SINCLAIR REFINING COMPANY

1839 May 24, 1948

Translation Book 135
Reel 186
Frame 2499-2506

S-114

Report on the Carbonisation Operations
Carried Out on Pile M 1/II; 2-23-40, 9:00, to 2-25-40, 17:00

1) Carbonisation feed stock and construction of the pile.

Before starting the construction of the pile, dirt and the decomposed shale were taken off by surface mining. The shale which had thus been laid open was much permeated by layers of loam and dirt. At that time freezing temperatures prevailed and the ground was frozen. For this reason it was not possible to sort out the shale as completely as desirable. The shale to be carbonized was taken up by a dredger, 3 m^3 at a time, and transported to the pile field in four-lorry trains. When the lorries were dumped at the slope, the material was automatically separated into its component parts. Up to a depth of 80 cm, fines and dust came to lie at the surface. The big pieces of shale, the sides of which were longer than 300 mm., were broken up by hand. The limestone was sorted out by hand and used for filling mud holes on the pile field. The shale broke into much smaller pieces than the Schönberg shale. Thus, some more fine shale has been produced. Whenever the charge of a train had been dumped a plough was coupled in front of the empty train. The plough pushed the material from the trucks and pressed it against the slope. Thus it became unnecessary to beat down the surface by hand.

The weather was most unfavorable during the entire construction period. After the initial period of frost ... followed a heavy snow-fall. When the construction of the pile was terminated it started thawing and later on raining. Before we started the construction of the pile we put a substructure of lime-stones below the tubes which form the grate protecting the grate chamber with slate plates and surrounding the tubes with fine shale for a distance of 6 m. from the grate towards the receiver as a protection against scorification and as a preventive measure against the sucking in of false air.

2) Kindling material.

(a) Kindling shale.

The material for preparing kindling shale has been specially sorted out from the dredger. By means of open box cars (0.8 m^3) it has been transported to the grate of the grizzly (Schüttelrieb). There it was sorted out again. When the kindling shale was ready, the pieces which were 10-50 mm. long, were placed upon the well-roughened surface of pile 1. This pile had been constructed from very poor material to begin with; then it had been exposed to changing weather conditions for about two months. For this reason a specific amount of kindling shale was applied, e.g. 137 kg/$m.^2$, as against the results of the Schönberg experiments, calling for a charge of 60 kg/$m.^2$. For, we wanted to make sure that the surfaces would really catch fire.

The amount of kindling shale has not been taken into consideration when we computed the output, assuming complete combustion.

1840

(b) Peat.

We found that pulling apart the small peat sods by hand results in less abrasion dust and mold than breaking them up with hatchets. We made an experiment mixing the specific amount of peat (10 kg/m.^{2}) with a small quantity of minced machine-cut peat, but that turned out a complete failure.

(c) Wood shavings

As wooden ignition agents we chose one part wood shavings and one part excelsior. The latter material stood the test very well, for it keeps smoldering for a longer time after it has burnt out and it is better at igniting the subjacent peat than the hitherto used shavings. We shall try to reduce the quantity of shavings and excelsior which had been used hitherto.

Table I

Ignition Agents	Tons	kg/m.^{2}
Kindling shale	98	137
Peat	7.0	10.0
Wood shavings	0.7	1.0

3) Ignition and progress of the experiment

After the ignition agents had been put on, the electric current failed owing to enemy action. Since the repairs took a longer period of time we protected the ignition material against rain and snow by covering the pile with canvas. The following day at 9:00 a.m. the horizontal surface was ignited and three hours thereafter the lateral faces. Contrary to our misgivings, the ignition of the entire pile was a success, excepting the right slope which had to be re-ignited 8 hours after the test had been started. Ten hours after the start of the experiment tube (1) was closed. Up to that time it had been regarded as an auxiliary tube for the slope. The progress of the experiment was easy to observe since all the tubes of the grate had been equipped with U-manometers.

The negative pressure rose initially in a uniform manner in all tubes, but after the tenth hour it dropped substantially in the medium tubes. Later on, however, it rose again; only tubes (8) and (13) lagged behind. All through this test, the temperature in these tubes was hard to control. In the ninth hour the (?) with 4 filters was started (filter (4)) having the most porous oil receivers (Oeltauchgrube) was kept in reserve). Filter (2), (3) and (5) failed frequently. They had to be disconnected. During the entire operation only one filter separator was available. The repairing of the failing filters has been started right away. It has been found that in setting them up the hookup of the heating wires used for heating the insulators had been mixed up. Apparently some of the filters have become defective for this reason.

(Confer Table II)

4) Measuring spots

For checking operating conditions, thermometers and U-manometers were mounted at the following spots:

At all of the tubes of the grate, at the receiver, at the measuring diaphragm in the pile field at building 52b in front of and behind the E.C.R., in front of and behind the blowers and in the by-passing pipe line to the combustion furnace.

In order to have the blowers safely under control, pressure and negative pressure, temperature and number of amp. have been checked at each of the individual blowers in the E.C.R.; moreover, the heating of the insulators, the intensity of the current and the tension for each of the filters have been checked.

5) Gas-throughput and O_2 content

The course of the experiment has been plotted giving on the abscissa the operating hours, the date and the time by hours, on the ordinate at the left-hand the gas throughput in $m^3/hr.$ (at 20° and 1 hr.) and at the right-hand the percent by volume in the residual gas.

On starting operations all the three blowers were started with a total gas throughput of 57, 500 $m^3/hr.$; that is, 62 m^3/m^2, in order to make sure that the ignition would be a success. After the ignition had succeeded, one blower was switched off (in the ninth hour) and the gas throughput was reduced to 29,500 $m^3/hr.$ This rate has been maintained up to the thirty-ninth hour when it was again reduced to 14,500 $m^3/hr.$ because the temperature had substantially gone up. In principle, we wanted to maintain a carbonisation velocity of 7.5 cm/hr. A maximum velocity of 10 cm/hr. should not be exceeded. Towards the end of the experiment (50 hours) the temperature at the tubes of the grate went down. Therefore, the gas throughput had to be raised to 23,000 $m^3/hr.$ The O_2-contents, determined in the residual gas dropped quickly to 9% up to the ninth hours, and to about 5% up to the twentieth hour, remaining at this rate until the end of the experiment with slight fluctuations.

Gas samples to be analysed have been drawn at the 21, 32 and 14 hour from the receiver of the pile. In all of these cases we found the O_2-contents to be 3%. We found several leaks and sealed some of them. False air might most readily be sucked in because the condenser in the first tap line was not impeccably packed. We tried to seal this leakage by packing it with clay, but at the hot gas pipe the clay dried quickly and cracked.

6) Yields of condensate and how it was worked up.

The following containers were available for collecting the condensate:

(a) One 6 m^3-container at the end of the gas pipe-line at the level of the 2, connecting line (building 52a).

(b) One 6 m^3-container in the long gas pipe-line which has a cooler effect and rises steeply in front of the E.C.R. by virtue of the terrain (building 52b).

(c) The 100 m^3 raw-product reservoir.

(d) The phenol water ditch.

The first condensate was obtained in the 5th hour at building 52a(water). The first oil condensate was obtained in the same container during the 8th hour. During the entire carbonisation period nothing but water was obtained at building 52b. The entire condensate obtained has been pumped to building 11, insofar as it did not flow automatically to this place from building 9 and 10, following the natural incline.

The condensate obtained in the raw product reservoir has been heated by the immersion heaters. The phenol water separated and was pumped to building 50 and the emulsion to building 100. Here the emulsion was heated up to 70° by means of the two preheaters and introduced into one of the three settling vats. One of the two excelsior filters has been passed empty, the dismulgan unt has been by-passed because dismulgan was not available. The water and the sludge separating out in the settling vat at a temperature of 80° were drained into the sludge ditch. When the setting up of the centrifuge will be terminated we shall work up the sludge.

Table III

Condensate Obtained	To	%
Building 52a	72	44.7
" 52b	24	14.9
" 9 and 10	65	40.4
Total condensate obtained	161	100.0

7) The pile after the termination of the carbonisation

When the experiment was terminated the phenol water collected in Building 50 was pumped off and used for quenching the adjacent pile which the carbonization had spread to. Dirt was kept ready in case surface fires should evolve due to the condensation and ignition of residual oil remaining in the pile. Only here and there, however, appeared small oil spots at the rim of the slope without igniting. The entire length of the pile collapsed in the middle and towards the slope in a depth of no more than 50 cm., without crater formation. The pile cooled quickly without forming incandescent slag on the surface. After putting the receiver farther back, slag has been stripped from the slope (about 20 m^3) for readying the second field. We found thereby and likewise on drawing out the tubes that the pile had been carbonised uniformly and completely in a downward direction. Only over a few of the tubes of the grate a slight layer (about 20 cm. thick) of sintered material had been deposited.

Operational and output data

Pile surface	730	m^2
Pile Volume	1110	m^3
Pile weight	1700	tons
Pile height	3.30	m *
Bulk weight	1.5	to/m^3 **
Period of operation	51	hr.
Velocity of carbonisation		
Velocity of carbonisation in 1/3	3-4	cm

Remarks: * The height has been measured from the midst of the tubes of the grate.

** No suitable balances being available, we used the figure determined for Schömberg shale.

Velocity of carbonization in 2/3	6	cm.
" " " in 3/3	8-10	cm.
Oil obtained	27.8	to
Water in oil	2	%
Oil obtained minus water	26.46	to
Emulsion	24	to
Water in emulsion	77	%
Oil in emulsion	5.5	to
Total oil obtained	31.96	to
Total water obtained	130.0	to
Total gas throughput	15.585.0	m^3/hr.
Gas throughput/shale	900	m^3/to = 7.7% ***
Oil contents according to Fischer	4.5	%
Total oil content of the pile	76.5	to
Yields according to Fischer	41.75	%

Remarks:

*** This value is only an estimated one. An average sample could not be drawn, for the wiring for a circulatory pump which is being set up had not been terminated. We analyzed the upper layer finding only traces of water. A sample withdrawn from the bottom near the sludge pipe showed a water content of 4.7%.
We have not included in our ouput figures the amount of condensate which remained in the pipelines, preheaters and excelsior filters.

**** Because of the lack of suitable laboratory equipment we made the assumption that the pure shale contained 5% of oil and deducted from this figure 10% for decomposed shale, dirt and limestone.

6) Withdrawing the tubes

The tripod which had been developed at Schömberg in the course of several months for withdrawing the tubes had not yet been completed. Two days after the carbonization had been terminated we started withdrawing the tubes with temporary equipment, expediting at the same time the completion of the Schömberg equipment. Contrary to our expectations, eight of the tubes could be withdrawn comparatively readily without major efforts. The receiver has sometimes been used as an anchorage. None of the tubes were damaged or corroded excepting tube (8) which broke apart at a round weld seam. It must be noted that this weld seam had been improperly made. It was evident that it had been partly burust before the tube had been used.

Ten grating tubes are still in the pile. They will be withdrawn when the tube-drawing equipment will be ready. This will be done with tripot tubes, mainly because the receiver suffered a break when the pulley was attached to it on drawing the middle tube.

9) Technical defects observed during carbonization

The most important defect is the lack in careful construction of the "Tauchgruben" at the blowers into which the waste waters of the main gas lines are

obtained on the pressure and on the suction side. While the cavities permit only a maximum plunge of 200 mm, the pressure was much higher on the pressure side of the blowers. This fact caused carbonisation gas to flow in a continuous stream from the "Gruben" into the entire blower station rendering the servicing very difficult, yea, perilous for the operators. .....

Several flange connections were leaking, in particular within the blower station; in the main gas line drainage was lacking owing to an adverse gradient; suction baskets were missing at the suction lines of the sludge ditches....

M. Beth

MB/HH

Att. (Graph may be traced upon request).

cc: All Division Heads