URMISCC/4-12

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REPORT

- on -

The SAMPLE of OIL producing SHALE from TASMANIA.

The sample received consisted of a block of soft fissile tough slaty material. It contained a number of fossils, which I have been informed belong to the Permo-Carboniferous period. The shales are therefore of great age.

They are impregnated with minute specks and small patches of resinous-looking material, from which the oil is obtained by distillation. The shales are not oil bearing in themselves for no solvents for oil will extract any. The heat of distillation breaks up the resinous mineral into water, oil and gas.

Late investigations have shown that the resinous patches consist of the spores of extinct plants. These spores accumulated in the muds formed at the time, probably in swampy places and near the mouths of rivers. The muds themselves in course of ages became consolidated into a shaly rock entrapping the organic remains.

The organic oil-producing mineral has approximately the following composition -

Carbon	79.3
Hydrogen	10.4
Sulphur	5.3
Oxygen	5.0
	100.0

It has a specific gravity of 1.18 thus being slightly heavier than water.

It is not altered by the action of most chemicals, caustic alkalies have no effect on it, neither has hydrchloric acid: strong sulphuric acid converts it into gases and carbonizes or chars it; strong nitric acid converts it into carbonic and sulphuric acid.

It burns with a smoky flame and with a disagreeable odour.

Heating in a closed vessel without access of air breaks it up into water, sulphuretted hydrogen, carbon di-sulphide, permanent gases and oily products possessing a foetid odour.

The parcel sent over was crushed and small assay samples taken for proximate analysis, it yielded :-

Moisture at 212°F 1.44%

The steam-dried sample gave -

Volatile matter (including water, gases and oil) . . .	38.6%
Fixed Carbon	2.4%
Ash - slaty material	59.0%
	100.0%

The ash contained no sulphur; the sulphur must, therefore, have all passed over with the gases and oil. The percentage of sulphur present in the original shale was 3%. The amount which would pass over for every 100 lbs. of oil and gases would be 7 lbs.

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A larger parcel of several pounds was then distilled from a closed retort under conditions favourable for giving the maximum quantity of oil.

The products obtained were

(1) Residue which remained in the retort -

Slaty material	59.6
Fixed carbon	2.4

(2) Products which volatilized -

Crude oil	24.4
Gases	8.5
Water containing Ammonia . .	5.1
	100.0%

The gases evolved during distillation were exceedingly offensive, but could be made use of in practice for heating the retorts and thus destroyed.

The mixed oils on distillations were dark in colour and possessed an offensive odour. They were very fluid and had a high specific gravity, namely 0.92.

The amount of oil the sample gave was at the rate of 546.5 lbs. per ton of shale or 59.4 gallons per ton.

The water which distilled over would yield ammonium sulphate at the rate of 1.9 lbs. per ton. This is a very low yield, but probably under working with a large amount of material, the aid of superheated steam and a scrubble for the gases, larger amounts would be obtained. I further consider the single sample sent may not represent the true ammonia value of these shales.

The crude oil obtained by the first distillation could not be safely used on account of its low flash point, or,

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in other words, inflammable vapours would be given off at such a low temperature that its value as a fuel would be largely discounted.

The oil obtained was re-distilled so as to eliminate the volatile crude naptha oils.

On distilling the oil the naptha oils started to come off freely at 138°F and continued to come until the temperature reached 300°F, at the latter point the oil in the retort started to split up into gases. Distillation was, therefore, stopped at this point. The weight of the lighter oils so removed was 6.5% on the oil distilled. These had a specific gravity of 0.75. These ha d a very offensive odour, were very volatile and formed explosive mixtures with the air.

Treatment with caustic soda and sulphuric acid removed these objectionable odours, and on re-distillation an oil (naptha) having a not unpleasant aromatic smell was obtained. This crude naptha had a specific gravity of 0.75 and was clear, bright and transparent. It contained 2% of sulphur and unless this were removed it could not be used for internal combustion in motors or oil engines owing to the corrosive nature of the acid products formed.

The dark heavy oil remaining in the retort had a S.G. of 0.9[illegible] and on further distillation 0.970 but in spite of its weight for such an oil it was mobile and fluid, and easily converted into a spray suitable for burning in air. Its odour was objectionable but much less pronounced than the crude oils first

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obtained, or the crude naptha.

The amount of this crude oil freed from its naptha products per ton of shale would be 55 gallons. The sulphur present was 2.324 per cent.

The naptha and crude oil were both tested for their heating powers.

The calorific power of the naptha was 16,605 B.T.U.

: : : : : crude:oil : 21,915 B.T.U.

These values are higher than those of oils other than the Caucasian heavy crude oils which run 22,000 British Thermal units. The Scotch shales oils give an oil having 18,000 B.T.U.

Tests were also made on the crude oil with a view to refining it but, although sulphuric acid gave a comparatively large amount of acid tar or about 10%, caustic soda had no apparent action on the remaining oil. On removing the caustic soda and re-distilling, the oil remaining was dark green in colour -

Heating to 170° F gave . . . . . .5% of naptha

: : 220° F : . . . . . 4% gasoline oil

: : 230° F - the oil started to split up and leave a red deposit.

: : 300° F another 4% clear oil distilled over.

The residual oil was dark green in colour and without offensive odour, the naptha oils had an aromatic smell.

Since the oil was decomposing rapidly in the retort and very little clear oil was being formed, further heating would

have split the oil largely into coke and permanent gases. (9)

By distillation of the sale and then of the first crude oil a residual oil consisting of about 90% of the crude oil will be left, and about 8% of naptha-crude obtained as a distillat or assuming 60 gallons to the ton of crude oil 54 gallons of residual oil and 4.8 gallons of crude naptha.

The naptha is useful for solvents or it might be purified and fractionalized into gasoline and motor spirit or petrol. Most of the petrol power would escape with the gases unless the latter were scrubbed.

The residual oil would serve as fuel in such cases where the amount of sulphur present would not be injurious, it would not for instance be suitable for steel melting and copper refining.

Experiments have shown that it is eminently suitable for coating sulphides in the acid oil flotation processes. The sulphur could be removed and the oil rendered more valuable as a fuel by treatment with oxide of copper, the copper unites with the sulphur forming sulphide of copper, the copper unites with the sulphur forming sulphide of copper, the latter may be regenerated by roasting.

If the crude oil were fractionated and refined by means of caustic soda and sulphuric acid I consider that nearly one half of it would be destroyed: the use of sulphuric acid and soda would also add materially to the cost.

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SUMMARY of RESULTS.

Crude naptha from 1 ton of shale	4.8 gallons.
Crude black oil from 1 ton of shale	54.0 :
Shale residue left after distilling 1 ton	12 cwt.
Sulphur in crude shale	3%
Sulphur in shale residue, after retorting . .	None.
Sulphur in naptha crude	2%
Sulphur in crude oil after distilling off the naptha	2.324%
Calorific Power of Naptha	16,605 B.T.U.
Calorific Power of crude oil	21,915 B.T.U.
Ammonium sulphate which could be made from the liquor which condensed with the oil from a closed retort from 1 ton of shale	1.9 lbs.

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25/7/10.

Notes on Shale-Oil Industry.

Table of Costs &c. re work on Scotch Shales.-

Cost of retorting each ton of shale in Scotland	I/6
Cost of mining and delivering at retort mouth	4/- to 5/-
Cost of manufacture of sulphate of ammonia . .	2/3
	7/9 to 8/9

Miners paid 6/3 per day of 10 hours.
Workmen paid 5/9 per day of 10 hours.

The seams worked are 3, 5 or 7 feet thick and run from 22 to 40 gallons crude oil per ton. Some seams 12 feet thick are worked for 20½ gallons per ton, but as a rule the poorer the seam in oil the richer it is in ammonia. These poor seams give 60 lbs. of sulphate of ammonia per ton.

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of PLANT .- A modern retort will cost in Scotland £70 for each ton of shale put through daily, assuming 100 tons per day were to be treated the cost would be £7,000: each retort will deal with 4 to 5 tons daily so that a nest of from 20 to 25 would be necessary for this output. This is for retorts only.

If a naptha recovery plant, ammonium sulphate plant stills and all accessories for the full treatment of oil are taken into consideration the cost per ton of shale treated daily would be £140.

An estimate given by Messrs. A.F. Craig & Co., Edinburgh, for 5 benches of 64 retorts each, with all condensers, engines, pumps, tanks, boilers, sheds and connections.	43,700
A naptha recovery plant for same	1,550
An ammonium sulphate plant for same	4,320
Brickwork for these plants	16,200
	£65,770

This plant would deal with about 800 tons of shale per day and give crude oil, crude naptha and sulphate of ammonia.

The cost of a smaller plant would be proportionately higher since the buildings and machinery would remain of the same type as before, the number of retorts alone being reduced.

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Estimate of value of similar shales in Canada yielding 60 lbs. crude oil and 67 lbs. sulphate of ammonia per ton by Dr.R.W.Ells (for the Geological Dept., Canada).-

Crude oil valued at 1¼ per gallon	6	3
Sulphate of Ammonia at 1½ per lb.	8	4½
	14	7½

It is to be noted that the price for the crude oil is about the market price in Canada owing to the competition with American natural oils, the sulphate of ammonia being the more valuable product. The Canadian oil referred to contains 0.62% sulphur and has a heating power of 18,474 calories.

Price of Naptha in Scotland (1909). . . .	7d. per gallon.
Kerosene oils	6d. to 7¾ per gallon.
Gas oils for engines, &c. . . .	£5 per ton.
Lubricating oils	£5/10 to £8. per ton.
Solid Paraffin	2½d. to 3½d. per lb.
Still Grease	£4/10 per ton.
Still Coke (left in retorts) . .	£2/10 per ton.
Sulphate of Ammonia	£12 per ton.

Cost of mining and manufacturing 1 ton of shale in Scotland 8/3 - profit 3/4 per ton.

The approximate quantities of these materials produced in Scotland :-

			£
Spirit or Naptha	2,500,000	gallons.	73,000
Kerosene	17,000,000	:	500,000
Gas Oils	38,000	tons	190,000
Lubricating oils	40,000	:	280,000
Solid Paraffin wax	22,500	:	675,000
Sulphate of ammonia . . .	50,000		600,000
Still Coke	5,000	:	12,500
			£2,330,500

2,545,582 tons shale for 63,000,000 gallons crude oil, or about 8¾ per gallon on crude oil.