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MOBIL RESEARCH AND DEVELOPMENT CORPORATION

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TECHNICAL MEMORANDUM NO. 67-20

SUMMARY OF ACTIVITIES OF RETORT OPERABILITY TASK FORCE

ANVIL POINTS OIL SHALE RESEARCH CENTER

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The primary object of the Anvil Points Oil Shale Research Center TECHNICAL 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 TECHNICAL 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 TECHNICAL MEMORANDA have not been edited in detail.

(1) Mobil Research and Development Corporation, Project Manager

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

SUMMARY OF ACTIVITIES OF RETORT OPERABILITY TASK FORCE

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SUMMARY OF ACTIVITIES OF RETORT OPERABILITY TASK FORCE

I. INTRODUCTION

In the early part of 1967, considerable difficulty was experienced in operating Retort No. 3 in a stable fashion. This difficulty was particularly pronounced while processing small and wide range shale, that is, the 1/4 to 1 inch and 1/4 to 2 1/2 inch fractions. The ultimate symptom of inoperability in this retort, and in the Gas-Combustion Retort in general, is serious channeling of shale and gas flow. In Retort No. 3, these disturbances could be inferred from the development of nonsymmetrical vertical temperature profiles on the east and west sides of the retort and from divergence of the shale flow rates in the six feed pipes at the top of the retort. Generally, when the retort was shut down in this condition, a clinker was found to be wedged into and supported by the air distributor.

A task force was established by the Program Manager to attack the operability problem which existed when Retort No. 3 was operated with either small or wide range shale (Reference 1). This task force was responsible for testing and demonstrating a solution to this problem using bench scale and model studies. Its members were K. I. Jagel, Jr., Chairman; D. Liederman; R. L. McGalliard and L. J. Skowronek. The group was organized on May 15, 1967 and operated intensively with its full membership for a five-week period. Vigorous activity with curtailed participation by some members continued until about September 15, 1967.

Prior to the establishment of this task force, some preliminary work related to this problem was done by J. W. Payne, P. W. Snyder, Jr., J. Wei and J. Zahner, all of Mobil Research and Development Corporation, and by various staff members at Anvil Points.

This memorandum will review and summarize the work done by the task force established at Anvil Points and will also review earlier work dealing with the mechanism of retort operability failure.

II. SUMMARY AND CONCLUSIONS

The clinkers which are closely associated with the operability problems of the Gas-Combustion Retort are caused, primarily, by the wedging of carbonaceous agglomerates in the air distributor assembly. When these agglomerates are held in this hot, oxidizing environment, they burn at high temperatures and form clinkers.

Carbonaceous agglomerates are formed by localized liquid accumulations in the bed. These localized accumulations may be initiated by pieces of rich shale or by locally high concentrations of fine shale in the shale bed. These fines are more heavily wet with oil by the impaction of mist or by the surface condensation of oil than large shale pieces.

These accumulations are self-aggravating because:

1. the high local concentration of oil increases the scrubbing efficiency of the bed, therefore, more mist is removed from the gas stream, thus increasing the local liquid concentration;

2. the flow of gas through these regions is restricted causing a local cooling of the bed which causes:

    a. increased local condensation

    b. a low energy region in the bed to which contiguous liquid flows.

As liquid is vaporized and cracked from these local accumulations, a tarry, carbonaceous residue is left behind which binds pieces of shale and dust together to form cohesive masses. This occurs at a temperature of about 700 F. The cohesive masses of shale, dust, and tarry binder are converted to dry hard agglomerates at temperatures between 800 F and 1200 F.

The formation of cohesive masses and carbonaceous agglomerates has been avoided in several small scale retorts by disengaging a stream of oil from the shale bed at 600 F. This has resulted in improved operability and yields, when compared to the conventional Gas-Combustion Retort. This has been done by causing flooding to occur in a controlled fashion at a point in the retort that liquid can be disengaged.

This principle has been applied in Retort No. 3 with limited success. Some improvement in the allowable gas rates that could be used with 1/4 to 1 inch shale in Retorts No. 2 and 3 was achieved. However, yields were lower than desired. Because of the press of time, experimentation was directed toward proving the principle rather than developing a completely feasible commercial system.

A proposed improved application of this principle to Retort No. 3 is described in this memorandum. The cyclones and the inclined grates of the device tested in Retort No. 3 have been eliminated thus simplifying the design. These changes and other improved features of the proposed device have resulted from a careful review of Retort No. 3 operating experience with a liquid disengaging device and from related model studies.

A commentary on Technical Film No. 67-1 which presents some of the model studies that have been carried out in conjunction with this work is presented in the appendix to this memorandum.

III. DETAILED DISCUSSION

A. Retort Operability Failure Mechanism

When Retort No. 3 became inoperable, it was usually due to serious channeling of shale and gas flow. A clinker was usually found resting on, or wedged in, the air distributor.

1. Clinker Formation From Carbonaceous Agglomerates

Because of the general occurrence of clinkers when Retort No. 3 became inoperable, a bench scale study was undertaken to establish how clinkers were formed.

Preliminary work in September, 1966 had shown that shale could sinter and vitrify at temperatures likely to be met in the Gas-Combustion Retort. When shale was subjected to a temperature of about 2000 F, the carbonates were rapidly decomposed and the shale structure was weakened. Some sintering and loss of particle identity also occurred. If the shale were heated to 2100 F, or higher, for about fifteen minutes, vitrification occurred as indicated by a complete loss of particle structure, and by a glassy appearance.

A sketch of the terminal clinker that was formed in Retort No. 3, during Run C990, is shown in Figure 1. A decarbonized, vitreous portion of this aggregate exists around the air distributor and rests on the yoke. Its composition is indicated by Sample 1. Samples 2 and 3 are also substantially free of organic carbon but most of the inorganic carbon, in the form of carbonates, remains. Sample 4 is relatively rich in coke carbon compared to either a raw or spent shale. This may be inferred from their coke carbon to ash ratios. (Coke carbon rather than organic carbon is used in this argument because of the wide difference in oil content among these samples.)

The fact that organic decarbonization has proceeded to the extent that it has in Samples 1, 2, and 3 suggests that this aggregate was in place for some time before the retort was shut down. Vertical temperature profile data support this inference. Sample 4 is probably a remnant of a larger organic carbon-rich aggregate existing on the east side of the retort. A multiparticle aggregate, which is rich in fuel and which is located near the point of air injection, is felt to be the most likely precursor to the massive clinkers observed in Retort No. 3.

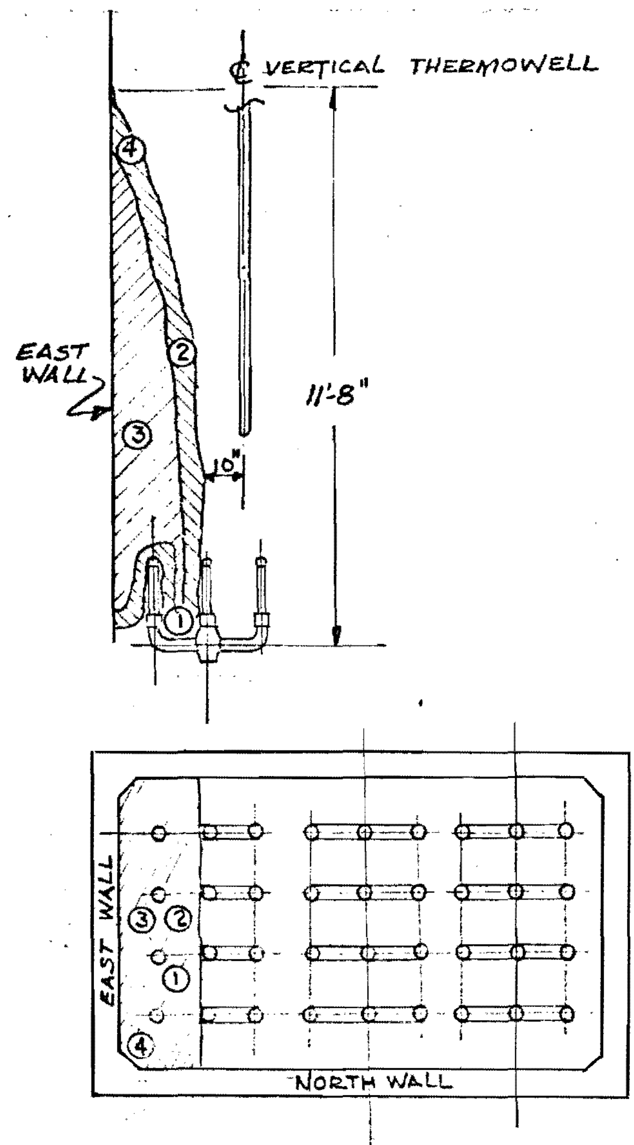
Such carbon rich aggregates may also be found in the spent shale discharge from a stable operation of

FIGURE 1

CLINKER FOUND ON SHUTDOWN - RUN C990

	1	2	3	4	Raw Shale	Spent Shale
Fischer Assay, Gal/Ton(1)	0.3	0.1	0.3	10.2	25.8	2.0
Oil, Wt %	0.1	0.1	0.1	3.9	9.8	0.8
Water, Wt %	0.2	0.2	0.3	0.7	0.9	0.8
Gas + Loss, Wt %	0.0	0.3	0.3	1.0	1.8	0.4
Spent Shale, Wt %	99.7	99.4	99.3	94.4	87.5	98.0
Chemical Analyses(1)						
Mineral CO_2, Wt %	1.26	17.3	12.7	19.0	17.2	16.3
Total Carbon, Wt %	0.54	6.46	2.56	12.5	16.0	7.7
Total Hydrogen, Wt %	0.54	0.20	0.07	0.85	1.68	0.37
Ash, Wt %	99.2	79.6	85.0	72.0	69.6	80.3
Organic Carbon, Wt %	0.20	1.73	(0.00)	7.31	11.3	3.25
Organic Hydrogen, Wt %	0.52	0.18	0.04	0.77	1.46	0.30
Organic (C/H) Wt Ratio	--	9.61	--	9.49	7.74	10.83
(Organic C/Ash) Wt Ratio	--	0.0292	--	0.1015	0.1623	0.0405
(Coke C/Ash) Wt Ratio	--	0.0198	--	0.0508	0.0341	0.0303

(1) Dry Basis



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Retort No. 3. A grab sample of uncrushed spent shale was taken during a recent operation with 1/4 to 2 1/2 inch raw shale. The roll feeder was in use at that time so that some crushing of the spent shale occurred as it was discharged. Sixty percent of the spent shale passed through a four-mesh screen. Of the remaining 40%, 93% was identifiable as spent shale particles, 6% were small carbon-rich aggregates, which have generally been called agglomerates, and 1% were small clinkers. The carbon-rich portions of this sample are the fines and the agglomerates. These results are summarized in Table 1. The clearest indication that these two components are organic carbon-rich is the ratio of organic carbon to ash. This ratio is independent of the extent of carbonate decomposition and therefore gives an unambiguous criterion for deciding whether or not a fraction is organic carbon-rich. If one breaks up the agglomerate fraction so that the carbonaceous binder can be separated from the pieces of spent shale that it binds, it is found that this material has an organic carbon to organic hydrogen ratio of 9.20 and an organic carbon to ash ratio of 0.0410. This organic carbon to ash ratio is about 30% higher than that for the spent shale composite analysis, more than twice as high as for burned spent shale particles, and more than ten times that for clinkers. These relatively high ratios suggest that the binder is a pyrolysis product of shale oil.

The primary goal of the bench scale study was to find out how these carbonaceous multiparticle aggregates which appear to be the precursors of clinkers are formed. J. W. Payne, of Mobil, pointed out that agglomerates of shale bound by a carbonaceous binder could form when shale oil refluxes on shale. Independent experiments were subsequently undertaken in this laboratory to explore the implications of this observation. Agglomerates were prepared by allowing oil to reflux and percolate over small particles of shale contained in a glass tube. The light material distilled off, while the remaining shale oil thickened and became tarry, and formed a cohesive mass with the shale particles. As this mass was heated, heavier ends cracked, depositing a shiny, black coke which bound the shale into a strong, hard agglomerate.

The strength of the agglomerate depends, to a great extent, upon the ratio of shale oil to shale used. Strong agglomerates were formed when enough shale oil was used to fill the interparticle voids in the bed of shale. If shale is closely screened, the void fraction is about 0.45 to 0.50. It has been observed that with closely screened shale, about a 1:1 ratio of shale oil

TABLE 1

SPENT SHALE ANALYSIS FROM A TYPICAL STABLE OPERATION OF RETORT NO. 3

	-4 Mesh Fines	+4 Mesh Particles			Composite
Weight % in Total Sample	60%	40%			
		Burned Spent Shale	Agglomerates	Clinkers	
Weight % in +4 Mesh Fraction		93%	6%	1%	
Chemical Analyses[1]					
Mineral CO_2, Wt %	16.6	14.6	17.0	2.82	15.8
Total Carbon, Wt %	7.47	5.49	7.53	1.12	6.7
Total Hydrogen, Wt %	0.71	0.28	0.43	0.11	0.54
Ash, Wt %	79.0	82.6	78.9	96.9	80.4
Organic Carbon, Wt %	2.94	1.50	2.89	0.35	2.39
Organic Hydrogen, Wt %	0.27	0.20	0.37	0.08	0.25
Organic (C/H) Wt Ratio	10.89	7.50	7.81	4.38	9.56
(Organic C/Ash) Wt Ratio	0.0372	0.0182	0.0366	0.0036	0.0297

[1] Dry Basis

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to shale gives the hardest agglomerates. The void fraction of a shale bed is reduced if dust is mixed with the shale, therefore, it may be anticipated that the amount of oil needed to form strong agglomerates would be reduced. This has been observed to be so.

The temperature at which the shale oil was refluxed and percolated over the shale also had an important effect upon the consistency of the agglomerate. When furnace temperatures were held in the region of 600 to 800 F, the end product was a tarry plastic cohesive mass. At temperatures from 800 to 1200 F, hard agglomerates were formed. An experiment, in which shale oil was refluxed and percolated on glass beads, showed that not even shale was necessary to form an agglomerate, but that the cracking and coking of the oil on a substrate of any kind was all that was necessary. Coke separated from the beads, contained 86% carbon and 2.7% hydrogen.

It was also found that shale oil bottoms coke very easily. An experiment, in which a 20% bottoms fraction was heated in the presence of shale, showed that a hard agglomerate could form in less than 5 minutes at 1000 F.

In glass retort experiments, which will be described below, it was shown that shale oil heavy ends which percolate in a bed of shale become heavier, polymerize, and, thus, could form agglomerates in a fairly short time. In summary, the conditions found necessary to form cohesive masses and agglomerates were the following:

a. <u>Presence of Oil</u>

   (1) The strength of the agglomerate increases with the amount of oil.

b. <u>Reflux and Percolation</u>

   (1) The strength of the agglomerate increases with the number of cycles.

      (a) Approximately six cycles are needed to form moderately strong agglomerates.

      (b) Each cycle involves loss of light material overhead, leaving a heavy residue which cracks, cokes, and polymerizes.

c. Aggregate of Solid Material

(1) The strength of the agglomerate seems to increase with decreasing particle size (or better fit).

d. Heat

(1) At temperatures of about 600 to 800 F, refluxing and percolation cause the formation of a tarry, plastic cohesive mass.

(2) At temperatures of 800 F up to at least 1200 F, the cohesive mass will form hard dry agglomerates.

Clinkers were formed from these laboratory prepared agglomerates at various oxygen concentrations for a number of combinations of time and temperature. These experiments were carried out by placing the agglomerate, surrounded by spent shale, in a stainless steel crucible fitted with a gas preheating coil. The apparatus was purged with a flowing stream of nitrogen and put into a muffle which had been preheated to the temperature desired for the experiment. After fifteen minutes, during which the apparatus heated up to the muffle temperature, a mixture of nitrogen and oxygen adjusted to the desired concentration and flow rate was substituted for the nitrogen purge. The experiment proceeded in the muffle for fifteen minutes, generally. At the end of the experiment, the apparatus was removed from the furnace and purged with nitrogen as it cooled. It was found that:

(a) At 21% O_2 (simulating air at the outlet of the air distributor) for three initial temperatures - 2000, 1500 and 1250 F, the agglomerates and shale form hard vitreous clinkers with blow-holes caused by gas channeling. The agglomerates and shale were fused into one mass at 2000 and 1500 F initial temperatures. Some of the shale particles were discrete at 1250 F, but vitrified. At 1000 F, the shale and agglomerate still retained their original shape but were free of carbon. The agglomerate was porous, and softer than originally. At both 1500 and 2000 F, clinkers were formed after only 30 seconds exposure to 21% oxygen. These clinkers are formed because the total effect of the initial temperature plus the temperature rise, as the binder burns, is sufficient to exceed the shale vitrification temperature.

(b) At 5% O_2 and 2000 F, the material was vitrified. The agglomerate and shale were separated, but there were aggregates of 10 to 20 pieces of shale fused together. There was an apparently diffusion-controlled burned zone, 1/8-inch thick, on the outside of the agglomerate with carbon remaining inside this zone.

The sample ignited at an initial temperature of 1500 F, appeared sintered, but was not vitrified like the one at 2000 F. Unburned carbon remained inside the agglomerate which was still relatively hard. There was no agglomeration of the surrounding shale particles.

The sample at 1000 F was more gray, compared to the orange tinge of samples previously described. This suggests that carbon remained, not only in the agglomerate, but in the individual surrounding shale particles - which were not agglomerated. The structure of the agglomerate was spongy; the surface soft. But the agglomerate was still fairly strong under compression and would probably hang up in the retort if it were large enough.

(c) An experiment was run at 1500 F and 21% O_2 using no agglomerate, but just spent shale containing about 2.6% organic carbon. This is almost double the organic carbon in normal spent shale. Since the shale was the loose material from the agglomerate-making experiments, it probably had a higher carbon concentration on the surface. This formed a massive vitreous clinker with some loose pieces of shale on its periphery. This indicates that a mass of spent shale with an excess of carbon on it (possibly from refluxing and percolation of shale oil) can also form a clinker without first having formed an agglomerate. This is a rather improbable mechanism for the formation of the massive multiparticle agglomerates which are generally found in the air distributor assembly when Retort No. 3 becomes inoperable.

The results of these experiments are shown in graphic form on Figure 2. The figure is divided into four zones which roughly describe what characteristics the shale and agglomerates had, after having been subjected to the described conditions of oxygen concentration and temperature. The boundaries should be considered fuzzy areas - not sharply defined.

The clinkers formed in the laboratory by these experiments resemble the hard, vitreous portion of the clinker pictured in Figure 1.

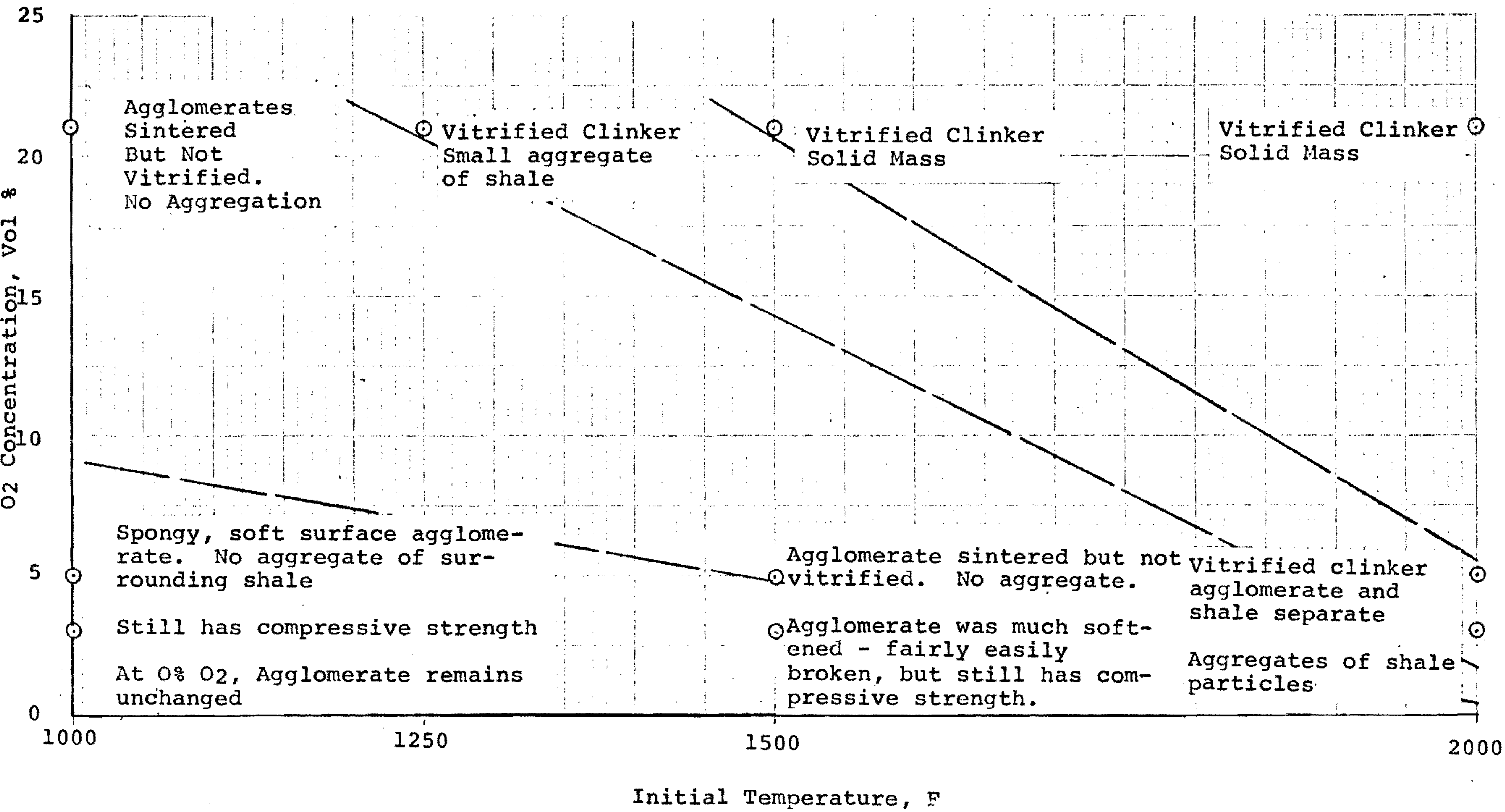
This series of experiments, coupled with the observations of the carbonaceous aggregates associated with the clinkers formed when Retort No. 3 failed, indicate that the most probable clinker formation mechanism of Retort No. 3 is by way of agglomerate precursors. These carbonaceous agglomerates are formed by the refluxing, revaporization, and thermal cracking of shale oil.

2. <u>Clinker Formation From Combustion of Recycle Gas</u>

An alternate mechanism that has been proposed for clinker formation is that clinkers are formed due to high temperatures which occur when recycle gas burns.

FIGURE 2

CLINKER FORMATION FROM CARBONACEOUS AGGLOMERATES
(In each case, agglomerate was preheated for 15 minutes at initial temperature)



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Adiabatic reaction temperatures for various mixtures of recycle gas and air have been calculated. Temperatures which are high enough to cause clinker formation are noted. The problem is aggravated by poor mixing of these two gas streams.

In this calculation, it has been assumed that the recycle gas enters the combustion zone at 1100 F and that it has the following composition:

H_2O	15.00 Mole %
CO_2	23.37
O_2	0.25
N_2	52.03
CH_4	1.36
CO	2.72
H_2	3.91
Others	1.36
	100.00 Mole %
lb C/MSCF	12.3
lb H/MSCF	0.63

Air has been assumed to enter this zone at 200 F. The stoichoimetric oxygen concentration in a mixture of this recycle gas and air is 8.23%. The calculated results are shown in Figure 3.

Although sufficiently high temperatures for clinkering may be reached by this mechanism, it is doubtful that the stoichoimetric ratio of recycle gas to air needed for clinkering to occur could exist over a sufficiently extensive region so that the multiparticle aggregates which are observed could be formed.

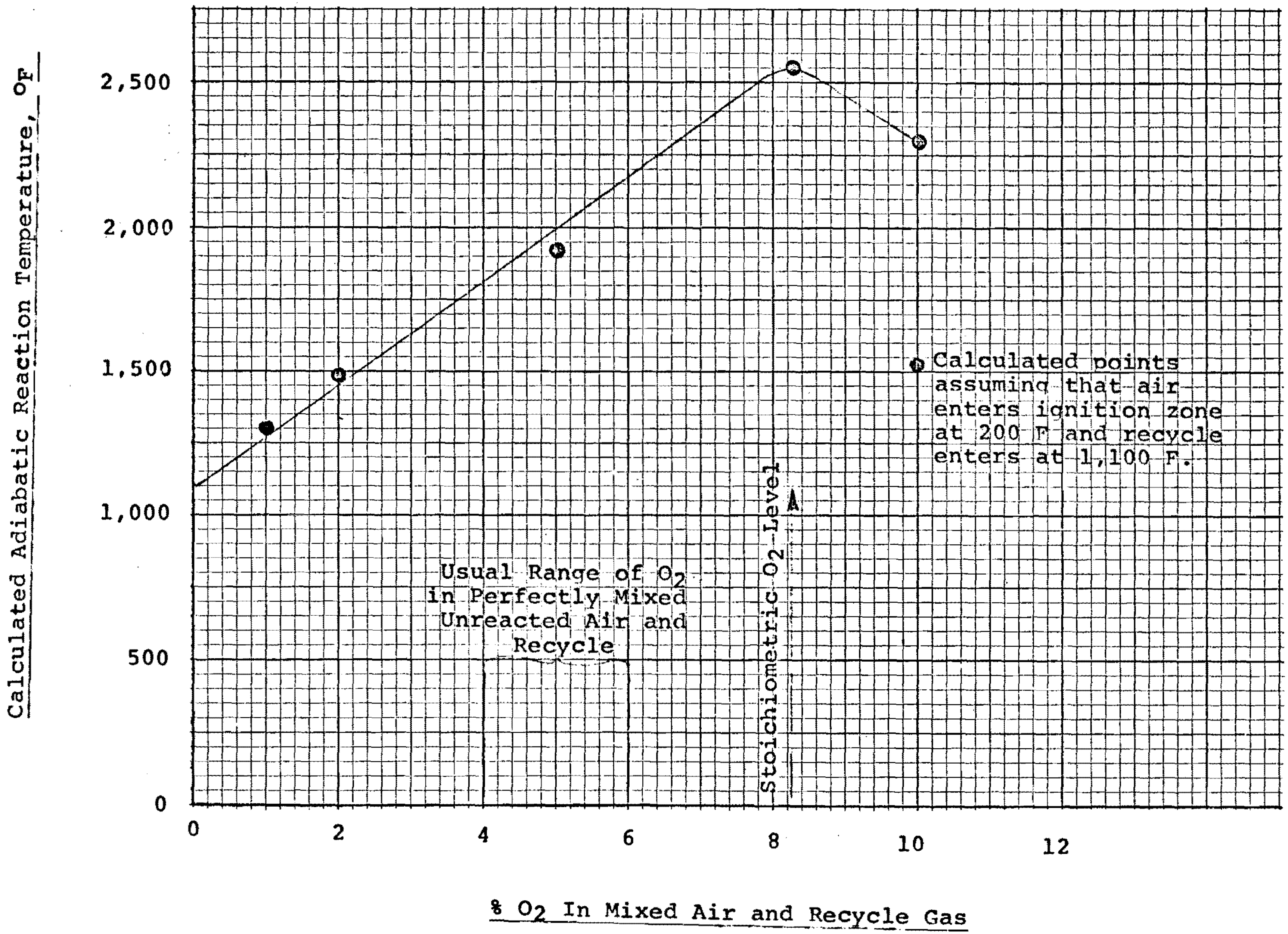
3. Decomposition of Agglomerates Without Clinker Formation

Carbonaceous agglomerates can be broken up by burning off the coke binder using mild oxidation conditions. When agglomerates were exposed to conditions of 1500 F and 3% oxygen for fifteen to thirty minutes, diffusion-controlled burning of the agglomerate occurred. These agglomerates were fairly resistant to compressive forces where organic carbon had not been burned off, but their surfaces were easily abraded. After all of the organic carbon was burned off, they could be more easily crushed.

This process probably occurs to some extent in a Gas-Combustion Retort in which agglomerates are being made

FIGU[illegible] 3

EFFECT OF O_2 CONTENT OF MIXED AIR AND RECYCLE GAS ON THE CALCULATED ADIABATIC REACTION TEMPERATURE OF THE MIXTURE



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at all times. The larger the agglomerates, the less effective this oxidative decomposition is, because of the diffusion-controlled nature of this process.

Since large agglomerates, which will not pass through the air distributor, are the critical problem, it is doubtful that this process is very effective in clearing the air distributor of multiparticle aggregates.

4. Statement of Retort Operability Failure Mechanism

The critical step in the failure of operability of the Gas-Combustion Retort, as conventionally conceived, is the local accumulation of liquid in the moving shale bed. These local accumulations are probably started by rich pieces of shale or by locally high concentrations of fine shale in the shale bed. These fines are more heavily wet with oil by the impaction of mist or by the surface condensation of oil than large shale pieces.

These accumulations grow in size because of their self-aggravating nature. This is due to

- the high local concentration of oil which increases the scrubbing efficiency of the bed. Mist is removed from the gas stream thus augmenting the local liquid concentration;
- the restricted flow of gas through these regions which causes:
    - increased local condensation;
    - a low energy region in the bed to which contiguous liquid flows.

As liquid is vaporized and cracked from these local accumulations, a tarry carbonaceous residue is left behind which binds pieces of shale and dust together to form cohesive masses. This occurs at a temperature of about 700 F. This chain of events may also be triggered by any other mechanisms which cause local flow nonuniformities in the shale bed. The larger the retort cross section is, the greater the probability that such nonuniformities will occur.

If this cohesive mass remains intact until its temperature is increased to about 1200 F, it will become a dry, hard agglomerate with a carbonaceous binder. For the large, multiparticle aggregates which result in retort inoperability, it is necessary that the agglomerate become wedged in the air distributor hardware. When this occurs, the carbonaceous binder burns causing a sharp temperature rise. If the initial temperature of the agglomerate and the oxygen concentration are high enough, vitrification will occur. This burning is a diffusion-controlled process, therefore, if sufficient heat generation to cause vitrification is to occur, the agglomerate must be exposed to a high oxygen concentration for some period of time. An additional minor factor that adds to the temperature rise, is the decrease in the heat dissipation due to the nonflowing zone in the shale bed.

If the carbonaceous agglomerate becomes a clinker, the massive gas and shale channeling, which ultimately renders the retort inoperable will occur.

B. <u>Strategies For Achieving Retort Operability</u>

Three strategies have been considered in achieving satisfactory retort operability. The first of these is the conventional approach of optimizing hardware and process conditions. With this approach, a few weak cohesive masses are formed, probably; (ideally, none would be formed). These weak masses are broken up due to shale movement before they become agglomerates. It has been difficult to scale up this method of achieving operability as indicated by the experiences of going from Retort No. 1 to Retort No. 2 or Retort No. 2 to Retort No. 3.

A second strategy is the approach suggested by P. W. Snyder, J. Wei and J. Zahner of Mobil. This method deals with agglomerates, if they are formed, by four techniques. Dilution gas is used to moderate the combustion zone intensity while an oxidizing atmosphere in the combustion zone assists in the gentle decomposition of these agglomerates. If they do not decompose, the air distributor configuration is open enough to handle fairly sizeable masses. If these agglomerates persist to the spent shale drawoff device, they are broken up by the crushing action of a roll feeder.

The third strategy is the one taken by the task force established at Anvil Points. It is a fundamental solution to the problem of carbonaceous agglomerate formation. The method, in this case, is to prevent oil accumulation in the bed and thus prevent the formation of cohesive masses. The studies in small scale retorts, in flow models, and in Retort No. 3, that will be described in the subsequent sections of this memorandum, are intended to either define the stated retort operability failure mechanism further, or to provide design information to be used in applying this strategy.

C. Small Scale Retort Studies

A number of small scale retorts have been developed which shed additional light on the mechanism discussed above. With one of these units, which had a fixed bed, shale flow was simulated by moving the furnace up the retort at a rate equivalent to the desired shale rate. The other three units were true moving bed units.

1. Moving Furnace Fixed Bed Retort Studies

This retort consisted of a one-inch glass tube filled with shale and heated by an electric furnace which was moved up the bed at a rate which simulated a shale rate of 500 lb/(hr)(ft^2) in the retort. Nitrogen was used as a sweep gas at a rate of 20,000 SCF/T. Generally, the unit was operated with gas entering the bottom and leaving the top of the retort. This retort is shown in Figure 4.

The experiments which have been conducted with this equipment are summarized in Table 2. The independent variables in these experiments were the shale properties of assay and size. The dependent variable was relative agglomeration, which is defined as the ratio of the specific volume of agglomerates (cc of agglomerates per gram of raw shale charged) for the test experiment, and the specific volume of agglomerates in the base case experiment.

The base case experiment was carried out at the shale and gas rates noted above using 25 gallon per ton, 4 - 8 mesh shale. The assay of this shale is in the commercially attractive range; the size is considerably smaller than the size usually considered desirable for processing in a vertical shaft kiln such as the Gas-Combustion Retort. This size was chosen because small shale accentuates the mechanisms which result in oil accumulation in the shale bed. In the base case experiment, 0.15 cc of agglomerates per gram of raw shale charged were observed. By definition, the base case relative agglomeration is 1.00. A flooded layer of liquid above the furnace was observed.

a. Liquid Loading

The first effect that was studied was that of <u>liquid loading</u>. As shale richness increased, relative agglomeration increased markedly to 2.47. The retort was badly flooded in this experiment. However, it was found that relative agglomeration was reduced to about 0.10 if about 10% of the

FIGURE 4

MOVING FURNACE FIXED BED
RETORT

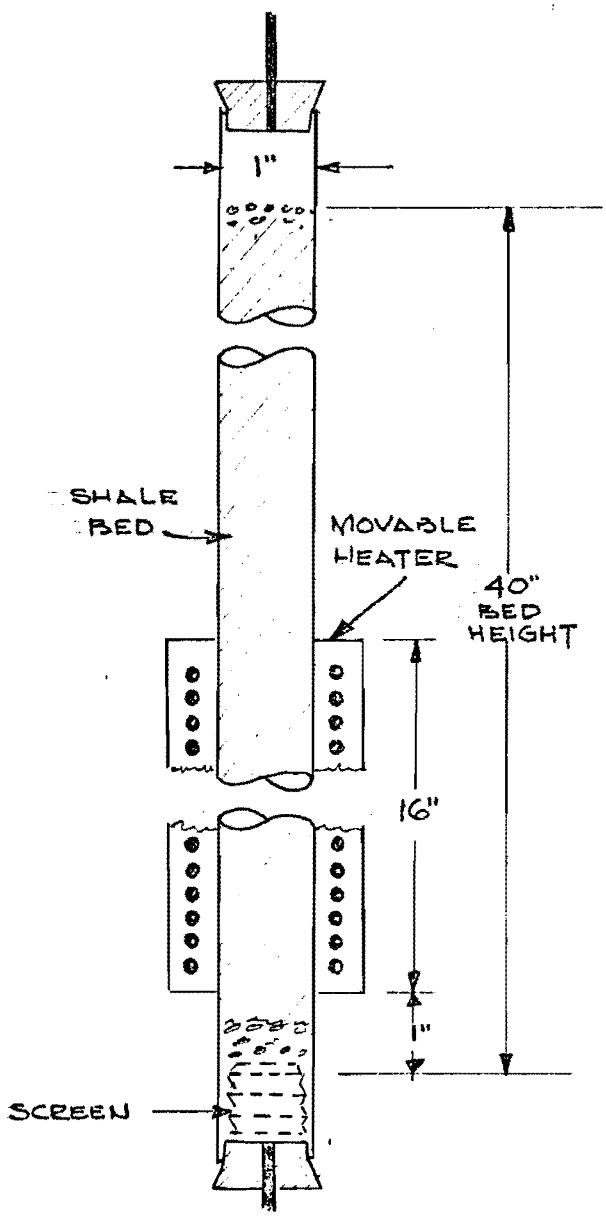

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TABLE 2

MOVING FURNACE FIXED BED RETORT STUDIES
(All Runs at 500 Mass Rate and 20,000 SCF/T; Furnace Temperature 1100 F)

	Shale Properties Size	Shale Properties Assay	Relative Agglomeration
Base Case	4-8	25	1.00
Liquid Loading Effect			
1. All gas and liquid overhead	4-8	41	2.47
2. 10% of liquid removed above furnace, remainder plus gas overhead	4-8	41	0.10
3. All gas and liquid removed above furnace	4-8	25	0.80
4. Cocurrent retorting	4-8	31	0.93
5. Gravel in furnace during startup	4-8	25	0.74
Size and Size Range Effect			
1. Large Shale	2½-3	25	0.67
2. Small Shale	14-28	25	1.53
3. Mixture of large shale and small shale	62%-2½-3 38%-14-28	25	1.47

Dust - Liquid Interaction

1.	Large Shale plus dust dispersed in bed (Raw shale preheated by gas flow)	90%-2½-3 10% thru 65	25 SS	0.62
2.	Large Shale plus dust dispersed in bed (Raw shale preheated electrically)	90%-2½-3 10% thru 65	25 SS	0.23
3.	Large Shale plus dust introduced with gas (Raw shale preheated by gas flow)	89%-2½-3 11% thru 65	25 SS	0.55
4.	Large Shale plus dust introduced with gas (Raw shale preheated electrically)	89%-2½-3 11% thru 65	25 SS	0.33

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liquid product were removed from the zone immediately above the furnace by means of a capillary tube. This capillary tube was moved upward with the furnace. The liquid withdrawn was about equal to the liquid holdup in the flooded zone. It was observed that when this portion of liquid was removed, no flooded layer formed. As mentioned previously, mist is trapped by the shale in two ways: impaction by the shale and scrubbing of the mist from the gas by liquid. A third mechanism which causes the oil to wet the shale is surface condensation. The oil removal technique used in this experiment is intended to minimize the scrubbing of mist from the gas by liquid, by reducing liquid accumulation.

When all of the liquid product and gas were removed from the retort by a tube whose end was in the zone immediately above the furnace, it was found that the relative agglomeration was intermediate between that for no oil removal and 10% oil removal. This is believed to be due to the poor preheating of the shale that occurred in this case. The shale was rapidly heated from a low temperature to retorting temperature in the top few inches of the furnace. The consequent high liquid accumulation in this zone caused some agglomeration.

One other approach to reducing liquid accumulation is to simulate a cocurrent retorting zone by moving the furnace downward as gas is flowed downward. (In this experiment, gas was put into the upper end of the retort and removed from the lower end.) The relative agglomeration was improved by this technique but not as strikingly as in the case when a small portion of liquid, corresponding roughly to the holdup in the flooded layer, was removed from the bed. This suggests that the heavy oil held at the juncture of particles may be an important cause of agglomeration.

The final experiment dealing with liquid loading was similar to the base case experiment, except that gravel was put in the furnace zone of the retort during warmup. Relative agglomeration was reduced by this technique so that it might be concluded that reducing the average oil loading is an effective device for reducing agglomeration.

b. <u>Shale Size and Size Range</u>

The second effect that was studied was that of <u>shale size and size range</u>. This was studied initially by repeating the base case experiment but using 2 1/2 to 3 mesh rather than 4 to 8 mesh shale. No flooded layer was observed in this experiment but moderate liquid holdup throughout the bed was noted. The relative agglomeration was reduced in this case. This suggests that significant agglomeration can occur even though the bed is only moderately loaded with liquid.

This effect was studied further with 14 to 28 mesh shale. This shale size caused such severe flooding that little mist left the top of the shale bed. Entrainment of massive droplets occurred and relative agglomeration increased in this case. When a mixture of two-thirds 2 1/2 to 3 mesh shale and one-third 14 to 28 mesh shale was evaluated, it was found to operate like the finer portion of the mixture. The relative agglomeration was about the same as that obtained with the 14 to 28 mesh shale alone. The small particles of shale adhered to the large particles.

c. Dust-Liquid Interaction

The third effect studied in this unit was that due to dust-liquid interaction. In this group of experiments, dust was either dispersed in the shale bed, or was injected with the sweep gas. In either case, improved operation, as indicated by lower relative agglomeration, was achieved if the bed was kept dry by removing the oil mist directly above the furnace and preheating the shale electrically so that no condensation occurred in the bed.

d. Effect of Additives on Oil Shale Agglomeration

Proposals have been made for the use of additives on shale to mitigate agglomerate formation. Several additives that have some surface active and solid lubricant properties were tested: barytes, bentonite, and talc. Another criterion was that they are inexpensive.

These materials were tested by mixing them with 4 to 8 mesh shale, and testing the mixtures in a retorting experiment carried out in the moving furnace fixed bed retort.

The effects of the additives were compared with shale to which no additives were added. The comparisons were made by carefully separating the agglomerated pieces of shale and measuring their loose volume and weight. The data are shown in Table 3.

TABLE 3

EFFECT OF ADDITIVES ON OIL SHALE AGGLOMERATION

Additive	Wt % Additive	Agglomerates cc/g Charge	Agglomerates Wt % of Charge
None	0	0.023	1.4
	0	0.022	1.3
	0	0.023	1.4
Barytes	0.01	0.020, 0.019	1.1, 1.1
	0.04	0.012, 0.024	0.6, 1.4
	0.2	0.029	1.9
	4.7	0.034	1.5
Bentonite	0.01	0.020, 0.022	1.0, 1.2
	0.04	0.023	1.2
	0.2	0.030	1.7
Talc	0.2	0.039	2.5
	1.1	0.042	2.3

Note: The shale used was -4 +8 mesh from which shale greater than 70 gallons per ton was removed by flotation. The shale assayed 25 gallons per ton as used.

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The conclusion from this data is that these additives do not lessen agglomerate formation; in fact, they may worsen it.

2. Studies With the 1-Inch I.D. Moving Bed Retort

The results obtained with the small static bed retort were encouraging enough to suggest that a moving bed retort using the same retort tube size (approximately one inch) could also be operated successfully. The two modes of operating this moving bed unit that have been tested are shown in Figure 5. Data are summarized in Table 4. The base case operation with this unit was carried out with 31 gallons per ton, 4 to 8 mesh shale at 500 lb/(hr)(ft^2) and at a nitrogen rate of 20,000 SCF/T raw shale. The retort was operated so that the raw shale was preheated by gas flow. As the unit operated, it gradually flooded. When flooded, the oil holdup in the bed was about 10% of the total oil produced by retorting. When nearly completely flooded, it was necessary to reduce the shale rate because of excessive pressure drop. (This experiment is shown in Technical Film No. 67-1 described in the Appendix.)

In a repeat of this experiment, a side arm was installed in the retort tube immediately above the retorting heater. The end of this side arm was plugged off so it was a dead end. As the retort was brought on stream, a small quantity of heavy, viscous liquid collected in this tube. Flooding did not occur as long as liquid continued to collect in the tube.

Flooding was initiated by increasing the rate of liquid accumulation in the zone above the furnace. This was done by decreasing the gas flow and increasing the shale rate momentarily. When the retort was brought back on condition, it was flooded. With the retort operating in this flooded condition, the gas and shale flow were cut off and the heater was turned off. An agglomerate quickly formed in the furnace zone. This was determined by breaking the retort tube. The heavy viscous liquid which had collected in the side arm was subsequently examined. It was found that it had a much higher specific gravity (approximately one), than shale oil and it could not be melted at 180 F.

To evaluate the <u>liquid loading</u> effect in this case, the equipment was arranged so that raw shale was preheated electrically to 700 F. Oil mist was removed from the unit immediately above the heater. No reflux layer formed in the run with air or the run without air. Although some dusty surfaces on the shale were noted in the air combustion runs, no clinker was formed even at air rates as high as 9000 SCF/T.

FIGURE 5

MOVING BED GLASS RETORT (1-INCH I.D.)

Raw Shale Preheated By Gas | Raw Shale Preheated Electrically

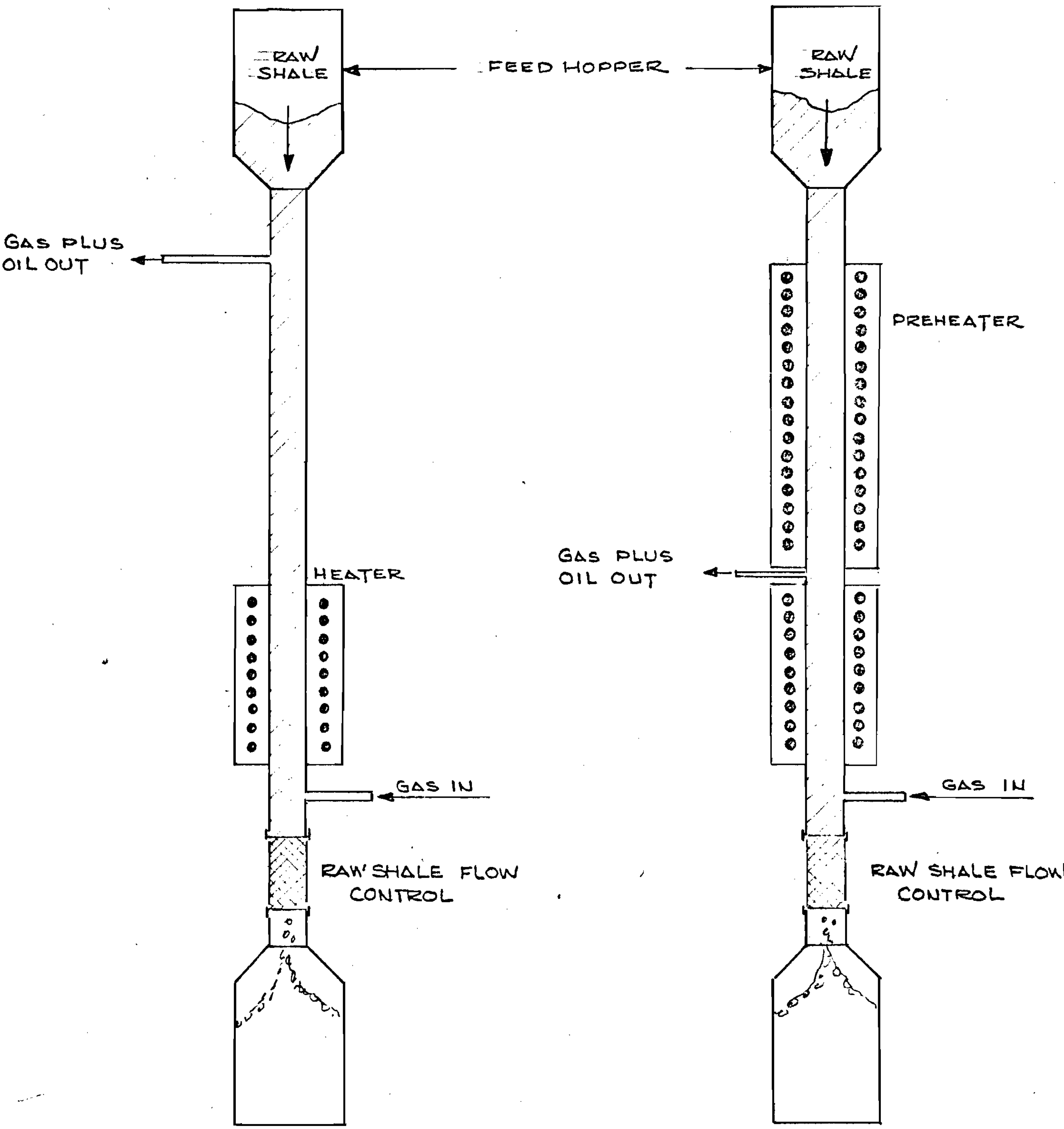

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TABLE 4

STUDIES WITH MOVING BED GLASS RETORT (1-INCH I.D.)

Base Case	Shale Properties: Fischer Assay, gallon per ton	Shale Properties: Size Mesh	Shale Flow	Time from Run Start	Height of Reflux	P
500 lbs/(hr)(ft^2) 20,000 SCF/T N_2 (Raw Shale preheated by gas)	31	4-8	Good	1.5 hour	0.0 inch	1.4" Hg
				2.5	8.5	1.7
				3.0	10.0	1.8
				3.3	12.3	1.9
Forced to reduce to 400 lbs/(hr)(ft^2) 20,000 SCF/T N_2				3.5	17.0	2.1
				3.7	22.0	2.5
				Shut down when liquid was splashed from vent		
Liquid Loading Effect						
1) 500 lbs/(hr)(ft^2) 20,000 SCF/T N_2 (Raw Shale preheated electrically to 700 F)	31	4-8	Good		No reflux layer	1.4
2) 500 lbs/(hr)(ft^2)	31	4-8	Good		No reflux layer	
15,500 SCF/T N_2 4,500 SCF/T Air				Strong Spent Shale, Dust on Surface		1.2
Increased Air to 9,000 SCF/T (Raw Shale preheated electrically to 500 F)			Good	Weaker Spent Shale, Dustier Surfaces, No Clinker		1.2

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3. Studies With the 2-Inch I.D. Moving Bed Retort

So that retorting with larger shale could be studied, a 2-inch I.D. glass retort, 9 feet high was constructed. Like the 1-inch retorts, it had a gas inlet port at the bottom and a gas-shale oil outlet port near the top.

Two relatively successful experiments were carried out in the 2-inch I.D. glass retort. In this unit, the shale was preheated electrically and the gas and oil vapor were disengaged from the bed at 700 to 750 F. The gas disengager used in this unit was a plenum which was concentric with the retort wall. The inner wall of this plenum was a screen cylinder which had the same inside diameter as the retort itself. The gas and vapor flowed through this screen into the plenum and then into the inlet of a condenser. Each of these experiments were ultimately shut down because of hard coke agglomerates which impeded shale flow. These formed on the surface of the screen of the gas disengager and were probably caused by the small but noticeable accumulation of oil in this region and in the region immediately below the disengager.

Because of this, experimental work with this unit was dropped, and investigations with a 3.6-inch metal retort were begun.

4. Studies with the 3.6-Inch I.D. Moving Bed Retort

a. Construction and Shakedown Operation

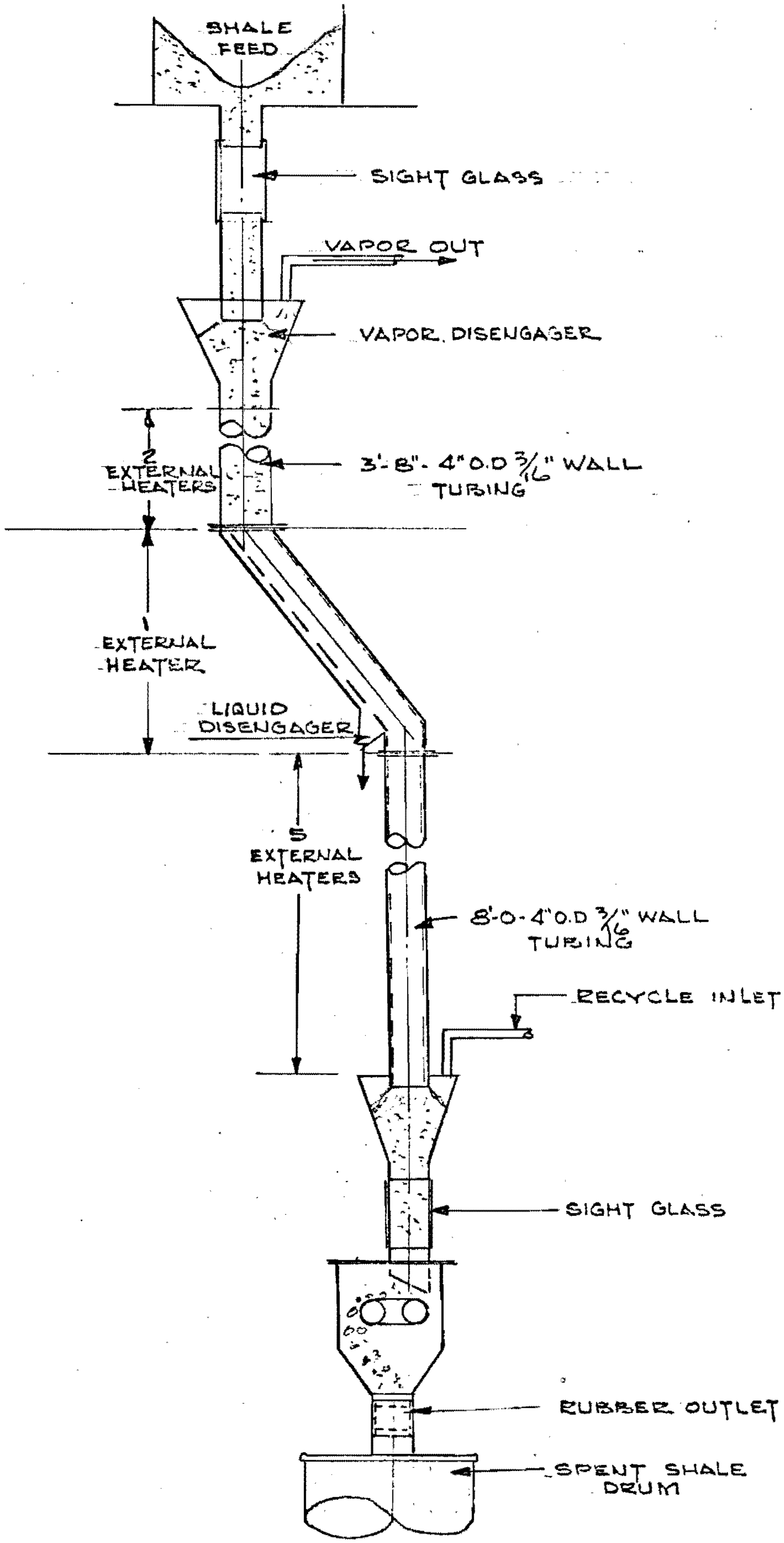
A 3.6-inch stainless steel moving bed retort was designed in cooperation with J. W. Payne of Mobil. A schematic of the design is shown in Figure 6.

The unique feature of this retort was a liquid disengaging system just above the retorting zone. The purpose of this system was to remove oil from the bed so that agglomerates, and thus clinkers, could be avoided.

It operates by allowing oil that has collected on the shale to flow off as the shale moves across a screen or grate.

The first experiments were done with a relatively inefficient liquid disengager shown in Figure 7. Nevertheless, liquid shale oil was disengaged from the bed at 700 F and at shale rates from 700 to 1000 lb/(hr)(ft^2) and at gas rates from 20,000 to 30,000 SCF/TRS. These experiments were of short

FIGURE 6



SCHEMATIC 3.6" RETORT

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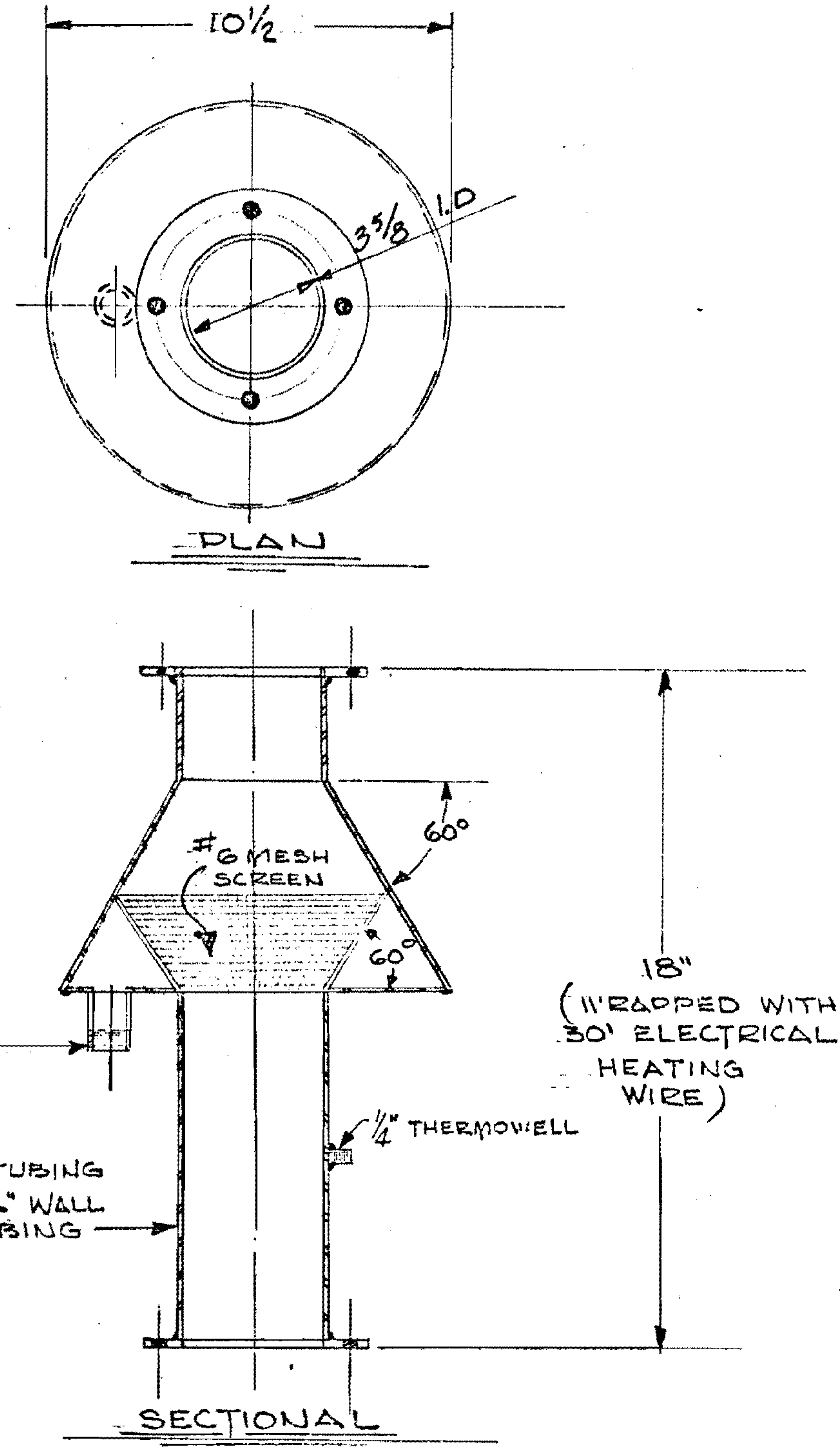
FIGURE 7

CONICAL LIQUID DISENGAGER



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duration and were terminated by mechanical difficulties in each case. In one of the experiments, a hard coke agglomerate was formed in the retorting zone, presumable because of the inefficient operation of the liquid disengager.

The subsequent experiments with this apparatus were done with an improved liquid disengager. These experiments are discussed in the following section. A flowsheet for the process as ultimately developed is shown in Figure 8. A drawing of the assembly and component part details is available as Drawing No. RC 166.

b. Operations

The first run (MBR3.6-3) with the disengager shown in Figure 9 was aborted because of several bad leaks in the system. Even so, it ran for about 2 1/2 hours. The run was at 930 lb/(hr)(ft^2) shale rate and 20,000 SCF/T gas rate. The disengager temperature had been about 650 F.

All subsequent runs were made with ceramic Raschig rings in the retort section at startup. This allowed stabilization of solids flow rate and unit temperature before shale retorting started. Table 5 summarizes the data obtained on the runs to be described below.

Run 4 was made at 930 lb/(hr)(ft^2) shale rate to test the operability of the disengager under the difficult condition of high shale rate. Operation was very good and yield was good at 85.5% Fischer Assay. In this run only, the disengager product was collected in an open bucket. Some of the lighter material flashed off and undoubtedly accounts for some of the deficiency in carbon balance. About 60% of the total oil was collected from the disengager. It was dry and had an API Gravity of about 17. The other 40% of the oil, along with most of the water from the process, was collected in a cooled cotton-batting demister. This product contained about 40% water. The gravity of this oil was about 30° API. The gas leaving the demister was about 85 F. The shale was fairly well retorted at this 930 lb/(hr)(ft^2) shale rate.

In Run 5, there was difficulty maintaining temperatures high enough in the retorting zone. Consequently the yield fell to 80.5% Fischer Assay and

FIGURE 8

3.6 INCH RETORT PROCESS FLOWSHEET

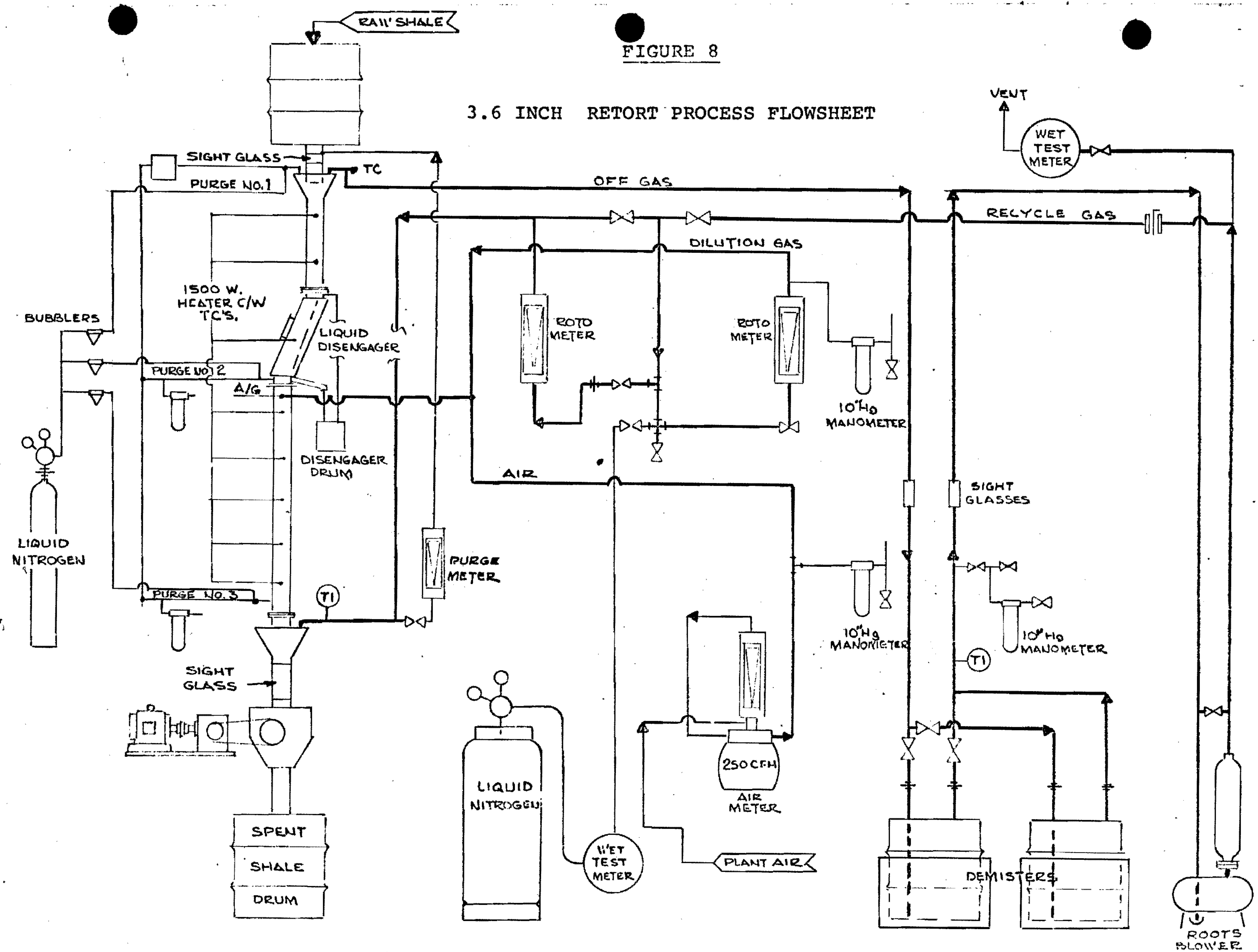
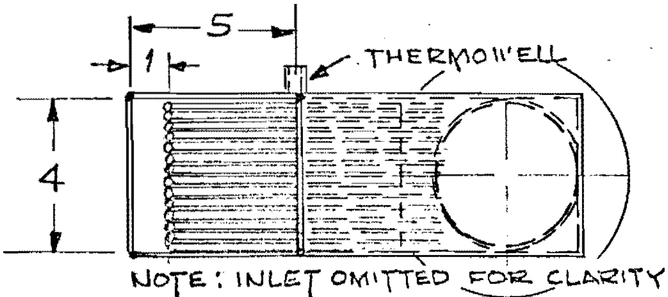
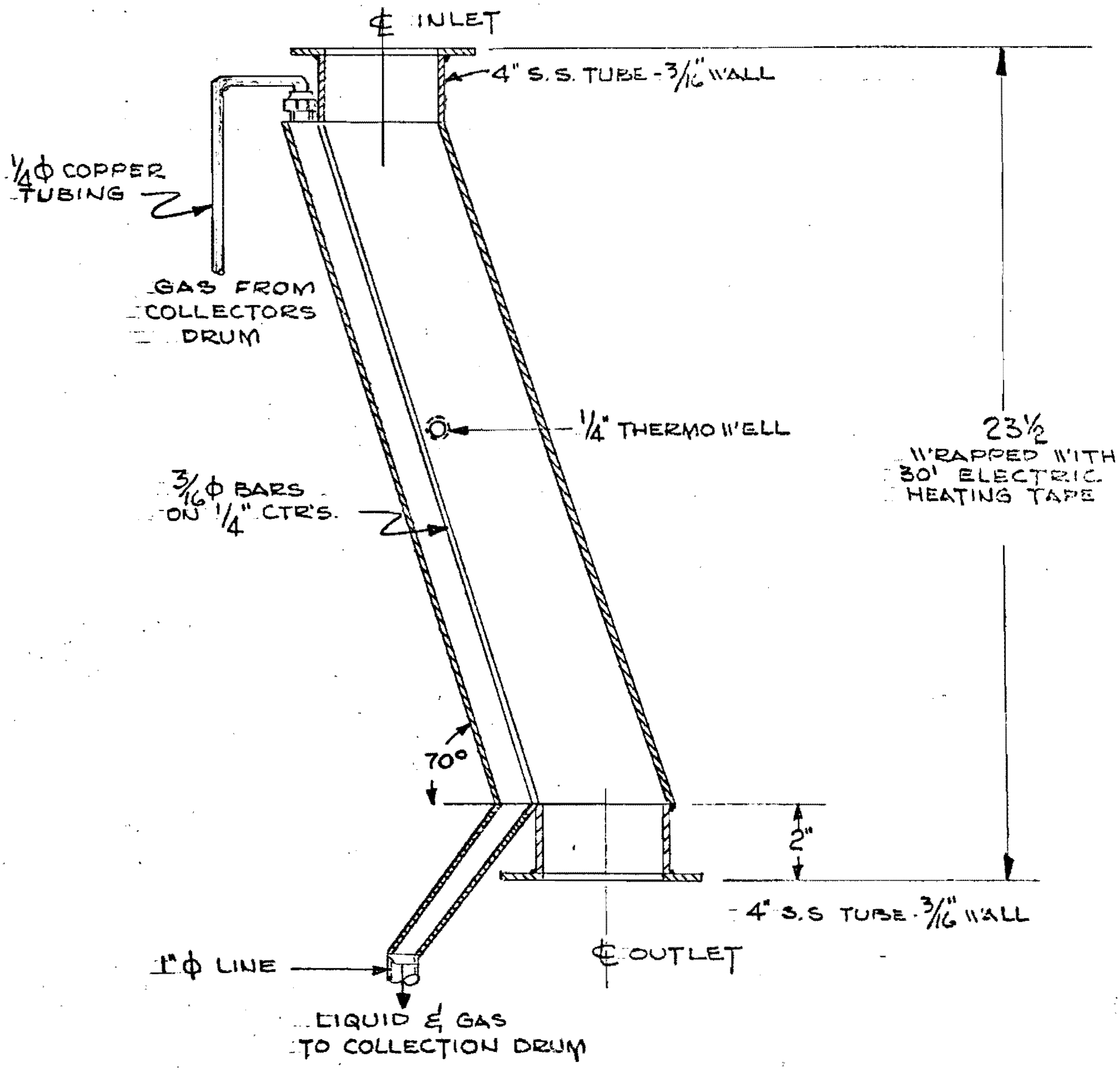


FIGURE 9



PLAN



SLOPED LIQUID DISENGAGER

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TABLE 5

OPERATING SUMMARY - 3.6-INCH I.D. MOVING BED RETORT

MBR3.6 Run Number	4	5	6	7	8	9	10	11
Operating Conditions:								
Raw Shale Rate lb/(hr)(ft^2)	930	930	450	930	450	930	500	500
Fischer Assay (gal/ton)	27.1	27.7	26.9	28.2	27.1	27.1	25.5	27.9
Nominal Size Range, (in)	←			1/4 to 1/2				→
Recycle (SCF/T)	20,000	20,000	20,000	20,000	20,000	20,000	20,000	20,000
Air (SCF/T)	0	0	0	0	0	0	5,000	0
Length of Run (hrs)	4	3 1/4	7	1 1/2	4	4 1/2	4	7 1/2
Was Disengager in use?	Yes(1)	Yes	Yes	No	No	Yes	Yes	Yes
Operating Data:								
Disengager Temperature (F)	640	550 - 650	600 - 650	--	--	600 - 700	600 - 700	600
ΔP. Top ("H_2O ft)	3.4	3.2	2.0	--	4.9	3.4	--	--
ΔP. Overall	3.0	4.0	1.9	4.5	3.3	3.2	--	--
Products Recovered:								
Oil Collected (vol % FA)	86.5	80.5	103.3	--	52.2	55.9	DISENGAGER LINE PLUGGED	95.4
Total oil in Disengager (vol %)	59.9	86.9	57.7	0	0	--		53.0
Total oil in Demister (vol %)	40.1	13.1	42.3	--	100	--		47.0
Gas Yield (SCF/T)(2)	208	387	565	--	--	--		--
Mineral CO_2 Dec. (% CO_2 chg'd)	5.4	5.4	6.6	--	--	--		--
Material Balances (as is)								
Ash (wt %)	95.5	95.0	98.5	--	--	96.9		98.3
Overall Balance (wt %)	96.1	97.3	98.8	--	--	103		96.2
Organic Carbon Balance (wt %)	80.5	89.0	91.6	--	--	--		--
Shale Oil Properties:								
Gravity (^{o}API) Composite	21.9	20.0	22.7	--	--	--	23.1	--
Disengager	16.9	18.2	15.9	--	--	13.4	--	12.0
Demister	29.5	32.1	33.1	--	24.0	29.1	23.1	27.3
Spent Shale:								
Fischer Assay (gal/ton)	0.9	4.6	0.2	--	--	8.4	--	0.0
Organic Carbon (wt %)	2.61	4.12	2.23	--	--	5.4	--	2.2
Overall Operational Performance	Very Good	Good	Excellent	Inop.	Inop.	Good	Fair	Excellent

(1)Oil drained into open bucket.
(2)Gas reported on nitrogen-free basis.

(Continued on next page)

OPERATING SUMMARY - 3.6-INCH I.D. MOVING BED RETORT

MBR3.6 Run Number	12	13	16	17	18	19	20	21
Operating Conditions:								
Raw Shale Rate lb/(hr)(ft^2)	500	500	300[1]	500	500	500	300	500
Fischer Assay (gal/ton)	27.8	28.2	29.1	28.6	29.4	28.2	37.7	51.5
Nominal Size Range (in)	← 1/4 to 1/2 →							
Recycle (SCF/T)	20,000 28,800	25,000	4,500	20,000	20,000	20,000	10,000	15,000
Air (SCF/T)	3,500 5,800	4,800	4,500	4,500	4,500	4,500	6,000	0
Length of Run (hrs)	7 1/2	7 1/2	12	7	7	7	9	2
Was Disengager in use?	Yes	Yes	No	Yes	Yes	Yes	Yes	Yes
Operating Data:								
Disengager Temperature (F)	550 - 650	600	--	600	500	400	500 - 600	600
$\triangle$P. Top ("H_2O ft)	--	--	--	--	--	--	--	--
$\triangle$P. Overall	--	--	--	--	--	1.7	1.7	--
Products Recovered:								
Oil Collected (vol % FA)	67.9	78.0	79	97.5	72.5	99.8	84.8	--
Total oil in Disengager (vol %)	70.3	--	0	54.5	69.6	56.8	47.5	--
Total oil in Demister (vol %)	29.7	--	100	45.5	30.4	43.2	52.5	--
Gas Yield (SCF/T)[2]	2,390	--	--	--	--	--	--	--
Mineral CO2 Dec. (% CO2 chg'd)					--	--	--	--
Material Balances (as is)								
Ash (wt %)	107	--	--	--	--	--	--	--
Overall Balance (wt %)	104	98.7	--	--	--	--	--	--
Organic Carbon Balance (wt %)	83.4	--	--	--	--	--	--	--
Shale Oil Properties:								
Gravity (°API) Composite	--	--	--	--	--	--	--	24.3
Disengager	15.1	15.1	--	15.2	16.0	14.0	12.9	--
Demister	26.2	26.8		24.7	23.7	29.3	27.9	24.3
Spent Shale:								
Fischer Assay (gal/ton)	0.7	0.0	0.0	0.0	0.0	0.5	2.4	--
Organic Carbon (wt %)			0.2					--
Overall Operational Performance	Fair	Excellent	Poor	Excellent	Poor[3]	Good	Poor	Inop.

(1) Could not operate at all above 300 mass rate
(2) Gas reported on nitrogen-free basis
(3) Considerable leakage around flanges

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4.6 gallons per ton was left in the spent shale. Actually, the 4.6 gallons per ton represents about 15% of the oil, which added to the 80.5 accounts for about 96%.

The gravity of the oil was 18 °API for the disengager and 32 °API for the demister.

Most of the oil was collected in the disengager because of the low temperatures in the furnace directly below it.

The overall pressure drops in both of these runs show incipient or actual flooding at this high mass rate. In any case, the pressure drops seems to stabilize at these levels - and the process was operable.

Run 6 was run at about 450 lb/(hr)(ft^2) shale rate. This was an excellent run which lasted seven hours, until the shale ran out. The yield was 103 vol % Fischer Assay. Run 11 was a check on this run and was made at essentially the same conditions (except that the shale rate was 500 lb/(hr)(ft^2)). Run 11 gave a yield of 95.4%; the average of the two runs is 99%.

The product split for both of these runs was similar; an average of 55% of the oil into the disengager and 45% into the demister.

Runs 7 and 8 were attempted in the straight pipe retort without liquid removal, at about the same conditions as Runs 5 and 6. Both 7 and 8 were aborted because of agglomeration which led to increased pressure drop and gas flow restriction. Some oil collected overhead from Run 8 had an API Gravity of 24.0, the same as Fischer Assay oil. The gas from the process also was very similar in composition to the gas from the Fischer Assay.

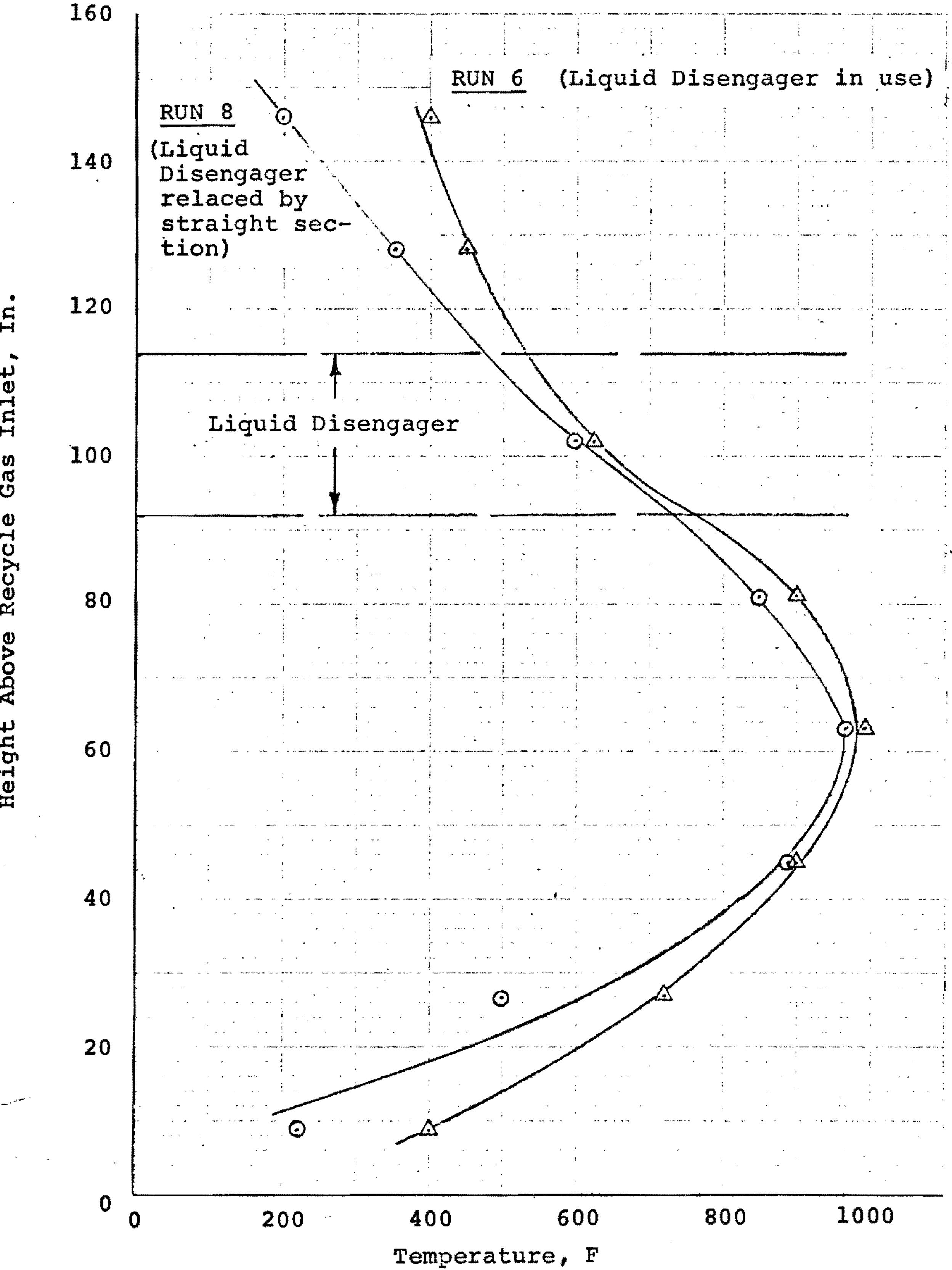
Temperature profiles for Runs 6 and 8, runs at 500 lb/(hr)(ft^2) shale rate, with and without the disengager, respectively, are shown in Figure 10. The residence-time above 700 F was about 40 minutes; this is sufficient to retort the shale completely.

Run 9 was an attempt to repeat Run 5, but with higher temperatures for better retorting. Operation was good until temperatures at the bottom got so hot that the bottom rubber seals melted. Retorting was even worse than in Run 5.

FIGURE 10

TYPICAL TEMPERATURE PROFILES FOR 3.6-INCH I.D. RETORT

450 lb/(hr)(ft^2) Shale Rate
20,000 SCF/T Recycle Gas Rate
No Combustion In Bed



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Run 10 was the first attempt to operate with combustion in the bed. Air at about 5000 SCF/T was mixed with recycle gas at the bottom of the bed. In this run, the disengager was found to be plugged; consequently, no oil was disengaged. An agglomerate formed in the bed above the disengager.

Run 11 was described above.

In Run 12, an attempt was made to operate the retort at 500 lb/(hr)(ft^2) shale rate, 20,000 SCF/T recycle gas rate, and 5000 SCF/T air. The oil disengager was to be at 600 F. Air was mixed with recycle gas and introduced at the bottom of the retort. The gas rate was increased and, finally, the shale rate was decreased to prevent the combustion zone from moving down out of the bed. Operation was only fair. The bed was flooded at times, and the product recovery system was not operating well for this run. An agglomerate, probably formed at the very end of the run, was found above the disengager section.

Before Run 13, the packing of the demister drum was improved for better product recovery. Run 13 was planned as a virtual repeat of Run 12, but with better control and better product collection. Operation of the retort during this run was excellent. However, as in the previous run, mixing air with recycle gas at the bottom of the retort caused the combustion zone to move down the bed. Increasing recycle rate to about 27,000 SCF/T, in an attempt to move heat up the bed, caused flooding.

After Run 13, three air distributor pipes, 120 degrees apart, were added about 40 inches below the bottom flange of the disengager. They protruded about 1/4-inch into the shale bed. Provisions were made for piping air and/or recycle gas into these air distributors.

Run 14 was an attempt to run without a disengager. This run was intended to find out if there were conditions at which a stable operation could be achieved in this retort when it was operated in the conventional manner. Startup was made with external heating, 300 lb/(hr)(ft^2) shale rate and 15,000 SCF/T recycle rate. Retorting took place in two intermediate furnaces, maintained at 900 F and 1000 F, respectively. About three hours after startup, when shale was probably down to the lower part of the retort, movement of the shale became more sluggish. Walnut-size agglomerates began to show up at the bottom sight-glass as the shale got harder to move. Flooding was indicated by increased pressure drop across the bed. Finally, the shale agglomerated to the point where it could not be moved, aborting the run.

In Run 15, another attempt to run the retort without the disengager was made. To decrease the chance of flooding, the recycle rate was held at 10,000 SCF/T, the temperature at the top of the retort was increased, and the bed height was effectively lowered by moving the retorting zone up into the upper part of the retort. The shale rate was held at 300 lb/(hr)(ft^2).

About three hours after startup (similar to Run 14), increasing temperatures in the two top furnaces indicated flooding. Liquid product splashed overhead and flowed into the demister. Shale flow became more difficult and finally stopped, aborting the run.

Run 16 was a final attempt to operate without the disengager - by any means possible. The run was started with external heating, at 300 lb/(hr)(ft^2) shale rate and 8000 SCF/T recycle rate. The retorting zone which was moved into the upper part of the retort was held at 1100 F. Shale flow soon became poor. About two hours after the start of the run, mist at the demister sight-glass cleared, indicating flooding. About one-half hour later, shale movement stopped and could not be started again.

Air was introduced via the air distributors in the hope of burning the agglomerate loose. After about two hours, during which the combustion zone moved from the middle to the top of the retort, heavy hammering broke the agglomerate and allowed shale movement.

The run was continued with about 4500 SCF/T each of air and recycle gas, both admitted to the air distributors. Retorting was maintained at the top of the bed. The run continued for another eight hours under these conditions - until the shale ran out. Shale flow was poor during this time and temperatures were hard to control. The spent shale was extremely well burned, containing only 0.2% organic carbon. Operation was generally poor. Yield was only about 80%.

Run 17 was run at conditions similar to Run 13, except that air was engaged at the air distributors instead of at the bottom of the bed. Conditions were: 500 lb/(hr)(ft^2) shale rate, 20,000 SCF/T recycle, and 4500 SCF/T air. The air and 4500 SCF/T of the recycle gas were mixed before the air distributors, making this, essentially, a dilution gas operation to minimize the possibility of clinker formation where the air was engaged.

In this run also, liquid product was noticed running into the demister drum. Mist was usually sparse during the run. However, operability was excellent, with smooth shale flow.

The yield of 98% with combustion was comparable to those for other disengager runs without combustion (Runs 6 and 11).

Run 18 was an effort to operate with a lower disengager temperature. Although this was a complete run, the overall operability was poor. A large amount of oil, as mist, was lost because of severe leaks around flanges on the retort. Therefore, results on this run are suspect.

Run 19 was begun after repairs had been completed on the retort flanges. The run was a second attempt at operating with a lower disengager temperature with other conditions the same as in other successful disengager runs: 500 lb/(hr)(ft^2) shale rate, 20,000 SCF/T recycle rate, and 4500 SCF/T air (mixed 1:1 with part of the recycle).

It was very difficult to maintain the disengager temperature at 450 F; the temperature varied between 300 and 550, averaging about 400 F. The overall ΔP remained at about 1.7 inches of mercury during the run. Very little mist was noted.

Shale movement was erratic and required frequent hammering to maintain it. The shale seemed to be hindered at the bottom of the disengager. The operability, nevertheless, appeared fair to good during the seven-hour run.

The yield was excellent at 100%. The product split was a typical 57% to 43% in favor of the disengager.

In Run 20, the purpose was to operate the retort with 37.7 gallon per ton shale at 500 lb/(hr)(ft^2) shale rate and 30,000 SCF/T recycle, with the disengager. The air distributors had been moved up about 18 inches for better heat distribution.

The retort was difficult to operate from the inception of the run. The disengager furnace could not be maintained at the desired temperature of 600 F. (The heating element in this furnace failed near the end of the run.)

Constant and violent hammering were necessary to keep shale moving. Apparently, the greater tendency of rich shale to agglomerate, accounted for the poorer shale flow.

The shale rate was reduced to 300 lb/(hr)(ft^2), and 6000 SCF/T air, mixed with recycle, was added through the air distributors. The total recycle rate was dropped to 10,000 SCF/T. Shale flow was still poor, hanging up either in the furnace below the disengager or in the disengager itself.

The overall pressure drop held at about 1.7 inches of mercury throughout the run, which lasted for about nine hours.

Operability was judged poor.

Run 21 was an attempt to run rich shale (51.5 gallons per ton) with external heating at 500 mass rate and 20,000 recycle and the disengager at 600 F. (The shale was analyzed after the fact, and was richer than desired.)

Good shale flow was noted for about an hour after shale first showed in the sight-glass at the top of the retort. That would put the shale at about the top quarter of the furnace directly below the disengager. At that time, shale flow became very bad and, only ten minutes later, flow stopped and could not be started again. Air was admitted to the unit in an attempt to free the shale, but this failed. When the retort was cooled down, a hard, massive agglomerate was found to fill it from Furnace 2 down through 6. No oil at all was found in the disengager, which indicates that it might have been blocked near the beginning of the operation.

D. Retort No. 3 Studies - Conventional Operation

A group of studies was carried out in Retort No. 3 while it was operating in the conventional manner, to establish a liquid loading profile in the vertical direction. The results were inconsistent and no firm conclusion could be drawn as to the liquid loading. The devices used to measure liquid loading were a tar camera, a cyclone, a cascade jet impactor, and sample probes intended to remove liquid from the bed. As a matter of record, these tests and test results will be briefly described.

1. Tar Camera Test

A known volume of mist laden gas was passed through a white sheet of filter paper mounted in the tar camera. Relative mist loading in the gas was determined by visual comparison of the discolored filter paper for the four sample tap locations. These locations are shown in Figure 11. This test indicated heavier mist loadings or larger particle sizes at sample taps 1.75 and 3.0 feet above the air inlet when compared to samples taken at taps 1.0 to 4.0 feet above the air inlet. This is shown in the following tabulation:

Height Above Air Inlet, Ft	Relative Loading	Mist Color
1.0*	Light	White
1.75	Heavy	Yellow
3.0	Heavy	Yellow
4.0	Moderate	Light Yellow

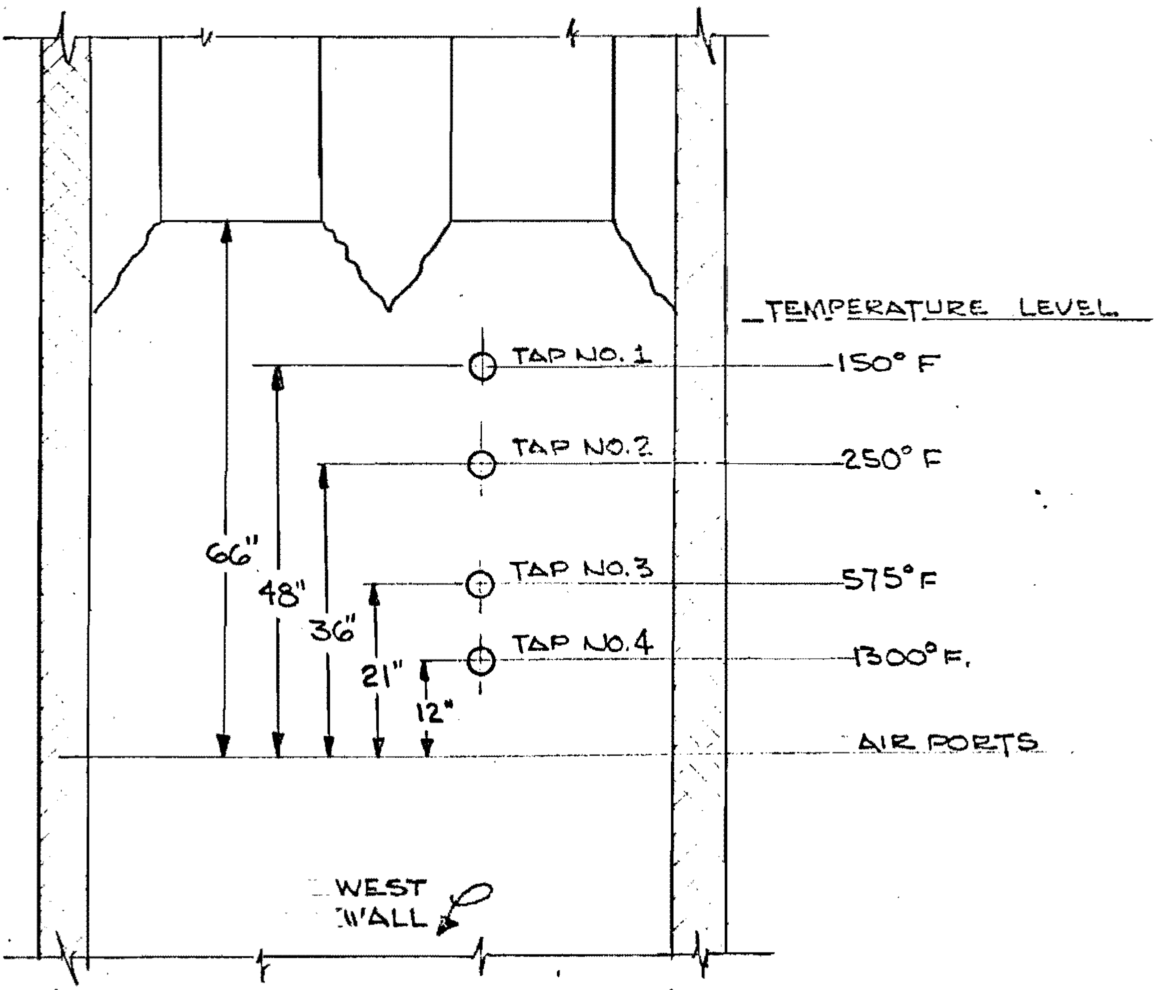
*No internal condensation is anticipated at this level. Rapid atmospheric quench is probably responsible for the white mist and the small amount of liquid present.

2. Cyclone Test

A two-inch cyclone was used to separate mist from mist laden gas flowing through the cyclone at about 30 SCFH. The liquid collection rates at each of the four taps are indicated in the table which follows. Again, higher loadings can be inferred from the higher liquid collection rates at the taps located 1.75 and 3.0 feet above the air inlet as compared to the liquid collection rates at 1.0 and 4.0 feet above the air inlet. Both this test and the tar camera tests were run during a 12-hour period when the retort was operating continuously.

FIGURE 11

LOCATION OF SAMPLE TAPS ON WEST END OF RETORT NO.3



Height Above Air Inlet, Ft	Liquid Loading, $\frac{lb}{MSCF}$ (Measured with Cyclone)	Specific Gravity of Recovered Liquid, ^{o}API
1.0*	0.2	--
1.75	0.8	18.0
3.0	1.4	18.4
4.0	0.1	--

*No internal condensation is anticipated at this level.

3. Cascade Jet Impactor Test

The cascade jet impactor tests were carried out at a later date than either the tar camera or the cyclone tests so it might be anticipated that, even using the same sample tap location and the same shale size, somewhat different results would be obtained. The results are, in fact, quite different. The curious part of this study is that substantial agreement was obtained between this data obtained with 1/4 to 1 inch shale using a horizontal sample probe and earlier data obtained in Retort No. 3 with 1 to 2 1/2 inch shale using a vertical probe. This is shown in the following table:

Height Above Air Inlet, Ft	Mean Mass Particle Diameter, μ	Geometric Standard Deviation	Liquid Loading, $\frac{lb}{MSCF}$
	1/4 to 1 Inch Shale (Horizontal Probe		
1.75	2.4	1.5	6.6
4.0	2.5	1.7	7.4
	1 to 2 1/2 Inch Shale (Vertical Probe)		
7.5	2.0	1.6	6.5
12.5	2.0	1.6	7.1

The cascade jet impactor data are inconsistent with the tar camera and cyclone test data. If a heavier loading or larger mist size does exist at 1.75 to 3 feet above the air inlet, there is an inherent limitation in the cascade jet impactor which clouds the results. One such limitation might be the inability of large mist particles to travel through the sampling tubing into the cascade jet impactor. A thorough study of the cascade jet impactor performance characteristics would be needed to resolve the inconsistency of these data.

4. Sample Probes Intended to Remove Liquid From the Shale Bed

Several attempts have been made to remove liquid from the shale bed of Retort No. 3 with sampling probes. A number of these experiments were made using a vertical probe. This probe is shown in Figure 12. The probe was to be operated by allowing gas and oil to flow into the oil sump in the lower end of the 2-inch pipe. The gas and liquid were to separate in the 2-inch pipe. Gas was to flow upward through the pipe and leave through the exit port. Oil was to collect in the oil sump until its level reached some point above the bottom of the interior 3/8-inch tube but below the inlet holes to the 2-inch pipe. Oil was to be pumped from this sump as long as a liquid seal could be maintained at the lower end of the 3/8-inch tube. When the seal was broken, pumping was to be interrupted.

When the probe was operated in this fashion, no liquid was removed from the shale bed over a range of elevations with respect to the air inlet with either 1/4 to 1 or 1/2 to 2 1/2 inch shale. In one instance, it was noted that the outer surface of the probe was coated with oil after one of these attempts.

On other occasions, the probe has been operated improperly so that a liquid seal was not maintained at the lower end of the 3/8-inch tube and it was erroneously concluded that oil was removed from the bed. This apparent removal of oil has been shown to be due to impaction of mist from mist laden gas as it flowed up the 3/8-inch tube.

Attempts have also been made to remove liquid from the bed with horizontal sampling probes mounted both flush to the retort wall and projecting three inches into the shale bed using various vertical locations. In no case was liquid oil removed from one of these taps.

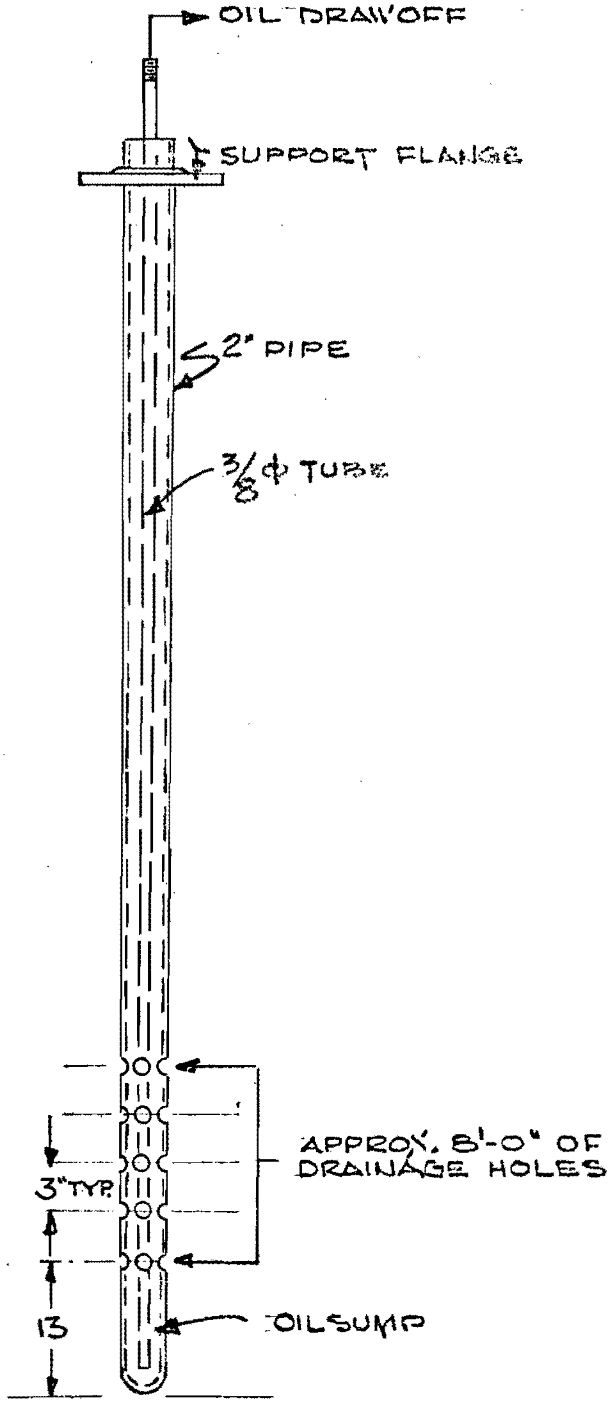
It has been concluded that these observations do not disprove the role of liquid in agglomerate formation; rather than that, it is felt that they emphasize the localized nature of the problem when marginal retort operability has been achieved.

E. Alternate Schemes for Preventing Localized Liquid Accumulation in Retort No. 3

A number of schemes for reducing localized liquid accumulation in Retort No. 3 were considered. These include:

FIGURE 12

SAMPLE PROBE TO REMOVE LIQUID FROM BED OF RETORT NO.3



-- Removing hot mist laden gas from the retort and circulating it with a hot blower through a mist recovery system to lower the mist loading and then returning the gas to the retort to preheat the shale.

-- Removing hot dry gas from the retort before any mist condensation occurs and condensing the oil externally, then reheating the cold dry gas and returning it to the bed to preheat the shale.

-- Disengaging oil from the bed of shale using specially designed hardware located within the retort.

In reviewing the possible alternatives in the light of project budgets of time and money, and ultimate process economics, it was decided that the third alternative was the most attractive. The first alternative required the development of a reliable hot gas blower that was capable of sustained operation under serious fouling conditions. This was impractical within the constraints of this project. The second alternative requires a large heat transfer facility. A preliminary research guidance economic study indicated that this was an economically unattractive approach.

F. Model Studies in Preparation for the Installation of a Liquid Disengager in Retort No. 3

1. Shale Flow Studies

A basic question that had to be answered early in this development was whether shale could flow through an array of hardware installed in the retort for disengaging liquid. An arrangement that had proven successful for handling a wide variety of shale sizes and which appeared to have some desirable features as a key element of a liquid disengaging system was the single-level drawoff assembly.

Three experiments were made to evaluate shale flow through the one-sixth scale plexiglass model of Retort No. 3 with a single-level drawoff assembly installed above the air distributor. The tests illustrated in Figures 13 and 14 were made with dry and water-wet shale and illustrate flow patterns to be expected with such hardware. The dry shale drawdown, Figure 13, was very even. The effect of coning both above and below the intermediate deck is clearly apparent. The water-wet shale drawdown, Figure 14, was somewhat erratic. However, most of the imbalance developed early in the drawdown and is probably related to filling the unit. After this water-wet shale drawdown, another was made with shale soaked with No. 1 fuel oil. The oil-soaked shale

FIGURE 13

"EMPTY BIN" SHALE FLOW THROUGH SINGLE LEVEL DRAWOFF ASSEMBLY WITH DRY SHALE

1/6 Scale Model 1/4 to 1 Inch Shale

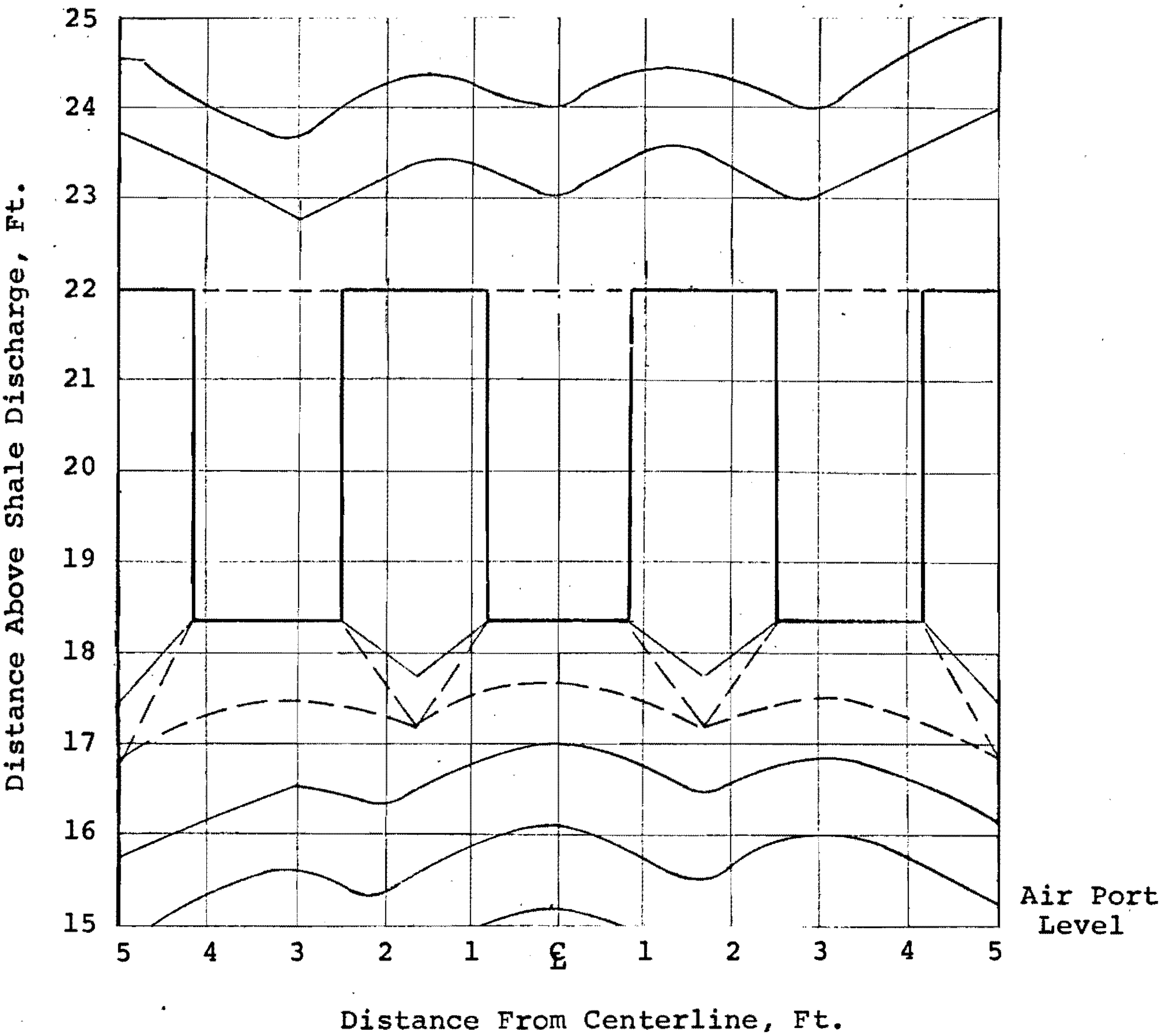


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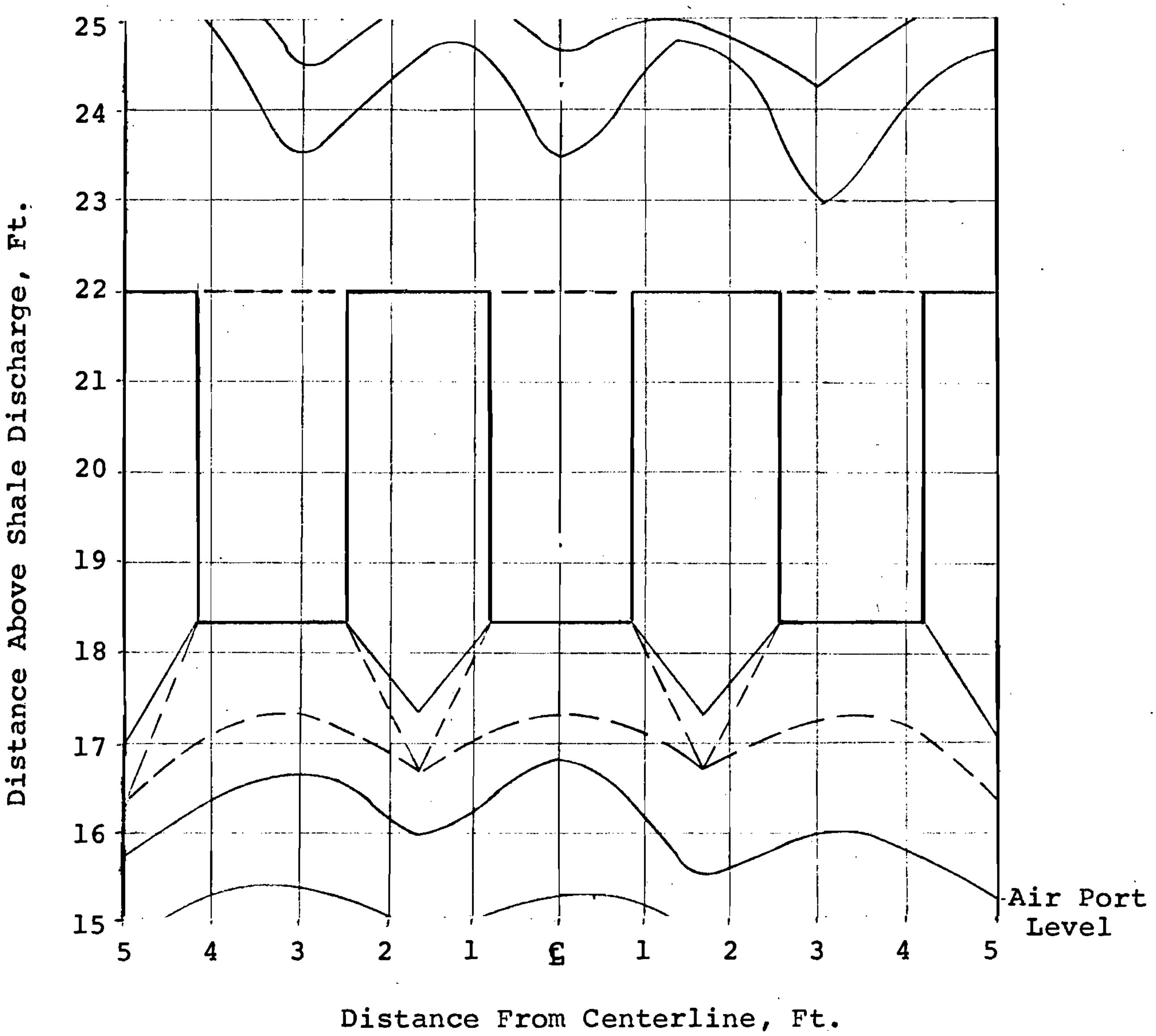
FIGURE 14

"EMPTY BIN" SHALE FLOW THROUGH SINGLE LEVEL DRAWOFF ASSEMBLY WITH WET SHALE

1/6 Scale Model 1/4 to 1 Inch Shale



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gave a somewhat smoother result than the water-soaked shale, but coning patterns were about the same as obtained with water-wet shale. It is probable, because of the increased surface area of the scaled 1/4 to 1 inch shale and the physical characteristics of the liquids, that both the water-soaked shale and the oil-soaked shale present more severe flow problems than actual 1/4 to 1 inch shale in a full-scale retort. This model data indicate that an intermediate-deck system will smoothly pass shale.

2. Conceptual Design of a Liquid Disengaging System for Retort No. 3

Since smooth shale flow through a single-level drawoff assembly could be expected with wet shale, it was decided to proceed with the development of a device using this assembly as a basic design feature.

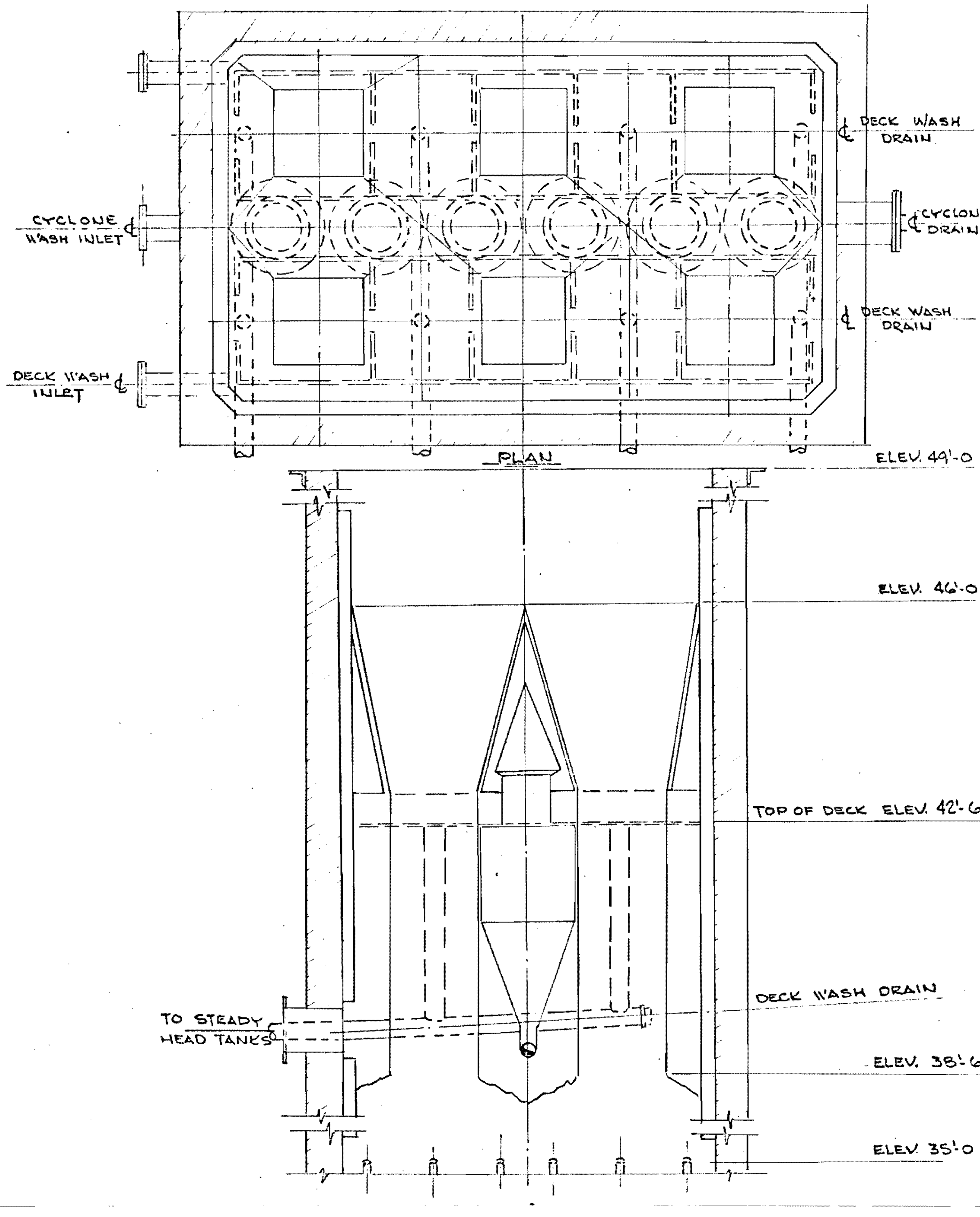
Briefly, the device which was developed, consisted of a modified single-level drawoff assembly installed above the air distributor with slotted, inclined grates mounted on the deck. As shale moved down through the retort, it was to flow over the inclined grates. Liquid oil was to drain through the grates and be collected on the deck of the single-level drawoff assembly. Gas, from the shale-gas disengagement zone created by the shale chutes, was to flow up through six cyclones, which were intended to remove oil mist and dust. Some gas would also flow up through the shale chutes; the gas split between the cyclones and the shale chutes was to be determined by the pressure balance which would occur between these parallel flow paths. The development studies that were investigated with model techniques involved the design of the cyclone and the shale chutes. The studies were full-scale model pressure drop evaluations of each element (Reference 2). The design that was developed from this concept and these studies is shown schematically in Figure 15.

G. Retort No. 3 Studies - Liquid Disengager Operations

A group of six runs (C1053 through C1058) were made in Retort No. 3 with various versions of the liquid disengager shown schematically in Figure 15 using 1/4 to 1 inch shale. The objective of these runs was to apply the principle of liquid disengagement in Retort No. 3. It was not anticipated that a commercial application of this principle could be demonstrated after a few weeks of experimentation, however. The device tested was not intended to be a scaled-up version of what had been studied in the small retorts; it was, rather, an application of the principle of liquid disengagement in a

FIGURE 15

LIQUID DISENGAGER RETORT INTERNALS



large scale retort. None of these runs was completely satisfactory. Although improved operability was realized in some of these runs, as indicated by the higher shale and gas rates that were achieved, considerable evidence of channeling of gas, liquid and shale generally existed, and lower yields for this shale size than were obtained in Retort No. 2 or in Retort No. 3 without the liquid disengager, were observed. Considerable operating difficulty was experienced in some of these runs because of plugging of either the inclined grates or the deck drain lines with chips and dust from the shale, and coke-like deposits. Each run will be reviewed separately but in a brief form. References to The Monthly Progress Memoranda will be given in each case so that a more detailed discussion of each run will be readily available. These runs are summarized in Table 6.

1. Run C1053 (Reference 3)

This run was the initial evaluation of the liquid disengaging concept in Retort No. 3. Startup with 1/4 to 1 inch shale proceeded smoothly and the unit was lining out at the 500 lb/(hr)(ft^2) shale rate when a bearing on the recycle blower motor failed. The unit had operated at 500 lb/(hr)(ft^2) shale, 4300 SCF/T air and 15,200 SCF/T recycle rate for about three hours when the emergency shutdown occurred. These were conditions at which even Retort No. 2 was inoperable. During the last eight hours of operation, an estimated yield of 85% Fischer Assay was achieved. The shutdown was particularly severe; all gas flows were shut off and hot shale was drawn from the unit under a steam blanket. No clinker was found, even though a fairly complex 54 bayonet air distributor had been in use.

The disengaging system did not remove as much oil from the shale bed as had been expected from the 3.6-inch I.D. retort studies. About 7 to 10% of the oil was recovered from the disengager during this run in Retort No. 3 compared to 50 to 60% usually recovered from the disengager with the 3.6-inch I.D. retort. This may have been due to a number of factors. The inclined grates which were made of 3/16 by 1 inch bars, were badly plugged with shale chips and dust. Secondly, the mist size may have been smaller in Retort No. 3 because of a higher cooling rate in the initial condensation region. The fact that the temperature at the deck was lower than desired would probably not decrease the disengagement of liquid by this device but it would probably have had an unfavorable effect on operability if the run had not been interrupted by an emergency shutdown.

TABLE 6

RETORT NO. 3 OPERATIONS WITH LIQUID DISENGAGER

Target Run Conditions	Remarks	Conclusions
Conventional 700/900 F startup at 300 lb/(hr)(ft^2) shale rate. Move directly to 500 lb/(hr)(ft^2) shale rate at Air - 4,300 SCF/T (cold) Recycle - 15,000 SCF/T	Smooth fast startup - early operation looked good until bearing failure on recycle motor terminated run after 18 hours. Liquid production from grates and cyclones averaged about 7.5% of total oil production compared to 50% in bench scale unit. Possible reasons: Grates severely plugged and liquid disengager temperature only 350 to 400 F rather than desired 600 F - no clinker. Preliminary data indicated yield level was 85% vol during startup and spent shale was free of oil.	•Inconclusive but encouraging. •Unit operated at 15,000 SCF/T recycle which is highest yet for any shale size. •Unit operated at 500 mass rate - a first in Retort No. 3 with 1/4 to 1 inch shale. •Unit operated with a 54 bayonet distributor which was unsuccessful previously.
Same as above.	Startup smooth but slow - liquid production at a low level (2 to 3% of total) from outset. Liquid disengager temperature still low - no change from C1053 - attributed to gas channeling. Yields started at the 81% level and deteriorated with time - corresponding increase of oil in spent shale - unit shut down because of low yields and liquid production rather than operability problems. Small clinker present - grates severely plugged.	•Bed height reduction aggravated gas channeling. •Improved grate design necessary to reduce plugging tendency. •More instrumentation necessary for improved understanding.
Same as above.	Startup smooth but slow as in C1054. Lateral temperatures in combustion zone more uniform in early stages. Temperatures in liquid disengager raised to 450 to 600 F level from 350 to 450 F. No measurable oil recovered from grid or cyclones from outset - high oil in spent shale	•Modified chute design has made bed temperatures more uniform. •Modified grid design improved plugging problem although there were considerable shale chips on the deck. •Oil recovery from disengaging section did not improve even

(Continued on next page)

Run No	Hardware Configuration
C1053	Installed grates to drain liquid from condensing zone. Installed internal cyclones to reduce mist loading leaving retorting zone. Installed 54 bayonet air distributor.
C1054	Same as above but reduced retorting bed height six inches to raise liquid disengaging temperature.
C1055	Same general configuration as C1054. Revised grate design to reduce plugging problem. Separated gravity liquid drain from cyclone system to pin point oil

TABLE 6 (CONTINUED)

RETORT NO. 3 OPERATIONS WITH LIQUID DISENGAGER

Target Run Conditions	Remarks	Conclusions
	(about 5 gallons per ton) throughout operation - increased severity in attempt to clean up spent shale - operation deteriorated quickly and unit was shut down with clinkers about 36 hours after startup.	though temperature was raised to level which was effective in bench scale unit.
Conventional 700/900 F startup at 300 lb/(hr)(ft^2) shale rate. Held 300 lb/(hr)(ft^2) shale rate, 5000 SCF/T air and 12,000 SCF/T recycle to give low liquid disengager temperature.	Non-symmetric temperature pattern developed about 6 hours after line burner reached 900 F. Suspected clinker formation 7 hours after line burner reached 900 F. Shutdown with clinker after 16 hours. No significant oil disengagement.	Failure followed usual 1/4 to 1 inch shale failure without oil disengagement.
Conventional 700/900 F startup at 300 lb/(hr)(ft^2) shale rate. Held 300 lb/(hr)(ft^2) shale rate, 15,000 SCF/T recycle in order to raise liquid disengager temperature and to keep liquid from running down downcomers.	Non-symmetric temperature pattern developed about 5 hours after line burner reached 900 F. Suspected clinker formation 6 hours after line burner reached 900 F. Unit continued to run stably for some time, however. Had to increase recycle to 18,100 SCF/T to raise temperature to 530 F. Gas channeling suspected. Disengaged 8 to 10% of oil. Shut down with clinker.	Similar failure to C1056 in spite of oil disengagement. Suspected that oil accumulated below deck during startup.
Same as C1057 except that it was not expected that high liquid disengager temperatures would be reached because of location of disengager with respect to air inlet.	This equipment arrangement was intended to prevent liquid accumulation at the top of the bed. The irrelevance of this was indicated by early clinkering. 2% of oil was disengaged.	Because of low disengager temperature, significant oil accumulated below the deck. This led to agglomeration and clinkering in the usual fashion.

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Run No	Hardware Configuration
C1055	production. Modified chute design to provide a more level shale bed in retorting zone (reduce gas channeling).
C1056	Same general configuration as C1055, but gas leaving cyclones was not allowed to flow through inclined gratings. It flowed through bypass lines to transverse distribution headers.
C1057	Removed cyclones and blanked 4 of 6 cyclone discharges. Installed dampers in two so that some gas could bypass downcomers. Removed crowsfoot baffles. Adjusted downcomer length to same length as C1053. Shut off deck wash.
C1058	Shortened downcomers so that distance between bottom of downcomers and air inlet was about equal to usual bed height above air distributor. Hats installed on bottom of downcomers.

2. Run C1054 (Reference 4)

After repairing the recycle blower motor, Retort No. 3 was again started up with 1/4 to 1 inch shale. The only significant mechanical modification during the interim between C1053 and this run was that the shale chutes were lengthened 6 inches so that the distance between the bottom of the shale chutes and the air inlet was 38 inches where it had been 44 inches. This change was intended to increase the deck temperature to 600 F. The unit operated for 63 hours from startup when it was shut down, under control, for mechanical modification. The lined out conditions reached were 500 lb/(hr)(ft^2) shale, 4300 to 4700 SCF/T air and 15,000 SCF/T recycle. About a day before shutdown, the shale drawoff system was shut down for 40 minutes. The unit recovered from this event and continued operating until shutdown. A single, small clinker was found wedged between adjacent bayonet manifolds when the shutdown inspection was made.

Temperatures below the liquid removal deck continued to be lower than desired during this run. About 400 F was generally observed while 600 F was the target temperature. This resulted in the accumulation of some oil below the deck. A sample of this oil had a 15.4 ^{o}API specific gravity and a 95 F pour point.

Yields which were lower than desired were obtained in the material balance periods of this run. This appears to be partially due to a low retorting zone residence time. Evidences of inefficient contacting of shale and gas were also apparent.

Lower recovery of oil from the disengager in this run was also observed. About 2 to 5% of the oil was recovered from the disengager in this run compared to 7 to 10% in the previous run.

The problem of plugging of the inclined grates and the deck drain system persisted in this run.

3. Run C1055 (Reference 5)

Following Run C1054, several significant modifications were made to the retort. These were:

1. Installation of an improved liquid withdrawal grid to reduce plugging.
2. Modification of the shale chutes to reduce coning patterns and therefore, gas channeling.
3. Modification of the oil wash system to allow determination of whether oil was collected by the collection deck or the cyclones.

4. Installation of temperature indicators in the inlet to each cyclone.

The first of these changes was implemented by installing round 3/16-inch rods as the component parts of the inclined grid in place of the 3/16 by 1-inch bars used previously. The desired effect of reduced plugging was achieved, although, more chips appeared to fall through the grate than previously.

The second change consisted of installing a "crowsfoot" baffle on the bottom of the shale chutes to obtain a more effective retorting zone by reducing extremes of the coning pattern. Although cold shale flow was good with these baffles, when the unit was shutdown, two were plugged with carbonaceous agglomerates. This indicates that high local liquid concentrations existed and these baffles acted as anchor points for the resulting cohesive mass. In retrospect, they appear to have been an undesirable modification of the liquid disengager.

Higher deck temperatures were reported for this run, however, it is not clear whether this was truly a temperature increase or a change in measurement method due to the installation of thermocouples in the inlet to each cyclone. In any event, no liquid was disengaged in this run.

Operation was poorer in this run than in the preceding two runs as indicated by the even higher spent shale assay (about 5 gallons per ton) at the same conditions of 500 lb/(hr)(ft^2) shale, 4300 SCF/T air and 15,000 SCF/T recycle. When air rate was increased to increase the retorting severity, the operation deteriorated, and clinkering soon occurred.

4. Run C1056 (Reference 6)

Based on a concurrent mechanical model study, the method used to re-engage gas with the shale bed after passing through the deck was modified. This study indicated that it was difficult to counterflow oil and gas through the inclined grates. Fine particles of shale were suspended by the gas and adhered to the underside of the inclined grate. This coating was an additional hindrance to oil flow. Bypasses around the grates were installed so that these effects could be minimized. The modifications made to the liquid disengager was to install transverse gas distributors at the upper part of the inclined grates. The six cyclones were discharged into a common plenum chamber. Six inverted U channels, one over each of the shale chutes, were opened to the plenum to serve as the transverse gas distributors.

The retort was again started up with 1/4 to 1 inch shale and brought to conditions of 300 lb/(hr)(ft^2) shale, 5000 SCF/T air and 12,000 SCF/T recycle. The unit ran, in a ragged fashion, for less than a day and was shut down with a clinker on the west side. Only small amounts of oil were recovered with the disengager. The inclined grates were relatively clean indicating that rerouting the gas to bypass the grates had been a beneficial change. One of the shale chutes was plugged with an agglomerate in this run. This indicates that liquid was again channeling down part of the liquid disengager that was to be relatively liquid free. Apparently, the crowsfoot baffle at the lower end of the chute was a complicating factor.

5. Run C1057 (Reference 7)

All of the changes that were made since C1053 were in the direction of reducing gas flow up the shale chutes, and, therefore, increasing the tendency towards liquid flow down the chutes. At the same time, results had deteriorated since C1053. Oil yields dropped, liquid removal from the bed was reduced, unretorted oil in the spent shale increased and operability suffered.

After Run C1053, it had been postulated that most of the oil that was recovered by the disengager was removed as mist by the cyclones from the low temperature (400 F) gas that entered the disengager. No direct measurement of the split between the oil removed by the inclined grates and the oil removed by the cyclones was possible in Run C1053. Prior to Run C1055, facilities for separately measuring the oil collected through the grates and with the cyclones were installed. Run C1055 was run with relatively high temperatures beneath the disengager deck. During Run C1056, gas rates were adjusted so that the same gas temperature entering the disengager was achieved as had been achieved in Run C1053. Very little oil was recovered from either the cyclone or the deck drain system, indicating that this hypothesis was incorrect. Further, operability was adversely affected, therefore, low gas temperatures entering the disengager do not appear to be desirable.

Prior to Run C1057, Retort No. 3 was modified to force a substantial amount of the gas up the shale chutes. At the same time the cyclones were removed. By-pass lines and valves were installed so that pressure drop across the liquid disengager could be controlled independently of gas rate. The crowsfoot baffles installed prior to Run C1055 were also removed and the 44-inch distance from the bottom of the chute to the air inlet was re-established. Retort No. 3 was started up with 1/4 to 1 inch shale at a shale rate of 300 lb/(hr)(ft^2) and gas rates of 5000 SCF/T air and 15,000 SCF/T recycle. The pressure drop across the deck was held at 14 inches H_2O and higher in order to insure that no liquid would drain down the shale chutes and into the retorting zone. During the course of the operation, the recycle rate was increased from 15,000 SCF/T to 18,100 SCF/T. (Air rates were reduced from 5000 to 4300 SCF/T.) This was effective in increasing the liquid disengaging temperature from 350 F to 530 F and reducing the oil content of the spent shale from six gallons per ton to two gallons per ton. However, the total liquid product rate and the percentage of the liquid product recovered by the disengager did not change materially during this period. The amount of liquid removed averaged 8 1/2% during the pretest period and held at this level in the next balance period. After 36 hours of operation, high temperatures were observed in the combustion zone and the unit was shut down shortly thereafter with a large clinker. There was a similar temperature peak early in the startup and there is evidence that a small clinker may have been formed at this time. In spite of this, the unit operated quite smoothly until the terminal upset.

6. Run C1058

A somewhat different approach was used in Run C1058 to test the liquid oil removal system in Retort No. 3. This technique was to prevent liquid accumulation at the top of the shale bed. (Heretofore, our efforts were directed toward disengaging liquid from intermediate levels in the condensing zone.) This attempt was unsuccessful and the unit was shut down after two days of operation. A very small portion of the total liquid product (roughly 2%) was recovered in the disengaging system. A second, concurrent mechanical model study indicated that liquid might accumulate at the top of the bed at shale rates in excess of 400 lb/(hr)(ft^2) and total gas rates of 20,000 SCF/T. Therefore, the retort was modified to obtain a conventional six feet of bed above the air distributors. Pyramidal hats were installed at the top of the shale bed to facilitate liquid entrainment into the disengaging system. In addition, the retort was started up with 1/4 to 1 inch at a shale rate of 400 lb/(hr)(ft^2) while holding gas rates at a high level (4500 SCF/T air and 15,700 SCF/T recycle) and an early transition to 500 lb/(hr)(ft^2) was made.

A shale bridge occurred early in the startup and there is evidence that some clinkering resulted at this time.

From the outset, the temperatures at the top of the bed ran considerably higher than expected (300 to 400 F compared to the normal 130 to 150 F). Thus, severe gas channeling is indicated which may explain the fact that the oil content of the spent shale ranged from four to nine gallons per ton throughout the operation. The recycle rate was reduced to 12,500 SCF/T at the 500 lb/(hr)(ft^2) shale rate in an effort to reduce the temperatures at the top of the bed. Although the temperatures decreased somewhat, the oil recovery did not improve. Therefore, the unit was shut down after the second day; two small clinkers were found.

H. Model Studies Concurrent With and Subsequent to Retort No. 3 Studies With the Liquid Disengager

1. Liquid Disengager Simulation

To better understand the problems in the disengagement of liquid in the retort, a simulation of the actual disengagement deck was constructed and operated in a plexiglass model. This model is shown in Figure 16. The gas rates used, simulated Retort No. 3 operation; the liquid loading was based on the data developed in the 3.6-inch retort. Air was used to simulate retort gas, and No. 1 fuel oil was used to simulate hot shale oil.

The gas split for the model was 96 ACFM under the liquid disengaging grate and 48.9 ACFM to the simulated shale chute. These gas flows are more meaningful when expressed as 0.74 cubic feet per square foot of grate area per second under the grate and 3.26 cubic feet per square foot of chute area per second to the shale chute.

The liquid loading was 0.0185 gallon per square foot of grate area per minute.

The model was constructed with a static shale bed; it was possible to vary the gas flow split and to separately drain off liquid from the shale chute and the chamber under the grate. In addition, dust could be injected into the gas stream.

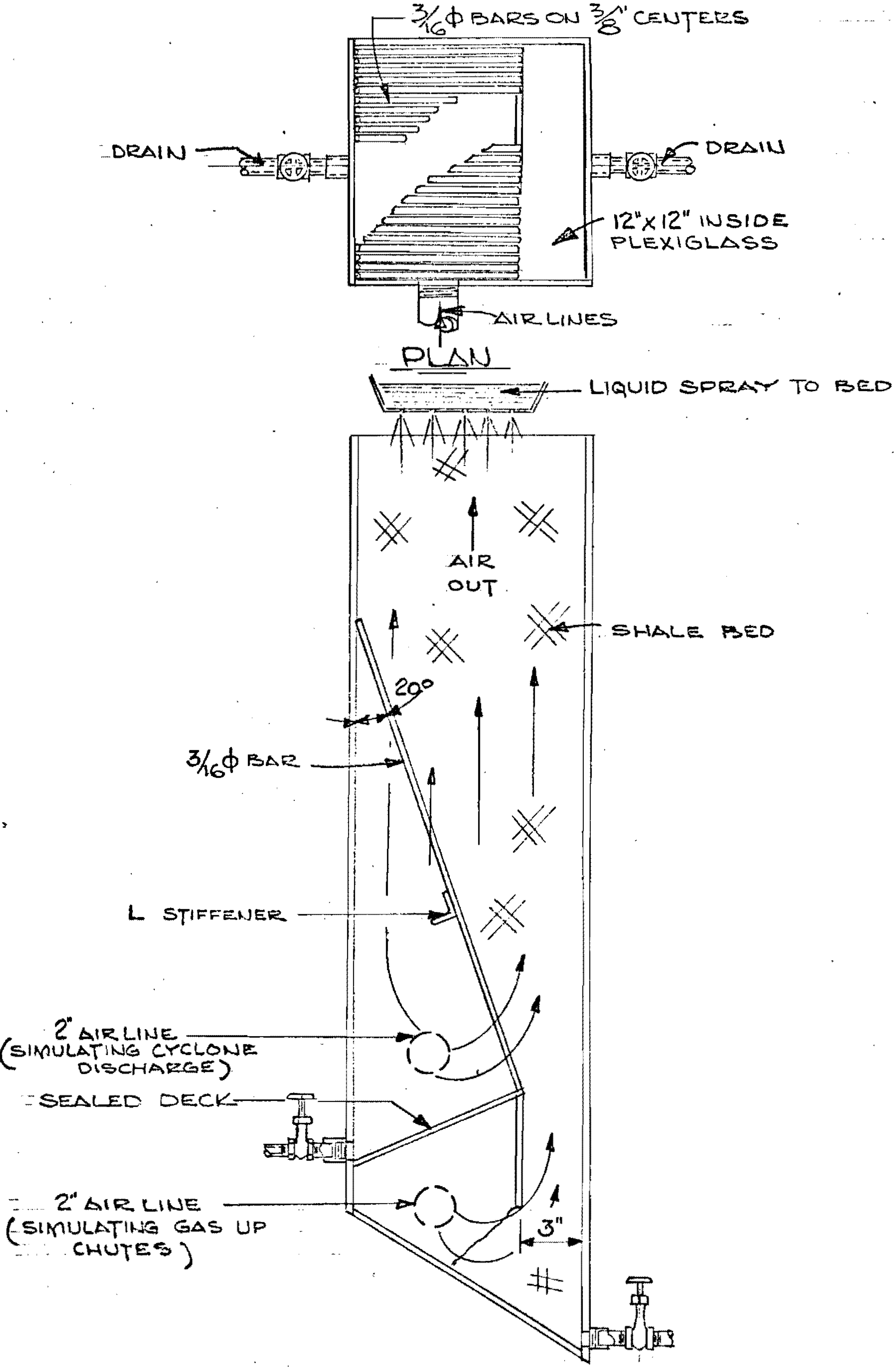
Run 1 - Gas Split Duplicating Retort No. 3 Operating Conditions

The model was filled with 1/4 to 1 inch shale that had been thoroughly wetted with No. 1 fuel oil. Gas flow split was set to simulate retort operating conditions. The liquid flow to the top of the bed was started.

Liquid disengagement from the bed was observed over the entire length of the bars with heavy streams dripping

FIGURE 16

LIQUID DISENGAGER SIMULATION



from the angle stiffener and very heavy flow from the bottom spacer stiffener bar.

The liquid split from the two chambers was about 85% from the grate chamber and 15% from the chute chamber. No fouling was observed on the grid. Turbulence of the liquid draining through the grid was violent.

Run 2 - All Gas to Shale Chutes

The model was operated with the gas rate at 145 ACFM; all of the gas flowed to the shale chute plenum. All other factors were the same.

The liquid disengagement from the bed was almost completely through the grate with only a very small fraction passing down through the chute.

Run 3 - Gas Split Identical to Run 1 - Dust Injected With Gas to Grid Plenum

All procedures followed in Run 1 were repeated but in addition, minus 8 mesh dust particles were injected into the gas line serving the grate plenum.

The dust particles were picked up by the air and the grate was soon completely fouled and plugged, the shale bed was observed to be flooded with liquid to the top of the grid bars. The liquid drained from the unit was split equally between the two chambers but at least 80% was held in the shale bed.

Run 4 - Gas Conditions Identical to Run 2 - Dust Injected With Gas to Grate Plenum

The model was started under the same conditions as Run 3 and the same plugged grid and flooding in the bed developed. The gas flow to the grate plenum chamber was cut off at that time and diverted to the shale chute chamber. Until then, little liquid was observed under the grate. When the gas flow was diverted, liquid was observed to spurt through the grate and accumulate on the bottom of the grate chamber. The grate was soon flushed clean.

An estimate of the liquid flow from either of the chambers in the last two runs was impossible due to plugging of the 1/4-inch drain.

These tests indicate that re-engagement of the gas through the grate can be detrimental to the objective of liquid drainage through the grate; particularly when dust is involved. Re-engagement of the gas above the grate should overcome the problem. Therefore, based on these observations, the cyclone gas was by-passed around the grates during Run C1056.

2. Gas Flow From the Shale Downcomer Into the Liquid Disengaging Section

The interaction of gas and liquid in a packed bed of 1/4 to 1/2 inch shale at the transition from the shale downcomer to the liquid disengaging section of a liquid disengaging device was also studied. In this simulation, air was again used in place of retort gas and No. 1 fuel oil was used to simulate shale oil. The equipment used is illustrated in Figure 17. Table 7 summarizes the data obtained. When the conditions simulated were 300 lb/(hr)(ft^2) shale rate, 20,000 SCF/T gas rate and either 150 or 600 F gas temperature, considerable liquid flow down the shale downcomers countercurrent to the gas flow occurred. When 500 lb/(hr)(ft^2) shale rate, 20,000 SCF/T gas rate, and 600 F gas temperature conditions were simulated, incipient flooding of liquid in the shale downcomers appeared to occur. At simulated conditions of 1000 lb/(hr)(ft^2) shale rate and 20,000 SCF/T gas rate with gas temperatures of either 150 or 600 F, flooding in the downcomers did occur and no apparent flow of liquid down the downcomers was observed.

3. Gas Flow From the Shale Bed Into the Shale Downcomer

The interaction of gas and liquid in a packed bed of 1/4 to 1/2 inch shale at the transition from the full bed of shale into the shale downcomer has also been studied. The same fluids were used as in the previous study. The equipment used is illustrated in Figure 18. When the model was operated near the loading point or the incipient flooding point, splashing and some local accumulation of oil occurred at the free shale surface. These conditions were observed at simulated 400 and 500 lb/(hr)(ft^2) shale rates, respectively, with a simulated 20,000 SCF/T gas rate and a 600 F gas temperature. The results are summarized in Table 8.

I. Significance of Model and Retorting Studies in Applying Liquid Disengagement Principle

In summary, the experiences with this liquid disengagement technique as practiced in the 3.6-inch retort, in Retort No. 3, and as simulated in the model studies, have led to the following conclusions:

--Disengagement of liquid appears to occur, in both Retort No. 3 and the 3.6-inch retort, by causing a controlled locally flooded zone in a region in which

FIGURE 17

SHALE DOWNCOMER-EXPANSION SECTION TRANSITION

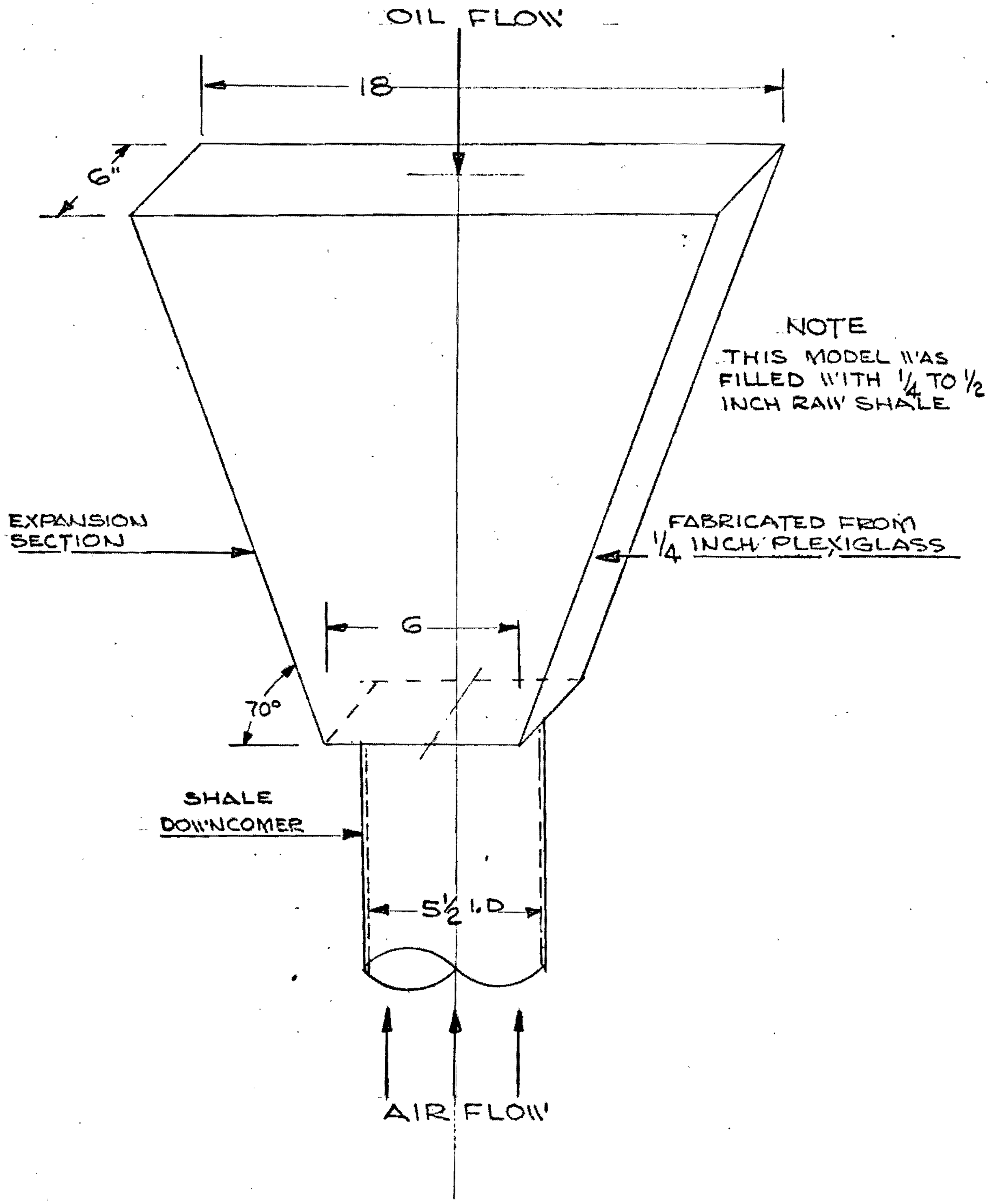


TABLE 7

VISUAL OBSERVATION OF LOADING AND FLOODING IN A PACKED BED OF 1/4 TO 1/2 INCH SHALE - TRANSITION FROM SHALE DOWNCOMER TO EXPANSION SECTION

Conditions Simulated						
Raw Shale, lbs/(hr)(ft^2)	300	300	400	500	1,000	1,000
Total Gas Rate, SCF/T	20,000	20,000	20,000	20,000	20,000	20,000
Gas Temperatures, ^{O}F	150	600	600	600	150	600

Liquid rate was chosen so that it simulated a refluxing liquid stream equal to 50% of the oil that would be recovered from 28 gal/ton shale at 90 Vol. % Fischer Assay yield.

Actual Rates						
Superficial Gas Velocity In Shale Downcomer Feet/Second	1.0	1.7	2.3	2.8	3.3	5.7
Volumetric Liquid Rate cc/min	19.6	19.6	26.2	32.7	65.4	65.4
Visual Observations	Liquid drained downward through shale downcomer. No significant liquid holdup other than surface wetting was apparent.	Some holdup at contact points of shale occurred in shale downcomer. A significant flow of liquid downward through the shale downcomer still occurred.	Increased holdup in shale downcomer occurred.	Liquid holdup in shale downcomer increased. Incipient flooding is believed to have occurred.	Flooded in shale downcomer. No apparent downward flow of liquid in downcomer. Separation of a liquid continuous bubbling zone in the expansion section occurred.	The bubbling zone in the expansion section became more agitated.

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FIGURE 18

SHALE BED - SHALE DOWNCOMER TRANSITION

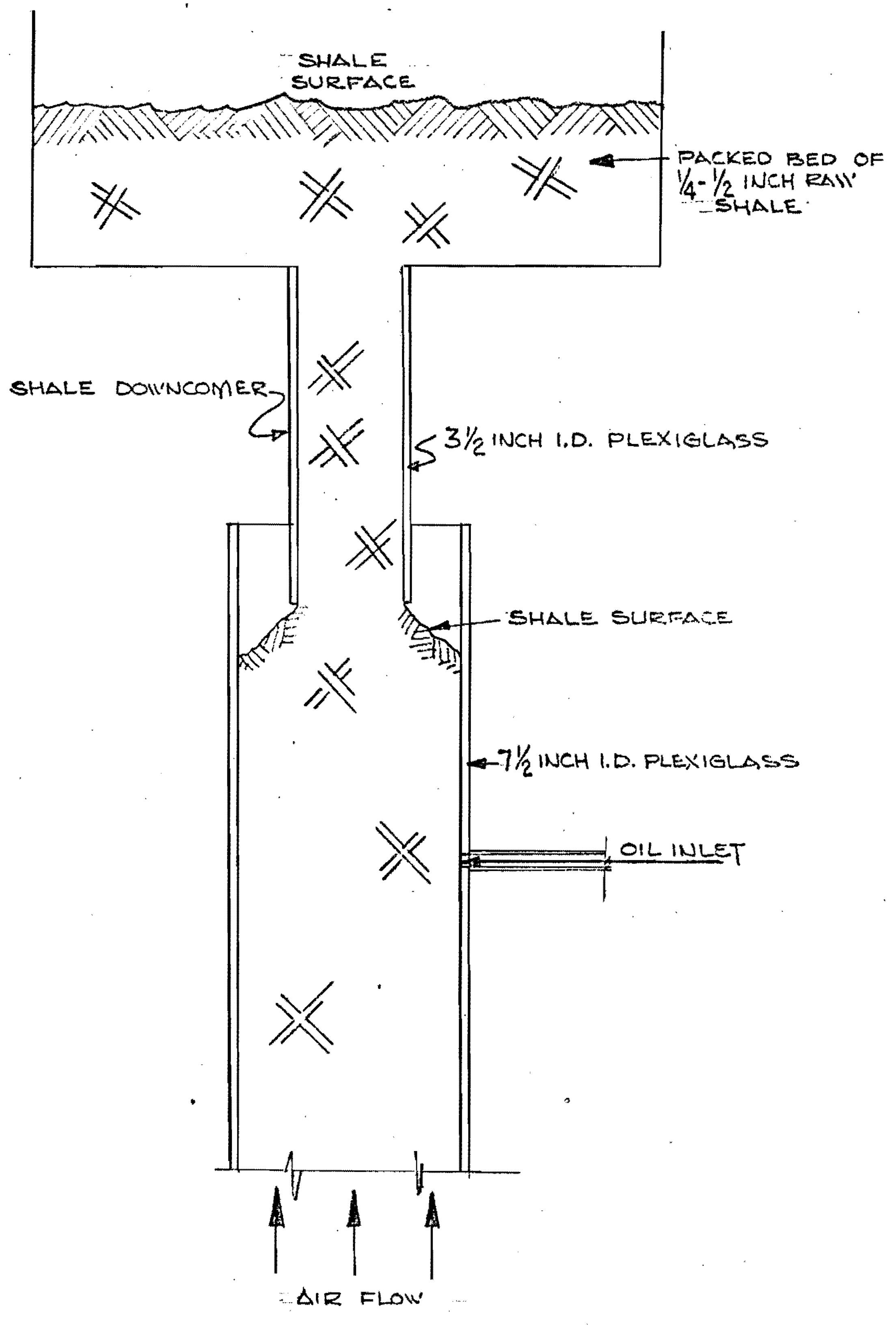

TABLE 8

VISUAL OBSERVATION OF LOADING AND SPLASHING IN A PACKED BED OF 1/4 TO 1/2 INCH SHALE TRANSITION FROM SHALE BED TO SHALE DOWNCOMER

Conditions Simulated						
Raw Shale, lbs/(hr)(ft^2)	300	400	500	300	400	500
Total Gas Rate, SCF/T	20,000	20,000	20,000	20,000	20,000	20,000
Gas Temperature, ^{o}F	150	150	150	600	600	600

Liquid rate was chosen so that it simulated an impacted liquid stream equal to 50% of the oil that would be recovered from 28 gal/ton shale at 90 Vol. % Fischer Assay yield.

Actual Rates						
Superficial Gas Velocity In Shale Bed, Feet/Second	1.0	1.3	1.6	1.7	2.3	2.8
Volumetric Liquid Rate cc/min	36.5	48.6	60.7	36.5	48.6	60.7
Visual Observations	Liquid drained downward through shale bed. No significant liquid holdup other than surface wetting. No splashing at (a).	No loading in shale bed. No splashing at (a).	No loading in shale bed. No splashing at (a).	No loading in shale bed. No splashing at (a).	Some significant hold-up of liquid in the shale at (a).	Incipient flooding. Significantly increased splashing at (a). Local accumulation of liquid at that point.

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--the gas temperature is about 600 F.

--a liquid disengaging facility is accessible to this controlled locally flooded zone.

--Accumulation of oil below this controlled locally flooded zone must be avoided. This may be done by controlling the temperature of this zone at about 600 F. The shale should be contained in confining hats below this controlled locally flooded zone so that oil does not accumulate at the free surface of the shale piles.

Conditions in the shale downcomers during Runs C1057 and C1058, in Retort No. 3, were in the flooded to superflooded range of operation. One might logically ask why these operations failed.

In Run C1057, it is likely that a significant quantity of oil condensed below the deck during startup. This may be inferred from the 350 F deck temperature observed early in this run and the concurrent symptoms of clinkering. (Eventually, in this run, 500 to 550 F deck temperatures were realized.) From the first of these model studies, it may be concluded that the upward flow of liquid with the gas into the shale downcomer would not occur at a 300 lb/(hr)(ft^2) shale rate with the gas rates and temperatures used. Local accumulations of liquid below the deck could, and presumably did, occur.

In Run C1058, confining cones were installed below the shale downcomers to prevent the local accumulation of oil at the free surface of shale where it flowed from the chute. The startup shale rate was also increased from 300 to 400 lb/(hr)(ft^2). These decisions were based on some preliminary parts of the second of these studies. At the same time, the shale downcomers were shortened without lowering the deck portion of the liquid disengager. The distance from the air distributor to the bottom of the shale downcomer was increased from about three to six feet. In effect, the bed height between the shale downcomers and the air distributor was now about equal to what would normally exist between the air inlet and the shale inlet chutes in the conventional process. The result that might be expected, occurred. Low deck temperatures (250 to 350 F) were observed. Even at the 400 to 500 lb/(hr)(ft^2) shale rate used in this operation, a significant local accumulation of oil below the deck would be expected to occur. Clinkering suggests that it did.

--Cyclones as designed and installed in Retort No. 3 are not a necessary or useful device for lowering liquid loading.

--Inclined grates are not a practical drainage device as installed either in Retort No. 3 or in the 3.6-inch retort.

Even if gas is not released below the grate in order to avoid fines adhering to the under side of the grate, the leakage of shale chips through the grate is a significant problem.

These conclusions have been kept in mind in developing the concept of the liquid disengaging device shown in Figure 19.

In this device, shale enters the top at a temperature greater than about 450 F and flows downward. (This temperature specification is not critical.) When the shale passes beyond a., it is in a relatively quiescent, isothermal region in which there is little gas flow. At b., it is exposed to sufficient gas flow so that any liquid which is associated with the shale will not flow downward. Most of the gas and the oil associated with the shale leave the shale bed at c. In this relatively low gas velocity, shale free region, oil is disengaged from the gas and drained off through suitable drainage piping. The gas which is now relatively free of liquid is reinjected into the shale bed at d.

The angle at α should be about 50^{o} to avoid the formation of a free shale surface at which liquid could accumulate and the angle at β should be sufficient to insure smooth shale flow with no inactive zones of shale in the zone between a. and b.

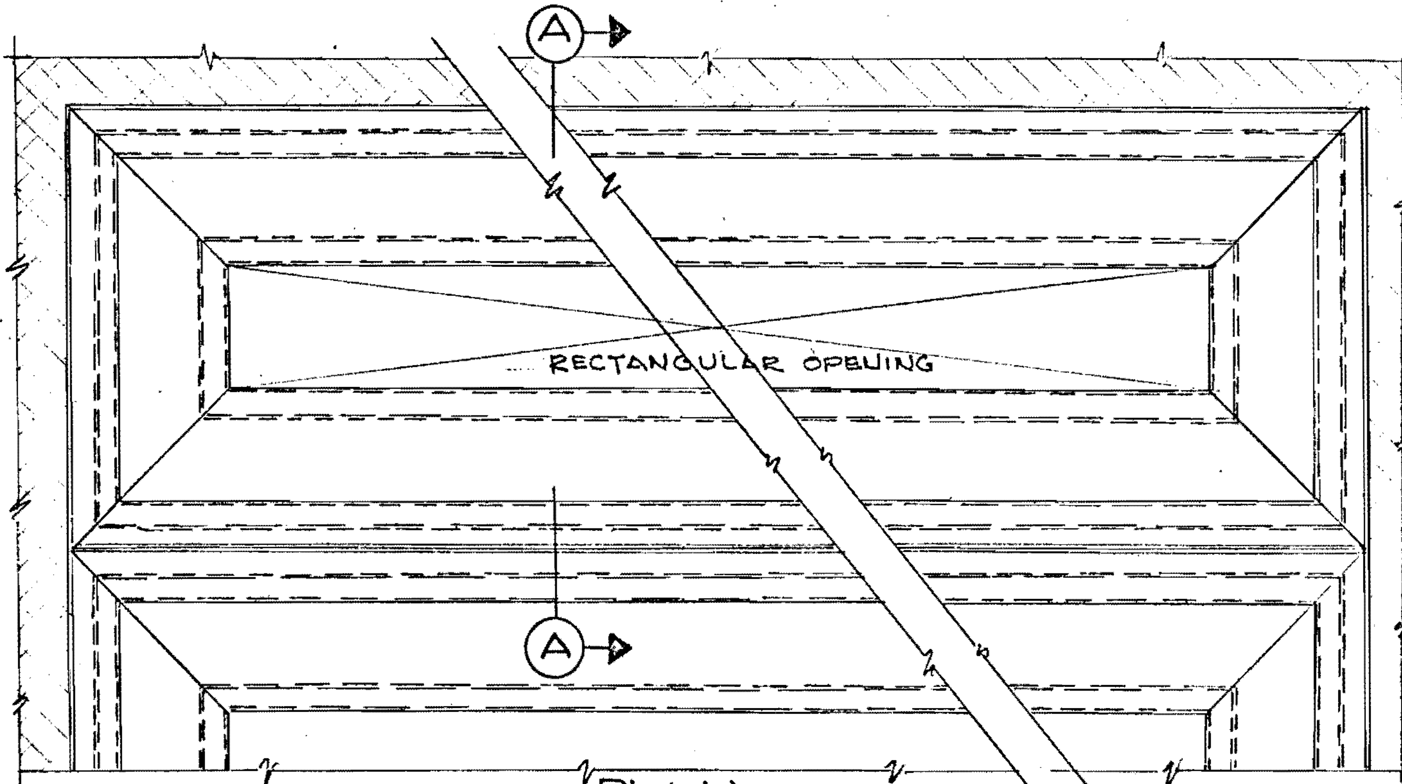
Dimension X should be sufficiently restricted so that the gas velocity through the shale bed at X is "superflooded". By this it is meant that no oil should flow downward counter-current to the gas flow but rather it should be conveyed upward and disengaged from the bed at c.

The device should be vertically located within the retort, so that condensation starts at or above dimension Y. Gas velocity at dimension Y should be high enough so that flooding will occur at very low liquid loadings.

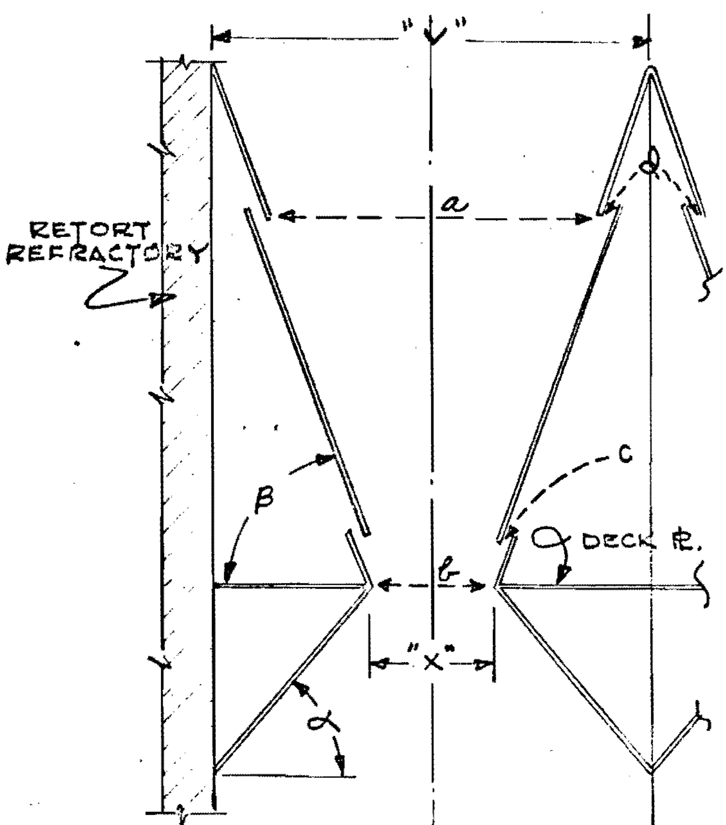
The location of this device in a retort of the same scale as Retort No. 3 is illustrated in Figure 20.

FIGURE 19

LIQUID DISENGAGING DEVICE



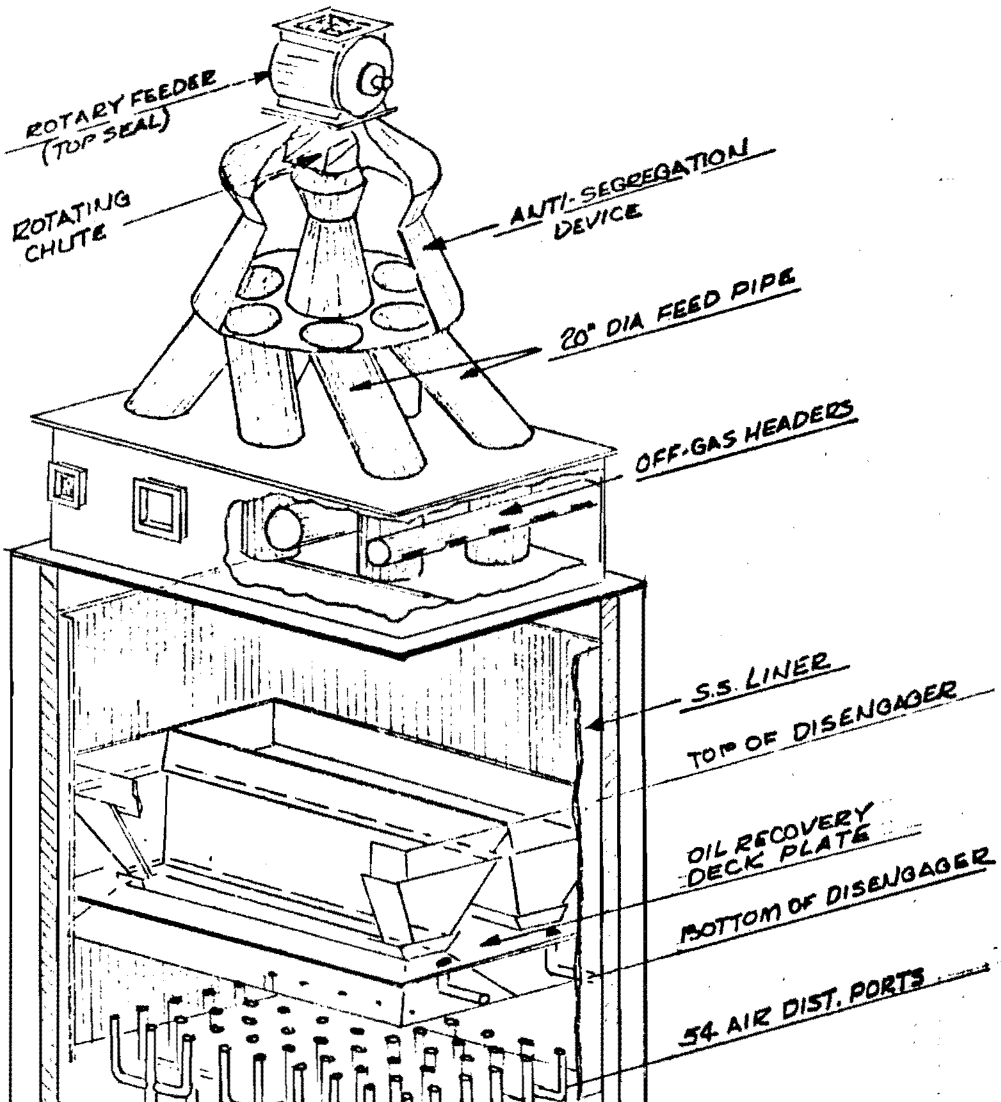
PLAN

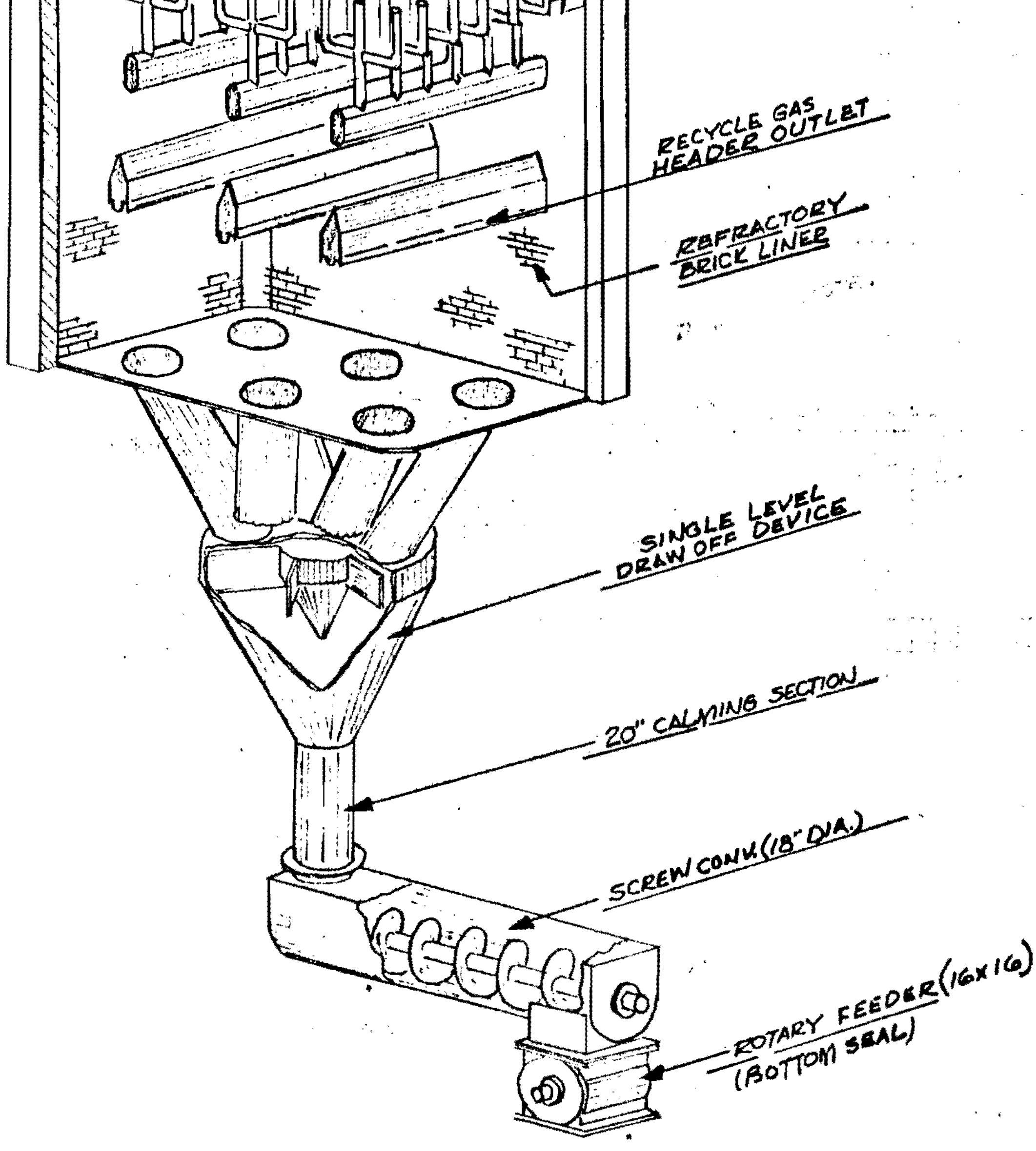


SECTION AA

FIGURE 20

RETORT EQUIPPED
WITH
LIQUID DISENGAGING DEVICE




RECYCLE GAS
HEADER OUTLET
REFRACTORY
BRICK LINER
SINGLE LEVEL
DRAW OFF DEVICE
20" CALMING SECTION
SCREW CONV. (18" DIA.)
ROTARY FEEDER (16X16)
(BOTTOM SEAL)

IV. REFERENCES

1. Letter, RHC/All Anvil Points Technical Personnel, File 102.06, May 15, 1967.

2. Monthly Progress Memorandum, Anvil Points Oil Shale Research Center, June 23, 1967, p. 48.

3. Monthly Progress Memorandum, Anvil Points Oil Shale Research Center, August 25, 1967, pp. 22-25.

4. Monthly Progress Memorandum, Anvil Points Oil Shale Research Center, August 25, 1967, pp. 25-27.

5. Monthly Progress Memorandum, Anvil Points Oil Shale Research Center, August 25, 1967, pp. 27-29.

6. Monthly Progress Memorandum, Anvil Points Oil Shale Research Center, September 26, 1967, pp. 19-21.

7. Monthly Progress Memorandum, Anvil Points Oil Shale Research Center, September 26, 1967, pp. 21-23.

8. Monthly Progress Memorandum, Anvil Points Oil Shale Research Center, September 26, 1967, pp. 23-26.

9. Technical Film No. 66-1, P. H. Gifford, "Studies of Behavior of Mist in Gas-Combustion Retorting Process," Anvil Points Oil Shale Research Center, October 7, 1966.

10. Technical Memorandum No. 66-6, P. H. Gifford, "Studies of Behavior of Mist in Gas-Combustion Retorting Process," Anvil Points Oil Shale Research Center, October 7, 1966.

APPENDIX 1

TECHNICAL FILM NO. 67-1

MODEL STUDIES OF THE OPERABILITY PROBLEM IN THE GAS-COMBUSTION RETORT

Approximate length of film - 350 feet	16 mm
Approximate projection time - 12 minutes	silent film

I. SUMMARY

This film is a compilation of five short filmed sequences that were assembled in the course of studying the retort operability problem.

The first of these sequences demonstrates with the one-sixth scale model of Retort No. 3 how agglomerates can become wedged in the air distributor assembly. The resulting channeling of shale flow is quite apparent.

The second portion of the film shows that liquid will flow to a low energy position in a flooded bed and that liquid may be drawn off laterally from a flooded zone. This withdrawal of liquid is aided by gas flow.

In the third part of the film, a small, glass, moving bed retort is shown in operation. The gradual accumulation of oil on the surface of the shale and between the shale particles in a retorting situation is clearly demonstrated. Reference is made to the earlier work of Gifford (References 9 and 10), particularly Technical Film No. 66-1, which shows this phenomena using 1/4 to 3 inch shale and oil mist from Retort No. 2. His work also shows the gradual accumulation of oil on the surface of the shale and the eventual flooding observed in this film.

The fourth part shows that the position of a flooded region in a shale bed can be controlled by the shape of the bed and that a superficial velocity exceeding 3 feet per second is needed to cause this flooded condition to exist using 1/4 to 1/2 inch shale. Gifford (References 9 and 10) had previously observed about the same limit using 1/4 to 3 inch shale and oil mist from Retort No. 2.

The final section of film shows that if flooding conditions exist in a bed of shale, liquid will accumulate at any free surface of shale contiguous to the flooded region. It is speculated that confining hats resting on these surfaces can prevent the accumulation of liquid in this way.

II. COMMENTARY

To assist in the synchronization of the commentary and the film, the titles are reprinted here just as they appear in the film.

A. One-Sixth Scale Model of Retort No. 3

Title

Model Retort No. 3

Scale 1" = 6"

Shale Size (equiv) - 1/4 to 1 inch

54 Risers - 3 Headers

on
N-S Orientation

4/10/67

1. Overall View of Entire Model (Front View)

The first sequence shows the general orientation of the shale drawoff system, the recycle gas distributors, and the air distributors. Although there are provisions for gas injection at the recycle distributor, none was used in the agglomerated shale flow experiment which is to follow. The shale inlet system was also removed. In the front view, the model is 20 inches wide. This represents the 10-foot dimension of Retort No. 3.

A close-up view of the air distributor assembly is also shown in this sequence since the point of the following sequence is the interaction of agglomerated shale with this assembly.

2. Agglomerated Shale Flow

Title

Agglomerated Shale Empty Bin Drawdown

The first view of this sequence shows the model full of shale with a simulated carbonaceous agglomerate located in the bed above the air distributor. This agglomerated shale has a dark gray shading to it when compared to the rest of the bed of shale. (A yellow line had been drawn across the face of the model at the top of the risers as a reference plane.) A number of red markers have been put in the shale bed near the top. As shale is drawn down, using the "empty bin" technique, the agglomerate can be seen to move into the air distributor region. As the agglomerate passes below the top of the risers, it flows unevenly. In a close-up view, a void in the shale bed can be seen to develop around the center part of the air distributor assembly. This void persists until the bed is drained. When it is drained, it is seen that part of the agglomerate is wedged into the center of the distributor assembly. Both front and side views of the wedged agglomerate are shown.

The position and the relative size of this simulated carbonaceous agglomerate are very similar to the clinkers observed in Retort No. 3 when the retort is emptied after it has become inoperable. This portion of the film helps visualize the sequence of events by which an agglomerate becomes wedged in the air distributor assembly and its effect on the shale flow around the obstructed region.

B. Simulated Flooded Zone in Retort

Title

Simulated Flooded Zone in Retort	
4-28	KIJ

1. Overall View of Apparatus

The apparatus shown in this sequence is a 2 foot by 2 foot box containing a bed of shale. This bed is supported on a screen and air is admitted to the plenum below the bed. Fine shale is located in the lower portion of the bed and 1/2 to 1 1/2 inch shale above it. The boundary between the zones is not horizontal; it has a vee in it. A screen fitted to the vee divides the zones.

2. Flooded Zone Experiment

In this angled front view, when water is poured into the apparatus with air flowing up through the bed at condensing zone conditions, the water floods at the screen. As might be expected, liquid flows to the low energy position in this configuration which is the point of the vee.

A close-up view of the side of the apparatus shows that liquid may be drawn off from a flooded portion of a packed bed and that liquid will flow across the bed with very low hydraulic head requirements. Gas flow through the drain ports assists liquid withdrawal.

A close-up of the flooded zone shows a free liquid froth above the fine shale.

This sequence indicates that if a flooded zone is established either by some random restriction to gas flow or by some feature designed into the bed, it is possible to remove liquid from that flooded zone. Gas flow assists liquid drainage from the flooded zone.

C. One-Inch Glass Retort

Title

	1 in. Glass Retort	
Top Drawoff		No Preheat
	500 Mass Rate	
	20,000 SCF/T gas	
	31 gal/T Assay	
		4-28-67

1. General Views of the Apparatus

The general orientation of the components of this retort are presented in the first sequence involving overall and panoramic views. The apparatus consists of a raw shale feed vessel, a one-inch glass tube retort fitted with a shale inlet and outlet at the top and bottom, respectively, gas inlet and outlet at the bottom and top, respectively, a furnace installed around the tube and a spent shale receiver. The shale movement is controlled by alternately pinching and releasing a piece of rubber tubing mounted in line with the one-inch glass retort tube and connecting this tube with the spent shale receiver. The retort gas used in this experiment was nitrogen and its rate was controlled by a rotameter.

2. Description of the Operation

The raw shale feed vessel and the retort tube were prepared by charging the vessel with 4 to 8 mesh, 31 gallon per ton raw shale. Shale and gas flow were started at the desired rates as the furnace was being heated to 1100 F. This heat-up was started at 12:00 on the clock. As the bed heated up, the shale appeared to darken. This is due to the gradual accumulation of oil in the bed between particles. By 12:20, oil mist is observed to be passing overhead with the offgas. This indicates that retorting is occurring.

At 1:00, 2:00, and 3:00 o'clock, it may be seen that the gradual oil accumulation is continuing. No demarcation of a flooded zone is yet apparent.

At 3:40, a line separating a zone of the shale bed heavily loaded with oil from one less heavily loaded, becomes apparent. This line is located about 14 inches above the furnace. By 3:48, this line moves up to a level 19 inches above the furnace. Finally, at 4:55, when the raw shale feed vessel runs out of shale, oil can be seen splashing from the shale bed into this vessel.

The mechanisms which cause this oil accumulation in the bed are, initially, condensation of oil vapor and impaction of oil mist on the surface of the shale. Ultimately, the scrubbing of oil mist from the vapor by oil already deposited is the major mechanism. These effects may be exaggerated in this experiment because of the small size of the shale, however, Gifford's film in which mist impaction in beds of 1/4 to 3 inch shale was studied (Reference 9) shows the same mechanisms at work, therefore, it can be considered a general mechanistic combination. In this film of Gifford's, shale, which was surface wet with oil, gradually accumulates more oil, due to impaction, as a mist laden gas is passed up through the bed. The amount of oil accumulated depends on the gas velocity through the bed. A discussion of this film and related studies have been reported in a technical memorandum.

D. Simulation of Gas Flow From a Shale Downcomer Into the Expansion Section (Liquid Disengaging Section) of a Liquid Disengager

Title

	Discharge from	
	Shale Downcomer	
	Into Expansion Section	
300 MR	20,000 SCF/T	150 F
1.0 FPS		9-6-67

1. Overall View of Apparatus

The object of the group of experiments carried out with this apparatus was to find the flooding gas velocity with 1/4 to 1/2 inch shale and with liquid loadings that might be expected to exist in the condensing zone of a Gas-Combustion Retort.

The general arrangement of the apparatus is shown in the first sequence. It is made up, from top to bottom, of a liquid feed tube, an expansion section which simulates the liquid disengaging section and a shale downcomer section. The expansion section is 6 inches by 18 inches at the top and 6 inches by 6 inches at the bottom. The inclined sides are at a 70° angle to the horizontal. This expansion section opens into the shale downcomer at its lower end. Air is admitted to the apparatus through two inlets on opposite sides of the shale downcomer approximately 7 inches below the expansion section. The shale downcomer is 5 1/2 inches in diameter and is attached on its lower end to a gas-tight receiver.

The apparatus is filled with 1/4 to 1/2 inch shale. Air, which is used to simulate recycle gas, is turned on at the desired experimental rate; then No. 1 fuel oil, which is used to simulate hot shale oil, is turned on at its desired experimental rate. The oil rate used is equal to fifty percent of the total oil that would be retorted from 28 gallon per ton shale assuming a 90% Fischer Assay yield. The shale bed is not moved in this apparatus. In setting gas rates, it is assumed that shale rate is based on the 5 1/2 inch diameter of the shale downcomer. The temperature indicated in the title was used in converting standard cubic feet to actual cubic feet of gas volume. The velocity indicated is a superficial velocity based on the shale downcomer area. The simulated shale rate is also based on this area.

2. Close-up View of Experiment 1

In this close-up view, the apparatus is operated at the conditions indicated in the above title. Oil trickles downward countercurrent to the air flow at this rate. The conditions chosen are intended to simulate the conditions that might prevail near the top of a Gas-Combustion Retort operated at 300 lb/(hr)(ft^2) shale rate.

Title

	Discharge From	
	Shale Downcomer	
	Into Expansion Section	
300 MR	20,000 SCF/T	600 F
1.7 FPS		9-6-67

3. Close-up View of Experiment 2

At this increased gas rate, which is intended to simulate conditions that might prevail in the condensing zone of a Gas-Combustion Retort operated at 300 lb/(hr)(ft^2) shale rate, oil continues to trickle downward countercurrent to the air flow, however, some oil hold-up at contact points of shale does occur.

Title

	Discharge From	
	Shale Downcomer	
	Into Expansion Section	
400 MR	20,000 SCF/T	600 F
2.3 FPS		9-6-67

4. Close-up View of Experiment 3

This experiment simulates the conditions in the condensing zone of a Gas-Combustion Retort operated at 400 lb/(hr)(ft^2) shale rate. The holdup observed in the previous experiment was increased by the increased gas velocity in this experiment.

Title

	Discharge From	
	Shale Downcomer	
	Into Expansion Section	
500 MR	20,000 SCF/T	600 F
2.8 FPS		9-6-67

5. Close-up View of Experiment 4

In this experiment, the simulated shale rate is increased to 500 lb/(hr)(ft^2). The liquid holdup in the shale downcomer again increased. Incipient flooding is believed to have occurred.

Title

	Discharge From	
	Shale Downcomer	
	Into Expansion Section	
1000 MR	20,000 SCF/T	150 F
3.3 FPS		9-6-67

6. Close-up View of Experiment 5

The conditions simulated in this experiment are those at the top of the retort at a 1000 lb/(hr)(ft^2) shale rate. Visual flooding occurs in the shale downcomer. No downward flow of liquid in the shale downcomer is apparent. The separation of a liquid continuous bubbling zone in the expansion section occurs.

Title

Discharge From		
Shale Downcomer		
Into Expansion Section		
1000 MR	20,000 SCF/T	600 F
5.7 FPS		9-6-67

7. Close-up View of Experiment 6

The condensing zone of a Gas-Combustion Retort operating at a 1000 lb/(hr)(ft^2) shale rate is simulated in this experiment. The bubbling in the expansion section becomes more agitated.

The last three experiments indicate that the superficial gas velocity in the shale downcomers should exceed at least 2.8 feet per second, and probably 3 feet per second, to prevent the downward flow of oil in these downcomers. This conclusion is probably dependent on shale size and liquid loading, however, essentially the same flooding velocity was observed by Gifford (References 9 and 10) in his experiments with 1/4 to 3 inch shale.

E. Simulation of Gas Flow From the Shale Bed Into a Shale Downcomer

Title

	Discharge From	
	Shale Bed	
	Into Shale Downcomer	
500 MR	20,000 SCF/T	150 F

1.6 FPS 9-6-67

1. Overall View of Apparatus

An overall view of the apparatus is shown in this first sequence. The unit consists of a gas disengaging zone, a 3 1/2 inch shale downcomer and a 7 1/2 inch shale bed. The gas disengager is only intended to minimize oil splashing and is not involved in this experimentation.

The shale downcomer, which is the 3 1/2 inch cylindrical section extends down through the plane at the top of the shale bed. This is done intentionally so that a coned free surface of shale will exist at the transition from the shale bed to the shale downcomer.

The object of these experiments is to find out the conditions of flow at which liquid will accumulate at this coned shale surface. Air, which is used to simulate recycle gas, is admitted to the apparatus through a concentric screened inlet at the bottom of the shale bed. Oil flows into the shale bed about 10 inches below the bottom of the shale downcomer. No. 1 fuel oil is again used to simulate hot shale oil. As in the previous group of experiments, the oil rate used is equal to fifty percent of the total oil that would be retorted from 28 gallon per ton shale assuming a 90% Fischer Assay yield. These experiments are also fixed bed. In this case, gas rates are based on the assumption that shale rates are calculated using the 7 1/2 inch diameter of the shale bed. The temperature indicated in the title was used in converting standard cubic feet to actual cubic feet of gas volume. The velocity indicated is a superficial velocity based on the shale cowncomer.

2. Close-up View of Experiment 1

In this experiment, which is described in the above title, it may be noted that the shale bed does not retain much liquid. It trickles downward. No accumulation of liquid at the coned surface of shale occurs, either. This condition is one that exists at the top of the bed of the Gas-Combustion Retort.

Title

	Discharge From	
	Shale Bed Into	
	Shale Downcomer	
500 MR	20,000 SCF/T	600 F
2.8 FPS		9-6-67

3. Close-up View of Experiment 2

This experiment, which is at the point of incipient flooding as determined in the previous group of experiments, does cause a significant accumulation of liquid at the coned surface of shale. This can probably be avoided by installing a confining hat on the surface of the shale pile.