#### DEPARTMENT OF MINES.

South Australia.

#### -RESEARCH AND DEVELOPMENT BRANCH-

REPORT No. R.D. 66.

#### HEAVY SANDS INVESTIGATION.

THE RECOVERY OF MONAZITE. RUTILE AND ZIRCON FROM MOANA BEACH SAND.

bу

B.E. Ashton. C.R. Sandman. and L.J. Weir.

MICROFILLMEN

DATE: July, 1957.

Copy No 21 of 25 copies.

This document consists of 16 pages, and 3 flowsheets.

CONFIDENTIAL.

### HEAVY SANDS INVESTIGATION.

# THE RECOVERY OF MONAZITE, RUTILE AND ZIRCON FROM MOANA BEACH SAND.

### -Contents-

|    |                                 | Page. |
|----|---------------------------------|-------|
|    | Abstract                        | 1.    |
| ı. | Summary                         | 1.    |
| 2. | Introduction                    | 2.    |
| 3. | Material Examined               | 2.    |
| 4. | Equipment                       | 3.    |
| 5• | Procedure                       | . 3.  |
| 6. | Operating Conditions & Results  | 4.    |
| 7. | Discussion of Results           | 11.   |
| 8: | Observations and Conclusions    | 14.   |
|    | Appendices: Flowsheet No. 1 2 3 | 16.   |

#### HEAVY SANDS INVESTIGATION.

# THE RECOVERY OF MONAZITE. RUTILE AND ZIRCON FROM MOANA BEACH SAND.

by.

B.E. Ashton.
C.R. Sandman.
and
L.J. Weir.

#### -Abstract-

A concentrate of heavy minerals was made from the beach sand deposit at Moana, South Australia. This concentrate was treated by magnetic and electrostatic methods to recover monazite, rutile and zircon. The valuable mineral product was in each case below marketable grade. The original sand contained 0.9 percent rutile, 1.4 percent zircon and 0.1 percent monazite. The recoveries were 51, 43 and 65 percent respectively.

#### 1. SUMMARY.

The minerals monazite, rutile and zircon occur in a beach sand deposit at Moana, 23 miles south of Adelaide. Laboratory tests to extract saleable concentrates of these three minerals have been made. Since monazite contains the strategic radioactive element thorium, greater emphasis was placed on the recovery of monazite than of the other two minerals.

The treatment adopted consisted of three basic forms of mineral separation.

- 1. Gravity separation.
- 2. Magnetic Separation.
- 3. Electrostatic Separation.

Tests were conducted in a manner approximating to plant practice.

The recoveries of monazite rutile and zircon were 67,
51 and 43 percent respectively. Because overlapping characteristics of the three important minerals prevented clean separation,
concentrates of market specifications were not produced.

#### 2. INTRODUCTION.

Concentrations of heavy minerals are known to occur at various points along the South Australian coast. The valuable mineral constituents of these beach sand deposits are rutile, zircon and monazite. Many other heavy minerals occur associated with them, and consist mainly of miscellaneous heavy silicate and iron oxide minerals.

Various accepted treatment methods are used to effect a separation of rutile, zircon and monazite, but the efficiency of separation is dependant on the characteristics of the minerals present which, for different deposits, are not necessarily constant. Hence a treatment method designed for one beach sand deposit may not be suitable for another deposit even though the sands contain the same economic minerals.

#### 3. MATERIAL EXAMINED.

The beach sand used for this investigation was obtained from the sand dunes at Moana. The sample was collected in collaboration with Mr. A. Black, consultant to South Australian Rutile Limited. It was approximately five tons in weight and was considered to be representative of the whole dune area, excluding the higher grade beach deposit.

The material was used since it was the only bulk heavy mineral beach sand sample readily available for large scale investigation of the recovery of thorium bearing and other valuable minerals.

#### 4. EQUIPMENT USED.

The following equipment was used in the investigation:

- (a) Humphrey Spiral (5 turn).
- (b) High tension electrostatic separator.
- (c) Induced roll magnetic separator.
- (d) Wilfley table.
  - (e) Stearns single disc magnetic separator.

#### 5. PROCEDURE.

#### 5.1 Initial Gravity Concentration.

For this work a 5 turn Humphrey spiral unit was used to produce a concentrate containing all of the valuable heavy minerals. Due to the wide range of mineral specific gravities it was found necessary to adopt multi-stage treatment of the various tailing and middling fractions.

The method finally adopted is shown in Flowsheet 1.

There was insufficient sample available to carry out this gravity concentration stage in cyclic fashion.

Prior to gravity concentration it was found necessary to screen the bulk sample on a 20 mesh screen to remove extraneous matter. The oversize fraction which represented less than 0.5 percent by weight of the total sample consisted mainly of organic matter, large pebbles and shell fragments.

#### 5.2 Production of valuable mineral concentrates.

For this stage the dried gravity concentrate was used as feed material. Previous experience with beach sand material of this type had shown that a complex method of treatment was required, involving gravity, magnetic, and high tension forms of electrostatic separation. Since in such a method appreciable amounts of material report in the

intermediate fractions, the investigation was carried out in cyclic manner. The aim of a cyclic test is to allow the intermediate fractions to reach equilibrium with regard to both weight and composition i.e., when the weight and composition of each of these fractions becomes constant. The cyclic test was carried out as follows:— the gravity concentrate was divided into six equal portions each forming the basis of a cycle. One of these portions was then treated in the manner shown in Flowsheet 2 as the first cycle.

This flowsheet produces a number of final products both valuable and reject in nature and also other products which have been considered as intermediate or "middling" fractions. These middling products were returned to the next cycle in the positions considered most appropriate. This procedure was repeated until the six cycles had been completed.

- 6. OPERATIONS CONDITIONS and RESULTS.
- 6.1 Operating Conditions.
- 6.1.1 Spiral Concentration.

The operating conditions for the various stages of spiral concentration are shown in Table 1.

#### TABLE 1.

Humphrey Spiral Operating Conditions.

Feed rate.
Feed pulp density.
Concentrate pulp density.
Wash water flow rate.

0.7 tons of dry solids/hour. 23 percent solids. 50 " " 440 gallons/hour.

#### 6.1.2 Electrostatic Separation.

The conditions for each stage of high tension separation are given in Table 2.

#### TABLE 2.

#### High Tension Separator Operating Conditions.

Feed\_rate.
Roll peripheral speed.
Potential.

Polarity.

Feed temperature.

25 lbs/inch width/hour.
370 feet/minute.
20-30 kilovolts depending on nature of feed.
Rotating roll earthed.
High tension electrode negative.
300 F.

#### 6.1.3 Magnetic Separation.

The operating conditions for each stage of magnetic separation are given in Table 3.

#### TABLE 3.

#### Magnetic Separator Operating Conditions.

Feed rate.
Roll peripheral speed.
Coil current.

30 lbs/inch width/hour.
120 feet/minute.
0.5 - 1.5 amps depending on nature of feed.

#### 6.1.4 Table Concentration.

The conditions under which the Wilfley table was operated for the various tabling stages are given in Table 4.

# TABLE 4. Wilfley Table Operating Conditions.

Foed rate.
Table speed.
Table stroke length.
Water feed rate.

70 lbs of dry solid/hour. 250 strokes/minute. 0.5 inches. 80 gallons/hour.

#### 6.2 Results.

#### 6.2.1 Results of Initial Gravity Concentration.

The percentage weight of each fraction produced during the gravity concentration stage is shown in Flowsheet 1.

Since rutile has the lowest specific gravity of the three valuable minerals present in the sand (monazite, zircon and rutile) the final spiral products, consisting of concentrate middling and tailing fractions, were assayed chemically for TiO<sub>2</sub>. This served as a measure of gravity concentration efficiency.

The weight percentage and TiO<sub>2</sub> content of each of the final spiral fractions are given in Table 5.

TABLE 5.

Results of Humphrey Spiral Gravity Concentrate.

| Fraction.                       | Percent<br>Weight of<br>Original<br>Feed. | TiO <sub>2</sub><br>Percent. | Distrib.<br>of TiO <sub>2</sub><br>Percent. |
|---------------------------------|---|------------------------------|---|
| Concentrate. Middling. Tailing. | 19.4<br>27.2<br>53.4                      | 10.1<br>1.18<br>0.27         | 81.3<br>A2.9<br>5.8                         |
| FEED.                           | 100.0                                     | 2.4                          | 100.0                                       |

The mineralogical compositions of the above three final fractions were determined and are shown in Table 6.

TABLE 6.

Mineralogical Composition of Humphrey Spiral Fractions.

| Fraction.   | Feed.             | Concentrate.                          | Middling.           | Tailing.    |
|---|-------------------|---------------------------------------|---------------------|-------------|
| Percent Weight of Spiral Feed.                        | 100.0             | 19.4                                  | 27.2                | 53•4        |
| Percent Weight of:                                    |                   |                                       |                     |             |
| Haematite.<br>Limonite.<br>Ilmenite.<br>Magnetite.    | 8.7               | ( 14.4)<br>( 8.1)<br>( 1.6)<br>( 0.8) | 8.6                 | 3.0         |
| Total rutile. Non-opaque. Ferruginous.                | (2.2)<br>0.9      | ( 10.8)<br>4.5                        | (0.3)<br>0.1        | (trace)     |
| Zircon.<br>Monazite.                                  | 1.3<br>1.4<br>0.1 | 6.3<br>6.8<br>0.7                     | 0.2<br>0.4<br>trace | trace       |
| Miscellaneous Heavy<br>Silicates.<br>Quartz, Felspar. | 11.6<br>76.0      | 43.2<br>13.6                          | 7•3<br>83•4         | 2.4<br>94.6 |
|   | 100.0             | 100.0                                 | 100.0               | 100.0       |

The results shown in Tables 5 and 6 indicate that a good recovery of the valuable constituents, monazite rutile and zircon, was achieved in the gravity concentrate. Although the middling fraction represented a considerable percentage weight of the original spiral feed, the valuable mineral content was low suggesting that this fraction could be considered as a tailing.

## 6.2.2 Results of Production of Valuable Mineral Concentrates.

As was stated in section 5.2 a six cycle locked-batch test was carried out based on Flowsheet 2.

The percentage weight of each of the final products from the last cycle is shown in Table 7. The final fractions are expressed as a percentage of feed to the gravity concentration sections.

TABLE 7.

# Percentage Weight of Final Products, and Based on Feed to Gravity Concentration Section.

| Product. (Identification as per Flowsheet 2).  | Percent Weight.                                      |
|--|--|
| 17 18 (Zircon concentrate). 22 23 (Monazite concentrate). 24 34 40 (Rutile concentrate). | 3.53<br>0.61<br>7.70<br>0.33<br>0.22<br>4.37<br>0.51 |
| Gravity Concentrate equivalent.  | 17.27  |

The summation of the final products gives 17.27 percent but for equilibrium to be established this figure should be 19.40 percent. This indicates that all the new feed was reporting in the final products, and equilibrium conditions were not quite attained. This difference is discussed later in Section 7.

These final products were subjected to mineralogical examination and in addition, the three concentrate products were assayed chemically.

The results of the mineralogical and chemical analyses are given in Tables 8 and 9 respectively.

TABLE 8.

Mineralogical Composition of Final Products.

| Product Numbers. |  |   |  |   |   |  |  |
|------------------|--|---|--|---|---|--|--|
| 17.              | 18.  | 22.   | 23.  | 24.   | 34•   | 40.  |  |
| 0.2              | <b>-</b> .   | 3.0   | 9.1  | 61.5  | 57.3  | 2.1  |  |
| 0,2              | -  | 14,2  | tr.  | 8.5   | 17.9  | 0.9  |  |
| tr.              | -  | tr.   | 1.1  | 3.1   | 6.6   | ••   |  |
| 0.2              | -  | 0.8   | tr.  | 2.2   | 1.1   | tr.  |  |
| (tr.)            | (0.9)  | (3.8)   | (10.1)   | (3.9)   | (2.9)   | (89.7)   |  |
| tr.              | 9.0  | 1.0   | 5.2  | 1.0   | -   | 79.8   |  |
| -                | _  | 2.8   | 4.9  | 2.9   | 2.9   | 9.9  |  |
| tr.              | 86.4   | -   | 12.3   | 1.1   |   | 2.6  |  |
| tr.              | 3.9  | -   | 25.2   | 2.2   | -   |  |  |
| 9•3              | 0.7  | 78.2  | 42.2   | 17.5  | 13.5  | 4.7  |  |
| 90.1             | •••  | •••   | -  | -   | 0.7   | ***  |  |
| 100.0            | 100.0  | 100.0   | 100.0  |   |   | 100.0  |  |
|                  | 0.2<br>0.2<br>tr.<br>0.2<br>(tr.)<br>tr.<br>tr.<br>9.3<br>90.1 | 0.2 -  tr  0.2 -  (tr.) (0.9)  tr. 9.0  -  tr. 86.4  tr. 3.9  9.3 0.7  90.1 - | 0.2 - 3.0  0.2 - 14.2  tr tr.  0.2 - 0.8  (tr.) (0.9) (3.8)  tr. 9.0 1.0  - 2.8  tr. 86.4 -  tr. 3.9 -  9.3 0.7 78.2  90.1 | 17. 18. 22. 23.  0.2 - 3.0 9.1  0.2 - 14.2 tr.  tr tr. 1.1  0.2 - 0.8 tr.  (tr.) (0.9) (3.8) (10.1)  tr. 9.0 1.0 5.2  - 2.8 4.9  tr. 86.4 - 12.3  tr. 3.9 - 25.2  9.3 0.7 78.2 42.2  90.1 | 17. 18. 22. 23. 24.  0.2 - 3.0 9.1 61.5  0.2 - 14.2 tr. 8.5  tr tr. 1.1 3.1  0.2 - 0.8 tr. 2.2  (tr.) (0.9) (3.8) (10.1) (3.9)  tr. 9.0 1.0 5.2 1.0  - 2.8 4.9 2.9  tr. 86.4 - 12.3 1.1  tr. 3.9 - 25.2 2.2  9.3 0.7 78.2 42.2 17.5  90.1 | 17. 18. 22. 23. 24. 34.  0.2 - 3.0 9.1 61.5 57.3  0.2 - 14.2 tr. 8.5 17.9  tr tr. 1.1 3.1 6.6  0.2 - 0.8 tr. 2.2 1.1  (tr.) (0.9) (3.8) (10.1) (3.9) (2.9)  tr. 9.0 1.0 5.2 1.0 -  - 2.8 4.9 2.9 2.9  tr. 86.4 - 12.3 1.1 -  tr. 3.9 - 25.2 2.2 -  9.3 0.7 78.2 42.2 17.5 13.5  90.1 0.7 |  |

Chemical Analyses of Valuable Concentrate
Products.

| Product.        | TiO <sub>2</sub><br>Percent | ZrO <sub>2</sub><br>.Percent. | Fe <sub>2</sub> 0 <sub>3</sub><br>Fercent. | Total<br>Rare<br>Earths<br>Percent. | ThO <sub>2</sub><br>Percent. |
|-----------------|-----------------------------|-------------------------------|--|-------------------------------------|------------------------------|
| 18-Zircon conc. | 7.5                         | 60.4                          | N.D.                                       | N.D.                                | M.D.                         |
| 23-Monazite "   | N.D.                        | N.D.                          | N.D.                                       | 11.7                                | 0.95                         |
| 40-Rutile "     | 86.2                        | 2.7                           | 4.5  | N.D.                                | N.D.                         |

N.D. denotes - not determined.

The estimated recoveries of rutile, zircon and monazite given below are based on the percentage weight and the mineralogical analysis of each of the three valuable concentrate products (shown in Tables 7 and 8 respectively).

#### Rutile:

The rutile concentrate represented 0.51 percent weight of the original spiral feed and contained 69.7 percent total rutile. However, since the iron content of ferruginous rutile makes it unsaleable, only the non-opaque rutile is of any value. The rutile recovery is therefore based only on the recovery of non-opaque rutile. Therefore the grade of the rutile concentrate should be considered as 79.8 percent and not 89.7 percent. The presence of this ferruginous material would seriously affect the market value of the concentrate.

Using the lower figure in conjunction with the nonopaque rutile content of the spiral feed, the recovery of nonopaque rutile is 45 percent.

#### Zircon:

The zircon concentrate represented 0.61 percent weight of the original spiral feed and contained 86.4 percent zircon, equivalent to a recovery of 38 percent of the zircon in the original feed.

#### Monazite:

Of the original spiral feed 0.33 percent weight was recovered as a monazite concentrate containing 25.2 percent monazite. This represented a recovery of 60 percent of the monazite present in the original spiral feed.

The monazite concentrate contained a large proportion of other minerals, mainly zircon and garnet. Since garnet is weakly magnetic a portion of this monazite concentrate was put through a disc pick-up type magnetic separator. It was found that 60 percent weight of the initial concentrate could be rejected as a magnetic fraction containing only 5 percent of the monazite in the initial monazite concentrate. Thus monazite hehaves as a non-magnetic mineral when subjected to low intensity magnetic separation.

Mineralogical Results of Up-grading Initial Monazite
Concentrate.

| Fraction.   | Magnetic.                                       | Non-Magnetic.   |  |
|---|---|---|--|
| Percent Weight of Spiral Feed.  | 0,20  | 0.13  |  |
| Percent Weight of:  |   |   |  |
| Monazite. Zircon. Rutile. Garnet. Haematite. Ilmenite. Magnetite, limonite. Miscellaneous Heavy | 2.1<br>0.7<br>4.0<br>60.5<br>27.5<br>3.3<br>1.4 | 60.2=percent Ln <sub>2</sub> 0 <sub>3</sub> 26.5= 19 " Zr0 <sub>2</sub> 7.0 1.2 |  |
| Silicate.   | 0.5   | 1.0   |  |
|   | 100.0   | 100.0   |  |

The non-magnetic fraction was analysed and gave the values listed below:

| Total Rare Earths.                           | 31.4        | percent. |
|--|-------------|----------|
| ThO  | 2.8         | 11       |
| ThO <sub>2</sub><br>ZrO2<br>TiO <sub>2</sub> | 30.8        | 11       |
| TiO2   | 30.8<br>8.2 | tt       |

It is apparent that clean monazite will contain approximately 4.7 percent ThO<sub>2</sub> and 52.3 percent rare earths.

During the tests the separation of the various minerals was determined by grain counts. However due to the small sample normally used in microscopic work and the inherent difficulties of obtaining a representative sample of such small size, this method was used for comparison only. All final products were assayed using chemical methods.

#### 7. <u>DISCUSSION OF RESULTS</u>.

#### 7.1 Initial Gravity Concentration.

Flowsheet 1 indicates the treatment method adopted for this initial concentration step. 'It was found necessary to retreat various fractions in order to achieve a high recovery

of valuable heavy minerals in the final concentrate. In plant practice where the continuous recycling of middling products is employed, it would not be necessary to adopt the same number of concentration stages.

The results in Table 6 show that good recoveries of the valuable heavy minerals were obtained in the concentrate fraction. As the loss of these minerals in the middling fraction is low, further work was based on the concentrate fraction alone, the middling fraction being considered as a tailing.

### 7.2 Production of Valuable Mineral Concentrates.

The method adopted (Flowsheet 2) was based on the theoretical behaviour of the mineral constituents of the original sand when subjected to electrostatic, magnetic and gravity forms of concentration.

The expected mineral distribution is shown in Flowsheet 3. This flowsheet indicates that it should be possible to produce clean monazite and zircon concentrates. It was anticipated that, due to the presence of non-magnetic limenite and haematite, the rutile concentrate would be contaminated by these iron exide minerals.

The results in Table 8 show that it was not possible to produce market grade products.

The monazite concentrate was contaminated to a large degree with zircon and other heavy silicates, mainly garnet.

From an examination of Flowsheet 2 it appears that any zircon reporting in the monazite concentrate must be feebly magnetic.

Subsequent treatment of the initial monazite concentrate (see Table 10) indicated that most of the garnet and iron oxide minerals could be removed by low intensity magnetic separation.

Unfortunately the monazite and zircon in the initial monazite concentrate appear to possess similar magnetic susceptibilities.

The rutile concentrate contained amounts of iron oxide minerals, ferruginous rutile and heavy silicates. Although this

-13-

concentrate could probably be upgraded in TiO<sub>2</sub> content by further physical or chemical methods, the recovery of non-opaque rutile in this concentrate (Product 40) is only 45 percent, which is too low to warrant further treatment.

For similar reasons attempts to remove the contaminating rutile from the zircon concentrate are not considered warranted.

From experience gained during this investigation such a separation would be very difficult.

It was shown in Section 6.2 that equilibrium conditions were not reached but it is reasonable to assumes that at equilibrium the percentage weight of each of the final products would be increased proportionately without any change in mineralogical composition. For the three concentrate products this would mean an increase in percentage weight and recovery as shown in Table 11.

TABLE 11.

Estimated Percentage Weights and Recoveries at Equilibrium Conditions.

| Product                      | Atta                 | ined.                | Expected at Equilibrium |                      |  |
|------------------------------|----------------------|----------------------|-------------------------|----------------------|--|
| (Flowsheet 2).               | Percent<br>Weight.   | Percent<br>Recovery. | Percent<br>Weight.      | Percent<br>Recovery. |  |
| 17<br>18(Zircon conc).<br>22 | 3.53<br>0.61         | 38                   | 3.95<br>0.69            | 43                   |  |
| 23(Monazite conc).           | 7.70<br>0.33<br>0.22 | 60                   | 8.65<br>0.37<br>0.25    | 67                   |  |
| 34<br>40(Rutile conc.)       | 4.37<br>0.51         | 45                   | 4.91<br>0. <b>5</b> 7   | 51                   |  |
| Gravity conc.                | 17.27                |                      | 19.40                   |                      |  |

<sup>\*</sup> Recoveries are based on the amount of pure mineral in the concentrates as a percentage of the minerals in the feed to the gravity concentration section.

#### 8. OBSERVATIONS and CONCLUSIONS.

It was found impossible to achieve clean mineral separations, due to overlapping of specific gravity, magnetic and electrostatic characteristics of the constituent minerals in the original sand.

If it were feasible to upgrade the final products to meet market specifications without lowering the recovery, the values of the concentrates per ton of original sand would be as follows:

|                              |                   |                    |                   |                   | T              | TAL.          | 10     | 9.            |
|------------------------------|-------------------|--------------------|-------------------|-------------------|----------------|---------------|--------|---------------|
| Monazite<br>Rutile<br>Zircon | (At<br>(At<br>(At | £150<br>£65<br>£17 | per<br>per<br>per | ton<br>ton<br>ton | of<br>of<br>of | conc.) conc.) | 2<br>5 | 10<br>11<br>0 |

The above values are based on the estimated recoveries under equilibrium conditions (Table 11) but it should be realised that the prices used are those current at the time of writing and are liable to vary at short notice.

The returns to be expected from the treatment of the dune sands are, at the moment, not sufficient to make the deposit attractive for commercial operation.

However, there are several means by which the economic position might be improved.

The chemical cleaning of particles prior to separation, the application of flotation and more careful control of temperature during electrostatic separation are three aspects worthy of further investigation. Also the grade of the naw material could be increased by inclusion of higher grade material from the beach.

The monazite is an important constituent of the sand and its probable future importance as a strategic mineral would appear to justify further work to ensure that recovery methods

would be available at short notice. Moreover, the fact that the large deposit is a borderline case in itself justifies further work.

There are similar but smaller deposits adjacent and in other parts of the State. Methods used for this deposit could probably be adapted for the adjacent material.

#### 1. APPENDICES.

#### (1) Flowsheet No.1

Initial Gravity Concentration.

#### (2) Flowshcet No. 2.

Production of Valuable Mineral Concentrates.

In this flowsheet the following points should be noted.

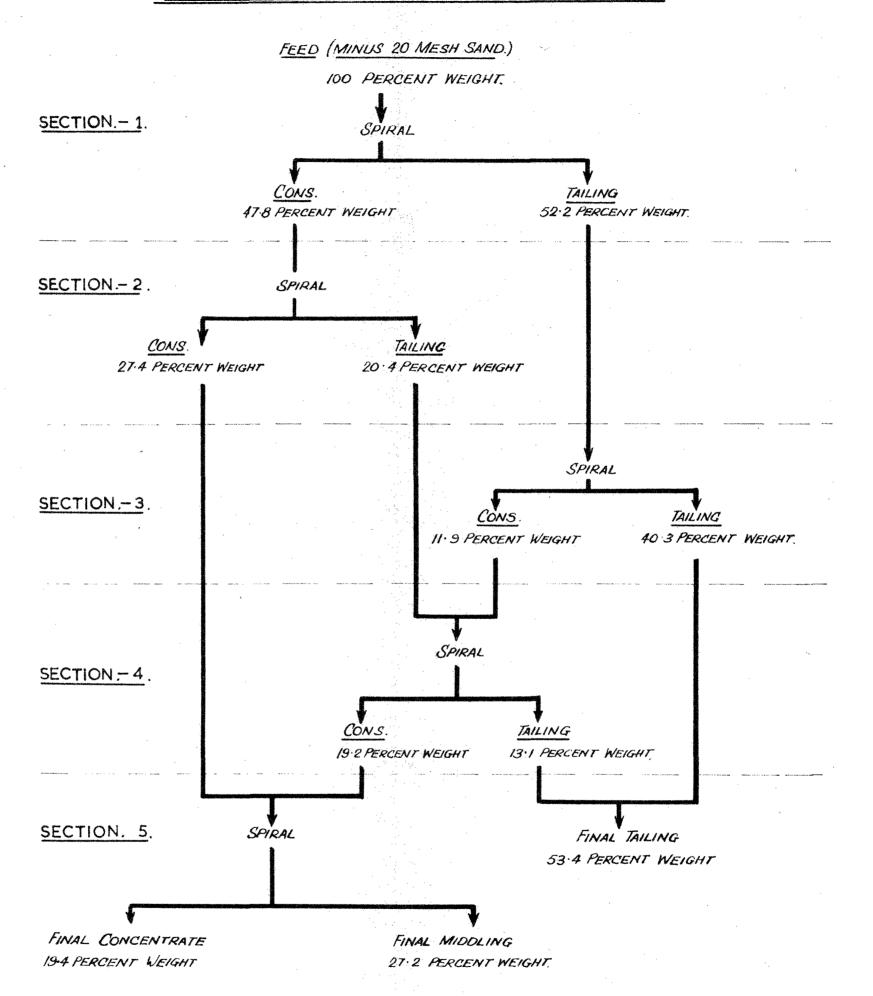
- (a) All percentage weights refer to the weight of any product as a percentage of the feed to the initial gravity concentration stage.
- (b) "H.T." Separation denotes High Tension Electrostatic Separation.
- (c) "N/C" denotes Non-conductor.
- (d) "C" denotes Conductor.
- (e) "I.R." Mag. Sep" denotes Induced Roll Magnetic Separation.
- (f) "N/M" denotes Non-magnetic.
- (g) "M" denotes Magnetic.
- (h) The "C", "M", and "T" immediately following Wilfley
  Table denotes Concentrate, Middling and Tailing
  respectively.

#### (3) Flowsheet No. 3

Mineralogical Flowsheet.

The abbreviations quoted for Flowsheet No.2 apply to this flowsheet.

## HEAVY SANDS INVESTIGATIONS FLOWSHEET No-1.



\_\_ N.B. PERCENT WEIGHTS EXPRESSED AS PERCENT WEIGHTS OF SPIRAL FEED"

SECTION, 1.

