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SOLVENT REFINED COAL (SRC) PROCESS

Quarterly Technical Progress Report, January—March 1979

February 1980

Work Performed Under Contract No. AC01-76ET10104

The Pittsburg & Midway Coal Mining Company
Denver, Colorado



U. S. DEPARTMENT OF ENERGY

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QUARTERLY TECHNICAL PROGRESS REPORT
FOR THE PERIOD
JANUARY, 1979 THROUGH MARCH, 1979

THE PITTSBURG & MIDWAY COAL MINING CO.
DENVER, COLORADO 80222

FEBRUARY, 1980

PREPARED FOR THE UNITED STATES
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
UNDER CONTRACT DEAC-0179-ET-10104
(Formerly EX-76-C-01-0496)

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A B S T R A C T

This report summarizes the progress of the Solvent Refined Coal (SRC) Project by The Pittsburgh & Midway Coal Mining Co. for the Department of Energy for the period January 1, 1979 to March 31, 1979. Activities included the operation and modification of the Solvent Refined Coal Pilot Plant at Fort Lewis, Washington; the Process Development Unit P-99 at Harmarville, Pennsylvania; and research at Merriam Laboratory in Merriam, Kansas. The Pilot Plant processed Powhatan No. 5 Coal in the SRC-II mode of operation studying the effect of coal particle size and system temperature on coal slurry blending and the effect of carbon monoxide concentration in the reaction feed gas on process yields. January and February were spent completing installation of a fourth High Pressure Separator on Process Development Unit P-99 to better simulate operating conditions for the proposed Demonstration Plant. During March, one run was completed at P-99 feeding Pittsburgh Seam Coal from the Powhatan No. 5 Mine. Merriam investigations included a study of the effect of iron containing additives on SRC-I operation, the addition of carbon monoxide to the feed gas, utilization of a hydrogenated solvent (Cresap process solvent) in the SRC-I mode under both normal and short residence time operating conditions, and development of a simulated distillation technique to determine the entire boiling range distribution of product oils.

I. Summary

During the period from December 25, 1978, to March 24, 1979, 693 tons of Powhatan No. 5 coal were processed in 654 hours of SRC II operation. Target raw coal feed rate was 2140 lb/hr and target slurry composition was 30% coal and 45% total solids. Operating conditions with a full dissolver were 2000 psig total pressure, 850° to 855°F outlet temperature and 86% to 90% hydrogen in the gas feed. The feed coal for most of this period was ground to approximately 98% minus 30 mesh and contained about 45% minus 200 mesh as compared to normally ground coal of about 80% minus 200 mesh.

In the early part of the period, there were numerous major curtailments which were attributed to processing operations from December 19 to December 26, 1978, when coarsely ground coal with a nominal size of minus 1/8 inch was used. The use of this coal appears to have resulted in substantial accumulation of heavy material including coarse mineral particles in piping and vessels throughout the slurry handling systems. Problems with plugging in slurry control orifices were frequently encountered. In most cases, the substance which was found in these orifices was a gravel-like accumulation associated with heavy hydrocarbon material. Frequent attempts to start up the pilot plant using normal 200 mesh pulverized coal were disrupted by such plugging incidents which indicated that this coarse accumulation was not being removed by normal flushing procedures.

In addition to the problems with coarse particles, record cold weather was experienced during this period which resulted in numerous frozen lines in the utility and gas systems as well as in the slurry processing system.

By the latter part of January, the pilot plant was operating reasonably well on minus 30 mesh coal. One SRC-II material balance run, 79SR-30, was completed at conditions similar to those proposed for the SRC-II Demonstration Plant. These conditions duplicated previous material balance runs which had been made with normal 200 mesh pulverized coal and the yields were comparable to previous results.

Separate experimental runs were conducted to determine the effects of increased concentrations of carbon monoxide in the reaction feed gas. Analyses of gas balance data from experiments conducted with concentrations of 5% and 10% carbon monoxide indicated that increasing feed carbon monoxide concentration increased carbon monoxide consumption and increased carbon dioxide production. These results were consistent with carbon monoxide consumption by a water gas shift mechanism.

Tests were conducted during February to determine whether coal slurry blending would be feasible at temperatures higher than the 350°F normally used. The results of the tests indicate that high temperature coal slurry blending is feasible, at least up to a temperature of 400°F with coal ground to a particle size distribution of smaller than 30 mesh. This study also showed the benefits

of feeding coarser particles; it was not possible to operate the coal slurry blending system at temperatures in excess of 370°F with a smaller particle size distribution, while operation with the larger particle size at 395°F was possible.

The Fort Lewis Pilot Plant continued to operate in the SRC-II mode processing Powhatan No. 5 coal until midnight February 27. Also on February 27, raw solvent was introduced into the feed for conditioning. Processing of raw solvent continued, interrupted by plugging in the downleg of the new Vacuum Flash Drum, until March 1 when a fire occurred at the Filter Feed Flash Vessel Recirculation Exchanger. Turnaround activities for conversion to SRC-I mode operation and for testing of the Lummus Antisolvent Deashing Unit began March 2.

During the turnaround, the spool piece was interchanged on Dissolver A for half dissolver operation and the top and middle quench lines were removed from the dissolver head. The bottom quench line was removed, cleaned, and replaced. A coke trap was installed upstream of High Pressure Flash Drum Level Control Valve 166A. All hydrogen lines were chemically cleaned to remove any buildup which might have contributed to recent compressor difficulties. Inspection of the inlet and outlet carbon steel lines of the Recycle Stripper indicated substantial thinning. Replacement of these lines with stainless steel required an extension of the shutdown since this work had not been anticipated. In the slurry preparation area, the pulverizer classifier section was replaced to return to operation with nominal 200 mesh coal. Dowtherm side-arm exchangers on the Filter Feed Flash Vessel and Precoat Slurry Pressure Vessel recirculation loops were rebuilt. A new reboiler bundle was installed in the Light Ends Column, the reboiler bundle of the Wash Solvent Column was repaired by plugging six tubes and a new reboiler shell of 316L stainless steel was installed. A new reboiler bundle of 317 stainless steel has been ordered for the Wash Solvent Column.

The new Slurry Preheater was received in the latter part of March. The foundation for this unit is in place and orders have been placed for the necessary equipment items. The target date for installation is August 1.

The waste treatment area operated at a sub-standard level throughout the reporting period. Problems were encountered with the biological Oxycontact Unit and phenol concentration in the effluent remained high in spite of continuous use of the charcoal filters.

Installation of the fourth High Pressure Separator on Process Development Unit P-99 was completed by mid-February. This new separator is located between the old Intermediate and Cold Separators and, like the Cold Separator, is designed to recover both an organic and an aqueous phase. The rest of February was spent in coming back on-stream and getting the new separator system working. During March, one run, Run 49, was completed feeding Pittsburgh Seam Coal from the Powhatan No. 5 Mine. Although the conditions

for Run 49 were quite similar to those for Run 48, except for a slightly higher recycle solids level in Run 48, distillate yield was lower and bottoms yield was higher for Run 49.

During the first quarter of 1979, activities at the Merriam Laboratory included a series of runs investigating the effect of iron containing additives on SRC-I operation. Pittsburgh Seam coals from the Blacksville No. 1 and Loveridge Mines were used. Addition of low levels (5-10 percent) of carbon monoxide to the feed gas was also studied while processing Pittsburgh Seam coal from the Powhatan No. 5 Mine in the SRC-II mode. The use of Cresap hydrogenated process solvent was investigated in SRC-I experiments under both normal and short residence time operating conditions with Kentucky 9/14 coal. Finally, development of a simulated distillation technique was initiated to determine the entire boiling range distribution of product oils.

II. Pilot Plant Operations, Engineering, and Maintenance

A. Coal Receiving and Preparation (Area 01)

Between December 25, 1978, and March 24, 1979, 693 tons of Powhatan No. 5 Coal were processed in 654 hours of SRC-II operation. The hourly on-stream factor, adjusted for downtime not directly caused by process problems or equipment failures, was 43.1%. A coal processing summary covering the first quarter of 1979 and overall process operating histories is shown in Table A-1.

Primary operating objectives during this period were to process coal which had been ground to approximately 98% minus 30 mesh. This ground coal had 45% minus 200 mesh as compared to normally used ground coal of about 80% minus 200 mesh. Target raw coal feed rate was 2140 lb/hr and target slurry composition was 30% coal and 45% total solids. One 28-hour material balance run was made on January 28-29. No unusual operating problems were attributed to feeding the minus 30 mesh coal, but on March 1 a leak and fire in Area 03 resulted in a plant shutdown and a decision to conduct a plant turnaround prior to switching to SRC-I operation using the Lummus Deashing System. The plant was down for turnaround through March 24, the end of the reporting period.

During plant operations in January and February, Area 01 problems caused approximately 147 hours of lost production. Two major outages accounted for most of this time. The first occurred when the slurry circulation lines plugged on January 17. The plugged lines were broken, then cleared with steam. The Slurry Blend Tank was also opened and cleaned at this time. Laboratory analyses on the material which plugged the lines showed it would dissolve in heavy distillate in a matter of minutes, had a low melting point, and had low pyridine insolubles. Clearing the plugged piping in Area 01 caused 55 hours of curtailment.

The second major outage occurred on February 25 during a period when attempts were made to determine the maximum operating temperature of the slurry blend system. Throughout this period, the preheater pumps required frequent flushing to reduce the tendency of the pumps to plug and pump speed to fluctuate. On February 5, both charge pumps failed and recycle slurry flow to the slurry mix system was reduced to prevent the blend tank from overflowing. Unfortunately, the recycle flow was then lost when the recycle air-cooled exchanger plugged and could not be cleared using normal procedures. During the subsequent shutdown, the bottom two rows of tubes in the exchanger were bypassed and the upper row of tubes, which had not been in service at the time and were therefore clear, were tied into the system. This change was made to minimize downtime and, more importantly, to improve control of recycle slurry temperature in order to achieve smooth operation at Slurry Blend Tank temperatures greater than 350°F. The plugged slurry recycle cooler caused 38 hours of downtime in this incident and 5 hours on February 21, again due to plugging. In the second case, the plug was cleared with a high pressure pump, flush solvent, and steaming.

About 4 1/2 hours of curtailment were required to remove the recurring buildup of granular, compacted material on the charge pump suction ballcheck valves during operation of the blend tank at elevated temperatures. During a plant shutdown on February 13, caused by problems in Area 04, the slurry system was modified by routing all the flow through the charge pump suction manifold instead of splitting flow at the suction manifold inlet. The resultant higher flows through the suction manifold appeared to reduce the settling of granular material in the suction manifold, but did not eliminate the buildup on the suction check valves.

Nine hours of curtailment occurred on January 17 when FCV-1152 in the slurry recirculation line plugged. Analysis of the material removed from the valve indicated 86 percent pyridine insolubles which appeared to be primarily gravel. Determination of the pyridine insoluble particle size showed that 83.9 percent passed 12-mesh, but only 15.8 percent passed 30-mesh. Plugging of this valve was therefore suspected to be a direct consequence of the previous processing of minus 1/8 inch coal.

Previous operation with minus 1/8 inch coal from December 16 through December 26 had resulted in damage to several of the charge pump ball checks. Since some of the balls appeared to be in better condition than others, hardness tests were run on two damaged and one undamaged ball. The two damaged balls had a Rockwell C hardness of 60, but the undamaged ball had a hardness of 66. The 200 chrome specification allows a hardness variation of 63 to 65. In addition, the damaged balls were visibly out of round. It is believed that at least part of

the problems with the ball checks experienced during the minus 1/8 inch run are attributable to these out-of-specification ball checks. Subsequently, fifteen new balls were examined for roundness and eight were found to be beyond specification. Purchasing has changed suppliers in order to assure that only specification balls are obtained in the future.

Excessive erosion was again noted in the No. 3 circulation pump which had to be removed from service February 21 to replace the impeller and the case. The case had only 0.085" of metal remaining and the impeller was almost completely eroded away. Service life of this pump was approximately 28 days with the minus 30-mesh grind. Previous service life had been 6 to 12 months with the normal 200-mesh grind, but only 3 to 5 days with the minus 6-mesh (1/8") grind.

Problems in maintaining the firing of the inert gas heater because of an unreliable flow meter led to a decision to try a short test using cold (100°F) carrier gas in the circulation loop. During the test, which was conducted February 20 and 21, the moisture content of the coal feeding the process increased from approximately 0.5% to approximately 2%. A noticeable increase of water in the slurry blend tank vent condensate system and a decrease in the horsepower requirement for the mix tank agitator were observed.

Plugging in the coal feed system was encountered throughout the operating period and totaled about nine hours of outage. Fifty-three incidents of plugging in the chute between the coal surge bin and the gravimetric feeder were recorded as well as nine cases of plugging at the variable-drive coal auger. The coal feed chute to the new mix tank plugged once and the slurry mix tank overflow line plugged three times. The slurry mix tank agitator bound once and stopped rotating.

The final phase of SRC II processing with Powhatan No. 5 coal was completed at midnight February 27. At that time, raw solvent was introduced into the feed for conditioning. Coal injection was stopped when a fire occurred at the Filter Feed Flash Vessel recirculation exchanger on March 1 and the shut-down continued through the end of the reporting period.

Inspection, modification, and maintenance work in Area 01 included:

1. Reinstallation of the classifier in the pulverizer and adjustment of the roller-race spring tension to its normal setting to enable production of the normal 200-mesh coal grind.
2. Relocation of the gravimetric feeder to eliminate some of the plugging problems which were experienced with the supply chute.

3. Unplugging the lowest of three rows of tubes in the recycle slurry air cooled exchanger using a portable high pressure pump. This exchanger is oversized and ordinarily only one-half of the top row of tubes is in service. Piping was installed during this shutdown to allow the optional use of the lowest row of tubes. The middle row is piped to steam to keep other tubes hot when off line.
4. Ultrasonic testing of the slurry circulation line (3" SL-87) from the circulation pumps to the new mix tank. Some metal loss has occurred and this line may require replacement during the annual shutdown.

B. Slurry Preheating and Dissolving (Area 02)

Area 02 was on-stream for 37 days during the January-March, 1979 reporting period. All operation was in the SRC-II mode using a full dissolver at a pressure of 2000 psig. The dissolver outlet temperature was maintained between 850° and 855°F. Hydrogen content of the feed gas was 86% to 90%, except during tests to determine the effects of increased concentrations of CO in the hydrogen recycle stream. Hydrogen purity then dropped to 75-80% as CO levels increased to 10%.

Area 02 problems were especially severe in the early part of the period and accounted for about 410 hours of curtailment during January. The majority of these problems were related to plugging of slurry lines and control valves. This plugging was directly attributed to the processing of minus 1/8 inch coal from December 19 through December 28, 1978. Operations were reasonably good during February, but on March 1 a fire in Area 03 caused a plant shutdown and the plant was down for turnaround for the remainder of the reporting period.

Plugging in the slurry transfer line (SL-13) caused several pressure buildups across the preheater coil during the period December 30 through January 2. To keep from backing slurry into the hydrogen lines, the system pressure was relieved by opening the isolation valve between the heater outlet and the flush solvent return system. After venting the heater outlet in this fashion twice, the vent line plugged. When the slurry transfer line plugged again on December 31, the remaining alternative was to reduce pressure in the reaction area through the pressure control valve on the recycle condensate separator and force the plug downstream. The plug was broken using a 1900 psi differential pressure, but the resulting mechanical stress displaced the transfer line approximately 8-10 inches away from its supports. The line was easily replaced but appears to have been permanently cold-sprung. Five incidents of plugging on January 2 finally forced a shutdown of the reactor area to hydroblast the line.

While the area was depressured, all the welds in line SL-13 were inspected by ultrasonic shear wave testing, but no faults were found. All Grayloc hubs and the line itself were inspected after hydroblasting. The inspection showed that the inlet nozzle to Dissolver A had approximately .015 inch metal loss on the 3/16 inch 347 stainless liner immediately downstream of the sealing surface as shown in Figure A-1. This may have been the result of using minus 1/8 inch coal, because it was not apparent in a routine inspection six months earlier. No corrective action is planned, however, the nozzle will be monitored in future shutdowns.

During the January 2 shutdown, the dissolver was opened for a volume measurement. Approximately 50-60 gallons of "rocks" in a thick slurry were dumped when the bottom Grayloc was removed. Essentially all of the "rocks", which resembled pea gravel, were too large to pass a 40-mesh screen. Selected analyses of this material are shown in Table A-2. Following subsequent operations on the unit using minus 30-mesh coal another sample of solids from the bottom of the dissolver was taken during the March shutdown and submitted for analysis. The results of this analysis are included in Table A-2 and show that the material was much finer than that found in the dissolver after using minus 1/8 inch coal.

The dissolver volume measurement showed 45 ft³ for one-half of Dissolver A, which represents no noticeable decrease in volume from normal values. Also during this time the bottom dissolver sampling quill, which had plugged previously, was removed and will not be reinstalled until some modifications are made to assure safe operation.

After circulation was restored January 6, both High Pressure Flash Drum level control valves (LCV-166A and B) plugged. Operations was able to clear LCV-166A by back-blowing from the Intermediate Pressure Flash Drum to the High Pressure Flash Drum, but LCV-166B had to be removed and cleaned.

On January 7 and again on January 9, a restriction in the Slurry Preheater Coil caused solvent to back into the hydrogen lines in Area 01. After the hydrogen lines were cleaned and blown dry with nitrogen, an attempt to begin circulation January 10 ended when LCV-166A and B plugged again. As before, LCV-166A was cleared by back-blowing, but the reaction area had to be depressurized to clear the heater coil and LCV-166B.

During this time, the High Pressure Flash Vessel was opened for inspection. Two of the upper baffles in the drum were dislodged from their support rings, and two of the lower baffles were in place but split in the middle and pushed upward. All four baffles, which are above the inlet nozzle, were replaced. In addition, there was a large deposit of 3/4 inch to 1 inch chunks of coke in the bottom head. A sample of this material was submitted for analysis and the

results are shown in Table A-3. The bottom corrosion rack was also completely fouled with this material. The High Pressure Flash Drum and the inlet and outlet lines to the Intermediate Pressure Flash Drum were hydroblasted to remove coke deposits and solids buildup. Maintenance also replaced the corrosion racks in the High Pressure Flash, the Intermediate Pressure Flash, and the Recycle Condensate Separator vapor spaces. A new double check valve assembly was installed in the hydrogen line to the Slurry Preheater inlet to prevent slurry from back-flowing into the hydrogen lines.

In this same period, the erosion/corrosion test loop on the dissolver effluent line was removed for inspection. The results of measurements on the loop are shown in Figure A-2 and indicate that there was no appreciable metal loss. During the March shutdown the erosion/corrosion test loop was reinstalled in the dissolver outlet piping.

Area 02 was repressured January 14, but a leak in the bonnet seal of a Walworth block valve between the dissolver and High Pressure Flash Drum prevented full startup. The bonnet of the Walworth block valve had originally been removed to facilitate hydroblasting of the piping in the area. To eliminate the necessity of removing this bonnet and the risk of having it leak, Engineering has designed an alternate hydroblasting access port.

On January 19, the area was depressurized to inspect for a possible leak in the preheater transfer line (SL-13) and to clear LCV-166A and B and LCV-175A and B, which were all plugged. After clearing the valves and pressure testing the heater outlet line, the system was flushed with solvent at 10,000 lb/hr to remove any traces of residue left from the minus 1/8 inch coal run. Startup was delayed again January 22 to repair a leak on a pressure transmitter fitting at the outlet of the preheater. It was this oil leak which had given the false impression January 19 that the heater outlet line was leaking. When circulation was re-established, LCV-175A and B plugged once again. LCV-175A was returned to service with taper trim in place of the standard Micro-Form trim to minimize trim breakage. LCV-166A then began plugging shortly thereafter, but was successfully cleared by back-blowing. Another delay was required to adjust the packing on the FCV-85 bypass valve.

On January 31, the Willis level control valve (LCV-166B) began leaking. Operation was switched to LCV-166A, in order to replace the trim in LCV-166B without depressuring the area. Upon switching operation to LCV-166A, it also plugged. Operations cleared this plug, but as it moved downstream it then plugged both 175A and B. In the process of trying to clear these valves, the trim in LCV-175B broke. The broken trim was changed under pressure, but replaced with a taper trim instead of the normal Micro-Form trim. About 12 hours of curtailment was involved in this work.

When the Willis control valve (LCV-166B) was repaired January 31, new valve discs made from tungsten carbide having less cobalt binder (K-602) were substituted for the Valenite 134 discs. The lower cobalt binder content discs were supplied by the manufacturer to determine if their use would improve trim service life. Service life did improve from 14 days to 16 days, but this increase is not considered to be significant.

Level control of the Recycle Condensate Separator was another problem during February. After LCV-172 began leaking February 3, control was maintained with a manual block valve until February 6, when the area could be depressurized to install new trim. Trim replacement, however, did not solve the problem. After the trim was inspected again February 13, it was determined that the bypass valve was leaking and that it had been responsible for the previous level control problems and not the level control valve itself.

On February 13, the reaction area was depressurized to repair a leaking safety valve (PSV-456) on the Intermediate Pressure Flash Drum. Repair of the safety valve required 25.3 hours of curtailment.

Area 02 continued to operate satisfactorily through March 1 and was shut down March 2 because of the problems in Areas 03 and 04. The following work had either been completed or was in progress as the end of the first quarter reporting period:

1. Piping on Dissolver A was switched to one-half dissolver (SRC-I) operation.
2. Both the top and middle dissolver quench lines were removed and the hydrogen lines capped. The bottom quench was removed for cleaning and reinstallation.
3. A 410 stainless steel sleeve was installed in the erosion/corrosion loop for high velocity slurry testing and the loop was reinstalled for service. The method of installation is shown in Figure A-3.
4. The bonnet was repaired on a 4-inch Walworth valve on the bypass line around the preheater outlet relief valve (PSV-404). Another 4-inch Walworth valve on the dissolver outlet line (4" SL-15) was repacked. Both jobs were done by ITT Henze, an outside contractor specializing in valve repair.
5. A permanent clean-out elbow was installed in the dissolver outlet line (4" SL-15) immediately downstream of the Walworth isolation valve.
6. The welded inlet nozzles on pressure relief valves for the high pressure and intermediate pressure flash drums and the preheater outlet were inspected for stress corrosion cracking by shear wave ultrasonic testing. None was

detected. However, these corrosion-susceptible 316 stainless steel nozzles will be replaced with a stabilized grade of stainless steel during the annual shutdown scheduled for July-August. Both pressure relief valves on the High Pressure and Intermediate Pressure Flash Drums (PSV 411 and PSV 456) were removed and reset.

7. Check valves were installed on the purge lines to the level transmitters on the High Pressure and Intermediate Pressure Flash Drums and the stripper. The design is similar to that installed downstream of FCV-85 in January. Springs were removed from the check valves before commissioning.
8. A new coke trap was installed upstream of the High Pressure Flash Drum level control valve LCV-166A. The trap consists of a cylinder containing a vertical baffle with a series of 1/8 inch diameter holes.
9. The slurry line from LCV-175 to Area 03 (2" SL-21) was inspected by ultrasonic testing and determined to be serviceable in spite of a significant metal loss. Results of the inspection are shown in Figure A-4. Plans are being made to replace the line with 316L stainless steel during the annual shutdown. The slurry line from LCV-175 to the Lummus Unit (1" SL-134) was also inspected by ultrasonic testing and found to be satisfactory. These inspection results are shown in Figure A-5.
10. Routine ultrasonic testing of the slurry recycle line (2" SL-114) from Area 01 to Area 02 showed significant metal loss, in some cases over half of the nominal wall thickness, as shown in Figure A-6. Further testing of the feed line to the stripper and the stripper bottom line to Area 03 (2" SL-118 and SL-110) also showed metal loss, but to a lesser degree. Results of the inspections on these lines are in Figures A-7 and A-8 respectively. Both SL-110 and 118 were completely replaced with 304L stainless steel; SL-114 was replaced with 304 and 316 stainless steel from Area 02 back to FE-1152 in Area 01. Fifty feet of SL-114 remain to be replaced during the annual shutdown.
11. The bypass valve on the Recycle Condensate Separator level control valve, LCV-172, was replaced in kind with a rebuilt 1-inch Rockwell 2500 psig globe valve. All block valves in this loop were also replaced.

C. Mineral Separation (Area 03)

On January 2, the Filter Feed Surge Pump was removed from service to repair a leak. The case and the impeller were severely worn and were replaced. In addition, the suction and discharge spools were replaced because of excessive thinning.

A new Lawrence pump, designed primarily to handle hot slurry, was installed for evaluation and for use as a spare.

Ultrasonic readings were taken on the Filter Feed Flash Vessel January 5, to determine if significant erosion had occurred during the processing of minus 1/8 inch coal. The results of this inspection are detailed in Figure A-9. Although no significant changes were detected on the vessel itself, the inlet recirculation nozzle showed increased erosion/corrosion and one reading showed a 30 percent loss of nominal wall thickness. Plans have been made to replace this nozzle as soon as possible.

On February 8, a leak caused by corrosion/erosion developed in the suction line of the Filter Feed Flash Vessel recirculation pump. Because of the severity of the leak, all lines in the vicinity of the leak were inspected. Although there was significant metal loss in the 6-inch suction line (Figure A-10), only the 4-inch suction line back to the 6-inch line was replaced with carbon steel. Piping in this loop will be inspected during the next turnaround and at that time, the 6-inch line may be replaced with austenitic stainless steel. As shown in Figure A-10, the corrosion/erosion is a maximum where the pipe reduces to 4-inches. The approximate liquid velocity in the 4-inch pipe is between 5 and 6.3 feet/sec; in the 6-inch pipe it is between 2.2 and 2.8 feet/sec. Corrosion, rather than erosion, is probably responsible for the metal loss in the 6-inch piping, since it occurred at low velocity.

The Recycle Process Water Tank was drained several times during the reporting period because of repeated buildup of emulsions in the tank. Engineering is currently looking at alternative processing methods to eliminate or reduce this and other oil/water separation problems.

Because of a leak and a subsequent fire March 1 at the Filter Feed Flash Vessel Recirculation Dowtherm Exchanger, coal feed was stopped and a planned turnaround was started. Inspection showed a pinhole leak at the inlet yoke and further inspection showed the entire tube side (slurry service) of the exchanger required replacement. All inner tubes and "U" bends on the east half of the six pass exchanger were replaced in kind with 2-1/2 inch schedule 80 carbon steel pipe. The west half of the exchanger was removed and new inlet and outlet yokes fabricated to accommodate the modification.

Work completed or in progress during the plant turnaround included the following:

1. All inner tubes (slurry service) and "U" bends in the Filter Feed Surge Vessel Recirculation Dowtherm Exchanger were replaced in kind with 2-1/2 inch schedule 80 carbon steel pipe.

2. The discharge line from the Filter Feed Recirculation Pump to the exchanger (3 and 4" SL-28) was inspected by ultrasonic testing and found to be very thin (see Figure A-11). The entire 4-inch line was replaced in kind.
3. An elbow on the Filter Feed Surge Vessel recirculation line (2" SL-32) was inspected and found to have lost approximately one third of nominal wall thickness. This line will be replaced on the annual shutdown.
4. The inlet nozzle to the Recycle Process Water Tank (RPWT) was raised above the water drawoff nozzle and an inlet baffle was installed. The radio frequency probe for the light oil level indication was removed and found damaged. It was replaced in kind and the probe nozzle was lowered two feet to give additional light oil volume. The heavy oil radio frequency probe was also found damaged and replaced in kind.
5. On March 19, Lawrence Pumps Inc. sent a representative to the pilot plant to inspect the excessive casing wear which had occurred on a 1 1/2" AL horizontal partially lined heavy duty slurry pump after 300 hours service pumping filter feed surge drum material (miscellaneous recovered liquids/slurries). This service has historically been one of the most severe encountered in the pilot plant. To alleviate the wear in future operation, Lawrence supplied a new 14" diameter impeller to replace the original 19" impeller. A casing with a new diffusion coating (surface hardness 140 Rockwell "C") will also be provided. To compensate for head loss resulting from the smaller impeller, pump speed is being increased from 1160 to 1980 RPM. Lawrence feels that this will reduce turbulence in the volute area. The combination of harder surface coating of the volute and reduced turbulence should markedly reduce erosion in areas unprotected by wear plates.

D. Solvent Recovery (Area 04)

The level control valve for the new Vacuum Flash Drum (LCV-1465) was cleared December 25, and process solvent circulation established through the drum. Later, however, the drum inlet control valve (FCV-1405) plugged. The 1/4 inch trim, which was broken, was replaced with 1/2 inch trim to reduce the frequency of plugging caused by the previous processing of minus 1/8 inch coal. Nevertheless, when circulation resumed December 26, it plugged again. In addition, the level control valve (LCV-1465) on the drum also plugged. When the drum was opened for inspection December 29, the coke trap was full of coke, the boot on the drum was coated with a heavy, tar-like substance which was too soft to hydroblast, and the control valves were plugged with small pieces of coke bridged together. The drum boot and FCV-1405 were cleaned with steam and the

down leg and LCV-1465 were hydroblasted. FCV-1405 plugged two more times in January, once with broken trim and stainless steel wire and once with 1/4 inch coal and gravel.

Faulty level indication on the new Vacuum Flash Drum allowed slurry to overflow to the Vacuum Flash Condensate Drum on January 18. The contents of the condensate drum were then transferred back to the Vacuum Flash Feed Accumulator for reprocessing. Pyridine insolubles content of the vacuum flash condensate increased to 12 percent during this upset.

Attempts to bring the old Vacuum Flash Drum on line December 29 were unsuccessful when the outlet control valve (LCV-219) plugged. The old vacuum system was in service January 4-5, until feedstock was no longer available.

From December 31 to January 1, the Light Ends Column was fed directly from the Filter Feed Surge Vessel for approximately 14 hours. This change in process lineup was required to replenish the heavy distillate inventory which was running critically low. The material in this vessel was of low pyridine insolubles content, and it was fed to the columns until the insolubles in the heavy distillate reached 4%.

On January 1, the cooling water block valve on the wash solvent overhead exchanger cracked when water froze in the valve. A cooling water system outage was necessary to repair the valve.

Plugging in the downleg of the new Vacuum Flash Drum was the major Area 04 operating problem during coal processing operations in the first quarter of 1979. Plugging occurred on February 9, 14, and 28 and on March 1, accounting for a total of about 48 hours of coal feed curtailment.

Preliminary tests to determine the severity of solids carryover from the new Vacuum Flash Drum at high feed rates and without a demister pad were completed February 12. During these tests, feed rates were increased from 1600 to 5000 lb/hr. Results showed no significant ash entrainment in the vacuum flash condensate at calculated vapor disengagement velocities of 4 to 6 feet/sec.

Area operation was stopped from February 22 until February 24 to make Dowtherm tie-ins to the Lummus Unit.

New thermocouples were installed on the old vacuum flash preheater coil February 11. Operation of the old vacuum flash system was not attempted during the reporting period.

Area 04 was shut down March 1 along with the rest of the plant for turnaround. Work completed or in progress during the turnaround included the following:

1. Inspection of the Wash Solvent Column reboiler bundle indicated generalized corrosion attack on all 316 stainless steel tubes. Metal loss has increased from 10% in March 1978 to 40% in March 1979. Hastelloy G and 317 stainless steel showed little or no attack but Incoloy 800 experimental tubes had thinned to the point of rupture. Fourteen tubes were intentionally plugged and the bundle was reinstalled. Results of the inspection and present condition of the reboiler bundle are shown in Figure A-12. A new 317 stainless steel bundle was ordered for installation during the annual shutdown.
2. The original carbon steel reboiler shell on the Wash Solvent Column was replaced with a 316L stainless steel duplicate. The condition of the shell at various inspections is shown in Figure A-13.
3. New 316L stainless steel feed lines to the Light Ends Column reboiler and the Wash Solvent Column reboiler were installed as replacement for the original carbon steel lines which were badly thinned. Figure A-14 contains the results of the inspection of the carbon steel lines which were replaced.

E. Gas Compression and Naphtha Unit (Area 05)

The gas compression area was responsible for 33 hours of coal feed curtailment during the processing operations in the first quarter of 1979. Compressor valve failures were the cause of these outages.

Compressor B valves failed on 5 occasions and resulted in about 21 hours of curtailment. Compressor A discharge valves were replaced twice and suction valves once, but these repairs did not require any curtailment. The third stage suction valves on the fresh hydrogen compressor failed six times. Failures on January 23 and February 2 entailed a total of about 12 hours of coal feed interruption. Failure of the third stage suction valve on the fresh hydrogen compressor also caused overpressure in the second stage and lifting of the second stage PSV. This required repair on 5 occasions. Both the fresh and the recycle hydrogen suction and discharge PSV's were repaired during the period but did not cause any curtailment. Ring and packing repairs were made while the plant was shut down in early January.

Several short duration power failures between January 31 and February 4 caused the fresh hydrogen compressor and both recycle compressors to trip. Even though time delay relays had been installed on the motors, none had been installed on the panel alarms for oil pressure and temperature. These alarms shut the compressors down each time. To prevent further problems, time delay relays were designed for the alarms and were installed during the March shutdown. Time delays have

also been ordered for the motor and panel alarms for the hydrogen syn-gas compressor and the inert gas compressors in Area 09.5. These relays will be installed with a new motor control center for Area 09.5 during the annual turnaround in August.

The absorption oil in the naphtha scrubber was changed from naphtha to middle distillate on January 23 to reduce foaming and carryover to the compressor suction. On January 29 the naphtha scrubber liquids went overhead into the compressor suction knock-out vessel and caused the compressors to shut down because of high liquid levels. This resulted in about 15 minutes of curtailment. There were no other operating problems. Eight transfers of middle distillate to the naphtha scrubber were made. On March 2, the naphtha scrubber liquid was pumped to the solvent area and the unit was steamed and secured for inspection. No maintenance was performed on this unit during the shutdown.

During the March turnaround the following work was done in the hydrogen system:

1. Hydrogen piping in Area 05 was chemically cleaned by Dowell Services, Inc. The lines were steamed, degreased, oxidized, and passivated and finally dried with nitrogen.
2. The fresh hydrogen compressor interstage coolers were inspected, hydroblasted, prefilmed, and reinstalled.
3. Both intercoolers on the fresh hydrogen compressor were removed for routine inspection. The tube side (hydrogen) appeared clean, but the shell side (cooling water) was fouled with large deposits of sludge and tuberculation. After both bundles were removed and hydroblasted, severe pitting of the bundles was evident. Because no spares were available, the bundles were hydrotested, reinstalled, prefilmed and returned to service. Identical carbon steel bundles were ordered and will be installed on the annual shutdown.
4. Both demister pads in the recycle hydrogen suction and discharge knock-out vessels were replaced with 304 stainless steel. The pad in the suction vessel was completely gone with only the supports remaining. The inlet and outlet lines were checked, but no trace of the pad was found.
5. Lubricator oil was changed from Molub - Alloy MA-30 to MA-828-30 for wet gas service. This was done on all the hydrogen and inert gas compressors, in an effort to reduce valve and ring problems.

F. Product Solidification (Area 08)

Flow rates to the Sandvik belt were limited because the belt had become bowed in the center causing vacuum bottoms to run off the sides of the belt when operated at normal rates. During the shutdown, a new belt was installed and the local controls for LCV-1465 and LCV-219 were relocated for improved safety.

SRC having an ash content above 20% and a melting point above 300°F was retained for future gasification tests.

G. Waste Treatment (Area 09.1)

Waste treatment area performance was substandard throughout most of the first quarter of 1979 and caused the plant effluent to exceed established discharge guidelines for phenol. The charcoal filters were in continuous operation except for one 14 day period in January when, because of excessive duty, the charcoal in the filters was expended before a new shipment arrived on site. The rate of introducing new bacterial cultures to the bio-unit was increased with little effect.

Unusually cold weather aggravated waste treatment problems early in this reporting period. On December 31, all of the alum lines as well as all of the acid lines were frozen. Flow was finally restored January 2. On January 10, it was determined by laboratory analysis that a shipment of 20 percent sulfuric acid used for pH control ranged in concentration from 6.2 percent to 9 percent. Consequently, the delivery pumps were unable to supply sufficient acid to properly control pH.

On January 4, waste treatment had to be shut down due to plugging in the line from the aeration section to the clarifier in the bio-unit. The plugging problem continued through January, although no shutdowns occurred. Periodic steaming of the line to maintain flow, however, upset clarifier operation.

Because of continued plugging in the line between the aeration section and the clarifier in the bio-unit, a professional diver was used to inspect the bio-unit. The inlet to the 8" pipe in the aeration section was found to be approximately two-thirds restricted by trash buildup. In the clarifier, more trash as well as sludge was found. The sludge buildup was confined to an area of the tank around the outfall line. Once these lines were cleared, no further difficulties were encountered.

In late February, the bio-unit effluent contained 25 to 30 ppm phenol and indicated inhibited activity of the micro-organisms. With the help of a consultant from Gulf Science and Technology Co., the problem was determined to be caused primarily by poor oil/water separation upstream and poor alum flocculation in waste treatment. Solutions to the immediate problem of reducing

phenol concentration included removal of the oil layer from the aeration section and addition of both powdered charcoal and new bacteria cultures to this section. In addition, the digester section of the bio-unit was pumped to the Fort Lewis sanitation system, phosphate levels were increased, and pH control was improved, although it continued to be a problem.

Waste treatment was completely shutdown March 20 to clean the surge reservoir of sludge accumulation.

H. Tank Farm (Area 09.2)

The final SRC-II distillate inventory for the five month Powhatan coal run showed 2,139 bbl of middle distillate and 763 bbl of heavy distillate. This inventory includes some material from the 1977-78 SRC-II run which produced the Consolidated Edison test burn shipment. The recovered yield from processing a total of 2,232 tons of Powhatan coal was estimated at 1,435 bbl of middle distillate and 599 bbl of heavy distillate. The total SRC-II fuel oil inventory was blended at a ratio of 2.9 parts middle distillate to 1 part heavy distillate.

I. Gas Systems (Area 09.5)

Inert Gas

On January 2, a pinhole leak was discovered on the outlet line of the inert gas receiver. A temporary patch was installed until new piping could be fabricated, but the line began leaking again on January 3. This time the entire unit was shut down and a 15 foot section of 2 inch line was replaced. The leak was believed to have been caused by corrosion from CO₂ and water in the inert gas.

The excessive water in the inert gas system indicated poor performance of the dryers immediately upstream of the receiver. Both dryers were removed from service and opened for inspection on January 15. The dessicant in both vessels was badly deteriorated and the electrical heating element on the west dryer was burned out. The dessicant support screen on the west dryer had also failed. After visual and ultrasonic inspection of the dryer shells was satisfactorily completed, the vessels were repaired and charged with new dessicant. A temporary heating element was fabricated and installed. The inert gas dryers were returned to service on January 20.

On January 25, MEA flow was established through the absorber to remove the CO₂ from the plant inert gas stream. The CO₂ concentration in the MEA reboiler became excessive and stripping steam was added to the MEA reboiler to help unload the MEA. The regenerator back pressure was lowered to approximately 3 psig to help the MEA regeneration and, on February 23, a second stripping steam line was hot tapped into the reboiler.

The plant inert gas stream has become relatively CO₂-free, but the loading on the MEA solution is still at the upper limit. Tests were still in progress at the end of this period to find optimum operating conditions.

Hydrogen Unit

On December 29, the process gas flow stopped because of freezing weather and icing of the pressure regulator. A problem with the reformer cooling water return pump gearbox caused the plant to shut down twice. The gearbox was replaced.

One of the operating objectives during February was to determine the effects of increased carbon monoxide concentration in the feed gas to the reaction area. (See Section IV-E for the results of these tests.) To increase the CO concentration in the feed gas, the low temperature shift converter was completely bypassed and the high temperature shift converter partially bypassed. Carbon monoxide concentration was increased to 5% on February 2 and to 10% on February 11. The carbon monoxide level remained at 10% for 4 days before being reduced to 5%. Full gas flow through the shift converters was restored February 21.

The syn-gas aftercooler was removed for routine inspection and found in much the same condition as the fresh hydrogen intercoolers with heavy fouling and tuberculation. After hydro-blasting, it was evident that the tube walls were too thin to be returned to service. The bundle was retubed in kind with carbon steel and was installed, prefilled, and returned to service on March 17.

DEA Unit

The DEA reboiler tube bundle was inspected when the unit was down for cleaning on January 28. Approximately twenty to forty tubes were found to be corroded significantly. In order to return the unit to service as soon as possible, plugging of the tubes was delayed until the March shutdown. Most of the corrosion was on the steam side of the tube and appeared to be the result of the formation of carbonic acid in the steam condensate.

During the March shutdown, the reboiler bundle was removed and 16 leaking tubes were plugged.

J. Dowtherm System (Area 09.8)

Relighting the Dowtherm heater after the burner tripped out continued to be a major problem. On one occasion, it took 15 hours to relight the heater. On another occasion, after the heater was shut down to repair welds at the Lummus Unit, it took 14 hours to relight the pilot.

The Dowtherm heater tripped out seven times during February: once on a false high stack temperature, once for an unknown reason and five times because of power dips. On February 13, it took 9.5 hours to relight the heater after a power dip. Operation was more reliable after readjusting the pilot light.

During the March shutdown, the burner controls were inspected and adjusted by a representative of North American Burner. These adjustments, along with the installation of a strainer in the fuel gas line, are expected to alleviate recurrent flame-outs and difficulties in relighting the heater after a flame-out.

K. Lummus Unit (Area 10.0)

A radiographic survey of all the welds on slurry piping two inches or greater was completed during January. None of the 85 stainless steel welds and only 81 of the 228 carbon steel welds were acceptable.

All weld repairs were completed, inspected and hydrotested during January and February. In addition, several heat exchangers were repaired by machining sealing surfaces.

The combination regulator-rotometers on FI-1040 A&B, FI-1032, FI-1033 and FI-1035 (purges under safety valves) were replaced with capillary tubes for controlling the flow and with magnetic rotometers for measuring the flow. The regulators were replaced because of their tendency to plug. The rotometers were replaced because the glass rotometers were inoperable with opaque fluids.

Studies conducted on the Lummus Unit during the turnaround indicated that the fractionation section could be operated at design conditions and achieve satisfactory separation. Lummus deashing studies will begin using Kentucky No. 9 and 14 coal pulverized to 80 percent minus 200 mesh. Other operating parameters will be similar to previous SRC-I mode operation. The Lummus Unit has been commissioned and is ready for testing as soon as coal solution slurry is available. An experimental program for the first phase of operating and testing of the Lummus Unit has been devised with assistance from Lummus personnel.

III. Pilot Plant Process Analytical Data

During the month of January, the plant was mostly down due to operating problems aggravated by use of minus 1/8 inch coal and to the severe weather in late December which froze much of the equipment. When repairs were completed, the plant operated in the SRC-II mode with Powhatan No. 5 coal during the month of February. In March, the plant was in a turnaround period most of the time following a leak and fire in Area 03; meanwhile the laboratory continued to monitor waste treatment, steam generation, and other areas as required.

The average analyses of the raw coal used during February are shown in Table 1.

TABLE 1
Average Raw Coal Analyses, Wt. %
February

Ash	10.70
Moisture	3.29

The average analyses of the dried, pulverized coal are reported in Tables 2, 3, and 4.

TABLE 2
Average Dried, Pulverized Coal Analyses, Wt. %
February

Carbon	71.52
Hydrogen	5.15
Nitrogen	1.20
Sulfur	3.64
Oxygen (by difference)	6.85
Ash	11.07
Moisture	0.57

TABLE 3

Average Analyses for Forms of Sulfur, Wt. %

February

Pyritic Sulfur	1.81
Sulfate Sulfur	0.08
Organic Sulfur	1.75
Total Sulfur	3.64

TABLE 4

Average Sieve Analyses of Dried, Pulverized Coal, Wt. %

February

-12 mesh	99.49
-30 mesh	98.01
-60 mesh	84.82
-100 mesh	65.06
-200 mesh	45.49

The average analyses of the stripper bottoms for February are shown in Table 5.

TABLE 5

Average Analyses of Stripper Bottoms, Wt. %

February

Water	0.09
Naphtha	0.03
Middle Distillate	1.65
Heavy Distillate	28.54
Vacuum Bottoms (P.I. included)	69.68
Pyridine Insolubles (as received)	22.68
Ash in Pyridine Insolubles (P.I.)	64.68

Laboratory-determined coal conversion on a moisture-ash-free (MAF) coal basis during the month of February was 93.94% by weight.

The average analyses of the Recycle Process Water Tank (RPWT) oil and water phases for February are reported in Tables 6 and 7.

TABLE 6

Average Analyses of RPWT Oil Phase, Wt. %

February

Naphtha	11
Middle Distillate	50
Heavy Distillate	39

Specific gravity @ 60/60°F 0.948

TABLE 7

Average Analyses of RPWT Water Phase, Wt. %

February

Phenols	1.05
Nitrogen	1.72

Typical fractional analyses of liquid products, based on ASTM D-86 distillation data, are reported in Table 8.

TABLE 8

Typical Fractional Analyses of Pilot Plant Liquid Products, Vol. %

February

<u>Laboratory Distillation Fractions</u>	<u>Naphtha</u>	<u>Middle Distillate</u>	<u>Heavy Distillate</u>
IBP - 350°F	96	1	0
350 - 550°F	4	98	1
550 - 850°F	0	1	99

The average analyses of the pilot plant vacuum bottoms produced during February are reported in Table 9.

TABLE 9

Average Analyses of Pilot Plant Vacuum Bottoms, Wt. %

February

Carbon	64.58
Hydrogen	4.02
Nitrogen	1.35
Sulfur	2.87
Oxygen (by difference)	3.26
Ash	23.92
 Fusion Point	 292°F
(Gradient Bar Method)	

Typical analyses of waste water streams are shown in Table 10.

TABLE 10
Process Waste Treatment Analyses

<u>January</u>	<u>Bio-Unit Feed</u>	<u>Bio-Unit Effluent</u>	<u>Plant Effluent (Composite)</u>
pH	6.7	6.9	6.8
Total Suspended Solids, ppm	36	106	1.0
Phenol, ppm	102	20	1.7
Chemical Oxygen Demand, ppm	1521	486	31
Biological Oxygen Demand, ppm	90	27	3.4
<u>February</u>			
pH	6.0	6.8	6.8
Total Suspended Solids, ppm	19	61	2.6
Phenol, ppm	189	42	7.1
Chemical Oxygen Demand, ppm	2458	1291	141
Biological Oxygen Demand, ppm	107	67	19
<u>March</u>			
pH	6.4	7.0	6.8
Total Suspended Solids, ppm	32	85	2.8
Phenols, ppm	24	8.1	0.5
Chemical Oxygen Demand, ppm	812	524	54
Biological Oxygen Demand, ppm	128	45	7.8

During the quarter, plant effluent had an average daily flow of 777,555 GPD, with a minimum of 552,600 GPD and a maximum of 883,200 GPD. Phenol content in effluent water averaged 3.1 ppm, with a minimum of 0.06 ppm and a maximum of 8 ppm. The oil and grease concentration in the effluent water averaged 5.1 ppm, with a minimum of 1.9 ppm and a maximum of 12 ppm.

Material Balance Runs Data

During the reporting period, one material balance run was successfully completed, and analyses on the feed coal during the run as well as on the products are presented in Table A-4.

SPECIAL STUDIES

Study on Bio-Unit Micro-organisms

In order to investigate difficulties experienced in the bio-unit, the laboratory initiated a special study program with the cooperation of Dr. Arthur Gee, Professor of Biology at Pacific Lutheran University.

Samples were taken from various locations within the bio-unit and an extensive study on a variety of micro-organisms was conducted. Summarized results of the studies are presented below:

1. The three types of dried micro-organisms being used (viz. Types VIII, CW-3 and Phenobac) have all performed well in phenol degradation under controlled conditions. However, only Phenobac stayed active when used in a complex environment such as the plant bio-unit.
2. In separate experiments, the rate of phenol degradation by micro-organisms was determined in culture media with increasing concentration of the following: phenol, process derived oil, and other soluble organic materials (e.g., mono- and diethanol-amine) associated with the process. In all cases, phenol degradation was inhibited by higher concentration of these organic materials.
3. For the micro-organisms to function properly, minimum residence time in the bio-unit should be 48 hours. That means maximum flow rate on the plant unit should be 75 GPM. Laboratory culture and measurements of oxygen demand by bio-unit digester sludge over both short (less than one hour) and long (5 days) time periods when the process unit was operating in a high flow rate (i.e., low residence time) condition have indicated that relatively high metabolic activity is retained by the digester cell mass. It is therefore supposed that recirculation of this sludge to the aeration section would be of benefit in maintaining bio-unit effectiveness during high rate operation.
4. The pH in the bio-unit should be kept between 6.5 and 8.0 for good micro-organism activity.
5. There is some indication that some mineral salts or trace elements are necessary for good micro-organism growth. As the feed to the waste treatment unit varies greatly, some essential elements or other nutrients may not always be present in sufficient quantities.

IV. Pilot Plant Special Studies

A. Dissolver Volume Measurement

On January 4, 1979, two measurements were made of the active dissolver volume. Using a previously developed water³ filling technique an average bottom section volume of 45.0 ft³ was obtained. This result is typical of previous measurements and represents a loss of approximately 5 ft³ or 5.4% of the nominal dissolver volume. The active reactor volume was also measured during the March turnaround and again determined to be 45.0 ft³. A comparison of these and previous measurements is shown in Table 11.

TABLE 11
DISSOLVER VOLUME MEASUREMENTS

<u>Date of</u> <u>measurement</u>	<u>Measured Volume of</u> <u>Bottom Section of Dissolver</u>
09/20/77	47.3 ft ³
12/09/77	47.7 ft ³
04/07/78	45.7 ft ³
08/30/78	46.2 ft ³
01/04/79	45.0 ft ³
03/05/79	45.0 ft ³

B. Slurry Flow Measurement

The recycle slurry flowmeter, FT-1152, was recalibrated during January. In recent months, flow measurement from this instrument has been used to develop the desired feed slurry composition. This was necessitated by the inaccuracy of the flow measurements estimated from the speed of the high pressure charge pumps which had been used in the past to estimate the recycle slurry flowrate. Recent calibration studies have suggested that both the response of the speed indicator and the efficiency of the positive displacement charge pumps may vary with time. Therefore, the charge pump speed is presently used only as a check on the reliability of the flowrate indicated by FT-1152.

The performance of the recycle slurry flowmeter has improved steadily as modifications have been made to correct deficiencies. This meter consists of a 6K Stellite quadrant-edged orifice plate, connected to a differential pressure transmitter via solvent-purged impulse lines. A schematic drawing of the present slurry flowmeter installation is shown in Figure A-15.

The quadrant-edged orifice plate flowmeter was chosen for this service because it has a theoretical flow coefficient which is insensitive to viscosity changes at low fluid Reynolds numbers. The Reynolds number of the recycle slurry steam has been estimated at less than 10,000 at typical process flowrates in 1" pipe. Conventional concentric orifice plates and Venturi tubes are generally not recommended for use at such low Reynolds numbers because of sizeable correction factors which must be applied to the flow calculation equation.

The quadrant-edged orifice plate flowmeter measurement response has been shown to be independent of Reynolds number between values of approximately 3,000 and 70,000.¹ The recycle slurry application is well within this range. The calculation of flow for this meter is based on the following equation:

$$\text{FLOW (lbs/hr)} = 2835 (D^2) (F_a) (C) [(h_w) (G_f)]^{\frac{1}{2}}$$

where:

D	=	pipe diameter, inches
F _a	=	orifice plate expansion factor
C	=	flow coefficient
h _w	=	differential pressure, inches of water
G _f	=	fluid specific gravity at flowing conditions

Initial calibration tests on this flowmeter in December, 1977, verified that the response of the meter was independent of Reynolds number, and that the theoretical flow coefficient and actual flow coefficient were comparable.

In the original installation, the flow of purge solvent was controlled by needle valves. To inhibit plugging of these valves, the purge solvent was filtered through a 5 micron Cuno cartridge-type filter followed by a 60 micron sintered metal filter. The 60 micron filter was found to plug immediately when put in service. Attempts to operate without this filter were only marginally successful, as solids would partially or fully plug the metering valves intermittently and create bias in the differential pressure measurements. Solids were also observed to accumulate in the differential pressure transmitter. These problems were apparently due to solids bypassing the Cuno filter elements.

These problems associated with the solids in the flush solvent have been overcome by three major modifications:

1. Installation of 1/8" capillary tubing in place of metering valves to control the purge solvent flowrate.
2. Location of the DP transmitter in a "high" point in the meter loop to inhibit solids settling in the transmitter.
3. Use of different filter elements, which seem to have greater resistance to temperature and solvent environment.

¹ M. Bogema and P. L. Monkemeyer, "The Quadrant Edge Orifice - A Fluid Meter for Low Reynolds Numbers", Trans. ASME, J. of Basic Engr., Vol. 82-3, September 1960, pp 720-734.

The modifications greatly improved the reliability of the quadrant-edge orifice plate flowmeter in recycle slurry service, and a second meter has been installed on the feed to the new Vacuum Flash Drum (FE-1405). Both of these meters are now in operation in service where other flow measurement devices were not successfully applied.

Recent recalibration of FT-1152 has indicated that the measured flow coefficient is within two percent of the theoretical coefficient. The ability of this instrument to retain its calibration in slurry service suggests that it is suited to application in high temperature slurry service. The recycle slurry flowmeter has been in service for more than eight months during SRC-II processing. During this period, the orifice plate and upstream and downstream piping have been periodically inspected for erosion and corrosion. The Stellite orifice plate has not shown any effect of erosion or corrosion. There has been some loss of pipe wall on the upstream and downstream piping, but this loss is typical of the erosion/corrosion problems in high temperature slurry service. To further improve the reliability of this instrument, a purge oil system is being installed to inject a metered amount of a "clean" purge oil (motor oil) into the transmitter impulse lines.

C. SRC-II Material Balance Run 79SR-30

Material Balance Run 79SR-30 was completed this quarter. The run began at 1000 hours on January 28, and was aborted at 1400 hours on January 29 due to a plant power failure. Target operating conditions for this run are shown below:

Dehumidified Coal Feed, lb/hr	2070
Feed Slurry Coal Concentration, w%	30
Feed Slurry Flowrate, lb/hr	6900
Feed Gas Hydrogen Purity, v%	85
Dissolver Pressure, psig	2000
Average Dissolver Temperature, °F	850
Feed Coal	Powhatan No. 5 (-30 mesh)

This run was made at conditions similar to previous Powhatan coal SRC-II material balance runs. The major difference between this run and previous experiments was the feed coal particle size distribution. Prior to MBR 79SR-30, the coal pulverizer was modified to produce a size distribution of greater than 99 w% smaller than 30 mesh.

Average operating conditions and raw product yields are presented in Tables A-5 and A-6 respectively. Elementally balanced product yields are reported on a moisture-and-ash free coal feed basis in Table A-7. The SRC yield reported for this run appears higher than in previous runs, but this is believed due to the lower fusion point of the vacuum bottoms product. Other yields are comparable to previous results.

D. High Temperature Coal Blending Tests

A program to test the feasibility of blending minus 1/8 inch coal with recycle slurry at temperatures in excess of the normal 350°F blending temperature was undertaken during February. In commercialization of the SRC-II process, the capability of blending coal and slurry at high temperatures would reduce the heat requirement of the slurry preheater and thereby reduce both capital and operating costs. Past experience in blending 200 mesh pulverized coal with hot slurry in the Ft. Lewis laboratory had shown that the minimum viscosity of the mix occurred at 350°F, and the viscosity increased rapidly as temperature increased thereafter. Tests at higher blend tank temperatures in the plant in March 1978 revealed that 200 mesh pulverized coal slurry could not be processed at Ft. Lewis at temperatures in excess of approximately 370°F due to failure of the slurry recirculation pumps, apparently caused by high slurry viscosity.

Subsequent laboratory tests suggested that the slurry viscosity growth was substantially affected by coal particle size. The swelling of coal, or absorption of solvent by the coal, was found to be highly related to coal particle size, with larger coal particles swelling at a rate significantly less than that for smaller coal particles. The amount of swelling was assumed to be indicative of the viscosity of the mix. Therefore, it was hypothesized that higher coal slurry temperatures could be achieved at Ft. Lewis if the coal particle size could be increased.

The December 1978 test in which 1/8 inch coal was fed into the process was a test of that hypothesis. Unfortunately, the large particle size mineral matter in the coal caused severe processing problems, resulting in line plugs and valve plugs in the plant. However, during the period of operation when coarse coal was fed, it appeared that the coal slurry viscosity was lower than at comparable conditions with pulverized coal. The best available indication of the comparative viscosity of pulverized coal slurry and coarse coal slurry was available from analysis of the differential pressure across the constant speed hydraulic drive of the slurry mix tank agitator. Such a comparison is presented in Figure A-16. The agitator differential pressure decreased significantly from the period of pulverized coal operation to the period of crushed coal operation, suggesting lower viscosity. It was not possible to achieve temperatures above approximately 290°F with the 1/8 inch coal

because this coal was dried only to approximately 3% moisture (whereas pulverized coal is dried to 1% moisture) and it was not possible to achieve recycle slurry temperatures in excess of 400°F at the recycle slurry cooler outlet. The cooler inlet temperature was approximately 580°F, but the convection losses from this oversized cooler were quite large, even with the cooler shell thoroughly insulated. This limited the maximum outlet temperature which could be maintained.

Modifications were made to the cooler in January and February to limit heat losses and provide the capability of operating at elevated blend tank temperatures. The pulverizer was also modified at this time to produce an intermediate particle size distribution (particles smaller than 30 mesh). A series of tests followed, in which the blend tank temperature was gradually increased to determine the maximum feasible operating temperature. A maximum temperature of 395°F was achieved with higher operating temperatures precluded by heat loss from the slurry cooler and difficulty in pumping the increasingly viscous slurry.

The effect of blend tank temperature on agitator differential pressure at various levels of temperature is shown in Figure A-17. The agitator DP appeared to be positively correlated with the blend tank temperature, i.e., as blend tank temperature increased, so did the agitator DP.

At the conclusion of the high temperature tests, the coal pulverizer temperature was decreased to minimize coal drying, and thereby increase the moisture content of the feed coal. The result of the step is evident in a significant and unexpected decrease in the agitator DP. Whether this decrease was due to the presence of moisture in the feed coal or a slightly lower blend tank temperature is not certain.

The results of the high temperature coal slurry blending tests indicate that high temperature coal slurry blending is feasible, at least up to a temperature of 400°F with coal ground to a particle size distribution of smaller than 30 mesh. This study also showed the benefits of feeding coarser particles. It was not possible to operate the coal slurry blending system at temperatures in excess of 370°F with a smaller particle size distribution. Operation with the larger particle size distribution at 395°F was possible.

E. Feed Gas Carbon Monoxide Addition Tests

The feed gas to the Fort Lewis pilot plant during SRC-II operation typically contains 2.5-3.0 V% carbon monoxide. During February, step changes were made which resulted in raising the concentration of carbon monoxide to 5 V% and 10 V%. (These changes resulted in a decrease in hydrogen purity to 75-80 V% at the highest level of carbon monoxide.) During this period the following nominal operating conditions were maintained:

Feed Slurry Coal Concentration	30 wt %
Dehumidified Coal Feedrate	2070 lb/hr
Recycle Slurry Flowrate	4000-4400 lb/hr
Dissolver Temperature	800-855°F
Dissolver Pressure	2000 psig
Feed Gas Flowrate	80 MSCFH

Due to numerous coal outages during February, it was not possible to maintain lined out operating conditions for periods long enough to establish the effects of carbon monoxide concentration on distillate or vacuum bottoms yields. However, it was possible to obtain gas yield data through on-line gas balances for each level of carbon monoxide concentration. These data are presented in Figure A-18.

The data in Figure A-18 include two periods of operation at a 2.5 V% carbon monoxide concentration in the feed slurry. The mean product yields at each nominal level of carbon monoxide concentration are also shown in Figure A-18, and these means are connected by a broken line. The means and standard deviations of the data shown in Figure A-18 are presented in Table A-8.

These data were subjected to a one way classification analysis of variance to test the null hypothesis that there was no significant difference in the mean yields of a given component at varying levels of carbon monoxide concentration in the feed gas. The analysis of variance results are reported in Table A-9. Comparison of the critical value of the F statistic at the $\alpha = 0.01$ level of significance ($F_{0.01, 2, 56} = 4.98$) with the calculated values indicate that the calculated F statistic for hydrogen consumption, carbon monoxide yield, and carbon dioxide yield is greater than the critical value, leading to rejection of the null hypothesis. Therefore, this test suggests that the yields of these components were significantly effected by the concentration of carbon monoxide. The analysis of variance results also indicate that a significant difference in hydrocarbon gas yield or hydrogen sulfide yield did not occur when the carbon monoxide concentration was increased.

Observation of the trends in product yields as shown in Figure A-18 suggests that with increasing carbon monoxide concentration in the feed gas, hydrogen consumption and carbon monoxide production decreased (carbon monoxide began to be consumed), and carbon dioxide yield increased. These results suggest a reaction mechanism whereby carbon monoxide is converted to carbon dioxide and the net hydrogen requirement for the process is decreased.

It is unfortunate that more definitive analysis of the effects of carbon monoxide on distillate and vacuum bottoms yields was not possible. It would have been of interest to determine whether the carbon monoxide concentration had any effects on the overall hydrogen reactions and the resultant yields of distillate and organic vacuum bottoms. However, the lack of significant change in the production of hydrogen sulfide suggests the desulfurization of the coal was not affected. This leads to the presumption that the sulfur concentrations in the product were also not affected.

F. Feed Coal and Recycle Slurry Particle Size Distributions

Three ranges of feed coal particle size have been processed in the pilot plant in the SRC-II operating mode. Typical particle size distributions of these feed materials, as determined by ultrasonic sieving, are shown in Figure A-19. These data appear to represent typical cumulative distribution curves for comminuted solids.

In an article discussing techniques for estimating the particle size distribution of comminuted solids from sieve analysis, use of the Plitt equation is recommended.² This equation is of the following form:

$$y = 1 - 2^{-(x)B}$$

where: y = cumulative weight fraction of particles of dimensionless diameter

x = dimensionless particle diameter = actual diameter : diameter @ 50 wt% recovery

B = exponent governing the shape of the distribution curve (obtained from nomograph in reference 2)

The sieve analysis of pulverized coal is normally only used for process control. Historical data were not available for particle sizes below 200 mesh. Thus an incomplete distribution is obtained for pulverized coal samples using historical data. However, the Plitt equation has been found to provide a good fit to historical data and, further, yields a reasonable distribution below 200 mesh. The curves in Figure A-19 for nominal 30 mesh and 200 mesh pulverized coal are fitted to sieve analysis data using the Plitt equation which then provides an estimated complete distribution. Particle size

²A. Zanker, "Short Cut Technique Gives Size Distribution of Comminuted Solids", Chem. Engr., 4/24/79, pp. 101-103.

distribution data are also presented in Figure A-19 for samples of recycle slurry obtained during 30 mesh and 200 mesh coal processing. These measurements were obtained in an X-Ray sedimentation technique developed by an outside laboratory (Micron Data Laboratories).

Comparison of the two recycle slurry samples indicates little difference in particle size distribution despite the large variation in feed coal size. This finding leads to the following hypotheses: consistent comminution of particles occurs in the SRC-II reaction system regardless of feed solids size; consistent retention of larger particles occurs within the reaction system; sampling or analytical problems consistently bias dissolver effluent particle size distributions, masking the presence of larger particles. It is hoped that the results of the continuing dissolver solids sampling program will clarify SRC-II reactor particle dynamics and lead to a better understanding of the particle size results obtained to date.

G. SRC-II Liquids Production

Weekly inventories of SRC-II liquids were calculated throughout the Powhatan coal SRC-II run period. The calculation procedure involved sampling and analysis of approximately 20 major in-process vessels as well as all tank farm vessel levels. The analytical and vessel gauge data were then compiled and used in a computer program which calculated a total plant inventory of naphtha, middle distillate, and heavy distillate.

Table A-10 and Figure A-20 show the results of the weekly inventory calculations from October 1978 through February 1979. A certain amount of scatter is evident in the inventory data shown, probably the result of random error associated with sampling and analysis of a large volume of liquid distributed among many vessels. The data do, however, show a consistent increase in inventory even during periods of unstable plant operation.

When the inventory data from Table A-10 are combined with shipment data, the production figures shown in Table A-11 result. Production here is calculated by adding inventory changes to shipments made during each interval.

Using the total production of SRC-II liquids from Table A-11, an overall plant material balance was calculated. The results, shown on Table A-12, are based on the following sources of information:

- | | |
|------------------------------|--|
| 1. Coal input: | Based on coal weighed into the plant by the gravimetric feeder |
| 2. Hydrogen input: | Based on the average consumption figure calculated from material balance runs made during the period |
| 3. Gas and water make: | Same basis as hydrogen input |
| 4. Recovered distillate: | From inventory and shipment records |
| 5. Recovered vacuum bottoms: | Weighed on truck scales as produced |

The data shown in Table A-12 indicate that substantially less liquid product was collected than would be expected based on material balance results. Similar losses have been observed in the past, and are apparently the result of a number of factors. Light naphtha is lost due to the lack of a pressurized light ends fractionator (debutanizer). Middle and heavy distillate are lost to emulsion formation which frequently occurs and makes oil/water separation difficult.

Additional heavy distillate is lost during startup and shutdown of the vacuum flash system when very low melt point product is diverted from the solidification system. Some loss also results from process degradation of heavy distillate used as seal flush. Unaccounted losses also probably result from pump drips and spills.

As a result of the continuing oil recovery problem documented above, a new waste oil recovery system based on liquid extraction is being designed, which should significantly improve overall plant liquid balances in the future.

H. Lummus Deashing Unit

1. Experimental Plan

A meeting was held during March to discuss the proposed experimental program for the Ft. Lewis Antisolvent Deashing Unit.

Lummus Company representatives outlined the proposed experimental program and estimated the duration of each test run. The program was very similar to earlier experimental programs proposed by Lummus. However, due to startup delays, the time frame of the schedule was shifted and the order of several tasks was changed. The proposed program is as follows:

1. Shakedown Run - to debug and check whether the design conditions are suitable for the demo-run. From April 15 to May 15.
 2. Demo-Run - to prove sustained operability. From May 15 to June 15.
 3. Capacity Test - to establish maximum capacity. From June 15 to June 30.
 4. Variable Test - to establish optimum operating conditions. From July 1 to August 15.
 5. New Control System Test - to test simplified flow control. From August 15 to August 31.
 6. Underflow Recirculation Test - to test the effect of underflow recirculation. From September 1 to September 15.
 7. Settler Temperature Test - to test the effect of settler temperature on unit operation. From September 15 to September 30.
 8. Alternate Feed Location - to test the effect of an alternative feed point. From October 1 to October 30 (including brief shutdown).
 9. Remove Feed Distributor - to test whether the feed distributor is necessary. From November 1 to November 30.
 10. Remove Overflow Distributor - to test whether the overflow distributor is necessary. From December 1 to December 22.
2. Design Operating Conditions for the Ft. Lewis Antisolvent Deashing Unit

The reaction area operating conditions for the Antisolvent Deashing Unit tests are summarized below:

Coal Type : Kentucky #9 and 14
Dehumidified Coal Feed: 50 tpd.
Recycle Solvent to dehumidified Coal Feed Ratio: 1.5
Hydrogen-rich Gas Feed Rate: 35000 SCFH (@ 93% Purity)
Water Quench to Dissolver Effluent Stream: 900 lb/hr
Dissolver Volume: Approx. 50 ft³
Dissolver Pressure: 1500 psig
Dissolver Outlet Temperature: 850°F

Under these operating conditions the composition of the feed to the Antisolvent Deashing Unit has been estimated to be as follows:

Flow Rate: 8230 lb/hr
Composition: 60.8 wt % Process Solvent
31.6 wt % Solvent Refined Coal
7.6 wt % Ash + IOM
Initial Boiling Point: 525°F
Specific Gravity: 1.17 at 60°F
Viscosity: 233 cSt. @ 100°F
10.3 cSt. @ 210°F

3. Antisolvent Deashing Unit Mass Balance Strategy

The mass balance envelope for the Deashing Unit is shown in Figure A-21. The input and output streams, their components, flow meter numbers and sample point numbers are listed in Table A-13. All of the listed flowmeters are tied into the computer.

For the purposes of mass balances it will be assumed during lined out steady-state operation that no accumulation of any of the components (ash, SRC, Process Solvent, antisolvent and water) is occurring in any of the five major vessels in the unit (Feed Surge, Settler, Stripper, Fractionator and Overhead Accumulator). See Table A-14 for a list of major vessel holdup volumes and level indicator numbers.

Due to inaccuracies in the flow meters and to a lesser extent in the analyses, the component mass balances for ash, SRC and antisolvent are not expected to be more accurate than $\pm 15\%$. The process solvent balance will be even more inaccurate due to numerous locally metered or unmetered purges or flushes. The water balance will be forced since there is no sour water flow meter.

I. Data Acquisition System

Much available computer time during January was used for format conversion of magnetic tape data to process all old data to the format required by Lummus Unit expansion. The work was completed during the March shutdown. All programs referencing data from magnetic tape have been modified to handle the new format.

A new routine was added to the user's library to read and report switch settings on the computer main panel. The routine was incorporated in the program "Kickoff", which was used to initialize the data collection system. The program can only be run now if the proper combination of switches are set prior to execution. This security system prevents users from inad-

vertently executing a program resulting in the loss of plant data.

The format for storing the date and time of average data was expanded. The year is stored separately in order to be able to distinguish 1970 from 1980. All programs referencing the averaged data were modified to use the new format.

A new program was written to read the digital contact inputs and display the bit pattern on the CRT console. The program will be used to test data supplied by the new chromatograph.

As a part of preventive maintenance, the cooling fans in the CPU cabinets were replaced with new high volume models. On January 15, a computer shutdown occurred, caused by a bad power supply. The paper tape reader was malfunctioning the entire period. It would not read tapes correctly, and in many cases the tapes could not be read at all. A different reader was installed and several changes made to the logic control board, but its operation is still only marginal.

Several maintenance programming tasks were completed during this reporting period. All programs referencing a loop titles file were modified to use a new file containing titles and engineering units. Modifications were made to the current loop value program to provide more information. Modifications were made to the chromatograph servicing routines to provide information on currency of data. The plant inventory program was modified to allow the calculation and output for selected vessels only, using specified lab analyses for each vessel. Other maintenance programming work during the period included modification of the following routines: Flow Data Report, Lummus Scan Setup, Lummus Scan Resume, Control Room Special Scan, Control Room Scan Resume, and Lummus Log. These routines all access a control loop code file which was modified to a byte format to increase program execution speed. This change also required modification of the file input-output utility routine, and modification of the data file update program to include the new loop numbers file.

Work was begun during February on a series of routines designed to produce TPB boiling curve material balance run results from plant laboratory special D-86 and Cushman analyses. A lab input program was developed to calculate TBP temperatures, cumulative cuts, and discrete cuts based upon either Cushman or special D-86 analyses. This program punches a paper tape containing both raw and processed data which will feed another routine designed to report inventory change in a selected number of vessels. When completed, the programs will report material balance results on up to ten discrete boiling fractions for a variable number of vessels included in the balancing envelope.

During the period new routines were written to expand the capability of program execution from the Lummus Unit control room. Programs available now include Current Loop Value, Scan Inhibit, Scan Resume and Chromatograph Log. These routines can be called from a switchbox installed in the unit control room. New system backup paper tape records were also produced during a period of plant downtime. These tapes contain all of the on-line system routines and are used to reload the operating system in the event of failure of the magnetic tape bulk data storage unit.

The computer system experienced a number of total system failures during this reporting period. Although the problem was not completely diagnosed, it was alleviated by replacement of several control cards in the CPU. Both the paper tape reader and punch failed and were replaced. One process data input module failed during the period, apparently as a result of a transient produced during an AC main power blip. Four unexplained system shutdowns occurred. Data from each shutdown was sent to Foxboro Company service representatives for analysis. Details of systems reliability during the period are shown on Table A-15.

J. Solvent Inventories

Three plant solvent inventories were completed this reporting period and the results are listed in Table A-16.

V. Pilot Plant Special Projects

A. Ground Coal Studies

Because of the problems encountered with the Jodal crusher and with the processing of minus 1/8 inch coal, the Jodal crusher system was removed from service and returned to the vendor.

One test was conducted with the unit to determine if a modification to the outer drum would significantly increase output. The test was terminated when the outer drum plugged with tar-like material during the initial startup. The major design difficulties encountered with this particular unit included insufficient capacity, blinding of the outer sizing screen with coal especially when wet, and loss of fines to the atmosphere.

B. Recycle Slurry Air-Cooler Exchanger Study

The overall heat transfer coefficient for the recycle slurry air-cooled exchanger was determined. The coefficient ranged from 3 to 5 Btu/hr-ft²-°F. The data and calculations are shown in Table A-17.

C. New Emergency Flush System

A proposal for an emergency flush system for the High Pressure Separator letdown loop was submitted to Engineering for consideration. The proposed system consisted of an air-driven portable double piston pump connected to the high pressure letdown loop. The pump would be capable of delivering up to 1/3 gpm at over 3000 psi pressure.

D. Storm Drain Surge Reservoir

During the last two years, a considerable amount of asphalt pavement has been added to the processing area. This has increased the flow of storm sewer water to the waste water treatment system. During heavy rains the extra storm sewer water exceeds the processing capacity of the waste water treatment system and causes the surge reservoir to overflow. To correct this problem, a purchase order was issued to Hallanger Engineers of Seattle on February 21 for design of a secondary surge reservoir for storm runoff.

E. New Slurry Preheater

The new Slurry Preheater from Heat Research Corporation was delivered in late March. The foundation was poured during February and the concrete showed excellent strength. Purchase orders for all stainless steel valves have been issued. The Spec 200 instrument components for the Foxboro computer have now replaced the valves as the critical path item. Target date for completion of the installation is August 1.

A proposed operating program was written for the new Slurry Preheater. This program was incorporated into a paper given at ORNL at a DOE project review meeting on "Coal Liquefaction Preheater Studies" on March 21. A report was compiled and issued later containing all the proceedings at this meeting.

The program proposed is aimed at obtaining data on:

1. Pressure drop along the coil versus gas and slurry rates.
2. Coking tendency at various conditions.
3. Erosion versus velocity limits.
4. Temperature gradient data using 14 skin and 7 immersion thermocouples.
5. Efficiency of the furnace and heat of reaction.
6. Limiting heat flux versus coking.

Figure A-22 is a simplified diagram of the preheater and auxiliaries, showing a bypass heat exchanger for hydrogen which will permit the gas rate in the coil to be varied over a wide range. Table A-18 lists some of the operating constraints imposed by the pilot plant. Table A-19 lists the more important dimensions of the new heater. Initial operation will be with a 467 foot long coil of 2 inch Schedule 160 321 SS tubing. If early data indicates that a smaller coil would be operable, a 1-1/2 inch coil will be fabricated.

Table A-20 lists the proposed runs to be made including SRC-II and SRC-I operations. Brief survey runs will be made at each slurry feed rate, varying gas rate to determine pressure and temperature effects. Then, a 3-week erosion test run will be made at a selected stable operating velocity. After this, the furnace will be shut down and bypassed to inspect the coil for erosion and coke deposits. Assuming no significant erosion has occurred, a new series will then be made at higher velocities and slurry rate. Coking will also show up quickly in pressure drop increase at constant rates of feed.

Table A-21 lists the principal types of data to be logged or computed.

F. Combustion Studies

Several programs have been completed by various agencies to determine combustion performance of SRC-II products. A study by Southern California Edison Company entitled "Factors Influencing NO_x Production During the Combustion of SRC-II Fuel Oil" is included as Appendix D of this report. "Preliminary Combustion Evaluation of Gulf Oil SRC-II Pyrolytic Fuel Using ORF's 10x10⁶ Btu/Hr. Vortometric Burner Facility" by Ontario Research Foundation and "Small-scale Combustion Tests of SRC-II Fractions and Blends" by Gulf Research & Development Company are included as Appendices E and F, respectively.

VI. Harmarville Process Development Unit P-99

The scheduled mid-December turnaround of Process Development Unit P-99 was continued through mid-February to complete installation of the new, fourth high pressure separator. This new separator is situated between the old intermediate and cold separators. In the future, the old intermediate separator will be referred to as the first intermediate separator and the new separator will be called the second intermediate separator. Thus, the dissolver off-gas will flow successively through the hot separator, the first intermediate separator, the second intermediate separator, and the cold separator.

A simplified flow diagram of the separator system is shown in Figure B-1. The second intermediate separator is designed to recover both an organic phase and an aqueous phase. Normal operating temperatures for the four separators are: hot, 750-800°F; first

intermediate, 575-625°F; second intermediate, 375-425°F; and cold, 100-125°F. Water is normally injected at the outlets of both the first and second intermediate separators. Part of this injected water is steam condensate and part is recycled process water. The purpose of injecting this water is to scrub out hydrogen chloride, ammonia, and some of the hydrogen sulfide in the dissolver off-gas.

A number of other modifications to the unit were made during the turnaround as indicated in the following list:

1. The internal thermocouples in the dissolver were repositioned to add more temperature readings in the bottom half, which is the reaction zone. This will provide a better indication of coking, should it occur.
2. A preheater was installed on the sparge gas stream to the hot high-pressure separator to cut down the amount of cooling occurring in the hot separator and permit better temperature control.
3. A metering bypass valve was installed around the recycle gas control valve to permit changing the valve trim size for a wider range of flow rates without shutting down the unit.
4. The surge tanks between the high-pressure letdown valves were modified to permit easier replacement of worn nipples.
5. Larger thermowells that will accomodate two thermocouples were installed on both the fractionator and the debutanizer columns. One thermocouple is for control, and the other is for the data logger.
6. Quick disconnect sections were installed on the mixing tank vent duct to simplify cleaning.
7. An internal thermowell was installed in the vacuum column bottoms receiver.
8. An absolute d/p cell pressure transmitter was installed on the vacuum column bottoms receiver to obtain the pressure in the flash zone.
9. A sample bomb system was installed to sample the recycle gas stream between the amine and kerosene scrubbers.
10. The amine and kerosene off-gas metering systems were repiped in order to facilitate checking McAfee gauge calibrations while the unit is in operation.

After completion of the installation of the new separator, the remainder of February was spent in getting back on-stream. A number of mechanical problems were encountered, such as both recycle compressors needing to be repacked and the belt on the coal feeder splitting and needing to be replaced. Also, some problems were encountered with operation of the new separator.

Coal feed to Run 49 was started on March 4. This was the second run on the shipment of Powhatan No. 5 Mine coal received from Fort Lewis and dried and ground by Empire Coke. Conditions for Run 49 were:

Pressure	2000 psig
Average Dissolver Temperature	457°C (855°F)
Nominal Slurry Residence Time	1 hour
Coal in Feed Slurry	30 wt %
Total Solids in Feed Slurry	44 wt %
Dissolver Gas Rate	50,000 SCF/T of coal
Dissolver Gas Purity	90 V %

During Run 49, several operating problems occurred which delayed getting lined out data. Early in the run, a rupture disk in the fractionation system blew out, necessitating a several hour coal outage and generally upsetting the unit. Upon recovering from this problem, several pressure buildups in the dissolver were experienced. This latter problem was apparently caused by the deposition of ammonium chloride in the cooler between the hot and first intermediate separators. This was largely overcome by raising the temperature of the cooler.

Run 49 was completed on March 28, at which time most of the process streams were sampled. Then the unit was changed to Run 50 conditions. Run 50 differed from Run 49 only in having a higher dissolver gas rate and purity to give a higher partial pressure of hydrogen.

Yields for Run 49 are given in Table B-1. Although conditions for Run 49 were quite similar to those for Run 48 except for a slightly higher recycle solids level in Run 48, Run 49 produced less distillate and more bottoms than Run 48.

VII. MERRIAM LABORATORY OPERATIONS

A. Introduction

During the first quarter of 1979, activities at the Merriam Laboratory included:

- A series of runs investigating the effect of iron containing additives on SRC-I operation with Pittsburgh Seam coals from the Blacksville No. 1 and Loveridge Mines.
- Addition of low levels (5-10 percent) of carbon monoxide to the feed gas while processing Pittsburgh Seam coal from the Powhatan No. 5 Mine in the SRC-II mode.
- SRC-I experiments with hydrogenated solvent (Cresap process solvent) under both normal and short residence time operating conditions with Kentucky 9/14 coal.
- Initiation of a simulated distillation technique to determine the entire boiling range distribution of product oils.

The conditions and results for all runs reported this quarter are summarized in Table C-1.

B. The Effect of Adding Iron Containing Compounds During the SRC-I Processing of Moderately Reactive Pittsburgh Seam Coals

Two Pittsburgh Seam coals, selected on the basis of their moderate reactivity, were used in the additive work.³ The first, which has been used in previous additive work³, was washed coal from the Blacksville No. 1 Mine. The second coal, from the Loveridge Mine, was used when the supply of Blacksville No. 1 was exhausted. Analyses of the two coals are similar and they were found to be of similar reactivity.

Additives investigated were pyrite (FeS_2), magnetite (Fe_3O_4), ferrous sulfide (FeS), and pyrite in combination with molecular sieve. The molecular sieve was added to determine the effect of a cracking catalyst.

The pyrite and magnetite were from the coal cleaning operation of the U. S. Steel Robena Mine, Greene County, Pennsylvania. The pyrite was found to be 80.3% FeS_2 and the magnetite 85.3% Fe_3O_4 based on the iron content. The pyrite and magnetite were finely ground by the Jet Pulverizer Co., Palmyra, New Jersey. The average particle size of the pyrite was 0.6 micron and that of the magnetite was 1.3 micron as determined by the Fisher Subsieve Sizer method.

The ferrous sulfide was technical grade granular material from the J. T. Baker Chemical Co. It was pulverized in a ball mill and sieved to pass 150 mesh. The theoretical iron concentration of 63.6 percent was used in determining the addition level. The molecular sieve was 13x (Linde). The material was hand ground with a mortar and pestle and sieved to pass 150 mesh.

Pyrite was added at a 7.5% level based on MF coal and levels of the other additives were selected to give a similar iron concentration (see Table C-1).

The effect of additives on recycle solvent, total oil and SRC yields is summarized in Table 12. Yields were determined by distillation of both unfiltered coal solution (UFCS) and filtrate. The results are, in general, qualitatively the same based on either method. Results based on distillation of filtrate should be considered more reliable due to an accelerated decomposition during distillation of unfiltered coal solution which may be caused by the additives.

³Solvent Refined Coal (SRC) Process, Quarterly Technical Progress Report for the Period January 1, 1978-March 31, 1978. The Pittsburgh & Midway Coal Mining Co., March 1979, FE/496-155.

TABLE 12

Effect of Iron Containing Additives on Yields*
(Wt % MF Coal Basis)

	Results based on distillation of filtrate	Results based on distillation of UFCS
<u>Pyrite</u>		
Blacksville Coal (DOE 288-289)		
Recycle Solvent	+8.0	+4.0
Total Oil	+6.9	+2.9
SRC	-4.5	-0.6
Loveridge Coal (DOE 293-292)		
Recycle Solvent	+7.7	+6.5
Total Oil	+9.1	+8.2
SRC	-7.5	-6.3
<u>Magnetite</u>		
Blacksville Coal (DOE 290-289)		
Recycle Solvent	+3.3	+3.7
Total Oil	+1.2	+1.7
SRC	-0.1	-0.7
Loveridge Coal (DOE 291-292)		
Recycle Solvent	+3.3	+8.1
Total Oil	+1.6	+6.3
SRC	-1.4	-5.8
<u>Ferrous Sulfide</u>		
Loveridge Coal (DOE 294-292)		
Recycle Solvent	+3.8	+5.0
Total Oil	+2.7	+4.1
SRC	-2.9	-4.0
<u>Pyrite + Molecular Sieve</u>		
Loveridge Coal (DOE 295-292)		
Recycle Solvent	+5.4	+9.1
Total Oil	+4.2	+8.2
SRC	-3.8	-7.5

* Changes in yields as a result of the additives.

Pyrite, which increased recycle solvent yield by about eight percent (based on coal), was the most effective additive investigated. Magnetite, producing a three percent increase in oil yield, was less effective.

The smaller effect of the magnetite may have been due, in part, to a shortage of H_2S available to form a catalytically active $Fe_{1-x}S$ compound from the Fe_3O_4 . Conversion of the 4.25 g of magnetite added per 100 g of coal to FeS would require 1.87 g of H_2S . In the uncatalyzed control runs (DOE 289 and 292) the H_2S yield was only about 1.3 g/100 g of coal. The H_2S generated from the coal was essentially quantitatively consumed in the magnetite addition run as only traces of it were present in the product gas or water. Since the amount of $Fe_{1-x}S$ which could be formed was limited by H_2S availability, addition of larger quantities of magnetite would not be expected to show any additional benefit.

The effect of pyrite and magnetite was significantly greater than in the earlier work with these materials. The difference is presumed to be due to the much smaller particle size used in this study (1 micron vs 75 micron).

The activity of the ferrous sulfide (FeS) was also somewhat lower than that of the pyrite. Ferrous sulfide (nonstoichiometric) is believed to be the active catalyst derived from pyrite, but the material formed in situ from pyrite is for some reason more reactive than the added technical grade FeS . This could be due to a difference in active surface area or the stoichiometry of the resulting $Fe_{1-x}S$.

Oil yield was increased by the pyrite/molecular sieve combination, but not as much as with the pyrite alone. The pyrite addition level in the former case was about 2/3 of the level used when pyrite was added alone and the oil yield increase was also about two-thirds of that observed with the pyrite addition runs. There is no evidence, therefore, that addition of this particular molecular sieve had an effect on oil yield.

SRC yields based on both distillation of filtrate and distillation of unfiltered coal solution are compared below. SRC yield based on distillation of filtrate averaged 0.8 percent (absolute) lower than that based on distillation of unfiltered coal solution. The largest difference in results (3.4-3.9%) was observed for the two runs with added pyrite, the most active of the materials investigated. In both runs with magnetite and in the run with the pyrite/molecular sieve combination, the SRC yield was lower when based on distillation of UFCs. These results indicate that the additives are affecting the distillation. While pyrite reaction products appear to promote a repolymerization during distillation, the magnetite and molecular sieve products may retard this repolymerization or may promote additional oil formation during distillation.

Run DOE-	SRC Yield		Difference SRC (UFCS) -SRC (Filtrate)
	<u>Based on Filtrate</u>	<u>Based on UFCS</u>	
288	57.6	61.5	3.9
289	62.1	62.1	0.0
290	62.0	61.4	-0.6
291	59.8	57.6	-2.2
292	61.2	63.7	2.5
293	53.7	57.1	3.4
294	58.3	59.4	1.1
295	57.4	55.9	-1.5

Changes in product composition were often only slightly beyond the experimental error in the analyses. Hydrogenation level was generally increased slightly by pyrite addition. There was also a very small decrease in desulfurization with the pyrite addition. The effect of magnetite addition on hydrogenation was not consistent. There was a significant increase in hydrogenation level of the SRC from the Blacksville coal but not in the hydrogenation level of the recycle solvent. Little change in hydrogenation level was observed with addition of magnetite to the Loveridge coal although there was a small improvement in desulfurization.

The changes in desulfurization with the pyrite or magnetite addition can be explained by changes in partial pressure of hydrogen sulfide. The added magnetite almost quantitatively removed hydrogen sulfide from the system. Ferrous sulfide had little apparent effect on product compositions.

C. The Effect of Low Level Carbon Monoxide Addition on the SRC-II Processing of Pittsburgh Seam Coal from the Powhatan No. 5 Mine

Three runs were made in this series: DOE 296R with pure hydrogen; DOE 297R-1 with 95% hydrogen, 5% carbon monoxide; and DOE 298R with 90% hydrogen, 10% carbon monoxide. The total reactor pressure was increased with carbon monoxide addition to keep hydrogen partial pressure essentially constant.

Frequent plugging problems related to carbon monoxide addition were experienced in lines and valves downstream of the intermediate temperature separator. The obstructions were found to be principally deposits of ammonium carbonate (or ammonium bicarbonate). These were caused primarily by increased carbon dioxide concentration in the off-gas. A contributing factor may have been a decrease in water available to solubilize the salts due to the shift reaction. Post-dissolver water injection was used to overcome the problem during DOE 298R.

As expected, carbon monoxide consumption was accompanied by production of an approximately equimolar quantity of carbon dioxide as shown below:

Run DOE-	CO Added		CO Consumption*			CO ₂ Production
	Mole % in Feed Gas	Moles hr	Wt % Based on Coal	Moles hr	% of Added Amount	Moles hr
296R	0	0	--	--	--	0.04
297R-1	5.1	1.24	1.4	0.18	15	0.18
298R	10	2.65	4.3	0.53	20	0.40

* CO in less CO out.

The difference between the carbon monoxide consumption of 0.53 moles/hr and the carbon dioxide production of 0.40 moles/hr in DOE 298R is not considered to be significant.

It was not possible to accurately determine water yield in run DOE 298R with water injection. It was, therefore, assumed equal to the amount obtained in the control run (DOE 296R) adjusted for water consumed in the shift reaction with carbon monoxide.

There were no significant changes in yields (except for the carbon dioxide production via the shift reaction) with carbon monoxide addition. Variation in yields is within the range normally expected for replicate runs. There is a 1% range for C₁-C₄ yield, 2.6% range for total oil, and a 0.6% range for SRC yield. The increased ammonia yield for run DOE 298R is presumed to be due to sampling problems brought about by the large quantity of injected water.

D. Use of Hydrogenated Solvent (Cresap Process Solvent) in SRC-I Processing of Kentucky 9/14 Coal

The hydrogenated process solvent was supplied by the Cresap facility of the Liquefied Coal Development Corporation, Moundsville, West Virginia. An analysis of the material is shown in Table C-2. The Cresap solvent was found to contain about 25 percent saturates and 75 percent aromatics.

Severe operating problems were experienced while using the Cresap solvent. The worst problem was repeated plugging of the atmospheric flash and high temperature, high pressure separator due to the limited solubility of the product SRC* in this solvent. Additional problems were encountered in pumping the feed slurry, as the coal did not remain suspended in the Cresap solvent.

The wet filter cakes obtained during product workup contained significant amounts of precipitated SRC. On the average, 68 percent of the product SRC was obtained from the filtrate and 32 percent from the wet filter cake.

*The SRC referred to here is that material which was soluble in pyridine and would also be soluble in normal SRC-I process solvent.

Yields for the SRC I process with the Cresap solvent are compared with those obtained with normal SRC I solvent below:

Yields, MF Coal Basis

	<u>Normal SRC I Recycle Solvent</u>	<u>Cresap Solvent</u>
	GU 127	DOE 287
Water	5.6	4.8
CO, CO ₂ , H ₂ S, NH ₃	2.7	2.5
C ₁ -C ₄	3.3	4.6
Naphtha (C ₅ -193°C)	5.0	4.7
Wash Solvent (193-249°C)	3.3	4.3
Recycle Solvent (>249°C)	5.4	14.9
Total Oil	13.7	23.9
SRC	61.4	49.8
IOM	5.5	6.0
Ash	9.6	9.9
Total	101.8	101.5
H ₂ Reacted	1.8	1.5

Under these conditions there was a substantially higher yield of recycle solvent (and total oil) obtained with the Cresap solvent. There was a decrease in SRC yield with the increased oil yield.

Elemental compositions of the SRC produced with the Cresap solvent must be estimated from the unfiltered vacuum bottoms residue as the filtered SRC samples were unintentionally discarded before analysis. Calculated ash free analyses are shown below. A correction for the estimated concentration of FeS in the unfiltered vacuum bottoms is included in the sulfur correction.

	<u>Ash Containing Vacuum Bottoms</u>	<u>Calculated Composition Ash Free SRC</u>	<u>Conventional SRC-I (GU 127)</u>
C, Wt %	74.05	86.77	86.35
H, "	4.60	5.39	5.79
S, "	2.10	0.96	0.84
N, "	1.72	2.01	---
Ash, "	14.66	---	0.23

E. Short Residence Time SRC I With Hydrogenated (Cresap) Solvent

Discussion of this run will be delayed until a later report.

F. Simulated Distillation By Gas Chromatography

Simulated distillation of the ASTM D-2887 type has been initiated at Merriam to determine the entire boiling range distribution of all product oils. In the past these have been characterized by only a three cut distillation.

Apparatus:

Gas Chromatograph - Hewlett Packard Model 5734A. Chromatograph equipped with dual matched columns, dual Flame Ionization Detectors (FID), dual Thermal Conductivity Detectors, and a Hewlett-Packard Model 7671A Automatic Sample Injector.

The chromatograph may be operated using either the dual FID detectors or the dual Thermal Conductivity Detectors. In addition, by using an effluent splitter both types of detectors may be operated simultaneously. To date, only the FID detectors have been used.

Columns - 18 inch by 1/4 inch stainless steel packed with either:

- a. 10% UCW-98 on 60/80 mesh Chromasorb G AW-DMCS.
- b. 10% UCW-982 on 80/100 mesh Chromasorb P AW.

Integrator - Spectra-Physics Model SP-4000 Chromatography Data System.

Procedure:

Calibration--A mixture of nineteen normal paraffin hydrocarbons ranging from $n-C_5$ to $n-C_{40}$ is injected into the gas chromatograph. The signal from the detector is sent directly to the data system where it is measured and integrated and the retention times of the n-paraffin hydrocarbons are recorded. The retention times, along with the respective atmospheric boiling points of the compounds, are then entered into a turnkey simulated distillation program and the calibration curve is generated within the integrator.

The calibration sample is run as needed to update the calibration curve. Calibration samples and product oil samples are injected into the chromatograph at identical instrument conditions which are specified below:

Instrument Conditions:

Initial Column Temperature	-30°C
Final Column Temperature	380°C
Initial Isothermal Period	2 min.
Programming Rate	11°C per min.
Carrier Gas	He
Carrier Gas Flow	60m l per min.
Detector Temperature	400°C
Injection Port Temperature	350°C
Sample Size	3 μ l

Oil samples are injected without solvent dilution and the signal is digitized by recording the normalized computed areas under the chromatogram at fixed intervals. There are two second intervals for the first thirty seconds in order to establish an initial zero baseline condition, and then thirty second intervals for the rest of the chromatogram.

Calculations:

All calculations are carried out with a Spectra-Physics proprietary simulated distillation program within the SP-4000 system. Temperatures are calculated and printed out at each 5% off interval plus the 0.5% off and 99.5% off intervals and are reported in degrees Centigrade.

Precision:

Repeatability is \pm one second of absolute retention time for each peak of the calibration mixture and is well within the limits of the ASTM method.

Accuracy:

In order to determine the applicability of this procedure to coal liquids, an aliquot blend of SRC-II product oils was analyzed and the resultant yield distribution was plotted along with the true boiling point yields of the same sample. The simulated distillation curve is compared with the true boiling point curve in Figure C-1. It should be noted that the true boiling point yields are expressed in weight % off whereas the simulated distillation yields are based on area %. No corrections have been employed for correcting the boiling points of polar compounds (e.g. phenols) which are known to deviate from true boiling point using this method. Maximum deviations appear at temperatures below 180°C and above 320°C. Between these values there is essentially no difference.

Conclusions:

From this comparison, and others, it is concluded that simulated distillation as described above is a viable alternative to the lengthy distillations needed to generate boiling point distributions of the product oils.

G. Merriam Maintenance and Modifications

1. Maintenance

- a. During runs with the hydrogenated (Cresap) solvent, the primary maintenance was cleaning of precipitated SRC from the bottoms of the atmospheric flash and the high temperature, high pressure separator. This was required as frequently as every three hours.
- b. During runs with hydrogenated solvent, solids also settled in the slurry feed vessels and in the feed pump checks causing erratic pump rates. Run DOE 287 was terminated when both Hills-McCanna pumpheads failed.
- c. Routine maintenance items included replacement of trim in the Gismo slurry letdown valve and the Research Control valves in the bottom of the intermediate and ambient temperature separators and gas letdown valves. It was also necessary to replace diaphragms in the PPI and Aminco compressors.

2. Modifications

- a. During Run 287 the unfiltered coal solution return line from the atmospheric flash to the product flask was replaced by a short line and the product flask was placed directly below the atmospheric flash. This facilitated removal of the precipitated SRC from the atmospheric flash.
- b. A larger filter was installed on the Lewa distillation feed pump. This Nupro 65 mesh filter will provide longer service and require less maintenance.
- c. Prior to run DOE 296R, the Aminco compression loop was converted to carbon monoxide. The following changes in the system were made:
 - i. A capillary was installed on the CO feed line to the compressor from outside the laboratory to limit the rate at which CO could enter the building.
 - ii. The output of the CO feed capillaries was connected to the output of the hydrogen feed capillaries. A short mixing chamber filled with stainless steel mesh was installed following the union of the two streams.
 - iii. In order to sample the feed gas without drastically affecting reactor pressure or the feed gas

flow rate, a branch off the feed gas line was installed with a capillary connected to a 90 cc sample bomb. The sample bomb can be filled slowly and then separated from the feed gas line. The gas in the sample bomb is then piped to the gas chromatograph for analysis. There was good agreement between the calculated and measured feed gas composition in the CO runs.

- d. In addition to the changes in the Aminco compression system, two Mine Safety Association carbon monoxide detectors were installed to sample the atmosphere at five locations. An alarm is sounded if the carbon monoxide level reaches 50 ppm.
- e. A Milton-Roy pump was installed in the reactor bay to provide a means to inject water into the line between the high temperature separator and the intermediate temperature separator. The purpose of the water injection was to eliminate the deposits of ammonium carbonate which had been forming during runs with carbon monoxide in the feed gas. The pump was operated for two days and then removed. Water leakage from the pump packing was excessive and the flow rate was erratic. The Milton-Roy pump was replaced by a Hills-McCanna pump. The Hills-McCanna pump was installed in the vapor-liquid separation bay with the versatility to pump water to the transfer line between the high temperature separator and the intermediate temperature separator, or to the top of the high pressure condenser located after the intermediate temperature separator.

VIII. Future Plans

The turnaround work detailed in this report at the SRC Pilot Plant will be completed and initial startup of the Lummus Unit will begin. After steady-state operation is attained, the primary Lummus objective will be to finalize target conditions for a demonstration run which is scheduled to begin May 15, 1979.

Process Development Unit P-99 will continue to focus on hydrogen partial pressure effects. Runs at conditions similar to Run No. 49 except for reactor outlet hydrogen partial pressures of 1330 and 1450 psia are planned. Higher severity vacuum flash operations and higher total feed slurry solids concentrations will also be explored.

Effective March 1, 1979, the Merriam Laboratory will operate under a separate contract, "Exploratory Research on Solvent Refined Coal Liquefaction". Future Merriam results will be reported under that contract (DOE contract No. DEAC-0179-ET14800).

A P P E N D I X A

SRC PILOT PLANT TABLES AND FIGURES

TABLE A-1

COAL PROCESSING SUMMARY - FIRST QUARTER 1979

	Coal Processed, Tons	On-Stream Time		Available Operating Time Adj. Hr. (a)	Average Feed Rate		On-Stream Factor		
		Days	Hours		Tons Per On-Stream Day	Pounds per On-Stream Hr.	Days, %	Hours, %	Adjusted Hr., % (a)
1979									
January	37	4	37	677	9.3	2,000	12.9	5.0	5.5
February	558	28	519	707	19.9	2,150	90.3	69.7	73.4
March	98	5	98	132	19.6	2,000	17.9	14.6	74.2
First Quarter	693	37	654	1,516	18.7	2,119	41.1	30.3	43.1
Year to Date	693	37	654	1,516	18.7	2,199	41.1	30.3	43.1

(a) Available operating time is adjusted for downtime not directly related to process problems or equipment failures.

TABLE A-2

SOLIDS SAMPLES FROM A DISSOLVER

<u>Sieve Analysis of Pyridine Insolubles</u>	<u>January 5, 1979 Sample Wt. % Finer</u>	<u>March 5, 1979 Sample Wt. % Finer</u>
6 Mesh	99.90	---
12 Mesh	72.92	91.84
30 Mesh	---	72.34
40 Mesh	0.87	---
60 Mesh	---	77.30
100 Mesh	0.27	69.90
200 Mesh	---	59.81
<u>% Ash in</u>	<u>January 5, 1979 Sample</u>	<u>March 5, 1979 Sample</u>
6 Mesh Holdup	89.81	---
40 Mesh Holdup	91.08	---
<u>% Pyridine Insolubles</u>	76.37	66.94

TABLE A-3

ANALYSIS OF DEPOSIT TAKEN FROM
HIGH PRESSURE FLASH VESSEL*

<u>Sieve Analysis on Ash</u>	<u>% Passed</u>		
	<u>Small Granules</u>	<u>Medium Lumps</u>	<u>Large Lumps</u>
12	82.43	50.41	71.49
20	48.58	37.65	55.77
40	28.00	28.42	41.62
100	12.65	17.89	26.85
140	9.17	14.98	23.11
200	6.73	12.74	20.28
	<u>% Ash</u>		
	38.96	24.95	16.61

*Sample taken January 13, 1979

TABLE A-4
LABORATORY ANALYTICAL RESULTS
ON
Material Balance Run No. 79-SR30

Start of Run : 1-28-79 @ 1000 hr. Coal : Powhatan No. 5

End of Run : 1-29-79 @ 1400 hr. Mode : SRC-II

Analyses on the composite of ground feed coal (sample point 105A) throughout the MBR run:

Carbon	<u>71.51 %</u>	Ash	<u>11.59 %</u>
Hydrogen	<u>5.24 %</u>	Moisture	<u>0.91 %</u>
Nitrogen	<u>1.41 %</u>		
Sulfur	<u>3.63 %</u>		
Oxygen (by diff.)	<u>5.71 %</u>		

Analyses on the composite of plant vacuum bottoms (sample point 8101) produced during the MBR run:

Carbon	<u>64.83 %</u>	Ash	<u>26.46 %</u>
Hydrogen	<u>3.72 %</u>	Fusion Point	<u>295 °F</u>
Nitrogen	<u>1.45 %</u>		
Sulfur	<u>3.02 %</u>		
Oxygen (by diff.)	<u>0.52 %</u>		

Analyses on the total liquid produced during MBR run (sample point 416) :

Carbon	<u>86.88 %</u>	Fractions:	
Hydrogen	<u>9.12 %</u>	Naphtha	<u>10.1 %</u>
Nitrogen	<u>0.99 %</u>	Middle Distillate	<u>43.3 %</u>
Sulfur	<u>0.27 %</u>	Residue	<u>46.6 %</u>
Oxygen (by diff.)	<u>2.74 %</u>		

Elemental analyses on liquid fractions from MBR total liquid (above):

	Naphtha	Middle Distillate	Residue
Carbon	<u>84.84 %</u>	<u>85.08 %</u>	<u>88.89 %</u>
Hydrogen	<u>12.82 %</u>	<u>9.20 %</u>	<u>8.24 %</u>
Nitrogen	<u>0.35 %</u>	<u>0.96 %</u>	<u>1.15 %</u>
Sulfur	<u>0.16 %</u>	<u>0.18 %</u>	<u>0.38 %</u>
Oxygen (by diff.)	<u>1.83 %</u>	<u>4.58 %</u>	<u>1.34 %</u>

TABLE A-5

MATERIAL BALANCE RUN 79SR-30RUN CONDITIONS

Raw Coal Feed:	2162
Net Dehumidified Coal Feed:	2099
Mbisture Free Coal Feed:	2080
Solvent Feed (as Seal Flush):	842
Recycle Slurry Feed Rate:	4128
Slurry Feed to Preheater:	7069
Feed Slurry Composition:	
Coal	29.6
Mid. Dist. (350° - 550°F)	2.3
Hvy. Dist. (550° - 850°F)	27.3
SRC	26.9
Ash (due to recycle slurry)	9.6
ICM (due to recycle slurry)	4.3
Deh. Coal Space Rate, #/hr/ft ³	22.8
Recycle Gas to Preheater, lb/hr	806.1
Purge & Quench Gas, lb/hr:	
Top	168.3
Middle	0
Bottom	0
Purge	114.8
Recycle Purge Gas*, lb/hr	30.0
Total Recycle Gas: (mass), lb/hr	1119.2
Total Recycle Gas: (volume), SCFH	82624
Recycle Hydrogen Purity (mole)	83.2
Hydrogen to Slurry Ratio: 1b H ₂ /100 1b Slurry	5.18
Slurry Heater Outlet Temperature, °F	749
Dissolver Pressure, psig	2002
Average Dissolver Temperature, °F	851
Dissolver Temperature °F	
4 ft	841
8 ft	847
12 ft	852
16 ft	854
20 ft	856
24 ft	855
28 ft	855
Run Duration, hrs	28

*Estimated

TABLE A-6

RUN 79SR-30 - RAW YIELD DATA

(Distillate Yields Based on TBP Distillation Technique)

<u>Component</u>	<u>#/HR</u>	<u>% MFC*</u>	
H ₂	-88.0	-4.2	
C ₁	124.0	6.0	} 15.2
C ₂	73.7	3.5	
C ₃	73.0	3.5	
C ₄	45.6	2.2	
C ₅ ⁺	0.0	0.0	
CO	-12.0	-0.6	
CO ₂	22.0	1.1	
H ₂ S	36.7	1.8	
Naphtha	138.2	6.6	} 37.4
Mid. Dist.	452.7	21.8	
lvy. Dist.	187.5	9.0	
SRC**	579.0	27.9	} 33.4
ICM	115.0	5.5	
Ash	242.2	11.6	
<hr/>			
TOTAL	1989.6	95.7	
H ₂ O + Loss	90.4	4.3	

* Average moisture free coal feedrate = 2080 lbs/Hr

** Average Fusion point = 312°F

TABLE A-7

ELEMENTALLY BALANCED YIELDS

<u>Component</u>	<u>YIELDS, %maf COAL FEED</u>	
H ₂	-4.4	
C ₁	6.8	} 17.3
C ₂	4.0	
C ₃	4.0	
C ₄	2.5	
CO	-0.6	
CO ₂	1.2	
H ₂ S	2.6	
NH ₃	0.6	
Water	5.7	
IQM	6.0	
Naphtha + C ₅ ⁺	7.1	} 40.0
Mid. Dist.	23.3	
Hvy Dist.	9.6	
SRC	31.6	
Fusion Point, °F	312	
% Solids in Vac. Btms.	38.2	

TABLE A-8

MEAN AND STANDARD DEVIATION DATA FOR CARBON MONOXIDE ADDITION TESTS

	<u>Data Set 1</u>		<u>Data Set 2</u>		<u>Data Set 3</u>		<u>Data Set 4</u>		<u>Data Set 5*</u>	
	<u>\bar{x}</u>	<u>s</u>	<u>\bar{x}</u>	<u>s</u>	<u>\bar{x}</u>	<u>s</u>	<u>\bar{x}</u>	<u>s</u>	<u>\bar{x}</u>	<u>s</u>
Carbon Monoxide in Feed Gas, vol%	2.44	0.12	5.09	0.47	10.06	0.42	2.56	0.14	2.50	0.14
Hydrogen Consumption, lbs/hr	88.7	4.74	91.5	5.14	60.9	7.76	73.8	7.64	81.2	9.79
Carbon Monoxide Yield, lbs/hr	1.1	2.13	-13.0	5.12	-17.1	13.51	-0.6	5.39	0.2	4.12
Carbon Dioxide Yield, lbs/hr	24.5	4.13	50.8	4.67	77.8	10.27	29.6	5.89	27.0	4.72
C ₁ -C ₄ Hydrocarbon Gas Yield, lbs/hr	311.2	11.63	325.9	10.16	329.5	21.68	368.6	22.73	339.9	34.13
Hydrogen Sulfide Gas Yield, lbs/hr	38.5	3.02	41.0	1.61	40.8	4.42	40.3	3.83	39.4	3.51

*: Data Set 5 is the average of all the data in Sets 1 and 4.

TABLE A-9

ANALYSIS OF VARIANCE

	<u>Sum of Squares</u>	<u>Degrees of Freedom</u>	<u>Mean Square</u>	<u>Calculated F*</u>
<u>HYDROGEN CONSUMPTION</u>				
Treatment	7246.2	2	3623.1	47.58
Error	4264.5	56	76.2	
Total	11510.7	58		
<u>CARBON MONOXIDE YIELD</u>				
Treatment	4148.9	2	2074.5	24.22
Error	4795.7	56	85.6	
Total	8944.6	58		
<u>CARBON DIOXIDE YIELD</u>				
Treatment	34454.1	2	17227.1	305.32
Error	3159.7	56	56.4	
Total	37613.8	58		
<u>C₁-C₄ HYDROCARBON GAS YIELD</u>				
Treatment	1855.2	2	927.6	1.15
Error	44996.5	56	803.5	
Total	46851.7	58		
<u>HYDROGEN SULFIDE YIELD</u>				
Treatment	32.2	2	16.1	1.10
Error	817.8	56	14.6	
Total	850.0	58		

* $F_{0.01, 2, 56} \approx 4.98$

TABLE A-10
SRC-II LIQUIDS INVENTORY*

<u>Date</u>	<u>Naphtha</u>	<u>Middle Distillate</u>	<u>Heavy Distillate</u>	<u>Total MD & HD</u>
10/2/78	116	839	260	1099
10/9/78	111	924	286	1210
10/16/78	169	1024	365	1389
10/23/78	185	1217	428	1645
10/30/78	186	1186	554	1740
11/6/78	192	1354	601	1955
11/20/78	296	1623	687	2310
11/27/78	308	1854	665	2519
12/4/78	274	1884	639	2523
12/11/78	278	1908	714	2622
12/18/78	326	1895	626	2521
1/8/79	426	1990	914	2904
1/15/79	420	1970	908	2878
1/22/79	441	1999	820	2819
1/29/79	412	2123	855	2978
2/5/79	455	2161	911	3072
2/12/79	380	2206	954	3160
2/19-79	367	2287	989	3276
2/26/79	380	2299	1016	3315

*All units are 42 gallon barrels

TABLE A-11CUMULATIVE PRODUCTION OF SRC-II LIQUIDS*

<u>Date</u>	<u>Naphtha</u>	<u>Middle Distillate</u>	<u>Heavy Distillate</u>	<u>MD + HD</u>
10/2/78	0	0	0	0
10/9/78	-5	85	26	111
10/16/78	53	185	105	290
10/23/78	69	378	168	546
10/30/78	70	347	294	641
11/6/78	76	515	341	856
11/20/78	180	784	427	1211
11/27/78	192	1015	405	1420
12/4/78	158	1045	379	1424
12/11/78	162	1069	454	1523
12/18/78	210	1056	366	1422
1/8/79	310	1151	654	1805
1/15/79	304	1131	648	1779
1/22/79	325	1160	560	1720
1/29/79	296	1284	595	1879
2/5/79	339	1322	651	1973
2/12/79	264	1367	694	2061
2/19/79	251	1448	729	2177
2/26/79	264	1460	756	2216

*Production=Inventory change + shipments (all units in 42 gallons barrels)

TABLE A-12

OVERALL MATERIAL BALANCE FOR SRC-II OPERATIONWITH POWHATAN COAL

10/2/78 through 2/26/79

<u>INPUT</u>	<u>QUANTITY</u>		<u>% MF COAL FEED</u>
M.F. Coal	2036	tons	100.0
Hydrogen ¹	83.5	tons	4.1
TOTAL	2119.5	tons	104.1
 <u>OUTPUT</u>			
Hydrocarbon Gas	321.7	tons	15.8
(C ₁ -C ₄) ¹	(7.7	MSCF)	(3.78 MSCF/ton of coal)
Other Gas ¹	77.4	tons	3.8
Recovered Distillate	433.7	tons (2480 BBLs)	21.3 ²
Recovered Vacuum Bottoms	780.0	tons	38.3 ³
Water ¹	118.1	tons (675 BBLs)	5.8
TOTAL	1730.9	tons	85.0
Loss	388.6	tons	19.1

¹ Based on Average Yield from Material Balance Runs 78SR-22 through 78SR-29.² Actual distillate recovered (Average MBR yield of distillate was 37.2% of MFC, or 2.05 BBL/ton)³ Actual vacuum bottoms recovered (Average MBR yield of V.B. was 41.5% of MFC)

TABLE A-13

FT. LEWIS ANTISOLVENT DEASHING UNIT MASS BALANCE COMPONENTS

<u>INPUTS</u>	<u>COMPONENTS</u>	<u>FLOW METER</u>	<u>SAMPLE POINT</u>
Slurry Feed	Process Solvent SRC Ash	1601	S1001 or 208
Antisolvent Makeup	Antisolvent	GA107	S1003
Stripper Steam	Water	1626	-----
Fractionator Steam	Water	1627	-----
Flushes, Process Flush Seal	Wash solvent Process solvent SRC	Numerous local Or estimate	PSA 414 FSA 428 SFA 417
<u>OUTPUTS</u>			
Underflow	Process Solvent SRC Ash	1629	S10B
Overflow	Process Solvent SRC Ash	1628	S1014
Antisolvent Purge	Antisolvent	1604	S1004
Sour Water	Water	----	-----

TABLE A-14

LUMUS UNIT MAJOR VESSEL HOLDUP VOLUMES

<u>Vessel</u>	<u>Equipment Number</u>	<u>Approximate Volume</u>	<u>Level Indicator</u>
Settler	DA-101	1500 ft ³	None (liquid full)
Surge Vessel	FA-101	160 ft ³	LT-1651
Stripper	DA-103	Negligible	LT-1652
Fractionator	DA-102	Negligible	LT-1653
Antisolvent Accumulator	FA-102	150 ft ³	LT-1654 (antisolvent level)
Antisolvent Accumulator	FA-102	Negligible	LT-1655 (water level)

TABLE A-15

COMPUTER SYSTEM RELIABILITY FOR
DECEMBER 25, 1978 THROUGH MARCH 24, 1979

<u>EQUIPMENT TYPE</u>	<u>% AVAILABILITY</u>
<u>Input Devices</u>	
A.) Operator's Console CRT	100.00
B.) Tape Reader	95.26
C.) Decwriters (Avg. 3)	100.00
D.) Magnetic Tape	100.00
E.) Process/Computer Interface	99.95
<u>Output Devices</u>	
A.) Tape Punch	95.83
B.) Decwriters (Avg. 3)	100.00
C.) Line Printer	100.00
D.) Magnetic Tape	100.00
E.) Operator's Console CRT	100.00
Central Processor	98.79
OVERALL SYSTEM	98.74

TABLE A-16

SRC PILOT PLANT SOLVENT INVENTORY

DECEMBER 25, 1978 THROUGH JANUARY 24, 1979

(All Units in Barrels)

SRC-II Mode

<u>Date</u>	<u>Time</u>	<u>PLANT INVENTORY</u>			<u>TANK FARM INVENTORY</u>			<u>TOTAL</u>			<u>HD + MD TOTAL</u>
		<u>Naph</u>	<u>MD</u>	<u>HD</u>	<u>Naph</u>	<u>MD</u>	<u>HD</u>	<u>Naph</u>	<u>MD</u>	<u>HD</u>	
1-08-79	0800	19	144	500	394	1805	382	413	1949	882	2831
1-15-79	0600	9	213	519	398	1655	357	407	1868	876	2744
1-22-79	0600	30	231	456	398	1666	320	428	1897	776	2673

TABLE A-17

RECYCLE SLURRY AIR COOLER DATA AND CALCULATIONS

Date	1/29/79	1/30/79	12/16/79	12/19/79	2/21/79
Time	1000 Hrs	0845 Hrs	0910 Hrs	0900 Hrs	0800 Hrs
Slurry Flowrate (lbs/hr)	4270	4150	4350	3950	4000
Stripper Bottoms Temp. (°F)	620	649	643	646	648
Slurry Inlet Temp. (°F)	556	578	580	546	572
Slurry Outlet Temp. (°F)	385	390	440	446	455
Blend Tank Temp. (°F)	355	357	370	390	343
Ambient Air Temp. (°F)	25	22	38	38	40
Outlet Air Temp. ^(a) (°F)	340	364	337	383	388
ΔT Log Mean ^(b) (°F)	305	303	317	283	296
Inside Area (Ft ²)	440	440	220	220	220
Heat Flux (Btu/Hr) ^(c)	438,000	468,000	365,000	237,000	281,000
Over Heat Transfer Coefficient (Btu/Hr-Ft ² -°F)	3.3	3.5	5.2	3.8	4.3

(a) Louver closed and fan off during operaiton.

(b) Based on average air temperature.

(c) An average slurry heat capacity of 0.6 Btu/Lb was assumed.

TABLE A-18

PREHEATER OPERATING CONSTRAINTS
FOR TYPICAL SRC-II AND SRC-I OPERATING CONDITIONS

	<u>SRC-II</u>	<u>SRC-I</u>
Slurry Feedrate to Preheater (LBS/Hr)	5000 - 8500	6000 - 13000
Dissolver Residence Time (Minutes)	80 - 45	37 - 22
Dissolver Temperature (°F)	850 - 860	845 - 855
Dissolver Pressure (PSIG)	2000	1500 - 2000
Hydrogen Feedrate to Preheater Inlet (SLF/HR)	10000 - 64000	40000 - 60000
Hydrogen Feed Temperature (°F)	60 - 360	60 - 360
Slurry Feed Temperature (°F)	300 - 370	150 - 200
Preheater Outlet Temperature (°F)	700 - 810	740 - 760
Feed Coal Size (Max. - Min.) (Mesh)	-30 - 200	-30 - 200
Preheater Pressure Drop, Maximum (PSIA)	500	500
Coal Feed Concentration in Slurry (WT%)	20 - 35	25 - 45

TABLE A-19

NEW SLURRY PREHEATER DIMENSIONS

Coil Size	2" SCH 160 Coil	1-1/2" SCH 160 Coil
Material	321 SS	321 SS
Length (FT)	467	467
Coil Wall Thickness (IN)	0.344	0.281
Coil I.D. (IN)	1.689	1.338
Flow Area (FT ²)	0.01552	0.00976
Internal Surface Area per Length (FT ² /FT)	0.442	0.350
Total Internal Surface Area (FT ²)	206	163
External Surface Area per Length (FT ² /FT)	0.622	0.498
Total External Surface Area (FT ²)	290	233
Coil Shape	Rounded Rectangle	
Radius of Bends (FT)	1.75	1.75
Number of Turns	13-1/2	13-1/2
Skin Thermocouple Locations	West End of Each Coil Turn	
Immersion Couples	Every Other Turn West End	
Differential Pressure Taps	Every Other Turn West End	
Inlet-Outlet Pressure & Immersion Couples	Outside Fire Box	
Coil Inlet-Outlet	West End	
Number of Burners	6	6
Location of Burners (double-fired)	Floor	Floor
Firing Control Point Outlet	Immersion Themocouple	
Flue Gas (excess air) Control	Stack O ₂ & Stack Damper	
Fuel Type	Natural Gas	
Air Control	Natural Draft	

TABLE A-20

PROPOSED SLURRY PREHEATER RUNS^(a)

Operating Mode	SRC-II				SRC-I	
Uninsulated Coil Length, Ft	467 Ft				467 Ft	
Coil Size	2" SCHD 160 Coil		1½" SCHD 160 Coil		2" SCHD 160 Coil	
Run Number	SRC-II-A	SRC-II-B	SRC-II-C	SRC-II-D	SRC-I-A	SRC-I-B
Slurry Feedrate, lbs/hr	5000	7500	8500	8500	10640	8500
Superficial Slurry Velocity, Ft/Sec.	1.2	1.9	2.1	3.3	2.6	2.1
Preheater Inlet Temperature, °F	350	350	350	350	150	350
Preheater Outlet Temperature, °F	750	750	750	750	750	750
Feed Coal Size, Mesh	-30	-30	-30	-30	-30	-30
Type Feed Coal	Powhatan	Powhatan	Powhatan	Powhatan	Kentucky	Kentucky
Dissolver Temperature, °F	860	860	860	860	850	850
Dissolver Pressure, PSIA	2000	2000	2000	2000	1500	1500
Dissolver Residence Time, Min.	80	53	47	47	24	30
Heat Input, Btu/Hr	1.56 x 10 ⁶	2.2 x 10 ⁶	2.5 x 10 ⁶	2.5 x 10 ⁶	2.9 x 10 ⁶	2.3 x 10 ⁶
Heat Flux per Area, Btu/Hr-Ft ²	7600	10600	12100	15300 ^(b)	14000	11000
Maximum Preheater ΔP, PSI	500	500	500	500	1000	< 500
Recycle H ₂ Purity, Mol %	85	85	85	85	95	95
Inlet gas feed rate, SCF/Hr and (Superficial Gas velocity, Ft/Sec)	32500 (6) 48800 (9) 65000 (12)	32500 (6) 48800 (9) 65000 (12)	32500 (6) 48800 (9) 65000 (12)	20400 (6) 30600 (9) 40800 (12)	32500 (6) 48800 (9) 65000 (12)	25000 (6) 42500 (9) 85000 (12) ^(b)

3-Week Erosion Run

Total Inlet Velocity, Ft/Sec	10	12	14	(select: 10-16)	12	8
------------------------------	----	----	----	-----------------	----	---

(a) All values of target conditions are estimates. The actual operating range may be limited by operating constraints.

(b) Run conditions may be limited by preheater ΔP.

TABLE A-21

DATA FROM NEW PREHEATER

Variables to Measure, Calculate or Test:

Inlet Temperature

Outlet Temperature

Pressure Inlet, Outlet, ΔP

Pressure Gradient Through Coil (9 points)

Skin Temperatures on Coil (14 points)

Fluid Temperatures in Coil (9 points)

Hydrogen Gas Rate (gas velocity)

Slurry Rate (velocity)

Sum of Velocities

Solids Concentration Maximum 50% Total

wt% Ash + IQM

wt% Coal

Type of Coal, Size of Coal Particles

Viscosity, Viscosity Gradient (compute from P, T gradients)

Outlet Slurry Composition (Need sample point, apparatus, technique)

Heating Rate, Flux

Coking Tendency

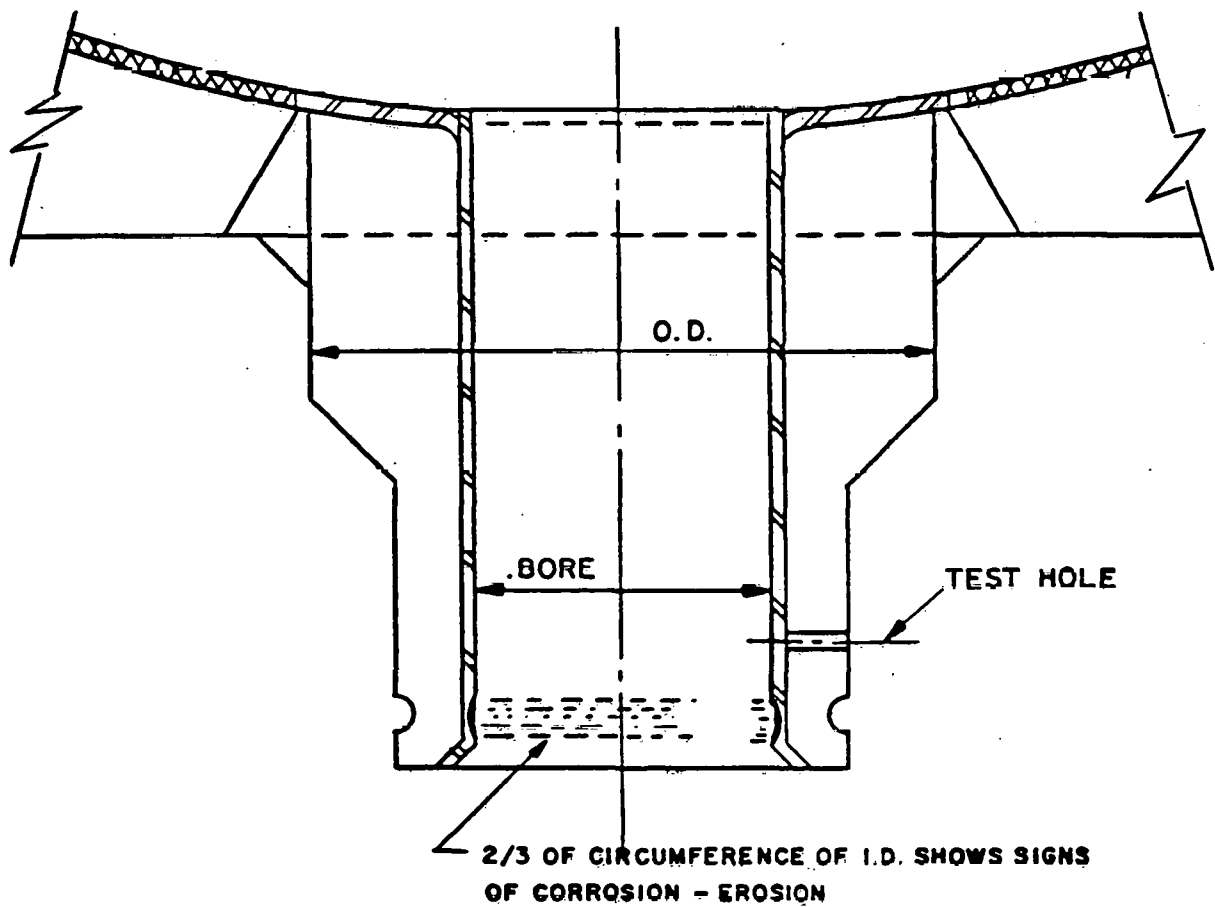
Coil Erosion - Corrosion (measure while shut down)

Furnace Efficiency, Excess Air, Flue -Gas Temperature

Fuel Gas Rate

FIGURE A-1

4" INLET DISSOLVER A NOZZLES



NOMINAL WALL THICKNESS = .125"

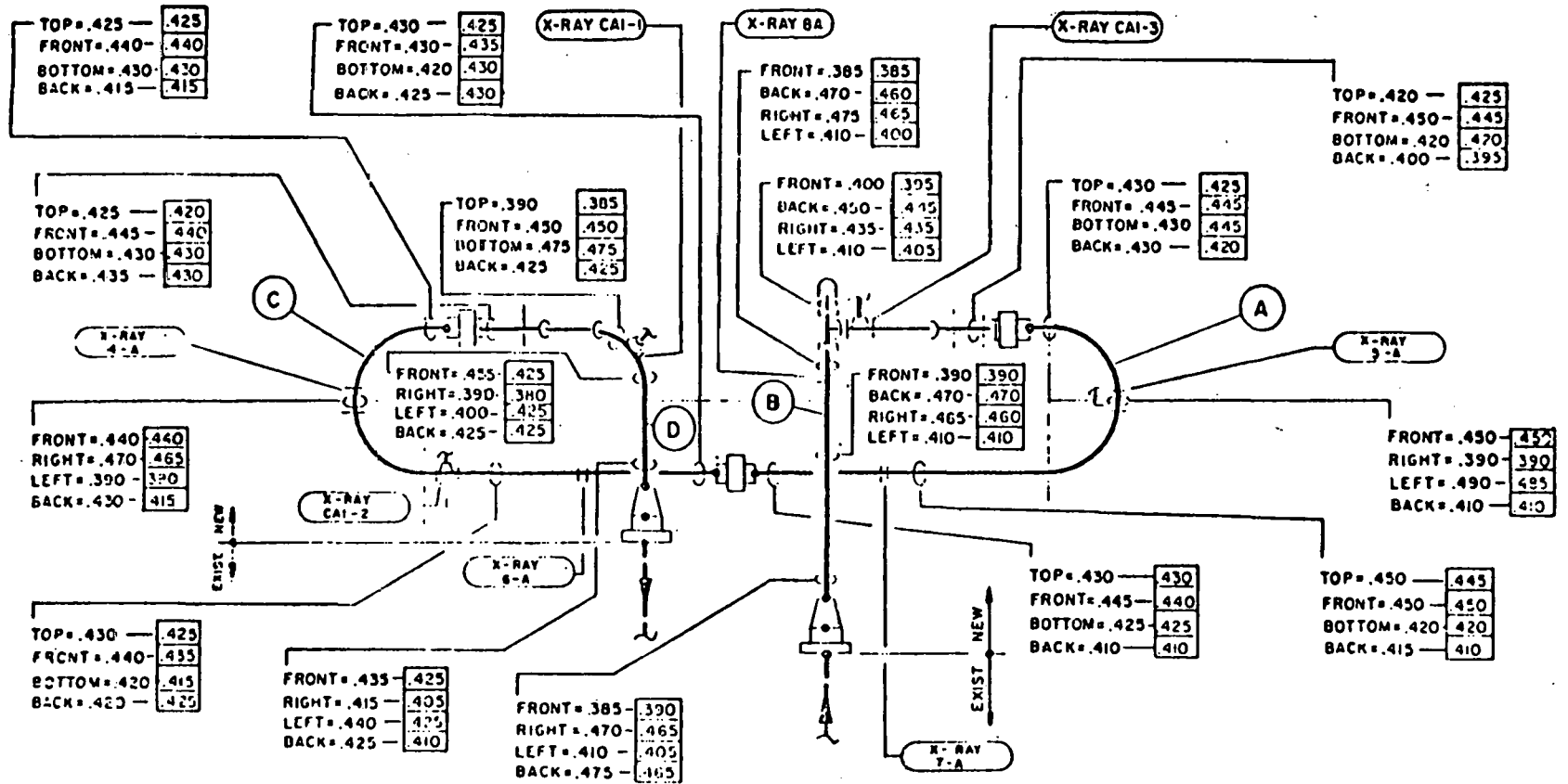
INSPECTED 1-4-79

SHOWS APPROXIMATE METAL LOSS OF .015"

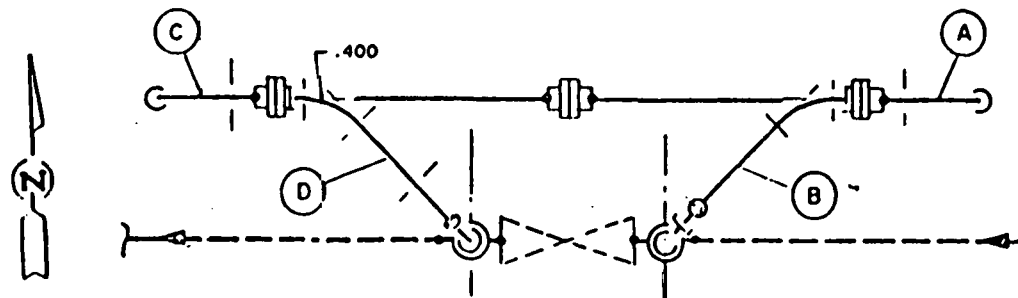
FIGURE A-2

CORROSION / EROSION TEST LOOP

ALL UNITS ARE IN INCHES



ELEVATION



PLAN

NOTE:

"FRONT" IS SOUTH FACE
 "BACK" IS NORTH FACE
 "RIGHT & LEFT" ORIENTATION
 IS LOOKING NORTH.

LEGEND:

INSTALLED READING = 9/5/78
 □ = INSPECTION DATE: 2/20/79

OPERATING TIME (> 630°F.) = 1789 HOURS

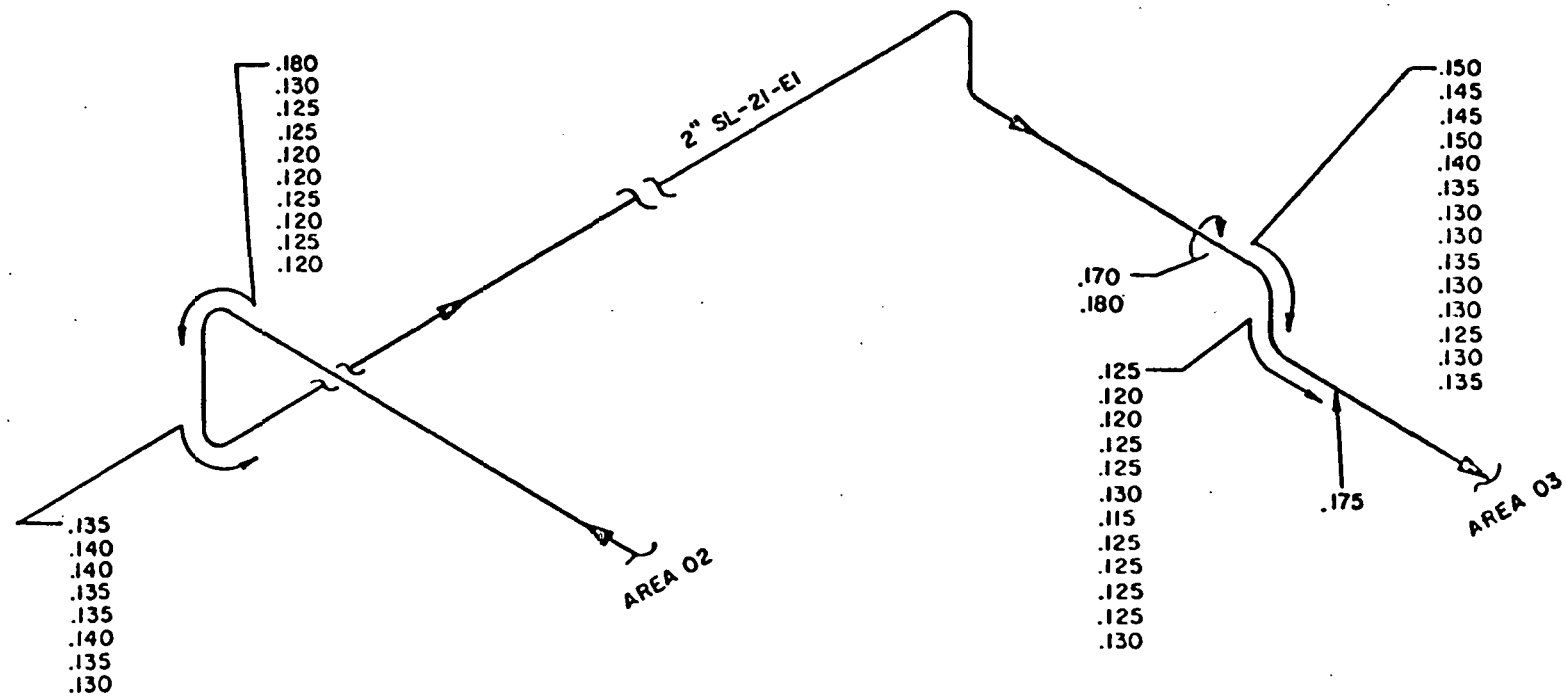
INSPECTED BY: L.G.F.
 DRAWN BY: R.L.M.

FIGURE A-4

WALL THICKNESS INSPECTION OF LINE SL-21-E1

AREA 02-03

ALL UNITS ARE IN INCHES



INSPECTION DATE : 3/15/79

2" CS SCH 80

NOMINAL WALL THICKNESS = .218

INSPECTED BY: L.G.F.
DRAWN BY R.I.M.

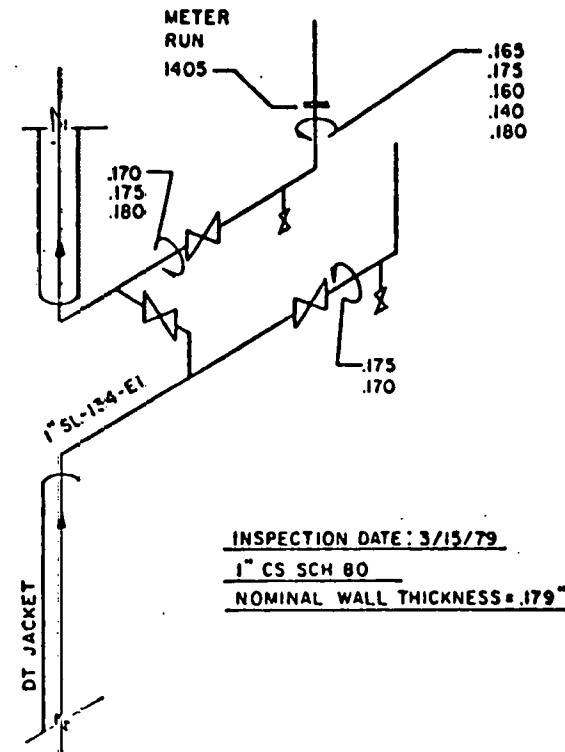
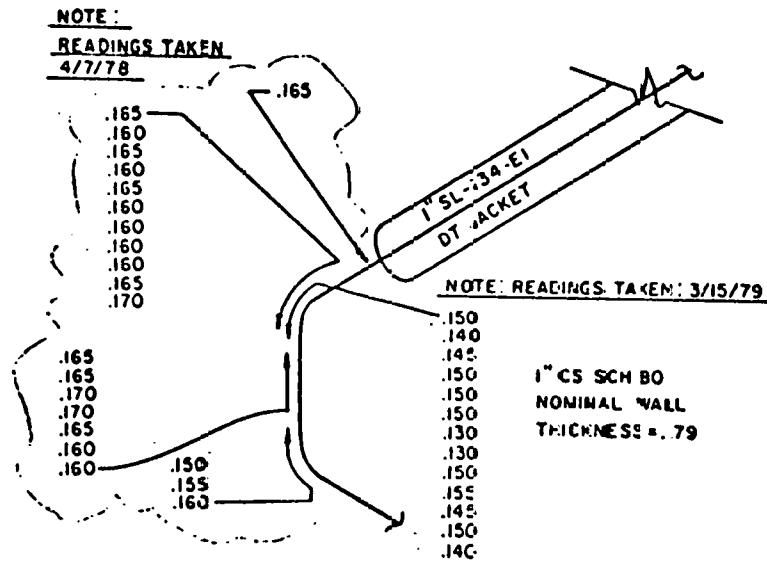
FIGURE A-5

WALL THICKNESS INSPECTION OF LINE SL-134-EI

AREA 02

ALL UNITS ARE IN INCHES

82



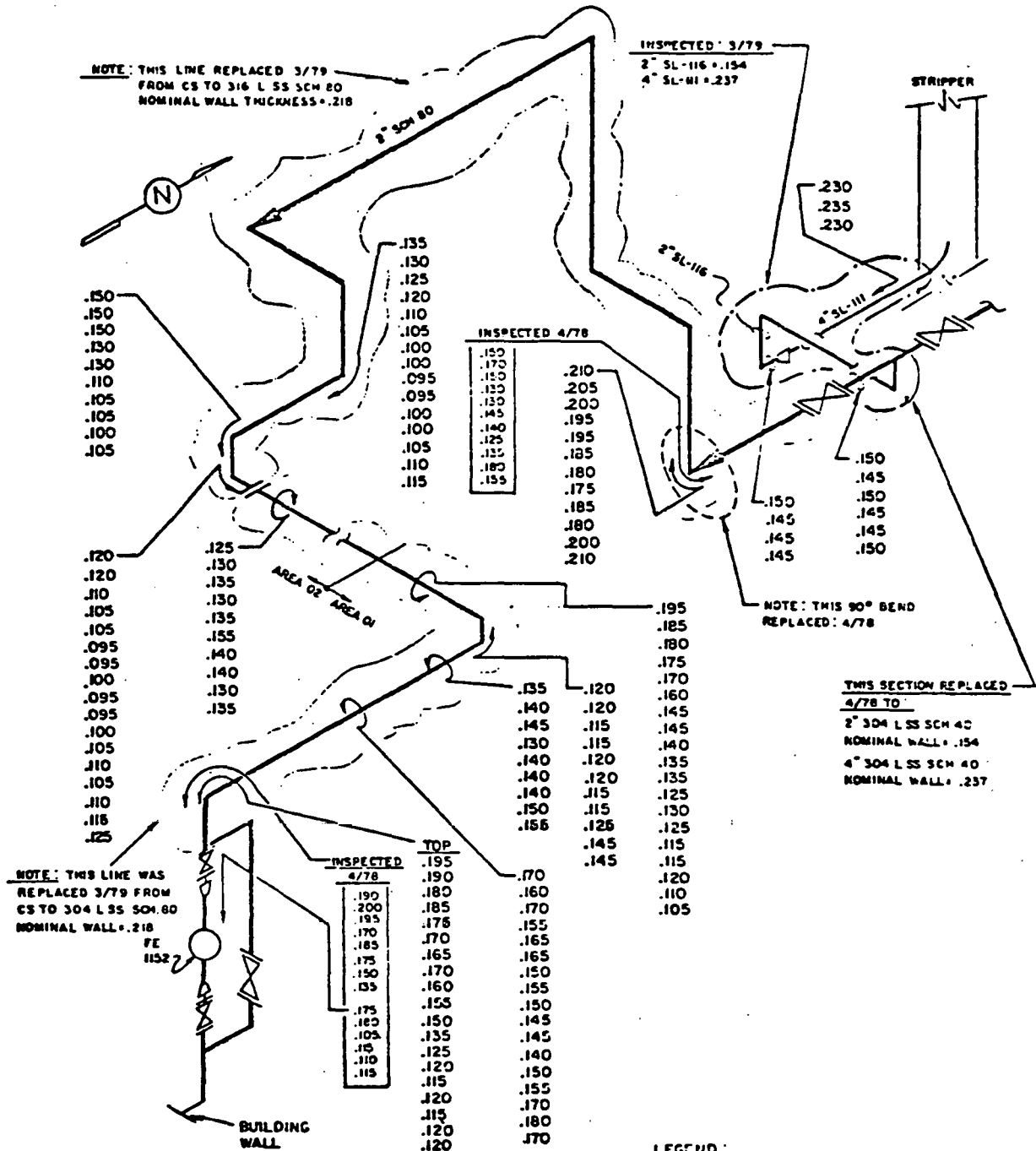
INSPECTED BY: L.G.P.
DRAWN BY: R.I.N.

FIGURE A-6

WALL THICKNESS INSPECTION OF LINE SL-II4-E3

AREA 02

ALL UNITS ARE IN INCHES

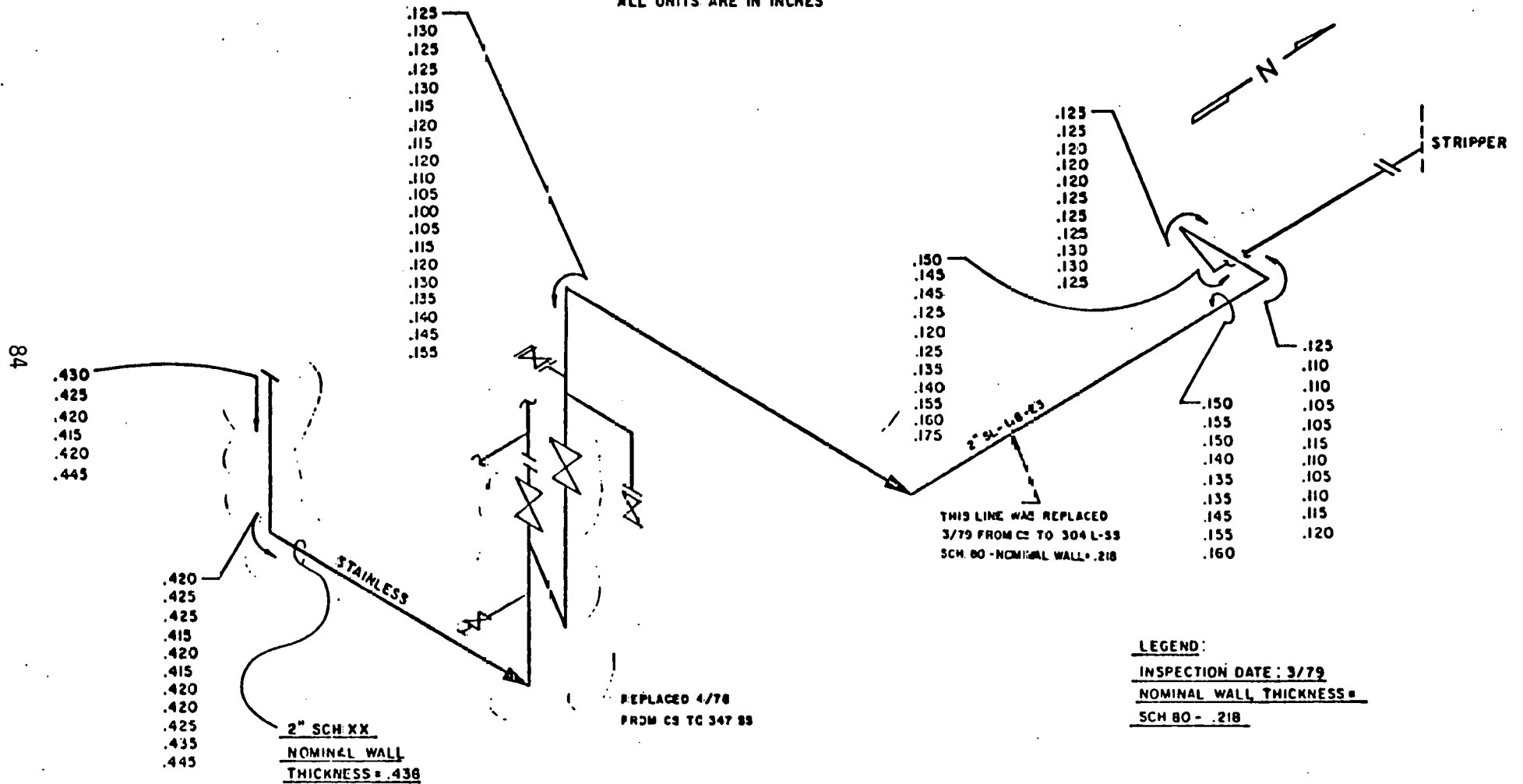


LEGEND:
INSPECTION DATE: 3/79
NOMINAL WALL THICKNESS =
SCH. 40 = .154
SCH. 80 = .218

FIGURE A-7

WALL THICKNESS INSPECTION OF LINE SL-118-E3
AREA 02

ALL UNITS ARE IN INCHES



INSPECTED BY: L.B.P.
DRWN BY: R.I.M.

FIGURE A-8

WALL THICKNESS INSPECTION OF LINE SL-110-E3
AREA-02

ALL UNITS ARE IN INCHES

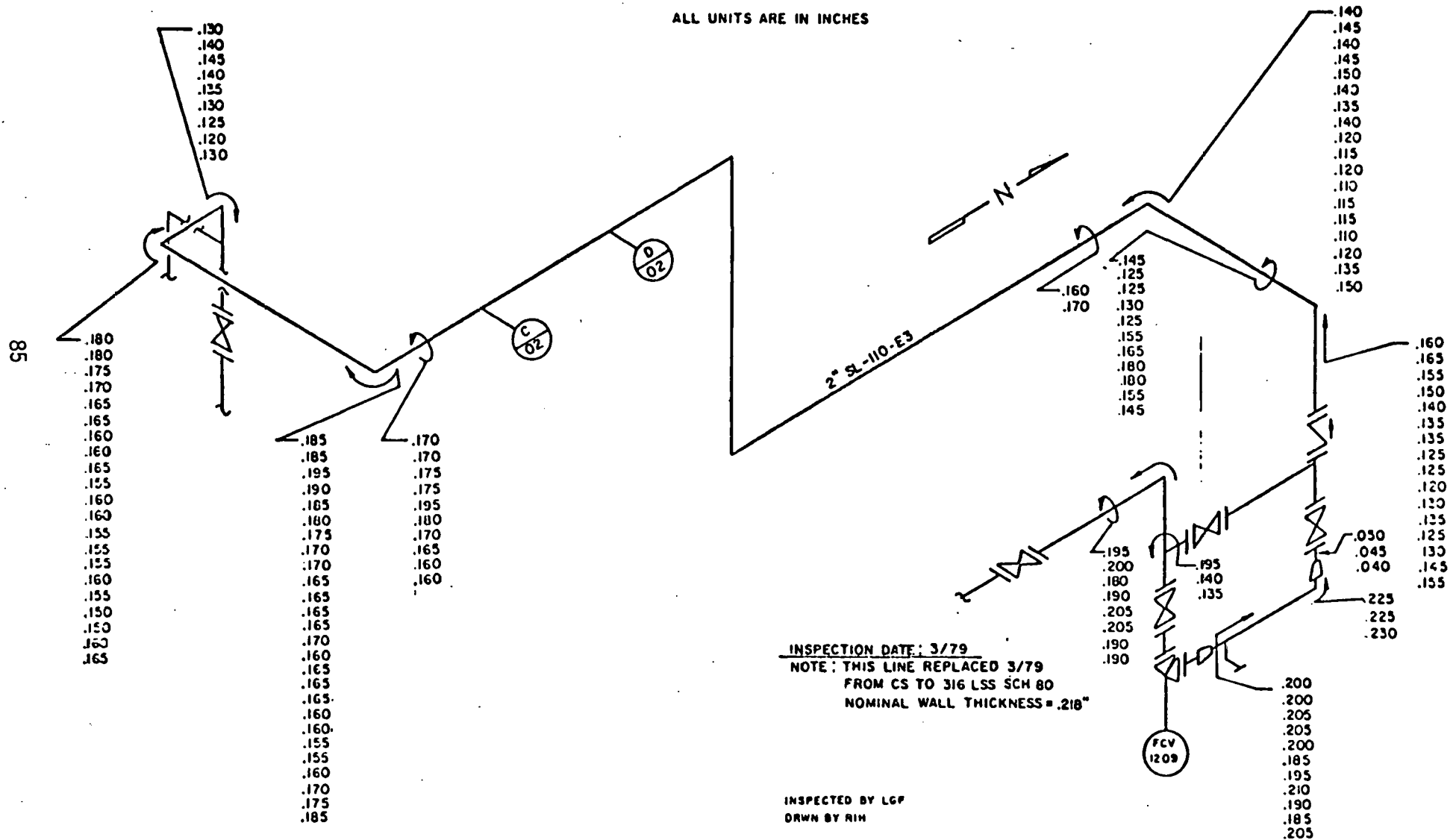


FIGURE A-9

WALL THICKNESS INSPECTION OF
FILTER FEED FLASH VESSEL, EQUIP. NO. 03D75506

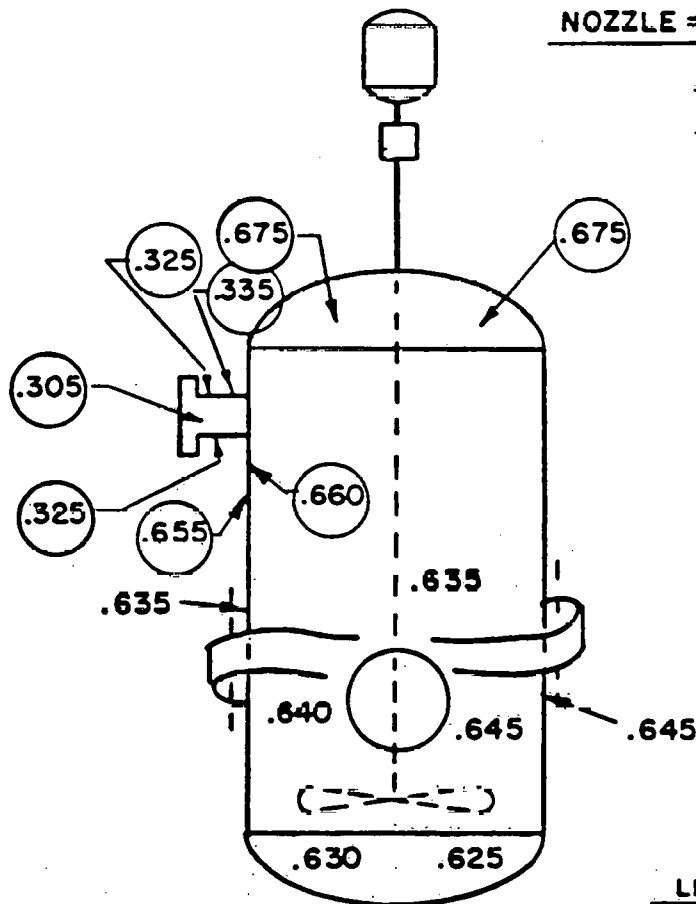
ALL UNITS ARE IN INCHES

NOMINAL WALL
THICKNESS = 0.687 "

TOP & BOTTOM = 0.687 "

NOZZLE = 4" SCH 120

.438" WALL
SA 106 B



LEGEND:

FIRST LOWER INSPECTION-4/78
TOP INSPECTION ○ - 1/5/79

6'-0" I.D. x 9'-0"

DESIGN 175 PSIG @ 175°F

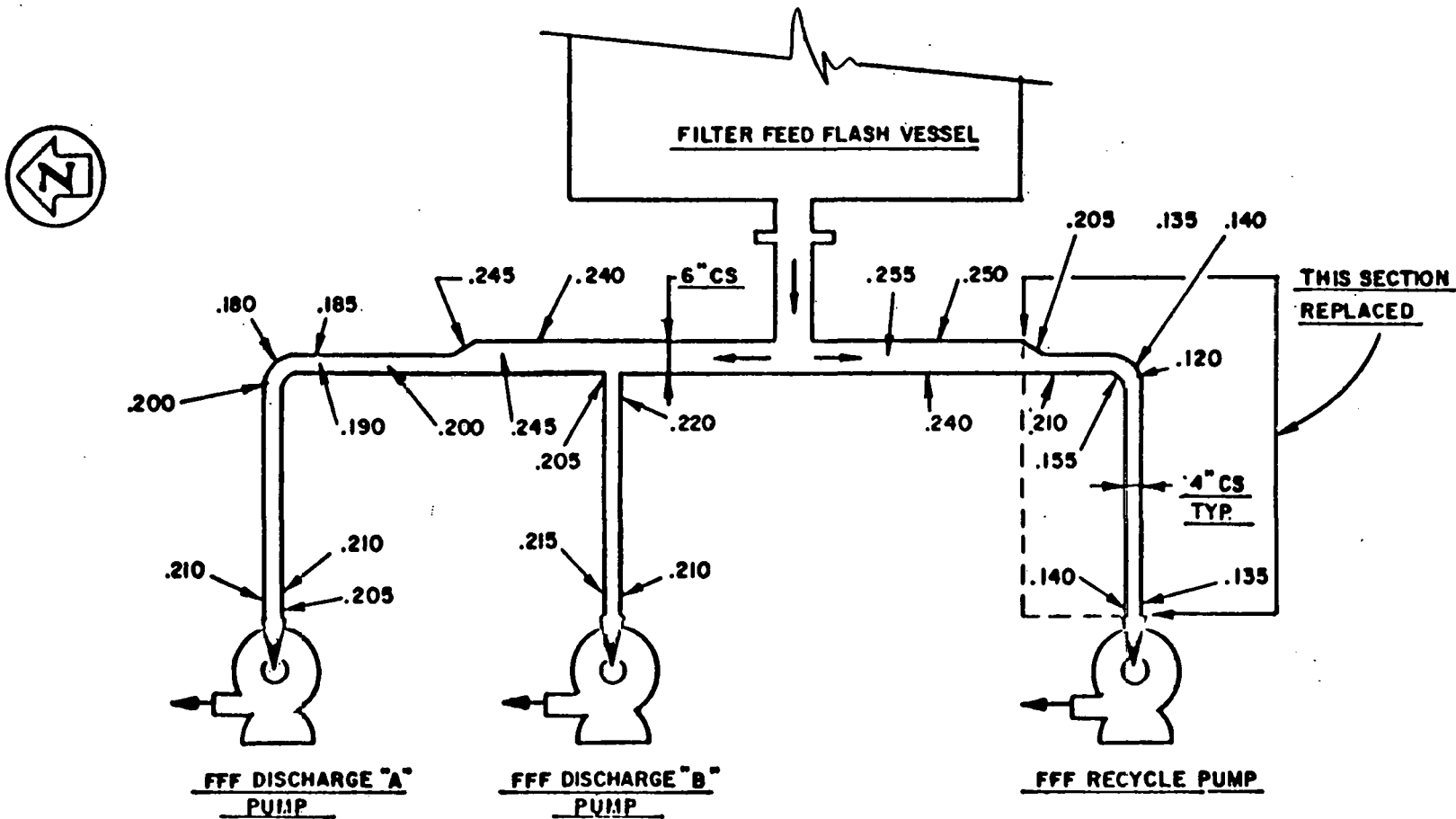
INSP. BY: L.F.
DRWN BY: RH

FIGURE A-10

WALL THICKNESS INSPECTION OF
SUCTION PIPING TO FILTER FEED FLASH VESSEL

(SL-25-E1)

ALL UNITS ARE IN INCHES



INSP. BY: L.F.
DRWN BY: R.H.

INSPECTION DATE: 2/9/79
6" SCH. 40 CS NOMINAL WALL = .280"
4" SCH. 40 CS NOMINAL WALL = .237"

88

AREA 03

ALL UNITS ARE IN INCHES

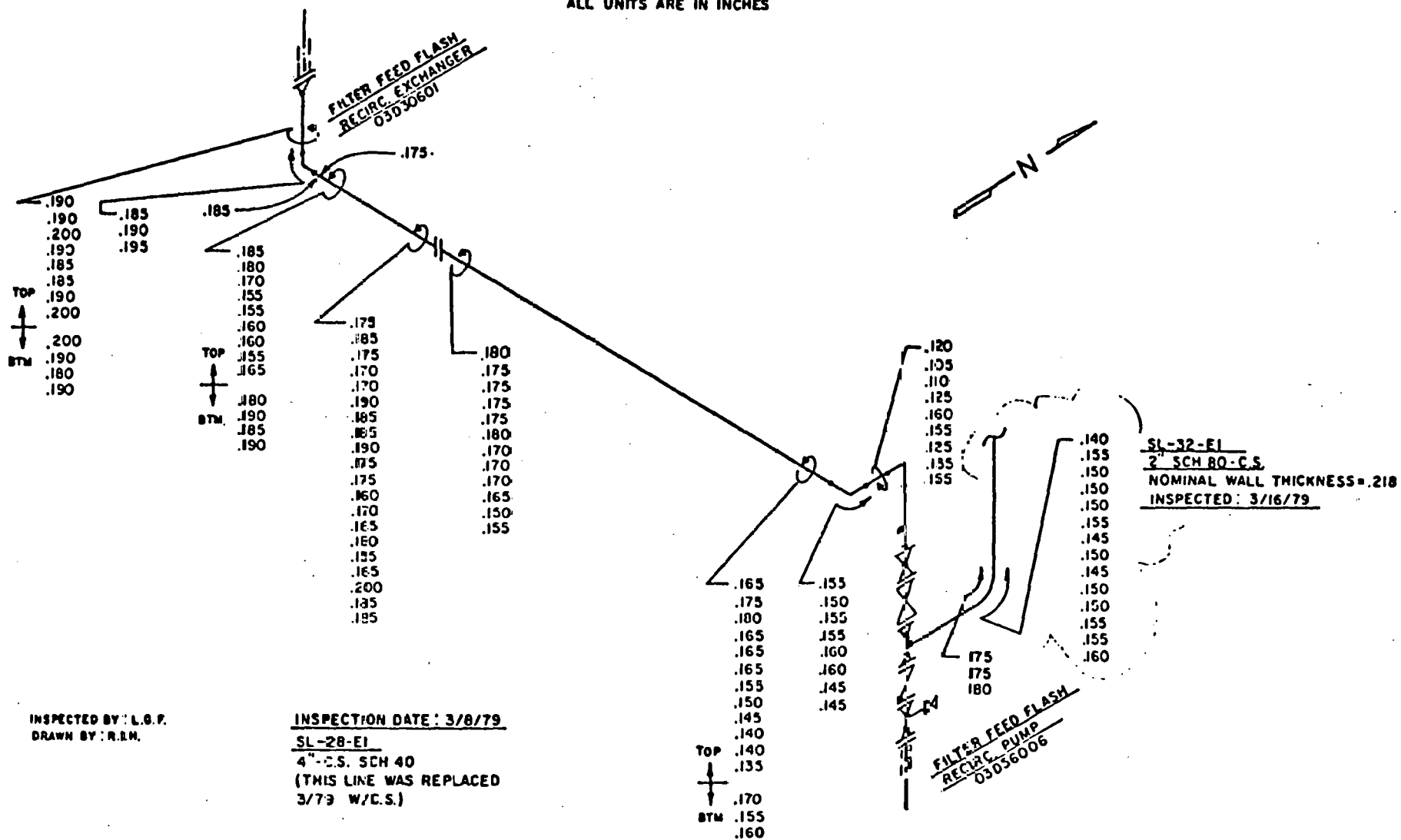
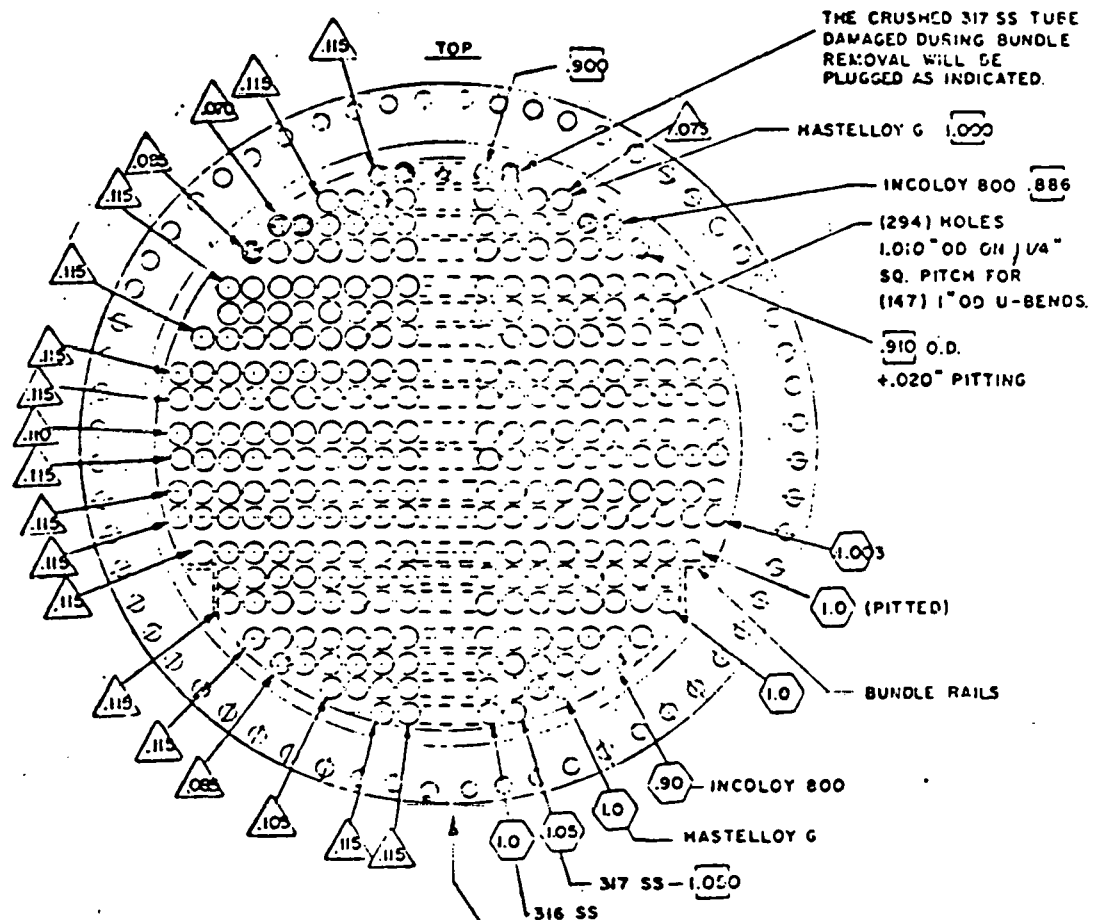


FIGURE A-12

TUBE THICKNESS INSPECTION OF WASH
SOLVENT COLUMN REBOILER

ALL TUBES ARE 316 SS WITH EXCEPTION OF SIX TUBES WHICH ARE OTHER MATERIALS AS LISTED:	
ORIGINAL (3-18-77) WHEN INSTALLED 316 SS WALL THICKNESS .120" 316 SS O.D. 1.0"	HASTELLOY G WALL THICKNESS .083 HASTELLOY G O.D. 1.0"
317 SS WALL THICKNESS .115", 317 SS O.D. 1.05"	INCOLOY 800 WALL THICKNESS .095" O.D. 1.0"

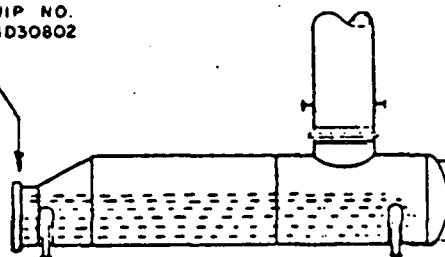


NOTE - NEW TUBES INSTALLED MAR. 77

LEGEND:

- ⊙ = PLUGGED TUBES
- △ = 4-19-78 (WALL THICKNESS) INCHES
- ⬡ = 4-19-78 (TUBE O.D.) INCHES
- = 3-8-79 INSPECTION (WALL THICKNESS) INCHES

EQUIP. NO.
04030802



INSPECTED BY L.S.F.
DRAWN BY R.H.

FIGURE A-13

WALL THICKNESS INSPECTION OF WASH SOLVENT COLUMN REBOILER

ALL UNITS ARE IN INCHES

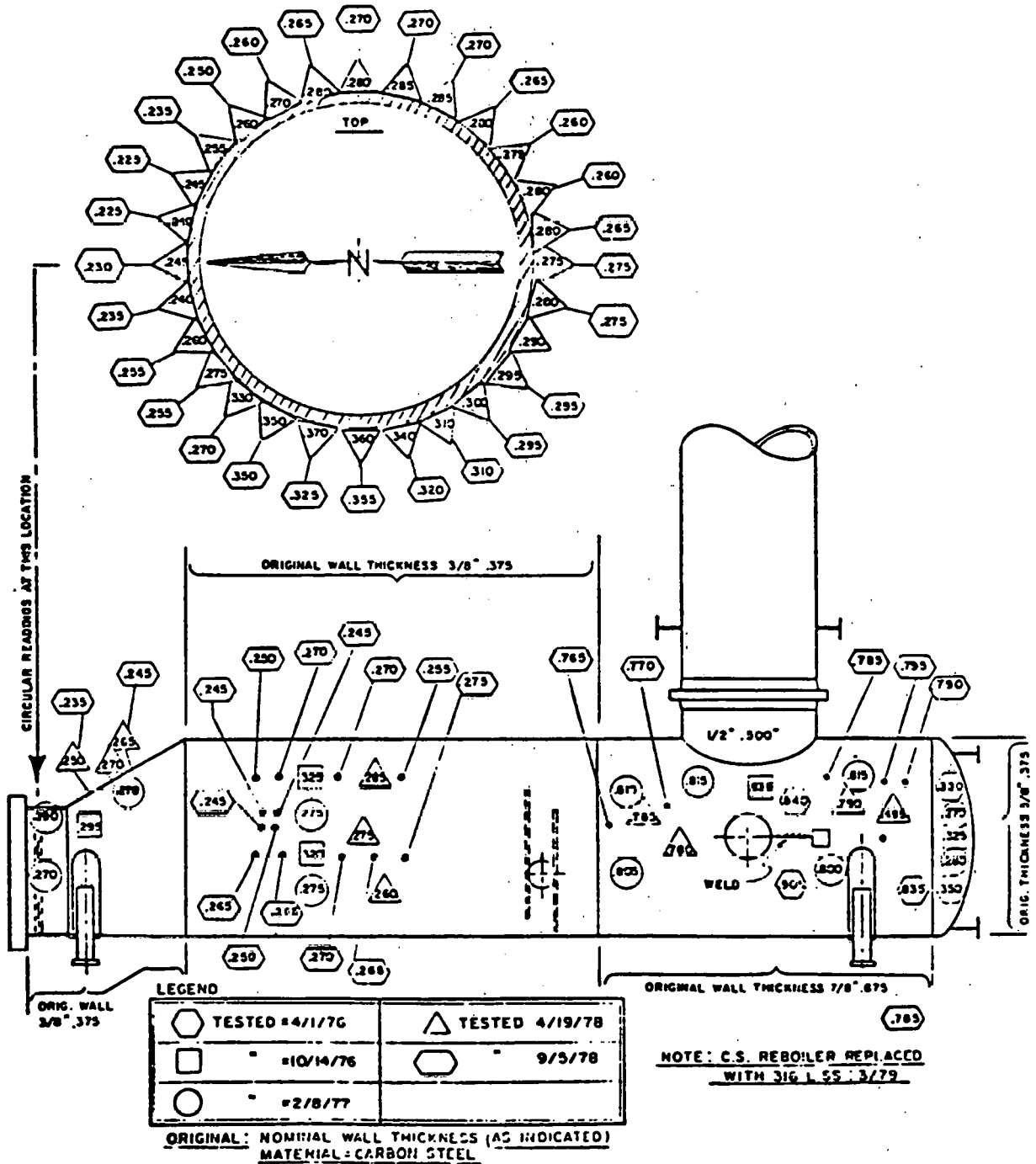
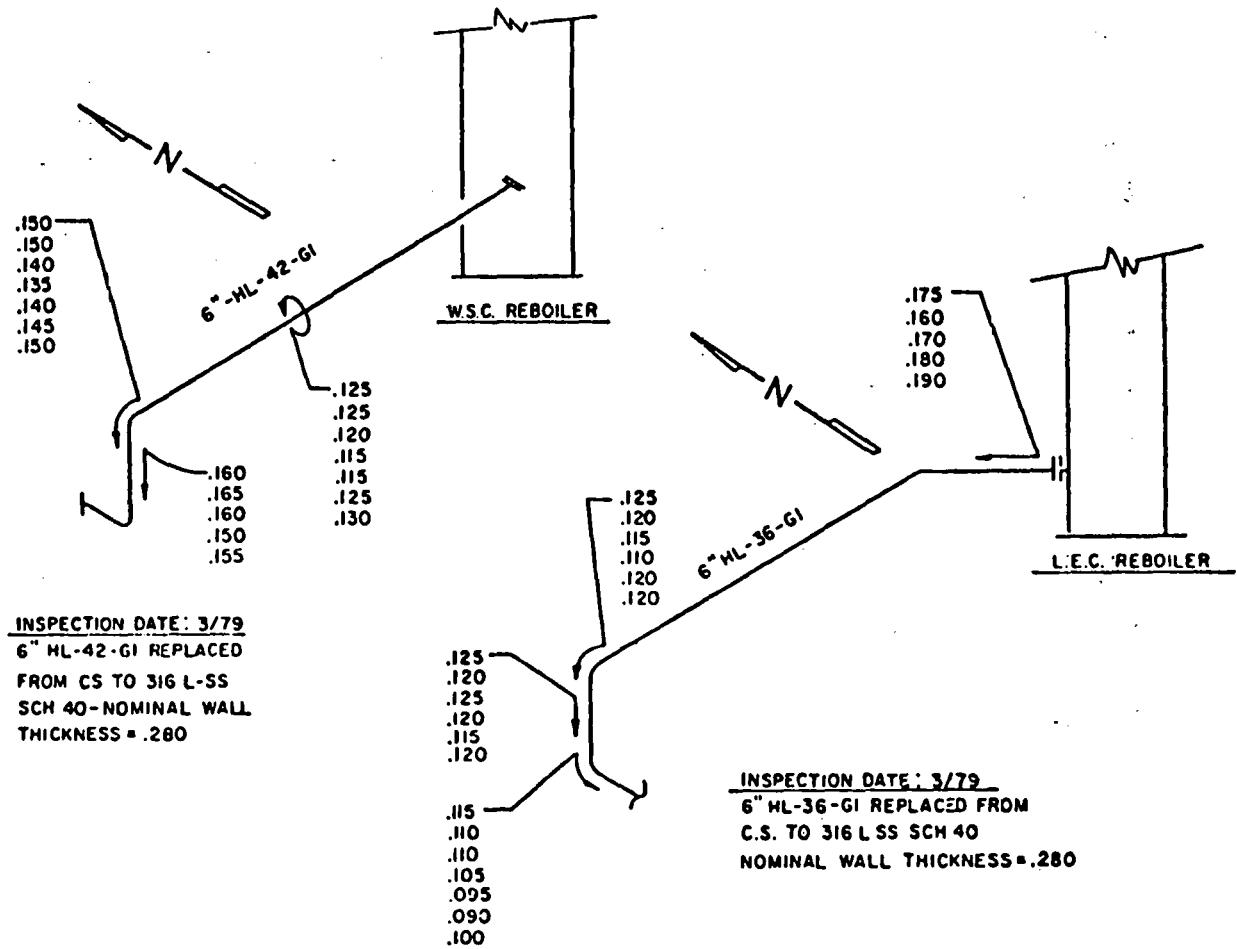


FIGURE A-14

WALL THICKNESS INSPECTION OF LINES HL-42-GI

AND HL-36-GI

ALL UNITS ARE IN INCHES



INSPECTED BY: L.G.P.
 DRAWN BY RIM

FIGURE A-15

QUADRANT-EDGED ORIFICE PLATE FLOWMETER CONFIGURATION - 1/79

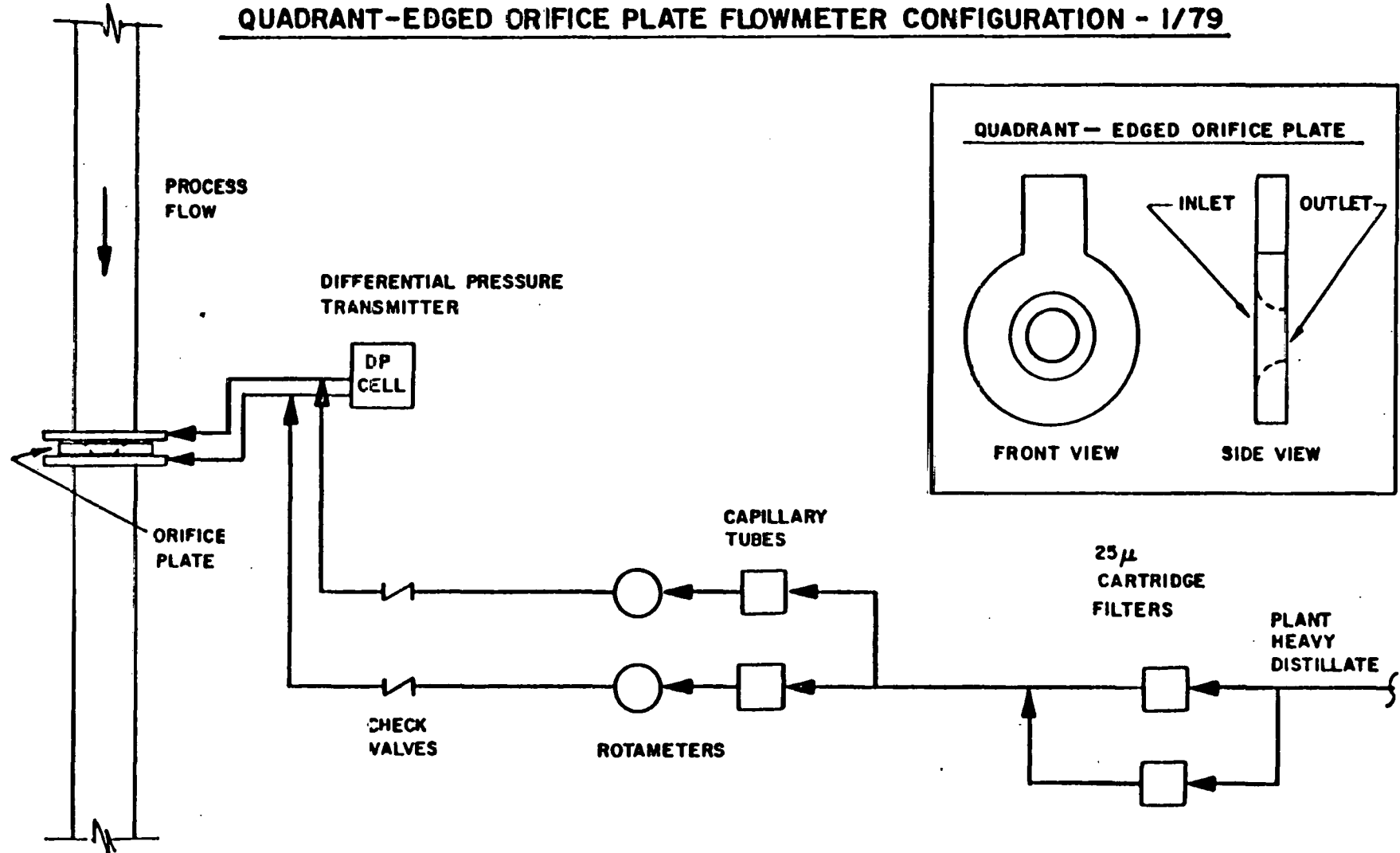


FIGURE A-16

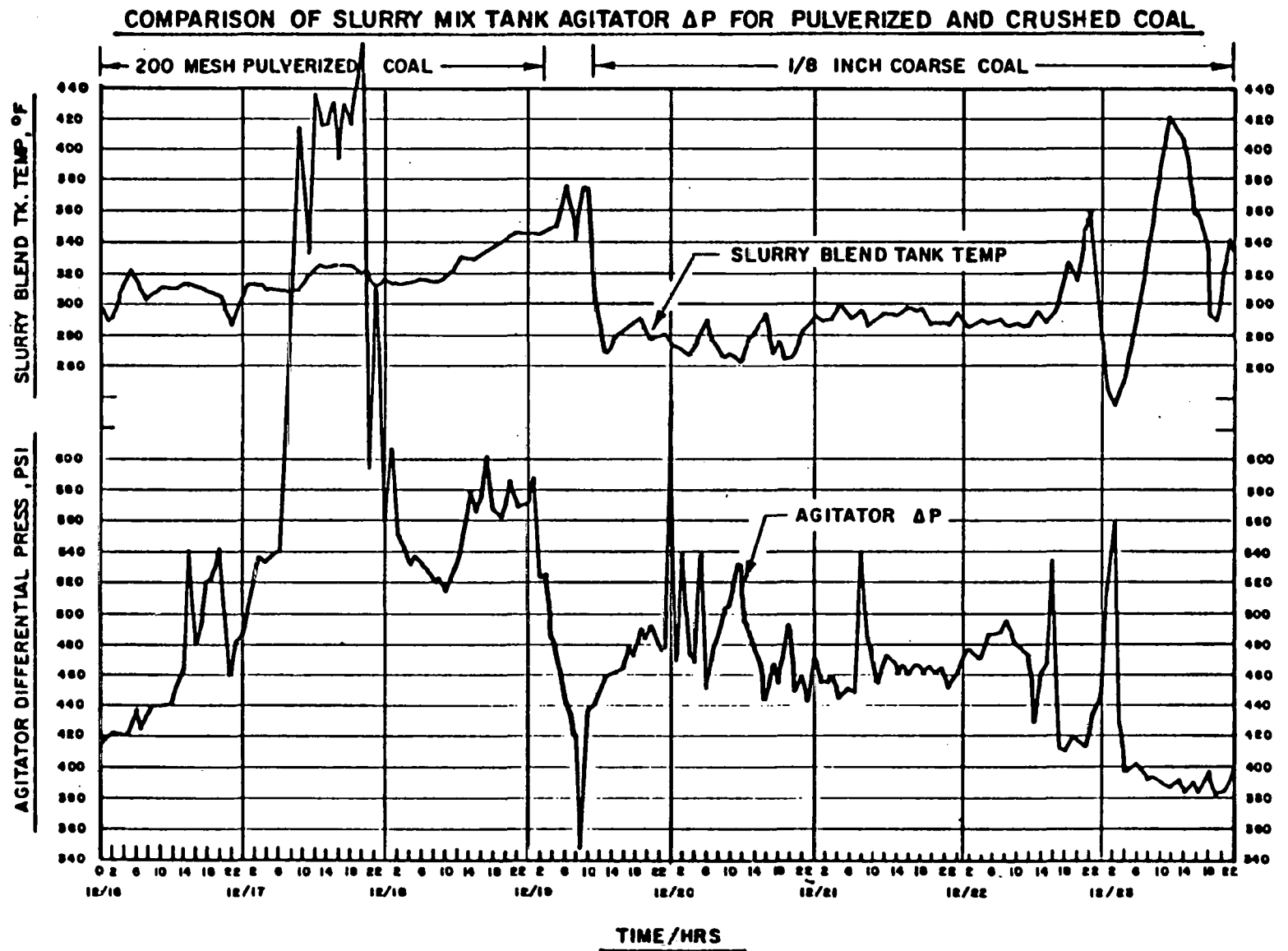


FIGURE A-17

SLURRY BLEND TANK TEMPERATURE AND AGITATOR ΔP

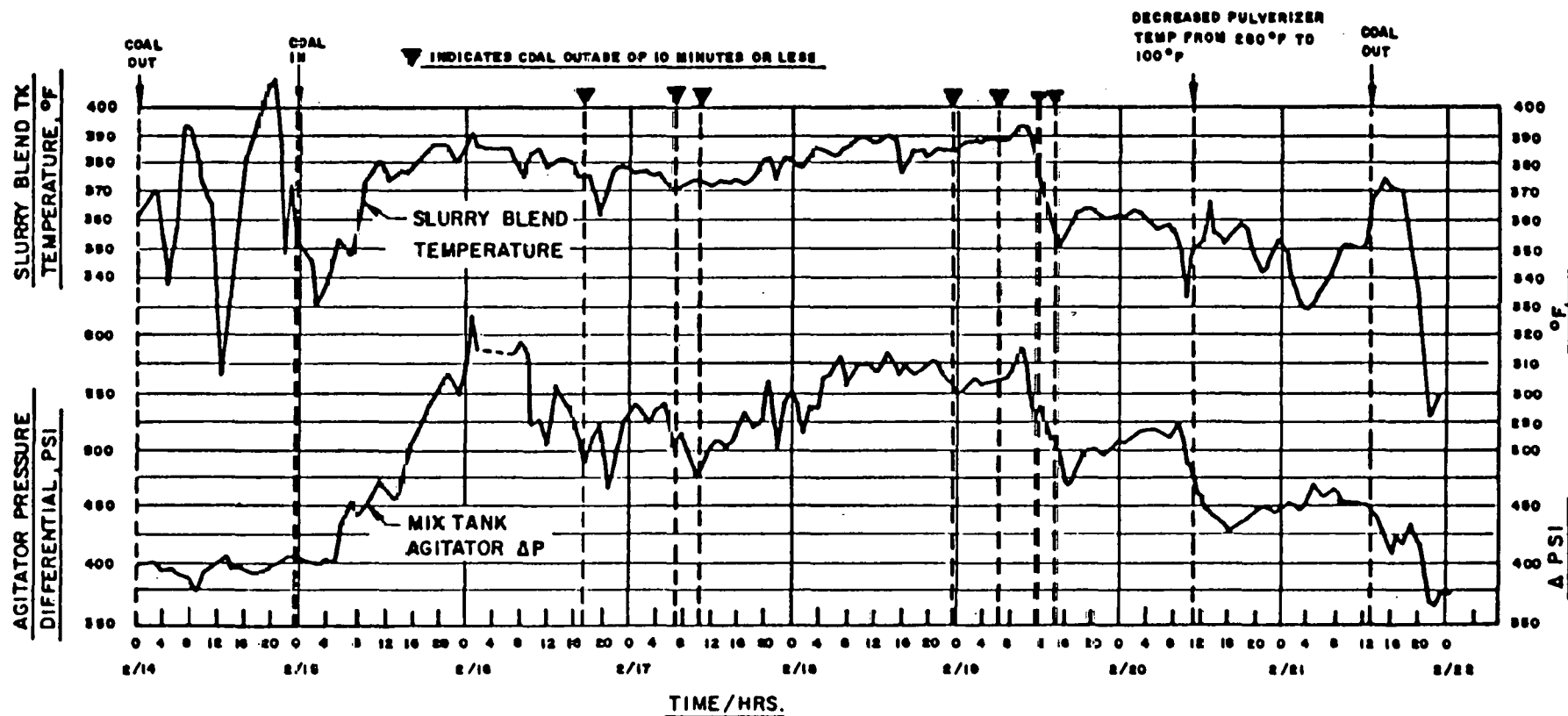


FIGURE A-18

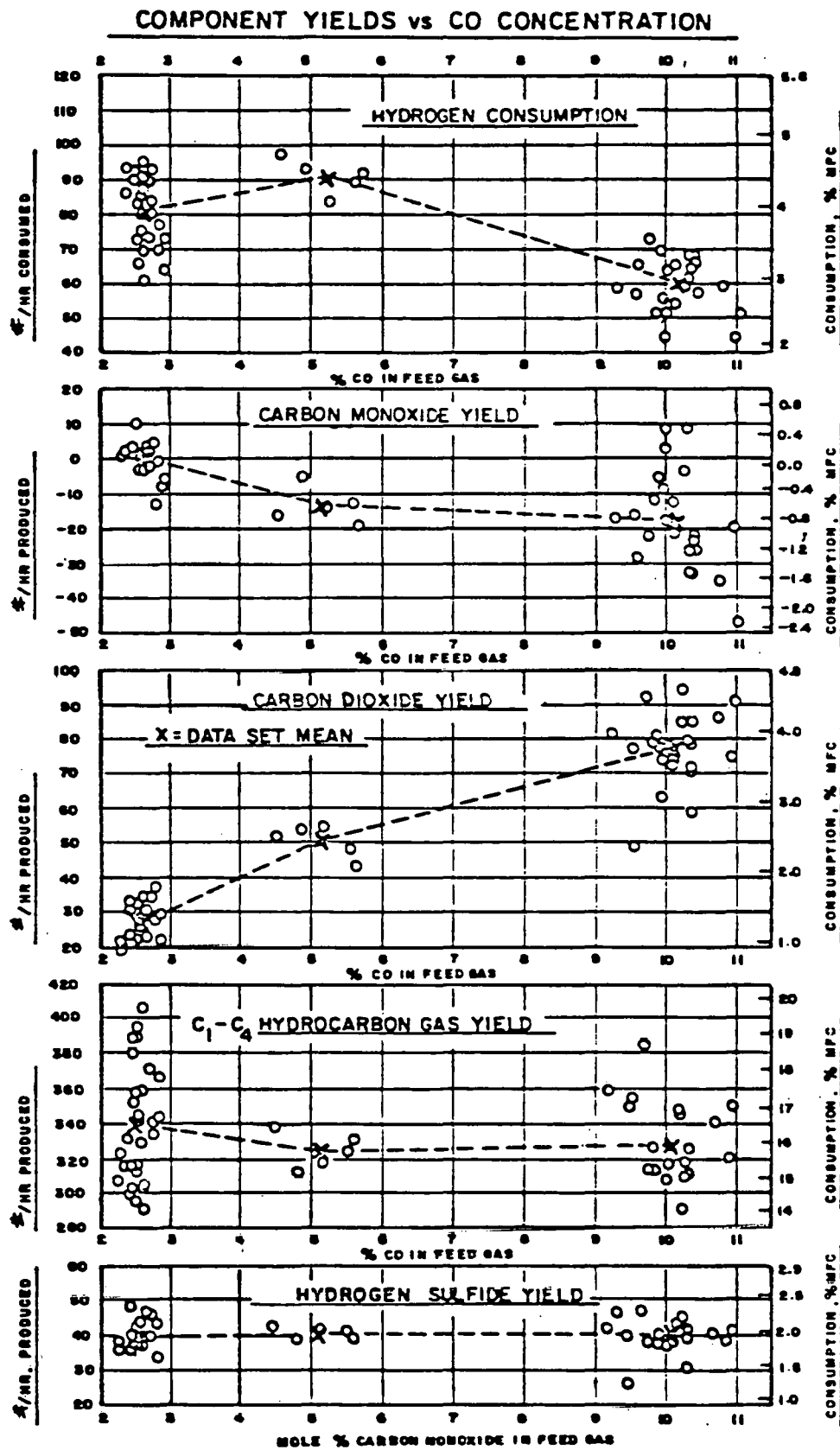


FIGURE A-19

COAL FEED AND RECYCLE SLURRY PARTICLE SIZE DISTRIBUTION

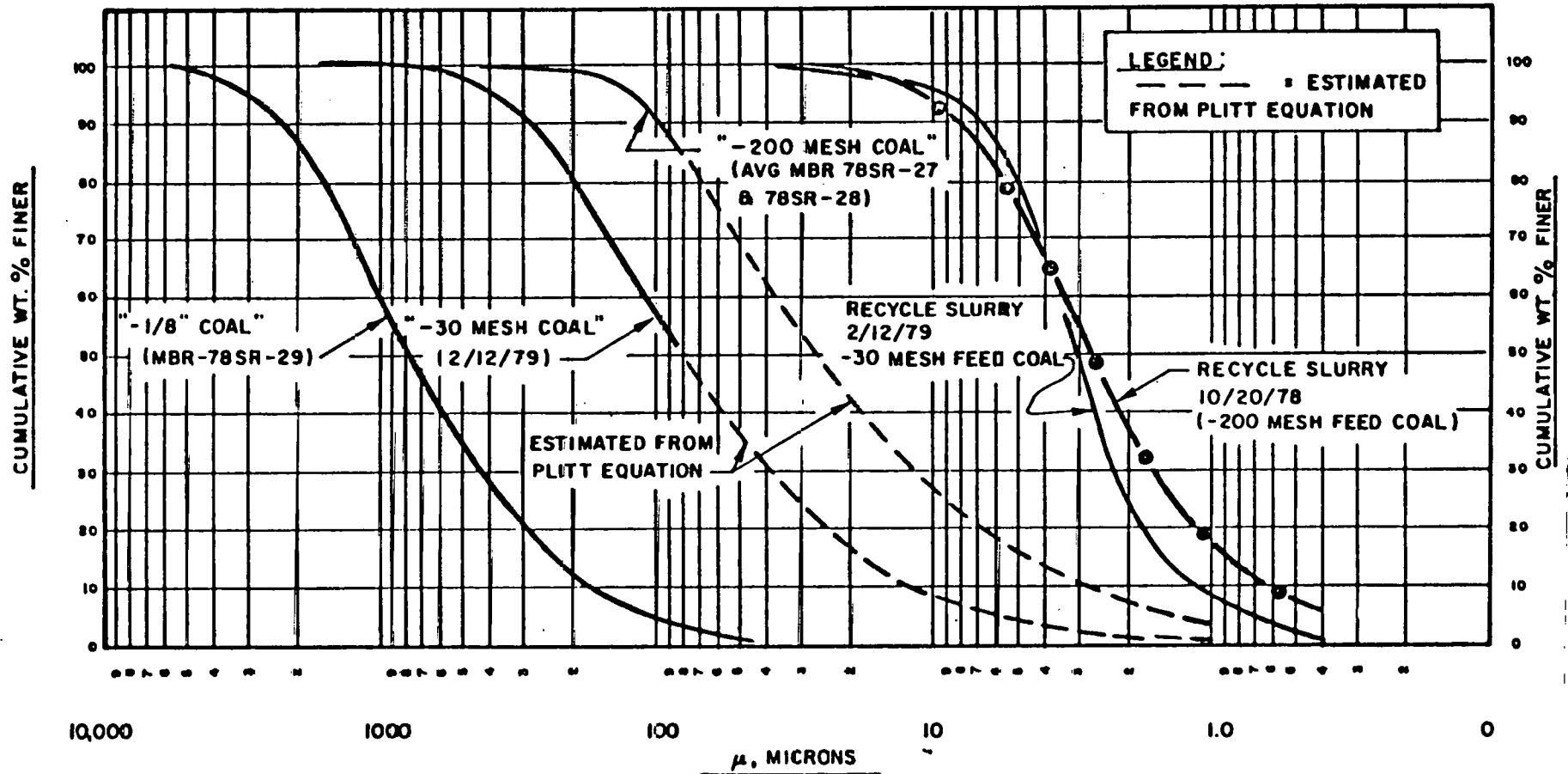


FIGURE A-20

SRC-11 LIQUIDS INVENTORIES

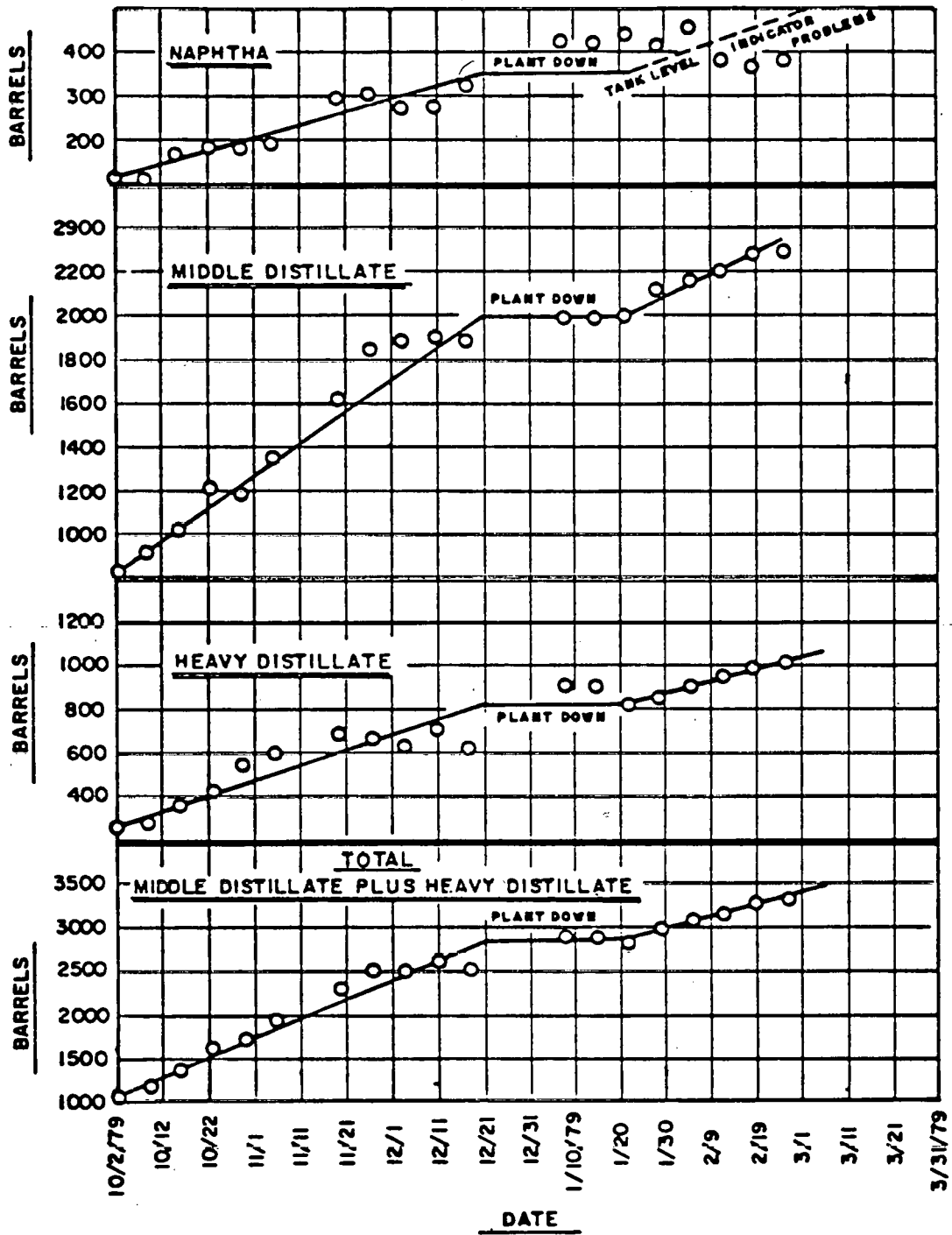


FIGURE A-21

FT. LEWIS ANTISOLVENT DEASHING UNIT MASS BALANCE ENVELOPE

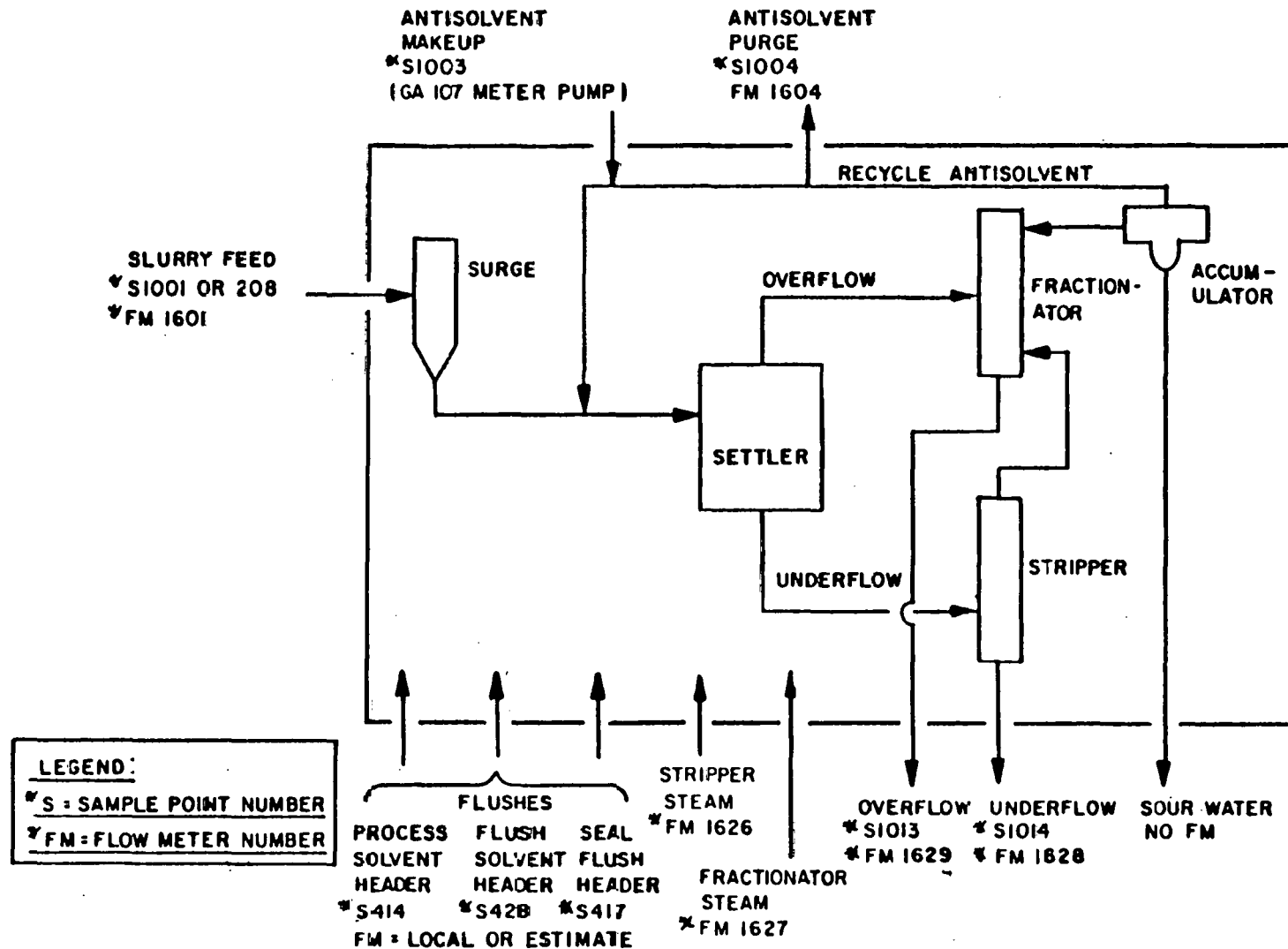
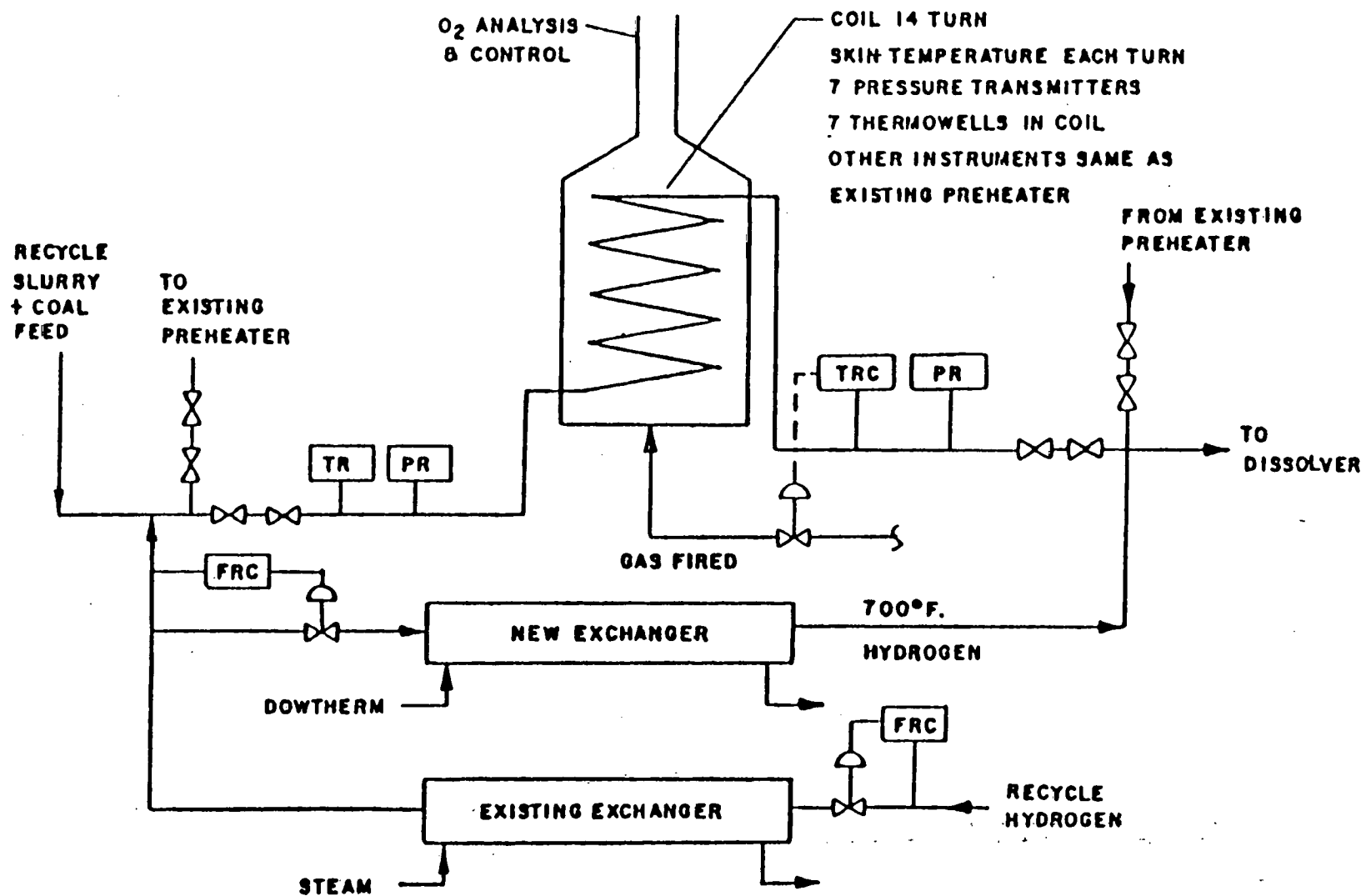


FIGURE A-22

SIMPLIFIED FLOW DIAGRAM OF NEW HEATER



A P P E N D I X B

PROCESS DEVELOPMENT UNIT P-99

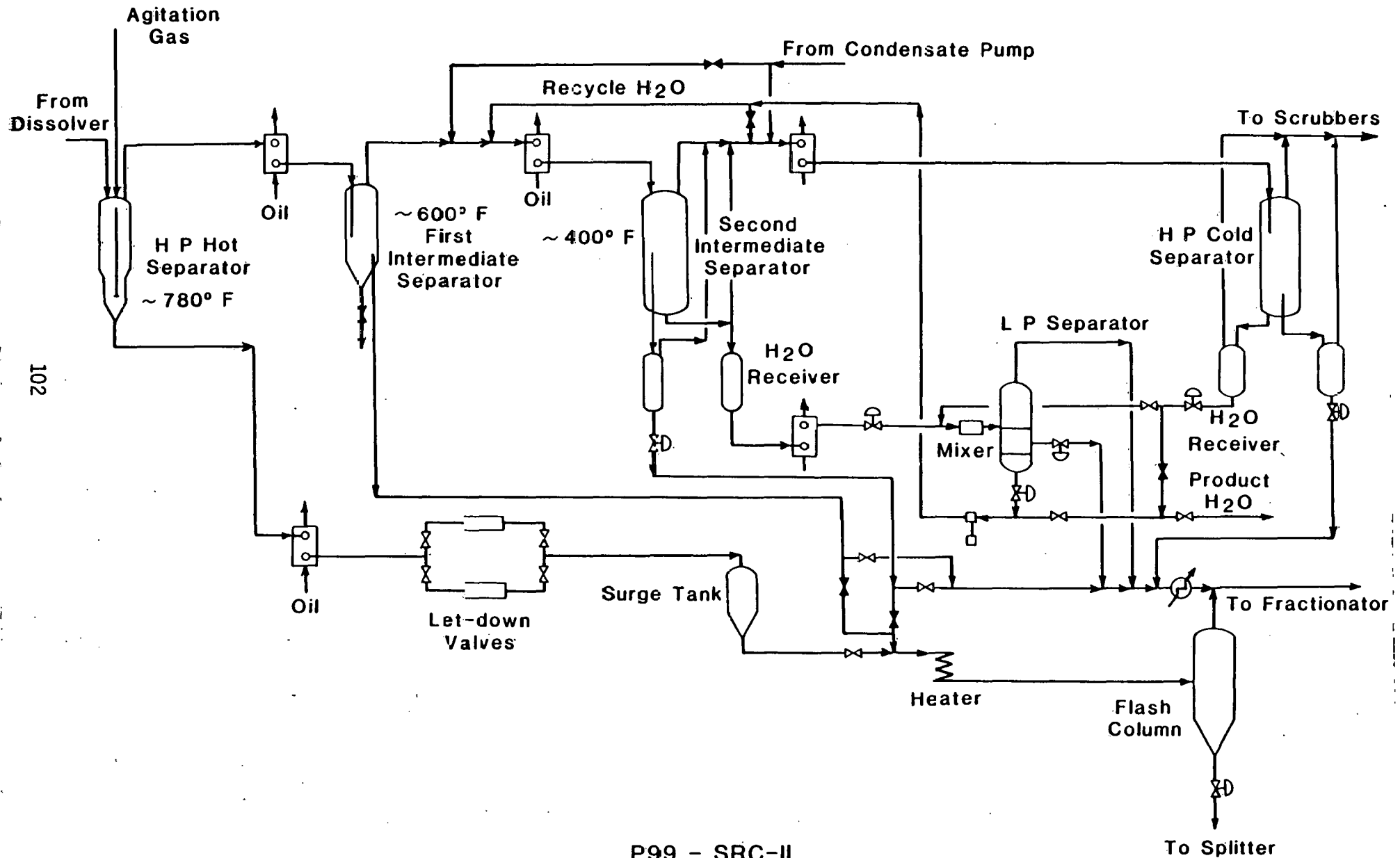
TABLES AND FIGURES

TABLE B-1

SRC-II YIELDS
FOR RUN P99-49

<u>Run No. P99 -</u>	<u>49</u>
Hydrogen	-4.2
Methane	5.8
Ethane	4.4
Propane	4.3
Isobutane	0.2
N-Butane	<u>2.0</u>
Total C ₁ -C ₄ Gases	16.7
Ammonia	0.4
Hydrogen Sulfide	2.2
Carbon Dioxide	1.0
Carbon Monoxide	0.1
Hydrogen Chloride	<u>0.1</u>
Total Other Gases	3.8
Water	7.8
C ₅ - 380°F	7.9
380 - 550°F	14.4
550 - 900°F	<u>8.7</u>
Total Distillate	31.0
900°F + Pyridine Solubles	26.2
Insoluble Organic Matter	6.4
Ash	<u>12.3</u>
Total 900°F + Product	44.9

FIGURE B-1



P99 - SRC-II

HIGH PRESSURE SEPARATOR SYSTEM

A P P E N D I X C

MERRIAM LABORATORY TABLES AND FIGURES

TABLE C-1

SUMMARY OF MERRIAM PROCESS CONDITIONS,
YIELDS AND PRODUCT ANALYSES

Conditions	DOE 287	DOE 288	DOE 289	DOE 290
Solvent	Cresap ^a	← Recycled →		
Coal	Ky 9/14	← Pittsburgh Seam ^b →		
Reactor	← 415 cc Tubular ^c →			
Nominal Slurry Residence Time, min.	22.6	26.6	26.1	26.0
Coal Feed Rate, lb/hr/ft ³	73.6	62.8	63.9	64.3
Average Dissolver Temperature, °C	447	448	448	448
°F	837	838	838	838
Dissolver Pressure, psig	1410	1900	1900	1900
H ₂ Feed				
Wt % based on slurry	1.96	3.56	3.50	3.49
MSCF/ton of coal	19.1	34.6	34.0	33.8
Additive	--	Pyrite	--	Magnetite
Additive level (% based on MF coal)	--	7.5	--	4.3
Slurry Formulation				
% Coal	39.0	39.0	39.0	39.0
% Recycle Solvent	61.0	58.1	61.0	59.3
% Additive	--	2.9	--	1.7
Yields				
H ₂ O	4.8	3.0	2.7	3.5
CO	0.3	0.3	0.2	0.2
CO ₂	0.9	0.6	0.7	0.7
H ₂ S	1.3	2.7	1.4	--
NH ₃	--	0.2	0.1	0.1
C ₁	2.4	2.4	2.7	2.3
C ₂	1.1	1.3	1.5	1.3
C ₃	0.7	1.0	0.9	0.9
C ₄	0.4	0.6	0.5	0.4
Total C ₁ -C ₄	4.6	5.3	5.6	4.9
Naphtha, C ₅ -193°C	4.7	6.1	5.9	5.9
Wash Solvent, 193-249°C, 380-480°F	4.3	8.3	9.6	7.5
Recycle Solvent, >249°C, >480°F	14.9	8.7	0.7	4.0
Total Oil	23.9	23.1	16.2	17.4
SRC	49.8	57.6	62.1	62.0
Insoluble Organic Matter	6.0	6.1	6.6	6.6
Ash	9.9	6.1	6.2	6.3
Total	101.5	105.0	101.8	101.7
H ₂ Reacted, gas balance	1.5	2.3	1.8	1.8
Additive Conversion byproducts	--	2.7	--	(0.1)
Product Analyses				
Recycle Solvent Analyses				
% C	90.03	88.06	88.67	88.39
% H	8.54	7.03	7.56	7.56
% S	0.31	0.40	0.39	0.35
% N	0.23	0.93	0.83	1.03
% O (by difference)	0.89	2.78	2.55	2.67
Specific Gravity	0.9931	1.0463	1.0539	1.0662
SRC Analyses				
% C	74.05 ^d	87.79	87.94	88.47
% H	4.60	5.80	5.51	5.77
% S	2.10	0.76	0.74	0.62
% N	1.72	1.97	1.96	1.98
% Ash	14.66	0.10	0.11	0.11
% O (by difference)	--	3.58	3.74	3.05

a) Hydrogenated solvent supplied by the Liquefied Coal Development Corp.

b) From Blacksville Mine No. 1, washed.

c) Tubular dissolver 11/16 inch I.D. by 7 feet 6 inches long, 415 cc volume.

d) Sample from run DOE 287 unfiltered.

TABLE C-1 (Continued)

Conditions	DOE 291	DOE 292	DOE 293	DOE 294	DOE 295
Coal	Pittsburgh Seam ^a				
Reactor	415 cc Tubular ^b				
Nominal Slurry Residence Time, min.	26.3	26.1	26.2	26.1	25.9
Coal Feed Rate, lb/hr/ft ³	63.6	64.0	63.9	64.0	64.5
Average Dissolver Temperature, °C	447	447	448	448	448
OF	837	837	838	838	838
Dissolver Pressure, psig	1900	1900	1900	1900	1900
H ₂ Feed					
Wt % based on slurry	3.53	3.50	3.51	3.50	3.47
MSCF/ton of coal	34.2	33.9	34.0	33.9	33.7
Additives	Magnetite	--	Pyrite	FeS	Pyrite + Mol. Sieve
Additive level (% based on MF coal)	4.3	--	7.5	4.9	5.1 + 2.0
Slurry Formulation					
% Coal	39.0	39.0	39.0	39.0	39.0
% Recycle Solvent	59.3	61.0	58.1	59.1	58.0
% Additive	1.7	--	2.9	1.9	3.0
Yields					
H ₂ O	4.5	3.2	3.9	3.6	4.2
CO	0.3	0.3	0.3	0.2	0.3
CO ₂	1.1	1.0	0.9	0.9	0.8
H ₂ S	--	1.3	2.5	1.2	2.3
NH ₃	0.1	0.1	0.2	0.1	0.1
C ₁	2.2	2.4	2.3	2.2	2.2
C ₂	1.3	1.4	1.4	1.4	1.3
C ₃	0.9	0.9	0.9	0.9	0.9
C ₄	0.4	0.4	0.5	0.4	0.4
Total C ₁ -C ₄	4.8	5.1	5.1	4.9	4.8
Naphtha, C ₅ -193°C	5.3	5.9	6.5	6.3	6.2
Wash Solvent, 193-249°C, 380-480°F	6.4	7.6	8.3	6.0	6.0
Recycle Solvent, >249°C, >480°F	5.4	2.1	9.8	5.9	7.5
Total Oil	17.1	15.6	24.6	18.2	19.7
SRC	59.8	61.2	53.7	58.3	57.4
Insoluble Organic Matter	6.7	6.5	6.5	7.7	7.1
Ash	7.6	7.5	7.2	7.4	7.4
Total	102.0	101.8	104.9	102.5	104.1
H ₂ Reacted, gas balance	2.1	1.8	2.2	1.8	2.4
Additive Conversion byproducts	(0.1)	--	2.7	0.7	1.7
Product Analyses					
Recycle Solvent Analyses					
% C	88.11	88.24	88.32	88.18	88.45
% H	7.47	7.43	7.61	7.40	7.58
% S	0.37	0.40	0.47	0.44	0.40
% N	1.00	0.92	0.95	0.98	0.96
% O (by difference)	3.00	3.01	2.65	2.90	2.61
Specific Gravity	1.0646	1.0566	1.0597	1.0661	1.0582
SRC Analyses					
% C	87.46	87.61	87.55	87.47	87.33
% H	5.69	5.77	5.64	5.63	5.90
% S	0.72	0.78	0.80	0.77	0.75
% N	1.90	1.90	1.88	1.87	1.87
% Ash	0.08	0.14	0.08	0.15	0.28
% O (by difference)	4.15	3.80	4.05	4.11	3.87

a) Loveridge Mine

b) Tubular dissolver, 11/16 inch I.D. by 7 feet 6 inches long, 415 cc volume.

TABLE C-1 (Continued)

Conditions	DOE 296R	DOE 297R-1	DOE 298R
Coal	Pittsburgh Seam ^a		
Reactor	DOE 1		
Nominal Slurry Residence Time, hr	1.01	1.05	1.05
Coal Feed Rate, lb/hr/ft ³	21.2	20.5	20.5
Average Dissolver Temperature, °C	455	455	455
°F	851	851	851
Dissolver Pressure, psig	1800	1935	2065
Feed Gas Composition			
Mole % H ₂	100.0	94.9	90.0
Mole % CO	0.0	5.1	10.0
H ₂ Feed			
Wt % based on slurry	3.96	3.94	4.09
MSCF/ton of coal	49.8	49.5	51.4
Slurry Formulation, wt %			
Coal	30.0	30.0	30.0
Recycle Coal Solution	61.5	61.5	61.5
Recycle Solvent	8.5	8.5	8.5
Slurry Blend Composition, wt %			
Coal	30.0	30.0	30.0
Middle Distillate (193-288°C, 380-550°F)	4.4	4.0	4.4
Heavy Distillate (>288°C, >550°F)	30.2	30.5	28.8
SRC	21.6	21.9	22.5
Ash (from recycle coal solution)	9.6	9.4	9.8
Insoluble Organic Matter (from recycle coal solution)	4.2	4.2	4.5
Total Solids	43.8	43.6	44.3
Yields			
H ₂ O	7.0	6.9	4.3
CO	0.3	--	--
CO ₂	0.7	2.7	5.1
H ₂ S	2.2	1.8	2.2
NH ₃	0.4	0.5	0.8
C ₁	5.7	5.6	5.7
C ₂	3.6	3.7	3.8
C ₃	2.9	3.0	3.2
C ₄	1.3	1.4	1.7
Total C ₁ -C ₄	13.5	13.7	14.4
Naphtha, C ₅ -193°C	10.0	9.7	9.2
Middle Distillate, 193-288°C	20.2	17.1	21.5
Heavy Distillate, >288°C	9.6	11.1	9.9
Total Oil	39.8	37.9	40.6
SRC	25.0	26.3	25.6
Insoluble Organic Matter	5.0	5.1	5.1
Ash	11.4	11.3	11.1
Total	105.3	106.2	109.2
H ₂ Reacted, gas balance	5.3	4.8	4.9
CO Reacted, gas balance	--	1.4	4.3
Lineout Index	1.00	1.07	1.06
Product Analyses			
Heavy Distillate Analyses			
% C	89.19	89.20	89.18
% H	7.36	7.25	7.19
% S	0.49	0.53	0.52
% N	1.25	1.21	1.24
% O (by difference)	1.71	1.82	1.81
Specific Gravity	1.0870	1.0860	1.0859
Vacuum Bottoms Analyses			
% C	64.68	64.20	63.39
% H	3.72	3.78	3.68
% S	2.91	3.01	2.96
% N	1.29	1.29	1.31
% Ash	27.23	27.55	27.78

a) Powhatan Mine No. 5

TABLE C-2

Analyses of Cresap Process Solvent*

API ⁰ @ 60 ⁰ F				12 ⁰					
D-1160 Distillation									
Vol %	IBP	5%	10%	30%	50%	70%	90%	95%	EP
T, ⁰ F	446	470	481	499	525	610	778	830	871
T, ⁰ C	230	243	249	259	274	321	414	443	466
Carbon				Hydrogen			Sulfur		
90.8 wt %				9.1 wt %			0.1 wt %		
Pour Point							< -25 ⁰ F		
THF Insolubles							0.06 wt %		
+880 ⁰ F							1.27 wt %		

*Analyses and solvent provided by Liquefied Coal Development Corporation, Moundsville, West Virginia.

Distillation Curve^a of Cresap Solvent

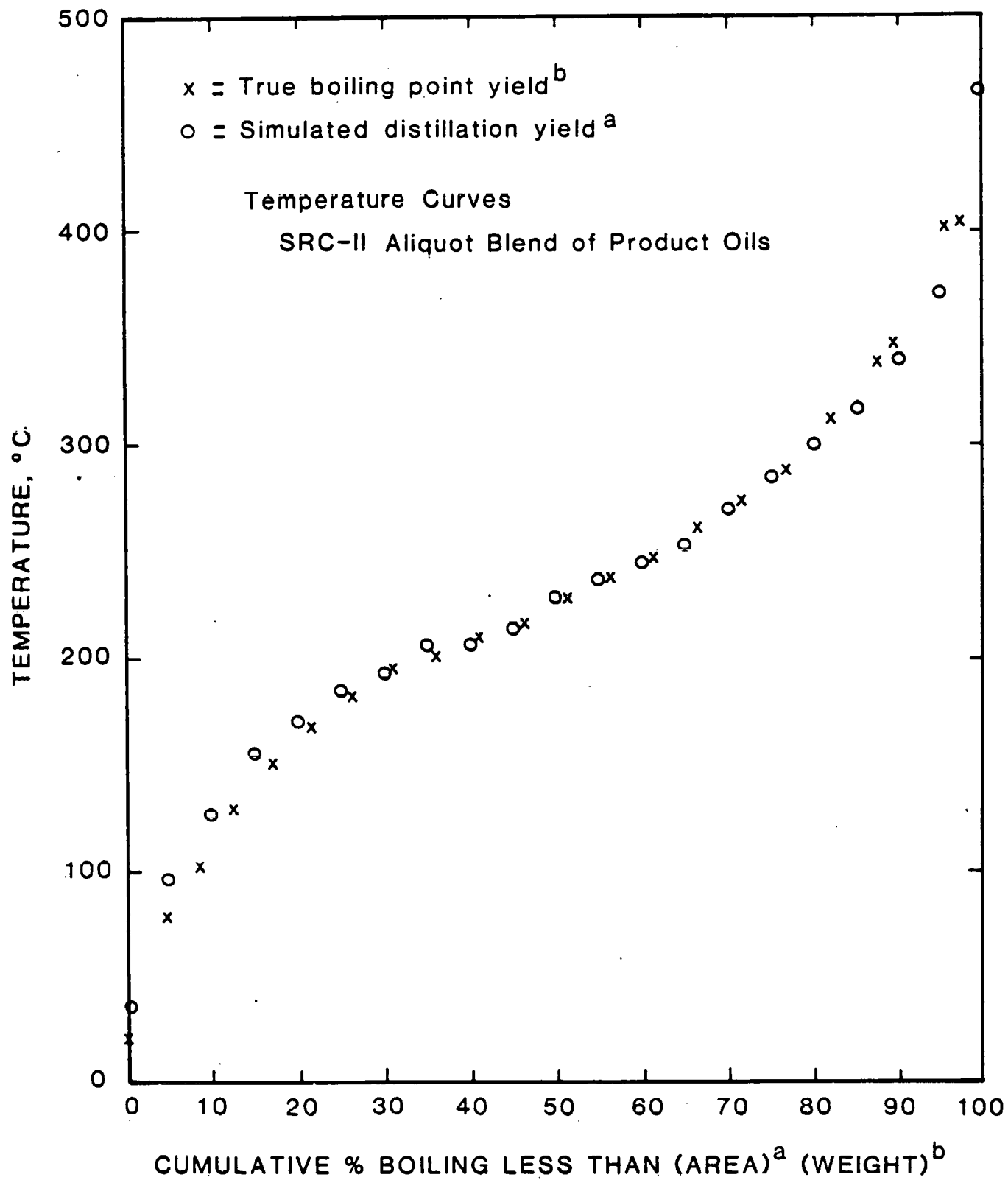
Cut No.	Pressure mm Hg		Temper- ature, ^o C	Corrected Temperature, ^b ^o C	Wt %	Σ Wt %
1	732	(ibp=175 ^o C)	220		0.93	0.93
2			235		8.94	9.87
3			240		6.35	16.22
4			245		8.12	24.34
5			250		8.70	33.04
6			255		6.09	39.13
7			260		5.89	45.02
8			270		8.03	53.05
9			288		9.75	62.80
10	2.0		135	304	3.77	66.57
11			150	321	4.64	71.21
12			175	352	7.14	78.35
13			200	383	9.58	87.93
14			225	423	7.60	95.53
15			243	438	2.94	98.47
Residue					1.40	99.87

a) Vigreux Column (40 cm), 10:1 Reflux ratio.

b) K = 10 correction.

FIGURE C-1

COMPARISON OF SIMULATED DISTILLATION AND TRUE BOILING POINT CURVES



**FACTORS INFLUENCING NO_x PRODUCTION
DURING THE COMBUSTION OF
SRC-II FUEL OIL**

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FOR

**WESTERN STATES SECTION/THE COMBUSTION INSTITUTE
1979 FALL MEETING**

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ABSTRACT

Combustion tests of SRC-II fuel oil produced by the Pittsburgh & Midway Coal Mining Company at the Fort Lewis SRC Pilot Plant funded by the United States Department of Energy (DOE) were performed in a 10×10^6 Btu/hr subscale combustor to identify parameters that influence NOx production during the burning of the fuel. The tests were conducted burning SRC-II fuel oil in its pure form as well as blended with #6 or diesel oils. Parameters identified to most significantly influence NOx production included fuel atomization, excess O₂ and fuel nitrogen content and vaporization characteristics. The tests were performed using a developmental low NOx burner proprietary to Southern California Edison Company, and the lowest NOx emission level achieved when burning pure SRC-II fuel oil was 269 ppm (corrected to 3% O₂). The test results suggested that consideration should be given to the optimization of the burner hardware especially for SRC-II usage in order to achieve further reduction of NOx emissions.

1.0 INTRODUCTION

Nitrogen oxides (NO_x) are formed in combustion processes by two basic mechanisms; the thermal fixation of atmospheric nitrogen at elevated temperatures within the flame zone, and the conversion of organically bound nitrogen to NO_x during the combustion process. NO_x formation by thermal fixation, normally referred to as thermal NO_x, is essentially dependent upon the flame temperature and the concentration of atmospheric oxygen and nitrogen within the flame zone. The conversion of organically bound nitrogen to NO_x, however, is a complex function of the fuel properties and the burner design and operating variables.

Research on the reduction of thermal NO_x formation has been in progress for many years. More recently, attention has been given to the formation of NO_x from nitrogen compounds contained within the fuel. This source of NO_x gains importance as high nitrogen content fuels derived from coal and shale grow in use.

Among the coal-derived synthetic fuels that may become commercially available in the near future is Solvent Refined Coal (SRC-II) fuel oil produced by the Pittsburgh & Midway Coal Mining Company. This new fuel, being derived from the plentiful sources of domestically available coal, offers an attractive alternative to our nation towards becoming energy self-sufficient. The SRC-II fuel oil has a definite advantage over direct coal combustion since it is compatible with existing hardware for oil-fired industrial and utility installations and is significantly easier to handle and transport.

However, the relatively high nitrogen content of SRC-II fuel oil (nominally 1.0%) may pose a potential NO_x emission problem with its use. The incremental cost of nitrogen removal coupled with the resultant change in other characteristics from denitrofication processing may render the product suitable for higher-value fuel applications and thus reduce its potential for consideration as an alternate boiler fuel. It is, therefore, been considered important to identify methods of minimizing NO_x production during the combustion of SRC-II fuel oil.

This paper describes a test program conducted by the Research and Development Department of Southern California Edison Company under contract to the Gulf Mineral Resources Company to identify parameters that may influence NO_x production during the combustion of SRC-II fuel oil. The objectives of the test program included:

- Determine the effect of fuel atomization, type of atomizing media, and combustion stoichiometry on NO_x formation when burning pure SRC-II fuel oil, and blends of SRC-II fuel oil made with No. 6 or diesel fuel oils.
- Identify parameters influencing the conversion of the SRC-II fuel-bound nitrogen to NO_x during the combustion process.

- Evaluate the smoking tendencies of SRC-II fuel oil using commercially available smoke suppressants and examine the effect of reducing the operating level of excess O_2 on NO_x production.

The test program was performed in the TRW subscale (10×10^6 Btu/hr) combustion test facility located in Redondo Beach, California. A developmental low NO_x oil burner proprietary to Southern California Edison Company was used in performing the tests.

2.0 DESCRIPTION OF HARDWARE

2.1 THE COMBUSTION FACILITY

The TRW subscale combustion facility provides the capabilities for testing at a heat rating of up to 10×10^6 Btu/Hour. A schematic layout of the facility is provided in Figure 1. The facility's main components are comprised of a windbox, an air register assembly, a primary combustion chamber, an uncooled combustor extension, and an exhaust stack.

Combustion air is supplied to the combustor through a refractory lined windbox of a relatively large volume to dissipate swirl within the combustion air flow field. The air is then ducted to the primary combustion chamber through a special air register assembly designed to further minimize the swirl, maintain uniform combustion air velocity profile across the burner throat and provide an axisymmetric combustion air envelope to surround the flame. The register design produces a parallel combustion air flow field to reduce the NO_x emission level achieved by the developmental burner.

The primary combustion chamber consists of a water jacketed cylindrical assembly that is three feet in internal diameter and fifteen feet long. Since the combustor wall is water cooled, the heat dissipation to the wall is higher than generally experienced in actual furnace installations. This high dissipation of heat probably reduces the average burner flame temperature such that a reduction in thermal NO_x formation results. Heat dissipation to the walls is reduced by installing a segmented flame side inner liner fabricated from stainless steel plates with an air gap between the flame side and the water cooled furnace wall.

Combustion products exit the water cooled primary combustion chamber into an uncooled combustor extension. This extension is also three feet in diameter and fifteen feet long. The extension is installed to provide longer residence time for the reacting gases at elevated temperature and hence, it minimizes the tendency for smoke emission. Combustion air injection ports are provided at the entrance of the uncooled combustor extension to introduce secondary combustion air into the reacting gases in the event the burner is operated in the primary zone under fuel-rich stoichiometry. The capability to introduce the secondary air at the exit of the

extension section is also provided by the ducting arrangement in case longer residence time is desired under the fuel-rich conditions.

Combustion air at ambient temperature is supplied to the combustor by a forced draft fan rated at 4000 SCFM and 1 psig. The combustion air fan ducting is arranged such that flue gas recirculation from the combustor discharge, of up to 15% of the total combustion air volume, may be mixed with the combustion air. Variation in the combustion air flow rate is achieved by two dampers installed in series at the inlet ducting to the combustor. An indication of combustion air volume flow rate is attained by a manometer reading of static pressure drop across the register assembly and by the output of a pitot tube installed in the center of air ducting to the windbox.

Fuel atomization may be accomplished in the facility by utilizing steam, air, natural gas, or flue gases as the atomizing medium. Steam is supplied by a gas-fired, 50 horsepower, York Shippley packaged boiler. Pressure during the use of air or flue gas as the atomizing medium is delivered by a 100 SCFM Root compressor. In the case of handling flue gas, the gas is cooled to less than 200°F in a special water cooled heat exchanger before it is introduced to the compressor. The natural gas atomization is supplied from a utility natural gas supply-line.

A schematic layout of the fuel supply system is also presented in Figure 1. The system consists of three inter-meshed circuits for the handling of diesel, #6 and SRC-II fuel oils. Each of the three fuel circuits utilizes a 0-2 gpm Tuthill centrifugal pump rated at 400 psig maximum pressure output. The desired fuel flow rate and blend ratio are achieved by regulating fuel flow in the various circuits using the valving arrangement shown in Figure 1. The fuel flow rates are monitored by three Foxboro turbine-meters that are installed in the various fuel circuits.

2.2 LOW NO_x BURNER

The burner used in the test program is a developmental low NO_x burner proprietary to Southern California Edison Company. The burner, as shown schematically in Figure 2, is a steam atomized oil gun that consists of two concentric pipes for the delivery of fuel and atomization steam to the burner tip. Fuel is injected through a ring of circumferentially located orifices that are directed radially. Atomizing steam is injected through continuous steam slots positioned on both sides of the oil orifices. As the oil departs from the orifices, it is sheared and accelerated by the higher steam velocity forming an oil mist whose dispersion characteristics into the combustion air flow stream can be modulated and controlled. The dynamic interaction between the radially accelerated fuel mist and axially flowing combustion air establishes a thin umbrella-shaped burner flame.

Adjustment of the Low NO_x burner hardware provides the ability to control mixing and local fuel stoichiometry within the flame by

changing fuel injection velocity (oil orifice number and diameter), atomization steam gap proximity to the oil manifold, atomization steam mass flow and momentum, and combustion air velocity profile within the burner throat. The interaction of fuel and injected steam with combustion air in the low NO_x burner establishes two extensive furnace gas recirculation fields which adds to the burner NO_x control capability and augments the flame stabilization process. These recirculation fields are established, as shown in Figure 2, one external to the burner flame at the discharge of combustion air into the furnace, and the other within the core of the umbrella-shaped flame. The strength of the recirculation field within the core of the flame is enhanced by discharging atomization steam through continuous circumferential atomization steam gaps. The injection of steam in a continuous curtain also provides dynamic stabilization of the burner flame through its interaction with the combustion air flow.

3.0 DATA ACQUISITION AND REDUCTION

3.1 TEST PROCEDURES

Testing was performed to evaluate, as indicated earlier, the effect of burner operating variables on NO_x emission from pure SRC-II fuel oil and blends of SRC-II fuel oil mixed with diesel or #6 fuel oils. Operating variables investigated during the study included fuel blend ratio, degree of fuel atomization, and level of excess O₂. The firing of the facility was limited to 6×10^6 Btu/Hour to minimize SRC-II fuel oil consumption.

Special test procedures were adopted to expedite the data acquisition while maintaining the accuracy of the results. The testing consisted of evaluating NO_x levels for different SRC-II fuel oil blends under a variety of burner operating conditions. Burner variables tested included four different atomization pressures of 1.0, 1.5, 2.0, and 2.5 psig.; and four excess O₂ levels of 6.5, 7.1, 7.8, and 8.5 percent. For each of the atomization pressures, the minimum excess O₂ that can be maintained in the combustor without resulting in smoke emission was determined and termed the "Smoke Limit".

To avoid visible smoke emission, the facility was started on diesel fuel while maintaining sufficiently high excess O₂ within the combustor. After a reasonable warm-up period had elapsed, fuel switch over was performed, and the SRC-II fuel oil blend ratio for the specific testing condition was then established. A cavitating venturi was placed in the fuel supply line to the combustor to ensure that fuel flow was set at a constant value, controlled only by the feed system supply pressure. The cavitating venturi was installed downstream from the location where fuels are blended in the piping in order to provide accurate regulation of the total fuel flow.

Upon establishing the desired fuel blend ratio and flow rate to the combustor, combustion air flow was then reduced slowly using a

screw driven damper assembly installed in the ducting at the inlet to the windbox. The screw mechanism allowed the adjustment of the damper setting in very fine increments and permitted resetting of the damper to repeat the test conditions if needed. The combustion air was initially reduced until light visible smoke appeared and then air was slightly increased to eliminate the smoke. Excess O_2 as established under these conditions was then considered the "smoke limit" and the NO_x emission level was determined. To establish the smoke limit at a different level of atomization, excess O_2 was first increased within the combustor before changing burner atomization pressure to avoid combustion upset. After the new atomization pressure was adjusted on the burner, combustion air flow was again reduced in the manner described earlier to establish the new smoke limit. The smoke limit was determined for the various fuel blend ratios at each of the four selected atomization pressures.

In order to identify the effect of excess O_2 on the emission characteristics of the various fuel blends, excess O_2 was increased within the combustor to four different preselected excess O_2 levels. To expedite the data acquisition, it was elected to scan the entire range of atomization pressure while maintaining the excess O_2 and fuel blend ratio constant. This was preferred since it was significantly easier to reproduce the atomization pressure on the burner gun than attempting to reset excess O_2 level for a specific fuel blend ratio. Establishing the desired operating level of excess O_2 was achieved by repeatedly processing the combustor flue gas through a gas chromatograph to identify actual excess O_2 level within the furnace. If O_2 was not at the desired level, readjustment of the damper assembly was made to obtain the required operating O_2 level.

3.2 GAS SAMPLING TRAIN

Samples of the gaseous effluent were collected through five stainless steel, water cooled probes located near the exit of the uncooled combustor extension. The probes were installed in a plane perpendicular to the products of combustion flow with one probe installed in the center of the combustor and the remaining four spaced 90° apart in a circumferential arrangement. The five sampling lines were manifolded together outside the combustor to a single stainless steel line which was used to deliver the sample for conditioning and analysis. Shut-off valves were provided on each of the five sampling probes to allow sampling from all five probes simultaneously or from any combination of probes. The gas samples were conditioned before analysis in a moisture trap and a filter to dry the sample and remove particulate matter. The gaseous constituents determined during the test program included O_2 , CO , CO_2 , NO_x and SO_2 . A Carle Instruments Gas Chromatograph was used to analyze for O_2 and CO_2 and a Thermal Electron Chemiluminescence was used for the NO_x measurements.

3.3 DATA REDUCTION

3.3.1 Combustion Calculations

Combustion calculations were performed to provide a theoretical determination of the combustor air volume and air/fuel ratio at the burner, and to quantify the velocity of combustion air within the burner throat. The calculations were based on the ultimate analysis of the fuel burned in the combustor during a specific test. The chemical composition of the fuel blends burned during the various test runs was proportionately calculated using the fuel blend ratios and the ultimate analysis of the individual fuels used. The composition of all three fuels tested was determined by an independent laboratory and the results are included in Table 1. A detailed summary of the performed data reduction is included in SCE final report #79-RD-7.

3.3.2 Fuel Nitrogen Conversion Efficiency to NOx

The effect of fuel bound nitrogen on NOx emission was quantified by calculating the conversion "efficiency" of the fuel nitrogen to NOx under the various test conditions. This conversion efficiency was determined by calculating the incremental increase in NOx as a result of increasing the nitrogen weight fraction within the fuel blend. The variation in the nitrogen content of the fuel was obtained by blending the SRC-II fuel oil with low nitrogen containing fuels such as diesel or #6 fuel oils.

In order to determine the incremental increase in NOx emission due to the fuel nitrogen, it was required to obtain a base NOx emission level for each of the fuel blends without the presence of the organic nitrogen in the blend. This was achieved by substituting diesel for SRC-II fuel oil assuming that the two fuels have similar physical properties with the exception of their nitrogen content. The difference between NOx levels obtained by the combustion of the SRC-II and diesel blends was attributed to fuel produced NOx. Combustion calculations were then performed to determine NOx concentration in the flue gas (ppm, corrected to 3% O₂) that may result from the complete conversion of 1% by weight of fuel nitrogen to NOx. Since the ultimate analysis of the fuel blends changed with the variation in SRC-II fuel oil blend ratio, NOx concentrations at 1% fuel nitrogen conversion were calculated for each of the tested fuel blends. The conversion efficiency was then calculated according to the formula:

$$\text{Conversion Efficiency (percent)} = \frac{\Delta(\text{NOx})}{\Delta N (\text{NOx @ 1\%})} \times 100$$

Where:

$\Delta(\text{NOx})$ Is the increase in NOx emission attributed to fuel nitrogen (ppm corrected to 3% O₂)

ΔN Is the incremental increase in fuel nitrogen content due to SRC-II blending (percent by wt.).

(NOx @ 1%) Is the NOx concentration in the flue gases (ppm corrected to 3% O₂) when 1.0% of fuel nitrogen is completely converted to NOx.

4.0 RESULTS AND DISCUSSION

Several parameters were identified during the test program to influence NOx emission from the combustion of pure and various blends of SRC-II fuel oil. The lowest NOx level achieved by the developmental burner when burning pure SRC-II fuel oil was 269 ppm at 6.5 percent excess O₂, (the O₂ levels achieved in this subscale combustor facility were generally higher than what is obtained in a full scale utility application due to the cold wall effect and shorter residence time available in the combustor). The most important of the parameters identified to influence the emission levels were fuel atomization, excess O₂, and the properties of the fuel blend. The effect of each of these variables on NOx emissions as identified during the study is discussed below. Due to the large magnitude of data collected, only selected tests were plotted in this paper to show the trends illustrating the effect of the various variables.

4.1 EFFECT OF FUEL ATOMIZATION

The effect of fuel atomization on NOx emission was investigated for SRC-II fuel oil/#6 fuel blends of various proportions. Burner atomization pressures between 1.0 and 2.5 psig were tested, and NOx emission was determined at the smoke limit and four other discrete excess O₂ levels (6.5, 7.2, 7.8, 8.5 percent).

NOx emission was found, as shown in Figure 3, to increase with improved atomization. The NOx data presented in Figure 3 were obtained at the smoke limit but similar trends were also experienced at the higher levels of excess O₂. As the proportion of the high nitrogen-containing SRC-II fuel oil in the fuel blend was increased, a vertical shift in NOx emission curves was progressively noted. The magnitude of this shift was more significant at SRC-II blend ratios of 20 and 100 percent. At 20 percent SRC-II fuel oil blend ratio, for example, the increase in NOx emission over levels obtained with pure #6 oil was in excess of 50% when using 2.5 psig atomization pressure. At 100% SRC-II fuel oil a similar percent increase in emission levels was obtained over the levels measured at 60% blend ratio using the same atomization pressure. More modest shifts in the NOx curves were obtained for SRC-II fuel oil blend ratio between 40 and 60 percent.

The sensitivity of NOx emission level to atomization varied substantially as a function of SRC-II proportion within the fuel blends. The increase in emission with atomization became more pronounced as higher SRC-II fuel oil blend ratios was used. The

difference between the NO_x curves was, as a result, maximum at the highest atomization pressure and it progressively increased with higher SRC-II fuel oil blend proportions. With pure SRC-II fuel oil the increase in emission with atomization was more dramatic and appeared to be of an exponential nature.

While improved fuel atomization has achieved a progressive reduction in the operating level of excess O₂, lower O₂ concentration within the furnace did not provide a reduction in NO_x emission. As shown in Figure 4 the reduction in excess O₂ with improved atomization has produced a modest increase in NO_x emission with the low nitrogen containing fuel (#6 fuel oil) and a dramatic increase with the high nitrogen fuel (SRC-II) fuel oil. In spite of the fact that SRC-II fuel oil is high in aromatic content, a very low smoke limit was demonstrated by the fuel at a sufficiently high atomization pressure. It appeared that the light consistency of SRC-II fuel oil enhanced fuel atomization and vaporization resulting in the demonstrated low excess O₂ levels.

The increase in NO_x emission with improved fuel atomization is mainly attributed to the enhancement of mixing between the fuel and combustion air within the burner flame. Breen⁽¹⁾ explained that initial mixing between combustion air and fuel produces pockets of combustible air/fuel mixtures that ignite and burn at a very rapid rate. The formation of these pockets establishes an adiabatic flame front zone within which thermal NO_x generation is significant. As a larger portion of the fuel, due to improved atomization, evaporates and mixes with the air in this primary combustion zone, higher levels of thermal NO_x are produced.

Fuel nitrogen conversion to NO_x is also affected by mixing between the fuel and combustion air. Heap et al.⁽²⁾ investigated NO_x produced from fuel nitrogen in an atmosphere that contained no molecular nitrogen to isolate the effect of thermal NO_x formation. Oxidizer used in the combustion process consisted of a mixture of carbon dioxide, argon, and oxygen. The study conclusively showed that different atomizer designs influenced NO_x production from the organically bound nitrogen over a wide range. Sarofim et al.⁽³⁾ postulated that as fuel atomization and mixing efficiency is enhanced, an increasing fraction of the fuel will be burned under locally lean fuel conditions resulting in an increase in the conversion efficiency of the fuel nitrogen to NO_x. This postulate was supported by the results of Appleton and Heywood⁽⁴⁾ who found an increase in the conversion of the nitrogen content of a doped-kerosene with increasing pressure of atomization and by Haebig et al.⁽⁵⁾ who determined that the conversion of fuel nitrogen to NO_x increased with decreasing sooting tendency of a number of burners.

The significant vertical shift in NO_x emission curves, shown in Figure 3, at 20 and 100 percent SRC-II fuel oil blend ratios, is again associated with fuel nitrogen conversion. The data indicated that the impact of fuel nitrogen on the emission level is not linear but a rather complex function of fuel nitrogen content and the atomization and evaporation qualities of the fuel. It appeared

evident that the incremental increase in NOx emission is not directly proportional to SRC-II fuel oil blend ratio and therefore, for a specific NOx regulatory limit, a certain optimum blend ratio between SRC-II fuel oil and #6 fuel is expected to maximize SRC-II fuel oil usage. It is noteworthy that while poor fuel atomization (low atomization pressure) has produced low NOx values, operation in this mode, in this subscale combustor, would require a sacrifice in fuel economy and efficiency due to the need to operate at a relatively high level of excess O₂ to avoid smoke emission.

The effect of the type of atomizing gas used, on the combustion qualities of pure SRC-II fuel oil was also investigated during the program. The gases tested consisted of air, steam, flue gas, and natural gas. The change in smoke limit and NOx emission as a function of atomization pressure was evaluated with each of the gases and the results are presented in Figures 5 and 6, respectively.

Data presented in Figure 5 show that for the most part, a reduction in smoke limit was generally achieved using higher burner atomization pressures. The smoke limit reduction was significant in the case of steam and natural gas and was somewhat minimal for air and flue gas. The use of natural gas provided a significant reduction in smoke limit throughout the tested range of atomization.

A possible explanation for this reduction is that natural gas established gaseous flames which enhanced the prevaporization of the injected fuel and hence, improved its mixing and combustion efficiency. It was interesting to note that air has a minimal impact on smoke limit and that flue gas achieved lower smoke limits than air. While the smoke limit varied substantially as a function of atomization pressure and the type of gaseous medium, the lowest NOx levels were consistently obtained with poor fuel atomization. The minimum NOx levels were achieved, as shown in Figure 6, with steam as the atomizing gas and, in general, NOx levels progressively increased with higher atomization pressure except for air, where the emission decreased with improved atomization.

4.2 EFFECT OF EXCESS O₂

The effect of excess O₂ on NOx emission was studied for several SRC-II fuel oil/#6 fuel blends. The variation in excess O₂ during the tests ranged between the minimum at the smoke limit for the various blends to a maximum of 8.5 percent. NOx variation with O₂ for different burner atomization pressures was evaluated at the same increments of O₂ levels to provide a common basis for the data.

NOx emission was found to increase linearly with the increase in excess O₂. Data presented in Figure 7, obtained using an atomization pressure of 1.0 psig, clearly displayed this linear dependence. Similar trends of NOx variation with excess O₂ were also obtained using higher burner atomization pressures. For

the most part, the fitting of the NO_x-O₂ results to a straight line relationship was, in general, acceptable and showed an average correlation coefficient in excess of 0.9. The linear dependence of NO_x on excess O₂ agreed with what is typically experienced in full-scale utility boiler application. Similar to the data presented in Figure 3, a vertical shift in the NO_x curves was noted due to the increase of SRC-II fuel oil proportion in the fuel blends. A more substantial shift in the curves was noticeable at 20% SRC-II fuel oil blend ratio than at other blend ratios. The sensitivity of NO_x to excess O₂ also varied as a function of SRC-II fuel oil proportion in the fuel blends with the greatest rate of increase in emission occurring at high SRC-II fuel oil concentration. By fitting the NO_x excess O₂ data at different atomization pressures to the straight line relationship, it was apparent, as shown in Figure 8, that the slopes of these lines progressively decreased with improved fuel atomization. In addition, data in Figure 9 showed that the slopes of the lines for the various SRC-II fuel oil blends progressively diminished by the reduction in the nitrogen concentration within the fuel.

The increase in NO_x emission with excess O₂ is again attributed to an increase in both thermal and fuel NO_x production. The linear dependence of NO_x on excess O₂ suggested that an increase in thermal NO_x occurred due to the higher O₂ concentration within the furnace. Since the slope of the straight lines for the NO_x curves progressively decreased with improved atomization (Figure 8), it is conceivable that the postulated thermal NO_x increase is mainly associated with post burner flame combustion rather than the combustion in the primary flame zone. This is plausible since, as improved atomization was introduced, less fuel became available for the post flame combustion and therefore a reduced burner sensitivity to excess O₂ was experienced. The change in the slope of NO_x curves as a function of SRC-II fuel oil blend ratio is, on the other hand, related to fuel nitrogen conversion. The steeper slopes invariably experienced at high SRC-II fuel oil blend ratio (Figure 9) indicates that fuel nitrogen conversion to NO_x was enhanced with the high excess O₂. In addition, as in the case of thermal NO_x, the improved fuel atomization maximized the conversion of fuel nitrogen of NO_x in the primary combustion zone which also contributed to the reduced sensitivity of NO_x to excess O₂.

Increasing excess O₂ level within the furnace may have also indirectly impacted the mixing processes within the burner flame. At the highest excess O₂ level tested (8.5 percent), combustion air velocity within the burner throat was increased by as much as 30%. This higher velocity could have introduced intense turbulence within the burner flame which generally contributes to an increase in both thermal and fuel bound NO_x production.

The sensitivity of NO_x emission to excess O₂ appeared to be also influenced by the distillation characteristics of the fuel blend burned. This was clearly illustrated when NO_x emission levels obtained with a 60/40 fuel blend ratio of SRC-II fuel oil/diesel

and SRC-II fuel oil/#6 were compared over a wide range of excess O₂ in Figure 10. The data showed that while NO_x dependence on excess O₂ is fairly linear for the SRC-II fuel oil/#6 fuel blend, it is more parabolic in shape for the diesel blends. Since #6 fuel oil contained a higher nitrogen fraction than the diesel fuel, it was rather surprising to note that higher NO_x emission levels were consistently obtained with the SRC-II fuel oil/diesel blends. Considering the fact that the amount of data obtained with SRC-II fuel oil/diesel blends was rather limited, it is difficult to formulate an exact explanation of the observed trends based on the available data. The discussion of the effect of fuel properties on organic bound nitrogen conversion to NO_x contained in Section 4.3 may explain however, to some degree, this observed difference in the NO_x levels.

Since O₂ level appeared to critically impact NO_x formation, tests were performed using pure SRC-II fuel oil blended with commercially available smoke suppressants to reduce the operating level of O₂ and in turn control NO_x emission. Two additives were tested during the program, Amergy 5000 and CI-2. The Amergy 5000 is a "combustion improver" manufactured by the Drew Chemical Corporation to be used in coal and liquid fuel fired installations. The CI-2 is a fuel oil combustion catalyst manufactured by the Ethyl Corporation.

Testing was first performed using the additives in the blend proportions recommended by the manufacturers. The concentrations used were 115 ppm for the Amergy 5000 and 25 ppm for CI-2 (by volume). The results of the testing with these concentrations showed a minor decrease in excess O₂ and NO_x levels. By consulting with the additive manufacturers, it was decided to increase the additive blend concentration to 500 ppm for the Amergy 5000 and 115 ppm for CI-2.

The results of tests performed with the higher additive concentrations for excess O₂ and NO_x are presented in Figures 11 and 12, respectively. The data showed that CI-2 at 115 ppm concentration was more effective in reducing NO_x and O₂ levels than the Amergy 5000 at 500 ppm concentration. In general the reduction in NO_x emission was correlated to lower excess O₂ within the combustor. The percent reduction in excess O₂ varied randomly for the different atomization levels, and the maximum O₂ reduction did correspond to the maximum reduction in NO_x levels. The percent reduction in NO_x emission achieved by the CI-2 ranged between 6-11 percent and for Amergy 5000, between 2-6 percent. Reduction in O₂ ranged between 12-20 percent for CI-2 and between 2-4 percent for Amergy 5000.

In the absence of data that explains the mechanisms by which the additives achieved the reduction in excess O₂ and, in turn, NO_x emission, it is difficult to correlate the test results to the combustor operating variables. The data showed conclusively, however, that additives, if used in sufficiently high concentrations, provide an effective reduction in NO_x emission. The trade-

offs between the cost of the additives and the attained reduction in the emission level is felt to be the factor that will determine the feasibility of considering additives as a NO_x control technique. Another factor to be considered, in using additives in a practical combustion application, is the impact of additive use on the operation and maintenance characteristics of the combustor.

4.3 EFFECT OF FUEL PROPERTIES

The observed variation in NO_x emission with the changes in burner atomization pressure and operating level of excess O₂ (Figures 3, 4, 7, 8, and 9) suggested that the properties of the various fuel blends have influenced the NO_x emission level in a complex fashion. Since a major portion of the NO_x generated is produced from fuel nitrogen, it appeared of practical interest to identify parameters that may dictate the conversion of fuel nitrogen to NO_x.

Using the compiled data base, the conversion efficiency of fuel nitrogen to NO_x was calculated as described in Section 3.3.2 for four different nitrogen concentrations. The change in the nitrogen content of the fuel was obtained by blending SRC-II fuel oil with either diesel or #6 fuel oil. The conversion efficiencies were determined for burner atomization pressures of 1.0 and 2.5 psig and for excess O₂ levels of 6.5, 7.1 and 7.8 percent. A summary of the nitrogen conversion data is included in Tables 2 and 3.

Nitrogen conversion to NO_x was found as shown in Figure 13 to decay with the increase in the nitrogen weight fraction within the fuel. As a result, as the nitrogen fraction within the fuel was increased, a decreasing portion of the fuel nitrogen was converted to NO_x. The results agreed with the data reported by Fenimore⁽⁶⁾ during a study of nitric oxide production from fuel nitrogen in premixed ethylene flames and with the conversion curve developed by Mansour and Jones⁽⁷⁾ during the combustion tests of Paraho shale oil in a utility boiler. The data also showed that the conversion efficiency is affected by the vaporization characteristic of the fuel blend. By comparing conversion efficiencies for SRC-II fuel oil/diesel and SRC-II fuel oil/#6 fuel blends, it was evident that higher conversion efficiency was obtained with the SRC-II fuel oil/diesel blend. The difference in the conversion efficiencies for the two blends was more significant at low nitrogen concentration and poor fuel atomization.

The degree of fuel atomization appeared to also impact fuel NO_x production. As indicated in Figure 13, higher atomization pressures have consistently produced high nitrogen conversion rates. The impact of atomization on nitrogen conversion was more significant for the SRC-II fuel oil/#6 blend. The maximum difference between the conversion efficiencies for the two levels of atomization occurred at a low nitrogen weight fraction.

The decay in fuel nitrogen conversion to NOx with the increase in nitrogen concentration within the fuel supports the postulate(8) that NOx emission from the nitrogen contained in the fuel is the result of two competing mechanisms. The first is a NOx producing mechanism which increases NOx emission in proportion to the increase in the nitrogen content within the fuel. The second is a destruction mechanism by which NOx, formed early in the combustion process, is consumed by chemical species generated by the delayed evaporation and pyrolysis of residual fuel components. While the exact nature of the chemical species that affect the NOx reduction is not fully identified, it is believed that it is related to heavy hydrocarbon fragments and nitrogen containing radicals (HCN, NH, NH₂, etc.) produced from the fuel bound nitrogen.

The higher conversion efficiency displayed by SRC-II fuel oil/diesel blends supports the possibility that heavy hydrocarbon fragments may indeed be an effective mechanism for NOx reduction. The data showed that in the presence of a heavy residual oil such as #6, a net reduction in NOx emission is achieved. This explains why the 60/40 fuel blend (Figure 10) of SRC-II fuel oil/#6 showed lower NOx levels than the SRC-II fuel oil/diesel blend. This could also explain why higher atomization pressures, which generally enhance fuel vaporization, resulted in an increase in conversion efficiency especially in the case of SRC-II fuel oil/#6 blend. The results also explain NOx sensitivity to excess O₂ where the presence of excess O₂ may have inhibited the reduction effect of NOx by combustion produced species.

The use of hydrocarbon species as a NOx reducing agent was studied by several other investigators. Among those were Wendt et al.(9) who demonstrated using an experimental laboratory burner that NOx may be reduced to molecular nitrogen by the injection and burning of methane downstream of the primary combustion zone. KVB Engineering Inc.(10) experimentally also showed that NOx in a gas turbine exhaust may be destroyed in a combined cycle installation by reaction in the downstream boiler's fuel rich flames. Furthermore, tangentially fired Combustion Engineering boilers generally displayed lower NOx levels than faced or opposed fired boilers due to the creation, in the center of the boiler, of a fuel rich vortex which provided a reducing environment for NOx formed in the primary combustion zone.

The decay in the conversion efficiency to lower levels, as the nitrogen weight fraction within the fuel is increased, supports, on the other hand, the possibility that nitrogen containing radicals produced by the fuel provide a reducing agent for NOx formed in the primary combustion zone. This appears plausible since studies performed by Axworthy et al.(11), Song et al.(12) and Kahn et al.(13), among others, showed that in a locally fuel rich environment, ammonia reducing radicals (NH₂, NH) and other nitrogen containing compounds such as HCN are produced by the fuel nitrogen. It therefore appears that, as much as the fuel nitrogen produced NOx, it also achieved a reduction of NOx resulting in the progressive decay of nitrogen conversion

rate. As expected, locally fuel rich stoichiometry within the burner flame enhanced fuel pyrolysis and thus the production of the radicals. This, further explains why improved atomization, which generally enhances fuel vaporization and mixing within the flame, has consistently resulted in higher conversion efficiency of the fuel nitrogen.

The effect of excess O_2 within the combustor on fuel nitrogen conversion to NO_x is presented in Figure 14. The data showed, as expected, that an increase in O_2 level produced a corresponding increase in the nitrogen conversion efficiency. The variation in the conversion efficiency with O_2 was fairly linear for high nitrogen concentration but more or less irregular for the low nitrogen concentration. Improved fuel atomization again produced an increase in the conversion efficiency. In addition, the high nitrogen concentration showed consistently lower conversion efficiency than that obtained with low nitrogen concentration. The noted O_2 effect on nitrogen conversion is again in agreement with Sarofim⁽³⁾ postulate that locally fuel lean stoichiometry enhances fuel NO_x production. The O_2 effect also explains the increase in conversion efficiency with improved atomization which generally induces rapid vaporization and mixing in the primary combustion zone. The data also supports the effect of burner stoichiometry on nitrogen conversion as reported by Mansour and Jones⁽⁷⁾ and explains the dramatic reduction in NO_x emission experienced by Arand and Muzio⁽¹⁴⁾ during the combustion of SRC-II fuel oil in the off-stoichiometric firing mode.

It is evident based on the results presented above that NO_x emission from SRC-II fuel blends is influenced by burner design and operating variables and by the properties of the fuel blend used. No attempt was made during this program to optimize burner variables to reduce NO_x emission levels. It is felt, however, that with good understanding of the factors that influence fuel nitrogen conversion to NO_x , the design of burner hardware optimized to minimize NO_x emission from SRC-II fuel oil is a reasonable and achievable goal. Based on the presented results, burning this new fuel in some conventional steam or mechanical atomized burners designed to achieve intense mixing and efficient combustion of fuels, would not produce the potential minimum NO_x levels. Burner hardware required to control NO_x formation from the new fuel may have to provide, as discussed earlier, delayed mixing and control of local fuel stoichiometry within the flame.

It is also evident, based on the results, that fuel properties could impact NO_x emission levels. The data showed, for example, that the fuel atomization (surface tension and viscosity) and vaporization characteristics are important in determining the conversion level of fuel bound nitrogen to NO_x . It is therefore reasonable to assume that the proportions by which the medium and heavy distillates of SRC-II fuel oil are blended may have a significant effect on the NO_x levels. While high proportions of the heavy oil fractions would be desirable to provide delayed evaporation and pyrolysis for the production of NO_x reducing

radicals, such a high concentration could dictate excessive excess O₂ levels which may adversely affect NO_x emission. On the other hand, larger proportions of SRC-II fuel oil medium distillate may be tolerated and result in an optimum NO_x reduction and low excess O₂ operation if mixing intensity and local fuel stoichiometry within the burner flame are controlled by the appropriate burner design.

5.0 CONCLUSIONS AND RECOMMENDATIONS

The conclusions and recommendations that can be made based on the presented data are primarily as follows:

5.1 CONCLUSIONS

- SRC-II fuel oil can be burned in a combustion apparatus in its pure form or blended with other conventional petroleum products without experiencing fuel handling, fuel mixing, combustion instability or smoke formation problems.
- NO_x emission is increased with improved atomization of SRC-II fuel oil blends and with the increase in O₂ level within the furnace. The rate of increase in the emission levels is more pronounced at high nitrogen concentration within the fuel.
- Variation of NO_x emission with excess O₂ appeared to be influenced by the distillation characteristics of the fuel blends.
- Smoke limit for pure SRC-II fuel oil is influenced by the gaseous medium used for atomization and the lowest O₂ levels are achieved using natural gas as the atomizing medium.
- Smoke additives, when used in sufficient concentrations, are effective in reducing the smoke limit and NO_x emission from pure SRC-II fuel oil.
- Fuel nitrogen conversion efficiency to NO_x decreases with the increase in the concentration of organic bound nitrogen within the fuel. The NO_x emission from nitrogen contained in the fuel appears therefore, to be the result of two competing mechanisms: the first is a NO_x producing process which increases NO_x emission in proportion to the increase in the nitrogen content of the fuel; and the second is a destruction mechanism by which NO_x, formed early in the combustion process, is consumed by chemical species generated by the fuel during the combustion process.
- Improved fuel atomization and the increase in O₂ level enhanced fuel nitrogen conversion to NO_x.

- Nitrogen conversion is affected by the vaporization and atomization characteristics of SRC-II fuel oil blends.

5.2 RECOMMENDATIONS

- The combustion qualities and emission level for the medium and heavy cuts of SRC-II fuel oil should be examined separately to determine the compatibility of the two distillates with the various combustion applications.
- The handling of the two SRC-II fuel oil distillates in independent fuel networks to be burned in a single combustion apparatus in a dual firing arrangement may offer optimum reduction in NOx emission.
- The effect of the blend proportions of SRC-II fuel oil distillates on the emission and combustion of the fuel should be investigated in subscale and full-scale applications.
- Consideration should be given to the development of optimized burner hardware that is compatible with SRC-II fuel oil usage and which can potentially provide further reduction of NOx emission.

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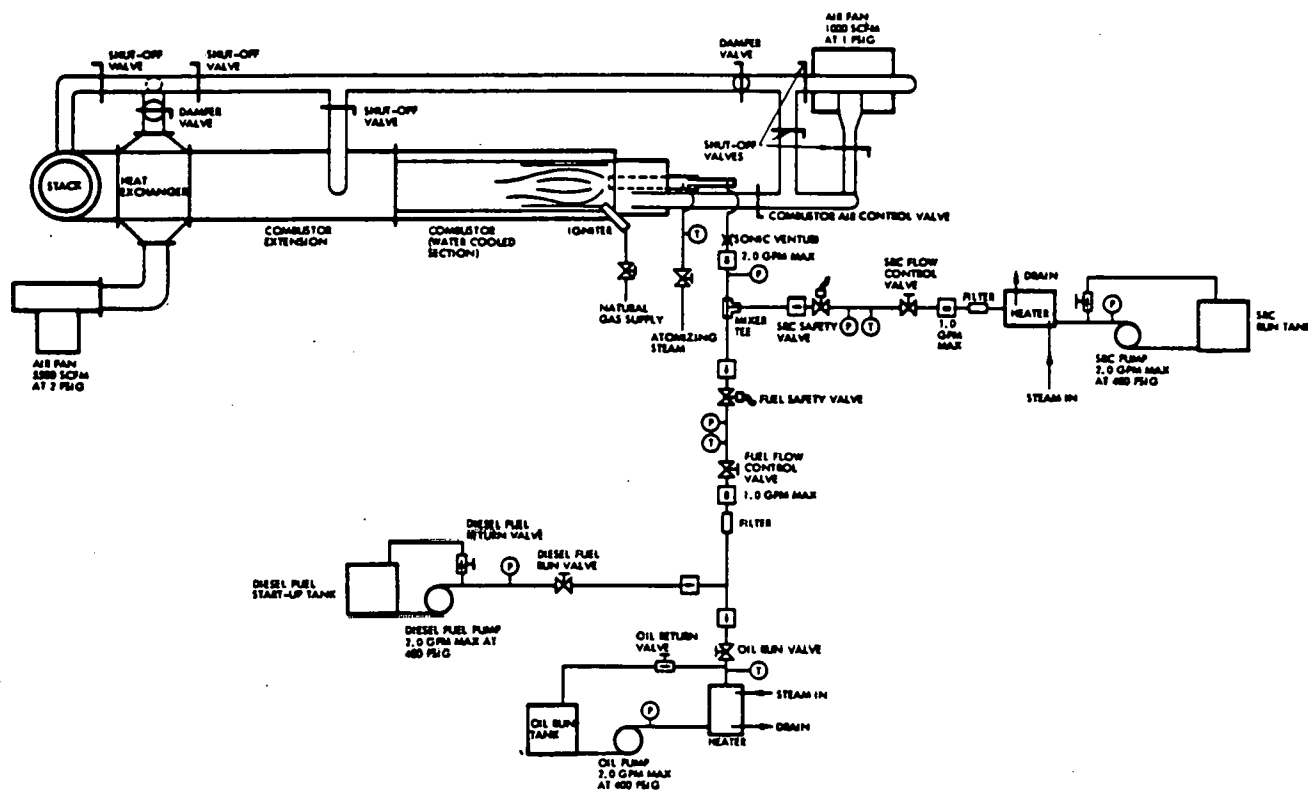


FIGURE 1 - SCHEMATIC LAYOUT OF TRW'S COMBUSTION FACILITY

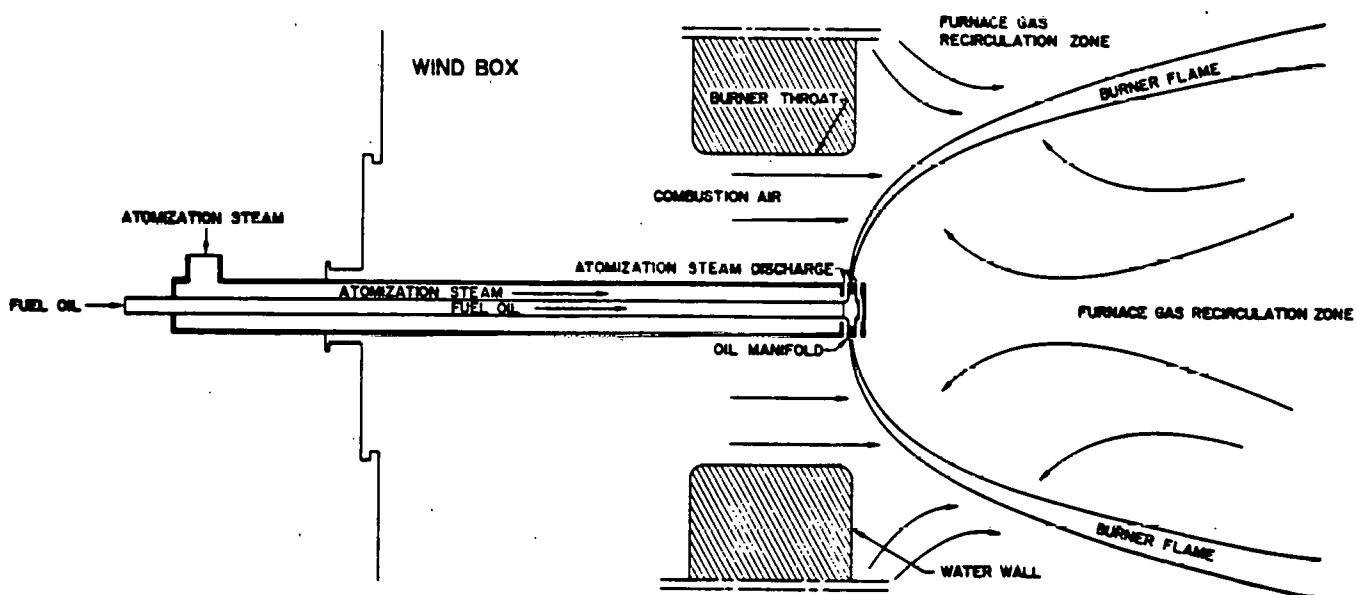


FIGURE 2 - SCHEMATIC VIEW OF SCE LOW NO_x BURNER

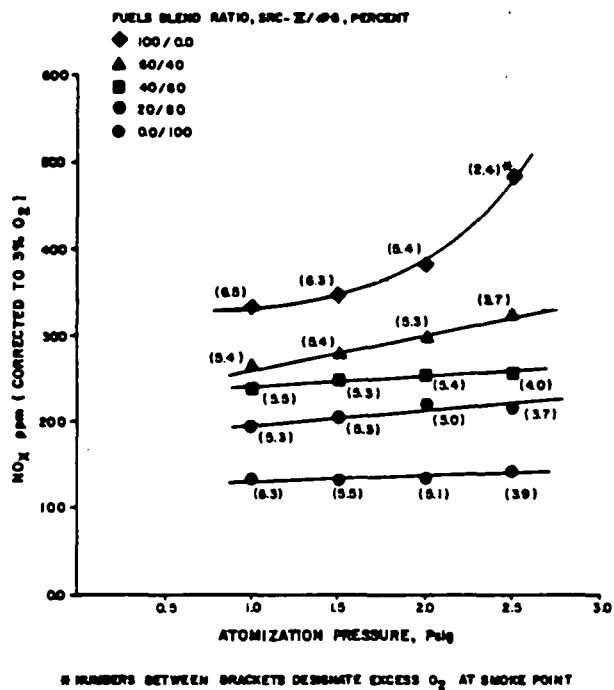


FIGURE 3 EFFECT OF FUEL ATOMIZATION ON NO_x EMISSION

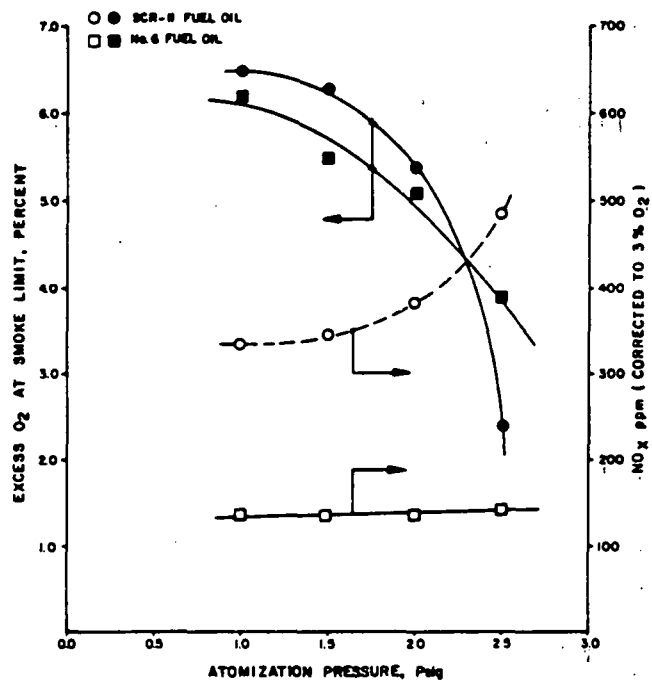


FIGURE 4 CORRELATION OF EXCESS O₂ TO NO_x EMISSION AT DIFFERENT ATOMIZATION PRESSURES

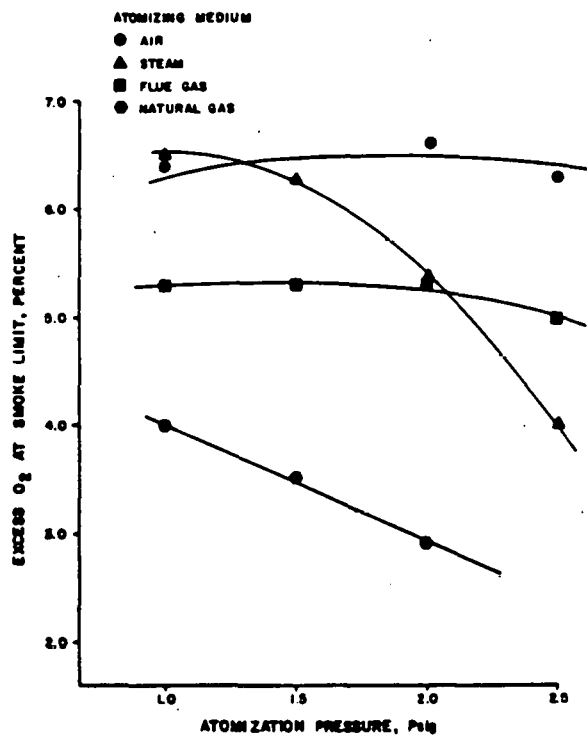


FIGURE 5 EFFECT OF TYPE OF ATOMIZING MEDIUM ON SMOKE LIMIT OF PURE SRC-II

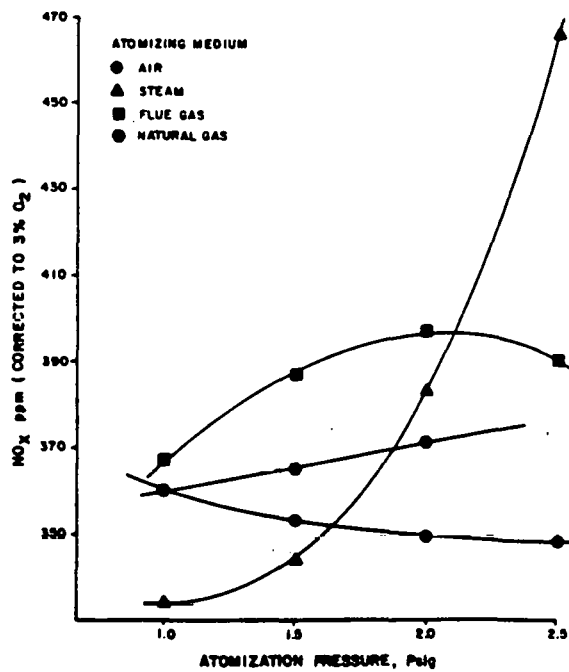


FIGURE 6 EFFECT OF TYPE OF ATOMIZING MEDIUM ON NO_x EMISSION FROM PURE SRC-II

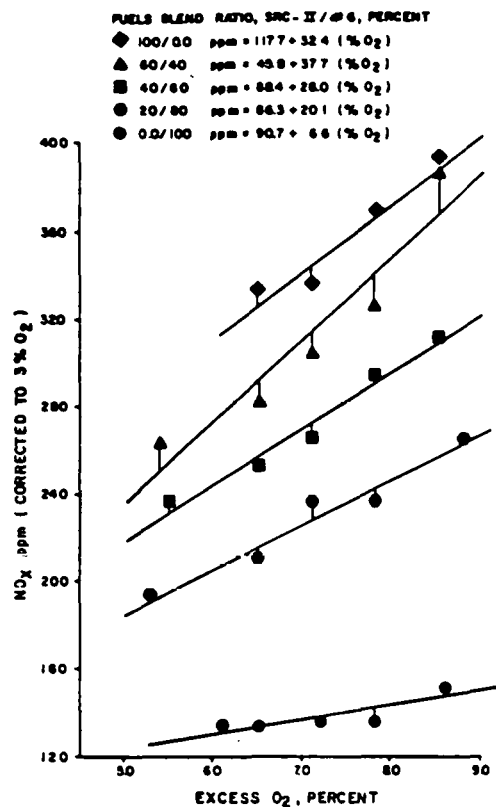


FIGURE 7 EFFECT OF EXCESS O_2 ON NO_X EMISSION

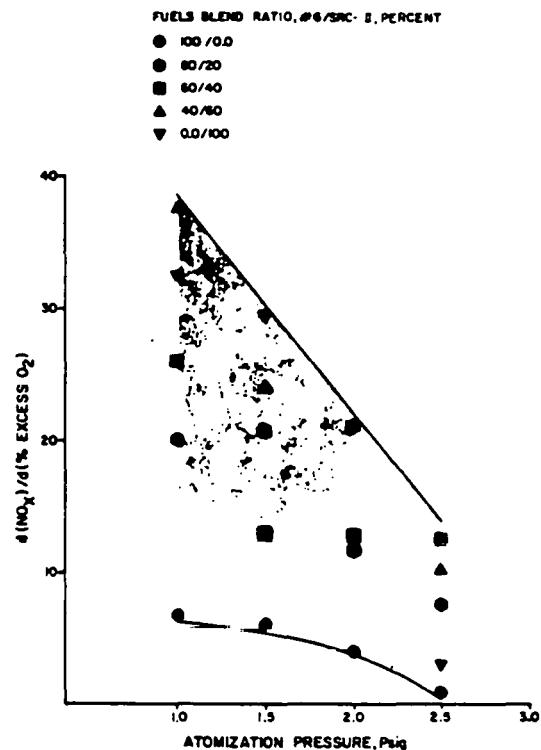


FIGURE 8 EFFECT OF ATOMIZATION ON NO_X VARIATION WITH EXCESS O_2

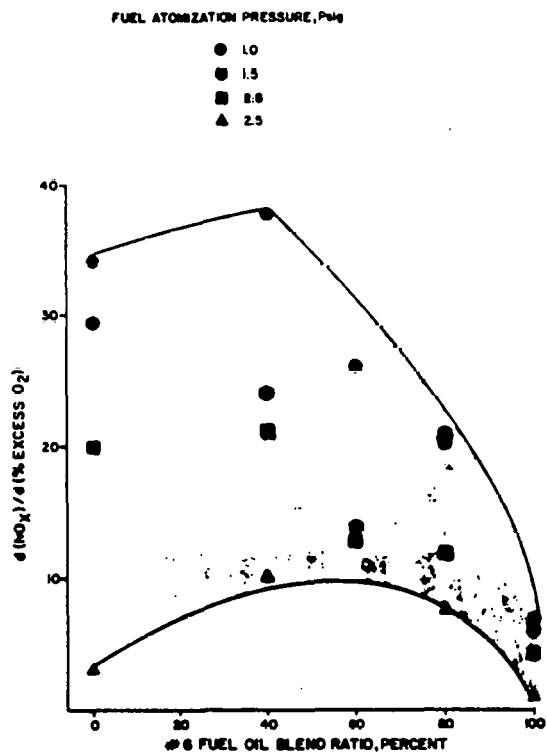


FIGURE 9 EFFECT OF FUEL NITROGEN ON BURNER SENSITIVITY TO EXCESS O_2

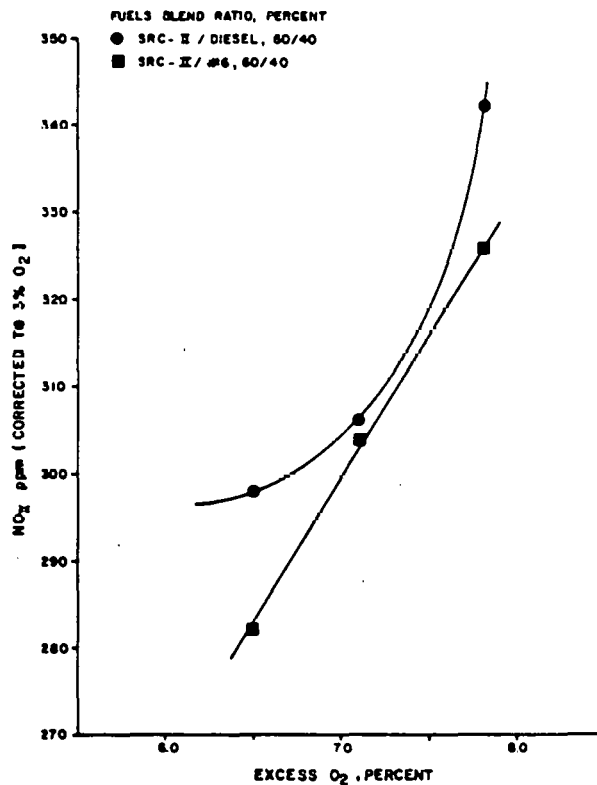


FIGURE 10 EFFECT OF FUEL VAPORIZATION CHARACTERISTICS ON VARIATION OF NO_X WITH EXCESS O_2

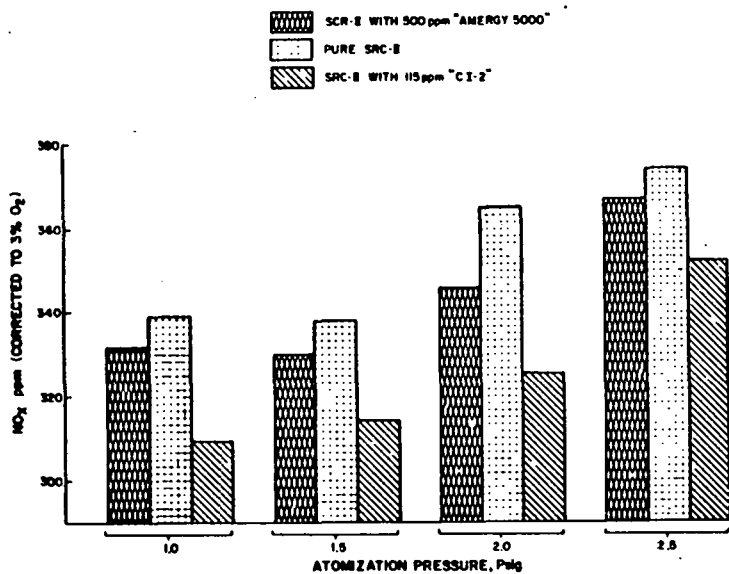


FIGURE 12 EFFECT OF COMMERCIAL SMOKE SUPPRESSANTS ON NO_x EMISSION FROM PURE SRC-II

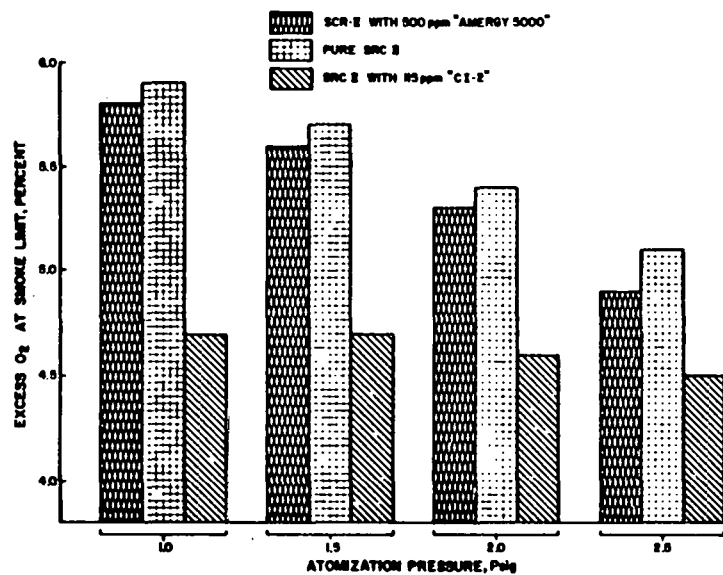


FIGURE 11 EFFECT OF COMMERCIAL SMOKE SUPPRESSANTS ON SMOKE LIMIT OF PURE SRC-II

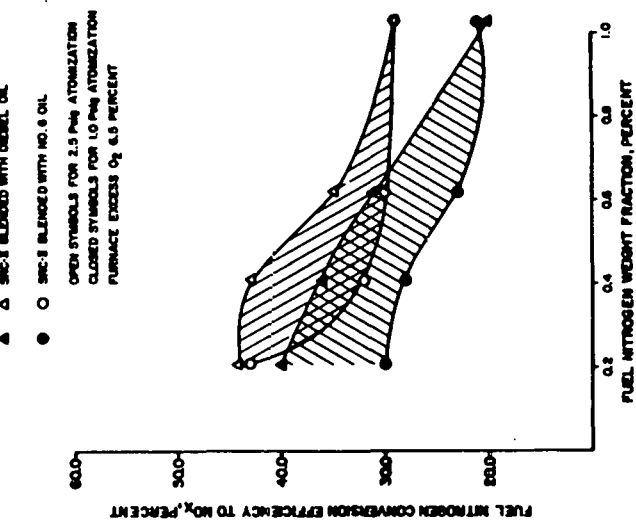


FIGURE 13 CONVERSION EFFICIENCY OF FUEL BOUND NITROGEN TO NO_x

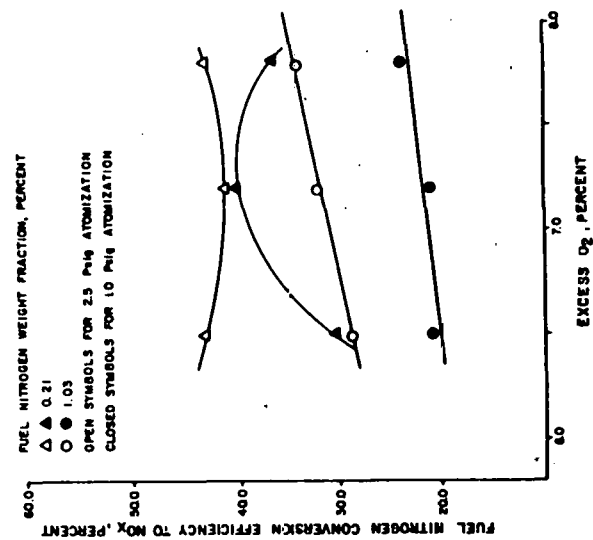


FIGURE 14 EFFECT OF ATOMIZATION AND O_2 ON FUEL NITROGEN CONVERSION EFFICIENCY TO NO_x

TABLE I
FUEL PROPERTIES

ELEMENT %	DIESEL OIL	#6 OIL	SRC-II	
			SAMPLE #1	SAMPLE #2
CARBON	86.50	85.80	86.34	85.98
HYDROGEN	12.76	12.65	8.74	8.60
NITROGEN	0.018	0.20	1.05	1.06
SULFUR	0.29	0.21	0.23	0.24
ASH	0.004	0.014	0.034	0.018
OXYGEN (BY DIFF.)	0.43	1.13	3.61	4.10
HEAT OF COMBUSTION				
GROSS BTU/LB		19,310	17,170	17,160
NET BTU/LB		18,160	16,370	16,380

Table 2: NO_x CONCENTRATION (PPM CORRECTED TO 3% O₂) IN
THE FLUE GASES WHEN 1.0% OF FUEL NITROGEN
IS COMPLETELY CONVERTED TO NO_x

Fuel Type	Blend Ratio Percent	Fuel Composition, Percent by Wt.					NO _x (ppm) @ 1% Fuel Nitrogen Conversion	V(exhaust). SCF/lb of fuel
		C	H ₂	N ₂	S	O ₂		
# 6	100%	85.8	12.65	0.20	0.21	1.13	1160	234
SRC-II	100%	86.34	8.74	1.05	0.23	3.61	1302	208
Diesel	100%	86.82	12.69	0.024	0.11	0.353	1149	236
SRC-II/#6 Blends	20/80	85.91	11.87	0.37	0.21	1.63	1186	229
	40/60	86.02	11.09	0.54	0.22	2.12	1213	224
	60/40	86.12	10.30	0.71	0.22	2.62	1242	218
	80/20	86.23	9.52	0.88	0.23	3.11	1271	213
SRC-II/Diesel Blends	20/80	86.62	11.90	0.23	0.13	0.51	1176	230
	40/60	86.63	11.11	0.43	0.16	1.66	1205	225
	60/40	86.53	10.32	0.64	0.18	2.31	1236	219
	80/20	86.44	9.53	0.84	0.21	2.96	1268	214
#6/Diesel Blends	20/80	86.62	12.68	0.06	0.13	0.51	1151	234
	40/60	86.41	12.67	0.09	0.15	0.66	1153	235
	60/40	86.21	12.67	0.13	0.17	0.82	1156	235
	80/20	86.00	12.66	0.16	0.19	0.97	1158	234

TABLE 3: FUEL NITROGEN CONVERSION EFFICIENCY

Fuel Type	Blend Ratio Percent	Burner Atom. Pressure psig	Excess O2 Percent	Base NOx (ppm) Without Fuel Nitrogen	NOx(ppm) with Fuel Nitrogen	Increase in NOx Emission	Increase in Fuel Nitrogen	NOx(ppm) @ 1% Fuel Nitrogen Conversion	Fuel Nitrogen Conversion Eff. Percent
Diesel/ SRC-II	0/100	1.0	6.5	58	334	278	1.03	1302	21.0
	40/60	1.0	6.5	58	298	242	0.62	1236	31.6
	60/40	1.0	6.5	58	236	180	0.41	1205	36.4
	80/20	1.0	6.5	58	159	103	0.21	1176	41.7
Diesel/ SRC-II	0/100	1.0	7.1	60	337	277	1.026	1302	20.7
	40/100	1.0	7.1	60	306	246	0.618	1236	32.3
	60/40	1.0	7.1	60	244	184	0.406	1205	37.6
	80/120	1.0	7.1	60	140	80	0.206	1176	33.0
Diesel/ SRC-II	0/100	1.0	7.8	53	369	316	1.03	1302	23.5
	40/100	1.0	7.8	53	342	289	0.62	1236	37.7
	60/40	1.0	7.8	53	255	202	0.41	1205	41.2
	80/20	1.0	7.8	53	149		0.21	1176	38.8
Diesel/ SRC-II	0/100	2.5	6.5	56	449	393	1.03	1302	29.3
	40/60	2.5	6.5	56	328	272	0.62	1236	35.5
	60/40	2.5	6.5	56	272	216	0.41	1205	43.7
	80/20	2.5	6.5	56	170	114	0.21	1176	46.1
Diesel/ SRC-II	0/100	2.5	7.1	59	492	433	1.03	1302	32.3
	40/60	2.5	7.1	59	363	304	0.62	1236	39.6
	60/40	2.5	7.1	59	275	216	0.41	1205	43.7
	80/20	2.5	7.1	59	175	116	0.21	1176	47.0
Diesel/ SRC-II	0/100	2.5	7.7	54	512	458	1.03	1302	34.1
	40/60	2.5	7.7	54	379	325	0.62	1236	42.3
	60/40	2.5	7.7	54	280	226	0.41	1205	45.7
	80/20	2.5	7.7	54	161	107	0.21	1176	43.3
#6/ SRC-II	0/100	1.0	6.5	58	334	276	1.03	1302	20.7
	4/60	1.0	6.5	107	282	175	0.62	1215	23.2
	60/40	1.0	6.5	115	253	138	0.41	1213	27.8
	80/20	1.0	6.5	135	211	76	0.21	1186	30.5
#6/ SRC-II	0/100	1.0	7.1	60	337	277	1.03	1302	20.7
	40/60	1.0	7.1	104	304	200	0.62	1242	26.0
	60/40	1.0	7.1	121	265	144	0.41	1213	29.0
	80/20	1.0	7.1	137	237	100	0.21	1186	40.1
#6/ SRC-II	0/100	1.0	7.8	53	369	316	1.03	1302	23.7
	40/60	1.0	7.8	105	326	221	0.62	1242	28.7
	60/40	1.0	7.8	127	295	168	0.41	1213	33.8
	80/20	1.0	7.8	146	237	91	0.21	1186	36.5
#6/ SRC-II	0/100	2.5	6.5	56	449	393	1.03	1302	29.4
	40/60	2.5	6.5	115	347	232	0.62	1242	30.1
	60/40	2.5	6.5	130	290	160	0.41	1213	32.2
	80/20	2.5	6.5	139	247	108	0.21	1186	43.4
#6/ SRC-II	0/100	2.5	7.1	59	492	433	1.03	1302	32.4
	40/60	2.5	7.1	115	335	220	0.62	1242	28.6
	60/40	2.5	7.1	127	298	171	0.41	1213	34.4
	80/20	2.5	7.1	143	246	103	0.21	1186	41.6
#6/ SRC-II	0/100	2.5	7.6	54	512	458	1.03	1302	34.3
	40/60	2.5	7.8	112	382	270	0.62	1242	35.1
	60/40	2.5	7.8	131	304	173	0.41	1213	34.8
	80/20	2.5	7.8	139	246	107	0.21	1186	43.0

A P P E N D I X E

Report No. ENG. R-79-13
Preliminary Combustion Evaluation of
Gulf Oil SRC II Pyrolytic Fuel
Using ORF's 10×10^6 Btu/Hr.
Vortometric Burner Facility
for

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February 13, 1979

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Report No. ENG. R-79-13

Preliminary Combustion Evaluation of
Gulf Oil SRC II Pyrolytic Fuel
Using ORF's 10×10^6 Btu/Hr.
Vortometric Burner Facility

for

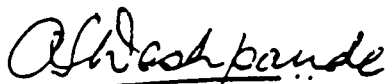
Gulf Oil Canada Limited

SUMMARY

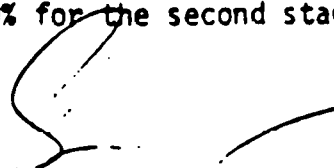
The Ontario Research Foundation undertook a series of preliminary combustion tests using Gulf's SRC II Pyrolytic fuel and No. 6 fuel oil in a standard 10×10^6 Btu/Hr. Vortometric burner.

The test objective was to determine whether the SRC II fuel could be burned in a standard Gulf Vortometric Burner and to evaluate the exhaust quality, particularly regarding NO_x emissions. Attempts were made to determine if NO_x emissions could be reduced by simple modifications allowing two-stage combustion.

Results from this preliminary evaluation show that the SRC II Pyrolytic fuel can be burned using a Vortometric burner without any modification and that two-stage combustion reduces the NO_x emissions for both the Pyrolytic fuel and the No. 6 fuel oil as compared to single stage combustion. The results also indicate that two-stage combustion is best achieved when approximately 50% of the total air is delivered to the burner and the remaining 50% for the second stage in the test configuration.



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1. INTRODUCTION

The Ontario Research Foundation (ORF) carried out preliminary combustion tests using Gulf's Pyrolytic fuel SRC II and No. 6 fuel oil on the existing 10 MBTU/Hr. Vortometric burner setup.

The primary test objective was to evaluate the performance of the SRC II fuel and No. 6 fuel with respect to exhaust emissions, in particular NO_x emissions, and to determine if two-stage combustion would result in reduced NO_x emissions. The staged combustion tests were carried out for various primary to secondary air ratios to evaluate the effect on NO_x emissions.

The tests were all conducted using a standard 10 MBTU/Hr. Vortometric burner firing into a 44 ft. long flame tube of 28" nominal diameter, as shown in Figure 2.

2. TECHNICAL DISCUSSION

In ordinary combustion, it is customary to assume that nitrogen present in the fuel or in the combustion air is inert and does not participate in the combustion reactions. However, when a mixture of nitrogen and oxygen is subjected to a high temperature ($>2800^\circ\text{F}$), such as provided by a flame, it will react to produce a significant amount of NO .

Emissions of oxides of nitrogen result from high temperature reactions of atmospheric nitrogen and oxygen in the combustion zone, as well as from the partial combustion of nitrogen contained in the fuel. The Pyrolytic test fuel with its high nitrogen content (approximately 1%, as shown in Table 4), has the potential for high NO_x emissions.

Numerous studies have been carried out indicating that nitrogen oxide emissions can be reduced by a number of different techniques such as reduced excess air, flue gas recirculation, staged combustion, etc.

Preliminary combustion tests at ORF were aimed at studying NO_x reduction using simple and cost effective two-stage combustion incorporating a standard Vortometric burner.

3. TEST FACILITY AND INSTRUMENTATION

3.1 Test Facility

The test facility used to carry out the program is shown schematically in Figure 2, which essentially consists of a standard MK 11B Vortometric Burner (10×10^6 Btu/Hr.) firing into a 44' long flame tube, approximately 28" diameter. The flame tube consists of eleven (11) 4 ft. stainless steel flanged sections which are connected together. The first eight feet of the flame tube is refractory lined with 2-1/2" fire brick and the remaining length is unlined.

Secondary air is introduced through a 4" x 4" opening in the side of the flame tube downstream of the atomizer. The second stage air is delivered to the port using a calibrated 4" diameter duct connected to a combustion air blower.

Primary combustion air was provided by a 10 H.P. blower rated at 2000 SCFM @ 18" W.C. A 10 ft. long, 8" diameter duct with a bell-mouth inlet was connected to the blower inlet.

The SRC II fuel was delivered, using a positive displacement gear pump connected to the fuel tank supplied by Gulf. The No. 6 fuel oil was delivered from the existing storage tank.

3.2 Instrumentation

Ambient air conditions were measured using a barometer, relative humidity meter and a thermocouple. Combustion air flows were measured using inclined manometers connected to calibrated Pitot tubes installed in the 8" diameter primary air duct and the 4" diameter secondary air duct. The SRC II fuel flow was measured with a positive displacement No. 2 fuel oil meter with $\pm 1\%$ rated accuracy and the No. 6 fuel was measured using positive displacement No. 6 fuel meter with $\pm 0.2\%$ accuracy. The temperature and pressure of the fuels and atomizing steam were measured using temperature and pressure gauges.

The exhaust emissions were measured using analytical instruments and equipment contained in a mobile laboratory. Samples of the flue gas were extracted from inside the flame tube (approx. 22 ft. downstream of the atomizer), using a stainless steel line connected to the analysers listed below. All of the flue gas sampling instruments were calibrated using calibration gases at various times during the tests.

Flue Gas Emission Instrumentation

<u>Emission Measured</u>	<u>Type of Instrument</u>
O ₂	Paramagnetic
CO ₂	Nondispersive Infrared
CO	Nondispersive Infrared
THC	Flame Ionization
NO	Chemilluminescent
NO _x	Chemilluminescent

4. TEST PROCEDURE

Preliminary attempts at two-stage combustion tests were carried out using the setup shown in Figure 1. Although the secondary air injection was uncontrolled, the Pyrolytic fuel provided sustained

combustion. These tests proved that introduction of secondary air did not quench the flame or cause flame-out.

The test burner setup (Figure 2) was operated with natural gas until steady state conditions were reached. The burner was then fired using the SRC II fuel at nominally 5×10^6 Btu/Hr. and 11% excess air. Subsequent two-stage combustion tests for various primary to secondary air ratios were carried out using the same firing rate and excess air parameters for comparison.

Each combustion test was run until the analyser readings stabilized. On completion of each test, the air flow was adjusted to a different primary to secondary air ratio (while maintaining the overall excess air constant).

Tests using No. 6 fuel oil were carried out in a similar manner and at nominally the same operating conditions described above.

5. TEST RESULTS

Tables 1, 2 and 3 show the readings taken during the test program using the SRC II fuel and the No. 6 fuel oil. Figures 3 and 4 show the effect of primary/secondary air ratios on NO_x and CO emissions for No. 6 fuel oil and the SRC II Pyrolytic fuel oils for nominally constant overall excess air and firing rates.

6. CONCLUSIONS AND RECOMMENDATIONS

- 6.1 The preliminary tests carried out have proven that Gulf's SRC II Pyrolytic fuel can be used in a standard 10×10^6 Btu/Hr. Vortometric burner without any major modifications.
- 6.2 Two-stage combustion reduces nitrogen oxide emission levels for both the SRC II Pyrolytic fuel and No. 6 fuel oils.

- 6.3 The nitrogen oxide emission levels for the SRC II Pyrolytic fuel were minimized when approximately 50% of the total air is used as primary air and the remaining 50% is introduced for secondary combustion. When No. 6 fuel oil was used, the minimum NO_x was found to occur at approximately the same ratio.
- 6.4 Results indicate that as nitrogen oxide emission levels decrease, carbon monoxide (CO) emissions increase. This can be explained since the combustion reaction temperature is reduced through two-stage combustion, resulting in increased production of CO.
- 6.5 The optimum primary/secondary combustion air distribution ratio will depend on many factors such as the geometry of the combustion chamber, the overall excess air ratio, the firing rate, etc. Further studies are essential to determine correlation between these variable parameters in the context of the Gulf Vortometric burner.

TABLE 1

PROJECT: GULF PYROLYTIC FUEL ACC. N° 36013

DATE: Feb. 7/79 AMBIENT TEMPERATURE: 23°F BAROMETRIC PRESSURE: 29.84 "Hg

TEST NO: 1 to 5 RELATIVE HUMIDITY: 35-40%

Time	FUEL: PYROLYTIC				ATMOSPHERIC FLUID: STEAM				COMBUSTION AIR								EXHAUST GAS ANALYSIS										COMMENTS
	Temp. °F	Pressure P.S.I.A.	Flow Rate S.C.F.H.	Firing Rate MBTU/hr.	Temp. °F	Pressure P.S.I.A.	Water Boiling	Calculated Flow Rate lb/hr.	Primary Air				Secondary Air				O ₂ %	CO ₂ %	CO PPM	THC PPM	H ₂ PPM	H ₂ O PPM	% EX. AIR	TOW. °F			
									Temp. °F	Wadlow Stat. Press. " H ₂ O	Velocity Pressure " H ₂ O	Flow Rate S.C.F.H.	Temp. °F	Wadlow Stat. Press. " H ₂ O	Velocity Pressure " H ₂ O	Flow Rate S.C.F.H.											
TEST NO. 1																											
9:38	48	23.5	0.5/123		317	50.5			26	1.6	0.48						0.4	16	140	14	680	640	1.83	853	possible		
9:40	45	23.5	0.5/123		315	48			26	1.7	0.44						0.5	140		770	680	2.3		fuel meter			
9:43	43	23.5	N.A.		NA	NA			NA	NA	NA														used test		
TEST NO. 2																											
SINGLE STAGE																											
10:57	43	23.5	0.5/64		300	51			26	1.8	0.43						0.2	152	50	3	640	670	2.77	862			
11:03	43 1/2	23.5	0.5/64 1/2		295	50			26	1.8	0.44						1.0	163	1000	6	610	770		870			
11:06	44	23.5	0.5/64 1/2		295	49.5			27	1.8	0.44																
TEST NO. 3																											
TWO STAGE 75% PRI. AIR, 25% SEC. AIR																											
11:26	43 1/2	26	0.5/61		305	54			39	1.5	0.29						2.2	140	40	4	400	370	12.77	810	✓		
11:32	43 1/2	26	0.5/62		295	50			39	1.6	0.27						2.6	152	80	1.0	430	420		815			
11:35	43	26	2.5/320		295	50			39	1.6	0.27																
TEST NO. 4																											
TWO STAGE 50% PRI. AIR, 50% SEC. AIR																											
11:43	45	21.5	0.5/65		295	50			38	0.8	0.13																
11:46	44	22.5	0.5/64		295	50			38	0.9	0.125						2.1	144	70	4	140	120	11.25	807	✓		
11:50	43	22	0.5/62		290	50			38	0.9	0.14						3.3	150	220	1.0							
11:52	43 1/2	22	0.5/62		295	49			38	1.0	0.15																
TEST NO. 5																											
TWO STAGE 90% PRI. AIR, 10% SEC. AIR																											
11:57	43 1/2	20	1.0/116		295	50			38.5	1.5	0.365						2.1	162	2		500	500	7.37	825			
12:03	43 1/2	22 1/2	0.5/62		295	50			38.5	2.4	0.33						0.5	1000		570	570						

TABLE 2

PROJECT: GULF PYROLYTIC FUEL ACC. NO. 36013

DATE: FEB. 7/79 AMBIENT TEMPERATURE: 30°F BAROMETRIC PRESSURE: 29.84" Hg

TEST NO: 670 B RELATIVE HUMIDITY: 35-40%

Time	FUEL: PYROLYTIC				ATMOS. FLUID: STEAM				COMBUSTION AIR								EXHAUST GAS ANALYSES										COMMENTS
	Temp. °F	Pressure P.S.I.C.	Flow Rate S.C.F.H.	Firing Rate MBTU/hr.	Temp. °F	Pressure P.S.I.C.	Meter Reading	Calculated Flow Rate lb/hr.	Primary Air				Secondary Air				O ₂ %	CO ₂ %	CO PPM	THC PPM	H ₂ PPM	H ₂ O PPM	% EX. AIR	TEMP. °F			
									Temp. °F	Windbox Stat. Press. " Hg	Velocity Pressure " Hg	Flow Rate S.C.F.H.	Temp. °F	Windbox Stat. Press. " Hg	Velocity Pressure " Hg	Flow Rate S.C.F.H.											
	TEST NO. 6				REPEAT OF NO. 3				SINGLE STAGE				11-12% EX. AIR														
3-03	41.5	22.5	0.5/61		290	47			30	2.5	0.54						2.1	140	80	40	140	170	11.87	861	✓		
3-06	42	22.5	0.5/63		290	49			30	2.5	0.55						2.6	150	120		190	170					
	TEST NO. 7				TWO STAGE				60% PRI. AIR ;				40% SEC. AIR														
3-31	42	23	0.5/61		285	40			30	1.7	0.26					1.15		140	30	7.6	300	290	13.15	838	✓		
3-32	42	23	0.5/60		285	40			30	1.8	0.275					1.16		27	40	140							
3-34	40	22.5	0.5/60		285	42			30	1.8	0.275					1.16											
	TEST NO. 8				TWO STAGE				40% PRI. AIR ;				60% SEC. AIR														
3-42	38	22.5	0.5/61		285	41			30	0.8	0.09					2.6		2.3	140	80	54	80	270	11.57	813	✓	
3-45	37	22.5	0.5/60		290	42			30	0.8	0.1					2.55		2.4	100								
3-49	37	22.5	0.5/60		290	42			30	0.8	0.1					2.55											

TABLE 3

PROJECT: GULF PYROLYTIC FUEL Acc. No. 36013DATE: Feb. 2/79
TEST NO: 2 to 14AMBIENT TEMPERATURE: 30
RELATIVE HUMIDITY: 35-40%BAROMETRIC PRESSURE: 29.84" Hg

Time	FUEL: # NO. 6 *				ATOMIZING FLUID: STEAM				COMBUSTION AIR				EXHAUST GAS ANALYSES										COMMENTS																																																																																																																																																																																																																																																																																																																																																																																
	Temp. °F	Pressure P.S.I.G.	Flow Rate L.B.P.H.	Firing Rate MBTU/hr.	Temp. °F	Pressure P.S.I.G.	Water Loading	Calculated Flow Rate LB/hr.	Primary Air				Secondary Air				O ₂ %	CO %	CO PPM	H ₂ PPM	H ₂ O PPM	H ₂ PPM		H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM	H ₂ PPM

PITTSBURGH & MIDWAY COAL MINING COMPANY
 SOLVENT REFINED COAL PILOT PLANT
 P.O. Box 199 DuPont, W. Va. 25827
 Phone: 206-954-5155

Laboratory Tests Report

on
 SAMPLE SHIPMENTS

Shipment No. 679-S

Shipment Sample No. 1342

Date Sampled 9/8/78 Time Sampled — Sample Point Blend Tank

Date Tested 9/14/78 Time Tested — Tag No. S.S.# 1341R

Sample Kind SRC-II Fuel Oil (5.75/1 Middle & Heavy Distillates)

Sample Shipped to: Mr. Craig Henderson

G. O. C. Co.

TABLE 4

ULTIMATE ANALYSIS OF SRC II PYROLYTIC FUEL

Sp. Gravity 60/60°F	<u>0.995</u>	Fusion Point (Gradient Bar)	<u>—</u> °F
Density @ 60°F	<u>—</u>	% Ash	<u>0.04</u>
Kin. Vis. @ 77°F	<u>—</u>	% Sulfur	<u>0.30</u>
@ 100°F	<u>4.02</u>	% Water	<u>Trace</u>
@ 210°F	<u>1.22</u>	<u>—</u>

Distillation: ASTM D-86 @ atn. mm Hg

Initial Boiling Point	<u>340</u> °F	70%	<u>500</u> °F
5%	<u>380</u> °F	80%	<u>530</u> °F
10%	<u>402</u> °F	90%	<u>595</u> °F
20%	<u>408</u> °F	95%	<u>698</u> °F
30%	<u>432</u> °F	End Point	<u>712</u> °F
40%	<u>446</u> °F	Recovery	<u>97</u> %
50%	<u>460</u> °F	Residue	<u>3</u> %
60%	<u>478</u> °F	Lost	<u>—</u> %

Elementals :

% Carbon	<u>85.91</u>	%	<u>—</u>
% Hydrogen	<u>8.74</u>	%	<u>—</u>
% Nitrogen	<u>0.97</u>	%	<u>—</u>
% Sulfur	<u>0.30</u>	%	<u>—</u>
by difference % Oxygen	<u>4.08</u>	%	<u>—</u>

Remarks : Conversion Carbon Residue 0.51%; Flash Point (Distillation Closed Tester) 17.3°F; Pour Point -55°F;
Coal Tar Residue 24.2%; Btu/lb 17,100.

Analyst : W. S. [Signature]

Chemist : [Signature] 147

TABLE 5

ANALYSIS: GULF FUEL OIL #6C1.5

GRAVITY	10.7
FLASH PT. °F	122
VISCOSITY (FUROL @122°F)	426
POUR PT. °F	+40
% SULFUR	1.01
% ASH	0.097 @1000°F
% ASH	0.02 @1500°F
% WATER	NIL
WT% NITROGEN (N ₂)	0.201
SEDIMENT (BS&W)	0.05
WT% SULFUR	1.29

METALS (SEMI-QUANTITATIVE) AS % OF ASH

AL	1-10	Ni	1-10
Ba	0.1-1	Si	>10
Ca	1-10	Na	1-5
Cu	0.1-1	Ti	<<1
Fe	>10	V	1-10
Pb	0.1-1		
Mg	0.1-1	Ni GCM 195	- 14.9 (ppm of sample)
Mn	0.1-1	V GCM 195	- 30.5 (ppm of sample)
Mo	0.1-1	Na GCM 130	- 6.19 (ppm of sample)

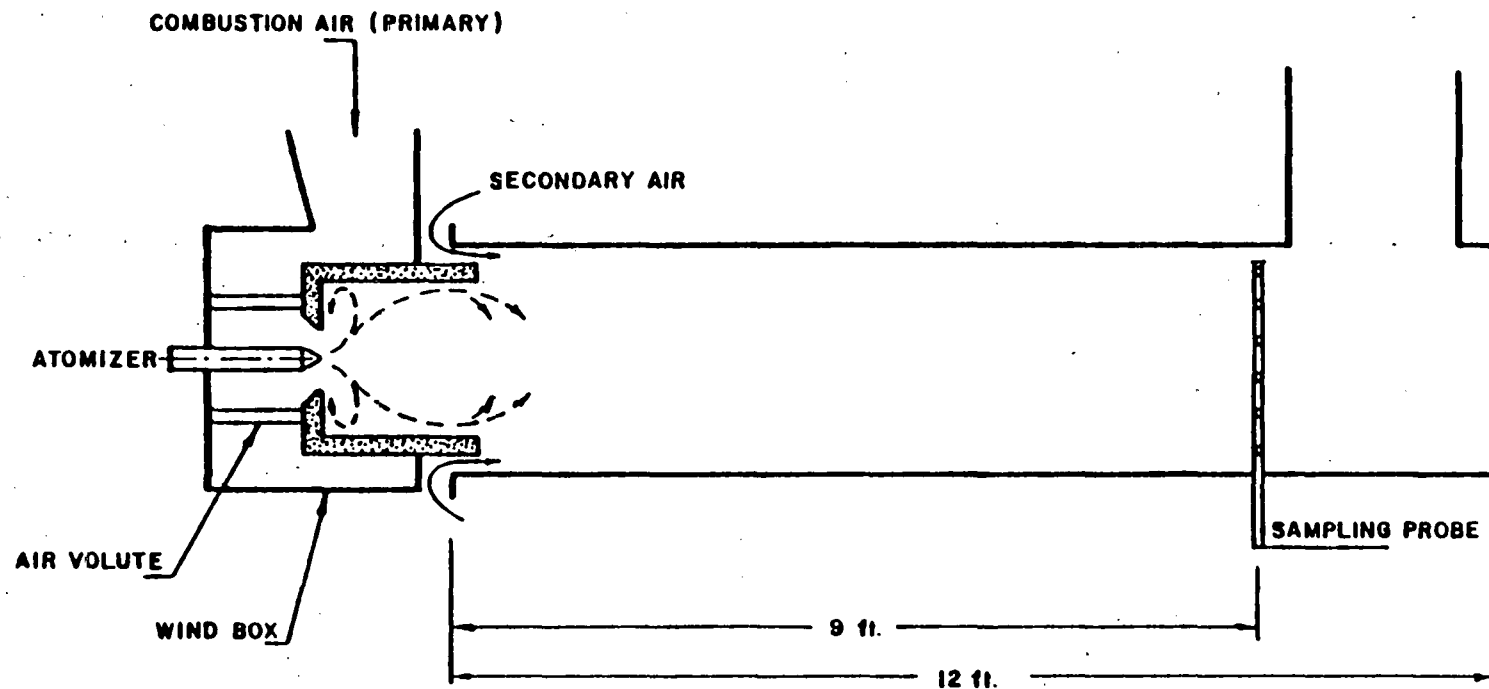


FIGURE I .

VORTOMETRIC BURNER WITH COMBUSTION TUNNEL SEPARATED TO ALLOW FOR TWO STAGE COMBUSTION
PRELIMINARY TRIALS

NOTE: Test Bed Facility employs a proprietary configuration and has not been included.

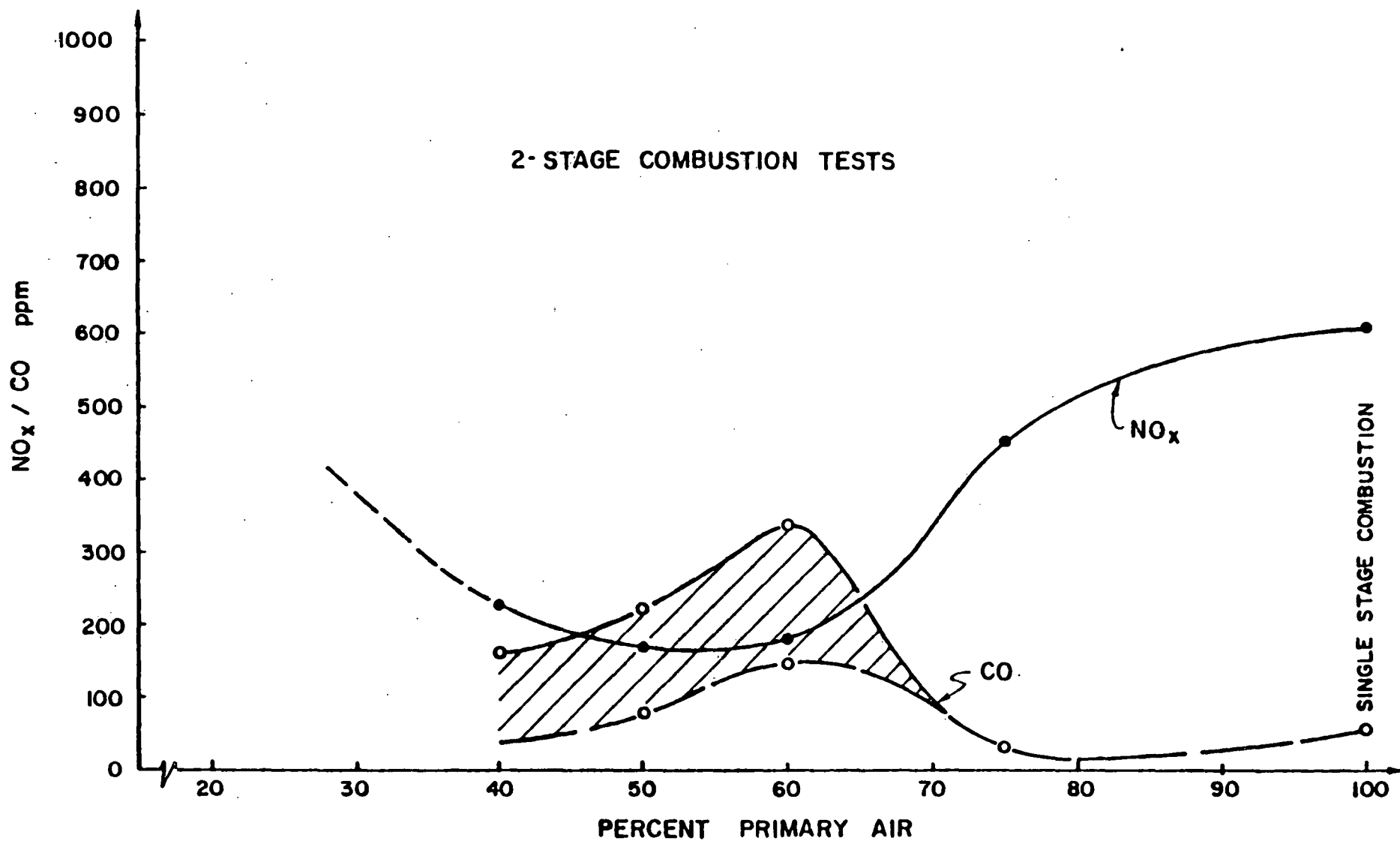
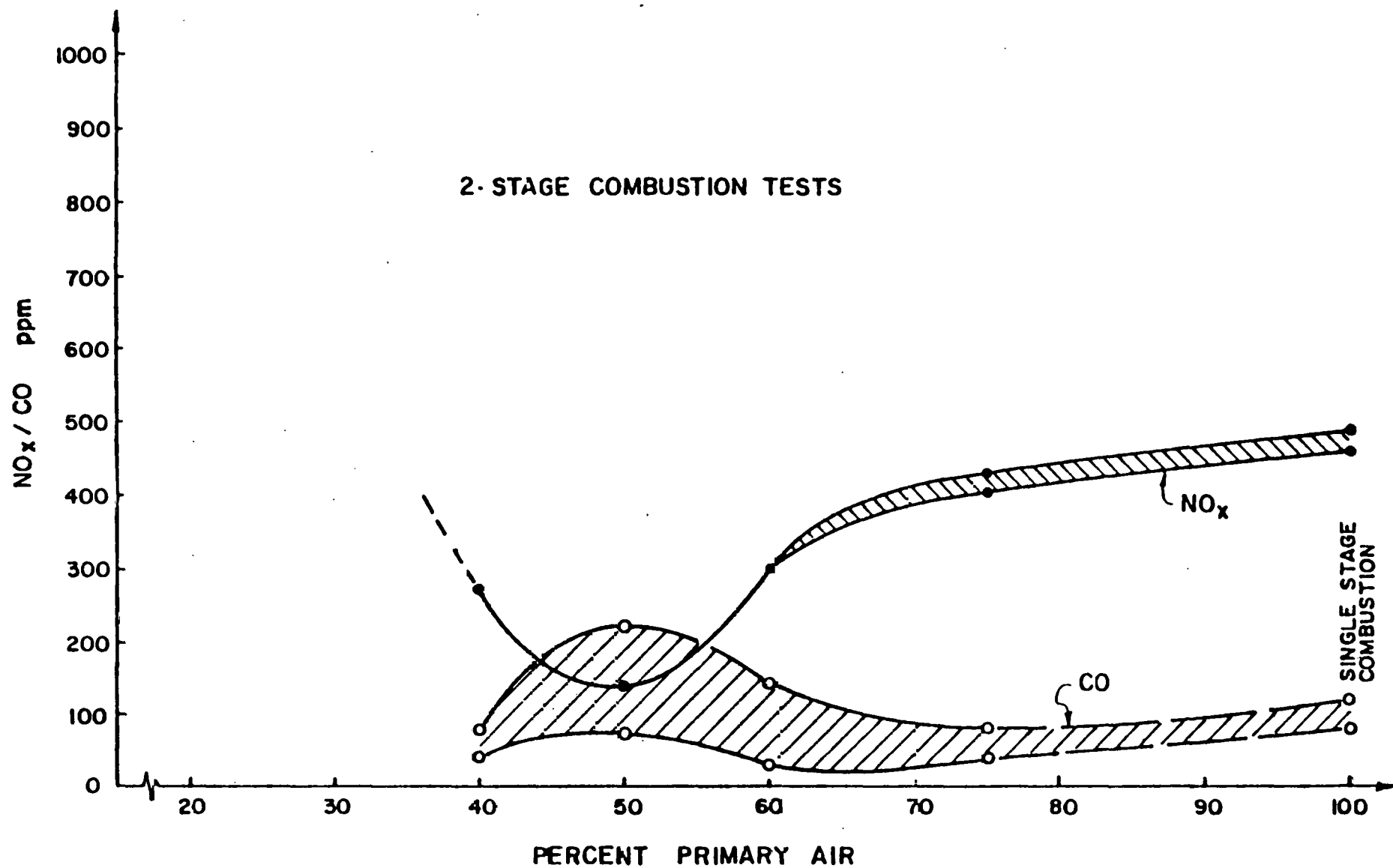


FIGURE 3. No. 6 FUEL - NO_x & CO EMISSION vs % PRIMARY AIR.
5M BTU/HR. AT \approx 12% EXCESS AIR



**FIGURE 4. GULF SRC II FUEL - NO_x & CO EMISSION vs % PRIMARY AIR
5M BTU HR AT ≈ 12% EXCESS AIR**

A P P E N D I X F

GULF RESEARCH & DEVELOPMENT COMPANY

Pittsburgh, Pennsylvania

SMALL-SCALE COMBUSTION TESTS OF SRC-II
FRACTIONS AND BLENDS

by

L. P. Walkauskas

J. E. Haebig

Chemicals and Minerals Division
Report No. 624RJ024
May 25, 1978

APPROVED BY:


R. H. Graham

DATE: May 26, 1978

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I. SUMMARY

This report presents the results of combustion tests of SRC-II liquid products produced at the Ft. Lewis, Washington, pilot plant from Western Kentucky coal. These combustion tests, together with laboratory characterization of these liquids, comprise an addendum to a DOE contract for the production of some 3000 Bbl of SRC-II. The resulting information is intended to facilitate the matching of further SRC-II production strategies to the optimum markets. Combustion emissions data were correlated with fuel properties and combustion conditions in order to clarify the fuel quality which the marketplace might require. Test equipment available at Harmarville in the Refining and Products Division and the Chemicals and Minerals Division was used to obtain data relating to residential furnaces and to industrial-size boilers. Various SRC-II distillation cuts and blends with petroleum-derived stocks, to be described presently, were selected to examine the effects of fuel properties on combustion behavior.

Our observations with a 1-gph furnace permit several conclusions regarding fuel viscosity, hydrogen and nitrogen content, and soot and NO_x emissions. Conventional "gun-type" furnaces such as the one used in this study would require an SRC-II fuel whose viscosity was within the present No. 2 oil ASTM specification, in order to satisfy the mechanical requirements for functioning of the pressure-atomizing nozzle. The SRC-II middle distillate (M.D.) and the 200-345°C cut from a blend of 2 volumes of M.D. and 1 volume of SRC-II heavy distillate (H.D.) met this viscosity requirement, but the 2/1 blend itself exceeded it. In spite of their low hydrogen contents, the M.D. and 200-345°C cuts produced no more soot than No. 2 fuel oil, probably because of the rapid air-fuel mixing provided by this common but contemporary burner, in contrast to older burner designs. The pure SRC-II fuels produced 500-600 ppm NO_x ; blending with at least 75% No. 2 fuel oil would be required to reduce NO_x emissions to a reasonable level, for example, 225 ppm, which is a federal limit on larger sources.

The 10-gph boiler which was used in this study represented some of the combustion conditions in commercial and industrial boilers. Such boilers have oil preheating means and air or steam atomization, and could burn SRC-II-containing oils extending into the No. 6 fuel oil viscosity range. In these tests, up to 25% SRC-II H.D. could be blended into low-nitrogen residual fuel oils without exceeding the limit of 225 ppm NO_x , and without increasing particulate emissions. However, important variations of NO_x emissions with burner operating conditions were found, confirming the need for low- NO_x burner design efforts. Our tests with SRC-II blends exhibited the usual decrease in NO_x emissions with decreasing excess air (approaching the conditions of two-stage combustion), supporting the effectiveness of staged combustion demonstrated by KVB's tests of the 2/1 SRC-II blend.

In our single-stage tests, decreasing excess air also caused increased carbon particulate emissions, which is the major disadvantage of staged combustion. Blends of H.D. with four different types of residual oil stocks were tested, and the nature and quantity of these particulates varied considerably with the properties of the blends. Therefore, a staged combustion test of particulate emissions from one SRC-II blend cannot be extrapolated with certainty to other blends. In contrast to utility boilers, adaptation of an industrial boiler to staged combustion could require some rebuilding, unless staging burners are developed.

In contrast, staged combustion in utility boilers has been demonstrated widely and provides the combustion conditions most likely to burn out the soot which formed during the staged combustion of SRC-II. Utility boilers are also more likely to have particulate collection systems.

Recommendations for further work include low- NO_x burner design based on droplet vaporization and combustion mechanisms, tests of water-oil emulsions to suppress smoke from staged combustion, and an engineering simulation of radiative heat transport and carbon particulate burnout in a pilot furnace.

II. INTRODUCTION

As part of the SRC-II liquid product evaluation, combustion tests to determine combustion characteristics of various blends of SRC-II heavy and middle distillates with each other and with petroleum-based fuels were undertaken. Since SRC-II liquids have lower hydrogen and higher nitrogen contents than corresponding petroleum fuels of similar boiling ranges, both soot and NO_x emissions were expected to be higher for the SRC-II fuels. One means of combating this expected increase in pollutants is to blend SRC fuels with petroleum stocks containing lower nitrogen and higher hydrogen contents. Combustion tests of the raw fuels and blends are necessary to determine to what extent the emissions problem is greater for SRC-II fuels than for conventional fuels and whether blending with conventional fuels can aid in emission reduction without significantly altering the combustion characteristics of conventional fuels.

SRC-II middle distillate (200-290°C boiling range) and a blend of middle distillate and heavy distillate boiling in the range 200-345°C (Blend X1152AOH) were tested in a small boiler equipped with a pressure atomizing burner to simulate a typical home furnace arrangement.¹ Both the middle distillate and the 200-345°C fraction are within the distillation range and viscosity specifications for No. 2 fuel oil and as such could be good fuels for residential furnaces.

SRC-II heavy distillate has a viscosity in the range of No. 5 fuel oil and may find use in commercial- and industrial-sized combustion equipment. Heavy distillate itself and blends of heavy distillate with No. 5, No. 6, a hydrotreated No. 6 oil, and a highly viscous vacuum tower bottoms sample were tested in a two-pass fire tube boiler with air atomizing nozzle to simulate industrial boilers.

The results are reported in two sections below, the first for SRC-II middle distillate and Blend X1152AOH, and the second for the heavy distillate. (See Table I for properties of blend components.)

III. COMBUSTION TESTS OF SRC-II MIDDLE DISTILLATE

A. Experimental

Figure 1 shows a schematic of the Oakmont boiler and its associated equipment. The burner is fired horizontally at 1 gallon per hour (gph) (atomizing pressure = 100 psig) into a firebrick-lined combustion chamber approximately one cubic foot in volume. Flame gases pass upward into the boiler section where they are cooled to around 500°F. Gases are sampled from the flue immediately above the boiler section. Appendix A contains a more complete description of the burner-boiler combination.

Flue gases are analyzed for CO and CO₂ (NDIR analyzers), O₂ and NO_x (chemiluminescent analyzer). An ASTM smoke tester (D 2156) is also positioned in the flue. Particulate sampling was accomplished by means of a B-P probe sampler. Further details of the gas analysis procedure and particulate sampling may be found in Appendix B.

B. Procedure

The experimental procedure for these tests is as follows:

- 1) At a constant firing rate, and at some selected air to fuel ratio (A/F), measurements of the ASTM smoke number and flue gas composition (CO₂, CO, O₂, and NO_x) are made.
- 2) The A/F ratio is varied randomly and the above-mentioned data collected. At the beginning of each run and at various times throughout the runs, a reproducibility check using No. 2 oil is made. At selected air/fuel ratios, particulate samples are collected on filters, as described in Appendix B.
- 3) Excess air (over that theoretically needed for complete combustion) is computed from the measured CO₂ concentra-

tion and the fuel composition; plots of smoke number and NO_x concentration as a function of the excess air are made. The particulate loadings in $\mu\text{g}/\text{l}$ (micrograms per liter) are entered on the relevant smoke under plots of Oakmont boiler data and also Pacific boiler data.

This procedure was followed for No. 2 oil (baseline data), SRC-II middle distillate, Blend X1152AOH, and for blends of the two SRC-II fuels with No. 2 oil. Two blends of each fuel with No. 2 oil were made: the first in the volumetric ratio 25% SRC-II to 75% No. 2 oil and the second 75% SRC-II and 25% No. 2 oil.

C. Results and Discussion

1. Particulate emissions. The results are plotted in Figures 2-6. It is apparent from Figures 2 and 3 that the slight difference in viscosity over the range of blends from pure No. 2 oil to pure SRC-II fuel (2.62-3.41 C.S. (centistokes) for middle distillate, 2.62-4.02 for X1152AOH) and the increase in C/H ratio (6.81-9.29 for middle distillate and 6.81-9.87 for X1152AOH) have very little, if any, effect on the sooting behavior of the SRC-II fuels with respect to No. 2 oil. This conclusion is supported by particulate sample results in which, under the same experimental conditions, the average particulate loading for No. 2 oil is 68 $\mu\text{g}/\text{liter}$ while that for a sample of 75% middle distillate and 25% No. 2 oil is 74 $\mu\text{g}/\text{l}$.

At no time during the combustion tests did the CO emission levels rise above 0.1% and for the majority of the cases, CO was not detectable with our analyzers.

The expectation that fuels having a higher C/H ratio produce more soot derives from experience with the home furnace designs preceding the "gun-type" burner used here, which first appeared in the 1950's. In these older designs, such as the rotary wall burner, high C/H fuel oils did produce more soot, because the air-fuel mixing which they provided was slower than that in "gun-type" burners. In other work,¹ a distillate

coal liquid (C/H 9.45) produced little more soot than No. 2 fuel oil, when a rapid air-fuel mixing gun burner was used.

Samples of the particulates collected from the Oakmont boiler with the B-P probe were examined by scanning electron microscopy (SEM). These soot samples consisted of a fairly thick layer on the fiber glass filter, and a portion of the soot layer was transferred with a micro-spatula to the SEM sample mount. This approach permitted micrography of the soot, whereas spray-mounting techniques failed. Plate 1 (magnification 300X) shows the homogeneous mass produced from No. 2 fuel oil. Plate 2 (10 000X) shows the substructure of this mass to be a porous mesh of chain-like agglomerates of subunits whose diameter is less than 0.1 micron. Combustion of a blend of 75% SRC-II M.D. and 25% No. 2 fuel oil in the Oakmont boiler produced soot (Plate 3) whose appearance was the same as that from No. 2 fuel oil.

2. NO_x emissions. The NO_x emissions observed are presented in Figures 4 and 5; the NO_x concentration (ppm) in the dried stack gas is plotted against the percent excess air. Most NO_x concentrations in this report are presented as measured and are not corrected to a single excess air basis. No. 2 fuel oil produced 65-75 ppm NO_x from oxidation of nitrogen in the combustion air ("thermal NO_x"). The NO_x concentration from the other fuels varied between 110 ppm and 550 ppm. The other fuels presumably produced nearly the same amount of thermal NO_x and the rest of the large amounts of NO_x originated from conversion to NO_x of a fraction (F) of the nitrogen chemically bound in those fuels, the remainder being converted to molecular nitrogen. NO_x so derived from the fuel is frequently termed "fuel-NO_x." This fraction was determined from these data by a computation which included subtraction of the "thermal NO_x" from the observed concentration and correction of the observed concentration for NO_x dilution by excess air. Increasing excess air caused increases in the NO_x concentration (i.e., fuel-NO_x) and the conversion fraction, due to faster oxygen admixture into the fuel spray. Figure 4 lists the nitrogen contents of the fuels (blends of MD) and also the maximum

observed values of the fuel-nitrogen conversion fractions F . These values also apply to Figure 5, blends of Fraction X1152AOH. Figure 6 also illustrates the increase in fuel nitrogen conversion with increasing excess air, by correcting for dilution of NO_x by excess air.

NO_x emissions from these fuels increased in the same order as their nitrogen contents. Pure SRC-II M.D. produced 560 ppm of NO_x (Figure 4, maximum value) at 65% excess air. Its nitrogen content was 1.10%, and 36% of that was converted to NO_x at 65% excess air. By blending the M.D. with No. 2 fuel oil, the NO_x emissions were reduced; for example, the fuel containing only 25% M.D. produced 250 ppm of NO_x . However, there is an inefficiency inherent in the blending approach, since the fraction of fuel-nitrogen converted increases regularly as it is diluted by blending, as shown by the data tabulated in Figures 4 and 5. In other words, NO_x reduction is not proportional to the reduction in nitrogen content obtained by blending SRC-II fuel with petrostocks.

NO_x emissions under combustion conditions of acceptable smoke levels (smoke number = 1.0, approximately 60% excess air) for this burner are in the range of 500-600 ppm for the pure SRC-II fuels and blends containing 75% SRC-II. The value falls off to the 200-250 ppm range for blends containing 75% No. 2 oil. These values are in excess of the 1974 federal EPA limitation for newly constructed boilers (227 ppm) for petroleum; blends of pure and 75% SRC-II fuels exceed the limitation for coal-fired boilers (500 ppm). Tests of the SRC-II M.D. and Fraction X1152AOH in a second high-efficiency furnace had been considered but would have been superfluous, since the burner having average efficiency, used here, was adequate to burn these SRC-II fuels with no more smoke than No. 2 fuel oil.

The sharp decrease in NO_x emissions with decreasing excess air levels indicates that another method of controlling NO_x emissions, staged combustion, would be effective; however, this method is practical only in industrial-sized or larger equipment.

In conclusion, SRC-II middle distillate and X1152A0H fuels produce no more smoke in this particular residential-type furnace than comparable petroleum fuels. However, NO_x emissions for pure SRC-II fuels or for blends containing mostly SRC-II are excessive at acceptable smoke levels and probably preclude use of such fuels in small units.

IV. COMBUSTION TESTS OF SRC-II HEAVY DISTILLATE

A. Experimental

Figure 7 is a picture of the Pacific steel firetube boiler used for these tests. A Cleaver-Brooks low-pressure, air atomizing gun burner (Figure 8) was installed just below the doors at the front of the boiler. A more complete description of the boiler and burner is given in Appendix A.

As in the tests with the Oakmont boiler, the burner is fired horizontally into the combustion chamber. The flame gases pass through the two banks of firetubes and exit to the stack at about 1100°F. Gases are sampled from the flue near the exit from the boiler section, as are soot emissions. Flue gases and soot emissions are analyzed as in the SRC-II M.D. combustion tests in the Oakmont boiler.

SRC-II heavy distillate (H.D.) was blended with four heavy petroleum fuels: No. 5 and No. 6 oil, a hydrotreated No. 6 oil containing 0.31% sulfur, and a sample of vacuum tower bottoms. The first three fuels were blended with the H.D. in the same 25/75 and 75/25 SRC-II/petroleum fuel ratios used with the M.D. But, because of the high viscosity of the vacuum tower bottoms, the blending ratios were 35/65 and 75/25 H.D./ vacuum tower bottoms.

The fuels were delivered directly from 55-gallon drums by pressurizing them with 6-14 psig instrument air. In this way, fuels could be heated in the drums with band heaters to obtain the proper operating conditions. The fuels passed through an insulated fuel line

to a burette (to measure fuel flow rates) and thence through a heated fuel line to the burner. The fuel delivery system was completely closed to prevent escape of fuel vapors inside the test facility. Along with the heavy distillate blends, samples of the middle distillate, a 200-345°C distillation cut (X1152AOH), a blend of 2/1 M.D./H.D. (X1152AB), and blends of the latter two fuels with No. 2 oil were tested in the larger boiler.

Figure 9 displays the results of baseline tests with No. 2 fuel oil. In addition to the excess air level, the firing rates (6 and 9 gph) and atomizing air pressures (10 and 15 psi) were found to have large but not unexpected effects on the smoke number. At lower firing rates, more excess air is required to suppress smoke; one cause of this is probably the lower velocity of the secondary air entering the flame zone through the swirl vanes, and the resulting lower turbulence and slower air-fuel mixing. The lower firing rate might also result in a lower effective flame temperature and slower soot burnout. Increased atomizing air pressure from 10 psi to 15 psi had the expected effect of diminishing soot emissions through accelerated air-fuel mixing. At a 9-gph firing rate, the end of the flame brushed the rear wall of the furnace (see Plate 6); at a 6-gph firing rate, the flame was considerably shorter (Plates 4 and 5).

These tests, together with other early tests of SRC-II fuels, revealed that there was no single "optimum" set of combustion conditions which simultaneously minimized smoke and NO_x and required low excess air. Furthermore, test results obtained at the 6-gph firing rate were as valuable as those at 9 gph because the emissions from various fuels differed more, facilitating the observation of differences between fuels; also, the parametric combustion behavior observed at 6 gph showed important differences from that at 9 gph. Thus, nearly all the fuel samples were tested at the four operating conditions represented in Figure 9. Some blends could not be tested at all conditions mentioned above because of their high viscosities; only those conditions under which a stable flame was produced were used in testing these fuels.

The results of the studies are presented below in two sections. In Section B, the light SRC-II fuels and their blends with No. 2 oil are discussed, and in Section C the heavy distillate and its blends with the heavier petrostocks. The SRC-II H.D. blends were preheated for combustion as described in Section C.

B. Tests of SRC-II M.D., Blend X1152AOH, Blend X1152AB, and H.D.

Figures 10 through 17 indicate the behavior of the various pure SRC-II fuels, including the H.D., in comparison with No. 2 fuel oil.

1. Sooting Tendency

Figure 10 displays results at a firing rate of 6 gph and atomizing air pressure (AAP) of 10 psig. Here, all of the SRC-II fuels produced more soot than did No. 2 oil. Upon increasing the atomizing air pressure to 15 psig (Figure 12), the same trend is followed but the lighter SRC-II fuels are shifted towards the No. 2 oil curve while the heavy distillate curve remains essentially constant.

At 9 gallons/hour and AAP 10 psig (Figure 14), the light SRC-II fuels and No. 2 oil have similar behavior and the two heavier fuels show a tendency towards less smoking than No. 2 oil. Upon increasing the atomizing air pressure to 15 psig (Figure 16) at 9 gph, there is very little difference in the sooting behavior of the fuels with the possible exception of the SRC-II H.D. which seems to produce less soot than the others.

Blends of two of the above fuels, X1152AOH and X1152AB (2/1 M.D./H.D.) with No. 2 oil were also examined in the Pacific boiler. Due to the small quantity of sample available, they were analyzed only at a firing rate of 6 gph. The trend in results shown in Figures 18 and 20 for both samples is as expected with the fuel blend containing 75% SRC-II fuel producing more smoke than that containing 25% SRC-II fuel.

2. NO_x Emissions

Plots of ppm NO_x (as measured) vs. % excess air are presented in Figures 11, 13, 15, and 17 for the various combustion conditions. Table II contains the fraction of fuel nitrogen converted to NO_x for each fuel and combustion condition.

At 6 gph and AAP = 10 psig (Figure 11), NO_x production from the fuels increases in the order: No. 2 < SRC-II M.D. < X1152AOH < 2/1 SRC-II M.D./SRC-II H.D. < SRC-II H.D., which follows the trend of increasing nitrogen content of the fuels. The data correspond to 17%, 16%, 19%, and 25% conversion of fuel nitrogen to NO_x for SRC-II M.D., X1152AOH, SRC-II 2/1, and SRC-II H.D., respectively.

Increasing AAP to 15 psig at 6 gph firing rate does not appreciably increase the amount of NO_x produced, as the maxima in the curves in Figure 13 are in the 220-230 ppm range, but the order is changed with No. 2 oil producing the least NO_x, the lighter SRC-II fuels about equal, and SRC-II H.D. higher at 300 ppm. Conversion of fuel nitrogen to NO_x is 17%, 17%, 16%, and 19% for SRC-II M.D., X1152AOH, X1152AB, and SRC-II H.D., respectively.

Figure 15 displays the effect of increasing the firing rate to 9 gph and AAP = 10 psig. Ranked in order of increasing NO_x production, the fuels are: No. 2 < SRC-II M.D. < X1152AOH = X1152AB < SRC-II H.D., with maximum NO_x emissions ranging from 50 ppm for No. 2 oil to the 320-360 ppm range for the light SRC-II fuels to the 500 ppm range for SRC-II H.D. Percent fuel nitrogen conversions to NO_x are 22%, 25%, 24%, and 31% for SRC-II M.D., X1152AOH, X1152AB, and SRC-II H.D.

At 9 gph and AAP = 15 psig, the trend observed for AAP = 10 psig holds but the maximum values of NO_x in Figure 17 appear to be lower, around 260 ppm for the light SRC-II oils and 360 ppm for SRC-II H.D. Percent conversions of fuel nitrogen to NO_x are also down to 22%, 17%, 16%, and 22%, respectively, for SRC-II M.D., X1152AOH, X1152AB, and SRC-II H.D., respectively.

Blends of Xl152AOH and Xl152AB with No. 2 oil (Figures 19 and 21) again show the expected trends: blends containing higher amounts of SRC-II component produce the greatest NO_x loadings. Blend Xl552A (75/25 Xl152AOH/No. 2 oil) showed an apparent trend for atomizing air pressure but, as this trend did not show up in any of the other fuels tested, we must assume it to be spurious.

3. Discussion of Low-Viscosity Fuels

Generally speaking, for a burner operating under reasonably efficient conditions, the results obtained here agree with those obtained in the Oakmont boiler for SRC-II M.D. and Xl152AOH: essentially there is little if any difference in the sooting tendencies of light (i.e., low-viscosity) SRC-II fuels and No. 2 fuel oil. In a similar combustion test of an SRC-II oil blend, KVB noticed the same trend in Bacharach smoke number for a fuel comparable to our blend of 2/1 SRC-II M.D./SRC-II H.D. and blends of this fuel with No. 2 oil.³ For a firing rate of approximately 20 gph in a boiler equipped with a pressure atomizing burner, the smoke numbers (at the same air/fuel ratios) differed very little for pure SRC-II fuel, 25/75, 75/25, and pure No. 2 fuel oil.

The H.D. differed from No. 2 fuel oil to a greater extent than the other SRC-II fuels did. At 6 gph, it produced much more smoke than did the lighter fuels; however, it appeared to produce less smoke at 9 gph. This latter result will be discussed further in Section III.C, together with smoke data from the other petroleum fuels.

Particulates collected from the Pacific boiler stack during combustion of light SRC-II fuels (e.g., Plates 7, 8, and 9) consisted of soot having the same microscopic appearance as that from the Oakmont boiler; no sparklers were visible in the flame (e.g., Plate 4). In contrast, sparklers were visible in the flame of many of the high viscosity fuels (e.g., Plate 5), including SRC-II H.D. fired at 6 gph. The particle micrographs from combustion of SRC-II H.D. (Plates 10 and 11) show some cenospheres among the soot, additional evidence for incomplete droplet vaporization and combustion in this case.

Similarly, the trends noted for NO_x emissions in the Pacific boiler parallel those found in the Oakmont boiler. Concentrations of NO_x emissions in the flue gases scaled roughly with the nitrogen content of the fuel. At a 6 gph firing rate, there is no effect due to changes in AAP. Increasing the firing rate from 6 to 9 gph slightly increased the maximum fuel-nitrogen conversion fraction observed. Increasing AAP from 10 to 15 psi tended to reduce the NO_x emission and fuel nitrogen conversion, while causing little change in the smoke emitted. This effect was most apparent in the case of SRC-II H.D. fired at 9 gph. One would expect increased atomizing air to enhance the atomization and admixture of fuel with secondary air, increasing the NO_x emissions and decreasing the soot formation. In the present case, the increasing flow of atomizing air might have increased the momentum of the jet of fuel and primary air and altered its shape, reducing the rate of admixture of secondary air, which could cause the observed effects.⁴ In general, the behavior of SRC-II H.D. discussed above may also be due to slower droplet vaporization resulting from its higher boiling range. These effects may be useful in finding improved burner design approaches and combustion conditions.

The sharp decrease in NO_x concentration with decreasing excess air parallels that found in the Oakmont boiler and indicates that staged combustion is effective in reducing NO_x emissions. The above-mentioned test by KVB has shown staged combustion to be effective in reducing NO_x emissions from the SRC-II 2/1 fuel. They also found the NO_x emissions to be dependent upon the firing rate.

C. Combustion Tests of High Viscosity Blends of SRC-II Heavy Distillate

SRC-II heavy distillate (SRC-II H.D.) was blended with four petroleum fuels: No. 5 oil, No. 6 oil, a hydrotreated No. 6 oil containing 0.31% sulfur (HDS product), and a sample of vacuum tower bottoms (VTB). The least viscous samples were tested first; means for preheating the drums of more viscous samples and the delivery tubing to the burner

were then installed for testing those samples. Table III contains the estimated viscosities of each sample at the nozzle tip during these tests. These viscosities were obtained from the temperatures of the samples at the nozzle, which in turn, were estimated from the measured temperature of the sample entering the burner drawer, and the additional heat input from the tube preheater in the burner drawer. The test procedure was otherwise similar to the procedure previously described. The smoke number and NO_x data are presented in Figures 22-38 in the following sequence: pure components, followed by blends of SRC-II H.D. with No. 5 oil, No. 6 oil, HDS product, and VTB.

1. Soot Emissions

Figures 22 through 25 are plots of smoke number vs. % excess air for the pure petroleum fuels and SRC-II H.D. The data for No. 2 oil are included for comparison.

At a 6 gph firing rate and AAP = 10 psig, SRC-II H.D. shows no more tendency towards sooting than the heavier petroleum fuels and all heavier fuels have a greater soot loading than No. 2 oil. Upon increasing the AAP to 15 psig, the difference between No. 2 oil and the No. 6, HDS product and SRC-II H.D. becomes greatest with No. 5 oil falling between the two. Pure vacuum tower bottoms were not tested due to the high viscosity of the oil.

At a 9 gph firing rate and AAP = 10 psig, HDS product and No. 2 oil show similar sooting tendencies, while SRC-II H.D. makes significantly less smoke. This trend is also apparent at AAP = 15 psig where HDS product smokes slightly more than No. 2; No. 6 oil, No. 5, and SRC-II H.D. produce even less smoke.

Thus, SRC-II H.D. produces no more smoke than the heavy petroleum fuels at a firing rate of 6 gph. At 9 gph, it produces less smoke in our test boiler. This is consistent with the smoke yields from No. 2 fuel oils reported above.

The main difference between the combustion behavior of the high viscosity oils and the low viscosity oils (Section III.B) is the cenospheres produced by the former. Plates 12, 13 and 14 are typical examples of the particulates sampling filters used in these tests. The filter in Plate 11 was taken when the smoke number was much higher than in the case of Plates 12 and 13, and the resulting higher ratio of background soot to cenospheres can be qualitatively seen in Plate 14. Plate 15 shows the detailed typical appearance of the cenospheres, and there is also visible a non-perforated sphere which is probably a fused ash particle. Plate 5 is typical of the "sparklers" visible in the flames of the most viscous blends (75% No. 6 oil or VTB). However, the concentration of the cenospheres in the flames was much lower in the tests of the other blends.

Blends of SRC-II H.D. with No. 5 oil (Figures 26-29) follow the expected trends at 6 gph. At AAP = 10 psig there is no apparent difference in sooting tendency among No. 5 oil, SRC-II H.D., and the two blends, corresponding to the trend seen for the two pure fuels. There is a slight separation at AAP = 15 psig following the tendency of SRC-II H.D. produce more smoke than No. 5 oil under these conditions.

At 9 gph and AAP = 10 psig, the sample containing 25% H.D. makes slightly more smoke than the 75% and pure H.D.; at AAP = 15 psig, SRC-II H.D., No. 5 oil, and 25/75 H.D./No. 5 are equal in sooting tendency, while the blend of 75/25 SRC-II H.D./No. 5 makes less smoke.

Blends of SRC-II H.D. with No. 6 oil (Figures 30-31) were fired only at 6 gph. Within the scatter of the data, which is fairly large, smoking tendencies are similar with the exception of the 25/75 blend which apparently makes significantly less smoke at 6 gph (AAP = 15 psig) than do the others. However, the presence of only two data points in the steeply sloping portion of the smoke curve for this blend precludes concluding that this behavior is real.

Blends of SRC-II H.D. (Figures 32-35) with HDS product again follow the expected trends at 6 and 9 gph, with the similar sooting tendencies (within experimental error), reflecting the similarities between SRC-II H.D. and HDS product.

Blends of SRC-II H.D. (Figures 36-37) with vacuum tower bottoms were also fired only at 6 gph due to their high viscosity. The trends shown here are again the expected ones with pure SRC-II H.D. and the 75/25 H.D./VTB blend showing similar sooting tendencies and the blend of 25% H.D. fractionally less. Overall, the SRC-II H.D. makes only slightly more soot at 6 gph than do the petroleum fuels and possibly makes significantly less at a firing rate of 9 gph. In general, the smoke emissions from the set of blends of each petroleum oil stock varied regularly with blend composition.

It would be desirable to relate the observed amounts of particulate emissions from the various petroleum oils and blends to their composition and properties, such as viscosity at atomization, boiling range, and asphaltene content. Others^{5,6} have demonstrated a correlation between asphaltene content and carbon particulate emissions, which seems to have a larger effect than the C/H ratio. Our data do not contradict such a relation, but it is obscured by the variation of other properties of the fuels simultaneously with their asphaltene content; size distribution data would also be needed to distinguish between soot and cenospheres. The petroleum residual oils and blends produced a smoke number which tended not to decrease to zero, even when the excess air increased greatly; this effect was probably due to the non-volatile components in the residual oil. Earlier tests¹ of coal liquids with and without non-volatile components produced a similar result, demonstrating another similarity between the combustion behavior of coal liquids and petroleum fuel oils.

2. NO_x Emissions

Figures 39 through 41 are plots of ppm NO_x (measured with the chemiluminescent analyzer) vs. % excess air for the pure petroleum fuels and SRC-II H.D. Data for No. 2 oil are also included for comparison. The fraction of fuel nitrogen converted to NO_x was also determined, and its values are presented in Table II. For all combustion conditions, NO_x loading in the flue gases correlated well with fuel nitrogen content. Thus, in increasing order of NO_x emissions the fuels are No. 2 oil < HDS product < No. 5 oil < No. 6 oil < SRC-II H.D. corresponding to fuel nitrogen contents of 0.01, 0.13, 0.31, 0.44, and 1.43%, respectively. As noted with lighter SRC-II fuels, increasing firing rate increases NO_x production, while increasing AAP results in a slight decrease in NO_x emissions. Also, NO_x emissions for pure SRC-II H.D. are again higher than the EPA limit of 250 ppm, but the sharp decrease in NO_x with decreasing excess air indicates that staged combustion should be effective in reducing NO_x emissions.

Figures 42 through 45 concern blends of SRC-II H.D. with No. 5 oil. The expected trends are noted for all combustion conditions. NO_x production increases with increasing fuel nitrogen content of the blends (shown in parentheses); No. 2 Oil (0.01) < No. 5 (0.31) < 25/75 H.D./No. 5 (0.60) < 75/25 H.D./No. 5 (1.19) < SRC-II H.D. (1.43). The blend of 25/75 H.D./No. 5 meets EPA requirements at all firing rates, but the blend of 75/25 H.D./No. 5 does not. Again, increasing firing rate increases NO_x production while increasing AAP results in a slight decrease in NO_x.

Figures 46 and 47 show the same trends for blends of SRC-II H.D. although they are not as pronounced as with No. 5 oil. At 6 gph AAP = 15 psig, all blends fall within the EPA limit for new boilers.

Data for blends of SRC-II H.D. with HDS product are presented in Figures 48 through 51. The above-mentioned trends also hold here, with fuel nitrogen content determining the relative amounts of NO_x emissions with decreasing excess air.

Finally, in Figures 52 through 54 are the data for blends of SRC-II H.D. with vacuum tower bottoms. Again, all of the above trends are followed for this set of fuels.

In general, the NO_x concentrations and fuel-nitrogen conversion fractions of the blends of petroleum residual oils varied with the combustion conditions and fuel nitrogen content in a manner similar to the dependencies shown by the samples containing only distillate material (Section III.B). NO_x emissions for pure SRC-II H.D. exceed the EPA limit of 250 ppm but blends of H.D. with petroleum-based fuels can reduce the NO_x loading of the flue gases. A sharp decrease in NO_x loading with decreasing excess air is noted for all fuels, indicating that staged combustion is a viable means of reducing NO_x emissions. The effects of firing rate and atomizing air pressure have also been examined. Increasing firing rate produces an increase in NO_x emissions, while increasing the AAP results in a slight decrease in NO_x . Although there have been predictions that less volatile fuels (such as SRC-II H.D.) would exhibit lower fractional fuel-nitrogen conversion (F), in our tests F for the H.D. was the same as (or slightly higher than) F for the M.D.

V. CONCLUSIONS

The following conclusions result from these tests:

(1) In the Oakmont 1-gph boiler, with a conventional moderately fast air-fuel mixing nozzle, SRC-II M.D. produced the same amount of smoke as No. 2 fuel oil. Fifty percent excess air was required to limit the ASTM smoke number to 1 (acceptable for residential furnaces), and 25% excess air was required to limit the smoke number to 8, an acceptable emission level from an industrial furnace.

(2) In the Oakmont boiler, at permissible smoke levels (25-50% excess air), combustion of M.D. produced 400-500 ppm NO_x , representing conversion of 35% of the chemically bound fuel nitrogen to NO_x .

(3) Lower NO_x concentrations were produced from blends of SRC-II M.D. with No. 2 fuel oil, but the reduction in NO_x was somewhat less than the reduction in the nitrogen content of the blends. This is because a greater fraction of the fuel nitrogen in the diluted blends was converted to NO_x (e.g., 56% of the nitrogen in the 25% M.D. blend).

(4) The 200-345°C distillation cut from SRC-II produced the same smoke and NO_x emissions as the SRC-II M.D. (200-290°C boiling range).

(5) The NO_x emissions from SRC-II liquids decreased rapidly with decreasing excess air, since more fuel nitrogen was being converted to N_2 and less was being converted to NO_x . This suggests that staged combustion in large boilers should effectively suppress NO_x emissions from the nitrogen in these SRC-II liquids.

(6) Several operating conditions were used with the 10-gph Pacific boiler and Cleaver-Brooks atomizing air burner; this unit tended to smoke more than the Oakmont boiler. No. 2 fuel oil, when burned at 50% excess air, produced a smoke number of 7 at a 6-gph firing rate and 10 psig atomizing air pressure (the smokiest operating conditions); at 9 gph and 15 psig (the cleanest operating conditions), the smoke number was 3.

(7) When the SRC-II M.D. was burned in the Pacific boiler, it produced only 220-350 ppm of NO_x , depending on the operating conditions; this corresponded to conversion of 17-22% of the fuel nitrogen to NO_x . When M.D. was burned in the Pacific boiler, it produced less NO_x and more soot, relative to the Oakmont boiler. These effects are both consistent with the slower admixture of combustion air into the fuel spray which probably existed in the Pacific boiler.

(8) Lower NO_x emissions resulted from blends of SRC-II H.D. with any of the four residual petroleum oil stocks used. Levels acceptable to EPA (e.g., 225 ppm) were met by blends containing 25% H.D.

(9) NO_x emissions underwent very marked decreases with decreasing excess air, indicating the probable effectiveness of staged combustion in suppressing NO_x from these fuels. However, the resulting copious particulate emissions varied greatly with fuel properties and should be assessed for each prospective application of fuel and equipment.

(10) The 2/1 SRC-II blend produced little more smoke than M.D. Micrographs showed it to be soot; in contrast, pure H.D. and its blends with residual oils produced some cenospheres.

(11) At the less efficient (smoky) burner operating conditions in the Pacific boiler, the smoking tendency of the residual oil blends increased with their viscosity. The smoke emissions seemed to be related only to the viscosity of the H.D., but not to its lower hydrogen content, making it a favorable blending stock for residual oil. Further tests would be needed to separately characterize the effects of residual oil asphaltene content and atomization viscosity on soot and cenosphere emissions.

(12) Under the more efficient burner conditions, the relative smoking tendency of H.D. was reversed in some cases and NO_x emissions were considerably reduced. This suggests that low- NO_x burner design may be feasible.

VI. RECOMMENDATIONS

(1) In the case of staged combustion, carbon particulate formation from coal liquids differs from that of petroleum fuel oils. The burnout of these particulates needs to be quantified and could be done advantageously in a correctly sized pilot furnace. Such particulates also greatly alter the radiative heat transport to the furnace waterwalls of a boiler, and the gas temperature distribution; a pilot furnace test would quantify this effect.

(2) The emulsification of a few percent water into fuel oils reduces smoke. We recommend a test of water emulsification as a means to reduce the amount of soot formed in the staged combustion of SRC-II. The test would determine the extent of the reduction of fuel-rich eddies, in which soot forms.

(3) The observed effects of altered air atomization pressure on NO_x show the potential for development of burner designs for minimum NO_x and smoke. The further design of such burners to accomplish a staged combustion without furnace rebuilding would greatly facilitate the use of SRC-II in the industrial boiler market sector, were a high cost alternative liquid fuel would be most acceptable.

(4) The effects of the properties of residual oils need further parametric testing, especially the effects of asphaltene content and oil atomization viscosity or temperature.

JEH:LPW:WPC(#1213)

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2. (a) Turner, D. W., and C. W. Siegmund, "Staged Combustion and Flue Gas Recycle: Potential for Minimizing NO_x from Oil Combustion," presented at AFRC/EPA, "American Flame Days," Chicago, IL, September 1972.

(b) Quinlan, C. W., and C. W. Siegmund, ACS Preprints, Div. of Fuel Chemistry, Vol. 23, No. 1, p. 139, paper presented at Anaheim, CA, March 1978.
3. Muzio, L. J., J. K. Arand, *ibid.*, p. 140.
4. Chigier, N. A., ACS Preprints, Div. of Petroleum Chem., Vol. 21, No. 4, p. 616, September 1976.
5. Goldstein, H. L., and C. W. Siegmund, Envir. Sci. Tech. **10**, (12), p. 1109, 1976.
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Table I

SRC-II BLENDS COMBUSTION TESTS - PROPERTIES OF COMPONENTS

	Dist. D86 10% Point °C/ 90% Point °C	% H	% C	% N Total	Viscosity cs. 100°F	Asphaltenes IP 143	Carbon Residue (con.)
<u>SRC-II Fractions</u>							
Middle Distillate (MD)	208/260	9.29	86.34	1.10	3.41	0.04	1.1
Heavy Distillate (HD)	307/431	7.50	89.89	1.43	90.15	2.69	1.5
67% MD + 33% HD (2/1)	209/368	8.47	86.62	1.21	6.89	.35	3.0 ^a
200-345°C (X1152AOH)	209/293	8.81	86.98	1.11	4.02	-	.25 ^a
<u>Petroleum Oils</u>							
No. 2	217/311	13.04	86.96	0.01	2.55	-	
No. 5	- / -	11.72	88.28	0.29	45.45	4.67	7.0
No. 6	- / -	10.97	89.03	0.44	35 ^b	0.70	7.5
HDS No. 6	- / -	12.49	87.51	0.13	90	3.09	3.13
Vac. Tower Btm (VTB)	- / -	11.19	88.81	0.52	106 ^b	0.19	7.03

^aon 10% bottoms.^bat 210°F.

JEH:qrn
C&MD
GS&TC
5/23/78

Table II

PERCENT OF FUEL NITROGEN CONVERTED TO NO_x

Fuel	Firing rate, gph Atomizing Air, psi	6	6	9	9
		<u>10</u>	<u>15</u>	<u>10</u>	<u>15</u>
#2 Oil		-	-	-	-
SRC-II MD		17	17	22	22
2/1 MD/HD		19	16	24	16
Dist. 2/1		16	17	25	17
75/25 MD/#2		-	43	-	-
25/75 MD/#2		-	-	-	-
75/25 2/1/#2		21	19	-	-
25/75 2/1/#2		24	26	-	-
75/25 Dist/#2		15	21	-	-
25/75 Dist/#2		28	26	-	-
#5 Oil		30	32	-	43
SRC-II HD		25	19	31	22
75/25 HD/#5		23	20	< 40	16
25/75 HD/#5		32	32	38	32
#6 Oil		-	31	-	34
SRC-II HD		25	19	31	22
75/25 HD/#6		24	20	-	-
25/75 HD/#6		-	33	-	-
HDS Product		45	36	45	45
SRC-II HD		25	19	31	22
75/25 HD/HDS		19	14	26	18
25/75 HD/HDS		32	25	42	30
SRC-II HD		25	19	31	22
75/25 HD/VTB		21	18	-	18
35/65 HD/VTB		-	16	-	-

JEH:WPC
C&MD
GS&TC
5/23/78

Table III

ESTIMATED SAMPLE VISCOSITIES AT NOZZLE, cs

<u>Fuel Sample</u>	<u>Firing Rate</u>	
	<u>6 gph</u>	<u>9 gph</u>
No. 2 oil	3.0	3.0
No. 5 oil	100	100
No. 6 oil	95	200
VTB	—	—
SRC-II MD	4.5	4.5
75/25 MD/No. 2	3.8	3.8
25/75 MD/No. 2	3.3	3.3
SRC-II HD	17	30
2/1 MD/HD	12	12
200-345°C	6	6
75/25 200-345°C/No. 2	5.0	5.0
25/75 200-345°C/No. 2	3.7	3.7
75/25 2/1/No. 2	6.8	6.8
25/75 2/1/No. 2	4.0	4.0
75/25 HD/No. 5	18	22
25/75 HD/No. 5	12.5	18
75/25 HD/No. 6	18	30
25/75 HD/No. 6	57	110
75/25 HD/0.3%S HDS	13.5	21
25/75 HD/0.3%S HDS	11	16.5
75/25 HD/VTB	18	30
35/65 HD/VTB	55	140
0.3%S HDS Product	17	75
345°C+ Residue	17	—

LPW:WPC
C&MD
GS&TC
5/23/78

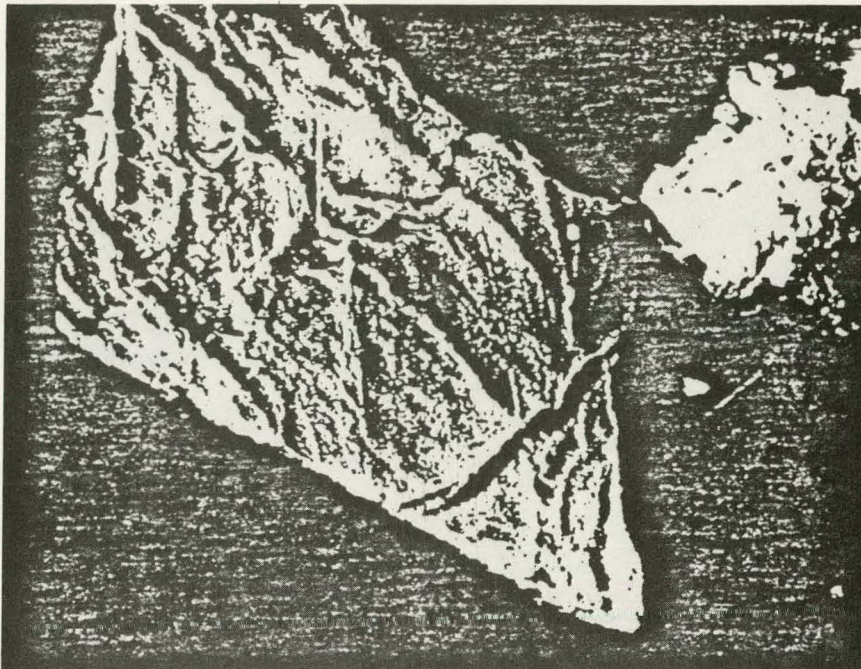


Plate 1. Particulate from Oakmont Boiler,
300X

Fuel - No. 2 Oil

Smoke No. - 9.8

Excess Air - 26%

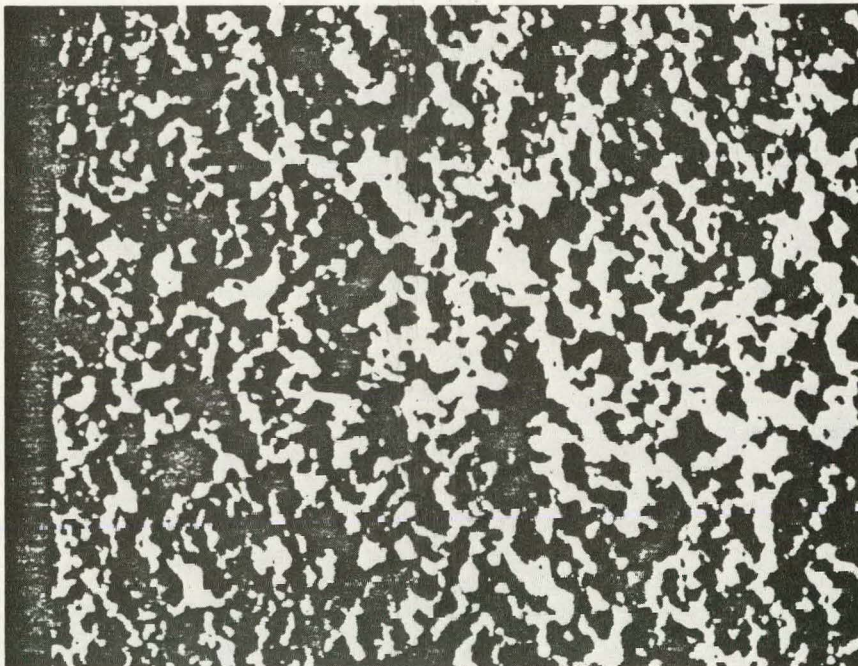


Plate 2. Same object as Plate 1,
10,000X

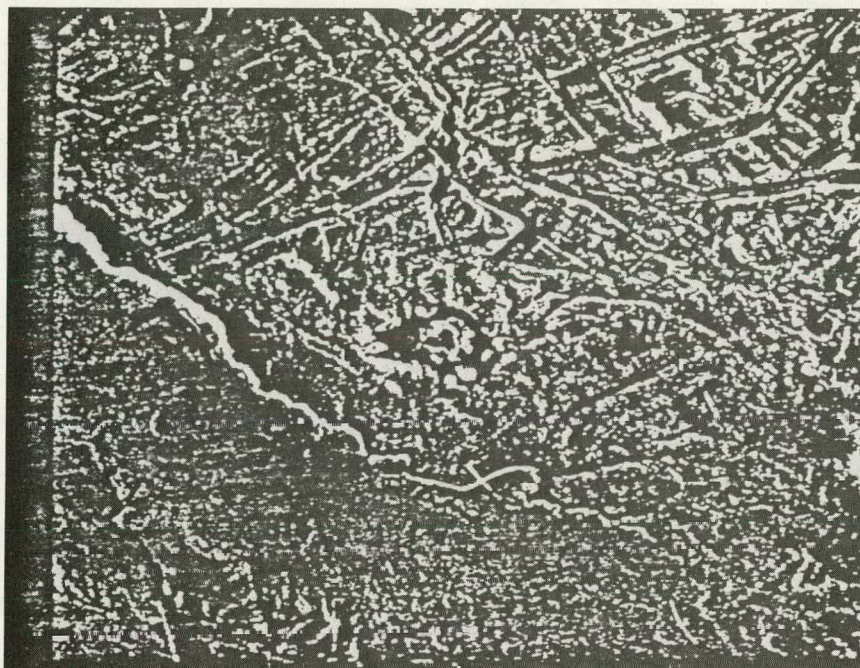


Plate 3. Particulate from Oakmont Boiler,
100X

Fuel = 75% SRC II MD, 25% No. 2 Oil

Smoke No. = 9.5

Excess Air = 24%

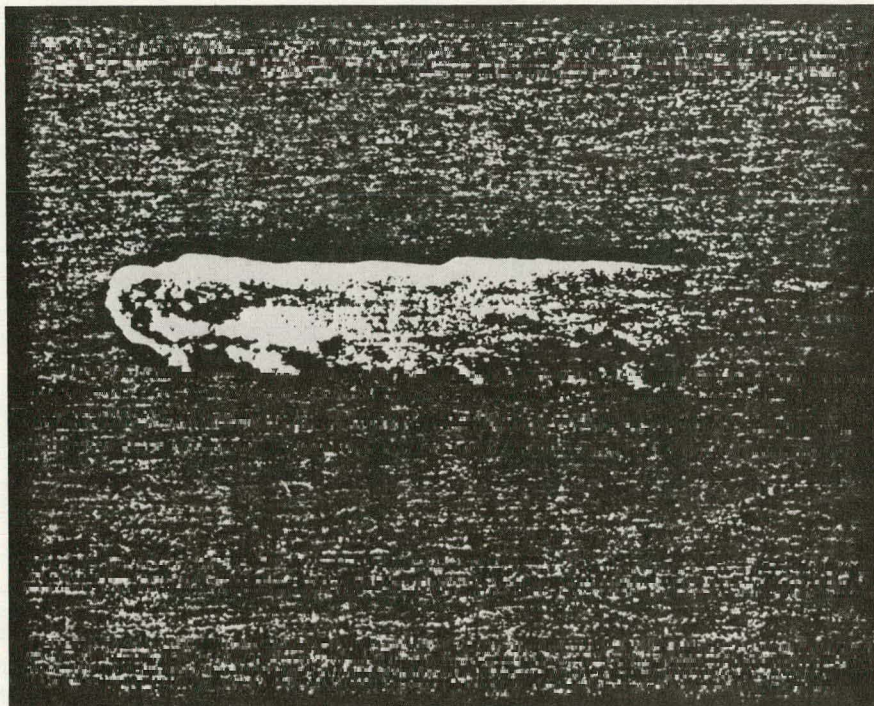


Plate 4. Flame in Pacific Boiler. View through 10" high horizontal slot in side wall of boiler. The root of the flame at the burner diffuser is visible at right hand side of photograph. Flame length is approximately 3:5'.

Fuel - SRC II Middle Distillate
6 GPH, 10 PSI Atomizing Pressure
Polacolor 2 film, f/22, 1/200 sec exposure

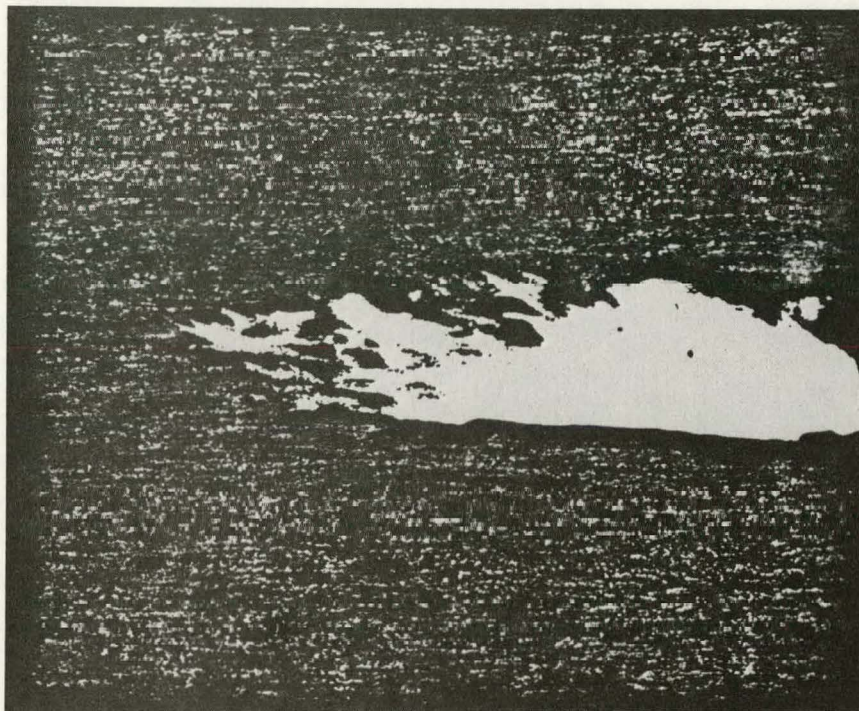


Plate 5. Same as Plate 4 except:

Fuel = No. 5 Fuel Oil

Aperature = f/8

"Sparklers" are visible following end of flame
at left side.

(Poor atomization conditions (no oil preheat, 6 gph, AAP = 10 psig) were intentionally used for this photograph, which also represents sparklers in tests of the most viscous blends.)

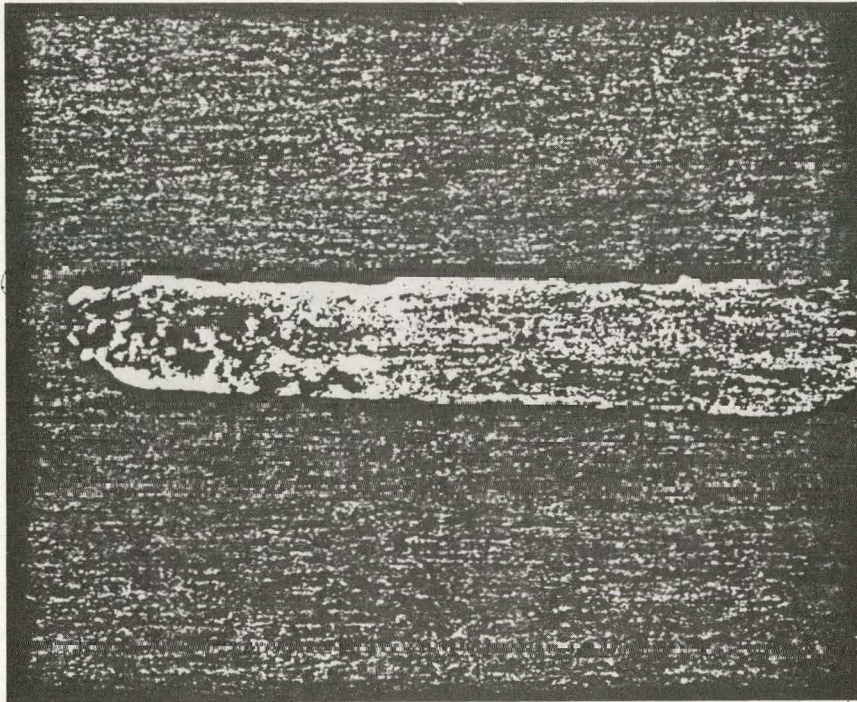


Plate 6. Same as Plate 4 except:

9 GPH firing rate, $f/45$

Flame length at least 4', impinging slightly on
rear wall, to the left out of the camera field
of view.

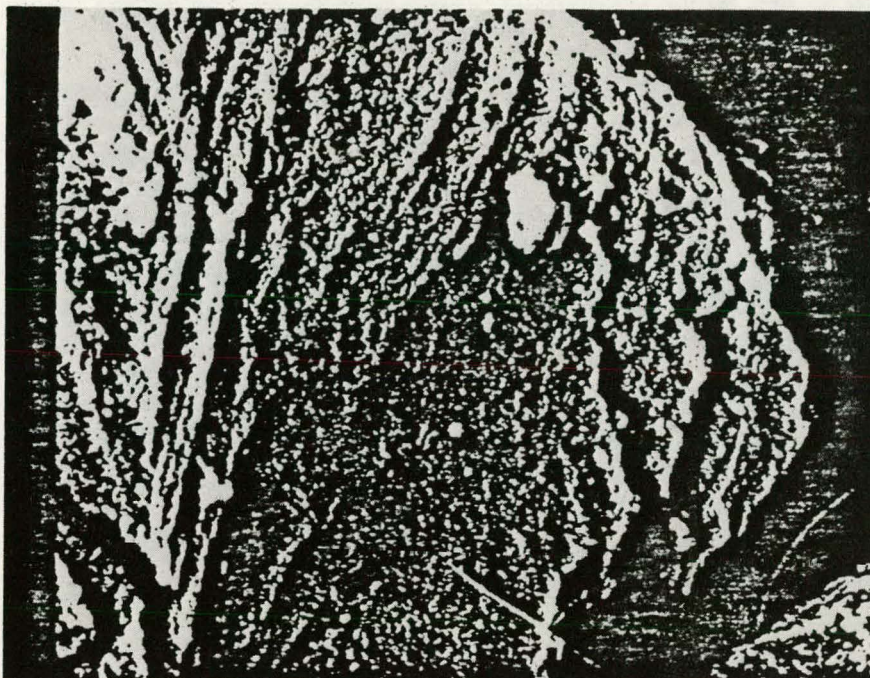


Plate 7. Particulate from Pacific Boiler,
300X

Fuel = SRC II 200-345°C

Smoke Number = 4.8

Excess Air = 50%

6 GPH, 15 PSI Atomizing Air P

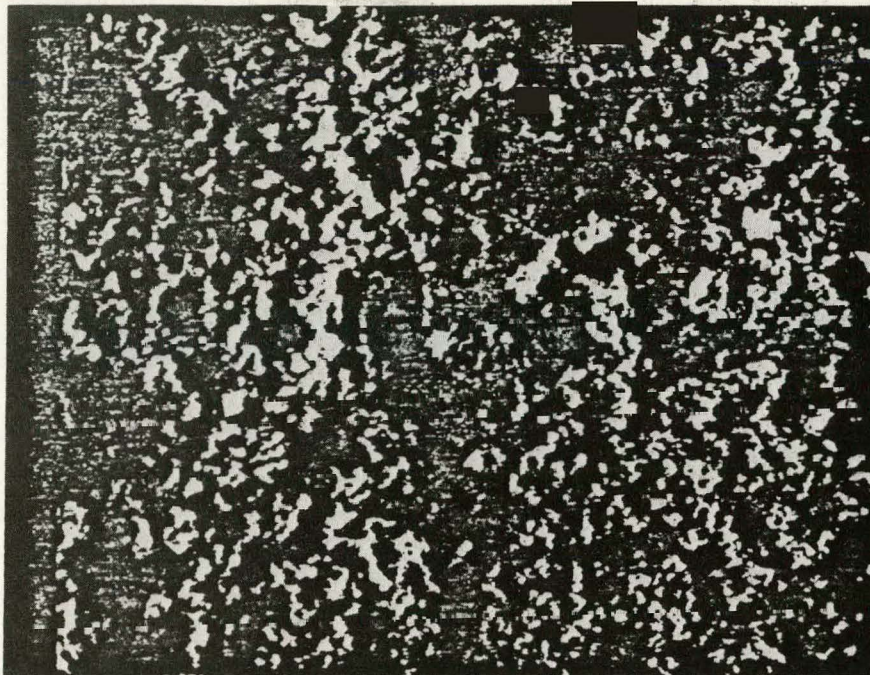


Plate 8. Same object as Plate 7, 10,000X

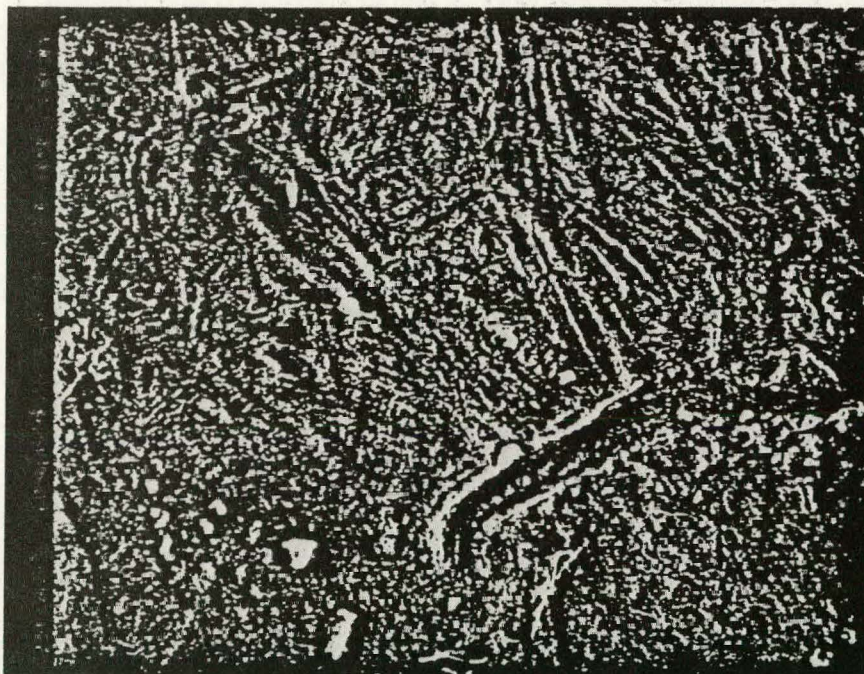


Plate 9. Particulate from Pacific Boiler,
100X

Fuel = 2/1 SRC II MD/HD

Smoke Number = 9.8 Excess Air = 23%

9 GPH, 16 PSI Atomizing Air P

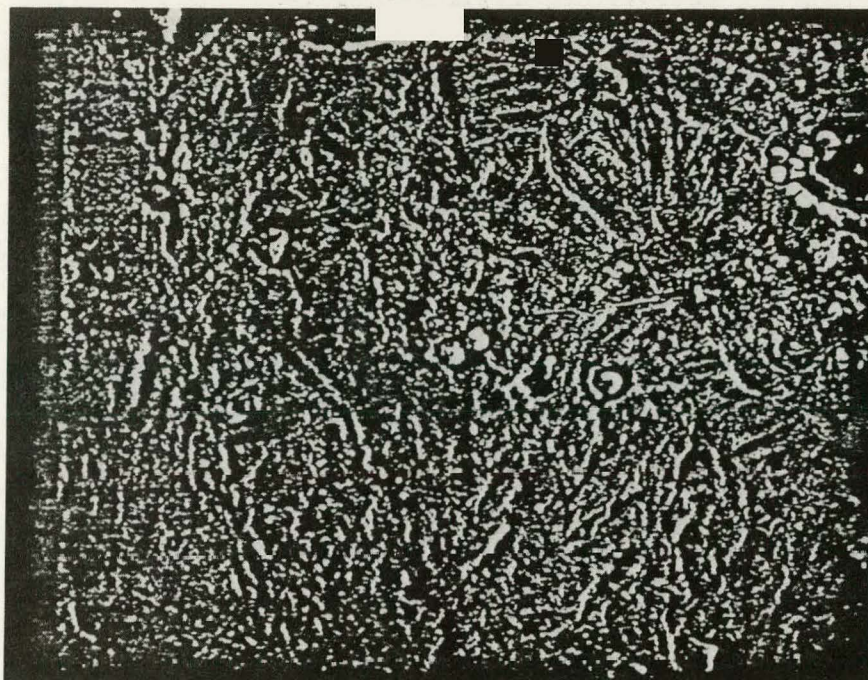


Plate 10. Particulate from Pacific Boiler,
100X

Fuel = SRC II HD

Smoke Number = 9.8

Excess Air = 19%

9 PGH, 15 PSI Atomizing Air P

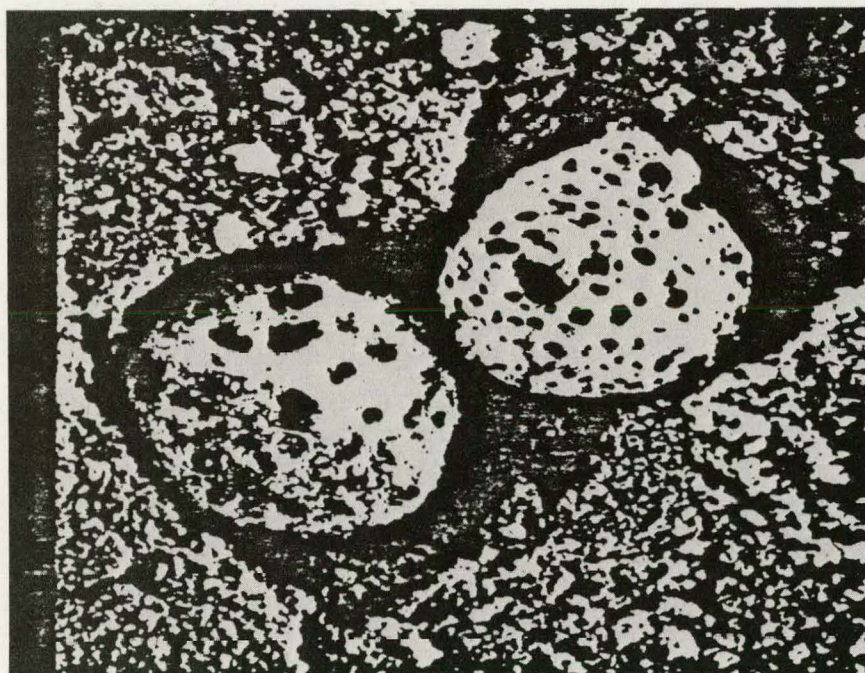


Plate 11. Same object as Plate 7, 1,000X

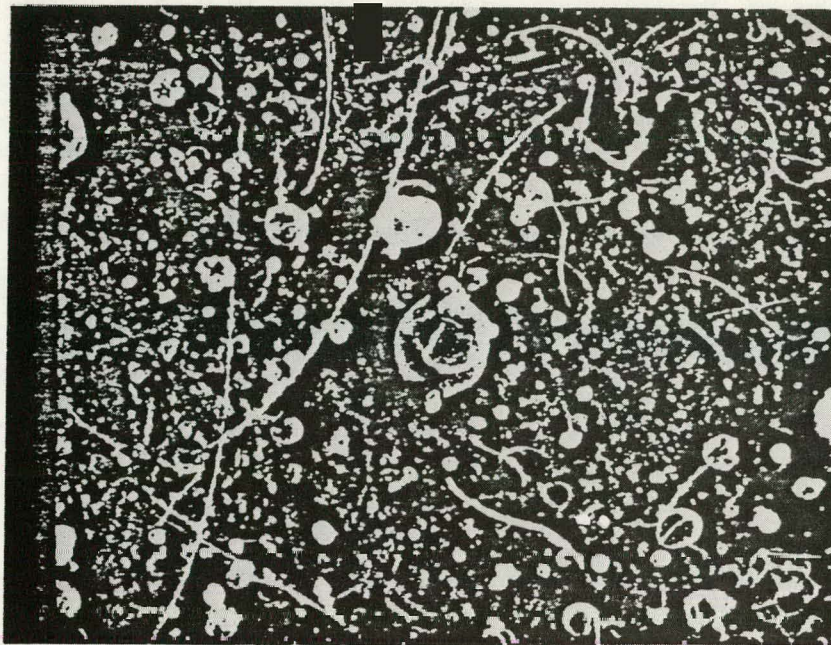


Plate 12. Particulate from Pacific Boiler,
100X

Fuel = 25% SRC II HD, 75% No. 6 Oil

Smoke Number = 5.9

Excess Air = 51%

6 GPH, 15 PSI Atomizing Air P

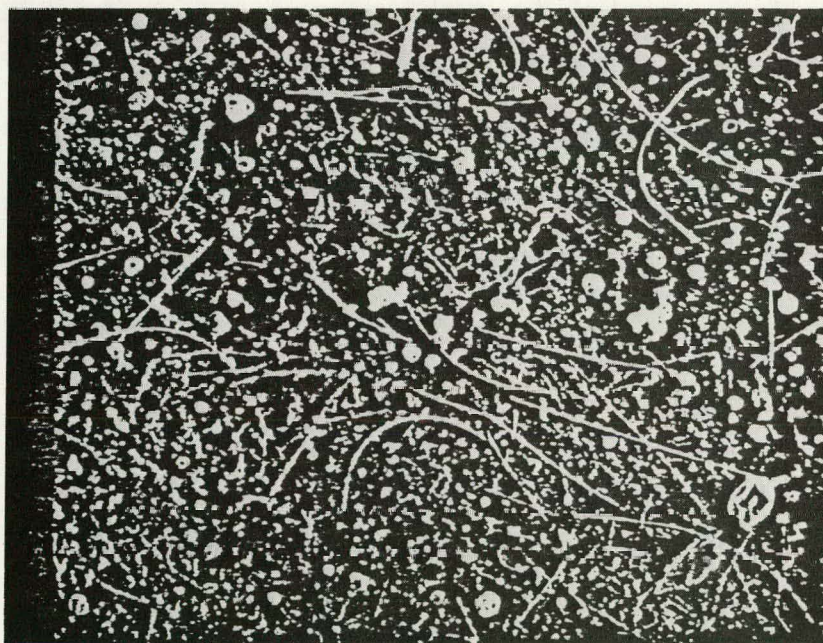


Plate 13. Particulate from Pacific Boiler
100X

Fuel = HDS Product

Smoke Number = 7.0

Excess Air = 68%

6 GPH, 15 PSI Atomizing Air P

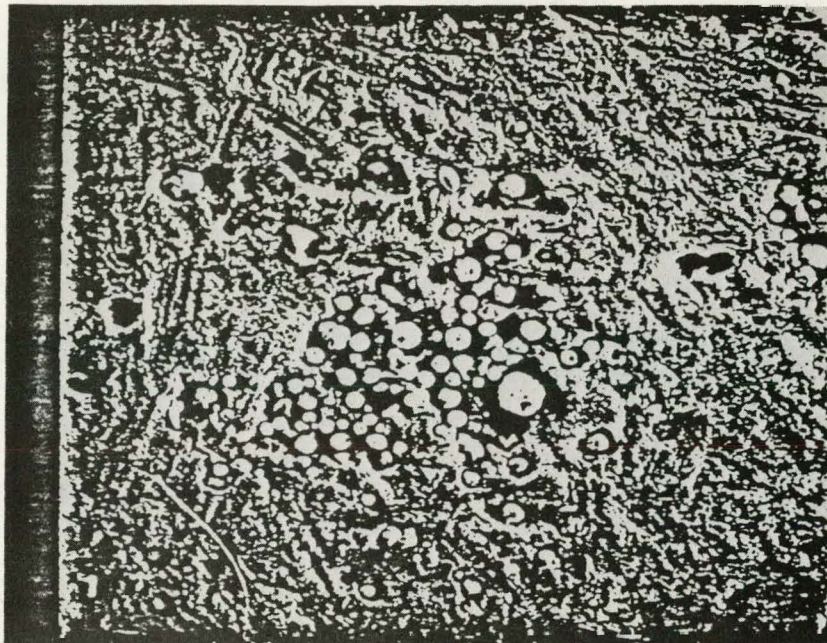


Plate 14. Particulate from Pacific Boiler,
100X

Fuel - HDS Product

Smoke Number = 9.9 Excess Air = 56%
6 GPH, 10 PSI Atomizing Air P

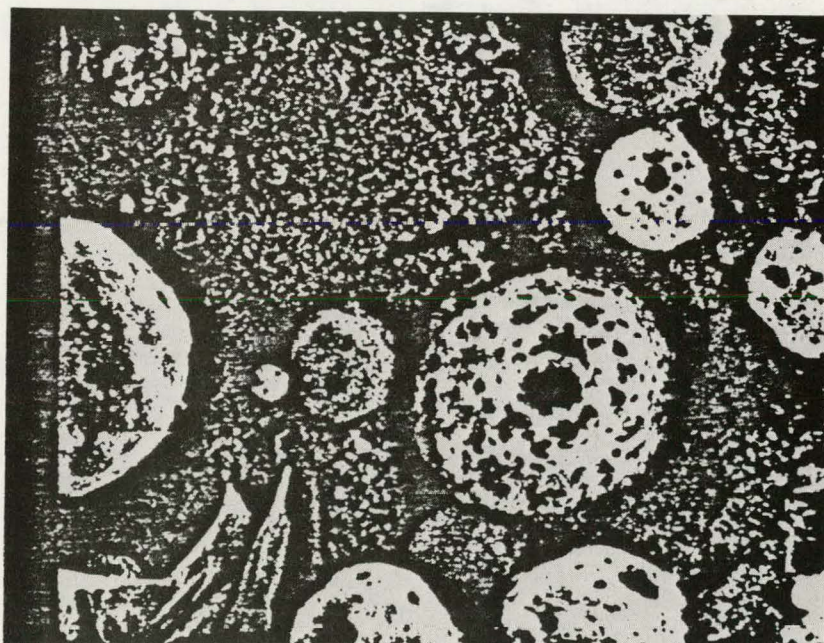


Plate 15. Same object as Plate 11.

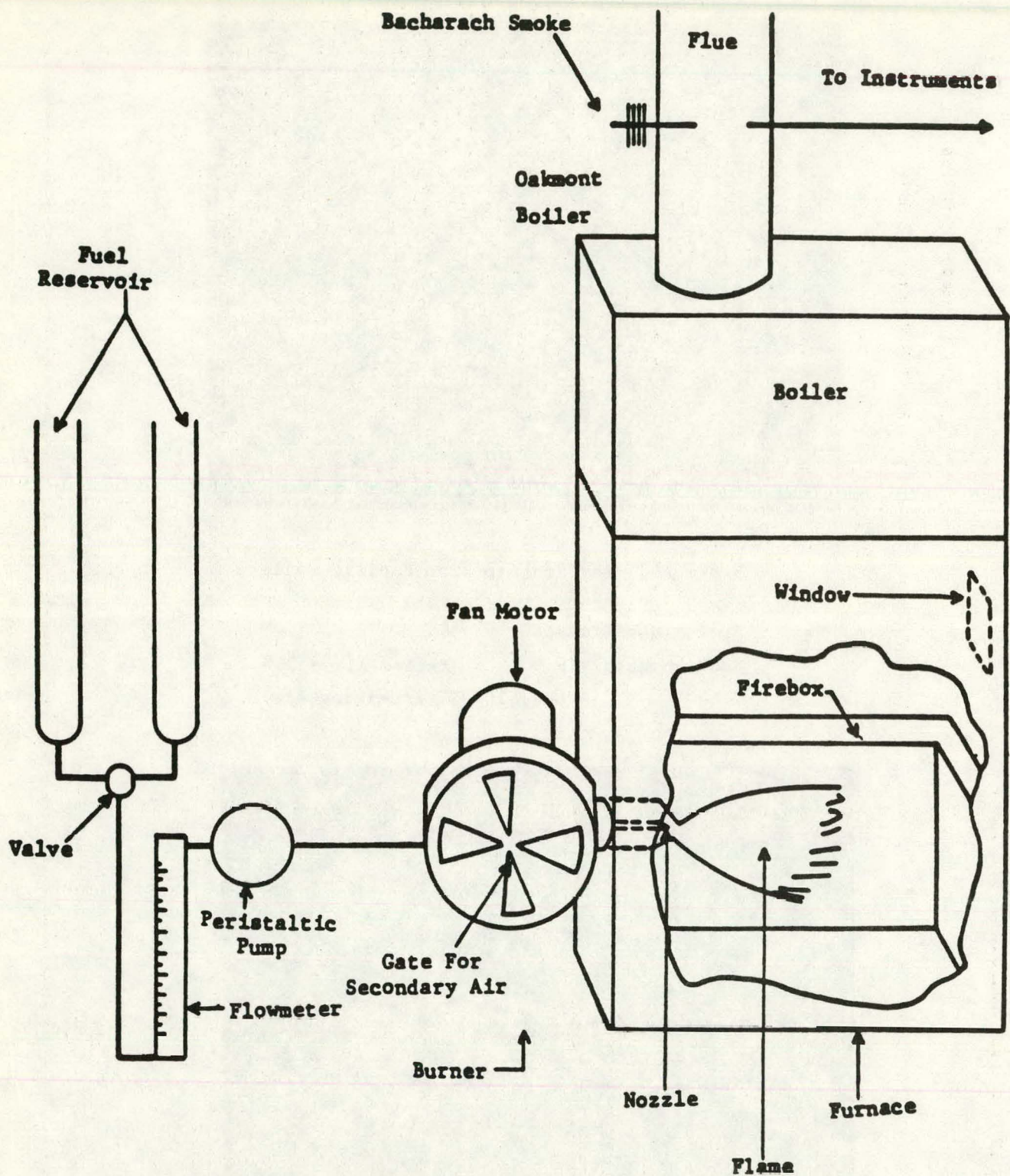
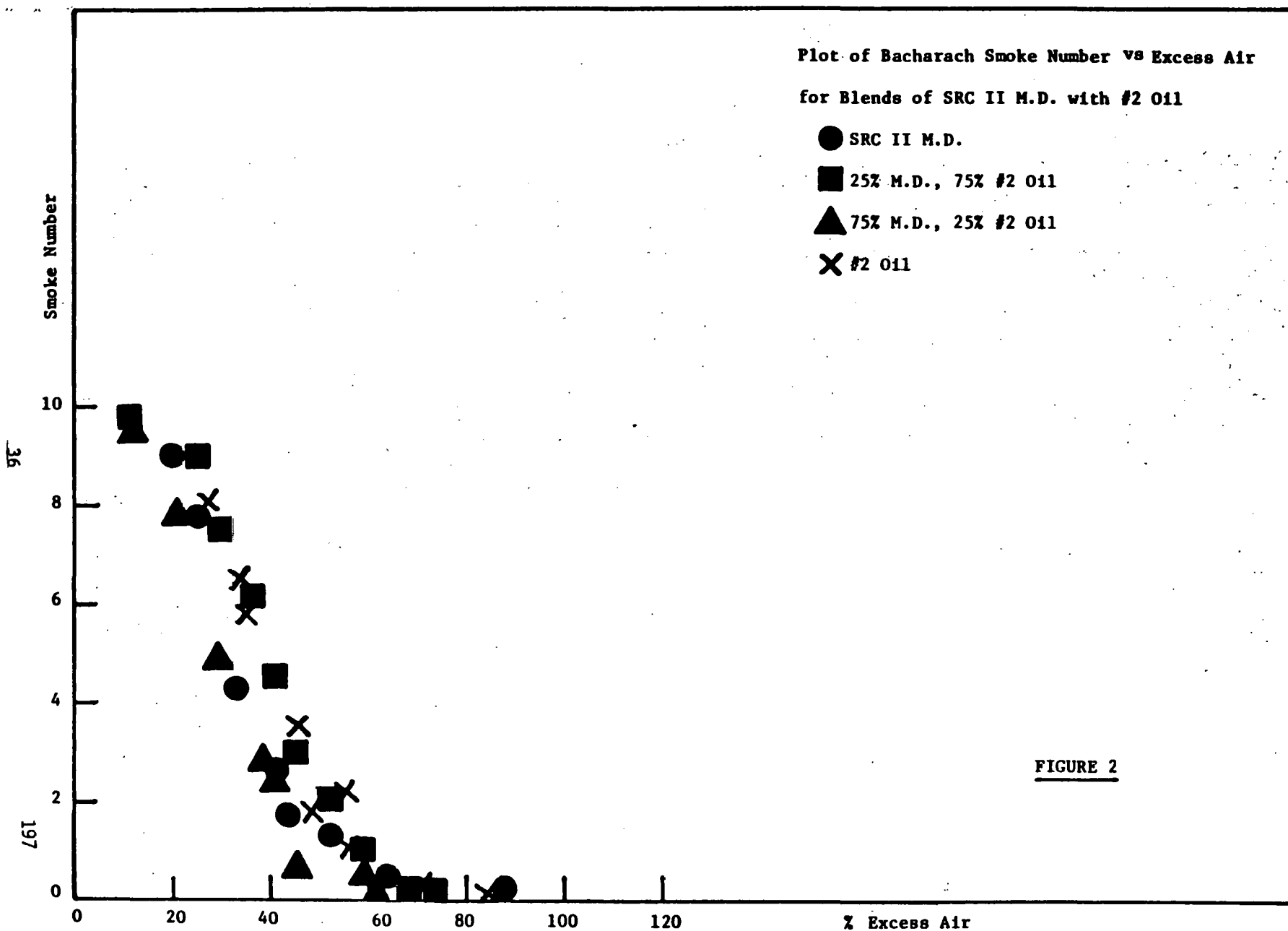
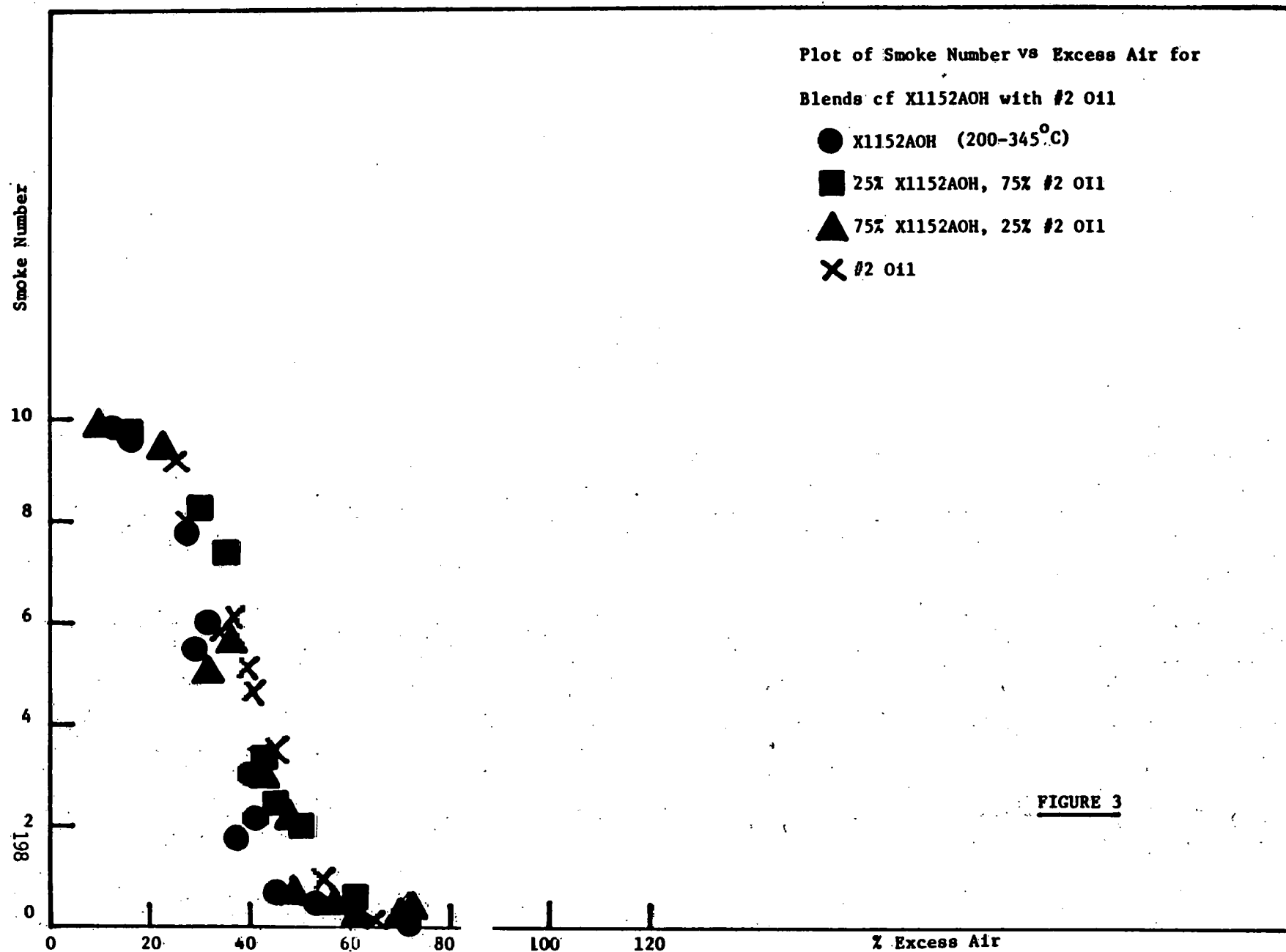


Figure 1
 "OAKMONT" RESIDENTIAL-SIZE FIREBOX
 BOILER, ASSOCIATED EQUIPMENT





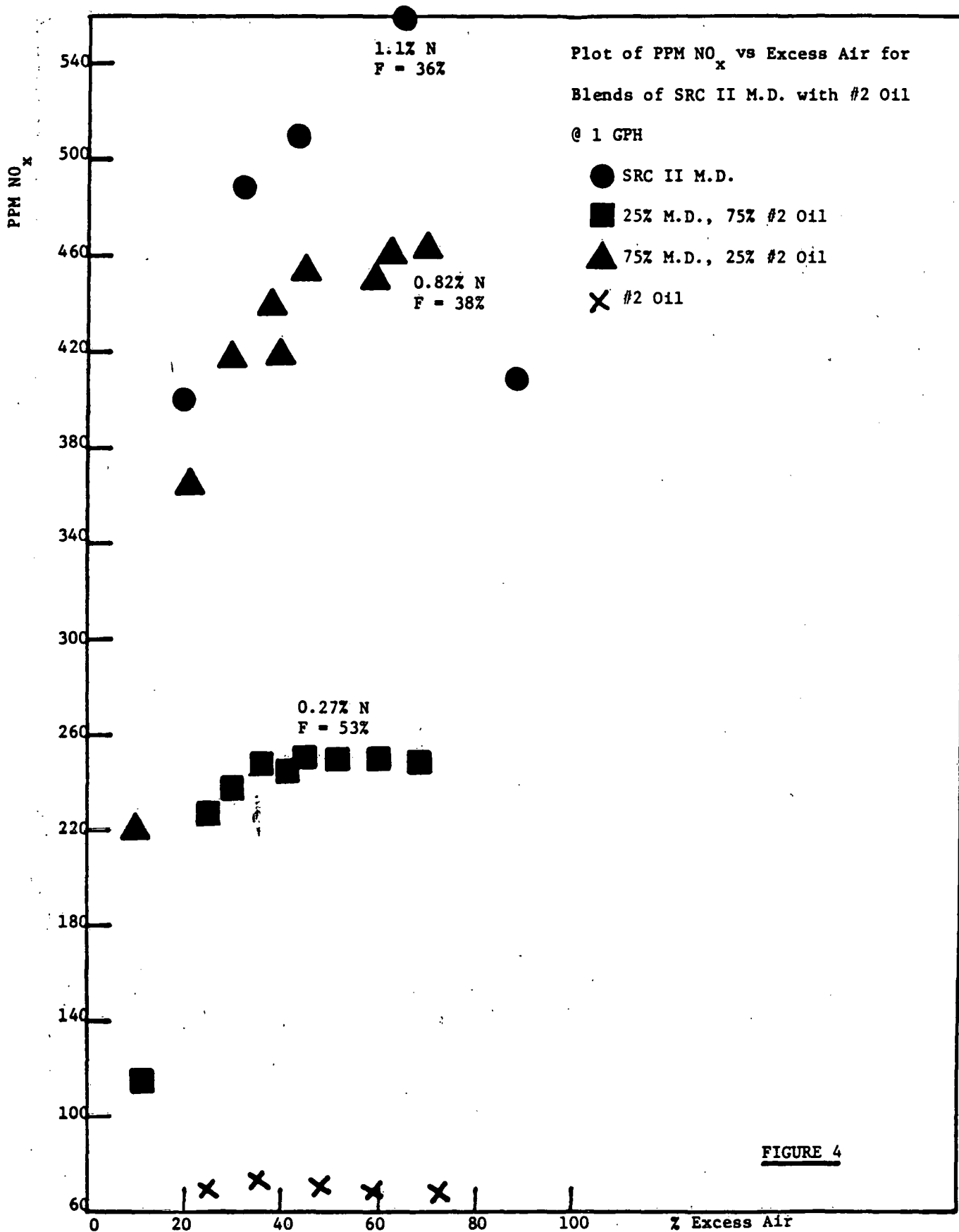


FIGURE 4

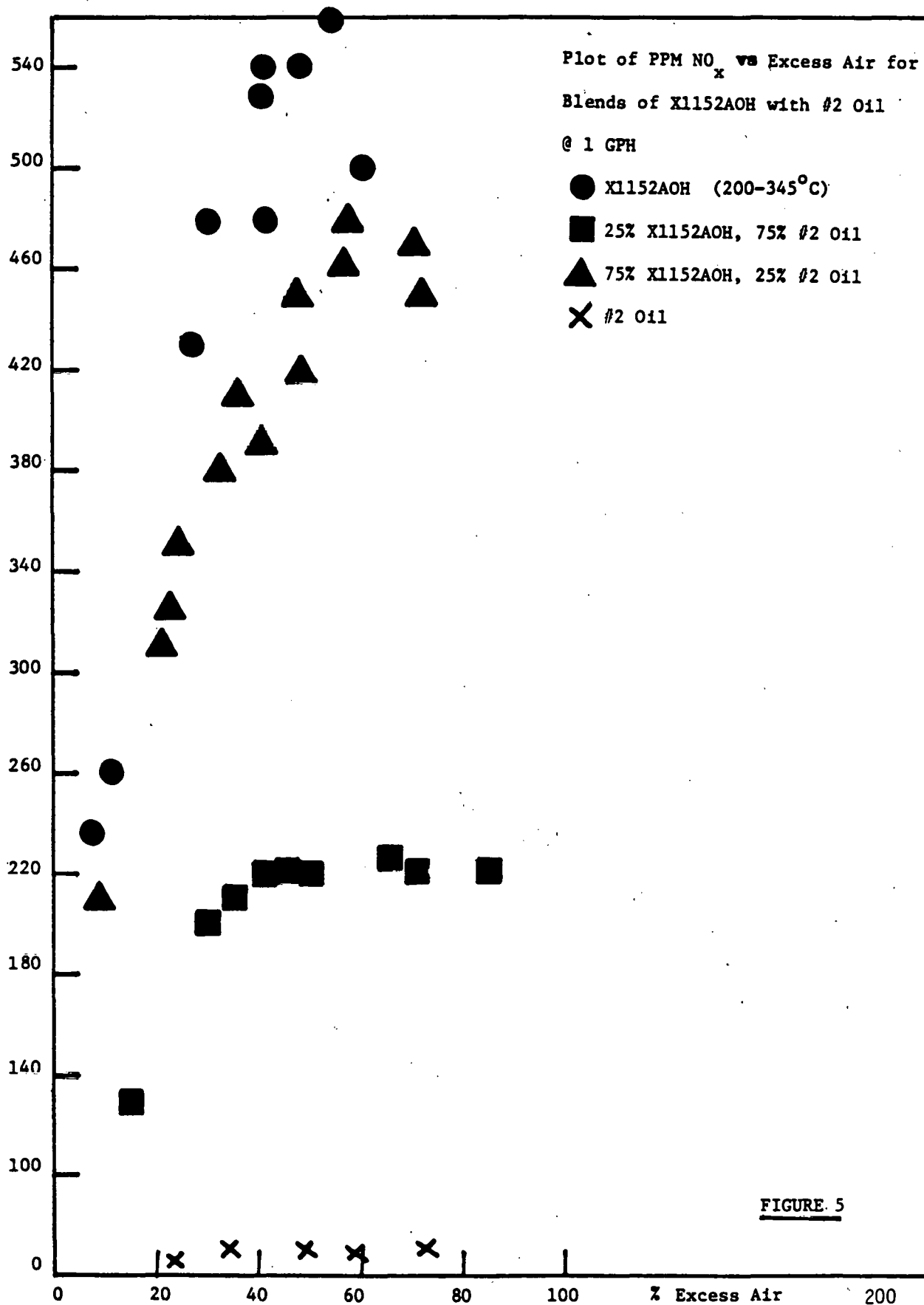


FIGURE 5

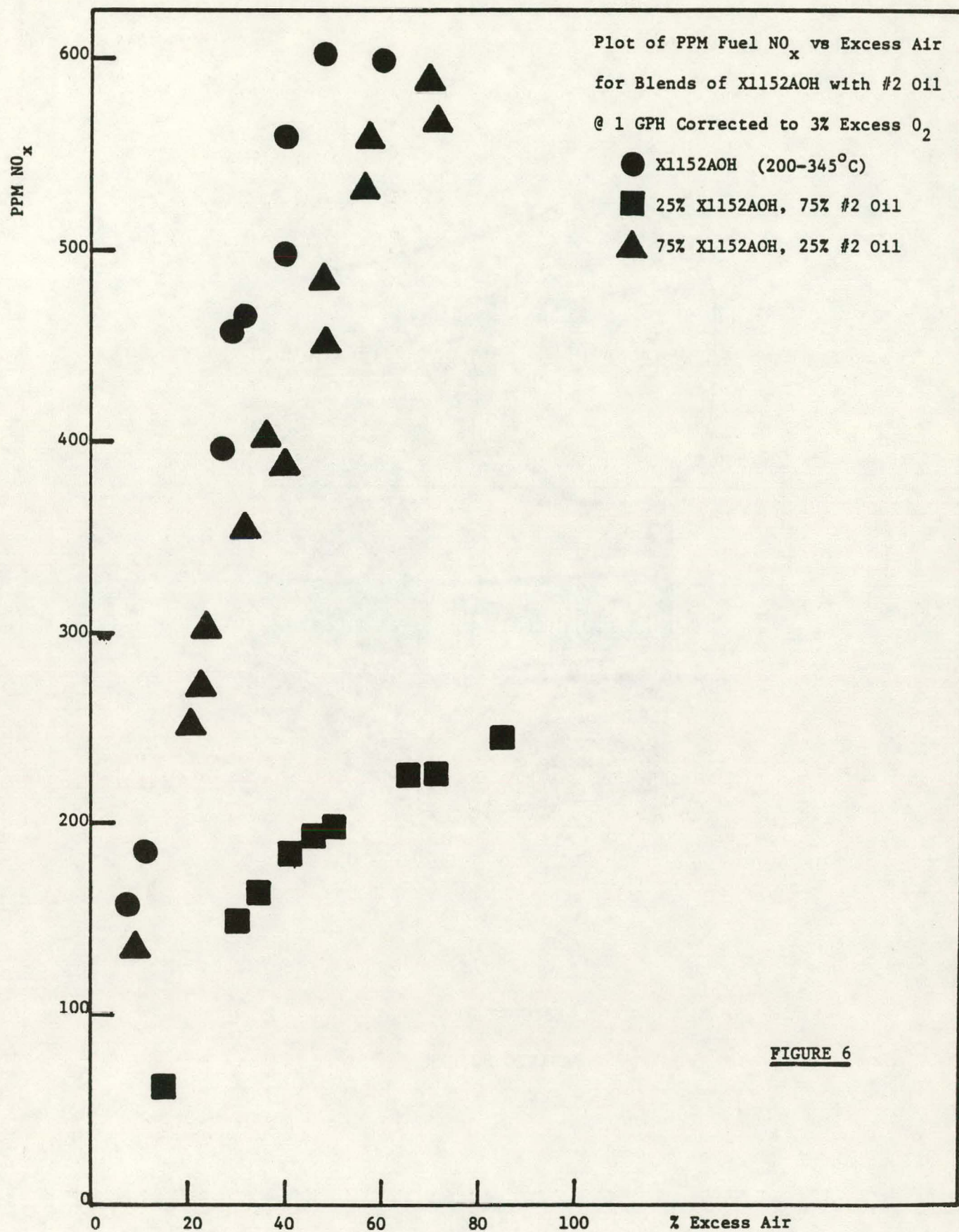


FIGURE 6

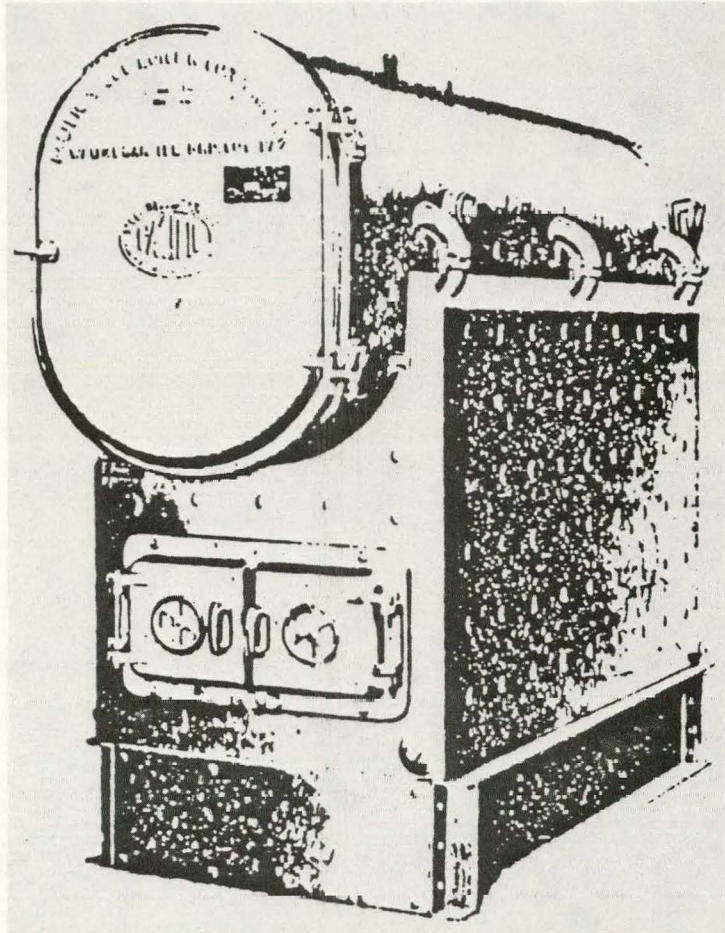


FIGURE 7
PACIFIC BOILER

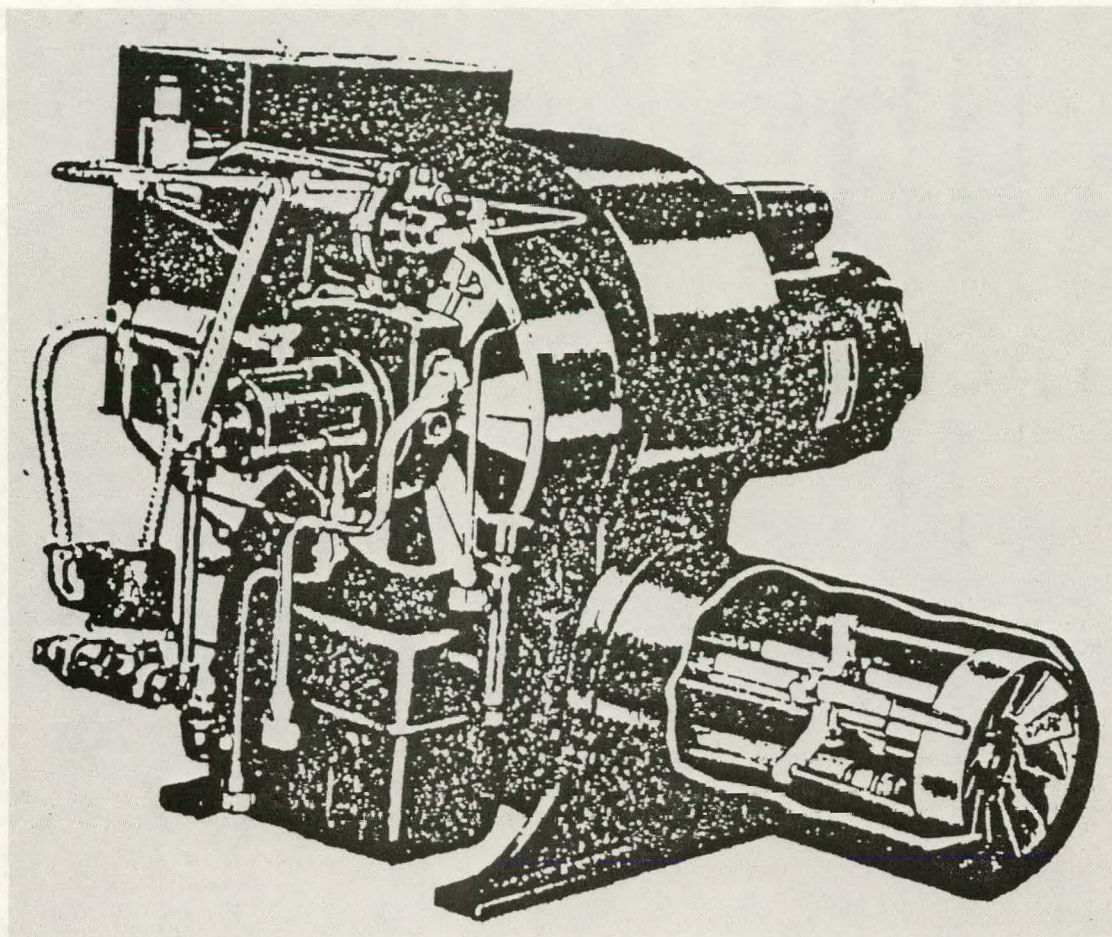


FIGURE 8
CLEAVER-BROOKS BURNER

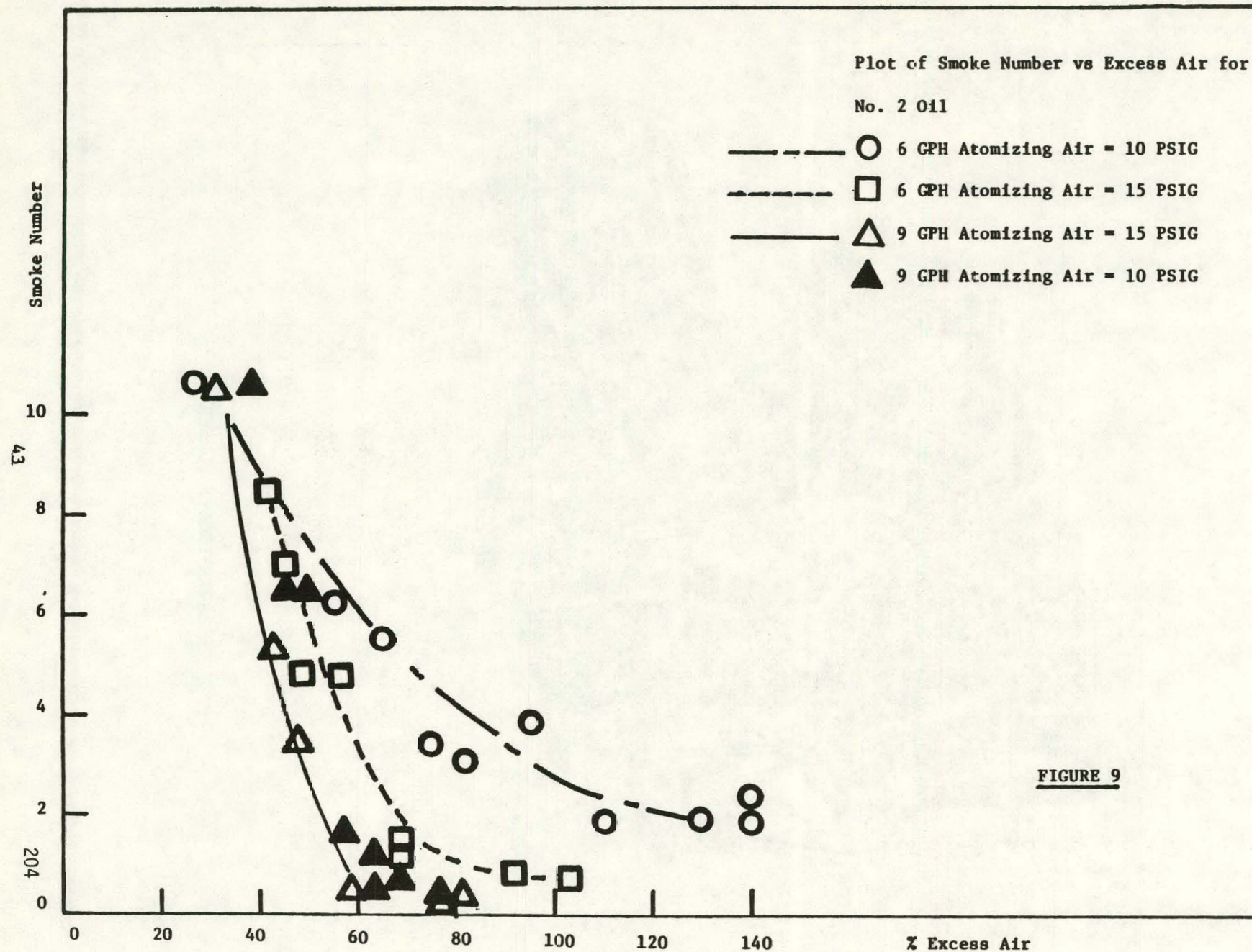
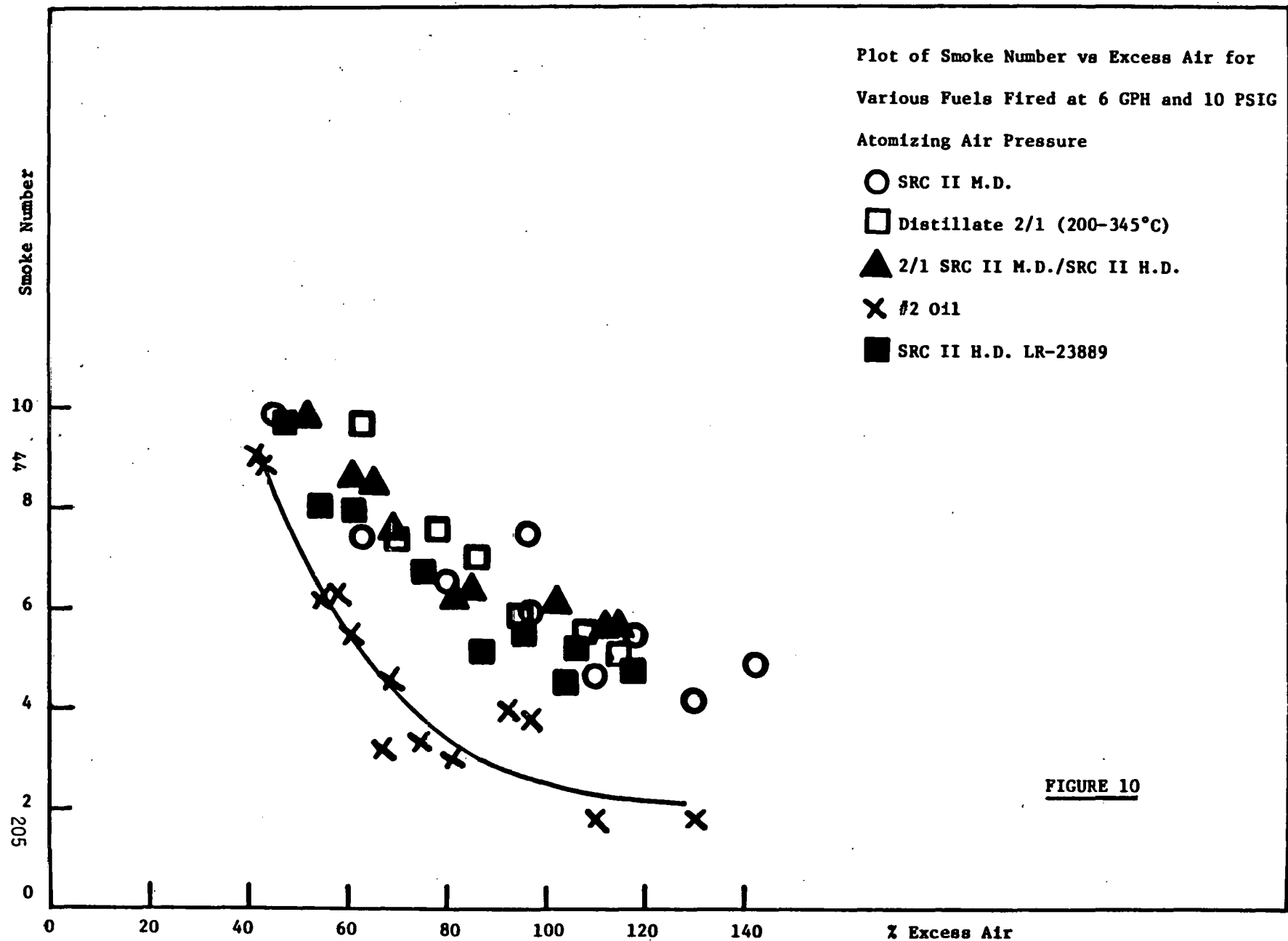


FIGURE 9



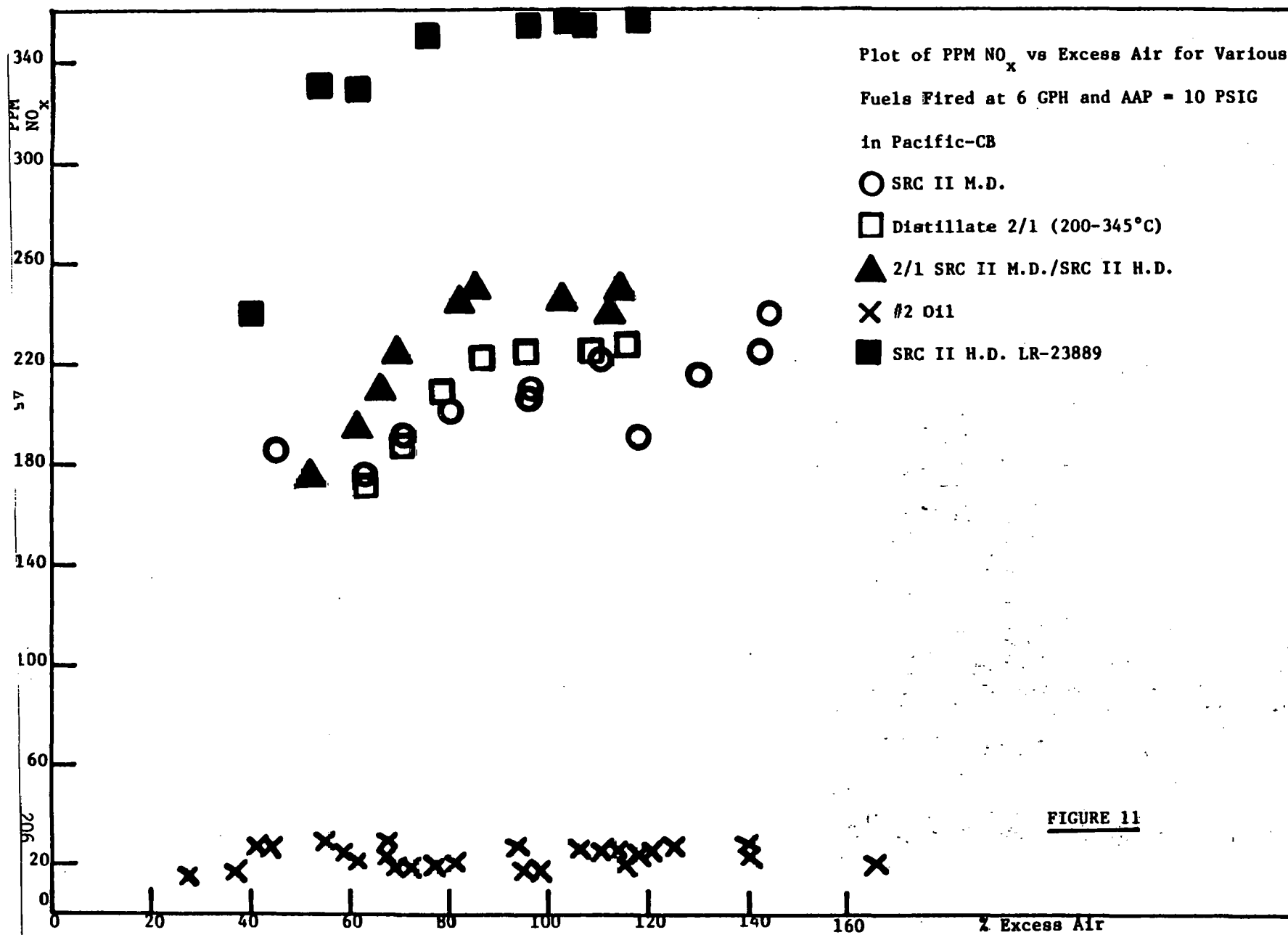
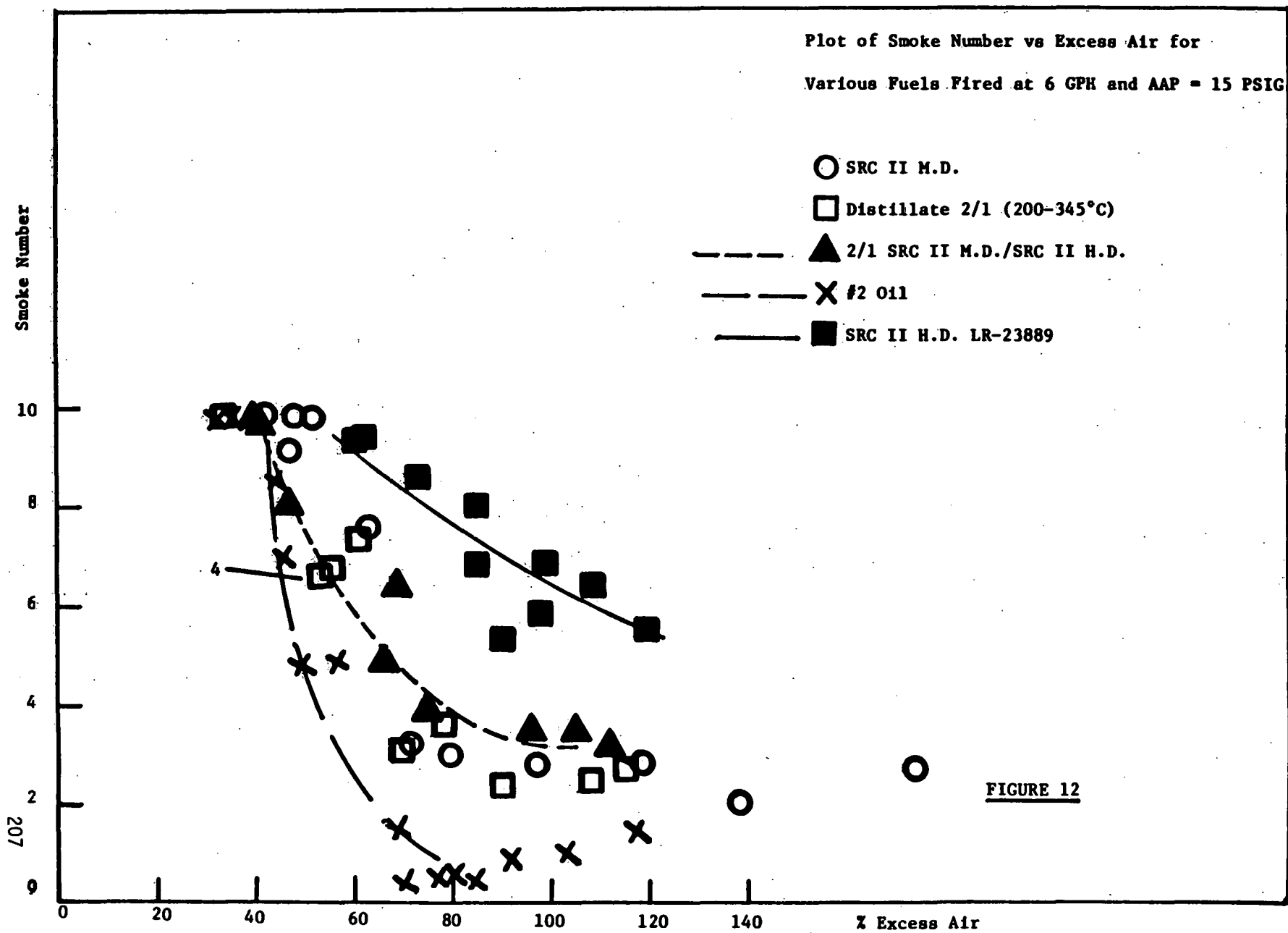


FIGURE 11



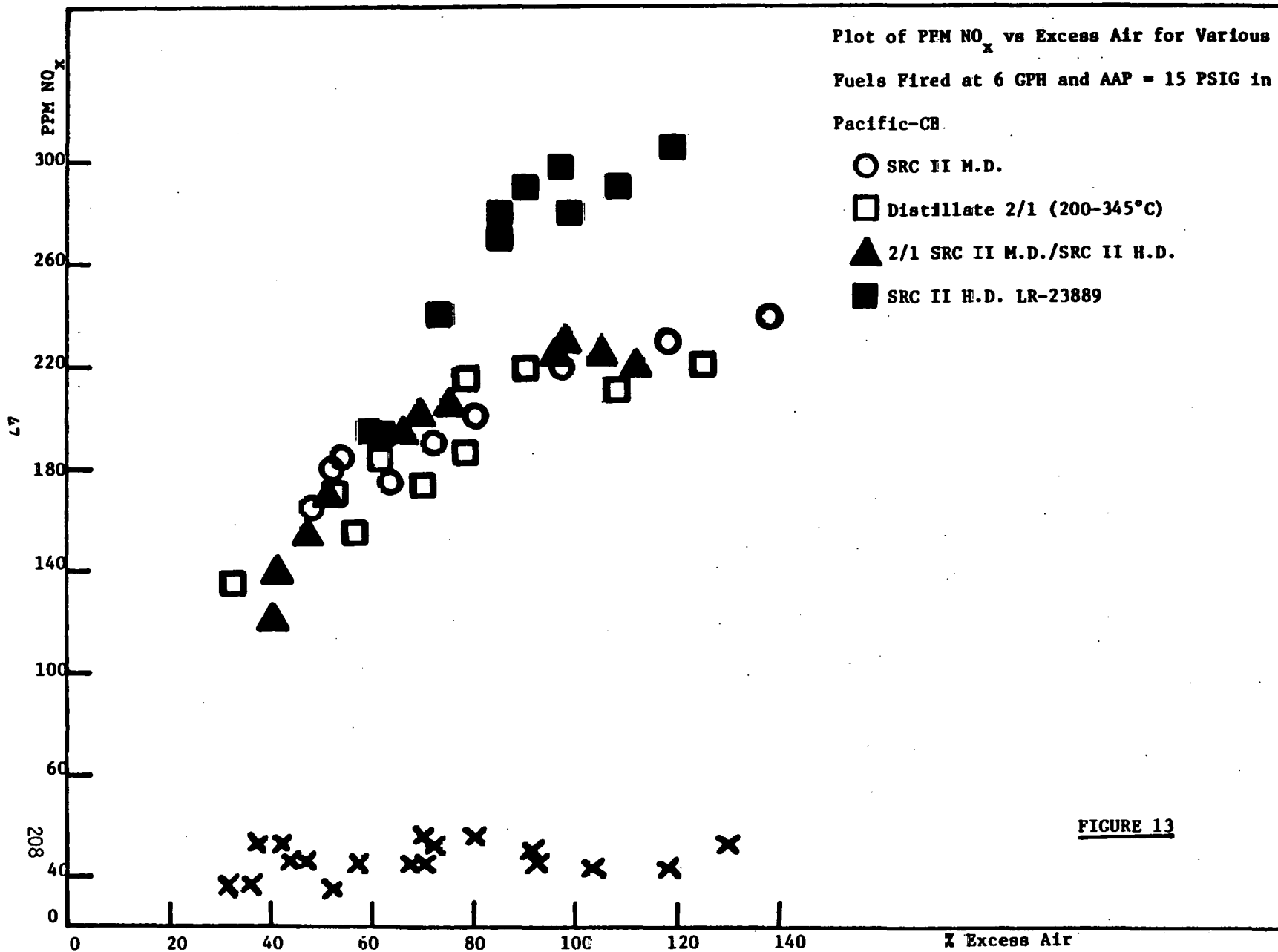
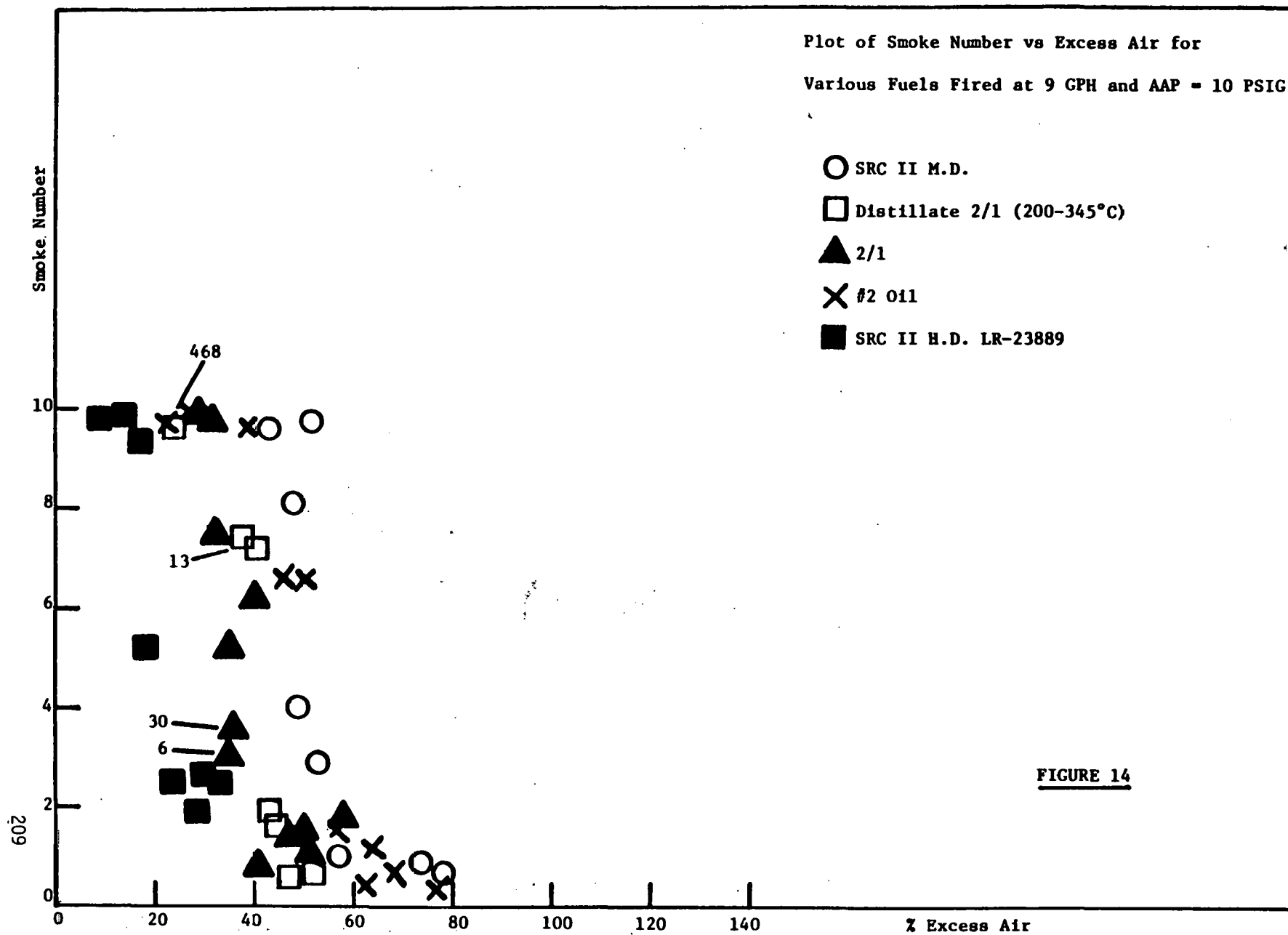


FIGURE 13



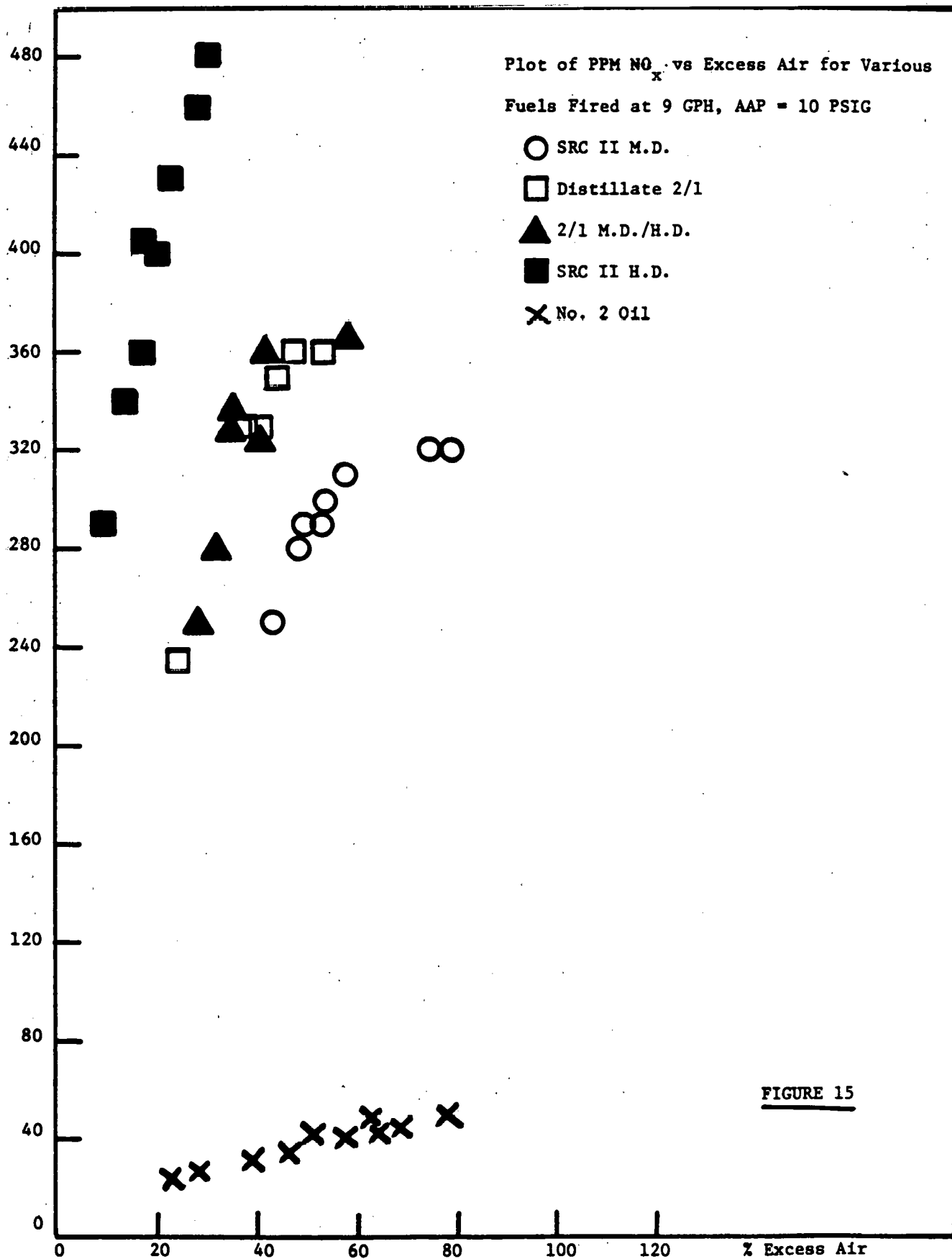


FIGURE 15

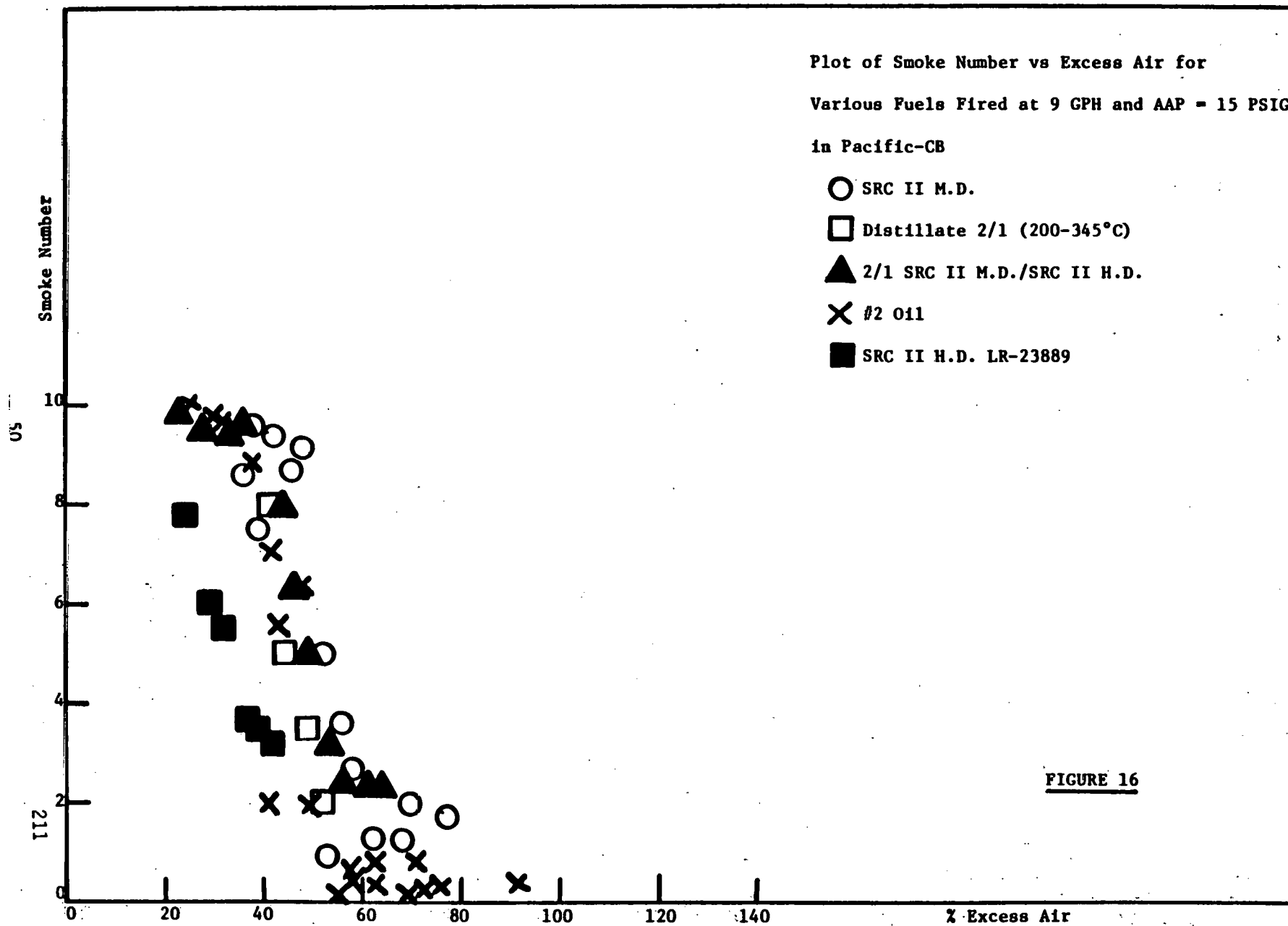


FIGURE 16

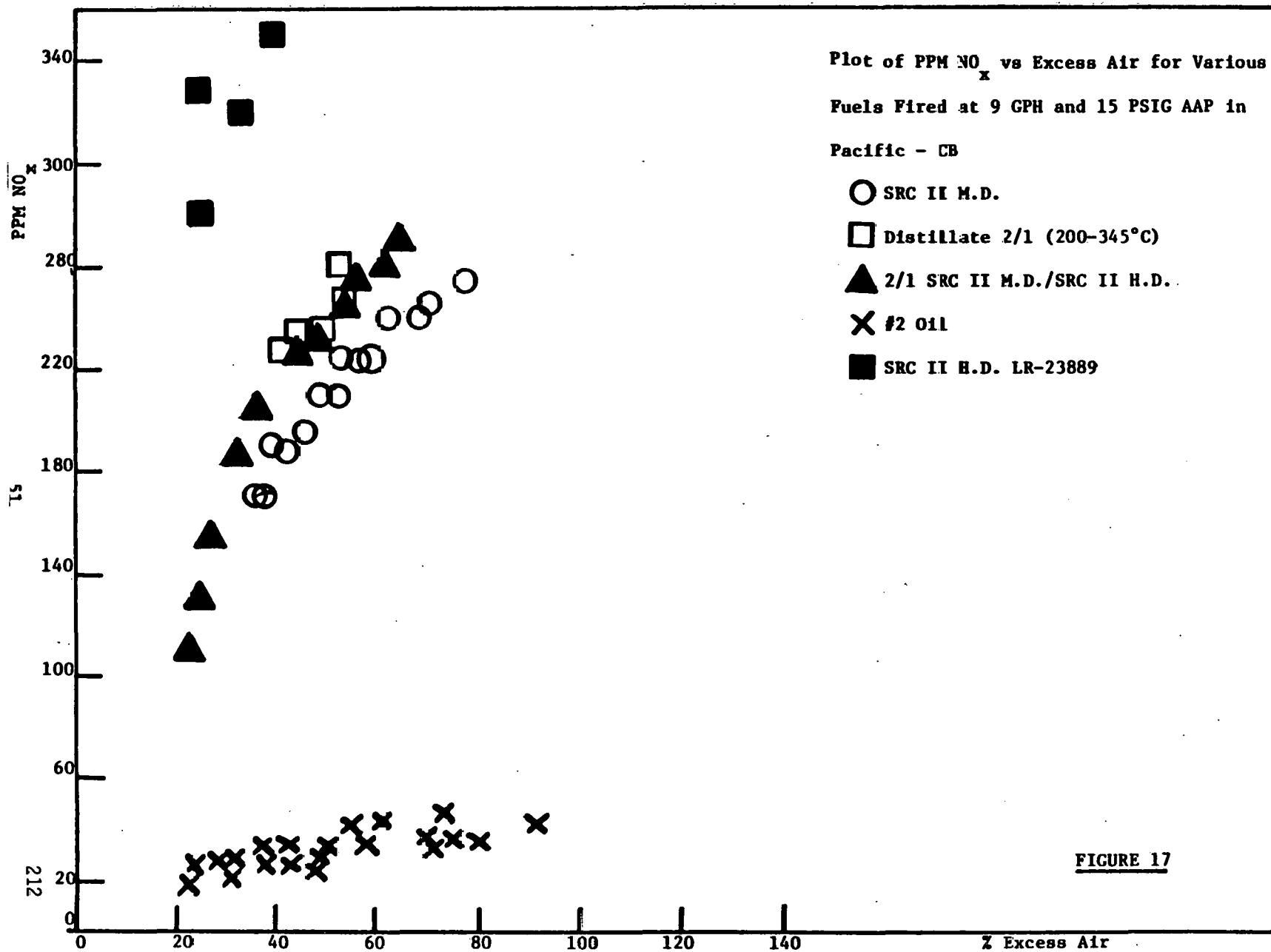


FIGURE 17

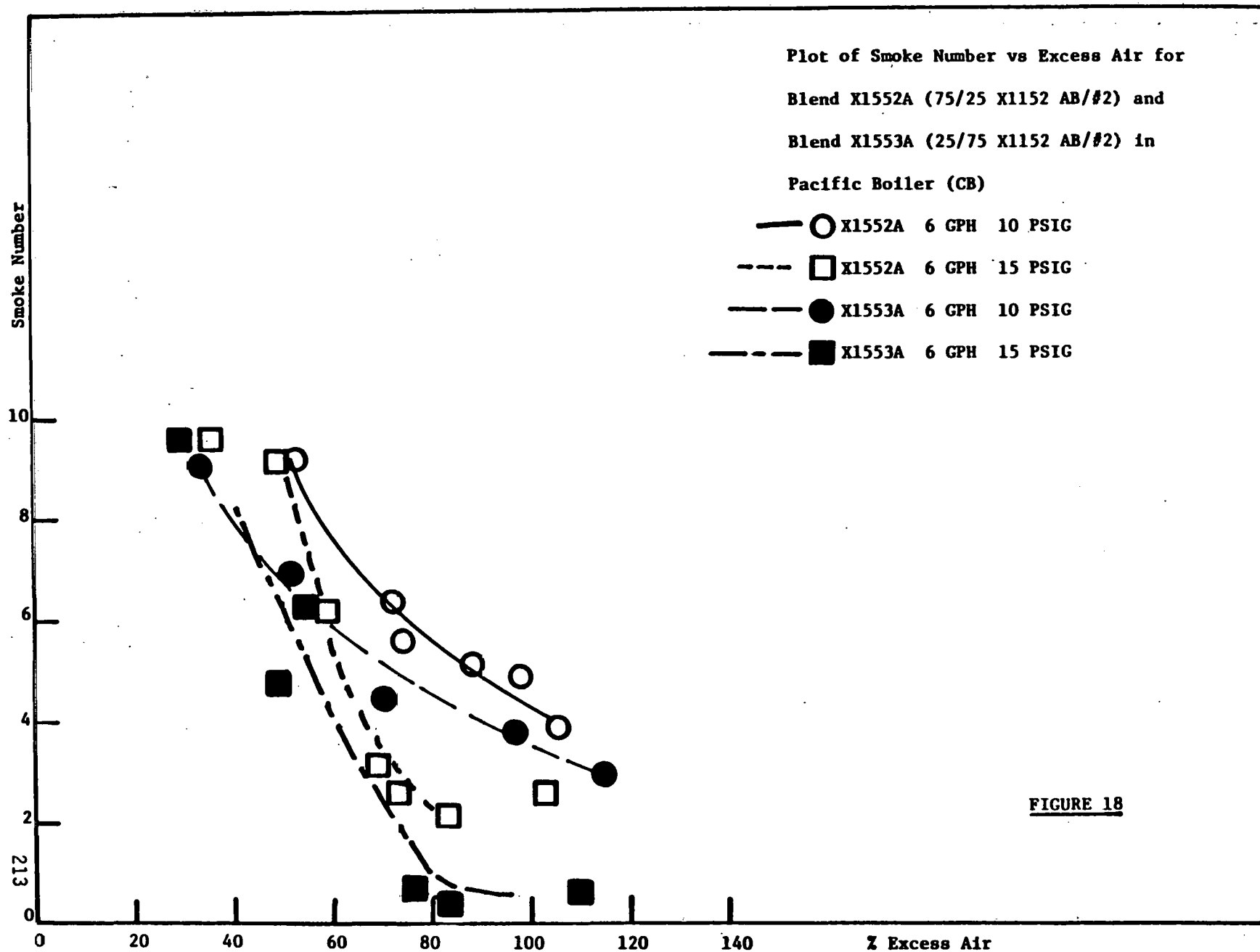


FIGURE 18

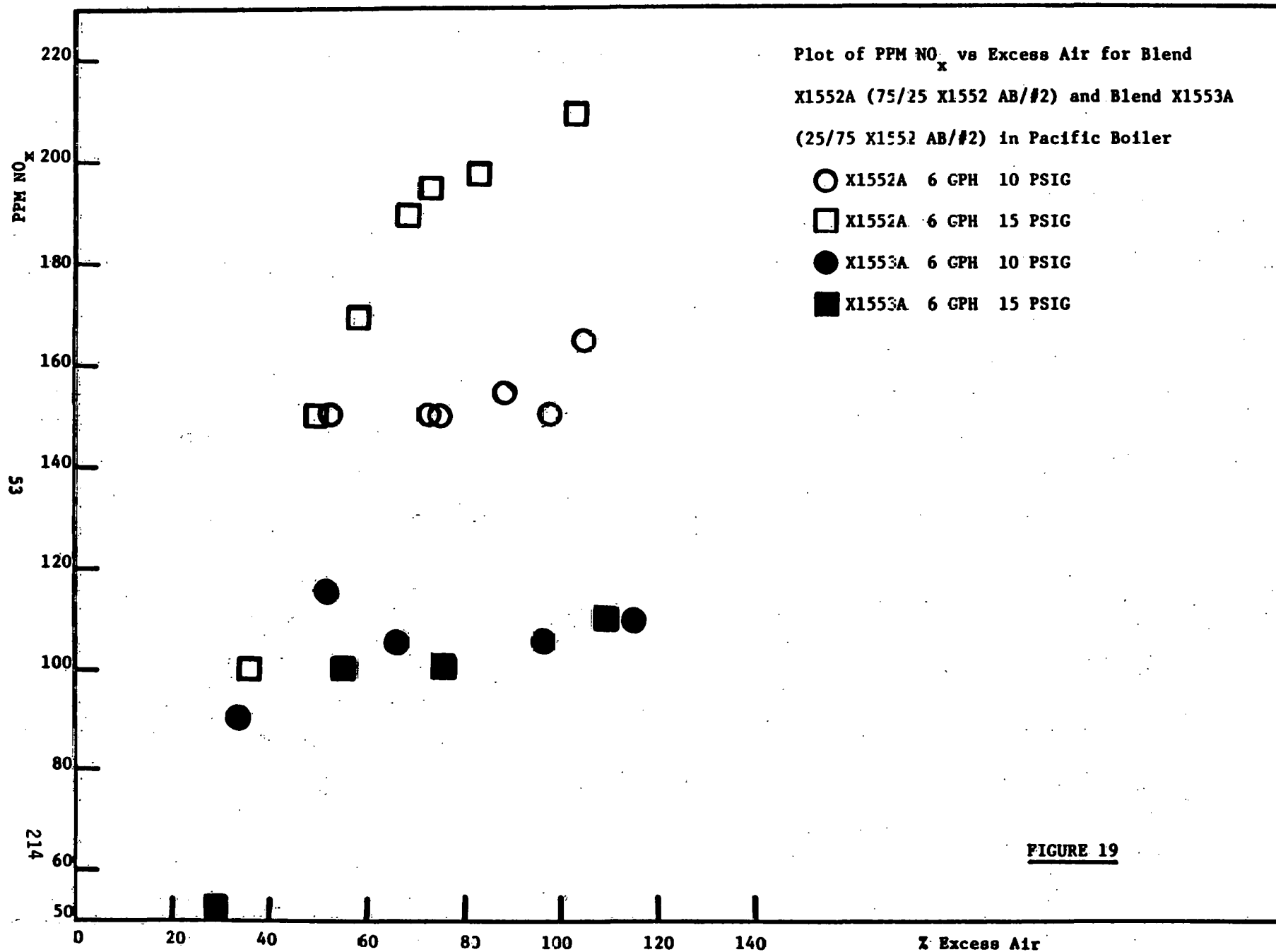


FIGURE 19

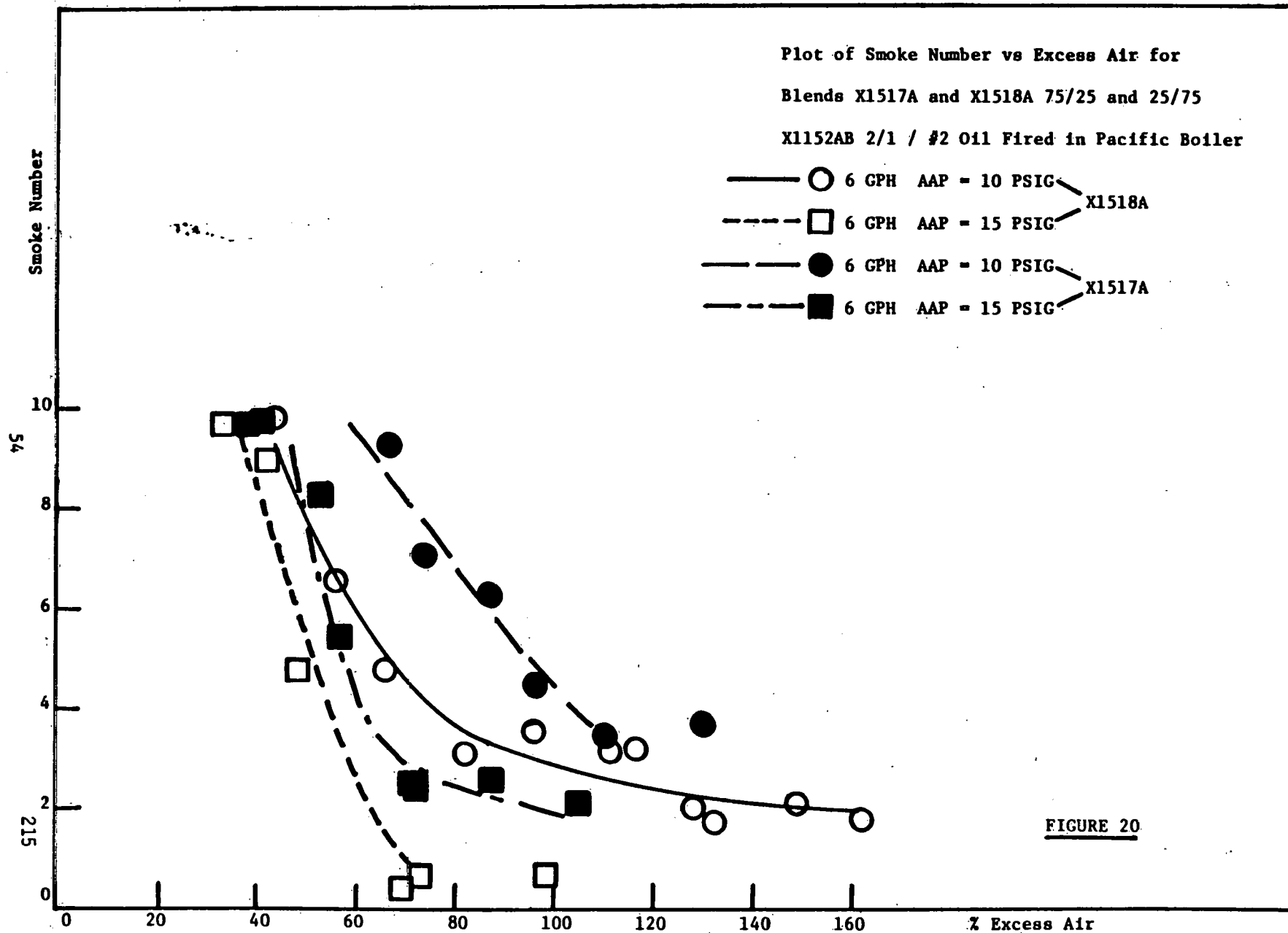
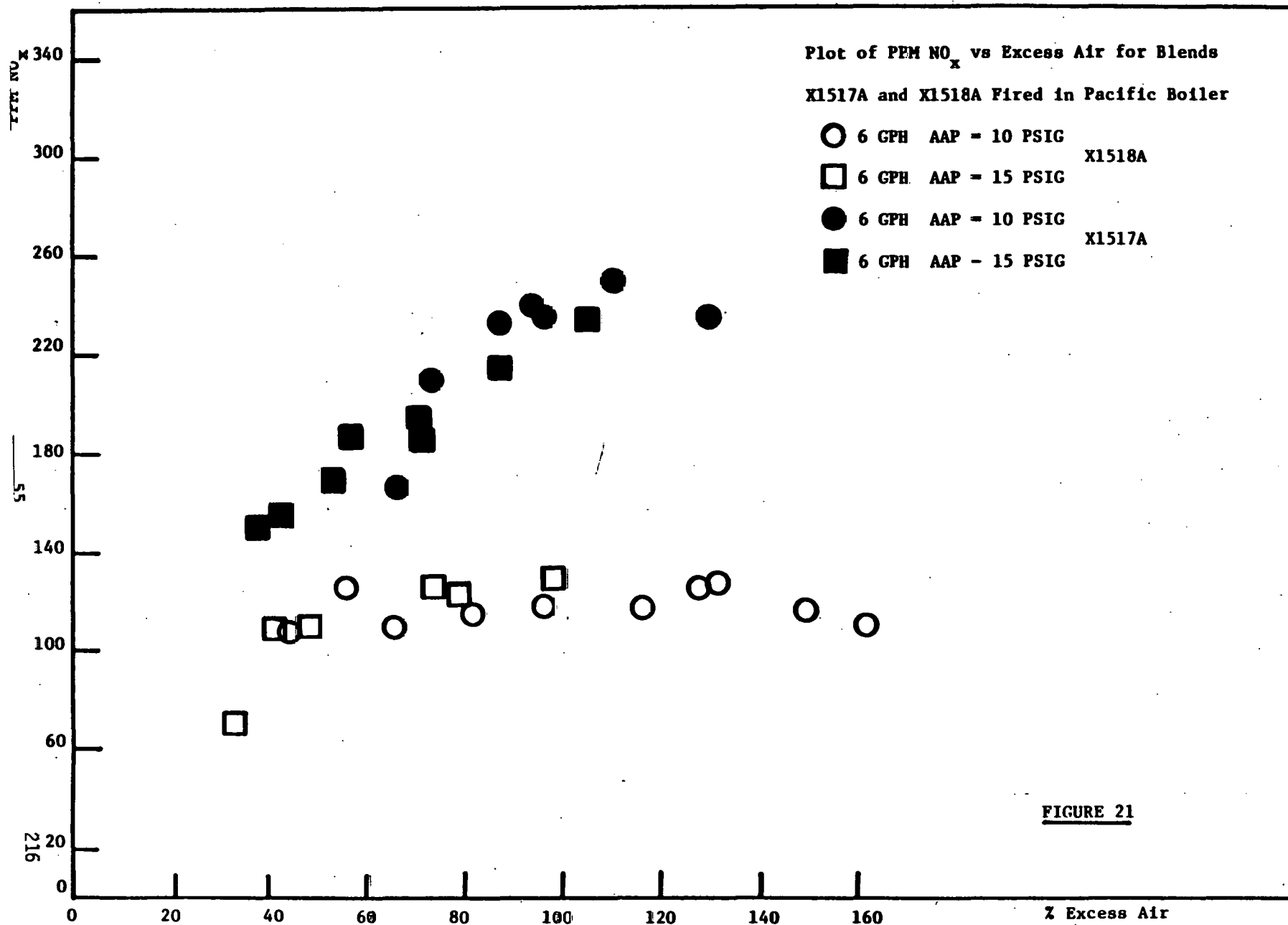
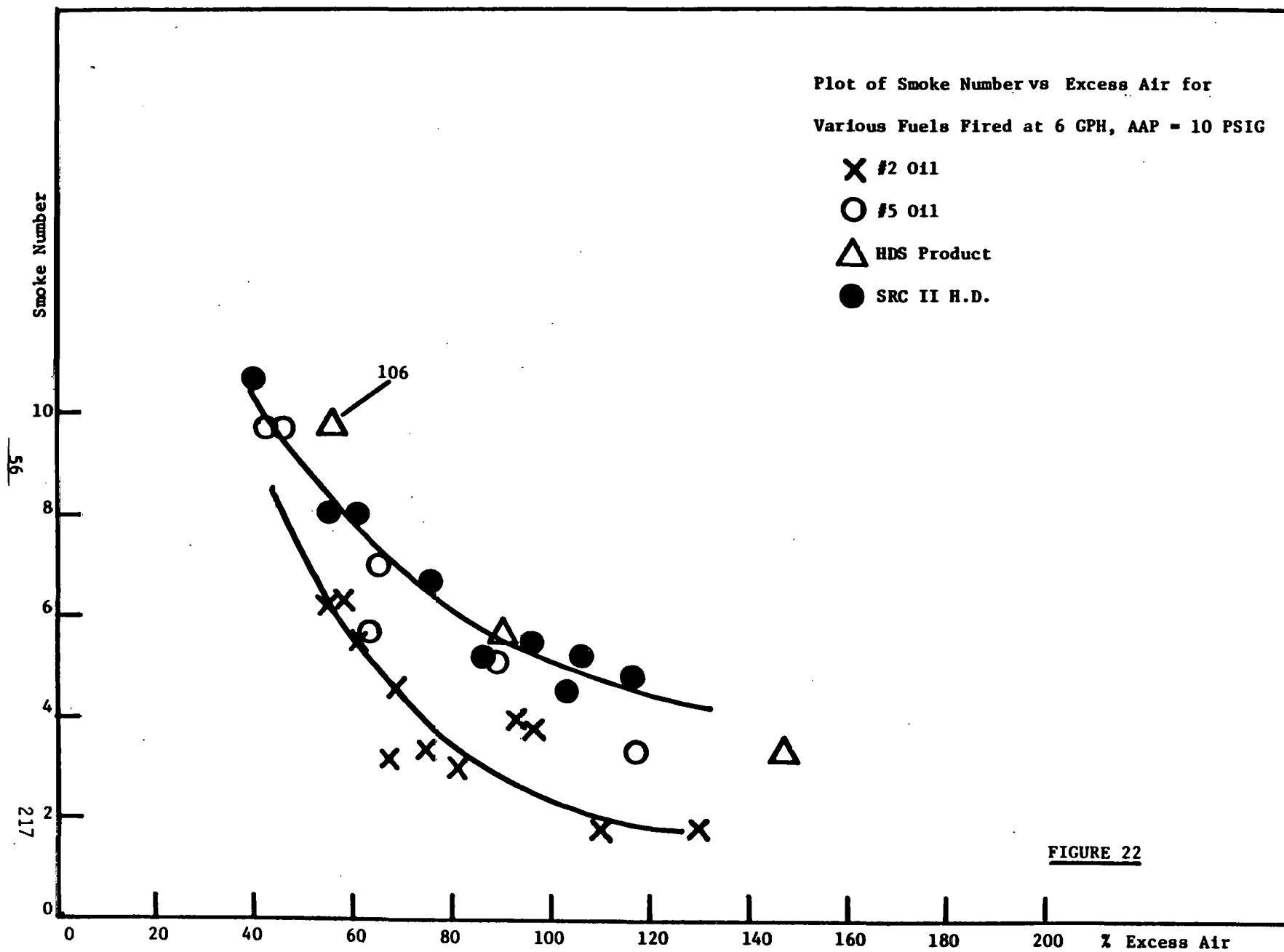


FIGURE 20





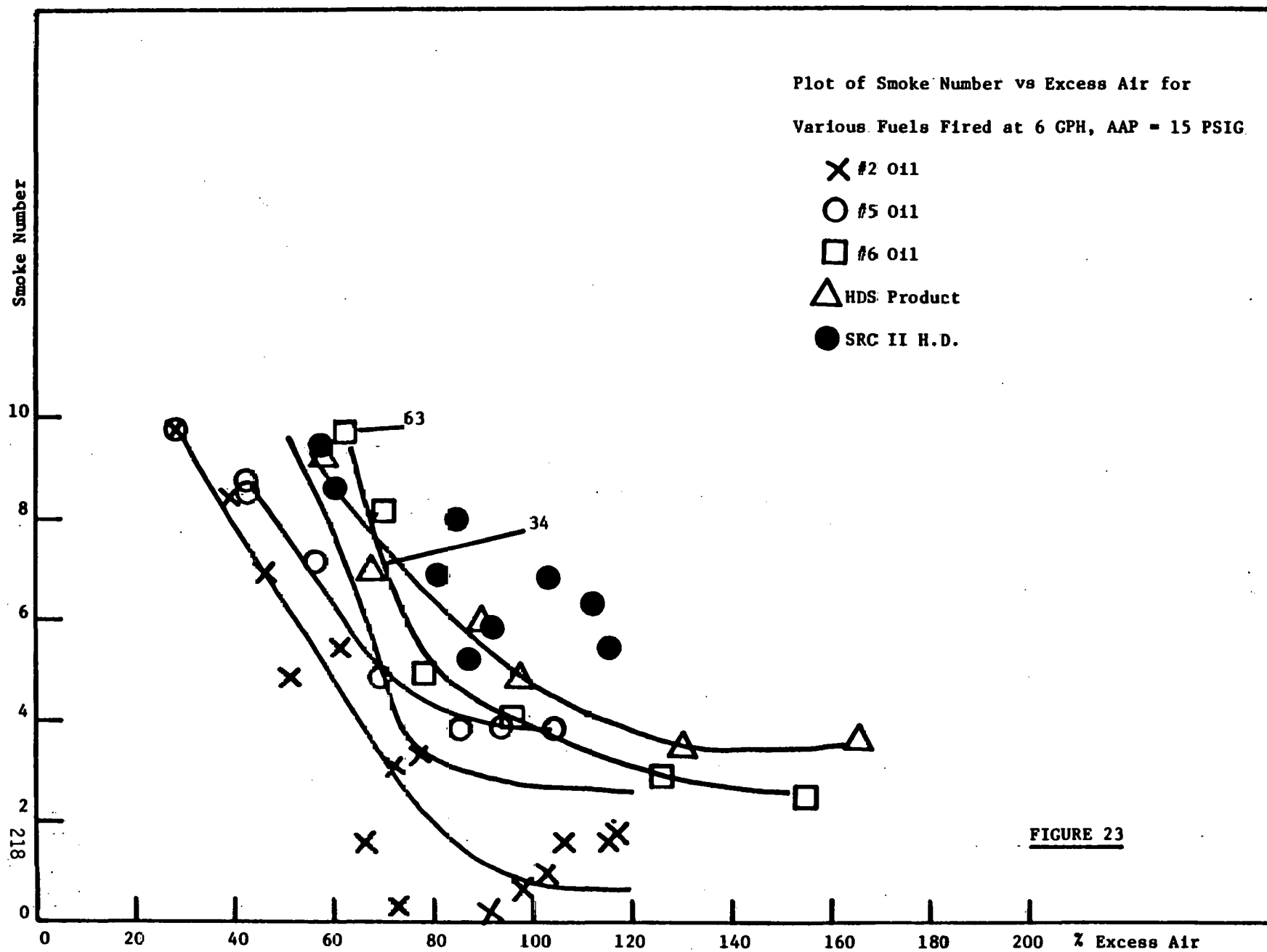


FIGURE 23

Plot of Smoke Number vs Excess Air for
Various Fuels Fired at 9 GPH, APP = 10 PSIG

X #2 Oil
 Δ HDS Product
 ● SRC II H.D.

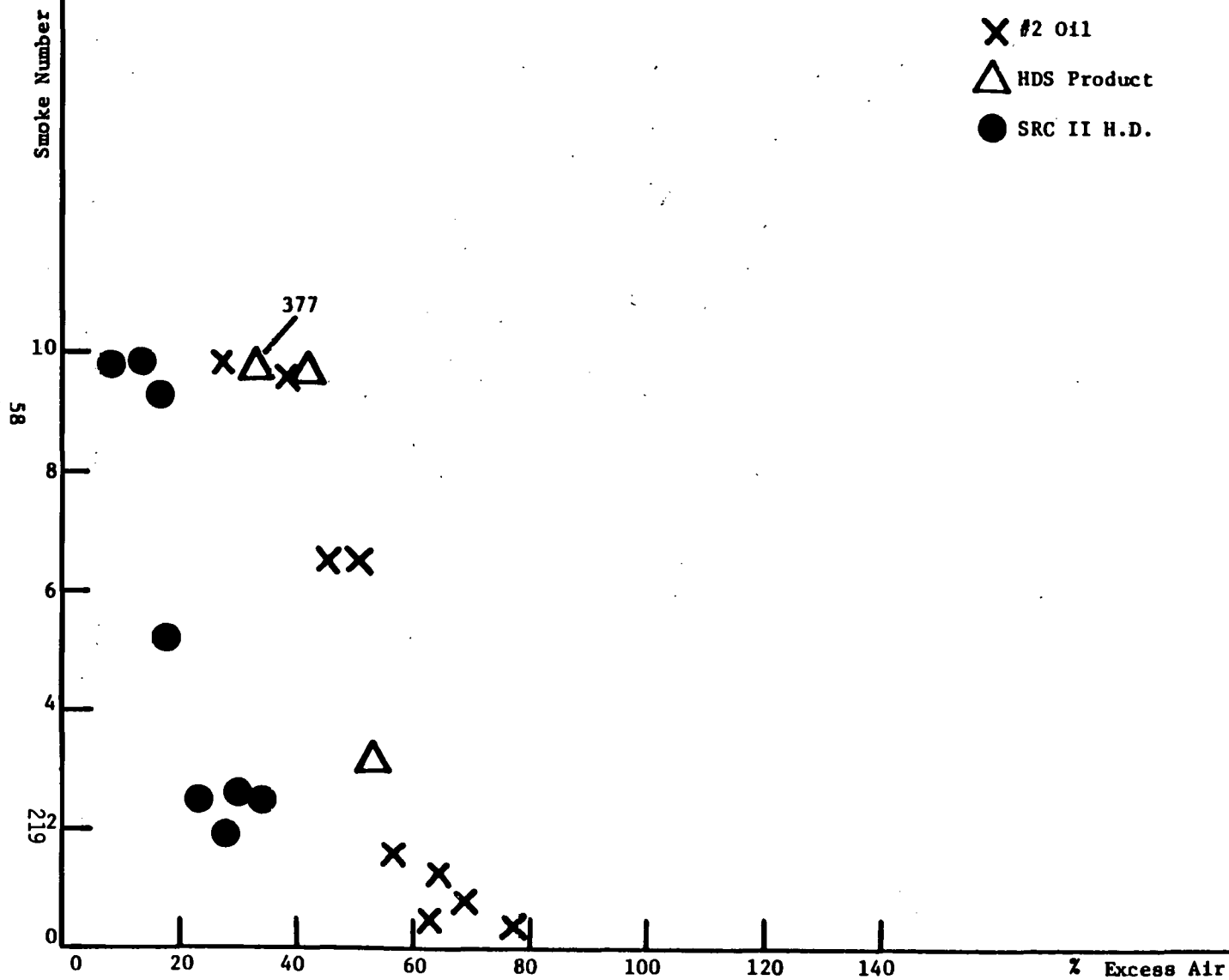
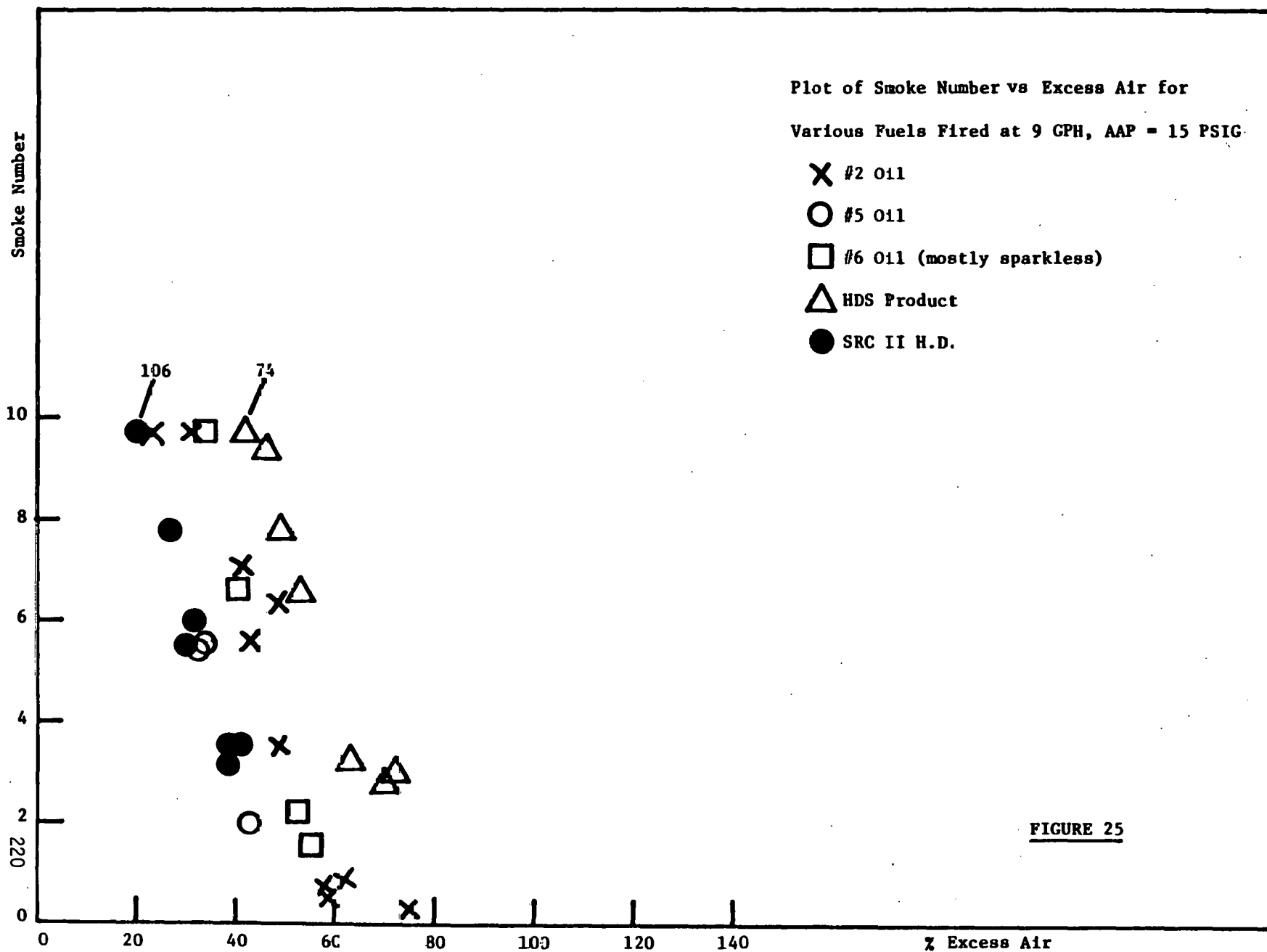
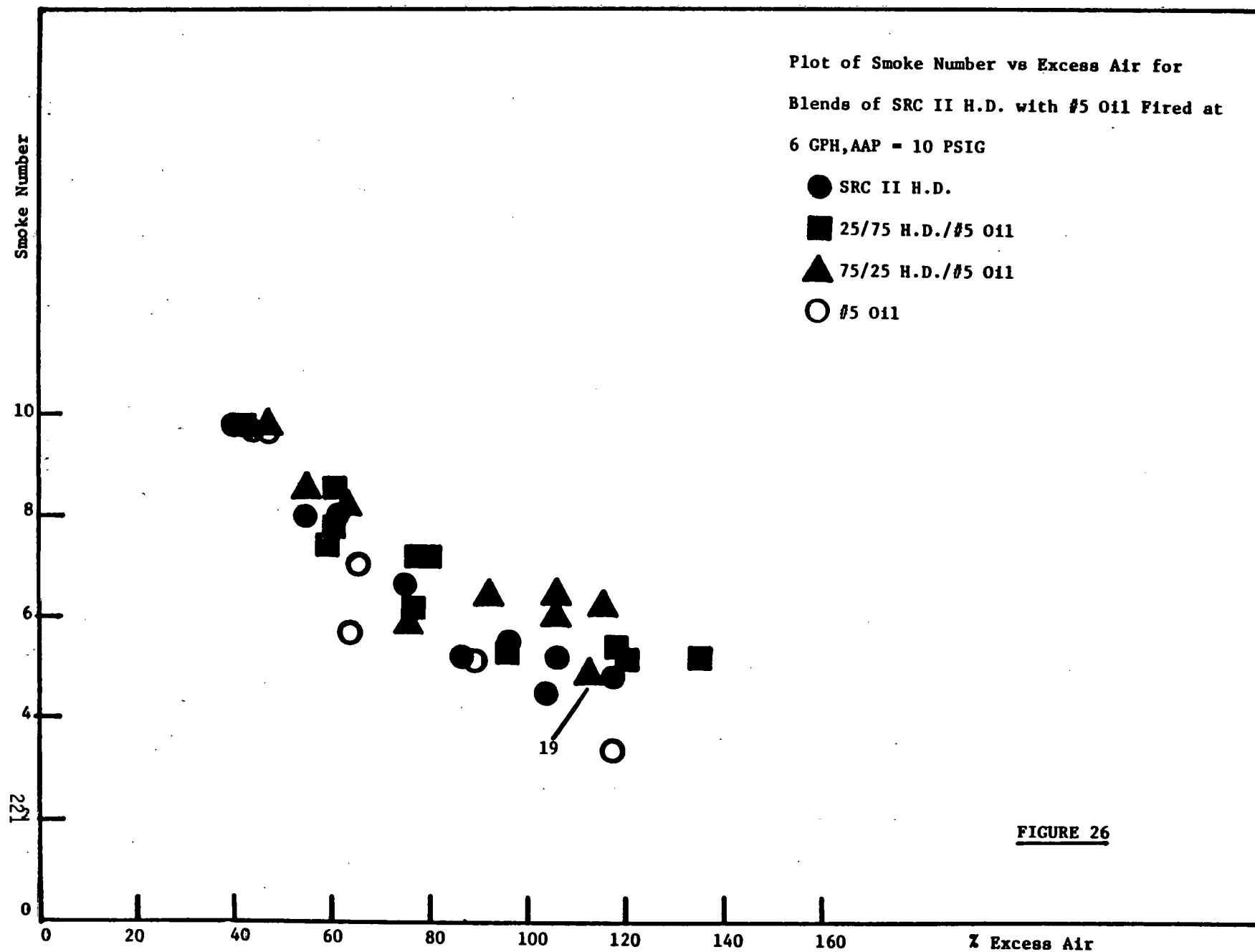


FIGURE 24





Plot of Smoke Number vs Excess Air for
Blends of SRC II H.D. with #5 Oil Fired at
6 GPH, AAP = 15 PSIG

- SRC II H.D.
- 25/75 H.D./#5
- ▲ 75/25 H.D./#5
- #5 Oil

Smoke Number

10
8
6
4
2
222
0

57

13

FIGURE 27

% Excess Air

0 20 40 60 80 100 120 140 160

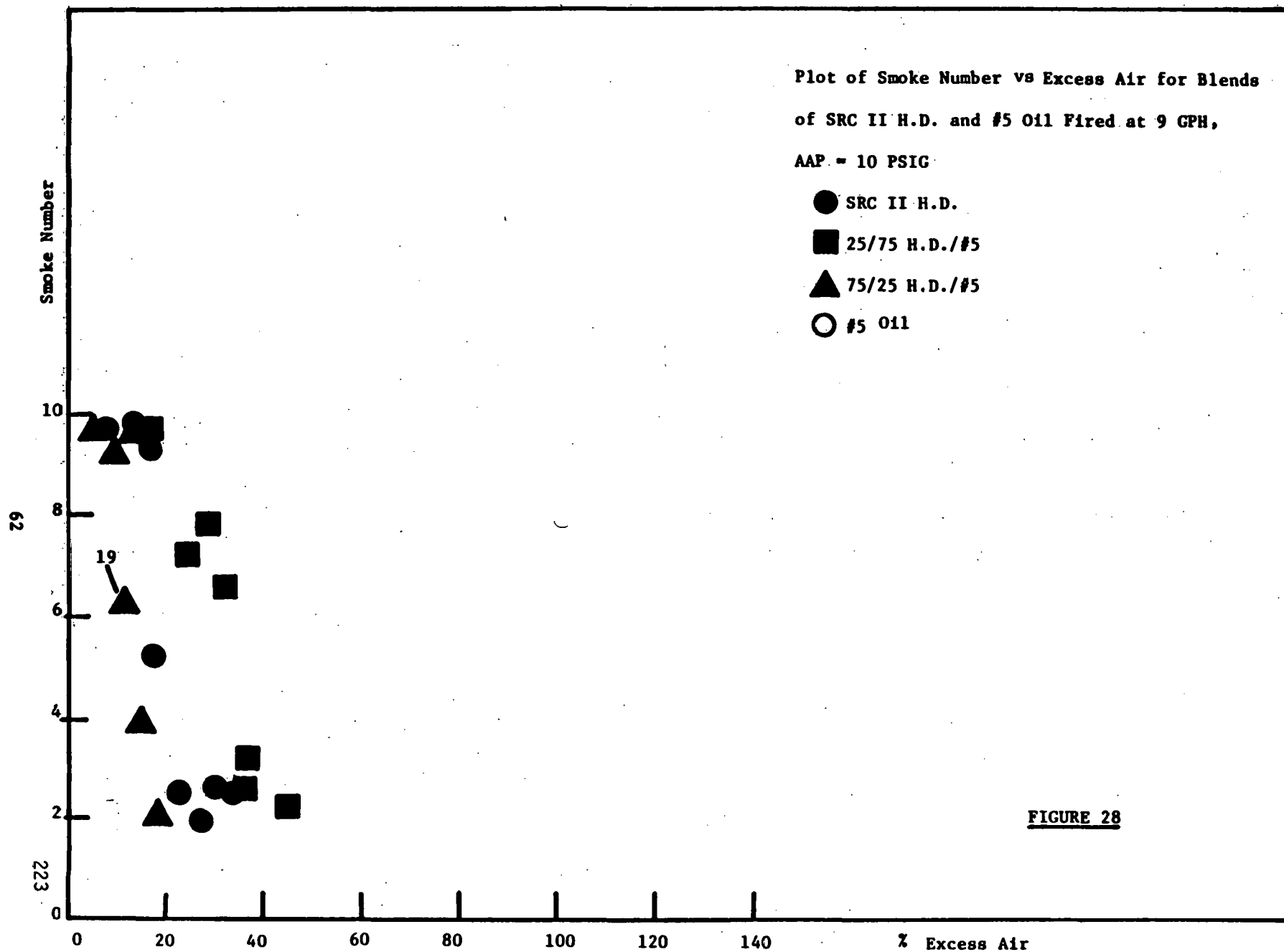


FIGURE 28

Plot of Smoke Number vs Excess Air for Blends
of SRC II H.D. with #5 Oil Fired at 9 GPH,

AAP = 15 PSIG

- SRC II H.D.
- 25/75 H.D./#5 Oil
- ▲ 75/25 H.D./#5 Oil
- #5 Oil

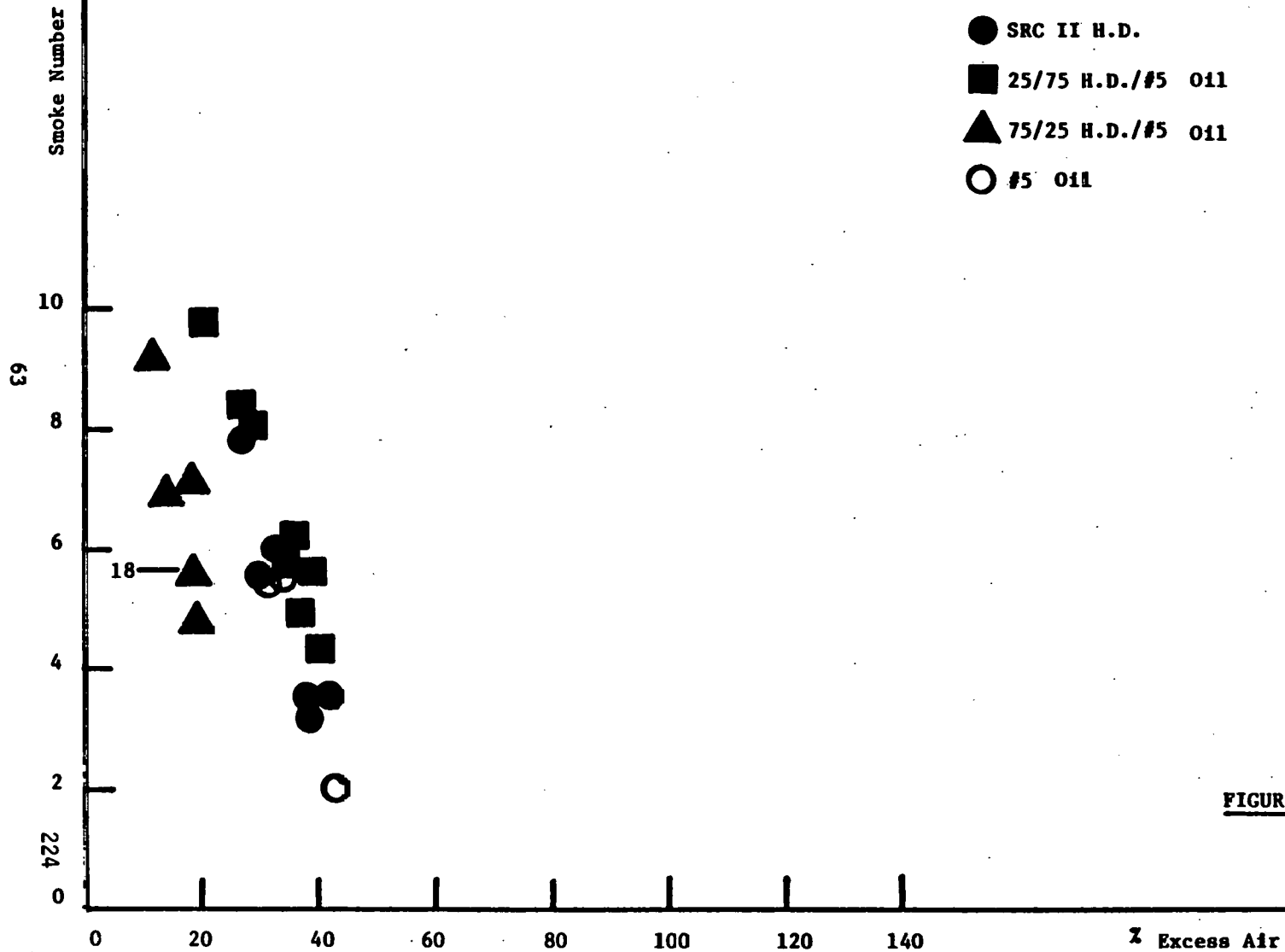


FIGURE 29

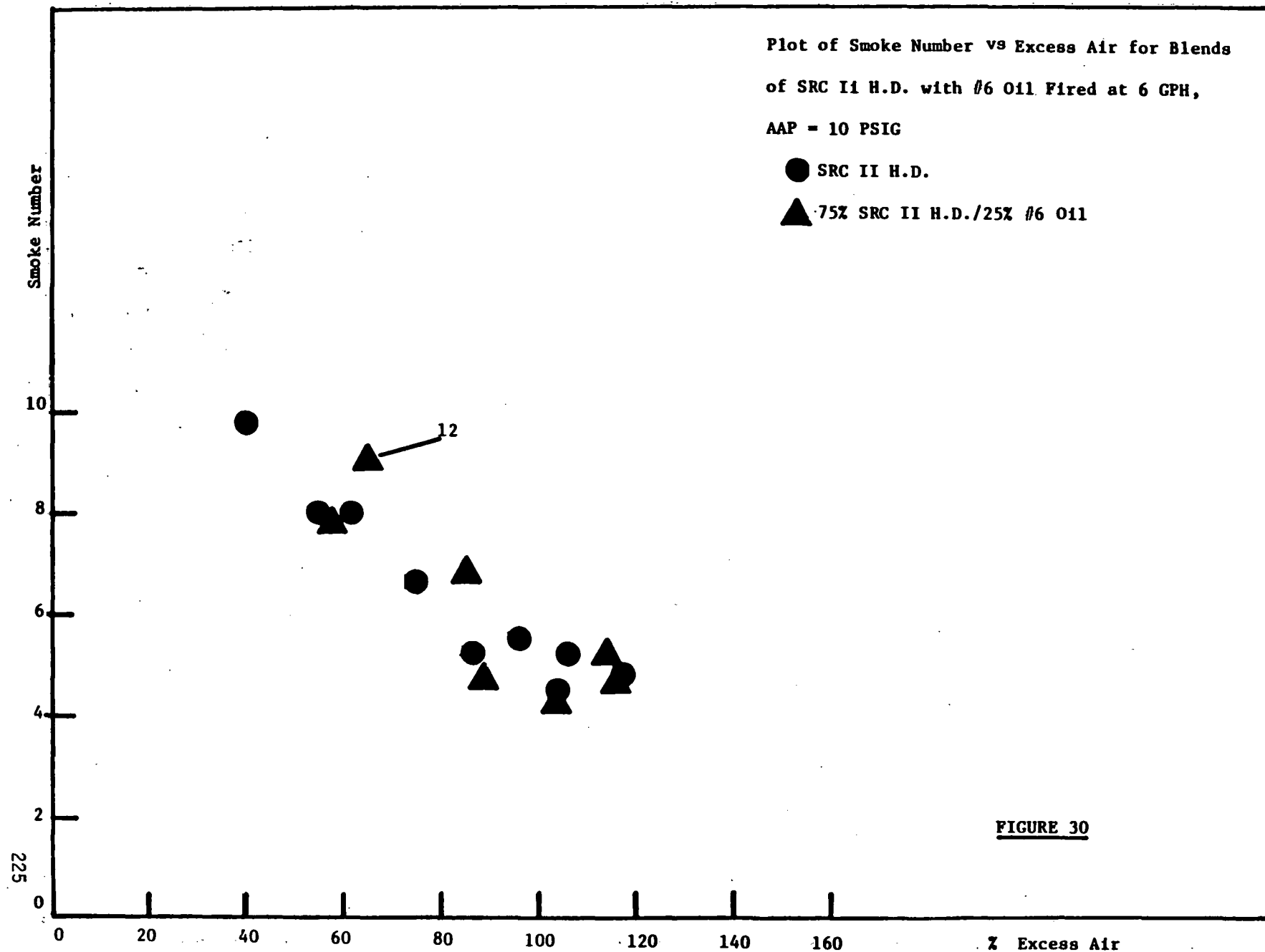


FIGURE 30

Plot of Smoke Number vs Excess Air
for Blends of SRC II H.D. W/#6 Oil Fired at
6 GPH, AAP = 15 PSIG

- SRC II H.D.
- 25/75 H.D./#6 Oil
- ▲ 75/25 H.D./#6 Oil
- △ #6 Oil

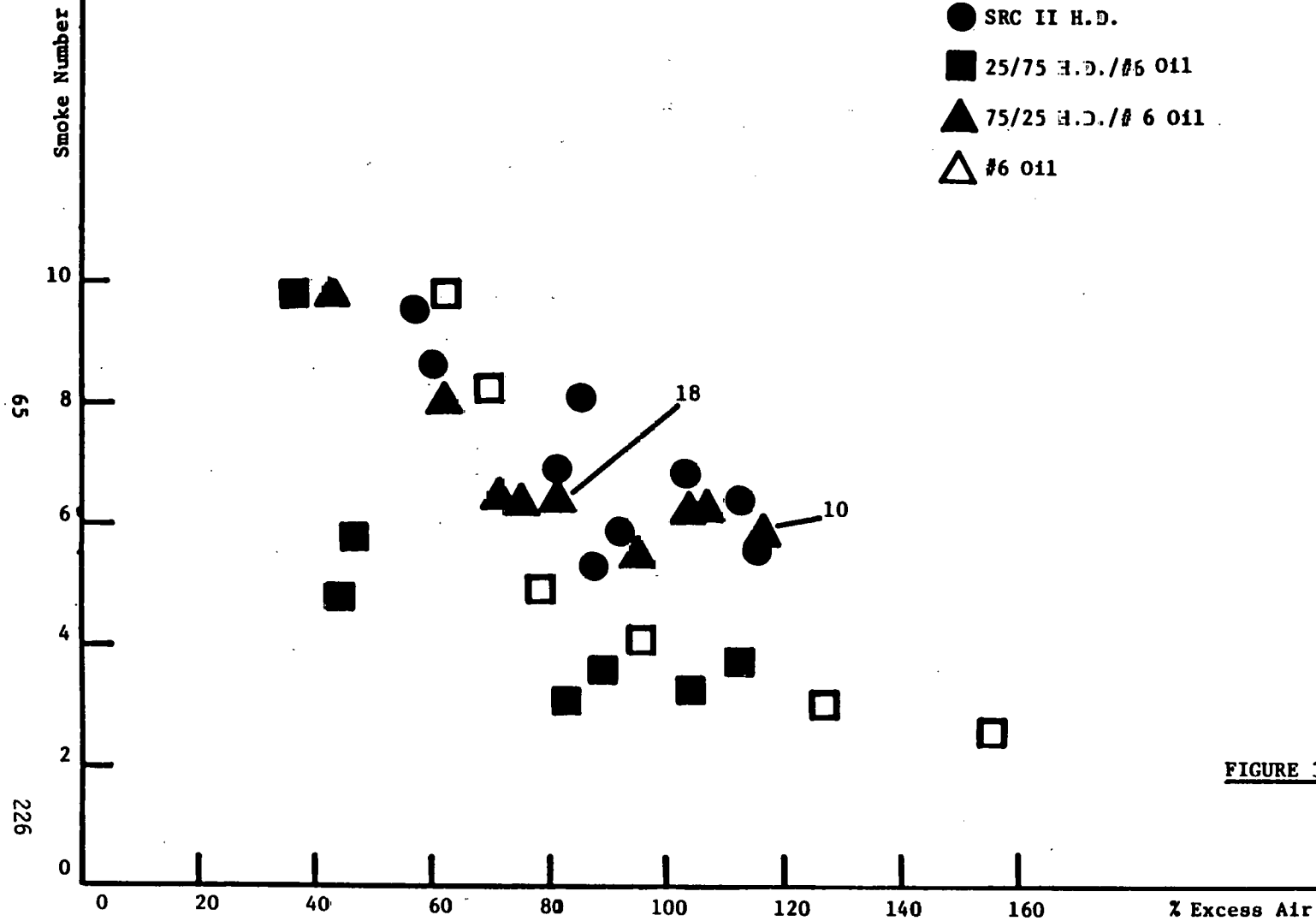


FIGURE 31

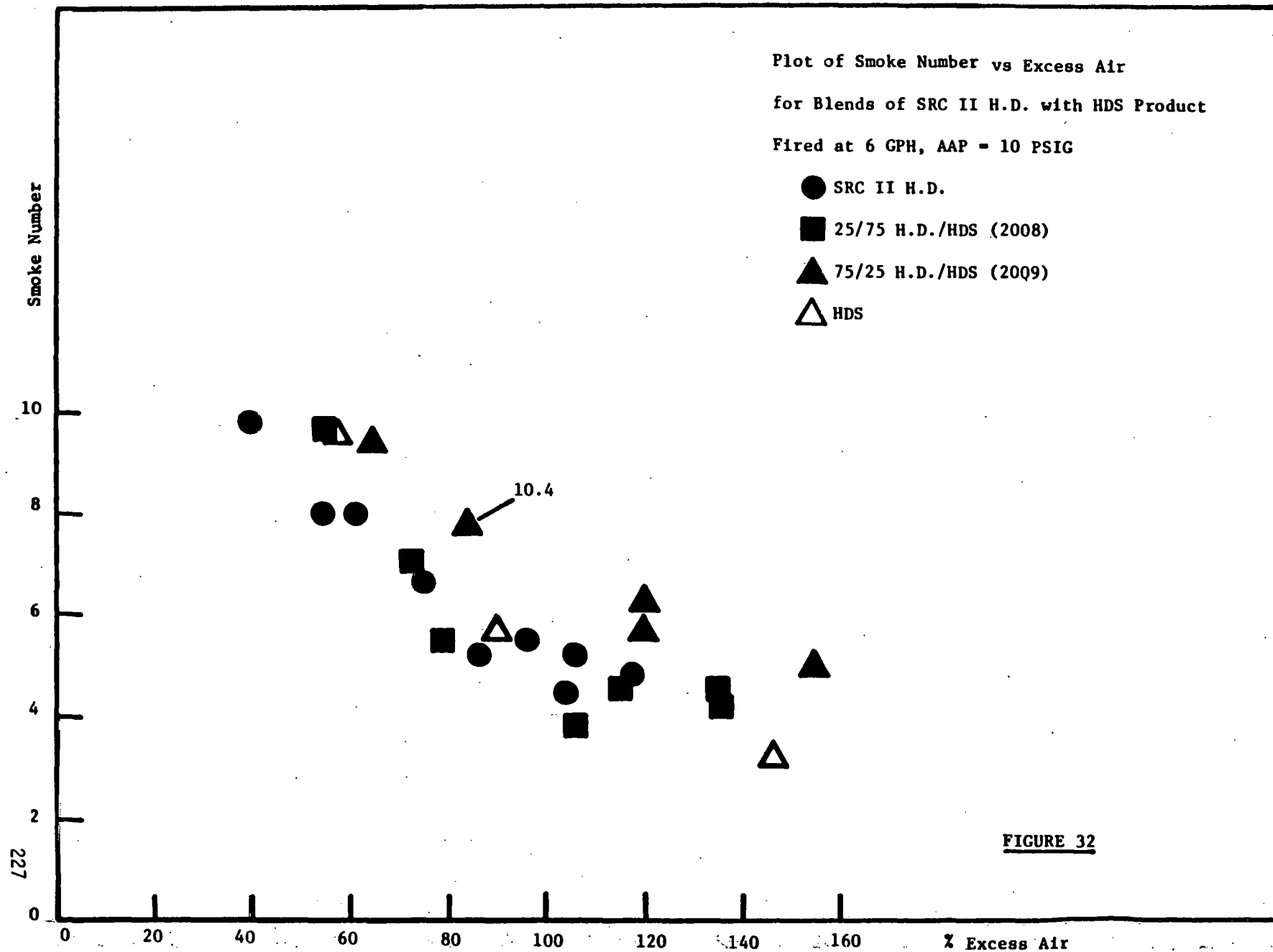


FIGURE 32

Plot of Smoke Number vs Excess Air
for Blends of SRC II H.D. with HDS Product
Fired at 6 GPH, AAP = 15 PSIG

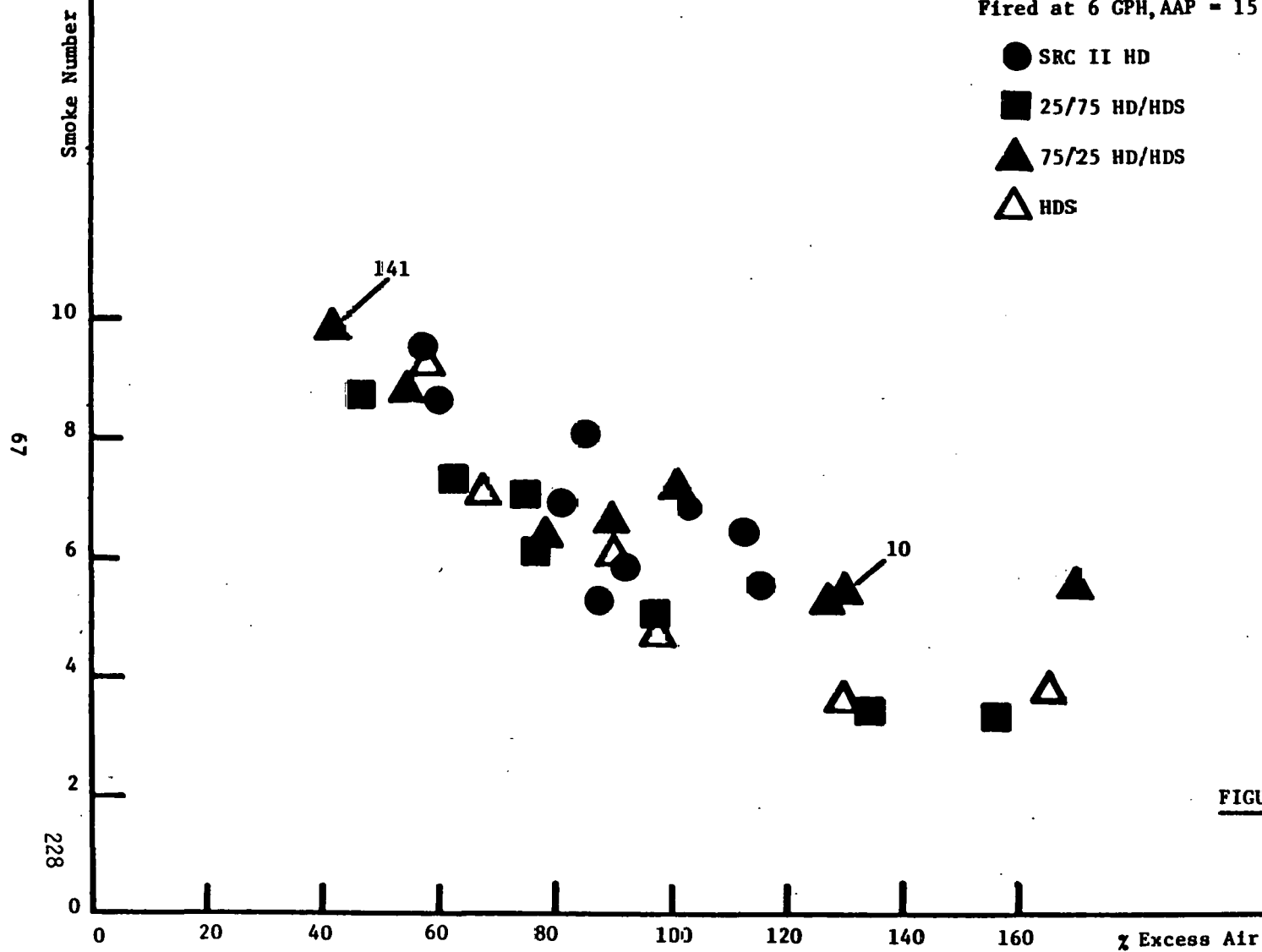


FIGURE 33

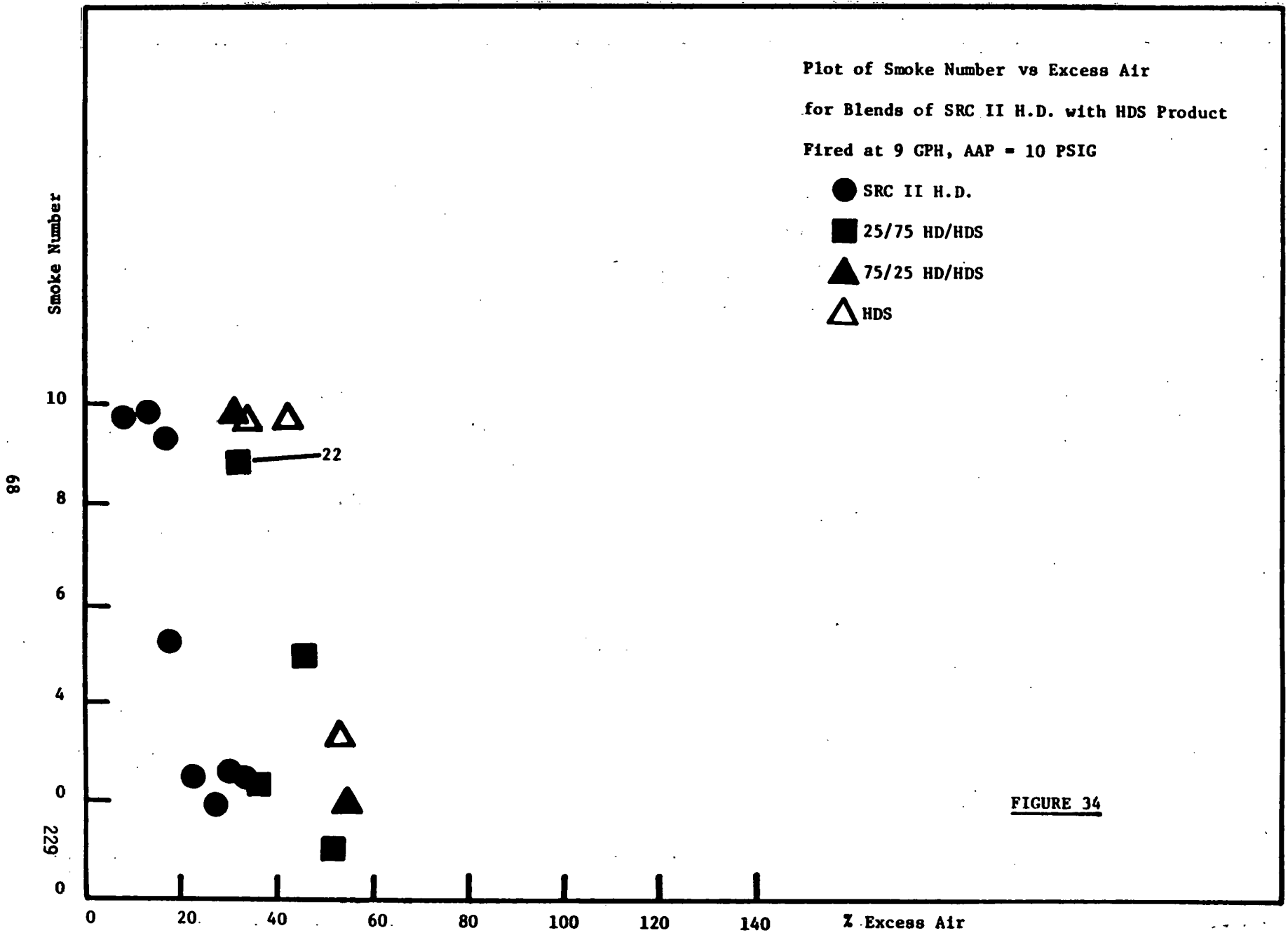


FIGURE 34

Plot of Smoke Number vs. Percent Excess Air

for Blends of SRC HD with HDS Fired at

9 GPH, AAP = 15 PSIG

● SRC II H.D.

■ 25/75 HD/HDS

▲ 75/25 HD/HDS

△ HCS

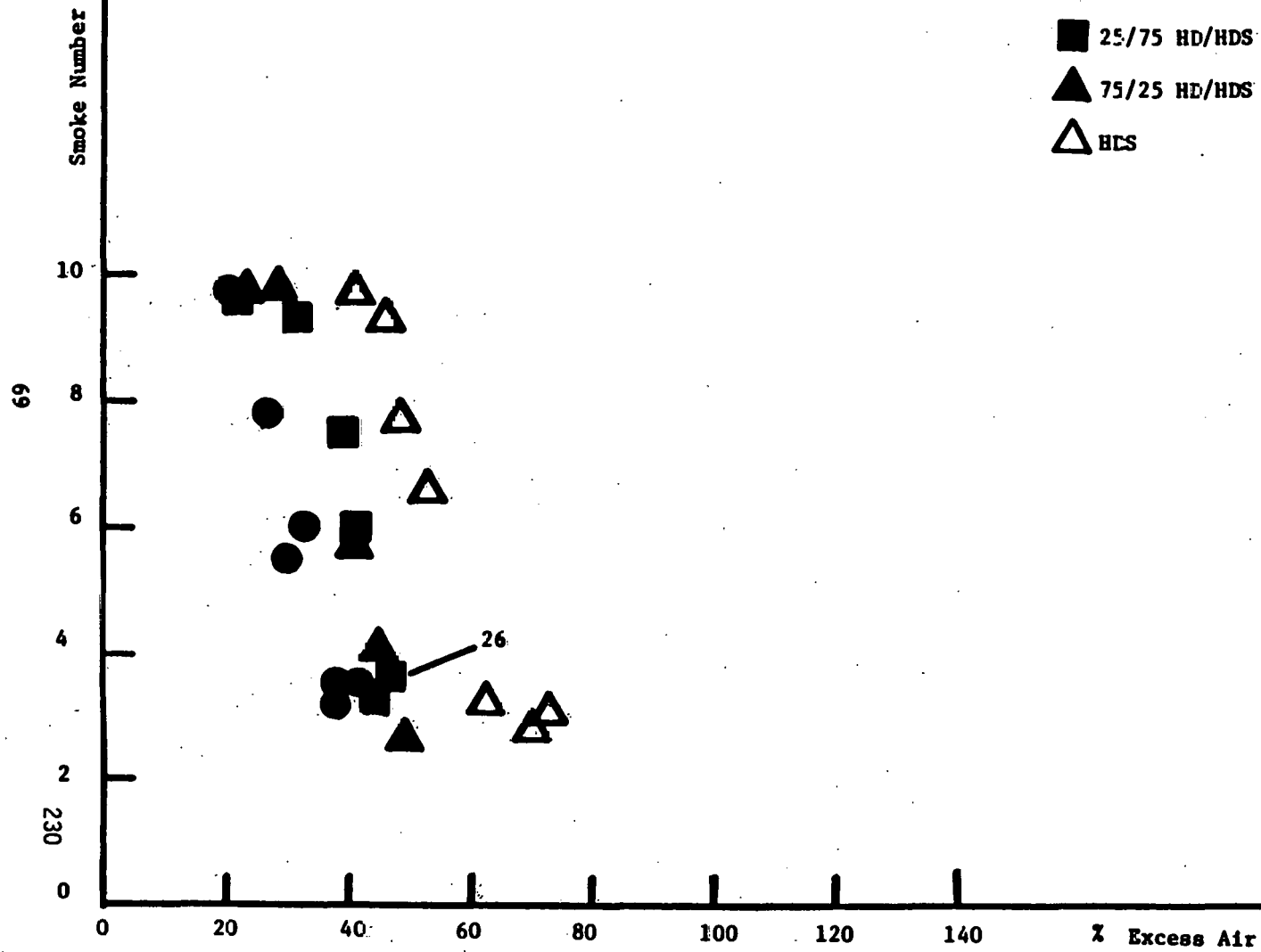
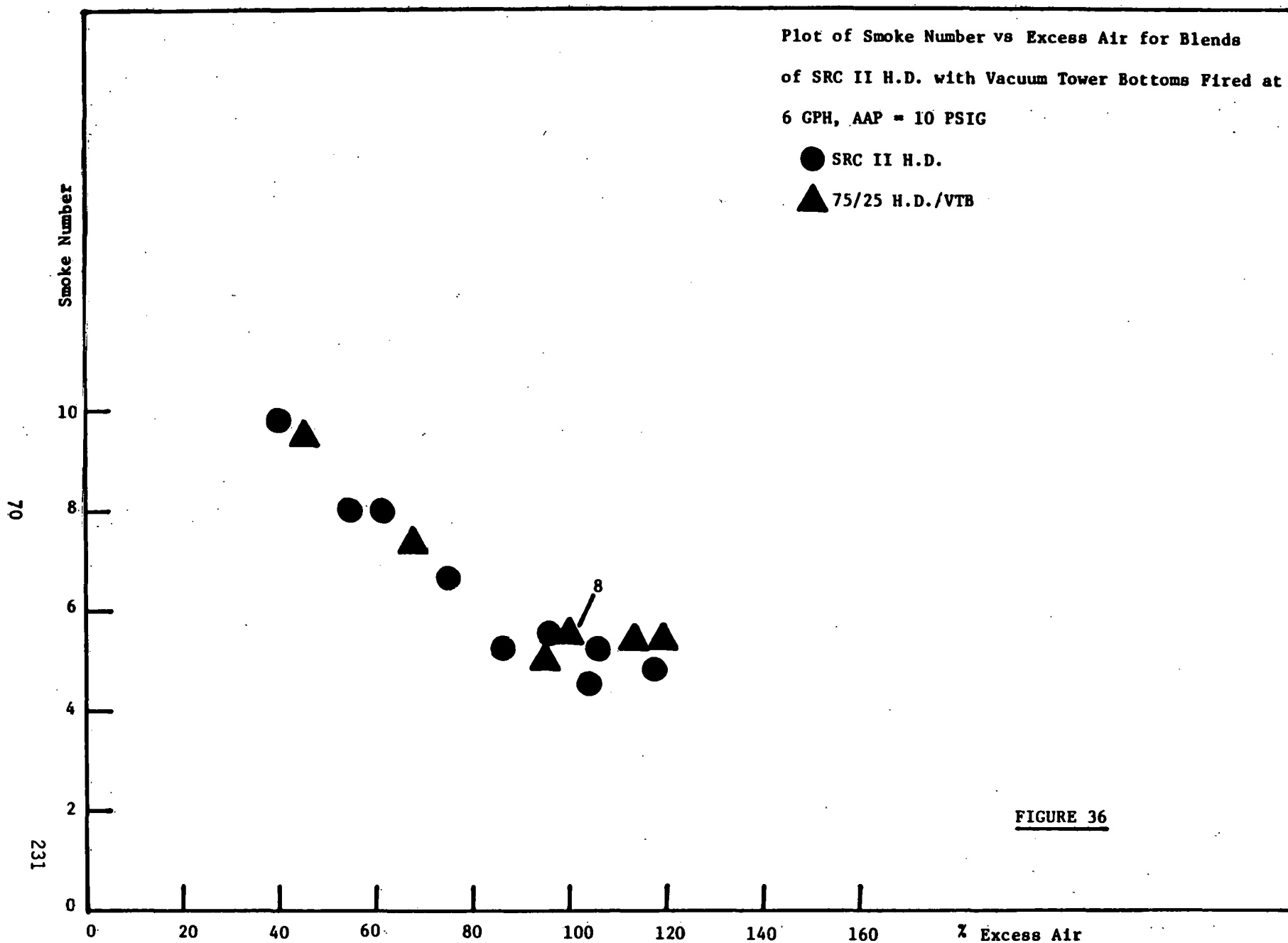


FIGURE 35



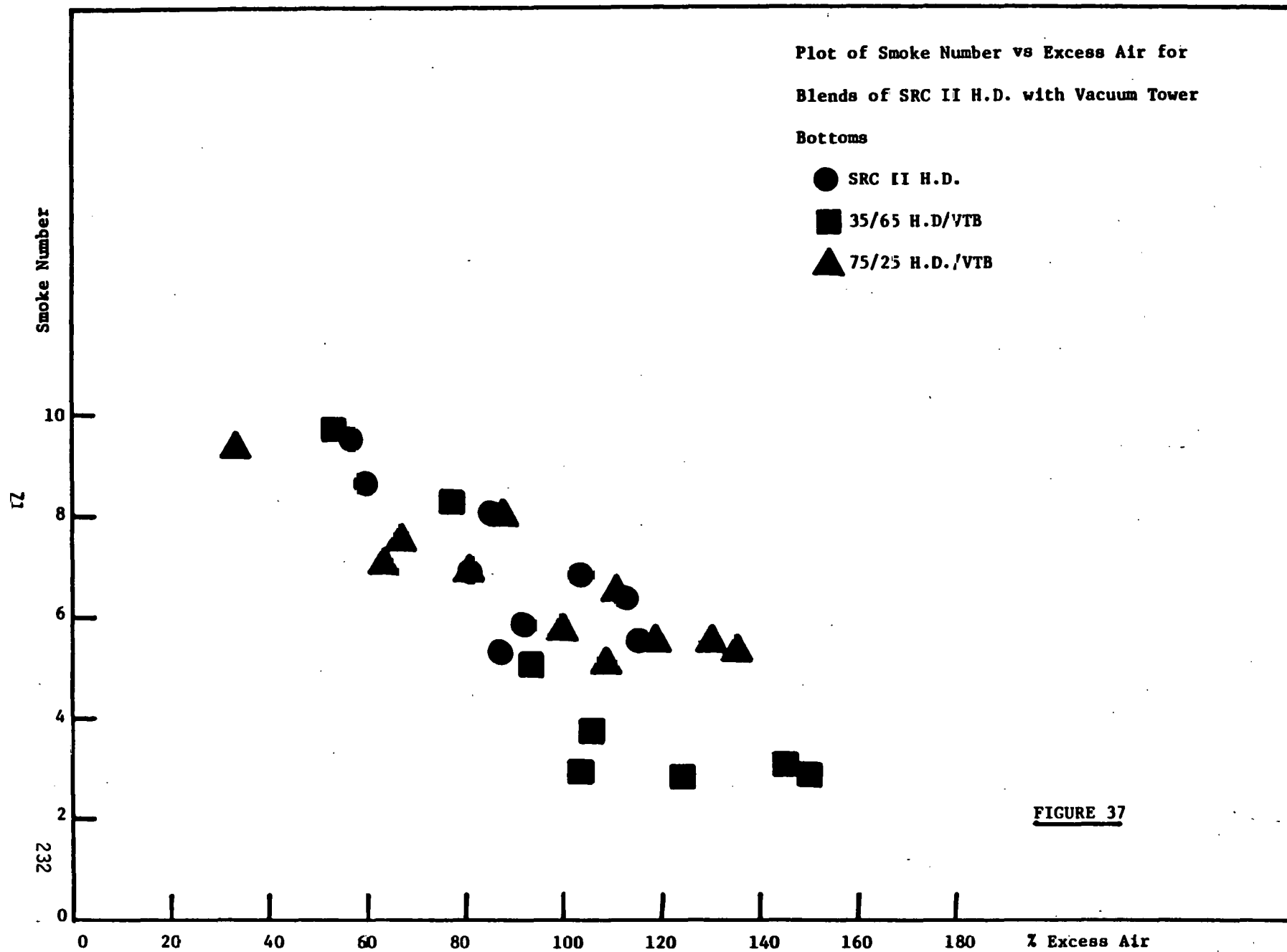


FIGURE 37

Plot of Smoke Number vs Excess Air
for 345°C Bottoms (SRC II)
at 6 GPH, AAP = 15 PSIG

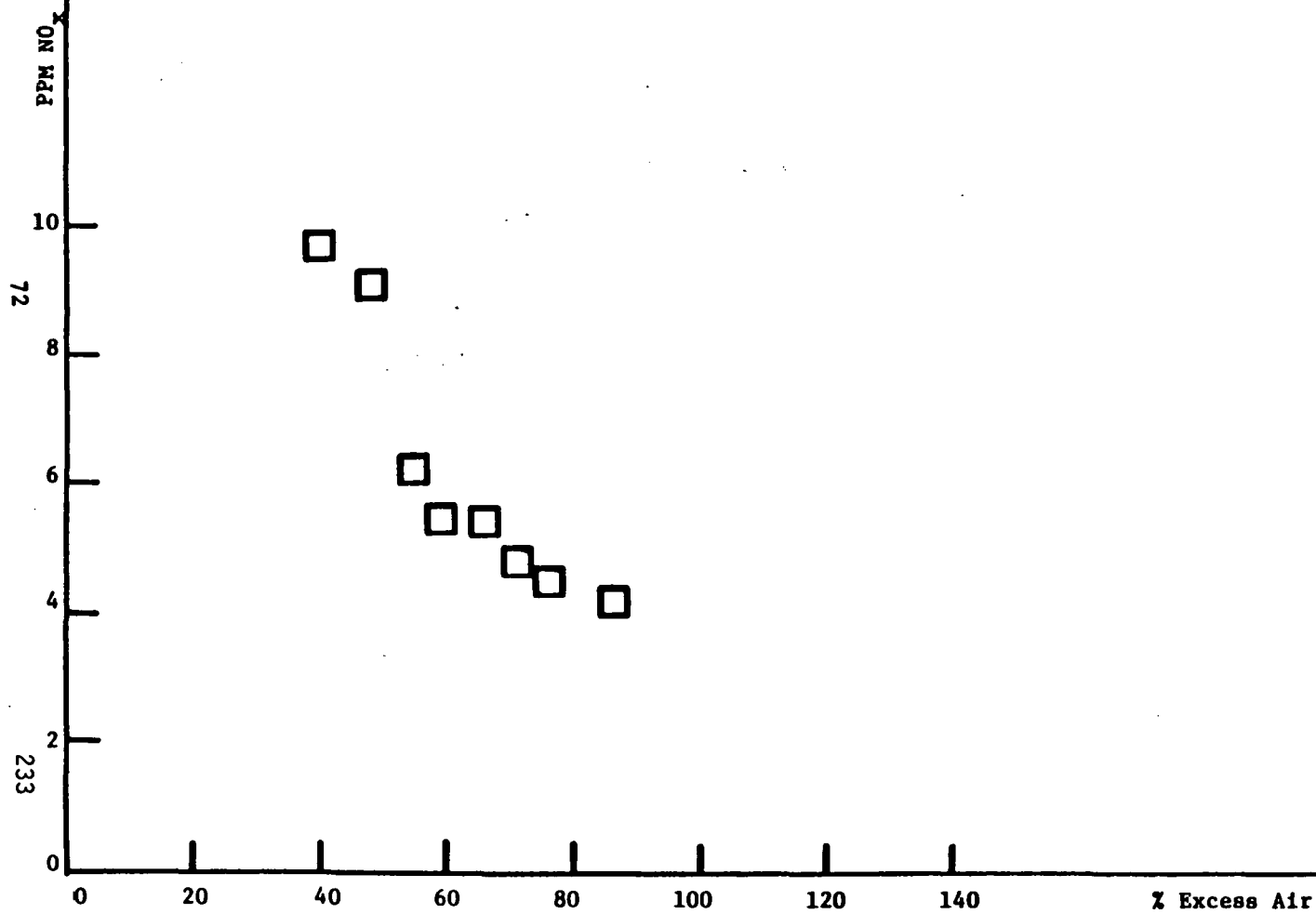


FIGURE 38

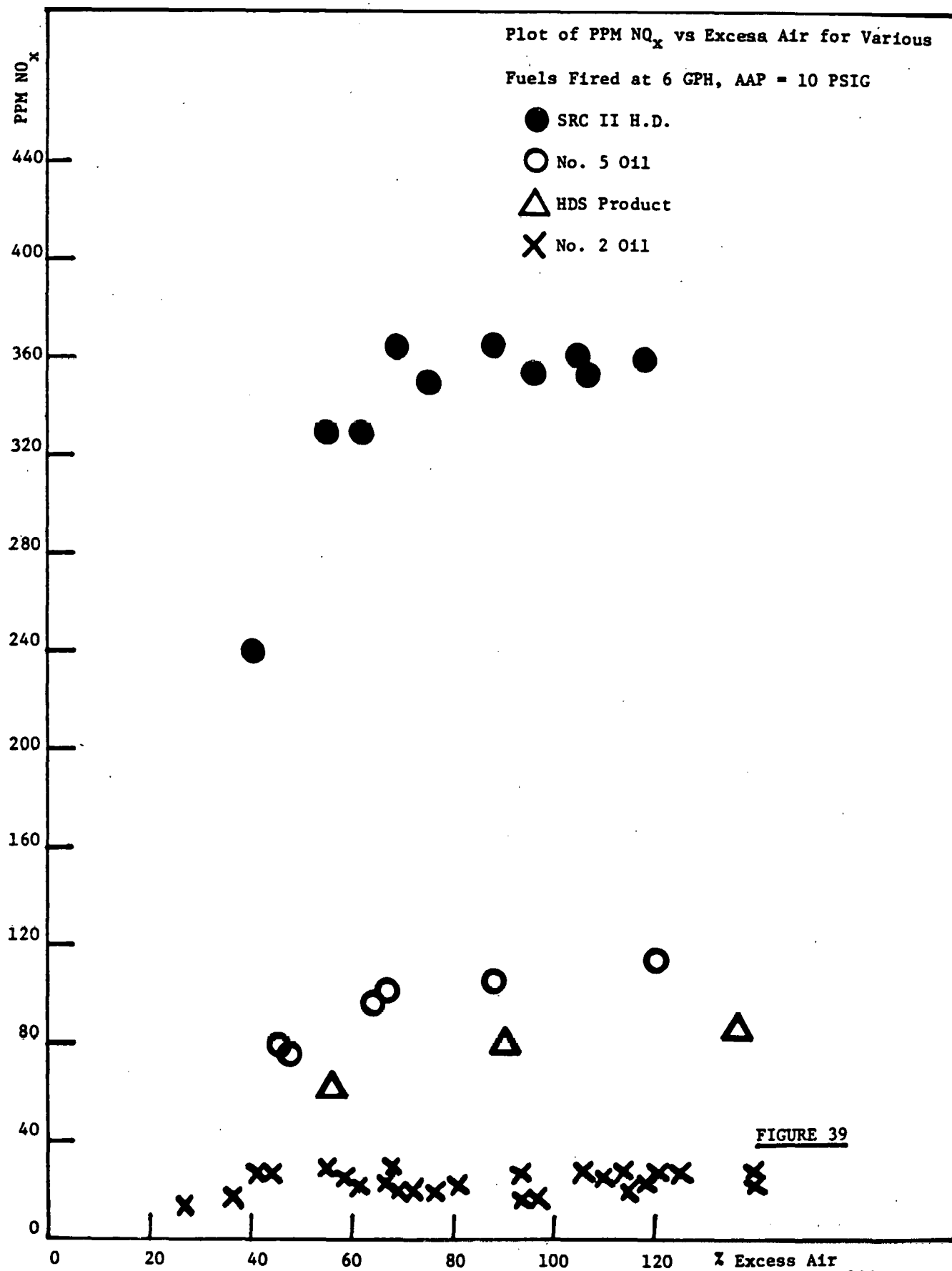


FIGURE 39

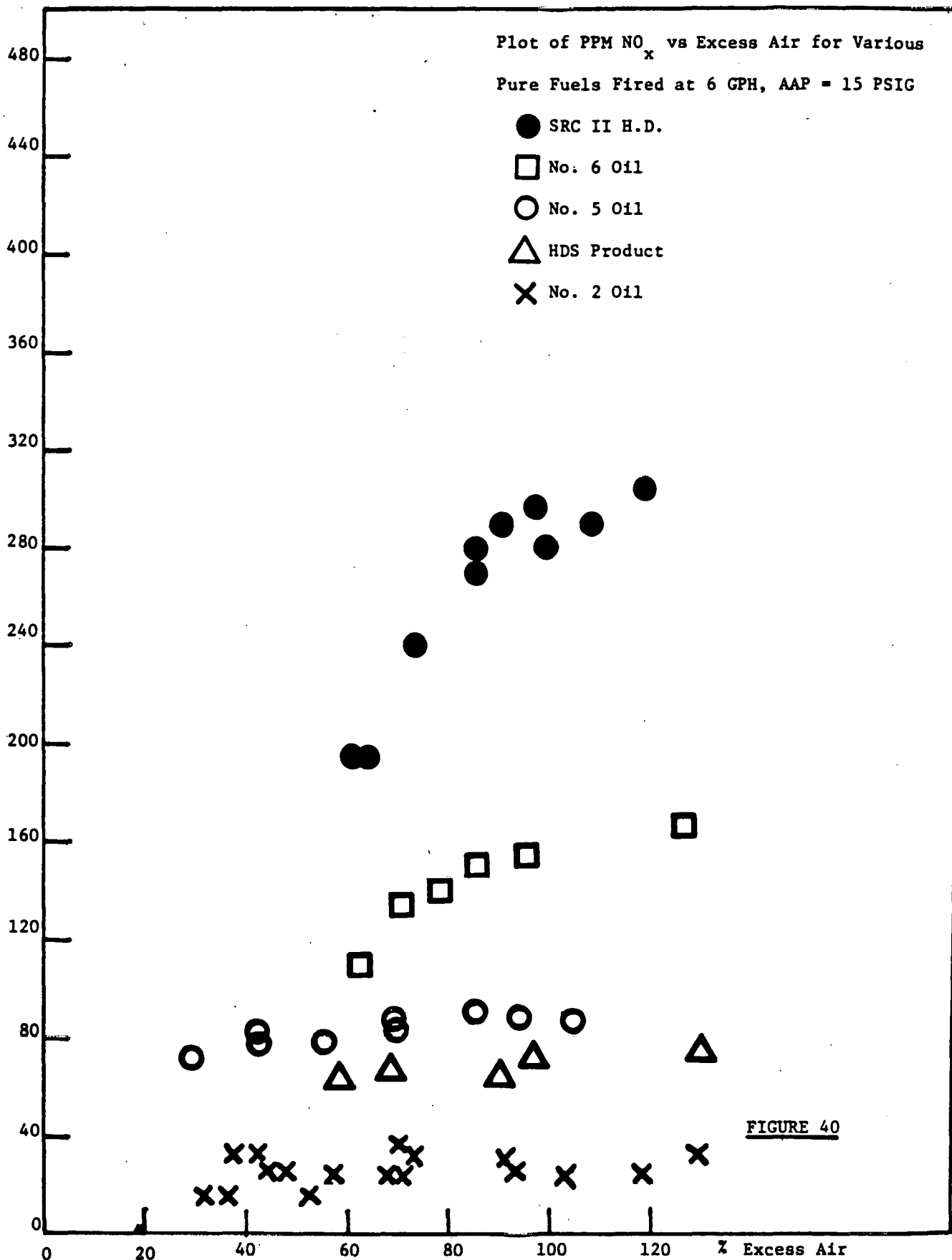


FIGURE 40

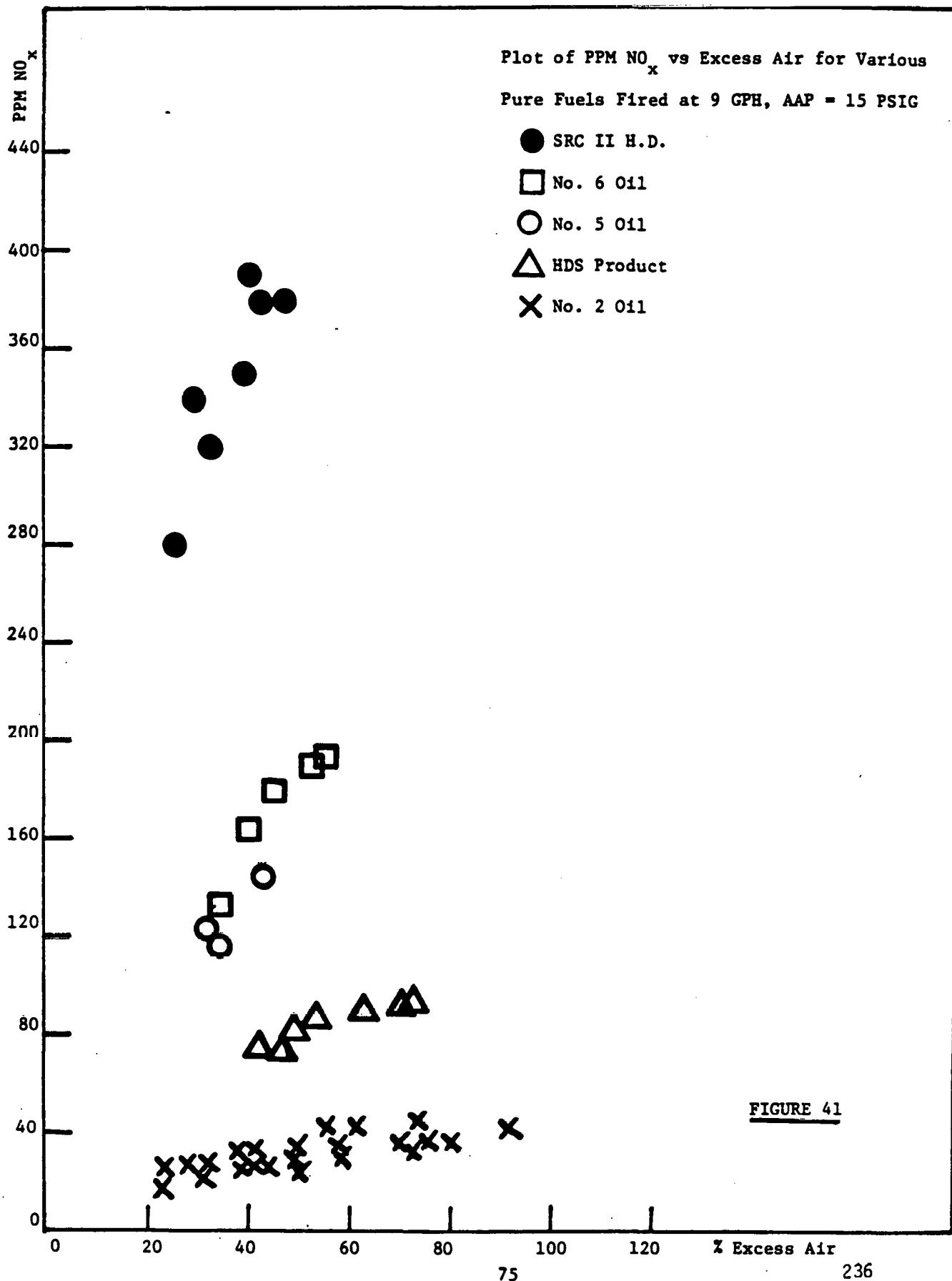


FIGURE 41

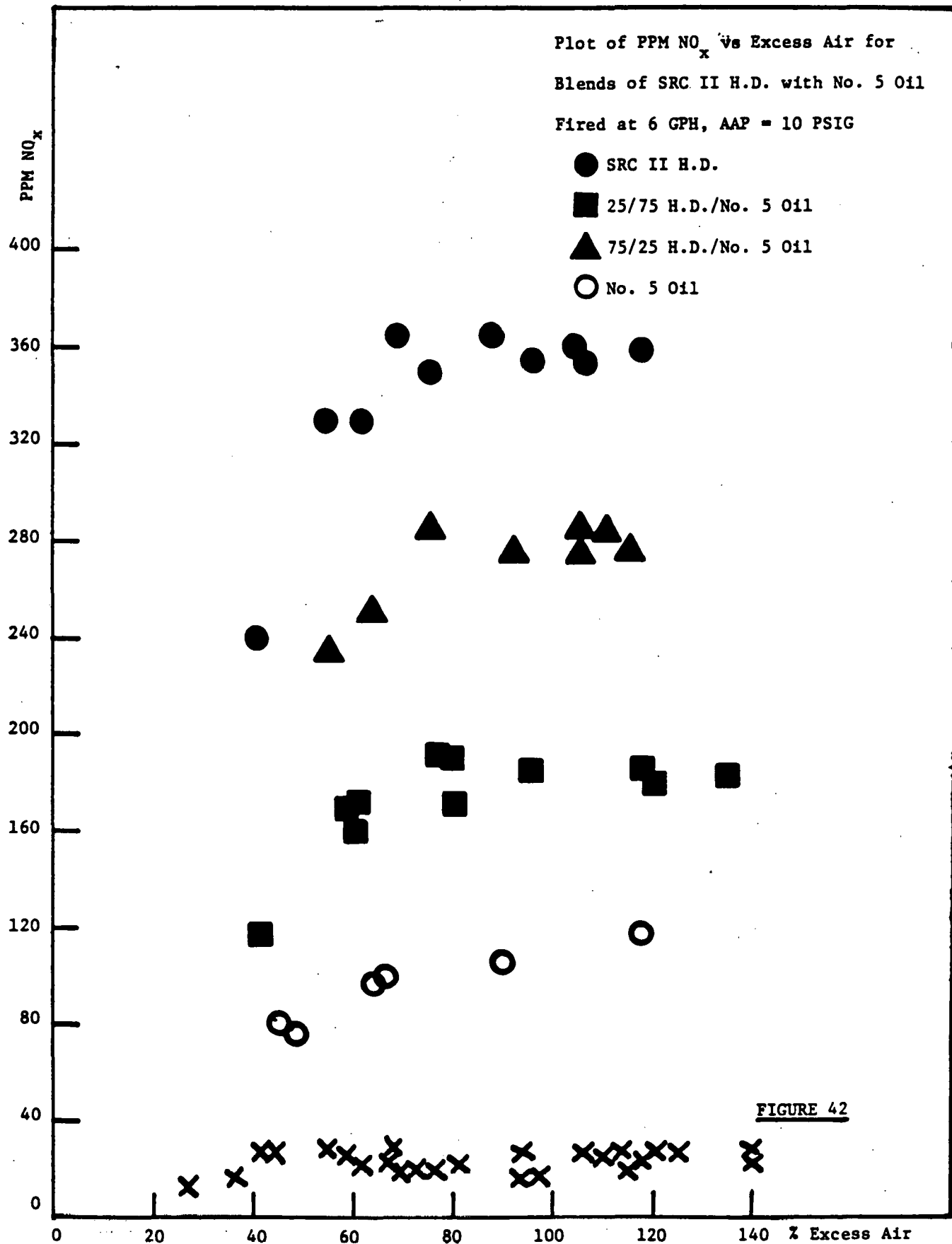


FIGURE 42

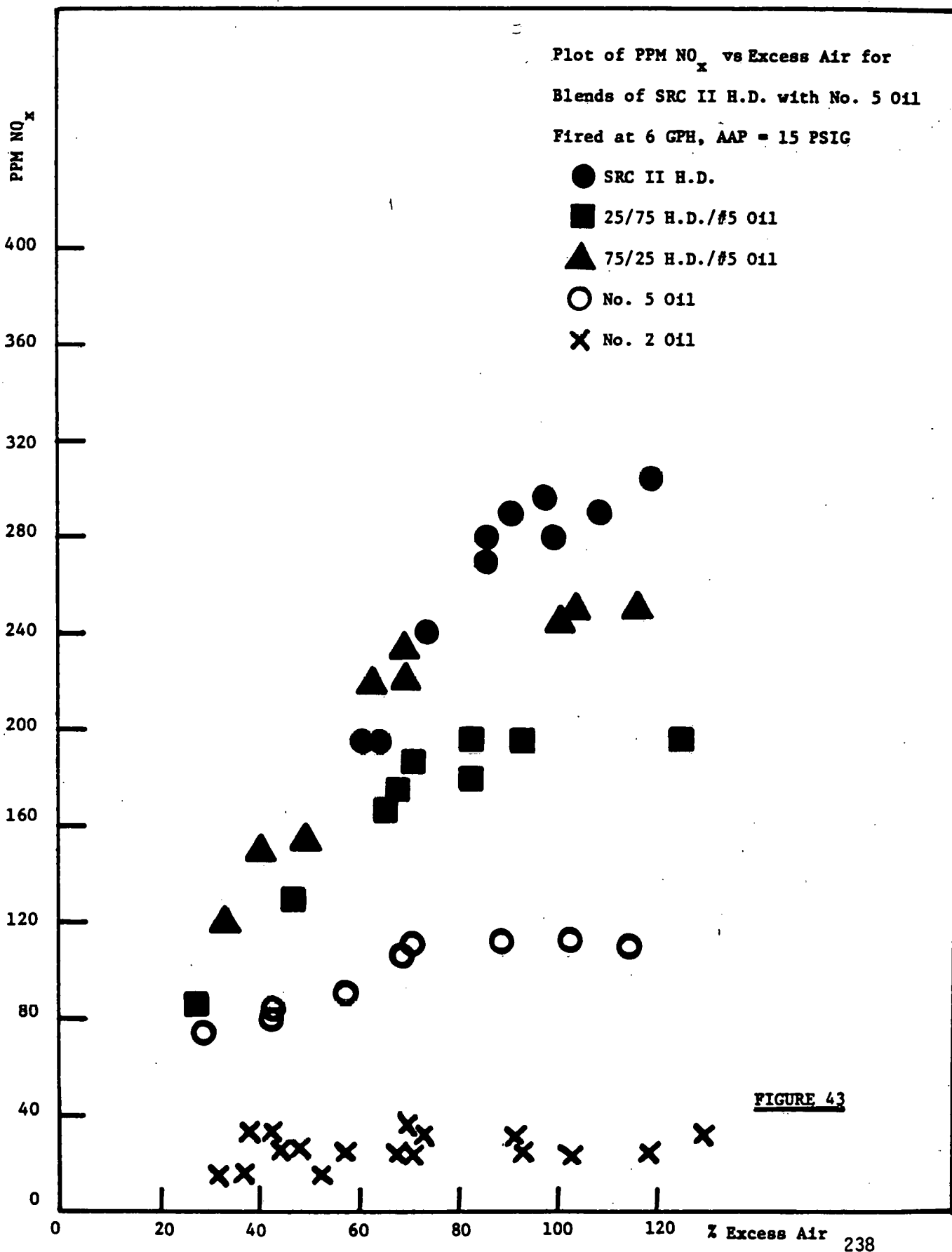


FIGURE 43

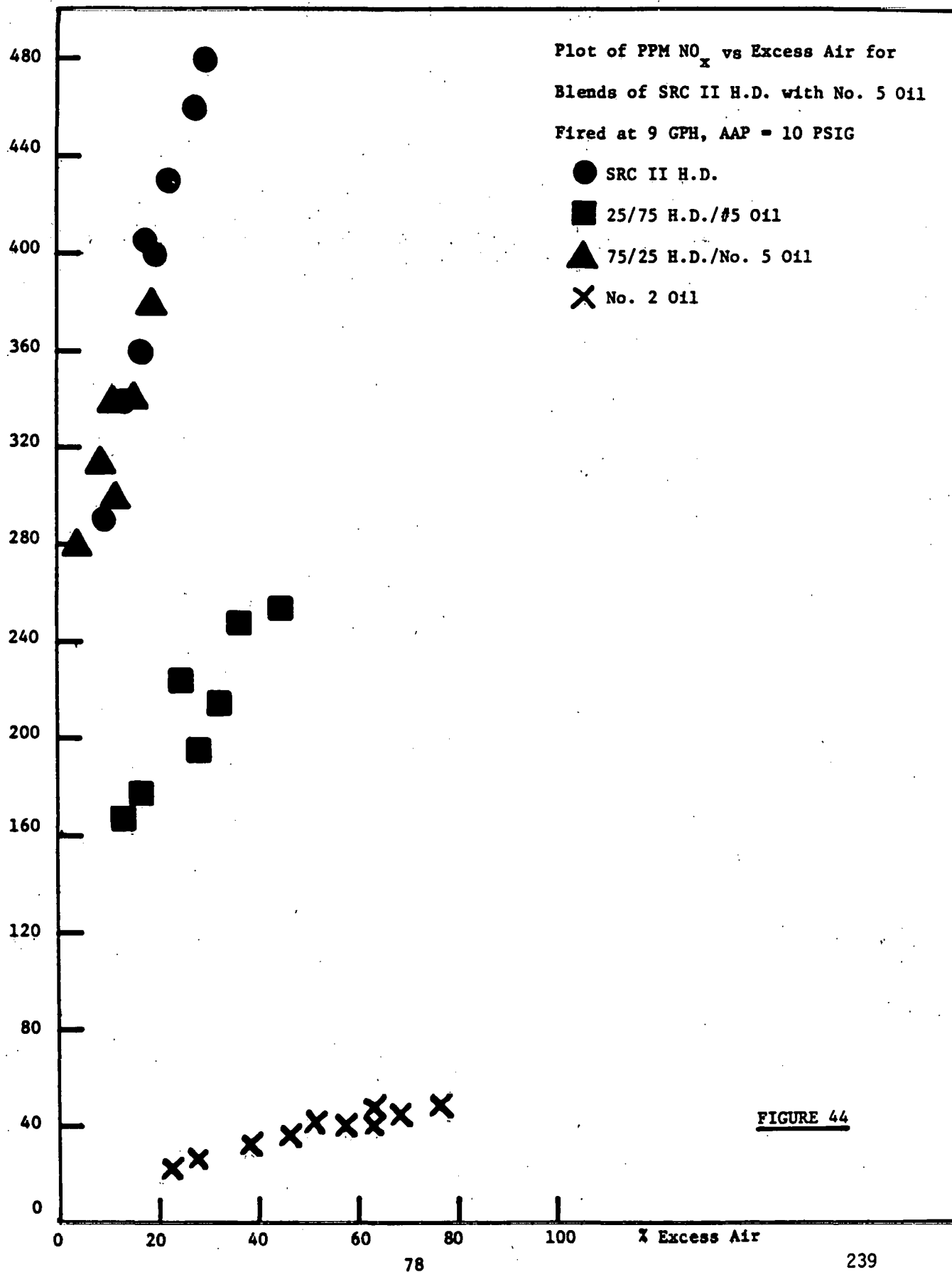


FIGURE 44

Plot of PPM NO_x vs Excess Air for
Blends of SRC II H.D. with No. 5 Oil
Fired at 9 GPH, AAP = 15 PSIG

- SRC II H.D.
- 25/75 H.D./No. 5 Oil
- ▲ 75/25 H.D./No. 5 Oil
- No. 5 Oil
- × No. 2 Oil

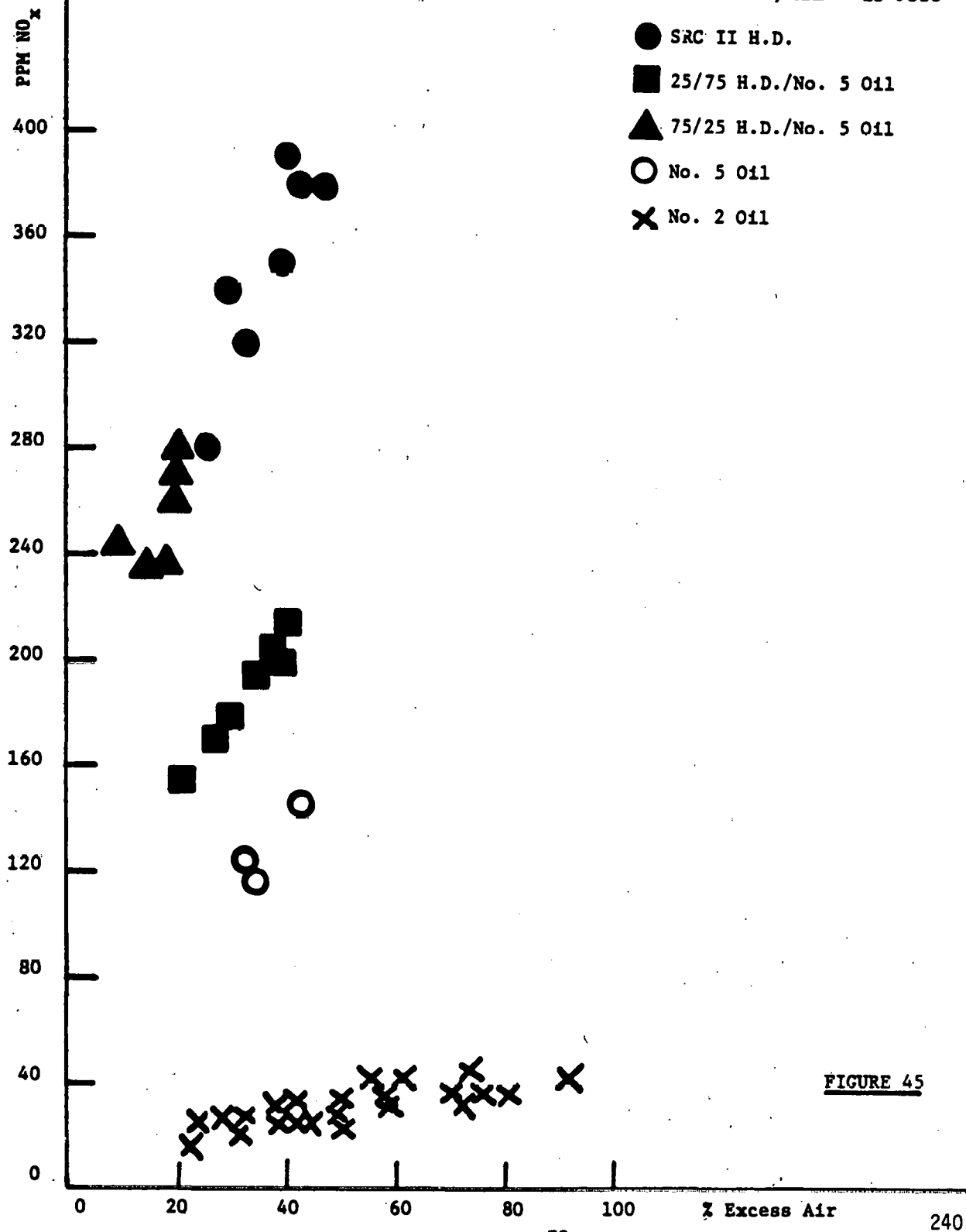


FIGURE 45

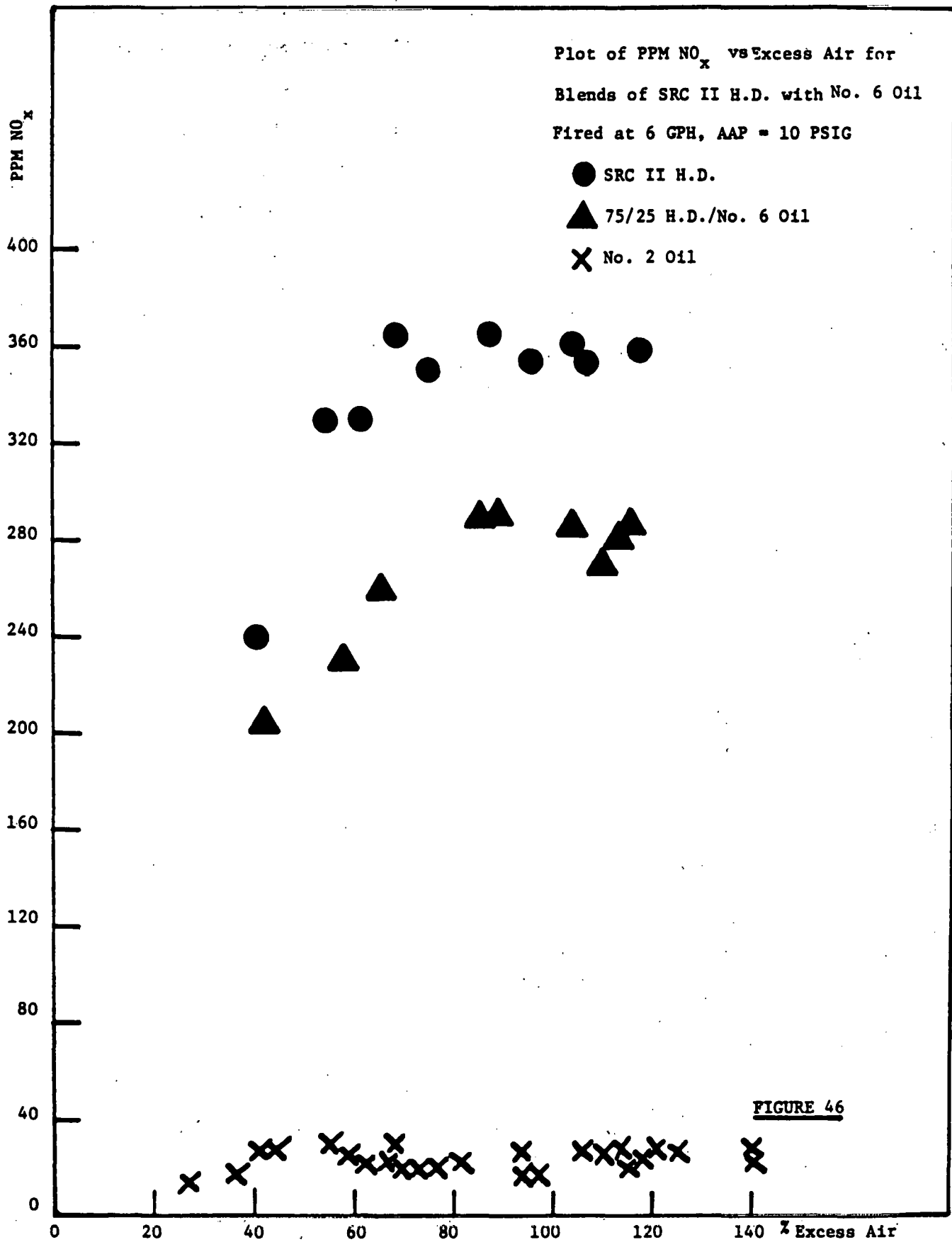


FIGURE 46

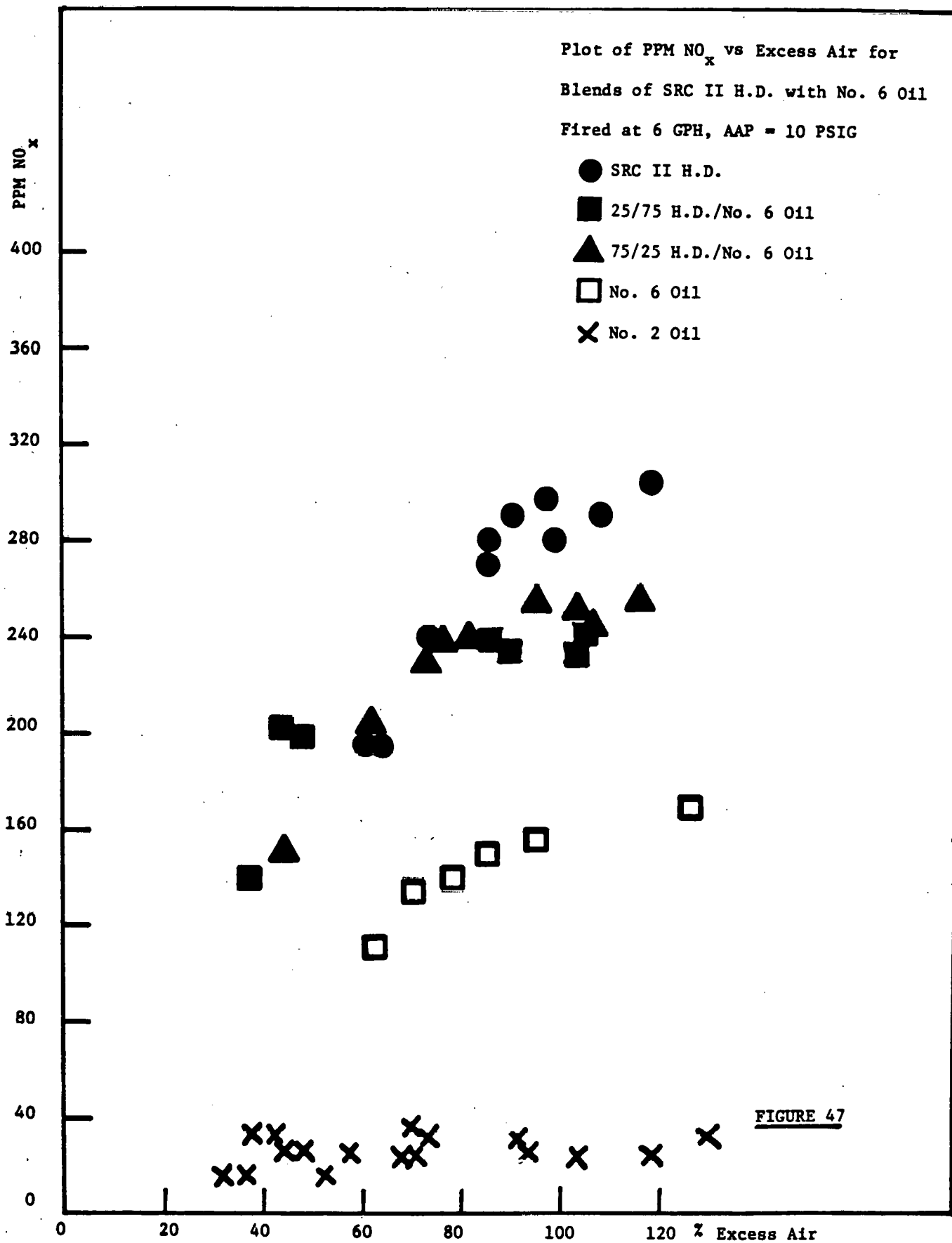


FIGURE 47

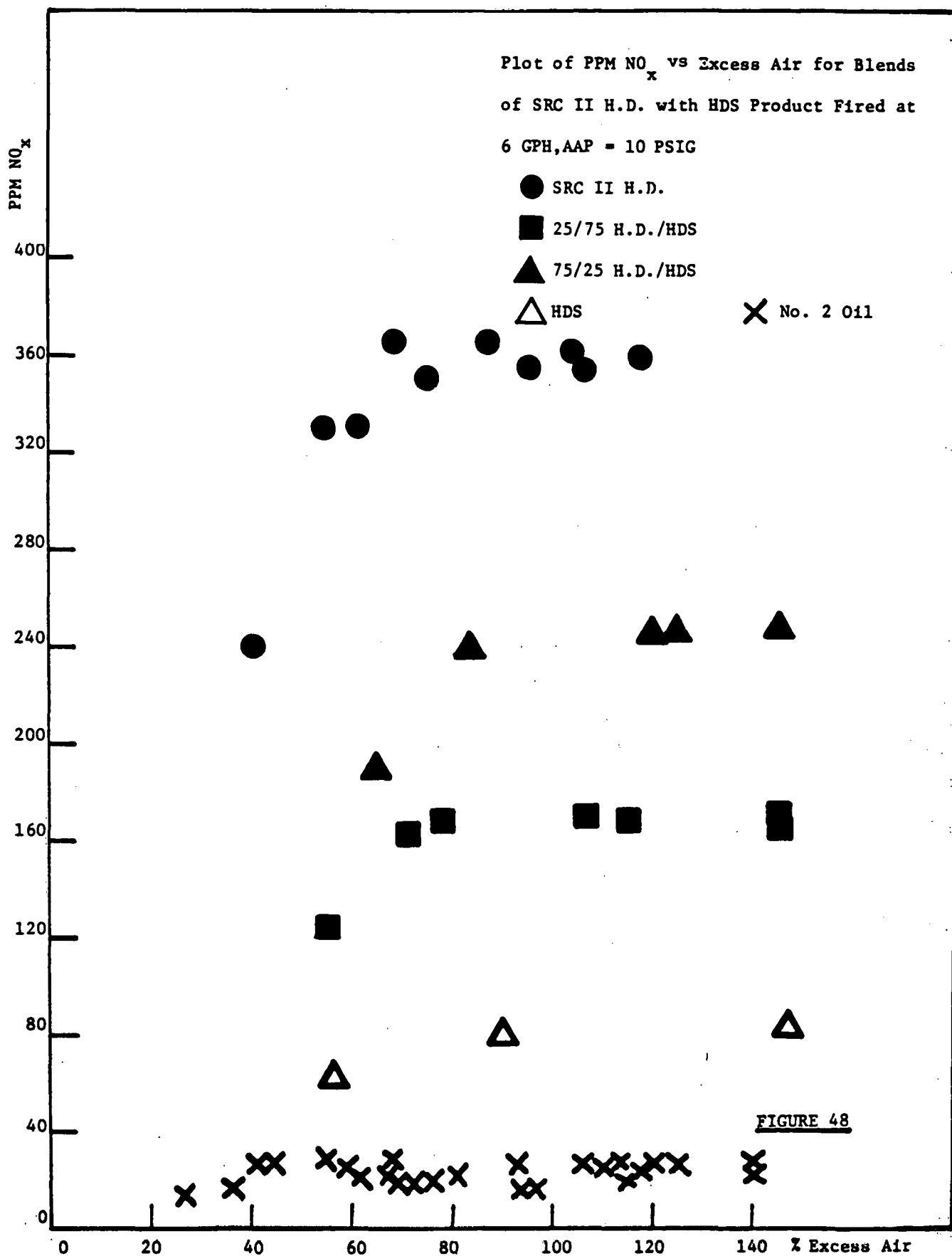


FIGURE 48

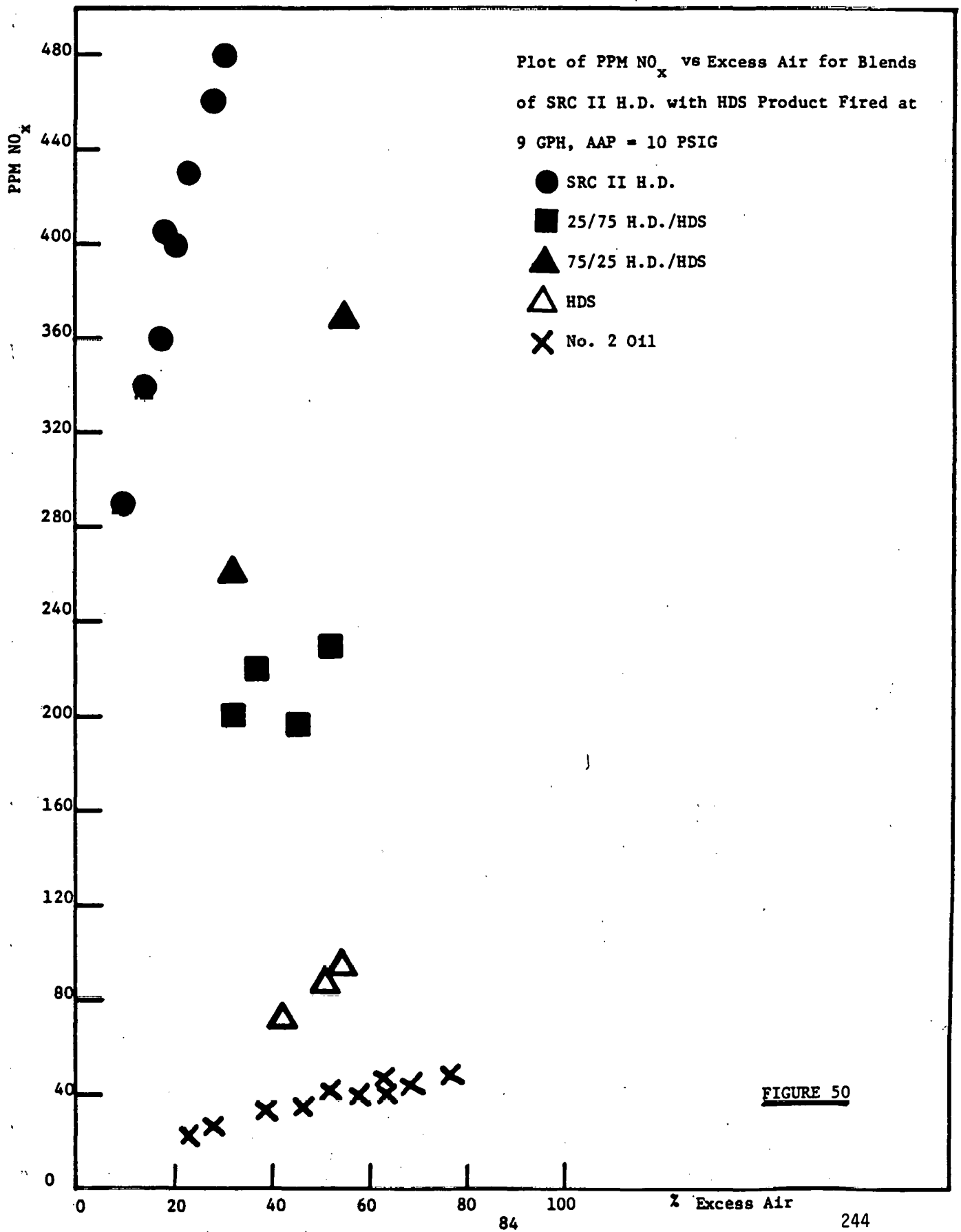


FIGURE 50

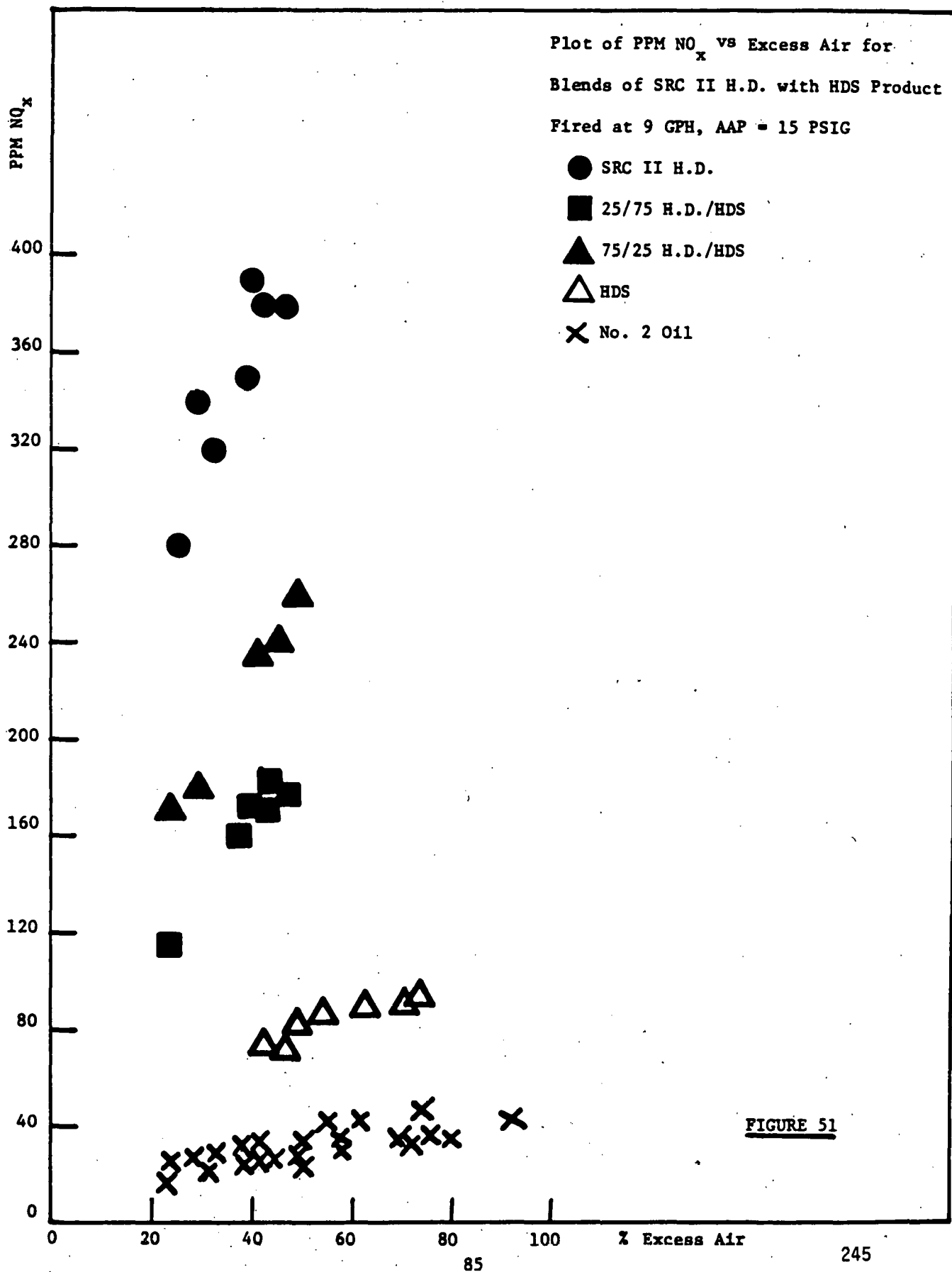


FIGURE 51

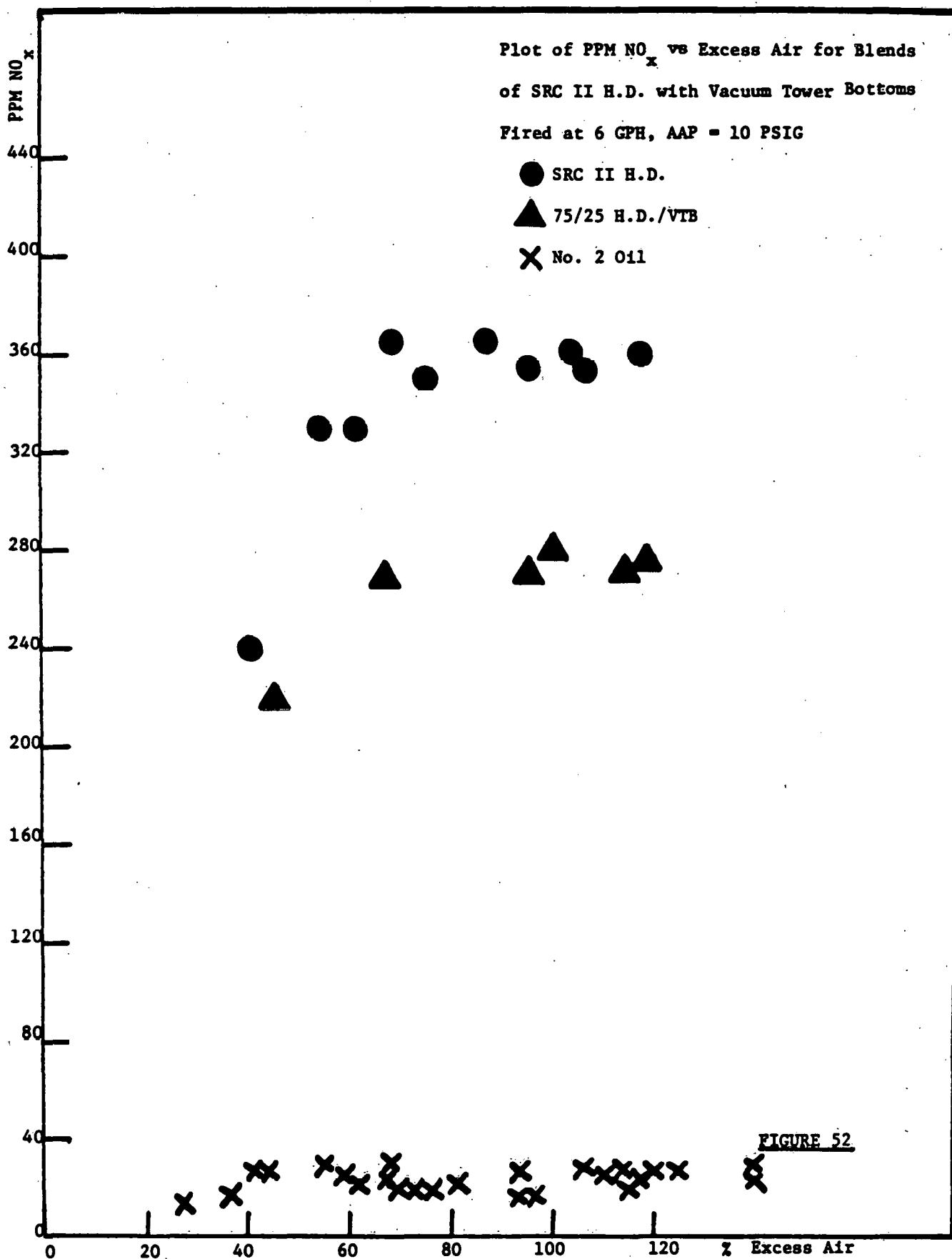


FIGURE 52

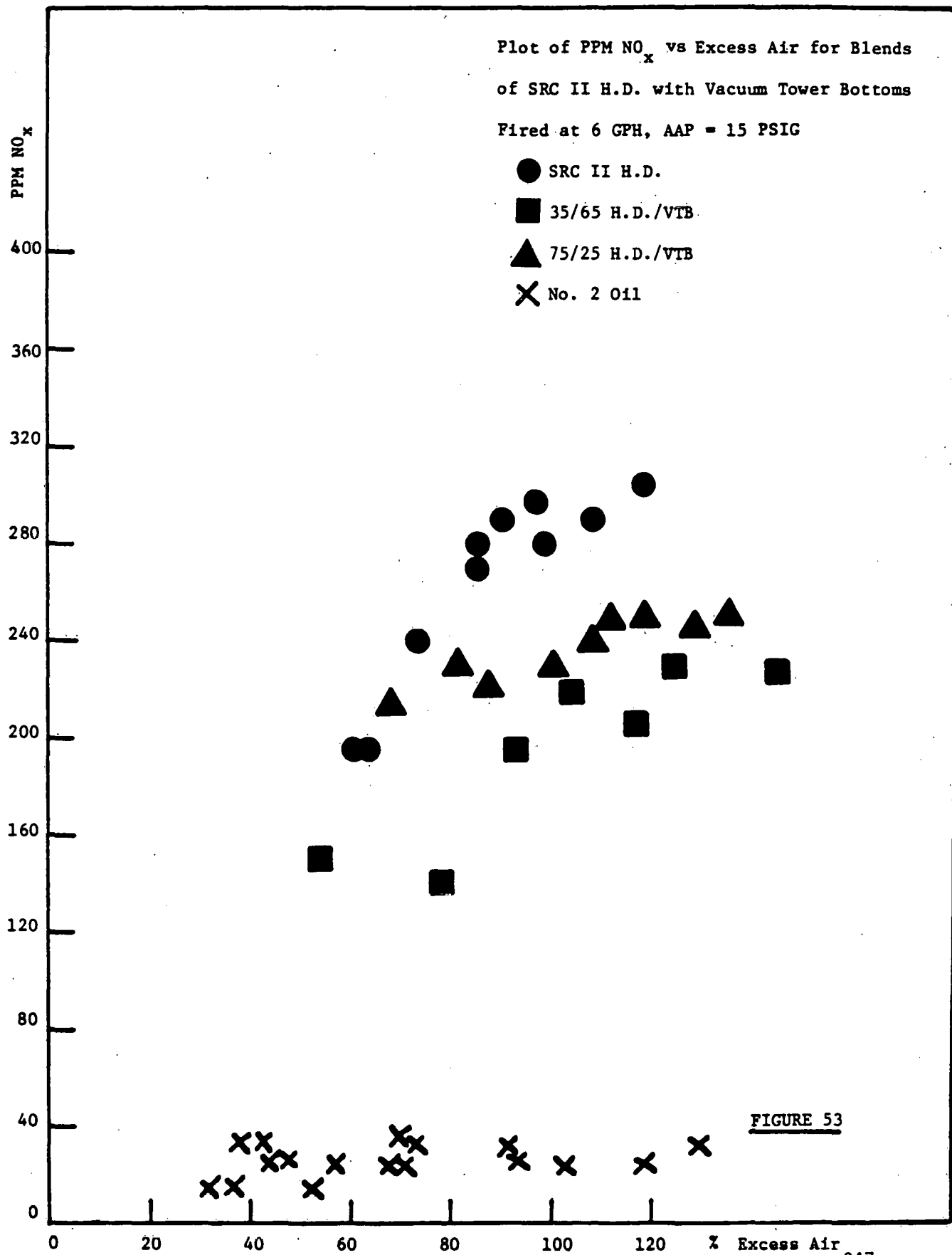


FIGURE 53

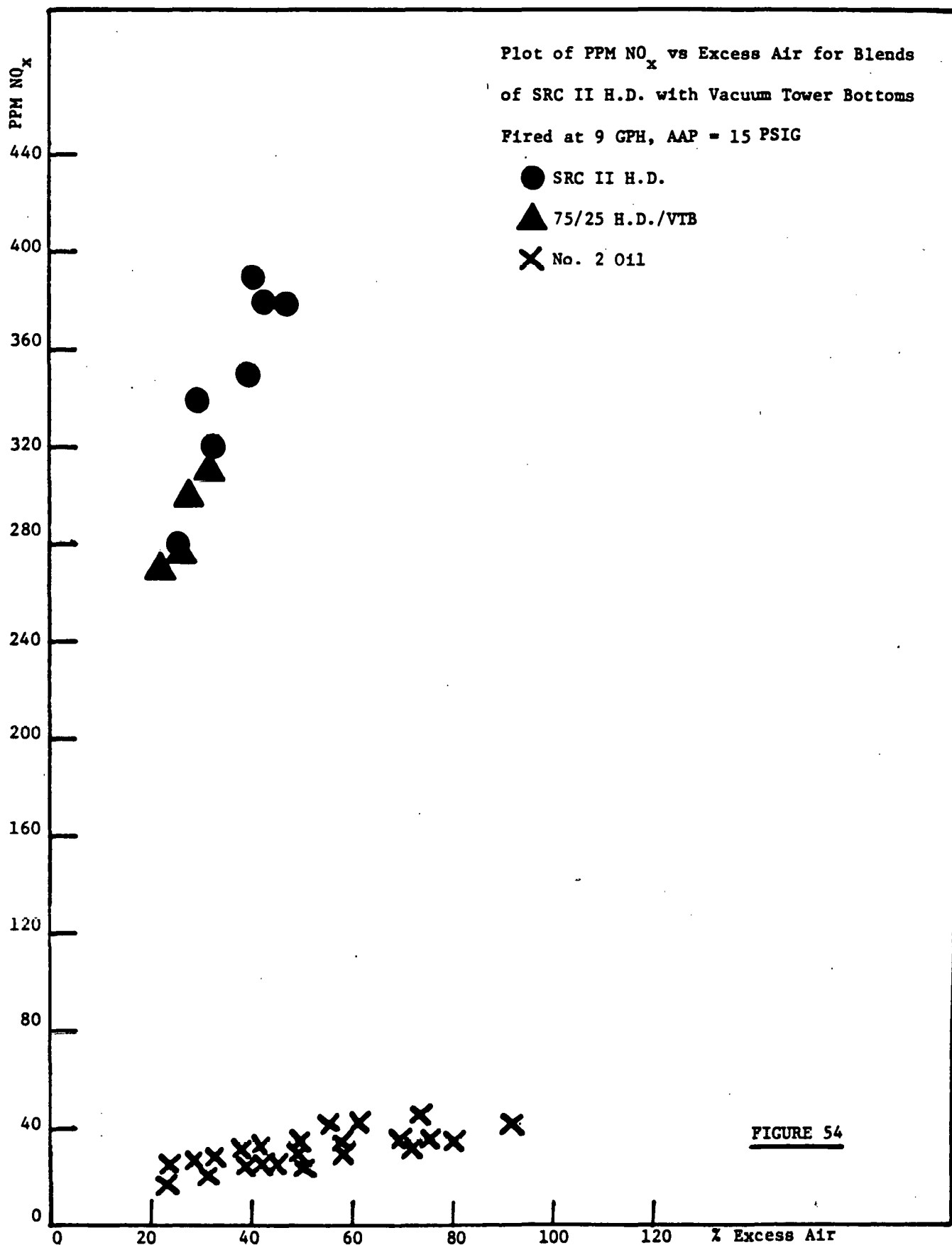


FIGURE 54

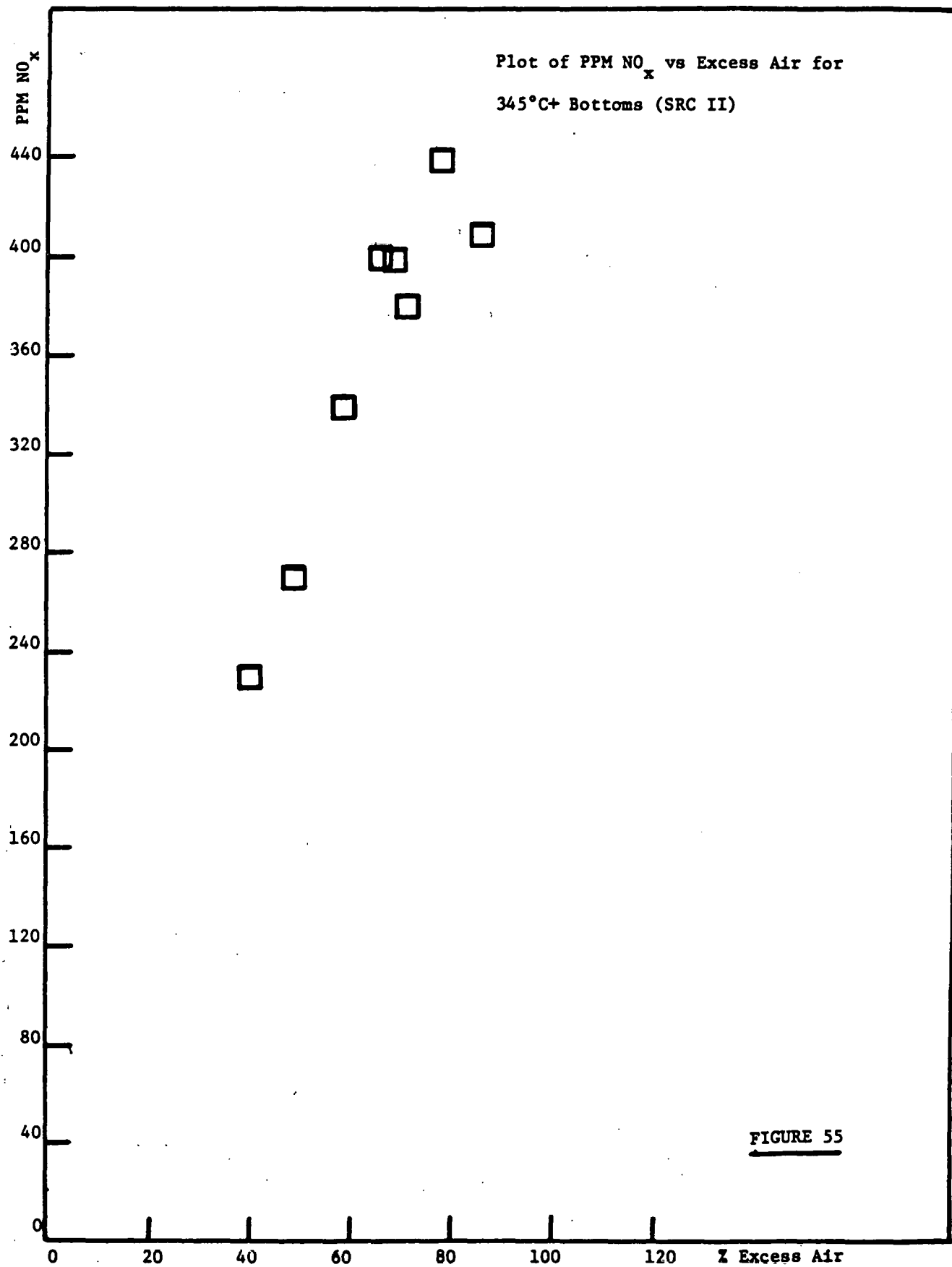


FIGURE 55

APPENDIX A

I. OAKMONT BOILER

The experimental set-up for the combustion tests of SRC-II M.D. consisted of the Oakmont boiler, a cast iron steam boiler Model S-86-OB manufactured by American Standard Division of American Radiator & Standard Sanitary Corporation. The boiler has a firebrick-lined firebox 15" x 11" x 14" high and is rated at 202 000 Btu/hr output. The burner used is a pressure atomizing Sun-Ray Model FC-134 with 0.5-2.0 gph capacity. It was operated with a 1 gph nozzle with 60° spray angle manufactured by Monarch Co. (Model F-80). Typical fuel atomizing pressures were 100 psig.

Samples of flue gas were taken via a 1/4" S.S. tube inserted into the flue just above the boiler section of the furnace. Also, at this level a Bacharach smoke sampling device (ASTM D2156) was installed.

II. PACIFIC BOILER

The boiler used for the combustion tests of SRC-II H.D. and blends of the H.D. with various heavy petroleum oils is a Model 3392 Pacific steel boiler. It has a capacity rating of 35 hp (at 10 gph input) and is equipped with two banks or passes of firetubes. The combustion chamber is firebrick-lined and the dimensions are 22"H x 25"W x 48"L.

The burner used for these tests is a low-pressure, air atomizing gun burner, Model AM4-CH HEV-E-Oil Burner manufactured by the Cleaver-Brooks Co. This unit is rated for a maximum 17.5 gph fuel flow rate and has a high-low modulating feature which controls both combustion air and fuel flow rate.

Flue gas samples and smoke samples (ASTM D2156) were again taken at a point immediately outside the boiler section of the unit.

APPENDIX B

Instrumentation

1) Stack Gas Analysis

Flue gases from both burners were pumped into a gas analysis cart which contained the pump, a condenser for removing water vapor from the gas stream, and the CO₂, CO, and O₂ analyzers.

The CO₂ and CO analyzers were LIRA Model 300 infrared analyzers. They were calibrated at the beginning and end of each test run and at several points during this run with a standard mixture of 13% CO₂ and 8% CO.

The oxygen analyzer was a Beckman Model 741 polarographic analyzer. It was calibrated with room air following the same procedure used for the CO and CO₂ analyzers, and zeroed using the CO₂ calibration gas.

NO_x emissions were measured using a Thermo-Electron Model 10 chemiluminescent analyzer. The analyzer was calibrated using a standard 430 ppm NO mixture and zeroed on room air several times during each run.

2) Soot and Particulate Measurements

As an indication of the sooting tendency of the various blends of fuels, a Bacharach smoke number sampler (ASTM D2156) was used. A calibrated reflectance meter was used to determine the smoke number from the sample filters.

Particulate sampling was accomplished using the B-P probe apparatus which samples particles from the flue gases isokinetically. This apparatus was developed at the B-P Research Center in England and was manufactured in France.

Flue gases enter the mouthpiece, which is located in the center of the stack. The gases flow through the filter where all soot emissions are trapped. The gases then pass through the finned condenser and ice-bath condenser to remove the major portion of entrained water vapor and are further dried by passing through a column of calcium sulfate. Finally, the gases pass through the rotometer and dry gas meter to the pump.

The regulator valve is used to maintain constant gas flow through the probe as indicated by the constant rotometer reading. The vacuum gauge and thermometer are used in conjunction with the gas meter to calculate total gas flow through the problem.

Before each test run, the probe and filter are cleaned and the filter media (quartz fiber, Gelman Type A) changed. The cyclone and filter are thoroughly dried in an oven for about an hour at 140°C before being placed in a desiccator to cool prior to weighing. A precision balance is used to obtain weights to the nearest 0.1 milligram. This same drying and weighing procedure is followed at the end of each run to determine the weight of trapped soot emissions. The particulate loadings in $\mu\text{g}/\text{liter}$ are entered on the relevant smoke number plots.