Catalysts for hydrotreating solvent refined coal (SRC-II)
by Ronald James Ramer

A thesis submitted in partial fulfillment of the requirements for the degree of MASTER OF SCIENCE in Chemical Engineering
Montana State University
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Abstract:
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A coking reactor seems to reduce catalyst deactivation for nitrogen removal. Nitrogen removal was found to be greatest for run times up to two hours.

Catalyst 3 (12.6% MoO_3, 10.3% WO_3, 2.8% NiO, 6.8% CoO) was the best catalyst in removing nitrogen and sulfur. The average pore diameter of the base (Norton 6176) is 152 Å. It removed nearly 90% of the nitrogen and 78% of the sulfur over a two-hour period. This represents a final sample with 0.12 weight percent nitrogen and 0.16 weight percent sulfur. Distillations done on two hour runs produced a 95-96% distillate yield under 700°F.

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Signature Ronald J Ramos

Date August 3, 1979

Patent Applied For.

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by

RONALD JAMES RAMER

A thesis submitted in partial fulfillment
of the requirements for the degree

of

MASTER OF SCIENCE

in

Chemical Engineering

Approved:

Lloyd Berg
Chairperson, Graduate Committee

R. L. Nickelson
Head, Major Department

Henry L Parsons
Graduate Dean

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ABSTRACT

Solvent Refined Coal (SRC-II) from Pittsburg and Midway Coal Mining Company's Fort Lewis Washington pilot plant was hydrotreated with commercial and Montana State University fabricated catalysts. Twenty-two trickle bed reactor tests and ten batch autoclave tests were executed.

The liquid products obtained from these tests were all analyzed for nitrogen content. Periodic samples were tested for sulfur content and distillate yields were measured whenever possible.

A coking reactor seems to reduce catalyst deactivation for nitrogen removal. Nitrogen removal was found to be greatest for run times up to two hours.

Catalyst 3 (12.6% MoO_3, 10.3% WO_3, 2.8% NiO, 6.8% CoO) was the best catalyst in removing nitrogen and sulfur. The average pore diameter of the base (Norton 6176) is 152 Å. It removed nearly 90% of the nitrogen and 78% of the sulfur over a two-hour period. This represents a final sample with 0.12 weight percent nitrogen and 0.16 weight percent sulfur. Distillations done on two hour runs produced a 95-96% distillate yield under 700°F.

INTRODUCTION

As the United States heads toward the twenty-first century, its demand for energy grows(1). As crude oil supplies decrease while prices increase, alternate sources of power become more and more lucrative(2). As the existing proposed solutions to this problem are studied, coal seems to be the most promising, immediate, solution.

It is estimated that coal accounts for about 80 percent of the total energy from oil, coal, and gas in the United States(1). Much of this coal would be relatively easy to obtain, as it lies in relatively thick beds under less than 1000 feet of overburden(3). Because the transportation system of the United States is almost exclusively dependent on liquid fuels, converting coal to a liquid fuel becomes most favorable(4,5).

Coal liquefaction processes are being looked into: Solvent Refined Coal (SRC), Exxon Donor Solvent (EDS), H-Coal, and Synthoil are a few examples. But, in the United States, each of these is merely in the development stage. Coal processes require large capital investment, abundant water, and have environmental and social problems (6). Along with these problems, coal is not of consistent

composition ranging from sub-bituminous and bituminous, to anthracite. Therefore, a strong energy policy must be taken now, otherwise coal liquefaction processes will not be economically competitive by the year 2000(7)!

This research is an attempt to upgrade the tar-like material obtained from the modified PAMCO SRC process, (SRC-II). The SRC-II is catalytically hydrotreated to remove unfavorable heteroatomic molecules (sulfur and nitrogen specifically) and improve the overall product. The reasons for the removal is to prevent catalyst poisoning in further refining steps and to reduce pollution effects of any final fuel made from the SRC-II.

BACKGROUND

SRC Process

Of importance to this work is the SRC process operated by Pittsburg and Midway Coal Mining Company. A 50 ton per day pilot plant is operated at Fort Lewis, Washington. Figure 1 shows a schematic diagram of the SRC-I process(8).

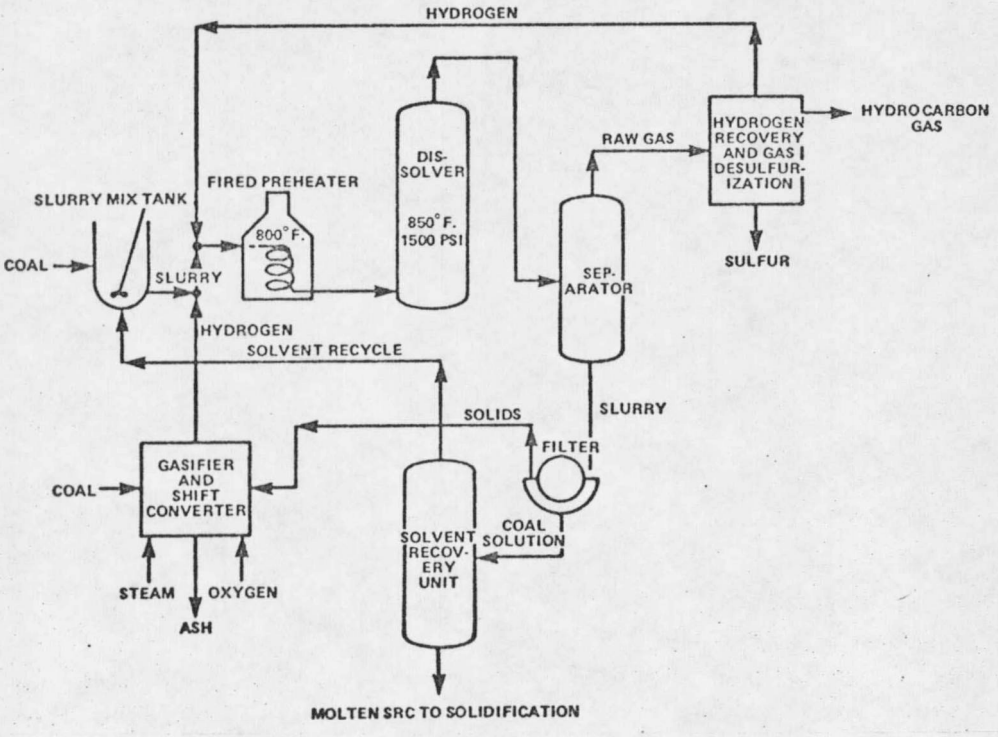


FIGURE 1. SRC-I PROCESS SCHEMATIC

The coal (Kentucky # 9) is pulverized and mixed with process-derived solvent in a slurry mix tank. The slurry is combined with hydrogen and is then pumped through a fired preheater and into a dissolver. In the dissolver, the coal is hydrogenated and thus depolymerized, leading to an overall reduction in product molecular weight and the dissolution of the coal. The solvent is also hydrocracked, yielding lower molecular weight hydrocarbons ranging from methane to light oils. The organic sulfur in the coal is hydrogenated to hydrogen sulfide.

After leaving the dissolver, the mixture passes to a separator where the gases are separated from the slurry of undissolved solids and coal solution. The raw gas is sent to a hydrogen recovery and gas desulfurization unit. The recovered hydrogen is then recycled and combined with the slurry from the slurry mix tank. Hydrocarbon gases are recovered and the hydrogen sulfide is converted to elemental sulfur.

The slurry of undissolved solids and coal solution is then separated in a solids separation unit. In a commercial scale plant, the solids would be sent to a gasifier-converter where they would react with coal, steam, and

oxygen to produce hydrogen for use in the process. The coal solution is passed to the solvent recovery unit and the final product (SRC-I) is produced(8,9).

In the making of SRC-II, which is of major concern to this research, the reactor effluent slurry is recycled for blending with the coal feed instead of distillate solvent. By increasing severity of operating conditions, the major product is converted from a solid (SRC-I) to a liquid distillate fuel (SRC-II). A schematic diagram of the SRC-II process is shown in Figure 2(9).

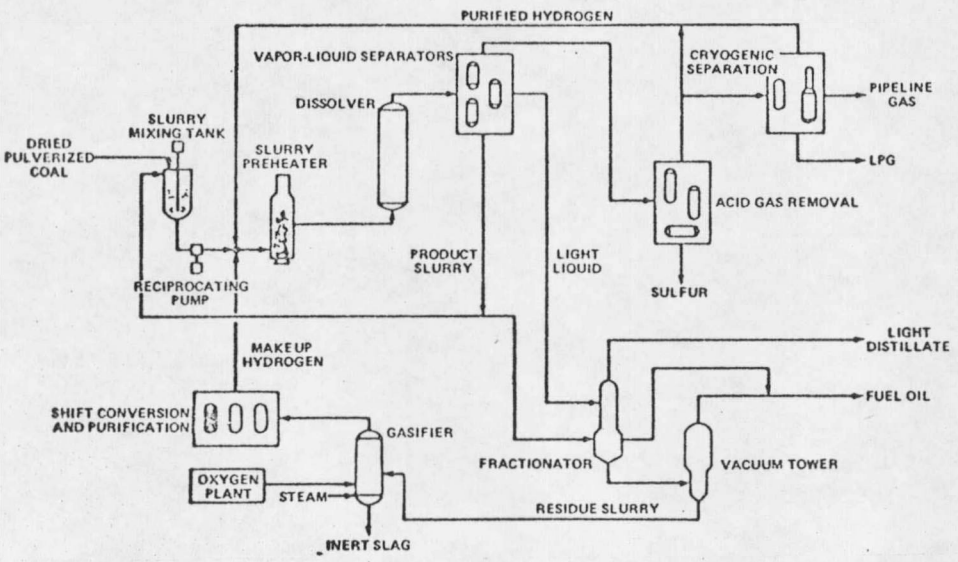


FIGURE 2. SRC-II PROCESS SCHEMATIC

The SRC-II process differs from the SRC-I case in that the dissolver slurry is split, one portion is recyled to provide solvent for coal slurry mixing, while the other is fractionated. The products from the fractionated system are naphtha, low-sulfur fuel oil, and a vacuum residue. This residue contains heavy oil, ash, and undissolved organic material from the coal(8).

Chemical Properties of SRC

The SRC process does not produce a one-step clean boiler fuel. Table I shows a typical analysis of SRC-II (10). Tables II and III show typical analyses of the SRC-II liquids obtained from Pittsburgh Seam Coal and Illinois No. 6 Coal(8). Pittsburgh Seam Coal is very simto Kentucky #9. The analysis for Kentucky #9 Coal is shown in Table IV(10).

Catalytic Hydrotreating of Synthetic Crudes

The hydrogenation of unsaturated hydrocarbons and the hydrogenolysis of heteromolecules are two main parts of hydrotreating. If SRC-II products are to be used as boiler fuels, a hydrogen to carbon mole ration of about 1:6 and nitrogen, sulfur and mineral levels below 0.5 weight percent (wt%) are desired. Sulfur levels are established

Table I

SRC Vacuum Flash Feed (SRC-II) as Received(10)

Sampled 1-24-77

% Carbon	87.43
% Hydrogen	7.15
% Nitrogen	1.17
% Sulfur	0.72
% Oxygen	3.72
% Ash	0.249

ASTM D-86 Distillation @ 640 mmHg

Volume, ml	Temperature, ^{o}F
IBP*	408
5	445
10	485
15	544
20	598
25	642
30	684
33.5	Final

Wt% recovered 69.2 Volume% recovered 69.7

*Initial Boiling Point

Table II

SRC II Liquids -- Pittsburgh Seam Coal

	Naphtha	Middle Distillate	Heavy Distillate
Naphtha, IBP-350°F, Vol. %	94	0	0
Middle Distillate, 350-550°F, Vol. %	6	100	0
Heavy Distillate, 550-850°F, Vol. %	0	0	100
Specific Gravity 60/60°F	0.817	0.976	1.087
Viscosity @ 100°F cSt	-	3.07	53.88
Pour Point, °F	-	-42	25
Flash Point, °F	-	163	-

Typical elemental analyses of the liquid products were:

	Naphtha	Middle Distillate	Heavy Distillate
Carbon, wt.%	85.17	87.03	87.18
Hydrogen, wt.%	12.38	8.69	7.30
Nitrogen, wt.%	0.52	0.98	1.17
Sulfur, wt.%	0.27	0.21	0.53
Oxygen (by difference), wt.%	1.66	3.09	3.85

Table III

SRC II Liquids -- Illinois No. 6 Coal

	Naphtha	Middle Distillate	Heavy Distillate
Naphtha, IBP-350°F, Vol. %	92	0	0
Middle Distillate, 350-550°F, Vol. %	8	100	1
Heavy Distillate, 550-850°F, Vol. %	0	0	99
Specific Gravity 60/60°F	0.817	0.976	0.087
Viscosity @ 100°F cSt	0.62	3.2	84
Pour Point, °F	-	-50	30
Flash Point, °F	32	160	325

Typical elemental analyses of the liquid products were:

	Naphtha	Middle Distillate	Heavy Distillate
Carbon, wt.%	84.62	85.41	90.02
Hydrogen, wt.%	12.09	9.05	7.04
Nitrogen, wt.%	0.58	0.83	1.27
Sulfur, wt.%	0.38	0.19	0.48
Oxygen (by difference), wt.%	2.33	4.52	1.19

Table IV

SRC Feed Coal Analysis, January 1977 wt%

Kentucky #9 Coal	
Carbon	71.35%
Hydrogen	5.07%
Nitrogen	1.44%
Sulfur	3.50%
O_2 (diff)	7.55%
Ash	10.12%
Moisture	0.97%
Sulfur Forms (wt% on coal)	
Pyritic Sulfur	1.63%
Sulfate Sulfur	0.09%
Organic Sulfur	1.76%
Total	3.48%
Average Mineral Residue Analysis (wt%)	
Carbon	27.61%
Hydrogen	1.39%
Nitrogen	0.54%
Sulfur	7.29%
Ash	63.17%
Pyridine Insol	96.98%

by current Environmental Protection Agency (EPA) standards (11). But, if an SRC-II product is to be used as a feedstock for a catalytic cracker, the nitrogen requirements are much more stringent. The catalysts used in catalytic crackers provide acid sites which facilitate cracking. If nitrogen is present, it acts as a poison and neutralizes these sites. On a commercial scale, catalytic cracking is maintained at a desired level by steadily increasing the severity of the reaction to the point that the reaction is economically feasible. The catalyst is then either regenerated or replaced. The desired nitrogen level in a feedstock is in the range of 100-400 ppm(12). However, several hydrocracking processes, such as Standard Oil of Indiana's Ultracracking and Union Oil's Unicracking process, can tolerate nitrogen levels of 0.3 wt%(13,14).

Since petroleum hydrotreating processes are controlled by fairly well established kinetics, the catalyst becomes the vehicle by which coal liquefaction is to be advanced. The important catalytic reactions are(15):

*Polyaromatic hydrogenation--polyaromatics are hydrogenated to monoaromatics or saturates

*Denitrogenation

*Desulfurization

*Removal of heavy metals - heavy metals are poisonous to cracking catalysts

The catalysts that perform best are made of metallic sulfides of tungsten (W), nickel (Ni), cobalt (Co), and molybdenum (Mo). Generally, the metal oxides deposited on a refractory oxide (SiO_2, Al_2O_3) are sulfided to obtain the metallic sulfide(10,16).

In hydrotreating, one of the major causes of catalyst deactivation is the deposition of carbonaceous material (7). It is believed that SRC-II has a high asphaltene content with an average size of 40-50 angstroms (Å) per molecule(19). Thus, the smaller pores of the catalyst tend to plug up – decreasing surface area and deactivating the catalyst. With large molecules, the problem of pore diffusion limitations is also encountered.

From this discussion, it can be seen than an attempt to fabricate various catalysts containing W, Ni, Co, and Mo or some combination of these is of importance to this research(16). High surface area and/or large pore diameter catalyst bases were used in an attempt to develop a good catalyst.

Research Objective

The objective of this research is to upgrade SRC-II. The amounts of sulfur and nitrogen are to be decreased and the amount of products recovered in ASTM D 86 distillation is to be increased.

Research was done by fabricating new catalysts and testing them. Some commercial catalysts were also tested. Hopefully, this research will aid the future development of synthetic crude.

Materials, Equipment, and Procedures

Feedstock Analysis. A 55-gallon barrel of SRC-II was obtained from Pittsburg and Midway Coal Mining Company's pilot plant operation in Ft. Lewis, Washington (10). The Kentucky #9 coal is from the Colonial Mine. Analysis of this coal is listed in Table IV. SRC-II analyses are listed in Table I.

Catalyst Fabrication. All catalysts fabricated at Montana State University were made with commercial supports and impregnated with metal salts using the incipient wetness technique. The following procedure was used.

The support was dried at 110°C for eight hours and calcined for eight hours at 450°C. The support was cooled in a dessicator and weighed. The amount of MoO_3, CoO, WO_3, or NiO desired was calculated. Experience would later give ratios of salt weight to desired metal oxide weight. The salts, ammonium molybdate, cobalt nitrate, ammonium tungstate-meta or nickelous nitrate, were used. The salt was then dissolved in water. Then, the solution and support were placed in a slowly rotating jar. A stream of air was directed in the jar and the catalyst was allowed to dry. The catalyst was then further dried at

110°C in air for eight hours and then calcined for eight hours at 450°C. The calcined catalyst was placed in a dessicator and allowed to cool. The cooled catalyst was then weighed to determine the amount of oxide added. No attempt was made to determine the exact form of the oxide on the support although the oxides described are assumed to be correct. The composition was reported as the weight percent of the metal oxide on the support.

Catalyst Presulfiding. Before a catalyst can be tested, the metal oxides of the catalyst are sulfied to obtain the metallic sulfide. Sulfiding also prevents reduction of the catalyst activity by hot hydrogen(19).

The apparatus used to sulfide the catalyst was a steel pipe two feet long with a thermowell running down the center. One quarter inch inert spheres were used to situate the catalyst in the apparatus. After the one inch inside diameter (I.D.) pipe was filled with catalyst and inert, it was heated with an electric pipe heater to 325°C for about 12 hours. Throughout the heating step, a stream of ten percent hydrogen sulfide in hydrogen was passed through the apparatus at approximately atmospheric pressure. After leaving the apparatus, the exit gas was

scrubbed with water and a 20% sodium hydroxide (NaOH) - water solution. When the heater was turned off, the hydrogen mix was allowed to flow through the apparatus for a couple of more hours. After the flow was stopped, the catalyst was removed and stored in a sealed container. Extreme caution must be executed while handling hydrogen sulfide as it can cause death within a few seconds after a few inspirations. It fatigues the sense of smell in high concentrations; so, it gives no warning(20).

Batch Runs. Batch autoclave runs were made in a Parr series 4000 pressure reaction apparatus(21). The apparatus was heated in a rocking autoclave heater. The apparatus and heater-rocker are shown in Figure 3.

The autoclave was charged with 200 ml of SRC and 25 ml of catalyst. The head of the reactor was secured to the autoclave and torqued tight. A new copper gasket was torqued to 60 ft-lb with subsequent 10 ft-lb increases per run up to 100 ft-lb. The copper gasket was then replaced. Copper Anti-Seize Compound was used on all threaded connections to prevent bolt seizure. After the head of the reactor was secured, the gauge block-pressure gauge was attached to the autoclave head. The autoclave was then

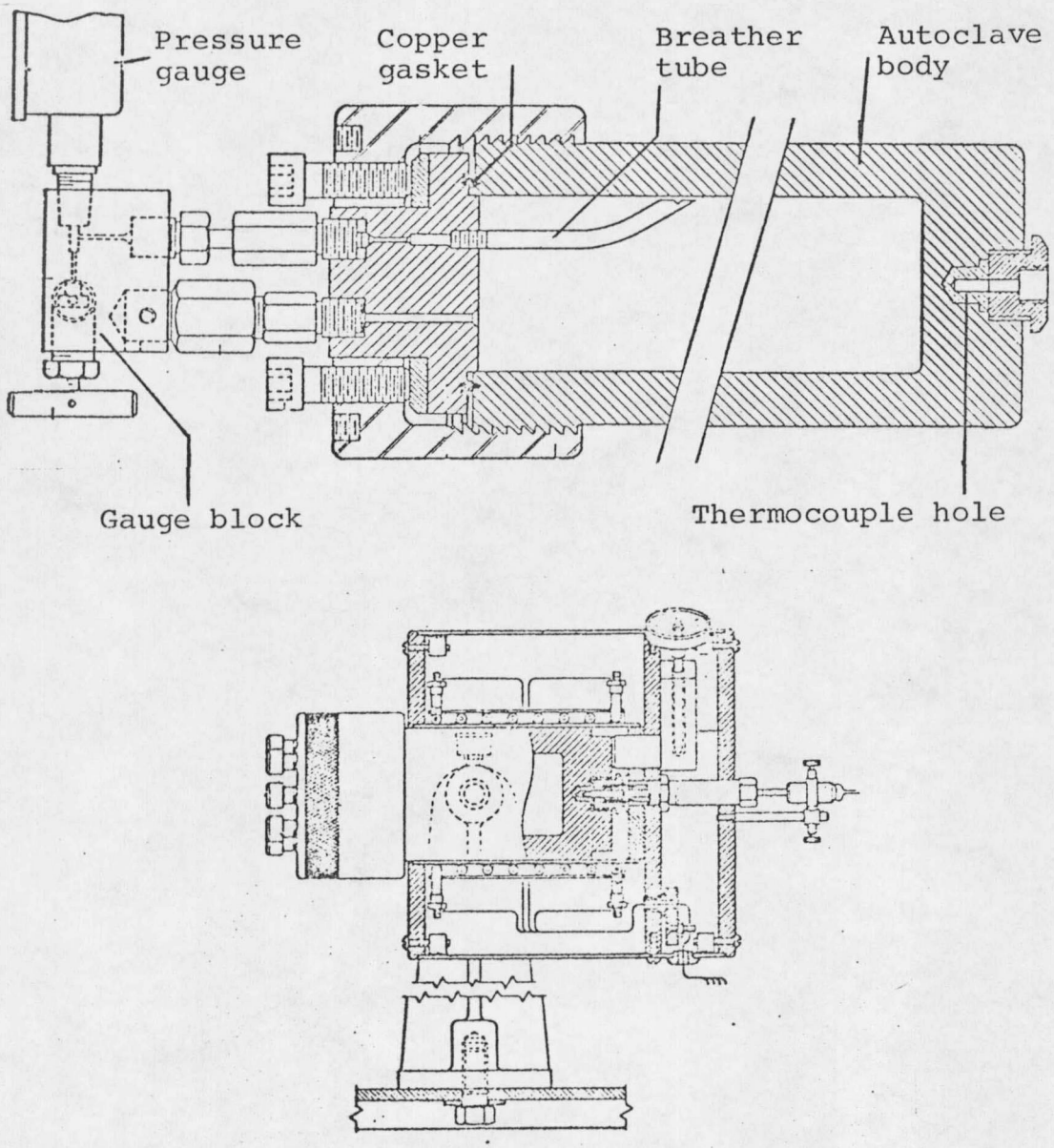


FIGURE 3. ROCKING AUTOCLAVE ASSEMBLY DETAILS

pressurized up to approximately 2500 psig with hydrogen using a Haskel gas booster(22) and checked for leaks. The autoclave was then placed in the reactor and heated up to about 425°C. An iron-constantan thermocouple, placed in the base of the autoclave, allowed for constant monitering of the temperature by a single point Micromax recorder(23). The temperature of the autoclave was controlled by adjusting a Powerstat variable transformer. Pressures and temperatures were recodered at 10 minute intervals for one hour. After the run was completed, the autoclave was removed from the heater-rocker and allowed to cool to room temperature. Thus, the approximate hydrogen consumption (cold loading pressure minus final room temperature pressure) could then be found. The gas was then slowly vented to prevent liquid product loss. The liquid was then separated from the catalyst and analyzed.

Continuous Trickle Bed Reactor. The trickle bed reactor was designed and built by the Chemical Engineering Department at Montana State University prior to this research. The schematic for the apparatus is shown in Figure 4.

The reactor consists of a one-inch I.D. schedule-80

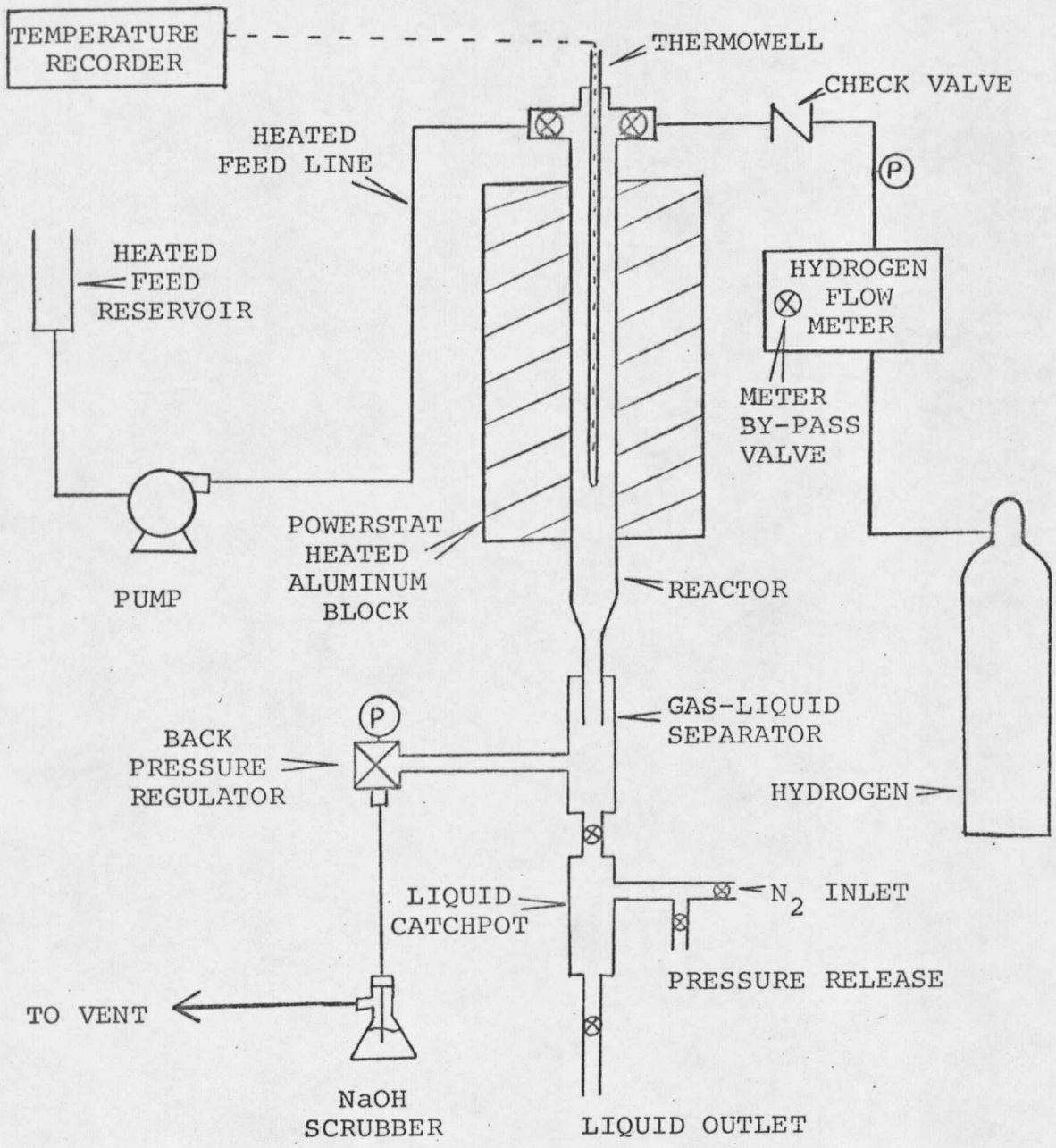


FIGURE 4. TRICKLE BED REACTOR

Inconel pipe 40 inches long. The top of the reactor is fitted with a 1/4-inch stainless steel cross. This allows the fitting of a 36-inch piece of stainless steel tubing (which runs down the center of the reactor and acts as a thermowell) and the fitting of two feed ports, one for the SRC-II and one for the hydrogen. The reactor is placed into the bore of a six-inch outside diameter (O.D.) aluminum block about three feet long. The block is wrapped with three sets of nichrome wire heating coils encased in ceramic beads. Each coil is connected to a Powerstat variable transformer which allows manual temperature control. Four iron-constantan thermocouples were placed in the thermowell at six-inch intervals. The uppermost point was 12 inches from the top of the reactor.

The reactor was loaded at the top with 175 cubic centimeters (cc) of 1/4-inch Denstone inert support(24) and 25 cc of 1/8-inch Denstone inert support to act as a preheat zone. The 60 cc of catalyst were mixed with 60 cc of 1/8-inch inert and loaded into the reactor. The bottom of the reactor was filled with 1/8-inch inert and a stainless steel screen cone was inserted as a plug support. A 1/4-inch I.D. reactor closure was then threaded

into the pipe. All threaded connections were sealed with teflon tape and/or Copper Anti-Seize Compound to prevent leakage. All continuous runs were packed this way except Run 22. In Run 22, the top of the reactor contained about 20 cc of 1/4-inch inert followed by a 180 cc catalyst —180 cc 1/8-inch inert mix.

SRC-II was pumped into the reactor by a Milton Roy Model MR-1 49 simplex packed piston pump through a 1/8-inch stainless steel feed line. The pump is equipped with a manually controlled micrometer adjustment for controlling feed rate. All lines and reservoirs containing SRC-II were heated with Cole-Parmer flexible heating cords (25) to prevent feed freeze-up.

Technical grade hydrogen passed through a regulator, a micrometering valve, a Brooks Thermal Mass Flowmeter (26), and a ball check valve, then to the top of the reactor.

The gases and liquids passed through the reactor to a gas-liquid separator. The gases passed through a Grove back pressure regulator. The teflon diaphragm allows the regulator to handle the corrosive gases. The gas then passed through a 20% NaOH-water scrubber and was then

vented . A wet test meter can be connected before the gas is vented to calibrate the Brooks Thermal Mass Flowmeter. The liquids passed through the reactor to a gas-liquid separator and into a pressurized catchpot. When a sample was taken, a valve between the catchpot and gas-liquid separator was closed. The catchpot was then depressurized and the sample was drained out the bottom. The catchpot was then repressurized with nitrogen and the valve reopened.

Continuous Trickle Bed Runs. After the reactor was packed as preciously discussed, it was placed in the aluminum block. The 50-50 mix of inert-catalyst was used because in single pass trickle bed reactors that contain a thermowell, conversion data correlates well with scaled up reactors(27). The catchpot system was then attached as was the hydrogen and SRC-II feed lines. The whole systems except the pump was then pressurized with nitrogen to about 1000 psig and checked for leaks. If no leaks were found, the system was depressurized to just above atmospheric pressure. Then the thermocouples,connected to a Leed and Northup Multipoint recorder, were inserted in the thermowell. The Powerstats to the aluminum block were

then turned on and the system was allowed to heat overnight.

When the run temperature was reached, the heating coils on the feed line and feed reservoir were turned on. The valve between the reactor and feed line was closed and the feed line was removed. The Brooks Thermal Mass Flowmeter was turned on and the meter by-pass valve was opened. The feed reservoir was filled and SRC-II was pumped through the feed lines. If the feed line is not filled, the pump will tend to cavitate on the pressurized system.

The reactor system was then slowly pressurized with hydrogen. When the system had reached the desired pressure, the by-pass vlave on the flowmeter was closed and the micrometering vlave was adjusted to obtain the desired flow rate. In all runs, except Run 22, 10,000 standard cubic feet per barrel (scf/bbl) of feed was used (28). In Run 22, about 67000 scf/bbl of hydrogen was used. The valve of the feed line was then opened and the pump started. The cocurrent flow in the reactor allows for better distribution of the liquid over the catalyst and higher liquid flow rates without flooding problems.

The SRC-II flow rate was controlled by use of a stop watch and timing the flow from a graduated side-arm attached to the main feed reservoir. The flow rate was checked many times to maintain even flow. In all runs, the flow rate was one liquid hourly space velocity (LHSV-(volume of liquid feed)/(volume of catalyst x hour)).

For Runs 1-5, samples were taken every three hours for 12 hours. In Runs 6-9, the reactor plugged up after about 2 hours. Therefore, only one two hour sample was collected for Runs 7-9. For Run 6, no sample was obtained due to technical error. Runs 10-22 each plugged as the reactor reached 1300-1500 psig. In Run 10, samples were taken as follows:

Time on Stream minutes	30	45	60	75	90	105	120	150	180	210	240	300
Sample	1	2	3	4	5	6	7	8	9	10	11	12

In only Run 11, were 12 samples obtained before plug up. In all the rest, between six, and eight samples were obtained before plug up.

After the last sample was obtained, the pump was shut off, the valve between the feed line and reactor was closed and the feed line was removed. The hydrogen was

shut off and the reactor system was depressurized. The catchpot system was removed and later cleaned with acetone. The Powerstats were shut off and the excess SRC-II drained from the reservoir feed lines. Acetone was used to rinse much of the excess SRC-II from the feed lines and reservoir. The reservoir was then filled with 30 W motor oil and pumped through the feed line. After it was sure that only oil was being pumped through the line, the line was hooked up, the valve opened, and the oil was pumped through the still hot reactor. The reactor was then removed from the aluminum block with asbestos gloves. The feed line and reservoir was cleaned with acetone and as most runs plugged up, only the material up to the plug could be knocked out.

A pipe with one end serrated was placed into the reactor with the thermowell inside the pipe. The other end of the pipe was attached to a drill and the plug was drilled. After about one inch was drilled, the thermowell generally was loosened enough so that it could be removed. The plug was then drilled out with a cement drill bit. The reactor was then rinsed with acetone. The system was then ready for the next run.

Analytic Procedures. Liquid products were analyzed for nitrogen and sulfur content. If possible, the extent of hydrocracking was determined.

Nitrogen content was determined on all samples by the Macro Kjeldhal method using 0.5 grams of samples and 40 grams of potassium sulfate(29,30,31). SRC-II contains about 1.17% nitrogen. Weight percent denitrogenation (%DN) was calculated as follows:

$$\frac{1.17\% - \text{wt}\%}{1.17\%} = \%\text{DN}$$

Sulfur analysis was done on all samples for continuous Runs 1-9, and for all batch runs. Sulfur analysis was done on selected samples for continuous Runs 10-22. The analyses were performed by the quartz tube combustion method using a Bico Brown Shell design sulfur apparatus (32,33). SRC-II contains about 0.735 wt% sulfur. Weight percent desulfurization (%DS) is calculated similarly to %DN.

The extent of hydrocracking was determined by ASTM-D-86 atmospheric distillation in a Lab-Line distillation apparatus(34). The size of the sample used for the distillation is 50 ml(10). This technique measures the

cumulative amount of product that boils below 700°F or when decomposition begins, whichever occurs first.

RESULTS AND DISCUSSION

SRC-II was tested with 28 catalysts in 22 continuous runs and 10 batch runs. Ten catalysts were tested in batch runs and 22 catalysts were tested in continuous runs. All samples taken from all runs were analyzed for nitrogen content. Many samples were tested for sulfur content and whenever possible, the amount of distillable liquids was found by ASTM distillation.

The data for each run can be found in the appendices. Appendix A gives a graphical representation of the data. Appendices B and C give detailed information about batch and continuous runs, respectively. Catalysts developed at Montana State University are designated by the prefix MSU (developed by Angong Yeh) or Catalyst # (developed by the author). See Table VII for a description of the bases of many of the catalysts.

Batch Run Tests

Batch tests were performed on SRC-II to test the activity of a catalyst. This has been done by previous researchers at Montana State University(10,28). To test the activity of the catalysts, a batch run was made on each. As continuous runs have previously been very long, the batch run would provide relatively fast testing of

many catalysts. The results of the runs are shown in Table V. Six Harshaw catalysts:

720 A-1-3-4
618 A-30-10-6
L3304-23-1
720 A-2-1-1
720 A-2-7-3
720 A-2-11-3

were received (See Table VI for catalyst description). Of the six, 720 A-1-3-5 was the best catalyst for removing nitrogen and sulfur. This catalyst was then tested in a continuous run. When the activity of this catalyst proved to be low, it was decided not to test further the other five Harshaw catalysts.

As catalysts were fabricated at Montana State University, batch runs were made on each of the catalysts. After Catalysts 1-4 were tested in batch runs, the author, not satisfied with the information received from batch runs, tested most of the fabricated catalysts in the continuous reactor. Catalyst 3, which showed no nitrogen removal ability in Batch Run 9, proved to be one of the best at nitrogen removal when run in Continuous Run 13 (See Table VIII). It was then decided that batch runs do not give a good representation of the activity of a catalyst. In the future, it would be advisable to discontinue

Table V

Data Summary of Batch Runs with SRC-II

Run	Catalyst	%DN*	%DS*	%Yield*
1	720 A-1-3-5	15.1	55.8	72.6
2	618 A-30-10-6	12.9	53.1	73.2
3	L3304-23-1	0.0	35.5	71.2
4	720 A-2-7-3	0.0	8.8	70.6
5	720 A-2-1-1	9.4	46.9	75.1
6	720 A-2-11-3	2.2	32.7	74.2
7	1	13.8	34.0	72.4
8	2	0.9	40.8	73.6
9	3	0.0	41.5	74.0
10	4	15.1	32.0	74.8

*%DN Based on 1.17 wt% N_2 in feed
%DS Based on 0.735 wt% S in feed
%Yield Based on volume% by ASTM distillations

Table VI
Catalyst Description

Catalyst	Run* CR	Run* BR	Base	Composition wt% MoO_3	WO_3	NiO	CoO	SiO_2	P	SO_4
Ketjen 153	1	-	--	15.0		3.0		1.1		2.0
Cyanamid HDS-20A Trilobe	2	-	--	16.2			5.0			
Nalco NM 502	3	-	**	14.0		4.0				
Nalcomo 477	4	-	**	14.0			3.3			
Harshaw										
720 A-1-3-5	5	1	--	15.3		2.9		5.6		
618 A-30-10-6	-	2	--	14.5		3.3		0.1	2.0	
L3304-23-1	-	3	--	14.9		3.0		4.5		
720 A-2-7-3	-	4	--	16.0		3.0		0.1	0.5	
720 A-2-1-1	-	5	--	14.8			2.8			
720 A-2-11-3	-	6	--		32.3	5.7				
Catalyst 1	-	7	Norton	11.7	17.0	1.1	2.6			
Catalyst 2	6	8	Norton		20.5	2.9	6.6			

*CR = Continuous Run BR = Batch Run **See Base Description-Table VII

Table VI (continued)
Catalyst Description

Catalyst	Run CR	Run BR	Base	Composition wt% MoO_3	WO_3	NiO	CoO	SiO_2	P	SO_4
Catalyst 3	13	9	Norton	12.6	10.3	2.8	6.8			
Catalyst 8	11	-	A		19.5	3.6	5.7			
Catalyst 10	22	-	A		22.6	3.4	5.4			
Catalyst 9	7	-	B		20.0	3.2	5.5			
MSU-C6-B	20	-	B	14.8		0.19	1.17			
MSU-C7-B	14	-	B	14.6		0.13	5.8			
Catalyst 4	12	10	C		21.5	2.9	5.9			
MSU-C11-C	17	-	C	11.1		0.1	1.6			
Catalyst 7A	9	-	D		20.2	3.1	5.9			
Catalyst 7	10	-	D		19.7	3.1	6.0			
MSU-C17-D	15	-	D	9.3	8.8	0.17	1.8			
Catalyst 6	8	-	E		21.5	2.7	6.2			
MSU-C18-E	21	-	E	29.3		0.1	0.1			

Table VI (continued)
Catalyst Description

Catalyst	Run CR	Run BR	Base	Composition wt% MoO_3	WO_3	NiO	CoO	SiO_2	P	SO_4
MSU-C19-E	19	-	E	18.9		0.1	3.5			
MSU-C21-E	18	-	E	9.5		0.4	1.9			
MSU-C22-E	16	-	E	9.8	9.2	0.3	1.4			

Table VII
Base Description

BASE	SURFACE AREA (A) m^2/g	PORE VOLUME (V) ml/g	PORE DIAMETER (Å) Median	PORE DIAMETER (Å) Average (4V/A)
Norton	250.0	.7000	---	125.0
A	323.2	.7183	90.2	88.9
B	232.4	.7215	137.3	124.2
C	214.6	.8397	161.0	156.5
D	211.4	.7943	190.0	150.0
E	146.95	.6841	420.2	186.2
**	300.0	.7600	---	101.3

BASE	PELLET SIZE (in)	COMPOSITION WT% Al_2O_3	SiO_2	P_2O_5
Norton	1/16	99.85	0.12	
A	1/32	96.0	4.0	
B	1/16	100.0		
C	1/32	98.0	2.0	
D	1/16	91.0		9.0
E	1/16	83.0		17.0
**	1/16	97.6	2.4	

**This is the base for Nalcomo 477 and Nalco NM 502.

the use of batch runs and concentrate on testing with continuous runs.

Continuous Run Tests

Continuous runs were made on 22 catalysts. Runs 1 and 2 were made to acquaint the author with the apparatus and procedure involved in making a continuous run. Table VIII shows the results of the continuous runs. In Run 2, Cyanamid HDS-20A Trilobe was tested to see if comparable results could be obtained with a previous researcher(10). When it was found that similar results could be obtained, more catalysts were then tested. Nalco NM 502 and Nalcomo 477 were investigated next. Each had high recommendations for use in hydrotreating units where desulfurization and/or denitrogenation treating is desired. These catalysts were tested in Runs 3 and 4 and proved to be only slightly better, if that, than previous catalysts.

Six Harshaw catalysts were recieved and tested in batch runs as previously discussed. Harshaw 720 A-2-3-5 looked to be the best and was tested in a continuous run. Though it proved to be fairly good at sulfur removal, it was extremely poor at nitrogen removal. Therefore, the

Table VIII
Continuous Run Data Summary with SRC-II: Runs 1-9

Run	Time (hour)	Catalyst	%DN	%DS	%Yield 425°F	%Yield 700°F
1	3	Ketjen 153	62.1	50.3	21.4	87.4
	6		31.9	46.9	23.7	86.5
	9		21.1	49.6	17.0	82.0
	12		17.7	51.4	17.1	85.5
2	3	Cyanamid HDS-20A	59.1	60.0	41.8	92.0
	6	Trilobe	23.3	56.6	17.6	76.5
	9		21.1	67.3	18.9	82.0
	12		22.4	61.9	--	--
3	3	Nalco NM 502	56.9	62.6	30.0	94.0
	6		30.2	64.6	--	--
	9		23.3	62.6	18.0	85.3
	12		28.4	59.2	21.1	85.3
4	3	Nalcomo 477	59.9	71.4	16.2	89.5
	6		24.1	66.7	14.1	84.2
	9		16.4	59.2	18.7	82.0
	12		12.1	44.2	16.3	82.0
5	3	Harshaw	37.5	72.1	21.2	89.8
	6	720 A-1-3-5	6.0	64.6	17.5	80.2
	9		5.2	66.0	13.2	72.0
	12		2.2	58.5	18.3	72.2
6	2	2	--	--	--	--
7	2	9	76.9	74.8	45.8	94.0
8	2	6	27.4	24.5	19.6	80.0
9	2 1/4	7A	72.6	77.1	36.0	94.6

Table VIII
Continuous Run Data Summary with SRC-II: Runs 10-13

Run	Time (min.)	Catalyst	%DN	%DS	%Yield 425°F	%Yield 700°F
10	120-2 hr	7	69.2	83.7	34.0	92.6
	180-3 hr		40.2	75.5	24.4	93.0
	360-6 hr		38.9	74.1	25.5	96.4
	540-9 hr		29.5	63.3	20.0	91.8
11	30	8	91.5	69.4		
	45		88.0	--		
	60-1 hr		87.2	--		
	75		92.3	81.6		
	90		85.5	--		
	105		79.5	--	0-120 min.	
	120-2 hr		78.6	--	32.0	96.2
	150		80.3	73.5		
	180-3 hr		81.2	--		
	210		82.1	--	120-240 min.	
	240-4 hr		85.5	34.7	33.8	96.6
	300-5 hr		52.1	--		
12	30	4	92.3	62.0	----------	
	45		93.2	--		
	60-1 hr		85.5	--		
	75		91.0	68.0		
	90		88.9	--		
	105		87.2	--		
	120-2 hr		90.6	78.2		
13	30	3	99.1	78.2	----------	
	45		98.3	--		
	60-1 hr		88.9	--		
	75		92.3	89.1		
	90		94.9	--		
	105		73.9	--		
	120-2 hr		92.3	79.6		
	150		81.2	--		

Table VIII (continued)
Continuous Run Data Summary with SRC-II: Runs 14-17

Run	Time (min.)	Catalyst	%DN	%DS	%Yield 425°F	%Yield 700°F
14	30	MSU-C7-B	82.1	71.4	----------	
	45		76.1	--		
	60-1 hr		75.2	--		
	75		71.8	60.8		
	90		70.9	--		
	105		75.2	--		
	120-2 hr		73.5	74.1		
	150		41.9	74.1		
15	30	MSU-C17-E	94.9	76.6	----------	
	45		86.3	--		
	60-1 hr		74.4	--		
	75		78.6	77.1		
	90		79.5	--		
	105		--	73.6		
16	30	MSU-C22-E	62.9	74.1	0-120 min.	
	45		83.8	--	28.9	94.7
	60-1 hr		83.8	--		
	75		74.4	71.4		
	90		68.4	--		
	105		60.7	--		
	120-2 hr		74.4	68.7		
17	30	MSU-C11-C	74.4	40.1	----------	
	45		94.0	--		
	60-1 hr		93.3	--		
	75		88.9	74.1		
	90		83.8	50.3		

Table VIII (continued)
Continuous Run Data Summary with SRC-II: Runs 18-21

Run	Time (min.)	Catalyst	%DN	%DS	%Yield 425°C 700°F
18	30	MSU-C21-E	52.1	55.1	----------
	45		88.0	--	
	60-1 hr		87.2	--	
	75		85.5	72.8	
	90		79.5	--	
	105		78.6	66.0	
19	30	MSU-C19-E	61.5	53.7	----------
	45		94.0	--	
	60-1 hr		76.1	--	
	75		84.6	61.9	
	90		80.3	--	
	105		87.2	77.4	
20	30	MSU-C6-B	55.5	54.4	----------
	45		81.2	--	
	60-1 hr		71.8	--	
	75		71.8	51.8	
	90		74.3	--	
	105		76.9	--	
	120-2 hr		64.1	56.3	
21	30	MSU-C18-E	60.7	58.6	----------
	45		85.5	--	
	60-1 hr		71.8	--	
	75		59.8	79.3	
	90		66.7	--	
	105		47.9	73.3	

Table VIII (continued)
Continuous Run Data Summary with SRC-II: Run 22

Run	Time (min.)	Catalyst	%DN	%DS	%Yield 425°F	%Yield 700°F
22	30	10	89.7	72.2	0-45	min.
	45		65.8	--	52.0	96.2
	60-1 hr		97.4	--		
	75		88.0	72.0	45-90	min.
	90		83.0	--	40.0	96.6
	105		88.0	--	90-120	min.
	120-2 hr		66.7	74.4	32.0	96.6

other five catalysts were not tested.

In Runs 1-5, each run went for 12 hours with no sign of reactor plug up. As previously discussed, samples were taken every three hours for these runs. All of the runs followed patterns of catalyst deactivation very similar to work done by Hass(10).

A variety of large pore diameter bases were recieved from Nalco (See Table VII). The effect of the difference in pore diameter was to be obtained by applying the same or nearly same metallic composition to each base. A composition of 20.0% WO_3, 6.0% CoO, and 3.0% NiO was chosen as a consistent composition. Norton 6176 base was first used to develop technique in catalyst manufacturing. Catalysts 1-3 were thus produced. The fabrication of catalyst 5 was inadvertantly aborted; therefore, no catalyst was available. Catalysts 4-9 were made on each of the other bases. The composition of these catalysts and other catalysts tested are shown in Table VI.

With the advent of Run 6 came the testing of the Montana State University fabricated catalysts. Though Run 6 was designed to run for 12 hours, as had previous runs, it plugged up after 2 hours. Run 6 was started at

325°C and heated up to 430°C over the two hour period. Yeh showed that this method reduced deactivation of catalysts(35). Unfortunately, no sample was saved from the run. Upon cleaning out the reactor, the coked up plug was found about one to two inches above the catalyst bed.

Run 7 was started at 335°C and heated to 430°C in one hour and ran another hour at 430°C until it also plugged up. Again, the plug was one to two inches above the catalyst bed. The plug would also occur at this position in the reactor for Runs 6-21.

Run 8 was run at 430°C throughout the run, as Runs 1-5 had been. It was hoped this would prevent plug up. Run 8 began to plug up after about 1 hour and 45 minutes. The SRC-II flow rate was increased greatly in an attempt to free the reactor from the coked plug. But, after about 10-15 minutes the reactor eventually plugged up. The low denitrogenation, desulfurization, and yield could be attributed to this unsuccessful attempt to free the plug in the reactor.

In Run 9, an attempt to stop plug up was done by reducing the run temperature to 390°C. The reactor plugged up again after two hours. At this time, the two hour

sample for Run 7 had been analyzed showing a 0.26 wt% N and 0.185 wt% S. This was by far the best nitrogen and sulfur removal yet!

In Run 10, catalyst 7 was developed similar to catalyst 7A used in Run 9. The run took place at 360°C in another attempt to prevent plug up. The run lasted nine hours before plug up. Comparing Run 9 to Run 10, the higher temperature run (Run 9) had a better nitrogen removal, as expected(10).

With the continued plug up of the reactor and the good nitrogen level of Catalyst 9 in Run 7, it was decided to take more samples at short time intervals. In Run 11, the temperature was increased to 390°C and samples were taken at 15 minute intervals up to two hours. This first sample was taken after 30 minutes to, hopefully, allow the reactor bed to be wetted. At two hours, samples were taken at 30 minute intervals for two more hours. A final one hour sample was taken as the reactor plugged up. The results show a nitrogen content well below 0.3 wt% for four hours of the run. Distillations would later show a final distillate yield of over 96% for the run through four hours.

Runs 12-21 were made testing Montana State University fabricated catalysts. The length of the runs varied from 90-150 minutes. For Runs 17-21, the first sample taken was very nearly the worst of the runs. This can be attributed to difficulty in cleaning our the catchpot from the previous run.

Run 22 was a final attempt to prevent reactor plug up. Since reactor plug up always occurred just before the catalyst bed, it was decided to increase the volume of catalyst in the reactor. A catalyst was made (Catalyst 10) with a base and composition similar to Catalyst 8 (Run 11). Three times as much catalyst was used. Hydrogen flow had to be reduced to 6700 scf/bbl due to equipment limitations. But, the reactor still plugged up after two hours at 430°C. Comparing the data, Figure 5, we see that the nitrogen content had not been affected all that greatly by the additional amount of catalyst in the reactor.

The larger amount of feed going through the reactor allowed for larger samples; therefore, a distillation could be made over a shorter time span. The final distillate yields for Run 11 and Run 22 were both above 96%.

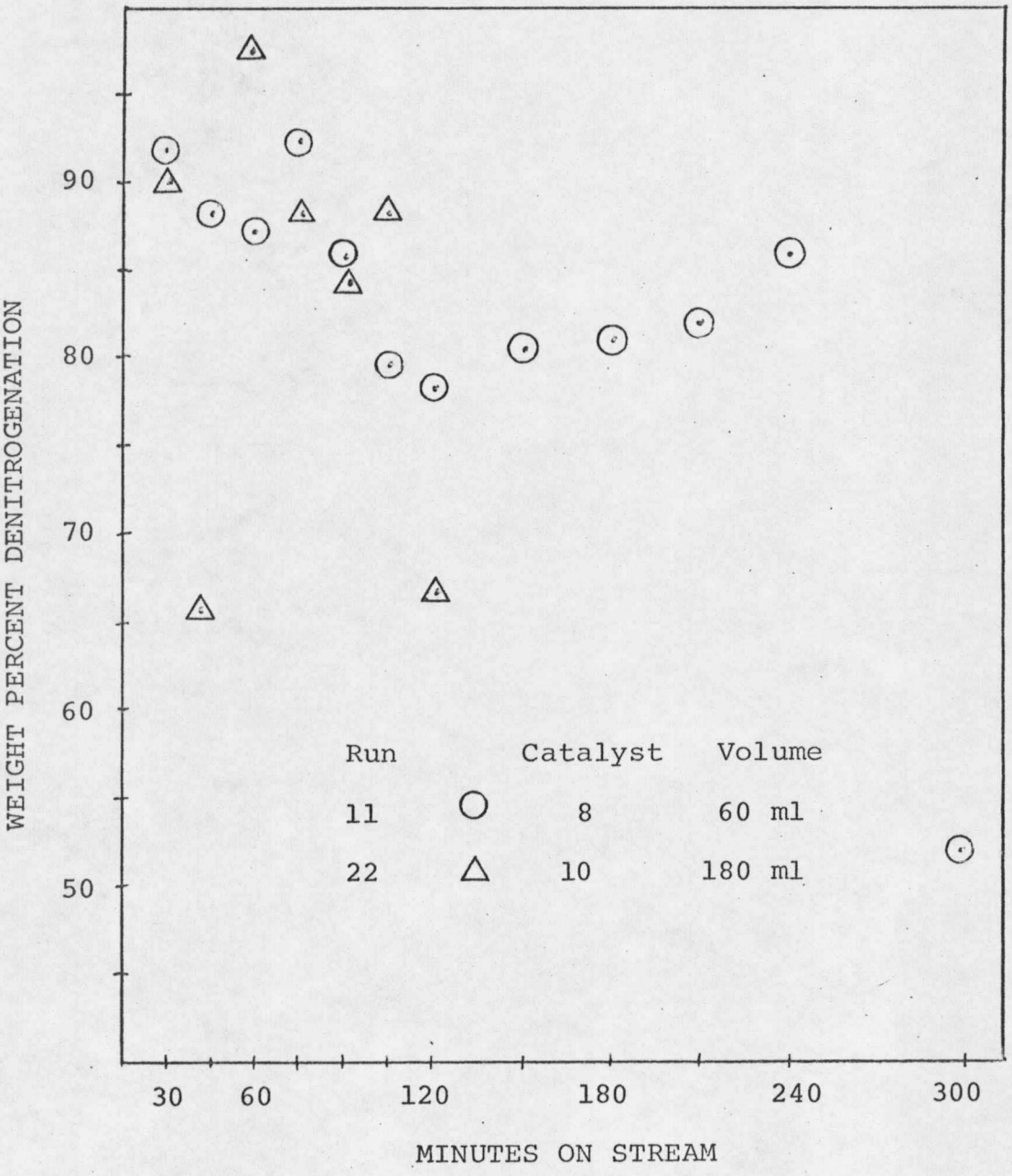


FIGURE 5. EFFECT OF CATALYST VOLUME ON NITROGEN REMOVAL

It is easier to obtain desirable sulfur levels than desirable nitrogen levels. From the Figures in Appendix A and past reseach(10), it can be seen that catalyst deactivation is very rapid with respect to nitrogen removal. But, it has also been shown that catalyst deactivation is much less rapid for sulfur removal(10). Therefore, for Runs 11-22, a sulfur analysis was done only on periodic samples. Sulfur analysis was done on all samples for Runs 1-10. The large number of samples in Runs 12-22 would make it extremely difficult for the analysts to analyze all samples. This was another reason for reducing the number of samples tested.

To determine distillate yields, ASTM distillations were done on all samples for Runs 1-10. For Runs 11-22, a sample could not provide enough material for a distillation. After nitrogen and sulfur analyses were done, samples were added together in an attempt to obtain enough samples for a distillation(50 ml). Only in Runs 11, 16, and 22 was enough sample available for analysis. In Table VIII, the volume percent yield obtained below 425^{o}F is also listed. Run 22 with its large catalyst volume gave the best yield below 425^{o}F.

The coking up problem that occurred in the reactor will be discussed here. The coke up presents a problem in that once the reactor plugs up the run must be shut down; thus, no more information can be obtained about catalyst deactivation. Also, it is difficult to clean a plugged reactor. The plug up occurred above the catalyst bed in Runs 6-22. It is not understood why Runs 1-5 did not plug up while Runs 6-22 did. This shows that the catalyst probably does not cause plug up. Yeh tested many catalysts similar to the ones tested in this research (36). Not one of his runs plugged up. But, the data received on nitrogen removal differed greatly from that reported in this research. The following is an example. In Run 18, catalyst (MSU-C21-E) was tested. Yeh tested catalyst (MSU-C24-E) in his Run A-16. The catalysts both have the same base (Base E). The composition of the catalysts is as follows:

MSU-C21-E	9.5% MoO_3	0.40% NiO	1.9% CoO
MSU-C24-E	15.5% MoO_3	0.15% NiO	2.3% CoO

Though different, these two catalysts are close enough to expect them to produce similar results when tested with

SRC-II at the same conditions. The two were tested under similar conditions. However, Run 18 plugged up while Yeh's Run A-16 did not. The following data was obtained after the two were tested:

Nitrogen Content wt%

Time, min.	30	45	60	75	90	105	120
Run 18 MSU-C21-E:	.56	.14	.15	.17	.24	.25	
Run A-16 MSU-C24-E:	.22	.44	.53		.79		.73

% Denitrogenation

Time, min.	30	45	60	75	90	105	120
Run 18 MSU-C21-E:	52.1	88.0	87.2	85.5	79.5	78.6	
Run A-16 MSU-C24-E:	81.2	62.4	54.7		32.5		37.6

Figure 6 helps show the effect of the coking reactor compared with a noncoking reactor (Yeh's). It can be seen that the rate of deactivation is much less for Run 18 than for Yeh's Run A-16. The difference in the reactors and possibly the cause of reactor plug up is the length of the reactors. The reactor used in this research is about six inches longer than Yeh's reactor. This may have allowed time for the SRC-II to coke up. Therefore, it seems that the coking of the SRC-II before it comes into contact with the hydrotreating catalyst may greatly reduce the amount of catalyst deactivation.

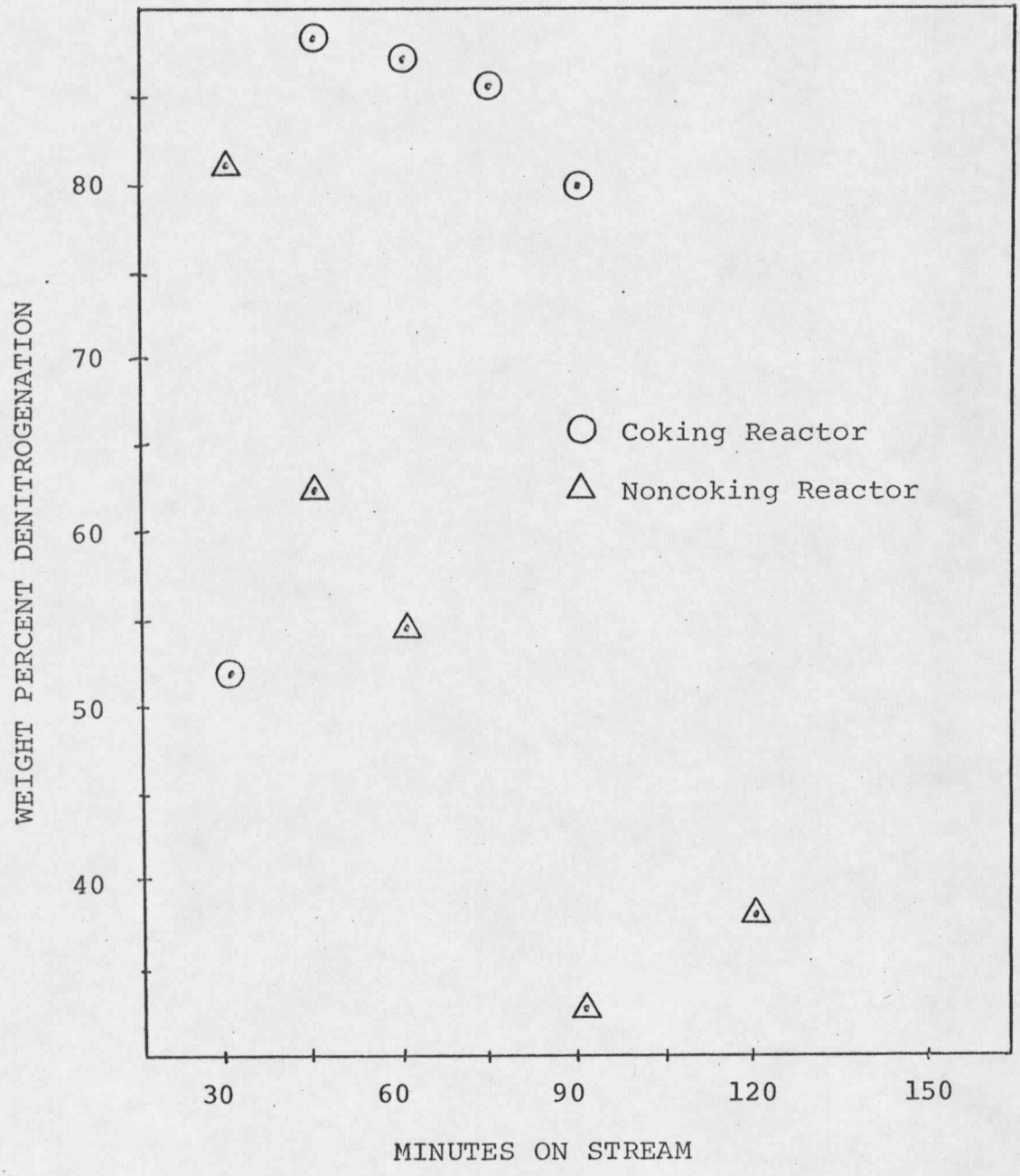


FIGURE 6. THE EFFECT OF REACTOR COKE UP ON NITROGEN REMOVAL

In the future, one catalyst should be tested in both reactors to varifly this statement.

The effect of the bases on nitrogen and sulfur removal can be seen in Figures 7-16. Due to the amount of data scatter and difference in catalyst composition, a single "best" base can not be chosen. Table IX gives an average percent denitrogenation and desulfurization for Montana State University fabricated catalysts. This "average" is based on the first two hours of the run or the whole run, whichever is shorter. From this table, Figure 17 and 18 are plotted against average pore diameter (4V/A). A, C, and Norton 6176 have a better nitrogen removal than B, D, and E. Bases D, and Norton 6176 have better desulfurization than A,B,C, and E. Of the distillations made (Runs 11, 16, and 22) all yields were 95% of better.

Overall, the best catalyst was Catalyst 3 tested in Run 13. It removed nearly 90% of the nitrogen (0.12 wt%) for most of the run. It also maintained sulfur removal above 78% (0.16 wt%).

In summation, it can be seen that nitrogen levels can be brought below 0.3 wt% and sulfur levels well

below 0.5 wt%. The coking of the feed and short run times seem to allow these levels to be reached.

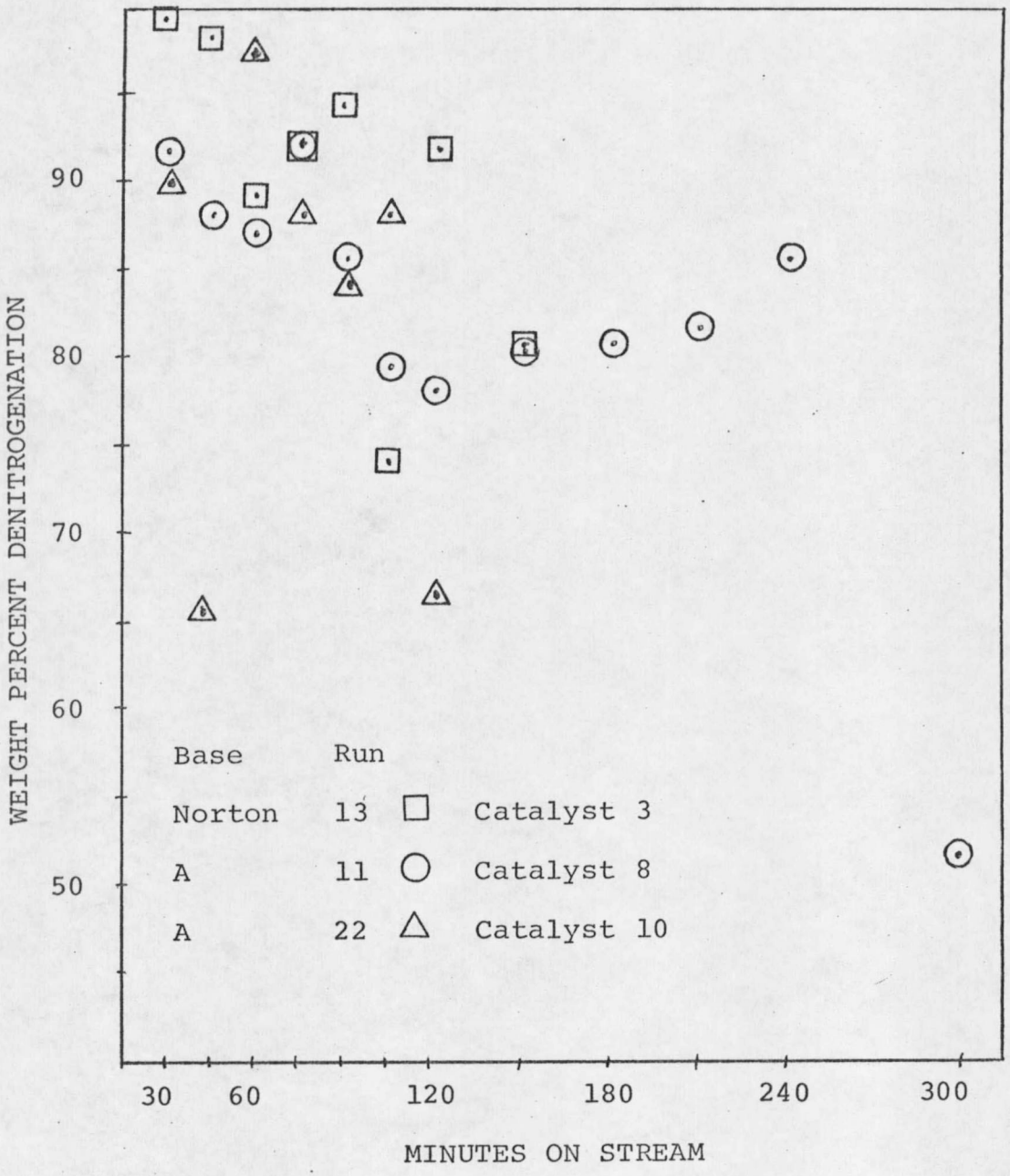


FIGURE 7. NITROGEN REMOVAL vs. RUN TIME FOR BASES A AND NORTON 6176

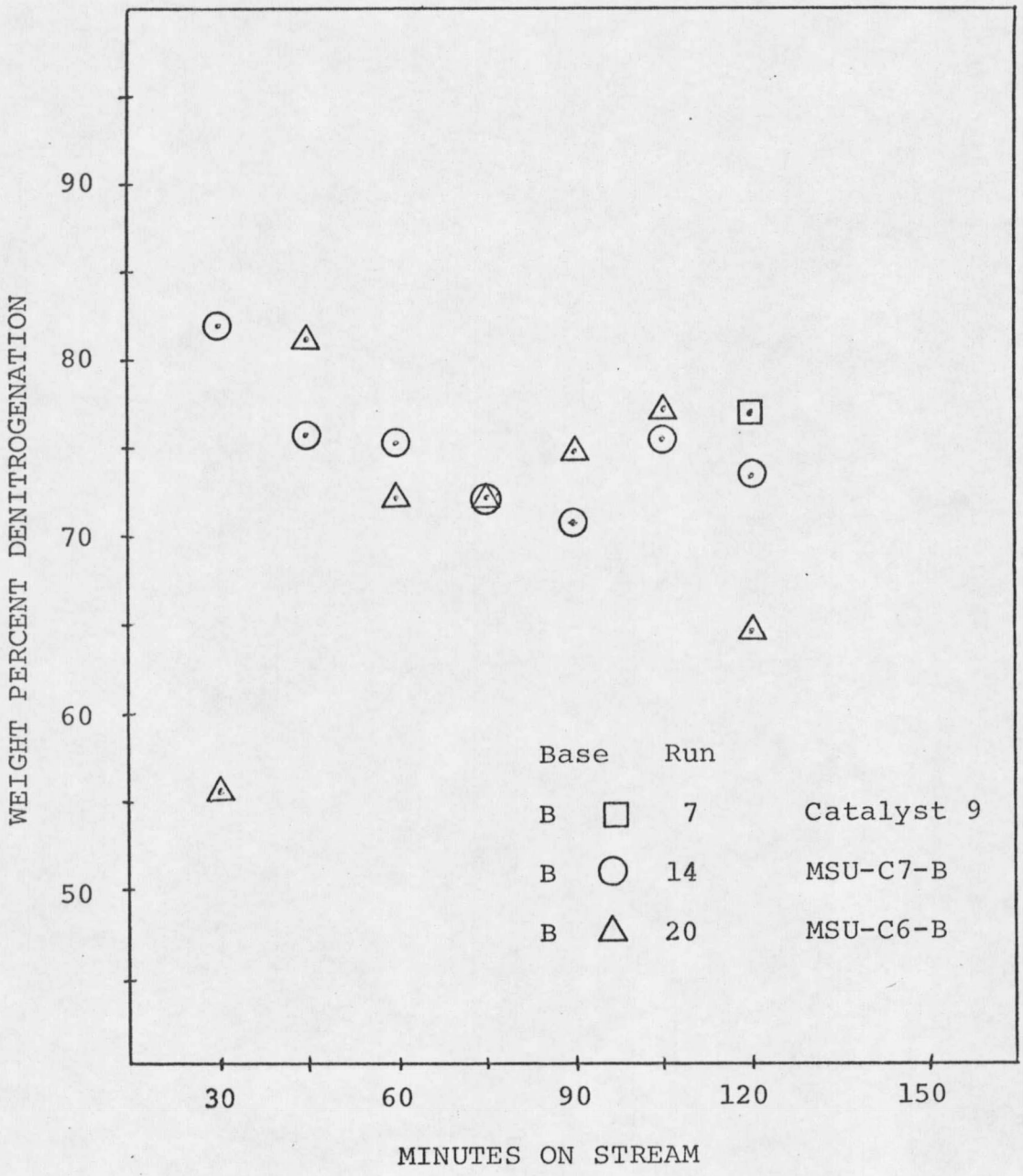


FIGURE 8. NITROGEN REMOVAL vs. RUN TIME FOR BASE B

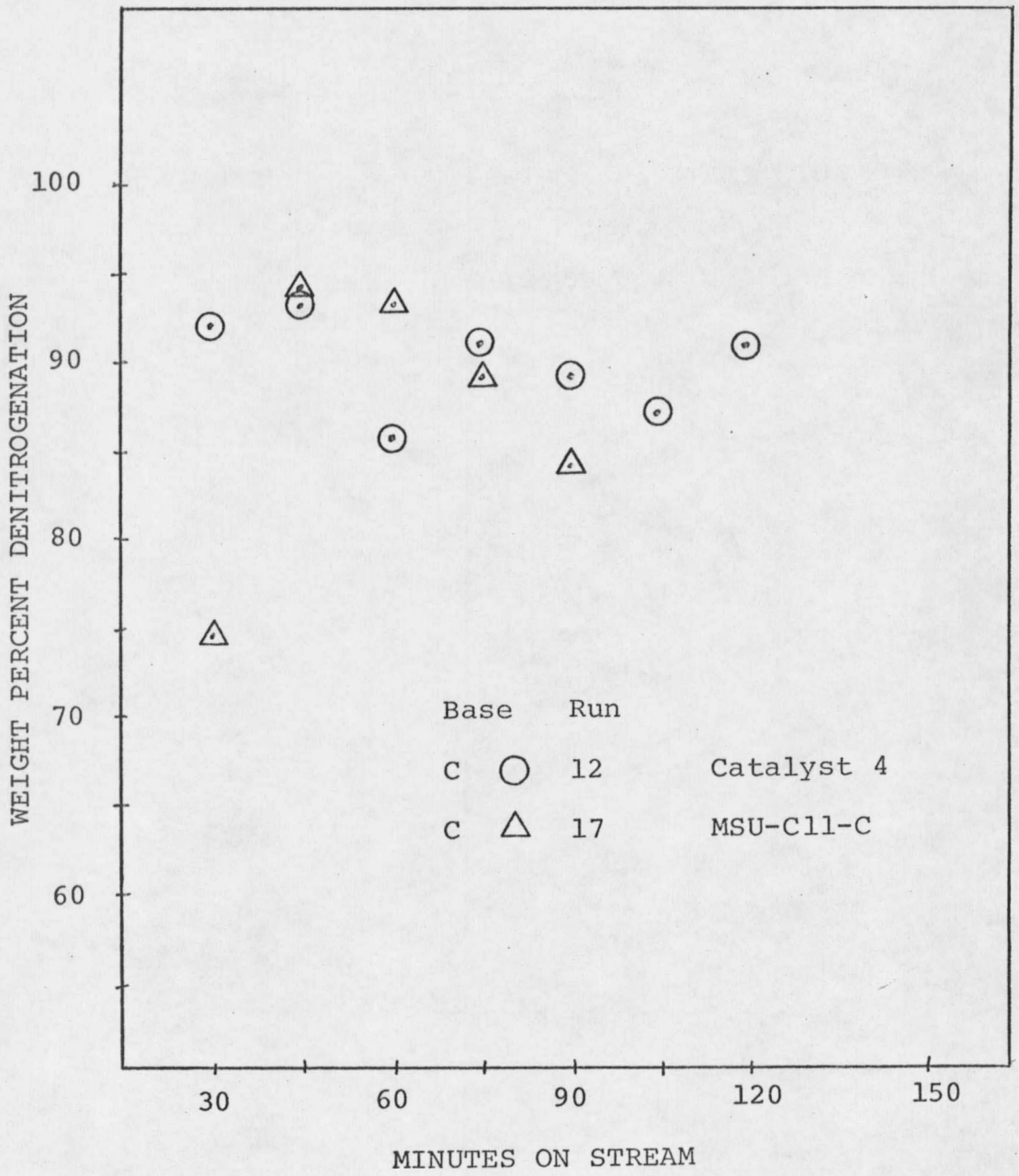


FIGURE 9. NITROGEN REMOVAL vs. RUN TIME FOR BASE C

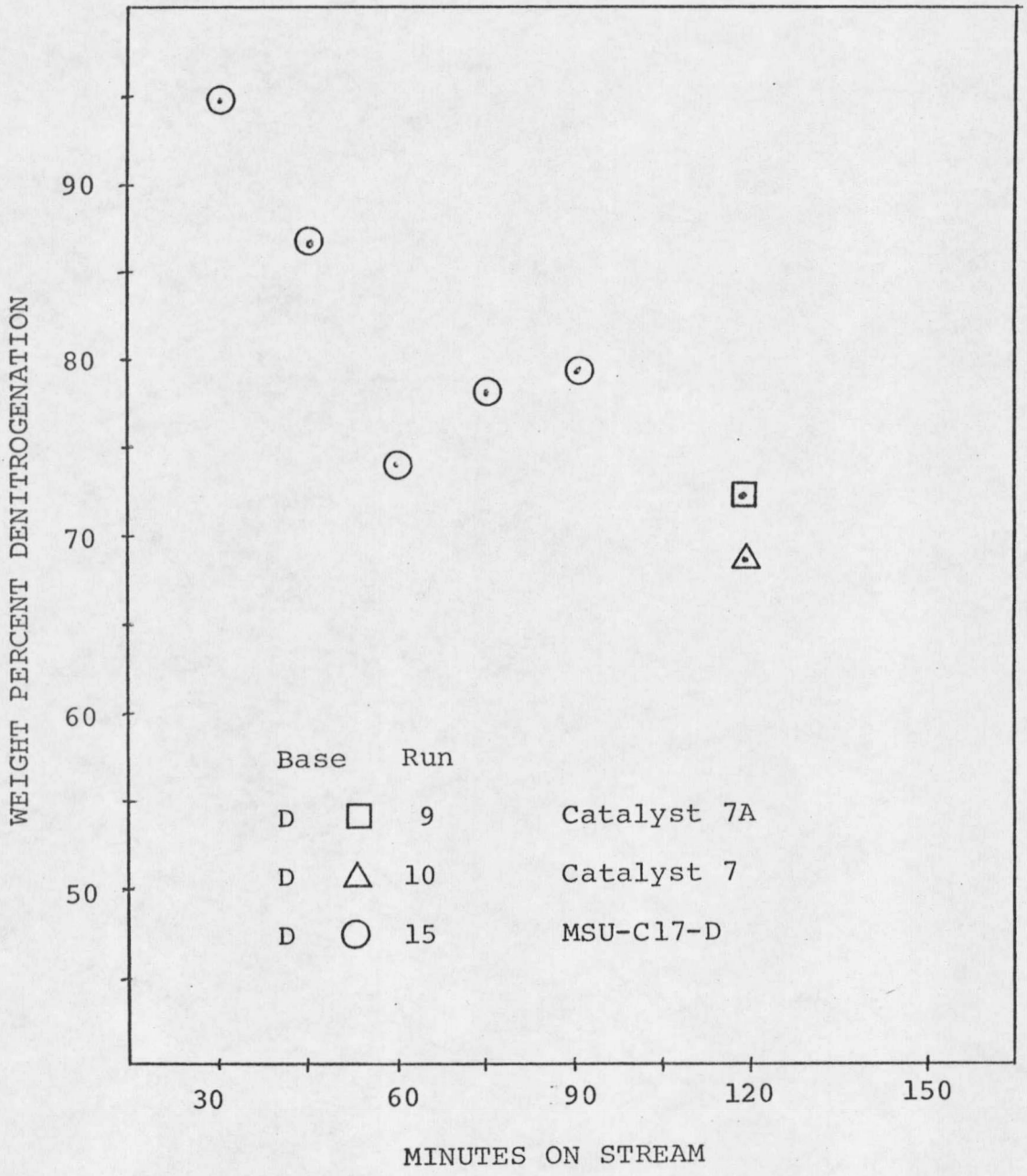


FIGURE 10. NITROGEN REMOVAL vs. RUN TIME FOR BASE D

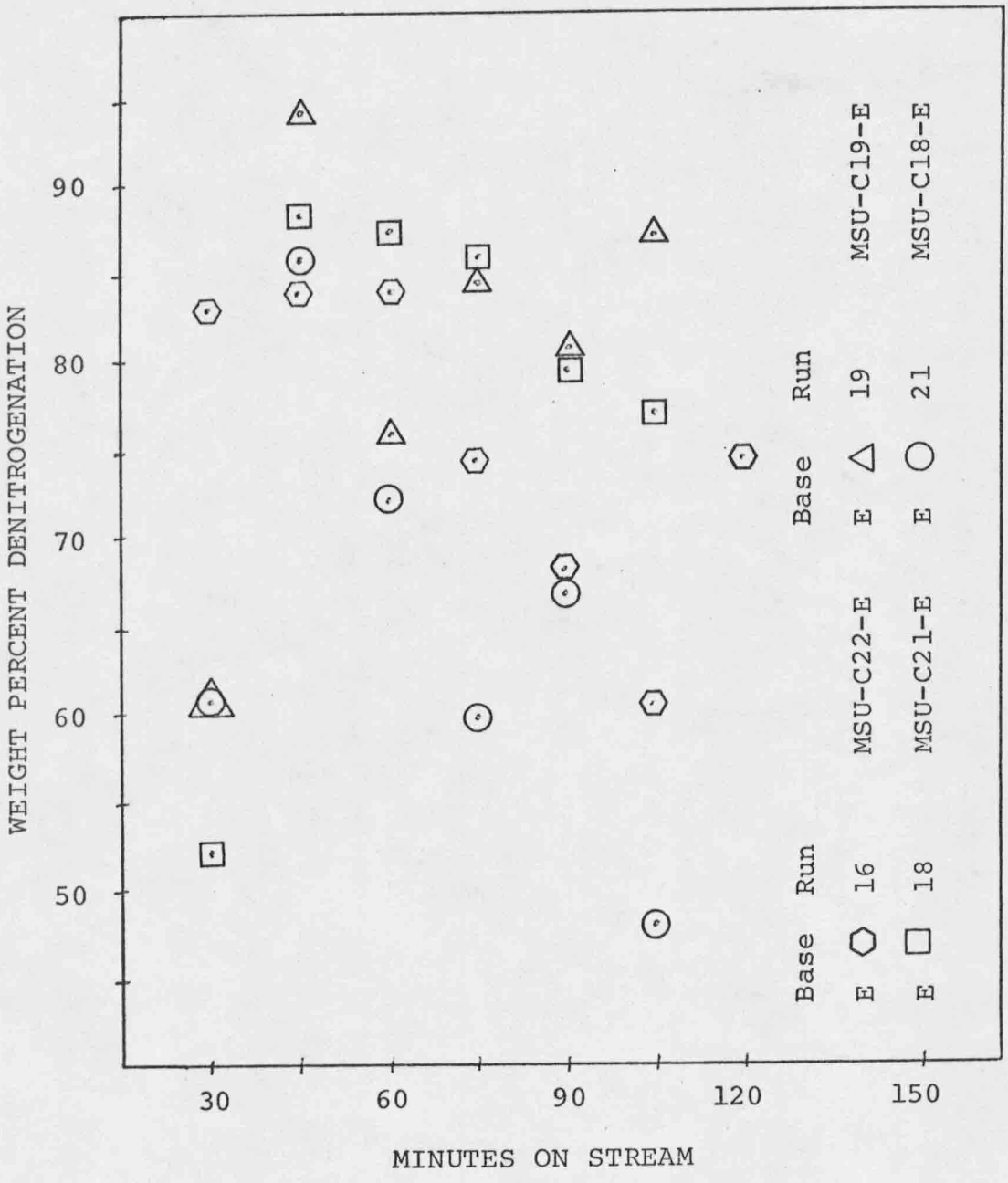


FIGURE 11. NITROGEN REMOVAL vs. RUN TIME FOR BASE E

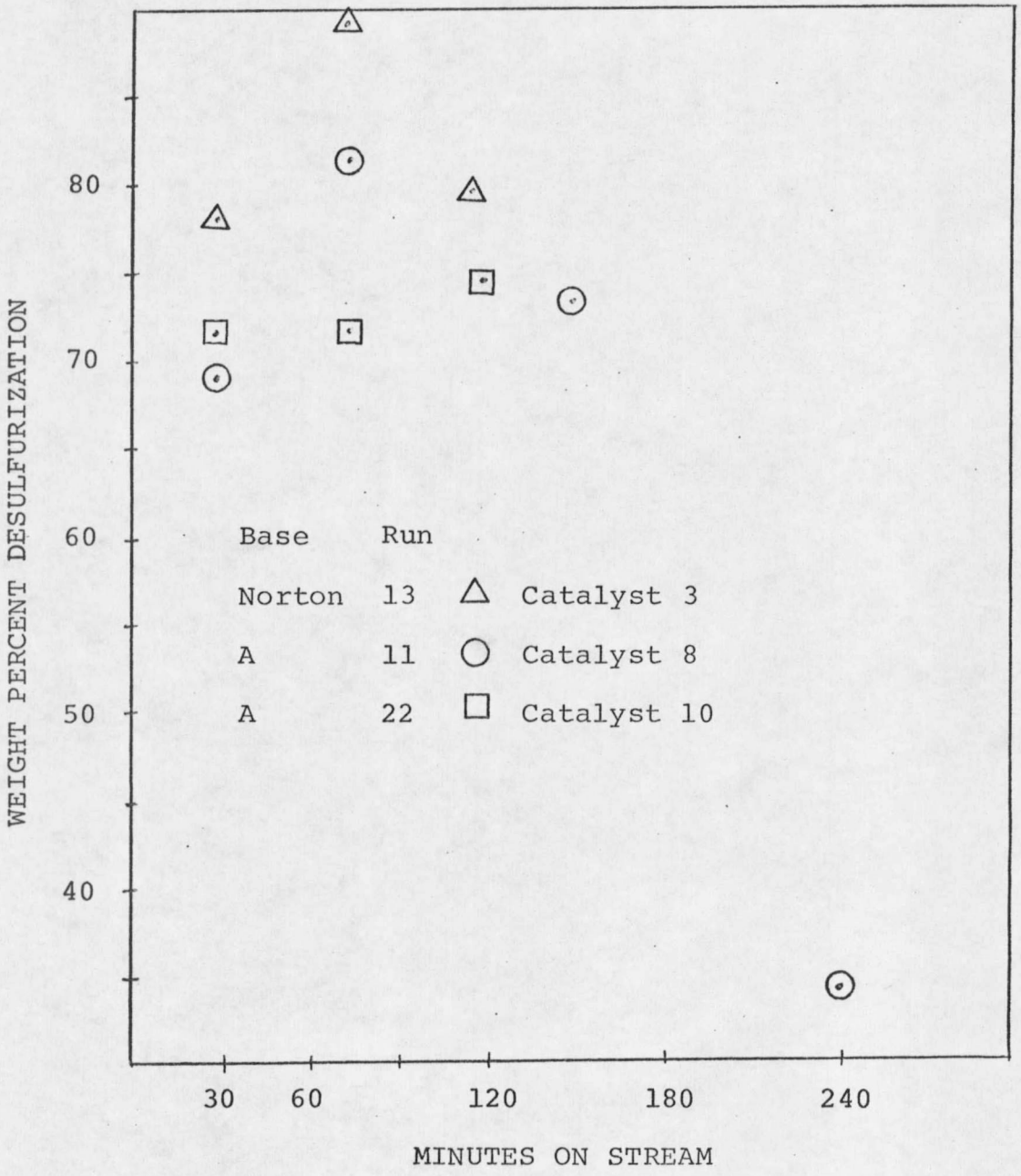


FIGURE 12. SULFUR REMOVAL vs. RUN TIME FOR BASES A AND NORTON 6176

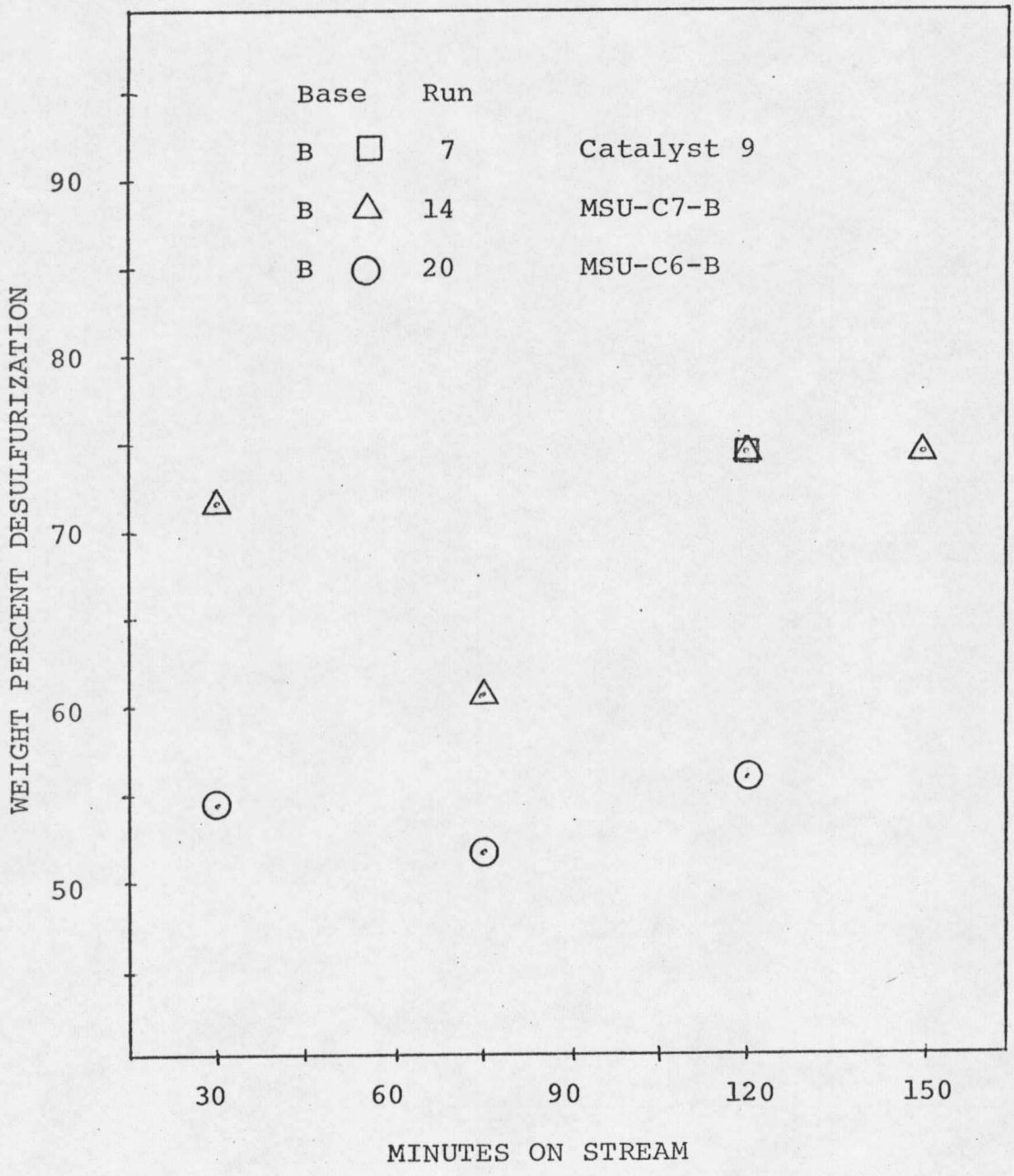


FIGURE 13. SULFUR REMOVAL vs. RUN TIME FOR BASE B

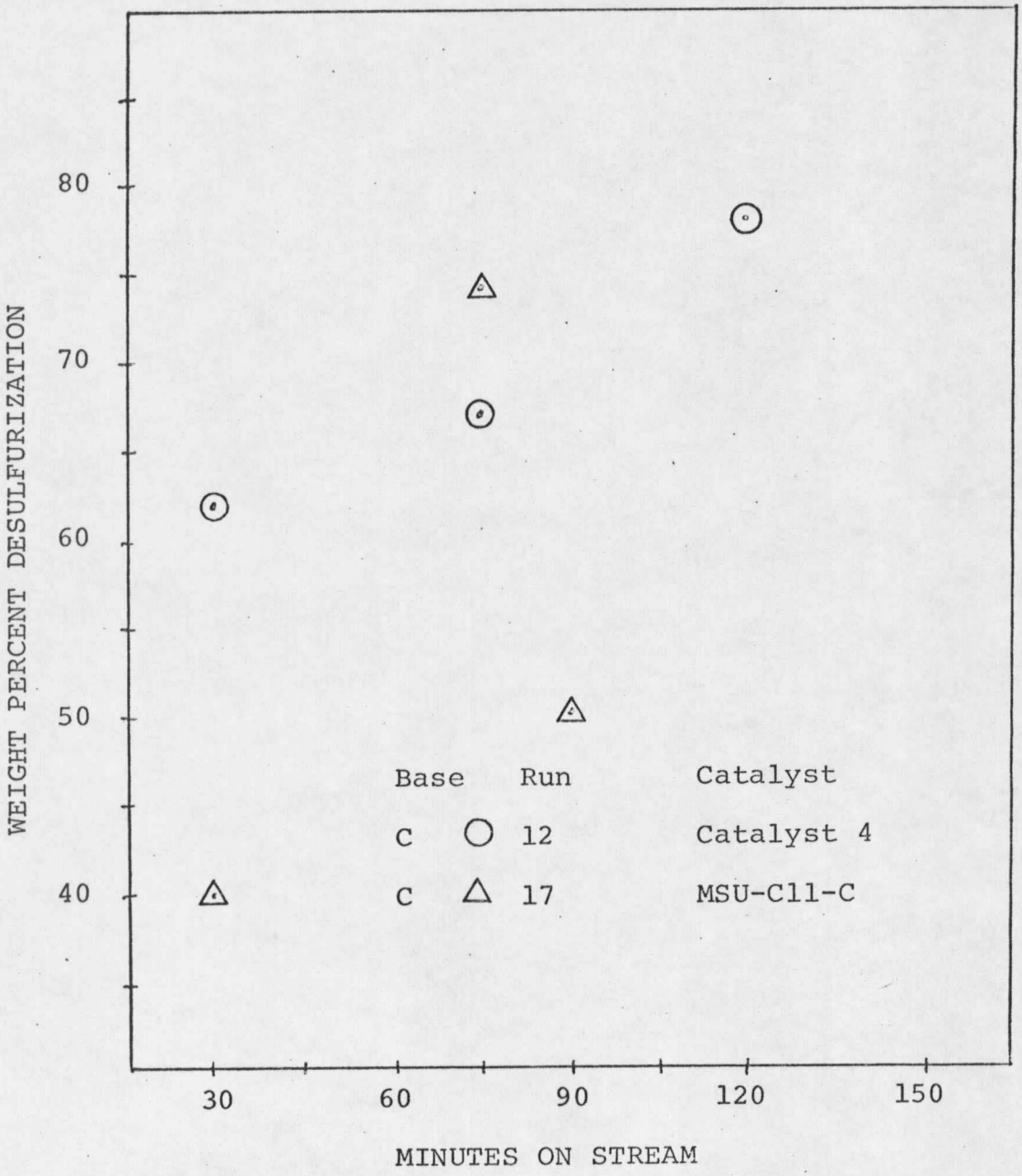


FIGURE 14. SULFUR REMOVAL vs. RUN TIME FOR BASE C

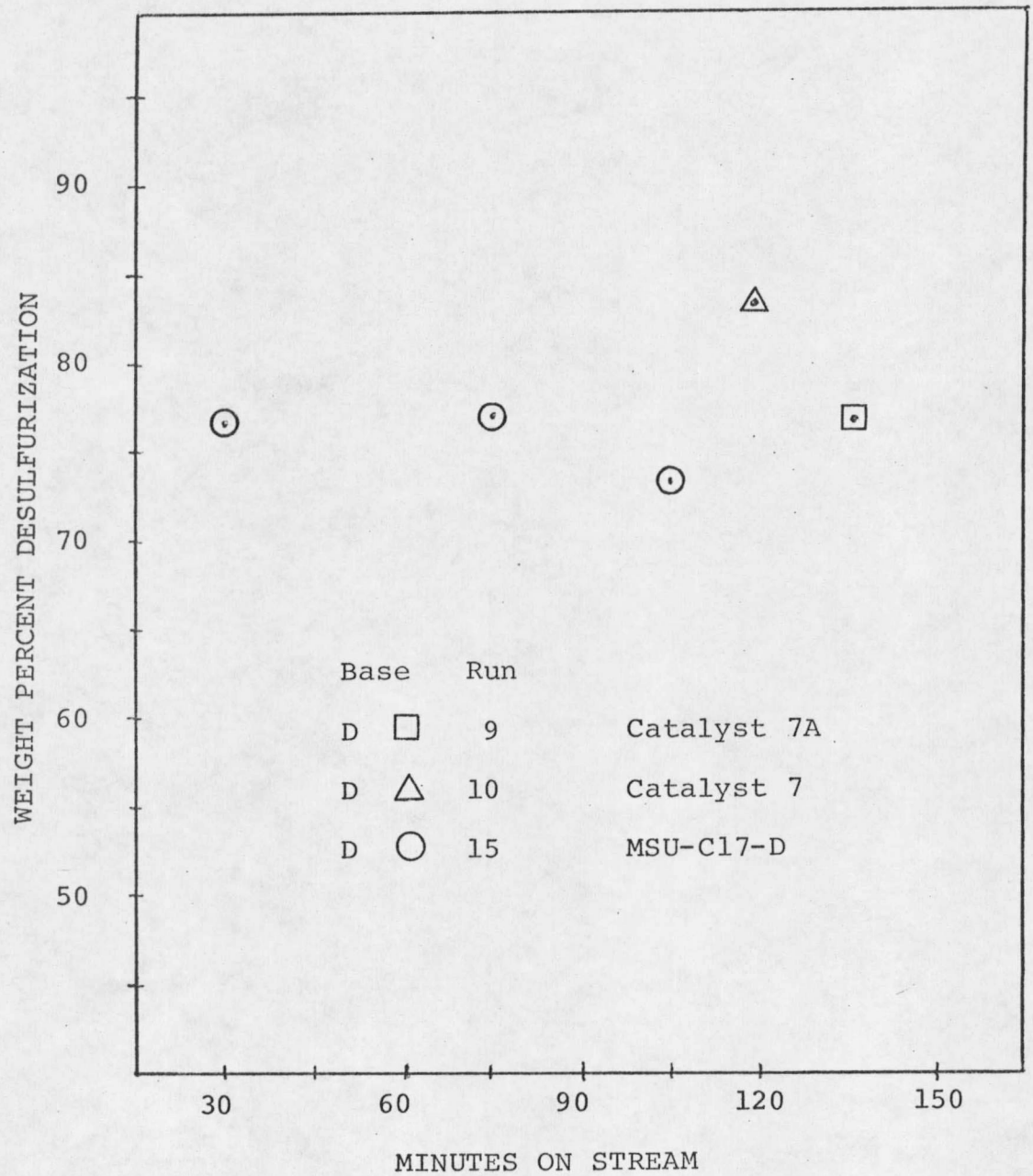


FIGURE 15. SULFUR REMOVAL vs. RUN TIME FOR BASE D

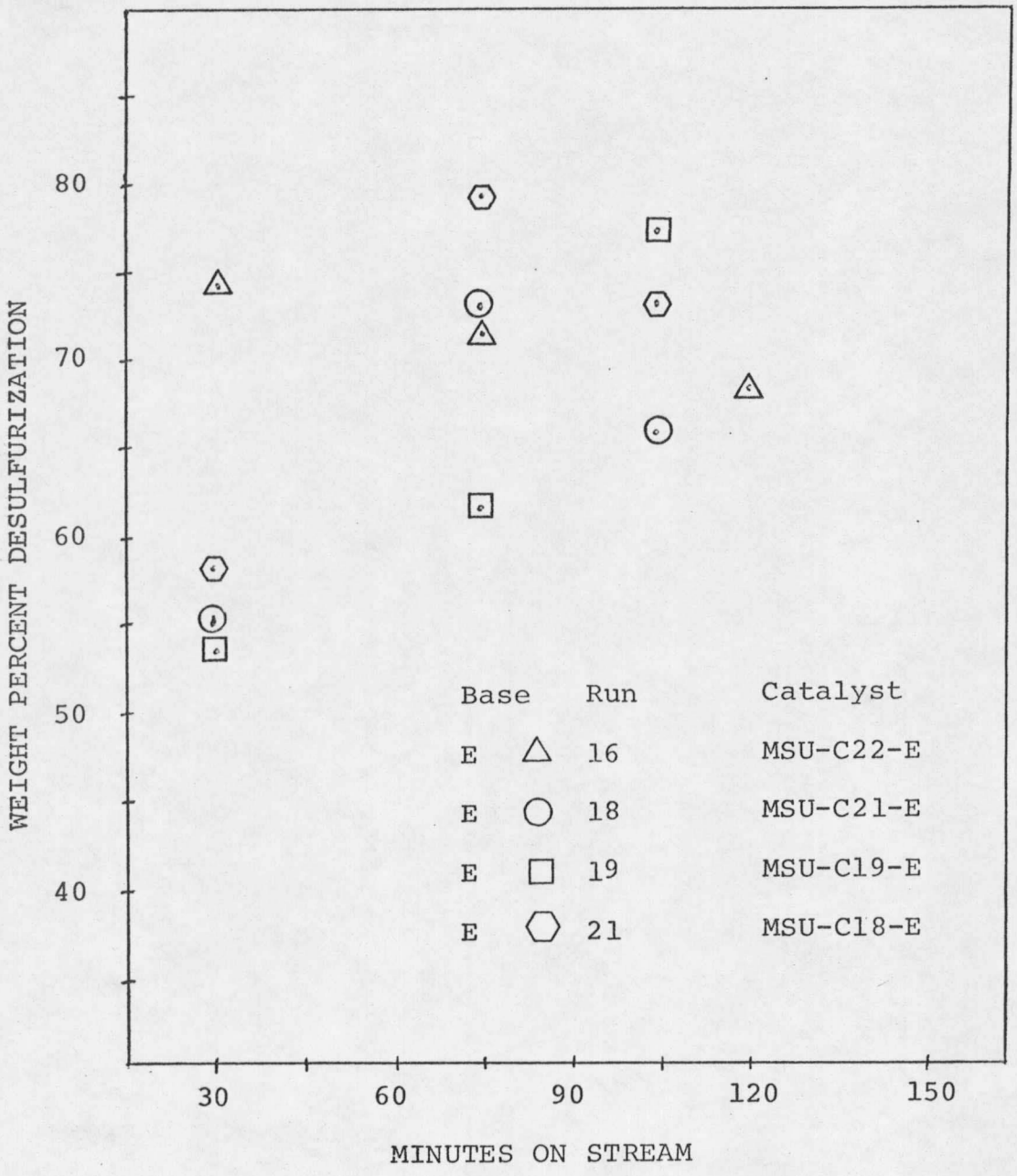


FIGURE 16. SULFUR REMOVAL vs. RUN TIME FOR BASE E

Table IX

Average Percent Denitrogenation and Desulfurization with Respect to Base Pore Diameter

BASE	AVERAGE PORE DIAMETER (4V/A) Å	AVERAGE PERCENT* DN	DS	RUN	CATALYST
A	88.9	85	75	11	8
A	88.9	86	73	22	10
B	124.2	75	73	14	MSU-C7-B
B	124.2	73	54	20	MSU-C6-B
B	124.2	77	75	7	9
D	150.0	73	77	9	7A
D	150.0	69	84	10	7
D	150.0	83	76	15	MSU-C17-D
Norton	152.0	91	83	13	3
C	156.5	90	69	12	4
C	156.5	90	55	17	MSU-C11-E
E	186.2	78	71	16	MSU-C22-E
E	186.2	84	65	18	MSU-C21-E
E	186.2	84	64	19	MSU-C19-E
E	186.2	69	70	21	MSU-C18-E

*Based on length of run or two hours whichever is shorter.

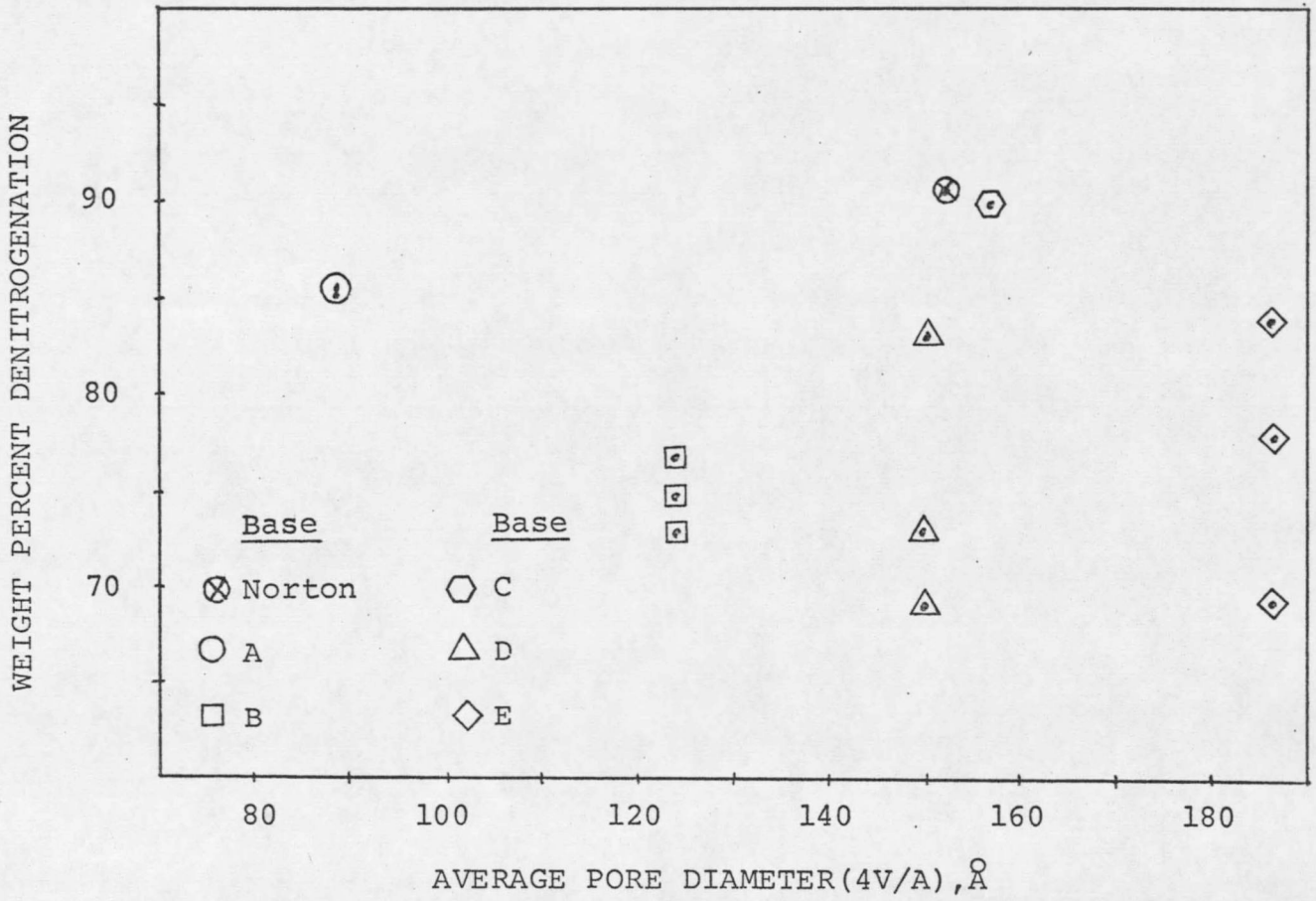


FIGURE 17. EFFECT OF BASES ON NITROGEN REMOVAL

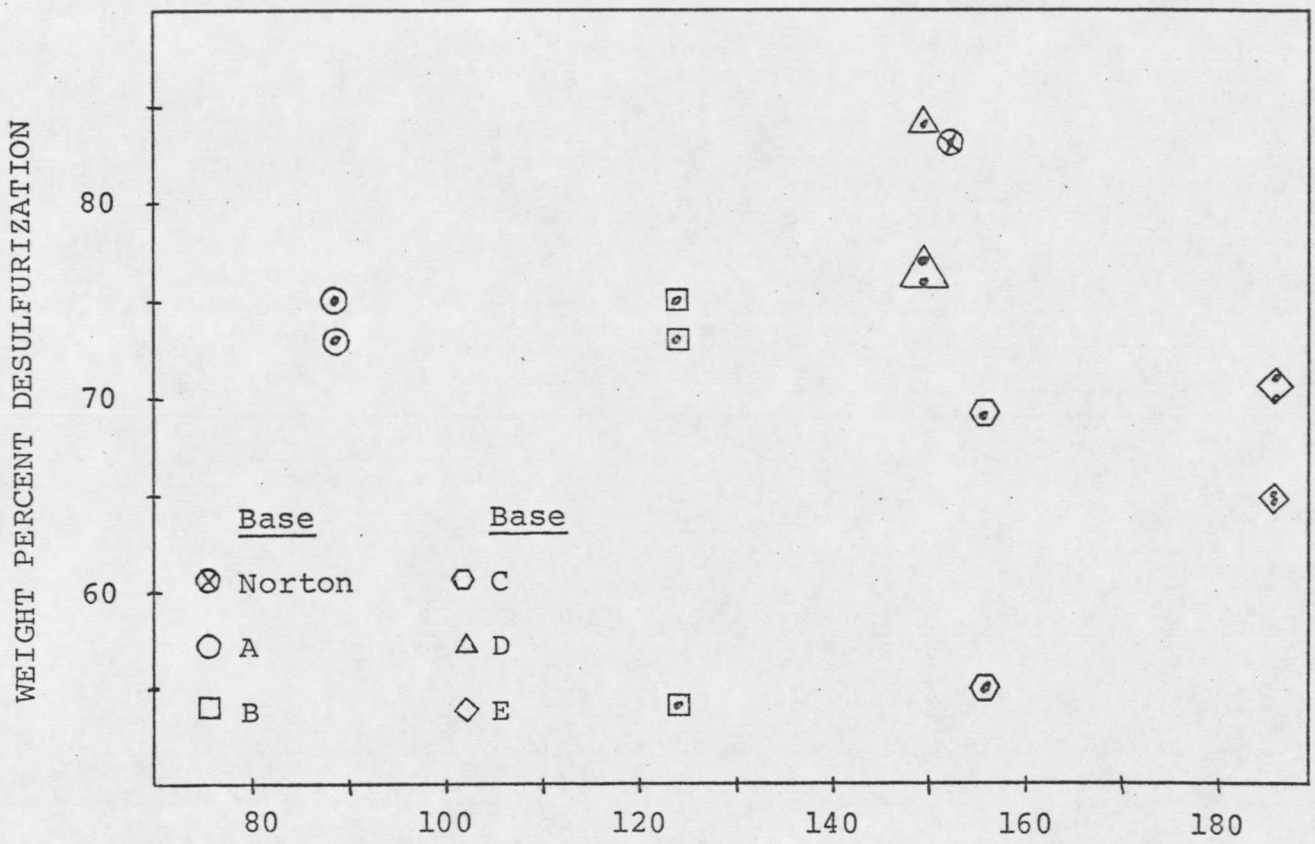


FIGURE 18. EFFECT OF BASES ON SULFUR REMOVAL

CONCLUSIONS

1. Batch runs do not satisfactorily pretest catalysts. Short continuous runs should be used.
2. Short runs can produce nitrogen levels below 0.3 wt% and sulfur levels below 0.5 wt%.
3. The precoking of the SRC-II appears to reduce the amount of catalyst deactivation in nitrogen removal.
4. Catalyst deactivation is rapid with respect to nitrogen removal; however, sulfur removal is not.
5. Larger catalyst beds do not greatly affect results.
6. Catalyst 3 (12.6% MoO_3, 10.3% WO_3, 2.8% NiO, 6.8% CoO) was the best catalyst in removing nitrogen and sulfur. The average pore diameter of the base (Norton 6176) is 152 Å.

RECOMMENDATION FOR FUTURE RESEARCH

1. Batch runs are not very reliable for pretesting catalysts; continuous runs should be made on all catalysts.
2. A multi-preheat zone reactor should be developed. This would allow continuation of a run by switching from the plugged up preheat sone to a new preheat zone.
3. Used MSU catalysts should be analyzed to determine the amount of carbon laydown.
4. Once used Montana State University fabricated catalysts should be regenerated and then retested.
5. Larger catalyst beds should be used to increase sample sizes. This would allow more distillation analyses to be done.
6. One catalyst should be tested in both Yeh's reactor and the author's reactor to varify the coking effects.
7. The temperature should be increased in a run using Yeh's reactor to see if coking will occur.

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LITERATURE CITED

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APPENDIX A

GRAPHICAL REPRESENTATION

OF

BATCH AND CONTINUOUS RUNS

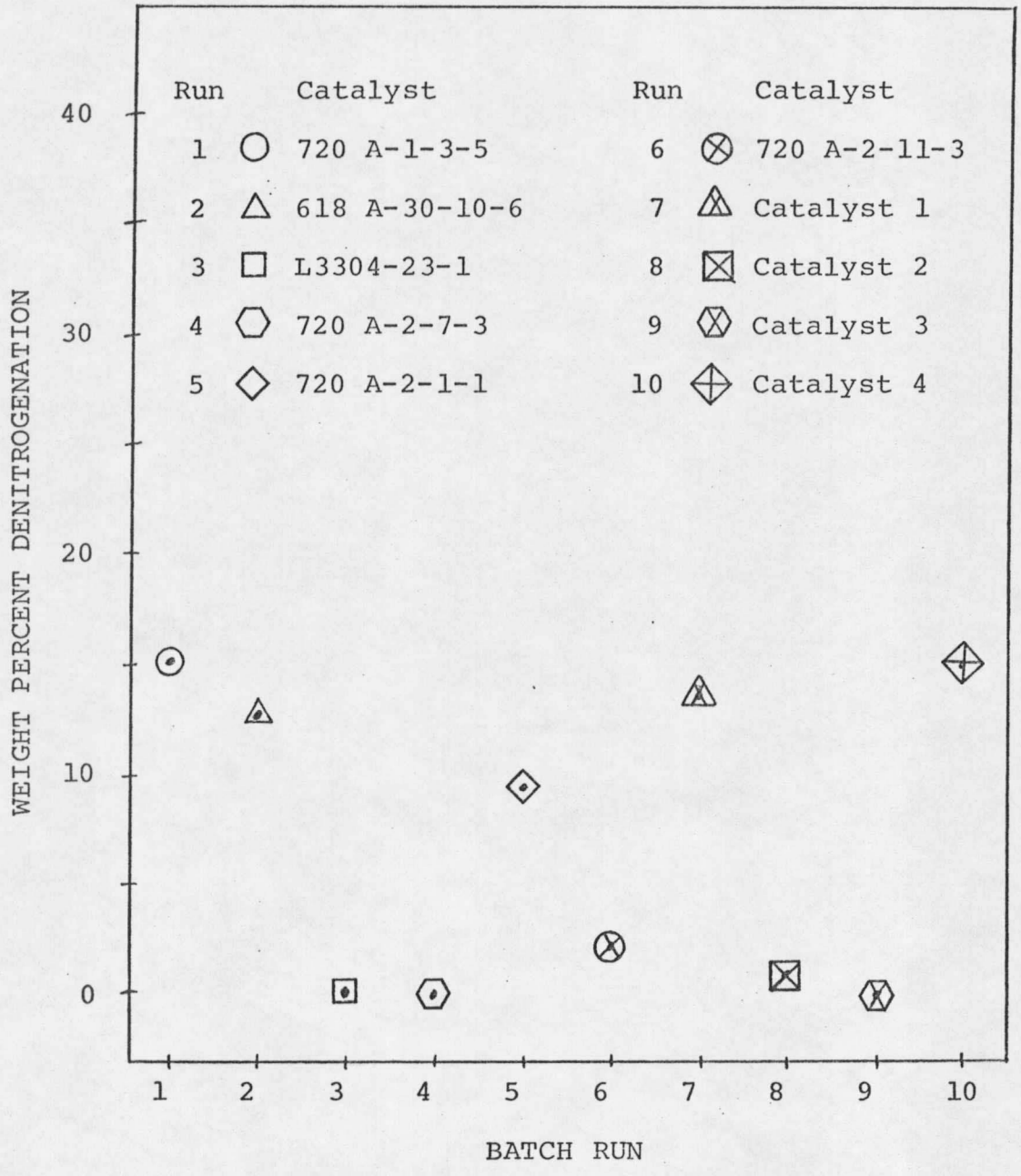


FIGURE 19. NITROGEN REMOVAL IN BATCH RUNS

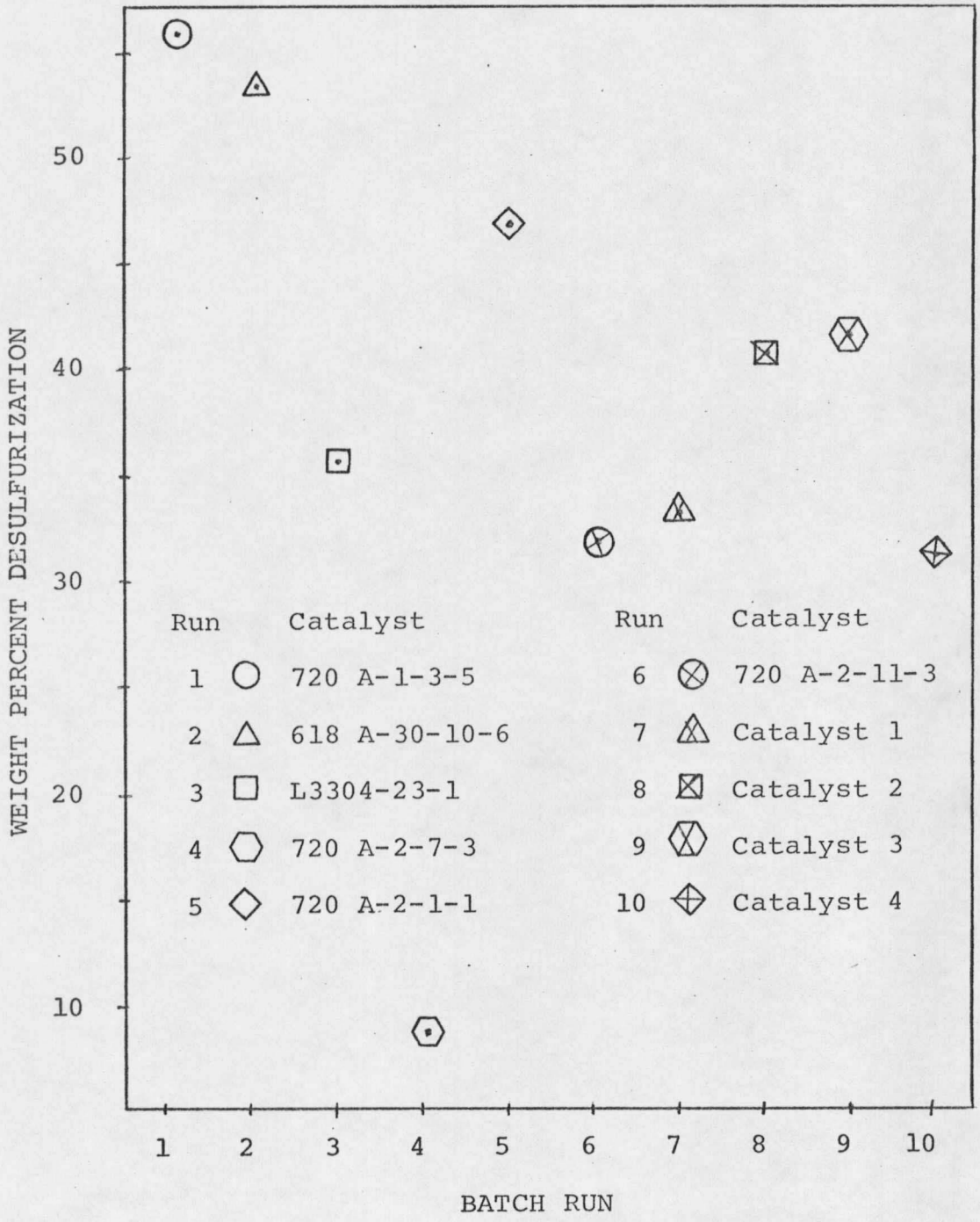


FIGURE 20. SULFUR REMOVAL IN BATCH RUNS

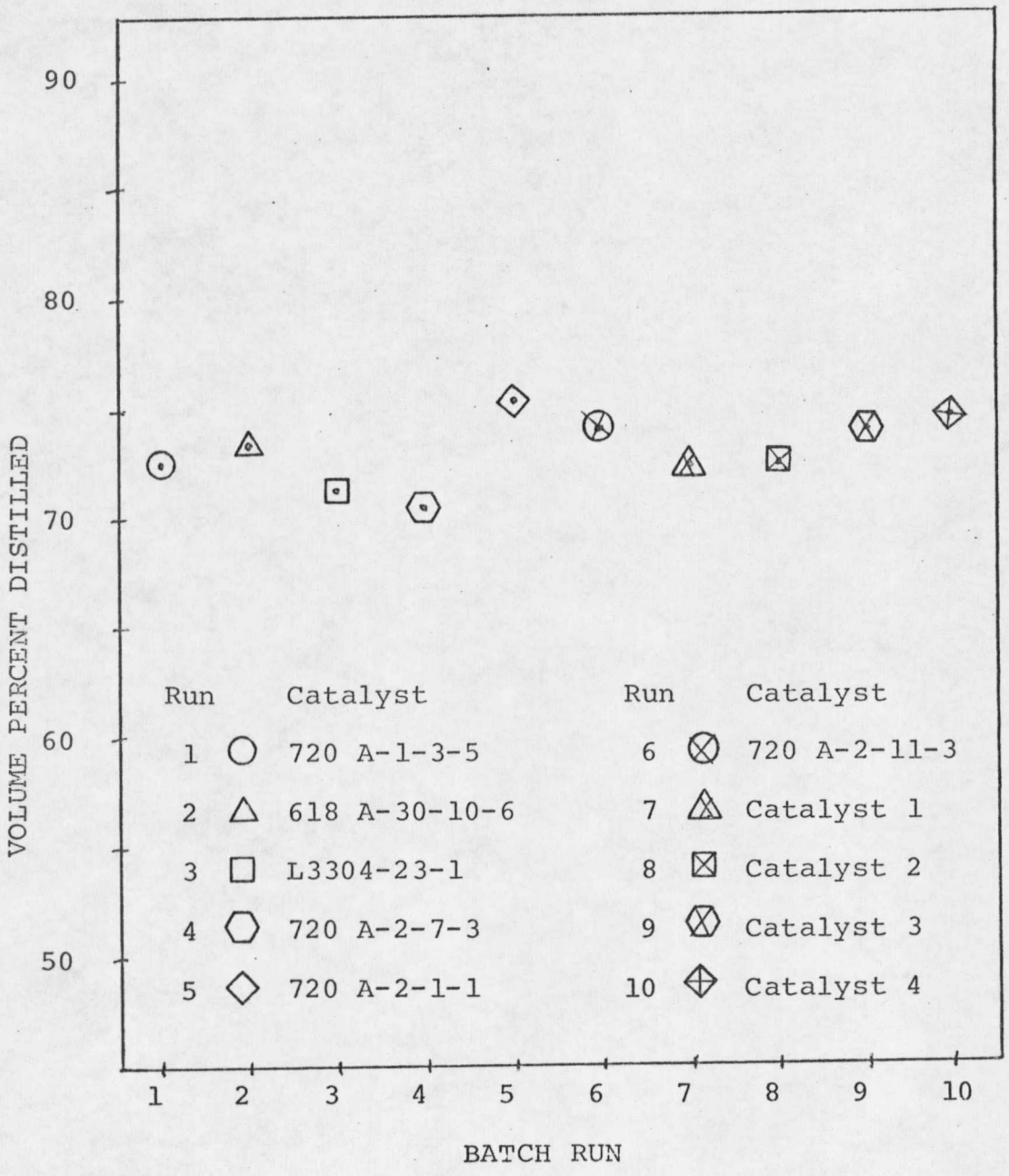


FIGURE 21. DISTILLATE YIELDS IN BATCH RUNS

FIGURES 22-31

THE EFFECT OF CATALYST DEACTIVATION

ON NITROGEN REMOVAL

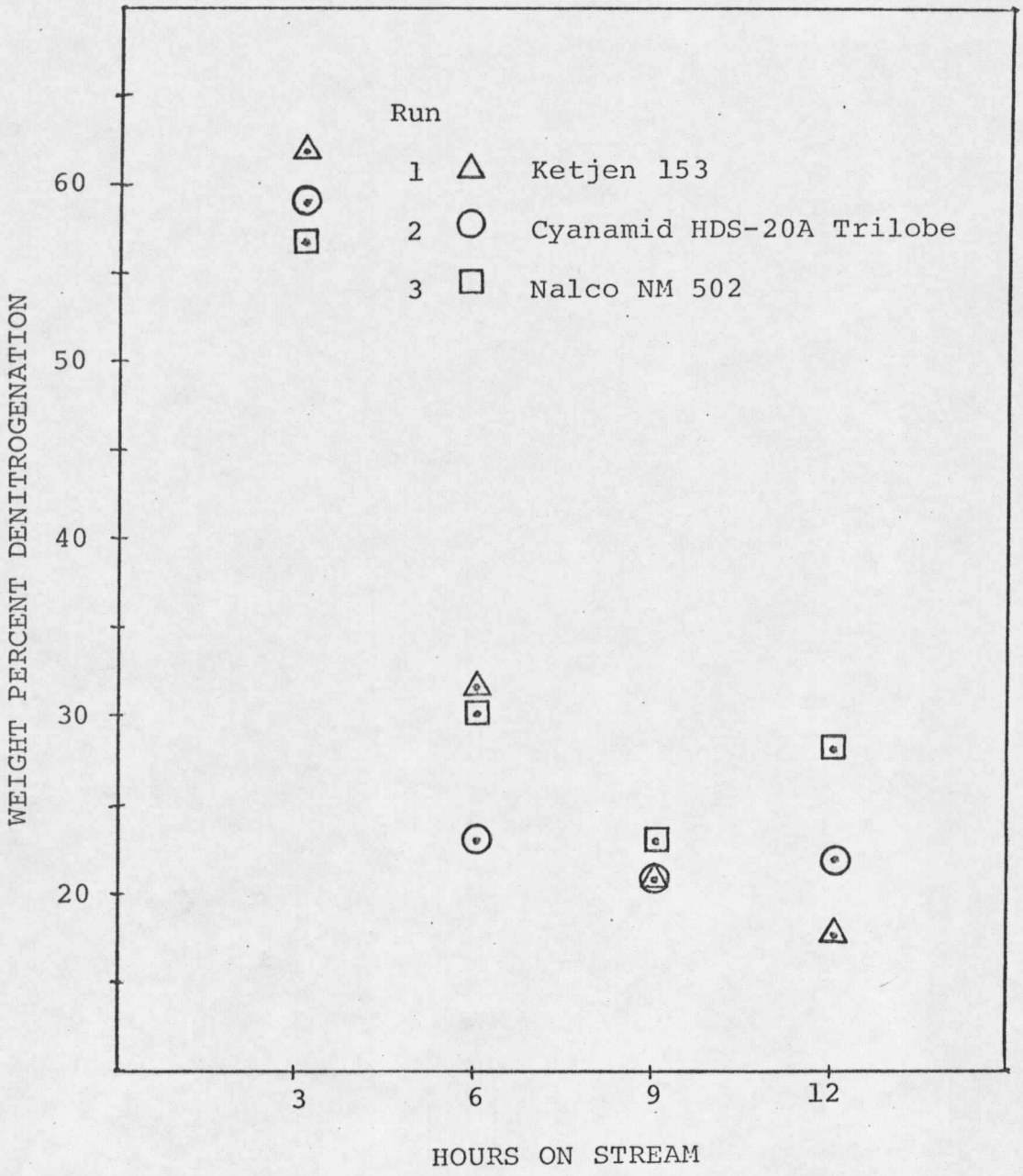


FIGURE 22. RUNS 1, 2, AND 3

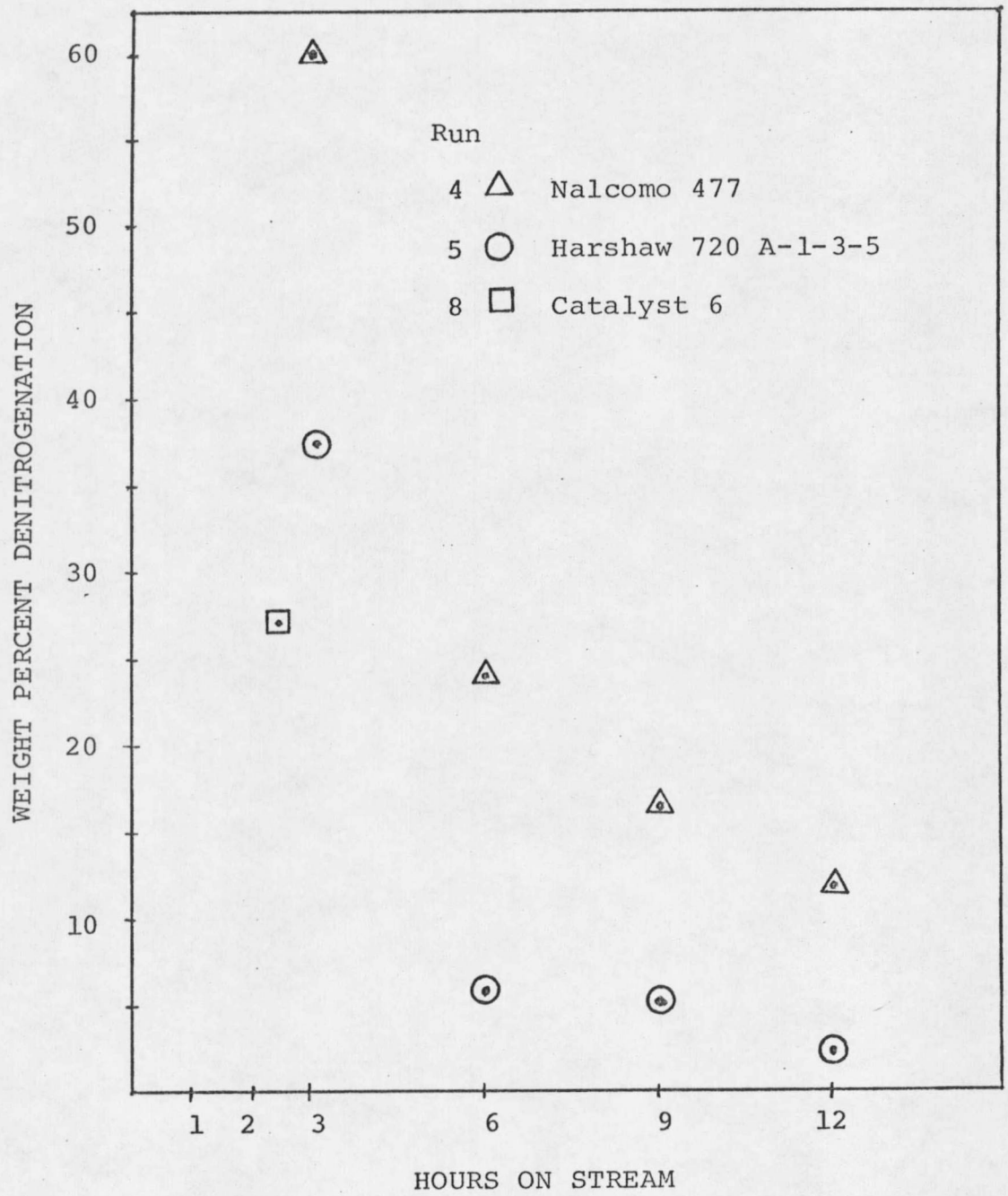


FIGURE 23. RUNS 4, 5, AND 8

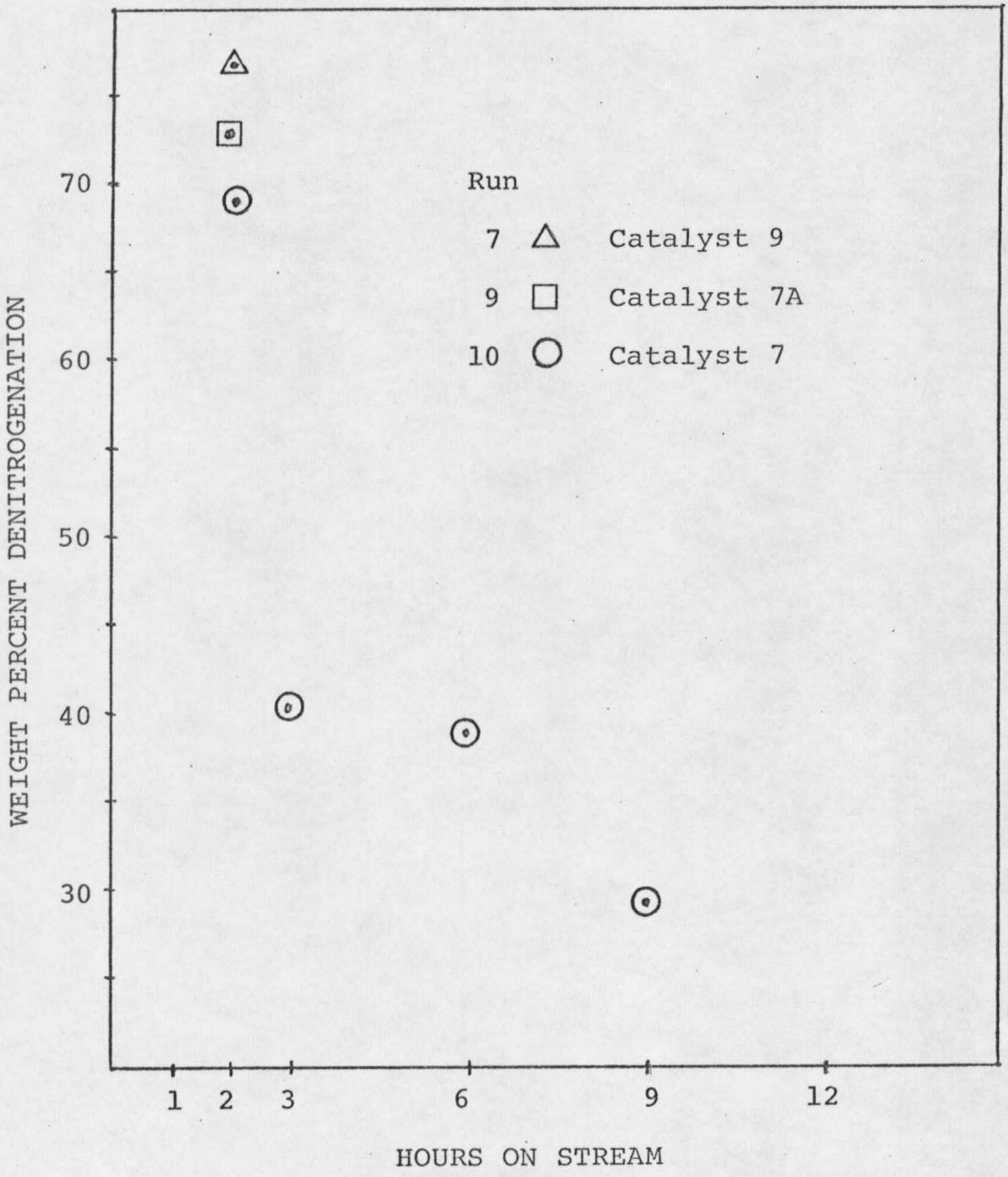


FIGURE 24. RUNS 7, 9, AND 10

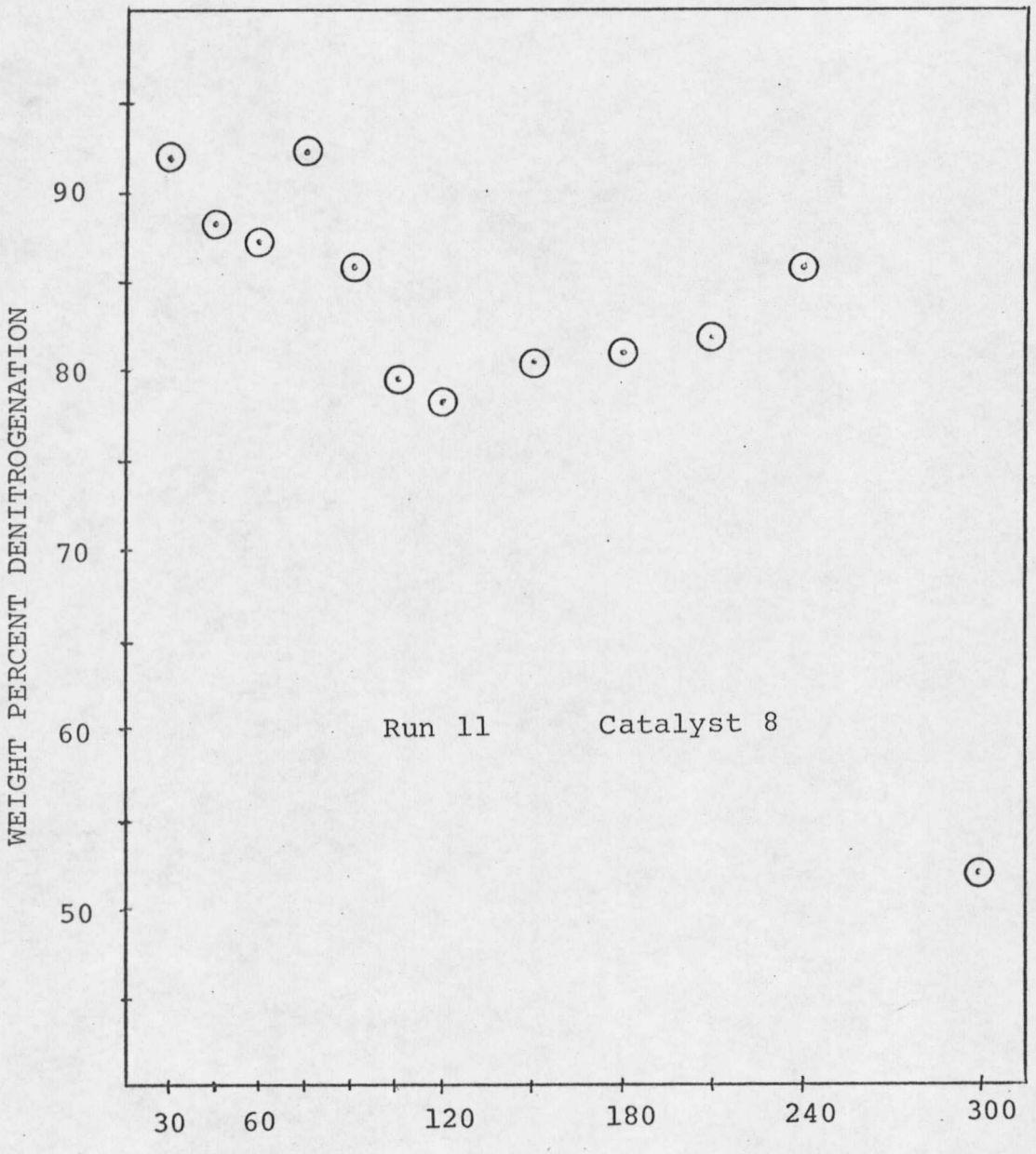


FIGURE 25. RUN 11

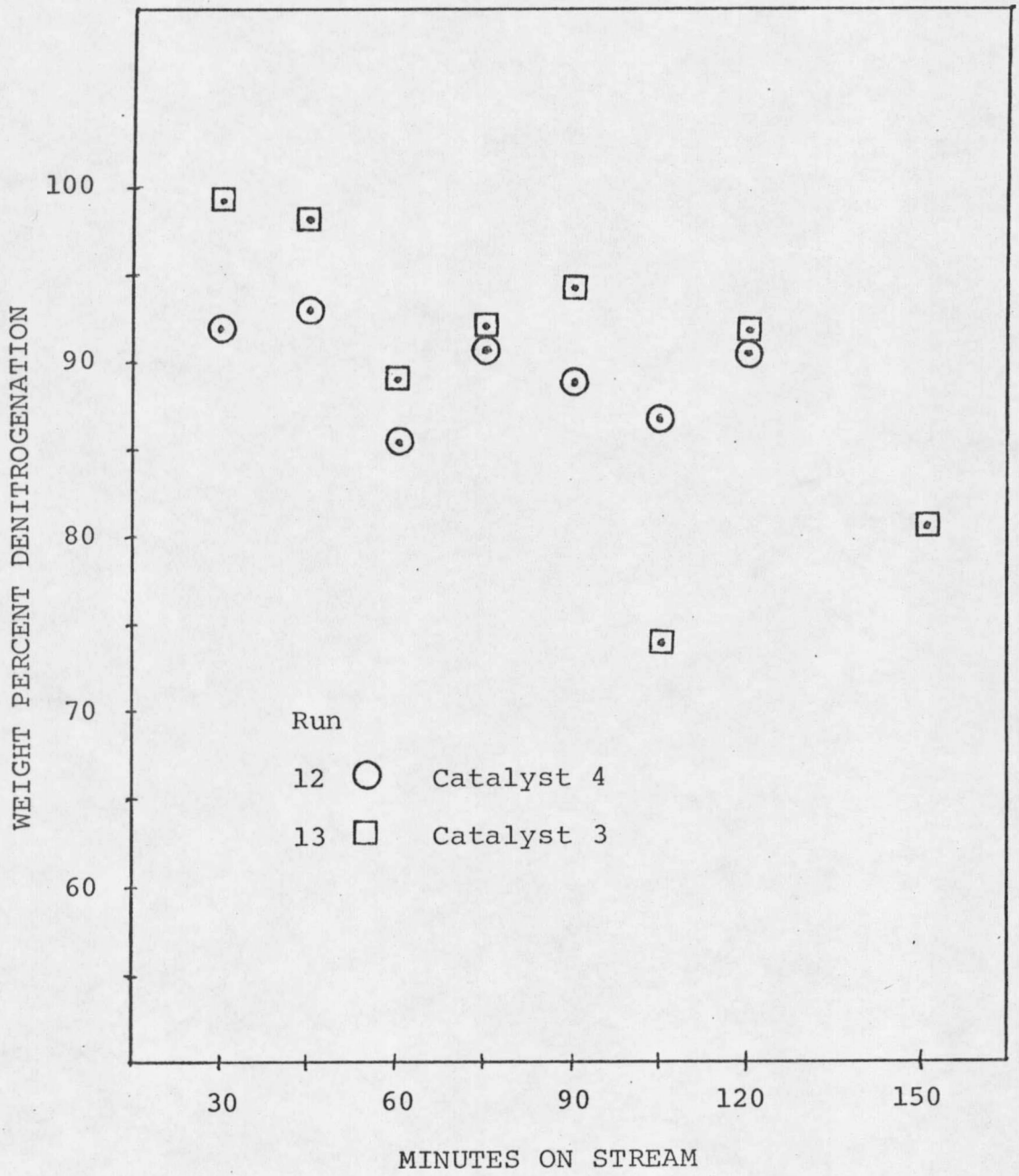


FIGURE 26. RUNS 12 AND 13

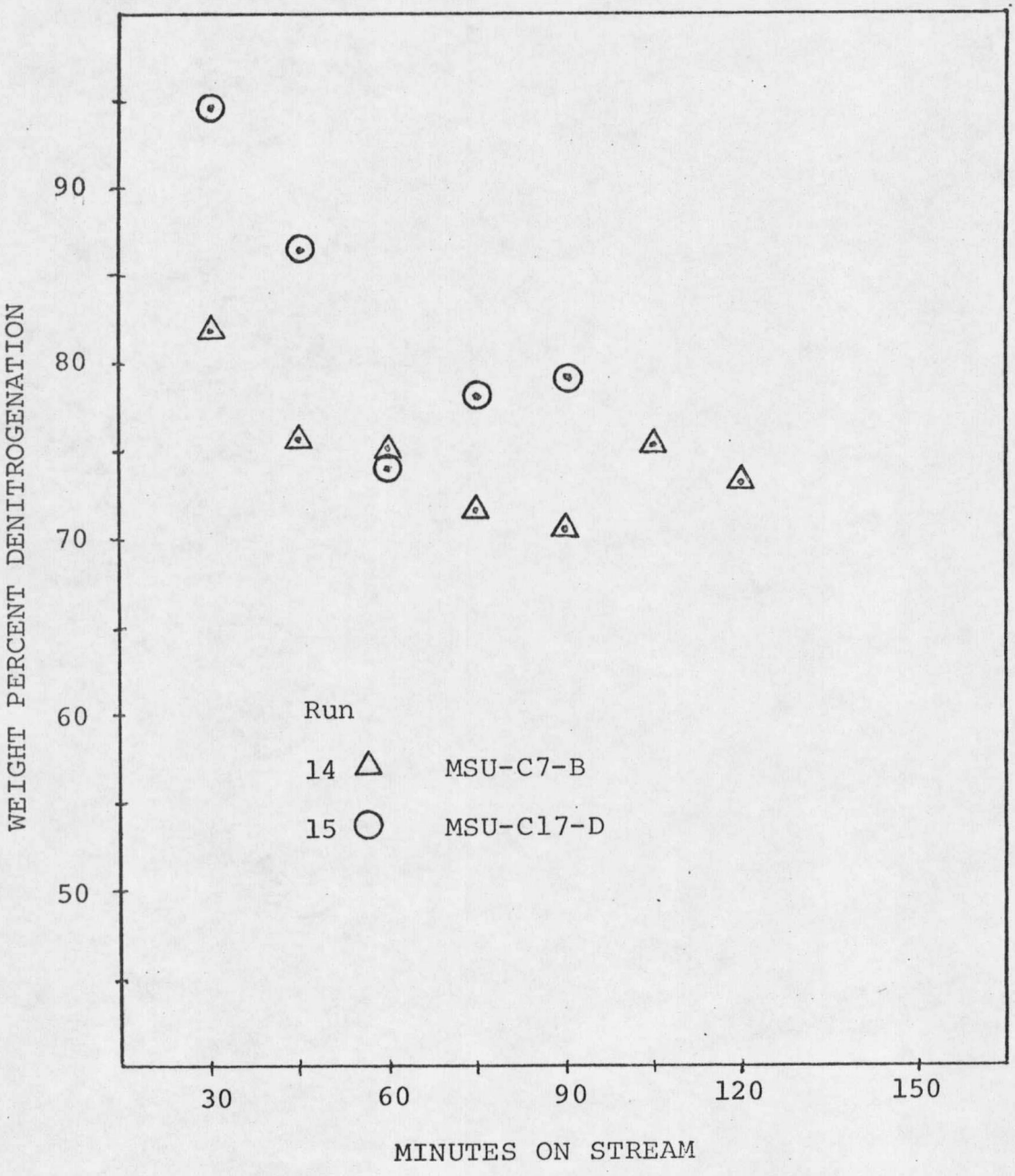


FIGURE 27. RUNS 14 AND 15

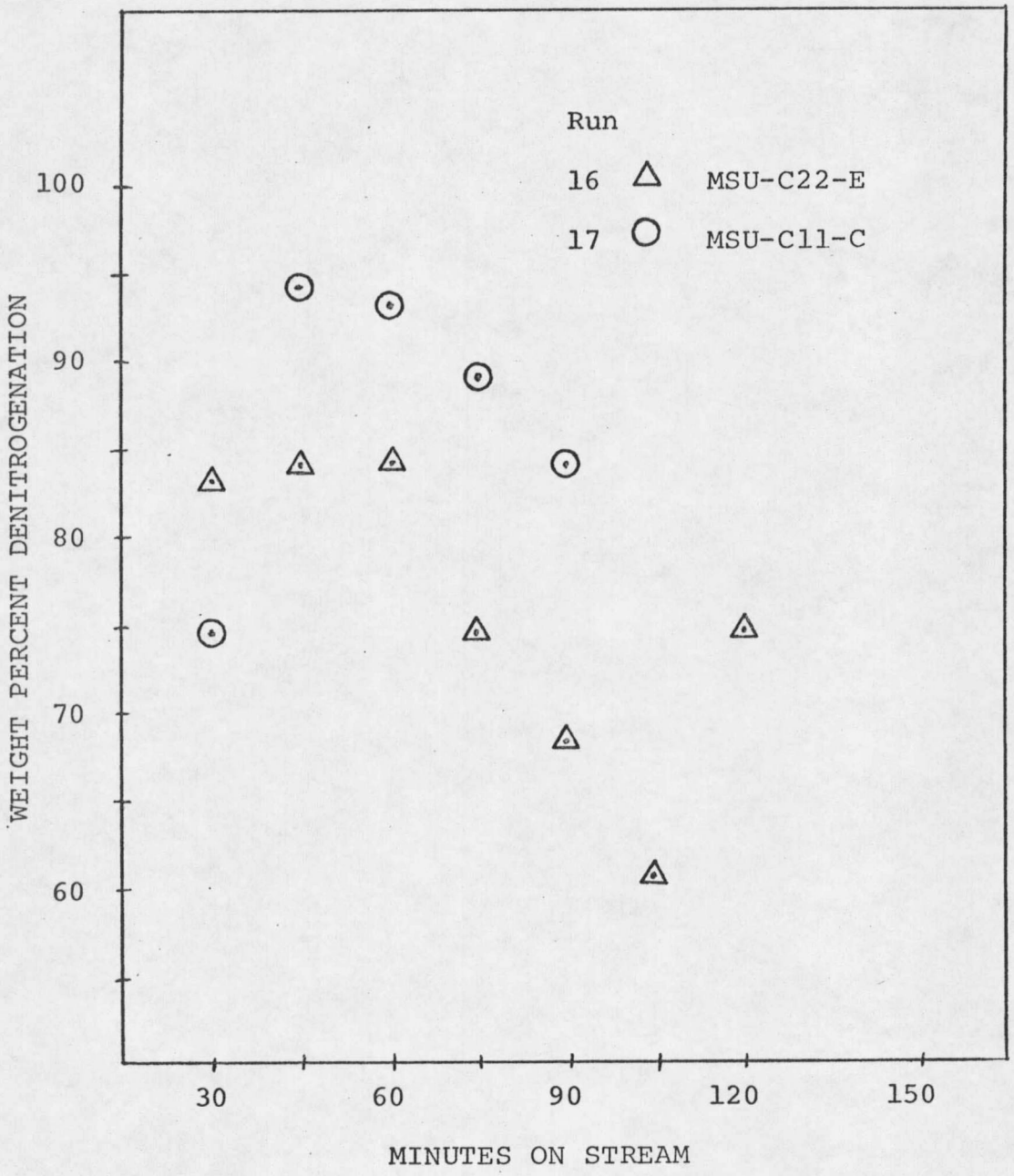


FIGURE 28. RUNS 16 AND 17

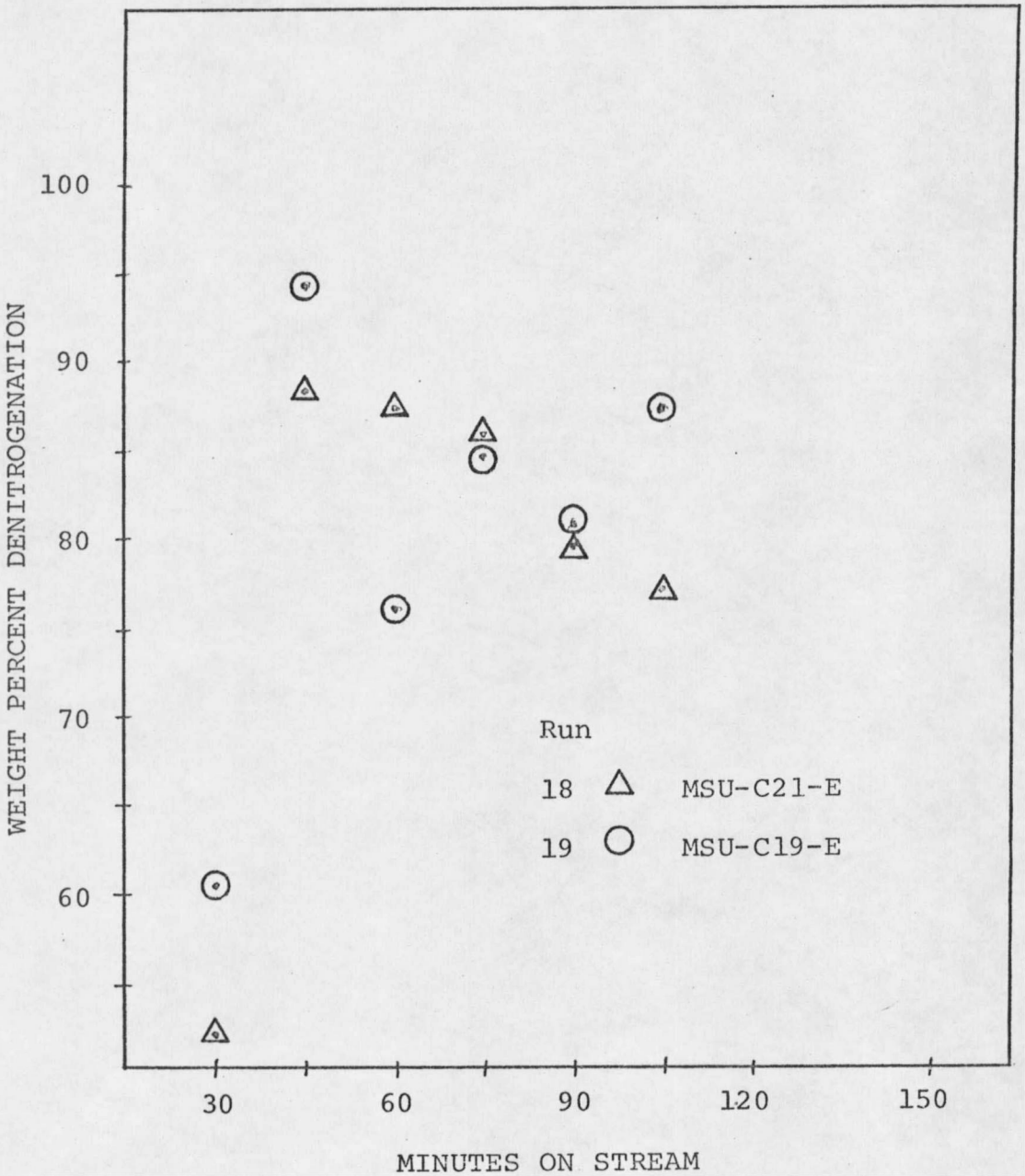


FIGURE 29. RUNS 18 AND 19

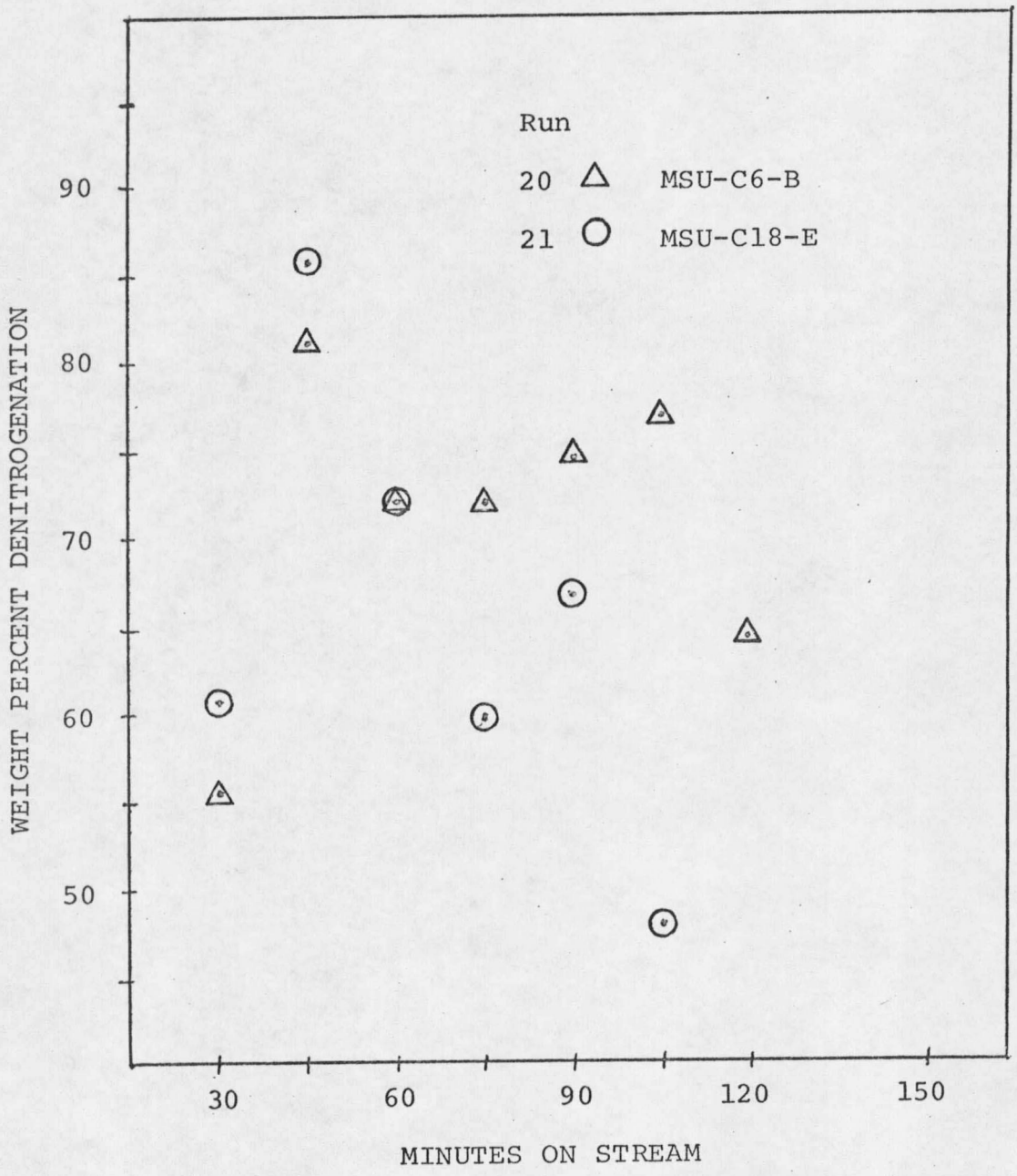


FIGURE 30. RUNS 20 AND 21

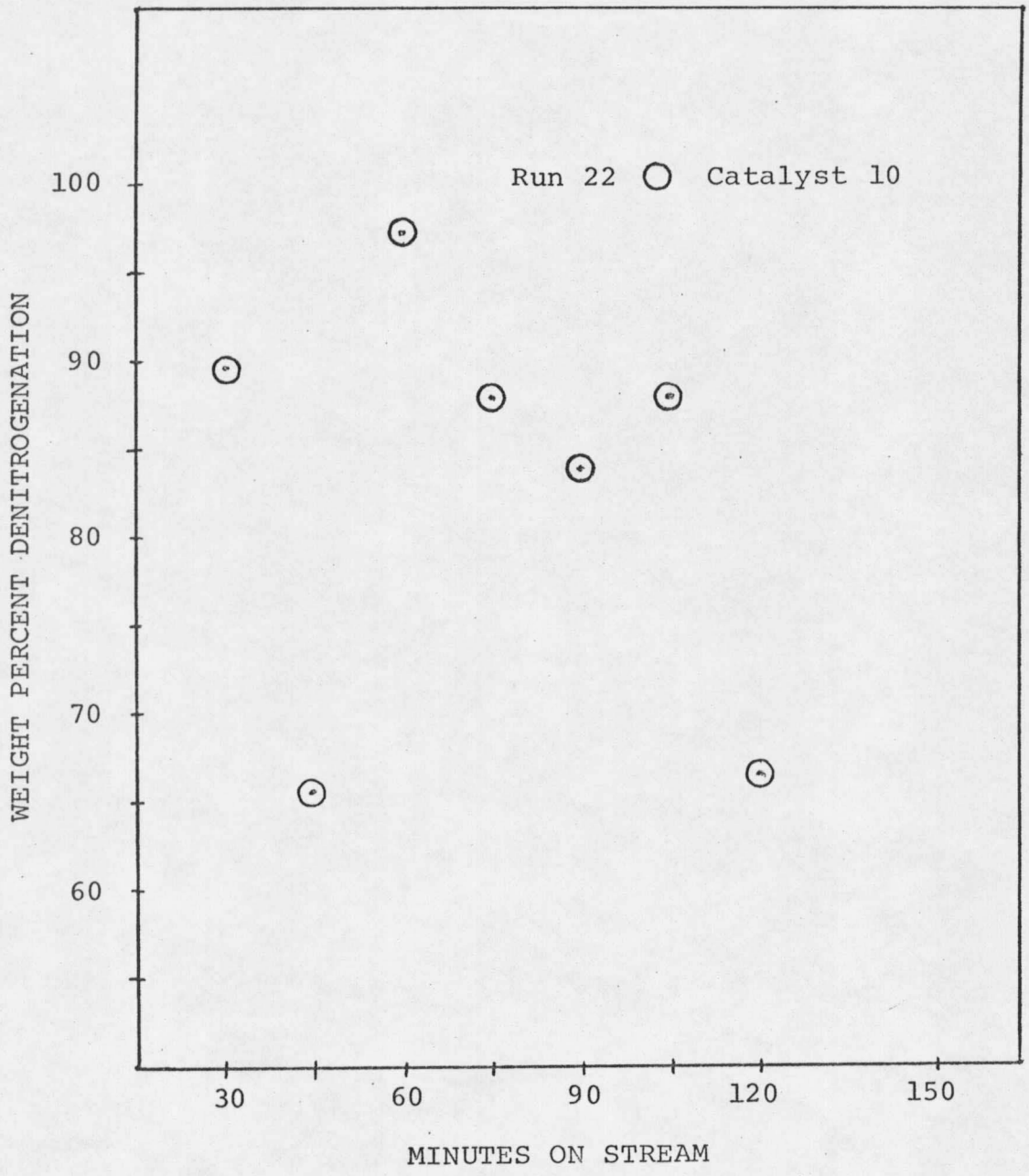


FIGURE 31. RUN 22

FIGURES 32-38

THE EFFECT OF CATALYST DEACTIVATION

ON SULFUR REMOVAL

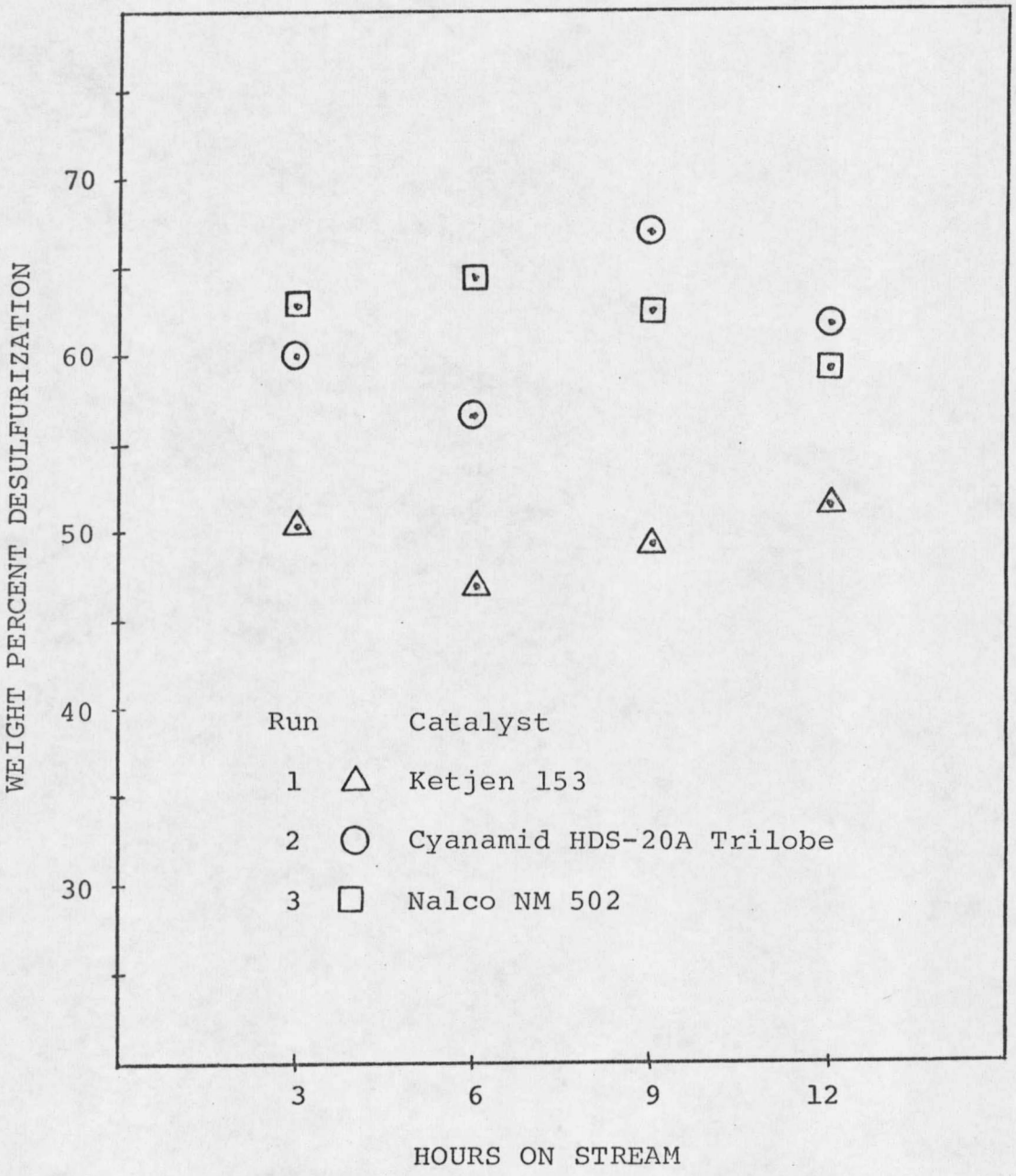


FIGURE 32. RUNS 1, 2, AND 3

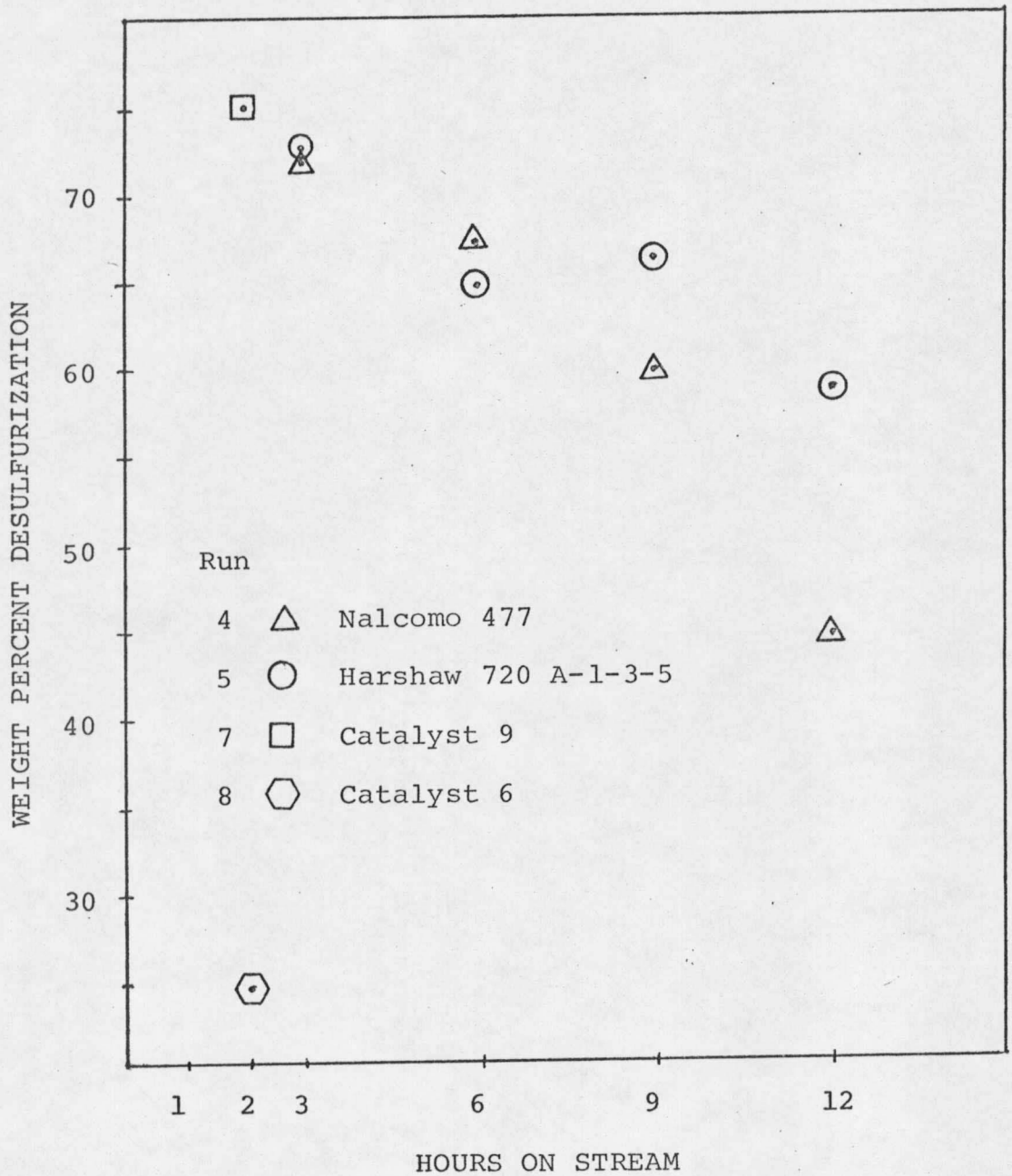


FIGURE 33. RUNS 4, 5, 7, AND 8

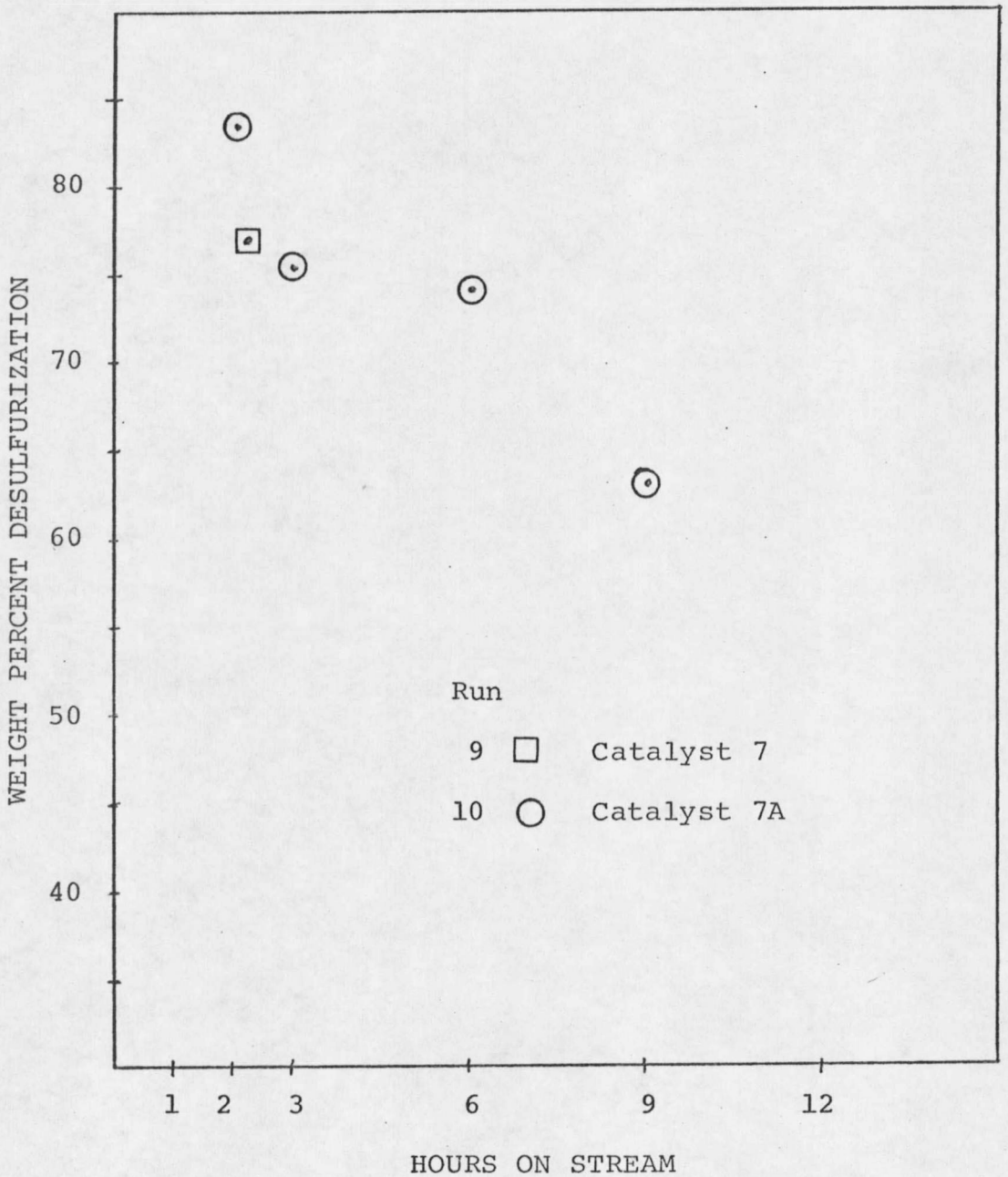


FIGURE 34. RUNS 9 AND 10

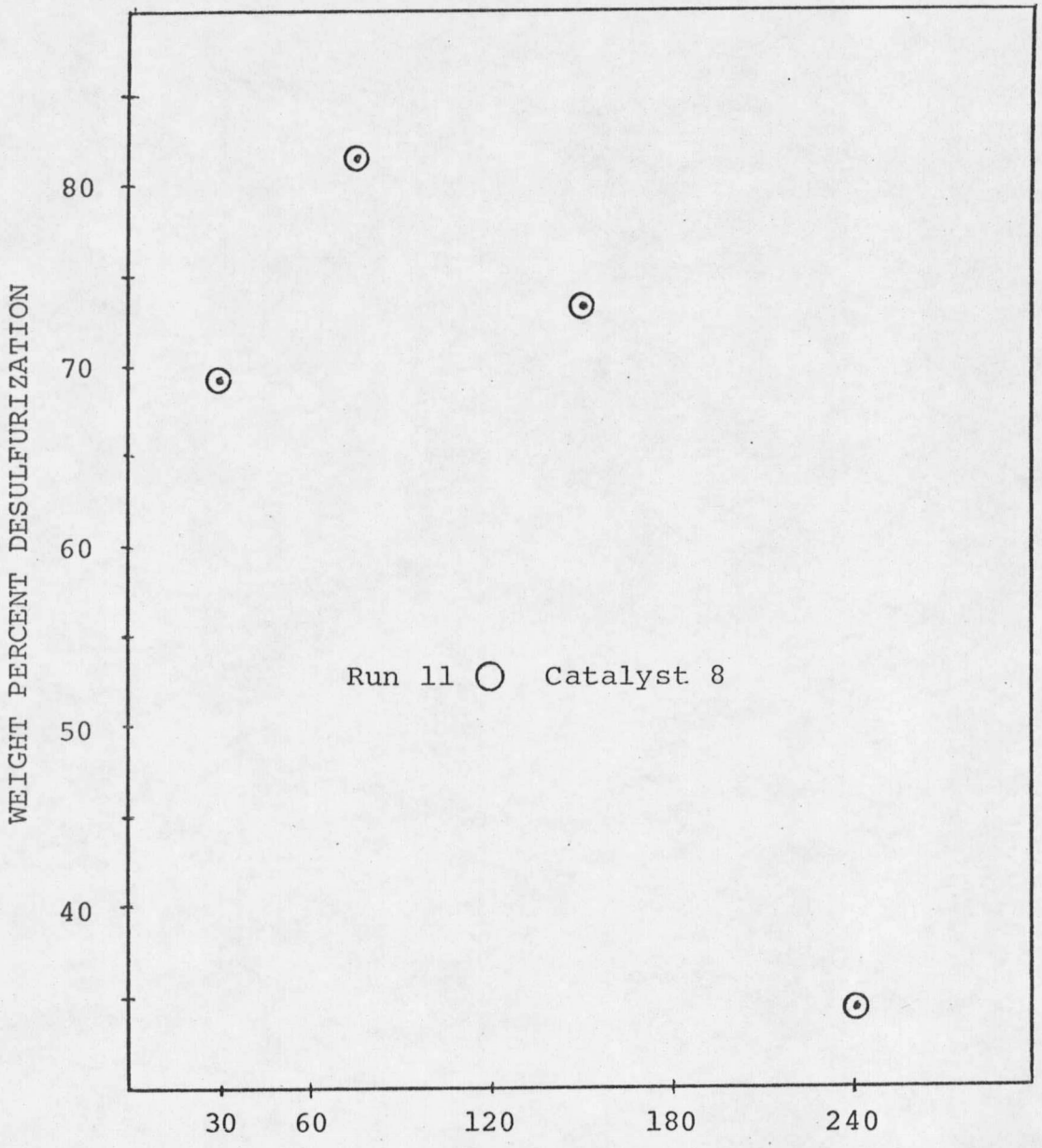


FIGURE 35. RUN 11

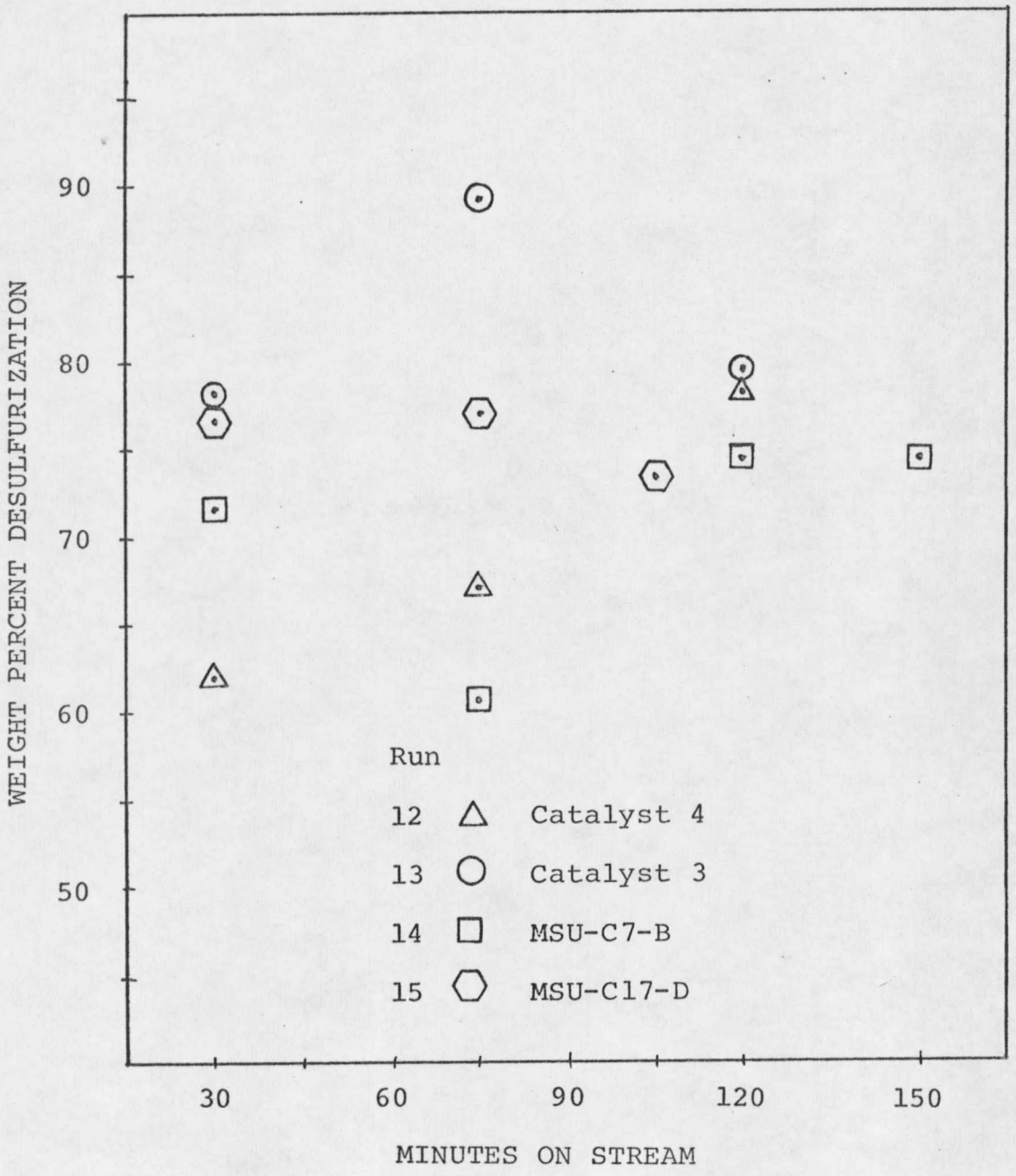


FIGURE 36. RUNS 12, 13, 14, AND 15

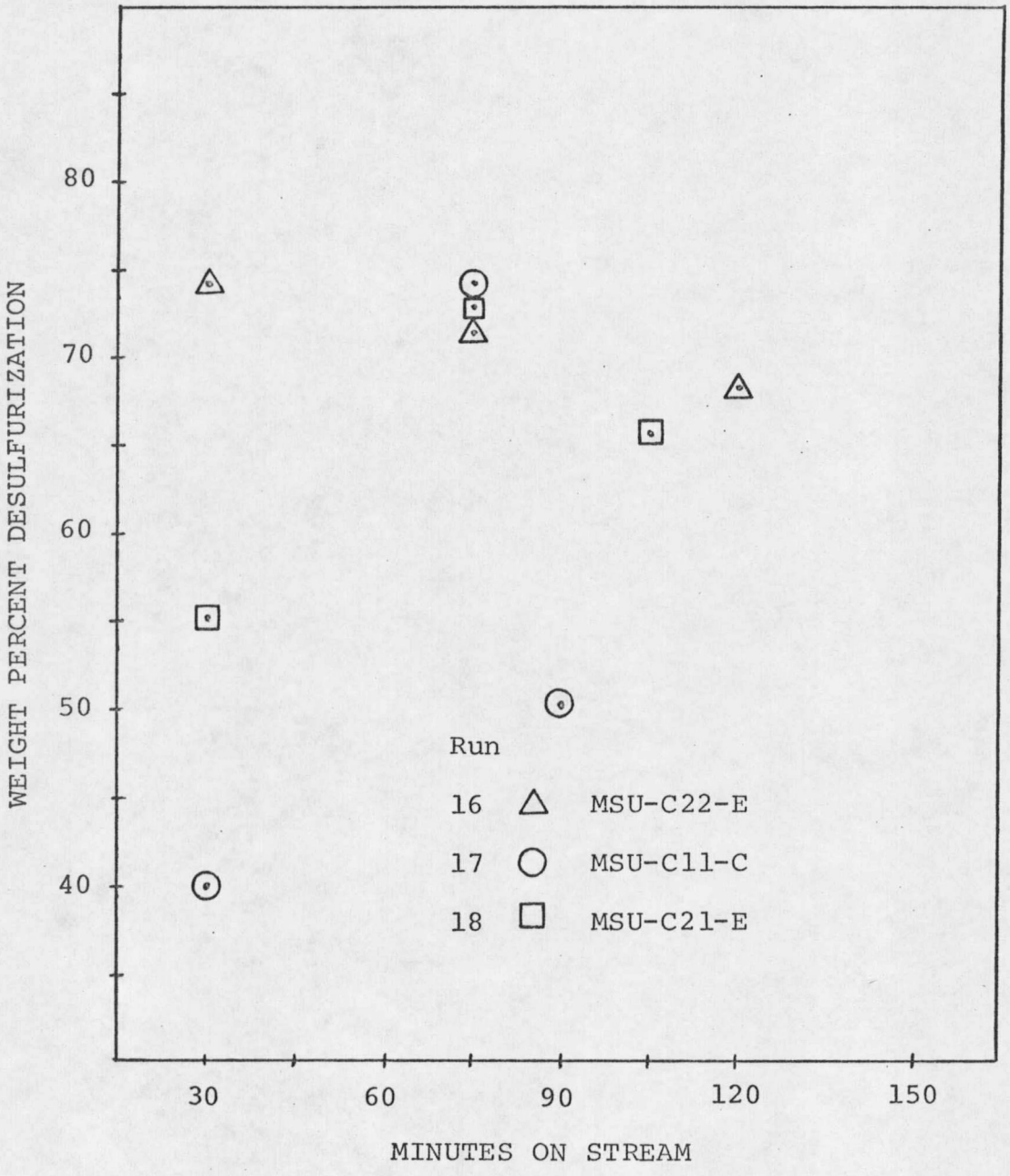


FIGURE 37. RUNS 16, 17, AND 18

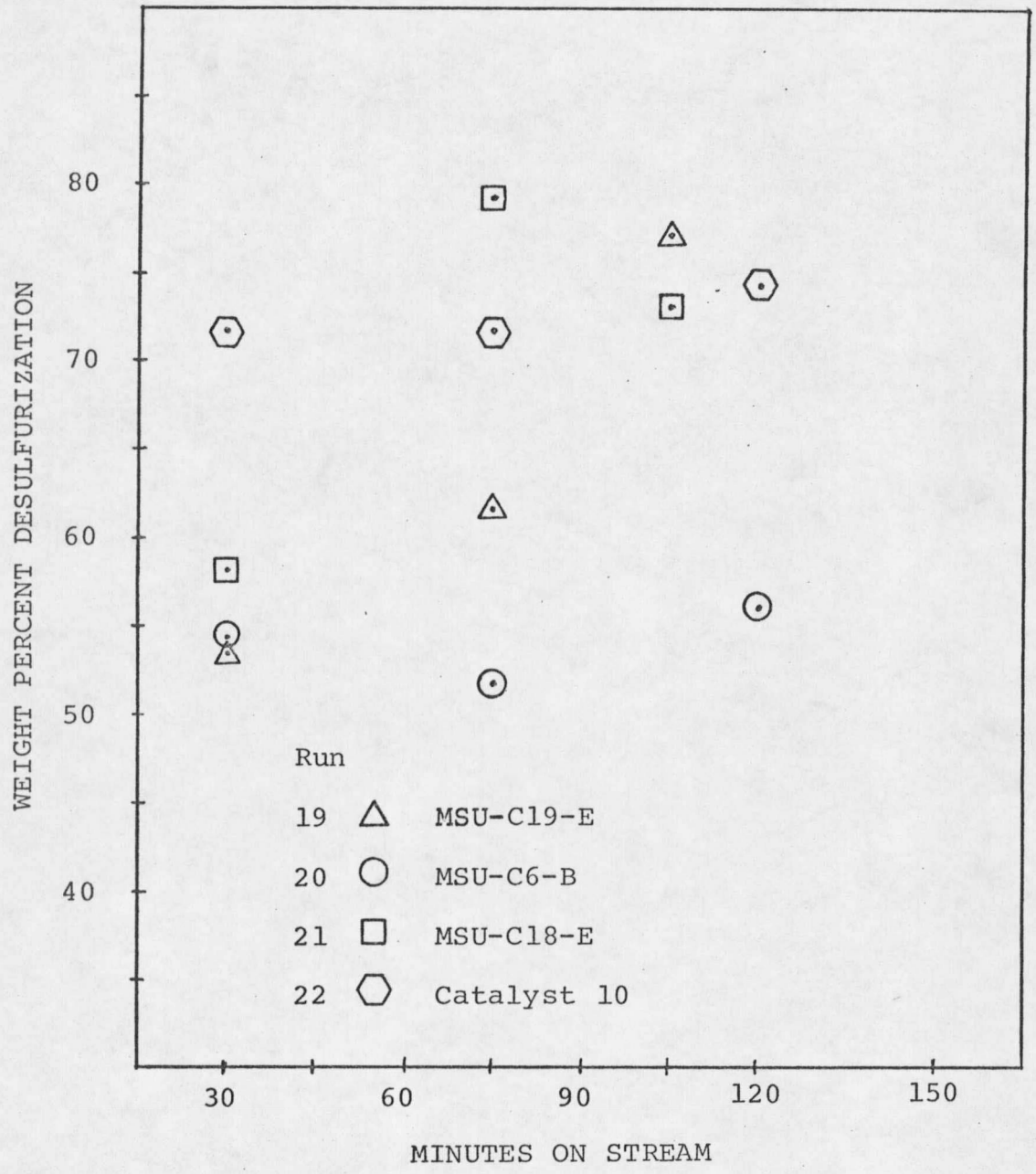


FIGURE 38. RUNS 19, 20, 21, AND 22

FIGURES 39-47

ASTM DISTILLATION

LIQUID YIELDS

VS.

TEMPERATURE

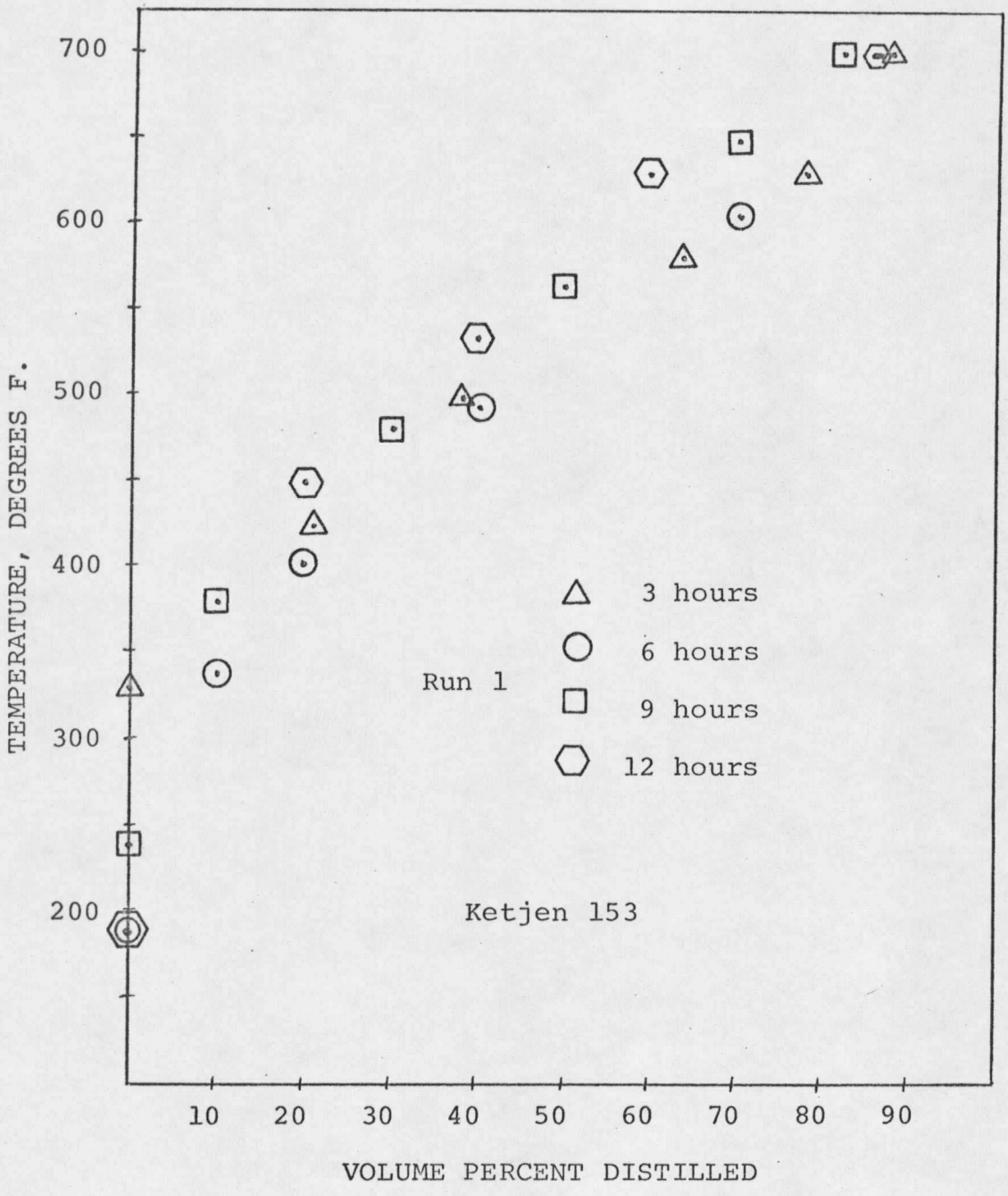


FIGURE 39. RUN 1

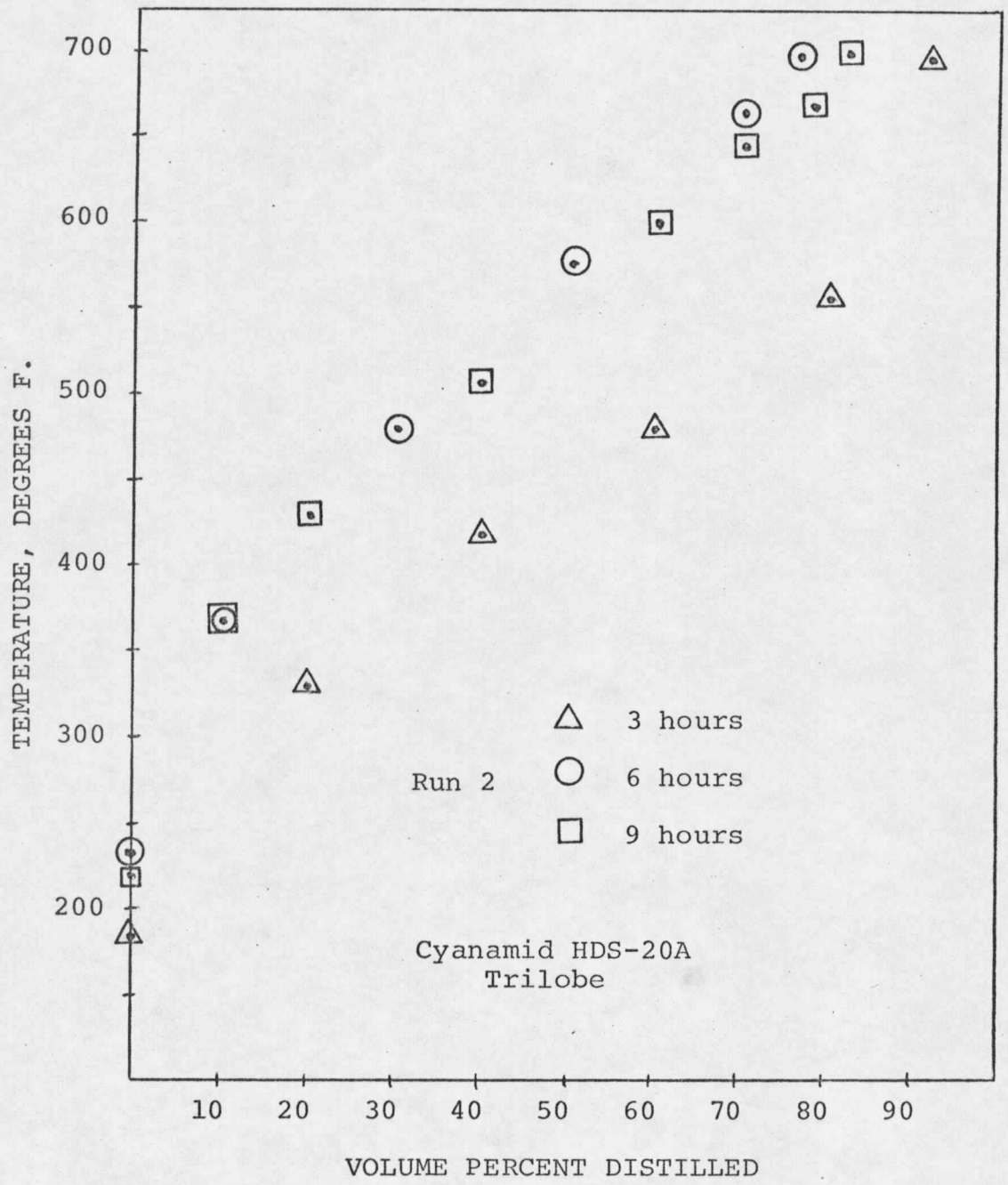


FIGURE 40. RUN 2

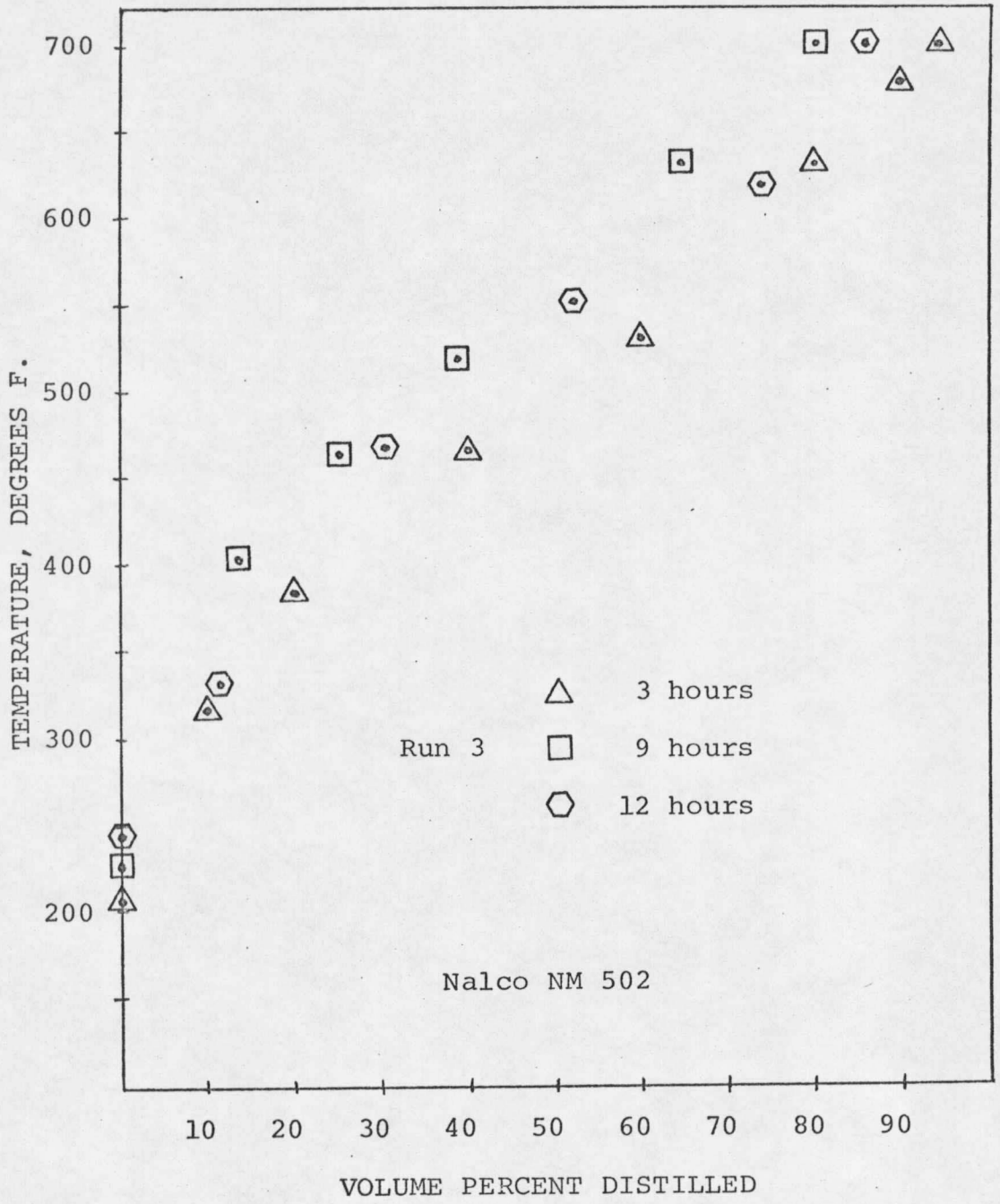


FIGURE 41. RUN 3

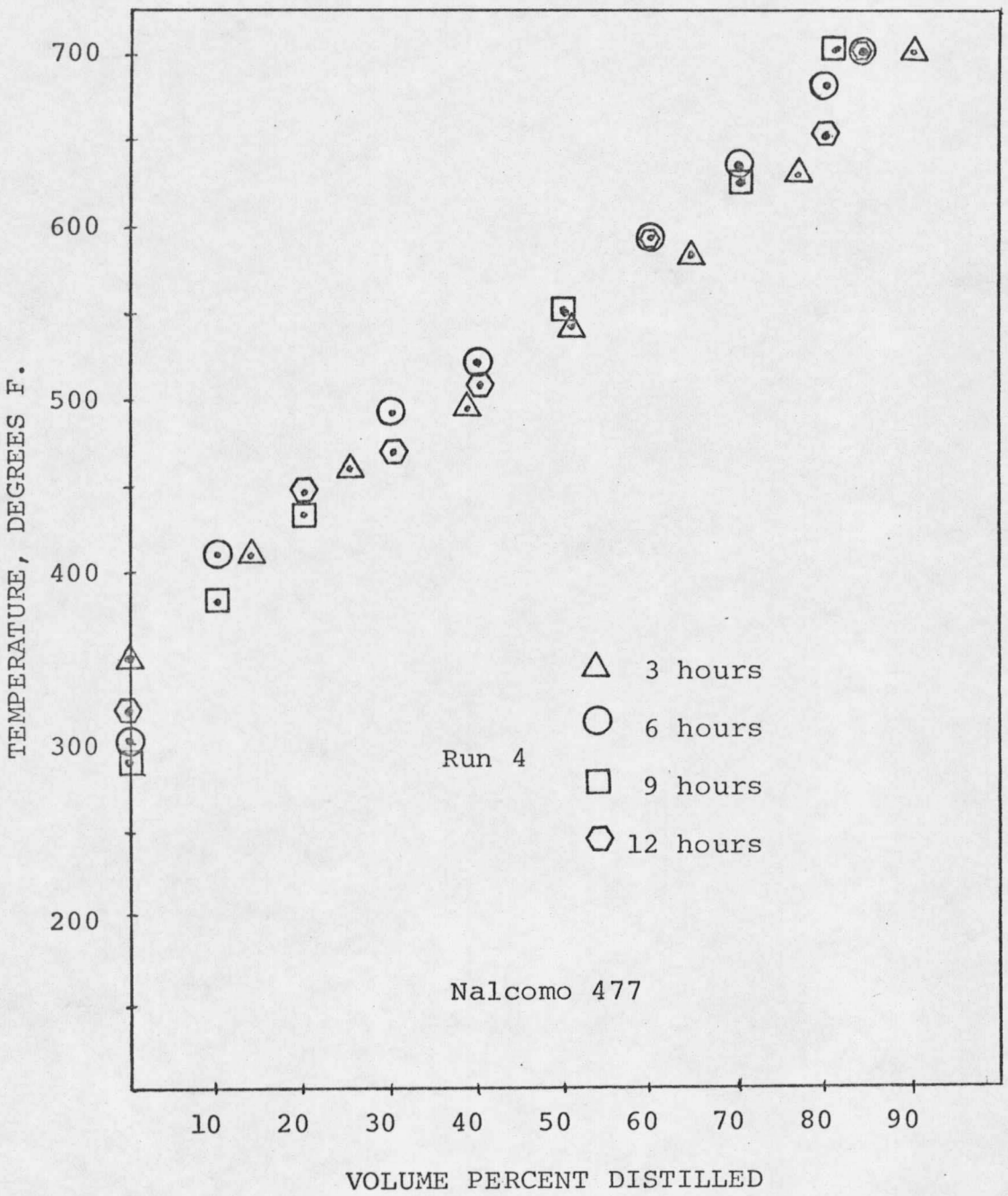


FIGURE 42. RUN 4

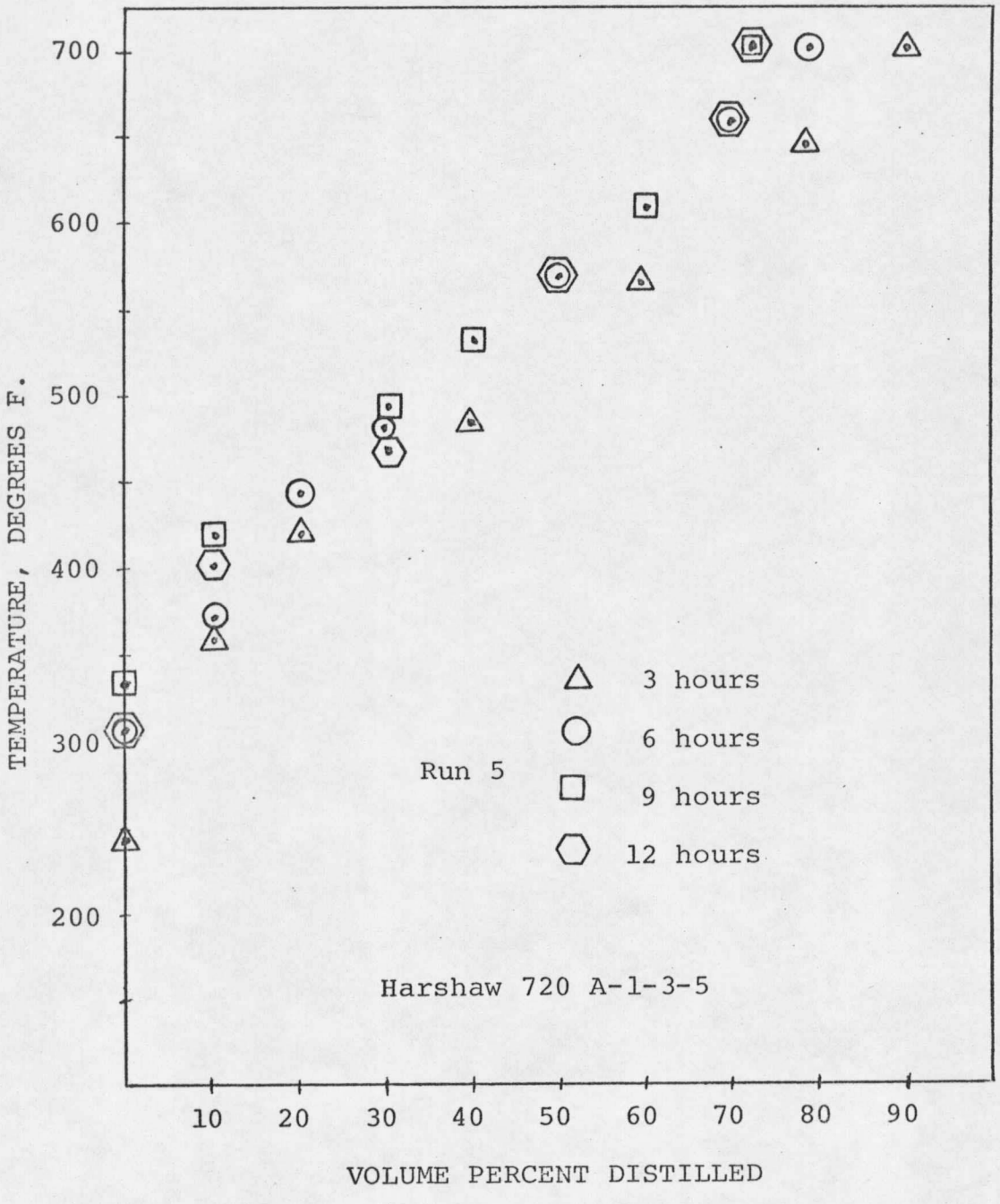


FIGURE 43. RUN 5

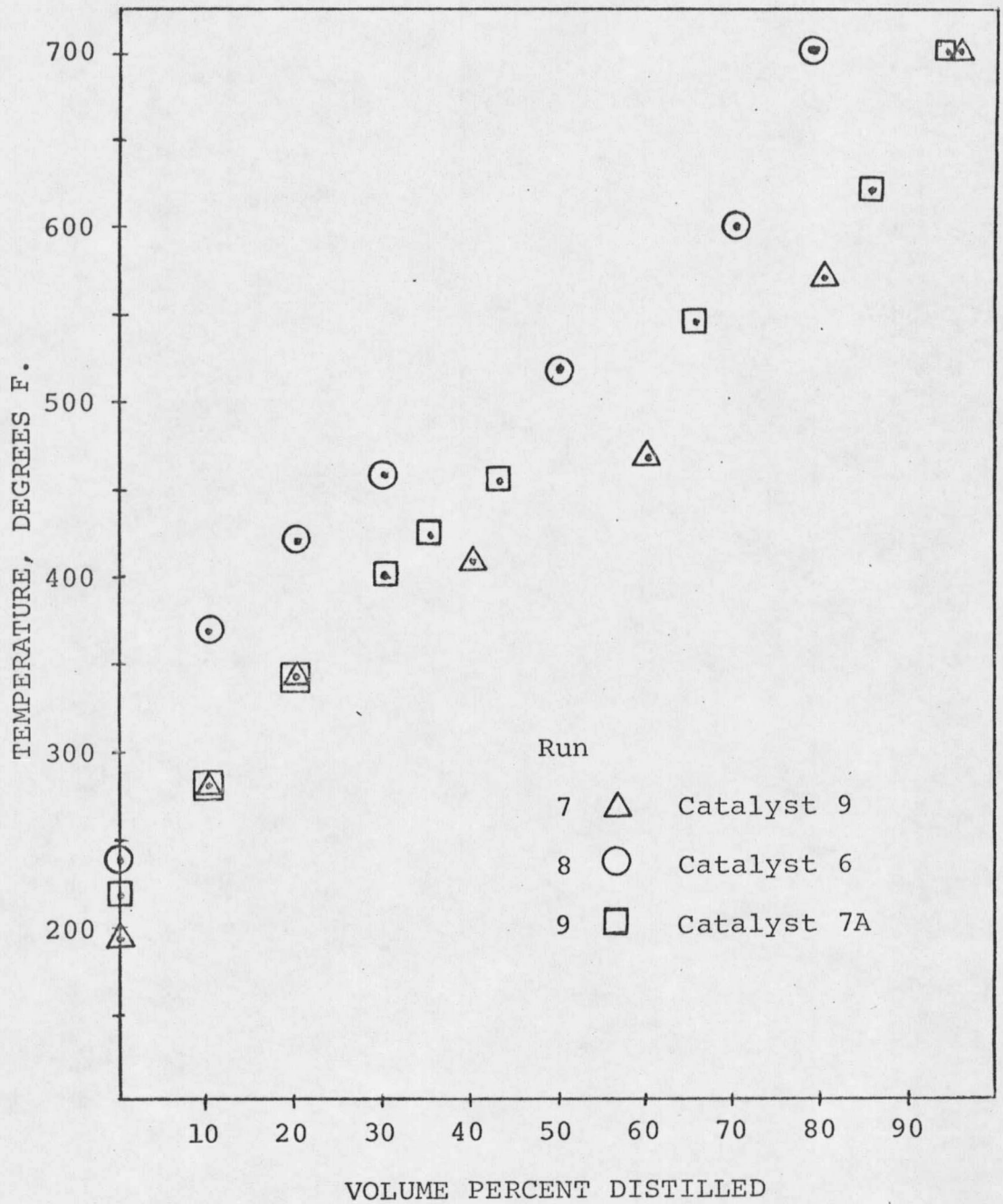


FIGURE 44. RUNS 7, 8, AND 9

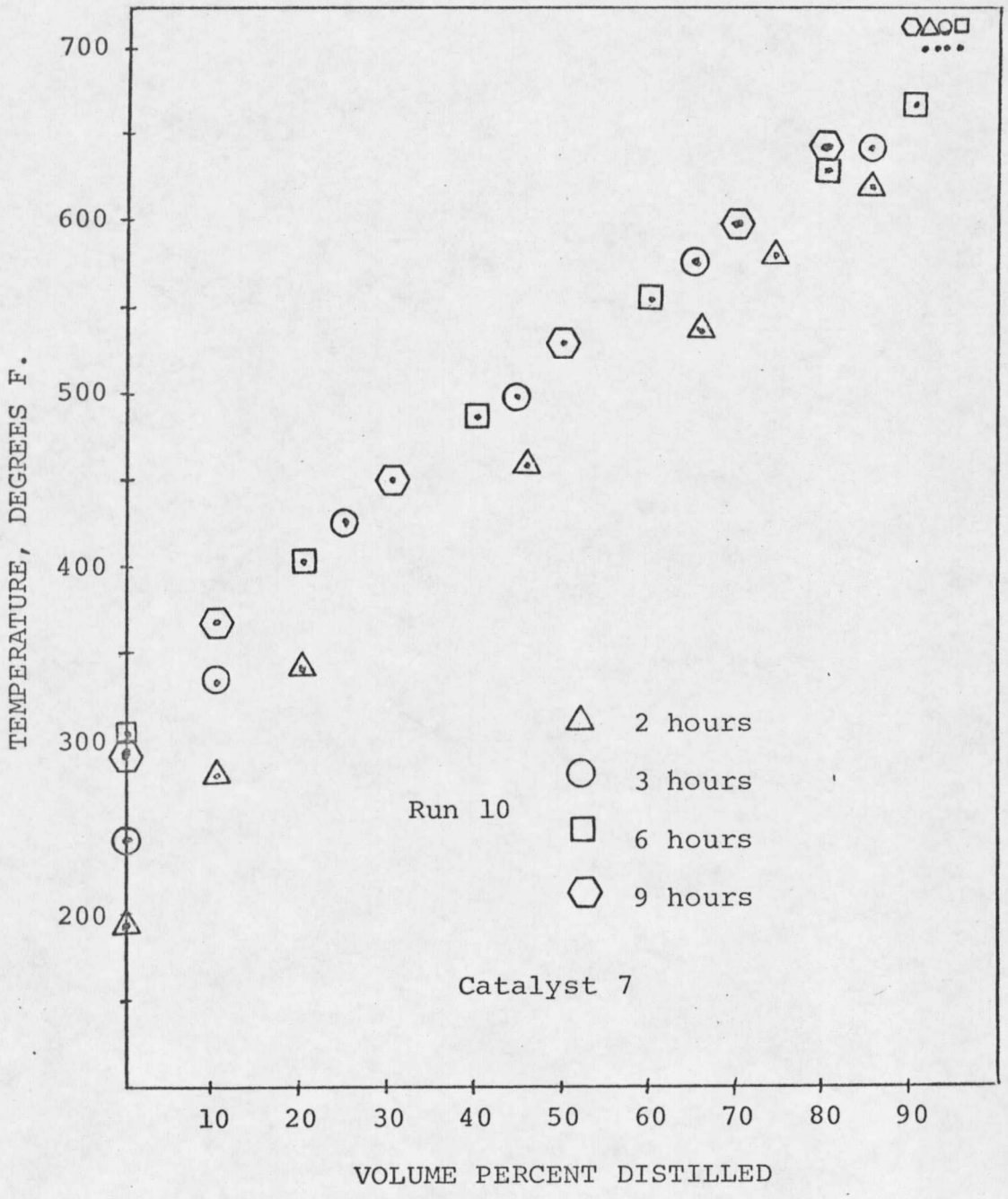


FIGURE 45. RUN 10

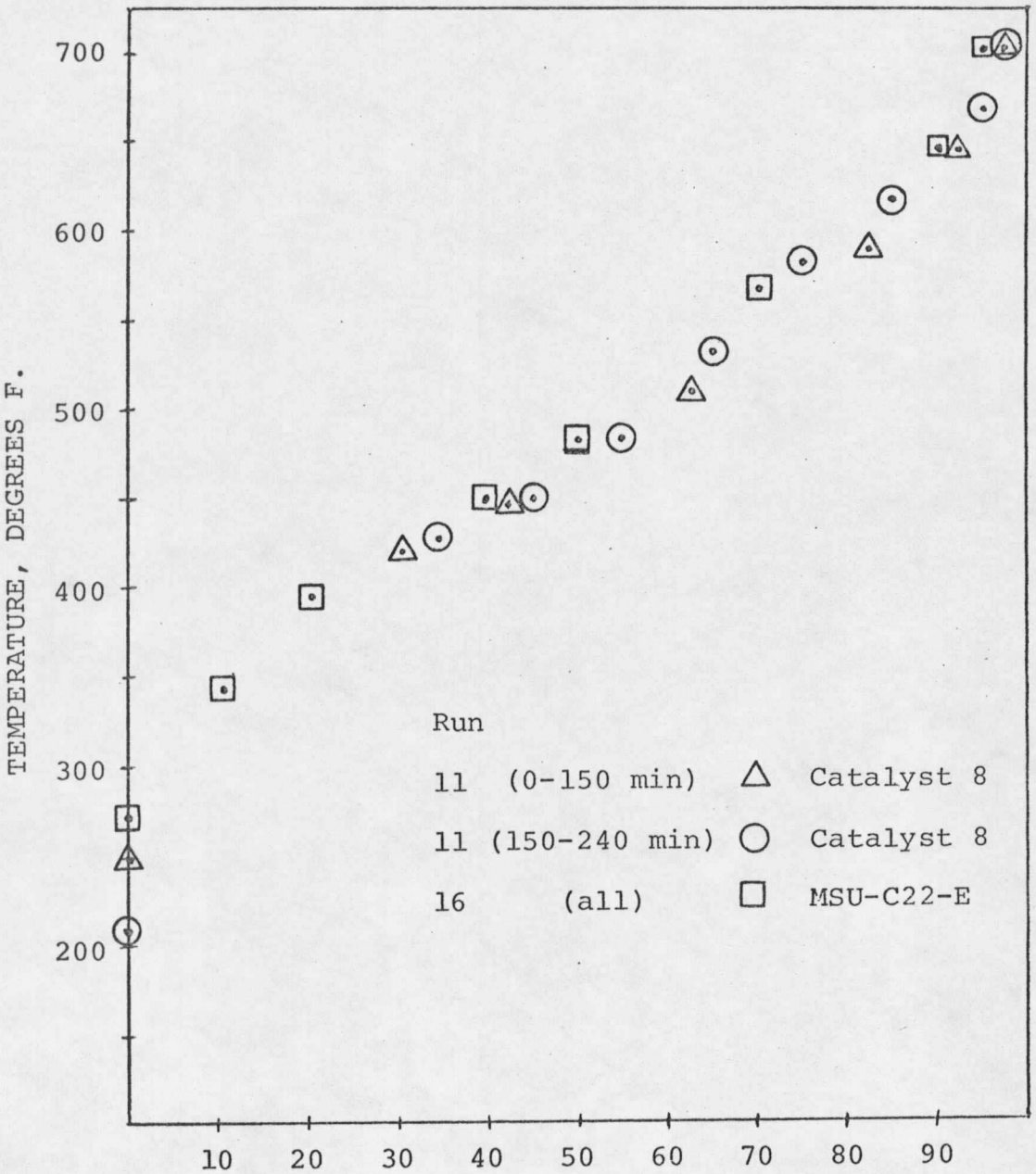


FIGURE 46. RUNS 11 AND 16

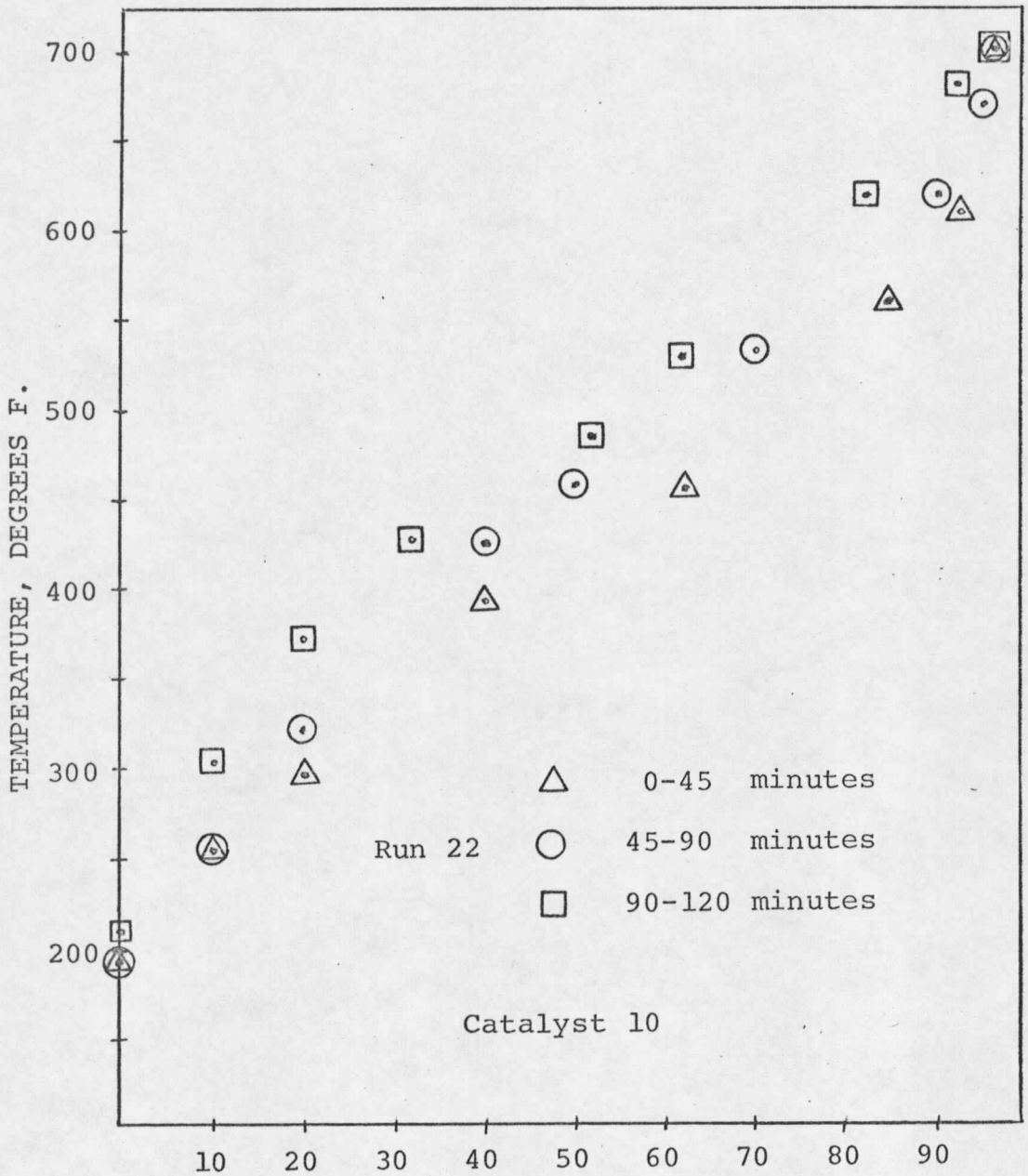


FIGURE 47. RUN 22

APPENDIX B

BATCH RUN DATA

RUN 1

CATALYST Harshaw 720 A-1-3-5

CATALYST COMPOSITION

METALS : 15.3% MoO_3 2.9% NiO 5.6% SiO_2

BASE : 100% Al_2O_3 1/8" SiO_2 Promoted

CATALYST SURFACE AREA, m^2/g: 161.0
CATALYST PORE VOLUME, ml/g : 0.36

CATALYST PORE DIAMETER (4V/A), Å: 89.0
CATALYST PORE DIAMETER (given), Å: 72.0

FEED CHARGE: SRC-II: 200g CATALYST, ml: 25

HYDROGEN PRESSURE, psig:

	Initial	Final
AT ROOM TEMPERATURE:	2500	850
AT RUN TEMPERATURE :	3600	2500

RUN TEMPERATURE, °C: TARGET: 415 RANGE: 408-422

TIME AT RUN TEMPERATURE, min: 60 RESIDENCE TIME, min: 135

MAXIMUM PRESSURE, psig: 4500

WT.% N_2: 0.985 WT% S: 0.325

ASTM DISTILLATION

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 36.3 ml

VOLUME:	IBP	5	10	15	20	25	30	34.9
°F :	306	416	453	498	552	591	640	682

RUN 2

CATALYST Harshaw 618 A-30-10-6

CATALYST COMPOSITION

METALS : 14.5% MoO_3 3.3% NiO 2.0% P 0.1% SiO_2

BASE : 100% Al_2O_3 1/16"

CATALYST SURFACE AREA, m^2/g: 192.0
CATALYST PORE VOLUME, ml/g : 0.5

CATALYST PORE DIAMETER (4V/A), Å: 104.0
CATALYST PORE DIAMETER (given), Å: 126.0

FEED CHARGE: SRC-II 200g CATALYST, ml: 25

HYDROGEN PRESSURE, psig:

	Initial	Final
AT ROOM TEMPERATURE :	2500	450
AT RUN TEMPERATURE :	3000	1850

RUN TEMPERATURE, °C: TARGET 430 RANGE: 430-435

TIME AT RUN TEMPERATURE, min: 60 RESIDENCE TIME, min: 149

MAXIMUM PRESSURE, psig: 4100

WT.% N_2: 1.01 WT.% S: 0.345

ASTM DISTILLATION

VOLUME OF CHARGE 50.0 ml FINAL VOLUME: 36.6 ml

VOLUME:	IBP	5	10	15	20	25	30	35
°F :	346	406	465	519	562	699	640	671

RUN 3

CATALYST Harshaw L3304-23-1

CATALYST COMPOSITION

METALS : 14.9% MoO_3 3.0% NiO 4.5% SiO_2

BASE : 100% Al_2O_3 1/8"

CATALYST SURFACE AREA, m^2/g: 187.0
CATALYST PORE VOLUME, ml/g : 0.37

CATALYST PORE DIAMETER (4V/A), Å: 79.0
CATALYST PORE DIAMETER (given), Å: 80.0

FEED CHARGE: SRC-II: 200g CATALYST, ml: 25

HYDROGEN PRESSURE, psig:

	Initial	Final
AT ROOM TEMPERATURE :	2500	625
AT RUN TEMPERATURE :	3550	2200

RUN TEMPERATURE, °C: TARGET: 430 RANGE: 427-431

TIME AT RUN TEMPERATURE, min: 60 RESIDENCE TIME, min: 60

MAXIMUM PRESSURE, psig: 4350

WT.% N_2: 1.16 WT.% S: 0.474

ASTM DISTILLATION

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 35.6 ml

VOLUME:	IBP	5	10	15	20	25	30	33.3
°F :	387	432	489	518	564	609	653	699

RUN 4

CATALYST Harshaw 720 A-2-7-3

CATALYST COMPOSITION

METALS : 16.0% MoO_3 3.0% NiO 0.5% P 0.1% SiO_2

BASE : 100% Al_2O_3 1/16"

CATALYST SURFACE AREA, m^2/g: 240.0
CATALYST PORE VOLUME, ml/g : 0.78

CATALYST PORE DIAMETER (4V/A), Å: 130.0
CATALYST PORE DIAMETER (given), Å: 141.0

FEED CHARGE: SRC-II: 200g CATALYST, ml: 25

HYDROGEN PRESSURE, psig:

	Initial	Final
AT ROOM TEMPERATURE :	2400	600
AT RUN TEMPERATURE :	3850	2800

RUN TEMPERATURE, °C: TARGET: 430 RANGE: 428-432

TIME AT RUN TEMPERATURE, min: 60 RESIDENCE TIME, min: 85

MAXIMUM PRESSURE, psig: 4250

WT.% N_2: 1.16 WT.% S: 0.67

ASTM DISTILLATION

VOLUME OF CHARGE: 51.0 ml FINAL VOLUME: 36.0 ml

VOLUME:	IBP	5	10	15	20	25	30	33.4
°F :	341	431	466	503	557	600	630	611

RUN 5

CATALYST Harshaw 720 A-2-1-1

CATALYST COMPOSITION

METALS : 14.8% MoO_3 2.8% CoO

BASE : 100% Al_2O_3 1/16"

CATALYST SURFACE AREA, m^2/g: 222.0
CATALYST PORE VOLUME, ml/g : 0.51

CATALYST PORE DIAMETER (4V/A), Å: 92.0
CATALYST PORE DIAMETER (given), Å: 110.0

FEED CHARGE: SRC-II: 200g CATALYST, ml: 25

HYDROGEN PRESSURE, psig:

	Initial	Final
AT ROOM TEMPERATURE :	2450	600
AT RUN TEMPERATURE :	4500	2250

RUN TEMPERATURE, °C: TARGET: 430 RANGE: 425-438

TIME AT RUN TEMPERATURE, min: 60 RESIDENCE TIME, min: 68

MAXIMUM PRESSURE, psig: 4500

WT.% N_2: 1.05 WT.% S: 0.39

ASTM DISTILLATION

VOLUME OF CHARGE: 51.5 ml FINAL VOLUME: 38.7 ml

VOLUME:	IBP	5	10	15	20	25	30	35
°F :	299	429	402	500	549	592	632	682

RUN 6

CATALYST Harshaw 720 A-2-11-3

CATALYST COMPOSITION

METALS : 32.3% WO_3 5.7% NiO

BASE : 100% Al_2O_3 1/16"

CATALYST SURFACE AREA, m^2/g: 160.0
CATALYST PORE VOLUME, ml/g : 0.52

CATALYST PORE DIAMETER (4V/A), Å: 130.0
CATALYST PORE DIAMETER (given), Å: 105.0

FEED CHARGE: SRC-II 200g CATALYST, ml: 25

HYDROGEN PRESSURE, psig:

	Initial	Final
AT ROOM TEMPERATURE :	2500	700
AT RUN TEMPERATURE :	4300	2550

RUN TEMPERATURE, °C: TARGET: 430 RANGE: 430-432

TIME AT RUN TEMPERATURE, min: 60 RESIDENCE TIME, min: 70

MAXIMUM PRESSURE, psig: 4600

WT.% N_2: 1.135 WT.% S: 0.495

ASTM DISTILLATION

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 37.1 ml

VOLUME:	IBP	5	10	15	20	25	30	35
°F :	299	429	402	500	544	592	632	682

RUN 7

CATALYST 1

CATALYST COMPOSTION

METALS: 17.0% WO_3 2.6% CoO 1.1% NiO 11.7% MoO_3

BASE : Norton 6176 1/16" 99.85% Al_2O_3-0.12% SiO-0.014% Na_2O

BASE SURFACE AREA, m^2/g: 250.0
BASE PORE VOLUME, ml/g : 0.7

BASE PORE DIAMETER (4V/A), Å: 152.0

FEED CHARGE: SRC-II: 200g CATALYST, ml: 25

HYDROGEN PRESSURE, psig:

	Initial	Final
AT ROOM TEMPERATURE :	2500	1100
AT RUN TEMPERATURE :	4400	2500

RUN TEMPERATURE, °C: TARGET: 430 RANGE: 430-436

TIME AT RUN TEMPERATURE, min: 60 RESIDENCE TIME, min: 65

MAXIMUM PRESSURE, psig: 4500

WT.% N_2: 1.01 WT.% S: 0.485

ASTM DISTILLATION

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 36.2 ml

VOLUME:	IBP	5	10	15	20	25	30	35
°F :	253	339	425	459	495	548	598	624

RUN 8

CATALYST 2

CATALYST COMPOSITION

METALS: 20.5% WO_3 6.6% CoO 2.9% NiO

BASE : Norton 6176 1/16" 99.85% Al_2O_3-0.12% SiO-0.014% Na_2O_3

BASE SURFACE AREA, m^2/g: 250.0
BASE PORE VOLUME, ml/g : 0.7

BASE PORE DIAMETER (4V/A), Å: 152.0

HYDROGEN PRESSURE, psig:	Initial	Final
AT ROOM TEMPERATURE :	2500	1250
AT RUN TEMPERATURE :	4800	3450

RUN TEMPERAUTRE, °C: TARGET: 430 RANGE: 424-437

TIME AT RUN TEMPERATURE, min: 60 RESIDENCE TIME, min: 59

MAXIMUM PRESSURE, psig: 4800

WT.% N_2: 1.16 WT.% S: 0.435

FEED CHARGE: SRC-II: 200g CATALYST, ml: 25

ASTM DISTILLATION

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 36.8 ml

VOLUME:	IBP	5	10	15	20	25	30	35	37.7
°F :	327	410	453	491	550	589	631	663	670

RUN 9

CATALYST 3

CATALYST COMPOSITION

METALS: 10.3% WO_3 6.8% CoO 2.8% NiO 12.6% MoO_3

BASE : Norton 6176 1/16" 99.85% Al_2O_3-0.12% SiO-0.014% Na_2O

BASE SURFACE AREA, m^2/g: 250.0
BASE PORE VOLUME, ml/g : 0.7

BASE PORE DIAMETER (4V/A), Å: 152.0

FEED CHARGE: SRC-II 200g CATALYST, ml: 25

HYDROGEN PRESSURE, psig:

	Initial	Final
AT ROOM TEMPERATURE :	2500	1200
AT RUN TEMPERATURE :	4500	3200

RUN TEMPERATURE, °C: TARGET: 430 RANGE: 427-432

TIME AT RUN TEMPERATURE, min: 60 RESIDENCE TIME, min: 58

MAXIMUM PRESSURE, psig: 4550

WT.% N_2: 1.17 WT.% S: 0.43

ASTM DISTILLATION

VOLUME OF CHARGE 50.0 ml FINAL VOLUME: 37.0 ml

VOLUME:	IBP	5	10	15	20	25	30	35	35.6
°F :	318	416	451	490	550	597	628	660	667

RUN 10

CATALYST 4

CATALYST COMPOSITION

METALS: 21.5% WO_3 5.9% CoO 2.9% NiO

BASE : Nalco 78-6008-C 1/32" 2.0% SiO_2-98% Al_2O_3

BASE SURFACE AREA, m^2/g: 214.6
BASE PORE VOLUME, ml/g : 0.8397

BASE MEDIAN PORE DIAMETER, Å: 161.0
BASE PORE DIAMETER (4V/A), Å: 156.5

FEED CHARGE: SRC-II: 200g CATALYST, ml: 25

HYDROGEN PRESSURE, psig:

	Initial	Final
AT ROOM TEMPERATURE :	2550	1100
AT RUN TEMPERATURE :	4700	3200

RUN TEMPERATURE, °C: TARGET: 430 RANGE: 428-433

TIME AT RUN TEMPERATURE, min: 60 RESIDENCE TIME, min: 67

MAXIMUM PRESSURE, psig: 4800

WT.% N_2: 0.99 WT.% S: 0.5

ASTM DISTILLATION

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 37.4 ml

VOLUME:	IBP	5	10	15	20	25	30	35	36
°F :	332	418	462	500	543	590	640	688	700

APPENDIX C

CONTINUOUS RUN DATA

RUN 1

CATALYST Ketjen 153

CATALYST COMPOSITION

METALS : 15.0% MoO_3 3.0% NiO 1.1% SiO_2 2.0% SO_4

BASE : 100% Al_2O_3 1/16"

CATALYST SURFACE AREA, m^2/g: 180.0
CATALYST PORE VOLUME, ml/g : 0.5
CATALYST PORE DIAMETER (4V/A), Å: 111.0

RUN TEMPERATURE, ^oC : 430 $\pm$5

RUN PRESSURE, psig : 1000

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, hours:	3	6	9	12
WT.% N_2 :	.44	.79	.92	.96
WT.% S :	.365	.390	.370	.360

ASTM DISTILLATION

3 hours VOLUME OF CHARGE: 38.9 ml FINAL VOLUME: 34.0 ml

VOLUME:	IBP	5	10	15	20	25	30	32
^oF :	330	391	442	449	530	582	624	641

6 hours VOLUME OF CHARGE: 51.0 ml FINAL VOLUME: 44.1 ml

VOLUME:	IBP	5	10	15	20	25	30	35	40	42.1
^oF :	191	339	401	459	496	536	569	603	655	673

RUN 1 (continued)

ASTM DISTILLATION

9 hours VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 41.0 ml

VOLUME:	IBP	5	10	15	20	25	30	35	39.2
°F :	239	381	444	482	521	563	603	642	702

12 hours VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 38.9 ml

VOLUME:	IBP	5	10	15	20	25	30	35	36.9
°F :	187	394	450	490	536	580	628	665	650

RUN 2

CATALYST Cyanamid HDS-20A

CATALYST COMPOSTION

METALS : 16.2% MoO_3 5.0% CoO

BASE : 100% Al_2O_3 1/16"

CATALYST SURFACE AREA, m^2/g: 230.0
CATALYST PORE VOLUME, ml/g : 0.52

CATALYST PORE DIAMETER (4V/A), Å: 90.0

RUN TEMPERATURE, ^oC : 430 $\pm$10

RUN PRESSURE, psig : 1000

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, hours:	3	6	9	12
WT.% N_2 :	.48	.89	.92	.90
WT.% S :	.295	.320	.240	.280

ASTM DISTILLATION

3 hours VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 46.0 ml

VOLUME:	IBP	5	10	15	20	25	30	35	40
^oF :	186	248	332	386	418	456	482	530	560

6 hours VOLUME OF CHARGE: 51.0 ml FINAL VOLUME: 39.9 ml

VOLUME:	IBP	5	10	15	20	25	30	35	37
^oF :	232	368	442	483	529	576	615	666	672

RUN 2 (continued)

ASTM DISTILLATION

9 hours VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 41.0 ml

VOLUME:	IBP	5	10	15	20	25	30	35	39
°F :	224	368	432	474	510	556	600	646	672

12 hours - (insufficient sample volume)

RUN 3

CATALYST Nalco NM-502

CATALYST COMPOSITION

METALS : 14.0% MoO_3 4.0% NiO

BASE : 97.6% Al_2O_3 2.4% SiO_2 1/16"

CATALYST SURFACE AREA, m^2/g: 240.0
CATALYST PORE VOLUME, ml/g : 0.53

CATALYST PORE DIAMETER (4V/A), Å: 88.0

RUN TEMPERATURE, °C : 435 ±5

RUN PRESSURE, psig : 1000

LIQUID HOURLY SPACE VELOCITY : 1

H_2 OIL RATIO, scf/bbl : 10,000

TIME, hours:	3	6	9	12
WT.% N_2 :	.50	.81	.89	.83
WT.% S :	.275	.260	.275	.300

ASTM DISTILLATION

3 hours VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 47.0 ml

VOLUME:	IBP	5	10	15	20	25	30	35	40	45
°F :	206	315	385	429	465	495	530	569	633	677

VOLUME:	45.5
°F :	679

6 hours - (insufficient sample volume)

RUN 3 (Continued)

9 hours VOLUME OF CHARGE: 39.0 ml FINAL VOLUME: 31.0 ml

VOLUME:	IBP	5	10	15	20	25	30
°F :	225	400	462	516	572	629	630

12 hours VOLUME OF CHARGE: 47.5 ml FINAL VOLUME: 40.5 ml

VOLUME:	IBP	5	10	15	20	25	30	35	40
°F :	240	333	422	464	508	553	592	620	674

RUN 4

CATALYST Nalcomo 477

CATALYST COMPOSITION

METALS : 14.0% MoO_3 3.3% CoO

BASE : 97.6% Al_2O_3-2.4% SiO_2

CATALYST SURFACE AREA, m^2/g: 256.0
CATALYST PORE VOLUME, ml/g : 0.55

CATALYST PORE DIAMETER (4V/A), Å: 88.0

RUN TEMPERATURE, °C : 430 ±10

RUN PRESSURE, psig : 1000

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, hours:	3	6	9	12
WT.% N_2 :	.47	.88	.97	1.02
WT.% S :	.21	--	.30	.41

ASTM DISTILLATION

3 hours VOLUME OF CHARGE: 39.2 ml FINAL VOLUME: 35.1 ml

VOLUME:	IBP	5	10	15	20	25	30	34.1
°F :	349	412	460	491	540	581	627	660

6 hours VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 42.1 ml

VOLUME:	IBP	5	10	15	20	25	30	35	40	40.9
°F :	300	410	447	489	519	560	593	629	677	689

RUN 4 (Continued)

9 hours VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 41.0 ml

VOLUME:	IBP	5	10	15	20	25	30	35
°F :	288	384	431	466	504	548	590	626

12 hours VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 41.0 ml

VOLUME:	IBP	5	10	15	20	25	30	35	40
°F :	318	389	446	470	512	552	592	625	650

RUN 5

CATALYST Harshaw 720 A-1-3-5

CATALYST COMPOSITION

METALS : 15.3% MoO_3 2.9% NiO 5.6% SiO_2

BASE : 100% Al_2O_3 1/8" SiO_2 Promoted

CATALYST SURFACE AREA, m^2/g: 161.0
CATALYST PORE VOLUME, ml/g : 0.36

CATALYST PORE DIAMETER (4V/A), Å: 89.0
CATALYST PORE DIAMETER (given), Å: 72.0

RUN TEMPERATURE, ^oC : 435 $\pm$5

RUN PRESSURE, psig : 1000

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, hours:	3	6	9	12
WT.% N_2 :	.725	1.090	1.100	1.135
WT.% S :	.205	.260	.250	.300

ASTM DISTILLATION

3 hours VOLUME OF CHARGE: 51.0 ml FINAL VOLUME: 45.8 ml

VOLUME:	IBP	5	10	15	20	25	30	35	40	43.9
^oF :	243	359	419	456	484	520	563	602	643	611

6 hours VOLUME OF CHARGE: 51.0 ml FINAL VOLUME: 40.1 ml

VOLUME:	IBP	5	10	15	20	25	30	35	38.5
^oF :	304	369	440	482	519	508	610	661	687

RUN 5 (Continued)

9 hours VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 36.0 ml

VOLUME:	IBP	5	10	15	20	25	30	35
°F :	334	416	444	490	530	568	609	598

12 hours VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 36.1 ml

VOLUME:	IBP	5	10	15	20	25	30	34.9
°F :	301	400	430	470	517	564	610	660

RUN 6

CATALYST 2

CATALYST COMPOSITION

METALS : 20.5% WO_3 6.6% CoO 2.9% NiO

BASE : Norton 6176 1/16" 99.85% Al_2O_3-0.12% SiO-0.014% Na_2O

BASE SURFACE AREA, m^2/g: 250.0

BASE PORE DIAMETER, (4V/A), Å: 152.0

BASE PORE VOLUME, ml/g : 0.7

RUN TEMPERATURE, ^oC : 335 to 430 in 2 hours

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME - (Reactor plugged up after two hours. The sample was lost; no analysis available.)

RUN 7

CATALYST 9

CATALYST COMPOSITION

METALS : 20.0% WO_3 5.5% CoO 3.2% NiO

BASE : Nalco 78-6008-B 1/16" 100% Al_2O_3

BASE SURFACE AREA, m^2/g: 232.4
BASE PORE VOLUME, ml/g : 0.7215

BASE MEDIAN PORE DIAMETER, Å: 137.2
BASE PORE DIAMETER (4V/A), Å: 124.2

RUN TEMPERATURE, °C : 335 to 430 in 1 hour then 1 hour at 430

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, min: 120

WT.% N_2 : .27

WT.% S : .185

ASTM DISTILLATION

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 47.0 ml

VOLUME:	IBP	5	10	15	20	25	30	35	40	45
°F :	194	281	343	385	411	435	468	520	574	600

VOLUME:	46.4
°F :	602

RUN 8

CATALYST 6

CATALYST COMPOSITION

METALS : 21.5% WO_3 6.2% CoO 2.7% NiO

BASE : Nalco 78-6008-E 1/16" 83% Al_2O_3-17.0%-P_2O_5

BASE SURFACE AREA, m^2/g: 146.95
BASE PORE VOLUME, ml/g : 0.6841

BASE MEDIAN PORE DIAMETER, Å: 420.2
BASE PORE DIAMETER (4V/A), Å: 186.2

RUN TEMPERATURE, ^oC : 430 $\pm$5

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, min: 120

WT.% N_2 : .85

WT.% S : .555

ASTM DISTILLATION

VOLUME OF CHARGE: 51.0 ml FINAL VOLUME: 40.0 ml

VOLUME:	IBP	5	10	15	20	25	30	35	40
^oF :	283	368	424	459	490	520	567	600	649

RUN 9

CATALYST 7A

CATALYST COMPOSITION

METALS : 20.2% WO_3 5.9% CoO 3.1% NiO

BASE : Nalco 78-6008-D 1/16" 91.0% Al_2O_3-9.0%-P_2O_5

BASE SURFACE AREA, m^2/g: 211.4
BASE PORE VOLUME, ml/g : 0.7943

BASE MEDIAN PORE DIAMETER, Å: 190.0
BASE PORE DIAMETER (4V/A), Å: 150.0

RUN TEMPERATURE, °C : 390 ±5

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, min: 135

WT.% N_2 : .32

WT.% S : .168

ASTM DISTILLATION

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 47.3 ml

VOLUME:	IBP	5	10	15	18	23	28	33	38	43
°F :	218	280	344	401	425	453	495	548	584	621

VOLUME:	46.6
°F :	630

RUN 10

CATALYST 7

CATALYST COMPOSITION

METALS : 19.7% WO_3 6.0% CoO 3.1% NiO

BASE : Nalco 78-6008-D 1/32" 91.0% Al_2O_3-9.0%-P_2O_5

BASE SURFACE AREA, m^2/g: 211.4
BASE PORE VOLUME, ml/g : 0.7943

BASE MEDIAN PORE DIAMETER, Å: 190.0
BASE PORE DIAMETER (4V/A), Å: 150.3

RUN TEMPERATURE, °C : 360 ±5

RUN PRESSURE, psig : 1050

H_2: OIL RATIO, scf/bbl : 10,000

LIQUID HOURLY SPACE VELOCITY : 1

TIME, min:	120	180	360	540
WT.% N_2 :	.360	.700	.715	.825
WT.% S :	.12	.18	.19	.27

ASTM DISTILLATION

120 min. (2 hours)

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 46.3 ml

VOLUME:	IBP	5	10	15	17	23	27	33	37	43
°F :	194	279	340	397	425	460	505	537	577	619

(continued.)

RUN 10 (continued.)

180 min. (3 hours)

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 46.5 ml

VOLUME:	IBP	5	12.2	17.2	22.2	27.2	32.2	37.2	42.2
°F :	241	334	425	466	498	534	575	607	641

VOLUME:	45.4
°F :	681

360 min. (6 hours)

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 48.2 ml

VOLUME:	IBP	5	10	15	20	25	30	35	40	45
°F :	301	343	401	445	487	518	554	590	629	670

VOLUME:	47
°F :	682

540 min. (9 hours)

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 45.9 ml

VOLUME:	IBP	5	10	15	20	25	30	35	40	45
°F :	298	370	425	450	482	527	568	599	639	671

RUN 11

CATALYST 8

CATALYST COMPOSITION

METALS : 19.5% WO_3 5.7% CoO 3.6% NiO

BASE : Nalco 78-6008-A 1/32" 4.0% SiO_2-96.0% Al_2O_3

BASE SURFACE AREA, m^2/g: 323.2
BASE PORE VOLUME, ml/g : 0.7183

BASE MEDIAN PORE DIAMETER, Å: 90.2
BASE PORE DIAMETER (4V/A), Å: 88.9

RUN TEMPERATURE, °C : 390 ±5

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, min:	30	45	60	75	90	105	120	180	210
WT.% N_2 :	.10	.14	.15	.09	.17	.24	.25	.23	.22
WT.% S :	.225			.135				.195	

TIME, min:	210	240	300
WT.% N_2 :	.21	.17	.56
WT.% S :		.480	

(continued.)

RUN 11 (continued.)

ASTM DISTILLATION

150 min. (Samples 1-8)

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 48.1 ml

VOLUME:	IBP	5	10	15	26	21	26	31	41	46
°F :	248	--	--	420	425	448	506	549	587	640

VOLUME:	47.4
°F :	660

240 min. (samples 9-11)

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 48.3 ml

VOLUME:	IBP	5	10	17	22	27	32	37	42	47
°F :	210	--	--	425	449	484	530	581	615	669

VOLUME:	47.6
°F :	671

RUN 12

CATALYST 4

CATALYST COMPOSITION

METALS : 21.5% WO_3 5.9% CoO 2.9% NiO

BASE : Nalco 78-6008-C 1/32" 2.0% SiO_2-98.0% Al_2O_3

BASE SURAFCE AREA, m^2/g: 214.6
BASE PORE VOLUME, ml/g : 0.8397

BASE MEDIAN PORE DIAMETER, Å: 161.0
BASE PORE DIAMETER (4V/A), Å: 156.5

RUN TEMPERATURE, ^oC : 425 $\pm$5

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, min:	30	45	60	75	90	105	120
WT.% N_2 :	.09	.08	.17	.105	.13	.15	.11
WT.% S :	.279			.235			.16

ASTM DISTILLATION -(insufficient sample volume)

RUN 13

CATALYST 3

CATALYST COMPOSITION

METALS : 10.3% WO_3 6.8% CoO 2.8% NiO 12.6% MoO

BASE : Norton 6176 1/16" 99.85% Al_2O_3-0.12% SiO-0.014% Na_2O

BASE SURFACE AREA, m^2/g: 250.0

BASE PORE VOLUME, ml/g : 0.7

BASE PORE DIAMETER, (4V/A) Å: 152.0

RUN TEMPERATURE, °C : 420 ±5

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, min:	30	45	60	75	90	105	120	150
WT.% N_2 :	.01	.02	.13	.09	.06	.305	.09	.22
WT.% S :	.16			.08			.15	

ASTM DISTILLATION -(insufficient sample volume)

RUN 14

CATALYST MSU-C7-B

CATALYST COMPOSITION

METALS : 14.6% MoO_3 0.13% NiO 5.8% CoO

BASE : Nalco 78-6008-B 1/16" 100% Al_2O_3

BASE SURFACE AREA, m^2/g: 232.4
BASE PORE VOLUME, ml/g : 0.7215

BASE MEDIAN PORE DIAMETER, Å: 137.3
BASE PORE DIAMETER (4V/A), Å: 124.2

RUN TEMPERATURE, ^{o}C : 420 $\pm$5

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, min:	30	45	60	75	90	105	120	150
WT.% N_2 :	.21	.28	.29	.33	.34	.29	.31	.68
WT.% S :	.21			.29			.19	.19

ASTM DISTILLATION -(insufficient sample volume)

RUN 15

CATALYST MSU-C17-D

CATALYST COMPOSITION

METALS : 9.3% MoO_3 0.17% NiO 1.8% CoO 8.8% WO_3

BASE : Nalco 78-6008-D 1/16" 91.0% Al_2O_3-9.0%-P_2O_5

BASE SURFACE AREA, m^2/g: 211.4
BASE PORE VOLUME, ml/g : 0.7943

BASE MEDIAN PORE DIAMETER, Å: 190.0
BASE PORE DIAMETER (4V/A), Å: 150.3

RUN TEMPERATURE, ^oC : 425 $\pm$5

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, min:	30	45	60	75	90	105
WT.% N_2 :	.06	.16	.30	.25	.24	---
WT.% S :	.172			.168		.194

ASTM DISTILLATION -(insufficient sample volume)

RUN 16

CATALYST MSU-C22-E

CATALYST COMPOSITION

METALS : 9.8% MoO_3 0.3% NiO 1.4% CoO 9.2% WO_3

BASE : Nalco 78-6008-E 1/16" 83.0% Al_2O_3-17.0%-P_2O_5

BASE SURFACE AREA, m^2/g: 146.95
BASE PORE VOLUME. ml/g : 0.6841

BASE MEDIAN PORE DIAMETER, Å: 420.2
BASE PORE DIAMETER (4V/A), Å: 186.2

RUN TEMPERATURE, °C : 430 ±5

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

TIME, min:	30	45	60	75	90	105	120
WT.% N_2 :	.20	.19	.19	.30	.37	.46	.30
WT.% S :	.19			.21			.23

ASTM DISTILLATION

VOLUME OF CHARGE: 49.5 ml FINAL VOLUME: 46.9 ml

VOLUME:	IBP	5	10	14.3	19.3	24.3	29.3	34.3	39.3
°F :	270	340	394	425	448	481	515	563	599

VOLUME:	44.3	46.3
°F :	640	695

RUN 17

CATALYST MSU-C11-C

CATALYST COMPOSITION

METALS : 11.1% MoO_3 0.1% NiO 1.6% CoO

BASE : Nalco 78-6008-C 1/32" 2.0% SiO_2-98.0% Al_2O_3

BASE SURFACE AREA, m^2/g: 412.6
BASE PORE VOLUME, ml/g : 0.8397

BASE MEDIAN PORE DIAMETER, Å: 161.0
BASE PORE DIAMETER(4V/A), Å: 156.5

RUN PRESSURE, psig : 1050

RUN TEMPERATURE, °C : 430 ±5*

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, min:	30	45	60	75	90
WT.% N_2 :	.30	.07	.08	.13	.19
WT.% S :	.440			.190	.365

ASTM DISTILLATION -(insufficient sample volume)

*The temperature may be higher due to temperature recorder difficulty.

RUN 18

CATALYST MSU-C21-E

CATALYST COMPOSITION

METALS : 9.5% MoO_3 0.4% NiO 1.9% CoO

BASE : Nalco 78-6009-E 1/16" 83.0% Al_2O_3-17.0%-P_2O_5

BASE SURFACE AREA, m^2/g: 146.95
BASE PORE VOLUME, ml/g : 0.6841

BASE MEDIAN PORE DIAMETER, Å: 420.2
BASE PORE DIAMETER (4V/A), Å: 186.2

RUN TEMPERATURE, °C : 430 ±5*

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, min:	30	45	60	75	90	105
WT.% N_2 :	.56	.14	.15	.17	.24	.25
WT.% S :	.33			.20		.28

ASTM DISTILLATION -(insufficient sample volume)

*The temperature may be higher due to temperature recorder difficulty.

RUN 19

CATALYST MSU-C19-E

CATALYST COMPOSITION

METALS : 18.9% MoO_3 0.1% NiO 3.5% CoO

BASE : Nalco 78-6008-E 1/16" 83.0% Al_2O_3-17.0%-P_2O_5

BASE SURFACE AREA, m^2/g: 146.95
BASE PORE VOLUME, ml/g : 0.6841

BASE MEDIAN PORE DIAMETER, Å: 420.2
BASE PORE DIAMETER (4V/A), Å: 186.2

RUN TEMPERATURE, °C : 430 ±5

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 10,000

TIME, min:	30	45	60	75	90	105
WT.% N_2 :	.45	.07	.28	.18	.23	.15
WT.% S :	.34			.28		.166

ASTM DISTILLATION -(insufficient sample volume)

RUN 20

CATALYST MSU-C6-B

CATALYST COMPOSITION

METALS : 14.8% MoO_3 0.19% NiO 1/17% CoO

BASE : Nalco 78-6008-B 1/16" 100% Al_2O_3

BASE SURFACE AREA, m^2/g: 232.4
BASE PORE VOLUME, ml/g : 0.7183

BASE MEDIAN PORE DIAMETER, Å: 90.2
BASE PORE DIAMETER (4V/A), Å: 88.9

RUN TEMPERATURE, °C : 415 ±5

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATION, scf/bbl : 10,000

TIME, min:	30	45	60	75	90	105	120
WT.% N_2 :	.52	.22	.33	.33	.30	.27	.42
WT.% S :	.335			.354			.321

ASTM DISTILLATION -(insufficient sample volume)

RUN 21

CATALYST MSU-C18-E

CATALYST COMPOSITION

METALS : 29.3% MoO_3 0.1% NiO 0.1% CoO

BASE : Nalco 78-6008-E 1/16" 83.0% Al_2O_3-17.0%-P_2O_5

BASE SURFACE AREA, m^2/g: 146.95
BASE PORE VOLUME, ml/g : 0.6841

BASE MEDIAN PORE DIAMETER, Å: 420.2
BASE PORE DIAMETER (4V/A), Å: 186.2

RUN TEMPERATURE, ^oC : 415 $\pm$5

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl :10,000

TIME, min:	30	45	60	75	90	105
WT.% N_2 :	.46	.17	.33	.47	.39	.61
WT.% S :	.149			.152		.196

ASTM DISTILLATION -(insufficient sample volume)

RUN 22

CATALYST 10

CATALYST COMPOSITION

METALS : 22.6% WO_3 5.4% CoO 3.4% NiO

BASE : Nalco 78-6008-A 1/32" 4.0% SiO_2-96.0%-Al_2O_3

BASE SURFACE AREA, m^2/g: 323.2
BASE PORE VOLUME, ml/g : 0.7183

BASE MEDIAN PORE DIAMETER, Å: 90.2
BASE PORE DIAMETER (4V/A), Å: 88.9

RUN TEMPERATURE, °C : 425 ±5

RUN PRESSURE, psig : 1050

LIQUID HOURLY SPACE VELOCITY : 1

H_2: OIL RATIO, scf/bbl : 6700

TIME, min:	30	45	60	75	90	105	120
WT.% N_2 :	.12	.40	.03	.14	.19	.14	.39
WT.% S :	.204			.206			.188

ASTM DISTILLATION

45 min. (Samples 1 and 2)

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 48.1 ml

VOLUME:	IBP	5	10	15	20	26	31	36	41	46
°F :	192	251	293	344	390	425	452	491	560	609

VOLUME:	47.5
°F :	620

(continued.)

RUN 22 (continued.)

90 min. (Samples 3, 4, 5)

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 48.3 ml

VOLUME:	IBP	5	10	15	20	25	30	35	40	45
°F :	189	249	321	392	425	457	491	530	581	619

VOLUME:	47.7
°F :	660

120 min. (Samples 6 and 7)

VOLUME OF CHARGE: 50.0 ml FINAL VOLUME: 48.3 ml

VOLUME:	IBP	5	10	16	21	26	31	36	41	46
°F :	208	301	370	425	451	480	526	567	619	680

VOLUME:	47.6
°F :	705

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