

Technical Article

The Use of Measured and Calculated Acidity Values to Improve the Quality of Mine Drainage Datasets

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Abstract: The net acidity of a water sample can be measured directly by titration with a standardized base solution or calculated from the measured concentrations of the acidic and basic components. For coal mine drainage, the acidic components are primarily accounted for by free protons and dissolved Fe^{2+} , Fe^{3+} , Al^{3+} , and Mn^{2+} . The base component is primarily accounted for by bicarbonate. A standard way to calculate the acidity for coal mine drainage is: $\text{Acid}^{\text{calc}} = 50 \cdot (2 \cdot \text{Fe}^{2+}/56 + 3 \cdot \text{Fe}^{3+}/56 + 3 \cdot \text{Al}/27 + 2 \cdot \text{Mn}/55 + 1000 \cdot 10^{-\text{pH}}) - \text{alkalinity}$, where acidity and alkalinity are measured as mg/L CaCO_3 and the metals are mg/L. Because such methods of estimating acidity are derived by independent laboratory procedures, their comparison can provide a valuable QA/QC for AMD datasets. The relationship between measured and calculated acidities was evaluated for 14 datasets of samples collected from mine drainage discharges, polluted receiving streams, or passive treatment systems, containing a total of 1,484 sample analyses. The datasets were variable in nature, ranging from watersheds where most of the discharges contained alkalinity to ones where all of the discharges were acidic. Good relationships were found to exist between measured and calculated acidities. The average acidity measurement was 239 mg/L CaCO_3 and the average acidity calculation was 226 mg/L CaCO_3 . Linear regressions were calculated for individual datasets and for the entire dataset. The linear regression for the entire dataset was: $\text{Acid}^{\text{calc}} = 0.98 \cdot \text{Acid}^{\text{meas}} - 8$, $r^2 = 0.98$. The good correlation between calculated and measured acidity is the basis for an easy and inexpensive QA/QC for AMD data. Substantial variation between measured and calculated acidities can be used to infer sampling or analytical problems.

Key Words: Acidity; alkalinity; coal mine drainage

Introduction

Reliable chemical analyses are an important component in designing mine water passive treatment systems, developing stream restoration plans, and assessing treatment and restoration efforts. Ideally, each mine water analysis includes a determination of all the major constituents, and the general accuracy of the measurements is determined from the balance of cations and anions. This analytical detail is not feasible for most mine water applications because of its high cost. Typically, a subset of chemical constituents is measured that includes parameters of regulatory concern. In the coal fields of the eastern U.S., this “regulatory” subset typically includes pH, acidity, alkalinity, Fe, Mn, Al, sulfate and total suspended solids. Many private laboratories can provide the regulatory analyses for one-third the cost of the more detailed analysis that would be appropriate for cation and anion balance. An objective method to assess the quality of this subset does not currently exist.

This paper describes a simple method to assess the quality of typical coal mine drainage analyses. The proposed method makes use of inherent inter-relationships of standard analytical parameters.

Specifically, the net acidity of a water sample, measured using the procedures described by the American Public Health Association (APHA 1999), is primarily a balance of alkalinity due to bicarbonate and acidity due to H^+ and acidic metals (Hedin et al. 1994, Kirby and Cravotta 2005a, Kirby and Cravotta 2005b). For coal mine drainage, the primary acidic metals are Fe, Al, and Mn. If the concentrations of the alkaline and acidic components are known, the net acidity of the water can be calculated. The correspondence of the calculated acidity value and the measured acidity provides an evaluation of the completeness and quality of the analytical results. The paper explores the relationship between calculated and measured acidity values for 14 data sets.

Background

Acidity Measurements

The acidity of mine drainage is determined by titration with base following oxidation with hydrogen peroxide and boiling (American Public Health Association 1999). The intent is to measure the amount of base (as CaCO_3) needed to neutralize the water to pH 8.3 under oxidizing conditions. The method involves the following steps:

- 1) the addition of a known quantity of acid (0.02N H₂SO₄), so that all of the alkalinity is eliminated and the pH is less than 4;
- 2) the addition of hydrogen peroxide (30% H₂O₂) followed by 2-5 minutes of boiling; and
- 3) the titration of the cooled solution with base (0.1N NaOH) to pH 8.3.

The addition of H₂O₂ assures that Fe and Mn are oxidized and form hydroxide solids during the base titration. Without H₂O₂ digestion, variable amounts of Fe²⁺ and Mn²⁺ can remain in solution at the end of the titration, causing an inaccurate measurement of acidity. Boiling exsolves dissolved CO₂, which should not be included in the titration because it exsolves naturally.

The net acidity of a sample is calculated from the difference of base additions (step 3) and acid additions (step 1). A positive result indicates that the sample has a net acidity. A negative result (more acid added in step 1 than base added in step 3) indicates that the sample has a net alkalinity.

The reporting of negative acidity values for samples with net alkalinity varies between laboratories and the standard method used. The 17th, 18th, and 19th Editions of APHA's Standard Methods of the Examination of Water and Wastewater provide the following guidance:

"...Report pH of the end point used as follows: "the acidity to pH ___ = ___ mg CaCO₃/L." If a negative value is obtained, determine the alkalinity according to Section 2320."

Section 2320 describes the conventional alkalinity determination, which does not contain a hydrogen peroxide provision for waters with hydrolysable divalent metals. The method is unclear about the reporting of a negative result. Most laboratories reported samples with negative results as having "0 acidity", "less than 0 acidity", or "negative." No laboratories, in the author's experience, reported the negative acidity value unless a special request was made. In these cases, the negative result was considered a non-standard method and usually reported as a FYI (for your interest) result.

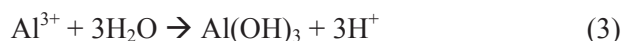
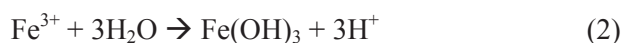
The 20th Edition of Standard Methods revised the reporting instructions:

"...Report pH of the end point used as follows: "the acidity to pH ___ = ___ mg CaCO₃/L." If a negative value is obtained, report the value as negative. The absolute value of this negative value should be equivalent to the net alkalinity."

Laboratories have begun to recognize the change and report negative acidity values.

Acidity Calculations

Mine water acidity arises from metals that undergo hydrolysis reactions and from protons (measured as pH). The principal metals in coal mine drainage are Fe²⁺, Fe³⁺, Al³⁺, and Mn²⁺. The acidity-producing hydrolysis reactions are shown below.



Acidity also is derived from free H⁺ ions, which are measured as pH.

Alkalinity arises primarily from bicarbonate ion.



Bicarbonate is only present, in concentrations measureable by titration with acid, when the pH is greater than 4.5. Alkalinity is conventionally reported as mg/L CaCO₃.

The total acidity can be approximated from equations 1-5 and the summary equation 6.

$$\text{Acid}^{\text{calc}} = 50 \cdot (2 \cdot \text{Fe}^{2+}/56 + 3 \cdot \text{Fe}^{3+}/56 + 3 \cdot \text{Al}/27 + 2 \cdot \text{Mn}/55 + 1000 \cdot 10^{-\text{pH}}) - \text{Alk} \quad (6)$$

where acidity and alkalinity are mg/L CaCO₃ and metals are mg/L.

Methods

This study used datasets from 14 monitoring programs where the goals were to characterize sources of AMD that pollute local receiving streams (13 sets) or to assess the performance of passive treatment systems (1 set). Samples were collected from sampling points by watershed association volunteers, Pennsylvania Dept. of Environmental Protection (PADEP) personnel, and Hedin Environmental (HE) personnel. In all cases, two samples were collected. A raw sample was collected for laboratory measurement of pH, alkalinity, acidity, sulfate, and total suspended solids, and an acid-preserved sample was collected for laboratory measurement of Fe, Mn, and Al. Samples were not filtered prior to acidification. Field measurements of alkalinity and pH were made for five of the monitoring programs. Whenever possible, acidity was

calculated using field pH and alkalinity. When field data were absent or considered unreliable, laboratory measurements of pH and alkalinity were used.

Acidity calculations were made using equation (6). Reliable iron speciation was only done by one laboratory for two datasets. For all other samples, the $\text{Fe}^{2+}/\text{Fe}^{3+}$ couple was assumed to be pH dependent. At pH less than 3, all iron was assumed to be Fe^{3+} ; at pH values equal and greater than 3, all iron was assumed to be Fe^{2+} . This split was based on the author's experience and a limited presentation of Fe^{3+} estimates made in Hedin et al. (1994). The robustness of this assumption will be discussed.

Five different laboratories were used to develop the 14 datasets. Most of the laboratories were not aware of the reporting change in the 20th Edition of Standard Methods and reported negative values as zero. As noted, this is not an accurate report of the measured acidity. For laboratories using this protocol, all samples with reported acidities of zero were excluded from the datasets and subsequent analyses. For laboratories that reported negative acidities, all data were considered in the analyses.

Results

Table 1 shows the average chemistry for each of the 14 datasets considered. The samples ranged widely in their characteristics. The Chart and Mill datasets were characterized by waters containing alkalinity, elevated concentrations of Fe, and low concentrations of Al. The Jon dataset was characterized by low pH

water with high concentrations of Al and low concentrations of Fe. The Cadog, Bear, and Botanical datasets were characterized by waters with very low pH and high concentrations of Fe and Al.

Table 1 shows average measured acidity (AM) and calculated acidity (AC) values. When all the datasets were combined, the average calculated acidity was 226 mg/L and the average measured acidity was 239 mg/L, a difference of 5%. (Relative differences were calculated from the difference of the measures divided by AM.) The individual datasets ranged from an under-calculation of 32% for the Chart dataset to an over-calculation of 17% for the HowMor dataset. Variation between the laboratories was apparent. Average relative differences are presented below:

- Laboratory B, two datasets, +16%
- Laboratory S, one dataset, +3%
- Laboratory G, four datasets, -6%
- Laboratory H, two datasets, -10%
- Laboratory P, five datasets, -14%

At two sites, two laboratories split the analytical responsibilities. At the Jon site (low pH, high Al, and low Fe) calculated acidities were approximately equal to measured acidities for both laboratories. At the Anna site (low pH, high Al, Fe, and Mn), the calculated acidities for both laboratories were less than measured acidities.

Table 1 also shows the linear regressions calculated for each individual dataset and for the entire dataset. Thirteen of the 14 datasets exhibited strong relationships

Table 1. Average chemical characteristics of the mine drainage datasets and linear regressions for the datasets, where $\text{Acid}^{\text{calc}} = a \cdot \text{Acid}^{\text{meas}} + b$, r^2

	Lab	N ^a	pH	Alk ^b	Fe	Mn	Al	SO ₄	AM	AC	Dif %	Linear regression	r ²
Dole Farm	S	86	4.3	7	48	6	8	575	178	183	+3%	AC = 1.13 AM - 17	0.98
Cadog	G	295	4.5	6	28	10	14	612	163	154	-6%	AC = 0.92 AM + 3	0.96
Chart	G	34	3.3	1	178	12	36	1,761	886	763	-14%	AC = 0.82 AM + 38	0.98
Elk	P	43	5.3	46	57	1	2	598	97	66	-32%	AC = 0.92 AM - 24	0.69
Botanic	P	146	3.7	3	5	2	10	298	89	66	-26%	AC = 0.79 AM - 4	0.97
Bear	P	46	3.3	1	23	2	38	545	320	309	-3%	AC = 1.01 AM - 17	0.98
Henry	G	345	4.5	7	95	7	20	631	378	378	0%	AC = 1.00 AM - 5	0.99
HowMor	G	13	4.3	5	31	7	8	373	120	118	-2%	AC = 0.96 AM + 3	0.98
Mill	B	43	5.6	88	175	39	<1	1,177	271	318	+17%	AC = 1.11 AM + 17	0.96
Jon - P	B	41	4.1	24	79	41	5	927	215	248	+15%	AC = 1.08 AM + 5	0.98
Jon - H	P	272	4.1	5	1	4	28	183	166	166	0	AC = 1.04 AM - 7	0.99
Anna - P	H	37	4.4	3	1	5	29	286	174	177	+2%	AC = 1.01 AM + 1	0.97
Anna - H	P	11	3.0	0	21	12	22	380	286	251	-12%	AC = 1.13 AM - 62	0.93
All data	H	72	3.0	0	19	11	20	538	305	238	-22%	AC = 0.78 AM - 6	0.87
All data		1,484	4.1	14	54	11	17	635	239	226	-5%	AC = 0.98 AM - 8	0.98

"N" is the number of samples; "Alk" is alkalinity; "AM" is measured acidity (note: the Farm and Bear sites included negative measured acidities, while the others did not); "AC" is calculated acidity; "Dif %" calculated (AC-AM)/AM; acidity and alkalinity are mg/L CaCO₃; Fe, Al, Mn, and sulfate are mg/L.

between AM and AC. The exception was the Chart dataset, which had the poorest relationship between AC and AM (32% difference) and the weakest statistic fit ($r^2 = 0.69$). This data set was characterized by samples containing elevated Fe and alkalinity. The net alkaline samples (47% of the dataset) were removed from analysis because the acidities were reported as zero; the author discovered that the laboratory was acidifying alkaline samples with a fixed amount of sulfuric acid. For samples with more than 100 mg/L alkalinity, the acid addition was not sufficient to neutralize all of the alkalinity. Incomplete neutralization of the pre-existing alkalinity may have contributed to the inaccuracy and variability of results. The laboratory has since corrected the procedural error. Recent analyses by the same laboratory of samples collected from the same Chart sites yielded a good correlation between calculated and measured acidity.

A perfect relationship between acidity estimates would yield a linear regression with 1.0 slope and zero intercept. The linear regression for the entire dataset had a slope of 0.98, an intercept of -8, and an r-square of 0.98.

Discussion

In order to use the relationship between measured and calculated acidity in a quality control context, one should appreciate the sources of error. If the controllable errors can be largely eliminated, then the relationship can be used to reliably identify samples with suspect analytical results and to alert laboratories to systemic analytical problems.

Errors Influencing Acidity Measurements

The hot peroxide method is a robust method if the protocols are followed. The primary error associated with acidity measurements is in the reporting of net alkaline samples. Many laboratories still report net alkaline samples as having zero acidity and the inclination is to assume that the alkalinity represents the net alkalinity. For samples that contain Fe or Mn, this assumption results in an erroneously high net alkalinity because a portion of this alkalinity is consumed during the oxidation and hydrolysis of Fe^{2+} and Mn^{2+} . If the laboratory reports negative acidity as zero, then all acidities with zero values should be removed from the comparison of measured and calculated acidity values. The laboratory should be informed that the reporting protocols for samples with negative acidity have been changed in the 20th Edition of Standard Methods (APHA 1999). If the original laboratory notes were retained, it is possible to recalculate and correct the acidity values.

Errors Associated with the Acidity Calculations

The acidity calculation (equation 6) includes several parameters whose measurement can potentially affect the accuracy of the acidity calculation.

Absence of field measurements of alkalinity and pH

Samples that contain both bicarbonate alkalinity and Fe^{2+} are unstable. If oxygen is introduced during sample collection or storage, iron oxidation and hydrolysis will occur in the sample bottle that is used by the laboratory for pH and alkalinity measurements. Iron hydrolysis reactions consume alkalinity and can change the pH. If the sample is net acidic, the pH can decrease from a field value of 6 to a laboratory value less than 4. If the sample is net alkaline, large changes in pH are unlikely, but a substantial decrease in alkalinity is possible.

In the absence of reliable field data, laboratory data must be used. The most substantial errors that result from the use of laboratory data occur for alkaline waters with high Fe concentrations. If an alkaline mine water sample contains 100 mg/L Fe, it is possible for the alkalinity to decrease by 180 mg/L between its collection and its determination in the laboratory. Because samples such as these commonly have measured acidities ranging from -200 mg/L to +200 mg/L, an error of 100-200 mg/L in the alkalinity determination is obviously a substantial source of error in the acidity calculation.

Figure 1 shows alkalinity measurements made for a discharge from an anoxic limestone drain. Field measurements were made within 30 minutes of sample collection. Laboratory measurements were made after transport to a central laboratory. Laboratory measurements were not made for at least 24 hours and could have been delayed for as long as 14 days and still conformed with sample storage recommendations (APHA 1992). The actual storage time for each sample was not known. The field measurements indicate that, after an early decline in alkalinity, the system consistently produced an effluent with 190-210 mg/L alkalinity. The laboratory data suggest a more variable effluent, with alkalinity concentrations ranging between 150 – 210 mg/L. This variability is largely due to errors introduced by variable sample storage. The lower values represent losses in alkalinity that occurred between sample collection and alkalinity measurement at the laboratory.

Acidity calculations made using laboratory alkalinity measurements suggest that the discharge is net acidic on occasions when the laboratory only measured 150-160 mg/L of alkalinity. The error in this assessment is

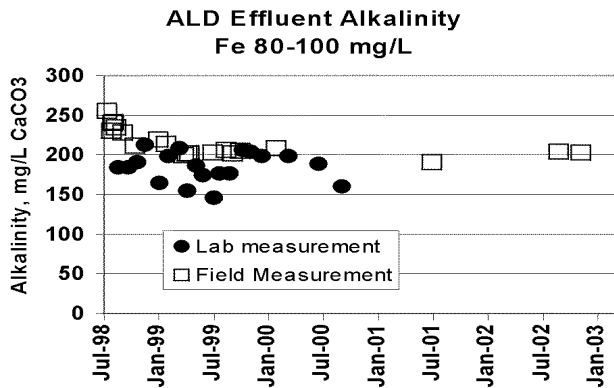


Figure 1. Differences between field and laboratory measurements of alkalinity for an anoxic limestone drain discharge

evident from the field alkalinity measurements and also from additional sampling of the passive system. (The final effluent never had a pH less than 6.)

Substantial pH changes can occur between sample collection and the laboratory when the water contains alkalinity, ferrous iron, and is net acidic. A delay of several days (a weekend) can result in a pH measurement that is 2-3 units lower than what was measured in the field. These changes, while notable, do not affect the acidity calculation substantially. At a pH of 3.5, the H^+ only contributes 16 mg/L to the acidity calculation. This increase in H^+ acidity is usually substantially smaller than the loss of alkalinity that occurred while the pH was decreasing.

The extent of the changes in pH and alkalinity depends on the amount of aeration that occurs during sample collection and storage, the storage conditions, and the length of storage. These are difficult parameters to standardize. The preferred option is to make alkalinity and pH measurements in the field. If field measurements are not possible, then samples should be collected from below the surface of the water (to avoid aeration), and the sample bottles should be filled to the top so that there is no headspace. The samples should be kept on ice and delivered to the laboratory as quickly as possible. The pH and alkalinity should be measured immediately on receipt at the laboratory.

Alkaline samples that do not contain Fe can undergo CO_2 degassing during storage, which can raise the pH without affecting alkalinity. Typically, the increase is less than one unit (increase from pH 6 to 7) and the effect on the acidity calculation is less than 1 mg/L.

Presence of suspended solids in the acidified sample

Solids that are collected in acidified bottles are generally dissolved by the acid and subsequently

measured by the metal analysis. Clay and silt particles will elevate aluminum concentrations. Iron oxide solids will elevate Fe concentrations. Solids do not undergo acid-producing hydrolysis reactions, so samples with substantial suspended solids will yield erroneously high acidity calculations.

The collection of a clean sample for the acid-preserved sample should be a high priority. Often the collection of suspended solids can be traced to disturbance of the sampling area by the collector. When a clean sample cannot be collected, the solids can be removed through filtration. If filtration is not possible, then the samples should be flagged as containing solids and should be considered poor candidates for acidity calculations.

Few mine drainage seeps contain suspended solids. In our sampling of hundreds of AMD discharges, we have only encountered a few mine discharges that contain suspended solids at their source. The most common occurrence of samples with suspended solids in our studies are samples collected from within systems treating Fe-rich water and samples collected from receiving streams during storm events. In passive treatment systems, it is common for ponds or wetlands to have water that contains visible iron hydroxide as suspended solids. Comparisons in the 1990's by the U.S. Bureau of Mines of filtered and unfiltered water samples from passive treatment systems indicated that the upper limit for particulate Fe was about 15 mg/L Fe (unpublished data collected by R. Hedin and R. Nairn, 1992-94). This amount of Fe, mistakenly considered as dissolved ferrous iron, would errantly overestimate acidity by 27 mg/L.

Suspended solids introduced by storm events into treatment systems and streams are usually re-suspended iron hydroxide and clay particles that, when dissolved, result in elevated concentrations of Fe and Al. It is not easy to adjust data to correct for this problem. Samples that are unavoidably contaminated with suspended sediments should be flagged as inappropriate for acidity calculations.

Ferrous/ferric iron assumptions

The acidity calculation differentiates between ferrous iron (Fe^{2+}) and ferric iron (Fe^{3+}). Methods exist for the determination of Fe^{2+} . No reliable low-cost method exists for Fe^{3+} , so ferric iron is determined from the difference of total dissolved iron and dissolved ferrous iron. Ferrous iron is rarely determined for mine drainage samples because it requires additional sampling (a separate sample preserved with hydrochloric acid), an added expense, and it is difficult to obtain accurate measurements.

So, for most of the datasets analyzed here, iron speciation was estimated from pH. Ferric iron has a very low solubility at pH values greater than 4, so any dissolved iron present in the pH 4-8 range can be confidently assumed as Fe^{2+} . At pH less than 3, ferric iron is highly soluble; it is commonly present in oxidized samples as Fe^{3+} and as one or more complexed ions.

At pH values less than 3, complexing of Fe and Al by OH^- becomes significant. In theory, these complexes could weaken the accuracy of the acidity calculation because these metals produce less acidity than is indicated by equations 2 and 3. However, sulfate complexes, such as HSO_4^- compensate somewhat (Kirby and Cravotta 2005a, 2005b).

The assumption that all samples with pH less than 3 are oxidized is debatable because some mine water samples with pH less than 3 have high Fe^{2+} concentrations. However, a compilation of all the samples with low pH indicated that the assumption results in a very good correlation between measured and calculated acidities. Figure 2 shows the relationship for samples with laboratory pH less than 3 and where all the iron was assumed to be Fe^{3+} . The average sample in this subset had a pH of 2.69 and contained 546 mg/L Fe, 86 mg/L Al, 15 mg/L Mn, and 3,029 mg/L sulfate. The average measured acidity was 2,171 mg/L, while the average calculated acidity was 2,130 mg/L.

For datasets containing many samples with pH less than 3, it is recommended that occasional measurements of Fe^{2+} be made so that an empirical estimate of the $\text{Fe}^{2+}/\text{Fe}^{3+}$ relationship can be made. However, it is the author's experience that laboratories without experience in ferrous iron measurements often provide inaccurate results. The laboratory should be required to assure its ferrous iron accuracy with rigorous QA/QC tests.

Using the relationship to identify sampling and analytical problems

If the sampling and analytical errors discussed above are minimized, then the resulting dataset should provide a good relationship between measured and calculated acidity values. The dataset summaries presented in Table 1 show that the relationship is robust and that it exists across datasets of varying water quality and different laboratories. On average, calculated acidities were within 5% of the measured acidities. The relationship can be used to identify problems with individual samples. Because sampling of mine drainage involves the collection of two samples at each location, paired samples can be

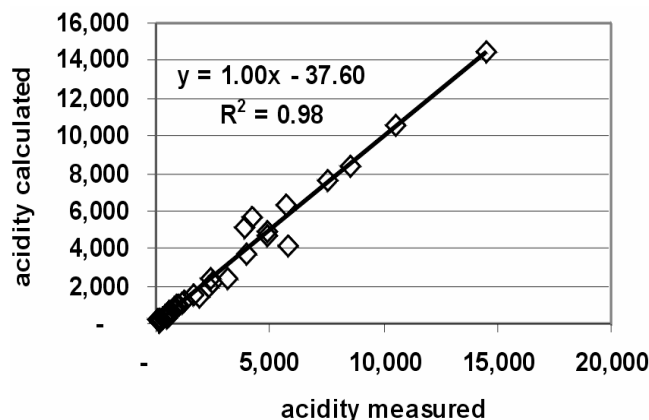


Figure 2. Measured and calculated acidity values for 48 samples with pH less than 3; all samples analyzed by Laboratory G

occasionally mismatched. If the mismatched samples vary in chemical composition, the error should be apparent from the disagreement of measured and calculated acidity values. It is then possible to contact the laboratory and try to sort out the problems or decide that the sample pair is invalid and exclude it from the dataset.

Summary

A method for evaluating the quality of mine drainage has been described that compares measured and calculated acidity values. In order to assure that the comparisons are as accurate as possible, efforts should be made to collect samples without suspended solids. The pH and alkalinity should be measured in the field or immediately after delivery to the laboratory, and the laboratory should report negative acidity values when appropriate. When these conditions are followed, it is possible to identify suspect data and suspect laboratory analyses by comparing measured and calculated acidities. The method is essentially a balance of acidic and alkaline components of the mine water samples. This approach provides reliable QA/QC at a fraction of the cost of conventional QA/QC done by balancing cations and anions.

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Important Dates

10th Feb 2007: submission of abstracts

10th Feb 2007: early bird registration

15th April 2007: submission of full Papers