

**SECTION C.2**

***VARIABLES AND UNCERTAINTIES IN THE  
GEOCHEMICAL CHARACTERIZATION OF MINERAL  
SAMPLES BY KINETIC METHODS***

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## **VARIABLES AND UNCERTAINTIES IN THE GEOCHEMICAL CHARACTERIZATION OF MINERAL SAMPLES BY KINETIC METHODS**

By

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### **Introduction**

In the geochemical characterization of mineral samples by kinetic methods there are many controllable and uncontrollable variables. Uncontrollable variables that may lead to inappropriate interpretation of results include:

1. Differences in particle size distribution between samples that generate differences in sample specific surface area.
2. Degree of potentially acid generating and neutralizing mineral liberation.
3. Humidity Cell and Column Testwork Equipment Arrangement.
4. Humidity Cell Procedure.
5. The effect of microorganisms.
6. Testwork reproducibility.

### **Sample Size Distribution and Specific Surface Area**

In kinetic ARD testwork, as in field behaviour, particle size, particle size distribution and individual mineral grain size are parameters that affect both acid generation and acid neutralization. For pyrite and pyrrhotite grains to oxidize, there must be exposure of these grains to the atmosphere. For neutralizing mineral grains to react with produced acid, such grains must be exposed to the acid. The degree to which individual grains are exposed depends to a great extent upon the size of the individual grains relative to the size of the rock sample in which they are contained.

Unlike Acid Base Accounting procedures, where samples are finely ground (100% -75  $\mu\text{m}$ , -45  $\mu\text{m}$  or -38  $\mu\text{m}$ ), samples for kinetic testwork may have significant size ranges. For example, for the ASTM humidity cell procedure (ASTM, 1996) the sample should be 100% passing 6.3 mm (1/4 inch), while column tests are commonly run on material less than about 25 mm and lysimeters may be operated with run-of-mine material that may be as coarse as 500 mm or greater. Although a specified mass may be used for a kinetic test (e.g. 1 kg for the ASTM

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humidity cell procedure (ASTM, 1996)), the sample specific surface area (usually expressed in square metres/kilogramme) depends upon the sample particle size distribution and the mineralogical characteristics of the sample. Because of these dependencies, two concepts from the mineral processing field must be introduced into ARD prediction:

1. Particle size distribution
2. Mineral liberation size (Liberation Size Determination and Surface Sulphur Determination Methods)

### Particle Size Distribution

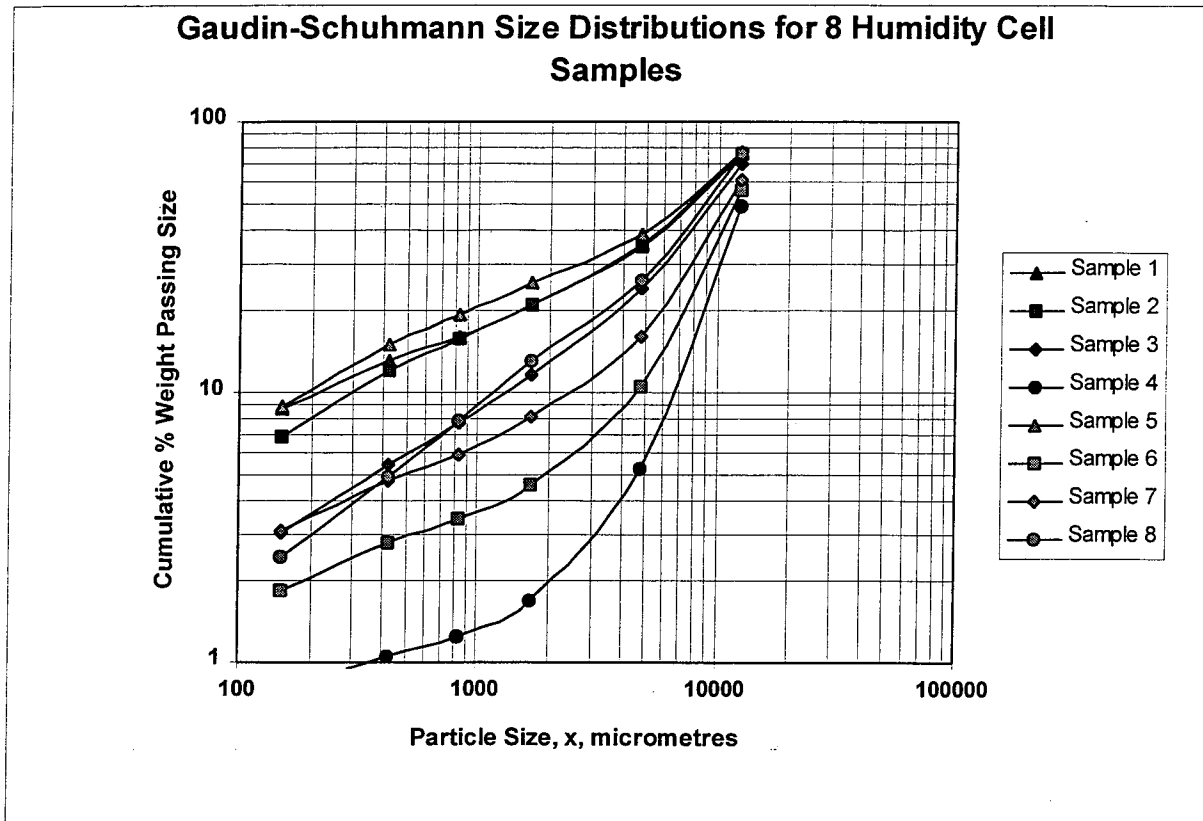
When rocks are broken (by blasting, crushing or grinding), particles are produced with a continuous size distribution from the coarsest particle in the product to the finest (which is theoretically a particle of zero size). The continuity of this particle size distribution may be broken when the fragmented material is subjected an imposed process such as screening or hydraulic classification - in which case any products of the process will have their own unique size distribution that will differ from the that of the original material.

Many methods, of varying complexity, have been developed to quantify the size distribution of particulates (Lynch, 1977, Bond, 1985, Lynch and Lees, 1985). One relatively uncomplicated method that has found favour in the North American metalliferous mining industry since 1940 is the Gates-Gaudin-Schuhmann equation, defined by:

$$y = [x/k]^m$$

where  $y$  is the fraction of the sample finer than size  $x$ , and  $k$  (*size modulus*) and  $m$  (*distribution modulus*) are constants for the particular distribution. If the logarithm of the cumulative % weight passing size  $x$  is plotted against the logarithm of particle size  $x$ , a relatively straight line is often obtained, with a slope equal to  $m$ , the *distribution modulus*, and an intercept at 100% cumulative weight passing of  $k$ , the *size modulus*. The size distribution is therefore characterized by two simple parameters,  $k$  and  $m$ . For any given group of size distributions, the higher the value of  $k$ , the coarser the size distribution, and the higher the value of  $m$ , the narrower the size distribution (and vice-versa). For a fixed mass of sample and value of  $k$ , specific surface area increases as the width of the size distribution increases - i.e. as  $m$  decreases.

The following Figure 1 shows Gaudin-Schuhmann size distributions for 8 samples of crushed drill-core from the same deposit prepared for humidity cell testwork by the same method. All of the curves appear to converge at a 100% cumulative % weight passing size of about 16 mm or 5/8 inch, thus the value of  $k$  for all samples is 16 mm or 16,000  $\mu\text{m}$ . The slopes, or gradients, of the curves range from approximately 0.5 for Sample 5 to approximately 1.1 for Sample 4, although the curves for Samples 4, 6, 7 and cannot be described as a straight line. The 8 samples thus have a range of values for  $m$ , from 0.5 to 1.1 and Sample 5 has the widest, and Sample 4 the narrowest, size distributions. Sample 4 would be expected to have the least specific surface area, and Sample 5, the greatest.



**Figure 1: Gaudin-Schuhmann Size Distributions for 8 Humidity Cell Samples**

A characteristic that is of considerably more use than the simple particle size distributions in the interpretation of ARD testwork is an estimate of sample total or specific surface area. For the above 8 samples, these estimates have been made, assuming all particles to be spherical with a maximum particle size ( $k$ ) of 16 mm and a minimum particle size of 2  $\mu\text{m}$  (chosen as being the likely finest sized particle retained in an operational humidity cell, and introducing a less than 1% error into calculations). Since testing screens are manufactured with apertures in series based on the fourth root of two, the geometric mean of the upper and lower screen size for each fraction should be used as a mean fraction particle size, rather than an arithmetic mean. The following Table 1 shows the calculated specific surface area (square metre per kilogramme) and the percentage of the total sample surface area contributed by the finest (-150+2  $\mu\text{m}$ ) fraction of the sample for the 8 samples.

**Table 1: Calculated Specific Surface and Percentage of Total Surface Area Contributed by -150+2 $\mu$ m Fractions for Data in Figure 1**

Sample Number	Calculated Specific Surface, square meter/kilogramme	Percentage of Total Surface Area Contributed by Minus 150 Plus 2 micrometre Fraction
1	12.0	93.2
2	9.77	90.8
3	4.51	86.4
4	1.28	79.1
5	12.5	92.0
6	2.75	86.9
7	4.42	89.6
8	3.86	82.7

The calculated specific surface area of the samples ranges from 1.28 (Sample 4) to 12.5 (Sample 5) square metre/kilogramme, which represents a range of almost an order of magnitude. In general the specific surface area values correlate well with the size distribution curves, in that the wider the size distribution, the greater the specific surface area.

**A difference of an order of magnitude between the specific surface area of humidity cell samples, from the same location, and prepared in a similar manner is significant. To be of any validity, comparisons between the results of humidity cell testwork on these eight samples would need to be on a normalized surface area basis, and not on a mass basis.**

The variation between the percentage of surface area contributed by the -150+2  $\mu$ m fractions is from 79.1% (Sample 4) to 93.2 (Sample 1), and this is also significant, representing an absolute difference of 14.1%.

### **Mineral Liberation Size**

The textural relationships between minerals within an ore, and their relation to process selection requires the introduction of the concept of liberation size. This is the size to which an ore must be crushed or ground to produce separate particles of either value mineral or gangue that can be removed from the ore (as concentrate or tailings) with an acceptable efficiency by a commercial unit process. Liberation size does not imply pure mineral species, but rather an economic trade-off of grade and recovery. The concept of liberation and liberation size may be extended to ARD prediction, where atmospheric leaching becomes the relevant unit process. Atmospheric pyrite and pyrrhotite oxidation requires that part of the mineral grain be exposed or liberated with respect to leaching. For any given pyritic or pyrrhotitic rock, the degree of pyrite or pyrrhotite mineral grain exposure increases with decreasing particle size, attaining 100% exposure or liberation with respect to leaching when all of the rock has been reduced in size to that of the smallest grain of pyrite or pyrrhotite.

**It is usually only at 100% exposure or liberation that acid generation potential determined by ABA (static) methods equals the acid generation potential of kinetic test or field samples.**

Thus grains pyrite or pyrrhotite in whole rock will have a lesser degree of exposure (less liberation) than those in blasted waste rock, and those in blasted waste rock will have a lesser degree of exposure (less liberation) than those in mill tailings. Size reduction corresponds to an increase in total particle surface area. Particle specific surface area is proportional to the reciprocal of particle size.

Liberation size can be estimated from petrographic studies, especially the microscopic examination of thin and polished sections.

Liberation size can also be determined by quantitative mineralogical testwork on size fractions of ground ore, although this is considerably less common because it is costly, time consuming, and often technically difficult. However for the determination of potential ARD generation problems with tailings, the methodology produces information that simple chemical analysis does not. Liberation size is a function of the relevant physical or chemical process and may differ greatly between processes (Mills, 1995). Since ARD generation is a sulphide (pyrite) leaching process and base metal concentration is usually by flotation from gangue sulphide (pyrite) these differences may have significance in ARD prediction. The liberation size of pyrite for both flotation and leaching (and gravity concentration if it is included in the mill flowsheet) is therefore a most important parameter. This information is extremely difficult to determine without quantitative mineralogical studies.

Size reduction for liberation naturally produces a size range of particles. This range is dependent upon the type of size reduction method used, and the hardness, texture, friability and degree of weathering of the rock and its constituent minerals.

For ARD prediction the important characteristics of ore or waste are:

1. The amount of pyrite and/or pyrrhotite in the ore or waste.
2. The degree of pyrite and/or pyrrhotite liberation produced by size reduction with respect to potential oxidation and leaching.
3. The presence of, and association with pyrite of, trace metal sulphides.
4. The amounts of leachable metallic sulphide minerals in the ore or waste
5. The degree of liberation of leachable metallic sulphide mineral produced by size reduction with respect to leaching.

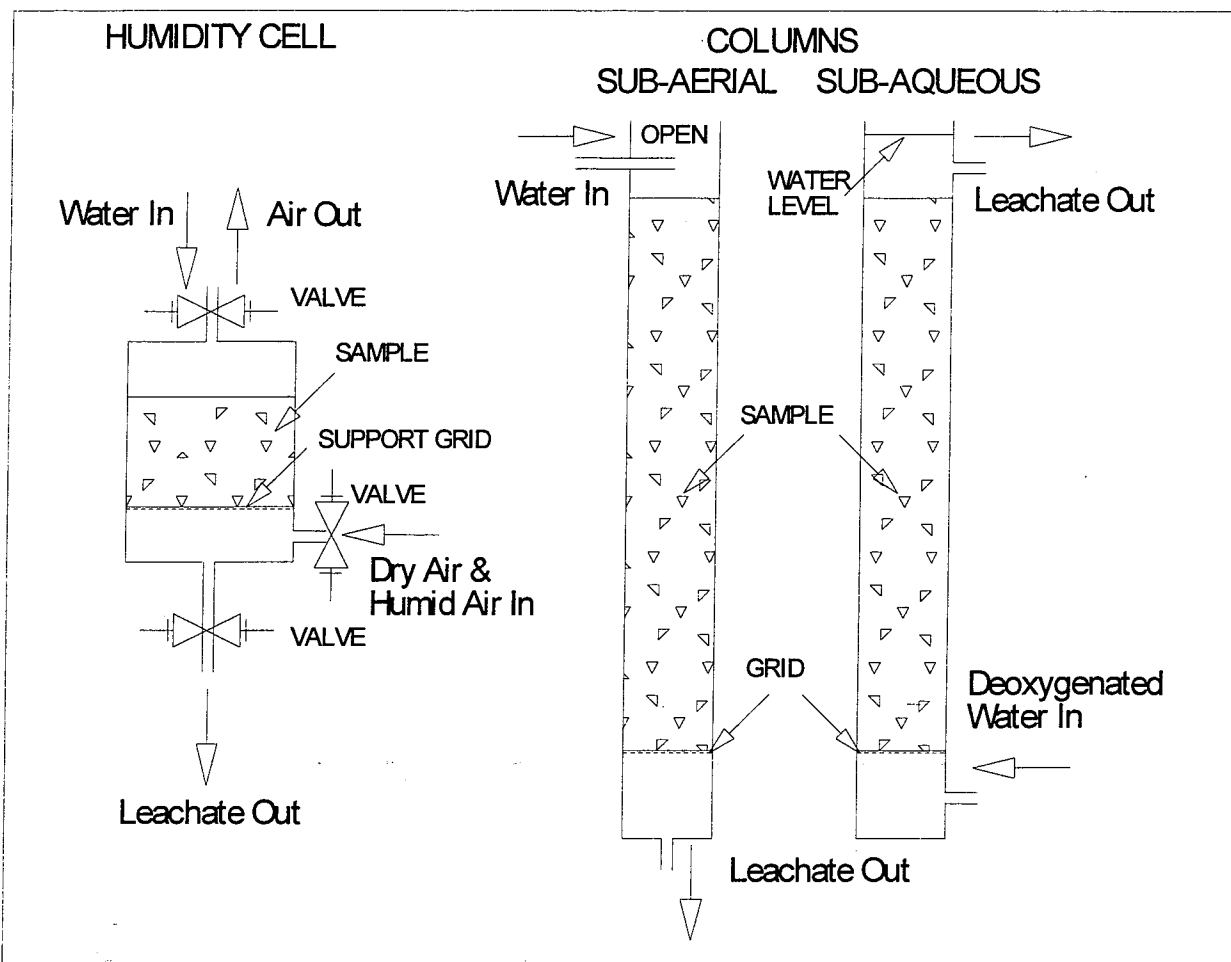
Most of the mineralogical testwork conducted in the field of ARD has focussed on the products of pyrite oxidation and the products of ARD neutralization within tailings impoundments. It is considerably easier to determine particle size distribution and liberation characteristics for

tailings than it is for waste rock dumps. For existing tailings, size distribution may be representatively determined from testing screen results, while liberation characteristics may usually be obtained using visible light microscopic techniques. For existing waste rock dumps the acquisition of representative size distributions is very difficult since particle size ranges from the coarsest material produced by blasting to the finest material retained after rain and snow infiltration and flushing. Liberation characteristics for waste dump material may be (and usually are) inferred from examination of the finer dump material. Where only drill core is available, liberation characteristics can be determined by microscopy techniques, but estimated size distributions require information on the proposed mining method and milling flowsheet. Metallurgical testwork is usually conducted concurrently with ARD testwork, so that tailings from testwork programs are normally available for ARD study.

### **Humidity Cell and Column Testwork Equipment Arrangement**

The following Figure 2 shows generic schematic diagrams for the arrangement of a humidity cell, a sub-aerial testwork column, and one possible configuration for a sub-aqueous testwork column. There are other possible configurations for sub-aqueous testwork (Lawrence, 1995). Humidity cells have, in the past, varied considerably in dimensions. However the laboratories servicing the mining industry have now adopted a limited degree of standardization. In *Designation: D5744-96 Standard Test Method for Accelerated Weathering of Solid Materials Using a Modified Humidity Cell* (ASTM, 1996), a cell 203 mm (8.0 inch) in height by 102 mm (4.0 inch) diameter is specified for material crushed to 100% passing 6.3 mm (crushed core or waste rock, and coarse tailings), and a cell 102 mm (4.0 inch) in height by 203 mm (8.0 inch) diameter is specified for material passing 150  $\mu$ m (fine tailings). Price (1997), while recommending similar dimensions, states that the dry and humid air flow should be directed across the surface of tailings samples, rather than through the samples from below. In both of the above arrangements the sample mass is 1 kg which typically gives a bed depth of about 80-120 mm in a 102 mm diameter cell and 20-40 mm in a 203 mm diameter cell, depending upon sample bulk density. Soregaroli and Lawrence (1998) have reported the use of a 3 kg sample with a 102 mm diameter cell and a 15 kg sample with a 254 mm diameter cell, and compared results with those obtained with a 1 kg sample and 102 mm diameter cell. Other configurations are in use, including those where the humidity cell dimensions and proportions more closely resemble those of a column than those of a "conventional" humidity cell.





**Figure 2: Schematic Arrangements of Humidity Cell and Sub-Aerial and Sub-Aqueous Columns**

For details and specifications of dry and humid air supply systems, materials of construction and other mechanical details see *Designation: D5744-96 Standard Test Method for Accelerated Weathering of Solid Materials Using a Modified Humidity Cell* (ASTM, 1996). The dry and humid air systems must be capable of delivering a controlled rate of 1 to 10 l/min to each humidity cell, and flow meters are required for flow rate measurement (ASTM, 1996). Columns for sub-aerial and sub-aqueous testwork are typically 76, 102 or 152 mm (3, 4 or 6 inch) in diameter, and from about 1 m to more than 3 m in height.

### Humidity Cell Procedure

The humidity cell operational procedure is a cyclic one, during which the sample is subjected to three days of dry air permeation, three days of humid (water saturated) air permeation, and one day of water washing (with a fixed volume of water). Caruccio (1995) has suggested that coal spoils from the Eastern United States should be subjected to six days of humid air, rather than

three days of dry and three days of humid air. In addition, some Canadian workers have recently used a three day dry, three day humid, three day dry, three day humid, one day leach cycle

The procedure is usually referred to as an *accelerated* weathering procedure (ASTM, 1996) because it is designed to *accelerate* the natural weathering rate of potentially acid-generating samples and reduce the length of time for which testwork must be run. The artificial nature of the procedure is a consequence of regulatory constraints, rather than natural ones.

Designation D 5744-96 (ASTM, 1996) states:

"1.1 This test method covers a procedure that accelerates the natural weathering rate of a solid material sample so that diagnostic weathering products can be produced, collected, and quantified. Soluble weathering products are mobilized by a fixed-volume aqueous leach that is performed, collected and analyzed weekly. When conducted in accordance with the following protocol, this laboratory test method has accelerated metal-mine waste-rock weathering rates by at least an order of magnitude greater than observed field rates.

1.6 This test method is not intended to provide leachates that are identical to the actual leachate produced from a solid material in the field or to produce leachates to be used as the sole basis of engineering design.

1.7 This method is not intended to simulate site-specific leaching conditions. It has not been demonstrated to simulate actual disposal site leaching conditions."

The ASTM Procedure (ASTM, 1996) requires a minimum test duration of 20 weeks, while Price (1997) recommends a minimum of 40 weeks. It is common in Western Canada for humidity cells to be run for periods in excess of two years (104 weeks). It is possible that some tropical areas of the world experience climates similar to that of a humidity cell, but for North American mines the atmospheric conditions of the humidity cell are considerably harsher than the natural ones to which waste rock and tailings may be exposed. This is because:

3. Testwork is usually conducted in a laboratory at room temperature, which is normally greater than the atmospheric temperature to which mining waste material is, or will be, exposed. Lower temperatures slow both chemical and biological reactions involved in acid generation.
4. Testwork ensures a rigorous dry air/moist air/water cycle to accelerate sulphide oxidation and to maximise oxidation product flushing. Most sites experience neither the regularity of the dry air/moist air cycle nor the regularity and intensity of wet precipitation corresponding the water cycle of the humidity cell.
5. The water flush cycle of the humidity cell is conducted to ensure as completely as possible the wetting and flushing of the entire sample. Precipitation influx through waste rock dumps, and to a lesser extent tailings, is non-uniform due to channelling, and complete wetting and flushing is not achieved. Thus the conditions of full oxygen and water supply to liberated sulphide minerals will not be achieved in practice.

6. Size reduction of waste rock or drill core, or the use of only fine fractions of waste rock, to meet the requirements of the humidity cell procedure produces sample material with a substantially greater specific surface area ( $\text{m}^2/\text{kg}$ ) than the real waste rock, and a substantially higher degree of both sulphide (acid generating) and neutralizing minerals liberation. Therefore, on a sample mass basis, a humidity cell test will potentially produce more acid (mass) and more soluble metals (mass) in a given time than the corresponding waste rock.

A humidity cell test will usually determine *if* a given sample will "go acid", but not *when* the material from which the sample was taken will "go acid" since the operation of the humidity cell has intentionally accelerated sulphide mineral oxidation. Similarly, the accelerated rate of oxidation and acid production will result in an accelerated rate of oxidation products generation as dissolved metals and/or precipitated metal compounds. That is, the metal concentrations in the weekly leachate (wash cycle) are likely to be higher than those generated in the field.

Prior to start-up of a humidity cell test, the sample material must be weighed, and characterized by ABA analysis, ICP multi-element analysis, whole rock major element analysis, screen sizing and mineralogical examination. The same weighing and characterization procedure must also be conducted on the humidity cell sample residue at the completion of the humidity cell test. It is particularly important that Acid Generation Potential (AP) and Neutralization Potential (NP) of the initial and final samples are determined using identical methods. It is equally important that the initial and final samples are adequately characterized mineralogically, because some samples undergo complex mineralogical changes during the extended period of a kinetic test. Without "before" and "after" mineralogical information it is sometimes difficult or impossible to evaluate kinetic test data.

Samples from material containing soluble sulphate minerals or previously oxidized material may require pre-treatment prior to humidity cell testwork in order to remove minerals or chemical compounds that may either mask acid generation or delay it.

The preparation of humidity cell test samples should avoid size reduction processes (such as crushing) if possible. Tailings samples can usually be tested "as is", while Price (1997) recommends the use of only the minus 2 mm fraction of existing waste rock dumps. Drill core must be crushed to provide samples suitable for humidity cell tests. Price (1997) recommends 80% passing 6 mm for future waste rock and ore, and 80% passing 150  $\mu\text{m}$  for future tailings. The reduction of drill core to 80% passing 150  $\mu\text{m}$  requires laboratory crushing and grinding that may produce size and mineral liberation characteristics very different from those of a real mill using several stages of crushing, screening, grinding, classification and other unit operations. It is therefore preferable that samples used for kinetic testwork on future tailings are those produced from metallurgical testwork.

A further particle size-related concern in humidity cell (and column) testwork is the presence of clay minerals (such as illites and smectites), with particle size less than 2  $\mu\text{m}$ , in the test sample. In the above arrangement diagrams, the sample is shown supported by a grid which, in practice, is covered with a filter medium to prevent fines loss during flushing. While this filter medium must be chemically inert and non-retentive to water, it must also be porous enough for easy flow

of leachate while retaining the test sample. In practice a compromise is made with the use of polypropylene felt, or equivalent, with a filament diameter of 22  $\mu\text{m}$  (ASTM, 1996). Such material is unlikely to retain particles of less than 2  $\mu\text{m}$ , and samples containing significant quantities of such particles will produce a leachate from flushing that will require further filtration (typically at 0.45  $\mu\text{m}$ ) before dissolved metals can be determined.

The operation of humidity cells is straightforward and can be significantly automated with the use of peristaltic pumps and timers. An initial flushing of the cell is conducted by adding 750 ml (for a 1 kg sample cell) de-mineralized water to the top of the cell so that the sample is thoroughly wetted. After a period of 2 hour (4 hour for tailings samples) the leachate is collected, weighed (for volume determination) and analysed for pH, conductivity, acidity, alkalinity, sulphate and dissolved metals. The measurement of leachate Redox potential, Eh, may also prove useful in test interpretation. For the following three days dry air is delivered to the cell at a predetermined rate, then for three more days humid air is delivered to the cell (again, at a predetermined rate). The air flow rates used may be a generic standard, or they may be determined as site-specific parameters. On the seventh day the sample is flushed with 500 ml (for a 1 kg sample cell) de-mineralized water, allowed to stand for 2 hour (4 hour for tailings samples), and the leachate is collected, weighed and analysed for the parameters noted above. The seven day cycle is continued until the test is terminated, whereupon the sample residue is removed from the cell, dried, weighed and subjected to ABA analysis, ICP metals analysis, major component whole rock analysis and mineralogical examination. Price (1997) and ASTM (1996) describe the procedure in detail.

Weekly (cycle) leachate analyses ( $\mu\text{g/l}$  or  $\text{mg/l}$ ) are used with leachate volume (l) and sample initial mass (kg) to calculate anion and cation leaching rates ( $\mu\text{g/kg/week}$  or  $\text{mg/kg/week}$ ). These leaching rates, along with conductivity, pH (and Eh) are plotted against cycle number (week number). The resultant curves indicate the progression of acid generation (sulphide oxidation) and neutralization. There are a variety of ways to plot the humidity cell data including cumulative anion or cation leached and residual anion or cation not leached (initial minus cumulative leached). Normally leachate data is plotted on a week by week continuous basis to monitor humidity cell progress, particularly with respect to pH. It is usual to terminate humidity cell tests after pH has stabilized at a constant value (and after the minimum number of cycles recommended by the appropriate regulatory agency). However, the presence of soluble sulphate minerals or the presence of previously oxidized material may mask or delay the onset of acid generation as noted earlier, and some samples may take 60 weeks or more to stabilize (Price, 1997). The point in time at which humidity cells are terminated is usually, therefore, site-specific, and may require considerable judgement by the project supervisor.

### Effect of Microorganisms

A subject of considerable uncertainty at this time is whether or not humidity cells should be inoculated with bacterial cultures such as *Thiobacillus ferrooxidans*. ASTM Designation D5744-96 (ASTM, 1996) includes instructions for the preparation of a *T. ferrooxidans* culture for humidity cell use, and for the method of introduction of the inoculum to the rock sample. It seems likely that samples of existing waste rock, ore or tailings would not require inoculation, whereas drill core samples might require inoculation. However, since it is generally accepted that

a consortium of bacteria, rather than *T. ferrooxidans* alone, is required for the bacterial enhancement of sulphide oxidation rates, and since even *T. ferrooxidans* is site specific (Leduc and Ferroni, 1994), the potential benefits of an inoculum of laboratory-prepared *T. ferrooxidans* are questionable.

### Testwork Reproducibility

The reproducibility of humidity cell data is not well documented. ASTM (1996) give comparisons for duplicates of two samples subjected to humidity cell testing for 120 weeks and shown in Table 2. There is no information on either the origin of the samples or their characteristics. Data is given for sulphate generation (release) rate.

**Table 2: Comparison of Sulphate Release Rates from Duplicate Humidity Cell Tests on Two Samples (from ASTM, 1996)**

TEST DURATION (20-WEEK INTERVALS)	AVERAGE SULPHATE RELEASE RATES (mg/kg/week)			
	SAMPLE 1		SAMPLE 2	
	CELL 1	CELL 2	CELL 1	CELL 2
0-20	182.5	184.1	523.3	558.7
20-40	321.8	254.6	887.1	868.1
40-60	354.8	197.5	1985.6	1481.1
60-80	306.5	192.1	1834.5	1292.1
80-100	294.0	235.0	1763.4	1247.6
100-120	227.6	141.3	3165.0	1660.7

The differences between duplicates in this example are significant, but may represent variability between sample splits, rather than variability in cell performance. In this case it would have particularly interesting to compare ABA analytical data and mineralogical information for the samples to determine if sulphide liberation characteristics were responsible for the differences.

Further discussion of the above subjects may be found on the Internet site *Acid Rock Drainage at Enviromine* (Mills, C. and Robertson, A.M., Eds.), <http://www.enviromine.com/ard>.

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