# METALLURGICAL TESTING METHODS
# (RENISON, TASMANIA

Procedures used at Renison and other mines on the west coast of Tasmania, originally compiled from many sources.   A few of the figures from the original document were mining.  Reference to them has been left in on the hope that someone can provide them.

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Appendix 1

# 1    INTRODUCTION

## 1.1    Scope of the Manual

This laboratory manual attempts to collate operating and test procedures at the CMS laboratory with the following objectives in view:

- to standardise laboratory unit mineral separation procedures so as to facilitate close reproducibility of experimental conditions and hence of resulting data.

- to improve on existing laboratory skills such that ambiguous clear-cut procedures are evolved commensurate with high overall efficiency.

- to rationalise procedures to ensure compatibility with the highest possible standards of laboratory safety.

To enable easier comparison and acceptability of CMS laboratory data with those from outside laboratories, reference is made to relevant Australian and International standards.

## 1.2    Laboratory Safety

The wide diversity of testing procedures utilised at the laboratory necessitate <u>the highest priority to be accorded to adoption of safe working practices</u>.  These are underlined at various portions of the manual and it is recommended that they be strictly adhered to.

## 1.3    Ore Characterisation

Ore characterisation studies the mineralogical relationships within a potential ore and the detailed responses of that ore to specific physical and chemical upgrading procedures.  It forms the basis of metallurgical process design or process modifications.

Techniques used in ore characterisation include:

- crushing, grinding and screening tests with associated assaying and mineralogy to determine breakage (work index) and liberation modes;

- physical beneficiation steps like heavy liquid separation, flowing film tests, magnetic and electrostatic separation;

- physico-chemical concentration processes, e.g. flotation; and

- chemical concentration procedures like leaching.

Table 1.1 **(Missing)** illustrates the information obtainable from these tests, by applying experience, and good mineralogical backup, it should be possible to make reasonably accurate estimates of recovery.

A useful technique in the assessment of tin ores is the natural grain size determination, arrived at by sizing unbroken grains of liberated cassiterite after selective acid dissolution of the host gangue. Figure 1a *(missing)*, for instance, displays the natural grain size distribution of a number of tin ores and the recovery achieved at the Cleveland mill on these ores. Natural grain size data curves are particularly valuable since they indicate the amenability of ores to gravity separation and cassiterite shapes, association and competence is also provided.

It is recognised that the validity of sophisticated test work hinges on correct sampling and preparation techniques. Specific procedures are outlined. With large numbers of samples it is important that, apart from normal precautions to prevent cross-contamination, sample comminution preparation procedures at CMS, i.e. techniques of jaw and rolls crushing and pulverisation, should take into account liberation characteristics of the ore-body evident from preliminary size analysis data, rather than achieve a blanket size reduction to a specific fine size for all ores.

Heavy liquid analysis, derived from washability tests in the coal industry, constitute an initial assessment of any specific ore body. These tests yield qualitative data on liberation, and the amenability to gravity separation, preconcentration techniques and flotation.

Subsequent release analysis, flotation, magnetic separation and leaching tests provide specific metallurgical data of particular relevance to recovery and grade.

The following are generalised ore characterisation steps:

- Metallurgical analysis of head grades and associated size distribution studies;
- A simplistic model analysis may be required.

An x-ray fluorescence scan on unknown samples is also recommended.

- Suitability of the ore for heavy liquid testing is assessed by the requirement that combined $CO_2$ + Fe + S is < 50% and $SiO_2$ is > 30%.

  Laboratory heavy liquid testing will be recommended for samples < 4kg. Larger bulk samples with a topsize up to 40mm may be tested with an Erickson cone.

Independent of the above steps, ore characterisation studies should desirably envisage:

- Detailed mineralogy to establish qualitative, association patterns of mineral/gangue complexes and full Model Analysis; and
- Comparison of metallurgical response of ore to a standard mill circuit recovery (through flotation or other associated ore concentration processes) to determine the maximum liberation point for the unknown ore.

With regard to heavy liquid testwork it is desirable that initial sink/float tests down to 38 microns be repeated to check for consistency of response.  It should be routine practice to submit selected heavy liquid products, say 3.31 sink/float fractions for mineralogical examination.  On the basis of these heavy liquid results that would yield a first pass liberation point, it may be necessary to selectively leach specific sink fractions and concentrate the residues, say by super panning to assess extent of visual (microscopic) liberation.  With regard to flotation, a normal CMS flotation grind, for both sulphide and oxide ores, is selected from liberation studies on heavy liquid tests.

The ultimate aim of all ore characterisation exercises should be the determination of detailed and optimum metallurgical response to specific unit concentration steps, specifically grades (enrichment ratios) and recoveries realistically attainable, to enable onward plant design.  It is likely that in most cases, full scale-up to mill operating tonnages may be difficult without intermediate scale pilot-plant testing.


# 2      SAMPLE PREPARATION, CRUSHING AND GRINDING


## 2.1     Introduction

This section of the laboratory manual discusses aspects of sample preparation as well as operating details of individual crushing, grinding and classification equipment.  The individual steps outlined are recommended for guidance purposes.  A suggested time frame, where applicable, is indicated for some unit operations (sequence of steps) to help with planning specific test programmes.


## 2.2     Sample receipt and labelling

- Identify and record sample details in workbook.

- Investigate and record client requirements.

- If samples are bulky and require size reduction, reduce to a workable size as per Gy's sampling equation (Appendix 1).

- Pack samples into plastic bags each with detailed identification labels.

- If samples are not to be worked on straight away, store them in the sample storage area.

## 2.3    Sample preparation

Laboratory sampling procedures involve either:

- Coning and Quartering; or
- Riffling.

### 2.3.1    Coning and Quartering

The method which is used for sampling large quantities of material say 20kg, consists of pouring or forming the material into a conical heap upon a solid surface (e.g. a steel plate) and relying on radial symmetry to give four quarters when the heap is divided by a cross. Two opposite corners are taken as the sample the other two set aside.  The portions chosen may be further reduced by a repetition of the process, until the required size of sample is obtained.

Operator skill defines the accuracy of this form of sampling.

- Procedure of Coning and Quartering an approximate 50kg sample

  Starting sample weight (approximately 50kg)

  1. Set up adjacent to work area.
  2. Clean steel plate.
  3. Spread out sample and mix thoroughly into conical heap.
  4. Quarter.
  5. Repeat quartering.
  6. Bag sample – replace container to storage with excess sample.

  It is expected that steps 1 – 6 should take less than 30 minutes.

### 2.3.2    The Riffle

This sample splitter is an open V-shaped container under which a series of chutes are at right angles to the long axis, giving a series of rectangular slots of equal area.  These alternatively feed two collection trays.  The sample whose particular size allows free movement through the slots (the largest particle being one-third the riffle opening) is poured into the feeder and becomes split into equal portions.  After repeated cycles the desired sample size is obtained.

Procedure for the Riffle

1. Set up sample and riffle, ensuring that the riffle is initially clean.
2. Riffle once.
3. Repeat riffling, discarding every alternate sample.
4. Bag sample and label – store excess sample.
5. Clean riffle.

The above sequence of steps should take 15 minutes.

## 2.4      Drill Core Samples

The sample of core may be delivered a whole or split core, usually in lengths not exceeding 150mm.  Each core sample should be weighed so that any future compositing may be done on a weight basis if needed.  Prior to any crushing, a portion of each core is retained in its original condition for mineralogical examination, and depending on a visual assessment, a slice may be cut along or across the axis of one or more pieces of each core.

Procedure for Drill Core

1. Weigh sample and record.
2. Select for mineralogy; bag, label and record.
3. Jaw crush, riffle split – bag one half and label.
4. Rolls crush other half, maybe twice – clean rolls-crusher.
5. Riffle out head sample.  Bag both head and remainder – label.

Processing of a drill core sample (steps 1 – 5 inclusive) should be completed in approximately 30 minutes.

## 2.5     Crushing

This is the first stage performed in the size reduction of a sample leading to some liberation of minerals from gangue and is a dry operation involving use of a jaw crusher, followed by screening and rolls-crushing.

The laboratory sample preparation room is equipped with a 200 x 130mm jaw crusher and a 200 x 150mm rolls-crusher, both manufactured by Altec WA.

Materials fed to these crushers must be limited to a certain top-size and feed rate as prescribed in the maker's instruction manuals.  Lubrication requirements and the procedure for the alteration of settings are set out clearly in these manuals, which are kept near the machines.

SAFETY GLASSES AND EAR MUFFS ARE PROVIDED AND MUST BE USED

If a large amount of sample is being dealt with, then after jaw-crushing the sample may be riffled to reduce the quantity to be rolls-crushed Crushed product from the jaw-crusher is screened at 3.35mm and any oversized fed to the rolls until all crushed sample passes 3.35mm, in three stages as outlined previously.

When changing the gap between the rolls, adjust both nuts equally so that the rolls remain parallel to each other.  Gap openings can be set with standard lead cylinders, the diameter of the latter being equivalent to the jaw opening.  Apply lock cut procedure.

THE POSSIBILITY OF CROSS CONTAMINATION MUST BE CONSIDERED AND AVOIDED BY THOROUGHLY CLEANING THE MACHINES BETWEEN SAMPLE CRUSHINGS.

Procedure for Sizing a 1kg sample

Steps:

1.  Jaw and rolls crush if required – Riffle out head sample.

2.  Wet screen at 38 microns; dry the products and record their weights.

3.  Riffle out 200 gram of the oversize and screen at the desired sizes.

4.  Weigh each fraction including the –38 micron fraction and record.

5.  If required, cyclosize the –38 micron fraction as per procedure.

6.  Weigh each fraction from the cyclosizer and record.

7.  All samples being sent for metallurgical analysis should be pulverised and neatly bagged and labelled.

8.  Ensure that each work area inclusive of screens, buckets, beakers, pulverisers etc. are thoroughly cleaned after use.

9.  Calculate and tabulate distributions from returned assays.

The overall sequence of steps 1 – 8 inclusive should take about 2½ hours.


## 2.6     Pulverising

This operation prepares the sample for metallurgical analysis by reducing to a particle size, usually –10µm, so that the mineral is exposed to dissolution by the normal assay solvents or meets the requirements of x-ray fluorescence determination.

Pulverising grinders used in the sample preparation room are of two types:

*   the disc pulveriser, generally for sample >1kg, where the material is fed through a hopper and enters a space between the discs, one of which is revolving, the other stationary. Fineness of product is determined by a screw adjustment;

- a ring pulveriser consisting of a covered bowl with concentric heavy rings between which the sample is poured. During grinding the rings rotate with a vibratory motion for a pre-set time interval. Care must be exercised to avoid overloading as this will affect the fineness of grind.

  To ensure that in every case, sufficient pulverising is achieved minimal pulverising time should be ~2½ minutes. Further, it is recommended that one in every two hundred samples is tested for consistency in liberation. Difficulties in attaining metallurgical balances often can be traced back to insufficient pulverising.

CROSS CONTAMINATION CAN BE AVOIDED BY CLEANING ALL PARTS AND SUBSTITUTING CLEAN QUARTZ FOR THE SAMPLE AND GRINDING IT BETWEEN EACH SAMPLE RUN.

Procedure for Ring Pulveriser (≤70 grams) Sample:

1. Clean the pulveriser with clean quartz only.

2. Introduce sample* into the pulveriser and pulverise for a minimum of 2½ minutes.

3. Assess further size reduction requirements. The product should be 100% passing 10 microns and not have a gritty feel. It may be necessary to repeat step 2.

4. Empty pulveriser with the brush provided or with the assistance of an air jet, bad and label sample.

* If sample appears to be damp, submit it to drying under the lights before pulverising.

## 2.7    Bond Grindability Test

### 2.7.1    Purpose

The grindability test is designed to give a measure of the grinding characteristics of a material by finding the amount of size reduction resulting from a controlled energy input.

### 2.7.2    Principles

Grindability of a material depends upon its hardness, structure, the manner in which it is fractured, water of combination, hygroscopicity, tendency of flocculate and agglomerate, combustibility and sensitivity to changes in temperature. The grindability is useful in evaluating power requirements for grinding. To design a particle breakage system it is important to know the energy required to achieve a given size reduction. The most commonly used energy-size relationship is Bond's third law equation.

$$W = Wi \frac{\sqrt{F} - \sqrt{P}}{\sqrt{F}} \times \frac{100}{\sqrt{P}} \qquad \text{.................................... 1}$$

Wi = work index of the material comminuted.
   It is a relative measure of the resistance of the material to fracture.

F, P = feed and product 80% passing size in microns

W = energy required, kWh/short ton.

Useful forms of equation 1 are as follows:

$$Wi = \frac{W}{\frac{10}{\sqrt{P}} - \frac{10}{\sqrt{F}}} \qquad \text{.................................... 1 (a)}$$

$$P = \left[ \frac{10\, Wi\sqrt{F}}{W\sqrt{F} + 10Wi} \right]^2 \qquad \text{.................................... 1 (b)}$$

For the ball mill grindability test, the work index is calculated from the following revised equation:

$$Wi = \frac{44.5}{(P_i)^{0.23} \times (Gpb)^{0.82} \times \left[ \frac{10}{\sqrt{P}} - \frac{10}{\sqrt{F}} \right]} \qquad \text{.................................... 2}$$

where:

   Pi = sieve size tested (microns)
   Gpb = ball mill grind ability
   Wi, P & F have the same meanings as in equation 1.

Equipment required:

Laboratory ball mill, 34.02cm x 34.02cm with following ball charge:

| Ball diameter (cm) | No. of balls | Total weight (gm) |
|---|---|---|
| 3.68 | 43 | 7880 |
| 2.97 | 67 | 7847 |
| 2.54 | 10 | 511 |
| 1.90 | 71 | 2143 |
| 1.55 | 94 | 1744 |
| | | 20.125kg |

- Agitator rolls.

- Measuring cylinder, 700ml volume.

- Set of screens, including the following sizes:
   850µm, 600µm, 425µm, 300µm, 210µm, 150µm

- Ro-tap machine, with timer.

- Drying facilities.

- Pressure filter.

## 2.7.3   Sample Preparation

- Obtain about 5 – 6kg of material to be tested and dry thoroughly.

- Crush dried material minus 3360µm.

- Thoroughly blend the crushed product and then split out a sample for screen analysis.

## 2.7.4   Procedures

1. Carry out wet and dry sizing on the split sample.  The percentage of undersize from the screen which is the required size (297µm in this case) is the percentage of finished material in the feed.

2. Fill the 700ml test can with ore and compact by shaking.  Add more ore as necessary until further compaction ceases.

3. Weight and transfer ore to the ball mill.

4. Grind dry for 100 revolutions.

5. Empty the ball charge and ore through a coarse screen to separate the balls from the ore.

6. After mill and balls are swept clean, screen and ground ore through the mesh screen to be tested, using coarser protective screens if necessary.  (Note: due to the large sample size, it will probably be necessary to screen the sample in a number of smaller portions.)

7. Determine and record:    weight oversize;

   weight undersize;

   corrected undersize

   (assume weight differences are
    associated with undersize only)

8. Add to the oversize a quantity of new  unsegregated feed equal to the weight of corrected undersize.

9. Return the oversize and new feed to the ball mill.

10. Calculate the number of revolutions to produce a circulating load of 250%.

11. Allow the mill to rotate the required number of times.

12. Repeat steps 7 through 11 until the net grams of sieve undersize produced per mill revolution reaches equilibrium and reverses its direction of increase or decrease.

13. Screen entire mill charge through required mesh screen.   Do screen analysis on oversize and undersize.

14. Plot cumulative weight percent passing VS particle size (in microns) for feed, final cycle oversize and final cycle undersize.

15. Determine $d_{80}$ for feed and last cycle undersize ($d_{80}$ is the screen opening in microns through which 80% material passes).

16. Calculate average grams of undersize produced per revolution for the last three cycles (Gpb).

17. Calculate work index using equation 2.

Calculations:

Number of revolutions required to produce 250% circulating load can be calculated using following formula:

$$X = \left[ \frac{W}{3.5} - \frac{(Y)\,(P)}{100} \; \frac{R}{U} \right] \qquad\qquad ....................................... \ 3$$

where:

| | | |
|---|---|---|
| X | = | number of revolutions required. |
| W | = | weight of original feed. |
| Y | = | corrected weight undersize previous cycle. |
| P | = | percent undersize in original feed. |
| R | = | revolutions previous cycle. |
| U | = | actual undersize produced previous cycle. |

Determination of $d_{80}$ values:

$$d_{80} = X + Y(Z - X) \qquad\qquad ...................................... \quad 4$$

where:

$d_{80}$ = 80% passing size.

X = micron size of screen through which nearest percentage below 80% passes.

Z = micron size of screen through which nearest percentage above 80% passes.

Y = ratio of the difference between 80% and the nearest cumulative percent below 80% AND the difference between the nearest cumulative percent above 8p% and the nearest cumulative percent below 80%.

Example for the determination of $d_{80}$:

| Sizes μm | Cum % passing |
|---|---|
| 841 | 98.35 |
| 495 | 92.71 |
| 420 | 84.32 |
| 297 | 78.65 |
| 210 | 65.30 |
| 149 | 52.18 |

$$d_{80} = X + Y(Z - X)$$

from above sizing chart:

X = 297

Z = 420

$$Y = \frac{80 - 78.65}{84.32 - 78.65} \quad \frac{1.35}{5.67} = 0.238$$

$d_{80}$ = 297 + 0.238 (420 - 297)
297 + 0.238 (123)
325μm

Ball Mill Grindability (Gpb) = (gm/rev) average of last three grinding cycles to give 250% circulating load.

Precautions:

There should be consistency in compaction of ores in the 700ml test can. This is normally done by shaking.

Accurate observations on grinding revolutions are essential.

Dry screening procedures should be consistent.

Gpb calculations should aim for maximum accuracy.


## 2.8 Comparative Grindability Test

### 2.8.1 Purpose

The test is designed to give a reasonable indication of the grinding work index of an ore material relative to one or more known standards. It is not applicable to mill tailings material.

### 2.8.2 Basic Equipment

Set of screens @ 75 to 2440 micron sizes.
Ro-Tam machine

Ball mill rolls

CMS ball mill No.1 with approximately 10kg of ball charge distributed as follows:

| Ball Diameter | % |
|---|---|
| 1 ½ " | 20 |
| 1 " | 50 |
| ¾ " | 30 |
| | 100 |

Procedure

1. Crush the known and test samples to -3350 micron using either the jaw or rolls crusher.

2. Riffle split the crushed sample. Weight out approximately 300 grams for feed sizing and exactly 1000 grams for grinding.

3. For sizing feed:
   - wet screen at 70μm and dry the product.

- dry screen the oversize (+75μm) on 2440, 1680, 1016, 600, 300, 150 and 75μm.

For grinding:

- place 1000 grams of the known sample in the ball mill, introduce the ball charge, add 625ml of water.  Secure mill lid tightly and grind for 10 minutes.

Wash the pulp thoroughly from the mill and the charge, wet screen the pulp @ 75 microns and dry product.

Dry screen the oversize (+75μm) product on 1680, 850, 600, 425, 210, 106, 75μm.

Plot feed and product sizings on a log-normal graph sheet as cumulative weight % passing versus particle size.  Each particle size is determined by calculating the geometric mean size (GMS) of two successive screens used.  For example, GMS of −3350 + 2440μm size range equals to:

$$\sqrt{3350 \text{ x } 2440 \text{μm}}$$

$$= 2830 \text{μm}$$

Repeat above procedure for the unknown sample.

Read off $F_{80}$(μm) and $P_{80}$(μm) from the plots and calculate the comparative grinding work index (CWi) of the unknown.

Note:   $F_{80}$  =   the diameter in micron which 80% of the feed weight passes.
         $P_{80}$  =   the diameter in micron which 80% of the product weight passes.

Calculation

The formula to use is:

$$\text{CWi x} \left[ \frac{1}{\sqrt{P80}} - \frac{1}{\sqrt{F80}} \right]_C = \text{RWi x} \left[ \frac{1}{\sqrt{P80}} - \frac{1}{\sqrt{F80}} \right]_R$$

where R refers to the reference standard sample of known Wi and C refers to the unknown sample.

Caution

For the comparison to be valid, the following rules of thumb should be noted:

- Feed size distributions of the reference sample and unknown should ideally be similar.

- Grinding conditions must be identical.

- Ideally, comparative ores should have roughly similar grinding characteristics; that is, similar slope and shape in size distribution curves. For example, hard siliceous ore ad soft talcose ore are not compatible.

# 3 LABORATORY SIZING

## 3.1 Introduction

Since the effectiveness of practically all mineral processing operations is a function of the size of the particles treated, a thorough knowledge of the size characteristics of the materials handled is of the greatest importance. Hence, the significance of consistent and reliable laboratory sizing techniques. The two most important sizing methods are:

1. Screening

2. Classification (Elutriation)

### 3.1.1 The ASTM Sizing Scale

The ASTM scale is based on a geometric progression, i.e. ore in which successive grades are in constant ratio, e.g. $\sqrt{2}$ . For practical manufacturing reasons, the reference screen opening is that of the so-called 200 mesh screen. This standard screen is a woven screen having 200 wires per linear inch, and likewise 200 openings per linear inch, in which the opening is a square having an edge of 74 microns or 0.074mm. Graphical Representation of Sizing Analyses

- Cumulative plot where the ordinate is the percentage of the total weight coarser than a given size, the abscissa, the particle or screen opening (mm or microns).

- Plot of percentage of the total weight included within consecutive divisional sizes (i.e. the amount retained by each screen and passing the screen immediately larger) (ordinate) versus actual screen opening in mm or micron. This type of plot could be misleading in that uneven emphasis is placed on the various size ranges. Difficulties could also be experienced in dealing with the material finer than the finest screen to which the sizing operation is conducted. Useful data can often be obtained by using a log scale for particle size (abscissa) versus cumulative percentage retained (ordinate) or direct percentage retained by individual size.

- In studies of size distribution in crushed products, a particularly useful graph is obtained by plotting the logarithm of the percentage in each size grade against the logarithm of the size. Another graph that has been adopted is one in which the ordinate is the log-log scale of the reciprocal of the cumulative percentage in each size grade, and the abscissa is the logarithm of the size.

### 3.1.2 The Ro-Tap Testing Sieve Shaker

This machine causes a circular motion of the material on the screen; the frame in which the screens are placed is provided with a translatory motion at one end and a circular motion at the other so that there is eventual movement of the material on the screen. Besides, a blow is delivered to the nest of sieves, once for each revolution of the frame. A nest of sieves, consisting generally up to six screens, and occasionally 12, is shaken in the machine for from 5 – 20 minutes depending upon the extent to which it is desired to approach the end point of the screening operation.

### 3.1.3 Preliminary Comments on Sieving Procedure

If proper care is taken not to overload the screens, and the material is deslimed (i.e. rejection of −38 micron fraction) and dried, machine screening gives excellent results. Most satisfactory results are obtained when the amount of material on each screen, at the end of the screening operation, does not exceed that required to form a layer one particle deep. However, a bed of particles of extremely fine particles to coarser particles or to each other through electrostatic action or the presence of minute amounts of moisture can be overcome by a combination of wet and dry screening.

## 3.2 Dry Screening

### 3.2.1 Purpose

Dry screening is used to determine the size distribution of particles coarser than 38 micron. For an accurate determination, it is necessary to deslime at 38 micron before commencement of dry screening.

### 3.2.2 Technique

Sieve Analysis is accomplished by passing a known weight of sample material successively through finer screens and weighing the amount collected in each screen to determine the percentage weight in each size fraction.

### 3.2.3 Equipment Required

1.  Set of standard screens, including bottom pan and lid. The usual range of sizes includes 4760 micron, 2380 micron, 1190 micron, 841 micron, 595 micron, 297 micron, 210 micron, 149 micron, 105 micron, 74 micron, 53 micron, 44 micron, although not all of these sizes will necessarily be used in a given test.

2.  Ro-Tap machine, with timer.

3.  Balance, accurate to 0.01 gram.

4. Bristle brush for cleaning screens.

5. Weighing pan.

### 3.2.4 Sample Preparation

1. Ensure material to be dry screened is absolutely dry for accurate results.

2. Split material to get the approximate sample size required, as per schedule below:

| Limiting Particle Size microns | Standard Charge grams |
|---|---|
| 4760  -  2380 | 1000 |
| 2380  -  841 | 500 |
| 841  -  420 | 250 |
| 420  -  210 | 100 |
| -  210 | 50 |

### 3.2.5 Procedure

1. Starting with the finest screen, stack the screens on the bottom pan in order of increasing micron size.  The top screen will be the coarsest one in the nest.

2. Accurately weigh the feed sample.

3. Place the sample on the top screen, and secure the lid in place.

4. Place screens in Ro-Tap machine and shake for 20 minutes.

5. Tare a weighing pan on the balance.

6. Starting with top screen (coarsest one), transfer the screen fraction to the weighing pan and accurately record and weight.  Use a brush to remove wedged particles from the screens.

7. Always brush up and down a screen, never brush with a circular motion.

8. Repeat steps 6 and 7 for each screen fraction.

9. All data and calculations are to be recorded on the standard screen Analysis Data sheet.

10. Plot weight per cent of each screen fraction, and the cumulative per cent undersize.

### 3.2.6    Calculations

Size Distribution
wt% of a screen fraction =  $\dfrac{\text{Weight of Screen Fraction}}{\text{Total Sample Weight}} \times 100$

Cumulative per cent undersize
cum.% passing =  $\dfrac{\text{Total wt. of fractions finer than the screen}}{\text{Total sample weight}} \times 100$

Also, cum.% passing =  100 -  (Total per cent of fractions <u>not</u> passing the screen)

Eighty per cent passing size
-    Plot weight per cent undersize as a function of particle size in microns.  The particle size given by 80% undersize on the curve represents the 80 per cent passing size.

-    Determination of $d_{80}$ values (without extrapolution from a graph):

$$d_{80} = X + Y (Z - X)$$

where   $d_{80}$  =  80% passing size

              X   =  micron size of screen through which nearest percentage below 80% pass.

              Z   =  micron size of screen through which nearest percentage above 80% passes.

              Y   =  ratio of the difference 80% and the nearest cumulative per cent below 80% AND the difference between the nearest cumulative per cent above 80% and nearest cumulative per cent below 80%.

The following will serve as an example:
Determine the $d_{80}$ from the following sieve analysis:

| Sizes Microns | Cum. % Passing |
|---|---|
| 841 | 98.35 |
| 595 | 92.71 |
| 420 | 84.32 |
| 297 | 78.65 |
| 210 | 65.30 |
| 149 | 52.18 |

$$d_{80} = X + Y (Z - X)$$

From sizing chart:

$$X = 297$$
$$Z = 420$$
$$Y = \frac{80 \quad - 78.65}{84.32 \quad - 78.65} = \frac{1.35}{5.67} = .238$$
$$d_{80} = 297 + .238 (420 - 297)$$
$$= 297 + .238 (123)$$
$$= 297 + 28$$
$$d_{80} = 325 \text{ micron}$$

### 3.2.7 Weighing of Residue

- Accurate weightings compatible with an overall accuracy of ± 0.2% are mandatory for all screen products.

- For screen undersize from rinsing operations, do <u>not</u> remove undersize from filter paper before weighing.
    - Tare a clean filter paper of same size and type as that used as undersize.
    - Weigh undersize plus filter paper.
    - Weight shown on tared balance will be the weight undersize.

  By following this procedure, no material is lost with the filter paper or with a transfer operation.

- For screen fractions, each one should be weighed separately.

  NOTE:  Always be sure that the balance is properly zeroed before commencing a weighing.

## 3.3 The Cyclosizer[1]

### 3.3.1 Purpose

Size analysis of sub-sieve size (-38 micron).

### 3.3.2 Principles

One of the most widely used methods of sub-sieve sizing in modern mineral-processing laboratories is the Warman Cyclosizer which is extensively used for routine testing and plant control in the size range 8 – 38 micron for minerals of specific gravity similar to quartz (s.g. 2.7) and down to 4 micron for particles of high specific gravity, such as galena (s.g. 7.5).

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[1] Adapted from "Mineral Processing Technology – B. A. Wills"; Pergamon Press, 2nd Edition.

The cyclosizer consists of five unit cyclones arranged in a series such that the overflow of one unit is the feed to the next unit. The individual unites are inverted in relation to conventional cyclone arrangements, and at the apex of each, a chamber is situated so that the discharge is effectively closed. Water is pumped through the unites at a controlled rate, and a weighed sample of solids is introduced ahead of the cyclone. The cyclosizer is manufactured to have definite limiting separation sizes at standard values of the operating variables, viz. water flow rate, water temperature, particle density and elutriation time.

The tangential entry into the cyclones induces the liquid to spin, resulting in a portion of the liquid, together with the faster-settling particles, reporting to the apex chamber, while the remainder of the liquid, together with the slower settling particles is discharged through the vortex outlet, and into the next cyclone in the series. There is a successive decrease in the inlet area and vortex outlet diameter of each cyclone in the direction of the flow, resulting in a corresponding increase in inlet velocity and an increase in the centrifugal forces within the cyclone, resulting in a successive decrease in the limiting particle-separation size of the cyclones.

### 3.3.3    Equipment

- Warman Cyclosizer
- -38 micron sized material
- Beakers

### 3.3.4    Procedures

1. Switch on both power points at wall and check hot and cold water main valves open. Also check hot water control valve open. Also check that automatic water temperature (23° ± 2°C) is correctly set and operational.
2. Plug in sample chamber.
3. Switch on pump.
4. Open rotameter valve.
5. Close pot discharge valves (after removing air).
6. Check the room temperature.
7. Set timer to five minutes (Feed Cycle) and open sample chamber slowly to feed particles within five minutes.
8. After feed time (five minutes) buzzer sounds. Set timer to separation time (20 minutes) and adjust rotameter flow down to 185 (as for 5).
9. After separation time (20 minutes) buzzer sounds; adjust rotameter flow up to 200.
10. Discharge CS5 pot product in to 1 litre beaker and return plastic hose to drain.
11. Repeat discharge of CS4, CS3, CS2 and CS1 in that order as for (10)
12. Shut off rotameter valve.
13. Open all valves.
14. Switch off pump.
15. Switch off power points at wall.
16. Dry, weigh and pack each fraction.

17. Collect –5 micron fraction and determine requirement for further separation (ie.beaker decantation).
18. Send each fraction for assaying.
19. Report the results in table form.

N.B. If separation time, water temperature, flow rate or s.g. of solids (2.7) are different to above, correction factors will be required for cyclone cut sizes.

For this machine cut sized on quartz are:

| CS | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|
| **Micron** | 45 | 34 | 24 | 16 | 11.5 |

Precaution
Read the Warman instruction manual carefully and inspect the cyclosizer, noting all the component parts and how to operate them. <u>Do not</u> operate the machine until you are sure of the procedure.

## 3.4     Additional References

| 1.   Australian Standard 1152-1973 | Test Sieves |
|---|---|
| 2.   Australian Standard 1289.C 6.1:1977 | Determination of the Particle Size Distribution of a Soil-Standard Method of Analysis by Sieving |
| 3.   Australian Standard 1774.19-1978 | The determination of Sieve Analysis and Water Content. |
| 4.   Australian Standard 2350.9 | Fineness of Portland Fly Ash Cement by the 45 micron Sieve |
| 5.   Australian Standard 1038, Part 18-1981 | Size Analysis of Coke |
| 6.   British Standard BS 1796:1976 | Method of Sieving |
| 7.   Specification of Test Sieves BS410:1976 | |

# 4    FLOCCULATION, SETTLING AND THICKENING

## 4.1    Flocculation

Flocculants are added to suspensions of solids in water to cause the individual particles to collect in the form of flocs.  The formation of flocs aids in improved settling rates, better overhead clarity and/or faster filtration rates.  There are <u>three</u> main methods for achieving aggregation of very small or colloidal particles in suspension:

a) Coagulation, i.e. aggregation caused by the compression of the electrical double layer of the solids by addition of simple electrolytes.
b) Aggregation caused by the action of hydrolysing electrolytes such as ferric or aluminium sulphate, the mechanism involved being a composite of both coagulation by the polyvalent ions and bridging flocculation by the precipitating hydroxides.
c) Flocculation, i.e. aggregation through the use of long-chain organic polymers that bridge between adjoining particles.  Flocculation is, by far, most import of aggregation methods used in the mineral industry.

There are basically three types of organic polymers (or flocculants) that can be used, nonionics, anionics or cationics.  The nonionics which are a neutral polymer molecule, have a wide application and can be used as flocculants for most solids in acid, basic or neutral conditions.  In some systems where flocculation is dependent upon the "ionic" charge of the particles a polymer with either a positive or negative charge will aid in the attachment to the solid particles and hence improve efficiency of flocculation.  The charge of the particles is dependent on a number of factors and is usually pH dependent.  If the charge on the particle is overall positive, then an anionic polymer will be most suitable and vice versa.

The solubility of polymers varies considerably, but in general is very low and therefore they should be prepared as a very dilute solution, usually less than 1% by weight.  Addition of the polymer aqueous solution should be very slow, with slow agitation.

The method of application is also important.  With most flocculants adsorption is rapid and irreversible, so thorough and complete dispersion of the flocculant throughout the slurry is a necessity.  The recommended techniques of application for maximum efficiency are as follows:

1. Application as a very dilute solution
2. Multiple point addition to the suspension
3. Gentle agitation or mixing, that is agitation sufficient to ensure distribution throughout the pulp but not violent enough to destroy rapidly formed flocs.

Consumption of flocculants usually range from 0.005 to 0.2 kg/tonne of dry solids.

## 4.2 Settling and Thickening Tests

### 4.2.1 Introduction

The choice of test method depends to some extent upon the temperature of the pulp, its flocculating characteristics, the required supernatant clarity, and the equipment available.

### 4.2.2 Methods

The Long Tube Method may be used for all materials which settle without a clearly defind interface. However, where the feed in question is well flocculated, either naturally or artificially, a simplified test method as indicated below may be used to obtain significant data.

Details of Simple Settling Tests
Simple Settling Tests are conducted to determine clarification performance for a pulp, flocculated naturally or because of chemical additives, of the type which yields a constant suspended solids concentration throughout the supernatant after a period of 10-15 minutes.

1.  Place pulp in a measuring cylinder and make up to say 1000 ml mark with water. Shake thoroughly after addition of flocculant.

2.  Allow to settle quiescently and note interface depth as a function of time (seconds).

3.  Flocculation is a time function following an empirical relationship.

    *$C = k\,t^{m}$ where C is the suspended solids concentration. Supernatant samples for suspended solids analysis should be taken after settling times of 10, 15, 30, 60, 90 and 120 minutes.*

4.  A log-log plot of this data usually will give a straight line of slope m.

5.  The static detention time calculated for the desired overflow clarity, and the observed bulk settling rate may be used, with proper scale-up factors that determine sedimentation area and depth requirements.

    For thickener sizing, the relevant relationship is:

    $$\text{Unit area } m^2 / tpd = \frac{0.0415}{R}\left[\frac{1}{C_G} - \frac{1}{C_F}\right]$$

    Where R = average settling rate, m/h
    $C_G$ = Initial Solids Concentration
    $C_F$ = Final Solids Concentration
    R is determined rate x 0.8 scale-up factor

> *Comment*:: An Allied Colloids clarity wedge can very often be used to rapidly assess effectiveness of flocculation or water clarity.

## 4.3 Beaker Decantation

### 4.3.1 General

Beaker decantation is a technique used to separate a mineral sample into two size fractions according to the differences in settling velocities of the particles. It is a technique which can be used to accurately split a sample at a pre-determined cut size but has some disadvantages in that it is a time consuming method especially when splitting at fine sizes. Also a dilute solids content is required to stop natural coagulation from occurring. A spherical shape is assumed for the calculations.

The procedure is based on Stokes Law which relates the settling velocity of a particle to its size and specific gravity through the equation.

$$V_T = \frac{g\,(P_P - P_F)\,(D_{ST})^2}{18 V_F}$$

*where*

$V_T$ = terminal settling velocity of particle

$D_{ST}$ = equivalent Stokes diameter of particle

$g$ = acceleration due to gravity

$V_F$ = fluid viscosity

$SG_P$ = specific gravity of particle

$SG_f$ = specific gravity of fluid

By calculating the settling velocity of a particle of the required cut size and specific gravity, the time for the particle to fall a certain distance unhindered through the liquid, can be calculated.

### 4.3.2 Procedure

Preliminary
- choose cut size
- choose specific gravity of mineral (eg. $SnO_2$ or $SiO_2$)
- Measure temperature of fluid and determine value of viscosity of that fluid.
- Determine specific gravity of fluid.

### 4.3.3 Equipment Details

- A settling vessel is required, the size of which depends on the amount of sample and the cut size chosen.  The vessel should be large enough to avoid any form of coagulation which would interfere with the unhindered settling of the particles.  The higher the settling height the longer the settling time – sometimes several hours.

- A syphon to remove the fluid containing the unsettled particles and decantation equipment, usually consisting of a plate with a tube attached with facilities to set at a predetermined depth.  Decant fluid can then pass over the plate and up the tube.
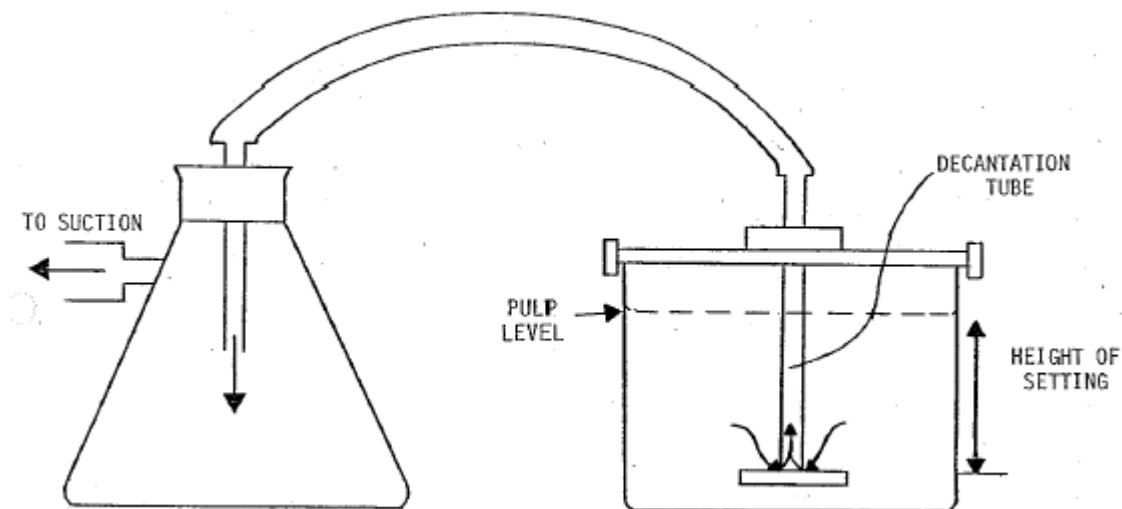


Figure 4a  Beaker Decantation Equipment Set-up

- Enclose entire system in a water bath to maintain constant temperature and viscosity and reduce disturbances to the sedimentation process, if complete accuracy is required.

### 4.3.3.1  Procedure

Place the sample in the sedimentation vessel and fill with water up to the required level.  If the sample was previously dry, complete rewetting of the sample must be performed.  Ultrasonics may help in the wetting process.  To ensure sample does not naturally coagulate, dispersants should be used, but only if no further processing of products is to be performed.  If natural coagulation does occur, more dilution may be required.

Check that all the decanting equipment is ready with the tube set at the correct height, but do not leave the tube in the liquid during the settling period.

Measure water temperature (to calculate viscosity of fluid) and maintain as constant as possible.

Agitate the vessel.

Commence settling time at completion of agitation.

Position the decantation tube delicately into position prior to completion of settling.

Remove the supernatant dispersion as quickly as possible.  Do not disturb the settled particles.

Repeat the above procedure several times until clear supernatant is obtained.  The separation efficiency is determined by the number of decantation steps.

Collect all the supernatant, flocculate, dry and weigh to obtain a weight balance.


# 5      FILTRATION


## 5.1      Introduction

Filtration is defined as the separation of insoluble solids from a liquid by forcing a portion of the liquid through a porous medium by a pressure differential, while the solids are trapped on the surface or in the depth of medium.  The two main methods that exist to create the required pressure differential are:

- Application of vacuum – vacuum filtration
- Application of high pressure to the slurry and squeezing the solids – pressure filtration

While vacuum filtration is adopted in a large variety of mineral processing industries, pressure filtration would be preferred where:

- The liquid is either too viscous
- The liquid is very close to the boiling point
- Solids are so fine and filter so poorly, or feed concentration is so low that a discharge cake cannot economically be formed, and a pre-coat filter cannot be considered for other reasons.


## 5.2      Leaf Filter Testing

The "leaf test" represents a small section of a continuous filter incorporating typical fliter medium support and filtrate drainage.  When used with techniques properly simulating the continuous filter, it can give results adequate for sizing production filters and predicting performance or determining the need for pilot testing.

Sample Size – Generally 15-20 litres of representative slurry.

Before testing, a representative portion containing sufficient solids to be meaningful should be taken for solids concentration determination.  The concentration is found by weighing the sample, filtering on a Buchner funnel fitted with two No. 1 Whatman papers, washing if necessary, drying at a suitable temperature and determine the net dry weight.  The solids concentration expressed in per cent is dry net weight x 100 divided by sample weight.


## 5.3      Test Data Format

The test data format (*missing*) calls for the minimum of essential data and calculated rates.  The feed % solids analysis on the bulk sample being tested is the "feed basis".   By dividing the cake dry weight by the sum of cake wet weight (vol x specific gravity), the "product basis" feed % solids is determined.  A significant difference between the two is evidence that the system is not at equilibrium due to filter thickening, inadequate or excessive agitation, or excessive loss of solids to the filtrate.

The actual vacuum throughout a test should be controlled and observed since this is a major factor in performance and sizing.


## 5.4      Preliminary Information

Reasonably complete information is needed on the material to be filtered.  E.g. slurry identification, specific gravity, pH, % solids, temperature, solids identification, particle size, specific gravity, liquore identification and specific gravity.

Further slurry information desirable is:  its physical nature; plant upstream process and possible variations to meet filtration objectives; the solids settling tendency and hence gravity thickening possibilities; whether solids are naturally flocculated or amenable to flocculation; temperature variations; flexibility for pH adjustment; aging effect inherent in the process, particle shape and variations with size; variations of the kind solids with size (eg. % ash may be higher in fine particles); porosity of individual particles; solubility of filter cake; inherent water of hydration; impurities; temperature of permitted oven drying.  Other useful liquor information is:  soluble solids content and their relation to saturation; viscosity; surface tension, vapour pressure variation with temperature; compatibility with flocculants.

Measure and record the filtrate volume.  When the cake has a mushroom shape requiring exclusion of the trim weight, the same proportion of filtrate must also be deducted.  Comment on the quality of the filtrate, testing for solids content when pertinent.  Measure and record the specific gravity and pH of filtrate.

In conjunction with any of the discharge methods, it is important to record observations regarding cake nature, any abnormality, cake release from the cloth, and condition of the cloth.

## 5.5 Laboratory Pressure Filtration

The laboratory batch pressure filters installed at CMS serve the primary purpose of dewatering solid-fluid suspensions and have not been designed for the performance of specific pressure filter tests.  However, by maintaining operating parameters constant e.g. slurry % solids, overall pressure applied and overall filtration time, it is possible to obtain comparative filterability data.

## 5.6 Steps for Leaf Testing

Stir the slurry with a spoon or spatula or an agitator to obtain a uniform suspension of the solids.

Turn on the vacuum source and adjust the test vacuum while pinching off the hose to the test leaf.

Submerge the leaf in the slurry, open the hose and simultaneously start the timer.  Stir the slurry as needed during form time.

At the end of planned formtime rotate the leaf up and out of the slurry just as a filter section and dry.  During the dry time slowly rotate the leaf, tilting it from it horizontal plane to half drain the leaf.  Dry for the planned time.  Note the time of any cake cracking.

Turn off the vacuum, quickly remove the tubing from the flask to break the vacuum on the leaf.  Elevate the leaf and drain remaining filtrate to the flask.  If necessary to facilitate draining, lift a portion of the cake off the cloth.

Explore the best method of cake discharge as detailed under 11 below.   If cake is mushroom-shaped, extending beyond the 0.1 ft$^2$  cloth area, trim off and separately weigh the trim.   Note: this correction is approximate.

Transfer the 0.1 ft$^2$  cake to a tared dish.  Again allow leaf to drain residual filtrate to the flask. Set the leaf aside.

Measure and record cake thickness and any variations.

Weigh the dish and contents.  Record the gross wet weight.  Put the dish in an oven at 105$^0$C or lower temperature if necessary.   Dry overnight, then record the gross dry weight. Alternatively, use drying lights until a constant dry weight of the filter cake is obtained.

## 5.7 Factors Affecting Filtration Rates and Cake Moistures

- Particle Size of Solids

  Generally the large the particle size, the higher the filtration rate in Kg/m$^2$/h and the lower the cake moisture. However, the validity of the last statement depends on other factors, e.g. distribution specific gravity of solids, absence of slimes and feed concentrates. For instance, a small average particle size, but with a narrow distribution range and no slurries, will have a high rate.

- Ratio of slimes to coarser particles

  The slimes or extreme fines in a filter feed slurry affect filtration rates to a vastly greater extent than their percentage. The residual cake moisture is similarly seriously affected. A particularly difficult slurry is one that contains relatively coarse particles and a number of very fine or slimy particles with little or no intermediate size.

- Admix of filter aids

  Filter aids like diatomaceous earth, perlite, powdered coal, fly-ash or paper pulp may be added to the flurry to increase its filtration rate and cake porosity.

- Feed solids concentration

  In general, the greater the percentage of suspended solids in a given slurry, the higher the cake filter rate in Kg/m$^2$/h and the lower the filter rate in m$^3$/m$^2$/h. Where maximum solids capacity is desired it is advisable to consider thickening the slurry by gravity. In some applications involving thickening with sludge recycle, particle size is actually increased and both cake and filtrate rates can increase.

- Filter Thickening

  Filter thickening normally occurs in a continuous filter rotating in a tank containing slurry wherein the solids in the filter tank increase in concentration and shift to a coarser size distribution. While an equilibrium concentration and size distribution is usually obtained, it may sometimes be necessary to dilute the pulp.

- Slurry pH

  Since slurry pH and particle dispersion are closely related, changes in pH could be one of the most effective methods to achieve flocculation and improved filterability, if the process can tolerate it.

- Flocculation/Dispersion of fine solids

    Flocculation is generally desirable for slurries of fine solids which are in a dispersed state and generally filter poorly.  The wide variety of polyelectrolyte flocculants provides room for a substantial improvement in filtration rates.  Effective use of flocculants, especially polyelectrolytes, on moderately high concentration filter feeds requires strong agitation to get good solids-flocculant contact.  A minimum of further agitation and minimum aging are important.

    Some slurries may be so viscous as to create filtering problems and a dispersant may be a better way to gain fluidity than dilution.

- Slurry Age

    Sometimes processes involve detention times whether international or not, which provide a conditioning effect, modifying filter performance.  Samples shipped for testing involve a risk that excessive aging may have some effect on filterability.

- Viscosity of liquor and temperature

    This is one of the most significant variables both in filtration rate and rate of dewatering to a minimum moisture.  Viscosity is closely related to temperature.  As temperature is increased, viscosity is decreased resulting in a higher filter capacity and lower cake moisture.

    As the same time, increased vapour pressure will help reduce moisture.

- Agitation Speed

    Some slurries, particularly with a wide particle size range, tend to classify in the test slurry container or the filter tube.  Increasing the agitation speed (or stirring) to a point that the coarse and fine particles are always thoroughly mixed may be desirable although too high a speed could limit cake thickness, prevent coarser particles from forming in the cake or cause delicate flocs to break down.

- Type of Filter Medium

    Filtering characteristics of fabrics depend mostly on the type of yarn and weave.  Yarns can be momo-filament, multi-filament, spun from staple fibre, or a combination of the latter two.  A high twist can make a multi-filament perform more like a mono-filament.

    Permeability and porosity are prime qualities in cloth selection.  The Frazier permeability rating, expressed as cfm/sq ft, is a measure of air flow at one-half inch water pressure through a dry cloth, and is comparable to per cent open area.

Porosity and particle retention may not be accurately indicated by permeability; there being no direct measure of porosity.

- **Filter cloth condition**

Cloth conditioning refers to the reduction of pore size or open area due to entrapment of fine solids in the interstices.

- **Applied vacuum**

The applied vacuum creates the pressure differential which is the driving force for filtration and dewatering. High vacuums give somewhat higher rates and lower moistures in all cases except for unusually incompressible cakes or where the filter medium becomes more conditioned. With coarse particles and porous caked lower vacuums (e.g. 5"-10" Hg) may be adequate. With such porous cakes, the vacuum capacity ($m^3 / m^2$) could be the important factor.

- **Cycle Time**

Cycle time of leaf tests is analogous to the filter drum speed, generally expressed in seconds or minutes per revolution. Generally the faster the drum speed, the higher the output. However, under these conditions, the cake is thinner and sometimes wetter, so discharge may deteriorate. At all times, a dischargeable cake must be produced. Any final selection of cycle or drum speed is a compromise of these conditions.

- **Surface Tension**

Lowering of surface tension through higher temperature or surfactants can significantly improve cake moisture of some materials. Its benefits are not predictable and it has no identifiable effect on filtration rate. Where surfactants are sued, vacuum capacity may be greatly reduced.

- **Cake Compression**

Cake compression is normally achieved as an adjunct to the filtration step to reduce cake moistures of compressible cakes.

# 6    AGGLOMERATION

## 6.1    Introduction

Agglomeration is the formation of aggregate by the sticking together of feed and/or recycle materials, and it includes the formation of agglomerate nuclei.  The main objective in agglomerating fines being the conversion of ores, minerals and chemicals of undesirable fineness into agglomerates characterised by a size consistency desirable for subsequent use or processing.  In metallurgical applications, the unit process of balling aims at achieving highly permeable large aggregates with a built-in ability to withstand large crushing forces.  The latter can be achieved by resorting to particular binder additions which confer structural integrity to the ball or pellet after a specified curing period.  The ability to form strong pellets then enables metallurgical recovery of finely disseminated metallurgical values by subsequent hydro or pyrometallurgical techniques.

## 6.2    Procedure

Homogenise the sample, and determine and record its chemistry, size analysis and (when necessary and possible) the specific surface area.

Where applicable add necessary quantity of binder e.g. Portland cement, lime or bentonite.

For a standard column leaching charge of 25kg, weigh out separately 3 kg for seed ball preparation.  Normally, you would require around 10% of overall charge.

Clean inner surface of balling drum and moisten very lightly.  It may be desirable to precoat inner surface of drum with a thin layer of material being agglomerated.

Start rotation of drum and add small quantities of ore in 50 gram amounts to the drum.  With a water spray bottle, ensure a fine adequate wetting of the ore surface.  The precise amount of water addition will be arrived at after some practice.

When sufficient seed balls have formed empty them onto a nest of sieves such that seed-balls only in the range 0.45 to 0.55 mm are accumulated.  The minus 0.45 mm balls will be re-fed into the drum, along with fresh fine ore.  The object of restricting seed-ball size range is to ensure a small size spectrum with regard to finished green balls.

Maximise build-up of seed-balls in the above size range.  Divide the rest of the charge to be agglomerated into say 3 batches and equivalently the number of seed-balls.

Introduce seed-balls into the balling drum and allow to cascade in drum.  Wet surface lightly with spray.

Start stop-watch and attempt to add fine ore as quickly as possible over a 3 minute time span maintaining a fine water spray addition.

Complete ball growth utilising all of the available fines which have occurred by now.

Allow rotation of green balls for a further 90 secs and then spread out prepared balls onto a plastic sheet for drying.

Set aside samples of green balls for moisture, green compression and dry compression testing.

# 7    HEAVY LIQUID/HEAVY MEDIA TESTING

## 7.1    Introduction

Heavy medium separation is applied to the preconcentration of minerals – mainly the production of a high weight, low assay product, which may be rejected as waste.

In principle it is the simplest of all gravity processes and is a standard laboratory method for separating minerals of different specific gravity.  Fluids of suitable density are used so that the minerals lighter than the fluid float and those denser sink (as shown in Figure 7a.)
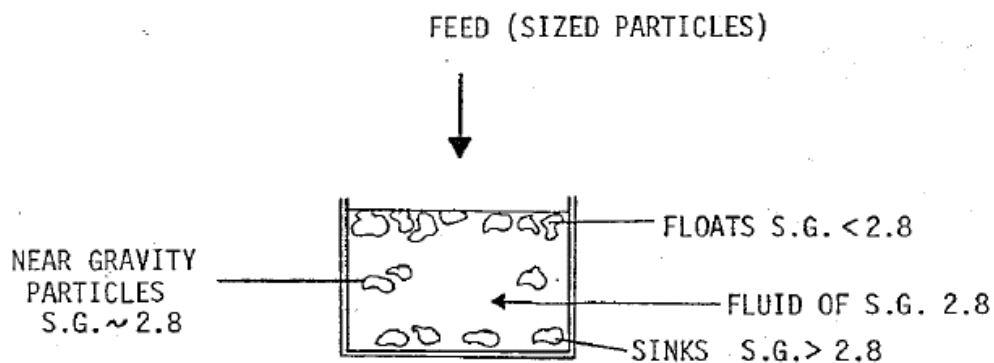


Figure 7 a   Principle of Heavy Medium Separation

### 7.1.1 The Heavy Medium

Heavy liquids are used in laboratories on small samples (50g to 10 kg) at specific gravities ranging from 0.80 to 4.00.

| SG | Liquid Used (S.G.) | |
|---|---|---|
| 0.80 – 1.60 | Benzene | (< 1) |
| 1.60 – 2.89 | Bromoform | (2.89) |
| 2.40 – 2.96 | Tetrabromoethane | (2.96) |
| 2.96 – 3.31 | Methylene iodide | (3.31) |
| 3.31 – 4.00 | Clerici's Solution | (4.00) |

The low S.G. range of liquids are used for coal/ash samples, while most mineral samples are separated in the range 2.50 to 4.0.

[Suspensions of ground material (known as media – usually magnetite, S.G. 5.1, and/or ferrosilicon, S.G. 6.8) are used on larger scale laboratory samples and in industrial applications.]

### 7.1.2 Laboratory Heavy Liquid Analysis

**Sample Preparation**

Closely sized samples are required for optimum separation. The ore sample to be tested is crushed to the required topsize and screened at at appropriate sizes (we and dry screening is carried out at 38-75 microns if these sizes are to be examined). A typical C.M.S. size distribution on crushed drill core would be 2440, 850, 300, 106 and 38 microns from a topsize of 3350 microns.

**Warning**

ORGANIC HEAVY LIQUIDS ARE TOXIC. INHALATION OF FUMES IS EXTREMELY DANGEROUS AND A THE FUME CUPBOARD WITH DOWNDRAFT VENTILATION MUST ALWAYS BE USED. SKIN CONTACT MUST ALSO BE AVOIDED (USE RUBBER GLOVES). HOWEVER, IF THE SAFETY PRECAUTIONS ARE STRICTLY ADHERED TO, THE OPERATION IS QUITE SAFE.

## 7.2 Procedure For Heavy Liquid Testing

The general procedure is as follows. The sample is placed in the liquid of lowest specific gravity and is split into two fractions. The float fraction is set aside to be weighed and assayed. The sinks fraction is washed in a suitable solvent, dried and placed in the liquid of

a higher specific gravity.  This process is continued in steps of suitable S.G. differences until a small final sinks fraction is obtained.  This is also dried, weighed and assayed.
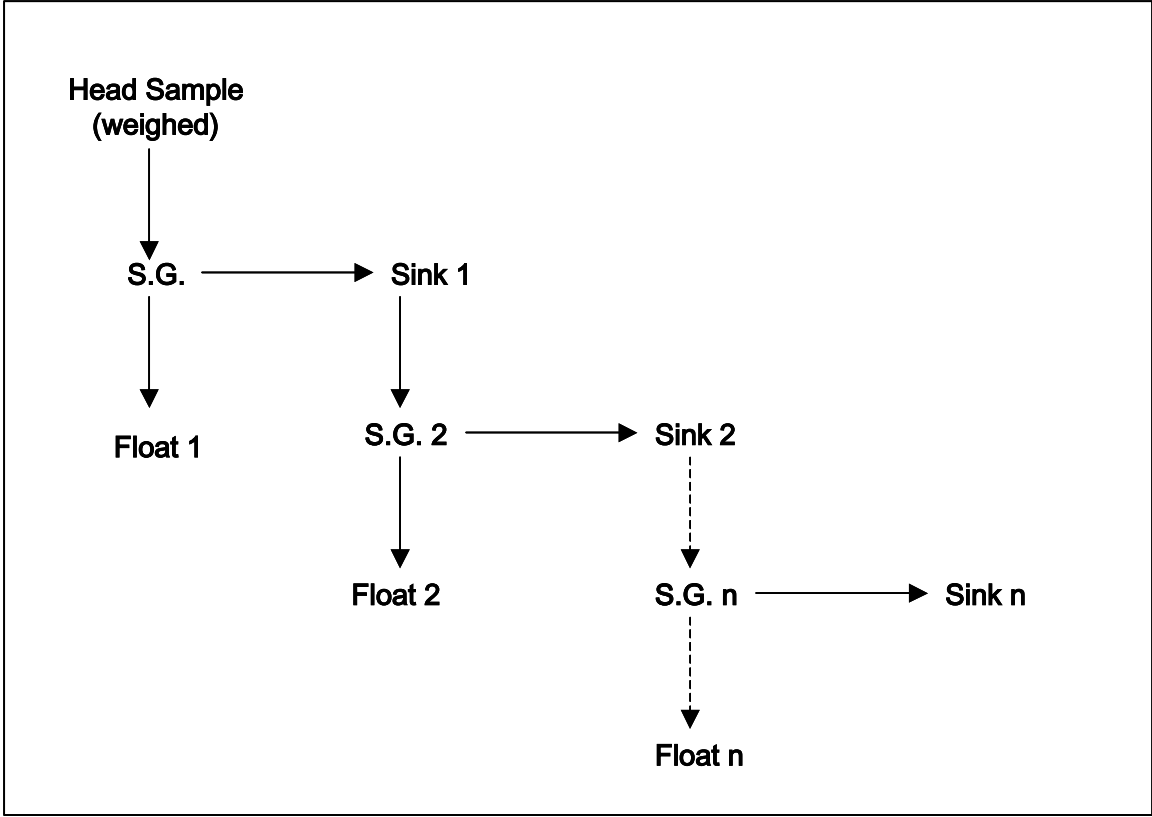


## 7.3    Results

After assaying the fractions for metal(s) content, the distribution of material and metal in the density fractions of the sample can be tabulated.  Table 1. shows such a tabulation:

| Size Fraction | Specific Gravity Fraction | % W.T. | Cum. % W.T. | % Sn | % Sn Dist. | Cum. Dist. % Sn |
|---|---|---|---|---|---|---|
| -850 micron +300 micron | -2.55 | 1.57 | 1.57 | 0.003 | 0.04 | 0.04 |
| | 2.55-2.60 | 9.22 | 10.79 | 0.04 | 0.33 | 0.37 |
| | 2.60-2.65 | 26.11 | 36.90 | 0.04 | 0.93 | 1.30 |
| | 2.65-2.70 | 19.67 | 56.57 | 0.04 | 0.70 | 2.00 |
| | 2.70-2.75 | 11.91 | 68.48 | 0.17 | 1.81 | 3.81 |
| | | | | | | |
| | 2.75-2.80 | 10.92 | 79.40 | 0.34 | 3.32 | 7.13 |
| | 2.80-2.85 | 7.87 | 87.27 | 0.37 | 2.60 | 9.73 |
| | 2.85-2.90 | 2.55 | 89.82 | 1.30 | 2.96 | 12.69 |
| | +2.90 | 10.18 | 100.00 | 9.60 | 87.31 | 100.00 |

*Table 1:  Heavy Liquid Analysis of a Tin Ore (after Wills)*

### 7.3.1 The Tromp Curve

As may be seen from the table, a separation at S.G. 2.75 would result in a float product of 68.48% of the weight contain 3.81% of the tin of that size fraction.  Laboratory separations in heavy liquids are "ideal" – all the sinks should report to the sinks and all the floats to the floats.  In industrial applications heavy media is used (rather than the organic heavy liquids) and inefficiencies in the separation are seen (i.e. sinks misreporting into floats and vice versa).  This is because particles with the same or near S.G. as that of the media have an equal chance of reporting to either fraction.  The efficiency of separation depends on the ability to separate material of s.G. close to that of the medium.  This can be represented by a Tromp or Partition curve, which relates to the partition coefficient (i.e. the percentage of feed material of a particular S.G. which reports to the sinks (or floats) product plotted against S.G.).  An example is shown in Figure 7b.
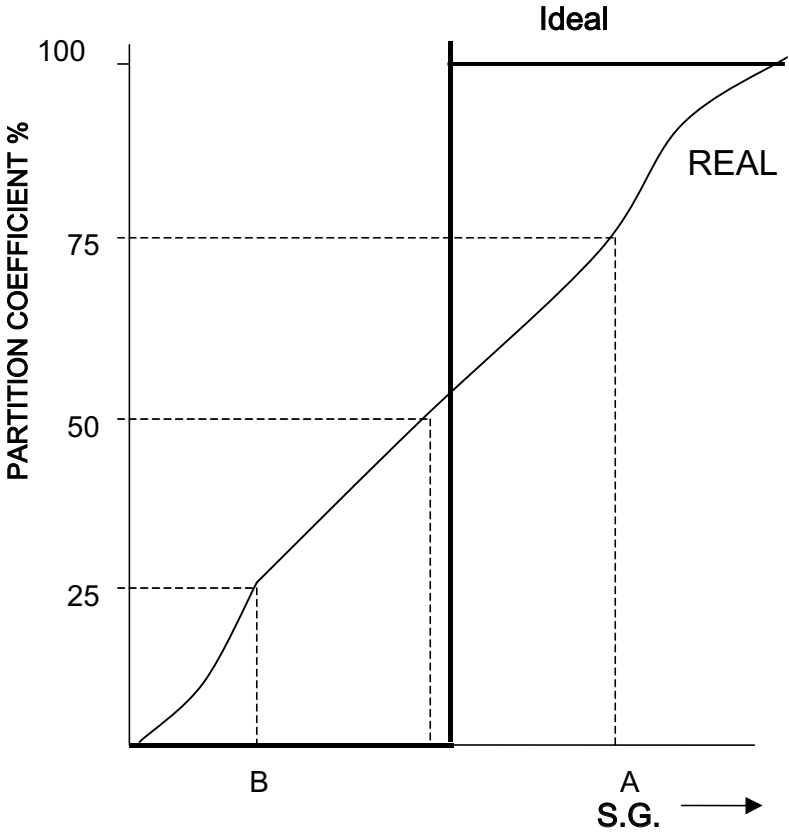


**Figure 7b:  Tromp or Partition Curve**

The ideal (heavy liquid) separation shows that all particles heavier than the separating density sink, and all those that are lighter, float.  The error of separation (known as the Ecart probable – Ep) is defined:

$$Ep = \frac{A - B}{2}$$

As can be seen the lower the Ep the more  idealised is the separation.  An ideal separation has an Ep of 0.0, while most practical Ep's are in the range 0.02-0.08.  (Full details of the construction of Tromp curves are given in Wills [1] pgs 267-274).

Testwork of this type can therefore tell:

        a)     The applicability of preconcentration to a particular ore, and also estimates of actual plant performance.

        b)     The efficiency of H.M.S. units in operation.

### 7.3.2    Washability Curves

Heavy liquid data can also be interpreted by washability curves.  To plot these curves, the axes shown on Fig. 7c are used.

**Figure 7c**
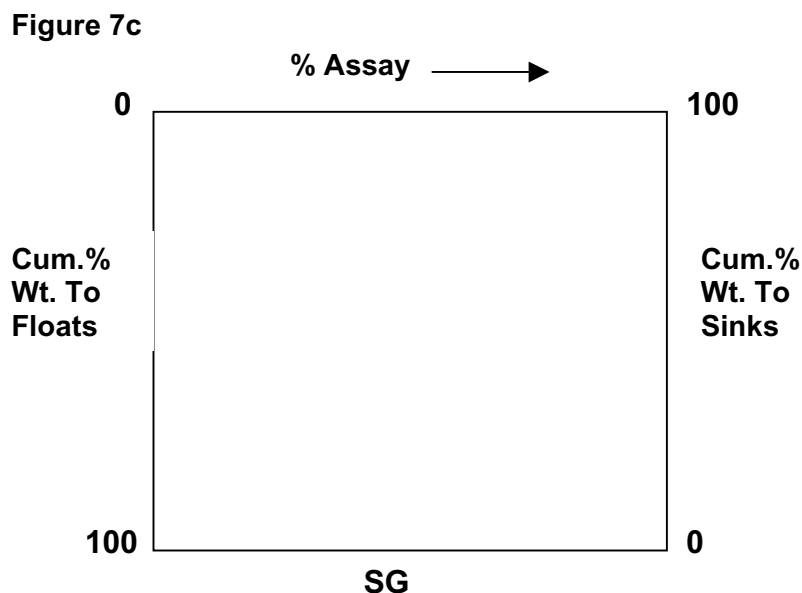


Specific Gravity Curve

The cumulative % wt. Floats or sinks is plotted against S.G.

If the material consists of two minerals completely liberated from each other, the curve will be a stepped line.  The width of the step indicates the S.G. difference between the minerals and

the height of the steps the relative amounts of the minerals present.  In practice, the minerals will not be completely liberated and a stepped curve will be obtained.

**Figure 7d**



Figure 7d (i)  (Liberated)
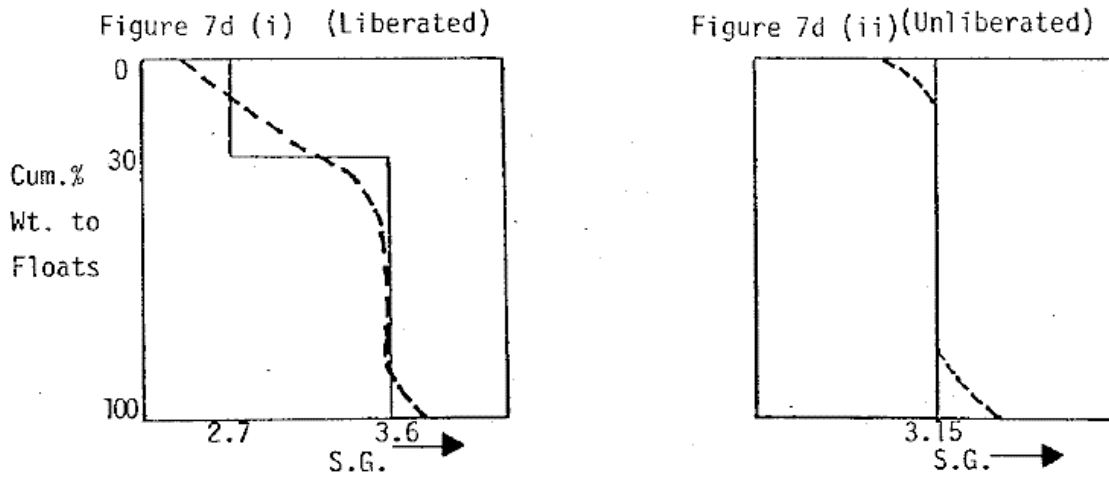
Figure 7d (ii)(Unliberated)

Figure 7d refers to an ore comprising
- 30% dunite (S.G. 2.7) – S.G. 3.15
- 70% chromite (S.G. 3.6)

Figure 7d(i) shows the curves obtained for completely liberated (solid line) and almost liberated (broken line) material.

Figure 7d(ii) shows the curves obtained for completely unliberated (solid line) and very poorly liberated (broken line) material.

S.G. values lower and higher than the theoretical minimum and maximum will often be otained due to the presence of closed pores and the inclusion of heavier minerals in the material.

The S.G. curve should not be used alone to indicate the ease or difficulty of separation because the width of the step depends on both the liberation of and the S.G. difference between the minerals.  The curve is also difficult to interpret when a mineral with two S.G. phases is present.

### 7.3.2.1  The Cumulative  % Assay of Floats or Sinks Curve

The cumulative % assay of the floats or sinks is plotted against the cumulative % weight of the floats or sinks.

**Figure 7e**
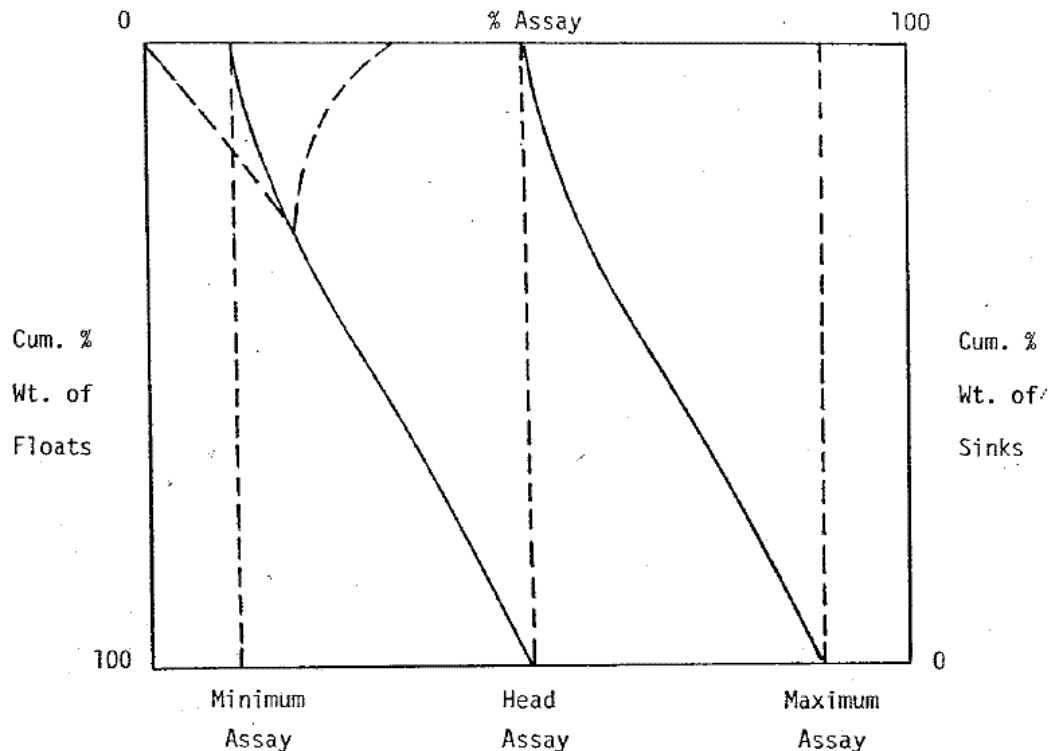


These curves indicate cumulative assay that the cumulative floats or sinks will have at a particular S.G. of separation.  They are used in conjunction with the S.G. curve and can thus predict the weights and assays of the two products of separation at any S.G.  Conversely the S.G. of separation can be found for a desired of concentrate or tails.

### 7.3.2.2  Cumulative Assay of Sinks Curve

At an S.G. lower than that of the lightest mineral, all the material sinks.  Therefore at 100% inks, the cumulative % assay of the sinks will equal the head assay.  As the cumulative weight of sinks tends to zero, their assay will tend to that of the richest particle which, if any of the particles are completely liberated, will equal the assay of the heavy mineral.

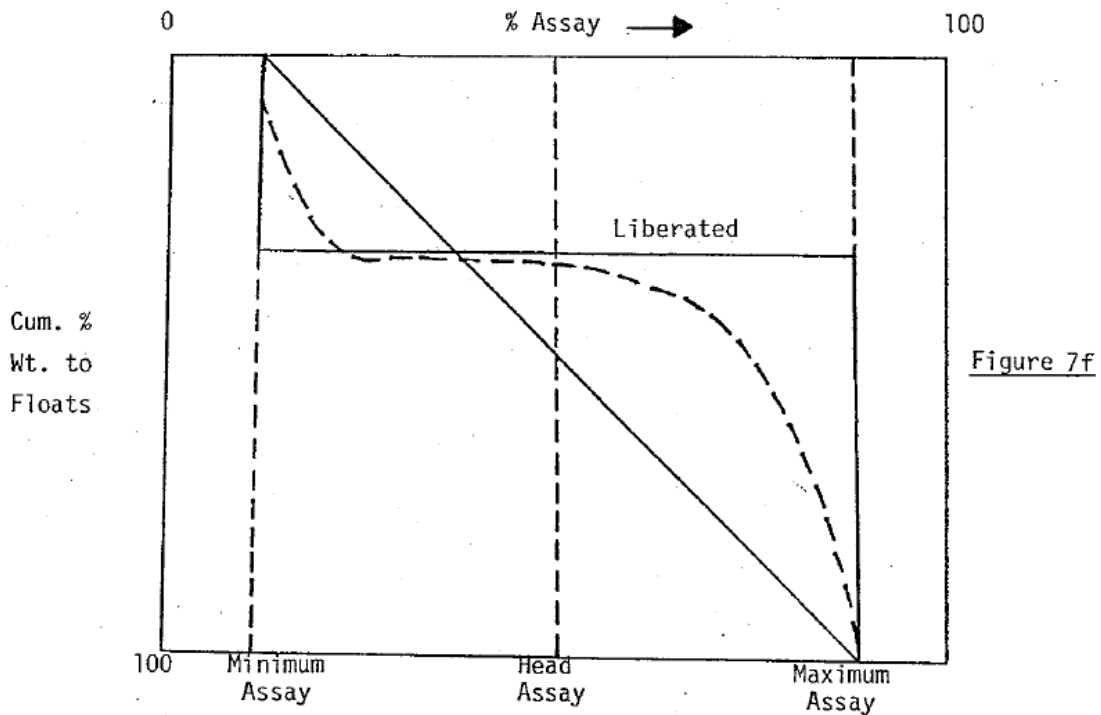### 7.3.2.3  Cumulative Assay of Floats Curve

At an S.G. greater than that of the densest component, the assay of the cumulative floats will equal the head assay.  As the weight of the floats tends to zero, the cumulative assay will tend to the lowest possible assay unless the valuable component is present in both low and

high S.G. forms.  The minimum assay will equal zero only if the light mineral exists in a completely liberated form.

### 7.3.2.4  The Characteristic or Elementary Assay Curve

The incremental assays obtained for each S.G. are plotted against the average cumulative % weight of sinks and floats.

**Figure 7f**



Figure 7f

This curve shows the assay of the richest particles in a float or sink.  If it is used in conjunction with the cumulative assay of floats or sinks curve, it indicates the spread of assay values obtained in a given product.

A material that contains equal amounts of all possible grades of particle will be shown by a straight line joining the maximum assay at zero sinks to the minimum assay at zero floats.

If the feed material is completely homogeneous, that is with no mineral liberation at all, the minimum and maximum assays will tend to the head assay and the characteristic Assay curve will be a vertical line at the head assay.

In practice the curve will have a reclining S shape.

- Clear inflexion points with a wide flat mid-section indicates the absence of middlings with the possibility of easy clean separation.

- Poor or indistinguishable inflexion points with a large slope in the mid-section indicates the presence of middling and a difficult separation.

In general, the steeper the curve, the more difficult is the separation at that point.

### 7.3.2.5 Specific Gravity Distribution (Tolerance) Curves

The three types of curve described so far completely define the behaviour of the material in a heavy liquid under ideal sink-float conditions.

In practice, we wish to use the data to predict the performance of a commercial process such as D.M.S., Jigs, Tables, etc.  In these processes, the S.G. of separation will vary between certain limits about a mean S.G. due to fluctuations in operation conditions.

This variation is expected to be:

| | |
|---|---|
| D.M.S. | ± 0.05 to 0.02 units, depending on control |
| Jigs, Tables | ± 0.10 to 0.15 units |

The accuracy of the split , in terms of the mean and characteristic assays of the products, will depend on the weight of material sinking or floating when the separating S.G. is varied about its mean value during separation.

To enable this to be read off the curves directly, it is usual to plot the S.G. Distribution curve. This is related to the slope of the S.G. curve and is calculated simply by finding the difference in the cumulative % weight floating or sinking between the stated tolerance limits for each mean separating S.G.  These differences are plotted on the cumulative % weight of sinks or float ordinates against mean S.G.

For coal, a table has been built up by experience to give the ease of separation for given weight percentages or material sinking within  ± 0.10 of the mean S.G. of separation.

| WT % Sinking within ± 0.10 of mean S.G. | Difficulty of Separation | Process | Type |
|---|---|---|---|
| 0-7 | Simple | Any high tonnage | D.M.S. JIGS TABLES |
| 7-10 | Fairly difficult | Efficient process – high tonnage | |
| 10-15 | Difficult | Efficient, medium tonnage.  Good operation | |
| 15-25 | Very difficult | Low tonnage Very efficient Expert operation | D.M.S |

The curve with two maxima will be formed in general, the area of interest being the portion from one of the maxima to the plateau region.

**Figure 7g**
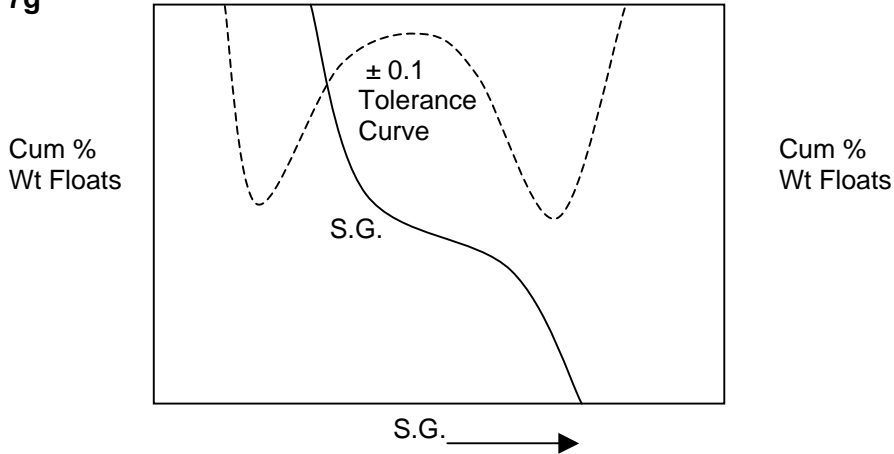


## 7.4    Heavy Media Analysis

Where large samples (both in topsize and weight) have to be examined it is not practical to use heavy liquids (being both expensive and time consuming).  A laboratory heavy media separator may be used which simulates the action of a heavy liquid bath.  Figure 7h schematically shows an Ericsson cone separator.  Sized fractions of particles in the range of 6-50 mm.



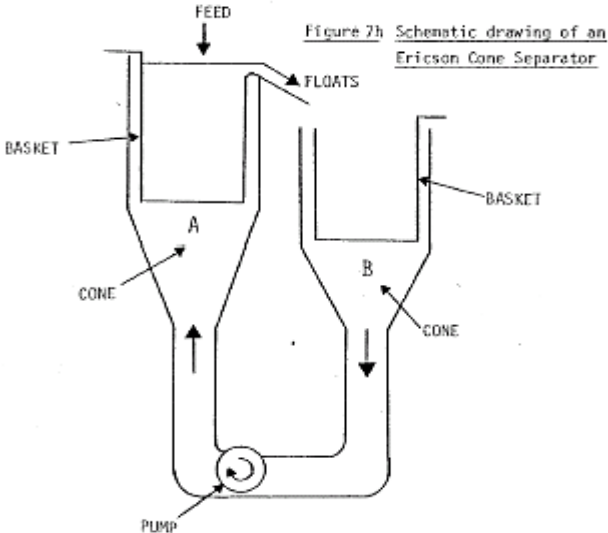**Figure 7h**

The media passes up through cone A and overflows into cone B.  Baskets are filled into the cylindrical sections of the cones and the feed put into cone A.  The floats are carried up and over into the basket in cone B while the sinks remain in the basket in cone A.  A similar analysis to the laboratory heavy liquid analysis is performed, at several media S.G.'s and particle size ranges.

# 8    MAGNETIC SEPARATION

## 8.1    Introduction

Magnetic separation  is a process used to separate materials from those that are less or non-magnetic.  All materials have a response when placed in a magnetic field, although with most, the effect is too slight to be detected.  The few materials that are strongly affected (magnetised) by magnetic fields are known as "Ferromagnetics", those lesser (though noticeably) affected are known as "Paramagnetics".

Ferromagnetics require relatively weak magnetic fields to be attracted and devices to sperate these materials usually have magnets that are permanently magnetised ("Permanent" magnets do not require electricity to maintain their magnetic fields).  Paramagnetics require stronger magnetic fields and these can only be achieved and maintained by  "electro" magnets (large wire coils around an iron frame – current is continuously passed through the coils creating the magnetic field within the iron.  The field is concentrated across an air gap in the circuit).

Both ferromagnetic (low intensity) and paramagnetic (high intensity) separation devices may be operated with dry solids or with solids in pulp form.  (A complete classification of magnetic separating devices is given in Wills "Mineral Processing Technology", pp. 338-356).

Magnetic separation has two major applications in mineral processing plants:

a)   The removal of tramp iron (which would deleteriously affect subsequent processes) from an ore stream.  This is usually achieved by a low intensity magnet suspended above, or at the head of, a conveyor.

b)   Separation of magnetic minerals from less magnetic minerals Table 8.1 shows the more common magnetic minerals and the range of field intensities that may be required to separate them.

**Table 8.1   Some Common Magnetic Minerals**

| | Mineral | Formula | Field Strength* |
|---|---|---|---|
| **Ferromagnetic** | Magnetic | $Fe_3O_4$ | ⟶ 1 |
| | Pyrrhotite | $Fe_7S_8$ | 0.5  - 4 |
| **Paramagnetic** | Ilmenite | $FeTiO_3$ | 8 – 16 |
| | Siderite | $FeCO_3$ | 9 – 18 |
| | Chromite | $FeCr_2O_4$ | 10 – 16 |
| | Hematite | $Fe_2O_3$ | 12 – 18 |
| | Wolframite | $(Fe,Mn)WO_4$ | 12 – 18 |
| | Tourmaline | | 16 – 20 |
| | | | |

(*The units given are kilogauss (kG) – C.G.S. system.  These are the units most commonly used.  The equivalent S.I. unit is the Tesla (T) * 1 Tesla = 10 kilogauss)

The extremes of field strength used are based on experience from a magnetic separation testing laboratory over many years.

As can be seen from the table, minerals from different deposits may have varying magnetic properties.


## 8.2     Laboratory Magnetic Separator

The laboratory separator is a high intensity dry device which uses a rotating iron disc to concentrate the magnetic field and remove the magnetic particles from the non-magnetic.  The separator is shown in Figure 8a.  It essentially consists of an electromagnet (the applied current is controlled by a rheostat) which produces a magnetic field across the airgap and a belt which passes through the air gap.  Mineral particles are fed from a vibrating feed onto the belt and through the magnetic field.  The magnetic particles are attracted to the disc above the belt and from there are removed from the field by the disc above the belt and from there are removed from the field by the disc's rotation – they fall from the disc and are collected at the side of the belt.  The non-magnetic particles are collected at the end of the belt.


## 8.3     Procedure for Using the Laboratory Separator

With reference to Figure 8a (***missing***):

a)   The machine should be thoroughly cleaned with compressed air, and the product collection trays placed in position.

b)   The sample to be tested should be closely sized (e.g. 425-300 microns, 75-45 microns) and Ferromagnetic particles removed prior to separation (a hand magnet inside a plastic bag may be used for this).

c)   Place the sample in the feed hopper.

d)   The air gap between the disc and the belt should be set to approximately treble that of the largest particle in the sample (NOTE:  The air gap cannot be adjusted when the current is on).

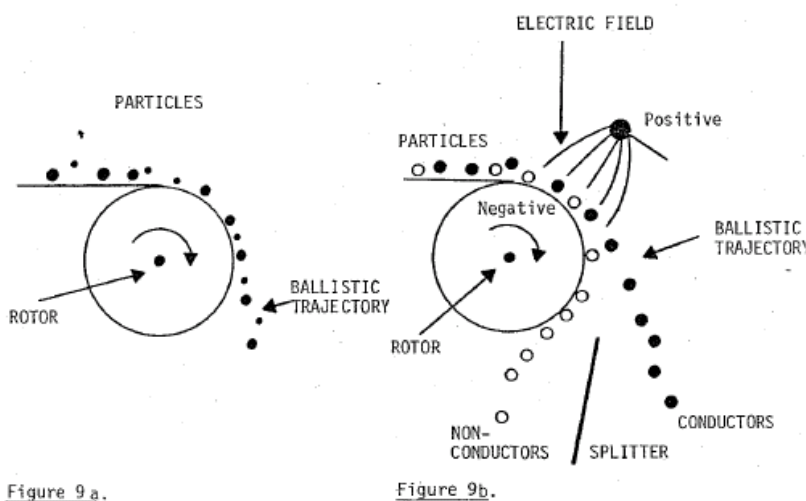e)   Press the mains "START" button and turn on the three switches (feeder, magnet and belt disc drive).

f)  Slowly increase the current to the required level (indicated on the ammeter) by turning the current rheostat – some experimentation may be necessary to determine the amps required (NOTE:  reduce current to zero <u>before</u> switching the magnet off).

g)  Slowly start the vibrating feeder and adjust such that a <u>single</u> layer of particles is distributed over the belt (periodic adjustment may be necessary).

h)  Once optimum separation is achieved, switch off the feeder, recombine the products in the feed hopper and commence separation.

i)  When the sample has run through, collect, weigh, label and bag the products.

j)  Repass the non-magnetics at a higher current if required.

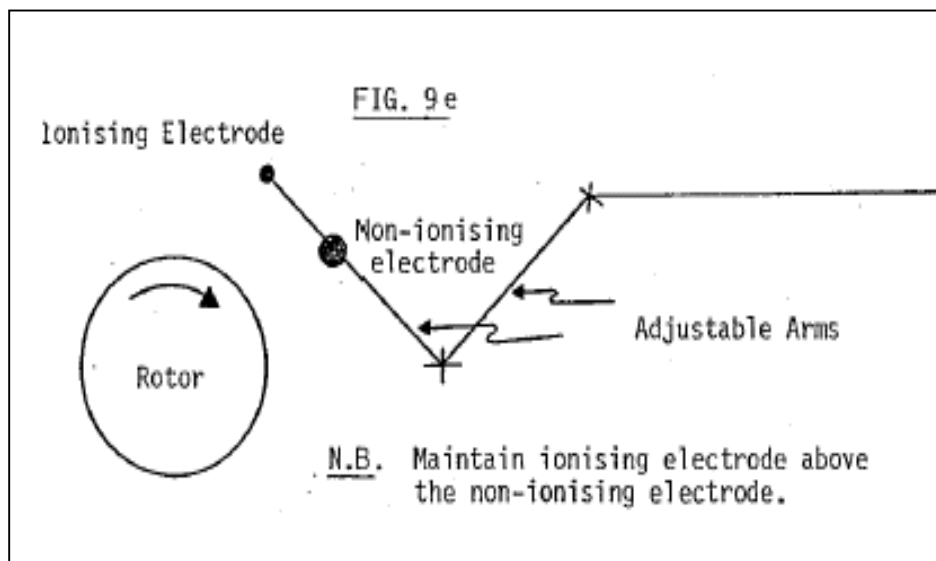k)  Reduce current to zero, turn off all switches, clean the machine thoroughly


# 9       INTRODUCTION

## 9.1    Electrostatic Separation

Separation of minerals utilizing differences in their electrical properties may be carried out using an electrostatic separator.  It will separate electrical conductors (minerals such as cassiterite and rutile) from those that are electrical non-conductors (minerals such as zircon and siderite).  The principle of separation is that as the particles to be treated are passed through an intense electric field each particle acquires a charge.  The conductors pass on their charge when emerging from the field while the non-conductors retain theirs momentarily.

Separation is effected by passing the particles on to a rotating metal cylinder (the roll); without any external forces all the particles will follow a ballistics trajectory when leaving the roll surface, as shown in figure 9a.



Figure 9 a.                Figure 9b.

FIG. 9 c

H.T. LOAD

0    A    5
~

H.T. VOLTS

0    K V    50

+
K W

OFF          Overload
O            X
ON           Press Reset
Filament

H.T. ON      OFF
O            X
             ON
High    Tension



FIG. 9 d

X Rotor Heater
ON/OFF Switch

350
+
RPM

0        700

Rotor Speed
D.C. Motor

O
Motor
Fuse

X
ON/OFF
A.C. Supply

O
Vibrator
Control



FIG. 9 e

Ionising Electrode

Non-ionising
electrode

Adjustable Arms

Rotor

N.B. Maintain ionising electrode above
the non-ionising electrode.

In the presence of an electric field, the rotor becomes negatively charged, while the particles are positively charged. The non-conducting particles retain these charges and are "attracted" (pinned) to the roll while the conducting particles lose their charges (to the roll) and follow the normal projectory as shown in figure 9b. Separation is affected by (adjustable) splitter plates.

**Laboratory machine**

This manufactured by Readings. There are two control panels –

a) the high tension (electric field control unit and
b) the rotor speed vibrating feeder control unit. The are schematically shown in figures 9c and 9d respectively. Relative humidity is an important operating parameter (moisture inhibits separation), and an infra-red lamp is positioned above the rotor to remove surface moisture from the feed particles and to maintain the rotor surface at a constant temperature.

**Procedure**

- **Sample Preparation**
  Particles to be closely sized as in magnetic separation.

- **Safety**
  The normal operating voltage of the machine is 30,000 volts (D.C.); consequently strict adherence to the safety procedure as indicated below is imperative.

  a. ensure machine off
  b. Clear machine with compressed air
  c. Adjust setting of electrodes as required
  d. Tighten nuts (subsequent vibration may undo them)
  e. Place protective view cover on
  f. Switch on power, rotor and heat lamp – leave on low speed for about 15 minutes before increasing to desired r.p.m. (normal range of 300-600 r.p.m.)
  g. Make sure K.V. knob is zero, switch on filament with key, and then switch on high tension switch – the light will indicate if it is on (figure 9b). Allow about 10 minutes for filament to warm up.
  h. Gradually increase voltage to that required. (N.B. At this point "arcing" may occur. This will be because of
     - High moisture content – check infra red lamp is on
     - Incorrect electrode setting (electrodes too close)
     - Too high a voltage
  i. Place feed (size appropriately and preheated under lamps) into hopper, and turn feeder on. Adjust feed rate such that a mono layer of particles are fed onto the rotor, and adjust splitter plates to produce the products required. Complete the separation.

On completion of testwork the different sections of the machine are switched off the following sequences:

> a) vibrating feeder
> b) high tension volts (gradually reduce to zero)
> c) high tension switch (light off)
> d) filament (use key)
> e) infra-red lamp
> f) rotor
> g) power supply
> n.b. clean machine thoroughly

**Mineral Separations using Electrostatic Separators**

In practice electrostatic separators will always be tested in several stages, as one pass separations producing clean products are rare. Inherent physical properties of mineral particles (specific gravity, grain size and composites, surface coating) and their interrelationships all affect the ease of separation. In addition the number of operating variables (temperature, voltage, rotor speed and electrode configuration) make a number of trials necessary to determine optimum conditions for separation.

**Additional Bibliography**

1. Readings 270 mm High Tention Separator Manual
2. Wills, B.A. "Mineral Processing Technology", 1979 (and late editions)
3. MacDonald, E.A. "Manual of Beach Mining Practice", 1973
4. http://www.carpco.com

# 10    GRAVITY SEPARATION / RELEASE ANALYSIS

## PART 1  SUPERPANNER

### 10.1    Purpose

Gravity separation of heavy minerals in the range 600 microns to 12 microns.

### 10.2    Principle

To simulate gravity performance of a given sample. Recovery and grade achieved are normally higher than in plant operation, but analyses of grade/recovery curves permits relative assessment of performance.

### 10.3    Equipment

The superpanner itself is a completely integrated machine which only needs suction lines to draw off concentrate tailings. The superpanner can simulate the hard knocking action of a shaking table or the gentle washing of a panner.

## 10.4 Sample Preparation

Individual sized fractions:

| Micron | |
|--------|------|
| - 600 | +212 |
| -212 | +106 |
| -106 | +75 |
| -75 | +38 |
| -38 | +12 |

The –38 + 12 micron fraction is obtained through cyclosizing.

## 10.5 Procedure

A 50 gram maximum of sample is recommended for the U-shaped deck of the superpanner. The superpanner itself has the ability to rock sideways and tilt in the horizontal. The knock that is applied to the end of the deck is like all other motions in that the degree of motion needed can be varied. The speed of the motion sideways and the interval of the knock can also be controlled. It is very important that water additions throughout the separation are such that the solids are kept in solution and that dry beds do not appear, thus hindering separation. Normally the deck starts in the horizontal with a hard fast knock and no side motion. It is hoped by this arrangement that the heavy mineral will fall through the sample onto the bottom of the deck (much like a jig).

After a period of time the knock is taken off and the deck angled towards the tail pipe and a gentle sideways motion is applied to remove the gangue from the top of the heavy mineral.

Once the heavy mineral is separated from the remainder of the sample it can collected into the concentrate holding bottle by means of the vacuum system. Tailing are normally taken off throughout the separation and re-superpanned several times. This enables production of a rougher concentrate with the final tail free of liberated cassiterite. The rougher concentrate can then be put back on the deck and a super concentrate of the highest possible grade removed (minimum weight – approxiametly 0.2g). Subsequently the remaining rougher concentrate may be re-superpanned and spread out along the deck of the superpanner where it can be split into three separate concentrates depending on S.G. (that is the proximity to the concentrate end of the deck). In the finer fractions it should be possible to attain the liberation size of the heavy mineral and recovery should hence be high. In the coarser fractions, however, high recovery is not always obtained because of unliberated cassiterite.

Note: High recovery and grade can only be achieved if the heavy mineral is liberated. The ability to quickly visually identify the heavy mineral and considerable operator patience are important attributes for high recovery.

## *PART II – MOZLEY SEPARATOR*

The separator enables release analysis of mineral grains of close specific gravity in the size range below 2 mm and can give additional data to heavy liquid results in the higher S.G. ranges.

It consists of a V profile stainless steel tray with a $1^o$ to $3^o$ slope, which can be made to oscillate with a horizontal amplitude of adjustable from 2 to 4 inces and a longitudinal amplitude of ¼ ".   An adjustable end knock action ;is also provided.

An irrigation pipe is attached to the tray's perimeter and a concentrate wash water pipe is situated centrally at the feed end of the tray.

Adjustments are provided for leveling, slope setting and a control panel houses a switch timer, water valves and rotameter.

The separator also has an interchangeable stainless steel tray which is flat and which can be used to predict slime table performance on ultra-fine materials.

After the wetted sample has been added to the tray the cyclic motion stabilizes the mineral particles and stratification takes place.  Heavy (usually valuable) mineral sinks to the tray surface and is moved "upstream" by the end knock action, "whereas the lighter (gangue) is carried downstream by the flow of the irrigation water to discharge via a tailings launder.

Interpretation may be done visually, microscopically or by assay analysis of the separator products.   These can be divided into middlings and concentrate, separately collected. Approximately 150 grams of sample may be used per test run.

Once established, test run parameters should not be varied for reproductibility to allow comparisons to be made between samples.


# 11     LEACHING

## 11.1     Theoretcial Overview

### 11.1.1   Leaching of Minerals

Leaching is the process of extracting a soluble constituent from a solid by means of a solvent.  In extraction metallurgy, it is the process of dissolving a certain mineral (or minerals) from an ore or a concentrate, or dissolving certain constituents from metallurgical poroducts such as a calcines, mattes, scrap alloys, anodic slimes, etc., to achieve either one or two purposes:

- Opening of ores, concentrates or metallurgical products to recover the metal values.
- Leaching easily soluble constituents (usually gangue minerals) in an ore or concentrate in order to have it in a more concentrated form, e.g. leaching of tungsten flotation concentrate with hydrochloric acid to dissolve away calcite and apatite.

Leaching is a heterogeneous process, involving three reaction steps:

- Diffusion of solvent through the pores in the solid particles, and of the dissolved substance outwards after solution.
- Chemical solution at the reaction site in the particle.
- Transport of the dissolved substance in the solvent away from the particle surface.

Factors controlling the rates of the above steps are temperature, agitation, concentration of leachant, particle size, leaching time and pH.

## 11.1.2   Leaching of Metals

Commonly leached metals could arise from any of the following:

1)        Naturally occurring metals in the native state, e.g. Au, Ag, Platinum metals, Cu.
2)        Produced as a by-product of a metallurgical process, e.g. Au, Ag, Pt, Se.
3)        Produced by metal oxide reduction, e.g. Cu and Ni.

Gold and Silver Leaching

For Gold and Silver, the most common leachants used are Na or K cyanides in the presence of oxygen, Thiourea ($NH_2$. CS.$NH_2$), also in the presence of oxygen, or Ammonium polysulphide ($NH_4$) $_2$Sx, especially for Gold.

Optimum physical conditions for cyanidation are:

1)  Maximum temperature of $85^oC$,
2)  Operating ph range of 9.5 – 11.5
3)  A (CN)/($^O2$) molar ratio of between 4.6 and 7.4

Minerals characteristically associated with Au and Ag are commonly pyrite, galena, sphalerite, arsenopyrite, stibnite, pyrrhotite and chalcopyrite and occasionally various selenium minerals, magnetite and uraninite.  Carbonaceous matter, if present in Au ores usually cause high cyanide consumptions since they absorb gold cyanide complexe. Metallic minerals that dissolve in cyanide solution can have either an "accelerating" or "retarding" effect on cyanidation.

"Accelerating agents" are small amounts of Pb, Hg, Bi and Thallium salts, the rapid dissolution of gold in the presence of these ions being possibly due to alteration in the surface character of gold by alloying with the displaced metals.

"Retarding" effects may be due to any of the following:

a) Consumption of oxygen from solution, e.g. Pyrrhotite accompanying gold in its ores decomposes in an alkaline medium forming ferrous hydroxide ($Fe(OH)_2$) and sodium sulphide each of which oxidize readily, depleting $0_2$ levels in solution.

b) Formation of complex cyanides, eg. Cu, Zn and iron minerals may dissolve preferentially in cyanide solution, thereby depleting the solution of its cyanide content.

c) Formation of thiocyanate: the sulphide ion liberated when the sulphide mineral reacts with cyanide and oxygen forms a thiocyanate which has no action on Au.

d) Adsorption on gangue mineral: Gold ores containing aluminosilicates or other silicates when finely divided in an aqueous alkaline medium often form colloidal Silica and Alumina to which ferric hydroxides with a strong adsorptive capacity for sodium cyanide get attached.

e) Film formation on the surface of the metal
   - As a consequence of build-up of a thin layer of sulphide ions
   - Formation of a layer of calcium peroxide at pH > 11.5 or
   - Films of insoluble cyanide, e.g. $Pb(CN)_2$ or of flotation reagent like potassium ethyl xanthate.

The effects of retardants can be ameliorated somewhat by addition of Lead salts like lead oxide, nitrate or acetate which, as insoluble lead salts, eliminate the sulphide ions as soon as they are formed. Additions of small amounts of Potassium permanganate also help eliminate difficulties due to sulphides by apparently oxidizing the sulphide ion to sulphate. Additionally, agitating the ore pulp in an alkaline medium helps decompose sulphide minerals, after which the solution is discarded. The sulphide-free pulp is then subjected to cyanidation.

Native Copper and Nickel containing ores are generally leached in aqueous ammonia solutions, important parameters being ammonium ion concentration and Oxygen partial pressure.

### 11.1.3  Leaching of Oxides

a) Cassiterite

Natural cassiterite on account of its crystalline nature is extremely difficult to dissolve in acids or alkalies  Artificial cassiterite on the other hand is amorphous and soluble in acids.  Cassiterite can easily be reduced however to Sn, which soluble in acids or alkalies.  Pressure leaching has

been applied to cassiterite concentrates using $Na_2S$ and NaOH solution at $400^0C$  Tin is recovered from the leach solution by exchange with Na amalgam whereby Sn enters the amalgam phase and sodium sulphide is regenerated.

b)   Wolfram and Scheelite:

Wolfram and Scheelite can be digested with excess concentrated Hydrochloric acid as follows; the digested mass being washed with water to remove iron and manganese chloride, the residue being then dissolved in hot ammonium hydroxide.  Ammonium wolframate is crystallized from the solution by evaporation.

$WO_3. FeO + 2HC1 \rightarrow FeC1_2 + H_2WO_4$
$WO_3. CaO + 2HC1 \rightarrow CaC1_2 + H_2WO_4$
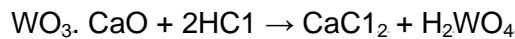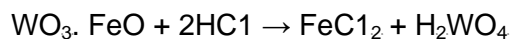
Leaching of wolframite with concentrated NaOH at high temperature in an autoclave yields a solution of sodium wolframate, while iron and manganese are precipitated as hydroxides:

$WO_3. FeO + 2NaOH \rightarrow Na_2WO_2 + Fe(OH)_2$
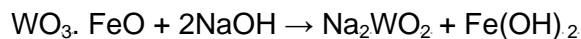
The solution of sodium wolframate is purified by precipitation with HC1:

$Na_2WO_4 + 2HC1 \rightarrow 2NaC1 + H_2WO_4$
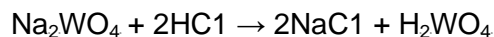
Alternatively, it is possible to leach calcium tungstate with a solution of $NH_4F$ and $NH_4OH$.  Tungsten goes into solution as $(NH_4)_2 WO_4$, while calcium is precipitated as $Ca F_2$.  Increasing the temperature, time or concentration of ammonium fluoride increases the rate of extraction. Tungsten from Scheelite can be extracted by leaching with excess sodium carbonate solution (30-50 g/1) at $200^oC$, the addition of small amounts of NaOH improving recovery.
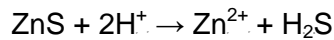
## 11.1.4   Leaching of Sulphides

Methods of leaching sulphide ores can be

1)   in absence of oxidizing agents
2)   in presence of oxidizing agents
3)   Bacterial leaching

11.1.4.1 Absence of oxidizing agents

a)  Acid leaching:
    Dilute acids dissolve some metal sulphides with liberation of $H_2S$, e.g.
    $$ZnS + 2H^+ \rightarrow Zn^{2+} + H_2S$$

    Three types of sulphides can be encountered.
    1)  Completely soluble sulphides:  Zns, CoS, NiS, FeS
    2)  Slightly soluble, e.g. $FeS_2$.
    3)  Insoluble:  PbS, CuS, $AS_2S_3$, $Sb_2S_3$, CaS

b)  Alkaili hydroxides:
    NaOH may be used to leach PbS and ZnS forming soluble plumbite
    ($Na_2PbO_2$) and zincate solutions respectively ($Na_2PbO_2$) respectively.

c)  Alkali sulphides:
    Sodium sulphide solution reacts with sulphides of arsenic, antimony, tin and
    mercury to form soluble thiosalts, additions of NaOH helping prevent
    hydrolysis of $Na_2S$.

d)  Alkali cyanides
    Sodium cyanide has a solvent action on sulphides.

e)  Double decomposition
    Copper sulphate can be used to separate Copper from Nickel in a sulphide
    ore or Copper from Fe in Cu –Fe sulphide ore.  $CuSO_4$ solution can also be
    used for removing Pb, Zn, and Fe from copper sulphide ores.

11.1.4.2 In presence of oxidizing agents:

a)  Ferric ion:
    Ferric sulphate or chloride react with metal sulphides liberating elemental
    sulphur, e.g.
    $$Zns + 2Fe^{3+} \rightarrow Zn^2 + 2Fe^{2+} + S$$
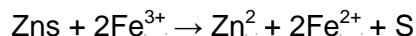
    And in the case of cuprous sulphide

    $$Cu_2S + 2Fe^{3+} \rightarrow CuS + Cu^{2+} + 2Fe^{2+}$$
    $$CuS + 2Fe^{3+} \rightarrow Cu^{2+} + 2Fe^{2+} + S$$
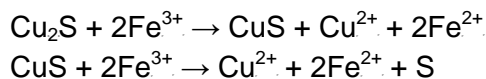
    During leaching with Ferric ion a critical balance between $Fe^{2+}$ and $Fe^{3+}$ ions
    in the leach solution is essential to maintain efficiency of leaching and
    subsequent electrolysis.  The ferric ion is also very sensitive to pH changes,
    hydrolysis and precipitation of basic ferric salts starting at pH 3.5.

b)  Chlorine water and hypochlorite:

Mixtures of hypochlorous and hydrochloric acid (the former a very powerful oxidizing agent) help leach sulphide ores, the overall reaction being

$$MS + C\ell1_2(aq) \rightarrow MC\ell_2(aq) + S$$
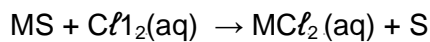
The technique can be used for Lead ($PbS$), copper ($Cu_2S$ and $CuS$), Iron ($FeS_2$ and $FeS$) and Molybdenum ($MoS_2$).

c) Nitric acid and Nitrates:

The oxidizing action of nitric acid and nitrates can be utilized to leach sulphides, but this is seldom adopted because of costs.

d) Concentrated sulphuric acid:

Most sulphides are oxidized to sulphate. From the sulphated ore, all of the zince, copper and iron can be leached with water while lead, silver, gold and antimony will remain in the insoluble residue.

## 11.2    Leaching Procedures

### 11.2.1   Performance of a Standard Bottle Leach Test

#### 11.2.1.1 Equipment Required

1) Winchester bottles, with tops
2) Plastic tape to seal tops of bottles
3) Flexible air lines for possible aeration requirements
4) Magnetic or mechanical stirrer for agitation
5) Timer, pH meter, thermometer and weighing device
6) Leachants, i.e. acid, alkaline or neutral solutions, or specific solutions
7) Filter:  vacuum or pressure
8) Titration systems

#### 11.2.1.2. Procedures

1) Dry the ore and crush it to minus 2380 pm (or as specified, otherwise).
2) Prepare the solution of required concentration (e.g. 1M $H_2SO_4$ requires 52 ml of con. $H_2SO_4$/litre of water).
3) Place 50 gm sample in the leach bottle and add 250 cc of prepared solution.
4) Allow leaching to take place for 1 hour under stirred conditions, ensuring the right conditions of pH, temperature, % solids, etc.

5) After leaching, discharge contents of leach vessel into clean pressure or vacuum filter without washing the vessel.
6) Filter pulp, and collect pregnant leach liquor undiluted.
7) Wash ad dry the residue and record it weight.
8) Analyse residues and pregnant liquors for relevant metal content.

### 11.2.1.3 Precautions

1) When handling concentrated acid, always wear eye protection, and always add acid to water. If water is added to concentrated sulphuric acid, the heat generated could vaporize the water and cause an explosion; by adding the acid to water, the heat is generated more slowly and is rapidly dissipated by the water.
2) Residues should be thoroughly washed of leaching reagents.

### 11.2.1.4 Calculations

a) $$\text{Acid consumption (mls)} = \frac{\text{dosage (g/t) x sample size (gms) x 1}}{\text{Specific gravity of acid x 445}}$$

b) Leaching recovery (R ) = $\frac{\text{head grade} - \text{residue grade}}{\text{head grade}} \times 100$
(%Metal Extraction)

c) Leach rate $= \frac{(\text{ original wt. } - \text{ final wt. })}{\text{time in sec x surface area}}$
(g/ time cm$^2$)

### 11.2.2   Bottle Leach Procedure for Cyanidation

1) Riffle out 400 grams of the ore.

2) Grind to pass 200 microns (˜13-15 minutes for hard rock ores;˜ 10 minutes for oxidized ore) grind at 35% solids.

3) Wet screen. Retain and dry fractions. Settling overnight of $-45\mu$ will be required. Decant and dry. Perform cyclosiing on $-45\mu$ fraction. Ensure to include $-45\mu$ material dry screened from remaining size fractions. (20 minutes on Ro-tap screen shaker).

4) Reconstitute sample – add to bottle along with same weight of 0.1% (1 gram/litre) NaCN solution, to give 50% solids. Adjust pH to 10.0 – 10.5, with measured lime additions.

5) Put bottle on rolls (with variable speed setting set at zero – this is sufficient agitation) – if using two bottles, place caps away from each other, and start timing.

6) Connect up air lines such that air is bubbled through solution.

7) When a sample of the pregnant cyanide solution is required, decant 100 mls of pulp, into a beaker. Return to the bottle any previous filtrate, along with 50 ml of cyanide solution (0.1%). Return bottle to rolls.

8) Filter pregnant solution from pulp, retaining and labeling the solution. 40-50 mls of pregnant solution is required.

9) When final sample (72 hours usually) has been taken, the residual cyanide must be determined. Procedure for this determination is outlined elsewhere.

10) Wash solids with warm water and dry, setting aside for final residue.

## Comments:

In lieu of cyanidation by bottle leaching on rolls, it may also be more convenient to bottle leach using magnetic stirrers with necessary air bubbled using flexible air lines.

Alternatively, a flotation cell could be utilized.

### 11.2.3   Column Leaching Procedure

1) Agglomerate ore if required. Cure for recommended time period. During this time the ore should be kept damp via a wet cloth spread over it.

2) Load ore into the column. This should be done with the column on an angle, and the ore poured gently in to prevent packing. The nylon mesh filter should be placed in position prior to filling the column.,

3) The column should be placed in position and cyanide solution made to concentration (usually 0.1% - ie. 1 gm/litre). Allow for 1 container (5 litres) of cyanide per day (plenty). The flow-rate from the top should be carefully adjusted to give a constant drip approximately 2 ml/min. Slightly more is acceptable. Periodic checks should be made to ensure an even flow rate, and also to check if percolation through the bed into the container below has actually occurred. This percolation time should be noted.

4) After 24 hours, and every 24 hours thereafter, the following procedure should be followed:

   i. The pH of the solution (underflow) should be taken. Appropriate adjustments should be made with line $(Ca(OH)_2)$ additions to the cyanide solution.

   ii. The feed flow rate can be measured by the volume loss incurred in the feed cyanide bottle. This should be reported as volume flow over an hourly period, say. The percolation flow rate can be measured by collecting an underflow sample over a 2 or

3 hourly period (around the 24 hour mark).  This should also be reported as volume flow/hour or ½.  In some circumstances this underflow may appear too erratic for the 2 hour period.  In this case, the volume flow over the past 24 hours would be more accurate.

5) When the test has been run for the appropriate number of days the bed should be flooded with hot water and left to percolate through.  The ore can be removed and spread upon sheet plastic to dry.

6) Once a day, the sample should be riffled down to several kg size, crushed and riffled down further to produce a sample for assay.

7) The pregnant cyanide solutions should also be sent for assay.

*Note*: *Take a final cyanide sample to determine residual cyanide by titration.*

### 11.2.4   Free Cyande Determination

1) Take a 10ml aliquot of pregant liquor.

2) Make aliquot to 60ml with distilled water.

3) Add 3 ml KI (10% solution)

4) Add 5 ml of 1.5% $NH_4OH$

5) Titrate with 0.10 N Ag $NO_3$
   then; $\underline{titre} = \%CN$
   $\quad\quad 100$

**Note:**

1. The endpoint is indicated by the first sign of a change from clear to slightly turbid conditions.  A black background helps to perceive this transformation, and a comparison with a sample at equivalence point is also useful.  If solution is already slightly turbid, a greed tinge will be noted at equivalence point.

2. **Reagent Preparation:**

   Distilled water used at all times
   Ag $NO_3$          -   originally N/50
   $\quad\quad\quad\quad\quad\quad$ -   dilute 1:1 to achieve N/100
   10% KI          -   10 grams KI per 100 ml $H_2O$
   1.5% $NH_4OH$   -   1.5 mls Ammonia per 100 ml $H_2O$

Ag $NO_3$ should be kept in a <u>dark</u> colored bottle, away from light.

# 12    FLOTATION

## 12.1    Theoretical Overview

Flotation processes can be classed as either:

> a)    Bulk flotation
> b)    Differential flotation

While all flotation processes are selective or differential in that one mineral or group of minerals is floated away from accompanying gangue, <u>bulk flotation</u> generally refers to separation of unlike mineral types such as sulfides from non-sulfides.  <u>Differential flotation</u> (exemplified, for instance, by the concentration and subsequent successive removal of Cu, Pb, Zn and Fe sulfides from a single ore) on the other hand, is restricted to operations involving separation of similar mineral types.

Froth flotation is a means of treating a pulp of finely ground ore so that it yields the valuable or desired mineral in a concentrate that will be amenable to further processing.  The process involves the imparting of a water repellent (hydrophobic) character to the wanted mineral particles by chemicals that are called collectors or promoters.  Under favorable conditions, these chemically coated particles become attached to the air that is bubbled through the pulp, and will thus "float" on the surface.

If the surface tension of the pulp is then reduced by a second chemical, called a frother, a stabilized froth containing the wanted mineral particles will form on the surface of the pulp. This froth can then be skimmed off to yield a concentrate in which the desired mineral is present in a much higher percentage than in the original ore.

## 12.2    Laboratory Flotation Testing

### 12.2.1    Pre-Flotation Procedures

When conducting a flotation experiment the following procedures should be completed before starting the tests.

#### 12.2.1.1 Planning the tests

The test objectives and therefore information required from the results should be established (e.g. sizings, assays, rates of flotation and treatment of products, etc).  Knowing the information that is required, the test can be planned with regard to the following:

- Cell type and size to be used
- Feed sample weight required for each test
- Water quality and quantity to be used
- Floatation density, etc.

**12.2.1.2 Reagents**

All reagents to be used during the test should be prepared at the required strengths prior to commencement.  The freshness of some reagents is important.  Remember the more dilute the reagent, the more accurate is the addition rate but the higher the volume addition to the cell.

**12.2.1.3 Preparation of Equipment**

All equipment should be thoroughly cleaned prior to the test (and after completion of the test), and all product trays and reagent dispensers properly labeled.

**12.2.1.4 History of the Sample**

It is sometimes helpful to gain information from previous tests of a similar type or performed on the same feed source, to become aware of any problems which may occur during the test (e.g. amount and nature of sulphides, feed size distribution, frothing problems, etc.)

## 12.3  Oxide Flotation

### 12.3.1  Flotation of Cassiterite Ores

**12.3.1.1 Reagents**

a) Collector

The collector is a most important reagent as it determines froth grade and recovery.  The optimum addition is controlled by several factors;  ore type, head grade, product grade and recovery.  The collectors used in cassiterite floation often have an effect on the froth properties.  Excessive collector additions can give froth properties.  Excessive collector additions can give froth stability problems (bubbles too stable) and hence selectivity problems.  The types of collectors used in the flotation of cassiterite are varied.  A number of factors control the choice of collectors used.  The following is a list of some of the collectors which are available:

**Collector Type**

1. Carboxylic acid derivatives
   e.g. Oleic Acid tall oil

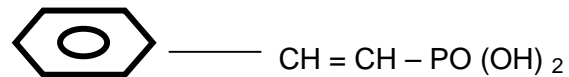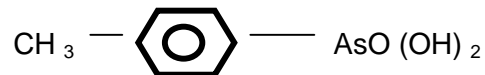$$R-C \begin{array}{c} O \\ \backslash\!\!\backslash \\ \diagup \\ OH \end{array}$$
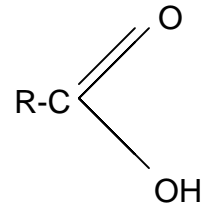
2. Sulfosuccinamates
   e.g.     S3903 – Cyanamid
            CA540 – Allied colloids

$$R - N \begin{array}{c} CH_2 - COONa \\ CH \!-\!\!-\!\!- COONa \\ C \\ CH_2 - CH \begin{array}{c} SO_3Na \\ COONa \end{array} \end{array}$$
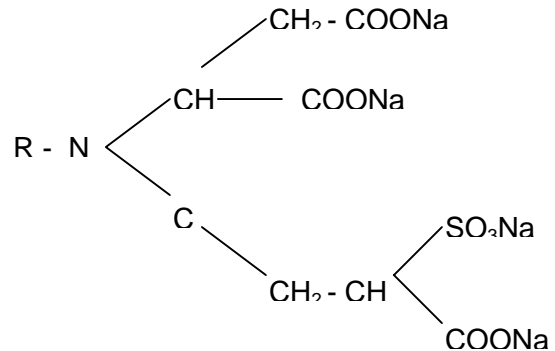
3. Phosphonic acid derivatives
   e.g. styrene phosphonic acid

$$\langle\!\langle O \rangle\!\rangle \!-\!\!-\!\!- CH = CH - PO(OH)_2$$

4. Arsonic acid derivaties
   e.g. p-tolylarsonic acid

$$CH_3 \!-\! \langle\!\langle O \rangle\!\rangle \!-\! AsO(OH)_2$$

b) Frothers

A frother should be used with extra caution in oxide flotation as excessive amounts of frother will alter the froth composition, increasing the recovery of water and hence increase the recovery of the gangue minerals.  The choice of frother for cassiterite flotation as recently become an important variable as the effects of various frother and concentrations have been investigated.  The frother MIBC has historically been the frother used but recent limitations with this reagent have led to the use of alternatives such as Aerofroth 65, Senfroth 41G and Dowfroth 250.

c) Depressants
   $Na_2SiF_6$
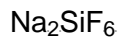
   The reagent sodium silico fluoride ($Na_2SiF_6$) is the most commonly used depressant in cassiterite flotation, acting both as a slime dispersant and also reducing the floatability of certain minerals, especially fluorite, tourmaline and other silicates. It is also thought to activate cassiterite in some circumstances. This reagent is added prior to the collector addition, but further additions in the cleaning stages can be of benefit. The rate of addition varies depending on the ore source, but 0.3-0.4 kg/tonne of feed is a good starting point.

   Other depressants which are occasionally used are:
   - Sodium silicate – slime dispersant but will depress cassiterite if added in large quantities
   - Lignin sulphonate – depressant for gangue slimes;
   - Cyquest – precipitation of heavy metal ions.

d) Modifiers (Acid)
e)
   It is necessary for the pH of the flotation pulp to be at the correct value and held relatively constant, for maximum selectivity and recovery.

   Cassiterite flotation is nearly always performed in an acid environment, the exact value depending on the ore source and collector to be used. Sulphuric acid is used for pH adjustment in most cases. Changes in pH can cause changes to the froth. A reduction in pH will give better selectivity but usually at a lower recovery. If the pH is increased the opposite will occur as persistent froths are formed.

   Alkali (NaOH)

   The addition of NaOH is sometimes used to increase the pH in the cleaning stages. The higher pH helps in dispersing slime and improving the % Sn grade.

   Readers are also referred to mining chemicals handbook, revised edition Cytec 2002.

12.3.1.2 Feed Preparation

The nominal size range for cassiterite flotation is between 2 and 100 microns. The top size is variable depending upon the liberation of the cassiterite particles. Cassiterite grains coarser than 100 microns are difficult to float whilst grains finer than 2 microns give selectivity problems. Hence the feed for cassiterite flotation should be reduced to 95% minus 100 microns and then deslimed at 2 microns to give a feed containing no more than 8% minus 2 microns. Information on techniques to achieve this feed size is given in page 34.

When a number of tests are to be performed, a bulk sample of flotation feed is placed in a well agitated vessel (cyclone rig) at the required density for flotation and the required volume

being sampled from the bulk for each test.  This is to ensure consistency of the feed for each test, with regard to size distribution, grade and sample weight.

It is important to have adequate sample for the number of tests required and to maintain the same sample quality, which will deteriorate with time.

## 12.3.2  Flotation of Tungsten Ores

12.3.2.1 Introduction

Scheelite ($CaWO_4$) and Wolframite (($Fe,Mn)WO_4$) are the principal minerals of tungsten, but other important minerals include ferberite ($FeWO_4$) and huebnerite ($MnWO_4$).  The high specific gravity of these minerals makes many of these ores amenable to gravity concentration methods, however, the fine dissemination of mineral in gangue for some ores may require finer grinding and recovery of mineral by flotation at finer sizes.

12.3.2.2 Flotation of Wolframite (Including ferferite and huebnerite)

Wolframite exhibits very similar flotation properties to that of cassiterite; hence it is floated in an acid medium using sulphosuccinimates (Cyanamid S3903 or Allied Colloids CA 540) or styrene phosphonic acid (SPA), and is floated as cassiterite.

12.3.2.3 Flotation of Scheelite

1) General practice for flotation of scheelite is in alkali circuit using either soda ash or lime for pH control.

2) Plant practice at King Island scheelite involves conditioning of feed pulp to 10.2 pH using $NaCO_3$ and floating a scheelite concentrate using oleic acid emulsified with akypomine. Rayflo-C, a wood derivative is used to control calcite and sodium silicate is used as a gangue dispersant.  This produces a 15-20% $WO_3$ concentrate which is then leached to produce an artificial scheelite product assaying 75% $WO_3$.

3) The Tempiute process is one approach used for the flotation of scheelite from ores having appreciable quantities of calcareous and siliceous gangue minerals.  The process basically consists of conditioning the ore with lime, followed by soda ash and sodium silicate as modifiers.  Scheelite is then floated using a mixture of oleic and naphthenic acids as collectors.  The addition of lime is not only useful for pH control but apparently aids in selectivity over gangue minerals.  50-56% $WO_3$ concentrate grades have been reported to 95% recovery.

## 12.4 Sulphide Flotation

Sulphide minerals possess excellent flotation properties; hence the prime importance of flotation as a concentration technique for sulphides.

### 12.4.1 Flotation Agents

a) Collectors for sulphide minerals :    Anionic collectors are most commonly used for sulphide minerals.  By reference to the table (below) it will be note that they are all structurally similar, each having a single sulphur atom double bonded to either a carbon or phrosphorus atom, hence all of the sulphide minerals can be floated with varying success by any of the sulfydric collectors.

| Types of Anionic Collectors of Sulphides | Structure |
|---|---|
| Xanthates | $S$<br>$\parallel$<br>$ROC - SNa$ |
| Thionocarbamates | $S$<br>$\parallel$<br>$RHN-C-0R^1$ |
| Dithiophosphates | $RO$<br>$\quad\diagdown \quad S$<br>$\qquad\quad \parallel$<br>$\qquad\quad P \text{——} SNa$<br>$\quad\diagup$<br>$RO$ |
| Thionocarbanilide | $S$<br>$\parallel$<br>$H_5C_6 - HN-C-NHC_6H_5$ |
| Xanthogen Formate | $S \qquad\quad O$<br>$\parallel \qquad\quad \parallel$<br>$ROC\ - S - COR$ |

b) Frothers:  The most common frothers in use are organic heteropolar compounds, the non-polar organic radical repelling water, the polar portion attracting water.  Essentially frothers lower the surface tension of water, the froth having just enough stability to act as a vehicle to transport the enriched mineral out of the flotation machine.  The quantity of frother required in sulphide ore flotation is generally less than 0.10 kg/tonne.  Examples of common frothers are pine oil, creosote, cresylic acid, amyl and hexyl alcohols and low viscosity completely water soluble liquids like polypropylene glycol methyl ethers (e.g. Dowfroth series).

c) Conditioners or modifiers may function as pH modifiers, Re-surfacing agents or precipitates for soluble salts.  These are summarized in the table inset.

---

a. pH
 e.g.  Lime (CaO), Soda Ash ($Na_2CO_3$),
        caustic soda (NaOH), Acid ($H_2SO_4$, HCℓ)
b. Resurfacing Agents to improve selectivity
   By either activation or depression of the resurfaced mineral
   Cations:  Ba, Ca, Pb, Zn, Ag
   Anions:  $SiO_3$, $PO_4$, CN, $CO_3$, S
   Organic colloids, Dextrin, glue, starch, etc

c. Precipitation or Ion removal
   Cations:  Ca, Ba
   Anions: CN, $CO_3$, $PO_4$, $SO_3$

---

Since almost all sulphide flotation circuits operate with an alkaline pulp for optimum metallurgy and the prevention of metallic corrosion, lime or soda ash for alkalimity regulation are employed.  Where the ore is very acid and lime difficult to obtain or where the minerals are floated from acid leached pulp, acid pH control is usually obtained with dilute $H_2SO_4$.

Examples of re-surfacing agents are the use of $CuSO_4$ in the flotation of sphalerite (ZnS) employing a xanthate collector wherein copper adsorbs on the sphalerite surface replacing zinc in the mineral lattice; use of lime (CaO) as a depressant for pyrite and other iron sulphide minerals when xanthate collectors are used; oxidizing agents like sodium hypochlorite (NaOCℓ) and potassium permanganate ($KMnO_4$) as depressants for Cu, Fe and Co sulphides in lime alkaline pulps; reducing agents like sodium sulphite ($Na_2SO_3$), or sodium bisulphate ($NaHSO_3$) to prevent excessive oxidation in Pb-Zn flotation or Na or K cyanides employed for the depression of Fe, Cu or Zn sulphide minerals.

### 12.4.2 Types of Sulphide Occurrences

     i. Copper Ores which can occur as:
        a) solid pyrtic ores where the problem is to separate the copper sulphides and the pyrites into independent concentrates, or
        b) impregnated (porphyritic ores), characterized by a relatively low pyrite content and presence of gangue minerals. Here the aim could be to separate the sulphide combination from the gangue.

     ii. Copper Zinc ores, typically combinations of some or all of the following:

| | |
|---|---|
| Chalcocite | $Cu_2S$ |
| Chalcopyrite | $CuFeS_2$ |
| Sphalerite | $ZnS$ |
| Pyrite | $FeS_2$ |
| Pyrrhotite | $FeS$ |

     iii. Lead-Zinc and Lead-Copper-Zinc-Iron ores

     iv. Copper Nickel and Cobalt ores

     v. Copper Molybdenum ores

     vi. Arsenic, Antimony and Mercury ores

     vii. Bismuth bearing ores

     viii. Gold Silver ores

### 12.4.3.1. Recommended Flotation Procedures: the following pertain to some of the sulphide occurrences.

Class I (i)    Bulk Copper Sulphide Ores
Object:      Differential flotation of copper while preventing excessive iron flotation.
Conditions: Use xanthates or similar collectors in a pulp made alkaline with lime (pH 8.5 to 12.5)

Class I (ii)   Porphyritic Copper Ore
Objective:   Separate Copper minerals closely associated with pyrite
Conditions: Rougher concentrate as per (i).

Regrind: Attempt to depress pyrite after regrinding by cyanide additions 0.0015 kg/t in addition to lime (pH =10). Typical reagent conditions as follows:

| Reagent kg/t | Primary Grind | Point of Addition Rougher | Regrind |
|---|---|---|---|
| Lime | 0.8 | - | - |
| Z-11 | - | 0.05 | - |
| Dowfroth 250 | - | 0.01 | - |
| NaCN | - | - | 0.0015 |

Class II    Copper Zinc Ores

Object:    Depression of Copper Sulphide with cyanides, sodium sulphide, zince sulphide or a combination of these reagents in an alkaline medium (pH 8.5 – 9.5).

Conditions: Collector – Butyl Xanthogenate or Cresyl dithiophosphate (0.03 – 0.05 kg. Tonne or a combination of these).

Frother:    Cresol or Terpineol

Depression: Zinc Sulphate 0.5 kg/tonne
Agents:    Zinc Sulphate (0.5 kg/t) and Cyanide (0.1 kg/t)
             $Na_2S$ (0.025 kg/t)

Class III    Lead-Zinc and Copper-Lead-Zinc-Iron Ores

Object:    The selective separation of galena and sphalerite, galena and copper sulphide and sphalerite and pyrite.

III (i)    Flotation of Galena

Collectors:    Sulfhydryl (20-40 g/t) (xanthogenates rather than dithiophosphates)
Carboxyl collectors for heavily oxidized mineral
Alkaline medium pH 9-10
Chromate and bichromate (effective galena depressants) can be used to separate galena and copper sulphide. When galena flotation is complete, sphalerite is activated with copper salts and zinc hence floated in a lime alkaline medium, to prevent a large proportion of the iron sulphides from passing into the zinc concentrate.

III (ii)    Selective Separation of Lead-Copper-Zinc Ores

Lead and copper minerals are usually separated into a join lead-copper concentrate.

Note: Copper minerals do not always pass into the lead concentrate. When relatively large amounts of cyanide are used for depression of copper, very often only part of the copper in the ore will accumulate in the Pb concentrate. Hence only moderate dosage of cyanide as a depressant is recommended ( ~0.3 kg/t).

The following two techniques are recommended for separating the collective Pb-Cu concentrate into Lead and Copper products:

1. Depress Cu sulphides with cyanide and float the Lead
2. Depress Galena with bichromate and float copper sulphides.

For alternative selective flotation of Pb-Zn-Cu ores, refer to paper by Jackman Scamardella and Tilyard, Mill Operators conference, Roxby Downs 1994. (Peter Tilyard@riotinto.com).

<u>Class IV    Copper-Nickles and Cobalt Ores</u>

IV (i)        copper-Nickel

Object:   collective flotation of Copper and Nickel from associated minerals like pyrrohotite and subsequent separation into copper and nickel concentrates.

Techique:  Copper-Nickel flotation with Butyl Xanthogenate and Pine Oil.  After several cleanings, collective concentrate is thickened and reground and the copper and nickel concentrate separated in alime cyanide medium, the copper concentrate as the froth product. Cyanide consumption 0.022 – 0.045 kg/tonne, lime 0.7 kg/t; pH 7.5 – 8.2.

IV (ii)       Copper and Cobalt from pyrite concentrate

Technique:  Ore is conditioned thoroughly in milk lime.  Extraction sequence is usually copper, pyrite-pyrrhotite and cobalt concentrate.

Collectors:   Copper minerals floated with diethyl and secondary dibutyl dithiophosphates (pH 10).

Iron sulphides are floated with amyl xanthogenate and mercapto-benzothiazole.

After floating the iron sulphide, sodium sulphide (small amount) is added to the pulp followed by $H_2SO_4$ and additional sodium sulphide.

Cobalt is floated at pH-4 with amyl xanthogenate and pine oil.

Class V      Gold Silver Ores

> The techniques adopted depend a great deal on modes of association of the precious metals, eg.  Au with the pyrites or Ag with Galena; in which case, the individual techniques of bulk sulphide rougher flotation and subsequent cleaning (or selective flotation of individual minerals to give, for example,  Cu rich, Pb rich, Fe rich and Zn rich concentrates) apply.

12.4.3.2 Standard Flotation Test

**Feed Preparation:**
Prepare a 3 kg charge from the weekly Que River ore sample.  This sample has been roll crushed to 80% - 1mm.

A further 20 g sample is required for the flotation head assay.

**Grinding:**
Using the 1 kg mill, grind the feed in three separate batches.  The grinding time for each batch is 14 minutes using 75% solids and 5 ml of 5% $Na_2S_2O_5$ (0.25 kg/t).

Screen the mill product with a 106 micron screen and reject the oversize.

**Cu Flotation:**
Using the 5.2 litre Agitair flotation cell make the flotation feed up to 45% solids with the −106 micron fraction.

**Conditioning:**
Add Na2CO3 (10%) if the pulp pH falls below 6.0 and condition for five minutes.

**Aeration:**
Add 1 ml of 5% sodium aerofloat collector (0.017 kg/t) and aerate for two minutes.  Again, add $Na_2CO_3$ if the pH falls below 6.0.

**Rougher Float:**
Add 0.05 ml of cresylic acid frother (0.0167 kg/t) and float for approximately six minutes.

Record the wet rougher weight, label the pan and dry.

**Pb Flotation:**

*Conditioning*:   Bring the pulp level up to the 5.2 litre mark by adding water.  Record the volume of water added.  Add 5 ml of 5% Zinc Cyanide depressant (0.083 kg/t) and increase the cell pH to 9.2 by adding $Na_2CO_3$.   Condition for five minutes.

*Pb Rougher:*   Add 1 ml of 5% Sodium Isopropyl Xanthate collector (0.017 kg/t) together with 0.02 ml of cresylic acid frother (0.006 kg/t) and float for 20 minutes.  Record the wet rougher weight, label the pan and dry.

*Pb Scavenger*:  Bring the pulp level up to the 5.2 litre mark, recording the volume of water added and adjust  the pH to 9.2 if necessary.  Add 0.4 ml of Sodium Isopropyl Xanthate together with 0.02 ml of cresylic acid and float for a further 5-6 minutes.  Record the wet scavenger weight, label the pan and dry.

**Zn Flotation**

*Conditioning*:  Bring the pulp level up to the 5.2 litre mark, recording the volume of water added.  Add 30 ml of 5% $CuSO_4$ (0.5 kg/t) and bring the pulp pH up to 10.0 using a 5% lime solution.  Record the volume of lime added.  1.5 ml of 5% Sodium Aerofloat collector (0.025 kg/t) should also be added and the pulp conditioned for a period of six minutes.

*Zn Rougher*:   Add 0.02 ml of Dowfroth 250 (0.007 kg/t) and float for 15 minutes.  Record the wet rougher weight, label the pan and dry.

*Zn Scavenger*:  Bring the pulp level up to the 5.2 litre mark, recording the volume of water added.  Using lime adjust the pulp pH to 10 if necessary.  Add 0.2 ml of Sodium Aeroflaot together with 0.02 ml of Dowfroth 250 and float till the froth is barren.  This should take approximately eight minutes.

Flotation Tail

Empty the flotation tail into labeled pans and dry.

**Preparation of Assay**

Record the dry weights of the Cu rougher, Pb rougher, Pb scavenger, Zn rougher, Zn scavenger and flotation tail.

Cut out a minimum of 20 g from each of these productions for XRF analysis at the Cleveland laboratory.

**Recording of Results and Assays**

All test results and assays should be recorded in the Que River log book.


**12.5    Standard Flotation Test Procedure**

   1)    -    Fill the flotation cell to the predetermined level and position the pH probe.  The calibration of the pH meter should be checked.  Note.   The level of the pulp will change when agitation is commenced and air introduced to the cell.

- Select the speed of agitation. This varies according to cell type, size and pulp density. A useful criterion is that the coarsest particles should not be allowed to settle on the bottom for more than one second. A speed of 800 rpm is usually used with a pulp density of 30-35% solids, in the 3 litre Agitair cell.

2) - Sulphide flotation (if required)
- Adjut pH to required value with $H_2SO_4$ / $NaOH$/$Na_2CO_3$ solutions and maintain throughout.
- Add $CuSO_4$ and condition for 5 minutes, followed by SEX/PAX mixture and condition for 5 minutes.
- Add frother as required and float off all sulphides until froth barren.
- Vanning of the sulphide sink is useful at this stage.

3) Oxide Flotation
- Adjust pH to required value with $H_2SO4$ / $NaOH$ solutions and maintain throughout. The pH value varies with the collector used.
- Add depressant (usually sodium silico fluoride) and condition for 5 minutes.
- Add collector and condition for 10 minutes
- Add frother as required and introduce air to produce bubbles.
- Remove the froth consistently and at a fixed depth.
- Collect concentrates at predetermined times.
- Add further collector and continue flotation if required.
- Vanning of concentrate and tails gives an idea of the performance.

### 12.5.1 Treatment of Products

- All concentrates are weighed wet to calculate water recovery.
- All products are dried and the dry weight recorded.
- Process products for required assaying, sizing and mineralogical analysis.

### 12.5.2 Record of Information

A complete record of the entire procedure used from sample preparation to treatment of products must be maintained. Included in this should be all reagent additions (and solution strengths), conditioning times and pH. A standard log sheet is available for this purpose.

### 12.5.3 Additional Notes

1. Flotation Mechanisms:

There are two main mechanisms by which a mineral can be recovered into the froth. The first is a "true" flotation mechanism, where the mineral surface becomes hydrophobic, due to absorption of collector ions, attaches itself to an air bubble, and rises to the

surface.  The second is called entrainment, where minerals are entained in the water layers between the air bubbles which make up the froth.  An increase in the recovery of water into the froth results in an increase in the rate of entrainment.  It is therefore important to reduce the rate of entrainment as this results in unselective removal of all minerals into the concentrate.  By choosing the correct reagent conditions as described in previous sections and maintaining the correct froth depth, the degree of entrainment can be kept to a minimum.

2.  Calculation of pulp density and reagent addition rates:

It is important to accurately know the pulp density for the flotation test to achieve both optimum flotation performance and the correct reagent additions.   The pulp density can be obtained by using Marcy density scales, or simply weighing a certain volume of pulp, wet and dry.   From the pulp density, the amount of dry solids in the cell can be calculated.

Dry Solids = Pulp Volume x SG (dry solids) x Pulp Density

If one wished to alter the pulp density in the cell, it is a simple calculation to determine the amount of water to be added or taken from the cell to achieve the new density required.

The exact addition of reagents is very important as the effect of reagents on the overall results has previously been shown to be critical.,  Knowing the amount of dry solids (in gms) in the flotation cell and the strength of the prepared reagents, the addition of reagent can be calculated.

Vol. Of addition (mls) = weight of dry solids (gms) x
Addition rate of reagent (kg/tonne) x Reagent strength (wt %)

e.g. if cell contains 1.2 kgs of dry solids and an addition rate of 0.3 kg/tonne is required and the reagent has been prepared to a 5% solution (i.e. 5 g of dry reagent, made up of 100 mls of solution).

Vol. of addition    = 1200 x 0.3 x .05
                    = 18 mls of 5% solution

Following the completion of the test it will be found that the total dry weight of all products is different from the earlier estimation.  One should then recalculate reagent additions using the exact dry weight figure, to find the exact addition rate of each reagent.

# Appendix 1

Determination of Sample Size:   GY's Method

Sampling is the means whereby a small amount of material is taken from the main bulk in such a manner that it is representative of that larger amount.

Sampling is dependent on probability; the sampling method devised by Gy being most often used to calculate the size of sample necessary to give the required degree of accuracy.  This method take into account the particle size of the material,  the content and degree of liberation of the minerals, and the particle shape.

Gy's basic sampling equation is

$$\mathbf{M} = \frac{Cd_3}{S^2}$$

Where    **M** is the minimum weight of sample required (grams),
            **C** is the sampling constant for the material (g. cm$^{-3}$ )
            **d** is the dimension of the largest pieces in the material to be sampled (cm)

And       **s** is the measure of the statistical error or the error committed by sampling, ie. The standard deviation of a normal distribution curve representing the random assay-frequency data for a large number of samples taken from the ore.

For most practical purposes, a 95 times in a 100 chance of being within prescribed limits is usually an acceptable probability level.

The sampling constant C is specific to the material being sampled, taking into account the mineral content, and its degree of liberation.

$$\mathbf{C} = f \times g \times \ell \times m$$

Where   **f** is a shape factor (0.5 normally except for gold ores, where it is 0.2)
         **g** is a particle distribution factor, usually 0.25, unless the material is closely sized, in which case a factor of 0.5 is used;  and $\ell$ is a liberation factor (0 for completely homogeneous material and 1.0 for completely heterogeneous material).

Gy devised a table (shown below) based on d, the dimension of the largest pieces in the ore to be sampled, which can be taken as the screen aperture which passes 90-95% of the material, and L, the size in cm at which, for practical purposes, the mineral is essentially liberated.  This can be estimated microscopically.  Values of $\ell$ correspond to the values of Top Size/Liberation Size, i.e. d/L, and can be estimated from the table:

| d/L | 1 | 1-4 | 4-10 | 10-40 | 40-100 | 100-400 | 7400 |
|-----|-----|-----|------|-------|--------|---------|------|
| $\ell$ | 1 | 0.8 | 0.4 | 0.2 | 0.1 | 0.05 | 0.02 |

**m** is a mineralogical composition factor given by

$$m = \frac{1-a}{a} \times [(1-a)\,r + at]$$

where **r** and **t** are the mean densities of the valuable mineral and gangue minerals respectively, and

**a** is the fractional average mineral content of the material being sampled.

Gy's equation assumes that samples are taken at random, and without bias, and is most applicable to streams of ore transported on conveyors or in pulp streams rather than heap deposits which are inaccessible to the sampler.

The equation gives the minimum theoretical weight of sample which must be taken.
Example:  Consider a lead ore, assaying about 5% Pb, which must be routinely sampled for assay to a confidence level of ± 0.1%.  Pb 95 times out  of 100.  The galena is essentially liberated from the quartz gangue as a particle size of 150 microns.

If sampling is undertaken during crushing, when the Pb size of the ore is 25mm, then

**d**  =  2.5 cm

**2s** $= \dfrac{0.1}{5} =$  0.02.  Therefore s = 0.01

$\dfrac{\mathbf{d}}{\mathbf{L}} = \dfrac{2.5}{0.015} =$  167, therefore from the table, $\ell = 0.05$

Assuming the galena is stochiometrically PbS, then the ore is composed of 5.8% PbS.

Therefore,   **a**  =  0.058,  r = 7.5,   t = 2.65

Therefore,  **m**  =  117.2  g.cm$^3$

**C** =   fxgx $\ell$ xm   =   0.5 x 0.25 x 0.05 x 117.2
=   0.73 g.cm$^{-3}$

**M** =   $\dfrac{Cd^3}{s^2}$  = 114 kg

In practice, therefore, about 300 kg of ore would have to be sampled in order to give the required degree of confidence.

If, however, the sampling takes place from the pulp stream after grinding to the liberation size of the ore, then d = 0.015 cm, and assuming that classification has given fairly close sizing,

$$C = 0.5 \times 0.5 \times 0.8 \times 177.2$$
$$= 23.44 \text{ g/cm}^{-3}$$

Therefore   M  =   0.8 g.

Such a small weight of sample would, however, not be cut from a pulp stream as it ;makes no provision for segregation within the stream, variations in assay and particle size et. With time however, it could be a guide for the increment to be cut at each passage of the cutter, the interval between cuts being decided from the fluctuations in the quality of the pulp stream.

Readers are also referred to Sampling of Particulate Materials, Theory and Practice, Pierre M Gy. Elsevier.

*Reference:  Mineral Processing Technique – B.A. Wills*