

# BENCH AND PILOT PLANT PROGRAMS FOR FLOTATION CIRCUIT DESIGN

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## ABSTRACT

Aspects of flotation test programs, ranging from preliminary evaluations through to full feasibility study development programs, are discussed. These aspects include: the objectives; level of data obtained, including evaluation of this data; limitations of the test programs; and practical requirements, including sample quantities, time frame and methodology.

Discussion of necessity and reasons for pilot plant evaluation and types of metallurgical deposit mapping programs are included as well as pitfalls encountered in bench and pilot plant flotation programs.

## INTRODUCTION

This paper discusses flotation flowsheet development for new mining projects and expansions from conceptual (or preliminary) flowsheet development through to pilot plant campaigns. Each phase is discussed with a strong focus on the objective of that phase, and a conceptual analysis of the methodology that one would apply in each phase.

The single most important issue to be addressed before any phase of metallurgical testwork is that of sample selection. Testwork results reflect the sample tested! The corollary of this is that poor sample selection can lead to poor or misleading metallurgical results. To get a truly representative sample, metallurgists must work together with the project geologist(s) and mine planner(s) to carefully select material for metallurgical testwork and establish logical compositing criteria.

The investigator should incorporate good qualitative and quantitative mineralogy in the flotation flowsheet development process. Understanding the nature of the sample mineralogy should drive testwork development and ultimately lead to the optimal flowsheet.

The phases of the flotation process development that will be discussed are:

- Preliminary scoping studies,
- Pre-feasibility studies, and laboratory based feasibility studies,
- Pilot plant testing, and
- Metallurgical mapping programs.

This paper focuses on sulphide mineral flotation but many of the comments can be applied to other mineral types.

## PRELIMINARY SCOPING STUDIES

### OBJECTIVES

The conceptual, or preliminary, stage of metallurgical flowsheet development has a few very specific objectives. These are to:

- a) Provide some broad understanding of the metallurgical department of the desired metal(s), or alternately, that there are no serious metallurgical concerns, (for example, 'is this a refractory gold ore?')
- b) Confirm that the metal(s) desired can be recovered from that sample(s) using classical flowsheets and technology and identify which specific flowsheet route is indicated,
- c) Establish an order-of-magnitude level of recovery for the desired metal(s) and some indication of the type of concentrate quality, and
- d) Produce indications for future testwork.
  - a) What flowsheet parameters need to be studied/optimized and are most economically sensitive?

## DATA QUALITY

Given that the objective of this phase is to understand the metallurgical department of the sample(s), it is not expected that statistically rigid methodology can and will be applied. It is important to 'scatter-gun' different approaches (i.e. flowsheets/reagent regimes) to look for what appears to be working and to establish preliminary

understanding of how the sample(s) responds to the various tests. What is its 'sensitivity' to those tests?

## DATA EVALUATION

The fundamental tool for data analysis is the 'grade-recovery' curve. It is preferable to prepare this curve with grade on the x-axis because frequently the flotation concentrate grade is set or controlled by smelter contracts. If gold were the desired metal, grade is typically recorded on the y-axis, as gold recovery by flotation often depends on the recovery of another mineral. For complex polymetallic flotation, another key tool is a "selectivity criteria" or the index of the desired metal versus an undesired metal, Gaudin's Selectivity Index is such an index. (Taggart 1945).

Test series are limited in the conceptual phase of flowsheet design and frequently tests are not duplicated. Test series focus more on various reagent combinations than optimization of reagents. Therefore, data evaluation in these cases is limited to identifying the tests which gave better metallurgical performance. Where test series are used (e.g. primary grind-size suite), these are best evaluated using a simple grade-recovery curve, that shows all tests.

## LIMITATION

Testwork at this stage is preliminary. The most significant limitations arise because (a) sample size is small and can be biased because this testwork occurs at an early stage of project development, geological sample selection is limited, or (b) the number of tests performed is limited.

Often for primary grind and regrind, a finer-than-optimum size is selected in this conceptual phase testwork to “minimize” poor liberation effects. These parameters are, of course, optimized in the pre-feasibility stage.

## PRACTICAL REQUIREMENTS

**SAMPLES.** Sample size is usually small and too often is less than 40 to 50 kg per sample. Sample selection is critical, but is often limited by sample availability. Composite samples are preferred to small, individual (one-meter) drill-core intersections. Compositing must be carefully considered and should be done after consultation with project geologists. It is best to composite drill core material. Samples such as assay rejects, reverse circulation drilling chips or old samples are usually very poor samples for metallurgical testwork and should be avoided if at all possible.

**TIMING.** Conceptual flowsheet development should be undertaken early in a project development history. It is important to understand the metallurgical deportment of the metals early in a project, as this can influence the economic decisions pertaining to the project. For example, “if we have a refractory gold ore, do we have sufficient precious metal value to sustain mine development?” If the answer to this question is “no”, then can anything metallurgical be done to significantly improve the economics?

## PRE-FEASIBILITY AND LABORATORY SCALE FEASIBILITY TESTING

### PURPOSE AND OBJECTIVES

The key objectives of the pre-feasibility and laboratory scale feasibility phase are to:

- 1) Identify the probable flowsheet and reagent regime required for the economic recovery of the desired metal(s) or, establish if there are any fundamental problems in establishing economic flowsheets,
- 2) Establish, with a higher degree of confidence the expected recovery(ies) and concentrate quality(ies) of the proposed flowsheet,
- 3) Study the variability of the metallurgical performance throughout the (known) orebody. This metallurgical mapping or (as it sometimes referred to) geo-metallurgical mapping program will be discussed in further detail later in this article. Effective laboratory-based pre-feasibility and feasibility studies routinely incorporate such mapping programs, and
- 4) Establish preliminary concentrator design parameters (i.e. grinding information [not discussed here], flotation retention time, reagent requirements etc.).

## DATA QUALITY

Pre-feasibility and feasibility studies require larger sample quantities and large sample sets. This permits more statistically based testwork with the better inherent data analysis and metallurgical conclusions. Some statistical based methodologies that can be applied are given in Griffith (1962). The application of statistical methodology is still limited and there are a number of possible reasons for this. There is a general discomfort in dealing with statistics, but a larger reason is that experience indicates that if we understand the nature of a certain type of ore we will apply a certain (known) flowsheet and reagent regime to that ore. This methodology is often the most expedient. However, in some instances, an ore does not respond well to conventional technology. In this case, the more rigorous methodology of statistical based testwork design and data analysis should be used to direct the testwork and resolve the complexity of flotation pulp chemistry.

## DATA EVALUATION

The recommended sample size for this phase of testing is large bulk composites

from which multiple charges can be composed. Normally, these charges are one or two kilograms each, but can be up to 10 kg. The composite must be well homogenized so that there is minimal variation in head assay and flotation testwork calculated heads. For non-nugget situations (i.e. not Au, Ag, PGE or Mo), the overall variation should be  $\pm 5\%$  (absolute), whereby expected analytical variation is about  $\pm 3\%$  (absolute). For nugget samples, the variation could be much greater.

Reliable analytical methods must support the metallurgical testwork and evaluation. Both the analytical method used and the statistical QA/QC used are important. It is also important to state the analytical method used and stress the need to always compare assays based on the method used. The authors have seen numerous examples of misleading information based around differences in analytical methods (for example, Mo by acid digest, AAS versus Mo by XRF (briquette or pyrosulphate fusion preparation)).

Data evaluation then follows simple analysis based around what set of conditions gave better metallurgical response (usually presented in tables). It is important to maintain ‘bridges’ or linking tests between ‘series’. This is usually achieved by repeating a ‘standard’ test. It is also important to track the results and consistency of the ‘standard’ test(s) throughout the program. (For clarification, it is possible that a ‘standard’ test will change throughout a large testwork program).

In analysis of metallurgical results, it is important to assess “to what extent is my less-than-desired metallurgical response (recovery and concentrate quality) a result of less-than-desired liberation and/or less than optimum chemical environment”. Quantitative information on liberation can be obtained by mineralogy. Even qualitative mineralogy can effectively guide the flotation investigation, (for example, “the sphalerite in the copper concentrate is mostly liberated.”) Therefore, use of mineralogy is one of the most important tools available to flotation investigators. The two critical questions that mineralogy helps answer are “what is the nature of my losses to the tailings,” and “what is the nature of my concentrate contamination?”

Knowing quantitatively the influence of the liberation on a metallurgical response means the effect of the given chemical processing environment can be inferred. This type of analysis is continuous and interactive during flowsheet development.

Generally, it is more expedient to focus on liberation early in a flotation flowsheet development program (i.e. primary grind and regrind(s)), to the point at which an economically acceptable trade-off point is established for these parameters. This is particularly important for the fine-grained polymetallic ores as lack of liberation can mask effects of reagent regime change (for example: "was my poor selectivity against pyrite due to poor liberation or poor chemical environment?")

## LIMITATIONS

As previously discussed, all metallurgical testwork is limited to the validity and furthermore, representivity of the sample(s) tested. Testwork is also limited to the breadth and completeness of the reagent regimes tested. The intent of the paper is not to discuss the enormous number of permutations of collector, frother, pH and type of modifier used, Eh, water chemistry, depressant, activator, dispersant conditions which can be tested to achieve the desired flotation control selectivity. A number of selected references on reagent regime selections are available

are given for this purpose (Bulatovic and Wyslouzil 1985; Bulatovic and Wyslouzil 1988; Bulatovic and Salter 1990; Bulatovic and Wyslouzil 1999; Bulatovic, Wyslouzil and Kant 1998; Bulatovic, Wyslouzil and Kant 1999; Agar et al. 1996).

## PRACTICAL REQUIREMENTS

**SAMPLES.** As previously mentioned the best sample(s) for testwork are composite samples. The compositing for a porphyry copper deposit metallurgical testwork program will be distinct to that of a massive sulphide ore or that of a vein hosted Au/Ag ore. Sample selection will take place for both grinding and flotation testwork at the same time and will follow similar logic. Relevant criteria include:

- Rock type
- Alteration type
- Mineralogy and/or head grade to assess variation in desirable metal(s) content, or major gangue mineral content (i.e. pyrite/pyrrhotite host).
- Oxidation states (for example, oxide zone versus a supergene zone versus a primary sulphide zone).
- Mining plan (such as year of mine production criteria).
- Unusual occurrences (e.g. highly faulted/fractured or folded zones, different mineralogy, etc.) These should be studied only if they are deemed to be geologically and economically significant to the ore distribution.

These same criteria can be used for identification of samples for a metallurgical mapping program. Metallurgical mapping programs are usually incorporated in a complete pre-feasibility or feasibility program.

Excessive compositing (i.e. production of large, overall composites) can mask valuable metallurgical response information and can give misleading conclusions about the actual plant performance. Therefore, it is generally recommended that the team create four to six composites.

The amount of sample required for pre-feasibility and laboratory scale feasibility testwork can vary from as low as 100-200 kilograms to as great as one to two tonnes of sample (per composite to be tested). An example of the latter extreme is testwork that studies Cu/Mo separation after production of a bulk Cu/Mo concentrate. Sample preservation is important as testwork can span many months. Surface oxidation will occur on exposed sulphide mineral surfaces with time. This has been found to compromise test results and give incorrect metallurgical information (Table 1).

It is clear from this, that sulphide mineral samples must be preserved. To achieve this, the samples must be kept as coarse as possible until testwork begins. Reverse circulation drilling and laboratory-reject products make very poor sample(s) for

TEST	SAMPLES	WT %	GRADE % Cu	RECOVERY % Cu	HEAD CALC % Cu	K <sub>80</sub> µm
Sample 1 New Drill Core	3 <sup>rd</sup> CI Conc	1.85	38.6	92.3	-	-
	Ro Conc	9.47	7.9	96.5	0.78	211
Sample 1 Old RC Chip Sample	3 <sup>rd</sup> CI Conc	1.49	37	68.2	-	-
	Ro Conc	13	5.68	91.4	0.81	197
Sample 2 New Drill Core	3 <sup>rd</sup> CI Conc	2.08	30.5	87.2	-	-
	Ro Conc	11.7	5.86	94.3	0.73	202
Sample 2 Old RC Chip Sample	3 <sup>rd</sup> CI Conc	1.4	30.9	55.9	-	-
	Ro Conc	14.8	4.4	84.5	0.77	194
Sample 3 New Drill Core	3 <sup>rd</sup> CI Conc	2.14	36.1	83.5	-	-
	Ro Conc	10.7	8.38	96.9	0.92	184
Sample 3 Old RC Chip Sample	3 <sup>rd</sup> CI Conc	1.61	37.3	68.1	-	-
	Ro Conc	12.3	6.47	90	0.88	191

Table 1 Selection of samples

metallurgical testwork. When the sample must be crushed (often to –10 mesh for flotation testwork) it should be preserved in sealed bags, ideally at sub-zero degrees Celsius (in a freezer), in an inert atmosphere (N<sub>2</sub> or Ar).

## TIMING

Embarking on a pre-feasibility or feasibility study is a corporate decision (outside of the domain of metallurgical investigation). Given that sample requirement for these program(s) and the associated metallurgical mapping program(s) are large, it is important that the sample availability and/or acquisition be considered before undertaking these programs.

## METHODOLOGY

A typical example of a pre-feasibility program for a porphyry copper ore will have the following components:

### SAMPLE CHARACTERIZATION:

- chemical
- mineralogical identification
- mineralogical liberation with respect to different size fractions
- petrography

### GRIND/TIME RELATIONSHIP: ROUGHER FLOTATION OPTIMIZATION:

- primary grind
- reagents
- flotation time

### CLEANER FLOTATION OPTIMIZATION:

- number of cleaning stages
- cleaner scavenger
- regrind (together with liberation analysis)
- reagents

### LOCKED CYCLE TESTS:

- flowsheet 'balance'
- recirculation load
- optimization of collector and frother
- recycle water
- final grade/recovery curve

This methodology places importance on the use of both locked cycle tests and mineralogy. These aspects are further discussed here.

## LOCKED CYCLE TESTING

A locked cycle test is a repetitive batch test used to simulate a continuous circuit. The basic procedure consists of a complete batch test performed in the first cycle which is then followed by similar batch tests which have "intermediate" material from the previous cycle added to the appropriate location in the current cycle. These batch tests, or cycles, are continued in this iterative manner for a number of cycles until, ideally, steady state is reached. The final products from each cycle, i.e. final concentrate and final tailings, are filtered and thus removed from further processing. At the end of the test, all the products, final and intermediate, are dried, weighed and subjected to chemical analysis. The test is balanced and a metallurgical projection is made. Typically we think of flotation for locked cycle testing, but any procedure can be locked cycle tested. A Bond grindability test is an example of a locked cycle test.

While the above description can be found in many classical textbooks (Taggart, 1945; Coleman, 1978; MacDonald and Brison, 1962; MacDonald, Hellyer and Harper, 1985), no discussion beyond the basic procedure is provided. In fact, comments such as "It is questionable whether in any case it approximates mill results any more closely than the standard batch test." (Taggart 1945) arise. It is truly surprising that our classic textbooks indicate that locked cycle tests are more art than science, and suggest that they can be of dubious value. None of the textbooks provide meaningful insight or discussion in:

- Preparation for a locked cycle test,
- The number of cycles to perform,
- How to assess if the test has achieved steady state,
- How to produce a valid metallurgical projection,
- Assess if the metallurgical projection is valid.

## OBJECTIVES OF LOCKED CYCLE TESTWORK

The purpose of the locked cycle test is to simulate continuous circuit behaviour

from batch testwork. There are at least three objectives in a locked cycle test:

- Metallurgical projection of continuous circuit behaviour,
- Assessment of circuit stability or "robustness," and
- Flowsheet or "network" development.

Locked cycle testing is the preferred method for arriving at a metallurgical projection from laboratory testing. The reason for this is simple: the final cycles of the test mimic a continuous circuit. In a batch test, the department of the intermediate streams to concentrate or tailings is unknown. In locked cycle testing these streams are recycled and, at the end of the test, the material in these streams should report to either concentrate or tailings. Thus it will be clear how the intermediate streams divide between concentrate and tailing.

Cycle tests are also used to assess the suitability of a flowsheet and reagent suite. If the cycle test does not come to steady state, then this indicates there are problems. Typical flowsheet problems stem from recovery intensive flowsheet (countercurrent) for ores with challenging mineral selectivity, or aggressive flotation in the recovery stages and too selective in the latter cleaner stages which forces a circulating load. Typical reagent problems stem from either too much or too little added, or a build-up of reagent in the circuit.

## BATCH TESTS LEADING UP TO LOCKED CYCLE TESTS

The batch testwork prior to a cycle test must be adequate to insure a reasonable chance of success. A failed locked cycle test is far more memorable in people's mind than a successful test.

Ideally, the batch tests leading up to a locked cycle test, have each separation stage optimized for reagents and flotation time.

A general trend in most batch testing is trying to achieve the highest possible recovery from the test. This emphasis provides an early estimate of the likely metallurgy for a sample, but is not optimum for cycle testing or a continuous

circuit operation. "Ultimate" batch test recovery is achieved by targeting very high stage recoveries (beyond optimum, i.e. incremental flotation rate of non-valorables greater than valorables), and often leads to more cleaner stages than may be required to produce the targeted concentrate grade. When this procedure is locked cycle tested, it invariably fails because the individual stage recoveries are too high and low grade concentrate and poor mass conservation usually result. In the batch test procedure, the intermediate streams report to tailings while in the cycle test there is no effective means of escape for this material as it will remain in the circuit. It has been observed that stage recoveries, of 85% to 90%, work out well for locked cycle and pilot plant testing.

### STEADY STATE, STABILITY AND MASS CONSERVATION

Although these terms are used interchangeably when locked cycle tests are discussed, they have different meanings. Mular and Richardson (1986) provide an excellent description of steady state. "At steady-state, the mass input rate equals the mass output rate, whether it is entire process that is being considered, or individual unit operations. For a system at steady-state, no material accumulates internally; each unit operation is functioning with an unchanging volume of material already in the circuit." This description of steady state highlights the need for stability and mass conservation.

Stability implies constancy. For example, the concentrate weight and grade remain the same for the last three cycles of the locked cycle test. Mass conservation implies "what goes in ... must come out." In the context of a locked cycle test, this means if 1000 grams of sample goes in, then 1000 grams must come out as final concentrate and tailing. However, mass conservation must also apply to the metal units. Thus, if 100 grams of chalcopyrite goes in, 100 grams of chalcopyrite must come out. Invariably, most people look for stability when studying locked cycle test results, as it is easy to see by looking at the data. Most people ignore mass conservation because it is not easily determined by quickly glancing

at locked cycle test data. Steady state implies both stability and mass conservation. A good locked cycle test achieves steady state.

### METALLURGICAL PROJECTIONS

Producing a valid metallurgical projection is one of the most important components of the test. It is the final numerical summary of the test's metallurgical performance. There are at least three different procedures used to generate the metallurgical projection.

- n-product formula (balance on assays of final products),
- SME procedure (balance on final product weights and assays),
- Concentrate production balance (balance on final concentrate weights and assays).

All three procedures will produce the same metallurgical projection for a test at steady state. None of the procedures are ideal for a test, which is not at steady state.

The following provides a brief description of the procedures. N-PRODUCT FORMULA. The n-product formula is a simple material balance technique that utilizes the assays from the final products to determine the mass balance. Taggart (1945) provides an excellent description. In the case of a simple ore with only a concentrate and tailing, the procedure uses the assay of the feed, concentrate and tailing;

$$C = F * (f - t) / (c - t)$$

The remainder of the balance is calculated once C (the concentrate mass) is determined.

In application to locked cycle test balancing, the weighted average assay for the final two to four cycles is used. One of the important requirements for using the n-product formula is that the circuit must have mass conservation, i.e. input material = output material. If the circuit does not have mass conservation, then the n-product formula will provide an erroneous result. Computer mass balance programs such as MATBAL, BILMAT or JKSimmet use essentially the same approach as the

n-product formula when applied to locked cycle tests.

SME PROCEDURE. The SME procedure is described in the SME handbook (Weiss, 1985). The procedure is more direct and should be easier to apply than the n-product formula. In the case of a simple ore with only a concentrate and a tailing, the concentrate is projected as the average mass and assay of the concentrate produced in the last few cycles of the test, and the tailing is projected in a similar basis. The feed for the test is then calculated as the sum of the products. This procedure is acceptable as long as the test has come to steady state. If the test has not achieved mass conservation, then it will be erroneous because it completely ignores the material that does not report to the final products.

CONCENTRATE PRODUCTION BALANCE. This procedure is a derivative of Weiss (1988) in which the concentrate is projected in the same way. However, the tailings are then calculated as the difference between the feed and the concentrate. This procedure does not overstate the metallurgy when the test does not have mass conservation. An overall premise is that the concentrate produced is the only concentrate produced. All other material must therefore be tailings. In many respects, this procedure resembles a month end production balance at an operating plant, because the smelters only pay for the concentrate received.

STUDY OF BALANCE PROCEDURES. Ounpuu (2001) compared the effect of these balance procedures on a test that achieved steady state and another test that did not. The steady state example had all three balance procedures yield the same metallurgical projection. The second example, which did not come to steady state, had the projected lead recovery vary from 75% to 85%. The difference arises from the assumptions inherent in each of the three balance procedures. Most balance procedures assume that the process is in steady state, or more importantly, that there is mass conservation. If this is not so, then most balance techniques overestimate the recovery. The concentrate production

balance does not overestimate the recovery, but may yield a conservative estimate. This technique has the benefit of at least predicting a laboratory recovery than can be achievable recovery. A reasonable metallurgical balance can be made for a test, which is unstable but has mass conservation. A test which oscillates around 100% mass conservation can have a valid metallurgical projection by using the number of cycles which comes closest to 100% mass conservation. Hence, a cycle test can use any number of cycles (greater than 2) for the projection, and the guideline for how many cycles to use for the prediction should be dictated by the number of cycles which provides a balance closest to 100% mass conservation.

However, a test which never achieves mass conservation will prove challenging to arrive at a good metallurgical projection. The options are, to repeat the test, or use the concentrate production balance procedure which may be conservative but at least yields an achievable result. Using the other balance procedures or trying to interpolate what the steady state metallurgy might be raises more questions than it answers.

How close to 100% steady state is acceptable: 100.00% steady state test has never been observed. Good tests will be at 100% for weight and 100%  $\pm$  2% for the metals. Any test which is >5% from 100% should be deemed as not near steady state, and thus the data viewed with caution. Any test which is >10% from steady state should be considered a bad test and must be ignored or repeated.

## HOW MANY CYCLES FOR THE TEST

How many cycles should be performed? We believe most tests should be conducted for a minimum of six cycles based on practical consideration. Tests should be conducted until they achieve steady state only. In a Bond grindability test, the cycle results are known prior to the next cycle, and thus, the number of cycles can be rigorously determined. This unfortunately does not apply to flotation locked cycle tests due to the long analytical turna-

round time needed to assess the results of each test.

Agar and Kipkie (1978) present a relatively simple numerical simulation technique that can be used to estimate the number of cycles and the potential stability for the test. This procedure provides a reasonable estimate of the test's potential for success, but is at best a pre-test estimation of the number of cycles required, and provides no indication of steady state during the test.

The technique found to have the most success was the tracking of the wet filter cake weights during the test. Each of the final products are weighed and recorded during the test. Target weights are established prior to the test so that the technician(s) can gauge the success of the test. The target weights can be derived from the weights produced during the batch tests, or using a simple calculation to account for the filter paper weight and the cake moisture content. The test is deemed to be in steady state when the all the target weights are being met for at least a few cycles in succession. Carrying out a test for twenty cycles does not necessarily ensure the test comes to steady state. If a test must be greater than nine cycles, then the operators are trying to artificially force the concentrate grade higher and the tailings grade lower than they naturally want to be.

The simpler the ore and process, the fewer cycles should be required. A simple, monomineralic ore with excellent liberation may only require four cycles for a good cycle test. A Cu-Zn ore of similar simplicity may require only five cycles. A complex Cu-Pb-Zn ore with poor liberation may not come to steady state after even nine cycles. It is felt that a minimum of six cycles should be planned for any locked cycle test, and the wet filter weights tracked during the test to monitor how well the test comes to steady state. The individual wet filter weights and the total wet filter weight should be tracked.

## MINERALOGY FOR METALLURGICAL FLOWSHEET DEVELOPMENT

The mineralogy of an ore defines the limit of any physical separation process, such as flotation. Therefore, understanding the mineralogy must underpin all stages of metallurgical flowsheet development.

Mineralogy for metallurgical investigation must focus more on the textural relationships of the ore and gangue minerals (e.g. occurrence, association, grain size and liberation) than mineral identification. Understanding the textural nature of the middling particles provides more useful metallurgical information than just the degree of liberation itself. It is the nature of the middling particles that will dictate the grind and regrind targets and the metallurgical results for a sample.

Quantitative and qualitative mineralogy are both valuable, but metallurgical development work should be backed up by quantitative mineralogy. Qualitative mineralogy can be used to rapidly guide a preliminary scoping program and, particularly for scoping reagent changes. Qualitative mineralogy can often be achieved using binocular microscopes located in the flotation laboratory. However, quantitative mineralogy, especially in the pre-feasibility and pilot stages allows a more detailed analysis of grind/regrind selection.

A hierarchical mineralogical methodology frequently used includes:

- Metal recovery (grade versus recovery)
- Size-by-size metal recovery
- Mineral texture recovery (for example, free sphalerite, simple binary sphalerite, tertiary and complex sphalerite binaries and pyrite recovery to a zinc concentrate)
- Size-by-size mineral texture recovery study (for example, the same mineral texture referred to above, but in five different size fractions, such as +100 mesh, -100 mesh + 200 mesh, -200 mesh + 38 micron, -38 micron +15 micron, -15 micron).

Detailed quantitative analysis requires a statistically based mineralogical methodology. Traditionally this was accomplished using intensive point counting, but recent developments (i.e. QEM\*Scan and some types of image analysis) mean this can be fully automated.

Papers which discuss the use of mineralogy in mineral processing are given (Grammatikopoulos 2002; Grammatikopoulos and Roth 2002; Petruk 2000).

## PILOT PLANT STUDIES

Pilot plant studies are often included in feasibility studies. A feasibility study does not necessarily need a pilot plant but most flotation flowsheets should and usually include a pilot plant study. The reasons for use of a pilot plant have been fully discussed (Engle 1978; Kuestermeyer 2000; Wilson and Dawson 1978) and a synopsis is given.

## RATIONAL

Typically, a pilot plant study provides much more confidence on metallurgical response and results than laboratory testwork. Therefore, the use of a pilot plant campaign is to reduce the technical and financial risk that result from scale-up and operation. Given this, a decision to have or not have a pilot plant campaign should be based on the need to resolve potential process risks and/or unknowns in the proposed flowsheet. Each laboratory flowsheet should be reviewed with respect to operation in an industrial setting, as well as new technology; water supply and/or water recycle; changes in the scale-up grinding criteria and the differences in product size curve of a SAG/AG grind versus a controlled laboratory grind, to highlight but a few areas.

## PURPOSE AND OBJECTIVES

The key objectives of a pilot plant are to:

- Define the concentrate quality and recovery (of desired metals) of a representative sample(s) or the ore(s) and the best known metallurgical process route.
- Produce a 'bankable' document that will be appended to the full feasibility study.
- Provide key engineering data for the concentrator design.
- Reduce design safety factors and improve accuracy of capital cost estimates.
- Study, define, control or optimize those interactions or processes that cannot be properly studied at a laboratory scale (for example, water recycle, bleed streams, regrind positioning or gravity concentration).

- Provide bulk sample for downstream processing (such as concentrate, tailings and water for thickening, filtering, further processing, disposal and others).
- Train metallurgical and operational staff on the flowsheet operation.
- Provide market samples for economic assessment.
- Provides psychological comfort to project financiers and due diligence consultants from a successful pilot plant demonstration.

## DATA QUALITY

The quality of metallurgical data from a pilot plant depends on the stability of the flotation circuit at the time that it is sampled (aside from sample homogeneity and chemical analytical technique). Typically, a flotation pilot plant is operated for several hours with minor reagent adjustments to obtain the reasonably correct conditions. The circuit operation is then frozen for a few hours prior to sampling the circuit under these stable conditions. The circuit should be sampled over several hours to produce a composite sample. Various process measurements are taken concurrent with sampling of the process. Circuit stability is documented with control assays (either separately taken or through on-stream analysis) and stability in key mass-flow rates (for example, cleaners-scavenger tailings). Only metallurgical results from stable pilot plant runs should be used in data analysis and conclusions.

## DATA EVALUATION

A typical pilot plant result or report can include:

### (I) SAMPLE DESCRIPTION

- chemical characterization
- mineralogical identification
- petrographic department information
- liberation criteria
- grindability nature

### (II) WATER DESCRIPTION

- open circuit
- recycle

### (III) BASELINE LABORATORY TESTWORK

- batch flotation tests
- locked cycle test(s)

### (IV) PILOT PLANT TESTWORK PROGRAM

- single tests conducted
- conditions for 'demonstration' or 'final' run

### (V) FINAL METALLURGICAL RESULT

- from 'demonstration' run
- comparison to locked cycle test
- projected grade/recovery curve for desired metal(s)

### (VI) DETAILED PILOT PLANT ANALYSIS

- flowsheets compared
- reagents compared

### (VII) ENGINEERING DATA FROM THE 'DEMONSTRATION' RUN

- grinding circuit (kWh/t, operating work index, etc.)
- rougher flotation (retention time)
- regrind circuit (kWh/t)
- 1<sup>st</sup> cleaner/cleaner-scavenger (retention times)
- other cleaner stages (retention times)
- mass flow data/mass balance
- water balance
- assay balance
- reagents / pH/ Eh
- media consumption
- size analyses of a variety of streams
- kinetic flotation analysis (if required)
- concentrate settling and filtration
- tailing settling and filtration
- tailing rheology (if required)
- pulp rheology (if required)
- column scale-up criteria (if required)

### (VIII) CONCENTRATE CHARACTERIZATION

- chemical (to expected smelter contracts)
- mineralogical composition
- liberation
- size
- % moisture
- transportable moisture limit

### (IX) EFFLUENT CHARACTERIZATION

- chemical
- toxicological

### (X) TAILING CHARACTERIZATION

- chemical
- mineralogical
- acid-generating capability
- leachate testwork
- rheology

**(XI) SUPPORT TESTWORK DURING PILOT PLANT CAMPAIGN**

- laboratory flotation testwork
- mineralogy
- liberation studies
- special plant surveys

**(XII) SPECIAL TESTWORK (AS DEFINED), FOR EXAMPLE: CONCENTRATE COMBUSTIBILITY**

- downstream concentrate hydrometallurgy
- downstream batch concentrate testwork (e.g. Cu/Mo separation)

**(XIII) REVIEW OF PILOT PLANT OPERATION**

- strengths
- weaknesses
- stability

A 'demonstration' or 'final' run in a pilot plant is a continuous pilot plant operation incorporating all the findings, with regard to flowsheet and reagent regime/addition rates from previous pilot plant operations, in such a way as to demonstrate the final metallurgical performance of the ore. This usually lasts 48 hours to five days, and is a continuous operation.

Locked cycle flotation tests are used to indicate stable metallurgical performance at a laboratory scale (given known sample, flowsheet and reagent regime). Therefore, a locked cycle test performed on the pilot plant feed sample(s) can be compared to the final 'demonstration' run metallurgical result. Such a locked cycle result should give the same metallurgical performance as the pilot plant. This comparison is considered important as it provides a connection of pilot plant with previous and future laboratory-based flotation testwork. It provides a baseline so that changes to the final piloted flowsheet can be assessed in a cost effective manner.

**LIMITATIONS**

The largest potential limitation of a pilot plant is sample representivity. Pilot plants can require between thirty to several hundred tonnes of material. The questions that then surround this are "how

does one obtain this amount of sample(s)," 'what does the sample represent?' and "what does one want the sample to represent." The sample should also be linked back to historical and future laboratory-based testwork, by the use of "bridging" testwork as previously discussed.

The next problem presented is the homogeneity of the pilot plant sample. Consistent head grade must be provided throughout a pilot plant campaign. Classical rod mill/ ball mill grinding circuit pilot plant configuration require a fine crushed feed (-1/2" to -1/4") that makes it easy to homogenize a pilot plant feed pile. SAG/AG mill pilot plant grinding circuits are usually fed with a top size of six-inch material. Homogenization of this material is more difficult. Samples that contain gravity recoverable gold can also be particularly problematic because of gold concentrating in the fines of the ore pile.

**PRACTICAL REQUIREMENTS**

Sample. Refer to the comments above on sample selection. There is a range of pilot plant sizes available. Some typical sample requirement sizes for pilot plants are:

THROUGHPUT	TIME	SAMPLE SIZE
1 t/h	4 week campaign	400 tonnes
0.5 t/h	4 week campaign	200 tonnes
0.15 t/h	4 week campaign	50 tonnes

Head grade of the desired metal(s) needs to be considered when sizing pilot plant feed rates. Cleaner flotation flowrates and cell volumes guide the throughput for pilot plants. Thus, higher grade ores can use lower feed rates, while lower grade ores need higher throughputs. Also, higher throughputs (>0.25 t/h) are recommended for complex poly-metallic flotation because these pilot plants are difficult to balance and control at low throughputs. Pilot plant size SAG mills consist of 5½' to 6' diameter mills that typically require 1-4 t/h feed rates, depending on the autogenous work index.

Pilot plants can be scaled from laboratory testwork, ideally using a projected mass balance from a balanced locked cycle test (although batch testwork can also be used). The authors use a flotation retention time scale-up factor of 2.5:1 to 3:1 for laboratory to pilot plant.

**METHODOLOGY**

A typical well-structured pilot plant program should potentially include:

- SAG/AG grinding circuit testwork
- Flotation circuit commissioning
- Selected specific tests on flowsheet/reagent
  - reagent optimization
  - flowsheet deviations (e.g. regrind location, column cells)
  - use of recycle water
- Continuous operation with selected final flowsheet without recycle water
- Continuous operation with selected final flowsheet with recycle water
- Product thickening and filtration testwork
- Product characterization
- Downstream product testwork



It should be pointed out that a pilot plant should not be used to scope reagents, but rather to optimize reagent additions, points of addition and requirements, given a closed system.

## METALLURGICAL VARIABILITY PROGRAMS

### PURPOSE AND OBJECTIVES

A well planned metallurgical mapping program can significantly reduce project risk and contribute to the completeness of a pre-feasibility or feasibility program. The key objectives are to:

- 1) Evaluate the ore variability on the basis of head grade, mineralogy, rock type, alteration type, location and/or mining year (mine plan).
- 2) Provide correlation between mineralogical testwork and metallurgical testwork.
- 3) Develop model(s) that forecast flotation performance based on some criteria such as head grade, mineralogy.
- 4) Assess robustness of the proposed flotation flowsheet to the established mineral variation.
- 5) Provide for an optimization process for the flowsheet/reagent regime that can be incorporated in a final concentrator design to make it more robust during mine life.

The use of metallurgical mapping programs are discussed in references (Winkers 2002; DiPrisco, MacDougall and Urbanoski 2000).

## SAMPLE

A well planned metallurgical mapping program consists of a matrix of samples that reflect the orebody. Commonly used matrices are rock type/ alteration type, and/or mineralogy; mine plan matrix (either as a stand alone matrix or combined with the above) or the simplest model can be to composite samples on a pre-established drill-core meterage.

The sample selection for the metallurgical mapping program must be done by consultation between the metallurgist, geologist and mine planners. An ideal program should consist of a representative large sample base (>20 samples, preferably >100), and the number of

samples should bear some correlation to the size, value and variability of the deposit. Medium to large deposits typically have a sample for each 1-5 million tonnes of ore.

## METHODOLOGY

All samples should be submitted to a standard characterization program that includes head chemical analysis, mineralogical examination and a standard batch flotation test (rougher and cleaners). Data analysis needs to be at two levels:

- 1) Metallurgical results need to be studied for trends in results versus matrix characterization (for example, alteration type versus metallurgical performance) and or metallurgical result versus primary metal(s) head grade(s) and/or versus some auxiliary metal analysis (for example, "does poor metallurgy of a copper concentrate correlate to zinc contamination/zinc in the feed assay?")
- 2) Dependent on the mineralogical methodology used, some attempt should also be made to relate specific mineralogical characteristics to metallurgical performance. Some examples could include the percent pyrite versus metallurgical performance or if using QEM/Scan (Winkers 2002; Sutherland, Wilkie and Johnson 1989), one could use the PSSA (Phase Specific Surface Area) factor versus metallurgical performance.

This type of analytical methodology calls for one number that reflects metallurgical performance. There are various numbers that one can use to do this. Some of these are referred to in reference (MacDonald and Brison 1962). These authors refer to a selectivity index (SI), where  $SI = A_r B_f / B_r A_f$  with  $A_r$  the grade of the constituent A in float,  $B_r$  is the grade of constituent B in float,  $A_f$  grade of constituent A in non float and  $B_f$  is grade of constituent B in non float. An option preferred by the authors is to use a recovery number at some standardized concentrate grade. Whatever number is used, it is important to be cognizant of the assumptions and limitations behind that number.

## CONCLUSIONS

This paper has outlined concepts, philosophy, controls and limitations of laboratory and pilot plant flotation flowsheet design. Many of the themes touched on in this paper appear timeless because so many of the questions being asked today are unchanged from twenty or thirty years ago.

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