

REPORT ON SAMPLE OF SHALE

RECEIVED FROM

THE LATROBE SHALE & OIL COMAPANY

TASMANIA

by

Edwin M Bailey

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REPORT ON SAMPLE OF SHALE

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THE LATROBE SHALE AND OIL COMPANY, TASMANIA.

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Sample of Shale:-

The sample consisted of three lots in bags, marked A. B. and C. respectively, representing three different sections, their thickness being stated as 19 inches, 9 inches and 14 inches. In addition to these there was a fourth lot in boxes, which we marked X.

Retorting Shale:-

The Shale was distilled in the full-size Experimental Pumpherson Patent Retort. This is a complete plant comprising Retort, Condensers, Exhauster, Receiving Vessels, Ammonia Scrubbing Tower and Circulating Pump, with steam and auxiliary gas supply for heating purposes.

Samples A. B. and C. were distilled in this retort separately and successively in the inverse order of their richness, as ascertained by a laboratory test of a small sample of each lot. Average samples were also taken daily as the shale was being charged to the retort, and the laboratory results obtained from these confirmed the yields obtained on the industrial scale.

Yields:-

The following table gives the yields of Crude Oil and Sulphate of Ammonia.

Lot marked	Crude Oil Gallons per ton 2240 lbs.	Sulphate of Ammonia Pounds per ton, 2240 lbs
A.	54.92	1.57
B.	9.92	3.56
C.	27.34	8.68
Average yields supposing the three sections to be worked together, taking into consideration the relative thickness and specific gravity of the various sections	33.67	4.54

Remarks on Retorting:-

Lot B. worked well in the retort, lot C. did not do so well, and Lot A. was somewhat difficult and troublesome, owing to the fact that the spent shale was of a very friable and pulverulent nature, and as it fell together in the form of powder it prevented the passage of steam up through the mass, while at the same time, the shale in the upper portion clung to the sides of the retort, thus interfering with the heating of the retort, and reducing its working capacity.

With regard to lot X., the quantity was insufficient for making an accurate test of the yield of Oil and Ammonia, but it was nevertheless retorted for the purpose of ascertaining how the mixture of the three sections - which it was supposed to be - would behave in the retort, in view of the difficulties experienced in working the richer sections, especially lot A. by itself, as indicated above. The mixture worked satisfactorily in the retort, so that if the three sections A. B. and C. were working together, no difficulty would be experienced in this respect.

As to the heat required for the retorting of the shale it was evident that on the large scale, with a bench of retorts at work, enough gas would be produced from the distillation of the shale itself, in presence of steam, to effect this; although with a single isolated retort, the amount of heat lost by radiation is so great that some auxiliary gas had to be supplied to maintain the proper temperature of the retort, even with the richer quality A., while a good deal of gas had to be used while the poorest section B. was being worked. In any case, other fuel, such as producer gas - from the shale itself if that were the cheapest source available - would have to be employed temporarily at the outset for the initial drying and heating of the retorts, and permanently to raise steam for the distillation of the shale.

The quantity of steam, however, required for the retorts would not be great, as the ammonia yield is so small that it would not pay to work up the liquor, and only sufficient steam

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would, therefore, be required to facilitate the working of the shale in the retorts.

The average throughput of shale - per 24 hours - was 3 tons of A., 3.35 tons of B. and 4 tons of C. In the case of the lot X, it was 3.7 tons. Working the three sections together, the average daily throughput may be taken at 3.5 tons of shale per retort. This is somewhat less than that for the average Scotch Shale worked in this retort, 4 tons being the usual daily working capacity. This less throughput would necessarily increase the cost per gallon of the crude oil produced.

The Shale was, in every case, well exhausted when dropped from the retort.

Yield of Naptha from Retort Gases.-

Tests were made on the gas from each lot of shale to ascertain how much Naptha could be obtained from the retort gases after passing through the atmospheric condensers, in addition to that present in the crude oil as collected. The tests were made by passing the gases through a series of scrubbing vessels containing oil - previously steamed to ensure its freedom from naptha or light oil - and then passes through a meter. This was done in each test continuously during 24 hours, so that a fair average sample of the gas was obtained. The oil was afterwards steamed, and the absorbed naptha derived from the gas was thus expelled, condensed, collected and measured, and the quantity obtained from the measured quantity of gas employed was calculated to the total quantity of gas - leaving the atmospheric condensers - obtained from the retort per ton of shale.

The results obtained were as follows:-

Naptha obtained by Scrubbing the Retort Gases:-

Average of A., B. and C. sections together, taking into consideration their relative thickness and specific gravity:-

Crude Scrubber Naptha 9.3 gals. per ton - 2240 lbs.

The Specific Gravity of the Scrubber Naptha was .745 at 60 deg. Fah.

It must be kept in view that the removal of all this Naptha would very materially affect the heating value of the gas, the reduction due to this cause being equal to about 25 per cent of the total heating value.

Analysis of Return Gas.

Carbon Dioxide	20.2
Oxygen	2.6
Nitrogen	30.4
Carbon Monoxide	4.0
Olefines	2.6
Hydrogen	30.1
Marsh Gas.....	10.1

Heating value per Cubic Foot at 60 deg. Fahr., and 30" Bar., equals 232 B.T.U.

Crude Products.

Ammoniacal Liquor.

This was an average strength of 3.1 lbs. Sulphate per 100 gallons, and as the total ammonia only amounted to the equivalent of 4.54 lbs. of Sulphate of Ammonia per ton of Shale, it could not, of course, be profitably worked up, and must be considered not only valueless, but actually an encumbrance, as on account of its polluting character, it probably could not be allowed to flow into a stream, nor could it be used for steam raising purposes on account of its corrosive action on the boilers. It might be dealt with by pumping to the top of the spent shale bin, through which it would be allowed to percolate.

Crude Oil.

The chief characteristics of the Crude Oil are given below:-

Sec.	Specific gravity at 60 deg. F.	Flash Point Close Test.	Temperature to which it may be exposed while still remaining liquid
A.	.9365	116 deg.F.	Flows below 20 deg. F.
B.	.9562	178 " F.	Very thick at 30 deg.F.
C.	.9467	150 " F.	Flows but thick at 20deg.F.
A. & C.	.9395	123 " F.	Flows below 20 deg.F.
A,B. & C.	.9405	124 " F.	Flows below 20 deg.F.

The Crude Oil contains 0.896 % of Sulphur.

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Analysis of Crude Oil - A.C. & C. mixed in proportion.

Sp. Gr. .9405; Flash Point, 124°F. - close test.

Remains liquid and flows below 20° Fahr.

100 volumes Crude Oil distilled with external heat and sufficient steam, gave :-

A. Crude Naptha	13.85%	Sp. Gr.	.831
B. Crude distillate	83.49%	" "	.9505

Total Products 97.34%

Loss on first Distillation 2.66%

Crude Distillate B. treated with 1.5% Acid - 1.84 sp. gr.

71.56% Sp. Gr., .9505

Loss on Acid Treatment 11.93%

then with 0.75% Caustic Soda Solution - Sp. Gr. 1.35-71.093%

Sp.Gr. .9453

Loss on Soda Treatment ~~12.397%~~ 0.467%

Total Loss on treatment 12.397%

Distilled, fractionating into:-

C. Light Oil	5.85%	Sp. Gr.	.851
D. Heavy Oil	59.598%	" "	.9465
E. Residuum	3.65%		

Total Products 69.098%

Loss on second distillation 1.995%

Crude Naptha A. treated with 1.5% Acid and 0.75%

Caustic Soda, and distilled with steam only, and fractioned gave:-

F. Finished Naptha	1.53	Sp.Gr.	.783
G. Naptha Bottoms	11.65%		

Total Products 13.18%

Loss on Treatment and distillation 0.67%

Light Oil C. and Naptha Bottom G. mixed together -

equal 17.50% - treated with 1.5% Acid and 0.75% Caustic Soda Solution, and distilled, fractionating, gave:-

H. Burning Oil	11.00%	Sp.Gr.	.812
I. do.	3.50%	" "	.862
J. do.	1.65%		.9066

Total Products 16.15%

Loss on Treatment and Distillation 1.35%

Heavy Oil D. treated with 2% Acid, 54.38% - Sp.Gr. .9426

Loss on Acid Treatment 5.218%

then treated with 1% Caustic Soda Soln. 51.00% - Sp.Gr. .9395

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Loss on Soda Treatment 3.38%
 Total Loss on Treatment 8.598%

Distilled, fractionating, gave:-

K. Gas Oil, or Fuel Oil	25.00%	Sp.Gr. .9135
L. - - -	19.69%	" " .958
M. Residuum	4.20%	
	<u>48.89%</u>	

Loss on Distillation 2.11%

Burning Oil H. treated with 1% Acid and sufficient weak and strong Soda Solution gave:-

Finished Burning Oil 10.50% Sp.Gr. .8093

L. Heavy Oil treated with 4.5% acid and sufficient weak soda 17.25%

Sp. Gr. .949 -N-

Loss on Treatment 2.44%

K. Gas or Fuel Oil treated with 3% acid and sufficient weak soda 23.42%

Sp. Gr. .9114 -O-

Refined Products:-

Product	Per Cent.	Sp. Gr.
Naptha F.	1.53	.783
Burning Oil H.	10.50	.8093
I.	3.50	.862
J.	1.65	.9066
Gas or Fuel Oil - Dark K -	25.00	.9135
Heavy Oil - Dark N -	17.25	
Total Refined Products	59.43	
Total Residuum E. & M.	<u>7.85</u>	
Total Products	<u>67.28</u>	

On further refining of the Fuel or Gas Oil, the yields were:-

Product	Per Cent	Sp. Gr.
Naptha F.	1.53	.783
Burning Oil H.	10.50	.8093
I	3.50	.862
J.	1.65	.9066
Gas or Fuel Oil - Pale O -	23.42	.9114
Heavy Oil → Dark N -	17.25	.949
Total Refined Products	57.85	
Total Residuum E. & M.	<u>7.85</u>	
Total Products	65.70	

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Notes on Refined Products.

Burning Oil H.

The Flash Point is 81° Fah. - Abel Close Test. The Oil burns well, with a good clear light, but with a somewhat sulphurous smell. It has a very pale straw colour after the finishing treatment, but on keeping a few days, the colour becomes very yellow. The smell of the oil is fairly good, but quite characteristic of the oil from shales of this nature, being very persistent and impossible to completely remove even by excessively high treatments and is quite distinct from the smell of the burning oil from Scotch Shale.

Gas or Fuel Oil - Dark K.

The Flash Point is 218° Fah. - Close Test - and thus conforms to the British Admiralty's requirements, which stipulate that the flash point of the Fuel Oil supplied to them must not be below 200° Fahr. The Colour is dark red by transmitted, and dark green by reflected light, and the smell is not unpleasant.

The Oils I. and J.

Could be mixed with the above as Gas or Fuel Oil, but the flash point of the mixture would probably be less than 200° Fah.

Gas or Fuel Oil - Pale O.

Produced from K. by further treatment, has a reddish-orange colour, with a slight greenish fluorescence, and has a good sweet smell, with only a trace of the odour characteristic of these products.

Heavy Oil - Dark N.

Is a dark opaque oil, with a slate coloured fluorescence, it is practically without smell. At ordinary temperature it flows, but shows minute crystals of solid paraffin, which, however, it was impossible to remove by cooling and filtration, as the whole mass passed through the filter cloth.

This oil could only be used in the manufacture of a coarse lubricating grease, and it would not be profitable to refine it so far, as it would be as good for this purpose in the unfinished state.

The same remarks apply to the Residuum - E. and M.

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General Observations.

The Crude Oil is of a very peculiar nature, being, like oils from other foreign shales I have examined, quite different in character from, and not so responsive to treatment with Acid as the Crude Oil from Scotch Shale. Further the impurities - removable by acid - present in the Crude Fractions appear to be of the same specific gravity as the purified oil itself, which is not the case with Scotch Shale Oils. But, while this is so, excessive amounts of acid and soda have to be employed to obtain even partially-refined products, and, even at the best none of these - with the possible exception of the Naptha and Gas or Fuel Oil - is of the usual standard marketable quality in this country.

Chemicals used in refining per 100 gallons. Crude Oil:-

<u>Making Dark Gas or Fuel Oil</u>		<u>Making Pale Gas or Fuel Oil</u>
Acid - sp.gr. 1.84	3.91 gals	4.66 gals.
Caustic Soda-		
sp.gr. 1.35	1.46 "	1.46 "
Soda Ash - 58%	3.61 pounds	6.55 pounds.

Cost of Manufacture-in Scotland.

Retorting of Shale at 1/2 per ton	equals 0.416 pence p. gall.
	Crude Oil
Refining of Crude Oil	equals 0.500 pence
Making Dark Gas or Fuel Oil -	
Total retorting and refining cost	<u>0.916</u> "

Financial outcome as a Commercial Enterprise - in Scotland.

Scheme A. Disposing of the Crude Oil as Fuel Oil. -

Assuming that a lower flash point than 200°Fhr. would be accepted.

Value of Crude Oil as Fuel Oil - F.P. below 200°F.	1.960 pence
	per Gal
Manufacturing cost of Crude Oil	0.416 " "
	<u>1.544</u>
Difference	

33.67 gallons Crude Oil at 1.544d. equals 4/4 per ton of Shale, available to cover cost of mining and Breaking Breaking Scotch Shale exceeds 4/4d per ton, and as it is, therefore, quite evident, that at the present price of Fuel Oil, - and in the absence of any information as to Mining Cost of the Shale under review - assuming the Mining Cost to be the same in both cases, there would be no margin of profit at all in working this Latrobe Shale.

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Scheme B.

Refining the Crude Oil and disposing of the Refined

Products:-

Value of Naptha	1.53% @ 3.63d.	equals	5.36d.	pr	100	gals.
Value of Burning Oil	10.50% @ 2.52d.	"	26.46d.	"	"	"
Value of Total Fuel Oil	30.15% @ 1.96d.	"	59.09d.	"	"	"
Value of Heavy Grease Oil	25.10% @ 1.50d.	"	37.65d.	"	"	"
			<u>128.56 d.</u>			

Total Value of Refined Products	1.288d.	per	gall.	Crude Oil
Cost of Manufacturing and Refining	0.916d	"	"	"
	<u>0.372</u>	"	"	"

It will be seen that there is here a surplus of only 0.372d. per gallon, compared with 1.544d. per gallon under Scheme A, and as moreover the refined products are not of standard quality, Scheme A, would be the only possible one that would be adopted, and this only if the Mining Cost were very much below that which prevails in Scotland.

The yield of Naptha from the Retort Cases has not been included in the foregoing estimates, as climate conditions in Tasmania might make it impossible to obtain the best results. In my opinion, the best results that could be looked for would be a yield of 4 gallons of Refined Naptha per ton of shale.

On the basis of Scotch values and Working & Mining Costs, & assuming that Naptha was recovered from the Retort gases by Scrubbing, the result would be :-

Scheme C. Disposing of the Crude Oil as Fuel Oil and Recovering and Disposing of the Scrubber Naptha.

Per Ton of Shale

Value of Crude Oil	33.67 gals. at	1.960 d per gal. =	5/5.993
Value of Naptha	4.00 " " "	3.63 d " " "	= 1/2.52
Total Value of Products			<u>6/8.513</u>
Manufacturing cost of Crude Oil	33.67 gals at	0.416 d =	1/2.007
Manufacturing cost of Naptha, including cost of production and refining, and of fuel to replace its heating power on the gas.			= 3.336
	Total cost of Products	<u>1 5.546</u>	
Exclusive of Mining Cost			1 5.546 →

Note. The above figures can be generally balanced if the Total cost is 17.546 d instead of 1/7.546

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Difference between Value and Manufacturing cost equals 5/3.17d.

Deducting Mining Cost - assumed- equals 4/4.00d.

Leaves for interest on Capital,
Expenditure and Depreciation,
etc. and Profit.....11.17d.

(Sgd.) EDWIN M. BAILEY,

Chief Chemist.

Pumpherston Oil Works,

Mid Calder,

Scotland.

13th April, 1911.