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Estland

Progress in the Treatment of Estonian Oil Shale

Mathematisch-Naturwissenschaftliche Fakultät der Universität Tartu

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Occurrence and Properties of Estonian Oil Shale

Occurrence.

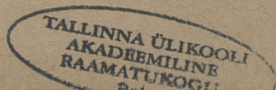
The oil shale of Estonia occurs in the Ordovician System, and covers an area of approximately 2,400 square kilometres, extending from Kadrina, on the west, to beyond Narwa on the east, the Reval — Narwa railway traversing the area.

The geology of the area has been dealt with in some detail by several geologists, chiefly, Bekker, Lamansky, Kutorga, Bonnema, Krutikov, and others, the firstmentioned having published a notable Memoir¹ on these deposits. Where the oil shale, known as Kukersite, is typically developed, it consists of seven beds, or seams, referred to locally as the A, B, C, D, E, F, and G seams, respectively, the lowermost being the A seam. The seams are inter-stratified with limestone bands.



Fig. 1. Open-cast Face at Kohtla Järve.

¹ "The Kukers Stage of the Ordovician Rocks of N. E. Estonia", by *Hendrik Bekker*.



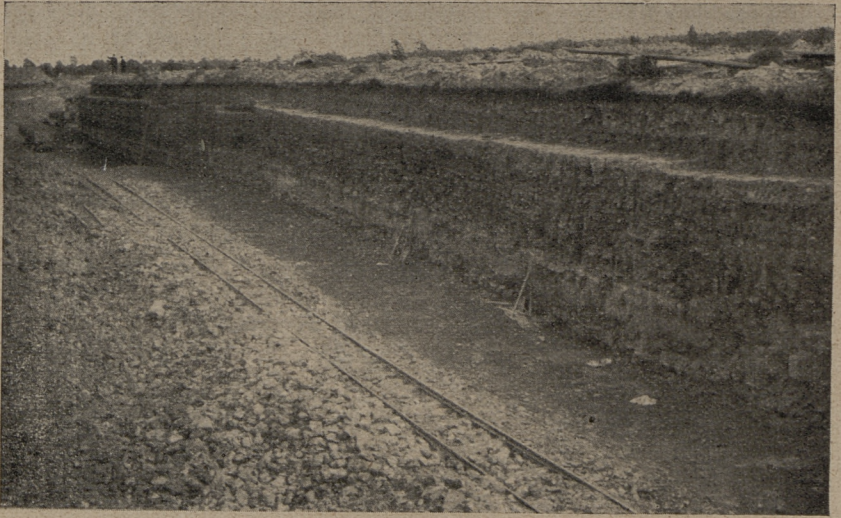


Fig. 2. Opencast Face at Eesti Kivioli.

A typical section, as developed in the Kohtla area, is shown in Fig. 1, while a number of exposures are illustrated in Figs. 2 to 5, showing the opencast workings at Eesti Kivioli. It will be observed that the deposit has a gentle dip of only about $\frac{1}{4}$ degree, and near the outcrop is overlain by a few feet of overburden, consisting of a foot or so of peat, and about two feet of glacial soil.

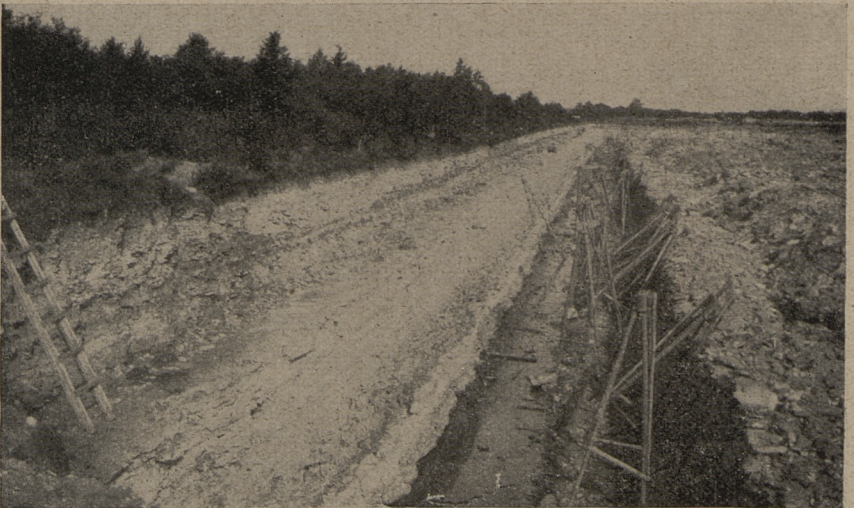


Fig. 3. Opencast at Eesti Kivioli.

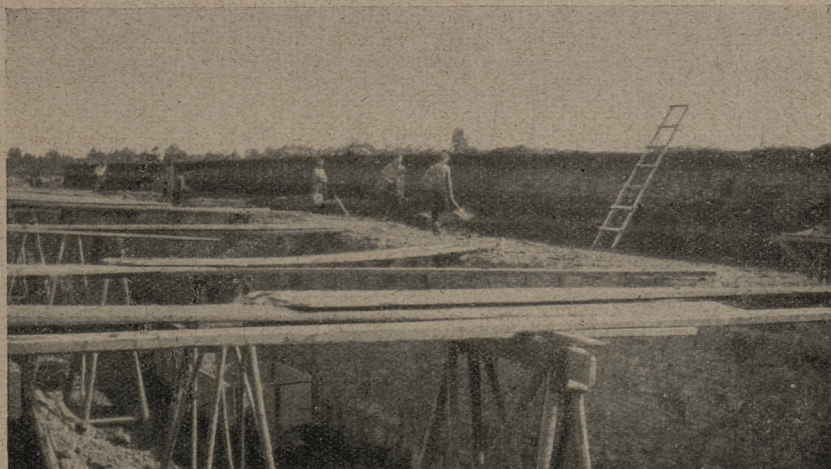


Fig. 4. Removing Overburden and Upper Seam of Shale at Eesti Kivioli.

The oil shale varies in colour from light brown to dark brown, is rather soft and easily mined. Its specific gravity varies from 1.2 to 1.8, but is generally about 1.5. It is easily kindled by means of a match, and burns with a smoky flame.

Some of the beds are highly fossiliferous; in Fig. 6 trilobites and bryozoa can be identified.



Fig. 5. Advancing Track at Face Eesti Kivioli.



Fig. 6. Fossiliferous Oil Shale.

Composition

An average proximate analysis of the oil shale is given in Table 1.

Table 1. Proximate Analyses (percentages)

Condition of Shale	Water	Ash	CO ₂	Ash and CO ₂	Organic matter	Calorific value cal/kg
Freshly mined .	18.2	30.0	7.9	37.9	43.4	3000 to 3500
Air dried	1.5	36.1	9.5	45.6	52.9	4200 to 4500

The composition of the organic matter varies within the following limits, in percentages:—

C 76.5—76.7; H 9.1—9.2; N 0.2—0.5; S 1.6—2.2, and O (by diff.) 11.2—12.2. This nearly corresponds to the empirical formula $(C_8H_{11}O)_n$.

The chemical composition of the organic matter of the various seams of Kukersite does not vary much, as will be seen from Table 2.

Table 2. Chemical Composition of Shale Seams (percentages)

Seams	A	B	C	D	E	F
Organic matter in air-dried 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 (by diff.) ² . . .	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

² Includes also the sulphur.

Table 3. Volatile Matter Tests on Shale (percentages)

Seams	A	B	C	D	E	F
Volatile matter at 900° C	56.7	60.1	54.2	42.7	56.6	50.4
"Fixed Carbon"	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

Oil Yields

The yield of liquid products varies with the nature of the fuel, the method of distillation, and the temperature of treatment. Table 4 shows the variation in the yield at different temperatures, as determined in a silica tube.

Table 4. Distillation of Shale at Various Temperatures

Temp. °C	Oil Yield per cent	Oil Yield gal/t	Gas Yield c. ft./t at 0° and 760 mm	Calorific value of oil B.T.U.
410	27.1	63.3	1900	—
500	29.7	72.9	2250	17,028
600	30.8	74.8	3000	17,428
700	27.5	65.0	4500	—
900	21.7	49.7	7200	—

Although higher percentage yields of oil have been obtained in the laboratory with other samples of shale, the above can be taken as representative.

Exploitation of Shale

Although Kukersite was first discovered about 140 years ago, it was not until 1916 that, owing to a scarcity of fuel in the Leningrad (then Petrograd) district, it was decided to investigate the possibility of exploiting the shale. After preliminary surveys and tests mining operations were commenced at Kohtla.

Work was interrupted during the Bolshevik Revolution and the subsequent German occupation, but in May 1919 the Estonian Government took over the Kohtla works, which have since been in continuous operation. Later developments were the opening of the Vanamoisa mine by the Estonian Oil Shale Development Syndicate, Ltd., the exploitation of the shale at Pussi by the Eesti Kivioli Company, and *Estländska Oljeskifferkonsortiet* at *Sillamägi*. Permits and concessions have since been granted to several other concerns.

Use of Raw Shale

The shale is used somewhat extensively in the raw state, not only in railway locomotives, but also in stationary boiler plants at various factories, chiefly in Tallinn (Reval). In one or two plants the shale is burnt with almost complete absence of smoke, but these cases are exceptions; the combustion of the raw fuel being generally accompanied by the production of volumes of dense black smoke. It goes without



saying that the use of raw shale in this manner should be discontinued at the earliest possible moment. This will come about when sufficient liquid fuel is produced locally on a sufficiently large commercial scale to meet the demands of Estonian industries.

Commercial and Semi-Commercial Distillation Plants

At present the largest shale distillation plant in operation in Estonia is that at the State Works at Kohtla. The plant consists of 6 retorts of the producer type, designed by J. Pintsch & Co., Berlin. At the Pussi mine a retort of the tunnel-kiln type is being employed on a fairly large experimental scale. The "Fusion" type of rotary retort was formerly employed at Vanamoisa, but is not now in operation. Another tunnel-oven type of retort has been experimented with at Sillamägi by a Swedish group. A modified form of the Scotch retort was operated at Tallinn from 1919 to 1921, but proved unsuccessful.

The yield of oil was 15% with this unit. Plans are now being prepared for the erection of a battery of Davidson Rotary retorts to be operated on the concessions held by the New Consolidated Goldfields, Ltd., London.

The Kohtla Plant

A photograph of the Kohtla plant is shown on Fig. 7, the retort house being on the left, and the boiler house in the middle distance. The Pintsch retort consists of a vertical cast-iron shaft lined with firebrick. Its external diameter is 2 m, and its height 5 m. The raw shale is graded to pass through a 4-inch mesh and retained on a 2-inch mesh, this grade having been found the most suitable size for treatment. In all producer-type retorts employed in the treatment of shale and coal careful grading is essential, hence, the practice at Kohtla is no excep-

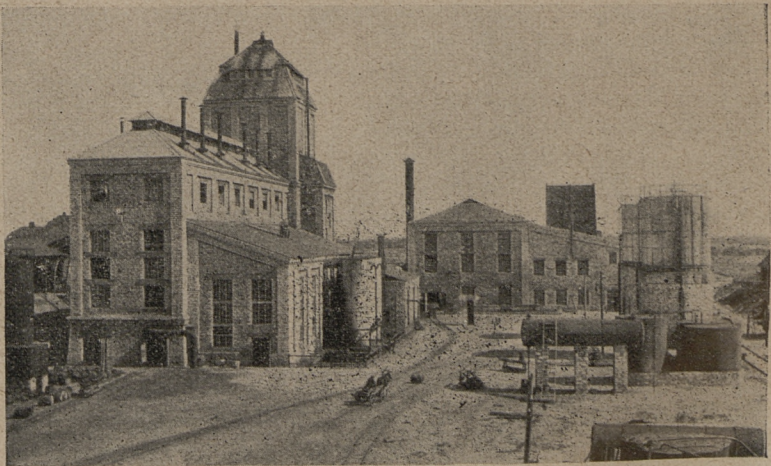


Fig. 7. Photograph of State Plant at Kohtla Järve.

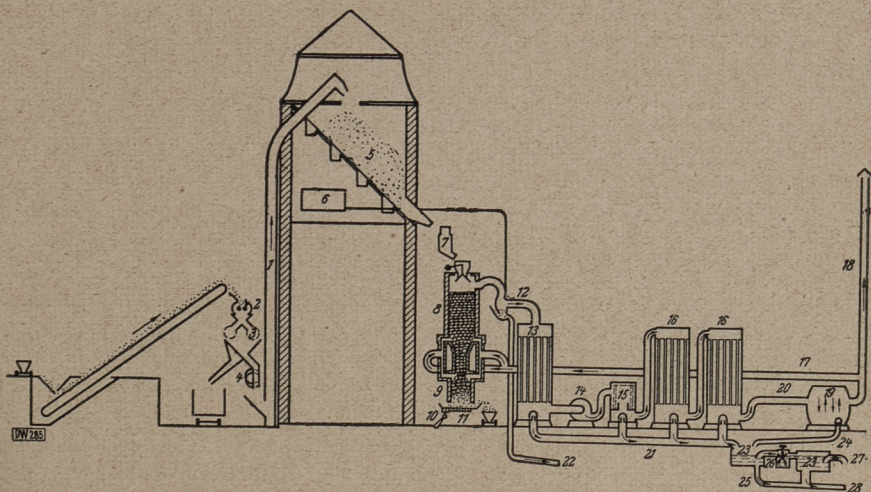


Fig. 8.

tion to the rule. A flow sheet of the retorting plant is shown in Fig. 8. The shale is charged through a bell hopper in the usual manner, and then descends by gravity through the producer.

The gases generated in the combustion chamber at the base of the producer enter annular flues at the top of the combustion zone, and pass into the charge undergoing distillation higher up in the retort. These gases, supplemented by cold permanent gas returned from the recovery plant, effect the distillation of the shale. The cold gas reduces the temperature of the combustion products to distillation temperature, namely, about 550° C. The volatile products leave at the top of the retorts, and pass successively through the cooler, exhauster, tar extractor, and condensers to the separating tanks.

Part of the permanent gas is used for firing Babcock and Wilcox boilers, in conjunction with raw shale. The chimney in the centre of Fig. 7 shows that combustion is effected smokelessly, the photograph having been taken while the boilers were steaming.

Properties of Crude Oil from Pintsch Retorts

An average sample of the crude shale-oil has the following properties.

Sp. gravity at 15°	About 1.0
Reaction	Neutral
Moisture	About 1%
Flash point	83° C
Insoluble in benzene	0.3%
Viscosity at 50°	5° E
Sulphur	About 1%
Calorific value	9600 cal/kg

The permanent gases still contain about 20 g of light benzine per m³, the calorific value of the gases (determined in Junkers calorimeter) being about 1240 cal/cm³.

The Kohtla crude shale-oil contains but very little benzine (motor spirit); the main marketable products being:—

1. “*Motornaphtha*”, or *Diesel oil*, which is obtained from the crude oil by distillation. The distillate is neutralised and the lower boiling constituents removed by steam. The properties of this product are:

Flash point (Pensky-Martens) ..	over 60° C
Viscosity at 50° C	1.0 to 1.3 E
Specific gravity at 15°	0.93 to 0.95
Sulphur	About 0.9%
Coke (crucible-test)	3 to 4%
Calorific value	9,700 to 10,000 cal/kg

Distillation Test:—

Up to 200° C	5 to 8%
200 „ 250° C	15 „ 20%
250 „ 300° C	20 „ 30%
300 „ 360° C	30 „ 40%

The oil was tested at Södertälje (Sweden) in a 15 HP-motor (Type D. PS.) giving 500 revolutions p/min. The results were satisfactory.

2. *Lubricating oil* fractions have not been produced so far on a large scale, although good lubricating oils have been obtained on a laboratory scale.

3. *Pitch* is the distillation residue of the crude oil, and has a melting point — of 70 to 90° C. The properties of this product are dependent on the method of distillation.

As the crude-oil is subjected at present to “air-blowing” to produce three grades of heavy oils and five grades of *bitumen*, or blown asphalts, the pitch is no longer a marketable product.

4. *Estobitumen* A, B, C, D, and E. The blowing of the oil is carried out in stills of 8 to 10 t capacity. The hot bitumen is passed into special tanks from which (before solidifying) it is poured into barrels.

On blowing, about 10% of the crude oil is obtained as distillate, and about 80% forms bitumen.

Properties of Estobitumens, A, B, C, D and E.

Melting points (Kraemer-Sarnow)	18 to 65° C
Specific gravities	1.07 to 1.09
Solubility in CS ₂	98.5 to 99.5%
„ „ CCl ₄	98 to 99%
Sulphur	0.9%
Mineral ash	0.3 to 0.5%
Flash points	180 to 220° C
Coke	25 to 35%

The estobitumens A, B, C are used in road making as surfacing tars and asphalt mastic. The products which have higher melting points

are used in the manufacture of roofing paper. Some other products are also manufactured from bitumen.

5. *Phenolate* is obtained from crude oil by treating the oil with 5 to 10% caustic soda solution. The phenolate is used as a disinfectant, and for the preservation of timber and railway sleepers.

The Eesti Patendi A/S System

This system is employed by the Eesti Kivioli Company at Pussi, the unit having been designed to treat 50 t of raw shale per 24h, but it has proved capable of treating 70 t per day. The tunnel oven is rectangular in area, so dimensioned as to allow the free passage through it of a tub, or tram, holding 680 kg of shale. Its length is 25 m, and it accommodates 20 tubs at one time, the tubs being distributed as follows: 5 in preheating chamber, 12 in carbonising section, 3 in the cooling chamber, where the carbonised shale is quenched with water. Each compartment of the tunnel is sealed by means of hydraulically-operated doors, controlled electrically from a control platform, shown in Fig. 9, which also shows the circulating fans, which will be referred to presently.

The steel tubs are of rectangular form, as shown in Figs. 10 and 11, and provided at the bottom with a short flanged pipe. The shale rests on a false bottom, or grid, perforated to allow the passage of volatile matter. Shale from $1\frac{1}{2}$ " to 4" is filled into the tub, on the top of which a wire net is placed.

During distillation the volatile matters expelled from the shale are circulated over and over again through the charge in each tub. To achieve this object there are provided at regular intervals along the

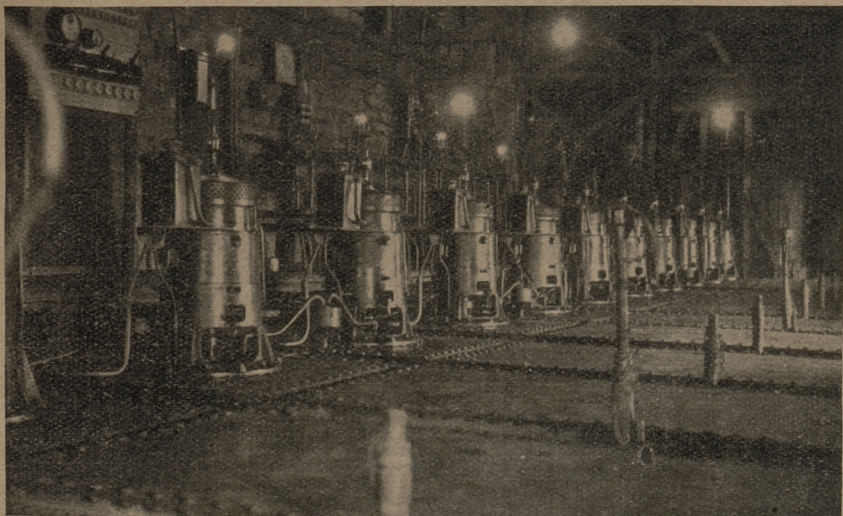


Fig. 9. Circulating Fans and Control Platform. Eesti Patendi Retort.

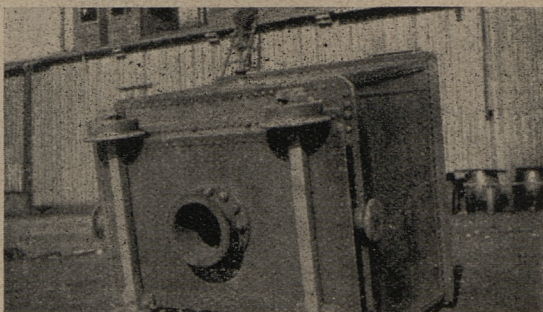


Fig. 10. Underside of Steel Tub.

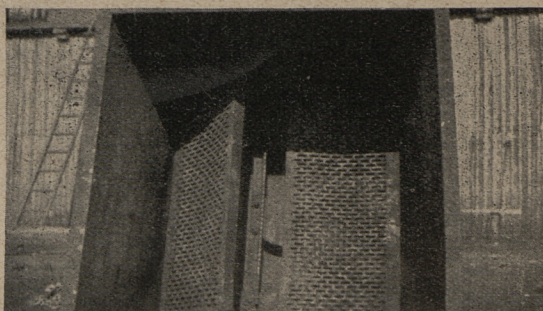


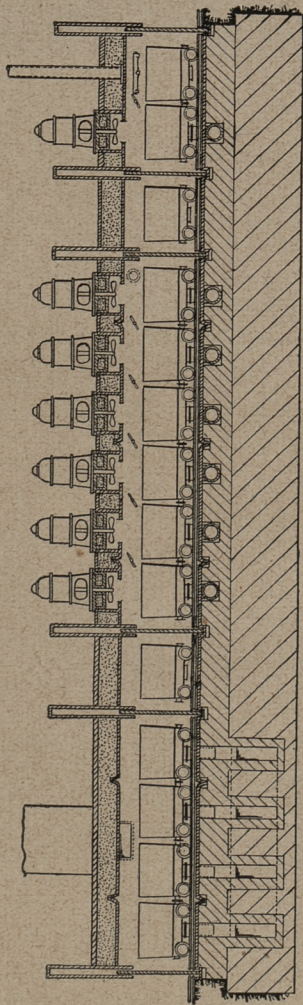
Fig. 11. Interior of Tub, Showing Grid, or False Bottom.

tunnel flanged pipes of the same diameter as those on the tubs. At each position in the retort the tub rests immediately above the circulation pipe and below an electrically-driven circulating fan. The volatiles are drawn by means of the fan through it, and forced through a horizontal duct leading to a distribution channel above vertical reheater tubes, which are located in a combustion chamber running alongside the retort. See Fig. 12, *a*, *b* and *c*. Passing through the reheater tubes the gases then enter a horizontal duct at the bottom, leading to the flanged pipe within the retort and below the tub.

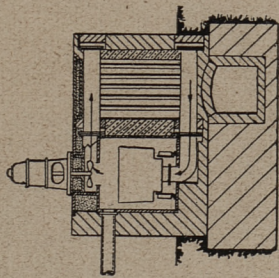
After a period of 12 to 14 min each tub is moved forward one stage, a spent charge being withdrawn from the cooling chamber, and a fresh charge put into the retort.

The unit was subjected to a 14-day test by the authors in the summer of 1929, when just over 70 t of shale per day was successfully treated. At that time the furnace was fired partly with gas and partly with crude oil. A view of the retort house is shown in Fig. 13.

(b) SECTION A-B



(c) SECTION J-K



(a) SECTIONAL PLAN

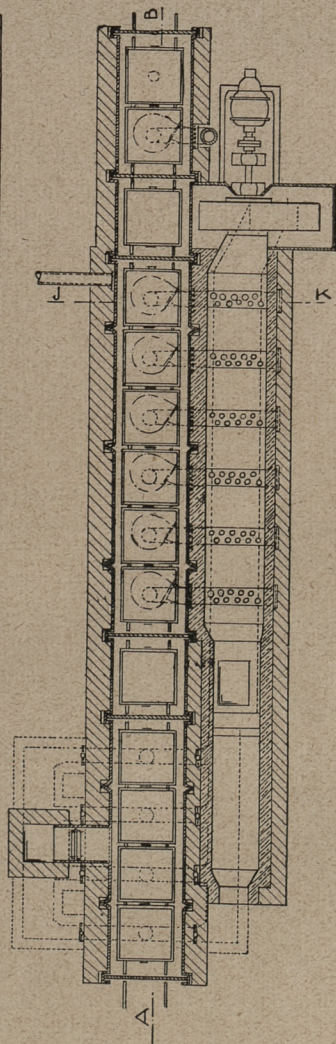


Fig. 12, a, b and c.



Fig. 13. View of Retört-House Eesti Patendi Plant.

Condensation and Separation of Products.

From the retort the vapours and fixed gases pass into the vapour main, which conducts the vapours to the air-sealed vertical condensers. The atmospheric condensers have sufficient cooling surface to separate from the vapours and fixed gases the heavy and medium oils³. The vapours of light oils (including benzine) and gases pass further to the water-cooled condensers, where some "medium oil" and light oils are condensed. The gases passing from the condensers still contain small quantities of light hydro-carbons (gas-benzine) not removed by the condensers. In order to separate these, the gases are passed into pipes, which are surrounded by a freezing mixture ("refrigerator").

Properties of Oils from Eesti Patendi Retort

1. *Mixture of Heavy and Medium Oils.*

Specific gravity at 15° C	1.01
Viscosity at 50°	7.0° to 7.8° E
Insoluble in normal benzine	About 10%
Mineral substances	0.01 to 0.02%
Moisture	6.4%

Engler Distillation

Up to 200°	1.0 to 2.0%
200 „ 300°	16 to 17%
300 „ 360°	44 to 52%

2. *Light Oil:—*

Specific gravity at 15° C	0.830
Sulphur	0.90%
Phenols	About 6.5%
Unsaturated compounds	About 30%

³ This mixture of heavy and medium oils forms about 77% of the bulk of the total oil.

Engler Distillation

IBP	64° C	
Up to 100°	2.0 to	3.0%
100 ,, 125°	8.0 ,,	10.0%
125 ,, 150°	13.0 ,,	15.0%
150 ,, 185°	17.0 ,,	23.0%
185 ,, 200°	7.6 ,,	8.8%
200 ,, 220°	10.4 ,,	12.0%
Residue (above 220°)	About	33%

3. Crude benzine (gasoline), gas benzine not included.

Specific gravity at 15°	0.767 to	0.770
Sulphur	0.72 ,,	0.74%
Unsaturated compounds	About	28%

Engler Distillation

IBP	48° C	
Up to 100°	21.5 to	25.3%
100 ,, 125°	21.7 ,,	23.8%
125 ,, 150°	18.8 ,,	19.4%
150 ,, 185°	15.0 ,,	16.0%
185 ,, 200°	4.3 ,,	4.6%
200 ,, 220°	5.4 ,,	5.6%

The Davidson Rotary Retort

Principles of Design. The aims of T. M. Davidson, the designer of this retort, have been to ensure continuous operation with low power consumption, ease of operation with minimum labour, the prevention of scale formation within the retort, the avoidance of cracking of the volatile products, and the utilisation of the sensible heat and the combustible matter in the shale residue after distillation for carrying out the distillation process, the heat derived from the residue being supplemented by stripped gas returned from the recovery plant.

A retort embodying these principles was erected at Colchester, England, in co-operation with the New Consolidated Goldfields Ltd., London. Other processes had previously been closely investigated by this Company, with a view to the treatment of Estonian shale on a commercial scale.

Description. The Davidson retort Fig. 14, consists of a rotary steel tube *A*, 50 feet long, and 4 feet diameter, rotated at a speed of 1 revolution in $1\frac{1}{2}$ min. The retort is set with a fall of 0.8%. The tube is externally heated, the temperatures in the heating flues being maintained at about 590° C. The interior of the retort is fitted with scrapers, which prevent the accumulation of scale, and keep the inner side of the shell clear. The shale is fed from the bunker *B* through the inlet *C*, the feed being continuous and automatic. In its passage through the tube the temperature of the shale is progressively raised until a final temperature of about 480° C is reached. As the retort rotates under

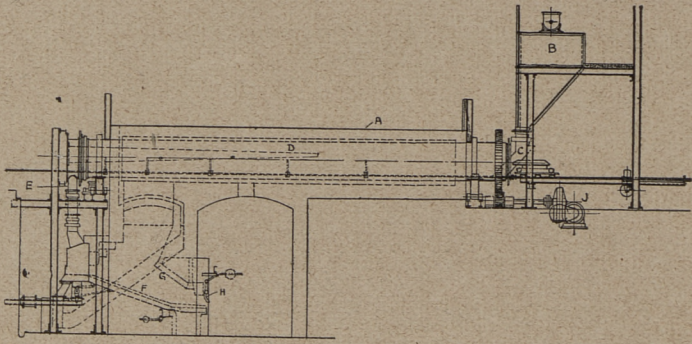


Fig. 14. The Davidson Rotary Retort.

load, the shale is turned over 200 or 300 times, and travels a distance of a few thousand feet over the retort shell.

Meanwhile, the volatile products are withdrawn through an offtake tube *D*, and pass into a dust catcher situated near the discharge end of the retort, see Fig. 15. The hot residue is discharged into a duct *E*, in which are fitted two hydraulically-operated knife valves to regulate the feed into the heating furnace *F*, on the step grate of which the residue is burnt. The grate is mechanically operated. To supplement the heat obtained from the residue, stripped gas is introduced at the back of the furnace arch at *G*, secondary air being admitted through the balanced curtain *H*, below which the ashes are discharged. The motor for driving the retort and the scraper gear is shown at *J*.

Operating Results. The retort erected at Colchester, working on Estonian oil shale, was operated under test conditions for a period of

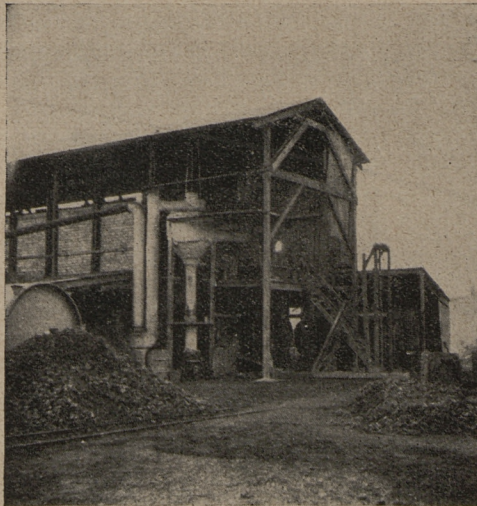


Fig. 15. View of Discharge End of Davidson Retort.

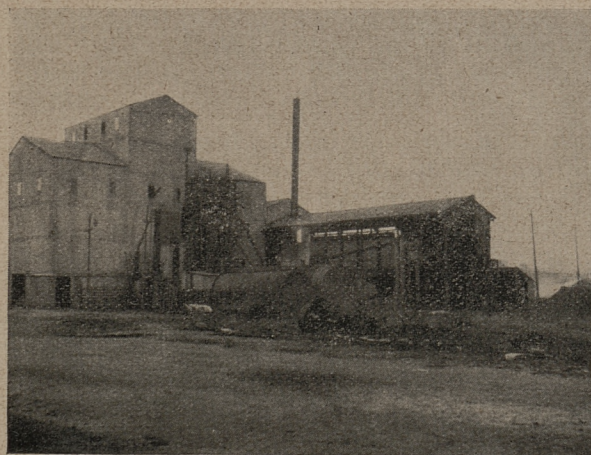


Fig. 16. View of Davidson Demonstration Rotary Retorting Plant.

14 days under the supervision of one of the authors. This test run proved quite successful, very satisfactory yields of oil being obtained, the retort working quite smoothly, with a low power consumption, and a minimum of manual labour. The heats were easily maintained with the Davidson furnace in operation, without the aid of external fuel, and combustion was complete and effected smokelessly, as evidenced by the chimney in the photographs, Figs. 15 and 16, which were taken while the retort was distilling shale.

Preparations are now being made for the erection in Estonia of a number of these retorts forming a 250-ton unit. This development is being undertaken by The New Consolidated Gold Fields Ltd. The new retorts will be 75 feet in length, and will incorporate a preheating section at the feed end.

Shale Oil from Davidson Retort. The following is an Engler Distillation of an average sample of oil obtained during the test run on dry shale in the Davidson Retort, September, 1929.

Moisture	0.8%
Spec. grav.	0.9398 @ 18° C
Phenols	30.66% by vol.
Unsaturated hydrocarbons (with H ₂ SO ₄ , density 1.84)	68.93% ,, ,,
Insoluble in normal benzine about ...	5%
Flash point	about 14° C

<i>Engler Distillation</i>		Spec. Grav. @ 18° C
Up to 150° C	8.07 grams	0.7483
150 ,, 175	4.96 ,,	0.7927
175 ,, 200	3.82 ,,	0.8179
200 ,, 225	4.05 ,,	0.8454

			Spec. Grav. @ 18° C		
225	„	250° C	5.24 grams	0.8739	
250	„	275	6.27	„	0.9056
275	„	300	7.66	„	0.9371

Another sample of the crude oil was steam distilled up to 200° C. The steam carried over 30.1% by volume. The crude shale oil is thus seen to be of very high standard of quality.

The Gröndal Retort

This retort, which is operated at Sillamägi by the Estlandska Oljeskifferkonsortiet, is also of the tunnel-kiln type, and the principles of operation are somewhat similar to those employed by the Eesti Patendi Company. The tubs are shallower, but of larger sectional area than in the case of the latter plant. The unit at Sillamägi has a capacity of about 250 t of shale per day.

The Crude Shale Oil as a Substitute for Well Petroleum and as a Source of Raw Materials for Chemical Industry

The crude shale oil must not be regarded merely as a substitute for well-oil, but also as a source of raw materials for manufacturing chemical products.

As regards the oils produced from the crude shale oil, i.e., motor spirit or gasoline, kerosene (lamp oil,) lubricating oils, etc., they will compete with the products from natural petroleum. The shale petrol or benzine used in Estonia is of high quality, it has a high compression ratio and good anti-knock properties. Two heavy fractions from the Davidson Retort were tested at the National Physical laboratory, Teddington and found to possess good lubricating properties.

The "heavy benzine" produced by distillation of light oil at *Eesti Patendi* Company's plant is sold to Czecho-Slovakia, where it is used as a good denaturant for alcohol.

The so-called phenolic bodies of Estonian shale oil are used at present for impregnating timber. The laboratory researches have proved that certain fractions of these phenols are suitable for manufacturing Bakelite products. Undoubtedly, there are other possibilities for the utilisation of some constituents of shale oil which do not occur in the well oil.

Zusammenfassung

In den letzten Jahren macht sich eine lebhafte technische Ausnutzung des Brennschiefers bemerkbar. Besonders zur Destillation des estnischen Brennschiefers, der sich durch ausgiebigen Ölgehalt auszeichnet, sind mehrere Retorten konstruiert; es gesellen sich zu der früheren von Pintsch konstruierten Generatorretorte, welche in Kohlla angewandt wird, noch einige neue Schwelanlagen, von denen folgende nennenswert wären:

1. Der Kanalofen mit Innenheizung der Estnischen Patent-Aktiengesellschaft (Kulschinskysche oder M.S.K.-Retorte). Diese horizontale Retorte, deren Durchmesser rektangulär ist, ist durch Schieber in drei Sektionen geteilt: in eine Trockenkammer, die 5 Wagonetten faßt, eine Destillationskammer für 12 Wagonetten und eine 3 Wagonetten fassende Kühlabteilung. Der Retorte parallel läuft der Heiz-

kanal, an dessen einem Ende sich die Feuerung befindet. Im Heizkanal sind die sog. „Überheizer“ untergebracht. Die Destillationsgase werden mit Hilfe besonderer Ventilatoren durch den „Überheizer“ geleitet und dann wiederum aufs neue durch die Brennschieferschicht usw., so daß somit die Destillations- und Öldämpfe einen langen Spiralweg zu durchlaufen haben, ehe sie in die Kühler gelangen können. Die Öldämpfe werden anfangs durch Luftkühler gekühlt, wo dann zuerst das Schwer- und zum Teil das Mittelöl sich kondensiert; späterhin in Wasserkühlern, wo sich von Stufe zu Stufe das Mittel- und Leichtöl mitsamt dem Benzin kondensiert. Das aus den Kühlern sich ausscheidende Gas enthält noch Gasbenzin.

2. Ungefähr nach demselben Prinzip arbeitet auch die Gröndal-Carlson-Zeidlersche Retorte des Estländischen Ölschieferkonsortiums. Die Schieber schließen hier nicht so dicht ab wie bei der Kulschinskyschen Retorte; die Wagonetten sind flach gebaut, von pfannenähnlicher Form, und die Überheizer sind unmittelbar unterhalb der Retorte untergebracht. Das Durchlaßvermögen dieser Retorte beträgt ca. 250 t/Tag.

3. Gegenwärtig beginnt man in Estland ein neues Destillationsunternehmen ins Leben zu rufen, wobei beabsichtigt ist, daselbst eine T. M. Davidsonsche horizontale, rotierende Retorte mit Außenheizung aufzustellen. Dieser Typus einer Retorte hat sich durch in England, in Colchester gemachte ausgiebige Versuche bewährt, wobei die Retorte 20 bis 25 t Ölschiefer, täglich durchließ. Das Anbacken des Brennschiefers an den Wänden der Retorte ist hier durch einen besonders konstruierten „Schaber“ („scraper“) verhindert.

Das spezifische Gewicht des aus der Davidsonschen Retorte erhaltenen Öles ist kleiner als dasjenige der Öle, die aus Retorten mit Innenheizung erhalten sind, während aber der Benzinprozent im Rohöl der Davidsonschen Retorte recht erheblich ist.

Es läßt sich das Brennschieferöl nicht als einfacher Ersatz für Erdöl betrachten, da sich in ihm viele Verbindungen finden, die dem Erdöl gänzlich fehlen und die sich als Rohprodukt für die chemische Industrie sehr gut eignen.

