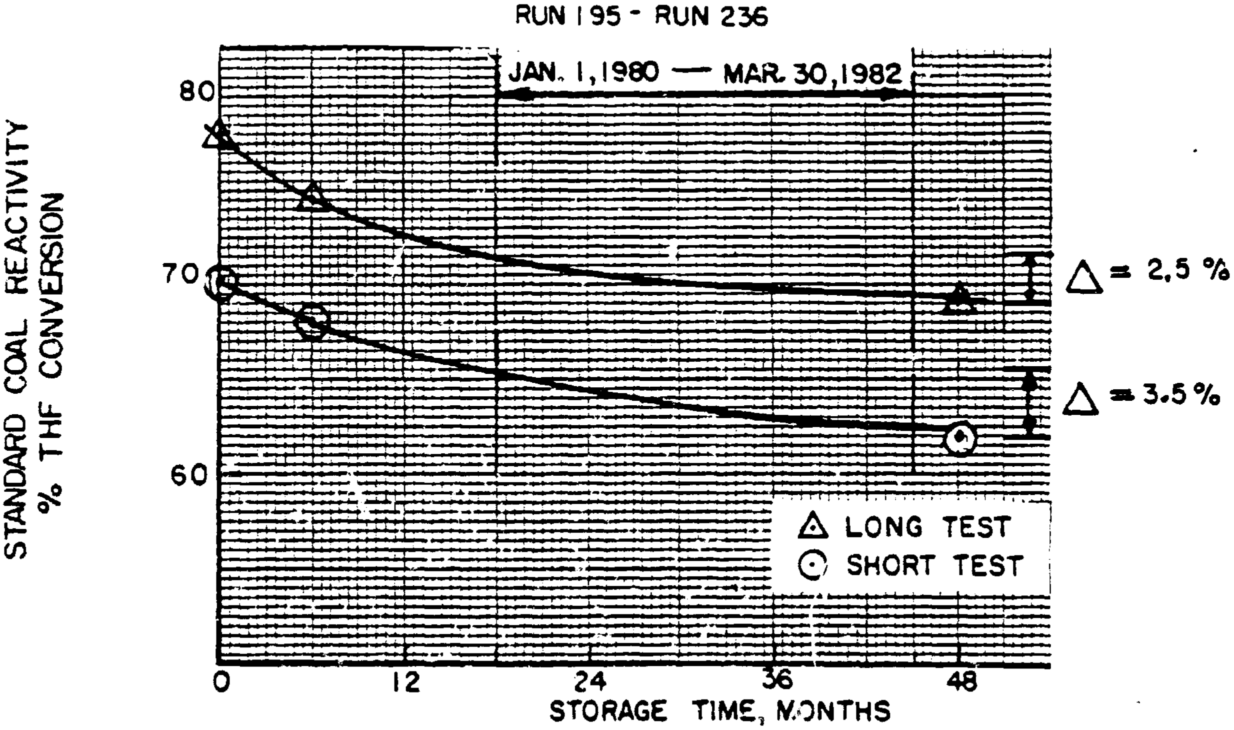


FIGURE 10 CHANGE IN COAL REACTIVITY WITH STORAGE TIME

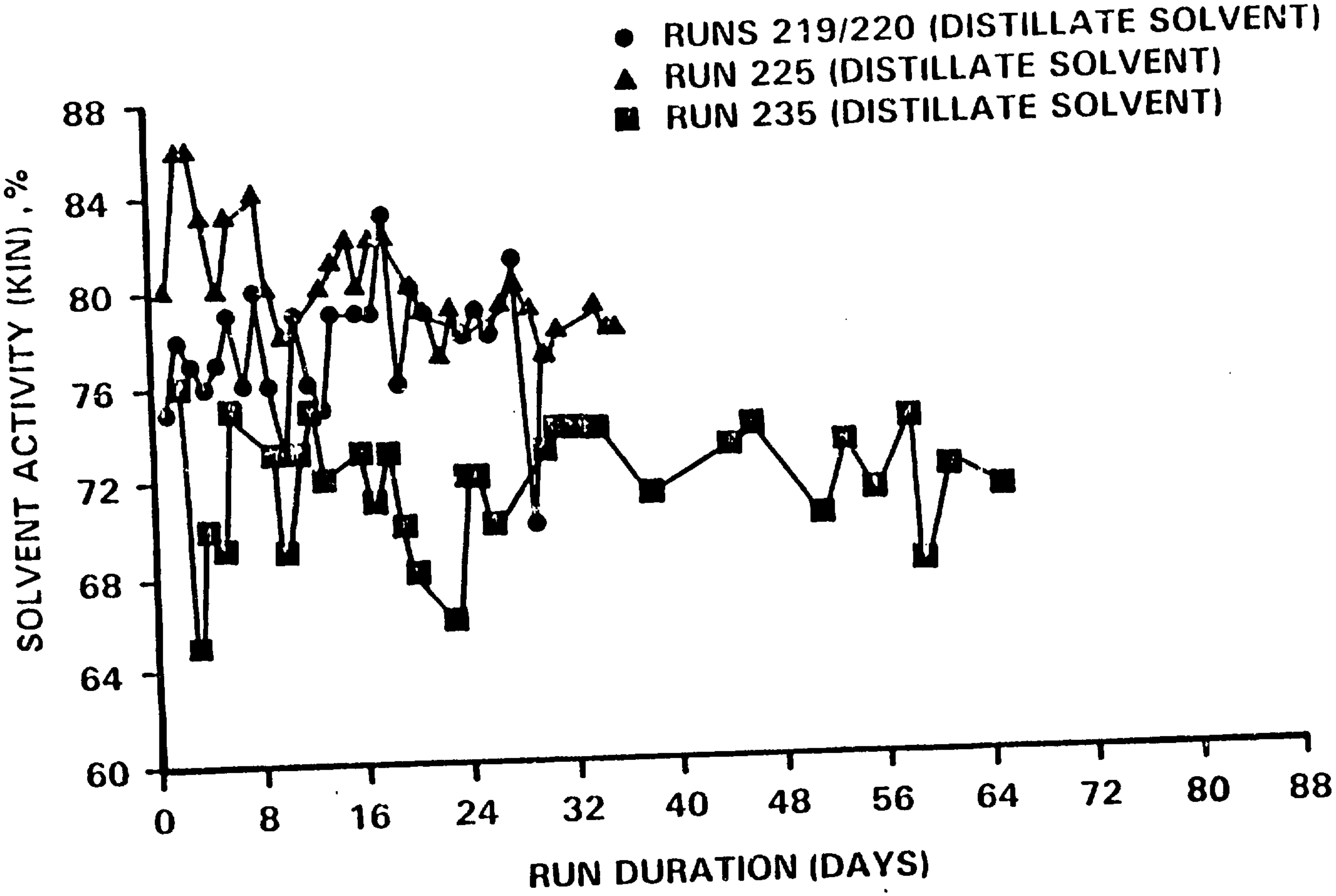


FIGURE II. STEADY STATE SOLVENT ACTIVITY-HIGH SEVERITY

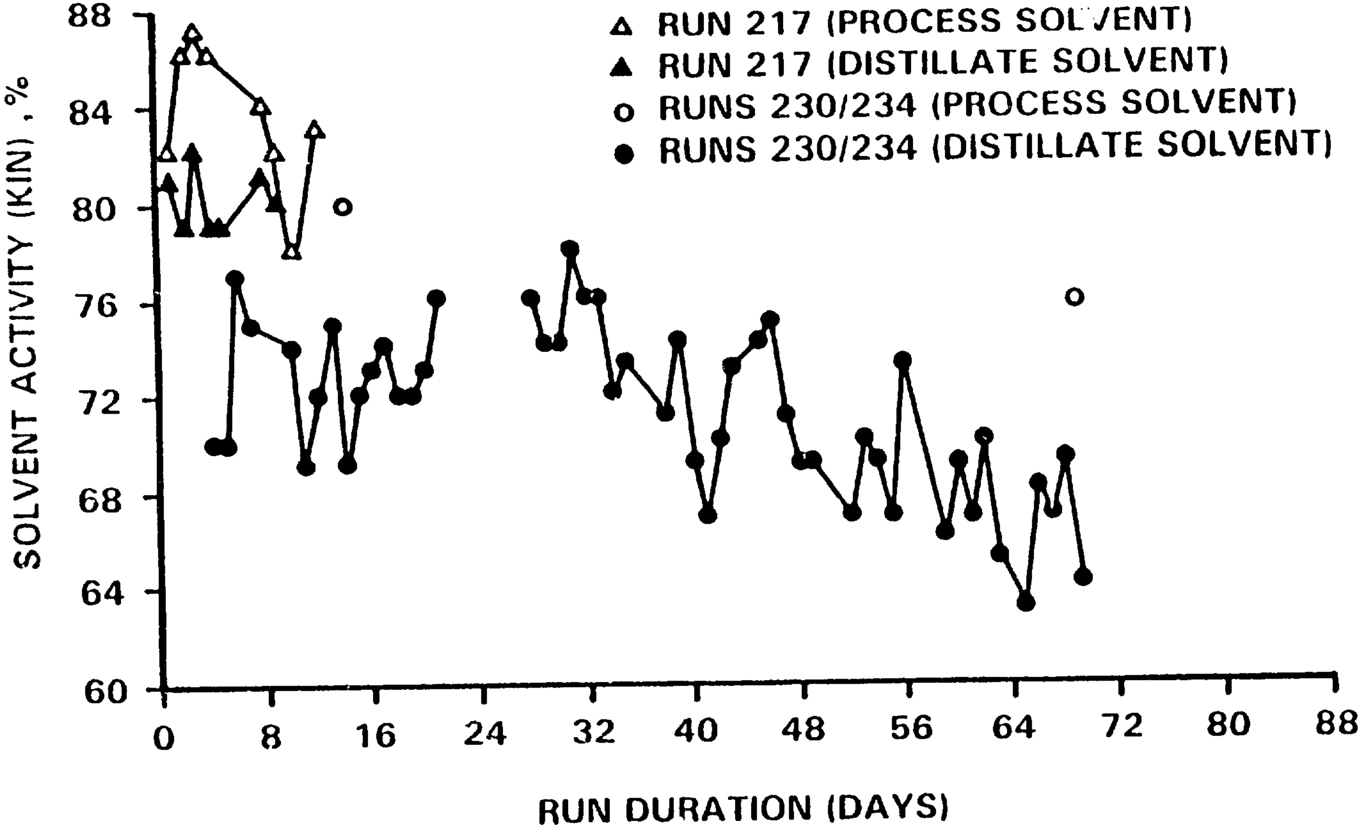


FIGURE 12. STEADY STATE SOLVENT ACTIVITY-LOW SEVERITY

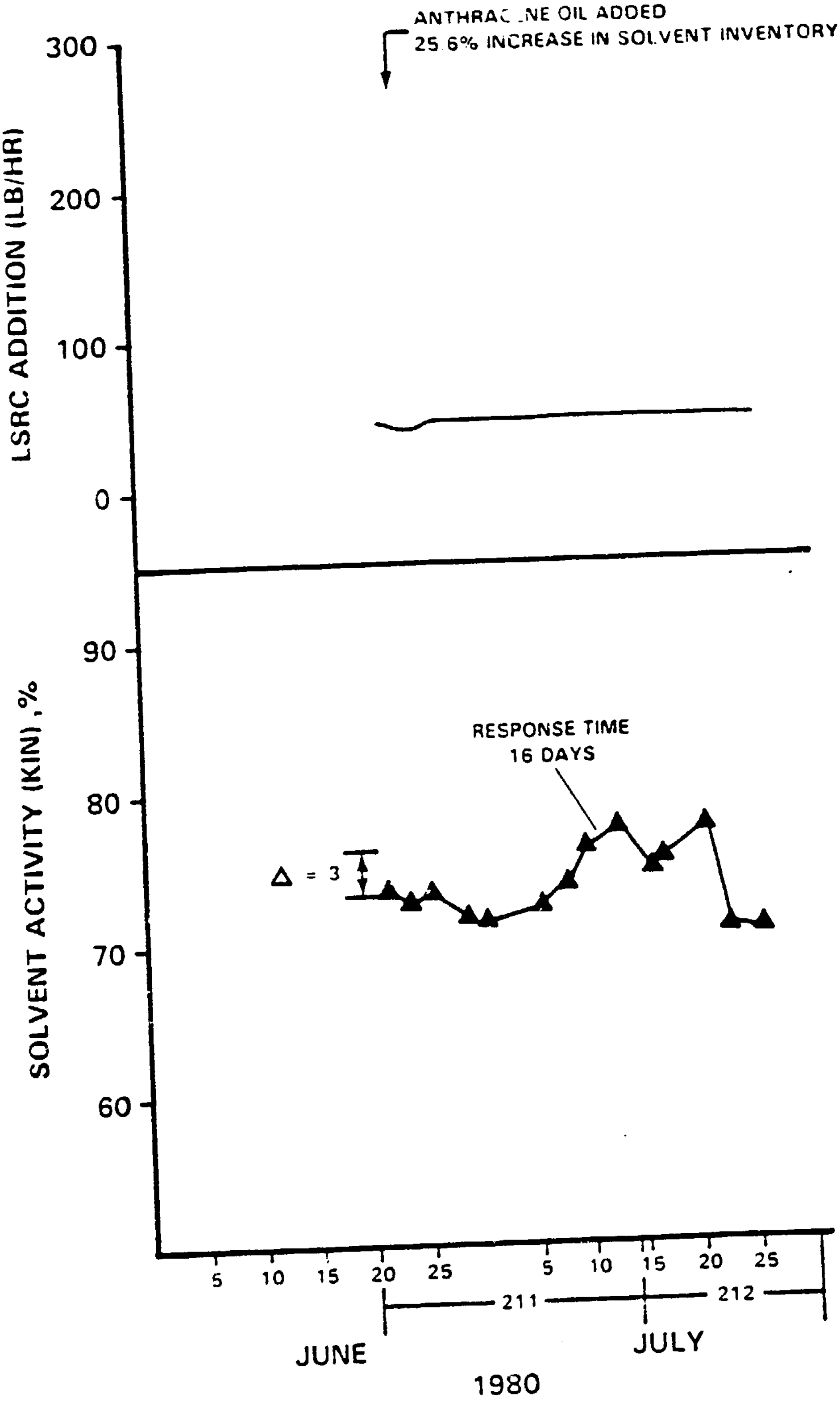


FIGURE 13. EFFECT OF ANTHRACENE OIL ADDITION ON SOLVENT ACTIVITY

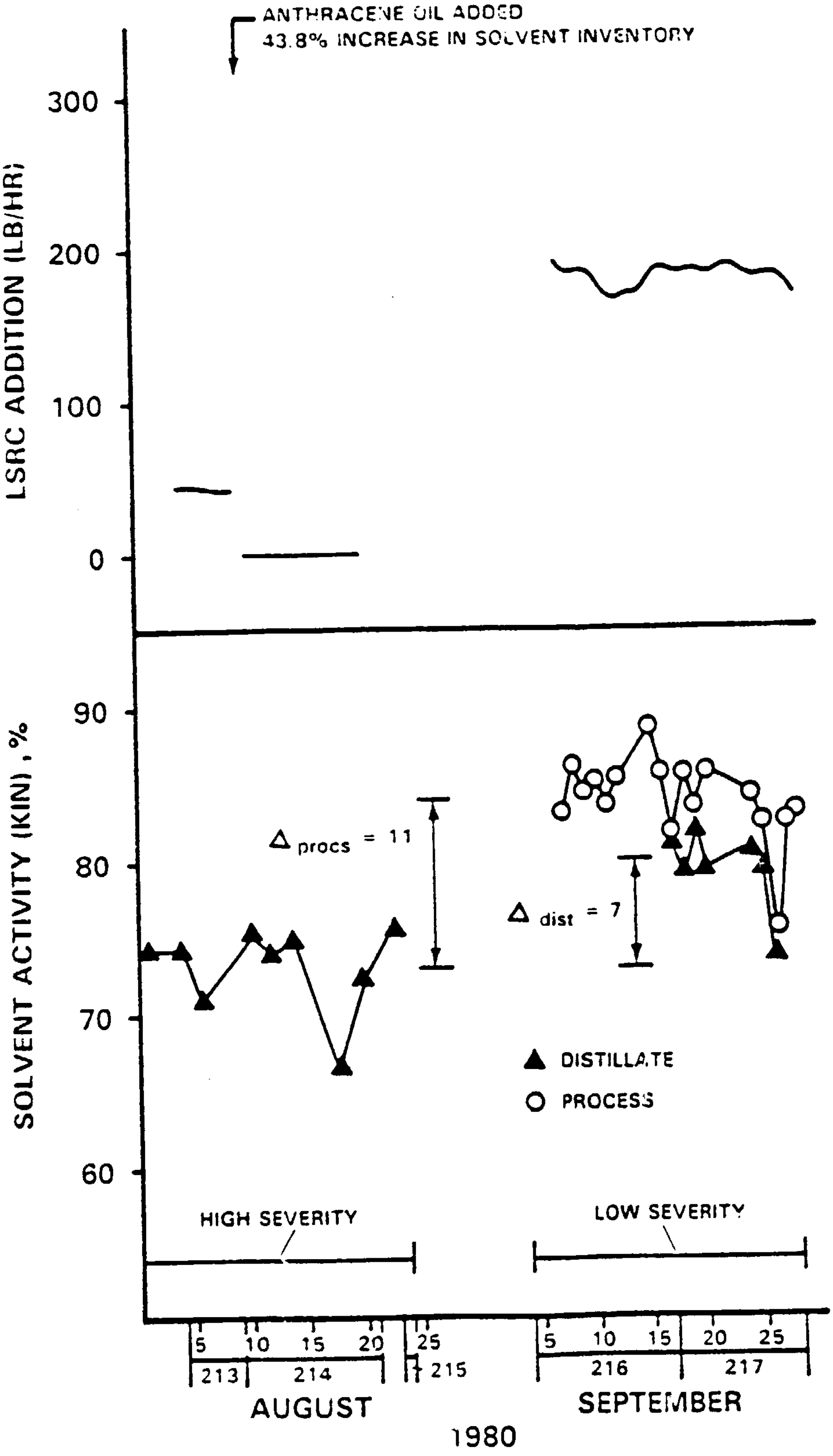


FIGURE 14. EFFECTS OF ANTHRACENE OIL, LSRC ADDITION, AND CHANGES IN SEVERITY ON SOLVENT ACTIVITY

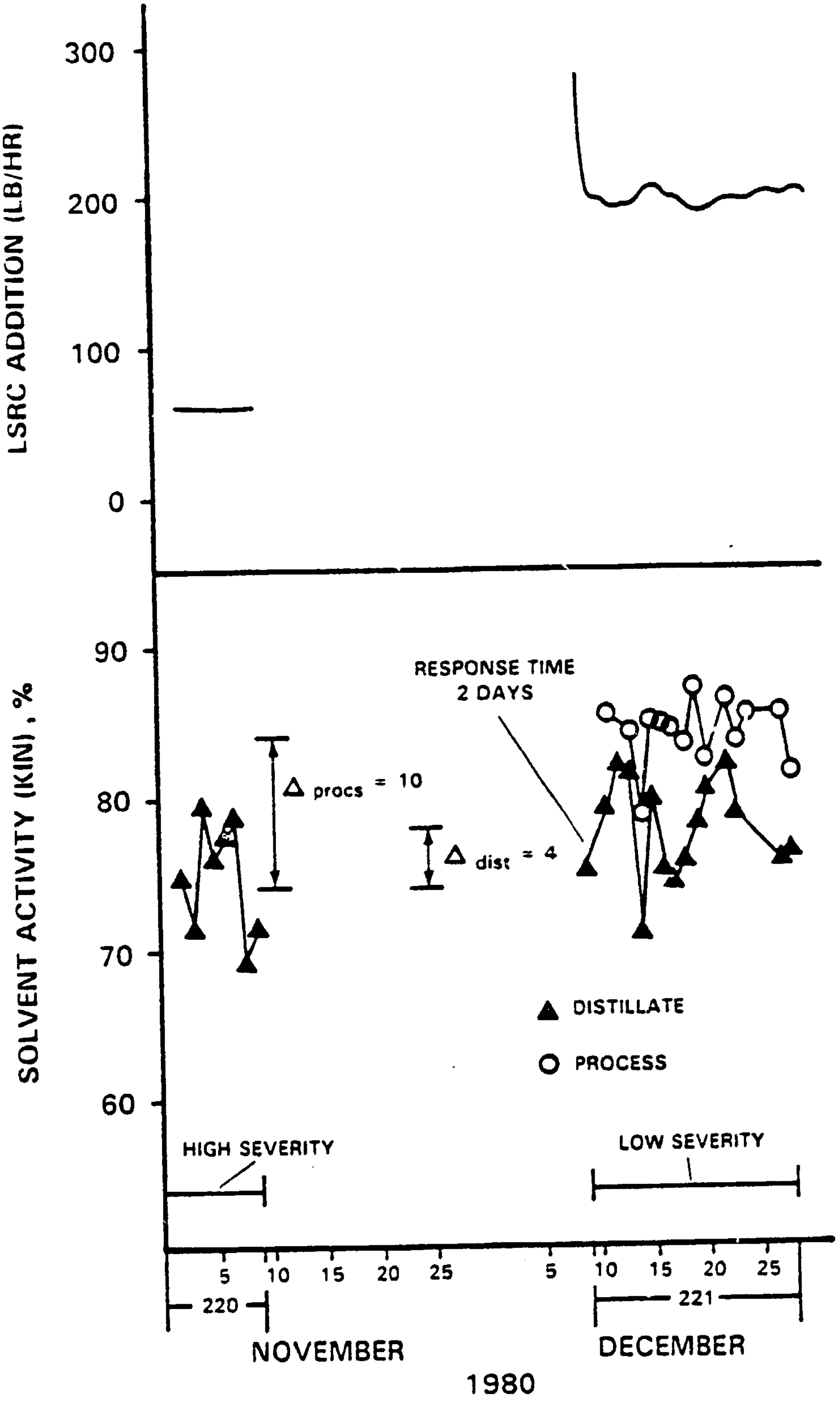


FIGURE 15. EFFECT OF LSRC ADDITION AND CHANGES IN SEVERITY ON SOLVENT ACTIVITY

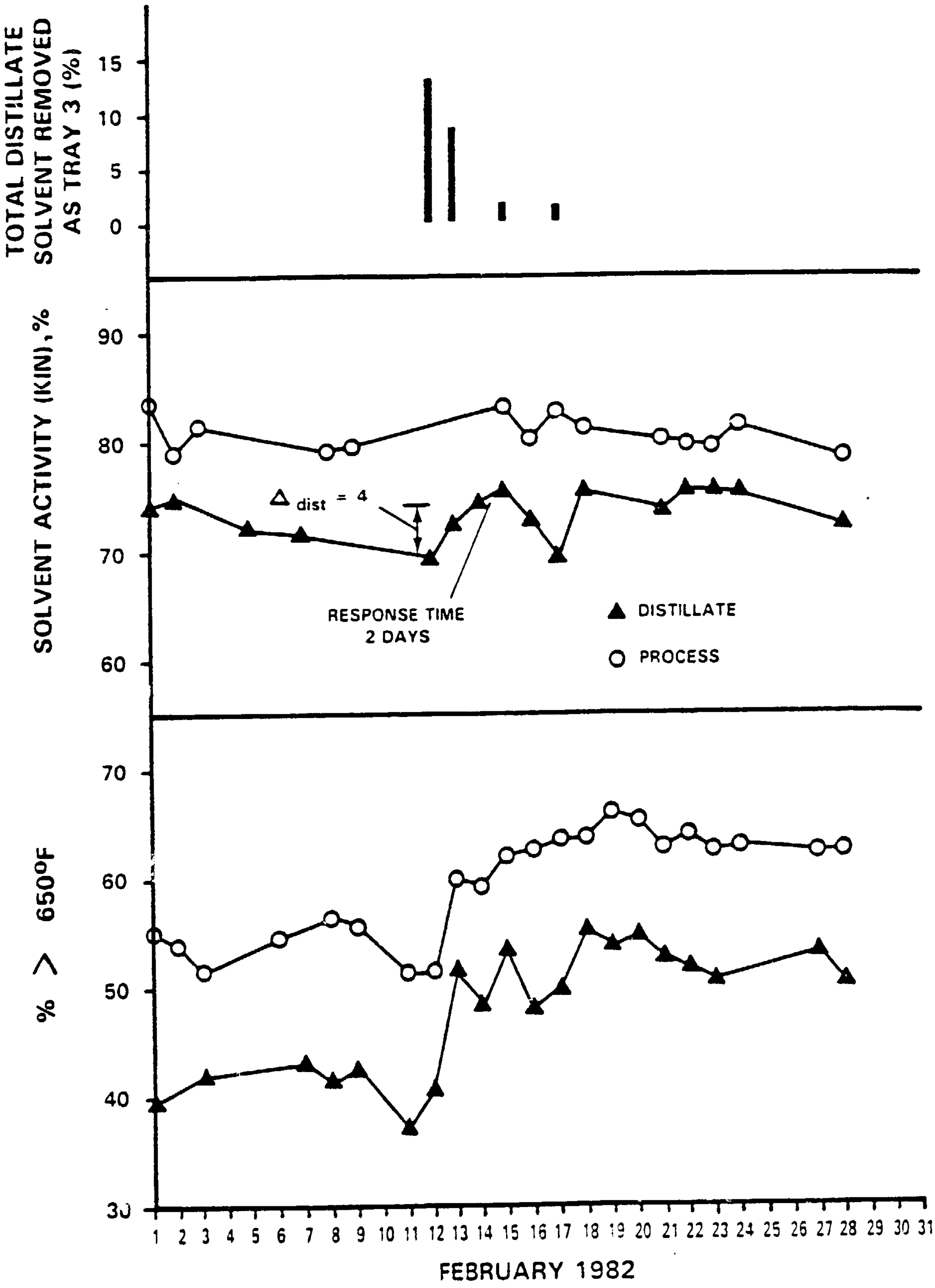


FIGURE 16. CONTROL OF SOLVENT ACTIVITY BY DISTILLATION

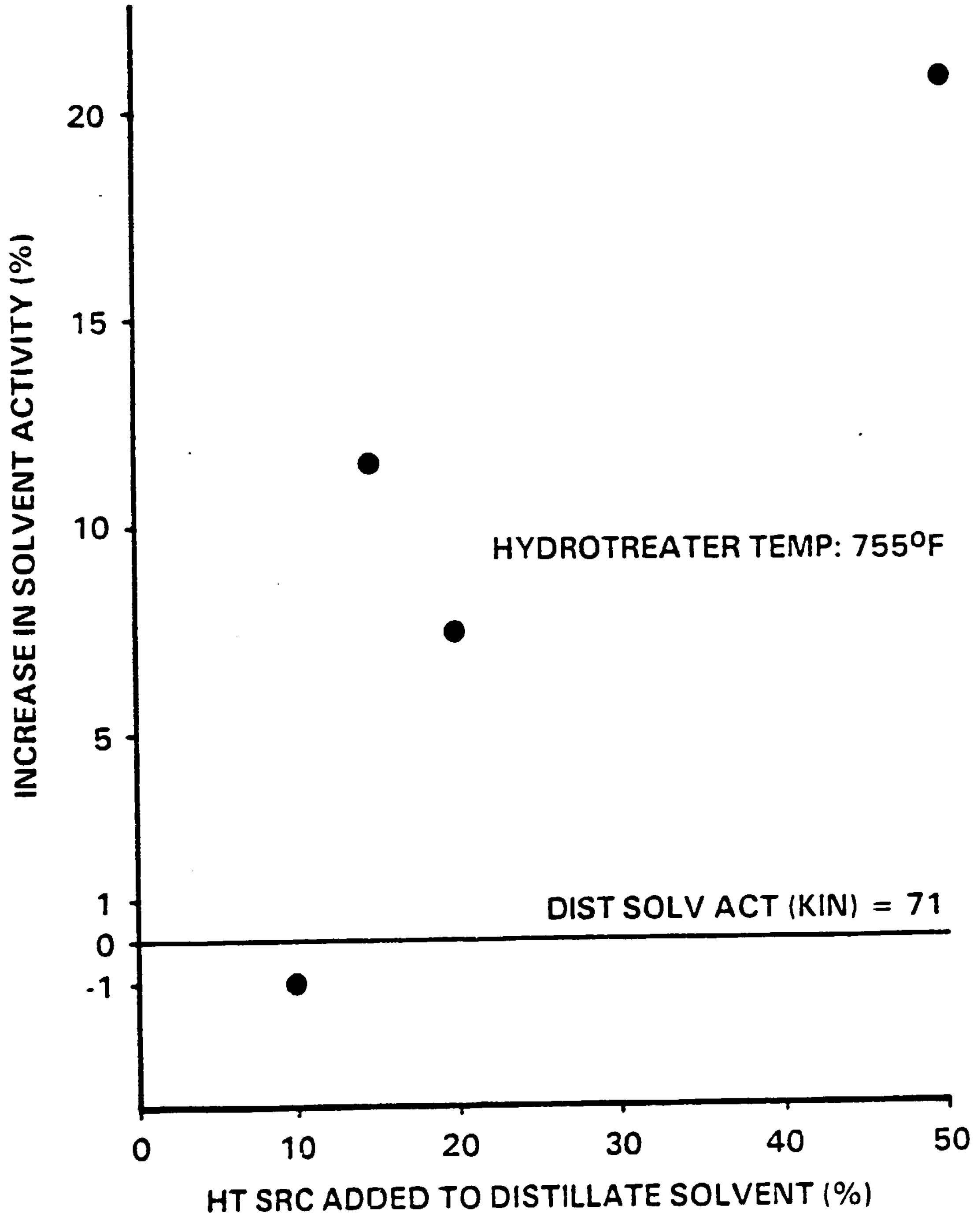


FIGURE 17. EFFECT OF HYDROTREATED SRC ON SOLVENT ACTIVITY

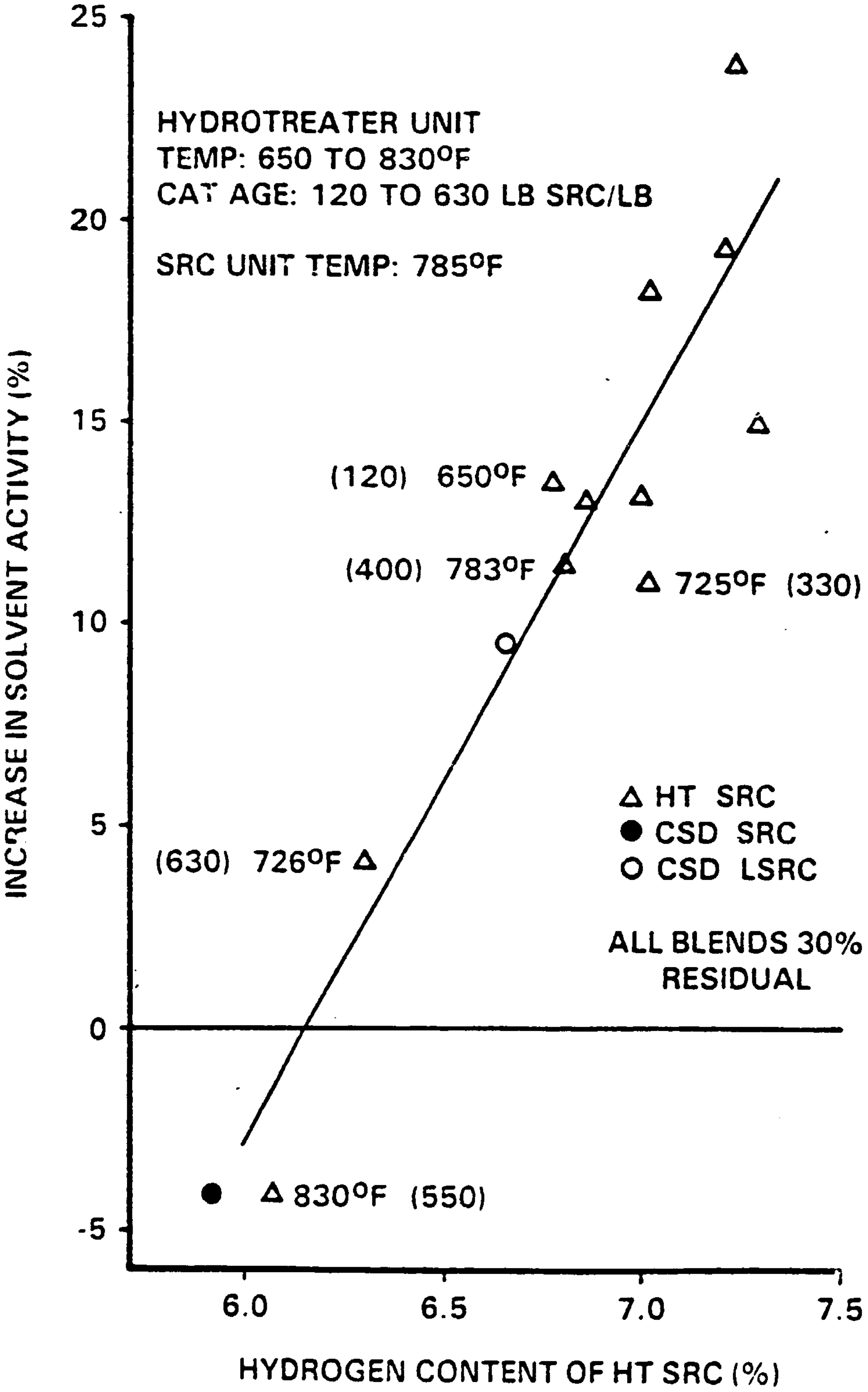


FIGURE 18. EFFECT OF HYDROGEN CONTENT OF RESIDUALS ON SOLVENT ACTIVITY

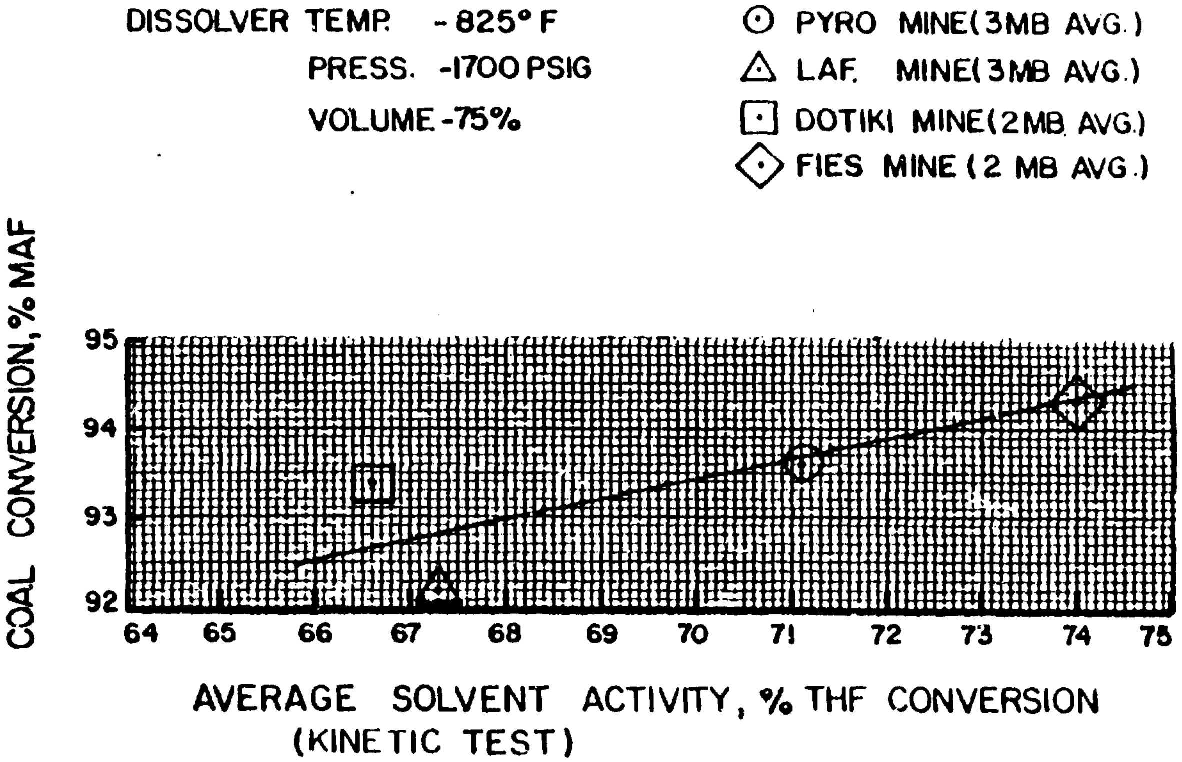


FIGURE 19. SOLVENT ACTIVITY VERSUS COAL CONVERSION

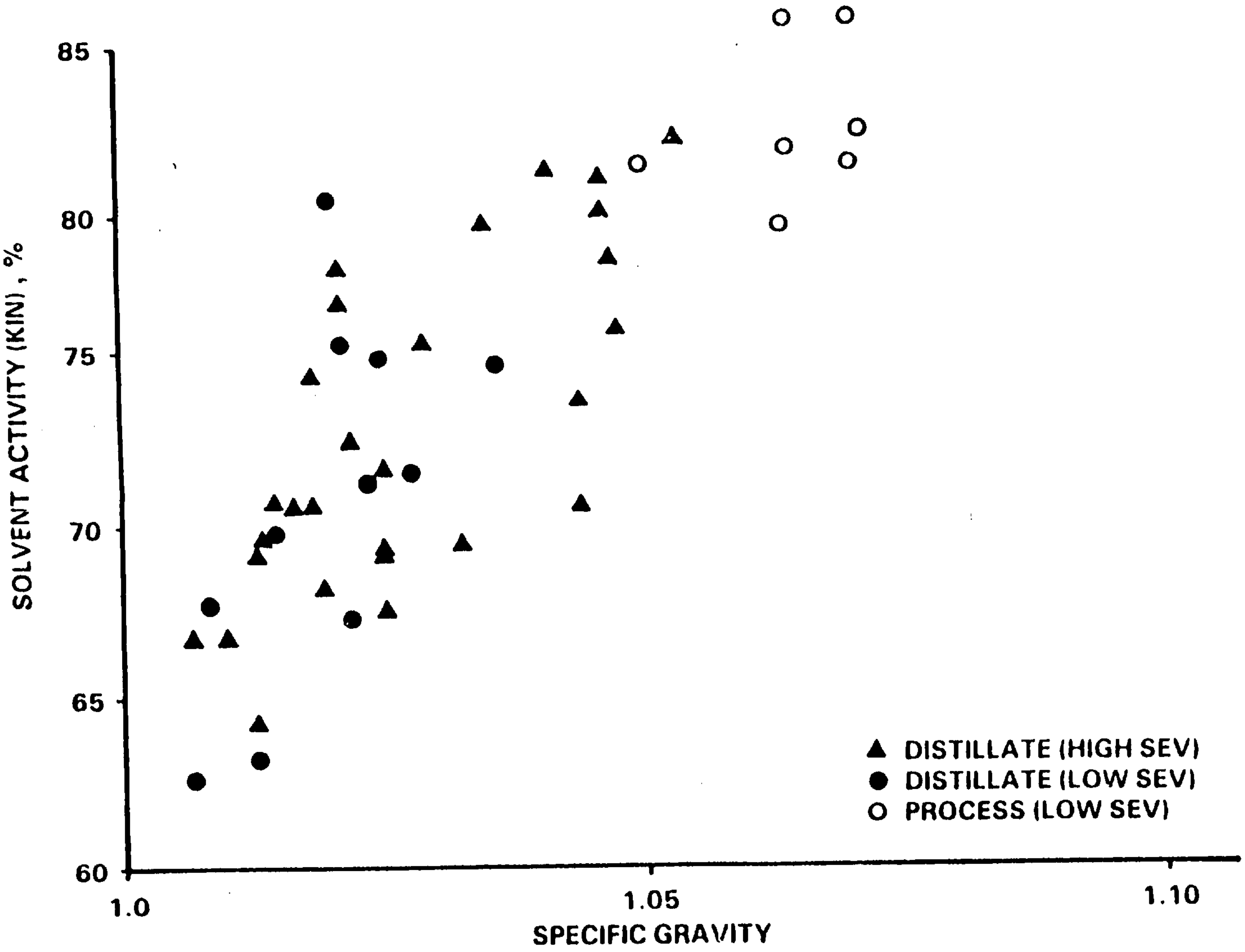


FIGURE 20. SPECIFIC GRAVITY VERSUS SOLVENT ACTIVITY

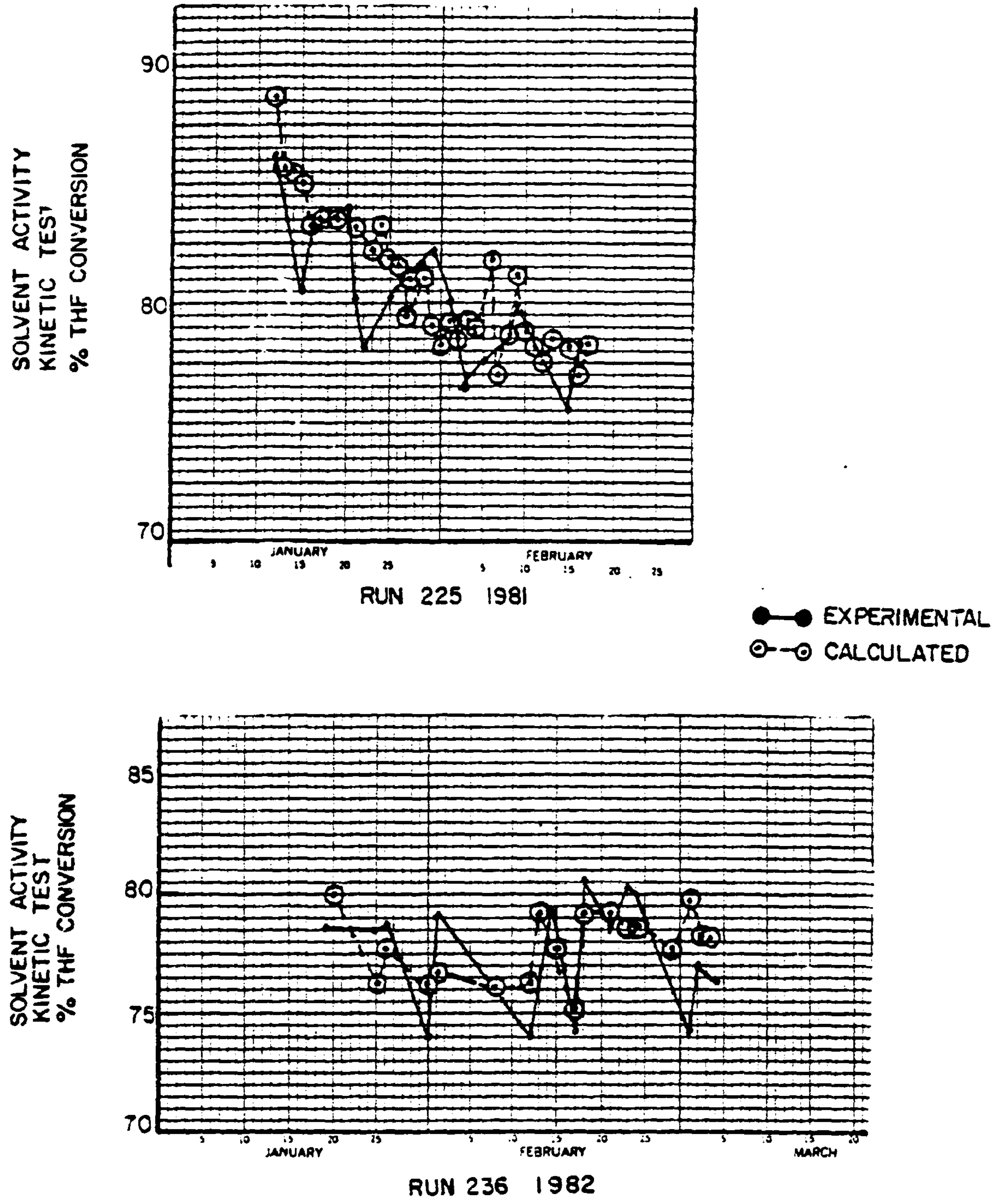


FIGURE 21. A COMPARISON OF SOLVENT ACTIVITY TRENDS WITH SOLVENT ACTIVITY TRENDS CALCULATED USING DISTILLATE SOLVENT SPECIFIC GRAVITIES IN A LEAST SQUARES EQUATION

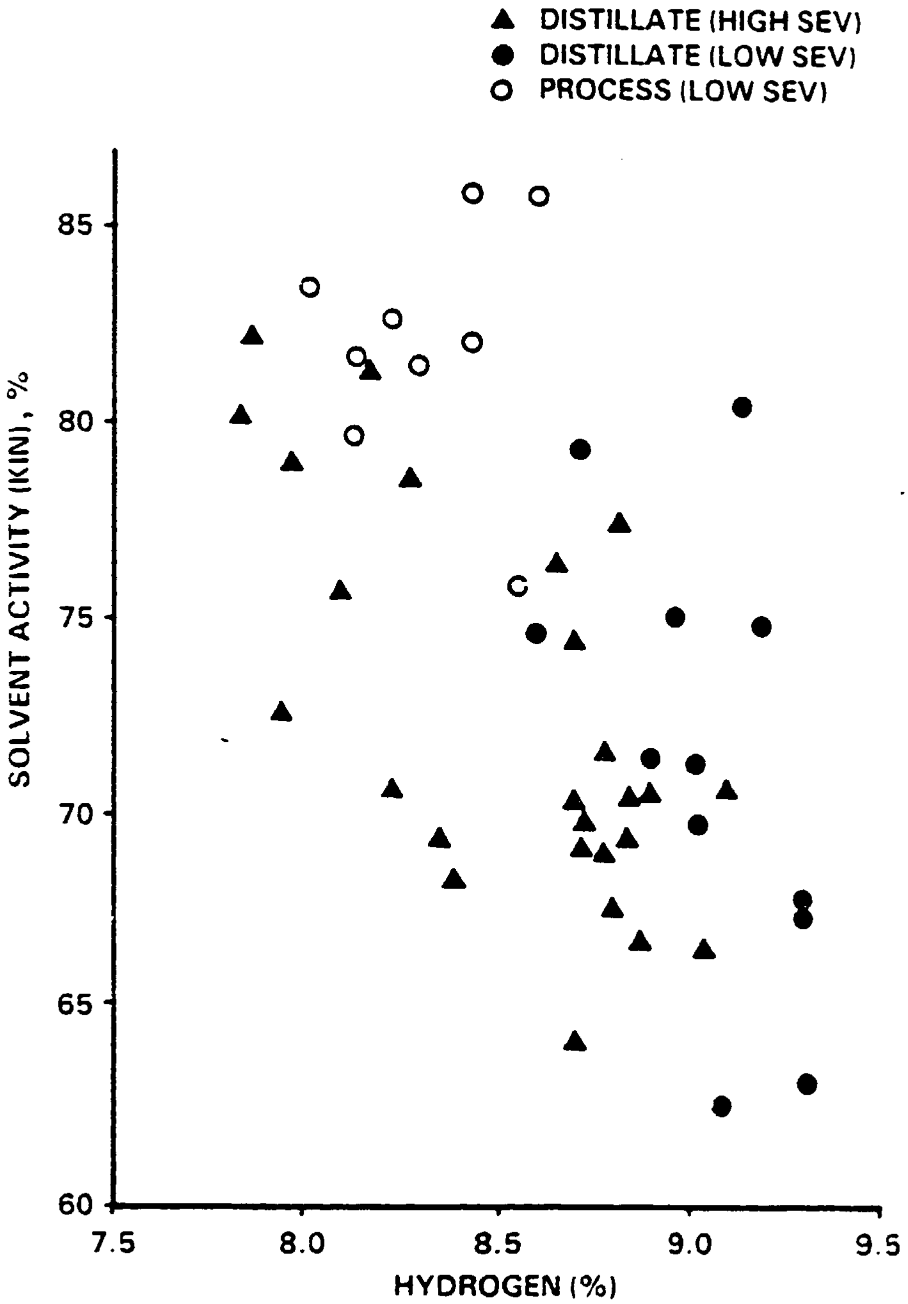


FIGURE 22. HYDROGEN CONTENT OF SOLVENT VERSUS SOLVENT ACTIVITY

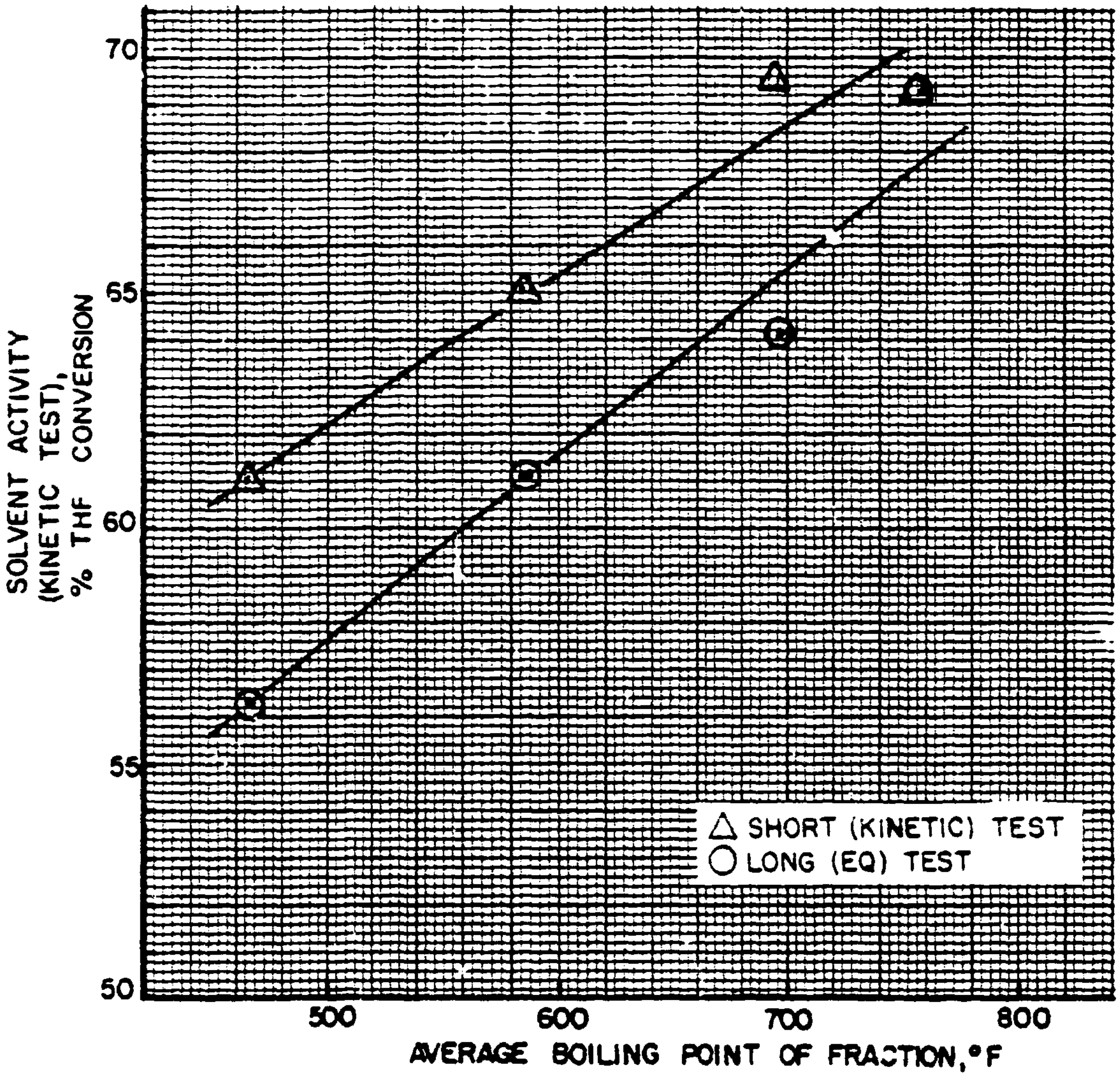


FIGURE 23. SOLVENT ACTIVITY VERSUS AVERAGE BOILING POINTS OF DISTILLATE FRACTIONS

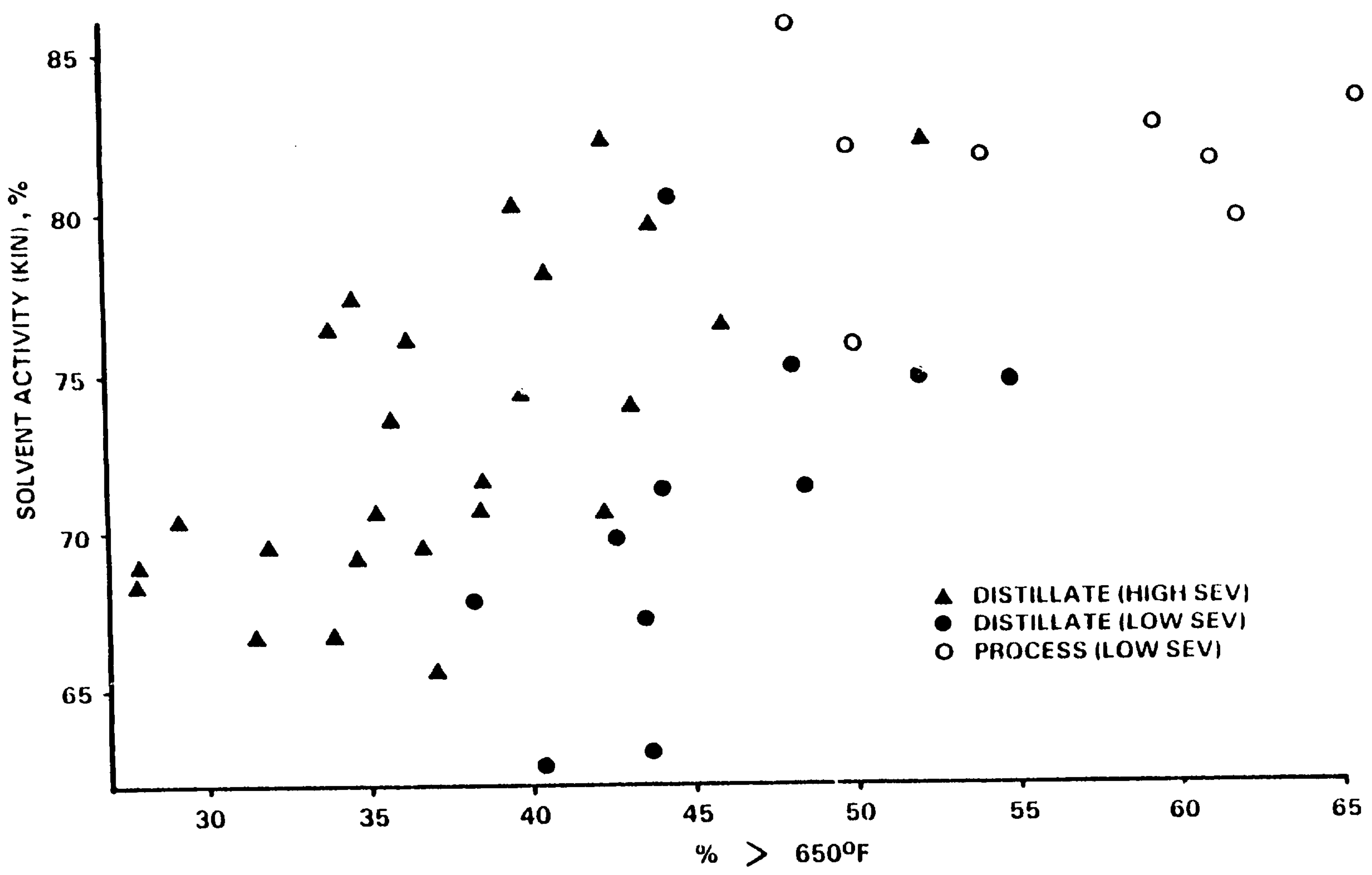


FIGURE 24. +650°F FRACTION IN SOLVENT VERSUS SOLVENT ACTIVITY

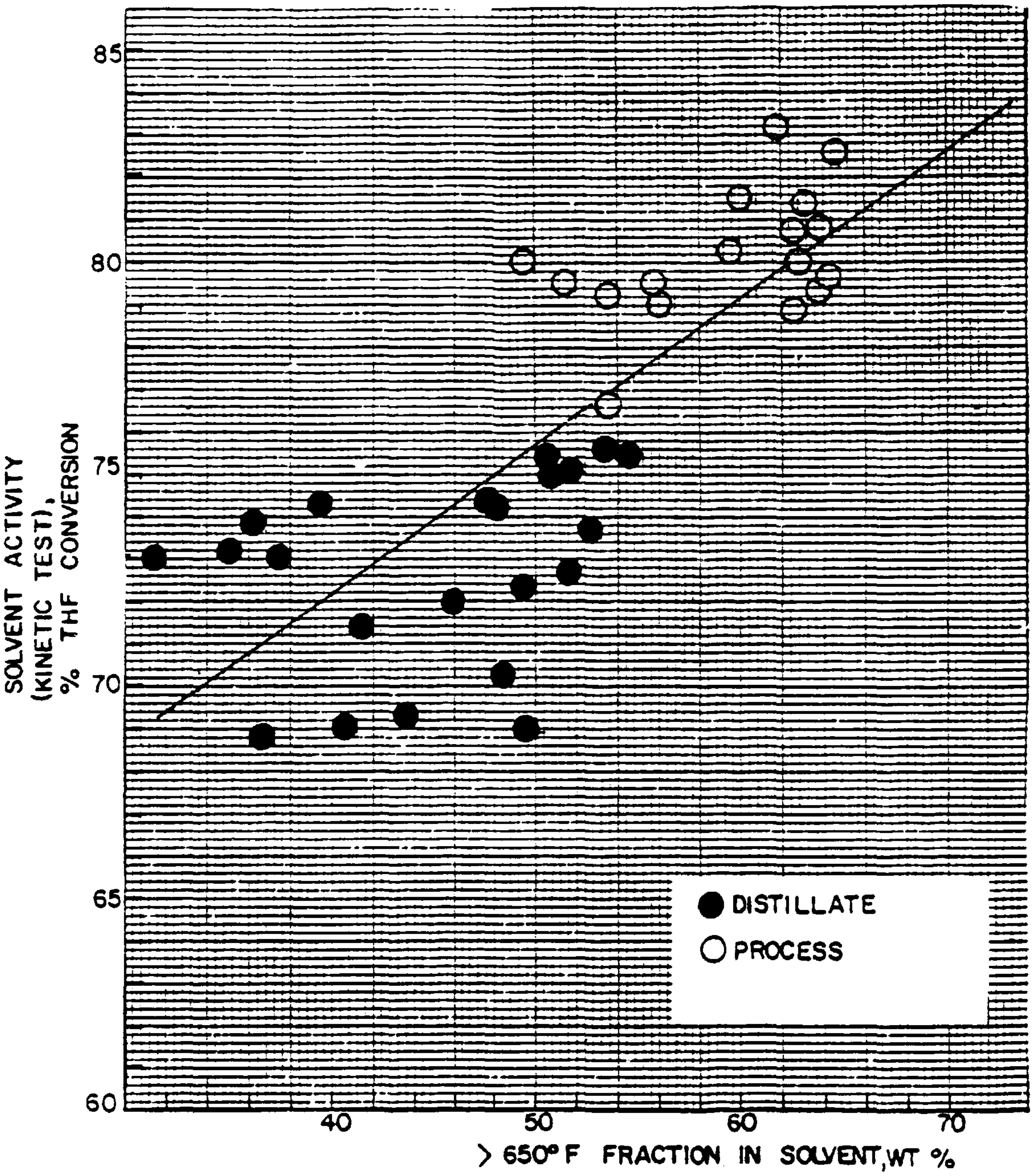


FIGURE 25. 650°F FRACTION IN SOLVENT VERSUS SOLVENT ACTIVITY (RUN 236)