

The cool, mist-laden gases pass first through a centrifugal separator, where a considerable part of the entrained oil droplets coalesces on the walls, then to a positive-displacement blower, where they are repressured to overcome pressure loss in the retort and recovery system. The action of the blower coalesces the finer droplets, and another centrifugal separator removes a large part of the recoverable oil. An electrostatic precipitator acts as a final clean-up unit. Part of the clean gas is recycled to the retort, and the remainder is vented.

Control of Mist Formation

Condensation of oil vapor within the retort as a mist depends on controlling the conditions that contribute to mist formation, namely, supersaturation at some point in the gas stream and the presence of suitable condensation nuclei.

Above the retorting zone two important physical actions occur:

1. There is a transfer of heat from the gas stream to the shale. The rate depends upon the temperature difference between the gas and solid.
2. There is a mass transfer of oil from the gas stream to the shale. The rate of this diffusional process depends on the difference between the partial pressure of the vapor in the gas stream and the vapor pressure of the oil film on the shale.

Therefore, in an operation such as the gas-combustion process, if a high rate of heat transfer from the gas to the shale and a low rate of mass transfer of vapor from the gas to the solid can be attained, the vapor-gas mixture will become supersaturated and if nuclei are present the vapor will condense as mist. The ultimate would be a situation in which the driving force for mass transfer equaled zero. If this were possible, the gas would be cooled below the saturation point, and if nuclei were present, the vapor would condense on the nuclei as a mist. Unfortunately, it is not possible to eliminate the driving force for mass transfer entirely, and a thin film of oil always condenses on the shale.

The heat-transfer rate can be increased by increasing the temperature differential between the gas and shale, using a smaller quantity of hotter gas to do the retorting. This does not have an appreciable effect on the mass-transfer rate, which depends on the partial-pressure differential, since the concentration of vapor in the gas stream is very small. Accordingly, the degree of supersaturation can be controlled by regulating the volume of retort gases.

It is believed that the principal source of nuclei for condensation is the inorganic material in the shale and that the number of nuclei produced is a function of the shale-bed temperature, more being produced at the higher temperatures. To provide an adequate number of nuclei at shale-bed temperatures that do not exceed those ordinarily required for successful retorting, it has been found advantageous to supplement the normal supply by adding inorganic salts to the shale.

Evaluation Run

A continuous 4-day run was made to demonstrate the operating characteristics and evaluate the performance of the gas-combustion retort when processing a blend of shale from the minable beds as rich as or a little richer than would be obtained in a commercial mining operation. The series of beds that is considered economically minable is about 70 feet thick in the area of the Bureau's mine and has an average oil content of about 27 gallons per ton based on the Fischer assay; the shale blend processed during the evaluation run averaged about 30 gallons per ton.

Within this 70-foot thickness, known as the Mahogany ledge, is a bed 8 feet thick that averages 50 gallons of oil to the ton and has unusual retorting characteristics. Pieces of this rich shale pass through a plastic condition when heated to retorting temperatures. The plastic particles act as a cementing material and bind other pieces of shale into a hard cluster. If these clusters become large enough, they prevent uniform flow of shale through a gravity-flow retort. Approximately 11 percent by weight of the Mahogany ledge has this plastic property.

For the 4-day evaluation run, the rich shale from the 8-foot bed was mixed with shale from all of the strata in the Mahogany ledge to produce a blend containing 12.6 percent by weight of 65-gallon-per-ton shale, as shown by sink-float separation in a heavy liquid medium. Thus, the retorting characteristics were determined on a charge material more difficult to process than that from a commercial mining operation. Properties of the blend are shown in table 5.

TABLE 5. - Gas-combustion pilot plant; properties of shale feed;
4-day evaluation run

Fischer assay:		
Oil content	gal. per ton	29.6
Oil gravity	°A.P.I.	23.0
Water from assay	wt. percent	1.5
Gas plus loss	do.	2.1
Mineral carbon dioxide	wt. percent	14.5
Screen analysis:		
-1.05 + 0.742 inches	wt. percent	19.7
-.742 + .525 inches	do.	44.1
-.525 + .371 inches	do.	24.7
-.371 + .263 inches	do.	10.1
-.263 + .185 inches	do.	1.0
-.185 + .131 inches	do.	.2
Pan + loss	do.	.2
Sink-float analysis:		
Specific gravity of medium		1.76
Float	wt. percent	12.6
Sink	do.	87.4
Float assay, ave.	gal. per ton	70
Sink assay, ave.	do.	24

From 24 tons of shale charged at a constant rate throughout the period, an average yield of 95.7 percent by volume of the Fischer assay was achieved at a retorting rate of 229 pounds per hour per square foot of cross-sectional area. Yields for each 24-hour period ranged from 92.8 to 99.8, indicating an over-all experimental reliability of plus or minus 3 percent. Recycle gas was supplied to the bottom of the retort at a rate of 71.4 std. c.f.m., and 16.2 std. c.f.m. of air was introduced into the air-gas mixer. Table 6 summarizes the operating conditions and product-yield data for the run.

TABLE 6. - Gas-combustion pilot plant; operating conditions and product-yield data; 4-day evaluation run

<u>Run history:</u>	
Date, 1951	10/22-10/25
Length of run	4 days
<u>Shale feed:</u>	
Fischer assay	gal. per ton 29.6
Total feed	tons 24.0
Feed rate	tons per day 6.0
<u>Specified conditions:</u>	
Retorting rate	lb. shale per (hr.) (Sq. ft.) bed area 229
Air rate	std. c.f.m. 16.2
Recycle gas rate	do. 71.4
Recycle gas:air ratio	4.4
<u>Resulting conditions:</u>	
Air required	std. c.f. per ton of shale 3,900
Recycle gas required	do. 17,200
Superficial linear gas velocity ^{1/}	ft. per sec. 0.73
<u>Temperatures, °F.:</u>	
Products	122
Retorted shale	212
Retorting zone	960
<u>Oil-yield summary (water-free oil):</u>	
Liquid oil collected	gal. per ton 28.4
Volume percent Fischer assay	95.7
Weight percent Fischer assay	97.6
<u>Product-gas production:</u>	
Wet gas	std. c.f. per ton 5,760
Dry gas	do. 5,040

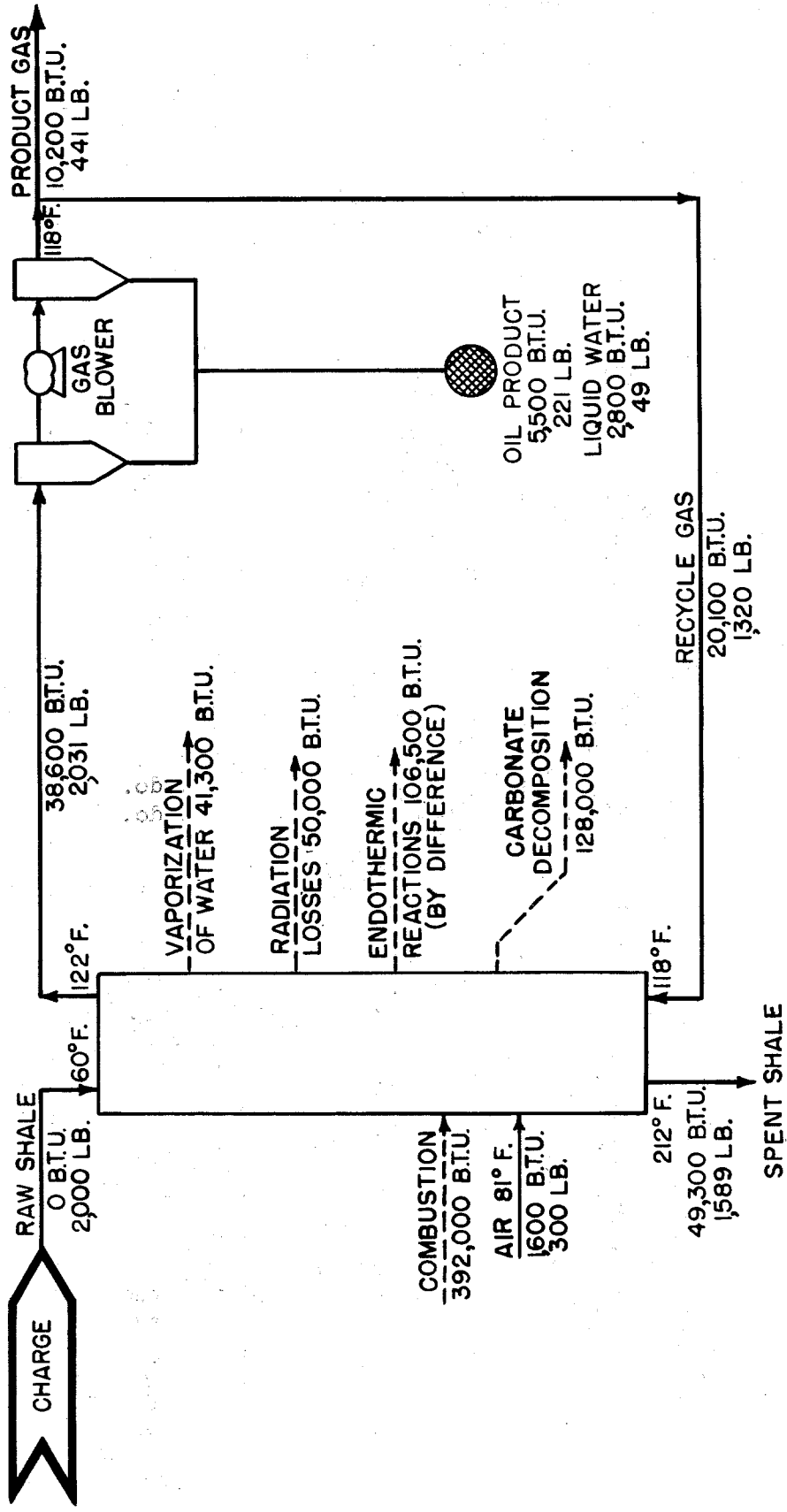
^{1/} Basis: Gas from retort (wet) corrected to standard conditions, retort empty.

Table 7 lists the properties of the retort products. The oil has properties similar to those of oils from N-T-U and other internal-combustion retorts. It is virtually dust-free, and the water produced with the oil is easily removed by gravity settling and decantation.

TABLE 7. - Gas-combustion pilot plant; properties of retort products;
4-day evaluation run

<u>Properties of crude oil:</u>		
Gravity	°A.P.I. at 60° F.	20.0
Specific gravity	at 60° F.	0.9340
Viscosity	S.U.S. at 130° F.	135.0
	S.U.S. at 210° F.	50.4
Ramsbottom carbon	wt. percent	2.51
Water	vol. percent	9.4
Nitrogen	wt. percent	2.09
Sulfur	do.	0.63
Flash point	°F.	240
Pour point	do.	85
A.S.T.M. distillation (corrected to 760 mm.)		
I.b.p.	°F.	290
5 percent	do.	446
10 percent	do.	497
20 percent	do.	580
30 percent	do.	647
40 percent	do.	694
Recovery at cut-off point of 699° F.	vol. percent	49
<u>Properties of product gas:</u>		
Gross heating value (saturated with water vapor at 60° F., 30 in. Hg)	B.t.u. per std. c.f.	85
Water-vapor content	vol. percent	12.4
Orsat analysis - (dry basis)		
CO ₂	mol. percent	24.7
Illuminants	do.	1.3
O ₂	do.	0.4
CO	do.	4.8
H ₂	do.	5.2
CH ₄	do.	1.3
C ₂ H ₆	do.	1.2
N ₂	do.	61.1
<u>Properties of retorted shale:</u>		
Fischer-assay oil content	gal. per ton feed	0.3
Mineral carbon dioxide	wt. percent	11.6
Organic content by wet oxidation	do.	4.1

The material balance shown in figure 24 is based on direct physical measurement of the streams, whereas the heat balance involves heats of reaction and sensible heat. Heat of combustion is calculated as in a furnace, the product-gas analysis being corrected for shale gas and mineral carbon dioxide produced in the process. A major item of heat consumption is the endothermic reactions by which kerogen is converted to shale oil. Other endothermic processes are the water-gas reactions and condensation of water in the piping system, which involves heat of vaporization. These are not directly measurable. Radiation losses were calculated from skin-temperature measurements.



BASIS: ONE TON OF SHALE FEED
 DATUM: 60° F.

Figure 24. - Material and heat balances, gas-combustion pilot plant.

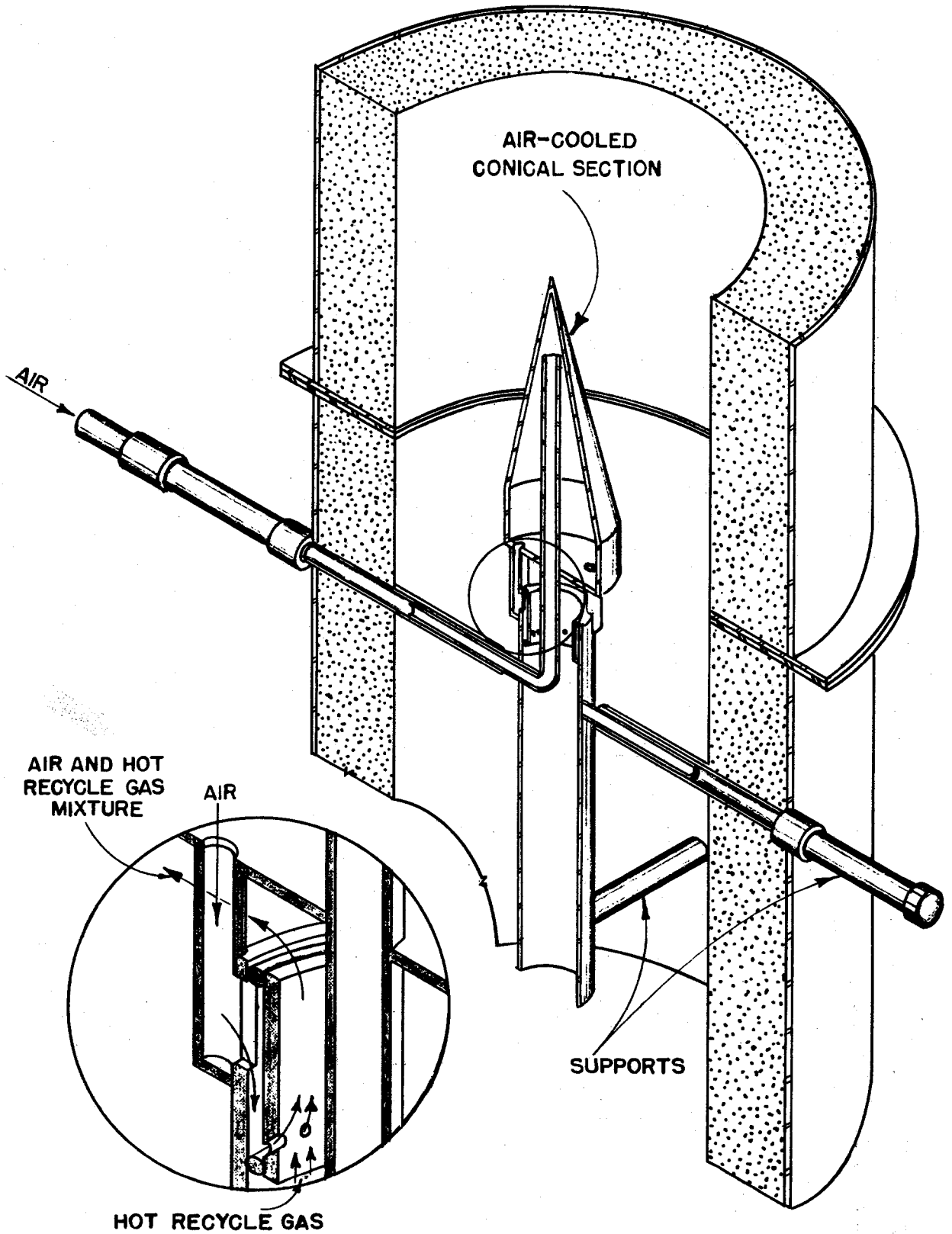


Figure 25. - Isometric section of center-type gas-air mixer, gas-combustion process.

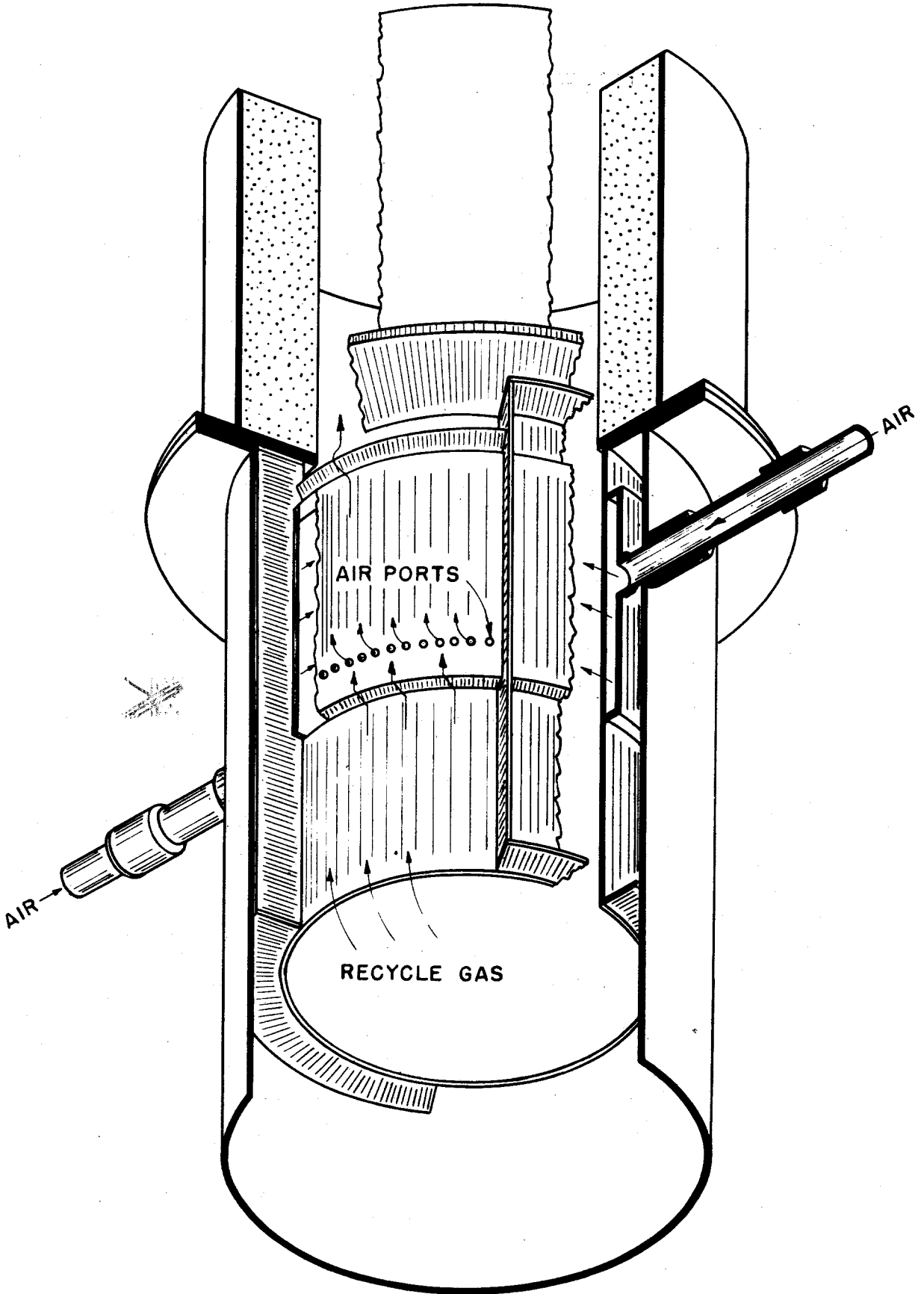


Figure 26. - Isometric section of wall-type gas-air mixer, gas-combustion process.

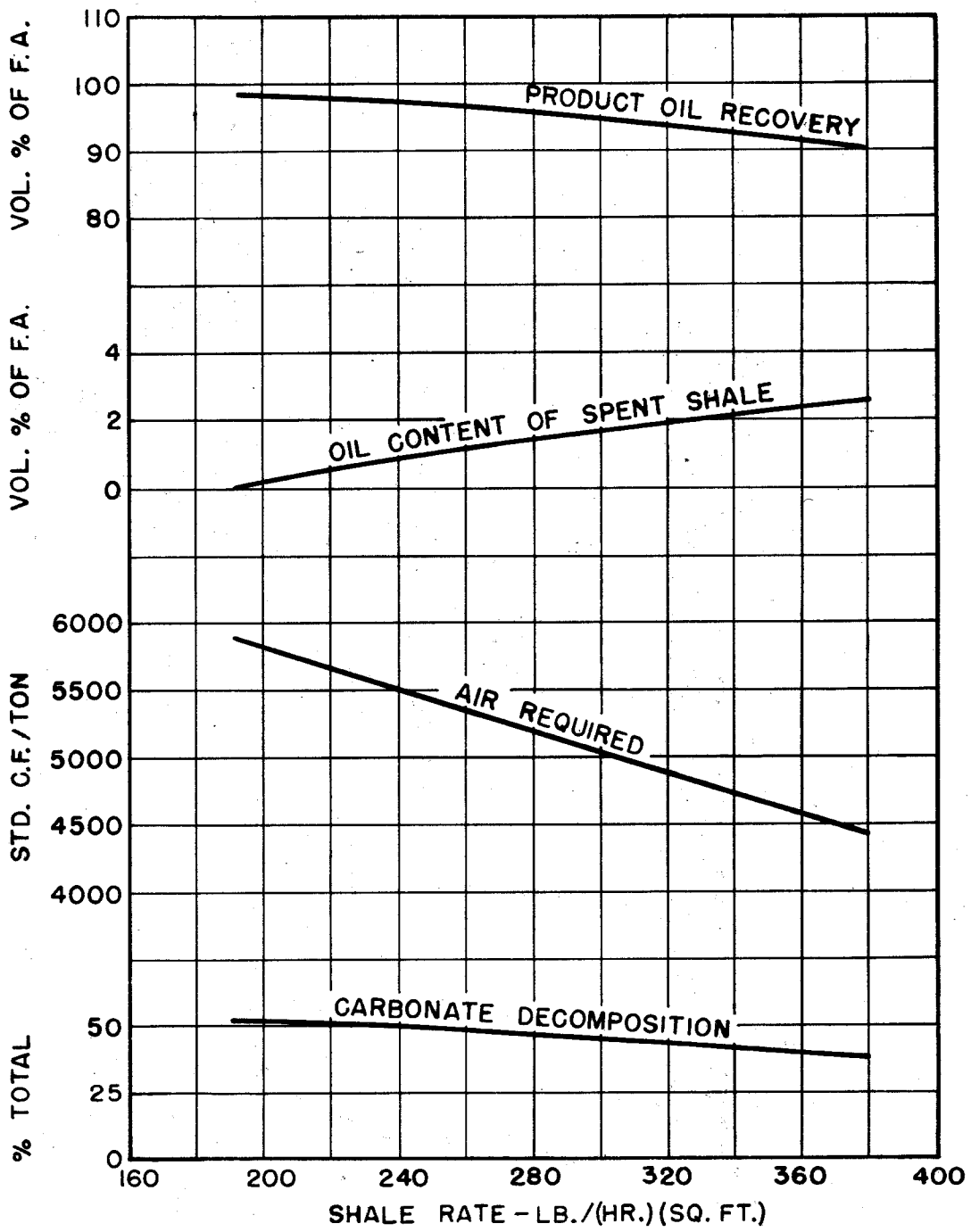


Figure 27. - Effect of shale rate on gas-combustion-retort operation.

Gas-Air Mixer Studies

An important feature of the gas-combustion process is withdrawal of a portion of hot recycle gas from the shale bed, addition of air to this gas, and injection of the mixture back into the shale bed. This is accomplished by means of the gas-air mixer, two general types of which are shown in figures 25 and 26, center and wall types, respectively. Information on the structure and functioning of the center type was given in connection with the description of the 6-ton-a-day pilot plant.

The primary purpose of the gas-air mixing device is to provide even heat distribution at the desired temperature level. The geometry of the mixer must be such that enough gas is entering it to prevent excessive localized temperatures which may cause the shale to fuse. On the other hand, the quantity of gas in comparison with the supply of air must be kept below combustible mixture proportions, so that combustion will not occur in the mixer.

The wall-type mixer is composed of two separate halves, mounted diametrically opposite each other on the wall of the retort vessel. Although the operating principles are the same for both designs, the greater cross-sectional area of the wall type offers less resistance to the flow of preheated gas and causes a larger proportion of the gas to enter it. Furthermore, because this type of mixer does not restrict the flow of shale, there is less danger of clinkering. Better results have been achieved at high shale-throughput rates with the wall mixer than with the center type.

Results of several representative tests with the wall mixer are shown by figure 27. The shale feed was a blend of the series of beds comprising the Mahogany ledge and had the following properties:

Fischer assay:		
Oil content	gal. per ton	24.0
Water content	wt. percent	1.4
Gas plus loss	do.	2.9
Mineral carbon dioxide	do.	17.0
Ignition loss	do.	31.1

Additives were used to increase the supply of nuclei, and the same proportions of air and recycle gas were used in each test.

The gradual decline in oil yield with increasing shale rate depicted by the upper curve is attributed to incomplete retorting at the higher rates, as shown by the spent-shale-assay curve. The degree of mineral carbonate decomposition during retorting is a measure of the temperature to which the shale has been heated. At the higher shale rates the residence time is less, and the shale does not reach as high a temperature or remain hot as long as at the lower rates. As would be expected, the heat duty was not as great when the shale rate was high as when it was low. This is indicated by the lower air requirements.

Oil-Recovery Studies

Through cooperative agreements, manufacturers have provided several types of equipment that have been tested and found effective for separating the oil mist from the gas stream after it leaves the retort.

Some of the particles are large enough so that they can be separated from the gas stream by a small-diameter centrifugal separator. Two such units, one supplied by the Western Precipitation Corp. and one by the Thermix Corp., have been tested and found satisfactory. A hydrostatic baffle-type wet collector furnished by the American Air Filter Co. also operated satisfactorily. A wet-entrainment-type packing furnished by the Metal Textile Products Co. was found effective in recovering the larger particles of mist.

It has been found that the centrifugal force produced in most types of gas blowers is sufficient to agglomerate the small mist particles into larger ones that can be separated by any of these types of equipment.

An electrostatic-type precipitator supplied by the American Air Filter Co. has been particularly useful in evaluating other types of mist separators because the effectiveness of this type of separator is not limited by the size of the mist particles.

Intermediate-Size Pilot Plant

Although the present gas-combustion pilot plant has proved versatile and has provided important retorting data, a point has been reached where these data can be extended but little because of small retort diameter, limited bed height, and inadequate shale-handling facilities. A new pilot plant with a maximum capacity of about 50 tons a day is being designed and will have several advantages over the 6-ton-a-day plant.

The new retort will be able to process 3-inch shale, approximately the size to be used for commercial operations, whereas the older unit cannot handle material larger than about 1-1/2 inches. The new pilot plant will be capable of rates up to 450 pounds per hour per square foot of cross-sectional area, whereas the upper rate limit for the existing plant is only about 300 pounds per hour per square foot. In the new retort, larger cross-sectional area will permit extension of the limits of investigation of gas-air mixer design. Mechanical shale handling will be a great improvement over the manual system now used. Not only will data be obtained over wider ranges, but they will also be more accurate. In addition to the usual experimentation, the new plant will be useful in pilot operations on the gas-combustion demonstration plant being built.

The 50-ton-a-day retort will consist of a steel shell with a hung refractory lining. It is planned to make the cross section rectangular, 28 inches by 48 inches, and the height will be 18 feet. The present design for the product oil-recovery system includes a dynamic precipitator and an entrainment separator. A belt conveyor equipped for weighing and sampling will feed the raw shale. Existing equipment may be used for spent-shale disposal.

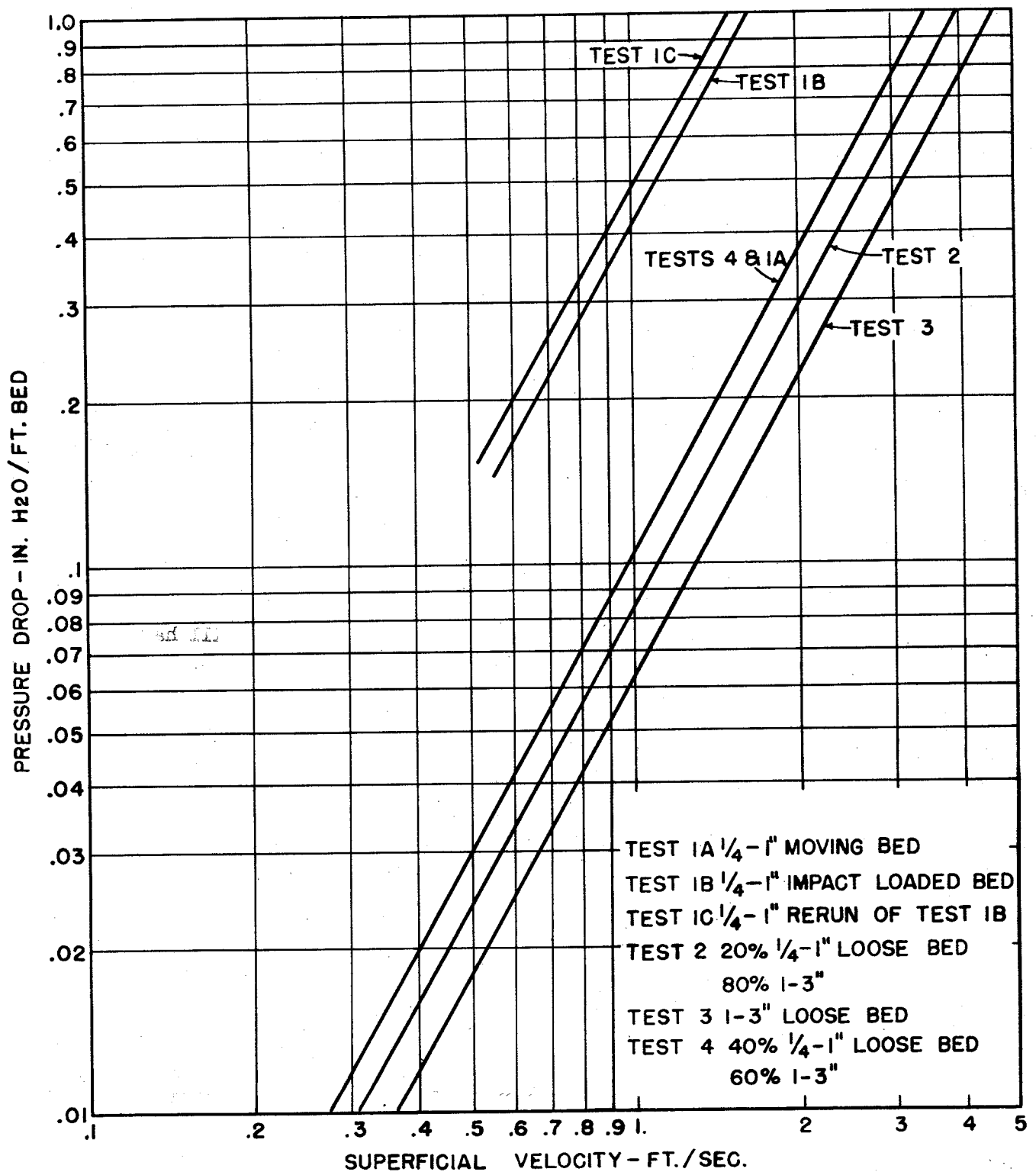


Figure 28. - Pressure drop vs. space velocity, gas-combustion pilot-plant retort.

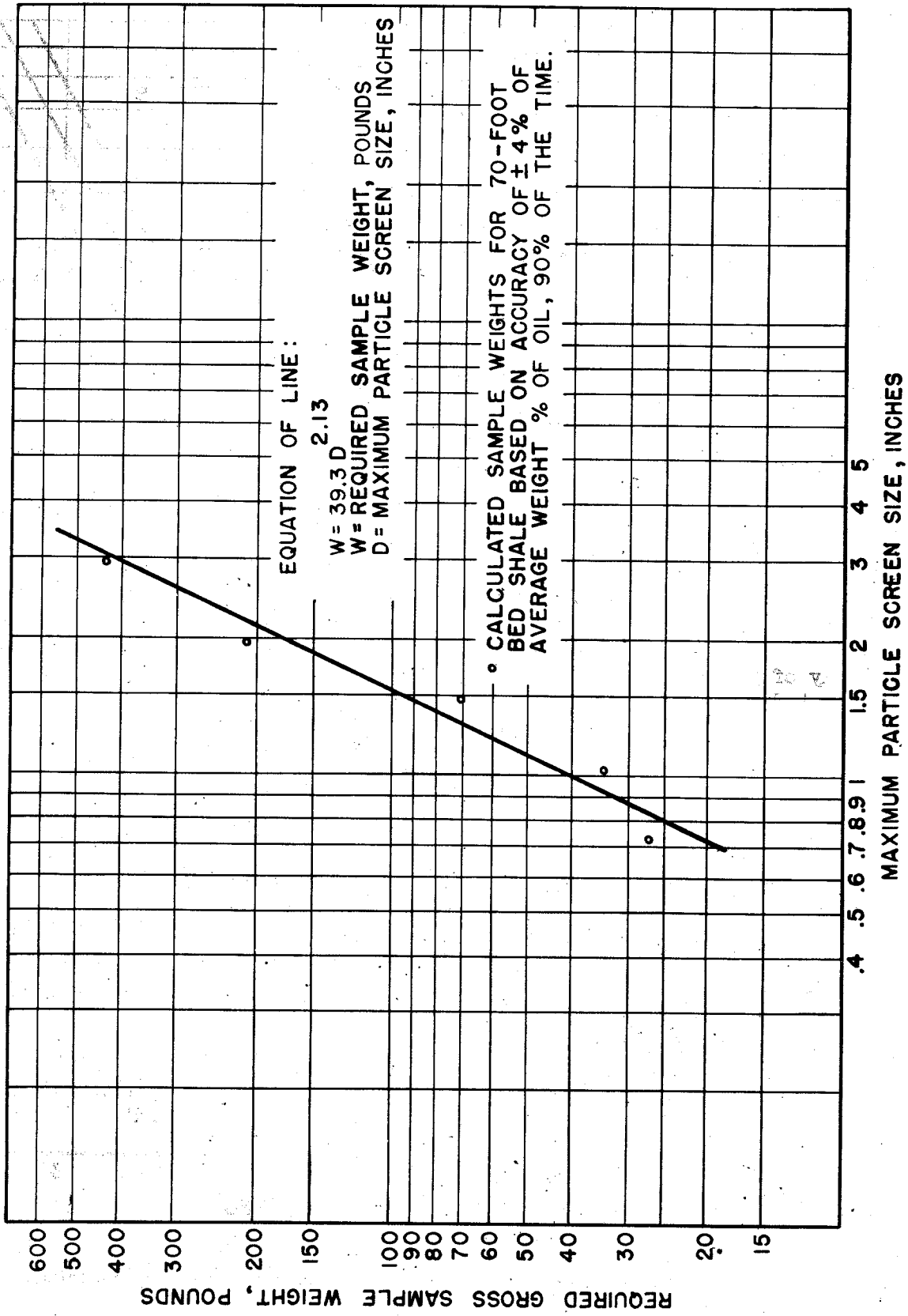


Figure 29. • Graph showing gross shale sample required as function of maximum particle size.

Gas-Flow Process

Pilot-plant studies of the gas-flow retort were discontinued early in the year to favor the gas-combustion process, which appears to have better commercial possibilities. However, it was recognized that considerable important data could be added to the fundamental knowledge of retorting by making several additional test runs. Beginning in September, it was found convenient to operate the unit for about 6 weeks, and several tests were completed. If the opportunity again arises, the plant will be operated for another short period to complete the final program.

The gas-flow process, has been described adequately in previous reports.^{2/3/} A complete report on the pilot-plant development of the gas-flow retort now is in preparation.

Miscellaneous Studies

Pressure-Drop Tests

Six tests were made in the gas-combustion retort to determine the qualitative effect of pressure loss in an air stream flowing through beds of crushed oil shale that had different void fractions. Increasing the void fraction by narrowing the particle-size range of the shale or by changing the packing arrangement of the particles in the bed decreased the loss in pressure at any given air rate. Figure 28 shows the results of these tests. The data have proved useful for retort design and process evaluation.

Sampling Raw Shale

A study of raw-shale sampling was undertaken and is still in progress. A literature study was made on statistical methods, and those that could be used were adopted. First, the variance of the Fischer-assay analysis was determined, and that variance included any errors incurred in the normal handling of raw-shale samples by the control laboratory. Next, a gross lot was split into five size consists, and the variance of Fischer assay was found for 10 equal sample increments from each size consist. These variances and the probability functions were used to calculate the number of increments required for a gross sample of predetermined accuracy for each of the five size consists. The weights of those gross samples were calculated from the number of increments and the weight of each increment. It was found that the required gross sample weight was a function of the maximum particle screen size in the lot of raw shale, and this function is shown in figure 29.

2/ Synthetic Liquid Fuels, Annual Report of the Secretary of the Interior for 1949, Part II - Oil from Oil Shale: Bureau of Mines Rept. of Investigations 4652, 1950, 70 pp.

3/ Synthetic Liquid Fuels, Annual Report of the Secretary of the Interior for 1950, Part II - Oil from Oil Shale: Bureau of Mines Rept. of Investigations 4771, 1951, 88 pp.