

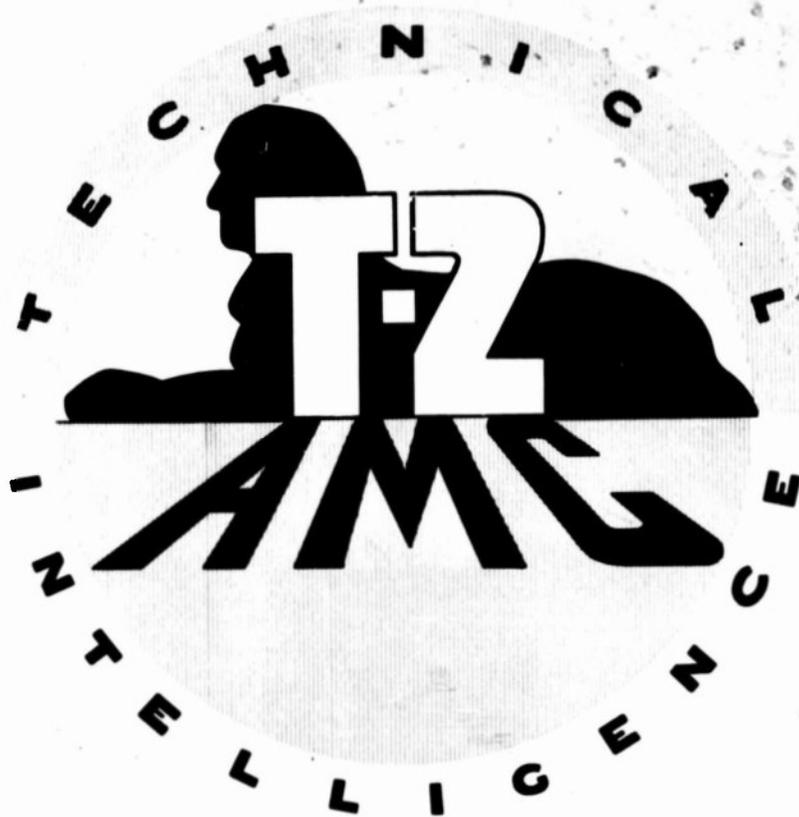
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FRENCH OIL SHALE INDUSTRY

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Reported by
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INTRODUCTION.

This is a brief report of visits to oil-shale distillation plants in France, made by William Odell and Emile L. Baldeschwieler during the period June 26 to July 1, 1945, inclusive. The French Government is exerting efforts to develop the known deposits of shale, particularly those in central and south central France, for the purpose of supplying a portion of their requirements of motor fuel and lubricating oil; that country does not have a natural ready source of supply of petroleum. Because of the efforts being made in France and also on account of the general interest of the Allied Nations in the production of oil from other raw materials than petroleum, it was desirable that information should be obtained as to advancements made in the art in France. The inspection trip was taken with the permission of the French Government and the authors were accompanied by a French engineer, M. Bernard de Hesseguier, who also traveled under permission.

The officers and other representatives of the companies whose plants were visited were most gracious and accorded us every facility for obtaining information from their records and notes.

I. - AUTUN.

The first plant visited was that of the Societe Minière des Schistes Bitumineux at Autun. It was found that the latter company is operating 3 batteries of 40 retorts each of Pumpherson type. These retorts are exact duplications of the well-known Scotch units in which shale is distilled in Scotland. Crushed shale varying in size from 4 inches down to about $\frac{1}{2}$ -inch is treated continuously by passing it downward through the vertical retorts which are externally heated by burning gases. The upper portion of each retort is made of metal (iron) and the lower portion, which is subjected to higher temperatures, is made of refractory material. The capacity of each retort was initially 3 tonnes per day of 24 hours of oil shale; this was at a time when super-heated steam only was passed upwardly through the mass of shale in process, but later results obtained when both air and steam were introduced adjacent to the bottom of the retort during processing showed an increase to 7 tonnes of shale per day per retort. Thus the total maximum capacity with all retorts operating is 840 tonnes of shale per 24 hours. The air is used internally to burn some of

the carbon which otherwise would pass out as carbon in the spent shale; in this manner additional heat is generated for processing without the consumption of additional extraneous fuel. Although there were 350m³ of gas generated from each tonne of shale treated, an additional amount of gas (producer gas separately generated) was required for heating the retorts. The amount of the producer gas used was the equivalent of 100 kgs. of coal per 100 liters of crude oil recovered; the coal contained 30% ash.

The shale treated at Autun appears to differ chemically from that of most American shales, at least the oil obtained from heat-treating the shale is said to contain less wax. The oil characteristics are not reported here because they are published rather completely elsewhere. However, it is noted that the shale being treated yields 6 to 7 percent of total readily condensable oils by the Fischer assay retort and that in plant operation the yield is 85 percent of Fischer yield. The shale contains 2.0 percent of total sulphur of which 1.5 percent is pyrite and the remaining 0.5 percent is organic sulphur. The softening point of the shale is 1100°C.

The mining is by underground methods, the shale being 325 feet below the surface in a single seam varying from 3.25 to 11 feet in thickness. The mining costs are high.

The number of men employed operating the plant are given as follows:

Men operating mine	500
Men in other operations	<u>400</u>
Total	<u>900</u>

Thus, at full capacity of 840 tonnes per day, the labor amounts to 1.07 men per tonne of shale mined and treated.

It is noted that the usual condensing and cooling methods did not remove all of the oil and that there was recovered by oil scrubbing the cooled gas 7 to 8 liters (1.85 to 2.1 gallons) per cubic meter of shale, that is, per 1.2 metric tonnes of shale. This amounts to 1.4 to 1.6 U.S. gallons per U.S. ton (2,000 lbs.) of shale treated. The total recovery of oil was 66 liters per tonne, of which 6 liters were obtained by oil scrubbing. In U.S. units the recovery was 15.8 U.S. gallons per

ton of 2000 lbs., of which 1.44 gallons was recovered by oil scrubbing. The crude oil has a specific gravity of 0.90.

The shale used had a relatively low lime content and there were no difficulties regarding residue disposal. A representative analysis of the spent shale is as follows:

Si O ₂	51.52 % by weight.
Ti O ₂	0.87
SO ₃	1.82
Fe ₂ O ₃	17.50
Al ₂ O ₃	6.61
Mn O	Trace
Ca O	5.75
Mg O	0.50
K ₂ O	5.07
H ₂ O	0.70
Loss on ignition.	<u>9.00</u>
	99.44

The company has concluded that the developments in the art of treating oil shale have been such that they are no longer satisfied in building this type of retort and have expressed their intention to replace these retorts as fast as they wear out by a different type of retort which appears to offer substantial economies and increased earnings. The retort which the company has decided to employ, replacing the Pumpherton unit, is that which has developed at St. Hilaire, and which is known as the Lantz retort.

II. ST. HILAIRE.

The next plant to be visited was the St. Hilaire (Allier) installation of the Société Chimique de la Grande Paroisse. A pilot plant has been in operation there since 1938, treating 80 tonnes of shale daily. The apparatus and process employed were unique and different from those employed elsewhere for treating oil shale. The retort which is vertical is comprised of two distinct but non-connecting sections; each section is traversable by a gas stream. The lower section is the distillation portion of the retort and has a rectangular cross-section. Its outer walls comprise a series of superimposed baffles which support the shale and which function as louvres through which gases can enter the retort. This section is encased in a metal shell and means are provided where-

by hot gaseous products evolved in the process are caused to pass horizontally through the shale confined between the baffles while said shale is passing continuously downwardly through the retort. A circulating fan is supplied for propelling the gases and a preheater is also connected in the system whereby the circulating gases are preheated to 450-500°C. before entering the retort. Separately generated producer gas is employed along with the excess retort gas in the preheater to supply the necessary heat energy. The waste or stack gas from the preheater is caused to pass through the shale in the upper section of the retort whereby said shale becomes heated to about 150°C. before entering the distillation step in the lower section. In this process the shale which is initially supplied to the retort in crushed 1-2" size pieces is heated gradually to a temperature not exceeding that of the circulating gases, namely 450-500°C. and the oil and vapors evolved are immediately removed from the shale in process and passed into the circulating system from which 10% is continuously withdrawn for the recovery of shale oil and gasoline.

The spent shale is discharged from the retort continuously into water from which it is removed by a screw conveyor. This quenching step not only cools the spent shale but generates steam through evaporation of water, which steam is mixed into the circulating gas stream. The spent shale discharged from the conveyor still contains 10 to 12% by weight of carbon which ignites in the air and burns in the residue pile in spite of the quenching treatment.

The shale used has a calorific value of 1900 calories per kilogram and that of the spent shale is 950. The oil content of the shale according to the Fischer retort test is 6.8% by weight; substantially all of this is recovered in the processing described. It is noted that the spent shale contains 1.2% of sulphur much of which is burned to sulphur dioxide in the discharging residue. During the processing there is evolved 25 m³ of gas per tonne of shale and the calorific value of this gas after removing the gasoline vapors therefrom is 6000 to 6500 calories per m³.

The company felt that the performance of the pilot plant was so satisfactory that they were warranted in building a larger size unit; one such unit designed to treat 500 tonnes of shale per day is under construction. It is estimated that the water requirements (make up water) will be 125 m³ per day for each 500 tonne unit.

In other words, 70 U.S. gallons of water are required for each 2000 lbs. of shale treated. The overall power consumption for handling and treating the shale from the mine clear through the plant handling and treating the shale from the mine clear through the plant is estimated to be 9-10 KW per tonne of shale. Two gas producers are installed for a 500-ton unit, one being reserved as spare. Sixty pounds of coke are used in gas producing for every 2000 lbs. of shale treated. The total fuel required for retorting in the 500-ton unit is 60 lbs. of coke and 856 cu. ft. of shale gas per ton of 2000 lbs. of shale. The coke used is: water 5%, combustible 75%, and ash 20%, and it has a calorific value of 5500 calories per kilogram or 9900 B.T.U. per lb.

The future plans of this company include expanding the capacity to 2000 tons of shale per day. A burning device has been developed whereby the heat energy represented by the 12% of carbon in the spent shale will be utilized in the future operations. This will no doubt eliminate the necessity of using coke for producer gas.

The total yield of oil is substantially 132 lbs. per 2000 lbs. of shale.

III - Lavernhe and Séverac-le-Château.

The next and last plants visited were at Lavernhe and Séverac-le-Château in south-central France. The Lavernhe plant was formerly operated by the Société de Bitumes et Schistes, a subsidiary of Laverhe Monhand et Aulnoye, while the Séverac plant was operated by the Compagnie Industrielle des Schistes Bitumineux de l'Aveyron (CISBA), a subsidiary of the Compagnie Alais Forges et Camaigne (AFC). These two present companies have now merged and are being operated under the name of Compagnie Industrielle des Schistes Bitumineux de l'Aveyron (CISBA).

It was found at Lavernhe that an entirely different type of retort for processing shale had been rather completely developed. This retort is known as the Marécaux retort, after the inventor who is a chemical engineer for CISBA.

This apparatus has been in operation on a pilot scale for more than a year, treating up to 30 tons of shale per day. The retort is vertical and comprises two concentric upright cylindrical shells which confine between them a mass of downwardly moving crushed shale in process. Means are provided for charging crushed

shale (3 to 25 mm) into the upper portion of the retort and for discharging the treated shale continuously at the bottom. The shale is externally heated through the outer cylindrical metal casing and the gases are withdrawn adjacent the bottom of the retort. The retort differs materially from the others in that the inner cylinder rotates slowly and has on its outer surface helical members which cause the particles of shale in the annular space to be agitated and turned over during their downward travel through the retort, thereby facilitating heating of the shale. No air is introduced and therefore there is no internal combustion in the operation of this retort. Means are provided, however, for introducing superheated steam at 400°C. into the annular mass of shale at a plurality of levels to facilitate distillation. The distillation gases are withdrawn into the inner cylinder substantially as they are formed in the shale mass, thus minimizing cracking of the oil vapors. All of the gas evolved from the shale is used for heating the retort along with an additional amount of producer gas. The shale charged is previously treated with the hot stack gases for the purpose of economizing heat and raising the temperature of the raw charge to 150°C. The rotating inner cylinder has a water seal connection which prevents air entering the retort at the top.

The total yield of oil from shale from this retort is 41.83 kg. per metric ton of shale treated which amounts to 92% of the standard Fischer retort yield. In addition, there is present in the gas prior to scrubbing 3.70 kg. of gasoline; this gasoline is not now being recovered because of the high content of H_2S in the gas and because the plant is only a pilot plant. The yield of gas per metric ton of shale treated is 13.6 m^3 and 25% of this gas is H_2S . The total amount of producer gas used in the pilot plant per metric tonne of shale treated is 330 m^3 . The steam used amounts to 33.4 kg. per metric tonne of shale. Although this furnace has not been tried on a larger scale, the smoothness of the operation of the pilot plant indicates that the operation should be equally smooth on a larger scale with substantially the same operating results. The shale residue contains 4% carbon.

The company is so satisfied that this type retort is economical and an improvement over standard practice that two commercial size units of 120 tons each are under construction at their plant in Severac.

Since the merger of the two companies, plans have been formulated for a large scale study of shale processing. It is proposed that there will ultimately be installed shale processing equipment capable of treating 3000 tons of shale per day. At this plant there is now under construction one Petit furnace, one Lantz retort and two Marecaux retorts as named above. The Petit retort is being installed chiefly because the material is available, and not because it offers any advantages over the other retorts developed in France.

Description of the Petit Retort.

This retort, developed at Grenoble with the help of CISBA on a pilot scale only, has never been operated on a scale larger than 8 tons per day. The process differs from others chiefly from the fact that the shale, prior to the distillation, is wet with the shale oil from previous distillations; the oil-wetted shale passes through a horizontal rotating, externally heated, cylindrical retort comprising two concentric cylinders. The shale in process is caused to pass serially through the inner cylinder and back to the reverse direction through the spaces between the said cylinders, discharging substantially at the inlet end of the retort. The object of the oil-wetting treatment is to cause solvent action and depolymerization of the kerogen.

Due to the fact that only a small portion of the retort heating surface is in contact with the shale in process at any one time, and because the general nature of the methods employed for applying heat to this retort, it is doubtful that it will be of interest in the treatment of American shales.

The writers wish to acknowledge the whole-hearted cooperation of the directors and officers of the companies contacted and their engineers who were most cordial and helpful to us in obtaining the picture of the shale situation in France. Very definitely, advances in the art have been made as a result of the researches of the French scientists.

W. W. Odell.
E.L. Baldeschwieler.

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ABSTRACT

Three French oil-shale distillation plants were inspected by representatives of U.S. Bureau of Mines and U.S. Petroleum Administration. French Government is attempting to develop known deposits of shale for the purpose of supplying a portion of their requirements of motor fuel and lubricating oil. Data are given on equipment, capacity, production of plants, and chemical composition of shale. Brief description of a new retort process developed in Grenoble is included.

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