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PRODUCTION RESEARCH
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NOTICE

The information contained in this report is regarded as confidential and proprietary. It is provided subject to the provisions regarding confidential, proprietary information contained in the Research Agreement among the Participating Parties.

SOCONY MOBIL OIL COMPANY, INC.

RESEARCH DEPARTMENT

MONTHLY PROGRESS MEMORANDUM
(Covering October 16 to November 15, 1965)

ANVIL POINTS OIL SHALE RESEARCH CENTER

Rifle, Colorado

November 24, 1965

CONTRIBUTORS:

- Mining Section
- Mechanical Engineering
- Retorting Section
- Analytical Laboratory Section
- Engineering and Economic Analyses

Signed by:

RH Cramer
R. H. Cramer
Program Manager

NOTICE

The primary objective of the Anvil Points Oil Shale Research Center MONTHLY PROGRESS MEMORANDUM is to advise authorized personnel employed by the Participating Parties[1] that various activities are in progress or that certain significant data have been obtained within the Research Center.

These MONTHLY PROGRESS MEMORANDA have been prepared to provide rapid, on-the-spot reporting of research currently in progress at Anvil Points. The conclusions drawn by project personnel are tentative and may be subject to change as work progresses. The PROGRESS MEMORANDA have not been edited in detail.

(1) Socony Mobil Oil Company, Inc., Project Manager

Humble Oil and Refining Company

Continental Oil Company
Pan American Petroleum Corporation
Phillips Petroleum Company
Sinclair Research, Inc.

MONTHLY PROGRESS MEMORANDUM

(Covering October 16 to November 15, 1965)

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CONFIDENTIAL

MONTHLY PROGRESS MEMORANDUM

(Covering October 16 to November 15, 1965)

I. TECHNICAL ADVISORY COMMITTEE

All six Participating Parties approved an extension of Stage I funding in the amount of $720,000. At the ninth meeting, the Technical Advisory Committee approved the budget submitted for the Stage I extension period for $720,000 to cover the period from about November 4, 1965 to about May 1, 1966.

The next meeting of the committee will be held in Denver on January 13, 1966.

II. MINING SECTION (G. R. Haworth and J. B. Sellers)

A. Production

The second and third leading rounds have been drilled and blasted at the north end of "Able" haulageway. There was practically no bootleg in any of the blast holes, which was an improvement over the first 30° V-cut round. The improvement was due to the fact that the V-cuts were not pulled independently of the remainder of the rounds, as had been the case in the first one.

The shale from the second round has been hauled to the crusher plant, the yield was 1,490 tons.

There have been numerous delays in the mining operations because of breakdowns on the Industrial Monkey, which is used for scaling, roof bolting and powdering. This unit is to be thoroughly overhauled during periods when it can be released from the mine.

Two weeks have been spent on surveying. New survey stations are being installed and at the same time the coordinates of old stations are being checked with those shown on the mine plans.

B. Mine Conditions

The condition of the roof in "Able" haulageway appears satisfactory. There is no indication of any movement having taken place in recent months.

The first pillar situated between "Able" and "Charlie" haulageways shows signs that it is coming under pressure. Cracks and fractures have developed in the faces of the pillar, although they are not serious at present, close observations must be kept on this area. The pillar will be instrumented to measure any convergence taking place.

C. Water Supply System

The rehabilitation work of the mine water supply from reservoirs on the top of the mesa has been completed. The cost of this work totaled approximately $3,000 compared with the estimate of $8,000.

D. Mine Road Maintenance

In the gravel pit work has finished on the crushing of gravel for resurfacing the mine road. The gravel is presently being spread on the road and there is one more mile to cover. The job will be complete by November 20, 1965.

III. MECHANICAL ENGINEERING (W. S. Bergen)

Retort No. 2 was in operation during this period testing two types of single level low velocity distributors over a wide range of process conditions.

Most revisions scheduled for Retort No. 1 incorporating multi-level air distributors were completed.

Summary

Retort No. 2

The two types of distributors tested were

1. A 16 pipe down flow distributor with a 9 1/2 inch triangular center configuration, and
2. An 8 pipe horizontal low slot velocity (35 to 50 ft/sec) distributor on an 11" X 13" rectangular center configuration.

The 16 pipe distributor was tested for seven runs and removed when an uneven shale flow problem developed. Yields at the two conditions tested were no better than those obtained at similar conditions with the eight riser configuration. This configuration had been installed to create a condition of more air injection points with a corresponding flattening of the temperature profiles in the combustion zone. Flatter temperature profiles were developed with this distributor system.

Eight revised low slot velocity risers were installed and all test work from Run B741 to date accomplished with these distributors. No fouling of the ports has been noticed. Yields were similar to those obtained on earlier runs.

A new eight riser system has been designed and fabricated to test air injection at velocities approximating 7 to 10 ft/sec. These will be installed the week of November 15.

Discussion

A. Retort No. 2

1. As noted, neither of the two riser configurations tested fouled in comparison with the fouling discussed in last month's report. This slot riser, though unprotected from shale at the slot face, appears operable.

2. New two inch pipe risers were designed for discharge velocities of 7 to 10 ft/sec. The openings are quite large (13/16 inch). A four inch diameter protective cap was installed over the slot to prevent shale from falling into the riser.

3. The liner in the combustion zone was reinforced and warping minimized. However, deposits continued to form on the

surface. On the November 9 and 10 shutdown, the liner in the combustion zone was removed. Corners were pinned and remain. Deposits on the wall surface at this point may be acting as a bridging agent.

4. The liner in the retorting zone was ground smooth on the November 9 and 10 shutdown. This surface has roughened since its installation. Special note will be made of the shale bed condition following these liner changes.

5. The DeLaval centrifuge was tested. It proved effective in separating the oil and water phases. It was not possible to use the unit more than eight hours at a time due to deposits in the bowl of the centrifuge. These deposits decreased separation efficiency. A considerable amount of time is required to clean the unit and its use has been discontinued. Investigation is underway to find a self cleaning unit.

6. The electrostatic precipitator efficiency remains at the 98.5 percent plus level with no further wall deposits.

7. A considerable amount of effort has been spent on the Richardson system:

    a. A recorder was installed to track the action of the fill and dump cycle. This addition has made shale accounting 100 percent reliable.

    b. Electrical problems have been traced in detail. Several relays were replaced and a new scale head installed. No faults have developed since these changes. Most of the problems with the system have centered around occasional failures of a relay or a relay contact. Since there are several dozen relays each with four to six contacts, the source of the problems have been difficult to find while keeping the unit in operation.

8. The Aerotec multiclone will be installed and tested on Retort No. 2 the week of November 15.

9. No additional work has been done to test isokinetic sampling lower than the 10 percent level.

10. The spent shale drawoff flow control system in Retort No. 2 is being reviewed. On occasion, an apparent shale holdup occurs in the retort. This in turn will speed the discharge rolls until the bridge breaks. The net effect is to create uneven rate of shale flow. Tests have been conducted holding the rolls on manual control and balancing it against the raw shale feed which is on rate control.

This appears effective in calming the retort shale drawoff rate although it will not prevent bridging. The controller (Gamm-O-Tron) sources will be spread and the system tested for response.

B. <u>Retort No. 1</u>

1. Revisions to the distributor section are complete. However recent studies have indicated the need for additional changes.

The installed perpherial ports in the hot gas distributors generate exit velocities of approximately 90 ft/sec when using air and dilution gas, and 350 ft/sec when using the external combustion with oxygen and dilution gas. Velocities not greater than 50 ft/sec will be designed into the lower two hot gas distributors.

The unit will also be tested using more than one hot gas distributor level at one time. Flow measuring equipment for this condition will also be installed.

IV. RETORTING SECTION (J. E. Lawson)

A. Retorting Group (R. L. Clampitt, D. P. Cotrupe, J. W. Hasz, K. I. Jagel, E. E. Turner)

1. Summary

The quench data obtained during the last period of Retort No. 1 operation are tabulated in this report. These data were obtained during Runs 708 and 710 which were attempts to reproduce Runs 507, 508 and 509 and during Runs 714 and 717 which were runs in which oxygen was used as the oxidant. In Run 714, combustion took place in the retort while in Run 717 essentially all combustion took place external to the retort in the line burner. When Runs 708 and 710 were compared with Run 509, it was apparent that in the top of the bed there was some evidence of fines agglomeration on the large pieces of shale. At a level of three to four feet above the air distributor the average particle size begins to decrease. This reduction in size is due primarily to the upward migration of fines from the combustion zone. The very high benzene extractable content of the fines particles indicates that they do adsorb large quantities of oil. The refluxing action of the fines in the zone two to three feet above the air distributor could account for the observed yield loss in Runs 509, 708 and 710.

In Run 717, in which oxygen was used as the oxidant in firing the line burner, essentially all the combustion took place external to the retort. The fines concentration profile indicates that the fines concentration at any level is as great and in a number of cases, greater for this operating condition than for Run 710 or Run 714, in which combustion took place in the bed. This is surprising since it has generally been felt that the combustion reaction and the concomitant carbonate decomposition is a primary cause of fines generation. One thing that is peculiar about Run 717 is the relatively high injection velocity of gas into the retort. This may be the reason for the unanticipated high fines concentrations observed in this run. Other possible explanations are that the controlling fines generation mechanism is not in the zone adjacent to the air or hot gas distributor. A third possibility may be that the high observed fines concentration may have been generated during a pre-existing condition in which combustion existed in the bed.

Retort No. 2 has been operated throughout the last month. The emphasis throughout this period of operation has been on the effect of air distributor design. Of the various designs evaluated, the most satisfactory riser-type distributor yet tested appears to be an eight riser, multi-level intermediate velocity distributor. It is planned to evaluate an eight riser low velocity distributor within the next month.

A tabulation of the thief results taken from Retort No. 2 during the current operation is also presented. A detailed discussion of these data will appear in a subsequent report.

It has been shown in a limited group of observations that it is valid to assume that the gas leaving the vent purge condenser is saturated with water at the temperature of the gas leaving the condenser.

Gas samples taken from the moving shale bed indicate that the oxygen is substantially consumed within a few inches of the air inlet ports for a number of difficult air distributor configurations and operating conditions. This test will no longer be run routinely because it is unusually time consuming.

A centrifuge has been rented from DeLaval to investigate the feasibility of continuously centrifuging the liquid product to separate shale oil and water. It has been shown that this type of separation is practical, if a self-cleaning centrifuge is available. The unit tested had to be shut down for cleaning every twelve hours of operation on T3 liquid product. The cleaning procedure took two or three hours. An investigation is now underway to see if a self-cleaning centrifuge can be purchased.

Chemical demulsification of shale oil-water emulsions is a difficult operation to carry out according to Tretolite. After evaluating five of our samples, they concluded that the characteristics of the emulsion varied from one sample to the next. All samples required heating to 190 to 200° F in order that their demulsifiers be effective. In one extreme case, it was found that 500 ppm of demulsifier were required to separate the oil and water phases using four hours of settling at 190 to 200° F.

Filtration of solids would help.

2. Retort No. 1

a. <u>Evaluation of Quench Data</u>

The results of Retort No. 1 quench experiments conducted after Runs 509, 708, 710, 714 and 717 are presented in Tables 1, 2, 3, 4 and 5 respectively. Runs 509, 708 and 710 were made at the same nominal conditions of 500 lb/(hr)(ft^2) shale rate, 5,400 SCF/T air rate, and 12,800 SCF/T recycle rate. In both Runs 714 and 717 pure oxygen was used as the oxidant. In Run 714, combustion took place in the retort while in Run 717 essentially all combustion

TABLE 1

RUN 509 QUENCH DATA - RETORT NO. 1

Spent Shale	Distance From Turntable, Feet													Raw Shale
	0-1	1-2	2-3	3-4	4-5	5-6	6-7	7-8	8-9	9-10	10-11	11-12	12-13	
	52.4	53.4	53.3	55.4	55.9	55.4	60.7	65.6	65.3	61.6	66.3	64.4	70.3	
	21.5	26.2	19.8	25.8	22.0	15.7	20.2	64.4	77.0	80.5	75.9	80.0	83.5	77.0
	52.6	50.0	52.0	50.2	52.2	52.8	49.4	26.2	20.4	19.5	23.9	18.6	14.0	21.7
	25.9	23.8	28.2	24.0	25.8	31.5	30.4	9.4	2.6	0.0	0.2	0.4	2.5	1.[illegible]
0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.6	23.2	28.5	26.5	28.3	26.6	27.1	27.1
13.9	13.5	17.1	13.1	13.5	13.3	14.3	13.2	16.8	16.4	16.9	16.7	16.4	15.7	17.0
5.92	5.73	8.15	5.72	5.63	5.78	6.16	5.72	15.5	16.8	17.3	16.6	16.7	17.6	16.3
2.13	2.06	3.50	2.15	1.94	2.15	2.26	2.12	10.9	12.3	12.7	12.0	12.2	13.3	11.7
0.13	0.15	0.39	0.21	0.20	0.28	0.28	--	1.56	1.72	1.81	1.68	1.71	1.88	1.73
0.12	0.08	0.30	0.12	0.08	0.08	0.11	--	1.22	1.52	1.64	1.49	1.55	1.69	1.57
84.3	84.2	79.5	84.2	83.5	83.0	81.8	84.1	69.6	67.8	66.9	67.5	67.3	66.8	68.0
0.09	0.21	0.85	0.74	0.65	0.63	0.42	0.39	0.08	0.10	0.14	0.21	0.26	0.26	0.34
--	0.14	0.21	0.13	0.13	0.13	0.14	0.13	0.39	0.43	0.46	0.41	0.43	0.46	0.39
	0.06	0.85	0.05	0.05	0.23	0.28	0.10	3.07	3.22	2.58	2.10	2.12	2.53	(1.3)

Packed Density, lb/ft^3

Screen Analysis
  ($D > 0.742$), Wt %
  ($0.093 < D < 0.742$), Wt %
  Pan ($D < 0.093$), Wt %

Analyses of Total Sample
  Fischer Assay, G/T
  Mineral CO_2, Wt %
  Total Carbon, Wt %
    Organic Carbon, Wt %
  Total Hydrogen, Wt %
    Organic Hydrogen, Wt %
  Ash, Wt %
  Moisture, Wt %
  Nitrogen, Wt %
  Benzene Extractable, Wt %

TABLE 2

RUN 708 QUENCH DATA - RETORT NO. 1

Spent Shale	Distance from Turntable, Feet 0-1	1-2	2-3	3-4	4-5	5-6	6-7	7-8	8-9	9-10	10-11	11-12	12-13	[illegible]
	51.4	84.5	60.5	57.4	62.6	75.8	75.9	84.8	84.2	70.8	74.6	74.4	70.8	
	18.4	16.2	20.6	18.5	18.6	12.0	29.5	56.8	69.3	79.6	79.7	82.2	82.6	72.4
	66.0	50.4	56.9	52.2	52.0	49.2	36.6	29.5	23.5	17.8	19.1	17.0	16.5	25.7
	25.6	32.5	23.0	30.0	29.9	38.5	34.2	13.6	7.2	2.8	1.5	1.1	0.9	1.9
0.0	0.0				0.0	0.0	7.5	21.0			24.0		26.6	27.2
14.2	11.2				13.3	14.3	17.5	16.1			17.7		16.7	17.6
5.75	4.79				5.49	6.48	10.2	14.8			15.5		16.6	16.7
1.87	1.73				1.86	2.58	5.4	10.4			10.7		12.0	11.9
0.15	0.23				0.26	0.32	0.77	1.41			1.62		1.76	1.73
0.13	0.15				0.15	0.17	0.60	1.26			1.47		1.63	1.58
84.4	86.6				84.0	82.2	74.9	70.8			68.9		68.3	67.3
0.10	0.18				0.20	0.22	0.21	0.11			0.27		0.22	0.51
--	--				--	--	--	--			--		--	--
--	0.14				0.18	0.24	1.55	2.77			1.93		1.65	--
	0.0	0.0	0.0		0.0	0.0	2.5	10.3			26.3		27.8	
	11.2				11.6	15.4	16.5	14.1			13.0		13.8	
	5.81	6.09			6.30	7.15	9.75	14.5			19.2		18.6	
	2.78				3.13	2.95	5.24	10.6			15.6		14.8	
	0.20	0.23			0.23	0.30	0.56	1.25			1.91		1.87	
	0.16	0.14			0.13	0.15	0.12	0.30			1.38		1.62	
	85.5				84.3	81.3	75.9	69.8			67.2		67.5	
	0.17				0.20	0.32	1.85	9.08			12.0		9.56	
	0.13				0.17	0.26	0.55	2.84			0.54		0.37	

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11/8/65

Packed Density, lb/ft^3

Screen Analysis
- $(D > 0.742)$, Wt %
- $(0.093 < D < 0.742)$, Wt %
- Pan $(D < 0.093)$, Wt %

Analyses of Total Sample
- Fischer Assay, G/T
- Mineral CO_2, Wt %
- Total Carbon, Wt %
  - Organic Carbon, Wt %
- Total Hydrogen, Wt %
  - Organic Hydrogen, Wt %
- Ash, Wt %
- Moisture, Wt %
- Nitrogen, Wt %
- Benzene Extractable

Analyses of Pan Fraction
- Fischer Assay, G/T
- Mineral CO_2, Wt %
- Total Carbon, Wt %
  - Organic Carbon, Wt %
- Total Hydrogen, Wt %
  - Organic Hydrogen
- Ash, Wt %
- Benzene Extractable, Wt %
- Moisture, Wt %

TABLE 3

RUN 710 QUENCH DATA - RETORT NO. 1

Spent Shale	Distance From Turntable, Feet													Raw Shale
	0-1	1-2	2-3	3-4	4-5	5-6	6-7	7-8	8-9	9-10	10-11	11-12	12-13	
	57.0	54.0	53.0	54.0	55.9	58.0	64.5	73.6	72.9	67.1	81.3	71.1	70.7	
	18.6	16.9	17.7	16.5	23.0	17.7	27.1	58.5	71.8	82.5	81.7	84.2	85.2	75.8
	53.5	51.4	54.6	51.9	76.4	52.1	39.4	25.2	20.5	16.0	16.9	14.8	14.5	23.0
	28.0	31.7	27.7	31.6	23.6	30.8	33.9	16.3	8.2	2.2	1.7	1.0	0.6	1.6
0.0	0.0				0.0	0.0	6.1	20.0			26.1		30.8	27.3
12.9	13.2				10.2	12.0	16.4	18.1			17.9		16.7	17.4
5.26	5.41				4.56	5.56	9.07	14.7			16.3		17.8	16.8
1.74	1.81				1.78	2.28	4.59	9.8			11.4		13.2	12.0
0.14	0.24				0.20	0.26	0.69	1.36			1.71		1.95	1.75
0.12	0.14				0.10	0.15	0.57	1.23			1.53		1.75	1.58
85.8	84.4				87.4	85.1	76.7	69.5			68.0		66.5	67.6
0.04	0.24				0.16	0.17	0.21	0.27			0.36		0.35	0.38
--	--				--	--	--	--			--		--	--
--	0.19				0.11	0.19	1.34	2.89			2.34		2.10	--
	0.0				0.0	0.0	1.5	8.1			26.5		26.3	
	10.8				10.0	11.7	15.4	14.2			12.5		13.9	
	5.86				5.60	6.53	8.54	12.7			18.3		17.6	
	2.91				2.87	3.33	4.33	8.8			14.9		16.8	
	0.26				0.22	0.27	0.43	1.02			1.84		1.90	
	0.14				0.13	0.16	0.30	0.88			1.48		1.47	
	85.5				86.6	84.0	79.0	73.7			68.2		67.9	
	0.32				0.18	0.25	0.98	5.83			12.0		7.79	
	0.35				0.25	0.30	0.31	1.12			0.85		0.95	

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11/8/65

Packed Density, lb/ft^3

Screen Analysis
- ($D > 0.742$), Wt %
- ($0.093 < D < 0.742$), Wt %
- Pan ($D < 0.093$), Wt %

Analyses of Total Sample
- Fischer Assay, G/T
- Mineral CO_2, Wt %
- Total Carbon, Wt %
  - Organic Carbon, Wt %
- Total Hydrogen, Wt %
  - Organic Hydrogen, Wt %
- Ash, Wt %
- Moisture, Wt %
- Nitrogen, Wt %
- Benzene Extractable, Wt %

Analyses of Pan Fraction
- Fischer Assay, G/T
- Mineral CO_2, Wt %
- Total Carbon, Wt %
  - Organic Carbon, Wt %
- Total Hydrogen, Wt %
  - Organic Hydrogen
- Ash, Wt %
- Benzene Extractable, Wt %
- Moisture, Wt %

TABLE 4

RUN 714 QUENCH DATA - RETORT NO. 1

	Distance From Turntable, Feet													
Spent Shale	0-1	1-2	2-3	3-4	4-5	5-6	6-7	7-8	8-9	9-10	10-11	11-12	12-13	Raw Shale
	66.2	60.8	61.2	62.0	60.8	69.4	71.6	71.1	73.1	74.7	68.4	72.7	70.8	
	15.3	19.0	18.3	16.4	19.9	20.3	57.5	71.2	74.0	79.4	74.8	75.4	78.6	75.1
	55.7	54.9	55.6	55.5	56.3	51.3	31.0	24.4	24.0	18.7	23.7	23.6	20.0	23.5
	29.0	25.8	26.0	28.8	24.1	28.4	10.3	4.1	1.4	1.1	1.6	0.8	1.4	1.4
0.0	0.0		0.0		0.0	2.2	16.8	27.3	25.6		23.5		23.9	26.8
16.2	14.9		14.9		12.5	16.0	17.8	17.1	17.4		17.8		17.8	17.4
6.38	6.07		5.88		5.56	8.08	14.1	16.6	15.9		15.0		15.6	16.5
1.93	2.00		1.81		2.15	3.71	9.24	11.8	11.2		10.1		10.7	11.7
0.17	0.23		0.27		0.32	0.51	1.32	1.66	1.65		1.53		1.62	1.74
0.15	0.12		0.15		0.04	0.20	0.88	1.48	1.47		1.37		1.45	1.57
82.0	81.4		82.3		83.8	78.5	70.8	67.6	68.2		69.4		68.6	68.0
0.06	0.23		0.26		0.74	0.28	0.23	0.18	0.26		0.23		0.29	0.29
--														--
--	0.13		0.20		0.51	0.90	2.51	2.60	2.24		1.92		1.62	--
	0.0	0.0	0.0	0.0	0.0	0.8	6.1	15.8		24.9		22.0		
	14.3	13.8		13.3	13.1	15.5	15.7	15.0		13.9	14.7	15.3		
	6.56	6.56		6.61	6.68	8.55	11.3	14.2		17.0	15.4	15.4		
	2.60	2.79		2.98	3.04	4.32	7.0	10.1		13.2	11.4	11.2		
	0.24	0.25		0.29	0.31	0.45	0.79	1.23		1.75		1.58		
	0.13	0.13		0.14	0.11	0.02	0.64	1.01		1.39		1.28		
	82.2	82.0	82.1	82.4	82.3	78.6	75.1	72.6		69.5	70.6	68.4		
	0.53		0.30		0.44	0.98	3.19	4.97		8.54	5.47	4.88		
	0.18	0.26		0.31	0.30		0.25	0.20			0.22			

JWHasz & KIJagel
11/8/65

Packed Density, lb/ft^3

Screen Analysis
- $(D > 0.742)$, Wt %
- $(0.093 < D < 0.742)$, Wt %
- Pan $(D < 0.093)$, Wt %

Analyses of Total Sample
- Fischer Assay, G/T
- Mineral CO2, Wt %
- Total Carbon, Wt %
  - Organic Carbon, Wt %
- Total Hydrogen, Wt %
  - Organic Hydrogen, Wt %
- Ash, Wt %
- Moisture, Wt %
- Nitrogen, Wt %
- Benzene Extractable

Analyses of Pan Fraction
- Fischer Assay, G/T
- Mineral CO2, Wt %
- Total Carbon, Wt %
  - Organic Carbon, WT %
- Total Hydrogen, Wt %
  - Organic Hydrogen, Wt %
- Ash, Wt %
- Benzene Extractable, Wt %
- Moisture, Wt %

TABLE 5

RUN 717 QUENCH DATA - RETORT NO. 1

Spent	Distance From Turntable, Feet													Raw
Shale	0-1	1-2	2-3	3-4	4-5	5-6	6-7	7-8	8-9	9-10	10-11	11-12	12-13	Shale
	64.9	65.0	63.7	63.7	64.0	64.3	70.4	68.3	67.6	67.6	68.9	70.5	69.4	
	15.2	12.3	19.6	13.4	15.4	18.0	52.8	63.1	74.0	82.8	83.0	71.8	84.4	78.4
	51.2	48.6	52.9	50.3	49.9	47.3	32.3	24.2	20.5	16.2	15.3	16.7	14.8	20.7
	33.6	39.1	28.2	37.1	35.0	35.8	15.7	13.6	3.9	1.1	1.6	1.3	0.8	0.9
0.0	0.0				0.0	0.9	9.7	25.3			29.0		23.3	27.6
19.4	18.1				18.3	18.3	19.9	17.4			17.3		17.0	17.3
7.52	7.58				7.27	7.67	11.7	16.5			17.4		14.7	16.9
2.22	2.63				2.27	2.67	6.3	11.7			12.7		10.1	12.2
0.24	0.29				0.32	0.41	0.89	1.66			1.88		1.55	1.81
0.20	0.20				0.21	0.32	0.78	1.54			1.73		1.58	1.61
78.5	78.3				78.5	77.8	72.2	68.2			66.8		69.8	67.4
0.04	0.12				0.12	0.10	0.15	0.15			0.23		0.28	0.32
--	--				--	--	--	--			--		--	--
--	0.12				0.09	0.40	2.75	3.64			2.47		1.6	--
	0.0				0.0	1.0	5.5	12.2			31.2		32.8	
	18.3				18.4	18.8	18.1	16.3			15.0		14.9	
	8.83				8.87	9.44	11.7	15.6			20.5		20.6	
	3.83				3.85	4.30	6.7	11.2			16.4		16.5	
	0.36				0.36	0.44	0.76	1.24			2.08		2.23	
	0.26				0.27	0.35	0.63	0.79			1.89		2.05	
	76.8				76.6	76.1	73.8	69.2			64.9		65.7	
	0.21				0.12	0.44	2.85	7.54			11.7		9.43	
	0.18				0.13	0.13	0.25	1.36			0.37		0.40	

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Packed Density, lb/ft^3

Screen Analysis
(D > 0.742), Wt %
(0.093 < D < 0.742), Wt %
Pan (D < 0.093), Wt %

Analyses of Total Sample
Fischer Assay, G/T
Mineral CO2, Wt %
Total Carbon, Wt %
Organic Carbon, Wt %
Total Hydrogen, Wt %
Organic Hydrogen
Ash, Wt %
Moisture, Wt %
Nitrogen, Wt %
Benzene Extractable

Analyses of Pan Fraction
Fischer Assay, G/T
Mineral, CO2, Wt %
Total Carbon, Wt %
Organic Carbon, Wt %
Total Hydrogen, Wt %
Organic Hydrogen
Ash, Wt %
Benzene Extractable
Moisture, Wt %

took place external to the retort in the line burner.

Figures 1, 2 and 3, plotted from the tabular data, graphically illustrate the particle size degradation that occurs as shale passes through the retort. Some preliminary conclusions that can be drawn from these figures are:

(1) Near the top of the bed small particles apparently agglomerate with large pieces of shale, probably with oil as a binder, to provide an increase in the greater than 0.742 inch particle size fraction.

(2) At a level of three to four feet above the air distributor, average particle size begins to decrease. This decrease in average particle is primarily due to fines that have migrated upward from the combustion zone. This is indicated by the low mineral CO_2 concentration in the pan fraction when compared to the total sample.

(3) At the air distributor level, the concentration of particles greater than 0.742 inch in diameter has decreased from about 75 wt % to about 15 wt %. The concentration of medium size particles with diameters between 0.093 and 0.742 inch increased from about 20 wt % to about 50 wt %. The concentration of small particles with diameters less than 0.093 inch has also increased from about 10 wt % to about 35 wt %.

(4) Average particle diameter increases somewhat immediately below the air distributor and then remains essentially constant throughout the spent shale cooling zone.

Presented in Figures 4 and 5 are plots describing weight percent benzene extractables as a function of height for both all the shale and the fine particles ($D < 0.093$). The very high benzene extractable (decomposed kerogen) content of the fine particles indicates that they do adsorb large quantities of oil. Although the pan fraction benzene extractable curve peaks at 12 percent about five feet above the air distributor the amount of oil involved at this level is small because of the low fines concentration. At a level about two to three feet above the air distributor, however, the fines concentration is sufficiently high to transport fairly large quantities of oil downward into the combustion zone.

Presented in Figure 6 is the packed bulk density data obtained as the quenched material was withdrawn from the retort. To obtain this data the net weight of material in each 30 gallon drum was divided by the approximate volume occupied in the drum. While the numbers are not

FIGURE 1

RETORT NO. 1 QUENCH TEST

WEIGHT % OF MATERIAL RETAINED ON SCREEN WITH 0.742 INCH OPENING

Symbol	Run
x	509
△	708
○	710

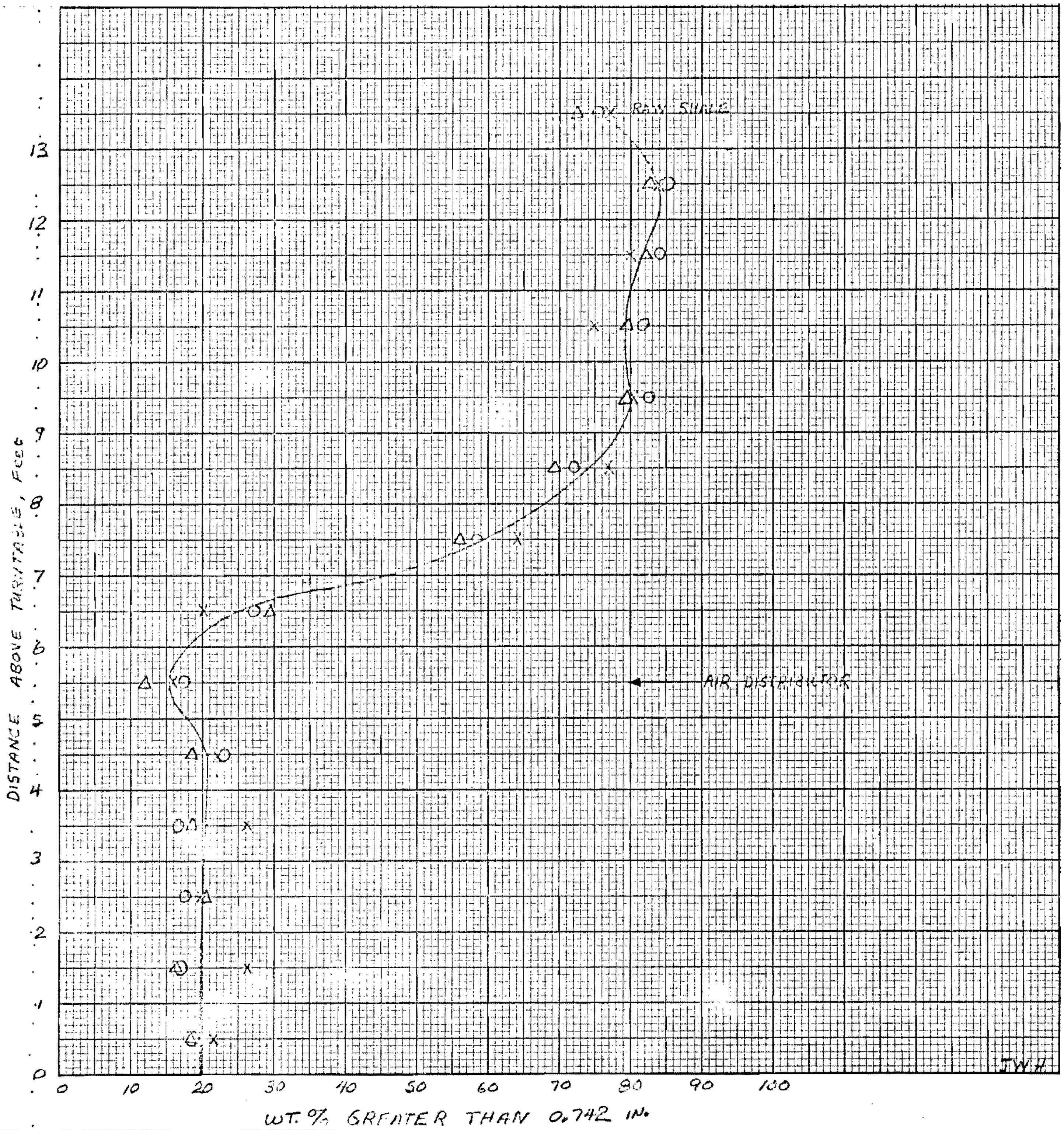


FIGURE 2

RETORT NO. 1 QUENCH TEST

WEIGHT % OF MATERIAL RETAINED ON SCREEN WITH 0.093 INCH OPENING - THROUGH SCREEN WITH 0.742 INCH OPENING

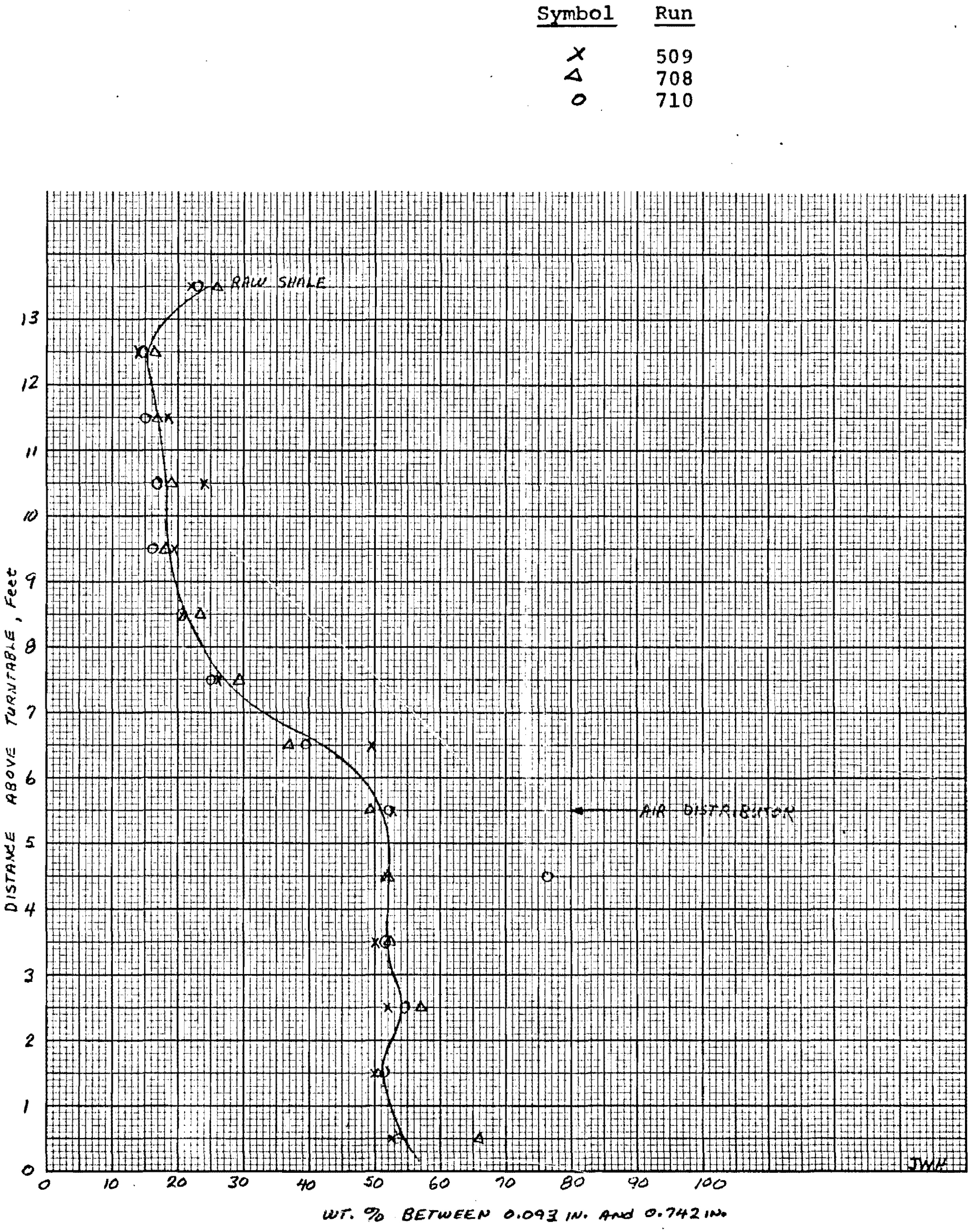


FIGURE 3

RETORT NO. 1 QUENCH TEST

WEIGHT % OF MATERIAL THROUGH SCREEN WITH 0.093 INCH OPENING

Symbol	Run
×	509
△	708
○	710

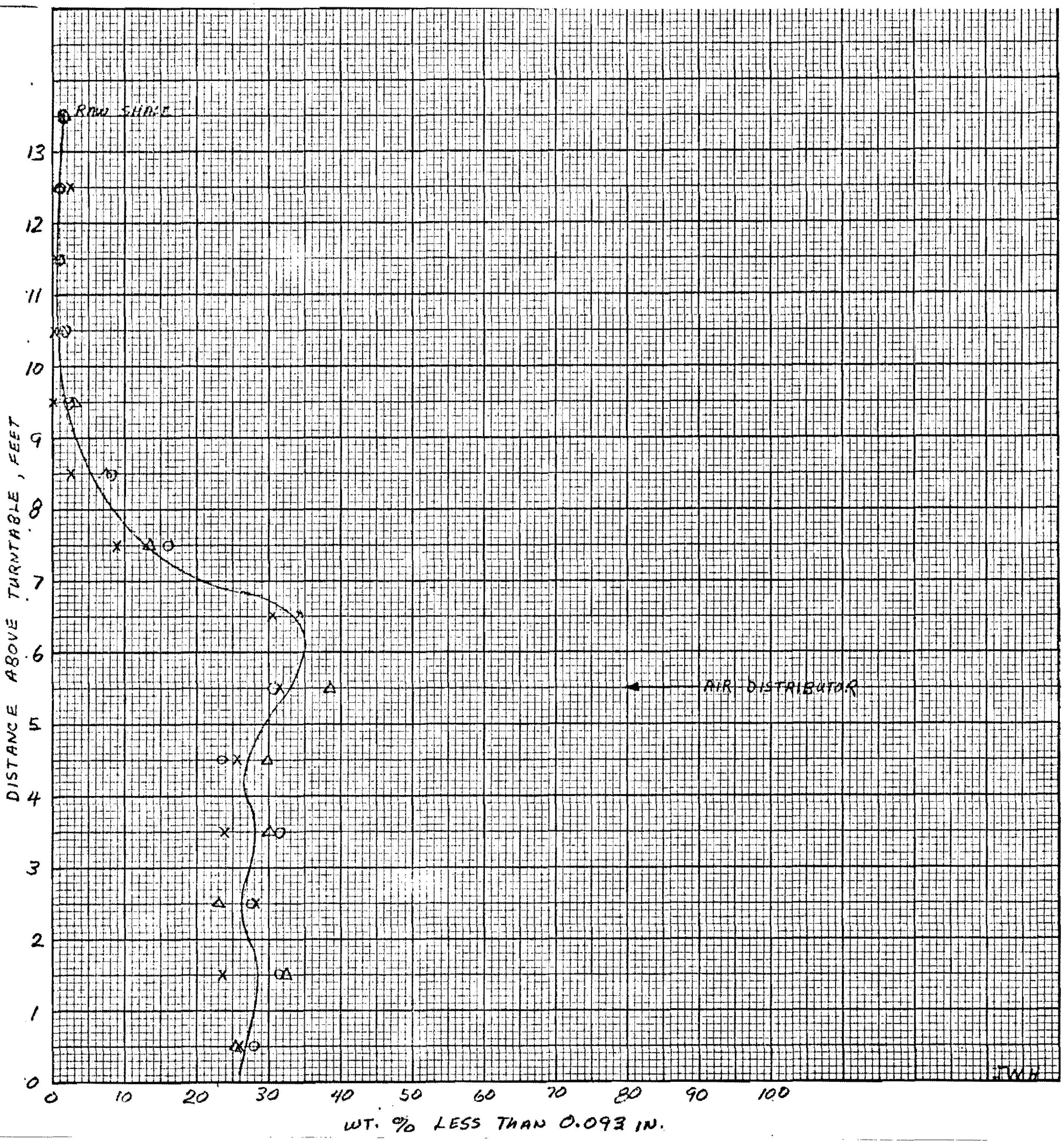


FIGURE 4

RETORT NO. 1 QUENCH TEST

BENZENE EXTRACTABLE MATERIAL, TOTAL SAMPLE

Symbol	Run
△	708
○	710

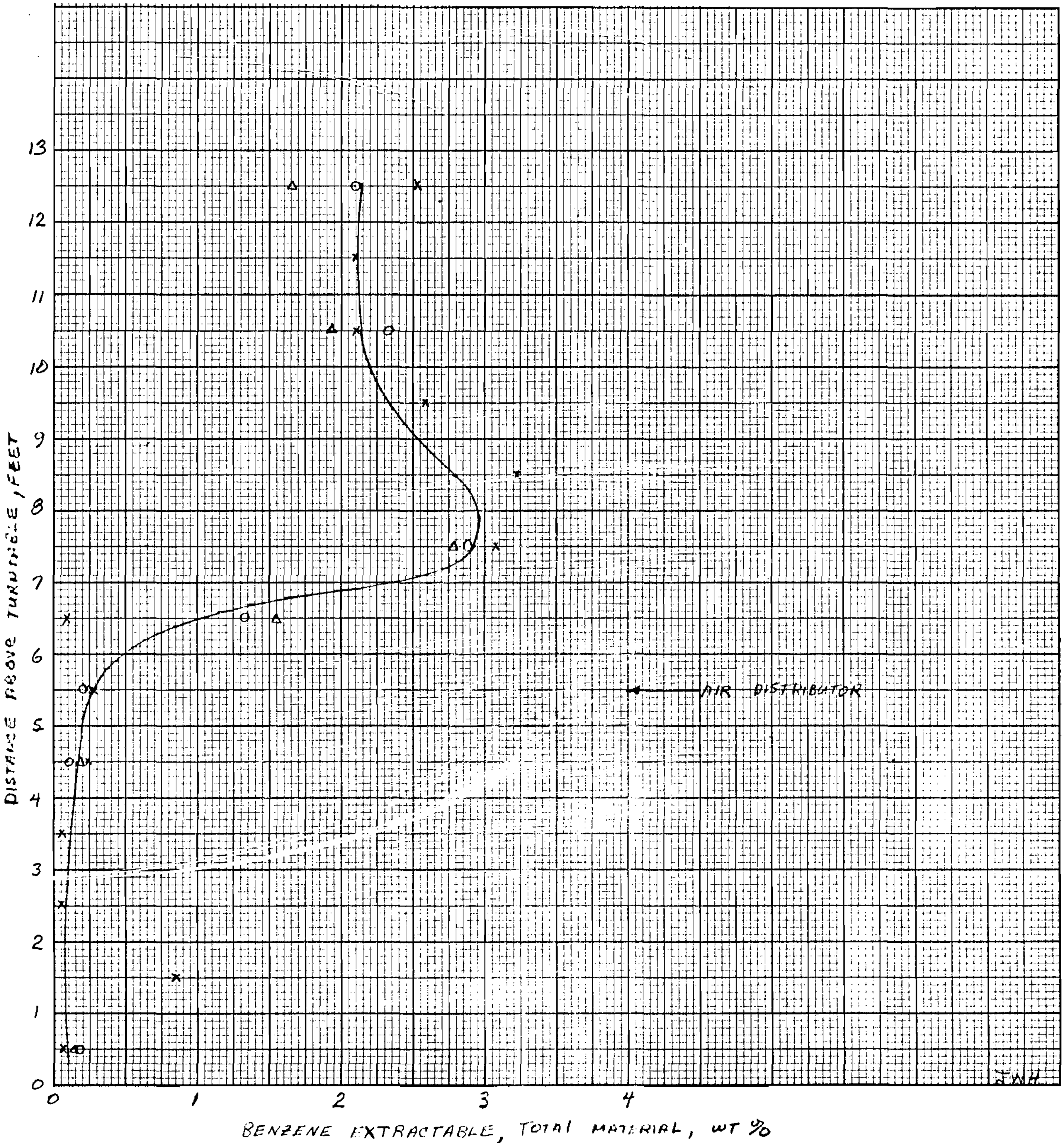


FIGURE 5

RETORT NO. 1 QUENCH TEST

BENZENE EXTRACTABLE MATERIAL, PAN FRACTION

Symbol	Run
△	708
○	710

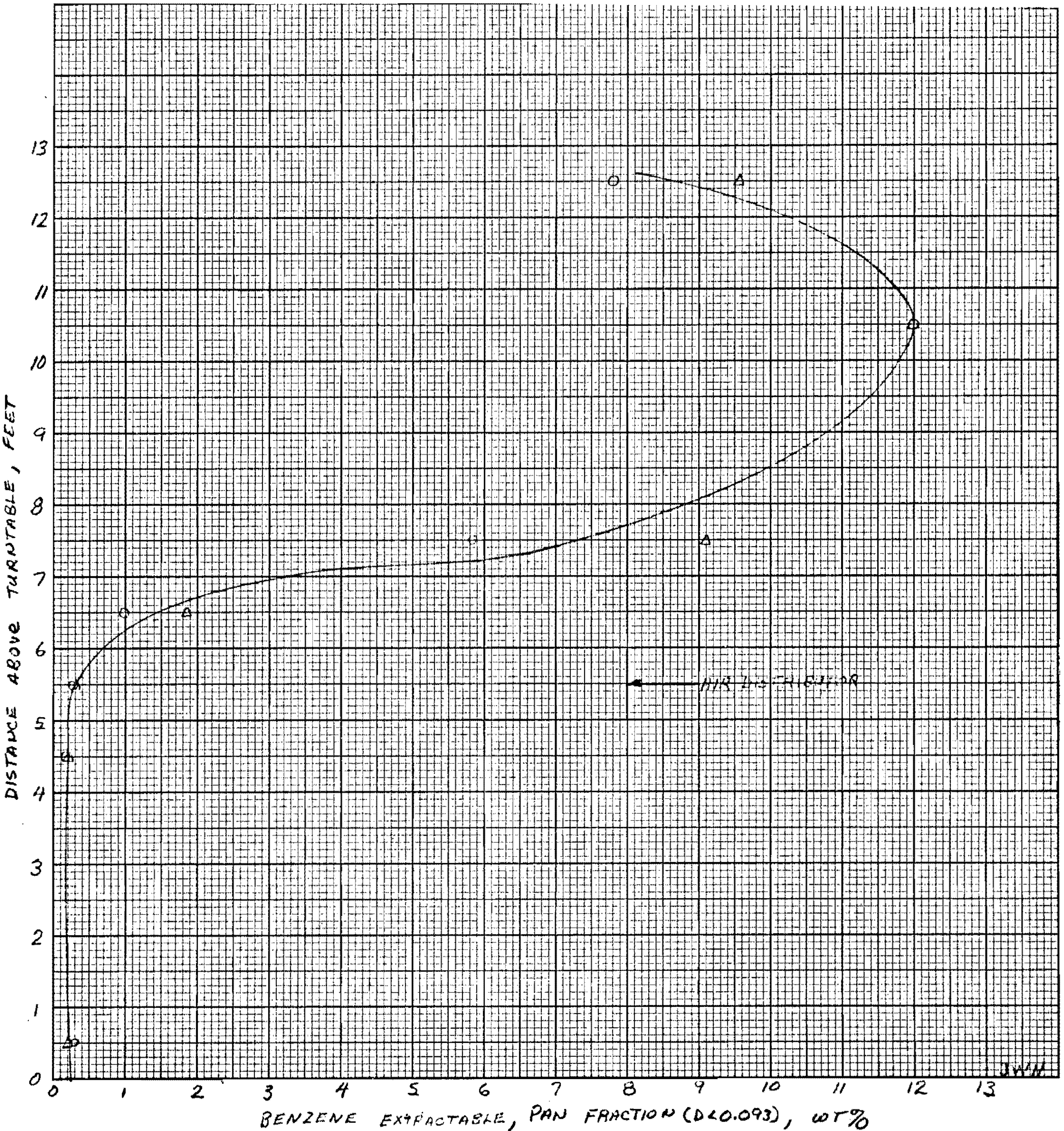
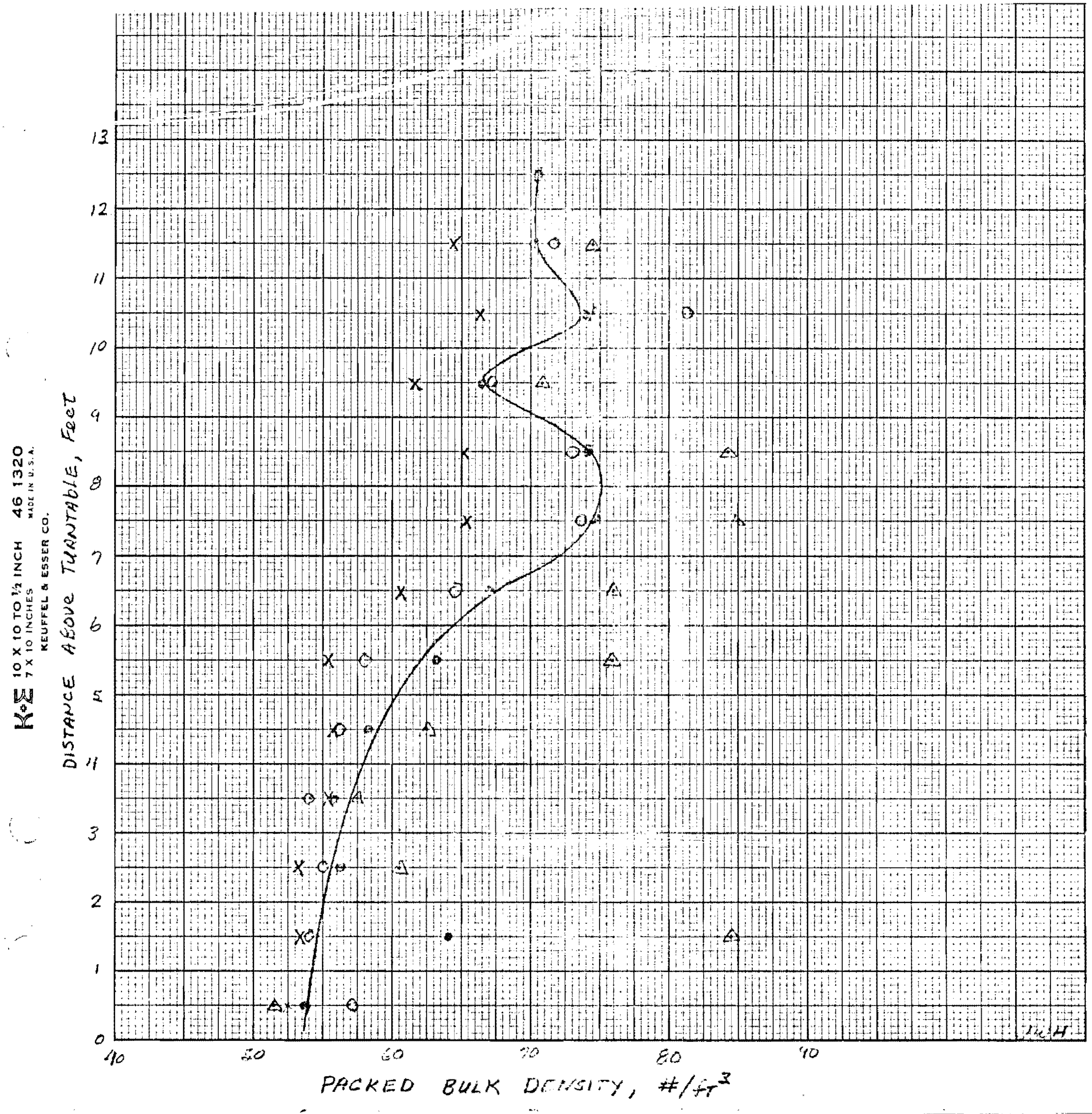


FIGURE 6

RETORT NO. 1 QUENCH TEST

PACKED BULK DENSITY AS A FUNCTION OF HEIGHT

Symbol	Run
X	509
△	708
O	710
●	Average



precise and do not define the actual bulk density in the retort, they do provide a basis for comparing the material present at each level.

The data for Runs 714 and 717 have not been analyzed in detail. However, there is one unexpected conclusion that can be drawn from these studies. If one compares the fines concentrations in the central portion of the retort for Run 717 with those of Runs 710 and 714, it is apparent that the fines concentration is as high or higher. The surprising factor is that there was essentially no combustion within the bed. This shows that combustion and its concomitant carbonate decomposition are not the only causes of fines generation. Because of the high temperature that existed in the hot gas injected through the hot gas distributor for Run 717, the gas entered with a high injection velocity compared to that attained in Runs 710 or 714. This may have resulted in the attrition of the retorted shale. Another possibility is that the causative factor in fines generation is in some other portion of the retort such as the retorting zone. Finally, it may be that the high fines concentration was due to some pre-existing condition in the retort in which combustion was carried out in the bed. A comparison among the fines concentrations obtained in Runs 710, 714 and 717 are tabulated below:

WEIGHT % OF SHALE FINER THAN .093 INCHES

Feet Above Air Distributor	Run 710	Run 714	Run 717
+ 3	8.2	1.4	3.9
+ 2	16.3	4.1	13.6
+ 1	33.9	10.3	15.7
0	30.8	28.4	35.8
- 1	23.6	24.1	35.0
- 2	31.6	28.8	37.1

3. Retort No. 2

a. Retort Operation

During the last month Runs B725 through B750 have been made on the No. 2 Retort. Runs B725 through B727 employed the single level eight riser air distributor design (Type VIII) used for Runs B718 through B724 reported previously. Nominal run conditions were 500 lb/(hr)(ft^2) mass rate, 16,000 SCF/T recycle rate and 4,400 SCF/T air rate. Average yield at this condition was 88.8 percent Fischer Assay. Unit operation was smooth and trouble-free.

At the conclusion of Run B727, the unit was shut down to permit alteration of the distributor configuration. During this shutdown, the unit was inspected and found to be in good operating condition. Partial plugging of some of the riser slots was observed, although none of the risers was completely plugged.

The distributor alteration made after Run B727 involved raising the level of four of the risers to a height eight inches above the original level, producing a two level distribution system. This system was employed in Runs B728 through B733 in an effort to determine the effect of physically expanding the combustion zone. It is realized, of course, that this particular configuration does not represent the optimum two-level air distribution system as it does not provide complete distribution at each level; however, it was designed to serve as an indicator of the relative merits of a two-level air distribution system.

Runs B728 through B730 were made at base case conditions of 500 lb/(hr)(ft^2) mass rate, 11,500 SCF/T recycle and 5,000 SCF/T air rate. Average yield at this condition was 77.0 percent Fischer Assay. Runs B731 through B733 were made at base case conditions of 500 lb/(hr)(ft^2) mass rate, 16,000 SCF/T recycle and 4,400 SCF/T air rate. Average yield was 85.8 percent Fischer Assay. The unit performed satisfactorily with the multi-level distributor, but not as well as with the previous single level distributors.

Upon completion of Run B733 the unit was shut down in preparation for the installation of another distributor configuration. This change consisted of the installation of a distributor with sixteen risers at a single level to test the possible advantage of increased coverage. During the shutdown period the retort was inspected and found to be in reasonably good operating condition. Small deposits (similar to that found in the previous inspection) were found on the east and west walls above the air distributor level and some of the riser slots were partially plugged. The roll feeders were checked and found to be functioning properly. The separation of the liner from the west wall was deemed sufficiently large to require repair and it was therefore drawn back to the wall and repinned. The new distributor configuration (Type X) was installed.

Runs B734 through B736 were made at base conditions of 5,000 SCF/T air rate and 11,500 SCF/T recycle rate. Average yield at this condition was 80.2 percent Fischer Assay. Unit operation was smooth and trouble-free.

Runs B737 through B740 were made at base conditions of 4,400 SCF/T air rate and 16,000 SCF/T recycle rate. Average yield at this condition was 85.5 percent Fischer Assay.

Unit operation, while fairly smooth, was not as satisfactory as desired. During Run B739 a void was noted in the northwest corner of the retort above the air distributor. It was also noted that considerable oil refluxing was occurring in the bed. The reasons for the relatively poorer operation are not defined.

The unit was shut down at the conclusion of Run B740 to install a modified version (Type XI) of the eight riser single level intermediate velocity (Type VIII) distributor used previously. Inspection of the 16 risers removed from the retort revealed rather severe plugging inside the risers. This is currently explained on the basis of oil entrainment from the air blower combined with a dirty air filter. Also, the riser temperature was probably hotter than in previous runs with vertical riser distributors because of the downward air injection angle.

Runs B741 through B744 were made with the Type XI distributor at base conditions of 500 lb/(hr)(ft^2) mass rate, 4,400 SCF/T air rate and 16,000 SCF/T recycle rate. The average yield for these four runs was 86.2 percent Fischer Assay. The actual recycle rate is probably somewhat lower than the target value of 16,000 SCF/T since an average gas loss of about 1,300 SCF/T was calculated. Yields might have been slightly higher had the target recycle rate been achieved. Overall operation at these conditions was smooth and trouble-free. A unit shutdown was occasioned at the end of Run B741 to remove the thief sampler from the retort. The sampler handle was severely bent during insertion into the bed through the port 23 inches above the air distributor. The cause of a raw shale feed system failure that occurred during the shutdown was traced to a loose power cable.

Runs B745 through B747 were made at nominal conditions of 400 lb/(hr)(ft^2) mass rate, 4,000 SCF/T air rate and 19,000 SCF/T recycle rate. Average yield for the series of three runs was 87.2 percent Fischer Assay. The actual recycle rate is probably somewhat lower than the target rate of 19,000 SCF/T as a result of a calculated gas loss of about 1,400 SCF/T. The average actual recycle rate for these runs is estimated to be about 18,000 SCF/T when the gas loss is deducted from the measured recycle. The cause of the high gas losses was traced to a plugged manometer used to indicate pressure balance between the bottom star feeders. Overall operation at these conditions was smooth and trouble-free.

Runs B748 through B750 were made at nominal conditions of 500 lb/(hr)(ft^2) shale rate, 5,800 SCF/T air rate and 11,500 SCF/T recycle rate. Average yield for this high air rate, low recycle rate condition was 83.8 percent Fischer Assay.

Better pressure balance of the bottom star feeders is reflected in this series of runs by the average calculated gas loss of 150 SCF/T. Operation was smooth at these conditions except for trouble experienced with the raw shale feed system. The raw shale feed system problems were traced to defective relays in the Richardson panel.

b. Evaluation of Thief Samples

A tabulation of the thief sample data obtained to date in our current period of operation (Runs B725 to B750) are presented in Tables 6, 7 and 8. In Runs B725 through B727 and in Runs B741 through B750 the following thief sampling points were used:

Thief Port No.	Inches Above Air Distributor
P_1	+ 23
P_2	+ 10 1/2
P_3	0
P_4	- 11 1/4

In all of these runs an eight riser single level air distributor was used. In Runs B728 through B733 an eight riser double level air distributor was used. With these runs, the port designations had the following significance:

Thief Port No.	Inches Above Top Air Distributor Port Level	Inches Above Bottom Air Distributor Port Level
P_1	+ 15	+ 23
P_2	+ 2 1/2	+ 10 1/2
P_3	- 8	0
P_4	- 19 1/4	- 11 1/4

The sixteen riser single level air distributor was used in Runs B734 through B740. This design prevented the use of thief ports P_3 and P_4, however, ports P_1 and P_2 were located 23 and 10 1/2 inches, respectively, above the air distributor port level.

Table 6 presents data obtained at 500 lb/(hr)(ft^2) mass rate, 5,000 SCF/T air, 11,500 SCF/T recycle rate and seven feet bed height and also at 400 lb/(hr)(ft^2), 4,000 SCF/T air rate, 19,000 SCF/T recycle rate and nine feet bed height. Table 7 summarizes data obtained at 500 lb/(hr)(ft^2) mass rate, 4,400 SCF/T air rate, 16,000 SCF/T recycle rate and nine feet bed height. Table 8 is a tabulation of data obtained at 500 lb/(hr)(ft^2), 5,800 SCF/T air rate and 11,500 SCF/T recycle rate.

TABLE 6

THIEF SAMPLES

500 Pound Mass Rate, 5,000 SCF/T air, 11,500 Recycle, 7 Feet Bed Height

B728				B730				B734		B735		B736	
P_1	P_2	P_3	P_4	P_1	P_2	P_3	P_4	P_1	P_2	P_1	P_2	P_1	P_2
67.8	47.4	23.1	49.2	48.2	43.0	20.3	27.7	57.6	39.3	53.6	43.9	45.3	50.0
18.5	28.3	54.7	41.0	14.3	29.3	37.9	45.8	15.3	19.9	18.7	9.2	13.3	18.9
1.6	3.2	7.2	3.4	1.2	6.7	9.8	7.6	1.9	2.1	2.6	1.1	1.4	3.1
2.5	5.6	6.9	3.0	4.0	8.4	14.2	8.5	6.8	6.1	7.2	5.8	6.2	9.2
9.6	15.5	8.1	3.4	32.3	12.6	17.8	10.4	18.4	32.6	17.9	40.0	33.8	18.8
23.6	15.3	0.6	0.0	21.6	5.7	0.6	0.0	26.0	19.2	22.9	18.2	21.1	19.1
15.2	16.3	16.1	16.3	15.3	16.9	16.7	16.5	16.1	14.6	16.3	13.9	14.4	16.5
16.0	13.3	6.78	5.88	16.5	10.6	8.27	7.10	17.9	15.0	16.8	14.9	15.4	14.9
1.60	1.12	0.25	0.15	1.52	0.67	0.31	0.22	1.67	1.32	1.58	1.26	1.41	1.26
70.1	73.0	81.7	82.3	70.4	76.2	79.7	80.9	67.8	72.6	68.9	73.6	72.2	71.4
1.30	1.0	0.05	0.0	0.9	0.4	0.2	0.3	0.8	0.8	0.7	0.6	0.9	0.7
3.04	1.12	0.09	0.09	2.52	2.10	0.22	0.11	2.22	2.17	2.19	1.97	2.85	2.00

400 Pound Mass Rate, 4,000 SCF/T Air, 19,000 SCF/T Recycle, 9 Feet Bed Height

B745				B746				B747				B748			
P_1	P_2	P_3	P_4	P_1	P_2	P_3	P_4	P_1	P_2	P_3	P_4	P_1	P_2	P_3	P_4
48.1	28.6	28.3	21.2	58.9	33.0	30.8	34.8	41.6	29.5	30.2	26.6	44.0	26.8	24.3	27.4
16.7	26.4	32.9	50.0	25.1	31.6	39.4	42.8	17.3	30.2	35.7	44.4	17.6	31.7	37.8	37.5
2.8	6.5	8.1	9.5	2.1	6.2	7.4	8.5	3.6	7.3	6.2	7.7	2.2	6.1	7.0	7.7
5.8	10.2	12.6	8.2	3.1	9.0	8.4	7.0	7.0	11.2	10.7	8.5	6.0	11.4	10.5	9
26.6	28.3	18.1	11.1	10.8	20.2	14.0	6.9	30.5	21.8	17.2	12.8	30.2	24.0	20.4	17.9
24.1	16.5	1.2	0.9	26.0	13.4	1.7	0.0	24.0	13.5	0.0	0.0	20.3	9.2	0.7	0.0
15.0	15.8	18.9	18.3	15.5	17.9	16.9	18.6	15.0	17.1	17.2	18.4	15.4	18.4	16.6	16.5
16.6	14.7	8.63	7.99	16.4	13.8	8.01	7.52	17.0	13.7	7.51	7.64	15.8	12.1	7.51	7.21
1.54	1.18	0.32	0.31	1.63	1.10	0.36	0.25	1.58	1.11	0.25	0.23	1.44	0.81	0.28	0.20
70.0	72.4	77.6	78.2	69.8	72.0	79.1	79.1	69.8	72.7	80.2	78.9	71.2	73.7	80.6	81.0
0.9	0.7	0.2	0.4	0.6	0.5	0.4	0.5	0.6	0.5	0.3	0.3	0.7	0.4	0.2	0.4
2.12	2.78	0.14	0.07	2.17	3.37	0.29	0.15	2.83	3.28	0.10	0.08	1.69	1.43	0.14	0.08

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Screen Analysis
$D > 0.742$
$0.185 < D < 0.742$
$0.093 < D < 0.185$
$0.0328 < D < 0.093$
Pan, $D < 0.0328$
Analysis of Sample
Fischer Assay
Mineral CO_2, Wt %
Total Carbon, Wt %
Org. Carbon, Wt %
Total Hydrogen, Wt %
Org. Hydrogen, Wt %
Ash, Wt %
Moisture, Wt %
Benzene Ext., Wt %

Screen Analysis
$D > 0.742$
$0.185 < D < 0.742$
$0.093 < D < 0.185$
$0.328 < D < 0.093$
Pan, $D < 0.0328$
Analysis of Sample
Fischer Assay
Mineral CO_2, Wt %
Total Carbon, Wt %
Org. Carbon, Wt %
Total Hydrogen, Wt %
Org. Hydrogen, Wt %
Ash, Wt %
Moisture, Wt %
Benzene Ext., Wt %

TABLE 7

THIEF SAMPLES

500 Pound Mass Rate, 4,400 SCF/T Air, 16,000 SCF/T Recycle, 9 Feet Bed Height

B726				B727				B731
P_1	P_2	P_3	P_4	P_1	P_2	P_3	P_4	P_1
42.0		41.9	44.9	5.2	34.0	31.8	0.2	41.7
14.6		33.5	37.8	5.8	33.6	38.0	35.3	18.3
2.8		6.1	4.1	3.1	9.6	5.2	20.7	2.6
4.9		6.6	4.8	10.7	9.7	7.0	21.0	6.2
35.7		11.9	8.4	75.2	13.1	18.0	22.8	31.2
19.4		0.0	0.0	13.5	0.2	0.0	0.0	18.7
16.0		16.8	14.5	13.6	18.9	12.8	9.69	15.9
16.0		6.71	6.44	17.0	8.36	6.09	6.37	15.7
1.37		0.18	0.14	1.17	0.27	0.13	0.13	1.36
70.6		81.6	83.5	72.3	77.9	85.3	88.2	71.4
0.06		0.07	0.4	0.14	0.08	0.08	0.08	0.07
3.51		0.05	0.03	8.02	0.15	0.04	0.05	2.84

B737		B738		B739		B740	
P_1	P_2	P_1	P_2	P_1	P_2	P_1	P_2
42.2	48.9	33.2	28.7	32.0	42.4	34.8	28.1
21.7	25.7	17.9	31.8	27.4	19.4	22.2	18.4
4.4	4.9	3.9	7.3	4.4	6.8	6.0	8.7
8.4	4.8	12.9	14.4	10.4	13.5	14.9	19.2
23.3	12.1	32.1	17.8	35.8	17.9	22.1	25.6
17.4	14.4	14.1	9.2	17.8	15.3	9.8	7.4
16.2	17.5	15.3	17.0	15.7	17.1	14.9	17.6
14.1	13.3	13.1	11.6	14.7	13.7	11.5	11.3
1.22	1.07	1.07	0.84	1.24	1.14	1.09	0.71
73.0	72.5	74.7	75.2	72.5	72.6	77.1	75.5
0.03	0.04	0.04	0.03	0.03	0.01	0.02	0.07
2.21	3.03	1.30	1.87	2.32	2.30	1.30	2.15

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Screen Analysis
D > 0.742
0.185 < D < 0.742
0.093 < D < 0.185
0.0328 < D < 0.093
Pan, D < 0.0328
Analysis of Sample
Fischer Assay
Mineral CO_2, Wt %
Total Carbon, Wt %
Org. Carbon, Wt %
Total Hydrogen, Wt %
Org. Hydrogen, Wt %
Ash, Wt %
Moisture, Wt %
Benzene Ext., Wt %

Screen Analysis
D > 0.742
0.185 < D < 0.742
0.093 < D < 0.185
0.0328 < D < 0.093
Pan, D < 0.0328
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Fischer Assay
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Total Carbon, Wt %
Org. Carbon, Wt %
Total Hydrogen, Wt %
Org. Hydrogen, Wt %
Ash, Wt %
Moisture, Wt %
Benzene Ext., Wt %

TABLE 7 (CONTINUED)

THIEF SAMPLES

500 Pound Mass Rate, 4,400 SCF/T Air, 16,000 SCF/T Recycle, 9 Feet Bed Height

B741 Pretest (2100 hrs)				B741 Pretest (0700 hrs)				B741				B742 Pretest			
P1	P2	P3	P4	P1	P2	P3	P4	P1	P2	P3	P4	P1	P2	P3	P4
46.1	36.9		59.2	33.4	29.7	45.5	36.7		20.6	45.5	38.7	47.7	51.8	37.5	63.4
21.6	21.8		27.1	14.5	24.9	32.4	48.7		26.6	32.4	47.3	28.5	25.0	31.0	21.9
2.8	6.0		4.2	3.0	8.3	5.6	4.8		7.9	5.6	4.6	0.8	2.7	7.2	4.1
5.8	10.9		4.4	8.6	14.5	6.5	3.6		14.6	6.5	4.4	2.5	2.1	9.9	4.6
23.7	24.4		5.1	40.5	22.6	10.0	6.2		30.3	10.0	5.0	20.5	15.4	14.4	6.0
24.5	16.3		8.1	7.0	3.1	2.6	0.0		5.2	2.6	0.0	25.6	27.3	13.1	21.7
14.5	17.2		18.3	16.2	18.1	16.5	18.6		18.3	16.5	14.6	15.4	16.3	18.3	18.6
16.5	14.5		10.9	14.6	12.1	8.81	6.65		10.9	8.81	6.01	16.8	18.0	12.5	15.3
1.57	1.18		0.76	1.17	0.79	0.48	0.15		0.52	0.48	0.18	1.57	1.76	0.95	1.42
70.7	71.4		75.0	77.5	73.9	78.8	79.5		75.1	78.8	82.9	69.5	67.2	72.6	69.2
0.10	0.10		0.08	0.04	0.09	0.04	0.05		0.09	0.04	0.08	0.15	0.11	0.25	0.13
2.18	1.90		0.57	1.77	2.50	0.23	0.01		1.20	0.22	0.01	1.86	2.75	0.07	1.19

B742 Pretest				B742				B743				B744			
P1	P2	P3	P4	P1	P2	P3	P4	P1	P2	P3	P4	P1	P2	P3	P4
42.3	33.9	33.0	45.1	32.2	34.7	40.4	38.6	39.1	34.3	48.4	49.0	41.7	21.9	23.1	34.7
21.3	21.9	33.8	39.2	14.6	26.3	36.5	36.7	21.7	29.8	35.6	31.4	17.3	15.8	37.1	39.3
3.2	6.3	7.2	4.8	3.9	7.6	6.2	7.0	4.0	8.6	5.4	6.7	3.4	5.7	7.9	8.2
6.0	12.9	11.1	4.5	9.2	11.5	7.8	7.1	8.1	11.8	5.1	5.2	8.3	11.5	13.0	8.4
27.2	25.0	14.9	6.4	40.1	19.9	9.1	10.6	27.1	15.5	5.3	7.4	29.3	45.1	21.9	9.4
20.4	12.0	0.0	0.0	13.9	3.9	1.6	0.4	23.5	2.2	0.0	0.0	17.8	6.6	0.4	0.0
15.7	17.0	17.1	15.2	16.2	19.6	19.6	16.2	15.8	18.8	18.4	16.0	16.3	18.8	16.9	18.8
15.7	13.1	7.52	6.57	13.8	10.7	8.69	6.75	17.2	9.66	7.30	6.47	17.2	11.3	7.80	7.38
1.39	0.98	0.25	0.18	1.07	0.55	0.36	0.27	1.59	0.51	0.26	0.15	1.26	0.68	0.26	0.24
71.1	73.5	80.2	82.3	73.2	75.1	77.1	81.6	69.5	76.9	79.8	82.3	71.6	74.4	80.0	78.8
0.01	0.01	0.02	0.04	0.05	0.04	0.03	0.09	0.03	0.09	0.04	0.04	0.05	0.07	0.07	0.06
1.83	2.64	0.07	0.02	1.85	0.75	0.35	0.02	2.05	1.08	0.10	0.09	2.27	1.49	0.05	0.05

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Screen Analysis
$D > 0.742$
$0.185 < D < 0.742$
$0.093 < D < 0.185$
$0.0328 < D < 0.093$
Pan, $D < 0.0328$
Analysis of Sample
Fischer Assay
Mineral CO_2, Wt %
Total Carbon, Wt, %
Org. Carbon, Wt %
Total Hydrogen, Wt %
Org. Hydrogen,Wt %
Ash, Wt %
Moisture, Wt %
Benzene Ext., Wt %

Screen Analysis
$D > 0.742$
$0.185 < D < 0.742$
$0.093 < D < 0.185$
$0.0328 < D < 0.093$
Pan, D 0.0328
Analysis of Sample
Fischer Assay
Mineral CO_2, Wt %
Total Carbon, Wt %
Org. Carbon, Wt %
Total Hydrogen, Wt %
Org. Hydrogen, Wt
Ash, Wt %
Moisture, Wt %
Benzene Ext., Wt %

TABLE 8

THIEF SAMPLES

500 lb/(hr)(ft^2), 5,800 SCF/T Air, 11,500 Recycle

	B749				B750			
	P_1	P2	P_3	P_4	P_1	P_2	P_3	P_4
Screen Analysis								
$D > 0.742$	49.7	28.1	27.8	33.6	48.1	39.3	32.3	22.2
$0.185 < D < 0.742$	35.2	33.5	50.0	44.5	27.4	21.7	37.5	43.1
$0.093 < D < 0.185$	3.4	8.2	7.9	5.3	3.2	5.6	7.1	8.5
$0.328 < D < 0.093$	3.4	12.1	7.2	4.4	5.2	11.3	9.3	8.5
Pan, $D < 0.0328$	8.3	18.1	7.1	12.2	16.1	22.1	13.8	17.7
Analysis of Sample								
Fischer Assay	28.0	8.6	0.0	0.0	11.6	2.7	0.0	0.0
Mineral CO_2, Wt %	16.6	18.4	16.3	17.8	16.2	18.1	16.4	16.8
Total Carbon, Wt %	17.6	12.1	6.88	6.89	15.8	10.8	6.74	7.12
Org. Carbon, Wt %								
Total Hydrogen, Wt %	1.74	0.82	0.25	0.16	1.48	0.68	0.21	0.19
Org. Hydrogen, Wt %								
Ash, Wt %	67.6	73.9	81.0	80.3	70.4	75.2	82.1	80.9
Moisture, Wt %	0.7	0.3	0.2	0.1	0.4	0.2	0.3	0.2
Benzene Ext., Wt. %	2.46	2.83	0.09	0.07	1.73	1.35	0.08	0.07

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c. Saturation of Gas From Vent Purge Condenser

Preliminary data taken on the gas from the vent purge condenser indicate that the gas is saturated with water as it leaves the vent purge condenser. (This assumption has been made up to this time without demonstrating it experimentally.) The relative humidity of the heated vent purge gas was obtained by an Electric Hygrometer-Indicator purchased from Hygrodynamics, Inc. The dew point of the heated purge gas is essentially the same as the temperature of the gas leaving the purge condenser. Additional data will be obtained to confirm this preliminary observation.

d. Sampling of Gas From a Moving Shale Bed, Primarily For Oxygen

A satisfactory procedure for determining the oxygen content of the gas in the retort was discussed in Monthly Progress Memorandum dated September 24, 1965. Essentially, the procedure entails the continuous removal of the gas from the moving shale bed through an air cooled one-fourth inch sample tube to a gas chromatograph. The data indicate that the combustion of oxygen takes place very rapidly. In most cases the oxygen decreases to a low level (0.1%) within a few inches of the air injection point. The detailed data are given in Table 9.

Since the value of the oxygen probe tests appear to be limited and since they take considerable time and effort it is planned to make the tests only in special cases, such as during testing of an entirely new concept in air distributor design.

4. Liquid Product Sampling and Analysis for Water Content

a. Evaluation of DeLaval Gyro-Tester Centrifuge

Before the recent improvements in liquid sampling and analytical procedures, consideration was given to the use of a continuous centrifuge. By removing water from the liquid product streams prior to sampling, errors resulting from sampling techniques could be minimized. Minimum water in laboratory samples would reduce problems associated with sample preparation for determination of percent water.

A high speed DeLaval Gyro-Tester was rented and installed immediately upstream from the T3 product tank. The centrifuge replaced the water decanting vessel which had been giving oil-water inventory problems.

Description of Gyro-Tester - 1/3 HP, 115/230V, Single Phase Motor RPM - 3450, Centrifuge Bowl RPM - 12,000, Bowl Capacity - 5 to 65 gal/hr (depends on solids content and viscosity)

The centrifuge was operated during Runs B745 through B748 on Retort No. 2. Below are data which indicate the general performance of the centrifuge.

CENTRIFUGE TEST RESULTS

Run No.	Entering Liquid Rate lb/hr	Centrifuge Output Rate lb/hr		Percent Water in Oil After Centrifuge	Hours of Test	Hours Centrifuge Operated
		Oil	Water			
B745-T-3-1	106.7	84.2	22.4	0.5	6	6
B745-T-3-2	111.0	82.5	28.5	4.4	6	6
B746-T-3-1	113.8	89.8	28.8	10.7	6	5
B746-T-3-2	101.3	96.8	13.5	18.5	6	2
B747-T-3-1	101.8	78.7	23.2	2.2	6	6
B747-T-3-2	111.0	86.8	29.0	2.1	6	5
B748-T-3-1	156.5	125.5	31.0	0.8	6	6
B748-T-3-2	162.0	130.7	31.3	0.5	6	6

An expected operating problem was encountered. Rapid internal plugging was experienced due to deposition of solids. After 28 hours of continuous operation the centrifuge bowl unit was disassembled. Solids were removed by hand washing in solvent.

Solids buildup in the centrifuge reduced efficiency. Water in the oil discharge was 0.4 percent after initial installation. At the end of the 28 hour run, water was up to 4.4 percent.

Additional work with the centrifuge indicated we could operate it approximately 12 hours before efficiency was severely affected.

While setting up the centrifuge and experimenting to find the optimum discharge nozzle, a heavy sludge was observed leaving with the water stream. Analysis of the sludge is listed below:

Water -	59.7 wt %	
Oil -	35.9 wt %	- (Gravity - 15.5° API)
Sediment -	3.3 wt%%	
Total	98.9%	

On the solids or sediment portion of the sludge various tests were run with the following results:

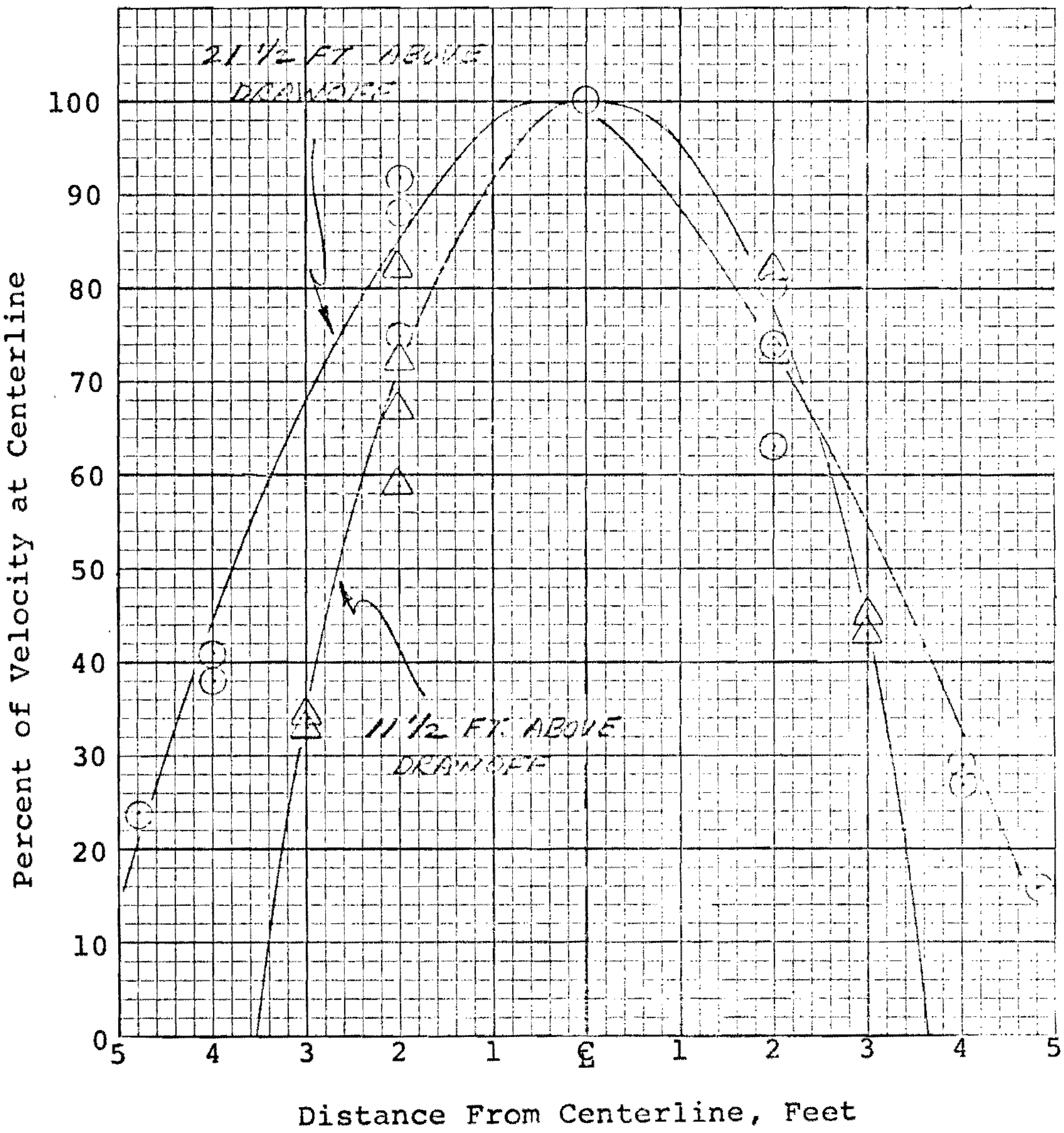
What was the [illegible] ? 15%

Carbon -	64.3 wt %
Hydrogen -	3.58 wt %
Mineral CO_2 -	2.01 wt %
Ash -	12.0 wt %

FIGURE 14

VELOCITY PROFILE

60° Cone
3/4 To 1 1/2 Inch Shale



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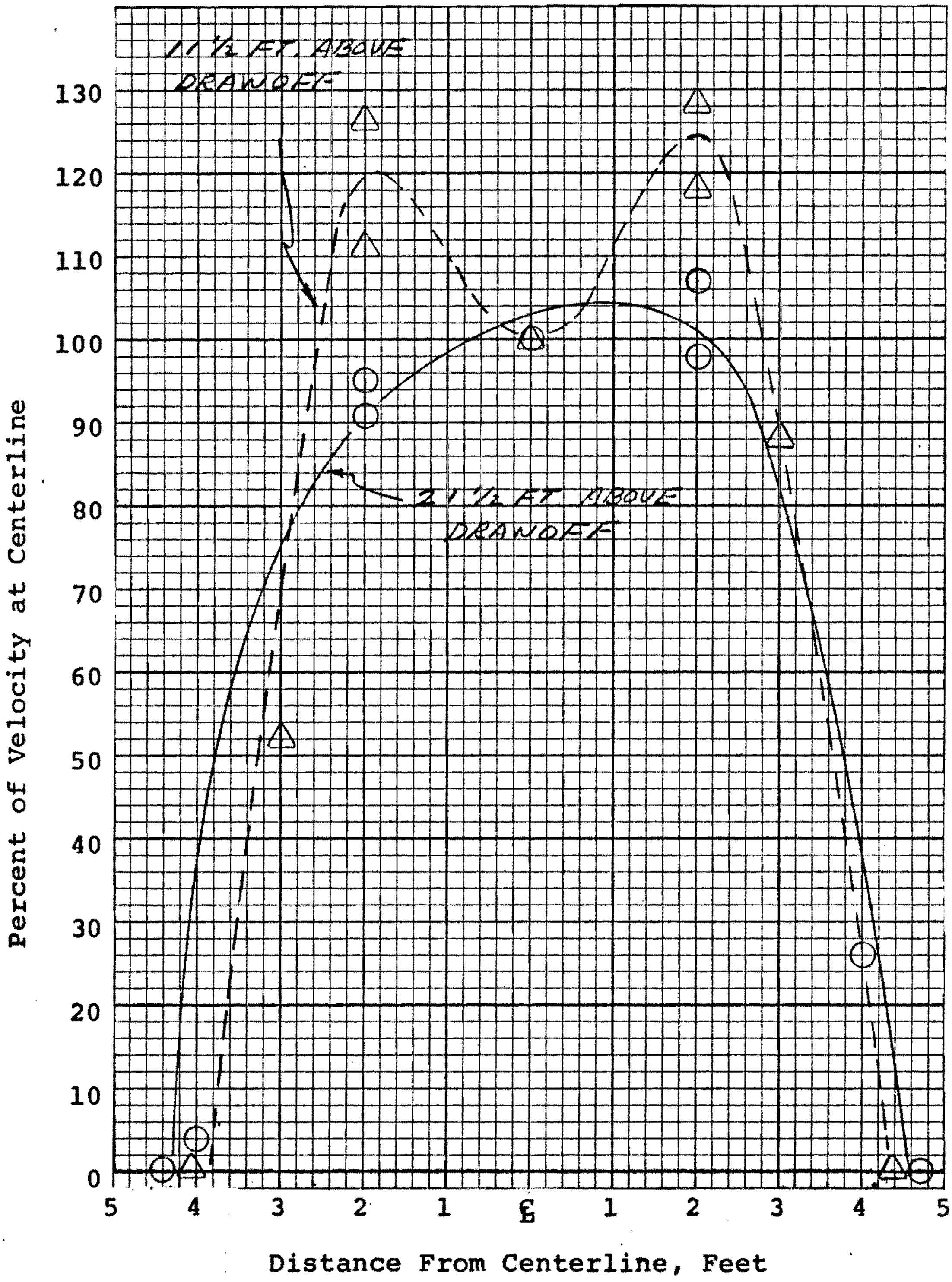
11/3/65

FIGURE 15

VELOCITY PROFILE

60° Cone
1/4 To 3 Inch Shale



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The ash appeared to be shale dust. Carbon content (64.3%) of the sediment was high. The solids had characteristics similar to carbon black.

The principle that water can be separated from the liquid product by centrifuging has been demonstrated. Because of time and cost involved in cleaning, the DeLaval Gyro-Tester was removed from service.

A properly designed centrifuge that is self-cleaning and that requires minimum maintenance could be utilized in a continuous operation. An effort is being made to locate a centrifuge more suited to these operations.

b. Chemical Demulsification of Shale Oil-Water Emulsions

Tretolite, a Division of Petrolite Corporation requested shale oil samples in their letter of September 8, 1965. They were interested in evaluating emulsions because the sale of emulsion treating chemicals is their business.

Five gallon samples from five different runs were submitted to Tretolite. The following excerpt from their letter of November 5, 1965 summarizes their conclusions:

"1. The five samples submitted to us varied in water content from 2-18% and solids content from trace quantities to 3%.

"2. The high concentration of asphaltines require an extremely good aromatic solvent for dilutions to determine water content by centrifuging. Benzene is adequate, while toluene and xylene are not suitable. Aliphatic solvents would be unacceptable.

"3. Due to the variations in each sample efficient dehydration and solids settling required a wide range of conditions. To manage the most difficult sample we used 200°F, four hours of settling time and chemical ratios of 500 ppm. Although the easier samples allowed a reduction in settling time and chemical concentration, the settling temperature of 190-200°F was still required.

"4. We evaluated a wide variety of chemical types on each sample and include the nine compounds with most promising capabilities. Of these nine (RN-2000 to 2008) RN-2004 was universally good, but not the best on any single sample. The others treated in a more specific fashion.

"5. To answer your questions regarding water determinations we compared the ASTM determination procedure with centrifuge samples and found them to be equivalent. The technique employed was similar to oil field practice. We used 50% benzene (vital since it avoids precipitation of asphaltic material), 50% of the wet sample and thoroughly mixed. To this charge was added 3 drops of "Tret-O-Lite" demulsifier F-46 solution and the tube was warmed and agitated gently. The tube was then transferred to a centrifuge and spun for 10 minutes at 2000 rpm. The water content was read (apart from the solids) and doubled to account for the 50% dilution. This figure duplicated the water-by-distillation within the accuracy of either method. The comparisons are lised below.

	ASTM results	Centrifuge Results
Sample A	12.0%, 12.3%	12.0%, 12.5%
Sample B	9.8%, 10.1%	10.0%, 10.2%
Sample C	3.2%, 3.2%	3.2%, 3.4%

Since there are problems with accuracy in either method and since the centrifuge technique is so much simpler and quicker, I would recommend it unless the solids content rises so high that a water layer fails to appear cleanly in the tube. We have included the chemical solution for this determination."

Additional study will be given to the possible use of these chemical demulsifiers.

B. Mechanical Models Group (T. C. Lyons, L. J. Skowronek, P. H. Gifford)

1. Dust Demonstration Model

A description of the Dust Demonstration Model and the initial results were presented in the Progress Memorandum of October 25, 1965. These data revealed that particles smaller than 28 mesh (0.0232 inch) could be suspended in the upper regions of the retort as a result of the gas velocities which are normally encountered in the combustion zone at a mass rate of 500 lb/(hr)(ft^2). Furthermore, it was demonstrated that the presence of an oily layer of shale in the top zone of the retort can suppress the elutriation of very small dust particles from the bed by causing them to agglomerate into larger particles. Recent studies have been made to investigate shale mass rate, shale size, area restriction in the combustion zone, and point of dust injection.

The effect of mass rate on the distribution of dust and fines is shown in Figure 7. At a mass rate of 300 lb/(hr)(ft^2), the particles recovered in the upper zone of the model were generally smaller than 60 mesh (0.0097 inch). This is in contrast to a mass rate of 700 lb/(hr)(ft^2)

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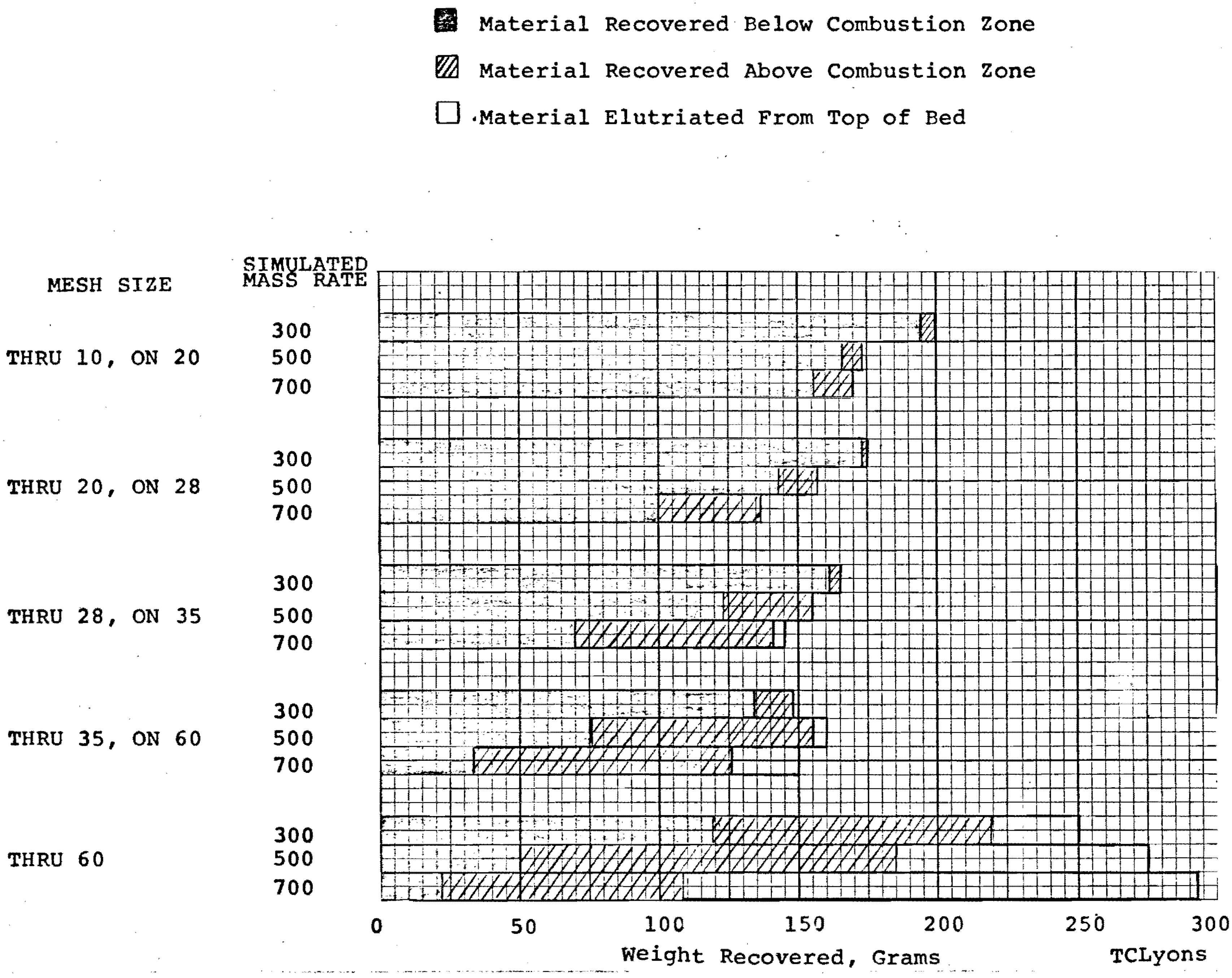
EFFECT OF MASS RATE ON DISTRIBUTION OF DUST AND FINES

3/4 TO 1 1/2 INCH SHALE



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where particles as large as 20 mesh (0.0328 inch) were recovered above the combustion zone in substantial quantities. Thus, this study seems to illustrate why the Bureau of Mines did not become aware of a dust problem at their normal rates. In addition, it demonstrates how operability and yields could be affected adversely by the high dust loading at the 700 mass rate.

Shale size does not appear to affect the distribution of dust to any large extent. The particle distribution obtained with 3/4 to 1 1/2 inch shale is compared with that of 1/4 to 1 inch material in Figure 8.

The effect of restricting the area of the combustion zone was also explored in the model. However, the point at which the dust is injected into the bed with respect to the restriction has a direct bearing on the distribution. Therefore, these two variables must be considered together. Up to this point, the dust has been injected into the bed (with the simulated combustion air) about one inch below the throat of the model. This situation simulates an air distributor which yields the maximum combustion temperatures (highest gas velocities) in the region one to ten inches above the injection point. A second situation is one where the burning takes place in the region around the air distributors and results in high temperatures in the restricted area. In order to simulate this condition, the dust and combustion air were injected directly in the middle of the throat area.

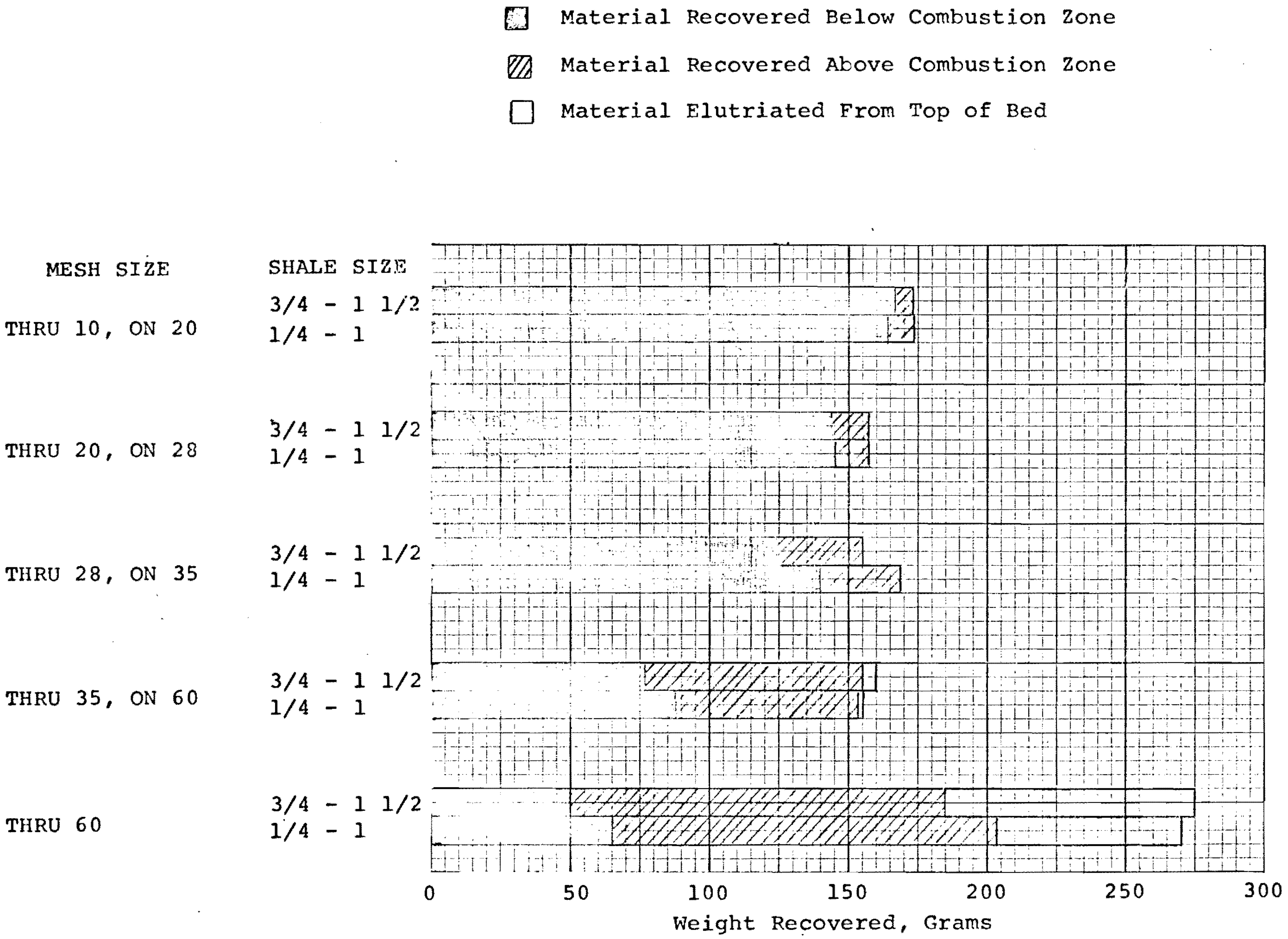
A comparison of the dust distribution resulting from the two different points of injection is shown in Figure 9. These data indicate that injecting the dust directly into the throat area results in slightly higher recovery of the smaller particle sizes in the upper section of the model.

After establishing the effect of the point of dust injection, the next step was to investigate the situation when a portion of the combustion zone area is taken up by the air distributor. (Previously, it was assumed that the distributor hardware did not take up any area). To do this, inserts were placed in the throat section to reduce the cross-sectional area to 85 and 70 percent of normal. These reductions in area had very little effect on the particle distribution when the dust was injected slightly below the restriction. These results are summarized in Figure 10. On the other hand, the distribution was affected when the dust was charged directly into the restricted zone. These data are shown in Figure 11. It should be noted that in the case of the 70 percent open area, the gas velocity or lifting power in the combustion zone at a 500 mass rate is slightly higher than at 700 mass rate and 100 percent open area. Thus, it would be expected that these two

FIGURE 8

EFFECT OF SHALE SIZE ON DISTRIBUTION OF DUST AND FINES

500 LB/(HR)(FT^2)



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10/21/65

FIGURE 9

EFFECT OF LOCATION OF INJECTION POINT ON DISTRIBUTION OF DUST AND FINES

SIMULATION: 500 LB/(HR)(FT^2) WITH 3/4 TO 1 1/2 INCH SHALE

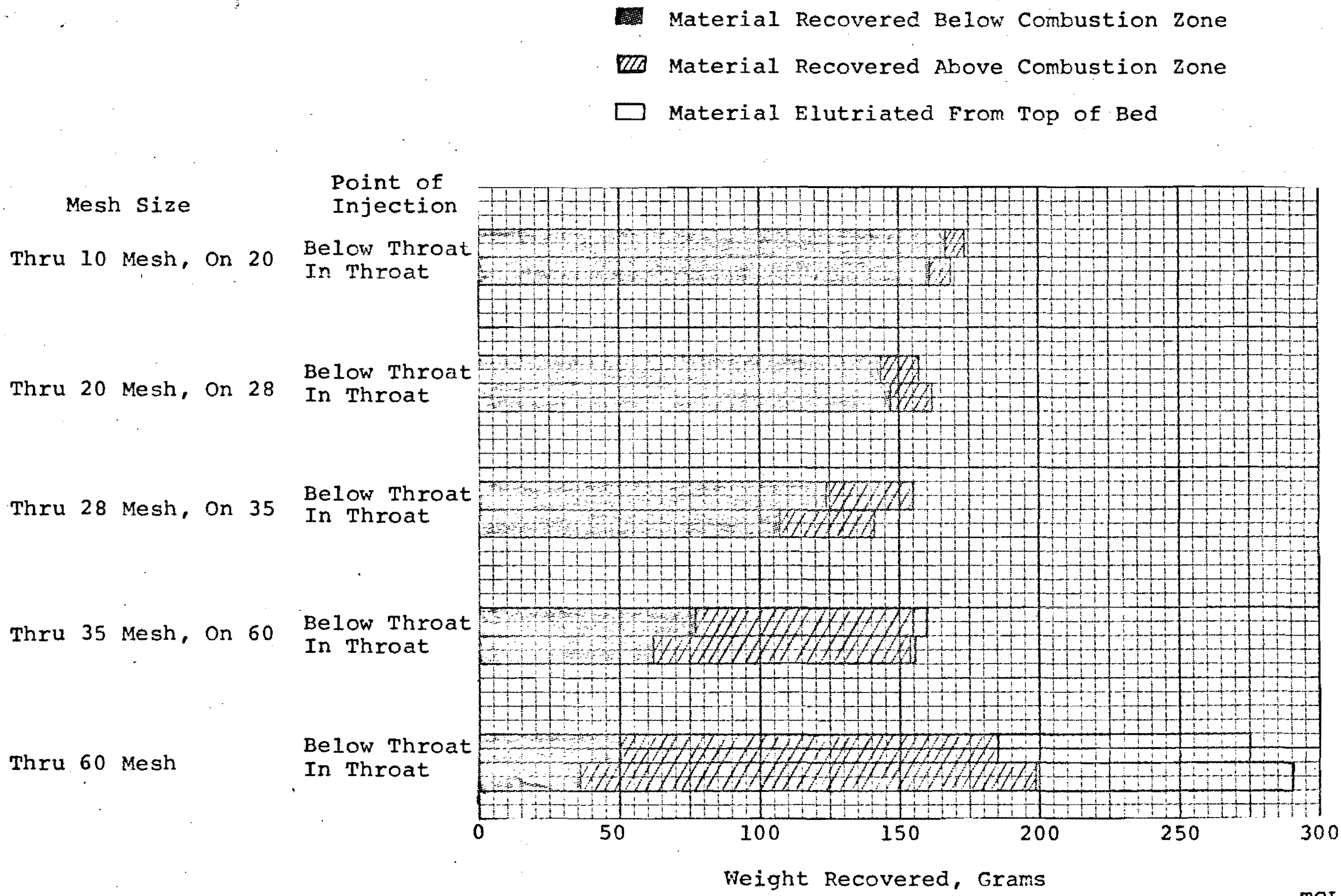

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FIGURE 10

EFFECT OF COMBUSTION ZONE RESTRICTION ON DISTRIBUTION OF DUST AND FINES

SIMULATION: 500 LB/(HR)(FT^2) WITH 3/4 TO 1 1/2 INCH SHALE

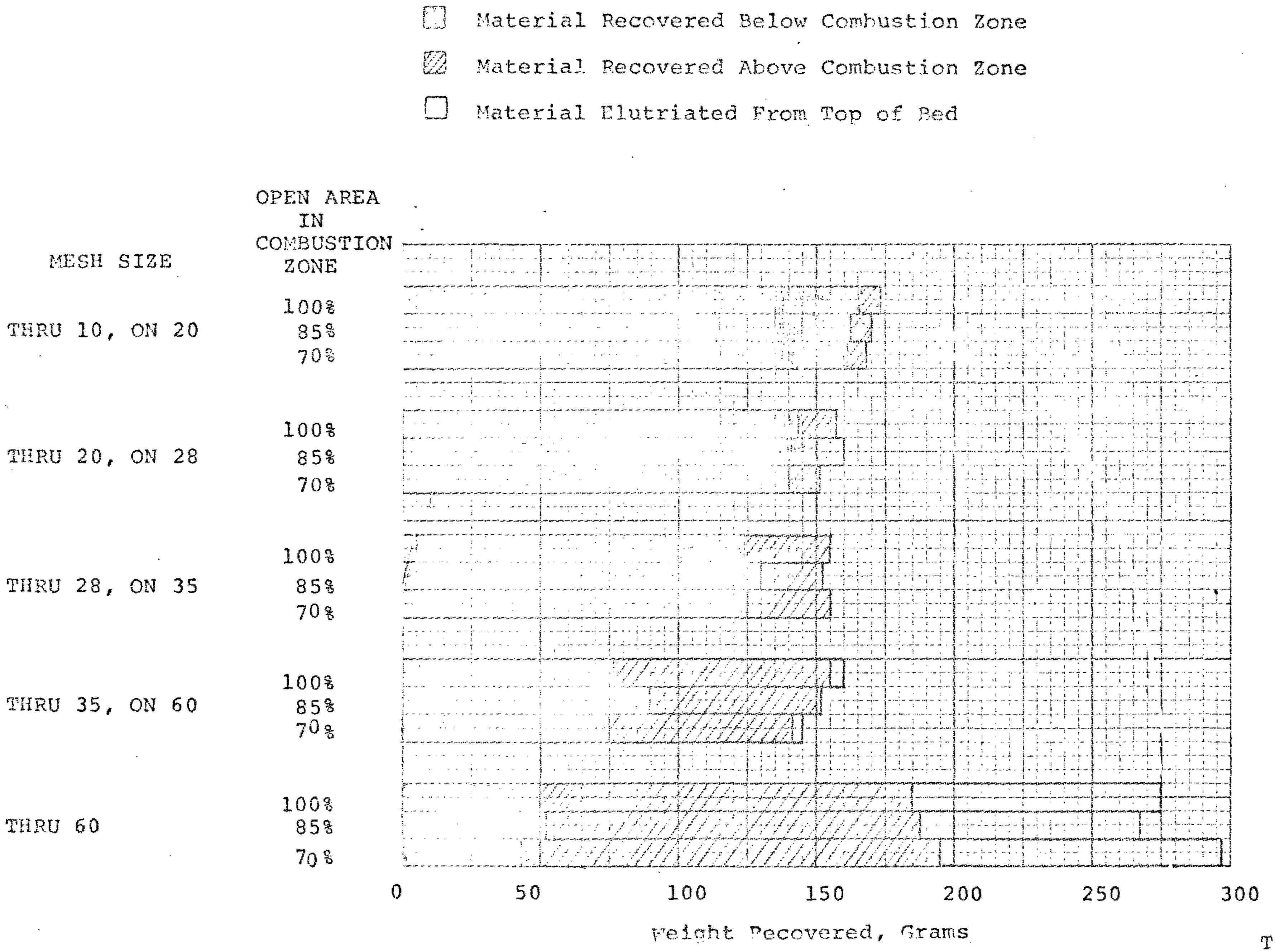

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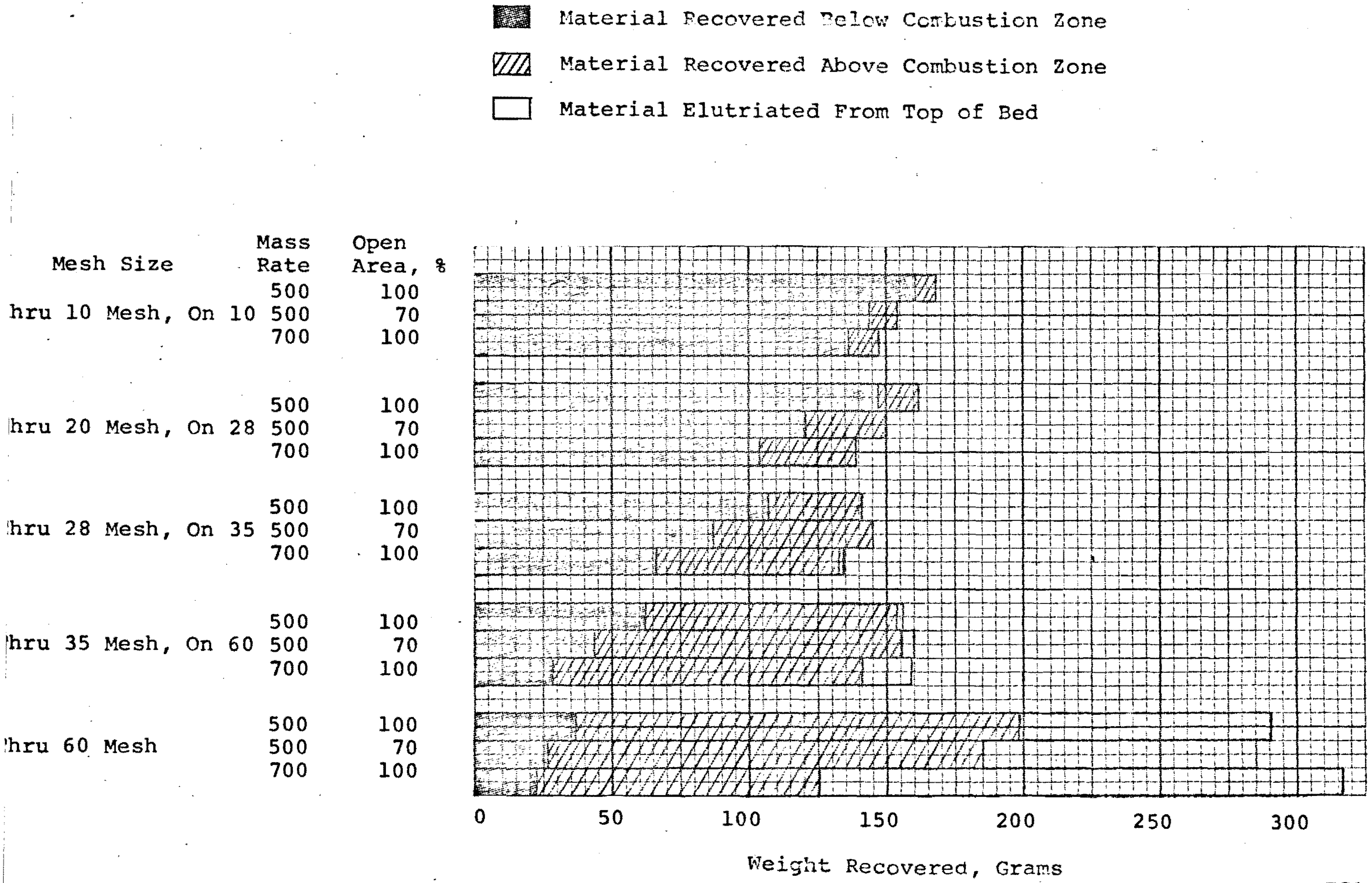
FIGURE 11

EFFECT OF MASS RATE AND COMBUSTION ZONE RESTRACTION ON DISTRIBUTION OF DUST AND FINES

3/4 TO 1 1/2 INCH SHALE

DUST INJECTED DIRECTLY INTO COMBUSTION ZONE



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conditions would give similar dust distributions above and below the combustion zone. The data indicate that the restriction forced more dust into the upper zone at 500 mass rate but it was not as great as that noted at the most 700 mass rate. This difference, although slight, is attributed to a looser bed (higher void fraction and lower gas velocities) as the shale passes through a restriction. This situation is most likely unique to the size of the model and probably would not be a factor if adequate spacing was provided for in a retort.

In any event, these studies show that the area taken up by the air distributor elements is only one factor and must be considered along with the location of the burning zone. The current efforts in the model area are being directed from studies of dust behavior in beds to investigation of dust generation.

2. Mist Simulation Study

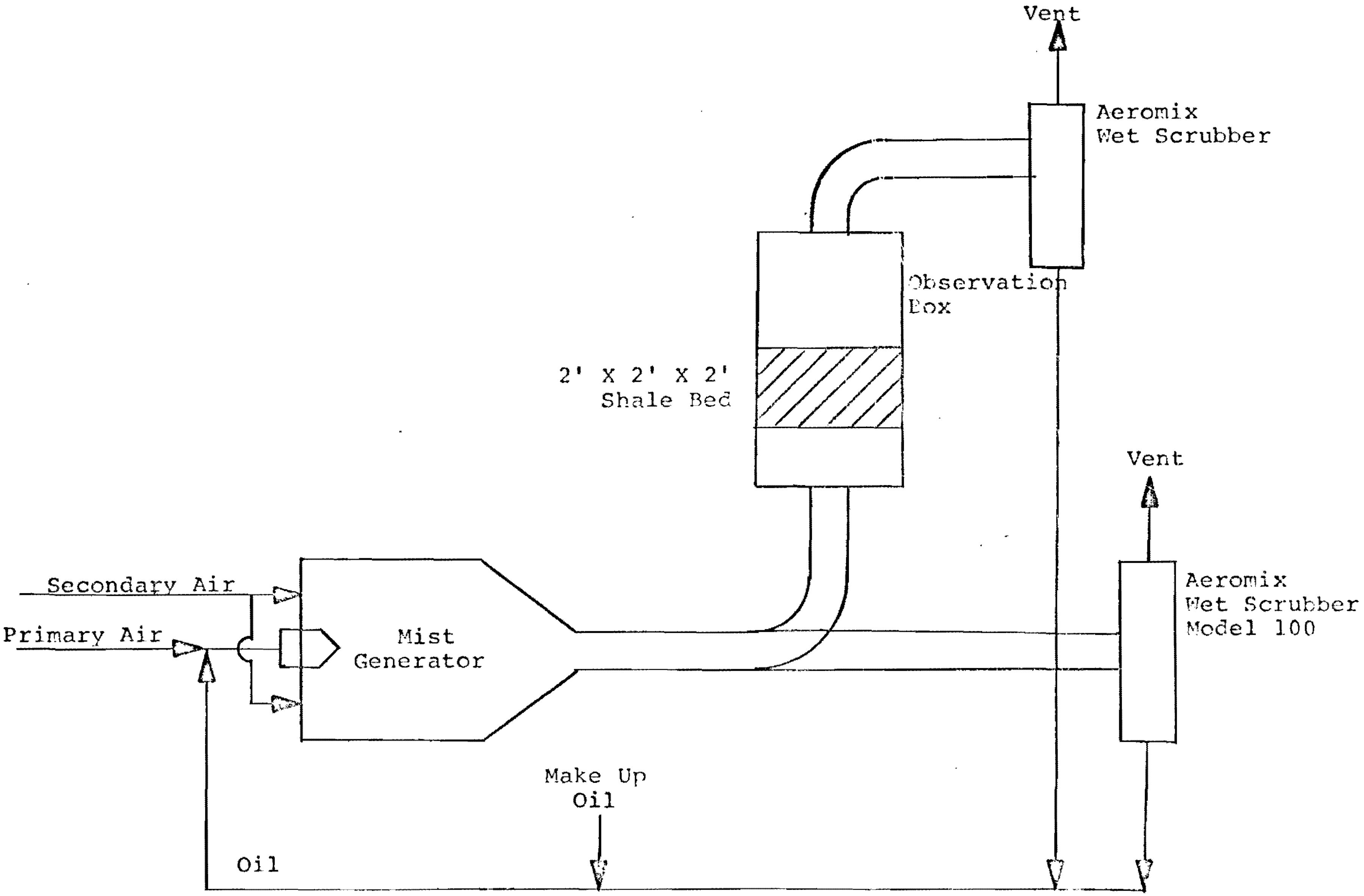
a. Description of Apparatus

The purpose of the mist simulation study is (1)to determine the relationship among the parameters - impaction probability, size distribution, and mist loading in a shale bed, and (2)to evaluate various mist recovery units for possible commercial application.

To attain these goals a model to test mist phenomena is being constructed. The process flow diagram is given in Figure 12. Three basic units are used - (1)the mist generator, (2)the Aeromix Wet Scrubber Model 100, and (3)the observation box. The mist generator is a 31 inch diameter chamber into which a Model 1811 Astrospray nozzle ejects the mist. To the nozzle are fed primary air and transformer oil at rates necessary to produce the desired mean mass diameter. Secondary air is added at the same end of the mist generator unit in order to adjust the mist loading to the desired level. The first mist recovery unit which will be tested is the Aeromix Wet Scrubber Model 100. This is a venturi type scrubber in which the recovered mist is recycled back to the scrubber in order to remove additional mist. A third air line, called tertiary air, has been installed in the system in order to provide variability in the mist generator while at the same time operating the Aeromix at the specified conditions. The third basic unit is the observation box. This box has a cross-sectional area of four square feet with a depth of bed of up to three feet. Two feet of bed depth will normally be used. It also has one side made of lucite so that when a flooding condition is reached it may be observed.

FIGURE 12

PROCESS FLOW DIAGRAM
MIST SIMULATOR



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JH

When studying the impaction of mist on a bed of shale the mist from the generator will be fed to the observation box in which a bed of shale has been placed and from there after mixing with a given amount of tertiary air to the Aeromix Wet Scrubber. When evaluating a certain mist recovery unit, such as the Aeromix, the mist from the generator will be fed directly to the unit. Other mist recovery devices will be evaluated in this manner but usually at much lower mist loadings. The amount of make-up oil required will provide an adequate measure of the efficiency of the unit.

b. Particle Size Distribution

The particle size distribution will in all probability determine to a great extent the efficiency of the various mist recovery units tested as well as the amount of impaction in the bed of raw shale. In order to determine this distribution a Cascade Jet Impactor Model CI-S-6 has been purchased and will be installed in a constant temperature box. The principle by which this unit operates is shown schematically in Figure 13. It will be used extensively throughout all of this work to determine the distribution into and out of either the mist recovery unit being tested or the distribution change when passing through a packed bed.

c. Nozzle Experiments

Before any work can be done on either mist impaction on shale or on the efficiency of mist recovery units a certain amount of exploratory work has to be done in order to characterize the Astrospray nozzle. This will entail determining the relationships between the mean mass diameter which the nozzle produces and the primary air and oil rates. Once this is accomplished the other main studies may be started.

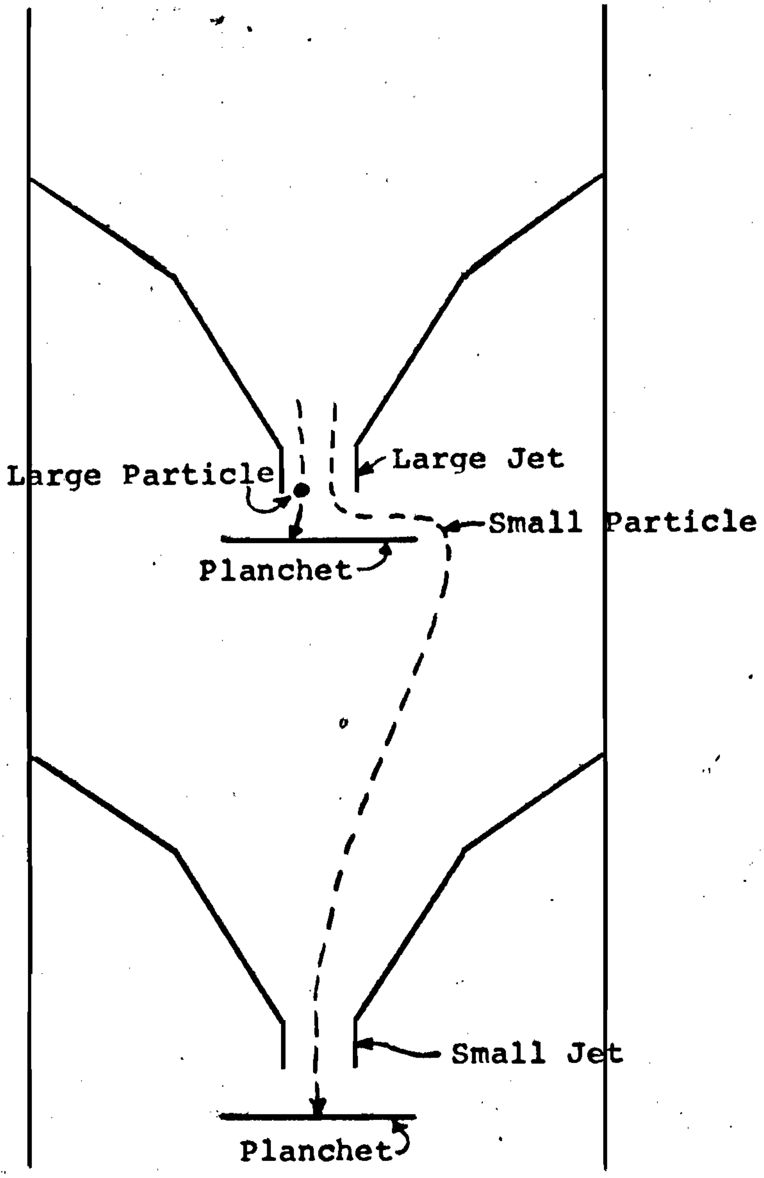
3. Retort No. 2 Studies

During the planning and construction stages of the mist simulator unit some development work has been done on the Cascade Jet Impactor. This work entailed measurement of the particle size distribution of the mist being produced in Retort No. 2. The results of some of these preliminary tests are presented in Table 10.

Early attempts at making this measurement were unsuccessful because the planchets used were too small with respect to holdup. That is, during the test the planchet would fill up and overflow into the next stage. This occurred with a sampling time of 30 seconds. The planchets were redesigned to give additional holdup while at the same time

FIGURE 13

SCHEMATIC DIAGRAM OF CASCADE JET IMPACTOR



PHGifford
11/12/65

JH

TABLE 10

SUMMARY OF RESULTS
RETORT NO. 2 OFFGAS MIST

Run No.	B-743	B-746	B-748P	B-749	B-751
D_{mmd}, μ	1.6	1.56	0.94	0.82	1.01
σ_g	1.65	1.64	1.52	1.54	1.55
Offgas Temp., °F	124	124	118	118	120
Raw Shale, lb/(hr)(ft^2)	493	399	502	493	294
Air, SCF/Ton RS	4,470	4,080	5,700	5,780	4,500
Recycle, SCF/Ton RS	16,000	19,200	11,300	11,400	15,300
Dilution, SCF/T RS	0	0	0	0	1,200
Bed Height, Ft	9	9	9	9	7
ΔP (Above) Inches H_2O/Ft	1.06	0.92	0.73	0.68	0.26
Yield, % FA	87.8	88.4	82.7	83.6	91.0

PHGifford
11/10/65

not appreciably changing any of the critical dimensions. Now with a sampling time of one minute all of the oil is retained.

Wall losses in the impactor were determined on two separate tests. They were found to be 14.4 and 15.6 percent of the total material collected. The corresponding loadings were from eight to nine pounds of oil per MSCF of gas. These losses will be checked periodically.

Future plans on Retort No. 2 entail the measurement of mist loading and particle size distribution at four locations in the mist recovery train. These are before the surge drum, before the low pressure cyclone, before the electrostatic precipitator and after the electrostatic precipitator. This information will be vital in designing an efficient mist recovery system.

4. Jenike Report on Solids Flow

Dr. Andrew Jenike has submitted his report covering the preliminary work that had been agreed to following his consulting visit late in July. This work falls into three parts: (1)Measurement of friction angles, (2)Bench scale models, and (3)Existing storage bins. The pertinent points presented in the report are briefly abstracted herein.

> "The kinematic angle of friction has been measured between raw and spent shale, and three different hopper wall materials.... The kinematic angle of friction is important in determining whether or not mass flow will occur in a given hopper and how steep a hopper made of a given material needs to be for mass flow to prevail. In order to correlate bench-scale observations with flow in full size retorts, the angle of friction was measured for two ranges of particle sizes 1/16 - 1/8, and 1/4 - 3/4 inch. The former is the range used in bench-scale tests, the latter in retorting....
>
> Dr. Jenike continues, "The results are exceptionally simple: the angle of friction is practically the same for both raw and spent shale: it is 20.5 to 23 degrees on stainless steel and plexiglass and [illegible] to 42 degrees on gunite;... The conclusion follows that the flow patterns observed in bench-scale model can be scaled up without any correction into operational retorts. This means that bench-scale models are very useful as design tools."

Dr. Jenike proceeded to recommend designs for two bench-scale models: A rectangular bin and a circular bin. The rectangular bin is 12 inches by 18 inches (42 inches high) whereas the circular bin is 11 1/2 inches I.D.

Concerning the existing 60 ton storage bins, Dr. Jenike concludes,

> "The bin is of the plug-flow type. This flow pattern causes segregation, piping and incomplete discharge, reducing the live capacity of a bin to a fraction of its total capacity.
>
> There appear to be two ways of correcting the situation.
>
> (a) Mass-flow hoppers. An internal stainless steel cone inclined at 20° from the vertical and placed inside the existing hopper will assure mass-flow of shale and will minimize segregation. Such a cone will reduce the total volume of the bin by some 500 cu. ft. but all the remaining volume will then be live.
>
> (b) Conical insert. .... The conical insert certainly is attractive in view of the small loss of capacity (60 cu. ft.). While the undersigned has not had personal experience with such inserts, the theory appears sound and is backed up by Johanson's experiments. In a new installation the undersigned would recommend a steep conical hopper (a) but under the present conditions he favors the insert (b)."

No action will be taken on Dr. Jenike's recommendations until the investigation of the mass flow bin concept is completed in the large scale flow model.

5. Shale Flow Studies in Full-Scale Model

The construction of the model has been completed and flow studies are underway. It will be recalled that the model is 10 feet wide by 20 feet high by 2 feet deep. A bottom sloped at 60° with the horizontal adds an additional eight feet to the vertical height. Continuous flow is achieved by means of a Syntron feeder and an existing bucket elevator. On-site storage is provided for complete inventories of four different shale sizes.

Current efforts are directed toward evaluating the mass-flow bin concept. It should be noted that the proposed design details of this model were discussed with Dr. Jenike during his visit last summer to insure that the model was sized properly for such an evaluation. He assured us that it was.

The initial studies revealed that the flow pattern was not uniform with a bottom sloped at 60° with the horizontal. The velocity profiles obtained with 3/4 to 1 1/2 inch shale at two different levels in the bed are shown in Figure 14. Note that at the 21 1/2 feet level, the velocity of the shale at the wall is roughly 15 percent of that at the center. The flow pattern is even poorer at the 11 1/2 feet level as the bed is static about 3 1/2 feet on either side of the centerline. In these studies, the shale was being circulated continuously and individual shale particles were observed (and timed) through the plastic face.

[illegible]

The flow pattern was also checked in a different manner by observing the top of the bed. In this type of study, the model is loaded with shale and the top is leveled. The bed is then drawn down and the point at which the surface breaks is designated the critical height or the point beyond which the flow is no longer uniform. (In this respect, it is similar to the "calming height" studies that were carried out in pipes and ducts and were reported previously.) The surface of the 3/4 to 1 1/2 inch shale broke immediately indicating the critical height was greater than 28 feet. This was observed at shale throughputs of 1,500 and 500 lb/(hr)(ft^2). (The previous calming height studies also revealed that velocity was not a factor.)

Also? No demonstrated connection between critical height and shale rate, except that they are both > 28 ft but not necessarily by the same amount.

Observations made with the wide range, 1/4 to 3 inch shale and the 60° slope angle led to the same conclusions. The velocity profiles are shown in Figure 15. At the 21 1/2 feet level, the velocity is reasonably uniform in the center four feet of the bed. However, beyond this point the velocity drops off rapidly. A rather unique velocity profile was observed at the 11 1/2 feet level. The reason for this unexpected shape is not known but the prevalence of small particles in the center zone of the model is probably a contributing factor.

A draw-down study confirmed that the flow throughout the entire model was non uniform. The inventory of 1/4 to 3 inch shale limited the initial bed level to 25 feet above the drawoff. The center portion of the bed broke immediately indicating that the critical height was greater than 25 feet.

In summary, the studies to date have revealed that a 60° bottom angle does not provide mass flow of oil shale. The model is currently being modified to evaluate a bottom which is sloped at 70°. Dr. Jenike's work indicates that this should be satisfactory.

70° didn't work either.

6. Shale Flow Movie

A movie has been prepared to supplement Technical Memorandum No. 65-4, "Basic Flow Characteristics of Raw and Spent Oil Shale" which was issued July 9, 1965. Copies are currently being made for each participating party. In addition, the government observer has reviewed the film and has requested a copy.

7. Cameron & Jones Shale Feed System

The Cameron & Jones patent dealing with a feed system was reviewed in light of our segregation studies. This system utilizes a rotating device which feeds a multiplicity of downcomer pipes. The technique appears sound although no physical dimensions were given so that pipe size and spacing could be judged.

V. ANALYTICAL SECTION (D. Liederman and R. Bernheimer)

A. Determination of "Others" in Vent Gas

The application of the Aerograph Gas Chromatograph with flame ionization detection has proven successful. The column for this separation of C_2 through C_5 hydrocarbons is 36 feet of 1/8 stainless steel tubing containing 10 percent dimethylsulfolane on 80 to 100 mesh C_{22} firebrick. The carrier is nitrogen. With this column, ethane and ethylene appear as a single peak. If we find it useful, we can separate these peaks on a second column containing activated silica-gel. Since small amounts of hydrocarbons above C_5 are also separated on the dimethylsulfolane column, we are adding a valve to the chromatograph in order to backflush the heavy components after detection of C_5's.

We are presently calibrating the instrument to determine sensitivities for the various gas components. An analysis of the recycle gas for the C_2 to C_5's should require about 1 1/2 to 2 hours.

B. Comparison of Fischer Assays by Core Laboratories and Anvil Points Laboratory

In order to determine if we can have available a rapid reliable Fischer Assay in case of equipment failure or temporary overload at the Anvil Points Laboratory, we have had analyses of our quality control samples run at Core Laboratories in Rifle. A summary of the results from that laboratory and ours is shown below:

Fischer Assay Quality Control Sample	Anvil Points			Core Lab		
	Oil, G/T (Average)	No. of Detn.	Standard Deviation	Oil, G/T Average	No. of Detns.	Standard Deviation
1	29.6	16	0.35	30.0	4	0.74
2	28.0	15	0.25	27.7	4	0.03
3	29.1	11	0.27	29.2	4	0.46

The only unacceptable data from the Core Labs were those for Control Sample No. 1. This sample, analyzed in two sets of duplicates on separate days, gave 30.8 and 30.8 for one set of duplicates and 29.0 and 29.4 for the other set. This was not a result only of the samples being analyzed on different days, since Control Sample No. 2 was analyzed also, in duplicate, on two different days, and its results are excellent. The Core Labs analyses for Control Sample No. 3 were all made on the same day. Results for the Anvil Points Laboratory were from single analyses by different analysts on different days.

Standard deviations were calculated, for simplicity, assuming single analyses by both laboratories.

We can conclude from the data that Core Labs is generally precise enough to act as an emergency source of analyses, but that it is out of control enough to suspect any single result.

C. Cooperative Program With Laramie For Sampling and Fischer Assay

We are currently engaged in a program in which our laboratory and the Bureau of Mines Laboratory at Laramie will both sample and analyze for Fischer Assay oil, three bags of raw shale. This procedure will check not only our analytical procedures as we have done in the past, but will show whether our sampling procedures, also, are equivalent.

D. Carbon Balance

In cooperation with the Retorting Section, we are working on several investigations to help close the carbon balance.

1. Spent Shale Dust

Some time ago, it was propounded that possibly the spent shale dust (which is lost) contained more carbon than the spent shale. If this were so, this would account for some of the carbon-balance discrepancy. Only recently, have we been able to obtain good spent shale dust samples from the Rotoclone stack. Results comparing spent shale and spent shale dust from two recent runs are shown below:

		Weight Percent			
Run No.	Material	C	H	Ash	CO_2
B748	Spent Shale	5.41	0.10	86.3	12.6
	Shale Dust	9.56	0.64	82.7	--
B751	Spent Shale	6.16	0.18	83.9	14.2
	Shale Dust	7.45	0.16	83.4	14.4

Although these data are preliminary, they do indicate one area of possible carbon-balance error.

R. Clampitt, who took the dust samples, suggests that a dust loss of approximately 60 lb/hr is not unreasonable. At an average carbon difference of about three percent, we might account for about 0.5 percent in carbon-balance. Work on this problem is continuing.

How about
the accumulation of
coke fines in the
retort?

This could be eliminated if they ran long enough to
(be sure of steady state condition)

2. Unmeasured Carbon in Vent Gas

It is apparent that our gas samples are not always representative of the vent gas since we sometimes observe a slight discoloration in the tubing leading to our gas holders at the Laboratory, and in the drying tube attached to the CEC process chromatograph in the Retort Building. We are presently doing work to determine the amount of what, we presume, is a very fine oil mist in the vent gas.

3. Differences in the Amounts of Moisture on the Raw Shale Weighed at the Bin and on the Lab Sample

Any difference in moisture content of the raw shale feed and the crushed sample delivered to the Laboratory will show up as a discrepancy in carbon balance. This difference in moisture will affect yield directly and carbon balance in almost direct ratio. Presently, we are determining moisture on some samples from the weighing area, so that we can compare these with the normal sample moisture contents. (An interesting phenomenon, that we have noticed before, was seen again when we dried the large pieces of shale from the Syntron at 220° F. At this low temperature, several pieces of shale exuded a substance that looked and smelled like shale oil. It is probably low-melting bituminous material.)

E. Raw Shale Density Distribution

Because our normal Fischer Assay merely assigns an average value to the raw shale entering the retort and does not indicate the shale richness distribution, a limited program was initiated to determine this parameter. It may be possible to relate this distribution to the operability of the retort, and perhaps also to the yields obtained.

The Bureau of Mines has already shown that shales of varying richnesses have different specific gravities. This work was based on different core hole samples and showed a smooth curve (but scattered) relationship for the yield versus specific gravity data; as expected, there was sometimes considerable scatter in the data.

Socony Mobil has a procedure for determining the density distribution of catalyst beads which consists of placing the beads in successively less dense liquids and measuring the amount of beads which sinks in each liquid.

We decided on a simple system which was based on the above principle. If we place a small sample (ca 10 g) of ground shales in a liquid of a specified density, the particles will either sink or float. As a first approximation, we assumed that the "degree of packing" at both the top and bottom of the column of liquid were equal; this is reasonable if we assume a normal distribution with no discontinuties.

The actual experiments were conducted in glass stoppered graduate cylinders (50 ml). Ten gram portions of each sample are placed in each of a series of 5 to 7 cylinders, along with an appropriate solvent mixture; we used the same solvents as Socony Mobil (1, 1, 2, 2 - tetrabromoethane (TBE) and carbon tetrachloride) in ratios varying from 100% CCl_4 to 40% CCL_4 - 60 percent TBE. The solvents were added to the 50 ml mark on the graduate. The latter was stoppered and vigorously shaken for about 30 seconds, then the graduates' contents were allowed to settle (several hours). The upper and lower levels of the solid layers were read and recorded. The graduates were shaken again and the process repeated several times. A summary of data is presented in Table 11.

The data indicate, at a glance, the large difference in distribution of raw shale when the Fischer Assay varies by a large amount. The data headed by percent TOP and percent BOTTOM represent an alternate method of presenting the data; they show the fraction of the total that these volumes represent.

We are currently investigating the richness distribution of raw shales from Runs 509, 710 and 725 to determine any difference among these retort samples. We will also help to correlate the shale richness distribution data to retort operations. We must also investigate the following parameters if we are to understand the method more completely: particle size, viscosity of liquids, use of liquids and container diameter

F. Gas Blending

The difficulties in working with copper tubing and fittings, mentioned in our last progress memorandum were circumvented by the use of a glass and rubber tubing system. We now have a system which will hold a good vacuum, and thus allow us to make accurate gas blends. We will use these blends to check our Matheson standards in the near future.

G. Emission Spectroscopy of Shale Samples

In order to ascertain whether there were any gross differences in metals content of shale and clinkers, we submitted samples of these materials to Esso Research and Engineering for qualitative emission spectroscopy. The results of these analyses are shown below:

TABLE 11

SUMMARY OF RAW SHALE DENSITY DISTRIBUTION

Fischer Assay gal/ton	Liquid (% CCl_4)(a)	Volume of Solids			
		Top±A.D.	Bottom±A.D.	% to P±A.D.	% Bottom±A.D.
46.4(b)(d)(e)	100	4.8±0.3	77.0±2.0	5.8±0.4	94.2±0.4
	95	9.8±2.3	66.0±7.0	13.1±3.8	86.9±3.8
	90	28.5±4.5	54.5±3.5	32.7±6.7	67.3±6.7
	85	51.0±3.0	40.0±7.0	57.3±6.8	42.7±6.8
	80	76.0±1.0	18.0±6.0	81.3±5.0	18.7±5.0
	70	81.3±3.3	0.1±0.1	99.9±0.1	0.1±0.1
27.0(c)(d)(f)	100	0.0±0.0	15.3±1.5	0.0±0.0	100.0±0.0
	90	0.1±0.0	16.8±1.1	0.1±0.0	99.9±0.0
	80	1.5±0.2	16.4±1.4	8.6±1.6	91.4±1.6
	70	4.9±0.2	13.1±1.0	27.5±2.4	72.5±2.4
	60	23.4±0.6	11.1±1.5	68.2±3.0	31.8±3.0
	50	29.5±0.8	4.9±0.3	85.8±0.6	14.3±0.6
	40	31.3±0.9	0.5±0.3	98.4±1.0	1.6±1.0

A.D. Average Deviation

(a) Remainder - tetrabromomethane

(b) Data measured as height in mm

(c) Data measured as volume in ml; last three graduates (60, 50 and 40% CCl_4) were 100 ml, and 20 g sample was used.

(d) 10 to 28 mesh sample used

(e) Average of two determinations

(f) Average of six determinations

RBernheimer
11/15/65

	Metals Content			
Sample	Major (10-100%)	Minor (1-10%)	Trace (0.1-1.0%)	Present (0.01-0.1%)
1. Clinker from B718 pretest	Si, Mg, Al, Ca	Fe, Na	Ti	B, Cr, Sr, K, Mn, Co, Ni, Cu, V, Mo, Pb.
2. Material scraped from sides of retort, Run 708	Si, Mg, Al, Ca	Fe, Na	Ti, Pb	B, Cr, Sr, K, Mn, Co, Ni, Cu, V, Mo.
3. Fines from SW corner after Run B702	Si, Mg, Al, Ca	Fe, Na	Ti, Pb	B, Cr, Sr, K, Mn, Co, Ni, Cu, V, Mo.
4. Solids from SW corner after B702	Si, Mg, Al, Ca	Fe, Na	Ti	B, Cr, Sr, K, Mn, Co, Ni, Cu, V, Mo, Pb.
5. Spent shale, B725	Si, Mg, Al, Ca	Fe, Na	Ti	B, Cr, Sr, K, Mn, Co, Ni, Cu, V, Mo, Pb.
6. Spent shale, 513	Si, Mg, Al, Ca	Fe, Na Na	Ti	B, Cr, Sr, K, Mn, Co, Ni, Cu, V, Mo, Pb.
7. Raw Shale, 588	Si, Mg, Ca	Al, Fe, Na	P, Pb V, K	B, Ni, Mo, Cu, Zn, Ti, Mn, Sr, Cr
8. Raw Shale, B720	Si, Mg, Ca	Al, Fe, Na	P, Pb, V, K	B, Sr, Ni, Mo, Cu, Zn, Ti, Mn, Sr, Cr.

The results are not completely consistent. The raw shales contain less Al, Ti and more Pb, P, V and K than the spent shales. Very small amounts of Zn and, in one case, Sr (No. 8), are also found in the raw shales, but not in the spent shales. All the clinkers appear identical and similar to the spent shales, except samples 2 and 3; they show more Pb than the other samples. At this time, we do not know whether this is the result of natural variations in the shale or just part of the experimental error inherent in qualitative emission spectroscopy analysis. The results indicate no radical differences in metals content of clinkers, spent shale, or raw shale. No further work along these lines is planned at this time.

H. Work Planned for the Near Future

Several investigations are being planned for the near future:

1. Coking Tendency of Shale Oil on Shale Dust

   Experiments in which we distill shale oil at low pressure in the presence of shale dust will show the tendency of coke formation in the retort. We will examine the coke agglomerate formed to determine physical and chemical properties.

2. Some centrifugation tests will be made with the DeLaval Gyro-Test Unit and Petrolite Corporation's emulsion breakers to see whether we can get reliable estimates of water-and-oil at higher "G" forces.

3. Investigation of more rapid ashing at higher temperatures will be made in an attempt to supply faster "quick-yield" data.

Note: The September 16 to October 15 Progress Memorandum contained an error on page 23, Section C1: It should read, "... limits (1 degree API corresponds..."

VI. ENGINEERING AND ECONOMIC ANALYSES (P. W. Snyder and J. E. Burchfield)

A. Pressure Drop in Retorts No. 1 and No. 2

1. Variation With Feed Particle Size - Retort No. 1

Last month's progress memorandum showed that pressure drop in Retort No. 1 was a function of gas rate to about the fourth power. It was also found the pressure drop was inversely related to shale mass rate. The data presented were for 3/4 to 1 1/2 inch particle size. Since Pressure drop is also a function of particle size, data for this factor have also been investigated.

Pressure drop data for 1/4 to 3/4 inch particles (Runs 588 through 600) have been plotted in Figure 16 along with data for 3/4 to 1 1/2 inch particles (Runs 552 through 576). All data are for 600 lb/(hr)(ft^2) mass rate except the one noted on the plot.

Although the pressure drops for the 1/4 to 3/4 inch particles are somewhat scattered they are generally about 35 percent higher than those for 3/4 to 1 1/2 inch particles. The pressure drop for the 1/4 to 3/4 inch particles, while higher than for the 3/4 to 1 1/2 inch particles, is nevertheless lower than expected. In packed beds, pressure drop is expected to be inversely related to the first to second power of particle size. Hence the large difference in particle size between the two sets of data should result in a much larger difference in pressure.

These data probably indicate then that the raw shale particle size does not represent the particle size in the bed. Dust in the combustion and retorting zones of the retort apparently have significant effect on bed particle size. Since raw shale particle size does not adequately represent the bed particle size, changes in feed size do not properly reflect change in pressure drop.

The parameter of pressure drop with mass rate seen for the larger particles may also be noted in the single point at 500 lb/(hr)(ft^2) mass rate. This lower mass rate point appears to have a higher intercept than the other data at 600 lb/(hr)(ft^2).

2. Retort No. 2 Pressure Drop

Data for Runs B676 through B700 and B718 through B733 on Retort No. 2 have been plotted in Figure 17. A dotted line representing the pressure drop on Retort No. 1 at 600 lb/(hr)(ft^2) has been plotted on the figure also for comparison with Retort No. 2 data.

FIGURE 16

PRESSURE DROP AS A FUNCTION OF GAS RATE AND PARTICLE SIZE - NO. 1 RETORT

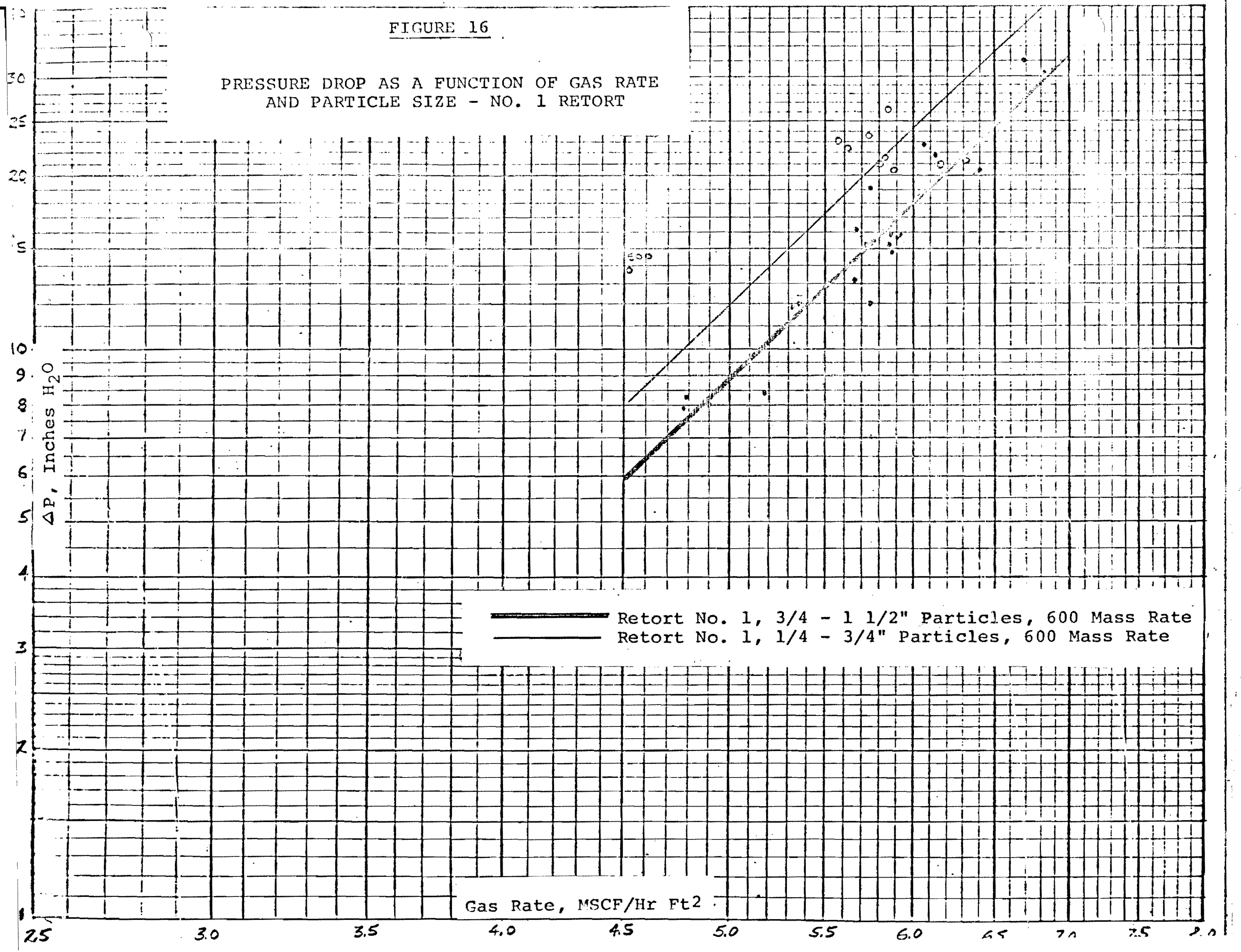
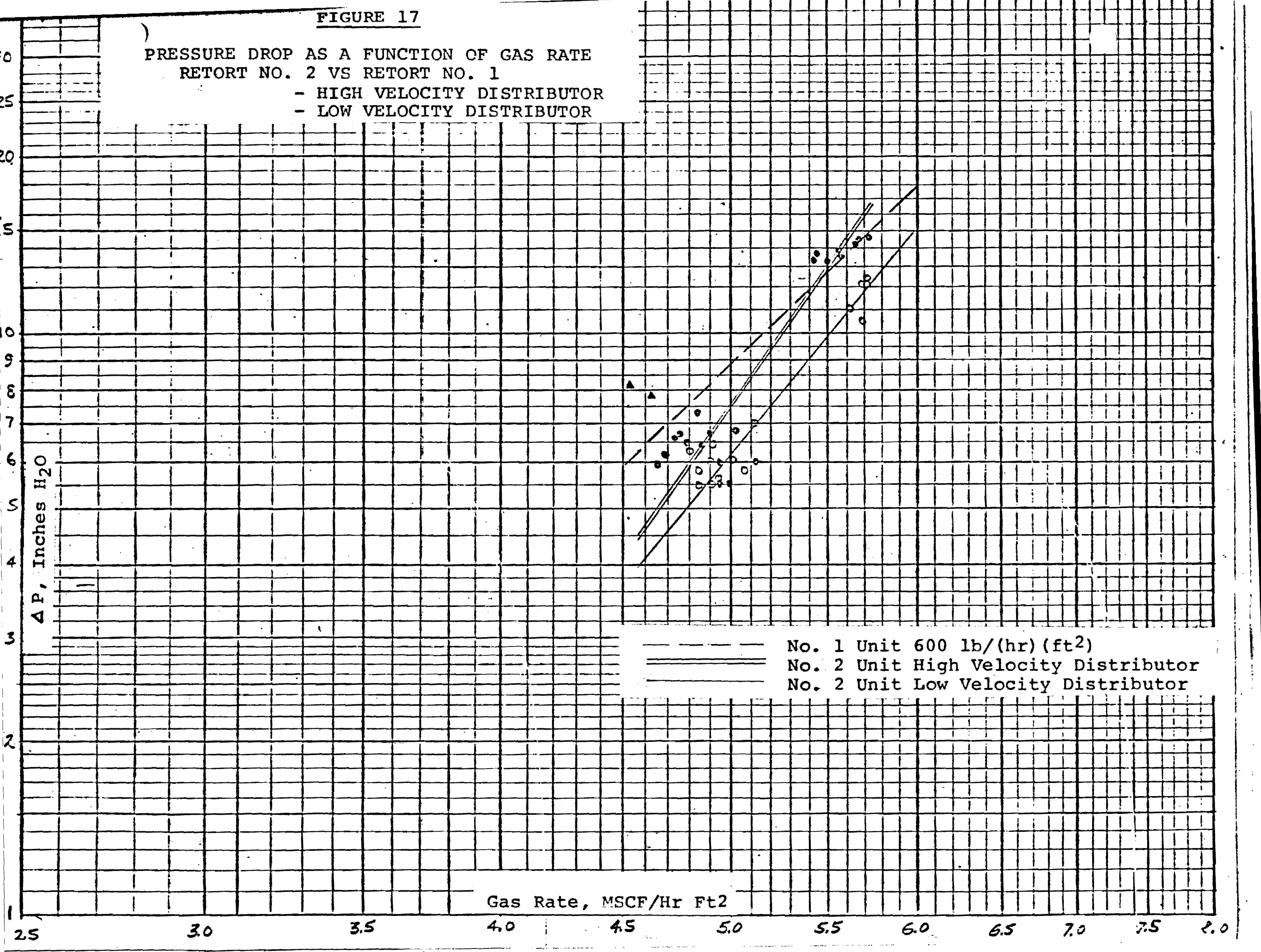


FIGURE 17

PRESSURE DROP AS A FUNCTION OF GAS RATE
RETORT NO. 2 VS RETORT NO. 1
- HIGH VELOCITY DISTRIBUTOR
- LOW VELOCITY DISTRIBUTOR



These data for Retort No. 2 are at about the same level or somewhat lower level than the data for Retort No. 1. However mass rate for the Retort No. 2 data is only 500 lb/(hr)(ft^2) whereas the Retort No. 1 line data were 600 lb/(hr)(ft^2). Had Retort No. 1 data been at 500 lb/(hr)(ft^2) mass rate, the pressure drop presumably would have been higher as shown by the regression analyses of Retort No. 1 data. Thus Retort No. 2 seems to give slightly lower pressure drops than Retort No. 1 for equivalent particle sizes, gas rates and mass rate. The slope also appears to be slightly greater in Retort No. 2.

The reason for the lower pressure drop in Retort No. 2 if it exists is not clear. It could be an indication of void or lower density zones in Retort No. 2 which is tied by time-temperature relationships to lower yields. It could also indicate a lower dust concentration in Retort No. 2, but this is difficult to reconcile with the somewhat lower yields in that unit.

The solid points in Figure 17 for Runs B676 through B700 represent data for the high velocity air distributor. The small circles for Runs B718 through B733 represent data for the low velocity slot distributor. The bed pressure drop appears to be lower for the lower velocity distributor. This lower pressure drop is believed to be the result of less dust formation at the air distributor.

Finally, the parameter of mass rate is again observed. Two runs on the high velocity distributor were made at 400 lb/(hr)(ft^2) and are noticeably higher in pressure drop than 500 lb/(hr)(ft^2) mass rate data at nearly the same gas rate.

B. Math Model Studies of the Effect of Recycle Rate

Math model runs at 12,000, 16,000 and 19,000 SCF/T of recycle have been compared to gain understanding of the effect of recycle rate. The temperature profile comparison is shown in Figure 18. The kerogen decomposition and oxygen consumption profiles are shown in Figure 19. The conclusions drawn from these comparisons are that increasing the recycle rate:

1. Increases the retorting residence time in the desired temperature range, i.e. more kerogen is decomposed at lower temperatures where there is less opportunity for cracking.

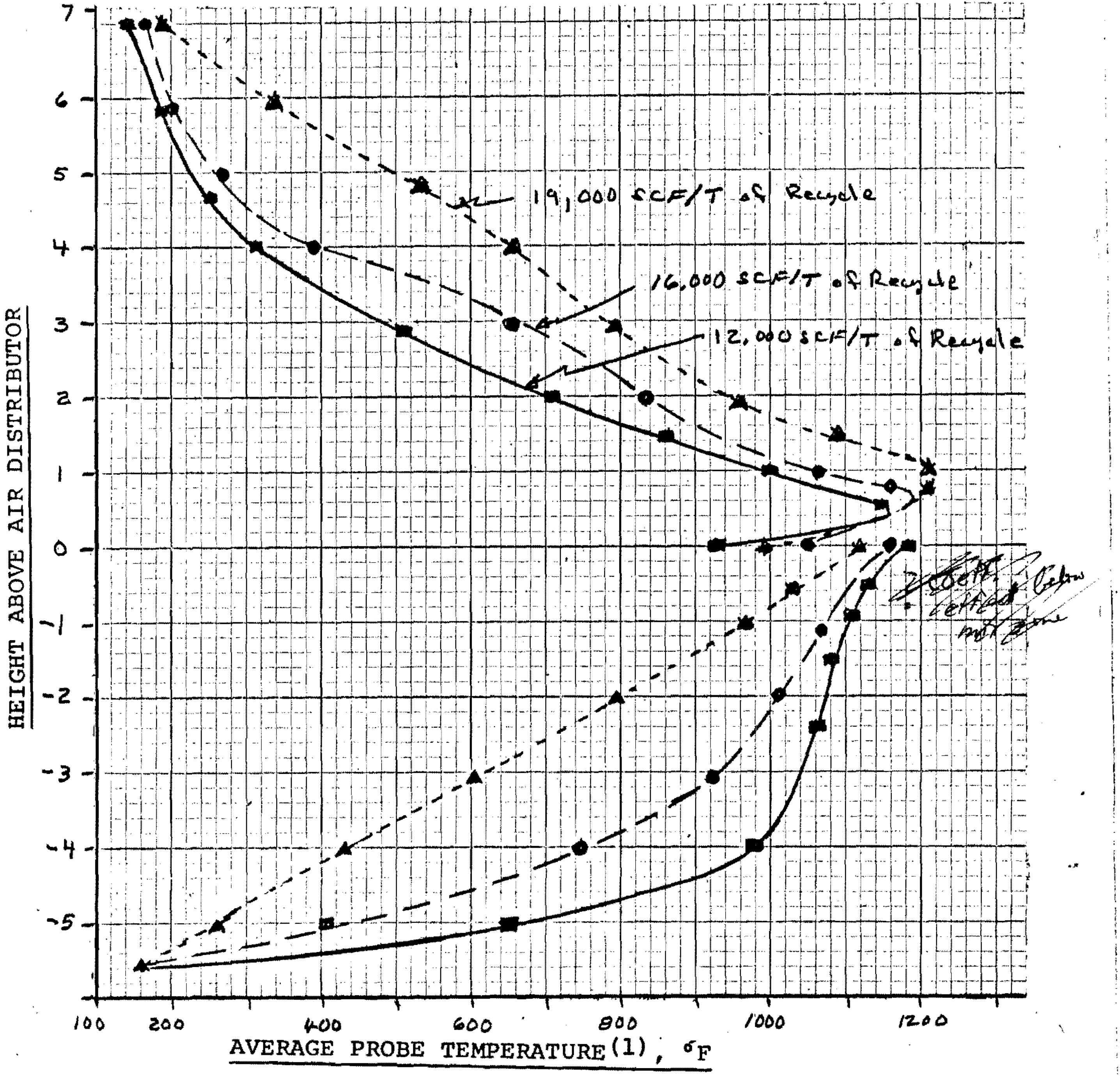
2. Moves the retorting zone further up into the bed reducing the opportunity of burning oil by reducing the overlap between the combustion and retorting zones.

FIGURE 18

MATH MODEL ANALYSES OF THE EFFECT OF RECYCLE GAS RATE

?

Operating Conditions:			
Recycle Rate, SCF/T	12,000	16,000	19,000
Shale Rate, lb/(hr)(ft^2)	500	500	500
Air Rate, SCF/T	5,300	3,900	4,000
Results:			
Kerogen Decomposed at 950° F shale Temp., %	75	83	86
Carbonate Decomposed, %	19	17	19
Retorting Residence Time Mins. (800-1,000° F Gas Temp)	5	8	9
Gas Temperature Profile:			
Symbol	■	●	▲



(1) Gas Temperature

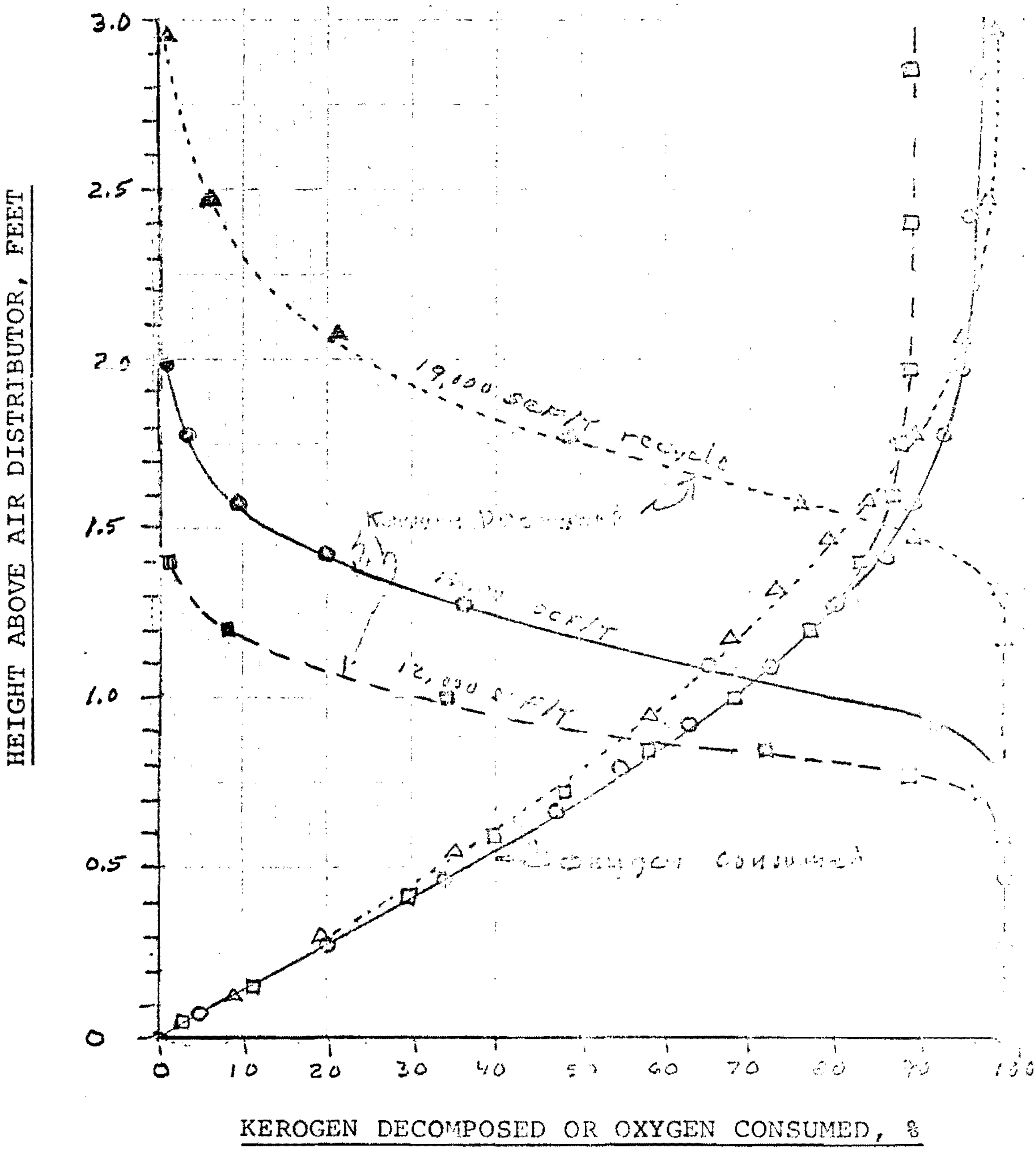
PWSnyder
11/15/65

FIGURE 19

MATH MODEL KEROGEN DECOMPOSITION AND OXYGEN CONSUMPTION PROFILES

Operating Conditions:

Recycle Rate, SCF/T	12,000	16,000	19,000
Shale Rate, lb/(hr)(ft^2)	500	500	500
Air Rate, SCF/T	5,300	3,900	4,000
Symbol	□	○	△



PWSnyder
11/16/65

3. Does not greatly change the combustion characteristics; combustion is 10 percent more complete at the higher recycle because the combustion temperatures are not quenched as rapidly as at the lower recycle rate.

4. Raises the offgas temperature. so what?

5. Reduces the quenching effect at the air inlet which should improve stability. - it could be predicted

   OK - so what?

6. Shifts the shale cooling profile to a more linear cooling rate and reduces spent shale temperature. No

As shown in Figure 20 the measure temperature profile for 16,000 SCF/T of recycle differ somewhat from the math model profile at this recycle. The measured profile is tending toward the refluxing type of profile also shown on Figure 20; therefore there is probably some oil-dust refluxing taking place in the actual pilot run at 16,000/SCF/T.

C. Shale Oil Condensation Temperatures Within the Retort

The Edmister phase diagram for predicting condensation temperatures at subatmospheric conditions has been calculated from the ASTM D 1160 distillation for a typical Gas-Combustion shale oil. This diagram is useful for estimating the condensation curve at the low partial pressures which exist in the retort. The resulting phase diagram is shown on Figure 21. At the partial pressure which exists in the retort, about 10 mm Hg, over three-fourths of the oil will be condensed below 650° F, but the first mist particle will be formed at about 900° F. Oil refluxing should cause changes in the temperature profile in the region of 300° to 650° F where most of the heat of condensation will be released. The "bubble point" line of the phase diagram shows why very little liquid boiling below 300° F (the IBP of typical shale oil) is obtained; since at the retort partial pressure, the bubble point for this 300° F IBP oil is about 140° F - typical offgas temperature. A few vapor-liquid equilibrium calculations will be made using vapor-liquid equilibrium "K"'s for the individual components and 100° F cuts for the shale oil as an independent check of the Edmister phase diagram.

FIGURE 20

MEASURED TEMPERATURE PROFILES VERSUS CALCULATED PROFILES

Operating Conditions:	Math Model		Pilot Retort No. 2 Runs 725 - 727
Oil Refluxing Ratio	None	7/1	--
Shale Rate, lb/(hr)(ft^2)	500	500	500
Recycle Rate, SCF/T	16,000	12,000	16,000
Air Rate, SCF/T	3,900	5,600	4,300
Gas Temperature Profile: Symbol	- - -	— —	●

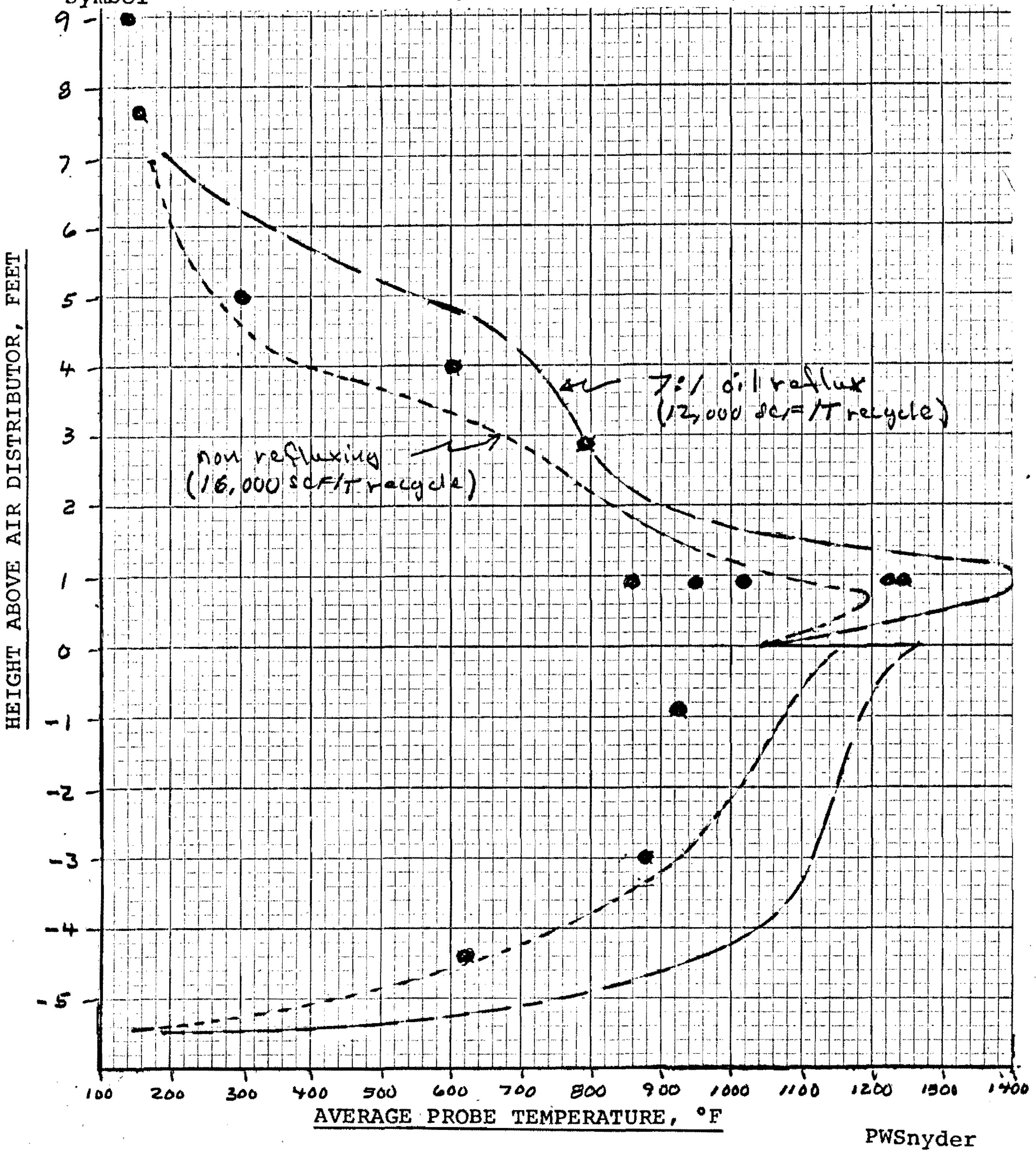


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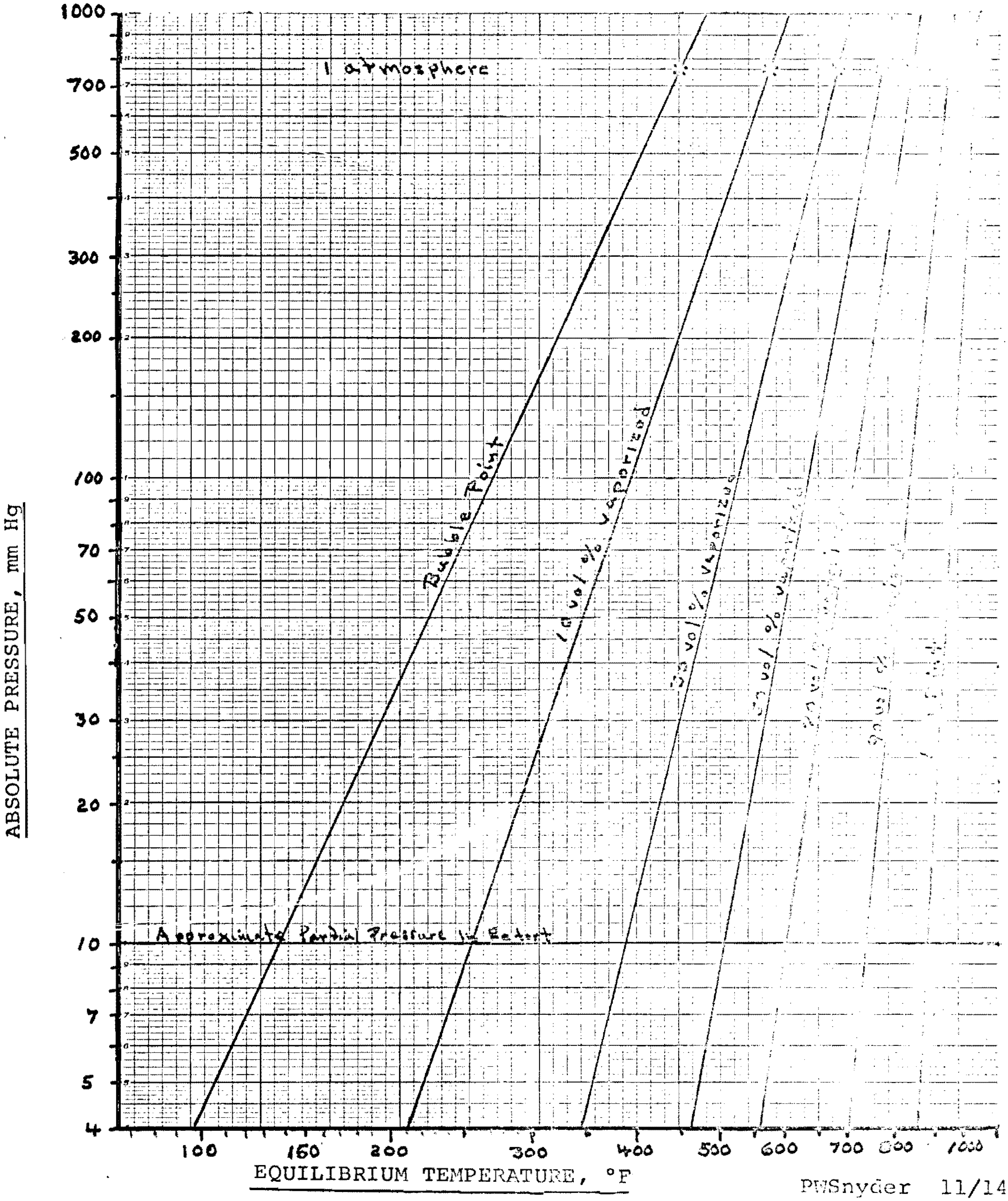
FIGURE 21

CALCULATED PHASE DIAGRAM FOR A TYPICAL SHALE OIL

Properties:		
	Gravity, °API	20.0
	Carbon/H_2 Ratio, wt/wt	7.6
	Vacuum Assay at 760 mm Hg, °F	
	10 vol %	500
	50 vol %	800
	90 vol %	1,050
	Vol Avg Boiling Point, °F	780
	Characterization Factor "K"	11.3
	Critical Temperature, °F	1,100
	Critical Pressure, psia	430



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