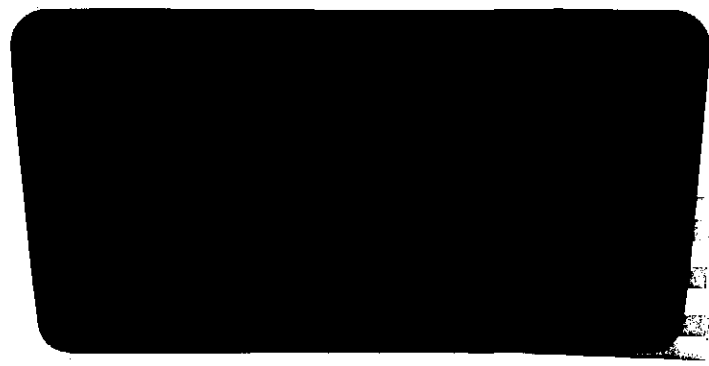


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ENDEAVOUR RESOURCES LIMITED

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No. 1433

ASPHALT FROM TASMANITE OIL SHALE
STAGE II

OPEN FILE

by

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SUMMARY

Background

In Stage I of this investigation, about 10 kg of tasmanite concentrate (ash content 17.0%) was prepared by grinding and refloating a batch of concentrate (ash content 31.8%) originally prepared in 1937 by the Tasmanian Department of Mines.

On the basis of a review of early work, it was recommended that the thermal conversion of the tasmanite concentrate to asphalt should be investigated on a small scale in the first instance; the bituminous products so obtained should be evaluated by means of a few standard tests.

A programme of experimental work along these lines was approved by Endeavour Resources Limited in a letter dated 24 March 1981.

Summary of Work Done

The tasmanite concentrate prepared in Stage I was progressively heated to $250 \pm 10^\circ\text{C}$ in a laboratory pugmixer which had provision for efficient stirring of the charge under a nitrogen atmosphere. There was little apparent change in the concentrate after a total of 12 hours at temperature.

A specially designed stainless steel reactor, again providing a nitrogen atmosphere and thorough stirring of the charge, was used to heat the tasmanite concentrate to higher temperatures. Ultimately, at about 390°C , fusion of the charge commenced, and after around four hours at temperature a bituminous product was obtained. Determinations of specific gravity, softening point and solubility in toluene confirmed that the product was similar in properties to native asphalt.

Conclusions

By heating the tasmanite concentrate in the absence of air in a stirred reactor, and finally holding at a temperature of 390°C for about 4 hours, a bituminous product is obtained, comparable in properties to native asphalt.

From this investigation, the temperature of 390°C appears to be necessary to achieve a reasonable rate of conversion to asphalt.

Recommendations

To permit more extensive testing and evaluation of the asphalt product, a larger batch should be prepared from the tasmanite concentrate on hand at AMDEL.

It is recommended that the detailed evaluation of the product should be carried out by a specialised laboratory, such as those of the South Australian Highways Department, or the Australian Road Research Board.

1. INTRODUCTION

In Stage I of this investigation, about 10 kg of tasmanite concentrate (ash content 17.0%) was prepared by grinding and re-floating a batch of concentrate (ash content 31.8%) originally produced in 1937 by the Tasmanian Department of Mines (Allen and Wong, 1981)*.

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, involving 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.

*ALLEN, R.J., and WONG, K.Y. (1981). 'Asphalt from Tasmanite Oil Shale - Stage I'. AMDEL Report No. 1385, April.

2. MATERIAL EXAMINED

Two samples of tasmanite concentrate were used for this testwork:

Bulk Concentrate No. 1

As described in AMDEL Report No. 1385 (Allen and Wong, 1981), this was produced by grinding the 1937 concentrate and re-floating, employing a rougher stage and two cleaning stages. This concentrate was found to have a residual ash content of 21.2%.

Bulk Concentrate No. 2

This was prepared from bulk concentrate No. 1 by conducting another two cleaning stages, giving a residual ash content of 17.0% (Allen and Wong, 1981)*.

*ALLEN, R.J., and WONG, K.Y. (1981). 'Asphalt from Tasmanite Oil Shale - Stage I'. AMDEL Report No. 1385, April.

3. EXPERIMENTAL PROCEDURE AND RESULTS

3.1 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).

3.2 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.

3.3 Pot Furnace Tests

Because the temperature attainable with the pugmixer was evidently not high enough, a new reaction vessel was designed and made, to suit an existing pot furnace. The arrangement as ultimately developed, is shown schematically in Fig. 3. A stainless steel baker containing the tasmanite was heated to a controlled temperature by the pot furnace. The beaker had a close-fitting lid which had an inlet tube for nitrogen gas, a thermocouple well, and a gap for escape of exhaust gases around the stirrer shaft. The exhaust gases were vented to the atmosphere via a small 'chimney' which allowed for further dilution with nitrogen; finally the emerging gases were drawn off by a vacuum fume extraction system.

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

3.3.1 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 at 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 re-melted. This final product was collected and weighed revealing that only 47% of the original weight of concentrate remained.

3.3.2 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 re-melted.

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.

A determination was made of the solubility of the asphalt product in toluene by the Australian standard (AS 23418) method. The percentage of the product insoluble in toluene was 23.3%.

3.3.3 Test III

A 300 g sample of bulk concentrate No. 2 (17% ash) was charged into the stainless steel beaker, and the temperature increased progressively to 390 ±10°C, and this temperature held for a total of 4 hours (i.e. 1.5 hours less than in Test II). The total time taken for heating up to 380°C was 6.5 hours, comparable to Test II.

After cooling to room temperature, the product was a shiny black solid material that, clearly had fused during the heating. The yield of product asphalt from Test III was 65% of the original dry concentrate. However, a determination showed that the softening point of this Test III product was 65°C, or 3 degrees higher than the Test II product.

Because a lower softening point (~50°C) was sought, the Test III product was reheated under nitrogen in the pot furnace, and held at 390 ±10°C for an additional 45 minutes, and then cooled. The softening point of this reheated product was 87°C.

4. DISCUSSION

The maximum temperature attainable with the pug mixer, viz 250°C to 260°C, was clearly inadequate to convert tasmanite concentrate to asphalt.

Furthermore, there was no sign of fusion when the pug mixer product was subsequently heated at 360°C in the pot furnace. By contrast, the conversion took place quite rapidly at 390°C which is close to the threshold of rapid pyrolytic decomposition, as shown by thermal analysis (Fig. 1). After 30 minutes at 390°C, fusion had commenced, and after 2 hours, the charge had completely melted. However, when more tasmanite concentrate was added, and the combined charge heated at 390°C for a total of about 3.5 hours, a highly carbonised product was obtained which presumably, had 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. After cooling, it was readily re-melted 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 (AS 2008-1980) does not include specifications for native asphalt, presumably because these have not been imported for roadmaking in recent years.

The toluene-insoluble matter (23.3%) would be fully accounted for by the ash content, indicating that the bitumen content is almost entirely soluble in toluene.

The initial product from Test III was very similar to that of Test II, judging from its appearance and softening point. However, further heating at 390°C did not have the desired effect of lowering the softening point of the asphalt; in fact, the reverse occurred. Clearly, the detailed time-temperature conditions are quite critical if overheating of the asphalt product is to be avoided.

*ASA10-1967, Class N90.

5. CONCLUSIONS

By heating the tasmanite concentrate in the absence of air in a stirred reactor, progressively increasing the temperature to 390°C, and holding this temperature for about 4 hours, a bituminous product is obtained. This product is highly soluble in toluene, and has a specific gravity and softening point comparable to native asphalt as specified in an Australian standard.

From this investigation, the temperature of 390 (± 10)°C appears to be necessary to achieve a reasonable rate of conversion to asphalt.

6. RECOMMENDATIONS

To permit more extensive testing and evaluation of the asphalt product, a larger batch should be prepared from the tasmanite concentrate on hand at AMDEL. The conditions of time and temperature established in this study should be used as a guide, and the progress of the conversion should be followed by determining the softening point of samples taken at intervals from the reactor.

It is recommended that the detailed evaluation of the product should be carried out by a specialised laboratory such as those of the South Australian Highways Department or the Australian Road Research Board.

7. ACKNOWLEDGEMENT

The author wishes to thank Mr Trevor Clarke of the South Australian Highways Department for guidance on the testing of asphalt.

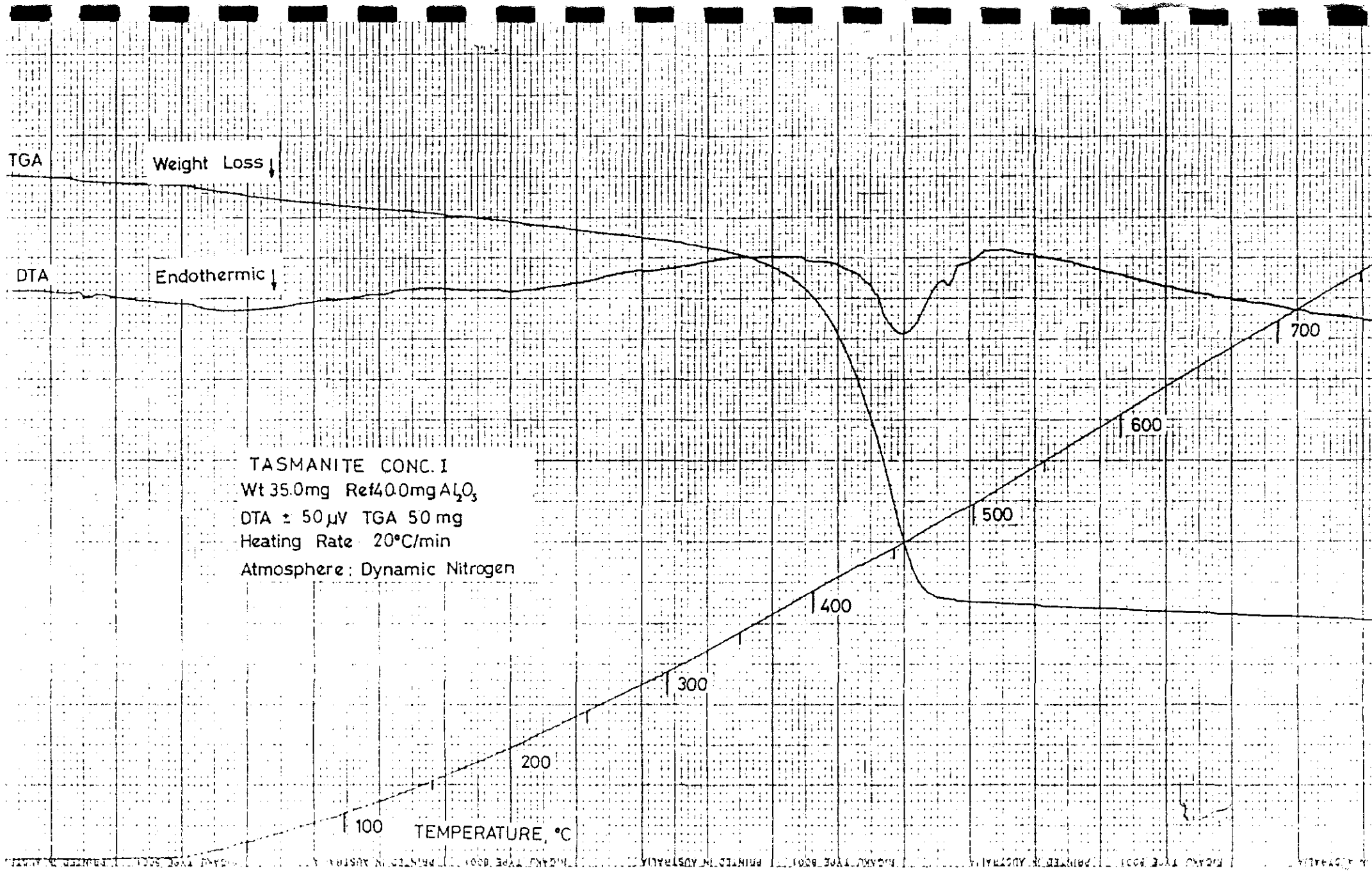


FIG. 1: THERMAL ANALYSIS OF TASMANITE CONCENTRATE IN NITROGEN

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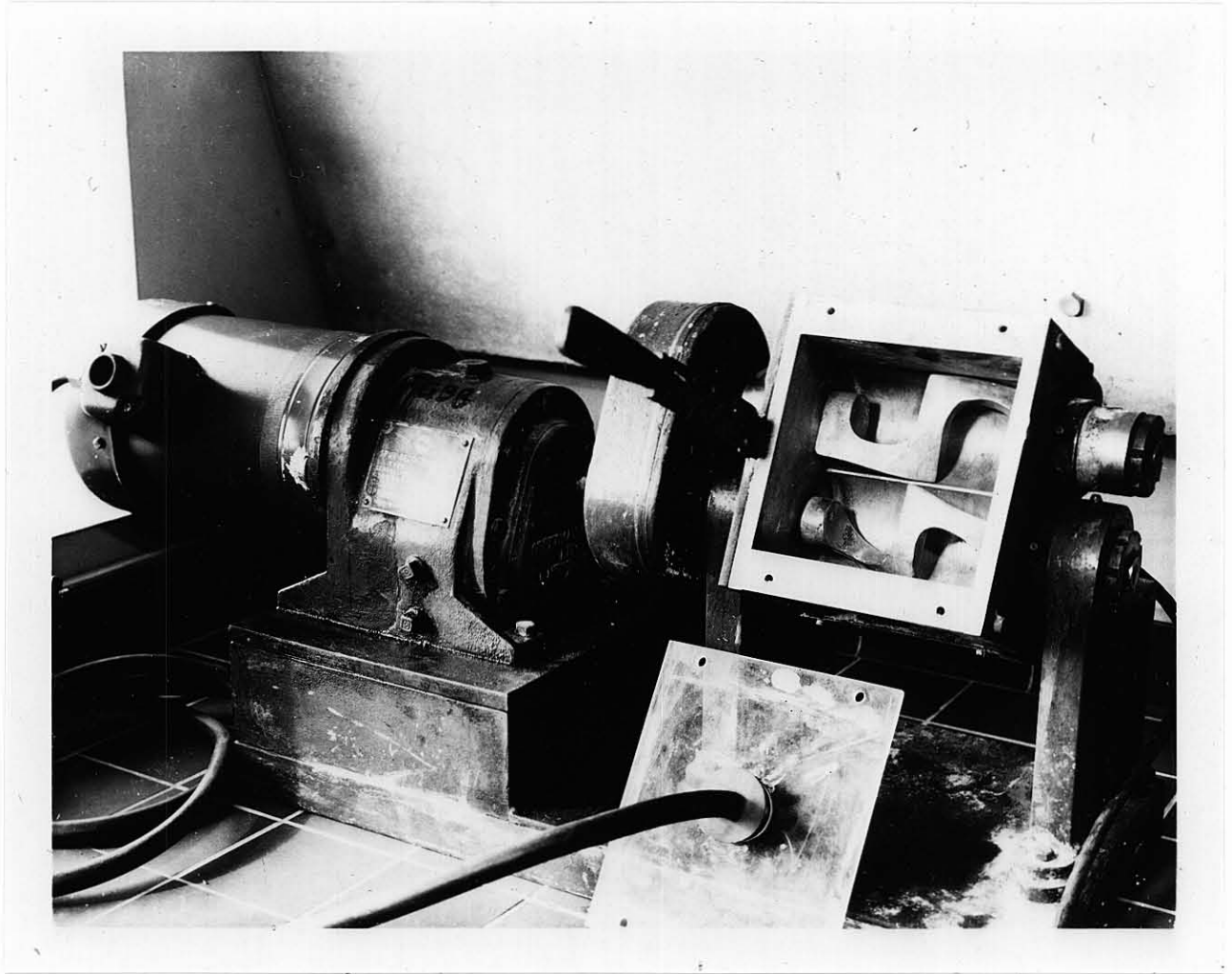


FIG. 2: LABORATORY PUGMIXER

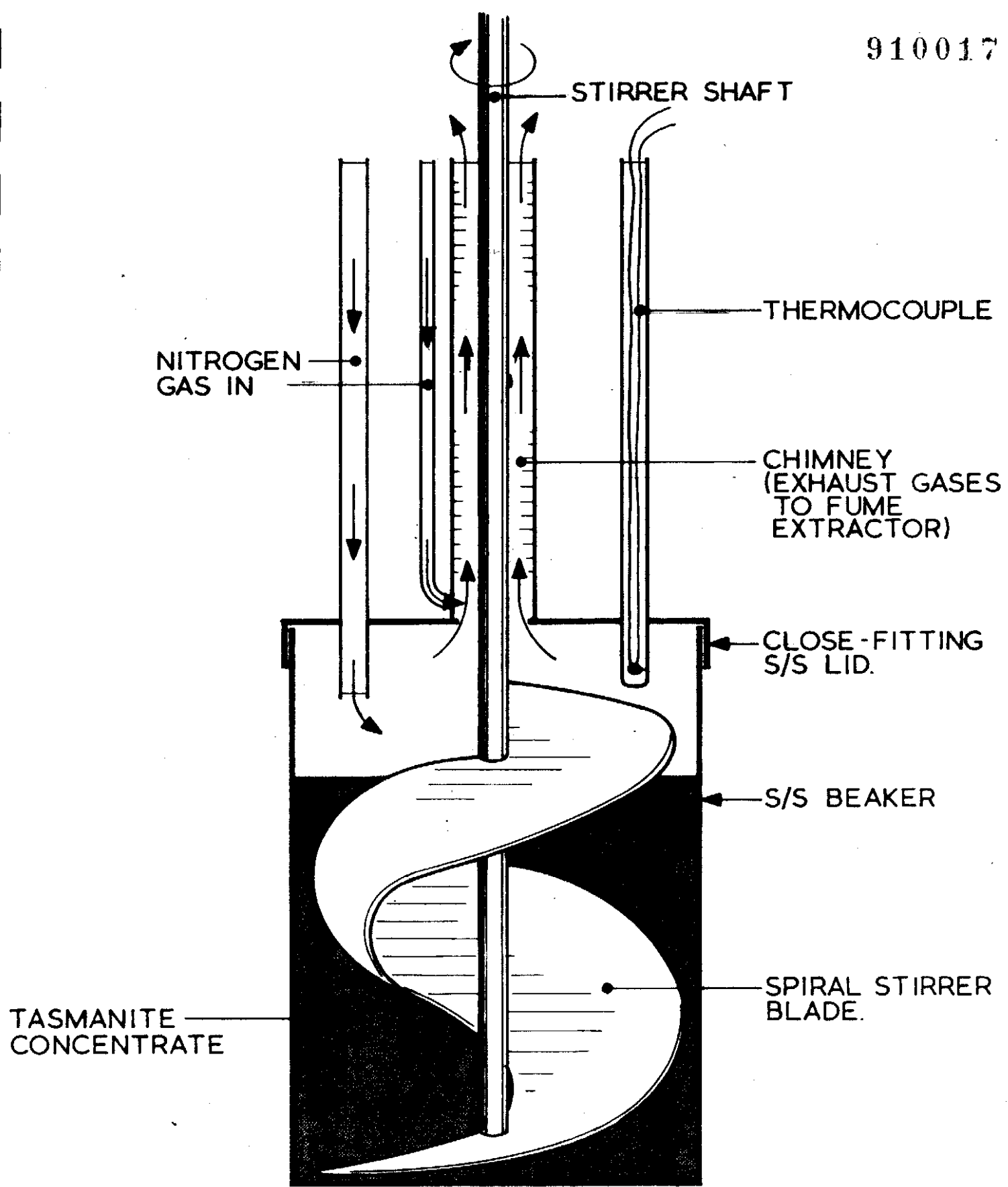


FIG. 3. STAINLESS STEEL REACTOR FOR CONVERSION OF TASMANITE TO ASPHALT.