

INVESTIGATIONS INTO THE MANUFACTURE OF ASPHALT FROM "TASMANITE"HISTORICAL:

The general purpose underlying these investigations is to ascertain if the deposits of Tasmanite oil shale in the Mersey Valley can be developed practically by a method offering better economic prospects than that of retorting the shale for oil. The latter industry has been adversely criticised, mainly on account of the average relatively low grade of the shale, and the poor quality of the oil derived from it.

The method of utilisation proposed is based on a fact well known in oil shale technology, that the oil bearing matter in the shale does not decompose directly under the influence of heat to form oils and gas, but passes by stages from a relatively insoluble state, infusible without decomposition, to an increasingly soluble solid or semi-solid, fusible bituminous substance, which on further heat treatment, may be partially or completely decomposed into oils, gas and carbon. (1) McKee, Ralph and Lyder on the Thermal Decomposition of Shales, Heat Effects J. Ind. Eng. Chem. 13, 613-18 (1921). (2) Franks and Goodier, Preliminary study of the Organic Matter of Colorado Oil Shales. Quarterly Colo. School of Mines, 17, No.4 Oct. (1922). (3) Maier and Zimmermann. The Chemical Dynamics of the Transformation of the Organic Matter to Bitumen in Oil Shale. Bull Univ. Utah, Vol. 14, No.7 pp. 62-81 (1924). Abstracts from the above references are printed in "Shale Oil" by Ralph McKee, 1925, published by the Chemical Catalog. Company, New York. Dr. Kurth of the University of Tasmania investigated this behaviour in the laboratory with reference to Tasmanite in the course of his investigational work on Tasmanite Shale, 1929-33, and a full account of his results is given in his Monograph on the Oil Shales of Tasmania and New South Wales.

In a paper published in Helv. Chim. Acta. Vol. XIV. 1930, Getzche, Vicari & Scharer describe work on the laboratory separation of the organic from the inorganic constituents of Tasmanite, and mention the formation of bitumen and oils by the heat treatment of the organic matter. Further work on the gravitational separation of the shale constituents is described by Dr. Kurth in his Monograph. Work carried out in the laboratory of the State Mines Department by Mr. W. St. C. Manson, chief chemist, was successful in obtaining an organic concentrate from the shale by froth flotation. As this offered prospects of commercial development, the State Government Mines Department continued the investigations to obtain further information on the products and the treatment methods necessary for obtaining them.

The investigations may be conveniently grouped under two main headings - (1) the concentration of the organic material (2) the conversion of the concentrate.

The objective of the work, if justified by the results, is the establishment of a plant on the shale field of a capacity sufficient to supply the Tasmanian bitumen requirements. Such a plant would require to treat 60 tons of shale per day, supplying 14.6 tons of organic concentrate per day for conversion to asphalt and oils.

This report concerns the conversion of the concentrate, and, while providing a summary of experimental results obtained, is principally directed to plant design features for larger scale operations.

The subject matter of the report is arranged under the following headings:

1. General description of the raw material, and of its behaviour under conditions relevant to the process.
2. A summary of the results of experimental work in the laboratory and with a small pilot plant including:
  - (a) a general description of the course of the reaction.
  - (b) a description of the reaction relative to temperature and time.
  - (c) data on four pilot plant tests.
  - (d) summary of results from typical laboratory tests.
  - (e) summary of pilot plant tests.
  - (f) yields adopted for discussion.
  - (g) description of the products.
3. General discussion of large scale handling methods based on experimental experience.
4. Discussion of the design for a large scale plant unit with particular reference to features arising from experimental work.
  - (a) General design - prevention of carbon formation
  - (b) Material of construction.
  - (c) Prevention of dust entrainment.
  - (d) Heating.
  - (e) Condensation.
  - (f) Plant control.
5. Summary & Conclusions.

GENERAL DESCRIPTION OF RAW MATERIAL:

1. The raw charge is in the form of a fine powder consisting of a mixture of finely/silicious clay and of amber coloured, resinous looking particles, presumably spore cases, whole and fragmentary, from fossil sporopollenine.

A screen analysis of a typical concentrate gave the following result:-

divided

<u>Size</u>	<u>% concentrate</u>	<u>Ash content of Fraction</u>	<u>Ash % of concentrate</u>
+44 mesh	0.46	32.8	0.15
+60 "	7.66	25.45	1.95
+85 "	19.47	29.4	5.72
+120 "	14.14	32.94	4.66
+150 "	12.06	34.8	4.19
+200 "	13.33	37.94	5.06
-200 "	35.13	48.06	16.88
			<u>38.61%</u>

The ultimate composition of the resinous material is (Kurth)

Carbon	-	78.5 %
Hydrogen	-	10.35 %
Nitrogen	-	0.64 %
Sulphur	-	4.7 %
by difference Oxygen	-	5.81 %

The relative quantities of inorganic filler and organic resinous material vary according to the concentration desired in the flotation plant. For practical purposes the percentage of filler may be taken as from 25% to 40% of the concentrate. For present purposes, the figure of 35% filler is adopted. This gives a concentrate weighing approximately 28 lbs. to the cubic foot, loosely packed, when dry. As delivered from the flotation plant the concentrate has a moisture content of about 40%. The appearance of the moist concentrate is somewhat deceptive, as it is relatively free, only slightly cohesive, and does not pug or become sticky. The dry concentrate is a free running powder.

The heat conductivity of the material is very low. With a charge of Tasmanite, ash content 66%, placed in a cylindrical retort of internal diameter 3 1/8" heated from the outside, Kurth found a temperature gradient of 36°C per inch for a heating rate of 1°C per minute.

The specific heat of the organic material may be taken as 0.38, and of the concentrate as 0.32 approximately.

The specific gravity of the organic material containing 5.4% ash was found to lie between 1.13 and 1.17. With 1% ash, Kurth found the gravities between the limits 1.08 to 1.12. The specific gravity of the inorganic gangue is approximately 2.5.

The organic material is partly soluble in certain organic solvents. Carbon tetrachloride extracts 2.6%, and pyridine dissolves a further 2.6% from the residue, a total of 5.2%. The extract was recovered as a reddish brown, soft, tacky resinous substance with a slight smell. Acetone and carbon disulphide also have a slight solvent action.

A calorific value was determined on a concentrate of ash content 12.36% by the Mines Department and gave a result of 14,736 B.Th. hs. per lb.

A retorting test on a concentrate of ash content 25.17% gave the following results:- based on dry concentrate.

Oil	=	53.3%
Liquor	=	2.3%
Residue	=	35.0%
Gas, scrubber spirit		
+ loss	=	9.4%
Specific gravity of oil	=	0.924
Oil yield per ton	=	129 gallons.

Tests by Kurth and by the Mines Department of New South Wales gave the following results - based on dry concentrate.

#### KURTH

Ash content of concentrate used	=	24.3 %
Oil yield	=	55.7 %
Liquor	=	2.23 %
Residue	=	35.4 %
Scrubbed gas	=	6.67 %
Specific gravity of oil	=	0.921
Oil yield per ton	=	136.

New South Wales Department of Mines (Grey-King-assay).

Ash content of concentrate used	=	30.5 %
Moisture content	=	1.03 %
Oil Yield	=	48.5 %
Liquor	=	3.8 %
Residue	=	40.1 %
Scrubbed gas	=	7.6 %
Specific gravity of oil	=	0.928
Oil yield per ton	=	117.1

#### BEHAVIOUR OF CONCENTRATE UNDER HEAT TREATMENT:

The following notes on the behaviour of Tasmanite under heat treatment at 300°C and 350°C are summarised from experiments carried out by Dr. Kurth, using a 10gram. charge, crushed through 20 mesh, placed in a hard glass tube. The vapour exit was formed by drawing the tube out to leave an exit of about 3 M.M. diameter. Heating was electrically controlled.

1. Heated to 300°C, relatively little change takes place, even after maintenance of the temperature for 220 hours, although prolonged heating tends to effect an internal rearrangement in the organic material favouring ultimate carbon formation at the expense of hydrocarbons. A very slow loss in weight takes place with a small oil yield, presumably from the soluble organic constituents. After 11 hours, about 4% of the organic material is recovered as oil. Any decomposition of the main body of the organic substance is very small.

2. Heated to 350°C, a moderately slow reaction takes place. After one hour at 350°C, the change is relatively slight. With increase of the time factor, there is a gradual increase in decomposition of the organic matter into a more soluble substance with increasing evolution of oil vapours

and gas up to a point. Beyond this point, the soluble matter decreases, and proximate analysis of the residual substance from extraction tests shows an increasing amount of fixed carbon and volatile matter. Evidently prolonged exposure to the temperature conditions involves a molecular rearrangement within the material. With a charge containing 66% of ash it takes about 20 hours, to attain the point of maximum solubility at a temperature of 350°C. After 50 hours heat treatment at this temperature, the residue was like an ordinary retort carbonaceous residue.

(3) Heated to 400°C, the decomposition rate is about ten times as fast as at 350°C, and, after two hours, the charge liquifies in the form of an asphalt with evolution of oil vapours and gas.

From the above, it appears that the charge can be preheated to 300°C, and possibly to 350°C, without any considerable reaction taking place, and without serious adverse effect on final treatment, provided the concentrate is not maintained at these temperatures for more than a short period before being raised to the final reaction temperature of around 400°C. At 300°C, several hours would have little effect, but at 350°C, it would be advisable to continue the treatment almost immediately.

Experiments on heating a relatively small volume of concentrate with a large heating surface, e.g. thin layers on a heated lead bath, showed that it was very difficult to obtain conversion to a liquid phase. In the majority of tests, the concentrate was recovered in a carbonised form. Similarly, if the concentrate were heated in pipes of as small a cross section as  $\frac{3}{4}$ " and ample provision were made for evolved vapours, a liquid phase was not obtained. Provided however, provision were made for the condensation and refluxing of the evolved oil vapours, the liquid phase could be obtained without noticeable signs of coking under suitable temperature conditions.

LABORATORY EXPERIMENTAL:

Tests carried out on small quantities of concentrate placed in crucibles or tins, loosely covered and treated in an air oven maintained at 390°C, for 2½ hours yielded material of slightly coherent nature, mixed largely with relatively unchanged spores. On cooling, the substance was of a somewhat rubbery nature. After 2 hours at 410°C, a carbonised layer was obtained at the heating surface, and the remainder of the charge was relatively little changed. Tests carried out by immersing the containers in a lead bath at 390°C, gave part of the concentrate as a coherent rubbery mass with the remainder relatively little changed. Higher temperatures gave part fully carbonised, part little changed and a small portion of a rubbery nature. Extraction of the mass with carbon disulphide, and recovery of the extract yielded a tough somewhat resilient bitumen.

When provision was made to keep the concentrate moving in contact with the hot walls a liquid phase was obtained over a wide range of reflux conditions.

The above tests indicate the necessity for preventing overheating by maintenance of as even a temperature in the charge as possible, and of providing for contact of

the heating mass with the oil vapours evolved which appear to aid the formation of soluble constituents.

(2) The apparatus adopted for laboratory testing consisted of a double jacketed mild steel pot, circular in cross section, 4.5" inside diameter x 3.5" deep, and holding 300-400 grms. of concentrate. The jacket was filled with lead, and heated by a Meker burner. A two bladed stirrer passed through a stuffing gland in the cover plate, and scraped the full area of the pot bottom and the sides to a height of  $\frac{1}{2}$ ". The cover plate was fitted with a pressure gauge, and a  $\frac{1}{4}$ " gas pipe offtake leading to a condensing system and a gas meter, and was bolted into position by means of 4 studs welded to the outside of the pot, the joints being made with red lead and linseed oil. A closed tube carrying a pyrometer passed from the outside jacket across the lead bath to the mid-point of the base of the inside pot, and in contact with the wall. A thermometer was inserted in the gas offtake, and the apparatus as a whole protected from drafts by a suitable arrangement of asbestos sheeting. Varying reflux effects were obtained by altering the length of the vertical  $\frac{1}{4}$ " offtake pipe.

Experiments were carried out under varying conditions of time, temperature and pressure.

With this apparatus, no difficulty was experienced in obtaining liquid asphalt even with the reflux pipe reduced to a minimum. The maximum temperature used in the lead bath was 520°C; and, in general 500°C. Varying pressures were tried, the normal being atmospheric. The usual time taken for completion of the reaction, starting from cold was from 2 $\frac{1}{2}$  to 3 hours, the time before appreciable reaction started being approximately 1 hour. The shortest time taken to complete the reaction after commencement of decomposition was 1 $\frac{1}{2}$  hours.

#### PILOT PLANT EXPERIMENTAL:

Following work on this apparatus, a small scale plant was erected in Launceston. The primary objective was to obtain, if possible, sufficient asphalt for a practical road test, by which the Public Works Department could gain information on the suitability of the material for their purposes. As the plant would be operated under conditions nearer to ordinary industrial practice than was the case in the laboratory, it was hoped that sufficient information would be obtainable from the operations to form the basis for designing a commercial plant. It should be noted that the experimental aspect of this work was subservient to the obtaining of sufficient asphalt for road tests, with the result that once conditions had been established for obtaining the asphalt, the work was largely standardised under them.

A sketch of the plant erected showing the essential features is appended, and the plant is more fully described later in this report.

From the work carried out, the course of the reaction by which the light sand coloured concentrate is changed to asphalt may be described in general terms as follows:-

GENERAL DESCRIPTION OF REACTION:

As the temperature rises, a slow evolution of non-inflammable gas takes place with little noticeable change in the appearance of the concentrate. This is followed by a considerable increase in the rate of gas, make, and the gas can be ignited. Traces of oil appear at this stage and the concentrate is slightly darkened in colour. A relatively very rapid evolution of gas follows, with a considerable increase in oil make, and the colour of the charge changes to a dark brown whilst still appearing relatively dry. This stage usually marks the maximum rate of gas flow, and, in many cases, but not necessarily, the maximum rate of oil make. The colour of the concentrate now changes to black with considerable oiliness, the material being less gritty and softer to the touch when rubbed between the fingers, but is still non coherent. As the change proceeds, the concentrate, on cooling sufficiently, can be pressed into a coherent mass, which, when cold, is somewhat resilient and rubbery in appearance but has no definite melting point. Shortly afterwards the charge appears as a frothy semi-liquid mass which quickly becomes a liquid showing considerable traces of partly changed spores. On cooling at this stage, the charge sets to a slightly resilient, dull surfaced semi-solid which readily melts to an asphaltic like liquid. On further heating, the charge changes to a soft bright bitumen of a grade approaching a light road oil. Continued heating simply distils oil from the asphalt, leaving a residual asphalt of a heavier grade.

During the course of the above changes, a yield of rich gas maintained with a flow of oil, the rate of which is controlled by the rate of heating. In the later stages, the rate of oil flow can be increased considerably without a proportionate increase in gas flow, the conditions evidently approaching more nearly an ordinary crude oil distillation as distinct from the earlier stages when the decomposition of the spore material is the controlling factor.

DESCRIPTION OF REACTION RELATIVE TO TEMPERATURE AND TIME

<u>Total time run</u>	<u>Rate of heating per hour</u>	<u>Temperature of charge</u>	<u>REMARKS</u>
95 minutes		315°C	Heating started from cold. Trace of water & gas over this period.
110 "	40°C	325°C	
130 "	15°C	330°C	Slight increase in gas rate but still very low approx. 90ft. 3 ton hur.
155 "	12°C	335°C	Gas rate about doubled and gas just igniting. Oil vapours in condenser.
180 "	12°C	340°C	Gas burning steadily. Oil beginning to distil. Charge is a non-coherent dark brown powder.
205 "	12°C	345°C	Very slow oil distillation.
230 "	12°C	350°C	The charge is a damp black free moving powdery mass, a sample

(Continued)

<u>Total time run</u>	<u>Rate of heating per hour</u>	<u>Temperature of charge</u>	<u>REMARKS</u>
			from which, after cooling sufficiently, compressed in the fingers to a coherent, somewhat rubbery mass, partly soluble in carbon-disulphide and showing considerable unchanged spores.
255 minutes	12°C	355°C	
289 "	12°C	360°C	The charge had liquified to a black asphalt but contained a large proportion of unconverted spores. On cooling a sample it set to a somewhat resilient solid. Steady evolution of oil vapours and gas.
370 "	13°C	380°C	Charge was a thin bright black asphalt, which, when cold would approximate a penetration of 300.

The following notes give data from four typical pilot plant runs. The times are shown in hours or decimals of hours, gas make in cubical feet per ton under atmospheric conditions, rate of gas make in cubic feet per ton per hour, oil made in gallons per ton, rate of oil make in gallons per ton per hour. Of the temperatures shown, T, represents the hottest point of the flue surrounding the converter shell, T2 a little over half way to the stack, and T3 the temperature of the charge in the early stages before shrinkage, and later the temperature of the vapours above the charge. The flue gases entering the stack were generally at a temperature of 325°C.

DETAILS OF TYPICAL PILOT PLANT TESTS

Test No. 16

Weight of dry charge = 168 lbs.  
Ash content = 35.4%

<u>Time run</u>	<u>Gas made</u> <u>ft. 3/ton</u>	<u>Gas rate</u> <u>ft. 3/ton</u> <u>hour</u>	<u>Oil made</u> <u>Gallon/</u> <u>ton.</u>	<u>Oil rate</u> <u>Gals./ton/</u> <u>hour</u>	<u>T1</u>	<u>T2</u>	<u>T3</u>	<u>Remarks</u>
2 hrs.	30 ft. <sup>3</sup>	15 ft. <sup>3</sup>	-	-	525°C	350°C	230°C	slow gas make charge little changed.
3 hrs.	86 ft. <sup>3</sup>	56 ft. <sup>3</sup>	-	-	530°C	370°C	265°C	Gas burning continuously. Traces of oil
3.6 4	340 ft. <sup>3</sup>	154 ft. <sup>3</sup>	not taken		550°C	385°C	305°C	Charge darkening
4.25			3.3	5				
4.42			6.6	19				
4.66			9.9	14				
4.91			13.2	14				
5	700 ft. <sup>3</sup>	360 ft. <sup>3</sup>			530°C	395°C	315°C	Charge dark brown colour non-coherent.
5.17			16.5	19				
5.33			19.8	20				
5.5			23.1	19				
5.71			26.4	16				Period of max- imum gas evolu- tion
5.93			29.7	15				
6	1073 ft. <sup>3</sup>	373 ft. <sup>3</sup>			530°C	400°C	325°C	Charge not

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<u>Time run</u>	<u>Gas made</u> <u>ft. 3/ton</u>	<u>Gas rate</u> <u>ft. 3/ton</u> <u>hour</u>	<u>Oil made</u> <u>Gallon/</u> <u>ton</u>	<u>Oil rate</u> <u>Gals./ton/</u> <u>hour</u>	<u>T1</u>	<u>T2</u>	<u>T3</u>	<u>remarks</u>
								Liquified. Damp black mass showing signs of coherence when pressed.
7	1260 ft. <sup>3</sup>	187 ft. <sup>3</sup>	33	3	535°C	280°C	290°C	Temperature set back while testing.
8	1380 ft. <sup>3</sup>	120 ft. <sup>3</sup>	36.3	3.3	550°C	400°C	320°C	Frothy liquid mass sets to resilient solid.
9.	1533 ft. <sup>3</sup>	153 ft. <sup>3</sup>	39.6	3.3	540°C	400°C	320°C	Liquid with unconverted spores.
9.6			42.9	5.5				
10	1693 ft. <sup>3</sup>	160 ft. <sup>3</sup>	46.2	8.2	550°C	400°C	335°C	Fully converted
			49.3					
<u>Test No. 22</u>								
			Weight of dry charge =		170 lbs.			
			Ash content =		37.2%			
2 hrs.	10 ft. <sup>3</sup>	5-ft. <sup>3</sup>	-	-	525°C	360°C	240°C	Slow gas make little change (gas burning continuously).
2.5								
3	88 ft. <sup>3</sup>	78 ft. <sup>3</sup>	-	-	515°C	380°C	280°C	Gas rate increasing rapidly. Traces of oil.
3.5								
4	345 ft. <sup>3</sup>	257 ft. <sup>3</sup>	3.3	3.3	500°C	390°C	310°C	Period of maximum gas.
4.58			6.6	6.5				
5	474 ft. <sup>3</sup>	129 ft. <sup>3</sup>	9.9	7.9	440°C	360°C	300°C	Charge darkening. Heating rate eased back.
6	645 ft. <sup>3</sup>	171 ft. <sup>3</sup>	11.0	1.1	400°C	340°C	290°C	Charge dark brown and non-coherent.

<u>Time</u>	<u>Gas made</u>	<u>Rate</u>	<u>Oil Made</u>	<u>Rate</u>	<u>T1</u>	<u>T2</u>	<u>T3</u>	<u>remarks</u>
7	753 ft. <sup>3</sup>	108 ft. <sup>3</sup>	12.5	1.5	470°C	370°C	290°C	Charge a non-coherent damp black mass.
7.33			13.2	1.3				
8	872 ft. <sup>3</sup>	119 ft. <sup>3</sup>			510°C	380°C	300°C	Signs of coherence. Sets to a black somewhat rubbery solid.
8.4			16.5	3				
8.91			19.8	6.4				
9	1041 ft. <sup>3</sup>	169 ft. <sup>3</sup>	20.4	6.6	520°C	390°C	320°C	Charge frothy semi-liquid setting to a black tacky resilient mass.
9.4			23.1					
10	1200 ft. <sup>3</sup>	159 ft. <sup>3</sup>	26.4	5.6	500°C	390°C	330°C	Liquid conversion incomplete
11	1325 ft. <sup>3</sup>	125 ft. <sup>3</sup>	33	6.6	475°C	380°C	330°C	

Test 38

Weight of charge = 158 lbs.  
Ash content = 26%

2 hrs.	7 ft. <sup>3</sup>	3.5 ft. <sup>3</sup>	-	-	540°C	345°C	200°C	Gas burning continuously. Traces of oil.
3	28 ft. <sup>3</sup>	21. ft. <sup>3</sup>	-	-	545°C	360°C	225°C	
3.5								Gas burning continuously. Traces of oil.
4	170 ft. <sup>3</sup>	142 ft. <sup>3</sup>	-	-	580°C	390°C	260°C	
4.83			3.54					Gas burning continuously. Traces of oil.
5	453 ft. <sup>3</sup>	283 ft. <sup>3</sup>			580°C	410°C	305°C	
5.16			7.08	10.7				Period of maximum
5.5			10.62	10.4				
5.7			14.16	17.7				Period of maximum
5.83			17.7	27				
6	836 ft. <sup>3</sup>	383 ft. <sup>3</sup>			550°C	400°C	300°C	Gas evolution

<u>Time</u>	<u>Gas made</u>	<u>Rate</u>	<u>Oil made</u>	<u>Rate</u>	<u>T1</u>	<u>T2</u>	<u>T3</u>	<u>remarks</u>
6.16			21.24	10.7				
6.4			24.78	14.7				
6.83			28.32	8.2				
7	1026ft <sup>3</sup>	190ft <sup>3</sup>			500°c	385°c	315°c	
7.41			31.86	6.1				
8	1200ft <sup>3</sup>	174ft <sup>3</sup>			535°c	385°c	300°c	
8.7			35.4	3				
9	1368ft <sup>3</sup>	168ft <sup>3</sup>			550°c	400°c	315°c	
10	1492ft <sup>3</sup>	124ft <sup>3</sup>	38.94	2.7	575°c	415°c	325°c	
10.17			42.48	20.8				
10.5			46.02	10.7				
10.83			49.56	10.7				
11	1652ft <sup>3</sup>	160ft <sup>3</sup>	53.1	20.8	575°c	425°c	335°c	
12	1880ft <sup>3</sup>	228ft <sup>3</sup>	64	11	575°c	425°c	335°c	Heat off at 12. Fully converted.

Test 37.

Weight of charge = 152 lbs  
 Ash content = 25.3%

<u>Time</u>	<u>Gas made</u>	<u>Rate</u>	<u>Oil made</u>	<u>Rate</u>	<u>T1</u>	<u>T2</u>	<u>T3</u>	<u>remarks</u>
2 hrs.	9ft <sup>3</sup>	4.5ft <sup>3</sup>	-	-	530°c	372°c	230°c	
3	78ft <sup>3</sup>	69 ft <sup>3</sup>	-	-	550°c	390°c	280°c	
4	321ft <sup>3</sup>	243ft <sup>3</sup>	Traces		560°c	410°c	300°c	
4.5			3.68					
5	604ft <sup>3</sup>	283ft <sup>3</sup>	7.36	7.36	530°c	405°c	310°c	
5.5			11.04	7.36				
5.92			14.72	8.7				
6	928ft <sup>3</sup>	324ft <sup>3</sup>			570°c	425°c	330°c	
6.08			18.4	23				
6.25			22.08	21				
6.5			25.76	14.7				
6.83			29.44	11.2				
7	1230ft <sup>3</sup>	302ft <sup>3</sup>			525°c	405°c	325°c	
7.5			33.12	5.5				
8	1422ft <sup>3</sup>	192ft <sup>3</sup>			545°c	420°c	330°c	
8.25			36.8	4.9				
8.75			40.48	7.3				
9	1606ft. <sup>3</sup>	184ft <sup>3</sup>	44.16	14.6	570°c	430°c	340°c	
9.25			47.84	14.6				
9.5	1732ft <sup>3</sup>	126ft <sup>3</sup>	51.52	14.6	575°c	430°c	355°c	
9.67			55.2	21.6				
	1901ft <sup>3</sup>		63.5					conversion complete Heat off Finish

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SUMMARY OF YIELDS FROM TYPICAL LABORATORY TESTS

<u>Ash content of concentrate</u>	<u>Time Under heat hours</u>	<u>Asphalt% by weight</u>	<u>Crude Oil% by weight</u>	<u>Aqueous liquor % by weight</u>	<u>Gas% by weight</u>	<u>Crude Oil Galls. per ton.</u>	<u>Gas Cubic ft. per ton.</u>	<u>Unaccounted for</u>	<u>REMARKS</u>
23.54%	8	82	8.3	1.3	3.4	21.1	1058	5	Medium reflux used Asphalt soft & ductile
36.6%	2.5	77.2	13.9	1.5	5.0	35.3	1555	2.4	No reflux used. Asphalt penetration approx. 150.
25.17%	6.	74.7	15.2	2.8	4.6	39.6	1431	2.7	Reflux considerable. Soft ductile asphalt.
24.74%	3	74	17.5	1.5	4.4	44.5	1370	2.6	No reflux soft asphalt.
29.7%	2.75	69.4	22.3	1.9	4.9	56.7	1524	1.5	No reflux
25.17%	3.3	65.2	23	2.2	6.9	60	2146	2.7	Reflux used soft asphalt.
25.17%	2.75	60	26.5	2.8	7.0	67.4	2178	3.7	No reflux, asphalt grade approx. 80 penetration.
24.15%	2.5	59	31.1	2.0	5.6	79.2	1742	2.3	No reflux, asphalt grade about 80 penetration.
19.5%	4.5	70	12.3	2.0	7.7	31.3	2395	8.0	Conducted under pressure of 50lbs. sq. in. soft ductile asphalt.
66.2%	2.5	82.7	10.7	2.5	2.5	27.2	778	1.6	Shale used for charge.

It will be seen that a noteworthy percentage of the charge is not accounted for in the above tests. The apparatus used was not suitable for accurate quantitative work, as it was difficult to recover the asphalt completely from the pot and stirrer at the conclusion of each test. Attempts were made to recover the asphalt completely by solution but were unsatisfactory. A removeable pot was tried, but condensation of oils around the pot made this unsatisfactory. In the majority of instances, the greater part of the loss can be credited to asphalt, but, owing to occasional vapour leaks past the stuffing gland, it has been considered best to segregate the figure. Vapour leakage would account for the greater part of the loss in the example of a test carried out under pressure.

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SUMMARY OF RESULTS OBTAINED FROM PILOT PLANT TESTS

<u>Weight of dry charge</u> lbs.	<u>Ash content</u>	<u>Heat-ing time</u> hours	<u>Maximum Temperatures Recorded °C</u>			<u>Asphalt % by weight</u>	<u>Crude oil % by weight</u>	<u>Gas % by weight</u>	<u>Total % by weight</u>	<u>Crude oil yields gal. per ton</u>	<u>Gas Yield cubic ft. per ton.</u>	<u>Maximum Oil rate gals/ton/hour</u>	<u>Maximum gas rate Ft<sup>3</sup>/ton/hr.</u>	<u>Time of Maximum Gas flow hrs.</u>
			T1	T2	T3									
182	27.5%	87	430	370	312	61.0	7.3	8.9	77.2	19.2	1.2	81		
214	34.8%	30	485	350	285	78.0	8.0	6.0	92.0	21	1844	1.62	136	12
115	30.0%	30.5	490	395	305	70.0	12.5	7.5	90	33	2300	1.35	146	19
120	30.0%	26	515	385	305	75.0	13.4	7.5	95.9	35	2296	3.4	168	8
176	36.0%	28	575	415	335	76.0	15.1	6.8	97.9	39.6	2125	6.3	170	6
177	37.8%	15.75	600	420	340	73.0	15.1	6.1	94.2	40	1896	12.6	234	10
110	37.8%	10	590	400	310	80.0	10.0	6.0	96.0	26.7	1883	31	306	8
160	37.8%	17.5	570	385	320	76	11.9	6.3	94.2	31.4	1950	8	294	8
165	38.5%	16	550	375	320	80.6	10.8	6.0	97.4	28.5	1840	4.5	230	8
164	35.0%	14	550	365	310	79.2	11.3	5.6	96.1	30	1720	6	219	6
169	36.0%	15	575	370	310	77	11.3	6.0	94.3	30	1870	5	239	6
160	36.0%	12.3	560	360	315	77.2	11.8	6.8	95.8	31	2100	5.25	252	3
164	36.0%	12	565	355	315	77.2	11.8	6.8	95.8	31	2100	4	300	5
164	35.0%	15.5	560	375	315		14.0	6.8		37.1	2100	20	341	4.5
168	35.4%	9.5	550	400	325	74.5	18.7	5.5	96.0	49.2	1711	20	373	5
162	37.2%	10.0	560	400	330		13.6	5.8		35.8	1804	10.4	332	4
165	40.0%	11.8	540	410	335		13.3	5.8		35	1804	25	312	8
162	39.0%	12	590	405	330	77.4	13.7	5.4	96.7	36	1680	17	304	8
163	37.2%	10	570	415	325		14.3	5.7		37.6	1773	14	419	5
161	37.4%	10.2	530	400	330		14.0	5.1		36.9	1572	14	236	5
170	37.2%	9.5	520	390	330	78.3	12.5	4.3	96.7	33	1325	6	263	4
168	38.0%	13.4	530	405	330		13.3	6.0		35	1845	13	320	6

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SUMMARY OF RESULTS OBTAINED FROM PILOT PLANT TESTS (cont.)

Weight of dry charge	Ash Content	Heat- ing Time	Maximum Temperatures Recorded °C			Asphalt % by weight	Crude Oil %	Gas % by weight	Total % by weight	Crude oil yield gal. per ton	Gas Yield cubic ft. per ton.	Maximum Oil rate gals./ton/hr	Maximum Gas rate ft <sup>3</sup> /ton/hr.	Time of Maximum Gas flow (hr)
			T1	T2	T3									
174	38.5%	12	530	405	330)		14.3	5.6)	37.7	1720	19	373	6	
174	36.0%	15	490	370	310)	77.6	10.0	5.5)	98.1	26.3	1711	7	270	9
146	28.0%	12	570	400	315)		18.9	6.98)	49.8	2148	.46	322	6	
163	25.3%	12	520	400	320)		16.7	5.7 )	43.9	1760	44	220	7	
170	27.1%	11	545	390	330)		16.9	6.0)	44.5	1852	19	290	4	
140	27.1%	11	500	370	320)	73.1	19.3	6.9)	96.4	51	2112	24	384	5
150	28.6%	14	500	385	320)		17.0	6.2)	44.8	1941	5	254	5	
165	28.6%	13	510	400	320)		17.7	6.1	44.9	1887	20	366	6	
155	25.3%	11.5	570	400	335	74.8	16.4	6.0	97.2	43.3	1820	36	462	7
148	25.3%	15	492	340	285	71.0	19.0	7.0	97.0	50	2186	23	424	6
159	25.3%	11.66	520	380	310	69.1	21.3	6.2	96.6	56.1	1845	22	451	5
153	25.3%	11	550	405	335	70.6	21.1	5.8	97.5	55.6	1800	15	424	5
167	25.3%	14	570	425	350)						40	403	7	
152	25.3%	9.33	575	430	355)						22	324	6	
158	25.5%	12	580	425	335)						28	383	6	
170	26.5%	12	565	435	350)	69.5	20.72	6.35	96.6	54.6	1975	13	355	6
166	25.5%	12	570	420	330)						40	350	7	
165	28.5%	12.75	560	415	330)						20	353	8	
173	29.0%	12.75	585	425	320)						11	360	10	
147	29.0%	11.5	600	415	320)						23	335	7	
511	27.1	(3 runs)				73	18.0	6.5	97.5	47.4	2022 Gross of 3 charges -			

SUMMARY OF PILOT PLANT RESULTS:

Excluding preliminary trials, a total of 13,195 lbs. of wet concentrate was used for asphalt conversion in a series of 45 tests. This quantity gave 7,265 lbs. of dry concentrate from which were obtained 5,390 lbs. of asphalt, 1,124 lbs. of oil, 440 lbs. of gas and aqueous liquor amounting to approximately 2% of the dry charge. The remainder of the quantity charged 2.3%, was mainly in the form of carbonaceous material, particularly in the earlier runs, asphalt not recovered and weighed and general manipulation loss.

PREOLENNA, NEW SOUTH WALES & TASMANITE SHALES

As a matter of interest, the process was applied to shale samples from Preolenna and New South Wales, but in neither case were the results encouraging. In the case of New South Wales shale, the product was a soft, non ductile, oily black mass containing paraffin wax. The shale appeared to pass through a stage somewhat similar to the intermediate stage of the Tasmanite conversion, when a dull black, non-ductile, somewhat resilient mass could be obtained, uneven and rough in texture and infusible without decomposition. The best result with the Preolenna shale gave a light, hard very short pitch, more like a semi-coked coal tar pitch.

A test carried out on a rich sample of Tasmanite shale gave sticky bituminous mass which compressed to a dense coherent block.

For the purpose of this report, and based on the results obtained, the yields of products from a concentrate of ash content 35% are taken as follows:-

REPRESENTATIVE

YIELDS OF PRODUCTS FROM CONCENTRATE ASH CONTENT = 35%

Asphalt	= 76%	= 1702 lbs. per ton
Crude Oil	= 12.5%	= 280 lbs. per ton = 33 gals/ton
Gas	= 5.5%	= 123 lbs. per ton = 1713ft <sup>3</sup> /ton
Liquor	= 2.0%	= 45 lbs. per ton
	<u>96.0%</u>	<u>2150</u>

DESCRIPTION OF PRODUCTS:

ASPHALT

The above Asphalt has the following composition

Bitumen soluble in carbon disulphide		<u>% of asphalt</u>
		= 50%
Filler insoluble in carbon disulphide	= 50%	
Ash content of filler	= 92%	= 46%
Volatile matter in filler	= 8%	= 4%

The volatile matter in the filler consists of volatile inorganic constituents such as water of constitution

in the clay and carbon-dioxide in the carbonates present, bitumen absorbed in filler and not removable by solvent, carbon formed during conversion if over treatment takes place and partially converted organic matter. The inorganic filler in the asphalt consists of finely divided clayey material and quartz. The mudstone which overlies and underlies the shale seam is of a somewhat similar composition. Although free from oil bearing organic matter, samples of this mudstone showed weight-losses on ignition, after pre-drying, varying from 3.78% to 6.46%. There is little doubt that part of the ignition loss on the filler is due to inorganic matter.

The composition of the ash free soluble bitumen varies according to the conditions under which it is obtained, but the following gives the characteristic groups present:-

Asphaltous acids	=	1.04%
Asphaltous acid anhydrides	=	.76%
Asphaltenes	=	30 - 32.9%
Oily constituents	=	26.8 - 39.5%
Resinous compounds	=	35.8 - 26.3%
Carbenes	=	negligible to 0.2%

The specific gravity of the total asphalt depends largely on the amount of filler present, typical figures being 1.33 at 77/77°F for an asphalt of filler content = 37.6% and 1.55 at 77/77°F for one of filler content 56%.

The specific gravity of the soluble bitumen depends on the grade, that of a light grade being 1.047 at 77/77°F.

The soluble bitumen is very ductile, and the penetration and ductility vary with the treatment. Tests have been carried out by fluxing the asphalt with residual oil obtained from the bye-product crude oil by distillation, and hardening the fluxed asphalt by blowing with air at 250°C, for varying periods. By this means, a product of any desired degree of hardness was obtained.

Tests were also carried out by heating the fluxed asphalt with varying amounts of sulphur, from 2% to 5%. The product was recovered as a tough, somewhat resilient mass.

Tests on suitably graded asphalt as a base for bituminous paint and varnish and for moulded articles indicated that it should be suitable for such purposes. If there are commercial possibilities for such side applications and for insulating and water-proofing uses, reports had best be obtained from specialists in these industries.

Experiments in the emulsification of the asphalt have been carried out in the colloid mill patented by Emoleum Ltd. of Melbourne with stabiliser as used for petroleum bitumen. With filler content as low as 2%, attempts to emulsify were unsuccessful. Further work is required to find a suitable stabiliser.

Test blocks were made with varying grades of asphalt and different aggregate mixes and submitted to Public Works Department Officers for examination, and the asphalt has been used by the Public Works Department in a road test. Reports have been obtained on its behaviour while being laid, and under traffic conditions.

Complete physical tests on the asphalt have not been carried out owing to lack of the special apparatus required.

#### CRUDE OIL:

The crude oil obtained is considerably lighter than normal crude oil obtained from shale. The specific gravity varies according to the conditions under which conversion of the concentrate takes place, and, in general, ranges from 0.85 to 0.88 at 66°f

During conversion, the oil obtained in the early stages was very dark in colour, that distilling later was **light verging** on water clear, although changing to a reddish wine colour on exposure to air. Samples were taken for distillation **analysis** at various stages of the test, usually representing the commencement, at quarter time, at half time, at three quarter time and at the finish. In every case, the distillate from the first sample went off colour at an earlier stage than with the others, and, on standing, darkened in colour till it became practically opaque. The other samples gave distillates light in colour when kept from contact with the air. In general it was found that the oils became progressively lighter in boiling range as the conversion proceeded, but considerable variation was noted. The effect is presumably due to cracking of the reflux, although any effect on the gas yield appears to be masked by the main re-action, as the lightest oils were obtained when the gas yield was relatively low, although the charge and setting were around the maximum temperatures.

Apart from the light grade, the oil is similar in character to a normal Tasmanite Shale Oil, and the information contained in the Report of the Tasmanian Shale Oil Investigation Committee, Geological Survey Mineral Resources No.8, Vol. 11, 1933, can be taken as applicable.

The crude oil and a heavy distillate from the oil with hydrogen sulphide removed, have been tested by the Engineering Department of the Tasmanian University as diesel fuels. Reports on their behaviour have been obtained.

The following analysis of the oil was obtained from Dr. ~~Knuth~~:-

#### DISTILLATION ANALYSIS A.S.T.M. 100 m.l. dry settled oil

Initial boiling point	(first drop)	75°c
	10% over	137°c
	20% over	172°c
	30% over	207°c
	40% over	235°c
	50% over	255°c
	60% over	269°c
	70% over	282°c
	80% over	293°c
	90% over	315°c

No water distilled over during the test.  
Specific gravity of the oil 0.8572 at 15°C.  
Saturation 50% ± 1% with three volumes of 1.84 Sp. gr. sulphuric acid.

Sulphur Content 2.31%

A standard refining test on another sample of the crude oil, using caustic soda solution, sulphuric acid of specific gravity 1.84 and plumite solution gave the following yields of finished products.

Motor spirit	=	18.5%
Power kerosene	=	29.4%
Residual fuel oil	=	43.0%
Treatment loss	=	9.1%

The details are as follows -

Volume of original sample = 400 ccs.

Initial boiling point = 78°C

10 ccs.	over	at	116°C	
20 "	"	"	125°C	
30 "	"	"	131°C	
40 "	"	"	137°C	10% at 137°C
50 ccs	over	at	142°C	
60 "	"	"	147°C	
70 "	"	"	151°C	
80 "	"	"	157°C	20% at 157°C
90 ccs	"	"	161°C	
100 "	"	"	164°C	
110 "	"	"	168°C	
120 "	"	"	178°C	30% at 178°C
130 ccs	"	"	182°C	
140 "	"	"	187°C	
150 "	"	"	191°C	
160 "	"	"	195°C	40% at 195°C
170 ccs	"	"	199°C	
180 "	"	"	206°C	
190 "	"	"	210°C	
200 "	"	"	214°C	50% at 214°C
210 ccs	"	"	219°C	
220 "	"	"	224°C	
230 "	"	"	233°C	
240 "	"	"	237°C	60% at 237°C
250 ccs	"	"	242°C	
260 "	"	"	247°C	
270 "	"	"	250°C	67.5% at 250°C

The light distillate to 237°C (240 ccs) from the distillate was re-distilled into two fractions.

	Initial boiling point	=	63°c
A.	63°c - 160°c	=	120 ccs
		=	30% of original crude oil.
B.	160°c - 230°c	=	118 ccs.
		=	29.5% of original crude oil.

SAMPLE A.

Given 10% of a 10% caustic soda solution, well shaken, settled, and tar run off.

	Washing loss	=	0.86%
Water washed	" "	=	0.5%
Given 3% of sulphuric acid, sp.gr 1.84, in 2 lots	Washing loss	=	7.7%
Water washed and neutralised	" "	=	0.86%
	Total loss	=	<u>9.92%</u>

The oil was distilled, the distillate given a sodium plumbite solution wash, and was redistilled. The final distillate had an end point of 142°c, and the yield was 74 ccs or 18.5% of the original crude oil. Apart from the lower end point, the spirit appeared to be similar to petrols obtained by similar treatment of normal crude oil from Tasmanite.

The residual oil from the treatment of sample A was combined with sample B, giving 150 ccs. Sample treated with 10% of 10% caustic soda solution -	washing loss	=	5.0%
Waterwashed	" "	=	1.0%
3% of sulphuric acid in two lots	" "	=	5.6%
Water washed and neutralised	" "	=	0.6%

The total treatment loss was 12.2%  
 Oil recovered = 130 ccs  
 Adding manipulation loss,  
 total loss = 13.3%

The oil was distilled.

Distillate 117.5 ccs	=	29.4%
Residual oil 12 ccs.	=	3.0%

DISTILLATION TESTS SHOWING VARIATION IN CRUDE OIL AT DIFFERENT STAGES OF CONVERSION

Total oil yield 2.5 gallons. Weight of concentrate 170 lbs.

A. Sample 1. from the first quart of distillate. Gas flow at time approximately 15 cubic feet per hour.

Initial boiling point	=	50°c
50°c - 100°c	=	2.2%
- 110°c	=	4.3%
- 120°c	=	6.4%
- 130°c	=	9.0%
- 140°c	=	10.2%

50°c	- 150°c	= 11.7%
	- 160°c	= 14.6%
	- 170°c	= 17.0%
	- 180°c	= 19.6%
	- 190°c	= 22.2%
	- 200°c	= 25.8%

Sample 2. 4th quart. Gas flow approximately 9 ft.<sup>3</sup>/hour.

Initial boiling point = 56°c

56	- 100°c	= 2.6%
	- 110°c	= 5.1%
	- 120°c	= 9.0%
	- 130°c	= 13.1%
	- 140°c	= 17.2%
	- 150°c	= 21.0%
	- 160°c	= 24.0%
	- 170°c	= 29.0%
	- 180°c	= 32.3%
	- 190°c	= 37.0%
	- 200°c	= 42.4%

Sample 3. 7th quart. Gas flow 12ft<sup>3</sup>/hour.

Initial boiling point = 48.°c

48.	- 100°c	= 3.6%
	- 110°c	= 6.4%
	- 120°c	= 10.0%
	- 130°c	= 13.3%
	- 140°c	= 17.0%
	- 150°c	= 20.8%
	- 160°c	= 25.5%
	- 170°c	= 31.1%
	- 180°c	= 34.1%
	- 190°c	= 39.0%
	- 200°c	= 45.7%

Sample 4. 10th quart. Gas flow approximately 9ft.<sup>3</sup> per hr.

Initial boiling point = 46°c

46	- 100°c	= 3.3%
	- 110°c	= 6.1%
	- 120°c	= 9.1%
	- 130°c	= 14.2%
	- 140°c	= 17.7%
	- 150°c	= 21.7%
	- 160°c	= 26.0%
	- 170°c	= 29.7%
	- 180°c	= 33.3%
	- 190°c	= 38.6%
	- 200°c	= 45.7%

B. Weight of charge = 174 lbs. Total oil yield 2.95 gallons.

Sample 1. First quart. Gas flow 17ft<sup>3</sup> per hour.

Initial boiling point = 66°c

66	- 100°c	none in receiver
	- 110°c	= 0.4%
	- 120°c	= 1.0%
	- 130°c	= 2.0%
	- 140°c	= 4.4%
	- 150°c	= 8.0%
	- 160°c	= 14.4%

66 - 170°c	= 18.0%
- 180°c	= 21.1%
- 190°c	= 27.0%
- 200°c	= 32.0%

Sample 2. 4th Quart. Gas flow 29 cubic ft. per hour.

Initial boiling point.

	= 49°c
49 - 100°c	= 2.4%
- 110°c	= 4.3%
- 120°c	= 6.6%
- 130°c	= 9.4%
- 140°c	= 12.0%
- 150°c	= 15.0%
- 160°c	= 19.0%
- 170°c	= 23.1%
- 180°c	= 26.3%
- 190°c	= 29.0%
- 200°c	= 34.4%

Sample 3. 7th Quart. Gas flow 26 cubic ft. per hour.

Initial boiling point

	= 50°c
50 - 100°c	= 3.3%
- 110°c	= 5.9%
- 120°c	= 9.8%
- 130°c	= 13.3%
- 140°c	= 17.7%
- 150°c	= 21.1%
- 160°c	= 26.4%
- 170°c	= 31.1%
- 180°c	= 35.5%
- 190°c	= 37.0%
- 200°c	= 41.1%

Sample 4. 10th Quart. Gas flow 7 cubic ft. per hour.

Initial boiling point

	= 53°c
53 - 100°c	= 3.7%
- 110°c	= 7.7%
- 120°c	= 12.2%
- 130°c	= 17.2%
- 140°c	= 22.7%
- 150°c	= 28.8%
- 160°c	= 34.4%
- 170°c	= 40.0%
- 180°c	= 48.0%
- 190°c	= 54.4%
- 200°c	= 62.2%

Sample 5. Taken from the last of the distillate after heating had been cut off. Gas flow 5ft<sup>3</sup>/hour.

Initial boiling point

	= 60°c
60 - 100°c	= 0.9%
- 110°c	= 2.2%
- 120°c	= 5.0%
- 130°c	= 9.1%
- 140°c	= 14.0%

60	- 150°c	= 18.9%
	- 160°c	= 25.0%
	- 170°c	= 29.0%
	- 180°c	= 34.6%
	- 190°c	= 40.0%
	- 200°c	= 46.1%

GAS

The density of the gas obtained from conversion of the concentrate varied at different stages of the test, figures obtained ranging from 0.0843 lbs per cubic foot at the beginning to 0.0658 lbs per cubic foot at the end, with an average value over the main decomposition period of 0.718, all reckoned at N.T.P.

The gas was not scrubbed to remove light hydrocarbons. In view of the relatively small scale of operations contemplated it is unlikely that scrubbing of the gas will be considered. Calorific values were not taken, but from the time when combustible gas appears till the finish the gas burns with a rich luminous flame. Despite the presence of a considerable percentage of hydrogen sulphide and carbon dioxide, the gas appears to be richer than normal coal gas, and should provide at least 9 therms per ton of concentrate.

DISCUSSION OF THE WORK IN RELATION TO PLANT DESIGN FOR LARGER SCALE WORK.

Probably the nearest approach under commercial operation to the conditions required is to be found in an industry established in the United States for the manufacture of asphalt from the mineral Wurtzilite. This industry is described in Abraham's book on "Asphalt and Allied Substances" from which the following brief account is abstracted.

"The insoluble, infusible wurtzilite, is crushed to break coarse lumps and fed to a horizontal cylindrical still through two charging hoppers, one at either end, provided with tightly fitting covers which are fastened into place before the fires are started. The bottom of the still is protected by a fire brick arch, and the products of combustion, after passing underneath the arch, are returned in three fire flues (one 10" in diameter and 2 by 6"), and thence back again in the space surrounding the still above the arch. The vapours generated by the wurtzilite pass upwards through two pipes joined to the top of the still near the ends, and connected with a single water cooled coil, which condenses the vapours and returns most of the condensate to the still. Best results are obtained if 2 to 5% of the distillate (based on weight of charge) is drawn off.

It takes from six to eight hours to raise the temperature of the charge to 400°f, then four to six hours to reach maximum temperature 580°f, which is maintained from twenty-four to thirty-six hours, and the contents are then allowed to cool to 450°f, and drawn through a valve at the bottom."

The general idea behind the above process is similar to that under discussion, but there are several vital differences which preclude the essential simplicity above, the most important difference being the temperature required, 750°f, in the case of Tasmania as against 580°f in the case of wurtzilite.

GENERAL DISCUSSION OF HANDLING METHOD FOR LARGER SCALE OPERATIONS.

The work to date has been limited to batch processing, and the results are not encouraging if large scale operations are considered solely on a batch basis. The heating, time and handling factors would probably result in a prohibitive cost.

Some consideration has been given to a plant working under continuous conditions. The tests on rapid heating of the concentrate, by having a heating surface relatively large to the quantity under treatment were not encouraging as there was difficulty in preventing carbonation of the material. The conditions would be approached by passing the concentrate through a heated rotating kiln. Similar difficulties do not encourage the use of a molten alloy bath as sometimes used for tar distillation. For continuous work, it appears that conditions which have made for relative ease of conversion to a liquid phase are closely paralleled in a plant of the Salerno type, a descriptive drawing of which is attached to this report. This plant has been designed primarily for retorting shale, and has also found application as a drier. In considering its suitability for the process under discussion, the following points should be noted:-

- (1) The liquid phase of the concentrate occupies approximately one quarter of the volume of the solid phase.
- (2) There is a possibility that the concentrate passes through a stage which might tend to ball up in the retort, causing intermittent variation in the through put rate. In a batch process, apart from a temporary effect on heat transfer, such a condition would be unimportant, as there is no question of transferring the material under treatment. Given sufficient time, under the influence of reflux oils and heat, the charge liquifies. In a continuous process, difficulties similar to those encountered in attempts to handle intumescing shales by continuous means may be encountered.
- (3) In the case of retorting shales the condition of the residue is unimportant, provided it has been efficiently exhausted of oil bearing material. Operations are therefore conducted with a reasonable working margin for control. In the case under consideration, the residue is the principal marketable commodity, and its value is seriously affected by over or under treatment. It is questionable whether the operation of a continuous plant under the requisite conditions would be sufficiently steady and capable of accurate enough control to maintain the desired results.
- (4) The salermo plant is an expensive type, and, of its nature a single unit would be designed to cope with the required throughput. Any hitch in the working of this unit would result in a complete cessation of output till remedied, with consequent standby losses.

Apart from the above considerations, the advantages of continuous working are obvious, particularly from the aspects of heating efficiency and wear and tear.

Consideration of the factors involved indicates that the process lends itself conveniently to some compromise between purely batch and continuous work, and it is suggested that development proceed along these lines.

It is recommended that the wet concentrate from the flotation plant filter be fed to a continuous drier and pre-heater of approximately 15 tons daily capacity, heated by the waste heat from a second section of the plant described later. At this stage the concentrate would be dried and raised to as near 300°C as practicable. This section of the plant would be in accord with standard practice, and, given the necessary data, manufacturers of drying equipment will be in a position to make recommendations as to type and cost. No difficulties need be anticipated which are not encountered in industrial drying practice.

From the drier the concentrate would be discharged to a closed heat insulated bin by a suitable conveyor, the bin to act as a charging reservoir for a bench of converters. A suitable conveyor running in a closed insulated casing would carry the hot concentrate across the bench of converters, each converter having a feed bin connected by chute with the conveyor casing, with suitable arrangement for opening or closing access to the conveyor. The feed bins to each converter would require an arrangement for positive feed by which sufficient concentrate was maintained in the bin to provide a seal against vapour leak from the converter while loading. The pressure in the converter at this period need not be more than a few inches water gauge.

It is possible that if air cannot be kept at a sufficiently low concentration in the plant between the drier and the converter, it may be necessary to omit pre-heating of the concentrate on account of combustion risk. Hot dry concentrate at an average temperature not exceeding 200°C, if left for some time in the pilot plant with the cover plate off allowing free access of air has been known to start a slow spontaneous combustion where in contact with the hot metal sides. Local temperature was probably well above 200°C at the point of combustion origin. The concentrate can be taken as considerably more inert than fine coal of similar size.

Assuming that each converter has a working capacity of 1.5 tons of dry concentrate, and that the bench contains five units with a sixth as standby, the following sequence of operations is deduced from experience with the pilot plant. The figures are not intended as a forecast of probable actual results, but are used to indicate the proposed sequence of operation.

<u>TIME</u>	<u>NO. 1 UNIT</u>	<u>NO. 2 UNIT</u>	<u>NO. 3 UNIT</u>	<u>NO. 4 UNIT</u>	<u>NO. 5 UN</u>
2hr. 24 mns.	1.5 tonsfd.	Fd.started			
4 " 48 "	Treating	1.5 tons fed.	Feed started		
7 " 12 "	"	Treating	1.5tons fed.	Feed std.	
9 " 36 "	"	"	Treating	1.5tons fed.	Feed std.
12 "	Liq.Fd.St.	"	"	Treating	1.5tons fed.
14 " 24 "	3 tons fed	Liq!fd.st.	"	"	Treating
16 " 48 "	Treating	3tons fed.	Liq.f.d.st.	Treating	"
19 " 12 "	"	Treating	3tons fed.	Liq.f.d.st.	"
21 " 36 "	Finished) & run off)	"	Treating	3tonsfed.	Liq.f.dng
24 "	Fd. std.	Run off	"	Treating	3tons fed.

It will be seen that it is proposed to increase the actual batch capacity of the unit converter by feeding a second charge when the first has liquified. The relatively small volume occupied by the liquid makes this possible, and the conversion of the second charge will probably be assisted by the presence of the asphalt. Small scale tests do not indicate that the first converted asphalt, will suffer from the longer period in the converter, as the temperature of the charge is low enough to prevent damage.

The above method affords a near approach to continuous operation, the heat lost during the run off period being relatively small, and the greater proportion of the plant being always in operation. There is also positive control of the required end point of the reaction, and the provision of a spare unit for use in the event of repairs, would not be a costly matter.

If the above general method of handling the conversion phase is accepted as offering the best prospects for economic working combined with positive control, it will be essential to have definite information on the actual performance of a proposed full scale unit converter, before details for a large scale plant can be finished. Experience in plant performance involving heat transfer has shown that there is serious risk in working by analogy from one system to another in which essential dimensions are altered.

It is therefore proposed to erect one such unit to obtain information on daily capacity, fuel requirements, temperature and volume of waste gases, make gas velocities and effect on dust entrainment condenser capacities, efficient utilisation of permanent make gas, and general engineering and mechanical behaviour. Some of the above data will have a bearing on drier design.

Since no drier will be available, it will be necessary to dry the charge in the converter as with the pilot plant. To gain information on the effect of doubling the charge, it will be necessary to run hot asphalt into a charge predried in the converter, but the final effect will be similar to that in the proposed plant practice.

#### DISCUSSION OF DESIGN FOR LARGE SCALE UNIT CONVERTER

The pilot plant unit was in the form of a close tank of U shaped cross section, 4 ft. long by twenty nine inches deep by sixteen inches wide, fitted with a  $1\frac{1}{2}$  inch steel shaft set along the longitudinal axis of the semi-circular tank bottom, and fitted with T shaped paddles arranged to sweep the whole of the semi-circular surface, with an end arm sweeping the end surface exposed to the heating flue. The tank was of  $\frac{1}{4}$  inch mild steel plate, and was fitted with a cover plate of the same metal bolted into position with suitable jointing material, the  $\frac{1}{2}$  inch bolts being spaced at 3 inch intervals. The tank interior could be conveniently exposed for access and examination by removal of the cover plate. A short section of 6 inch pipe with flanged end was welded to the cover plate, and fitted with removable cover for charging purposes. A length of closed  $1\frac{1}{2}$  inch pipe was welded to the underside of the cover, so that, with the cover in position, it just cleared the paddles. This was used as pyrometer pocket for taking the temperature of the charge before liquification and of vapour near the charge surface, after liquification. The vapour offtake was a 4 foot length of 3 inch pipe flanged at one end and bolted to the cover plate. A welded plate closed the top end, and was fitted with a

thermometer pocket and a vapour release valve. This valve was not used in practice, and was fitted as a precaution in the event of a condenser blockage. At a height of 3 feet 6 inches a 2 inch socket was welded to the vapour off take and connected the apparatus through a length of 2 inch piping to a water cooled coil 120 feet long of  $\frac{3}{4}$  inch piping. Condensed oil and liquor ran to a sealed trap, and permanent gas was metered, and passed through a safety trap to the furnace. A 2 inch run off pipe was welded to the end at the bottom and fitted with a steam plug valve. A 10 lb. pressure gauge was fitted for precautionary purposes, but during conversion the pressure was never sufficient to move the needle. Pressures of 3 lbs. per square inch were frequently registered during the drying period. Provision was also made for dip sampling of the charge. The stirring gear was driven by a 3 H.P. electric motor through a worm drive, the speed of the stirrer shaft being 2.4 r.p.m. Heating was by a 2 $\frac{1}{2}$ " non-rotary Major low pressure oil burner, the furnace arrangement being as shown in sketch.

Tests carried out with the above plant revealed certain features which require consideration in designing a larger scale unit.

#### CARBON FORMATION:

The U shaped cross section is unsatisfactory as it was found essential to keep any concentrate in contact with hot metal surfaces moving, otherwise there was risk of local carbonisation with the deposition of hard carbonaceous residue on the walls. In the pilot plant this was noted particularly at the junction of the semi-circular bottom with the vertical sides, the hottest part out of reach of the paddles. As first designed, a clearance of about  $\frac{1}{8}$ " was allowed between the paddles and the walls of the tank. This was always found to fill with carbon to the range of the paddles.

The original paddles had faces 2" wide. These operated satisfactorily with the dry concentrate, but with a wet charge, e.g. of 50% moisture content, a heavy resistance was set up due to a tendency on the part of the concentrate to pack at the point where the paddle met the vertical wall at a tangent. To overcome this, the faces on the paddles were cut away to a depth varying from one inch at the centres to  $\frac{1}{2}$ " at the ends. This allowed the concentrate to pass behind the paddle at the point where the packing tendency occurred.

Owing to the arrangement of the paddles on the shaft, there was a tendency with a wet concentrate for the paddles to act as a screw convey or resulting in a preferential accumulation at one end. This tendency disappeared as the concentrate dried and became more mobile. In an attempt to combat this movement, the paddles were given a set on the shaft against the direction of travel. This was only partially effective, and left a wider clearance space between the paddles and the walls than originally provided. This aggravated the carbon trouble on the walls.

It was decided to modify the paddles to allow for a clean scraping of the sides, to keep the charge moving in contact with the hot shell. To do this with the minimum of inconvenience and expense, sliding heads were fitted to the paddles, loosely studded to the original heads, free to move within a limited range radially from the shaft and also to a slight extent around the radial direction. The movable heads were held against the sides of the converter by two single wire steelsprings, sprung from a bolt set through the paddle arm, one spring on each side of the arm. By this means the original clearance space between the walls and the paddles, and the variation in the space due to the set given the paddles were eliminated.

This arrangement worked satisfactorily as a temporary expedient. Despite any temperature effect on the spring temper, the paddle heads were held well against the walls, except in two cases where insufficient play had been allowed on the studs to take up abnormal clearance. Following this alteration, the walls were found free from carbon within the range of the stirrers, the only noteworthy depositions being two small patches at the junction of the hot end wall with the sides, above the range of the end stirrer.

It will be seen from the sketch that the end of the converter to which the run off pipe was attached was in the hottest part of the flue. This arrangement was used to increase the small heating surface available. Trouble was experienced with carbonisation of the asphalt remaining in the run off pipe between batches, the cumulative effect being blockage of the pipe. In a larger unit, it would be preferable to keep both ends away from the flue gases, and lag them sufficiently to conserve heat.

From the above, it would appear that with the stirrers accurately fitted to a unit of circular cross-section, properly heated, there should be no trouble from carbon formation. It is, however, essential, that the paddles actually scrape the surface of the metal, or a carbon film will form to fill any clearance allowance, and seriously affect heat transfer.

Assuming a working capacity of 1.5 tons of dry concentrate per unit, it is suggested that the shell be 20 feet long by 3 ft. 6 inches diameter. Allowing for a 6 inch diameter paddle shaft the relevant data for a charge of 1.5 tons of dry concentrate would be -

Volume of initial charge, 1.5 tons,	=120	cubic ft.
Depth of charge in converter	2.15	feet
Free surface at rest	= 68	square feet
Free Gas volume	= 68.5	cubic ft.
Heating surface (approximate)	= 99	square ft.
Depth of charge after liquifaction	= 8 $\frac{3}{4}$	inches
Depth of charge after liquifaction of 3 tons	= 14 $\frac{1}{4}$	inches
Maximum temperature of flue	= 600 $^{\circ}$ c or 1112 $^{\circ}$ F	
Maximum temperature of charge	= 400 $^{\circ}$ c or 752 $^{\circ}$ F	
(Probable maximum 390 $^{\circ}$ c)		

Apart from the question of standing capacity of the unit, the suggested dimensions should be carefully scrutinised with reference to engineering considerations particularly from the mechanical and heating aspects.

The paddle shaft proposed is 6 inch outside diameter by 1 $\frac{1}{2}$  inch thick, hollow cast steel tube. The paddle arms would therefore be 18 inches long from the shaft perimeter to the shell of the converter, and would be operating at a speed of 3 revs. per minute. It is proposed to carry this shaft through stuffing glands at each end of the shell to roller bearings erected independent of the converter. The mechanical roundness of these proposals in view of the temperature, span and radius of action involved requires checking.

For heating a material with the physical characteristics of the concentrate, the diameter of the converter suggested is large, and much will depend on the effectiveness of the movement of the charge if a reasonable time factor is to be obtained. It may be considered preferable to reduce the standing capacity and increase, if necessary, the number of units.

MATERIALS OF CONSTRUCTION:

The choice of material for the construction of the shell depends mainly on heat, corrosion and abrasion resistance, combined with mechanical suitability.

Examination of the 1/4" mild steel shell of the pilot plant on completion of the experiments, showed that, in general, the metal surface appeared to be little affected. There was, however, definite evidence of local pitting. The total time under heat would be approximately one thousand hours (1000 hours) of which about one third would be under low temperature conditions of drying the charge. It should be noted that owing to the carbon deposition on the shell in the earlier part of the work, the metal must have been subjected at times to relatively severe conditions. There was also evidence at that time of local occlusions between the carbon layer and the metal. These local occlusions would be subjected to considerably higher temperatures than normal. Nevertheless the presence of hydrogen sulphide and complex organic sulphur compounds in the gases and liquid must be considered. In general, it is not considered that corrosion will seriously differ from that encountered in petroleum refinery practice.

Bearing in mind the temperatures involved and the possibility of corrosive effects, it is considered that the choice of material lies between a heat resisting cast iron and a suitable steel alloy.

A heat resisting cast iron as recommended by the British Cast Iron Research Association has proved itself very satisfactory as a retort metal for distilling Tasmanite shale. The corrosive agents were similar and temperature conditions more severe. This metal was used in the form of heavily ribbed castings 1 inch thick which were assembled by being bolted together across machined flanges. A suitable structure might be built of similar castings, semi-circular in shape and 4 ft. long, bolted together, with matching end castings. There is no doubt that the metal would be satisfactory from the temperature and corrosion resisting aspects, provided the method of assembly is sound. The proposed converters are not under steady heat conditions. This differentiates them from continuously operated retorts, and the comparatively poor expansive properties and tensile strength of cast iron might increase the liability to crack. Cast iron has also the disadvantage of weight. In view of the temperature variation when running off and charging, and its possible effect on the castings, especially in local positions such as around the run off pipe, a heat resisting steel is to be preferred, if a suitable alloy can be recommended by the manufacturers. In this case the structure would require to be welded throughout to avoid the projection of rivet heads complicating the stirring arrangements, unless countersunk rivets can be recommended, or a suitable type of flanged joint. Since only the bottom half of the converter is subjected to the flue gases, it may be possible to cheapen the cost of making the other half of mild steel. It is suggested then that the shell be made of heat resisting steel plate, 1/2 inch thick, and the upper half of 1/2 inch mild steel plate, welded throughout or rivetted through suitable flanged joints, subject to manufacturers recommendations and suggestions.

It will be noted that, after liquifaction, the charge will be below the level of the heating flue. Prevention of local over-heating of the metal will depend on the paddles carrying the liquid against the walls.

#### PREVENTION OF DUST ENTRAINMENT:

The design of the vapour offtakes should make provision for the prevention of dust carry over with the vapour stream, and consequent risk of condenser blockage.

At the vapour velocity in the pilot plant during conversion there was no trouble from dust carry over, but at maximum steam velocity during drying there was a tendency to dust entrainment, although the 3" rise pipe was clean to practically full bore after completion of the tests, and no cleaning of the  $\frac{3}{4}$ " condenser coil was necessary. On one occasion a temporary block in the coil during drying raised to 51 lbs/ins<sup>2</sup> the pressure when the system cleared itself.

The maximum velocity of gases and oil vapours during any conversion in the 3" rise pipe, was 0.97 ft. per second. During drying the maximum velocity of steam would be 4.9 feet per second, with an average of 3.25 ft. per second. The average vapour velocity during conversion would be very much less than 0.97 feet per second.

By adopting the maximum rates of gas and oil evolution and assuming that they take place simultaneously, seldom the case in practice, a unit with a charge of 1.5 tons of concentrate fitted with 4 by 1 ft. diameter, off takes will give a maximum vapour velocity in the off takes of 0.36 feet per second. With a 40% moisture content in the original charge and assuming a five hour drying period the maximum steam velocity if the unit is used as a drier would be around 1.5 ft. per second. At these rates there should be no serious trouble with dust, but adequate provision should be made for cleaning off take pipes and manifolds.

As a check against the velocity of entrainment, a series of tests was carried out by causing an air stream of varying velocities to pass up a glass tube of known sectional area. Concentrate was introduced into the air stream and the following results were noted:-

<u>Velocity of Gas Stream</u>	<u>Remarks</u>
8.3 feet per second	Entrainment considerable
6.7 " " "	" "
5.8 " " "	Noteworthy carryover
3.3 " " "	About balanced. Very slight carry
2.2 " " "	Entrainment negligible
1 " " "	Nil.

The above velocities are higher than would be expected from the minimum size particle present. When the concentrate is viewed under the microscope, the very fine particles are seen to have largely collected on the surface of the larger spore fragments. It is possible that this effect may give rise to the more rapid settling rate.

The following calculations were used in connection with the above data:-

CRUDE OIL DATA:

Specific gravity of crude oil	= 0.86
Distillation analysis	=
Initial boiling point	= 78°C
10%	= 137°C
20%	= 157°C
30%	= 178°C
40%	= 195°C
50%	= 214°C
60%	= 237°C
70%	= 255°C
80%	= 290°C
90%	= 355°C
Final boiling point	= 370°C
Mean boiling point	= $\frac{2466}{11} = 224°C = 371°F$
Molecular weight	= 150
Specific heat vapours	= .52
Specific heat liquid	= .64
Molal heat vapouration	= 16,500 B th.Us.
Maximum rate oil flow/ton/hour	= 40 gallons
	= 344 lbs.
Vapour temperature	= 570°F = 1030°R.

From  $W = \frac{MPV}{RT}$

We have V, the volume vapours at 570°F and atmospheric pressure equivalent to 40 glls. = 1722 Cub. ft.

GAS DATA;

The maximum rate of flow of permanent gas in cubic feet per ton per hour and N.T.P.	= 500 cub.ft.
The volume of permanent gas at 570°F & 760 mm	= 990 ft. cube
Total maximum combined vapour and gas rate in cubic feet/ton/hour	= 2712 cub. ft.
For 1.5 tons	= 4068 cub. ft.
Maximum rate per second	= 1.13 cub. ft.
With 4 + 1 foot diameter offtake maximum velocity	= 0.36 feet/sec.

DRYING:

With a charge of moisture content 40% and equivalent to 1.5 tons of dry concentrate, assuming a five hour drying period at 18 lbs. per sq. ins. absolute, the average volume of steam per second = 2.77 cubic feet. Taking the maximum at 50% in excess of the average figure, the maximum volume of steam per second becomes 4.16 cubic ft., or a maximum velocity of 1.32 feet per second.

Comparison of the above figures with pilot plant results.

TYPICAL TEST:

Maximum rate of oil flow	= 1 qrt. in 6 mins.
	= 2.5 glls. per hour.
	= 21.5 lbs. per hour.

Temperature of vapours at time = 570°F  
 Volume = 108 cubic feet  
 Maximum permanent gas rate = 32 cubic feet per hour  
 At 570°F = 63.3 cubic feet  
 Maximum total perhour = 171.3 cubic feet  
 Maximum velocity = 0.97 ft. per sec.

DRYING PERIOD IN PILOT PLANT:

Average rate of drying = 0.41 lbs. water per minute at 1lb/sq.in.  
 Maximum rate = 0.66 lbs. " " " " 3lbs/in<sup>2</sup>  
 At 1 lb/in<sup>2</sup> 24.4 cubic feet steam = 1 lb. of water  
 At 3 lbs/in<sup>2</sup> 21.9 cubic feet steam = 1 lb. of water  
 Average rate = .16 cubic feet per second, velocity = 3.25 ft/sec.  
 Maximum rate = .24 cubic feet per second, velocity = 4.9 ft/sec.

HEATING OF UNIT:

The pilot plant does not assist materially in estimating the probable heat requirements on account of the small scale and method of heating adopted.

In the earlier tests, the gases from the combustion flue passed directly to the back of the setting, there meeting secondary air fed from a pre-heating duct passing under and parallel to the combustion chamber. The mixed gases then passed over the walls of the converter. With this arrangement, insufficient time was allowed for proper mixing, and temperature control was difficult. In addition, direct radiation from the flame was reflected from the back wall of the setting to the metal wall, causing local carbonisation by its intensity. The arrangement was modified by blocking off the furnace flue as shown in sketch, and sending the hot furnace gases down into the secondary air duct. The secondary air was caused to rise over a hurdle just before this point with the object of setting up a swirling action to aid mixing and minimise stratification of the gases. The mixed gases then passed round the back flue before striking the converter shell at the point where pyrometer T1. was inserted. By this means direct radiation from the furnace was prevented, any radiation effect being through the brickwork. The furnace was consequently more in the nature of a hot air heater with low efficiency.

An actual test during a drying period by working out heat to steam and to charge relative to heat supplied, neglecting the heat required to raise the setting from the cold, gave an efficiency of 18%. Between the drying period and the time when appreciable reaction commenced, the heat absorbed as sensible heat was approximately 5% of the heat supplied. If this were approximately the proportion absorbed throughout conversion, the heat required per ton of concentrate would be 10.5 therms. In the absence of more reliable data, the figure of 11 therms per ton is taken as a safe working basis for the second heating stage of the proposed process.

The heating arrangement proposed is as shown on sketch. The combustion chamber 2 feet wide by 1ft.high extends for the full 18 foot length of the converter between the end walls, and is separated from the heating flue by surrounding the converter by a suitable fire brick arch or fire tile dividing wall. Air ducts for admission of mixing air pass underneath and parallel to the combustion chamber, the hot gases from the combustion are directed down towards the mixing air, and the mixture led upwards to the

flue surrounding the converter, passing along the walls of the converter to a waster head flue from which, normally, the gases would pass to a drier.

As the hot gases pass along the combustion chamber, heat will be given out in the following directions-

- 1. (a) By radiation from the roof to the converter shell and to refractory surface round the heating flue.
- (b) By conduction and convection through the roof to the flue gases in the heating flue.
- 2. (a) By radiation through the floor to refractory surfaces in the air preheating duct.
- (b) By conduction and convection to the air passing along the duct.
- 3. By conduction through the side walls. This should be minimised by suitable incorporation of insulated bricks in the setting.

It is desired to increase the radiation effect through the roof to a maximum by suitable construction of the roofing tiles or arch, if such a construction is practicable.

Sufficient air is allowed to pass along the air duct and mix with the exit gases from the combustion flue to reduce the temperature of the mixed gases to 1180°F at the point when they first meet the metal shell of the converter. It is also proposed to use this air duct as a combustion chamber for the permanent gases from the converter, if they are not required elsewhere. The gases will be supplied at approximately atmospheric pressure, and it is proposed to burn them with air supplied at from 1" to 3" water gauge pressure in a type of burner as sketched.

An attempt has been made to work out the probable result from such an arrangement, but the assumptions necessary were considered too numerous to be of practical value.

For discussion, the following figures are assumed (neglecting the gas)

Heat lost by radiation and conduction= 40%  
 Overall heat transfer to charge - = 18%  
 Heat carried away in the gases = 42%

Assuming fuel oil of specific gravity 0.946, calorific value 18,640 per lb. and consumption 4.75 gallons per hour or 45lbs per hour, we have

Total heat to setting per hour	= 839,000 Bth	Us.
Heat to charge	" " "	= 151,020BthUs.
Heat to flue gases	" "	= 352,380
Heat lost		= 335,600
	839,000	839,000

With a charge of 1.5 tons, the heat required has been taken as 16.5 therms. On the above assumption, the time required for conversion would be approximately 11 hours.

With a dry concentrate this should be a practicable figure. As previously stated, the main objective in erecting a single unit is to obtain the actual figures.

To dry a charge of 40% moisture equivalent to 1.5 tons dry, the heat required is -

Sensible heat in 1 ton water from 60°F to 212°F	= 340,480
Latent heat in steam	2,163,840
Sensible heat in concentrate	163,430
Total heat required	= 26.68 Therms
Heat in flue gases from converter per hour	= 352,380
Total heat in waste gases in 11 hours	= 38.72 Therms

The drier would require to operate with an efficiency of approximately 68%. Efficiency figures on rotary driers are quoted at from 40% to 65%. Under the above assumptions, it is probable that the heat to the drier would require boosting. Part of the permanent gas yield might be used. Assuming a drier efficiency of 50%, gas to the value of 14.6 therms would be required for boosting.

The total heat requirements for the conversion of 1 ton of concentrate of 40% moisture content (i.e. 1.66 tons of wet concrete) would, according to these figures, be 71 therms made up as follows - figures taken for 2.5 tons of wet concentrate of 40% moisture content i.e. for the normal charge of 1.5 tons of dry concentrate.

Heat required to dry concentrate	= 26.68 therms	= 25.0%
Heat required for conversion	= 16.5 "	= 15.4%
Heat lost in drier	= 26.68 "	= 25.0%
Heat lost in converter setting	= 36.91 "	= 34.6%
Total	= 106.8 "	= 100%

equivalent to 71 therms for 1.66 tons of wet or 1 ton dry concentrate.

The overall efficiency, reckoned on the basis of heat utilised in charge to heat supplied would be 40.4%.

With the oil from the process valued at 4 pence per gallon at the plant, the heating cost per ton on the above basis, allowing for gas would be approximately 11/5 or 2/9 per ton of shale.

It is hoped that further work with a continuous vacuum type of filter will enable the moisture content to be reduced below the figure of 40% taken above. If the practical results with the large unit do not materially reduce the above heavy heat requirements, the use of local coal might be considered in place of oil. The cost per therm would be one penny as against 2.25 pence for oil. At this difference, however, it is doubtful if the coal would show any overall advantage.

#### HEATING DATA

##### Pilot Plant Drying Period

Weight of wet charge	= 290 lbs
Weight of moisture	= 120 lbs
Weight of dry charge	= 170 lbs
Sensible heat in moisture	= 120 x 152 = 48,240 Bth Us
Latent heat	= 120 x 966 = 115,920 " "
Sensible heat of charge	= 170 x 242 x .32 = 13,165 Bth Us
Total	= 147,325 " "

Drying period from 60°F to 302°F fuel used) = 4.75 gallons  
 = 45 lbs  
 C.V. = 18,640 BthUs. lb.  
 Heat supplied = 838,800 Bth.Us.  
 Heat used = 18% approximately

### FURNACE CHAMBER ON PROPOSED UNIT

Sectional area of combustion chamber = 2 feet square  
 Area of roof radiating to converter = 36 " "  
 Area of floor radiating to air duct = 33 " "  
 Total area of side walls =  $2(1 \times 18) \times 4$  = 40 " "  
 Assumed fuel per hour = 4.75 gallons  
 Specific gravity = 0.946  
 Weight of fuel per hour = 45 lbs  
 calorific value taken = 18,640 Bth.Us pr. lb.  
 Theoretical air per perfect combustion = 13.96 lbs per lb fuel  
 Allow excess air and leakage = 100%  
 Total air per lb fuel = 27.92 lbs  
 Weight of flue gases per hour = 1260 lbs  
 Mean specific heat = 0.258  
 Theoretical flame temperature = 2640°F  
 Assume flue gas exit temperature from combustion chamber = 1620°F  
 Average combustion chamber temperature = 2130°F  
 Volume of flue gas at this temperature = 88,680 Cubic ft.  
 Average velocity in chamber = 12 ft. per sec.  
 Weight of flue gases leaving per hour = 1260 lbs  
 Temperature of gases leaving combustion chamber = 1620°F  
 Specific heat at 1620°F = 0.27  
 Heat content of gases = 530,700 Bth. Us.  
 Assume 800 lbs of air flowing in air duct per hour, and temperature of air at mixing point = 340°F  
 Specific heat at 340°F = 0.25  
 Heat taken up by air =  $800 \times 280 = .25$  = 56,000 Bth. Us.  
 Temperature above 60°F of mixed gases would be  $\frac{5600 \quad 530,700}{(800 \times .25) + 1260 \times .27}$   
 = 1086°F  
 Temperature of gases entering heating flue = 1146°F  
 Total flue gases per hour = 2060 lbs  
 Total air per hour = 2056 lbs  
 Theoretical air = 628 lbs  
 Under these conditions the flue gases would be expected to have a carbon dioxide content of approximately 5%.  
 Taking volume of flue gases at 37 cubic feet per lb.  
 Total volume of flue gases at temperature in converter flue = 76,220 cubic ft.  
 = 21 cubic ft. per sec. approximately

### CONDENSATION:

It is proposed to connect the four vapour offtakes in two sets of two by a pair of headers each leading to a water cooled condenser. Two old coil condensers totalling about 300 feet of 2 inch line are available and will be used if found in satisfactory order. It is proposed to use coil condensers for the single test unit, although properly

designed tubular condensers would give better results for any larger plant on account of the higher water velocity. As against this, cast iron piping in a box type of condenser would have greater resistance to corrosive action by the gases.

Experience with the 2 inch coils mentioned above in connection with crude oil stills, indicates that they should easily handle the vapour load. The following data gives some idea of the position with a 200 ft. coil under possible maximum load conditions.

maximum	
Adopting momentary/figures for oil and gas flow	
Temperature of vapours entering condenser	570°F
Mean boiling point of oils	371°F
Maximum rate of oil flow for 1.5 ton charge	= 60 gals.
	= 516 lbs
	= 110 Bth Us
Latent heat of vapours	
Heat to be abstracted for condensation	= 56,760 Bth.Us.
	= 0.64
Specific heat of oils	
Heat to be abstracted to cool condensed oils from 371°F to 150°F	= 72,983 Bth Us.
Heat to be abstracted to cool gas from 570°F to 150°F	
Maximum rate of gas flow per hour (1.5 tons)	= 148 lbs.
Mean specific heat of gas	= 0.5
Heat to be withdrawn	= 31,080 BthUs.
Total maximum heat to be withdrawn per hour	= 160,823 " "
Take cooling water entering at 50°F and leaving at 150°F	
Quantity of cooling water required	= 1608 lbs p.hr. = 2.7 gal/min.

Condensation takes place while cooling water rises from 115°F to 150°F

$$\begin{aligned} \text{Mean temperature difference during condensation} &= \frac{(371-115) - (371-150)}{2.3 \log \frac{371-115}{371-150}} \\ &= 239^\circ\text{F} \end{aligned}$$

Cooling takes place while the cooling water rises from 50°F to 145°F, and oil and gas cools from 371°F to 150°F.

Mean temperature difference (cooling period)

$$= \frac{(371 - 115) - (150 - 50)}{2.3 \log \frac{371 - 115}{150 - 50}}$$

$$= 166^\circ\text{F}$$

Take condenser coil, 200 feet long of 2 inch pipe overall cool area = 124 sq. feet

Assuming 0.25 of cooling area for condensation/area for condensation = 31 sq. ft.

then Mean temperature difference = 239°F

Heat to be removed = 54,760 Bth.Us.

Hence the overall heat transfer coefficient necessary = 8 approx.

Area for cooling = 93 sq. feet

Mean temperature difference = 166°F

Heat to be removed = 104,063 Bth.Us.

Necessary overall heat transfer coefficient = 7 approx.

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These temporary maxima should be attainable with suitable adjustment of the cooling water velocity.

If the converter is used as a drier, and it is desired to condense the steam, assuming a charge of 2.5 tons of wet concentrate containing 1 ton of water, approximately 250 feet of condenser pipe would be required with an overall coefficient of 35 for the condensing period and 24 for the cooling, if the concentrate were dried in five hours.

There is no need for making provision to condense all the steam unless to obtain a check on the moisture content of the charge. If the system were overloaded at any time owing to over rapid drying in the test unit, the steam may be partially blown to waste at the headers.

#### GENERAL CONTROL:

A weight balance on the concentrate charged and the products obtained will be kept. Gas volume will be measured by orifice and differential gauge installed in a suitable length of the return gas line to the furnace.

Suitably protected pyrometers will be placed close to the metal shell of the converter at each end of the heating flue, and at the base of the chimney. The temperature of the charge may be obtained by placing a pyrometer in a pocket welded to one end of the converter, as shown in the sketch. Vapour temperatures can be obtained by placing thermometers in suitable pockets welded to the top of the vapour offtakes.

Provision should be made in the setting for taking flue gas samples, if possible both before and after admixture of cooling air, and for sight holes and cleaning access to flues.

Provision should be made for the cleaning of offtake pipes and manifolds with the minimum of labour.

#### SUMMARY AND CONCLUSIONS:

It has been shown that an organic concentrate prepared from Tasmanite Shale can be converted under practicable conditions to an asphalt acceptable to the Public Works Department with a bye product yield of useful oil.

A general scheme for the daily conversion of 15 tons of concentrate has been outlined, and a general description of the full scale unit proposed to incorporate in the plant given. Since the choice of the most suitable plant is a debatable matter, qualified engineering opinion on the suggestions made in this report should be obtained before further expenditure on plant construction for the conversion stage is undertaken.

If the suggestions made are accepted as a working basis for continuing the investigation, a single full scale unit will be erected on the shale field, after constructional details are worked out in consultation with manufacturers.

Data from tests with this unit will largely determine the plant required for handling 15 tons per day, and will furnish useful information on the probable capital and operating costs of such a plant.

Considerable improvement on the figures used in this report will be necessary to bring the operating cost down to a reasonable figure. The figures used indicate an operating cost for drying and conversion of 18/6 per ton of concentrate or 4/6 per ton of shale.

A. WALKER, August, 1937.