CHAPTER 1.6

Laboratory Test Work and Equipment

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This chapter provides a broad introduction to metallurgical laboratory test work, and specifically the common types of equipment used in commercial laboratories for various unit processes. The discussion focuses on bench-scale testing, with large piloting equipment excluded.

Metallurgical laboratory test work always features as the basis of any mining and mineral processing operation. Test work typically commences shortly after initial exploration drilling and, thereafter, concurrently proceeds throughout the various phases of the project.

Initial test work, which together with drill-hole interval analysis for ore reserve/resource modeling, consists of simple bench-scale tests to determine overall physical and metallurgical (extraction, beneficiation, and chemical) characteristics of a limited number of sample composites. As a project develops toward construction, metallurgical laboratory testing continues at a higher level of complexity to further clarify physical and metallurgical characteristics over a greater range of samples, optimize processing parameters of differing treatment techniques and processes, and investigate variability across an ore deposit using a constant testing regime. In the final phases of project studies, test work is conducted on larger sample masses (bulk batch testing) to produce various final saleable products and tailings streams for marketing and vendor testing. Finally, pilot testing is often completed using small-scale continuous plant equipment to demonstrate the viability of a selected processing circuit inclusive of all recirculating streams and site water.

Ultimately, the test-work results form the basis of engineering design criteria for the final plant circuit issued for construction. In essence, the more thorough and comprehensive the metallurgical test work conducted in the study phases, the better the probability of success the operation has of achieving design objectives.

Test work often continues during mine site operation on plant samples for ongoing project optimization and investigations, marketing samples, toll treatment umpiring, and to produce data for metallurgical accounting purposes. In addition, brownfields expansion projects can see a repeat of the initial project development test work for newly discovered ore bodies, capacity increases, or different processing requirements to accommodate changing mineralization at mining depth.

METALLURGICAL TEST-WORK LABORATORIES

Laboratories that conduct metallurgical test work can be grouped into four major categories, which are discussed next.

Private Group and Commercial Laboratories

The modern commercial metallurgical laboratory had its genesis in the private laboratory infrastructure set up by the larger mining houses in the latter half of the 20th century. Similar to the university facilities, these laboratories were equipped with an array of equipment to encompass most mineral processing techniques, but with the specific purpose of testing site samples for industrial project development. During the same period, private businesses were also setting up laboratories with metallurgical testing as a sole core business and service stream, offering industry-competitive test work without the heavy cost of sustaining internal infrastructure through a cyclical market. As of 2018, with few exceptions, most of the large mining houses have closed down and/or divested in-house laboratory infrastructure to the independent private sector. In turn, many of those same independent businesses have been acquired by the three main testing and inspection corporations: ALS (formerly Australian Laboratory Services), Bureau Veritas, and SGS (formerly Société Générale de Surveillance), all of which have worldwide footprints with very large laboratory hubs that cover virtually all testing processes. There still remain some notable independent exceptions such as Hazen Research in the United States, and Mintek in South Africa. In most of the world's main mining regions, there are numerous small-scale laboratories providing niche and expert laboratory services.

Private Research Laboratories

These facilities are set up and maintained by the larger equipment vendors and manufacturers (e.g., FLSmidth, Outotec, Metso, Delkor, and many others) and can be very well equipped. However, testing focuses exclusively on the

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corporation's core intellectual property and/or processing techniques to match its equipment inventory (thickening, filtering, calcination/roasting, pumping, magnetic separation, etc.). The ultimate aim of all test work is to promote equipment sales, whether it be proving output parameters against equipment designs and models or general sample performance characteristics.

On-Site Mine Laboratories

These laboratories range in size commensurate with the processing operation but fundamentally are geared toward the preparation and analysis of plant control samples. Metallurgical testing equipment is limited to directly match the unit processes of the colocated industrial operation (small crushers, grinding mill and a flotation cell, leaching vessel or magnetic separator, etc.). Increasingly in recent decades, on-site laboratories have been downsized, with most metallurgical testing outsourced to better-equipped commercial laboratories, which precludes having to maintain the cost of a standing staff complement and equipment inventory.

University and Institute Research Laboratories

Situated on a university campus, these laboratories can be equipped with extensive small-scale equipment covering a wide range of comminution, beneficiation, and extraction unit processes. Some of the larger laboratories conduct a niche range of commercial services to augment private and government research grant revenue, but the primary purpose of these laboratories is conceptual research in conjunction with student learning.

The following sections describe laboratory equipment in terms of unit process, type and purpose, listing brands, and suppliers, if available, bearing in mind that some equipment is often custom fabricated. Where appropriate, some commentary is made on laboratory procedures related to the equipment and required sample masses.

CRUSHING AND SCREENING

As with most industrial-scale mineral processing operations, crushing is the first unit process requirement for most laboratory test-work programs. Avoidance of excessive fines production to produce an accurate and measurable target particle size distribution (PSD) is as imperative in the laboratory as it is in industrial practice. As such, closed-circuit screening at a target aperture is usually employed on crushed product at every stage (screening is discussed separately in the "Size Analysis" section). However, nominal crushing to a set crusher gap is sometimes conducted at very coarse sizes where an accurate PSD is not required, or where the sample is destined to be pulverized for assay.

Types of laboratory crushers tend to mirror industrial practice, but at smaller scale. Conventional single-toggle jaw crushers are typically used for initial coarse crushing through to midrange particle sizes. There are numerous manufacturers, makes, and models, of laboratory-scale jaw crushers, including Rocklabs, Essa (now part of FLSmidth), Jacques, and Shanghai Jianshe Luqiao Machinery. Laboratory-scale jaw crushers can range from 310 × 250-mm units that produce a 50–75-mm product, 250 × 150-mm units that produce a 40–15-mm product, and 100 × 75-mm units that can produce <6-mm products.

Finer top particle sizes suitable for treatment by laboratory rod and ball mills to produce material for metallurgical test work require secondary crushing using small-scale cone crushers, such as the Wescone, which can take up to 15-mm feedstock and produce a <2- to 3-mm product. The modern Rocklabs Boyd crusher is a dual-swing flat-faced jaw crusher that can take coarse 90-mm feed and produce a <3-mm product with multiple passes. Small rolls crushers can then take the cone or Boyd crusher product and generate finer distributions <1 mm; however, this is rarely required.

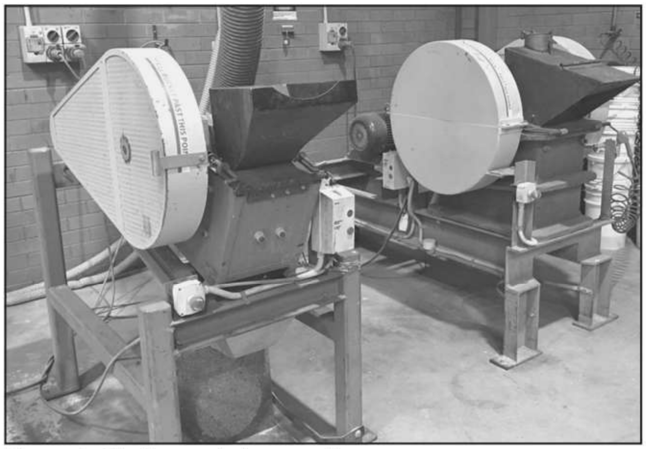


Figure 1 Single-toggle jaw crushers

Figure 2 Wescone crusher on left

Figure 1 shows two single-toggle jaw crushers decreasing in size right to left. Figure 2 is the accompanying fines crushing setup with a Wescone crusher on the left, a Boyd crusher located centrally, and the vibrating screen to the right installed for control crushing (i.e., product removal). This sort of setup facilitates the full scope of control crushing preparation from coarse to fine in a relatively efficient batch operation. All of the previously mentioned crushers have a degree of closed-side-setting adjustment to enable sample PSDs to be tailored to target.

Prior to commencing any crushing of test-work samples, process engineers and metallurgists need to carefully consider the particle size requirements for each component of downstream test work. Always starting from coarse through to fine, this determines the type of equipment and crushing methodologies employed.

Selection and collection of coarse-particle specimens must be conducted before crushing. These specimens can be taken directly from drill-core trays and/or from larger run-of-mine (ROM) particles and are used for tests such as uniaxial compressive strength testing, apparent specific gravity (SG), and Bond impact crushing test work. Once test-work composites have been defined and specimens selected, a preliminary phase of coarse crushing is often necessary to facilitate subsamples at various coarse size targets for hardness and comminution testing. When the coarse program is completed, and if required, any comminution reserves recombined with feedstock, then control crushing to mid and fine sizes for grinding can commence. The most common laboratory crusher product sizes for mill feed are <2.0 mm and <3.35 mm.

Crushing of small sample parcels for most bench-scale test-work programs (10–1,000 kg) is usually conducted by manually pouring the material into the crushers from buckets or suitably sized containers. Similar sized crushers as described earlier can be integrated with small conveyor systems to emulate continuous closed-circuit crushing systems for larger parcels between 2 and 100 t (metric tons).

BLENDING AND SAMPLE SPLITTING

Together with control crushing, accurate and precise blending and splitting of crushed samples, and downstream dried ground/processed samples, is a fundamental requirement of quality test work. This cannot be emphasized strongly enough, as any poor blending and splitting practices can compromise the veracity of subsequent test-work results and lead to incorrect conclusions. Best sampling practice should therefore be implemented throughout, and staff should be trained accordingly. There are several standards available from the International Organization for Standardization to assist in this training.

The objective is to either split out a small representative subsample from a larger mass, and/or produce a series of replicate charges for downstream test work. These test-work charges must have a high degree of precision to enable accurate comparative analysis of multiple parallel test-work programs.

Blending and splitting are invariably conducted in the same action. As for crushing, blending and splitting always commences at the largest size and mass and proceeds down to smaller quantities and size requirements. The equipment type and size and by extension, the methodologies used, are dependent on the mass to be blended.

Bulk and Large Samples

For bulk ore samples in the range of 2–6 t, large conveyor-fed rotary drum (usually 200-L capacity) dividers are available. These are usually custom engineered and fabricated. Figures 3 and 4 show 12-drum and 4-drum rotary splitters, respectively.

For larger ore parcels (>5 t), initial blending is often conducted using a skid-steer loader (Bobcat), which effectively is a large-scale cone and quartering equivalent. Large stockpiles are moved and blended three times before being spread out into windrows. The Bobcat then collects perpendicularly across the windrows to place the material into drums. At this point, the material is well blended; however, when a high level of precision is required on a plant feed, additional homogenization of the blended drums can be conducted using rotary splitters and/or large riffle splitters. The drums are presented to the splitters in a set order to ensure that every drum is mixed together.

Figure 3 Twelve-drum rotary splitter

Figure 4 Four-drum rotary splitter

Standard Samples

Standard samples measure in the 12- to 300-kg range. Well-equipped laboratories invariably use a rotary sample divider (RSD) to blend and split samples greater than 12 kg. RSDs are specialist laboratory machines whereby sample is poured into a feed hopper equipped with a vibrating feeder. The feeder transfers a slow continuous stream of material from the hopper into a rotating series of circular segmented containers. Each of the segmented containers is thus a representative split of the original mass. Laboratory RSDs are available in various sizes, from small-scale 12-segment units designed to process 12–100 kg, to large 18-segment units capable of handling 300 kg and more. With multiple feed and discharge drums in certain combinations with multiple passes, RSD units can be used to blend and split much larger parcels than the capacity of the feed hopper. The most common brand is FLSmidth-Essa, but there are many others, including Marc Technologies, Rocklabs, Qingdao Yosion Labtech, and Retsch, and local engineering shops can expediently custom fabricate to specification if required. Figures 5 and 6 showcase RSDs.

Blending is done by recombining the split segments back into the feed hopper three times before removing segment sub-splits. Selected segments from larger RSDs can be transferred to smaller RSDs to continue with the blending and splitting process down to very small subsamples from large masses.

Small Samples

Samples of <12 kg are typically blended and split using a standard riffle splitter. Riffle splitters are common items that can be purchased in various sizes from a vendor, with differing riffle vane widths to handle a range of particle sizes. Stainless steel (S/S) is the most common material of construction. Given riffle splitting is not unique to mineral processing, there are numerous suppliers available worldwide, including FLSmidth-Essa, Eriez, Marc Technologies, Westernex, Humboldt, and Sietronics. In each riffle, a diagonal plate forces the flow of material bidirectionally into two collection boxes. Figure 7 illustrates riffle splitters.

Blending is achieved by splitting the sample, recombining the halves, and resplitting three times. Subsampling is conducted by splitting the sample once and setting aside one half, then taking the other half and resplitting into quarters. This process is repeated until the appropriate size sample is achieved.

PULVERIZERS

The primary purpose of pulverizers is to take crushed and ground dry subsamples and very quickly impart high energy into the material to pulverize the sample into a fine powder (often referred to as *pulp*). Pulverized product PSDs are time dependent but are typically between a P_{80} of 70 and 20 µm.

Pulverizing is a batch process and occurs via a set of rings or a puck, oscillating at high speed within a closed bowl. The different bowl arrangements for the common Essa LM2 pulverizer are show in Figure 8. Bowls are manufactured in different sizes to facilitate various sample masses, typically from 50 g to 3 kg. Realistically, 5 g is a minimum requirement for a small bowl pulverizer.

Trace elemental contamination from the bowl, rings, or puck to the sample can be a significant issue in some pulverizing applications, and for this reason, bowls, rings, and pucks are made from a range of construction materials including carbon steel, chrome steel, tungsten carbide, and zirconia.

There are few metallurgical test-work applications where a pulverized product is suitable, because of the excessive fines generation. Pulverizers are mostly employed to prepare subsamples for assay analysis, and for this reason, sample-to-sample contamination is an extremely important consideration. Typically, a barren silica flush is conducted between samples to clean the bowl internals of any remnant of the previous sample. Free coarse gold, however, can sometimes smear onto the bowl internals and cannot effectively be removed with silica flushing. These bowls can, therefore, be reserved for very-high-grade samples.

Mechanical assistance and automation is becoming increasingly prevalent in modern laboratory pulverizers to increase sample processing productivity. The Herzog machines pictured in Figure 9 are a good example of modern automated pulverizers.

Figure 5 Rotary splitter, front view

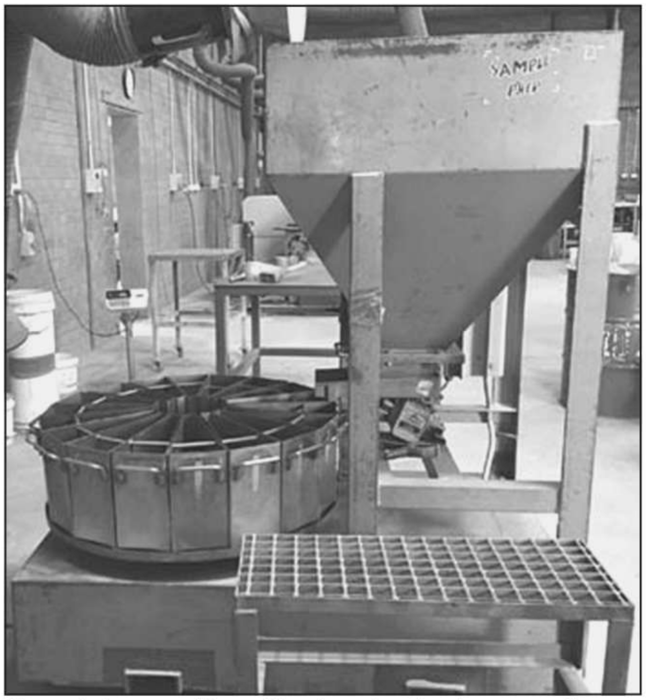
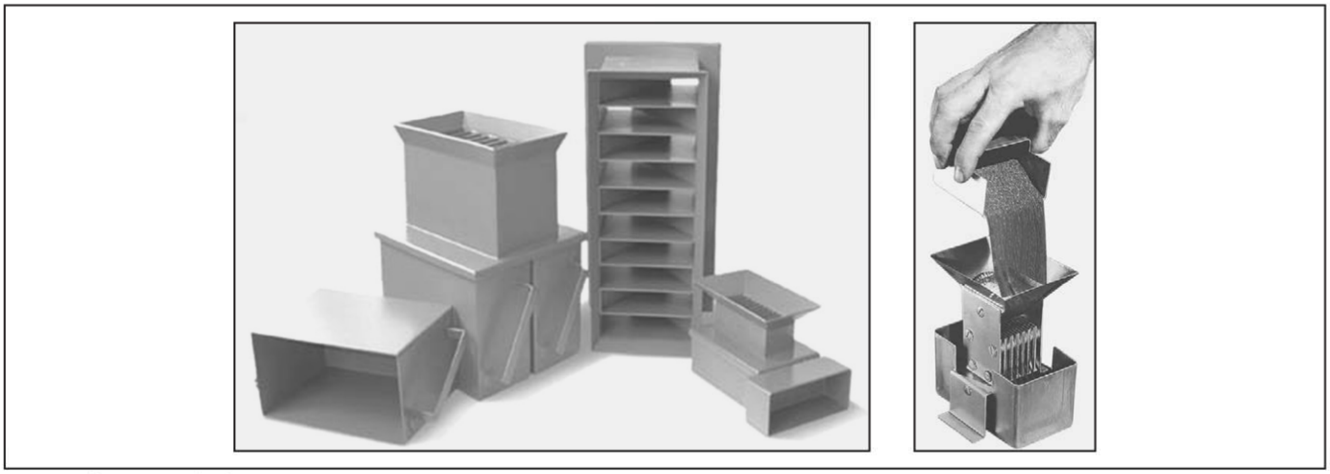


Figure 6 Rotary splitter, side view showing hopper and vibratory feeder



Courtesy of Cooper Technology

Figure 7 Types of riffle splitters

Figure 8 Essa LM2 pulverizer bowls

Figure 9 Herzog automated facility

SIZE ANALYSIS

Size analysis, sieve sizing, or screening is a critical component of metallurgical test work to determine PSD and is typically employed throughout every phase of a laboratory program. Individual chemical assays on discrete particle size fractions also serve to ascertain the metal deportment in the sample. Size analysis can be conducted wet or dry, and the employed methodologies and equipment are dependent on mass and PSD.

Dry Screening

Drying screening is routinely conducted on freshly crushed samples from 200 mm down to 2 mm, and on previously wet screened dried oversize material down to 25 µm. Laboratory screening of very coarse (+125-mm) particles is typically conducted by hand, using custom-cut frames or static screen mesh. Stacks of hand-shaken proprietary fabricated sieves can be used to screen 5- to 1-kg sample lots with particle distributions larger than 31.5 mm, as very little vibratory energy is required to allow passage of the particles through the screen mesh, and in fact, the particles can be oriented and selected by hand.

Although uncommon, orbital screen-shaking machines are available to sieve coarse particles from –125 mm to +10 mm. Notable suppliers are Marc Technologies and its version of the original *AC Cheers* screen design (Figure 10) that shakes a strapped-down stack of robust steel square 450-mm sieves. Endecotts also supplies the Titan 450 sieve shaker that can hold a stack of seven of the larger 450-mm-diameter circular sieves, with the added advantage of also being able to hold the smaller, finer aperture 200- and 300-mm sieves (Endecotts 2018).

Manufacturers of orbital sieve shakers accommodating the smaller mass-produced 200–300-mm sieves in the range of 30 mm to 20 µm are numerous and include W.S. Tyler (Figure 11), Endecotts, Haver and Boecker, Gaofu Sieving, Retsch, and Fritsch. All of these manufacturers supply the accompanying range of sieves over the full range of apertures. Typically, the sieves come in either S/S or brass casings; and in practice, there is very little difference in price or durability of the two materials.

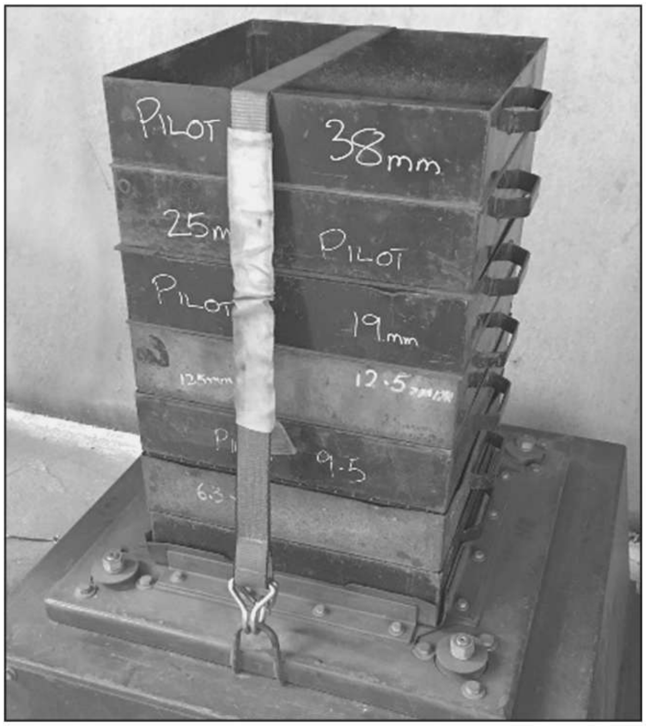


Figure 10 Steel square shaker with screens

Figure 11 Ro-Tap with screens

Material for the sieve mesh itself is S/S wire or for the very coarse apertures, punched plate. Brass wire mesh is often used down to 250 µm but is unsuitable for acidic applications. Woven S/S can now be manufactured down to 20 µm and has a much greater corrosion resistance in acidic oxygenated environments. Woven nylon fabrics can also be used for particles smaller than 45 µm.

For sample masses, coarse screening using +50-mm static screen mesh can treat large sample masses in the hundreds of kilograms, which can mean screening the entirety of the sample mass. However, appropriately sized representative subsample splits are required to process through the mechanical sieve shakers.

Dry screening efficiency is a function of open area and the number of particle strikes presented to the screen aperture. Thus, subsample feed masses to the uppermost sieve in the stack are dependent on the aperture range and, moreover, the size of the bottom sieve aperture.

Typical sample masses for the dry coarse sieving units (–50 +2.0 mm) range between 25 and 5 kg depending on the PSD. Dry screening of finer distributions between top sizes of <10 and <2 mm is invariably done on dried oversize fractions after wet screening to remove fines. For a bottom screen aperture of 300 µm and larger, a 200-mm-diameter screen stack can comfortably receive lots up to 1 kg. Below 300 µm, the lot size needs to be reduced to 200–500 g to prevent overloading and potential blinding of the finer apertures.

Wet Screening

Laboratory wet screening is routinely conducted to assist the passage of particles through the screen mesh, especially at finer apertures smaller than 500 µm. There is also an additional washing action of fines that otherwise adhere to larger particles, or agglomerate with each other, and provide a biased size distribution under dry screening conditions. The medium for wet laboratory screening is almost exclusively the local site tap water, although for water-repellent materials, a wetting agent or dispersant can be added.

Laboratory wet screening of coarser size distributions is always conducted in stages—from coarse to fine—as finer screen meshes are fragile and cannot withstand the impact and abrasion of larger particles. The number of stages is dependent on the range of the size distribution. For coarse top sizes larger than 20 mm, dry screening is usually conducted first with the fines washed and captured from the fractions, either collectively on the stack of screens or from individual fractions.

For samples and fractions of –20 mm +250 µm, wet screening is typically conducted using a single vibrating screen for each stage to achieve sufficient screening efficiency and thereby accuracy. The wet screening stages (apertures) can range in decreasing order from 6.3 mm, 3.0–1.0 mm, 800–500 µm, and 300–250 µm. In all cases, the captured screen undersize can be fed directly to the next screen aperture as a slurry. The amount of wash water applied to the screen typically increases with decreasing aperture size. It may, however, be appropriate to settle, decant, and/or filter each screen undersize prior to the next wet screening stage.

Size fractions or milled slurries smaller than 300 µm are by far the most common samples requiring size analysis in laboratory test programs. These samples can usually be screened directly down to 38 and 25 µm, thus only requiring one stage of wet screening. The exception is magnetic material, such as magnetite, which can form robust agglomerates at fine sizes and usually requires wet screening over every aperture, as described earlier for coarse fractions.

At the final wet screening stage, both the wet screen oversize and undersize are captured, filtered, and dried. A sample of the dried oversize is then dry screened over a nest of sieves as described earlier. Importantly, the dried wet screen undersize must be added to the dry screening pan undersize to achieve the correct size distribution. These two samples must be blended together if an assay subsample is required. It is not

Figure 12 Wet screening station

meaningful to only take the dry sieve nest pan undersize for assay, as the subsieve PSD is quite different.

Wet screening coarse material larger than 10 mm is usually conducted on larger sample masses using continuous 600–1,200-mm Kason- or Russell-style vibrating screens. Feed masses are adjusted accordingly to achieve an appropriate loading on the mesh. Wet screening below 10 mm is conducted on smaller subsamples using Eriez-style wet screening stations for 200-mm Endecott sieves, as shown in Figure 12. Feed masses are similar to dry screening described earlier and decrease commensurately with sieve aperture to prevent overloading of the mesh.

Cleaning and Inspection of Screens

Screens require regular inspection for aperture (mesh) damage and of aperture blinding. Damaged meshes require immediate replacement. Screens with mesh blinding can be cleared by careful brushing using soft wire brushes. This must be conducted on the underside of the screens to avoid forcing oversize particles through the mesh, which can damage or compromise the actual aperture opening size. Ultrasonic cleaners can be employed to dislodge blinded particles. This method minimizes damage to the screen cloth.

Cyclosizer

The Warman Cyclosizer still remains the most common method of subsieve analysis when assays are required on the subsieve fractions. Currently, the manufacturing license for Warman Cyclosizers is held by Marc Technologies in Western Australia, part of ALS Global. A photograph of a typical Cyclosizer is shown in Figure 13.

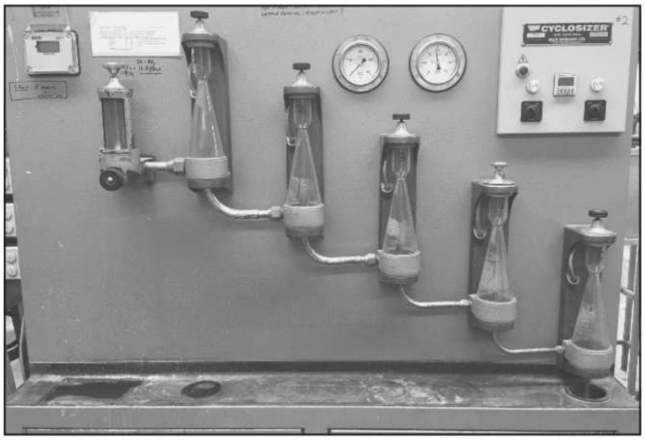


Figure 13 Cyclosizer

The practical feed size distribution for a Cyclosizer is <50 µm +10 µm. Feed masses are restricted to 50 g for higher-SG material, such as magnetite, and up to 80 g for lower-SG siliceous material. Any excess of feed mass can overload the top collection chamber of each cone, which deleteriously affects the resultant size distribution. Multiple passes of sample splits can be conducted to increase recovered fractional masses from each cone.

An accurate solids true SG determination for each sample is required, and care must be taken to apply the correct elutriation flow rate and correction factors for water temperature. It is also important to be aware of the calculated empirical nature of Cyclosizer results when combining with the sieve size distribution data. Even under the best testing conditions, there must always be a small stepwise variation at the transition point between methodologies. Selecting a final sieve size of 45 µm in place of 38 µm or 25 µm can smooth out the data transition.

Laser Sizing

Sizing using laser diffraction has become a widely accepted alterative to sieve sizing and Cyclosizers in certain circumstances, especially when no chemical analysis is required. Laser diffraction determines PSDs by measuring the angular variation in intensity of light scattered as a laser beam passes through a particulate sample dispersed in an agitated liquid medium. Larger particles scatter light at small angles relative to the laser beam, and small particles scatter light at wide angles. The angular scattering intensity data is then processed with software using the Mie theory to calculate the size of the particles, and particle size is reported as a volume equivalent sphere diameter (Malvern Panalytical 2018).

Theoretically, the technology can measure particles into the millimeter range; currently however, from a practical laboratory perspective, laser sizers are most effective for PSDs below 100 µm. Larger particles can suffer inherent error from the equivalent sphere calculations together with shape correction factors. The machines use very small subsamples of <1 g and so representative subsamples can be questionable for coarse PSDs. Coarser particles are also abrasive on the machine lenses. However, for a large proportion of primary grinds, and especially regrind products where syringe or dip sampling is effective on stirred slurries, laser sizers can provide an accurate and expedient determination of PSD.

Caution needs to be exercised with materials susceptible to agglomeration, such as clays and magnetite, as the beam

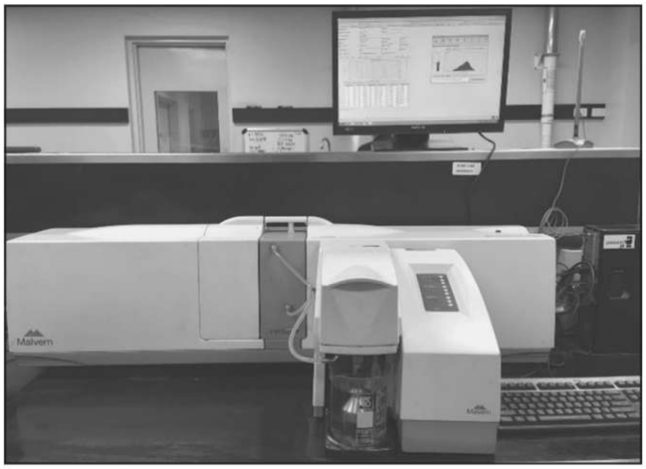


Figure 14 Malvern laser sizer

interprets agglomerates as a single particle and biases the size distribution result. In these cases, dispersants and/or ultrasonics can be employed to break up the agglomerates. A Malvern laser sizer is shown in Figure 14.

COMMINUTION TESTING

Different types of comminution testing are performed in metallurgical testing laboratories. Following are the most common.

JKMRC Drop Weight Test

The full drop weight test is a proprietary procedure developed at the Julius Kruttschnitt Mineral Research Centre (JKMRC) in Queensland, Australia. The test is conducted under license by approved laboratories using the drop weight apparatus.

Achieving the individual particle selections for a drop weight test typically requires between 75 and 90 kg of feed material consisting of either full PQ or HQ drill core or suitably sized ROM ore. The feed material is carefully control-crushed and screened for individual particle selection. Approximately 25 kg of actual sample material is selected in coarse-particle sets in the range of 63 to 13.2 mm (5× fractions of 30 particles) for drop weight impact breakage testing at three energy levels per fraction: 30 particles of –31.5 m +26.5 mm for apparent SG determinations and two 3.0-kg subsamples of –55.0 +38.0 mm for low-energy breakage and abrasion testing via a tumble mill. Where mass availability is limited, all unused material, including broken test products, can be recombined and prepared for further metallurgical testing.

The drop weight test is highly prescribed by the JKMRC proprietary data sheets, and all elements of the test must strictly adhere to the procedures and tolerance parameters. Raw laboratory data must be sent to JKMRC to generate the final test data and report. The test generates the appearance function (e.g., breakage pattern) of the ore under a range of impact and abrasion breakage conditions, which is subsequently reduced to three parameters: A, b (impact breakage), and t_a (abrasion breakage). These ore-specific parameters are used in the JKSimMet mineral processing simulator software and JKSimMet crusher model.

SMC Test

The SMC Test is another proprietary test developed by SMC Testing Proprietary Limited and is conducted under license using the exact same drop weight apparatus just described for the JK drop weight test. The intent of the test is to provide a cost-effective means of obtaining impact breakage parameters from drill core or broken ROM rock samples in situations where limited quantities of material are available.

Achieving the individual particle selections for an SMC Test typically requires approximately 25 kg of feed material consisting of either full or half PQ/HQ drill core. NQ core can also be used, or suitably sized ROM ore. Approximately 5 kg of actual sample material is selected to achieve a set of 120 particles within a single size fraction. The SMC Test can use one of three size fractions depending on the material characteristics.

The current recommended size fraction is the coarsest (–31.5 +26.5 mm range), as these particles provide superior confidence in the database. The next size fraction of –22.4 +19.0 mm is still common because it allows for the full use of quartered core feed samples, and the results are still reliable given the comprehensive data set gathered since the inception of the SMC Test. The finest size fraction of –16.0 +13.2 mm should only be used where the sample characteristics cannot provide the coarser particles. As per the drop weight test, all unused material, including broken test products, can be recombined and prepared for further metallurgical testing.

The SMC Test is also highly prescribed by the proprietary data sheets provided by SMC Testing Pty Ltd., and all elements of the test must adhere strictly to the procedures and tolerance parameters. Raw laboratory data must be sent to SMC Testing Pty Ltd. to generate the final test data and report, and this must include the source information of the sample. Raw data expressly cannot be provided to third parties.

The test generates the drop weight index (DW_i), which is a measure of the strength of the rock when broken under impact conditions and has units of kilowatt-hours per cubic meter ($kW \cdot h/m^3$), as well as the A and the b parameters. The test also provides an estimated value of the t_a, as well as the M_{ia}, M_{ic}, and M_{ih} comminution parameters. The M_{ia}, M_{ic}, and M_{ih} parameters together with the M_{ib}, which is obtained as part of the Bond ball mill grindability test, can be used to evaluate autogenous grinding mill/ball mill and high-pressure grinding roll/ball mill circuits. The A and b values can also be used directly in JKSimMet (refer to Chapter 2.5, "Modeling and Simulation") for plant design, expansion, and optimization. The SMC Test does not generate the crusher parameters, which must be obtained through a full drop weight test.

Bond Impact Crushing Work Index

The Bond impact crushing work index (CW_i) test (Bond 1946) uses selected specimens in the nominal size range of +75 –51 mm. The specimens are placed on a pedestal with the parallel surfaces (and the smallest dimension) perpendicular to the raised striking hammers, and subjected to progressively higher impact energy levels until fracture.

The selection of the CW_i specimens is particularly important for the test and also in the context of the test-work program. Consideration for specimen selection must be made in the initial stages of the preparation process, usually before any material is crushed. The selected specimens should be *natural* and represent the type of broken material to be presented in the feed to a full-scale crusher. Specimens should be of even aspect ratio and have two near-parallel faces for the hammers to strike. Specimens should not be dried. Typically, between 10 and 20 specimens are selected for each test, and obviously the larger sample set provides better statistical confidence in the results.

Worked samples, such as drill core or material cut with a saw, ideally should be avoided, but practically, this is often not possible when core is the only material available. As such, consideration must be given to the results in that worked specimens tend to give lower breakage energy values. Half core should only be tested as a last resort.

Under the best testing conditions, there is still a high degree of variability in Bond CW_i results, as there are many sources of variability relating to the quality of the contacts between hammers and specimens, the selection of the specimens, and the measurement of the force.

Bond Abrasion Index

Abrasivity is a term that relates to the ability of rock material to cause wear to structural materials after repeated contacts. Wear can be described as the process of damaging, diminishing, eroding, or consuming by long or hard use, attrition, or exposure.

The Bond abrasion index (A_i) test measures flow wear, which is the movement of material across a surface with little or no normal force (Bond 1963; Angove and Dunne 1997). The test relies on measuring the loss of mass from a metal paddle that counter rotates in a drum of rock particles sized to a specific range. The testing machine consists of an impeller rotating at 632 rpm within a contra-rotating drum. The impeller incorporates a Bisalloy 500-Bhn (Brinell hardness number) steel paddle.

The test requires 4 × 400-g charges control crushed and screened to –19 +12.7 mm. For planning purposes, a 5-kg subsample of –20-mm material, rotary or riffle split during the initial stages of sample preparation, suffice. The four lots of 400 g are successively processed for 15 minutes each in the testing machine. At completion of the test, the weight lost by the paddle is measured to 0.10 mg. The A_i is equivalent to the paddle weight loss in grams but is expressed as unitless value.

The test provides consistent results if the correct paddle metal is used and the other test parameters are closely followed. Various laboratories use slight variations in test parameters, such as drum speed, paddle material, and particle size, so caution should be exercised when comparing results from different sources.

Broad guidance for interpreting Bond A_i results is as follows:

- Nonabrasive, <0.01
- Moderately abrasive, 0.01–0.2
- Abrasive, 0.2–0.4
- Highly abrasive, 0.4–0.6
- Extremely abrasive, >0.6

Bond Rod Mill Work Index

Although rod milling is rarely used in modern comminution circuits, the Bond rod mill work index (RW_i) determination still holds significant value in observing the breakage behavior of larger particle sizes comparative with finer breakage mechanisms seen in ball mills (Bond 1961a; Angove and Dunne 1997). As such, RW_i is still a common requirement for a comminution program and is invariably conducted in conjunction with the Bond ball mill work index (BM_i) determination. The two indices are frequently compared as inputs to modern simulation programs that can mathematically model comminution circuits and predict power requirements.

Figure 15 Bond rod and ball mill comminution station

The Bond RW_i requires feed material to be control crushed to <12.5 mm, and so this is also conducted during the initial stages of a sample preparation program before any fines crushing. Typically, a 10–15-kg subsample of coarse crushed material is riffle or rotary split for a Bond RW_i and at least 8–10 cycles are allowed. In situations of limited mass, unused feed charges can be recombined and crushed further for downstream test work. Feed charges are prepared by filling and compacting a 1,250-mL cylinder. The closing screen size for a Bond RW_i is fixed at 1,180 µm.

FLSmidth-Essa is a well-known supplier of Bond mills, and in many cases, the units are fabricated and supplied by local engineering vendors according to the Bond specification. Modern engineering, together with electronics, allow for excellent sample and media handling ergonomics, safety interlocks, and revolution counters. Figure 15 shows a Bond RW_i and BW_i comminution testing station.

Regarding the laboratory Bond RW_i, over many decades, some laboratory regions have replaced the original wave liner mill profile for a smooth shell, which produces slightly higher RW indices in the order of 1.0 to 1.5 kW·h/t. This only has significance if results of one mill type are being compared to that of the other as outliers against the prevailing engineering data sets for the project or region.

Bond Ball Mill Work Index

With ball milling still a fundamental grinding unit process of many mining operations, likewise the Bond BW_i determination is a critical inclusion in most comminution test-work programs (Bond 1961b; Angove and Dunne 1997).

The Bond BW_i test requires material crushed to <3.35 mm, and so representative subsamples can be split at the final stages of sample preparation. For sample mass planning purposes, 10–15 kg comfortably allows for 8–10 test cycles. For low mass availability, 6 kg usually facilitates a minimum of six cycles for material of typical bulk density, however, this is insufficient for high bulk density samples such as magnetite.

Special care should be taken during the control crushing of the feed sample to avoid excessive fines generation. Overcrushed samples with a high fines distribution will bias results against the true BW_i. Zero cycles (whereby product material is screened out of the feed lot without milling) can be conducted on materials with naturally fine PSDs. There is a point, however, where it is practical for other tests, such as the Levin grind test (Levin 1989), to be used for finer feed size distributions.

BW_i differs from RW_i in that the closing screen aperture is routinely changed to suit the likely PSD of the project. The most common closing screen size is 106 µm, but engineers should be aware that this is not standard and that there is little value in assessing the grindability (BW_i, in kilowatt hours per metric ton) of a sample at a product size well away from the projected grind size target.

Rules of thumb for selection of closing screen apertures in the root 2 series relative to the target P_{80} are as follows:

- 1 × root 2 series above P_{80} for samples exhibiting medium hardness/competency
- 2 × root 2 series above P_{80} for very soft samples
- ½ × root 2 series above P_{80} for very hard/competent samples

Common selections are 75 µm (for finer distributions) or 150, 212, 255, 300, and 355 µm for coarser applications.

All aspects of the Bond BW_i test are highly prescribed and standardized, so theoretically, there should not be significant variances between laboratories testing the same samples and ore types. BW_i, however, is susceptible to errors in precision and accuracy if any elements of the test are not properly conducted.

Insofar as the test rig itself, the mill shell integrity, mill speed, and revolution counts, together with maintenance of the media, are elements that need strict control. The mill charge inside a Bond mill is shown in Figure 16. The prescribed mass of the steel ball media charge must be exact for every test, which can be achieved by minor manipulations of the ball distribution. However, balls must be replaced periodically to account for wear.

Ancillary practices, such as accurate sieve sizing and sample splitting, have a high impact on the BW_i result. Having trained technicians able to discern when the test has reached equilibrium is also imperative for meaningful results. Slippage in any of these areas gives decreased precision and accuracy error.

Figure 16 Ball media charge in a Bond mill

LABORATORY GRINDING MILLS

Grinding holds the same fundamental importance in the laboratory as in commercial operations, with most test-work programs requiring further size reduction following dry crushing and of course, the addition of water to change the material into a liquid-slurry medium. Laboratory dry grinding is rare and is not discussed in this chapter.

Laboratory grinding is specifically focused on achieving a target PSD and much less on specific energies and efficiencies. A very high degree of sample recovery from the mill is also imperative for mass reconciliation of the subsequent test, and for this reason, small-scale laboratory mills do not have the bolt-in liner/lifter arrangement seen in industrial mills, which can trap material. The mill shell and liners are usually integral with each other and mostly metal. Modern mills are increasingly smooth profiles, although lifters can be welded in, or cast inserts can be manufactured to slide into shell sleeves.

Grinding is usually carried out in small lots to match the test-work mass requirements, which often requires multiple grinds per test. Typically, 1-, 2-, and 10-kg sample splits of <2.0 mm or <3.35 mm crushed material are ground in discrete parcels to feed the downstream test work.

Achieving the target PSD is done using two methods: grind establishment or stage grinding. A grind establishment (or grind curve) is conducted when sample mass permits. Representative parcels of crushed ore are ground for various times, with the mill product sized over the appropriate sieve series. The P_{80} of the products at certain apertures can then be plotted with time to determine the grind time required to achieve a target PSD.

Stage grinding is conducted when sample mass is limited. In this procedure, the limited mass of material is ground for short increments, and the mill product for each grind is sized over the desired sieve aperture. The oversize is returned to the mill for another cautiously estimated time increment.

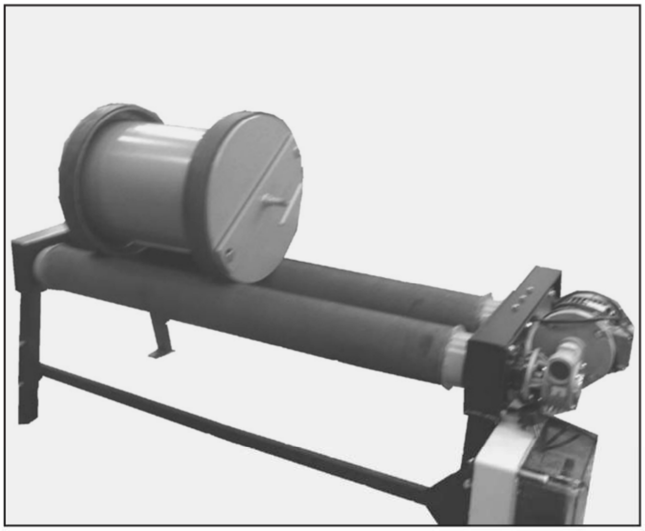
An alternative stage-grinding method is to take a small dip sample (from the agitated mill slurry charge), size it, and return the sample to the mill, then grind further for an additional time increment. However, inherent error from the dip sampling is introduced by this alternative method and it is not recommended.

One common request that process engineers often ask of laboratory metallurgists is to grind a bulk batch of slurry to an agitated tank, and then subsample for subsequent test work. Once again, the inherent subsampling error in this method is large, greatly affects the results of downstream testing, and should be immediately disregarded.

Mill selection is dependent on the sample material characteristics and the target PSD. Typical laboratory mills are rod, ball, and stirred media mills, which are discussed in the following sections.

Rod Mills

Despite being ostensibly obsolete in plant practice, rod milling still accounts for the vast majority of laboratory primary grinding. The reason for this is the natural tendency for tumbling rod media to preferentially grind the coarsest particles and thus prevent overgrinding and excessive fines production. The rod mill allows laboratories to accurately and consistently achieve a target PSD and thereby include grind size as an investigative variable, or alternatively, effectively eliminate grind size as a source of error in the test.



Courtesy of Eriez

Figure 17 Barrel mill and roller

The old-style independent barrel mill on a roller (Figure 17) is still in widespread use in many laboratories. It is a simple system whereby the grinding mill barrel is loaded with media, ore charge, and water on the bench, closed/sealed, and manually transferred on and off the rollers. Mill speed (measured in revolutions per minute) and grind time control are done through the roller mechanism. The manual lifting requirement introduces a weight limitation to the size of the barrels and also the potential for staff injuries. Some roller mill designs can have a central end opening to allow air into the grinding chamber, which can be advantageous to promote pulp-oxidizing conditions when using mild steel (M/S) media.

The modern suspended shaft and yoke design (Figure 18) alleviates most health and safety issues with a three-way pivot-locking pin arrangement that allows the supported mill barrel to be tilted up for loading and media removal and cleaning, down for slurry discharge, and horizontal for milling. Enclosures and cubicles, with associated timers and control interlocks, provide additional safety measures. The independent skid platform also improves general loading and discharge ergonomics over roller mills by allowing the entire grinding procedure to be conducted in the same space.

FLSmidth-Essa is a well-known supplier of laboratory grinding mills and Eriez also supplies a range of roller mills. Increasingly, there are many manufacturers that can be sourced quickly online by searching for "laboratory grinding mills."

The most common mill size is the 10-L barrel (~300 mm long × 210 mm in diameter), which grinds 1-kg charges. The suspended yoke design can also be fabricated in larger sizes such a 40-L barrels, which can grind 10–15-kg charges. Other types of mills are readily available, such as small-scale trunnion bearing mills and girth-driven tilt mills, but these are usually larger-volume units for bulk batch milling or small-scale pilot milling.

Materials of construction for the mill shells and rod media are typically M/S and S/S. The most common arrangement is a combination sealed S/S shell with S/S rods, as this has been proven to correlate well to industrial mills. However, all other combinations are available, that is, M/S shell–M/S rods, M/S shell–S/S rods, and S/S shell–M/S rods. The roller-style mill barrels are also available in ceramic, and ceramic rods can also be sourced.

Figure 18 Rod mill showing suspended yoke design

Rod mills comfortably grind charges down to a target particle size of a P_{80} of 38 µm. Finer grinds as low as 20 µm can be achieved with highly extended grind times; however, in these cases, a ball mill or stirred mill are more suitable.

Ball Mills

Laboratory ball mills are most often the exact same rod mill shells or roller barrels simply filled with ball media instead of rods, and most of the preceding points on overall design and materials of construction can be applied. Other media types can be used such as cylpebs, which are slightly tapered cylindrical grinding media with a ratio of length to diameter of unity (Shi 2004).

Graded ball charges do not preferentially grind coarse particles, and as such, ball mills can be expected to produce a greater proportion of fines for the equivalent rod mill grind time, especially for the coarser P_{80} target particle grind sizes. For the same reason, ball mills can be advantageous for finer target P_{80} size distributions and can realistically grind down to 20 µm in practical grind times. Finer particle size targets require a fine grinding (stirred mill) apparatus.

Fine Grinding Mills

General fine grinding duties in the laboratory to achieve target particle P_{80} size distributions of 25–10 µm can typically be facilitated through a stirred media mill. Applications include very fine grinding conducted as a second stage after rod or ball

Figure 19 Laboratory stirred mill

milling and regrinding of intermediate test product streams, such as flotation rougher concentrates.

Few laboratory equipment manufacturers supply small-scale stirred media mills suitable for metallurgical test work; however, the apparatus is simple, and custom fabrication can be readily achieved by local engineering shops. An example of a laboratory stirred media milling setup is pictured in Figure 19. In some cases, a simple overhead drill machine can be modified to suit. Typically, the *pots* are S/S to hold media charges of 1, 2, and 4 kg. The stirrers are usually simple horizontal rods and pins or rings attached to the shaft. Types of media vary from steel, zirconia, alumina, glass, and sand. Media size is generally in the range of 1–5 mm.

Many of the major industrial fine grinding mill suppliers, such as Glencore (IsaMill), Outotec (HIGmill [high-intensity grinding mill]), Metso (stirred media detritor [SMD] and Vertimill), and FLSmidth (VXPmill) do produce laboratory-scale testing rigs that can run batch continuous tests to determine specific energies for various sample streams and ore types. Mass requirements for these mills vary from 20 to 100 kg per test, which obviously precludes these machines for general laboratory duties; however, such machines can be applied in bulk batch grinding and piloting duties. A 20-L IsaMill is pictured in Figure 20.

GRAVITY SEPARATION

Although gravity separation encompasses an array of industrial unit processes in the recovery of heavy minerals and precious metals, from a laboratory bench-scale test-work perspective, an indicative value of gravity recovery (mass yield and grade) suffices for most engineering studies. Larger-scale bulk or continuous testing can be commissioned by equipment vendors on specific unit processes (i.e., spirals and jigs) to firm up design criteria closer to construction.

The two most common laboratory machine types to provide reliable indicative laboratory gravity separation data, however, are shaking tables and centrifugal concentrators, which are described in the following sections.

Shaking Tables

The two major laboratory shaking tabling options are the Wilfley 800 (0.8 m^2; 640 × 1280 mm) and the Gemeni GT60 (1.3 m^2; 894 × 1490 mm). The Wilfley table is shown in Figure 21, and the Gemeni table is shown in Figure 22.

Figure 20 IsaMill

The Wilfley table is available from Holman-Wilfley in the United Kingdom and Motive Traction in Australia. The table has an adjustable table angle and oscillation stroke. Laboratory machines typically have the end launder compartmentalized to capture up to 10 graded products across the width of the table, and the front tailings launder can also be split into two to four compartments. The Wilfley can be operated batch continuous with samples from 1 to 25 kg, or continuously at <75 kg/h. Feed slurry is typically via an agitated header tank at 40%–70% solids with wash water added between 2 and 12 L/min.

The Gemeni table is available through Mineral Technologies in Australia. The table is designed to operate on a flat floor but has an adjustable oscillation stroke. The table is double sided and can be operated with one side only or both. There are three launder outlets per side. Batch samples as little as 1 kg can be treated with one side and ~20 kg with both sides in operation. The Gemeni can be operated continuously at 40 kg/h at 60%–70% solids using an agitated constant header tank with wash water added at ~12 L/min.

Centrifugal Concentrators

Centrifugal concentrators are primarily applied to the gravity recovery of gold and platinum group metals but can also find test-work applications in other heavy metal ores, such as mineral sands, chromite, tin, tantalum, tungsten, iron ore, and cobalt, where there are sufficient differences in the SG of the valuable mineral compared to the gangue mineralization.

The two laboratory centrifugal concentrators of note remain the KC-MD3 Knelson, supplied worldwide by FLSmidth, and the Falcon L40, supplied worldwide by Sepro Mineral Systems.

The KC-MD3 Knelson concentrator is supplied with a 50-mm 4× rib bowl that spins at 60 *G* and collects ~60–80 g of product material per batch run. Typical batch evaluation masses for the Knelson concentrator range from 1 to 20 kg.

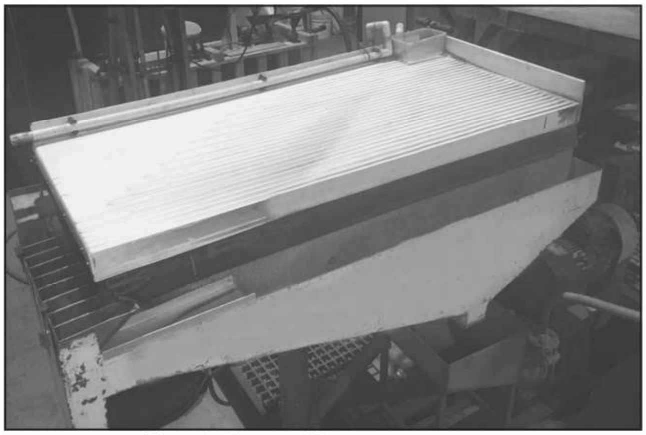


Figure 21 Wilfley shaking table

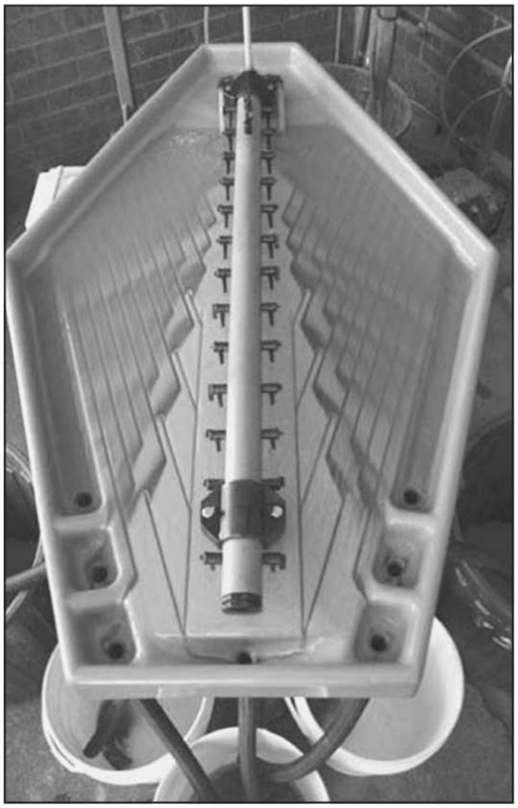


Figure 22 Gemeni shaking table

Figure 23 Knelson laboratory separator

Figure 24 Bowl for Knelson separator

Slurry feed is usually managed from a small header tank to ensure constant feed between 20 and 50 kg/h but can be manually tipped in for small samples. Fluidization water is supplied at up to 1 bar at 3–5 L/min and can be manually adjusted and measured prior to the test or by using a flowmeter. The KC-MD3 is pictured in Figure 23 and a photograph of the bowl in Figure 24. FLSmidth is currently also supplying two slightly larger laboratory units in the KC-MD4.5 and KC-MD7.5, which can process small pilot-scale flow rates (FLSmidth 2017).

The Falcon L40 is supplied with two types of 100-mm bowls that spin at 100–250 *G* using variable speed. The SB (semi-batch) dual-ribbed bowl takes fluidization water at <1.5 bar at 5–9 L/min and holds 80–120 g of product per batch run. The SB bowls have two bowl angels: 14 and 28 degrees. The 28-degree bowl is used for coarser samples. The UF (ultrafine) ribless bowl requires no fluidization water and holds ~200 g of product per batch run.

Typical batch test evaluation masses for the Falcon range from 1 to 50 kg. The slurry feed rate is typically managed from a header tank to ensure constant feed between 50 and 250 kg/h (with the lower feed rate for coarser samples) but can also be manually tipped in for small samples. The Falcon L40 is shown in Figure 25 and a photograph of the two bowl types in Figure 26, with the SB bowl on the left and the UF bowl on the right.

Figure 25 Falcon laboratory separator

Figure 26 Falcon semi-batch bowl (left) and ultrafine bowl (right)

Gravity assessment via centrifugal concentrators is routinely added in bench-scale test-work programs ahead of small-scale tests, such as flotation and leaching, and sometimes after testing, as required. The bowl concentrates are recovered for downstream testing and/or analysis. For gravity gold concentrates, the two main treatment methods are mercury amalgamation and intensive cyanidation. Because of the tendency to over-recover precious metals on a bench-scale relative to full-scale operation, mercury amalgamation is the preferred method, as it generally only recovers the coarse/free gold, whereas intensive cyanidation also extracts fine-grained or partially liberated gold particles. Thus the gravity recoverable gold (GRG) content can be overestimated.

For gravity testing of larger sample parcels, an assessment of the batch size needs to be made relative to the estimated heavy mineral content versus the bowl size. Laplante and Doucet (1996) have described the industry-accepted method for the determination of GRG, whereby 50–100 kg of sample is passed sequentially through the Knelson concentrator at three successive grind sizes, ranging from a P_{80} of 850 µm to 75 µm. At each stage, the Knelson concentrate is collected and subjected to size by assay gold analysis. The final-stage Knelson tailings is also subjected to size by assay gold analysis, and using these data, an accurate GRG content can be determined.

FLOTATION

Froth flotation remains a widely used industrial beneficiation technique for most base metal mines and an increasing number of other nonsulfide ore bodies, such as iron ores (reverse silica flotation), phosphates, oxides, and rare earth minerals.

In recent decades, the design of industrial flotation cells has advanced dramatically, with manufacturers now producing ever larger and increasingly more efficient cells for the modern beneficiation plant. Traditional small-scale benchtop flotation programs are therefore still required to determine the fundamental flotation performance parameters respective to the ores, such as grind size, residence times, and reagent optimization. In this regard, bench-scale flotation test-work procedures and equipment have remained relatively static, as described in the following sections.

Bench-Scale Flotation Cells

The two stalwart benchtop flotation cells used in the overwhelming majority of test-work programs worldwide are the Metso Denver D12 and the Agitair LA-500. Both machine types can be bought under the original specification and branding; however, equivalent copies are now available from many online suppliers.

Both types of cells share a similar overall configuration (Figures 27 and 28), with a flotation mechanism suspended over a cell (tank), supported by a rear column. The Denver mechanism can be raised and lowered into and out of the cell using a rack-and-pinion gear set in conjunction with a spring-loaded shaft and can be locked at any desired height. The Agitair has a fixed mechanism whereby the shaft/rotor is removed through a flange. Typically, both cell types have installed air/nitrogen injection with the appropriate pressure and volume gauges and manual regulators. The Denver cell, however, can operate fully self-aspirating if required. Other standard features are power actuation and rotor-revolutions-per-minute control. The differences between the two cell types lies in the rotor and stator (diffuser) design together with the associated cell. Figures 29 and 30 showcase each design for comparison.

The Agitair design shows the larger rotor sitting centrally in the stator, which is installed as part of the cell. The rotor supplies the energy to suspend the slurry in the cell as well as shear the air/gas bubbles, while the stator ensures a balanced fluid-flow pattern inside the cell to further diffuse/shear the air bubbles. Agitair cells typically run at rotor speeds of 600–900 rpm and can realistically treat PSDs smaller than 300 µm. Coarser sizes tend to sand around the stator.

The Denver cell has an integrated rotor and diffuser design, which comes in large and small sizes and either open or closed; as can be seen, the cell has no other installed parts. The proportionally smaller rotor in the Denver cell typically runs at higher speeds of 900–1,400 rpm and delivers commensurately higher energy inputs and tip shear. This does draw criticism from some industry sectors; however, the Denver cell can suspend PSDs up to 1 mm, which can be advantageous in flash flotation test-work applications.

Each cell design has its supporters and detractors in different laboratories and regions; however, the arguments are

Figure 27 Agitair LA-500

Figure 28 Denver D12

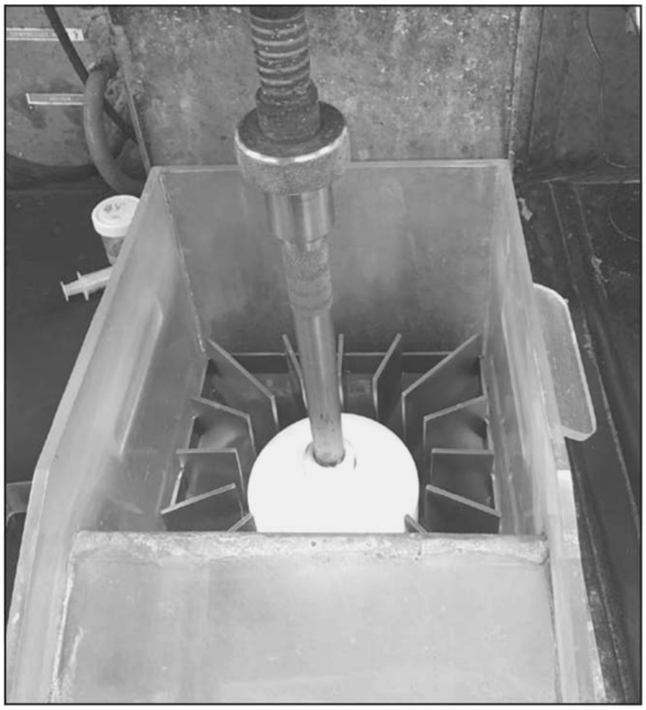


Figure 29 Agitair-type stator and rotor

Figure 30 Denver-type stator and rotor

Figure 31 Small continuous pilot flotation cells

largely moot if one cell type or another is used consistently throughout a test-work program and, moreover, matches with local engineering scale-up conventions.

Masses that can be treated in these bench-scale machines range from ~250 g to ~4 kg, which is facilitated by a range of cells and rotor sizes. Cell size selection is also related to the target test percent solids, which is typically 25%–40% for rougher tests and <20% for cleaning duties. A range of cell sizes is pictured in Figure 31 for both types of machines and usually includes 0.25, 0.5, 0.75, 1.0, 1.6, 2.2, 4.4, and 6.6 L for the Agitair cell, and 0.5, 0.75, 1.0, 2.0, 4.0, and 8.0 L for the Denver cell.

Materials of construction for the cells are clear Perspex, S/S, and ceramic. Agitair cells are usually Perspex, and Denver cells are typically S/S. Both rotors and cells can be purchased readily online and/or fabricated by local engineering shops.

Larger Batch Cells

At some point, most test-work programs for the larger feasibility studies require a larger mass of final flotation concentrate and also tailings to be produced to facilitate vendor testing (thickening, filtration, geotechnical, viscosity, etc.) and samples for marketing. This can be achieved by conducting multiple 4-kg tests; however, a more expedient and cost-effective method is to use a larger batch flotation cell. In this case, the Agitair cell type comes readily available in 20-L, 40-L, 75-L, 130-L, and 300-L cell sizes. Obviously, flotation residence times need to be scaled up (usually by two to three), and consideration must be given to rotor sizes and energy inputs comparative to the smaller cells. Some comparative optimization tests may be required to ensure similar test performance when using the larger cell size. An example of a 40-L Agitair bulk batch cell is shown in Figure 32.

Other Ancillary Equipment for Flotation Test Work

In the wider context of metallurgical test work, flotation testing cannot be conducted without a range of other ancillary laboratory equipment and infrastructure. This includes items such as a stop watch, a range of syringe sizes and droppers for reagent addition, beakers and stirrers for reagent storage and preparation, a pH and oxidation-reduction potential (ORP) meter and probe setup, hand scrapers and paddles for froth removal, wash bottles to clean the cell and probes between stages, and aluminum trays for capturing the recovered froth.

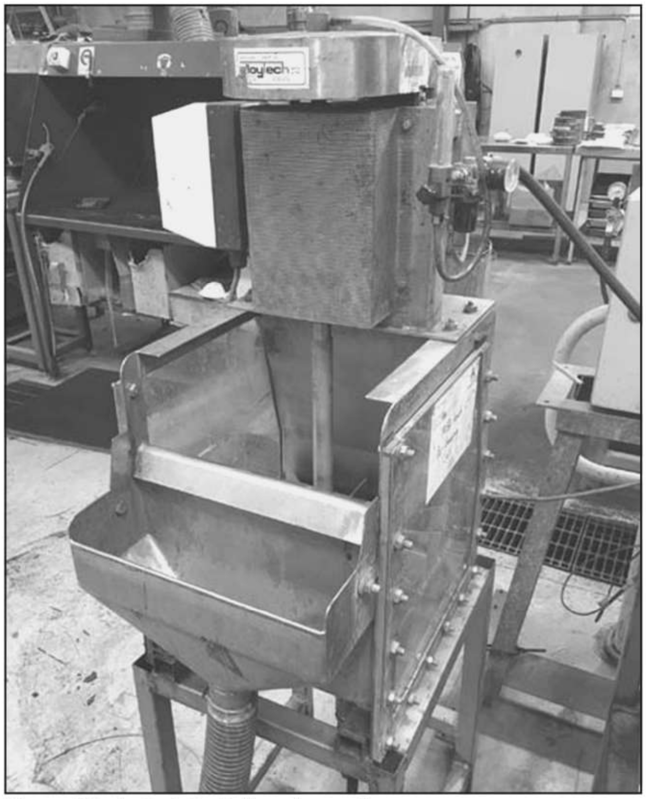


Figure 32 Agitair bulk flotation cell

Other post-test requirements include Buchner vacuum filtration flasks and funnels, vacuum filters, filter papers, sample tags, and drying ovens. Larger external infrastructure includes a compressed air system, vacuum pump system, nitrogen bottle storage and delivery plumbing, and possible air-extraction systems to remove noxious reagent odors from the laboratory. Figure 33 shows a selection of the benchtop ancillary items mentioned.

MAGNETIC SEPARATION

Laboratory magnetic separation is a frequent requirement for many metallurgical test-work programs, particularly for iron ore, mineral sands, lithium, and vanadium projects. In this regard, laboratory testing has always been particularly well served by the equipment manufacturers of industrial-sized magnetic separators of all forms, and this continues to be the case.

Generation of the magnetic field is via either permanent magnets or electromagnets, and there is a vast array of machine types available to handle either dry or wet applications. Common small-scale laboratory dry and wet units are discussed in the following sections.

Dry Magnetic Separators

Many dry magnetic separators are available with a small benchtop footprint. These are induced roll magnets, permanent rare earth magnets, and electromagnets. Typically, these machines can treat dry material from 10 μm to 5 mm at gauss settings between 0 and 16,000. With this range, the entire spectrum of highly susceptible particles to weakly susceptible

Figure 33 Ancillary equipment for batch flotation test work

Figure 34 Frantz laboratory separator

paramagnetics can be assessed. Sample masses range from as little as 5 g to 10 kg, although most of the units can effectively be continuously run if product containers are replaced as required.

Laboratory data can be easily scaled for industrial-sized plants. Common brand names and types are

- S.G. Frantz LB-1 magnetic barrier separator (United States),
- Sepor MIH(13) high-intensity induced-roll magnetic separator (United States),
- Mineral Technologies Reading magnetic separators (Australia),
- Mecal magnetic separators (Australia), and
- Eriez 534 rare earth magnetic roll separator (worldwide).

Bench-scale dry magnetic separators are pictured in Figures 34 to 37.

For test-work programs requiring larger masses, Eriez in particular produces a range of laboratory-scale dry low-intensity, medium-intensity, and high-intensity magnetic separators that can process tens to many hundreds of kilograms of sample. These machines have an extremely long lifespan of many decades when maintained correctly, with negligible loss of field intensity. As such, the age of a machine should generally provide no barrier to achieve accurate results when employed by competent laboratory staff.

Figure 35 Mecal magnetic separator

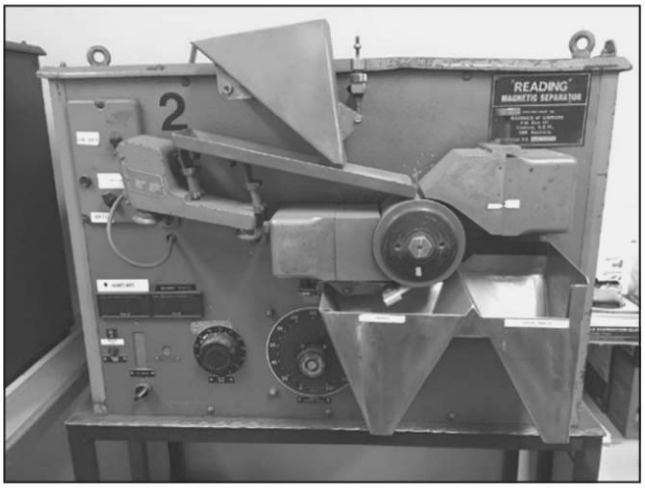
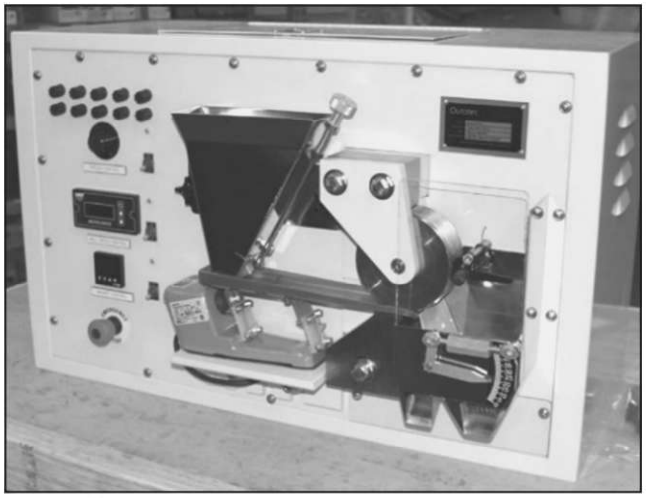


Figure 36 Reading magnetic separator

Wet Magnetic Separators

Wet magnetic separation is usually applied to samples post milling, typically <1 mm down to slimes fractions. Just as for dry magnetic separation, field strength is applied using either rare earth permanent magnets or current-induced electromagnets.

By far, the most common laboratory wet magnetic separation apparatus is the Davis tube, which remains essentially unchanged since its inception in 1921. Pictured in Figure 38, the Davis tube supports an angled oscillating glass tube placed between the poles of two magnets up to 4,000 G. Sample masses of 20 g are utilized for the test at PSDs of <1 mm. Typically, the sample charge is ground or pulverized to a range



Courtesy of Sepor

Figure 37 Sepor magnetic separator

Figure 38 Davis tube

between 150 and 20 µm. Wash water is applied to the tube and, in conjunction with the rotating oscillation action, facilitates a vigorous washing action to reject weakly magnetic particles. Multiple Davis tubes are often set up together with LM2 pulverizers to enable mass production of Davis tube wash tests, which are sometimes incorporated into both geological and metallurgical modeling.

Proprietary wet separators can be grouped into three broad categories:

1. Low-intensity and medium-intensity magnetic separators (LIMS and MIMS)
2. Small-chamber vertical wet high-intensity magnetic separator (WHIMS)
3. Vertical pulsating high-gradient magnetic separator (VPHGMS)

Laboratory-scale units are available in all types. Notable brands are Eriez, Steinert, Longi, and SLon/Outotec.

A well-known and versatile laboratory LIMS is the Eriez L8 drum electromagnet (Figure 39A), which can characterize 5–10-kg samples at 500–1,100 G in batch continuous mode. These units can also be used in small-scale pilot operations in cascade mode.

For WHIMS applications, the traditional small-chamber vertical-flow WHIMS units are still available from Eriez (Figure 39B); however, these units are not effective in recovering fines (<100 µm) and have largely been superseded by VPHGMS units.

The VPHGMS system involves a vertical ring of rod matrices rotating through a bath of slurry surrounded by a high-gradient electromagnet (up to 1.8 T [tesla]). Magnetic particles are attracted to the rod in the matrix, and the bath is continually pulsed to allow washing and separation of the nonmagnetic particles. The magnetics are then washed from the matrix into a concentrate trough once the ring rotates out of the field. Different-sized matrix rods and spacing configurations are available to treat various particle sizes up to 3 mm. Cooling water, at approximately 4 m^3/h, is required for the electromagnets in the VPHGMS units. To avoid excessive water consumption, a cooling tower or heat exchanger infrastructure is usually needed.

The laboratory SLon 100 VPHGMS is a system using a single matrix in a pulsating bath that requires continuous filling with water to maintain the level and wash the matrix. Sample masses at 230 g per test can be processed. The slightly larger SLon 500 and the equivalent Longi 500 can treat slightly larger ore samples of 30–60 kg. Both the SLon 100 and the Longi 500 are pictured in Figures 40 and 41.

DENSITY MEASUREMENT AND SEPARATION

This section describes the broad methodologies and equipment used for density measurements and heavy liquid separation.

Density Measurement

Density measurement typically falls into three categories: bulk density, apparent SG (or in situ SG), and true SG. Bulk density determinations are simple tests whereby a vessel of known volume is filled with mass of sample and weighed. The result can be uncompacted or compacted (effected by tapping or vibratory energy applied to the vessel). The vessels themselves are typically M/S or S/S pots of various volumes, typically 5 or 10 L. For finer crush sizes, the Bond rod and ball mill pots can be used.

Apparent SG, or in situ SG, determinations use the principle of individual particle pycnometry. This method calculates density based on the mass differential observed between weighing individual particles in air and suspended in water. It can measure density of quite large particles, such as drill-core specimens, and is only limited by the mass capability of the balance and the support screen aperture. Typically, 8–100 mm particles are readily processed. However, to provide accurate sampling statistics, large numbers of particles must be processed (100–2,000).

The equipment required is a balance with underpan weighing capability, underpan weighing cage, a suitable support frame, water tank, temperature measurement capability, and the ability to calculate the density of each particle in real time to allow separation into the various density bands. Wax coating can be utilized around porous or broken samples.

True SG determinations are most commonly conducted using a gas pycnometer, utilizing ideal gases such as helium. These machines determine the difference in gas pressure between a sealed reference chamber and a testing chamber, each of known volume. The reference chamber is first brought

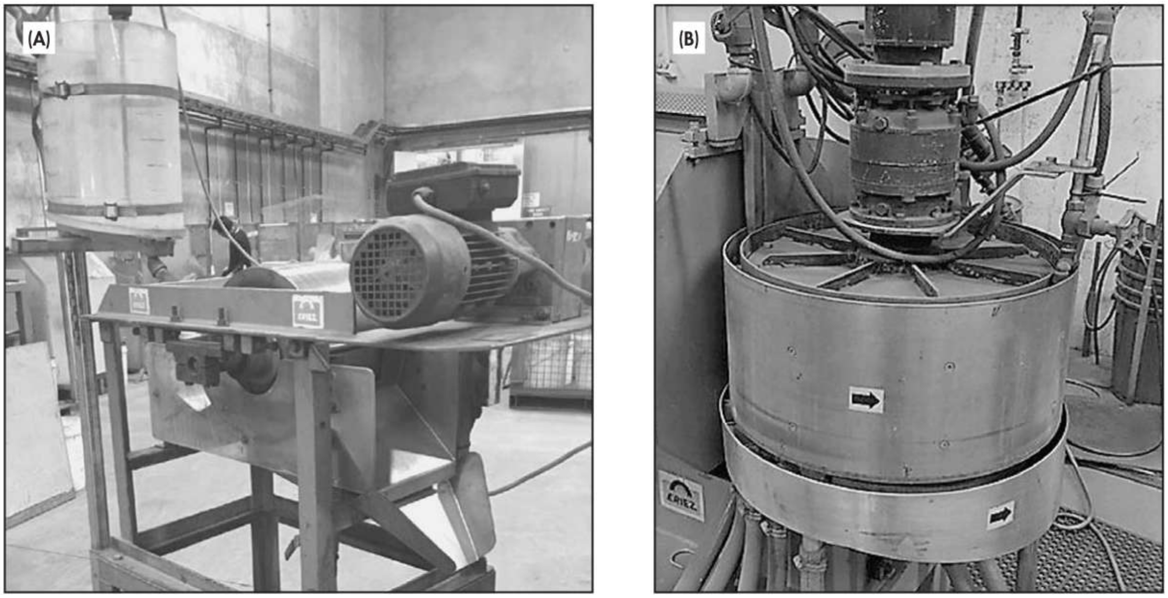


Figure 39 (A) Eriez L8 electromagnet and (B) Eriez WHIMS

Figure 40 SLon separator

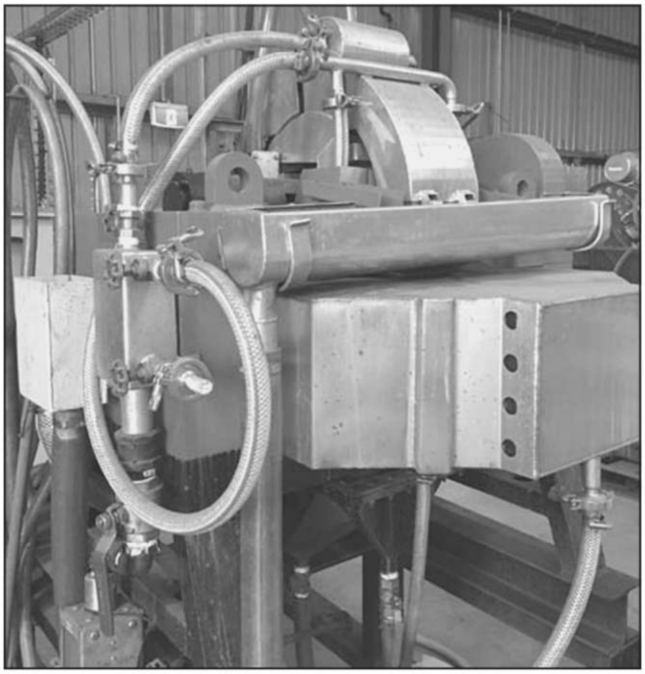


Figure 41 Longi separator

to a stabilized pressure, which is recorded. The gas is then released into the testing chamber containing a known mass of sample. The drop in gas pressure from the reference chamber is proportional to the volume of the sample in the testing chamber, and thus the true SG of the sample can be calculated using calibration standards. This density accounts for all voidage space in the sample, including surface pores accessible to the gas, but not internal or *locked* pores.

The machines usually come with different-sized chamber cups to cater for sample sizes ranging from 5 to 180 g, depending on the sample type and PSD. Samples charges are typically crushed and/or ground to a P_{80} of 75 µm to 2 mm. Well-known brands are the Quantachrome Ultrapyc 1000 and the Micromeritics AccuPyc 1330.

Heavy Liquid Separation

Heavy liquid separation (HLS) utilizes liquids of varying densities to separate ores or minerals based on the density of the minerals. A sample is mixed with a heavy liquid with a density intermediate between the mineral densities to be separated and allowed to separate by gravity. Any material with a density lower than that of the liquid floats, while material of higher density sinks. The two fractions are then recovered for further testing or analysis.

The two basic HLS techniques are static separation and centrifugal separation. Static separations are typically performed on coarse material with particle sizes in the range of 0.5 to 30 mm, while centrifugal separations are performed on samples with particle sizes <1 mm, with fines <38 µm usually removed from the sample. There is some overlap, and techniques may be adjusted for specific sample programs.

Some equipment is common to both techniques, while centrifugal separation uses some additional specialized equipment. Essentially all of the equipment can be sourced and purchased through generic laboratory equipment suppliers such as Rowe Scientific, Cole-Parmer, John Morris, and Retche.

Typical equipment inventory includes the following:

- Various sized beakers up to 2 L
- Buchner vacuum filtration flasks and funnels
- Measuring cylinders
- Hydrometers in the range of liquid densities used, for example, 2.0–4.5 kg/dm^3
- Coarse sieves and nylon sieve mesh (38 µm or similar)
- Plastic funnels
- Laboratory centrifuge (Boeco C28A or similar, equipped to use 100 × 40-mm tubes)
- Drying ovens
- Balances (4 kg with increments of two decimal places or better)
- Rotary evaporators (Buchi Rotavapor R-220 or similar)

Although it is possible to separate a sample over the full 0.5–30-mm size range, typically samples are sized into several narrower ranges. This improves the separation efficiency and enables an assessment as to whether mineral liberation is affected by particle size.

Sample mass is limited by the volume of the separation vessels; average sample density; the combined mass of the sample and heavy liquid required to separate it; and the ability to manipulate the combined mass of container, sample, and liquid. A 1.5-kg sample typically requires 1–1.5 L of solution to affect a separation, so assuming a density of 3.5 kg/dm^3, the combined mass may be in excess of 7 kg.

Typically, a sample is placed into a beaker and the appropriate heavy liquid added. The float layer is removed by scooping off the surface with an appropriate sieve or by decanting the float onto a sieve. Residual heavy liquid is then decanted off for recycling. If separation is required at more than one density, then the next-higher-density solution is added to the sinks, and the process repeated. Where it is necessary to change liquids to achieve the required density, the sample needs to be thoroughly washed and dried prior to the next separation.

For samples with particle sizes <1 mm, centrifugal separation is usually used. Sample mass is limited by the capacity of the centrifuge tubes to approximately 200 g. Particle size is limited by the vacuum equipment used to recover floats to ~1.5 mm on the coarse size and by the screen used to collect the samples to 38 µm. Finer sizes can be recovered by filtration with a consequent reduction in throughput. Separations on iron ores sized to 5 µm have been successfully completed and on unsized pulverized gold ores. Where it is necessary to separate larger masses than either process can accommodate, multiple batches of material can be processed and the products combined.

Table 1 Commonly used heavy liquids

Name	Density, kg·dm^{-3}
Lithium polytungstate (LST)	2.920
1,1,2,2-Tetrabromoethane (Muthmann solution [TBE])	2.967
Sodium polytungstate	3.100
Diiodomethane (methylene iodide [MI])	3.325
Thallium formate + thallium malonate (Clerici solution)	4.250

Heavy Liquids

The range of available materials has reduced considerably from previous decades because of the cost and toxicity of heavy liquids. Principally, these liquids are high-density organic compounds or aqueous salt solutions. Commonly used liquids are shown in Table 1.

With the exception of the polytungstates, all of the heavy liquids in Table 1 are considered hazardous and/or highly toxic. Clerici solution has a very high potential for causing harm at low exposures and requires special precautions during manufacture, handling, and use. In most jurisdictions, these poisons are available only to specialized or permitted users under strict regulations.

Solution Recovery

Heavy liquids currently vary in price from ~US$100/L (Muthmann solution) to US$5,600/L (Clerici solution), and together with extreme toxicity, HLS laboratories take steps to recover as much of the liquid as possible after test work has been completed. This reduces cost, minimizes risk or harm to end users of the samples, ensures accurate analysis of the products, and minimizes potential environmental harm.

For organic liquids, the samples are repeatedly washed with the solvent used to dilute the reagent, typically acetone because it is both inexpensive and readily available. All washings are recovered and filtered prior to reprocessing. For inorganic liquids, the samples are repeatedly washed with hot demineralized water. All washings are recovered, the water is removed by evaporation or distillation, and the distilled water is discarded. Discarded liquids must meet local requirements.

DEWATERING

Most slurry dewatering of bench-scale metallurgical test-work products is conducted using either vacuum or pressure filters.

Vacuum and Pressure Filters

Small, readily filterable samples of low percent solids and/or small masses (approximately <2 L and <300 g) can be dewatered quickly and efficiently using the standard Buchner funnel and vacuum flask. The flask is connected to the vacuum hose, and the funnel is pressed into the neck with a rubber stopper seal. A sheet of filter paper is inserted into the funnel and wetted, the vacuum applied, and the slurry sample poured into the funnel. Water and solution are sucked through the filter paper and captured in the flask. Once all solution has passed through, the vacuum is closed, and the filter paper with wet filter cake is removed, usually for oven drying.

Funnels come in various sizes and diameters, usually 100, 200, and 300 mm, and are constructed of either plastic or ceramic. Buchner funnels are typically off-the-shelf items that can be purchased through the main laboratory supplier chains such as John Morris, Cole-Parmer, and Rowe Scientific.

The next size apparatus is the benchtop barrel vacuum filter. These units also employ a standard vacuum flask to capture solution and consist of a base plate and removable barrel. Barrels are usually S/S. Operation is similar to the Buchner funnel with a filter cloth and paper placed on the base plate and held in place by the barrel. The barrel can then be lifted off the filtered sample and the paper and filter cake removed for oven drying or further testing. These benchtop pressure filters are also freely available in laboratory catalogs and also supplied by Marc Technologies and others. Both the Buchner funnel and benchtop barrel vacuum filter are shown in Figure 42.

Pressure filtering is required for larger test-work masses and volumes, and/or less filterable material types. The almost universal filter type for laboratory pressure filtering is the barrel filter, which comes as a benchtop model that can filter up to 6 L (Figure 43) and also as a floor-mounted model (Figures 44 and 45). The benchtop model is similar to the vacuum version except with a sealed removable lid and clamp frame. Compressed air is applied through a regulator valve and gauge through the top lid.

The floor-mounted pressure filter comes in either single free standing or skid mounted in sets of four to six. The central barrel is fixed with a filter cloth and filter paper placed on the base, which is then screwed into place in the frame. The sample is poured into the barrel, and the lid is then also screwed into place to create a seal. Often, a piece of solid bar is used as a lever to achieve firm pressure on the rubber ring seals in the base plate and lid.

The standard 200-mm-diameter × 400-mm-height barrel can effectively filter 10-L slurry volumes and masses between 100 g and 5 kg, depending on the material PSD and filterability. It is recommended to limit filter cake sizes to <3 kg. Manufactures of barrel pressure filters include FLSmidth-Essa, Marc Technologies, Sipor, and many local manufacturers that have generic designs. These units are classed as pressure vessels and need design certification to be compliant with the relevant government standards, together with regular structural integrity testing.

Two critical operating considerations of the barrel pressure filter are (1) thorough cleaning of the barrel around the base plate seal between samples and (2) seal maintenance. Slurry blowouts can occur under pressure as a result of dirty and/or worn seals, which result in loss of sample mass; poor cleanliness can result in cross-sample contamination.

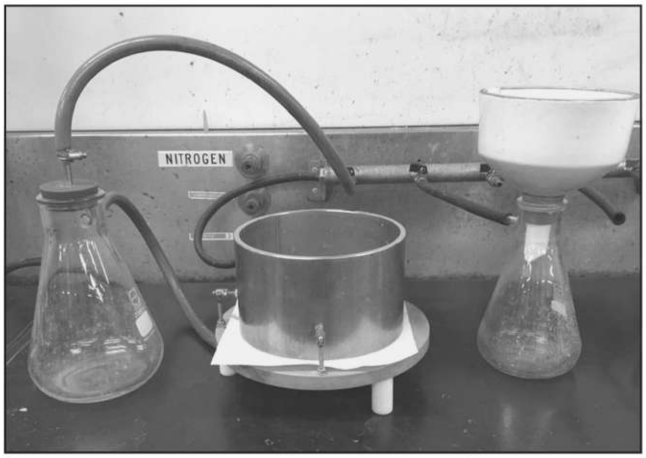


Figure 42 Barrel vacuum filter and Buchner funnel

Figure 43 Pressure barrel filter

Bench-Scale Settling, Thickening, and Filtration Test Work

Flocculant selection test work is undertaken on slurry samples with the aim of selecting one or two optimum flocculent types that ideally result in rapid solids settling and high supernatant solution clarity. The test requires 1 kg to evaluate a large array of different flocculant types and dosages. The test is performed in 100-mL measuring cylinders, where the lowest dosage of flocculant (i.e., 10 g/t) is added to the slurry at the conditions to be evaluated, mixed, and the time taken for the solids to settle past free settling recorded. Additional flocculant is then added and the test repeated sequentially up to the highest flocculant dosage (e.g., 100 g/t). A typical flocculant screening testing setup is shown in Figure 46.

The next level of bench-scale testing is a settling rate test based on the Talmage and Fitch method. These tests typically require ~1 kg of solids in slurry form, which can potentially evaluate as many as ten tests, limited only by the percent solids. The test is performed at a selected solids grind size and slurry percent solids in a 1,000-mL measuring cylinder. The required dosage of flocculant is added to the cylinder, mixed, and placed on the bench to allow the solids to begin settling. Using the graduated marks on the side of the cylinder, at specified time intervals, the volume of settling solids is recorded over a period of 24 hours. Notes on the supernatant clarity are recorded during the test. The data of time versus settled volume is then used to determine free and hindered settling rates as well as the calculation of the required conventional thickener area.

Due to the engineering guarantees required for modern construction contracts, settling test work for equipment design has moved in recent decades from the commercial laboratory to the equipment vendors. These vendors, including Outotec, FLSmidth, Delkor, Diemme, and Ishigaki, perform flocculation, thickening, and filtration test work in-house and sign off on performance guarantees on their own test work. This is especially the case for filtration test work, which requires

Figure 44 Floor-mounted barrel filter

complex apparatus and analysis to provide accurate information for equipment sizing.

Samples should be provided to the vendor laboratories at the appropriate grind size and under conditions where testing is required (i.e., leach feed or tailings, flotation concentrate or tailings, etc.). For both thickening and filtration test work, 15 kg of solids should be allowed for testing; however, where solids are not easily generated, that is, low mass pull flotation concentrates, a minimum of 6 kg should be allowed for evaluation. The sample should be provided to the vendor laboratory as a slurry (not filtered or dried prior to testing), and no flocculant/coagulant should be added.

VISCOSITY TESTING

Slurries generated from mineral processing operations and metallurgical test-work equivalents are invariably non-Newtonian fluids. Factors affecting the viscosity of a non-Newtonian fluid include percent solids, particle shape and size, temperature, and reagent additions. As all of these factors are target parameters in metallurgical test work, characterization of slurry viscosity is usually included at some point in most programs, from freshly milled feed to intermediate concentrates to final leach tails and so forth. Viscosity data assist design engineers in identifying potential rheology issues for a wide spectrum of shear rates for typical unit processes such as pumping, classification, agitation, and screening.

There are many different types of viscometers; however, rotational (cup and bob) viscometers are the most common in metallurgical laboratories and are ideally suited for efficient bench-scale testing of pre-prepared slurries taken directly from a range of test types. This category of viscometers is divided into two types. The Searle type has a rotating bob in a static cup of slurry, and the Couette type has the cup rotating the slurry around the stationary bob. Both types create a defined shear rate in the slurry and measure resistance to flow by the torque generated on the bob.

Figure 45 Barrel filter station

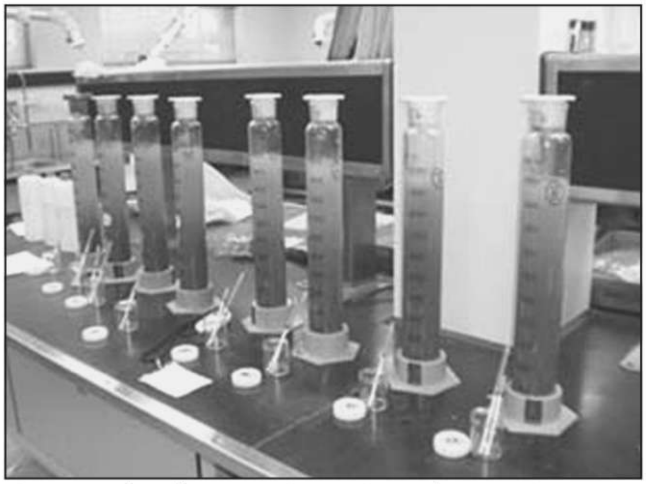


Figure 46 Flocculant screening station with measuring cylinders

The Searle type is by far the more common, given the design can be more flexible and temperature can be better controlled. Trusted brands that cover all laboratory viscometer needs are Malvern Panalytical Bohlin Visco 88, Brookfield Ametek digital viscometer series, and ThermoFisher Haake Viscotester series. The Bohlin Visco 88 and Brookfield DV1 viscometers are shown in Figures 47 and 48. Currently, most modern viscometers have color digital screens and electronic outputs (not shown in the figures).

Viscosity testing determines the results for viscosity (measured in centipoise or pascal-seconds) and shear stress (measured in pascals) across shear rates ranging from 4.2 to 209.9 s^{-1}.

The standard sample requirement for bench-scale slurry viscosity test work is 1 kg of ore ground to the required PSD and subjected to the relevant test conditions, that is, after cyanidation leaching, flotation testing, thickening, and so forth. It is important to evaluate the slurry as soon as practical to negate the effects of aging (i.e., shear thinning or thickening) and settling in the cup. A 1-kg sample mass is sufficient to evaluate many different conditions and percent solids, testing at an initial high slurry percent solids then diluting to a lower percent solids and testing again, or pH adjustment from lowest to highest adding lime at each testing point.

Some rules of thumb for the interpretation of the test results are as follows: slurry viscosity <100 cps at shear rates of 119.4 s^{-1} is considered acceptable for high-shear-rate environments such as pumping applications; and slurry viscosity <3,500 cps at shear rates of 4.2 s^{-1} is considered acceptable for low- and medium-shear-rate environments such as mixing and screening applications.

During pilot-plant-scale and full-scale operations, the use of a Marsh funnel (Fann Instrument Company 2013) can provide a simple measurement of slurry rheology by observing the time it takes a known volume of slurry to flow from a cone through a short tube. The Marsh funnel should not be used as a substitute for the aforementioned viscometers, as it only provides measurement under one flow condition. The sample requirements for the test are generally ~950 mL of the slurry under the conditions to be evaluated.

LEACHING

Leaching of various descriptions (e.g., water, acid, caustic, ammoniacal, and cyanidation) often constitutes a large component of metallurgical bench-scale test-work programs. Leaching test work is predominately by batch, using a range of different vessels and equipment. Test parameters, especially chemical resistance and temperature, determine the vessel materials of construction, and also whether the vessel operates at atmospheric pressure (i.e., <100°C) or as a pressurized vessel.

The various types of atmospheric leaching and pressure leaching vessels are described in the following sections.

Atmospheric Leaching Using Bottle Rolls and Agitated Vessels

The most expedient and cheapest method of atmospheric leaching is the bottle roll. The *bottles* themselves are, in reality, cheap plastic screw-top jars and barrels of various sizes (500 mL and 1, 2, 4, 6, 8, and 20 L). Glass and ceramic vessels can be used but are rare for minerals testing.

Typically conducted at ambient temperature, milled slurry at the target P_{80} is transferred to the bottle and made up to the desired percent solids before chemical conditioning (i.e., reagent addition and air-gas sparging). The lid is closed, and the prepared bottle is placed on a parallel pair of rollers (one drive roller and one neutral) for a nominated period whereby the rolling action provides the required mixing/agitation of the conditioned slurry to facilitate leaching.

The bottles can stay on the rollers for the entire test duration or, alternatively, be periodically taken off at nominated times for reagent monitoring-maintenance and subsampling for analysis to provide kinetic data. The roller apparatus is usually variable speed and often has an intermittent start–stop capability. Typical speeds for leaching of 2–4.5 L bottles are 30–40 rpm. Suppliers of bottle roller units suitable for metallurgical testing are available from FLSmidth-Essa, Marc Technologies, and Wheaton.

Small-scale agitated tanks, or vats, provide the next level of laboratory leaching capability at ambient or mild temperatures. The vessels are typically custom fabricated and constructed of clear Perspex in various volumes and design geometries, complete with side baffles, and represent miniature versions of industrial-sized leach tanks. S/S is also a viable construction material, as is high-density polyethylene (HDPE), but these are less common. Sample masses are commensurate

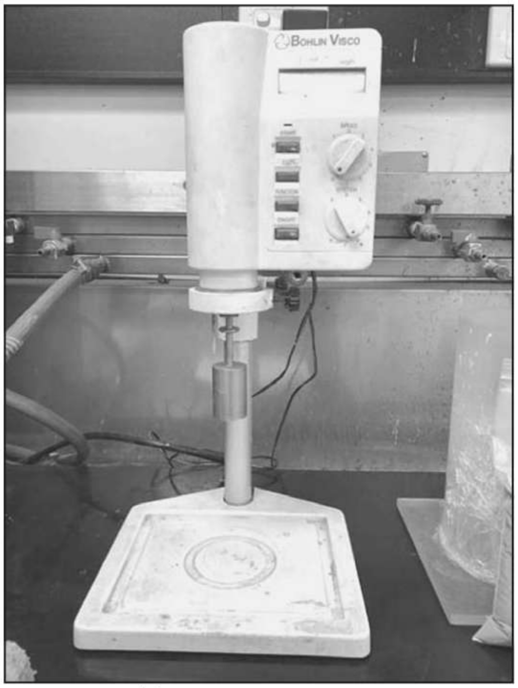


Figure 47 Bohlin viscometer

Figure 48 Brookfield viscometer

with the vessel size but typically range between 500 g and 20 kg on the bench. Agitation is provided by an overhead stirrer. The most common laboratory overhead stirrer is the IKA brand, which comes in a range of various powered units. All units are variable speed and some come with increasingly sophisticated digital displays and data outputs. Agitator speed is dependent on the agitator type, slurry PSD, and vessel volume. Typical agitator speeds for metallurgical leaching applications are around 200–300 rpm, sufficient to keep particles in suspension without excessive turbulence. The units allow for interchangeable shafts and impellor designs (i.e., flat blade, Rushton radial flow, axial flow, turbine, propeller, etc.).

Unlike the bottle roll, once the milled slurry has been placed in the vat, slurry conditioning, air-gas sparging, monitoring, and subsampling can take place continually or at any desired interval throughout the test. This is especially advantageous for testing the kinetic response of various test parameters. Heat input can also be facilitated with immersion heaters or external water baths, usually up to 40°C with Perspex tanks, and up to 70°C with S/S tanks or HDPE tanks.

Air or gas addition is supplied via a pressurized ring main with multiple outlets along the bench at each leaching station. Each outlet has an isolation valve followed by a regulated gas rotameter for flow-rate metering. A typical bottle roll testing apparatus is pictured in Figure 49, and Figure 50 shows a typical vat leaching setup.

Higher-temperature leaching applications, especially for acidic or corrosive conditions, are usually conducted in standard laboratory Pyrex glassware. These vessels are fabricated in a multitude of design shapes and volumes, with compatible ported lids, and can be sourced from any of the major laboratory equipment suppliers such as Cole-Parmer, John Morris, and Rowe Scientific. The upper volume sizes for standard glassware are usually limited to 5 L, and as such, sample masses for these setups are also limited to <2 kg. The overall configuration is similar to the preceding vat leaching apparatus, with an overhead stirrer providing agitation. Heat input, however, can be applied with a base hotplate up to 95°C, and outer insulation can be applied. The ported lids can also facilitate other additions such as condensers and scrubbers together with probe/pipette inlets. Figure 51 shows a typical Pyrex high-temperature leaching setup.

Column Leaching

Column leaching is the standard metallurgical testing method to assess heap leaching applications of coarse crushed PSD material, by either gold cyanidation or acid leaching. More detailed information can be found in Chapter 10.3, "Heap and Dump Leaching."

Sample crushed particle sizes range from 6 mm to 75 mm, or greater if investigating dump leach, and usually in 6.3-, 12.7-, 19-, 25-, 51-mm measurements, and so forth. Column heights are typically from 0.5 to 6 m. Preliminary test work is usually undertaken at 2-m height minimum, with confirmatory testing increasing in height to 4–6 m using customized rigs. Large-scale demonstration testing can be conducted in columns as large as 2-m diameter (or square cribs), requiring many metric tons.

Column diameter selection is based on an empirical factor of 6× the maximum particle diameter (i.e., a 50-mm crush size are performed in 300-mm-diameter column). Sample mass requirements are commensurate with the column height and diameter, PSD, and bulk density. Sizes and estimated masses for 2-m laboratory columns are shown in Table 2.

Columns are usually custom designed and fabricated from clear Perspex. Columns higher than 2 m are constructed in flanged 1-m sections to enable easier takedown and material removal at test termination.

Solution irrigation is facilitated with small positive-displacement peristaltic pumps (such as MasterFlex) that can take different-sized heads and tubing. The initial solution reservoir is typically a 20-L plastic bucket, which can be switched out during the test and also monitored and adjusted for chemical concentrations. Irrigation rates typically range from 8 to 20 L/m^2/h.

Cyanide solution strengths for gold leaching applications range from 250 to 1,000 ppm cyanide, but most often <500 ppm. Acid solution strengths for copper and uranium leaching applications range from 5 to 10 g/L free acid. Nickel columns usually require higher strengths at ~50 g/L. Cyanidation column leaching test durations range between approximately 30 and 70 days. Acid leaching column testing durations are much longer, for example, between 6 and 9 months.

Prior to actual leach testing, samples of the material are tested in columns for permeability, ponding, or ratholing. This is done by sealing the bottom of the column, flooding it

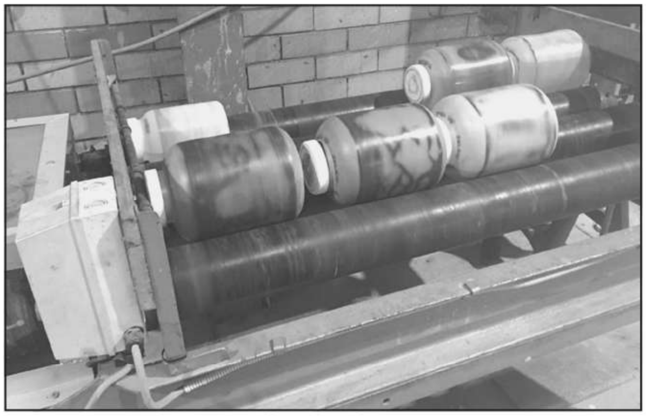


Figure 49 Leach using rolling bottle

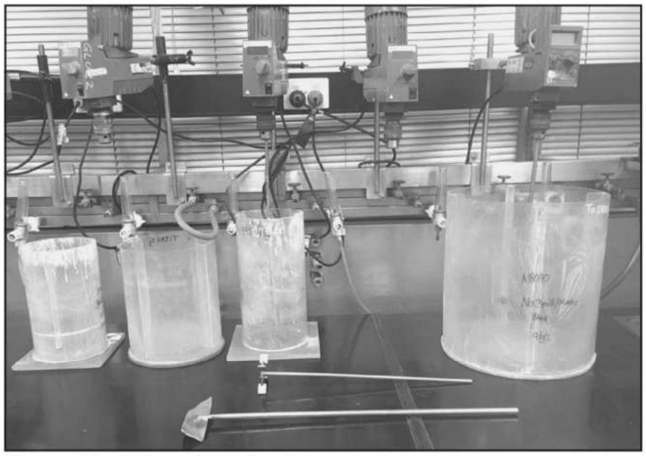


Figure 50 Agitated vat leaching equipment

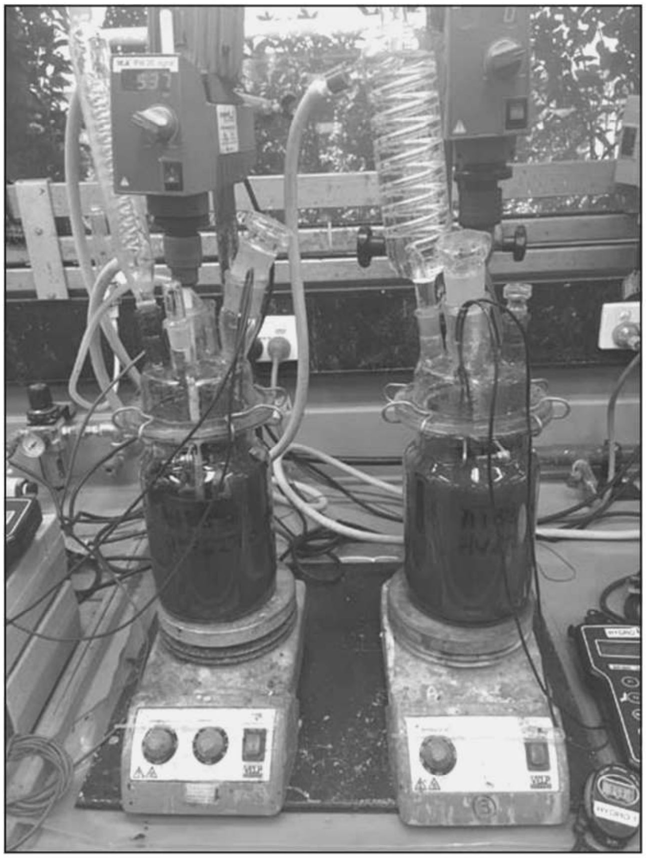


Figure 51 High-temperature leaching setup

Table 2 Laboratory columns

Diameter, mm	Height, m	Mass, kg
80	2	15
100	2	~24
150	2	~50
200	2	~95
300	2	~210
500	2	~580
1,000	2	~2,300

with water, and then examining the flow of water through the material and time of complete drainage to ensure that the total column contents are contacted by the water passage. If any concerns arise from this test, agglomeration of the material may be required.

Pressure Leaching Using Autoclaves

Higher-temperature laboratory leaching applications >100°C, such a high-pressure acid leaching (HPAL) or pressure oxidation (POx), require a pressure vessel or autoclave. Safety and regulatory requirements rise exponentially when dealing with high temperatures, high pressures, and highly corrosive solutions comparative to other bench-scale test work. These vessels require extremely corrosion-resistant materials of construction, such as titanium or tantalum, and precision fabrication to pass certification and, as such, are typically very expensive items to procure. In turn, this means the associated testing is also more expensive than atmospheric leaching; however, in many cases, there is no alternative to achieve acceptable extractions.

The most well-known brand in laboratory autoclaves is provided by Parr Instruments (United States), parts for which can be sourced via the John Morris Group. There are other alternatives; however, considering the safety risks, laboratory metallurgists need to be extremely confident of the construction schedule and certification. A single out-of-specification nut or bolt can result in rapid catastrophic failure under aggressive HPAL testing conditions.

Parr produces two sizes of autoclave (in U.S. gallons) at 3.8 and 19 L. The base vessel is situated in an insulated wraparound heater, and an immersion heater is also fitted to the lid. An internal agitator is connected via a magnetic coupling to an overhead drive mechanism. The lid has multiple ports to facilitate gas injection, nitrogen overpressure, temperature and pressure probes, and sampling bombs.

The 3.8-L autoclave takes sample masses of ~700 g, and the 19-L version can receive up to 3.5 kg. HPAL testing conditions operate at 220–250°C at steam pressures of 2,200–2,800 kPa with nitrogen overpressure up to 4,500 kPa. Maximum acid additions for the smaller unit are ~350 kg/t of sulfuric acid. Test durations range from 90 minutes to 4 hours. POx testing conditions operate at 105–220°C at oxygen overpressures ranging between 200 and 22,000 kPa. Test durations range from 5 minutes for partial oxidation to 3 hours.

The autoclaves also require a temperature and pressure control box, which can be sourced directly from Parr or from John Morris. A typical 3.8-L Parr autoclave skid arrangement is shown in Figure 52.

Leaching Ancillary Equipment

The following is a list of critical ancillary laboratory equipment and infrastructure associated with leaching:

- Grinding and sizing equipment as described in the preceding sections
- Pressure filters, filter cloths, and filter papers
- Drying ovens
- Air compressor and ring main plumbing
- Gas bottle storage and ring main plumbing
- Weigh scales and balances to handle small high-accuracy masses (100 g), midrange masses between 2 and 5 kg, and larger masses up to 30 kg
- ORP and pH meters and probes
- Dissolved oxygen meter and probe
- Burette and Pyrex beakers for titrations
- Pipettes, bulbs, test tubes, and racks
- Equipment for solution subsampling and assay analysis
  - A range of 50–100-mL plastic screw-top phials for solution and slurry subsamples
  - Aluminum trays and plastic buckets for sample collection
  - Aluminum tags, printable stickers, and a range of plastic bag sizes for sample storage

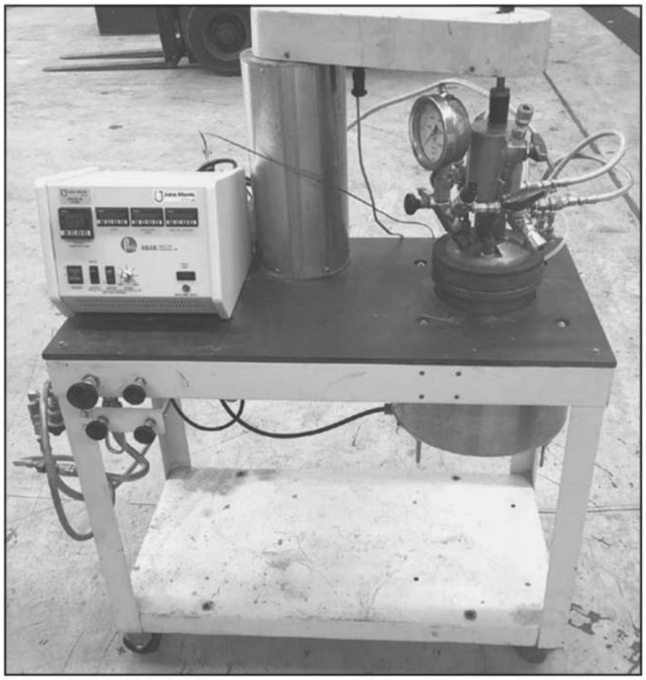


Figure 52 Parr autoclave

ACKNOWLEDGMENTS

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