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THE
OIL-SHALE INDUSTRY
OF ESTONIA.

BY

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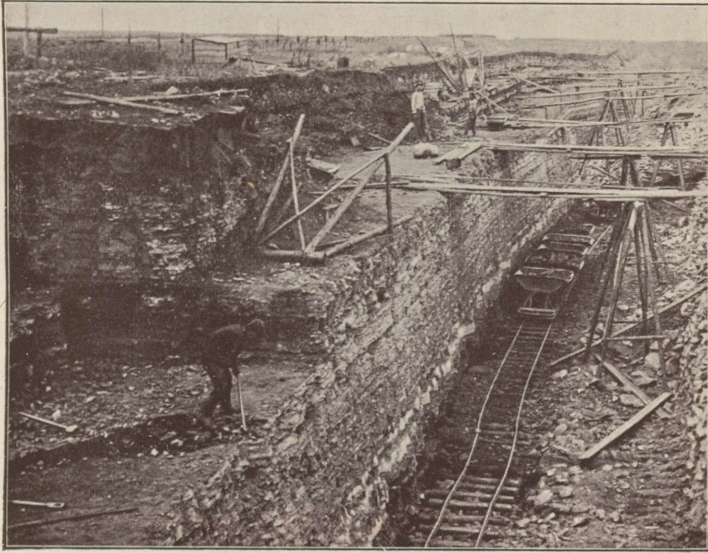


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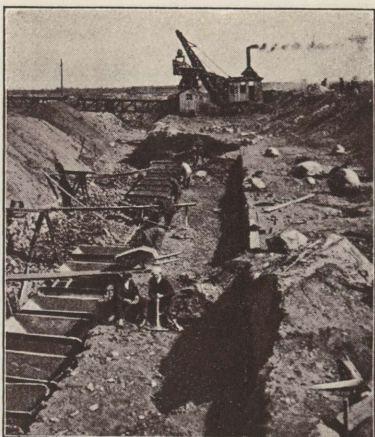
Oil-shale quarries at Kohtla.

The Oil-Shale Industry of Estonia.

THE DAWN OF THE INDUSTRY.

One hundred and thirty-five years ago an oil-bearing mineral was discovered in the North of Estonia. The mineral, known now as "Estonian Oil-Shale" or "Kukersite" was described in a report in 1798 as "a brownish laminated argillaceous or marly bituminous earth; it burns with flame, without giving an unpleasant odour. Shepherds burn this earth in piles."

The mineral seems to have been forgotten, and only fifty years later was it rediscovered by accident,



Oil-shale quarries at Kohtla, showing the work of a steam shovel.

when a stove, made of bituminous limestone and shale slabs, burnt down. In the middle of the last century an agricultural gazette published a paper, the author of which had taken the shale for "guano deposits". To disclose the mystery of these deposits, a chemical investigation was carried out at the University of Dorpat (Tartu) under the direction of Professor C. Schmidt. The chemical analysis proved the shale to be very rich in organic matter, but the percentage of phosphorus to be practically nil. The rich shale deposits remained unexploited until the Great War had destroyed the transport facilities in Russia, and the Petrograd district was suffering greatly from shortage of fuel. The Russian Fuel Commission then appointed a geologist, N. F. Pogrebov, to investigate the deposits of oil-shale and to estimate the supply available. In May 1916, Pogrebov and his fellow-workers investigated the oil-shale deposits over an area of about 50 square kilometres along the Tallinn (Reval) — Petrograd railway line, and a production of 2 tons of shale



Underground mine at Kohkla.

per square metre was estimated. Trials on a large scale carried out at the Petrograd Gas Works having given satisfactory results, it was decided to start mining operations at Kohkla for the production of 150,000 tons of shale annually. The preliminary work in this mine had to be suspended in October 1917, owing to the Bolshevist Revolution. During the German Occupation of Estonia, from February to November 1918, the Germans also tried to exploit the oil-shale, and samples were sent to Berlin for analysis. In November 1918, the Estonian Government took over the mine from the German authorities.

Up to this date only the preliminary work, which consisted of drainage and the building of a railway line $2\frac{1}{2}$ miles long to Kohkla Station, had been completed. A gravel pit about 50 metres long and 4—5 metres wide represented the whole mine, and the

only equipment left by the German authorities was 3 locomotives and 60 dumping cars.

The actual exploitation of oil-shale was started by the Estonian Government on May 5th 1919, after the Soviet Russian „Red Army“ had been defeated and thrown back over the Narova river, and has since then continued without interruption. For the working of the mine the “State Oil-Shale Industry“, financed by the State, was established, and under this administration two mines are now working: the Kohtla mine, open cut and partly underground mining, – and Kukruse mine, with underground working system only. The mines are situated 4–5 kilometres north of the Tallinn-Narva railway line, between the stations Jõhvi and Kohtla. In addition to these mines a third one, the Vanamõisa Mine, which was leased in 1923 to a private company, the “Estonian Oil-Shale Development Syndicate, Limited“, working with British capital, was established.

In 1922 a fourth mine, owned and managed by the “Eesti Kiviõli (Estonian Shale-Oil) Company“, started work between the stations Püssi and Sonda, and recently a fifth mine started operations at the station Maidla. The Püssi Mine is situated 6 km. (east) from the station Sonda. The mine has a drainage canal (length – 1,9 km., depth at the head 6 m.). A narrow gauge railway connects the mine with the experimental distillation plant, the length of the line being 3,8 km. The mine possesses 2 locomotives, 60 HP. each, and 84 dumping cars, with 1,5 cubic metre capacity each.

The company has an experimental distillation plant, a power station, a fully equipped chemical laboratory, work shop, dwelling houses, etc. The Government has so far issued over 30 permits for the prospecting of oil-shale.

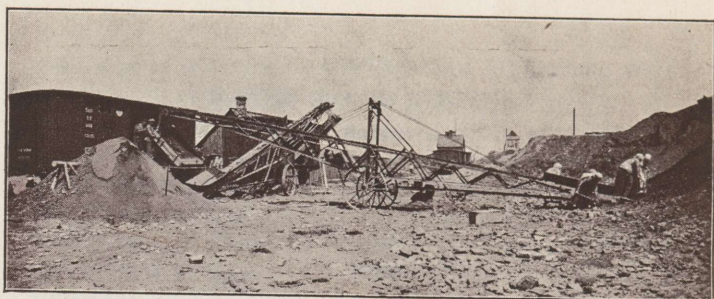
The mining of oil-shale has made rapid progress in the last six years, the annual production of oil-shale in the State mines having been as follows:—

Table I.
OUTPUT OF OIL-SHALE.
(In metric tons).

Years	State Oil-shale mines			Private companies			Total
	Kohtla	Kukruse	Vana- mõisa	E.O.D. Syn- dicate, Ltd.	"A./S. Kütte- Jõud"	"Eesti Kiviõli A./Ü."	
1918—1919	9.648	—	—	—	—	—	9.648
1920	45.844	—	281	—	—	—	46.125
1921	84.511	3.740	7.276	—	—	—	95.527
1922	127.410	11.522	—	—	—	3.200	142.132
1923	177.000	29.000	—	3.340	—	6.600	215.940
1924	194.710	32.935	—	712	—	1.713	230.068
1925	238.658			8.740	25.081	15.625	288.104
1926	334.130			7.677	48.458	37.414	427.679

At present the State Works at the Kohtla-Jaerve mine own 21 locomotives and more than 600 dumping cars. The railway line is 25 miles long, and the colony consists of 66 dwelling houses and 332 tenements. In addition there is a distillation plant, an experimental refinery, power stations, workshops, limekilns, storage rooms, etc. The new power station has a capacity of 500 kilowatts. The cost of the buildings and equipment amounted to 108 000 000 Estonian marks (approximately £ 67 500) and the cost of materials and rolling stock, — 58 000 000 Estonian marks (approximately £ 36 250).

There is also a post-office, bank, school, hospital, an efficient telephone system, etc. The "State Oil-Shale Industry" employs about 20 chemists and engineers, 200 clerks and foremen, and about 1 400 workmen.



Loading of oil-shale at Kohtla.

At the Vanamõisa mine an oil distillation plant with a pair of "Fusion" retorts has been erected. The company does not market the oil-shale, but uses it only for distillation at its own mine. The mining is carried out on the same system as in the Kohtla mine. The soil and glacial deposits are usually removed by hand and a steam shovel (excavator), and are transported to the places where mining operations have been completed. Operating in this way, the exploited land can be transformed again into ground suitable for afforestation. The underground working is also not difficult.

The oil-shale is the most popular and cheapest fuel in Estonia, and its use for general heating purposes is steadily increasing; factories and large industrial enterprises, railways, cement works, etc., are now using oil-shale as a substitute for coal.

Since the establishment of the "New Distillation Plant" at Kohtla, the Shale-Oil Industry has been put on a firm basis.

THE OIL-SHALE.

The Estonian Oil-Shale is one of the oldest and richest oil-shales in the world. The oil-shale deposits occur in the Middle-Ordovician strata, the whole

formation attaining a total average thickness of 2,2 metres, over an area of about 2400 sq. kilometres.

The colour of the oil-shale varies from greenish-yellow to reddish brown. Air-dried pulverised shale from the seams A and B looks very much like cocoa-powder. The freshly mined oil-shale is hard, only the weathered shale being soft and brittle. The hardness of oil-shale exceeds that of coal. The chemical composition of the organic matter in different seams does not vary greatly. The table shows the chemical composition of different oil-shale seams:

Table II.

COMPOSITION OF AIR DRIED OIL-SHALE.

(Analysis by Prof. M. Wittlich & N. Veshnjakov,
University of Tartu, Estonia.)

Seams	A	B	C	D	E	F
Sp. gr.	1,57	1,56	1,61	1,81	1,54	1,65
	%	%	%	%	%	%
H ₂ O . . .	3,1	1,3	2,7	2,1	2,3	2,3
O	35,6	37,5	35,8	25,4	37,4	34,1
H	4,2	4,5	4,2	3,1	4,4	4,1
C	9,9	9,6	10,1	7,2	9,6	9,2
N	0,1	0,1	0,1	0,07	0,1	0,1
S	0,7	0,5	0,6	0,4	0,8	0,5
CO ₂ . . .	10,3	13,1	8,6	10,4	9,4	12,3
SiO ₂ . . .	15,3	9,4	18,0	26,4	14,7	14,4
Fe ₂ O ₃ . . .	1,3	2,9	2,0	1,9	1,8	2,0
Al ₂ O ₃ . . .	4,0	2,1	4,0	4,6	4,1	3,5
CaO	13,3	16,8	11,9	13,8	12,6	15,6
MgO	0,6	0,5	0,9	1,4	0,7	0,6
Na ₂ O, K ₂ O	1,9	1,2	1,0	3,1	2,3	2,0
P ₂ O ₅ . . .						

t r a c e s.

According to the author's analyses the percentage of sulphur in all seams is slightly higher (1,0–1,4%) than given in the above table.

Table III.
COMPOSITION OF SOME OIL-SHALES.

Location	Oil Yield. Galls. per ton. Crude & Scrubber Naphtha	Per cent crude dist. to 275°	Volume of Gas Cub. Ft. per ton	Spent Shale, per cent	Sp. gra- vity, Crude & Scrubber Naphtha
Soldiers Summit Utah	49,68	37,2	1110	74,96	0,894
Grand Valley, Colo	36,71	38,5	943	80,58	0,899
Brazil	118,69	40,5	2146	34,0	0,870
Scotland	17,66	37,6	731	86,7	0,872
Estonia*).	74,0	39,0	—	40,0	0,938

Table IV.
CHEMICAL COMPOSITION OF THE ORGANIC
MATTER OF OIL-SHALE.

Seams	A	B	C	D	E	F
% of organic substance of air dried oil-shale	49,8	51,7	50,2	35,7	51,5	47,5
	%	%	%	%	%	%
C	71,5	72,4	71,3	71,1	72,4	71,6
H	8,4	8,7	8,4	8,6	8,5	8,6
O	19,9	18,7	20,1	20,0	18,9	19,6
N	0,2	0,2	0,2	0,3	0,2	0,2

Oxygen taken by difference (the figures include also % of S)

Table V.
COKING TEST OF OIL-SHALE.

Seams	A	B	C	D	E	F
	%	%	%	%	%	%
Volatile subst. . .	56,7	60,1	54,2	42,7	56,6	50,4
Coke	4,8	2,9	6,4	4,3	7,4	9,7
Ash.	38,5	37,0	39,4	53,0	36,0	39,9

*) Slightly different method was used for distillation.

The State Oil-shale mines are marketing the oil-shale in three different qualities which are described as follows:—

Table VI.
MARKETABLE OIL-SHALE QUALITIES.
(Analysis by Kohftla laboratory.)

Quality	I	II	III
Description of Shale	Sieved in lumps	Unsieved lumps and fine	Fine, soillike oil-shale
Percentage of moisture in summer	13—18	15—20	15—25
in winter	18—25	20—30	20—30
Percentage of ash (mineral ash plus CO ₂) in raw oil-shale . .	37—43	39—47	42—51
in the dry substance .	50	55	60
Percentage of combustible substance	38—44	31—39	28—34
Calorific Value . . .	3000—3500	2300—2700	2200—2400
Price F. O. B. at the Mine in E. Mks per pood (36 lbs.)	10	7,50	5,00
in shillings per ton .	7/9 ^d	5/10 ^d	3/10 1/2 ^d

The calorific value of an air-dried average sample is 4400 cal (9900 B. Th. Us.).

OIL-SHALE RESOURCES.

Recent drilling has shown that Kukersite strata worth mining cover an area over 2400 square kilometres, so that the investigated shale reserves amount to 5000 million tons. More oil-shale will certainly be found in unexplored areas, so that, including the probable shale reserves, the total amount would reach about 5500 million tons. The oil-shale strata in Estonia show almost an uninterrupted geological structure, and only thin out towards the West.

THE USES OF OIL-SHALE.

I. THE CONSUMPTION OF OIL-SHALE AS FUEL.

The consumption of oil-shale as fuel for all kinds of boilers has been steadily increasing. Experiments made during the year 1919–1920 showed that furnaces with fire-grates constructed for the burning of coal or wood were not suitable for burning raw oil-shale, and therefore furnaces of a special semi-producer type have been constructed. At present several large factories in Estonia have had their boilers fitted with special fire-grates and are using oil-shale for power production.

1 kg. of shale requires for complete combustion (theoretically) 8 m³ of air: the gases contain:

CO ₂	— 1,33	m ³
SO ₂	— 0,01	„
N ₂	— 6,30	„
	<hr/>	
	7,64	„

Percentage of CO₂ in gases = 17,4.

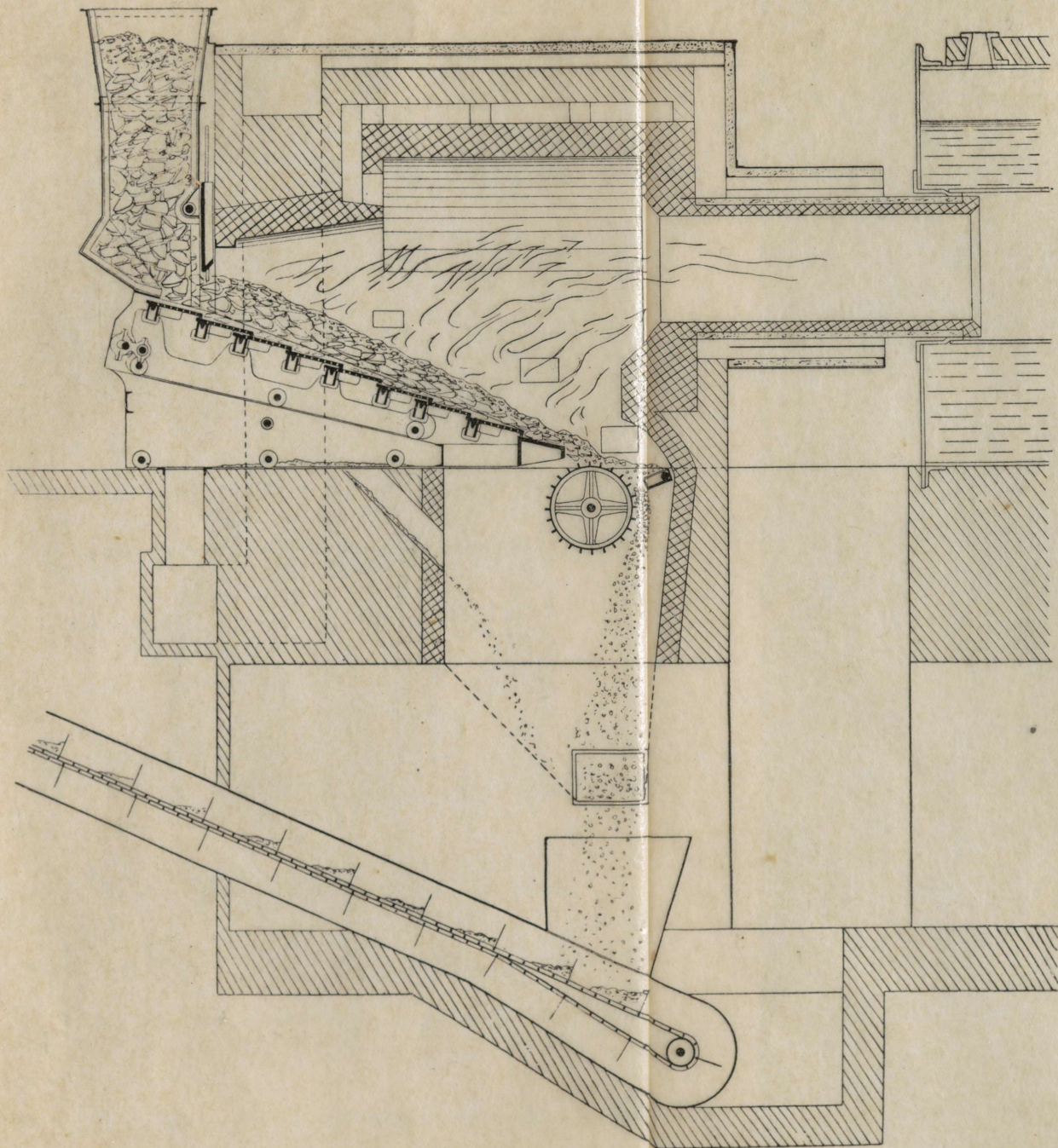
In February 1924, the Ministry of Communications appointed a Committee to inquire into the fuel problem of the State Railways. After investigation a final report was drawn up and accepted by the Committee on the 23-rd October, 1924, which may be summarized as follows: —

“At the present cost of fuels, oil-shale is the cheapest: if oil-shale is employed in the heating of locomotives instead of coal, there is an economy of 22 per cent, and if employed instead of wood, the economy is much greater, amounting to 43 per cent“.

At the oil-shale mines, oil-shale can be used for burning lime in kilns and for domestic use.

II. The biggest consumers of oil-shale have so far been the Portland Cement factories, the Cement works — Port Kunda and Asserin — using the cheap oil-shale of the III quality. The oil-shale is dried,

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Modern fire-grate for burning oil-shale.

Courtesy Baltic Mills, Ltd., Tallinn.

pulverized, and injected (by air) into revolving kilns. The oil-shale ash remains an ingredient part of cement clinkers. The quality of the cement is the same as obtained by using coal.

III. OIL-SHALE CONSUMED FOR THE PRODUCTION OF GAS.

The gas plants at Tartu (Dorpat) and Tallinn have only partially adopted oil-shale for gas production. The oil-shale is carbonized in old type, horizontal retorts, which of course are not quite suitable for the carbonization of shale. The yield of gas is 10000 cu.ft. (283 m³) per ton of oil-shale, the yield of tar 4–5 0/0. The chemical composition of the gases obtained at Tallinn is given below: —

Table VII.
CHEMICAL COMPOSITION OF KUKERSITE AND COAL GASES.*)

	Percentage of							Calorific value cal./kg.
	CO ₂	Heavy hydrocarbons	Benzol	CO	H ₂	CH ₄	N ₂	
Kukersite II quality. (March - April 1924, July 1926)	13,3	6,64	0,56	19,3	26,18	22,64	11,38	6000
Gas coal, British (January - April 1926)	3,1	2,62	0,28	8,04	49,0	30,56	6,4	5393
Gas coal, German (August 1926)	2,06	3,5	0,29	8,45	50,5	28,25	6,95	5500

*) From: Die Revaler Gasfabrik, by H. v. Winkler, „Das Gas- und Wasserfach,“ 1927, 16. Heft.

The use of coal for gas plants is preferred mainly for two reasons: 1) to obtain the extremely valuable by-product — coke, and 2) to avoid the cost of ash removal.

At present about 10—20 per cent of gas is obtained from oil-shale. The shale-gas does not require any special treatment for purification.

The annual fuel requirements in Estonia for oil shale alone is given below.

Table VIII.
OIL-SHALE REQUIREMENTS IN ESTONIA.

Portland Cement Works	170000 tons.
Other factories	340000 "
Railways	200000 "
Small consumers	40000 "
	750000 tons.

THE USE OF ASHES

The ashes remaining from oil-shale after carbonization and burning, when finely ground, have the properties of Roman cement and are used as a binding material for building purposes.

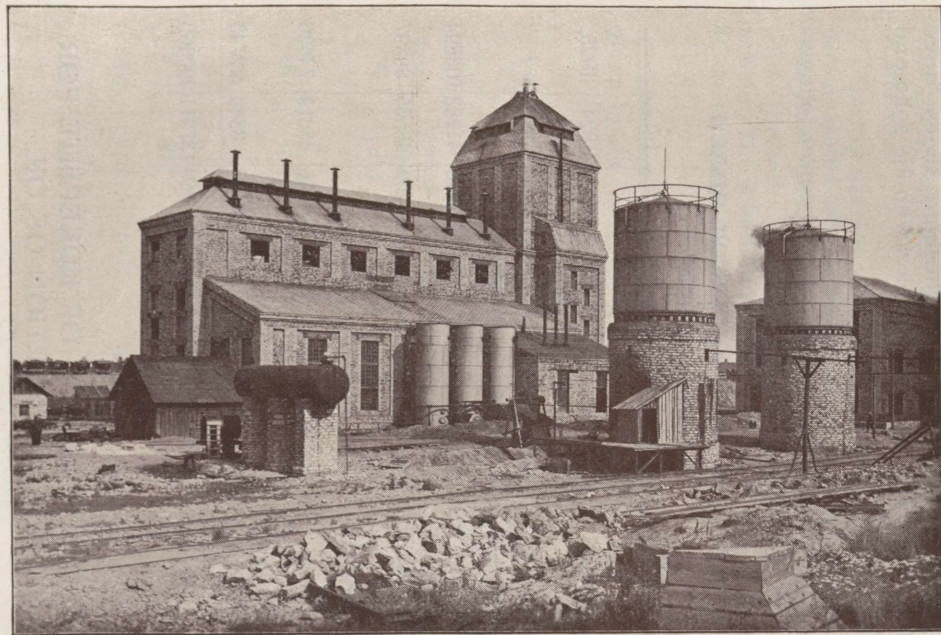
Table IX. shows the composition of the ashes: --

Table IX.
RANGE OF COMPOSITION OF ASHES.

CaO	26—50 ⁰ / ₁₀
SiO ₂	27—51 ⁰ / ₁₀
Al ₂ O ₃ , Fe ₂ O ₃	12—17 ⁰ / ₁₀
MgO	1,5—3 ⁰ / ₁₀
K ₂ O, Na ₂ O	3—6 ⁰ / ₁₀
S	0,5—3 ⁰ / ₁₀

Tensile strength of the ash test-pieces was as follows:

	After 28 days
Shale ash (in air)	12—17 kilogr.
" " (in water)	12—20 "
Portland cement	25—40 "
Good mortar	3—4 "



Oil-shale retorting plant at Kohla.

The shale ashes as a binding material have consequently half the value of Portland cement.

Up to the present the Oil-Shale Mines have chiefly supplied the home market with the solid and liquid fuels.

The consumption of oil-shale has been as follows :

Table X.
THE CONSUMPTION OF OIL-SHALE.
(In metric tons).

Consumers.	1918—19	1920	1921	1922	1923	1924	1925
Portland Cement Factories	1135	12288	37244	113554	102995	41283	103894
Railway	424	8923	9234	14903	34209	46565	89397
Fuel, Gas Factories and experiments	8089	24914	10824	5775	18856	45619	74626
Distillation plants	—	—	981	2202	3200	3344	22743
TOTAL	9648	46125	58283	136434	159260	137811	290660

THE DISTILLATION OF KUKERSITE FOR THE PRODUCTION OF OIL.

The production of oil from the shale is the most important of its uses. This main branch of the oil-shale industry has a world-wide importance, and is a most difficult problem to deal with.

LABORATORY EXPERIMENTS.

It is well known that the character and the yield of liquid distillates from a given fuel vary greatly, being dependent upon the temperature and the manner of distillation, as well as upon the nature of the fuel.

During the years 1919–1920 the author carried out a series of experiments at the Fuel Laboratories, Imperial College, South Kensington, London. The method of operation is described in an article "The Chemical Composition of the Estonian M.-Ordovician Oil-Bearing Mineral 'Kukersite'".

The results of the first series of experiments are given below: —

Table XI.
DISTILLATION OF OIL-SHALE AT VARIOUS TEMPERATURES.

Temp. °C	Percent- age yield	Yield of oil. Galls per ton	Yield of Gas cu. ft. per ton at 0° & 760 mm	Yield of Ammo- nia	Calorific value of Oil in B. Th. Us.
410	27,1	63,3	1900	—	—
500	29,7	72,9	2250	—	17028
600	30,8	74,8	3000	0,02	17428
700	27,5	65,0	4500	0,04	—
900	21,7	49,7	7200	0,11	—

The yield of the oil is also dependent upon the weathering of raw oil-shale. Table XII shows the results of distillation of four different samples of oil-shale.

Table XII.
RESULTS OF DISTILLATION OF OIL-SHALE, FROM
SEAM B. (KOHTLA), SHOWING THE EFFECT OF
WEATHERING UPON THE YIELD OF OIL.
(Analysis by Kohtla laboratory.)

	Un-weathered		Weathered	
	Moist	Dry	Slightly	Strongly
Analysis of the Shale.	‰	‰	‰	‰
CO ₂	12,6		10,4	6,6
Ashes	38,9	do	33,7	46,8
Organic matter . .	48,5		55,9	46,6
Distillation.				
Oil	34,1	33,0	33,2	21,3
H ₂ O	2,2	3,5	4,5	5,0
Coke	55,7	56,4	54,1	65,2
Gas (by difference)	8,0	7,4	8,2	8,5
Specific Gravity of oil	0,923	0,912	0,923	0,922
Analysis of Coke.				
CO ₂	21,5	21,2	18,4	10,1
Ashes	68,2	68,3	61,9	71,3
Organic matter . .	10,5	10,5	19,7	18,6
The coefficient of utilisation of organic matter.				
Oil	70,3	68,0	59,4	45,7
Water	4,5	7,2	8,0	10,7
Coke	11,8	12,6	19,1	26,0
Gas	16,5	14,6	14,5	18,2
	103,1	102,4	101,0	100,6
‰ of unsaturated compounds in oil	—	83,0	77,0	69,0

The distillation tests were carried out in Fischer's aluminium apparatus.

The results given in Table XII show that the yield of oil from weathered shale is lower than from the unweathered.

Frank E. Weston (late head of the Chemical Department at the Polytechnic, Regent Street, London), in a private report, gives the following average yields of oil and gas: —

Yield of crude oil	42 per cent
" " water free oil . .	40,8 " "
" " gas	12,0 " "

Average total volatile matter 54 per cent.

Sp. gravity of oil = 0,9545.

Fractionation of crude oil:

Light oil	6,7 per cent
Heavy oil	8,3 " "
Residuum	50,4 " "

Sulphur content of crude oil about 0,37 per cent.

Hydrogenation of kukersite. Two series of hydrogenation experiments have been carried out on kukersite (i) at the Oil-Shale Research Laboratory, University of Tartu, Estonia, by P. N. Kogerman & A. Tamm, and (ii) at the University of Zurich, Switzerland, by J. Kopwille.

The main results of those researches might be summarized as follows:

- (i) the total yield of oil does not increase to any considerable extent on hydrogenation;
- (ii) the oil obtained on hydrogenation contains more light oil; the oil is more saturated, and
- (iii) the amount of phenolic bodies in the crude oil decreases considerably;
- (iv) the crude oil (from hydrogenation) yields high grade light and motor oils.

EXPERIMENTAL DISTILLATION OF KUKERSITE ON A COMMERCIAL SCALE.

Four experimental distillation plants have been working on a semi-large scale in Estonia. (i) A modified, vertical Scotch retort, with external heating and superheated steam, erected at Tallinn, was running from 1919 to 1921. This plant proved to be uneconomical, although it yielded about 15 per cent of high quality crude oil. (ii) The second retort, of producer type, designed by J. Pintsch and Co.,

Berlin, started operations at the Kohlla Mine in 1921. The plant has now been rebuilt and equipped for refining shale-oil. A vertical cast-iron shaft oven with a fire-brick lining inside formed the producer. The external diameter of the producer was 2 metres and the height 5 metres. The shale sank in the producer by its own weight. The producer worked continuously and the throughput in 24 hours amounted to about 7 tons of shale. The yield of crude oil was about 20% of the weight of raw shale.

The analyses of the products of distillation are given in the Table XIII: —

Table XIII.

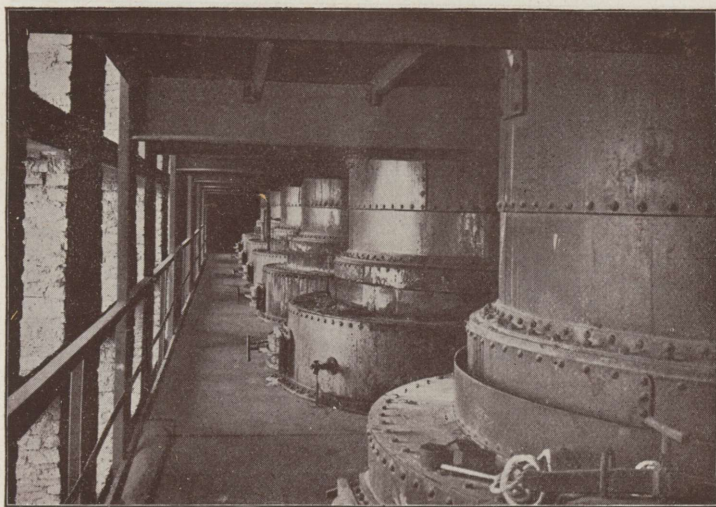
	Analysis of the oil, shale used	Analysis of semicoke obtained	Analysis of ashes obtained
	%	%	%
H ₂ O . . .	13,8	—	—
CO ₂ . . .	9,8	15,81	3,33
Ash . . .	34,8	68,38	89,83
Org. matter	41,6	15,81	6,24
	100,0	100,0	100,0

From the data given above, the yield of semi-coke and ashes is as follows: —

Table XIV.

YIELD OF SEMICOKE AND ASHES.

	Analysis of the oil-shale used	Analysis of semicoke obtained	Analysis of ashes obtained
	%	%	%
H ₂ O . . .	13,8	—	—
CO ₂ . . .	9,8	8,05	0,98
Ash . . .	34,8	34,80	34,8
Org. matter	41,6	8,05	2,65
	100,0	50,9	38,43



Pintsch's producer-retorts; Kohtla Oil-Works.

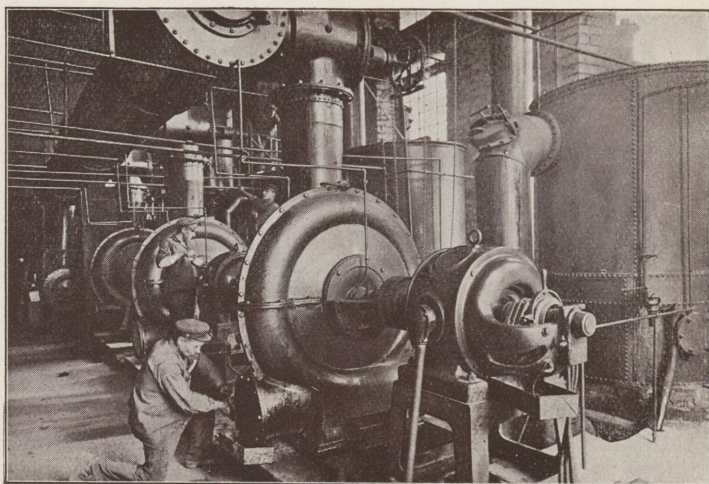
The semicoke contains on an average 15–17% of fixed carbon.

Sp. gravity of crude oil (with 1% of H₂O only) is 1,009 at 15° C, viscosity — 5,5 (Engler) at 50° C.

The ultimate composition of the oil is as follows:
 C — 81,26%, H — 10,15%, O — 7,26%, S — 1,08%
 and N — 0,25%. The dry shale-oil contains:

Neutral bodies (chiefly hydro-carbons)	72,1%
Phenols	22,4%
Carboxylic acids	4,0%
Bases	0,2%
Loss	1,3%
	100,0%

On the basis of the above-mentioned experimental achievements a large oil retorting plant with a battery of 6 retorts has been erected. The throughput of the new distillation plant is 200 tons



Kohtla retorting plant, showing exhausters and scrubbers.

of oil shale in 24 hours, and the quantity of crude oil would amount to 40 tons daily. The new plant started operations in 1925.

The working of the plant is illustrated by the attached scheme.

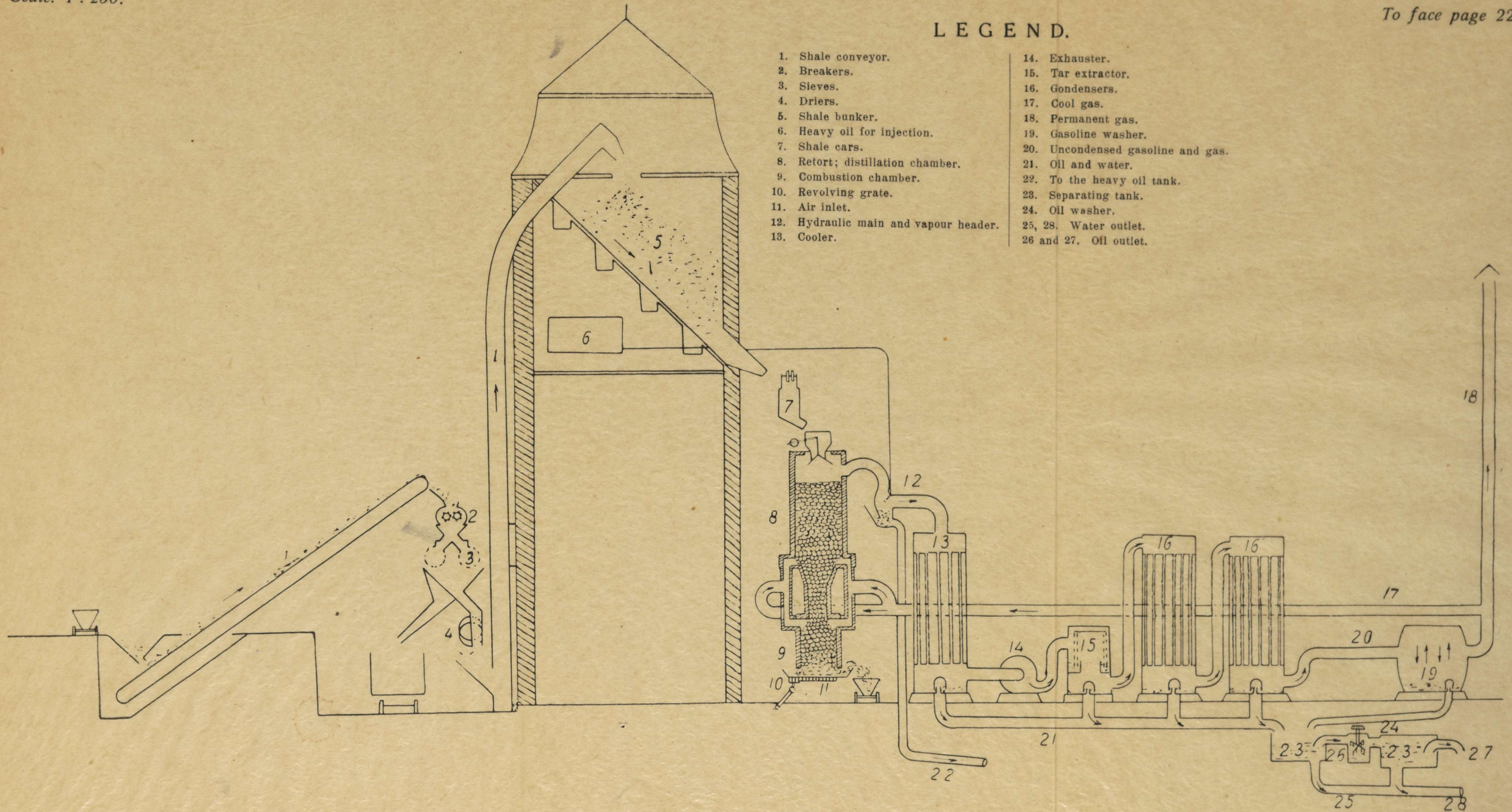
The circular producer-retort and pre-heater form one vertical unit. The shale is carried by a conveyor to the crushers (breakers), which consist of horizontal toothed rolls that revolve slowly toward each other. The distance between the rolls may be varied. From the breakers the shale is mechanically carried to the sieves on the top of the retort house and there discharged into the electrically driven cars. In the retort the shale sinks by its own weight and drops into the combustion chamber. The shale is heated by hot gases which pass from the combustion chamber to the retort through the annular flue canal. Oil vapours pass out at the tops of the retorts into the hydraulic main and from the main to the condensers.

Scale: 1 : 250.

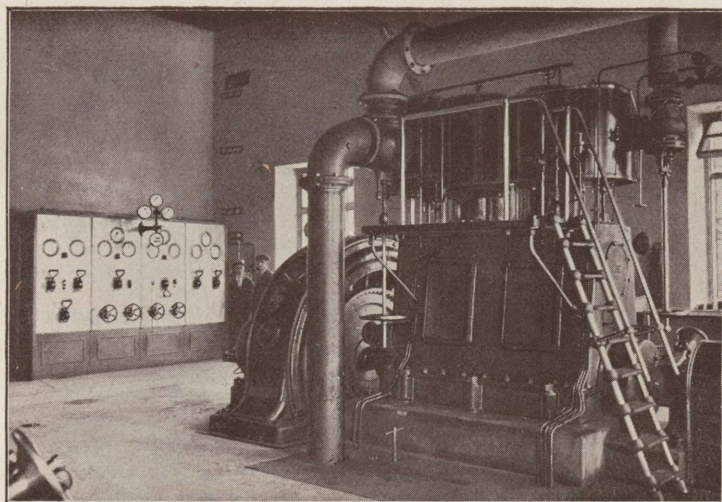
To face page 22.

LEGEND.

- | | |
|---------------------------------------|-----------------------------------|
| 1. Shale conveyor. | 14. Exhauster. |
| 2. Breakers. | 15. Tar extractor. |
| 3. Sieves. | 16. Condensers. |
| 4. Driers. | 17. Cool gas. |
| 5. Shale bunker. | 18. Permanent gas. |
| 6. Heavy oil for injection. | 19. Gasoline washer. |
| 7. Shale cars. | 20. Uncondensed gasoline and gas. |
| 8. Retort; distillation chamber. | 21. Oil and water. |
| 9. Combustion chamber. | 22. To the heavy oil tank. |
| 10. Revolving grate. | 23. Separating tank. |
| 11. Air inlet. | 24. Oil washer. |
| 12. Hydraulic main and vapour header. | 25, 28. Water outlet. |
| 13. Cooler. | 26 and 27. Oil outlet. |



Kohtla retorting plant.



Kohtla retorting plant ; power station.

The crude oil obtained in the new retorts is somewhat lighter than that obtained on distillation in the experimental retort, but the yield of crude oil is practically the same.

Properties of Crude Oil. — An analysis of the crude shale-oil, carried out in the Kohtla laboratories, gave the following: —

Sp. gr. at 15° C.	0,98 to 1,01.
Reaction	Neutral.
Moisture	About 1,0 per cent.
Flash point (Pensky-Martens)	Over 80° C.
Viscosity at 50°	3,5 to 4,5° E.
Cold test	—10° C.
Unsaturates	75 to 90 per cent.
Insoluble in normal benzine	1 to 2 per cent.
Coke test (in crucible) . . .	9 to 11 per cent.
Sulphur	Below 1 per cent.

Mechanical impurities . . . 0,05 per cent.
Calorific value. 9800 cal./kg.

Distillation test:

Up to 200° C. 3—5 per cent.
200 to 250° C. 10—12 per cent.
250 to 300° C. 10—12 per cent.
300 to 360° C. 30 per cent.

Diesel Oil — The properties of this product are: —

Flashpoint(Pensky Martens) Over 60° C.
Viscosity at 50° 1 to 1,3° E.
Unsaturates 40 to 50 per cent
Insoluble in benzine About 1 per cent.
Coke (in crucible) 3 to 4 per cent.
Sulphur 0,5 to 0,9 per cent.
Mineral matter Under 0,05 per cent.
Moisture About 1 per cent.
Calorific value. 10,000 cal./kg.

Distillation test:

Up to 200° C. 5—8 per cent.
200 to 250° C. 15—20 per cent.
250 to 300° C. 20—30 per cent.
300 to 360° C. 30—40 per cent.

Pitch. — The distillation residue of the crude oil has a melting-point of 70—90° C., and is suitable for use in the manufacture of roofing paper and as an insulation material.

Lubricating oil fractions have been investigated so far very superficially, although good lubricating oils have been obtained on laboratory scale.

Goudron: melting point (Kramer-Sarnov) 40—50° C. Insoluble in benzine 1—2 per cent.

Asphalt (mastic) m. p. about 100° C, bitumen content 30—35%. The shale asphalt is of good quality, and is used as a substitute for natural asphalts.

VANAMŌISA SHALE-OIL.

System of Retorting. — Horizontal, externally heated "Fusion" retorts are used, the retorts being manufactured by Messrs. Vickers, Ltd., and the oil condensers and benzol recovery plant by Messrs. Meldrums, Ltd, Manchester. The throughput of a single unit, two of which have been installed, is 24 tons of dry shale per 24 hours, the yield of oil being 50 to 60 galls. per ton of dry shale.

Properties of Crude Oil. —

Sp. gr. at 15° C.	0,950 to 0,969.
Viscosity at 50° C.	1,6° E.
Moisture	1,1 per cent.
Dust and free carbon	0,54 per cent.
Unsaturates	About 27 per cent.

(Note. — 1 vol. oil + 1 vol. benzine + 4 vols. H₂SO₄ 80%).

Distillation test (Engler):

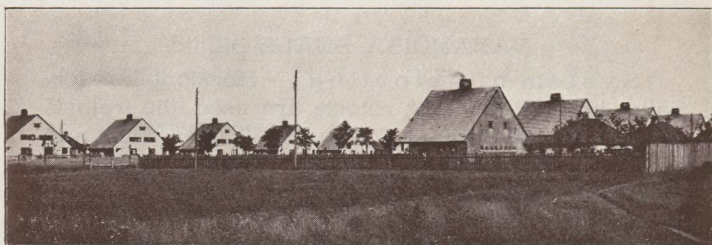
Amount of oil taken	92,2 g.
I. B. P.	70° C.
Up to 150° C.	7,7 c. c.
150 to 175° C.	5,9 c. c.
175 to 200° C.	4,6 c. c.
200 to 225° C.	6,0 c. c.
225 to 250° C.	5,9 c. c.
250 to 275° C.	6,9 c. c.
275 to 300° C.	6,4 c. c.

The shale was also retorted in the "Fusion Retort" in Great Britain, where a yield of about 63 gallons per ton of crude oil was obtained.

The oil analysed by F. Mollwo Perkin had been divided into two fractions, termed medium and heavy.

On analysis of the "Medium oil" the following figures were obtained: —

Sp. gr. at 15° C.	0,8462
Sulphur	0,93%
Calorific value	18 640 B. T. Us.
Flash Point	56° F.



Dwelling-houses at Kohtla.

This higher content of sulphur is probably due to the fact that other oil containing more sulphur than the Estonian had previously been passed through the Condensers.

Fractionation:

Up to 170° C.	60,25 0/0.
170—230° C.	17,00 0/0.
230—360° C.	12,50 0/0.
Residue above 360° C.	10,50 0/0.

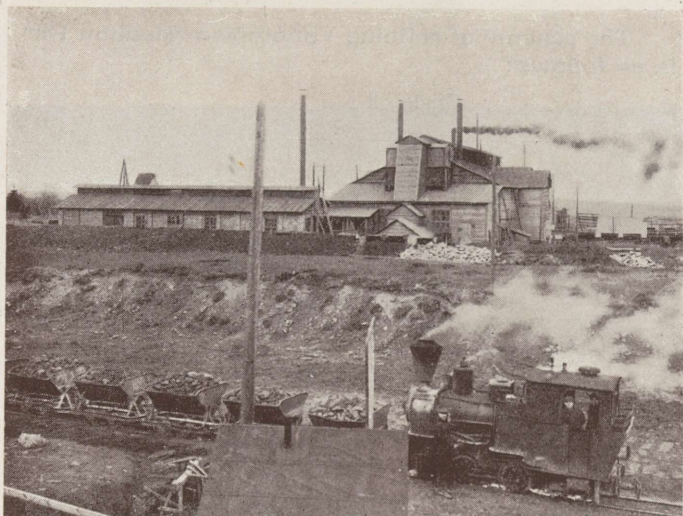
The following figures were obtained on analysis of the "Heavy Oil".

Sp. gr. at 15° C.	0,9273.
Sulphur	0,93 %.
Calorific value	18640 B. T. Us.
Flash Point	101° F.

This high content of sulphur may also be accounted for as in the Medium Oil.

Fractionation:

Up to 170° C.	14,1 0/0 (Spirit).
170—230° C.	15,4 0/0
230—360° C.	52,5 0/0
Residue above 360° C.	18,0 0/0 (Soft pitch).

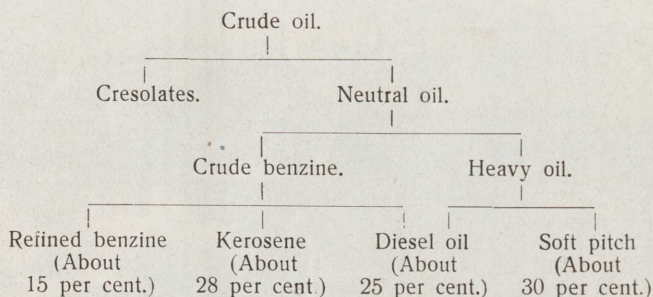


View of retorting plant of the Estonian Oil Development
Syndicate, Ltd., at Vanamõisa.

It will be noticed that the yield of spirit up to 170° C. in the Medium Oil is very high and on treating with a little acid and again fractionating is water white. This very high yield of spirit must, however, be taken in conjunction with the fact that the fraction from which it was taken was the topping from the heavy oil. The Heavy Oil contained 14,1% of Petrol.

Although the refinery is not yet equipped for the production of all the possible products from the crude oil, steps are being taken to instal the necessary plant as soon as possible. During the last years some refined products were obtained at the chemical works of Messrs. Richard Mayer & Co., Ltd., Tallinn, and these have proved highly satisfactory.

The scheme of refining Vanamōisa "Medium Oil" is as follows: —



Benzine. — A very high quality of aviation spirit and motor spirit is obtained: —

AVIATION SPIRIT.

I. B. P. 45° C.
 50 per cent. at 100° C.
 94 per cent. at 150° C.
 End point . . . 165° C.

MOTOR SPIRIT.

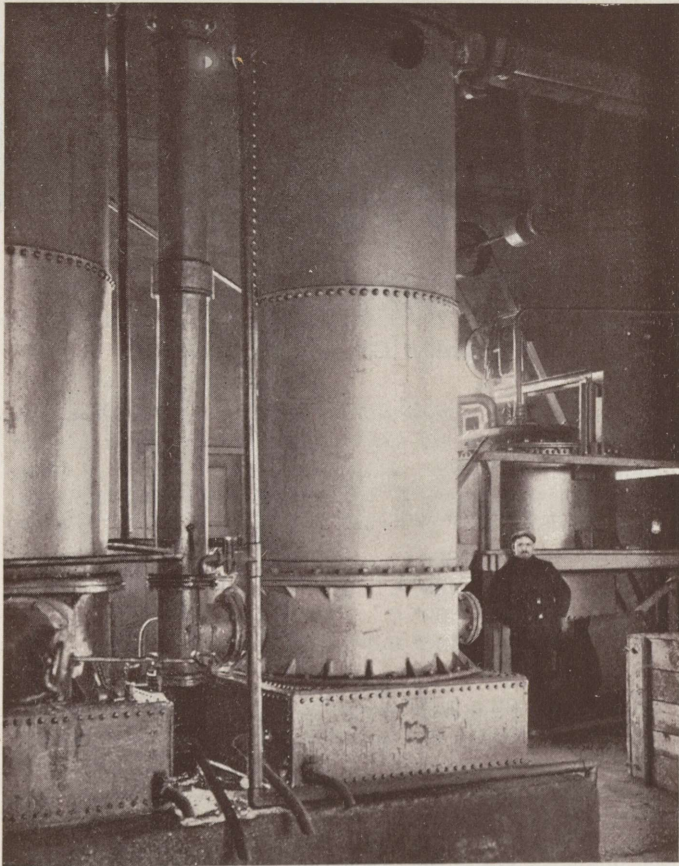
I. B. P. 58° C.
 28 per cent. at 100° C.
 87 per cent. at 150° C.
 97 per cent. at 170° C.
 End point . . . 178° C.

Colour: Water white.

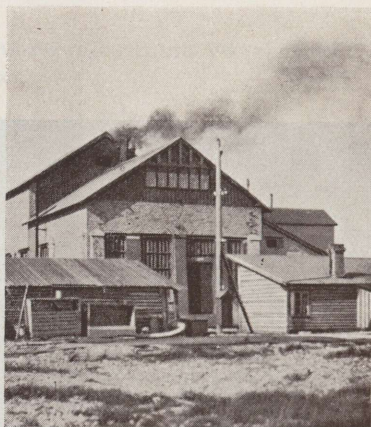
Kerosene. — The kerosene burns in an ordinary lamp with a somewhat smoky flame, owing to the high content of unsaturateds and the fairly high sulphur content, 0,68–0,77 per cent. The specific gravity at 15° C. is 0,813–0,850.

Diesel Oil — This has been used in Diesel engines with good results, the properties being: —

Sp. gr. at 15° C 0,899
 Calorific value 10,200 cal./kg.
 Viscosity at 20° C. 1,2° E.
 Flash point 55 to 60° C.
 Coke test 0,5 per cent.
 Asphalt (with benzine) Traces.
 Sulphur 0,62 per cent.



Vanamõisa retorting plant. Condensers and scrubbers.



"Eesti Kiviõli" Company plant at Püssi.

Distillation test:

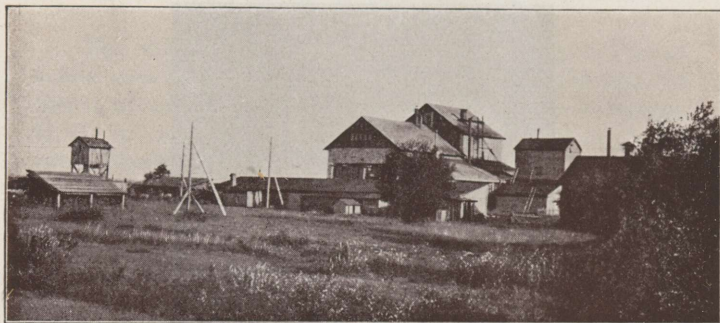
Up to 200° C.	5,0 per cent.
200 to 250° C.	39,0 per cent.
250 to 300° C.	36,0 per cent.
300 to 350° C.	15,0 per cent.

Lubricating Oils. — From the heavy oil cheap lubricating oils have been produced, the yield being 20–30 per cent., and the analysis of a sample supplied by the producers giving the following: —

Sp. gr.	1,011.
Viscosity at 50° C.	8,3° E.
Flash point.	152° C.
Asphalt	Traces.

PÜSSI SHALE-OIL.

System of Retorting. Distillation tests on a large scale have been carried out in an air-tight fire-brick chamber, designed and patented by the



"Eesti Kiviõli" Company plant, back view.

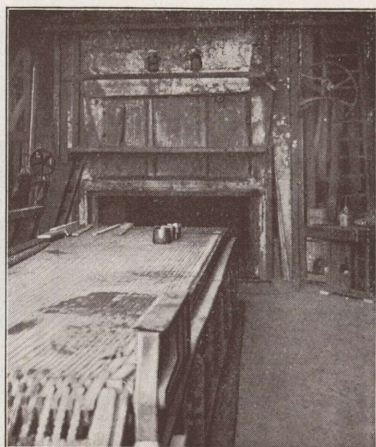
company ("Estonian Shale-Oil Co."). The dried and sieved shale lumps are heated on a sliding chain-grate by producer gases. The retort works continuously and puts through 25–35 tons of shale in 24 hours. The residue falls into the "coke-canal" and is discharged continuously.

PROPERTIES OF PÜSSI OIL.

Dependent on the temperature and manner of distillation, the specific gravity of the crude oil varies from 0,93 to 1,0, and the viscosity at 50° C from 2 to 10 E°. The yield of oil varies from 17,2 to 27 per cent on dry shale or 14,6 to 23 per cent on the freshly mined shale. The lower figures are obtained with the weathered shale only.

The distillation residue contains 12–20 per cent of carbon and is used for production of producer gas, alone or mixed with the shale. The yield of gases varies greatly, viz. from 25 to 120 cubic metres per ton.

A gasoline of good quality is obtained from the gases by washing.



Püssi retort, showing the sliding-grate.

The distillation test of a mixture of cracked light oil and gas-gasoline in the proportion actually obtained on distillation: —

I P. B.	. . .	40 ⁰ C	} sp. gr. 0,747
5 per cent	at	60 ⁰ "	
15 "	" "	74 ⁰ "	
25 "	" "	88 ⁰ "	
35 "	" "	100 ⁰ "	
45 "	" "	112 ⁰ "	
55 "	" "	123 ⁰ "	
65 "	" "	132 ⁰ "	
75 "	" "	142 ⁰ "	
85 "	" "	154 ⁰ "	
95 "	" "	180 ⁰ "	

The shale benzine has a sweet "Doctor-test" and shows a "negative" corrosion-test.

The total yield of crude benzine (gasoline) as obtained by washing of the gases, direct distillation and cracking amounts to about 10 per cent of the weight of dry oil shale.

The Püssi crude oil and some of its distillates were cracked and analysed in the laboratories of the Universal Oil Products Company, Chicago, U. S. A. By the courtesy of the director of the Estonian Shale Oil Company the author is able to quote some of the results obtained on cracking of the Püssi oil: —

CRACKING OF SHALE OILS.

1. The mixtures of shale-oil have been cracked on a no-residium basis with one throughput of the oil, producing a maximum gasoline yield of 38,7% (Mixture № 7-E. S. at 100 lbs. pressure). When the gas oil (P. D. Bottoms) is considered as recycling stock, an ultimate gasoline yield in excess of 45% and approximating 50% is obtainable.

2. Mixture № 7-E. S., which contains the heavy oil, medium oil, and undistilled light oil, makes the best charging stock when the gasoline yields are considered, for a maximum yield of over 53% was obtained.

3. None of the three mixtures lend themselves well to cracking on a fuel-oil-residue basis, and this mode of procedure is not recommended.

4. The yields of gasoline compared very favourably with the 50% yields obtained from shale oils from the United States, Australia, and France.

5. The rate of coke formation from any of these mixtures was approximately 70–75 tons per day based on a 1000 barrel throughput, which will necessitate a 40 ft. reaction chamber and a running cycle of one per day. The coke produced, however, will be of high quality, suitable for fuel or metallurgical purposes.

6. The rate of incondensable gas formation is rather high, but the calorific value of 1300 B. T. Us. per cu. ft. makes it a good fuel for the plant.

7. The raw cracked distillates produced from all three mixtures were treated to yield a water white, doctor sweet, negative corrosive and stable gasoline by the split plumbite procedure, using only 8 to 12 pounds of 66° Be. sulphuric acid.

8. These treated gasolines possess anti-knock properties very superior to any marketable gasoline we have encountered, and compare favourably with benzol blended motor fuels. That from Mixture № 7-E S. possesses automotive properties which will enable it to command a high premium over ordinary motor fuels when used in high compression engines. It may even be classed with the best grade of ethyl gasoline which is being sold on the markets in this country at the present time, and is applicable to any of the high compression engines now on the market."

"The results of analyses of three gasolines are shown in the following tabulation :

SOURCE	Unsaturated Hydrocarbons	Aromatic Hydrocarbons	Naphthene Hydrocarbons	Paraffin Hydrocarbons	Aromatic Hydrocarbon Equivalent	Kicardo's Compression Ratio
	0/0	0/0	0/0	0/0	0/0	0/0
Mixture 6ES						
Run 179	22,0	35,1	10,6	32,3	44,2	6,18
Plant 11						
Mixture 7ES						
Run 177	30,5	44,2	8,6	16,7	52,5	6,43
Plant 11						
Mixture 9ES						
Run 31	21,3	37,4	11,3	30,0	44,5	6,19
Plant 19						

"In carrying out the above analysis, a definite charge of the motor fuel is distilled to an end point of — say, 210°C. (410°F). The 210°C. fraction is treated with 80% sulphuric acid, and the percent decrease of volume owing to reaction solution calculated on the basis of the 210°C. fraction. The acid treated oil is washed with water, neutralized with a 10% solution of sodium hydroxide and then redistilled in the same apparatus until the vapour temperature in the Hempel column reaches 210°C. The volume of the residue of the second fraction to 210°C. is calculated as a percentage of the first 210°C. fraction and is the percentage of the unsaturated hydrocarbons which have been polymerized during the acid treatment. This, added to the reaction solution percentage, gives the total percentage of unsaturated hydrocarbons.

"The aromatic hydrocarbons are determined upon the distillate by the use of a nitrating mixture consisting of nitric acid 25%, sulphuric acid 58%, and water 17%, all by weight. The aromatic hydrocarbons are equivalent to 86% of the nitro lyer in volume, and this is calculated back to the original 210°C. fraction.

"The naphthene hydrocarbons are determined by the aniline method of Tizard and Marshall based on the lowering of the temperature of complete miscibility of aniline and the paraffins by naphthenes.

"Paraffin hydrocarbons are taken by difference.

"The unsaturated hydrocarbons and naphthene hydrocarbons are converted into aromatic automotive equivalents by means of Ricardo's data for toluene; 5% of unsaturated hydrocarbons being equivalent to 1% of aromatics and 4% of naphthenes being equivalent to 1% aromatics. The aromatic hydrocarbons are taken direct in evaluating the aromatic hydrocarbon equivalent. Ricardo's compression ratio is then calculated from the aromatic hydrocarbon equivalent.

“As regards anti-knock properties, these mixtures are the source of a very excellent grade of premium motor fuel. They are exceedingly high in aromatic hydrocarbon content, which makes them excellent anti-knock fuels. The motor fuels from No. 6—E.S. and No. 9—E.S. are the automotive equivalents of our best grade of Mid-Continent straight-run gasoline, to which 36% benzol has been added, while that from No. 7—E.S. is equivalent to the same gasoline in admixture with 47% benzol. For high compression motor purposes these motor fuels should command a premium over the ordinary gasolines on the market.

“They possess the highest anti-knock properties of motor fuels obtained from shale oil in the United States, Australia, or France which have been studied in our laboratories. For comparative purposes the following tabulation has been included to show how the motor fuel produced from these mixtures compares with that produced by cracking shale oils from various sources under the same operating conditions :

Shale-Oil from	Unsaturated Hydrocar- bons	Aromatic Hydrocar- bons	Naphthene Hydrocar- bons	Paraffin Hydrocar- bons	Aromatic Hydrocar- bon Equi- valent	Ricardo's Comp. Ratio
	0/0	0/0	0/0	0/0	0/0	0/0
U. S. Shale (Green River)	20,1	24,9	6,2	49,8	30,4	5,78
Australian Shale (New South Wales)	15,7	26,0	6,0	52,3	30,6	5,80
French Shale (Autun)	16,1	28,8	6,1	49,0	33,5	5,88
Estonian Shale (Estonian 7—ES)	30,5	44,2	8,6	16,7	52,5	6,43
(Estonian 9—ES)	21,3	37,4	11,3	30,0	44,5	6,19

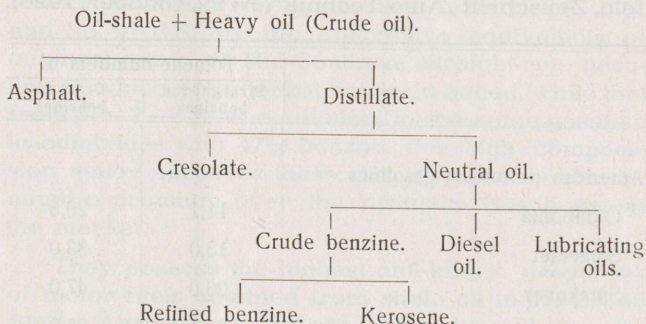
The following table, showing the comparison of toluene values of benzines, is taken from a recent paper on "Anti-knock benzines" by Prof. Dr. E. H. Riesenfeld („Klopffeste Benzine" von Prof. Dr. Riesenfeld, Zeitschrift „Auto-Technik", № 22, Oktober, 1926).

	Toluene-numbers of	
	Straight-run benzines	Cracked benzines
American petroleum-gasolines :		
Oklahoma	11,1	26,5
Somerset	13,0	33,0
Smakover	26,0	47,0
Panhandle	23,1	25,2
German petroleum gasolines :		
Wietzer		35,4
Other localities		32,8
Oil-Shale gasolines :		
Green River, U. S. A.		30,4
New Sth. Wales, Australia		30,6
Autun, France		33,5
Tallinn, Estonia		52,5
Gasoline from Brown-coals	30,6	

The above-cited experimental data clearly show the high quality of Estonian Shale benzines. Perhaps they are too rich for direct combustion in engines and could be used as "remedies" for straight-run benzines to improve their qualities.

Anyhow the toluene value and Ricardo's compression ratio of cracked Estonian Shale gasoline are higher than the values of any "non-synthetic oil" known so far.

A process has been patented by Messrs. E. Tram-pedach and R. Mayer, Ltd., Tallinn, for the direct production of asphalt from the oil-shale. The scheme is given below: —



Phenol Content. — The phenol content of all Estonian shale oils ranges between 15 to 20 per cent., the phenols being present in the form of higher phenols (cresols, xylenols, etc.). They have proved suitable for the purposes of impregnating timber such as railway sleepers, etc.

The light oils have been thoroughly tested by H. v. Winkler and L. Rubenberg, who used a two-stroke two-cylinder motor of 3,5 h. p., the speed of which was recorded and maintained, as far as possible, at 1500 r. p. m. Ten c. c. of the oils were tested, with the following results: —

Oil.	Time lasted. secs.
Normal benzine . . .	20,27
Kukersite benzine . . .	15,97
Kukersite light oil . . .	17,02
Kukersite benzol . . .	20,59

In all cases the exhaust gases were colourless and odourless.

The shale asphalt has been tested at the Material Testing Station in Tallinn by O. Maddison and F. Dreyer, with satisfactory results.

THE FUTURE OF THE OIL-SHALE INDUSTRY IN ESTONIA.

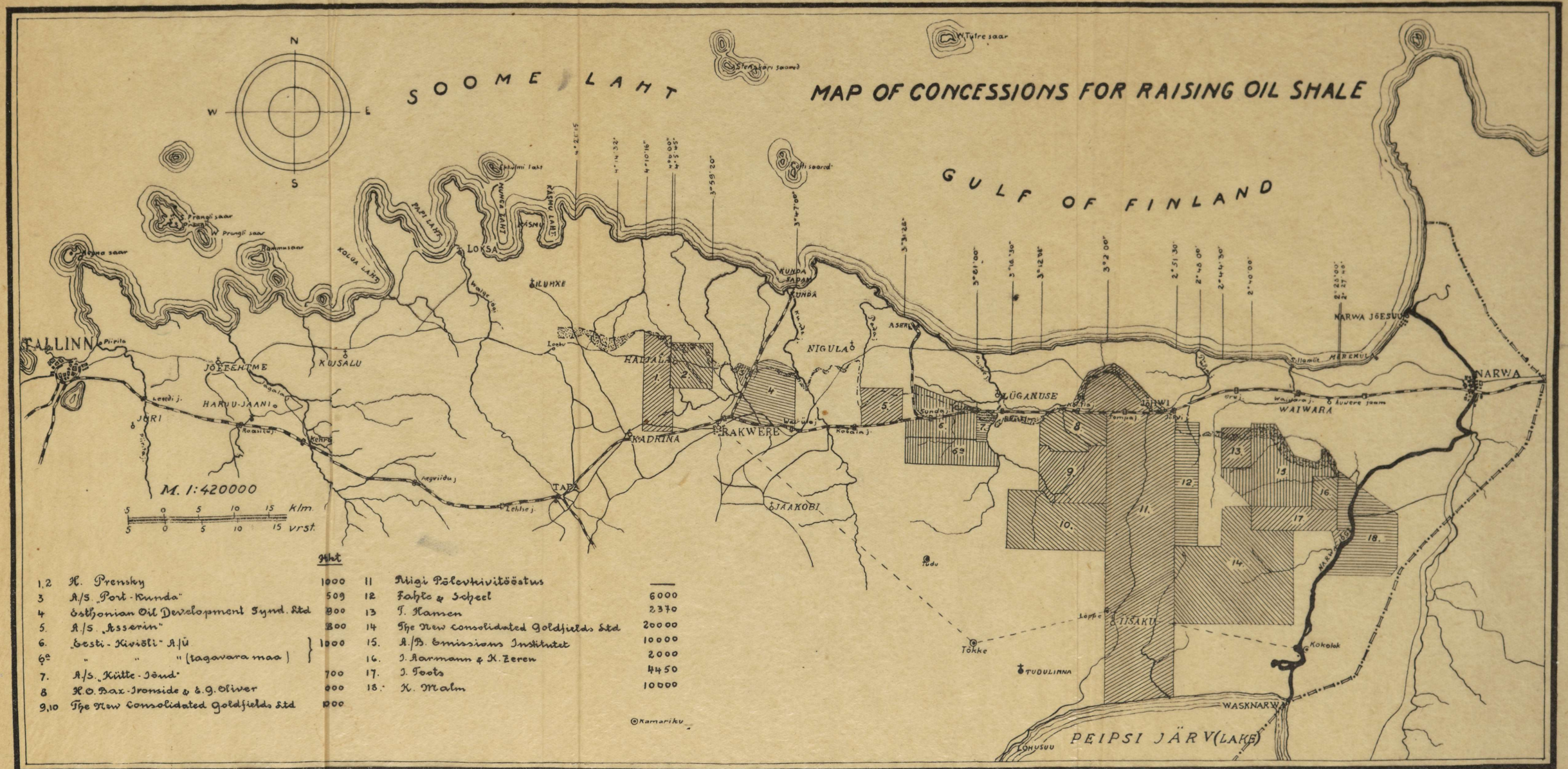
As already stated above, the mining of the shale is very easy, and the difficulty of the problem lies in the retorting of the shale. But this question is more of an economical than of a technical nature. The experimental trials on Estonian shale have proved that the oils obtained on distillation in externally heated retorts are usually lighter and easier to refine than the crudes obtained from producers at the same temperature. On the other hand a producer-retort requires fewer mechanical devices and probably lasts longer. To produce the highest marketable products, the crude oil or the distillates obtained on retorting by any present system should be cracked. As each crude requires its special method of treatment, the most economic method of refining for Estonian crude should also be worked out. The problem is already partly solved.

In the nearest future the Estonian shale-oil and its products may probably be able to compete on the European market with the products of natural well-petroleum.

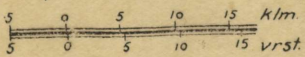


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9400		9400	
1,2	H. Prenskey	11	Siigi Põlevkivitööstus
3	A/S "Port-Kunda"	12	Fahle & Scheel
4	Estonian Oil Development Synd. Ltd	13	T. Klamen
5	A/S "Asserin"	14	The New Consolidated Goldfields Ltd
6	"Besti-Kiviõli" A.Ü	15	A. B. Emissions Institutet
6 ^a	" " (tagavara maa)	16	J. Rammann & K. Eeren
7	A/S "Kütte-Jõud"	17	J. Fests
8	H.O. Dax-Ironside & G. Oliver	18	K. Malm
9,10	The New Consolidated Goldfields Ltd		

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Area for possible open quarries Limits (southern) of the investigated area. © borehole.

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