

amdel

81-1608

5-10-81 882001

ASPHALT FROM TASMANITE OIL SHALE
STAGE II

Endeavour Resources Limited

PR No. 1

OD 3/495/2/3

September 1981

81-1608.



The Australian
Mineral Development
Laboratories

Remington Street, Frewville,
South Australia 5063
Phone Adelaide 79 1662
Telex AA 82520

Please address all
correspondence to
P.O. Box 114 Eastwood
SA 5063
In reply quote:

amdel

7 September 1981

OD 3/495/2/3

The Exploration Manager
Endeavour Resources Limited
PO Box 217
EAST MELBOURNE Vic 3002

ASPHALT FROM TASMANITE OIL SHALE - STAGE II

Progress Report No. 1

Investigation and Report by: R.J. Allen and B.H.J. Waters

Manager, Operations Division: Bruce E. Ashton

for Norton Jackson,
Managing Director

Pilot Plant: Osman Place
Thebarton S.A.
Telephone 43 8053

Branch Laboratories:
Perth W.A.
Telephone 325 7311
Melbourne Vic.
Telephone 645 3093

rp

1. INTRODUCTION

In Stage I of this investigation, about 10 kg of tasmanite concentrate (ash content 17.0%) were prepared by grinding and re-floating a batch of 1937 concentrate (ash content 31.8%) from the Tasmanian Department of Mines.

A review of early work on the conversion of tasmanite concentrate to asphalt left some areas of uncertainty concerning the detailed operating conditions for the conversion process. It was recommended that the conversion process, comprising stage heating of the dried concentrate to about 400°C in the absence of air, should be investigated on a relatively small scale (say 0.5 kg) in the first instance; the bituminous products obtained under a range of conditions should be evaluated by means of a few standard tests.

A programme of experimental work along these lines was proposed in AMDEL's letter of 3 February 1981. This work was approved by Endeavour Resources Limited in a letter dated 24 March 1981.

2. EXPERIMENTAL PROCEDURE AND RESULTS

2.1 Thermal Analysis of Tasmanite Concentrate

A sample of tasmanite bulk concentrate No. 1 (21% ash, 35 mg) was heated in the Rigaku Denki Model M8076 thermal analysis instrument, which permits the simultaneous recording of a DTA curve (heat changes in the sample) and a TGA curve (weight changes in the sample). The traces recorded are reproduced in Fig. 1.

Below about 370°C, the rate of weight loss was quite slow, but between 400°C and 500°C, nearly 60% of the sample weight was lost, due to endothermic decomposition to yield a high-carbon char (i.e. pyrolysis reactions).

2.2 Laboratory Pugmixer Tests

An existing laboratory pugmixer was adapted for use as a digester for thermal conversion of the tasmanite concentrate to asphalt. The mixing compartment (tilted for ease of viewing in Fig. 2), which has two counter-rotating mixer-blades and electrically-heated walls, was fitted with a new steel lid; the lid has a nitrogen gas inlet and an exhaust gas outlet, a thermocouple well, plus an inspection port with a sliding cover.

The mixer was charged with 418 g of the bulk tasmanite concentrate No. 1 (21% ash), and the temperature was progressively increased to 250 ± 10°C, the maximum operating temperature of the mixer. The charge was stirred continuously throughout, for a total of 12 hours at 250°C.

During this time some water and a small amount of tar were lost, but the physical condition of the powdery tasmanite concentrate was unchanged, except for a change in colour from a yellow ochre to a very dark brown.

2.3 Pot Furnace Tests

Because the temperature attainable with the pugmixer was evidently not high enough, a new reaction vessel was designed and made, to suite an existing pot furnace. The arrangement as ultimately developed, is shown schematically in Fig. 3. A stainless steel beaker containing the tasmanite is heated to a controlled temperature by the pot furnace. The beaker has a close-fitting lid which has an inlet tube for nitrogen gas, a thermocouple well, and a gap for escape of exhaust gases around the stirrer shaft. The exhaust gases are vented to the atmosphere via a small 'chimney' which allows for further

dilution with nitrogen; finally the emerging gases are drawn off by a vacuum fume extraction system.

The charge in the reaction vessel is continuously stirred at 8 rpm by the high-torque agitator which has a spiral-type impeller blade.

2.3.1 Test I

Using this reactor, 394 g of the tasmanite concentrate (previously held at 250°C for 12 hours) was heated to 300°C in nitrogen, and this temperature held for 1.5 hours. There was no evident change in the physical condition of the tasmanite, which was therefore brought to about 390°C for 0.5 hours. Examination of the product (330 g) revealed some blackening near the top of the reactor, and a small amount of quite hard, seemingly fused material adhering to parts of the walls and agitator. Microscopic examination of the hard material revealed that its surface was covered with globules that had apparently fused.

Further heating at about 360°C for 1.0 hours resulted in strong fuming, but examination of the product revealed little change. The product was therefore reheated at about 390°C for 1.5 hours, which resulted in complete fusion, and a great reduction in the volume. The cooled product had a flat, shiny black surface.

More tasmanite concentrate was added, and the combined charge heated to about 390°C for 40 minutes. This appeared to be insufficient to fuse the additional material, and the charge was again heated to 390°C for 40 minutes, yielding a product which was entirely fused. However, this product could not be re-melted by heating at 150°C in nitrogen on an oil bath.

In an attempt to lower the softening point of the asphalt product, it was heated again for 30 minutes at 390°C in nitrogen in the pot furnace, but the product still could not be re-melted at 150°C on the oil bath.

Finally the asphalt was heated for a further 2.5 hours at 390°C in the pot furnace, yielding a product which appeared to be highly carbonised, and which could not be remelted. This final product was collected and weighed revealing that only 47% of the original weight of concentrate remained.

2.3.2 Test II

Three hundred grams of the bulk concentrate No. 2 (17% ash) was charged in the stainless steel beaker, and the temperature progressively increased to 390°C and held for 2.5 hours with stirring under nitrogen. After cooling the reactor was opened and the product was found to be a dark, charcoal grey bulky power, no fusion being evident.

Reheating to 400°C for 3.0 hours yielded a product which had clearly been completely fused. Upon reheating on the oil bath to 150°C the asphalt product was successfully remelted.

Samples of the melt were transferred to brass rings for softening point determination, and to a pycnometer flask, for specific gravity determination.

The measured softening point was 62°C, and the specific gravity was 1.15.

3. DISCUSSION

The maximum temperature attainable with the pugmixer, viz 250°C to 260°C, was clearly inadequate for the conversion of tasmanite to asphalt.

Furthermore, there was no sign of fusion in the material subsequently heated at 360°C by the pot furnace. By contrast, the conversion took place relatively rapidly at 390°C. It is noteworthy that this temperature is close to the threshold of rapid pyrolytic decomposition as shown by thermal analysis (Fig. 1). After 30 minutes fusion had commenced, and after 2 hours at 390°C the charge had completely melted. Addition of more tasmanite, and heating at 390°C for a total of about 3.5 hours ultimately yielded a highly carbonised product which had presumably been overheated and excessively pyrolysed.

In Test II, the tasmanite concentrate was brought to 390°C to 400°C relatively rapidly. After a total of 5.5 hours at this temperature, the asphalt product was a shiny black, highly fluid material which was readily remelted on the oil bath at 150°C. The measured specific gravity (1.15) was very close to that specified in the 1967 Australian standard* for native asphalts. The measured softening point was 62°C, a little higher than the range (40°C to 51°C) specified in the 1967 Australian standard. It should be noted that the latest Australian standard (AS2008-1980) does not include specifications for native asphalt, presumably because these have not been imported for roadmaking in recent years.

*ASA10-1967, Class N90.

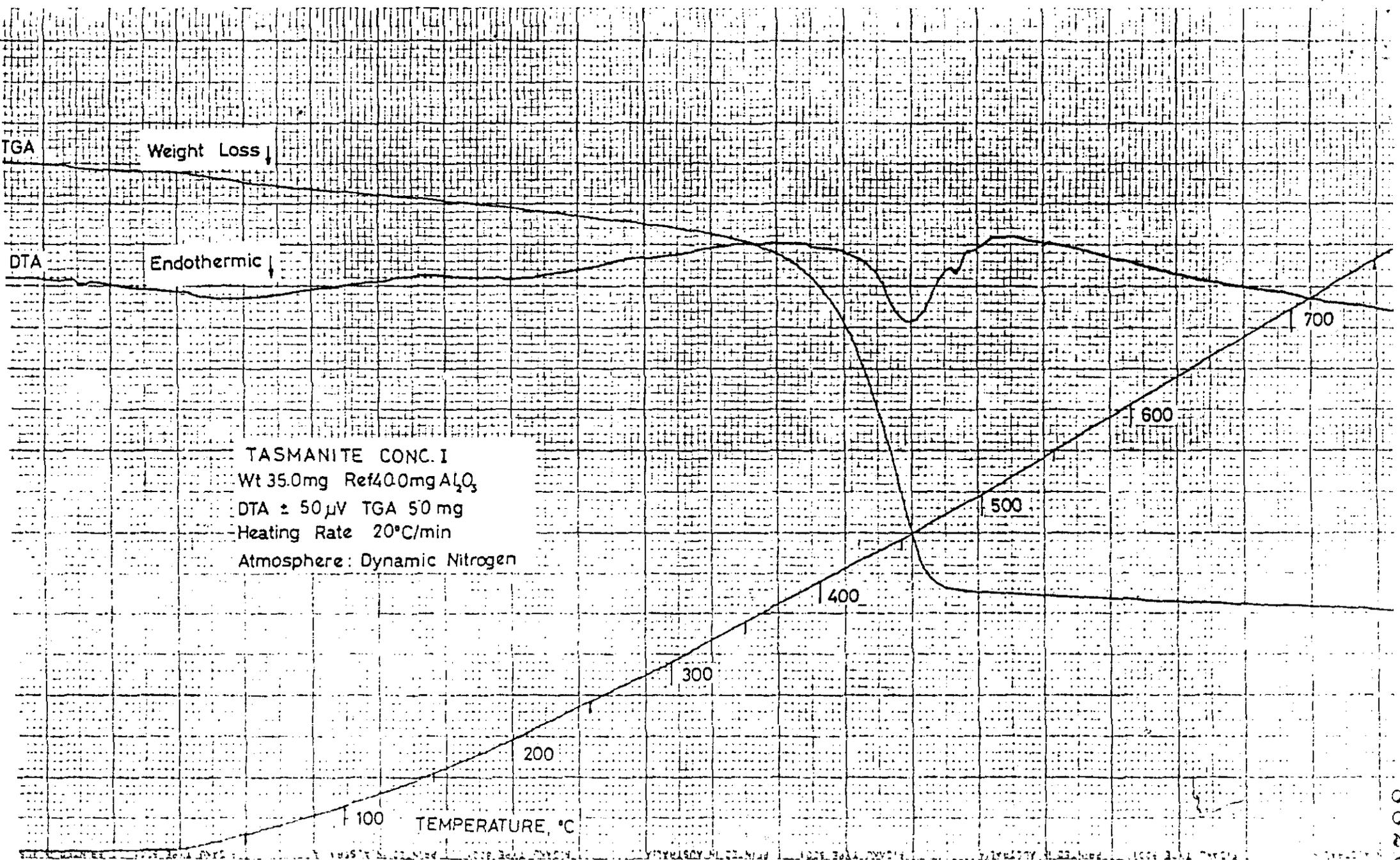


FIG. 1: THERMAL ANALYSIS OF TASMANITE CONCENTRATE IN NITROGEN

882006



FIG. 2: LABORATORY PUGMIXER

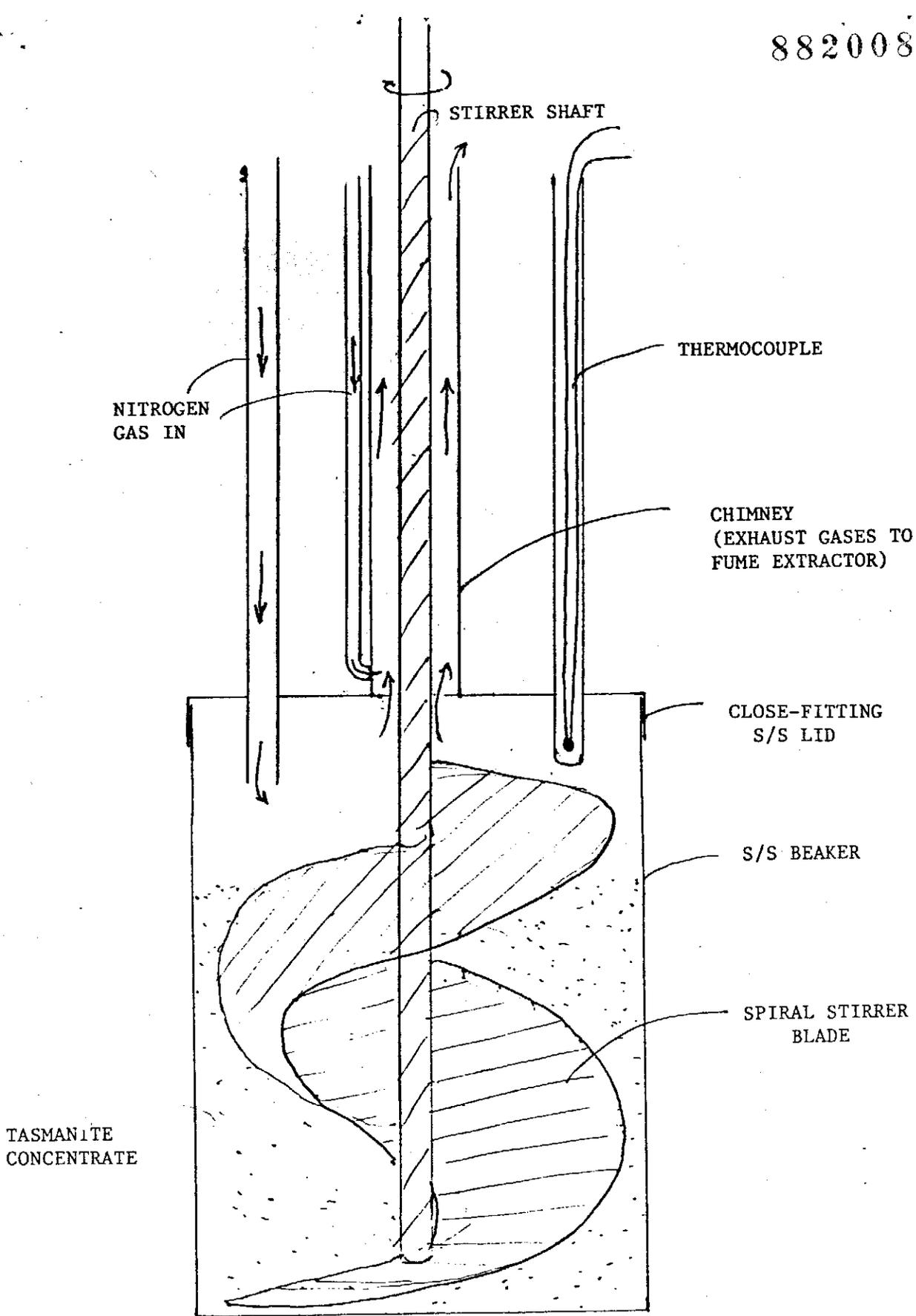


FIG. 3: STAINLESS STEEL REACTOR FOR CONVERSION OF TASMANITE TO ASPHALT