

EVALUATION OF MINE DRAINAGE ABATEMENT PROJECTS: COMPARISON OF RESULTS FROM SIMPLE FIELD TESTS OF WATER QUALITY WITH LABORATORY ANALYSES¹

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Abstract. Acid mine drainage (AMD) from surface and underground coal mines abandoned prior to August 3, 1977, has received additional attention in recent years. In response to this interest, the Mid-Continent Region of the Office of Surface Mining Reclamation and Enforcement has assisted in reconnaissance of AMD problem sites, design of passive treatment systems, and evaluation of AMD remediation measures. This paper will discuss the application and suitability of field test methods such as colorimetric analysis of key anions and cations. Field measurements were statistically compared with data generated from conventional laboratory techniques such as inductive coupled plasma (ICP) and ion chromatography. The purpose of this paper is to demonstrate the ability of low-cost field methods to supplement conventional analytical techniques. Important applications include: (1) reconnaissance sampling which can be followed by laboratory testing once field conditions are understood and (2) diagnostic tests to evaluate the performance of AMD treatment systems. Several field methods have been found to be comparable to laboratory tests, while others are useful only for an estimation of water quality. One test, the ferrous iron colorimetric method, is considered the standard method for this constituent and is recommended over laboratory testing. An evaluation of the use of field versus laboratory methods for determining total alkalinity is also included.

Additional Key Words: colorimetric analysis, inductive coupled plasma, ion chromatography, and AMD treatment.

¹ Paper was presented at the 2010 National Meeting of the American Society of Mining and Reclamation, Pittsburgh, PA *Bridging Reclamation, Science and the Community* June 5 - 11, 2010. R.I. Barnhisel (Ed.) Published by ASMR, 3134 Montavesta Rd., Lexington, KY 40502.

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Proceedings American Society of Mining and Reclamation, 2010 pp 6-21

DOI: 10.21000/JASMR10010006

<http://dx.doi.org/10.21000JASMR10010006>

Introduction

The Mid-Continent Region of the Office of Surface Mining Reclamation and Enforcement (OSM) has assisted in reconnaissance of AMD problem sites, design of treatment systems, and evaluation of constructed passive treatment systems since 1998. The first large-scale system built with OSMRE assistance was the rehabilitation of the Old Bevier Aerobic Wetland in Macon County, Missouri which the Missouri Department of Natural Resources constructed in 1990 and 1991 (Behum *et al.*, 2001, 2002; Kim *et al.*, 2004).

Data examined for this study contained sets of paired values, one from a commercial laboratory and one generated “in-house.” The latter values variously came from tests done on-site, tests run within several hours of sample collection (these tests were usually done in a hotel room), or tests run at a later time in an office laboratory. Time constraints or inclement weather, particularly low temperatures, often determined where in-house samples would be tested. Regardless of where and when the in-house samples were analyzed, the techniques were the same and all of these analyses could have been done on-site. Consequently, these samples will be referred to as field samples and their analysis was by field methods. At various times, any one of a number of commercial laboratories had tested water samples of the data set. The term “commercial laboratories” applies here to state environmental laboratories as well as business enterprises.

Test Methodology

Field and laboratory parameters used to characterize AMD have been those suggested by Hyman and Watzlaf (1995), Wildeman *et al* (1997) and Ball *et al* (1999) include dissolved metals (Fe, Al, and Mn) and sulfate. Water analyses typically have included the important measurement of Fe²⁺. Total and Fe²⁺ dissolved iron concentrations were determined in the field with a portable colorimeter. In this context, “total iron” refers to the sum of Fe²⁺ and Fe³⁺ iron concentrations. Dissolved Fe³⁺ iron values were calculated by subtracting Fe²⁺ iron from total dissolved iron. Additional field measurements included temperature, pH, redox potential (Eh), specific conductivity, salinity, dissolved oxygen, acidity, and, where the pH was > 4.5 total alkalinity. Either potentiometric or color-indicator titration methods were used for in-house determination of acidity and alkalinity.

Mine waters often have high concentrations of the analytes of interest. Samples then have to be diluted to drop concentrations within their prescribed range of the test method. Dilution can be a significant source of error, both in regard to the accuracy by which volumes are measured and in the use of the correct multiplier of the analytical results. Making accurate dilutions in the field is a challenge. Some of the earlier values reported in the data set apply to samples diluted through the use of graduated cylinders and eye droppers. For a number of years now, the practice has been to make field dilutions using plastic Class B volumetric flasks and pipettes with disposable tips. Glass Class A volumetric flasks are now routinely used for those samples processed in the office laboratory.

Water samples in this study were collected as prescribed by the 19th Edition of Standard Methods for the Examination of Water and Wastewater, hereafter referred to as Standard Methods. A split was separated from each collected sample drawn through a 0.45-micrometer nitrocellulose filter. In cases where the raw sample contained high turbidity, the sample was drawn through a glass fiber pre-filter. This split was then used for field metals analyses. Normally, colorimetric analysis of ferrous and total dissolved iron was made within 8 hours. Ferric iron was calculated by subtracting measured ferrous iron concentration from measured total dissolved iron. Some investigators have attempted to develop a colorimetric method to directly determine Fe^{3+} iron (Wildeman *et al*, 1997; Ball *et al*, 1999). However, these methods have not been widely accepted and commercialized. Samples for later colorimetric analysis and those to be tested by a commercial laboratory were preserved to a pH <2.0 with 1:1 HNO_3 acid (2 mL of acid per 1,000 mL of sample). Colorimetric analysis for metals was completed in the office laboratory within 6 months. Raw (unfiltered and non-preserved) samples that had a field pH > 4.5 were typically titrated on-site for alkalinity. Alkalinity was reported as zero for those samples that had a pH < 4.5. Some alkalinity data came from the inflection point titration (IPT) method (Rounds, 2006). IPT was run on a filtered sample using a digital titrator and pH probe. Sulfate in the raw sample split was determined within 28 days of collection by the colorimetric SulfaVer[®] method. The individual colorimetric tests are described below.

Measurement of acidity is critical to characterize mine drainage (Hyman and Watzlaf, 1995; Watzlaf and Hyman, 1995). The normal laboratory method for total acidity involves first treating the mine water sample with 30% H_2O_2 and heat, then titrating a known sample volume with NaOH. Sample heating and addition of H_2O_2 before the acid-neutralizing titrant is added is

intended to rapidly complete what would otherwise be a slower natural process of metal oxidation, hydrolysis, and consequent acid generation. The total acidity test as just described is also known as the hot acidity test. The special sample preparation required for total (hot) acidity generally precludes its use as a field test. Consequently, the data set used in this study did not contain values for acidity.

An alternative (or supplement) to the total (hot) acidity test is to calculate acidity. Calculation of acidity requires a field pH measurement and careful determination of the concentrations (mg/L) of the dissolved metals Fe^{2+} , Fe^{3+} , Mn, and Al. The formula typically used is:

$$\text{Acidity}_{\text{calc}} = 50[1000(10^{-\text{pH}}) + 2(\text{Fe}^{2+})/56 + 3(\text{Fe}^{3+})/56 + 2(\text{Mn})/55 + 3(\text{Al})/27]. \quad (1)$$

As in measured total (hot) acidity, the units are mg/L as a CaCO_3 equivalent.

Calculated total acidity (Hyman and Watzlaf, 1995) generally corresponds well with total acidity as measured in the laboratory. In the past, some laboratories mathematically incorporated alkalinity into the total acidity value, making comparisons to calculated acidity difficult. The procedure for total acidity has been clarified in recent editions of Standard Methods and is well described by others (Kirby and Cravotta, 2005a, 2005b). Kirby and Cravotta (2005a, 2005b) reported that the acidity calculation underestimates contributions from H_2SO_4 and H^+ while overestimating acidity due to Fe^{3+} and Al^{3+} but that these small errors tend to cancel out. Also, in some mine settings (for example, those having elevated Ni and Zn) the contributions to acidity by the hydrolysis of additional metals may become significant and should be considered (Hyman and Watzlaf, 1995).

Most all of the field measurements discussed here were taken with a Hach Company DR/890 colorimeter (Fig. 1) employing Hach reagents and methods. Products and technologies described below are for informational purposes only and should not be construed as endorsement.

Aluminum – Aluminon Method, Hach Method 8012 for water and wastewater

This method is adapted from Standard Methods (1995) but is not specifically approved by EPA. Typically, the preserved water sample is diluted to negate iron interference ($\text{Fe} > 20 \text{ mg/L}$) and to avoid exceeding the upper concentration limit of 0.8 mg/L. The pH is adjusted between

3.5 and 4.5 with 5.0 N NaOH standards solution prior to analysis. Ascorbic acid is added to remove any remaining iron interference.



Figure 1. A typical field colorimetric test kit, the Hach DR/890.

Aluminon indicator combines with Al in the sample to form a red-orange color. Depth of color is proportional to the Al concentration. A colorimeter such as the Hach DR/890 hand-held instrument measures the light transmitted through or absorbed by the colored sample. This test is time consuming, requiring approximately half an hour per analysis. That includes a 15-minute wait period for color development. The sample blank is prepared by adding a bleaching reagent to a separate portion of the Aluminon-treated sample. The presence of an Al complex may impact the accuracy of test results. Digestion is, therefore, required for all test methods prior to for analysis of total metals (Standard Methods, 1995).

Ferrous Iron—1,10-Phenanthroline Method, Hach Method 8146 for water and wastewater

The indicator, 1, 10-phenanthroline, reacts with Fe^{2+} iron to form an orange color in proportion to the iron concentration; ferric iron does not react. Preserved water samples often require dilution to avoid the upper concentration limit of 3.0 mg/L. This is a field test which

should be conducted as soon as possible to avoid the conversion of Fe^{2+} to Fe^{3+} iron by atmospheric oxygen inadvertently added during sample collection. A sampling-induced shift in the ratio of iron species is particularly likely for ground water or ground-water seepage—waters that often have naturally low dissolved oxygen concentrations. Hach Method 8146 is adapted from Standard Methods (1995) but is not specifically approved by EPA.

Total Iron – FerroVer[®] Method, Hach Method 8008 for water and wastewater

The FerroVer[®] reagent reacts with all soluble iron in the sample and most insoluble forms to produce soluble Fe^{2+} iron. Subsequently, 1, 10-phenanthroline reacts to form an orange color in proportion to the total iron concentration. The preserved water sample often requires dilution to avoid the upper concentration limit of 3.0 mg/L. The pH is adjusted between 3 and 5 with 5.0 N NaOH prior to analysis. This method is adapted from Standard Methods (1995) and is approved by EPA for analysis of wastewater (*Federal Register*, Vol. 45, No. 126, pp. 43459 (June 27, 1980); 40 CFR136.3, Table IB). Metals can form a complex with inorganic and organic species. As with Al, the presence of metal complexes may impact the accuracy of test results. Therefore, digestion is required for all test methods prior to analysis of total metals. Another colorimetric iron test method, the FerroZine[®] method, is also reported to provide accurate results for field determination of total iron (Ball and others, 1999) but was not evaluated in this study.

Manganese (High Range) – Periodate Oxidation Method, Hach Method 8034 for water and wastewater

This method has an upper concentration limit of 20.0 mg/L and is adapted from Standard Methods (1995) and is approved by EPA for analysis of wastewater [*Federal Register*, Vol. 44, No. 116, pp. 34193 (June 14, 1979)]. Again, digestion is required for analysis of total metals with all methods to eliminate the impact of metal complexes. This colorimetric method uses sodium permanganate to oxidize the sample. A purple color develops that is proportional to the manganese concentration. Iron (> 5 mg/L) interferes with the test, necessitating dilution. However, this often reduces the Mn level to below the detection limit of 0.2 mg/L. When this occurs, the colorimetric Mn low range test is run. Another problem is that highly buffered samples or those with extreme pH will exceed the buffering capacity of the test reagent set. Prior to testing, the pH must be adjusted to between 4 and 5 with additions of a 5.0 N NaOH standard solution.

Manganese (Low Range) – PAN Method, Hach Method 8049 for water and wastewater

This method is adapted from Goto and others (1977) and is approved by EPA for analysis of wastewater [*Federal Register*, Vol. 44, No. 116, pp. 34193 (June 14, 1979)]. Ascorbic acid is first added to reduce all manganese forms to Mn^{2+} . Subsequent addition of alkaline-cyanide reagent masks potential interferences from Fe (> 5 mg/L) and Al (> 20 mg/L). A low upper limit of this test (0.700 mg/L) will often necessitate dilution, but the low Mn detection limit of 0.07 mg/L allows for considerable dilution. The indicator PAN [1-(2-Pyridylazo)-2-Naphthol; $C_{15}H_{11} \cdot N_3O$] is added to form an orange-colored, metal-organic complex. The orange color is proportional to the Mn concentration. Prior to testing, the pH must be adjusted to between 4 and 5 with additions of a 5.0 N NaOH standard solution. The presence of metal complexes may impact the accuracy of test results by all methods. Therefore, digestion is required for analysis of total metals. The waste from this test is a hazardous cyanide compound that requires proper disposal.

Sulfate – SulfaVer[®] 4 Method, Hach Method 8051 for water and wastewater

This method is adapted from Standard Methods (1995) and is equivalent to EPA Method 375.4 for analysis of wastewater (Hach, 2004). Sulfate ions react with Ba to form an insoluble $BaSO_4$ precipitate. The precipitate forms turbidity which is proportional to the sulfate concentration. A stabilizing agent keeps the particles in suspension. The estimated detection limit is 4.9 mg/L. Considerable dilution is required for AMD samples because the upper concentration limit of this test is 70 mg/L.

Careful dilutions are required for all of the above mentioned tests. When small amounts of sample are added to a large dilution water volume, accuracy and precision are critical. Although difficult, these precautions are not impossible in the field environment. In practice this is accomplished by withdrawing a small amount of sample in an adjustable pipette such as the Hach TenSette[®] pipette (0.2 to 1.0 mL range), placing it into a 50 or 100 mL Class B polypropylene volumetric flask, and then filling the container with deionized water. Meticulous cleaning of all “glassware” is also critical, including a rinse with 10% HNO_3 acid solution to remove metals adhering to the container.

Alkalinity – Phenolphthalein and Total Methods, Hach Method 8203

The sample is titrated with H_2SO_4 acid to the end point (Fig. 2). The amount of acid added corresponds to the alkalinity. If the initial pH of the sample is more than 8.3 then the phenolphthalein alkalinity is determined by adding the phenolphthalein indicator then titrating with H_2SO_4 acid until there is a color change (sample changes from pink to colorless at pH 8.3). Total alkalinity is determined by adding the indicator bromocresol-methyl red and titrating to a pH of 4.5 with H_2SO_4 acid (color changes from green to light pink). This provides a measure of total alkalinity from which the hydroxide, carbonate and bicarbonate alkalinity may be determined. For samples with the initial pH < 8.3, the phenolphthalein alkalinity step is neglected. Bicarbonate alkalinity would then equal total alkalinity.



Figure 2. Measuring Alkalinity using a digital titrator.

Comparison by Analytical Method

To compare our analyses by field methods with laboratory results, a database was constructed with values collected since 2000. The data set contains 162 sampling events for which there was at least one set of paired water-constituent analyses. Samples from five states within the OSM Mid-Continent Region—Arkansas, Indiana, Illinois, Missouri, and Oklahoma—

are represented in the data set. The number of data pairs for a given constituent ranged from 51 to 114. The analytical values of this data set were not normally distributed. Transforming the data by taking the natural log did not produce normally distributed values in most cases. In general, good agreement was found between the field-capable colorimetric methods and laboratory testing by ICP (Fig. 3 through 9).

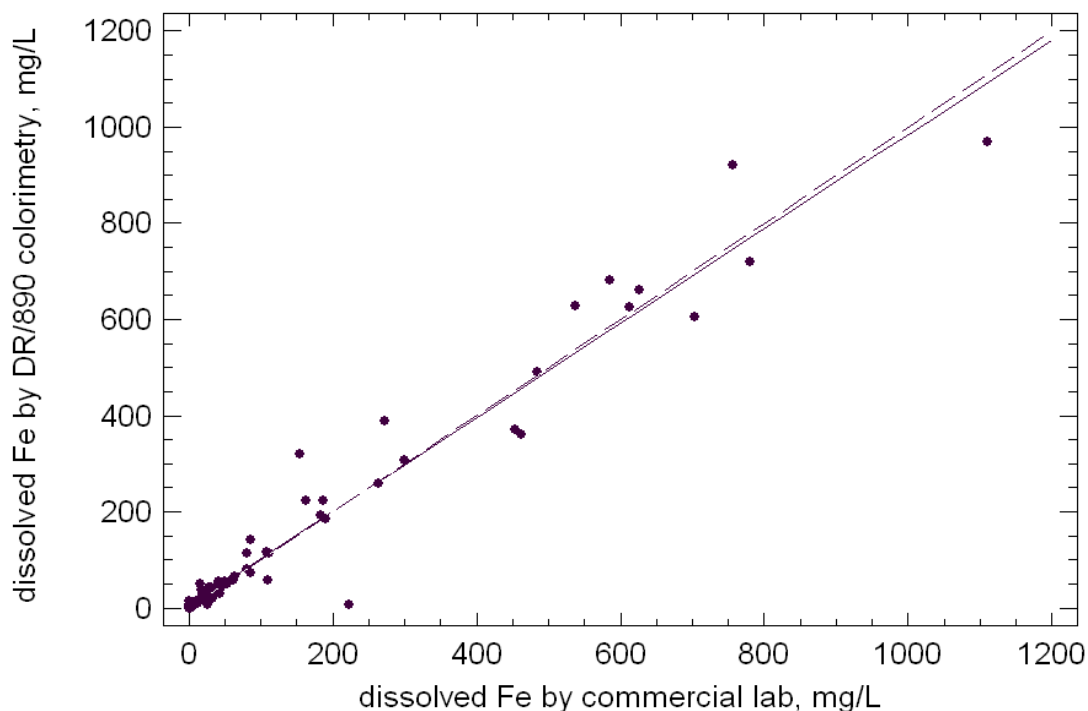


Figure 3. Scatter plot of dissolved iron with regression line (solid) and line of perfect correlation (dashed), N=114.

Comparison of colorimetric dissolved iron to ICP-measured dissolved iron, in particular, shows good agreement (Fig. 3). The line of perfect correlation ($Y=X$) nearly matches the regression line for the dissolved iron data. The equation of the regression line ($R^2 = 0.96$) is

$$\text{Field dissolved iron} = 3.8 + 0.98 \text{ lab dissolved iron} \quad (2)$$

Field and laboratory values for total iron exhibited a substantially lower coefficient of determination (Fig. 4, $R^2 = 0.44$). This is consistent with the findings of Wildeman *et al* (1997) and may be attributed to the absence of sample digestion in the field tests (i.e., some iron is adsorbed onto organics or clay particles). Based on a limited number of tests of Fe^{2+} iron, field analyses compared well with laboratory testing (Fig. 5, $R^2 = 0.98$). Colorimetric analysis is the accepted technique for determination of Fe^{2+} iron (Standard Methods, 1995).

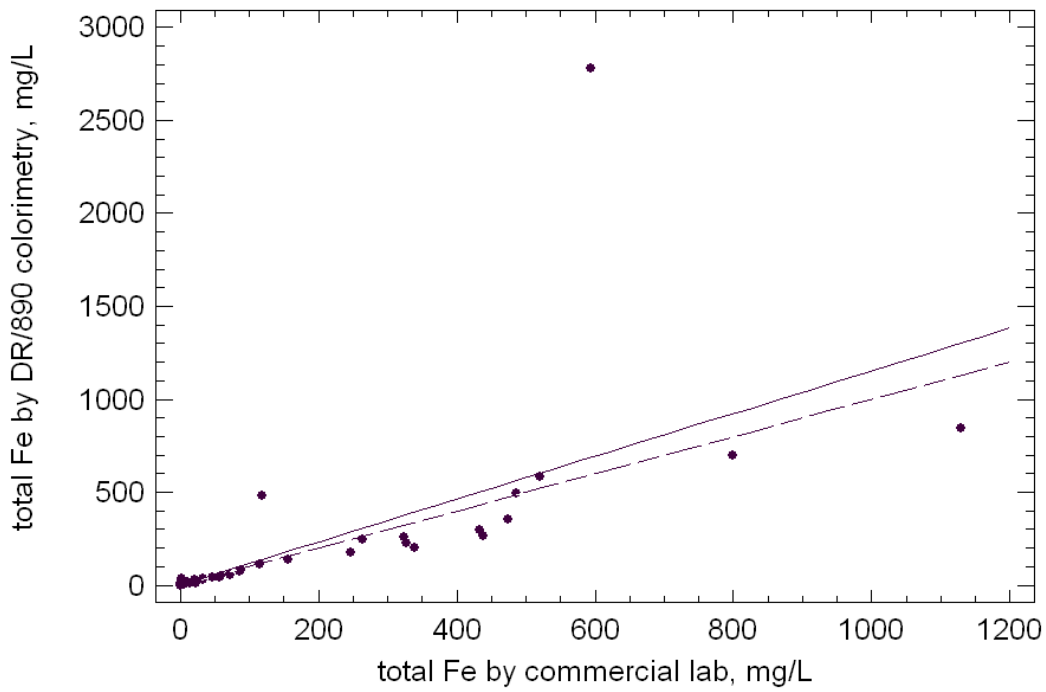


Figure 4. Scatter plot of total iron with regression line (solid) and line of perfect correlation (dashed), N=51.

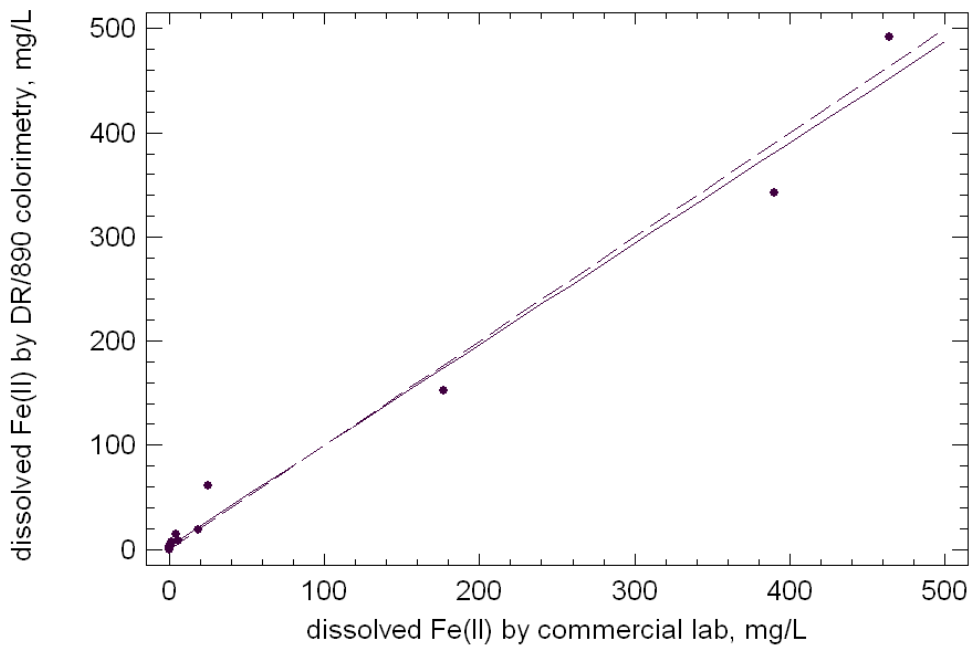
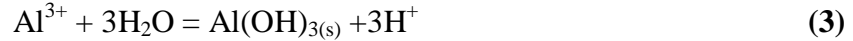


Figure 5. Scatter plot of dissolved Fe^{2+} iron with regression line (solid) and line of perfect correlation (dashed), N=21.

A larger deviation between field colorimetric tests and laboratory tests was observed for aluminum. Accurate determination of Al is necessary because hydrolysis of this metal produces a large amount of protons and, as a result, acidity.



Wildeman *et al* (1997) observed that colorimetric analyses of total aluminum were significantly lower than the lab results. This discrepancy was attributed to (1) the complicated nature of the Aluminon test procedure and (2) the use of deionized water without a pH adjustment that lead to precipitation of $\text{Al}(\text{OH})_3$. Results from the present study confirm this observation and show that the Aluminon test method produced values that were somewhat lower than those from laboratory tests for both dissolved and total Al (Fig. 6 and 7; $R^2 = 0.84$ and 0.96 , respectively). These data could have been affected by formation of ionic complexes or by dilution errors in tests where high Al was present; high Al content is a condition not uncommon with the Illinois Basin AMD.

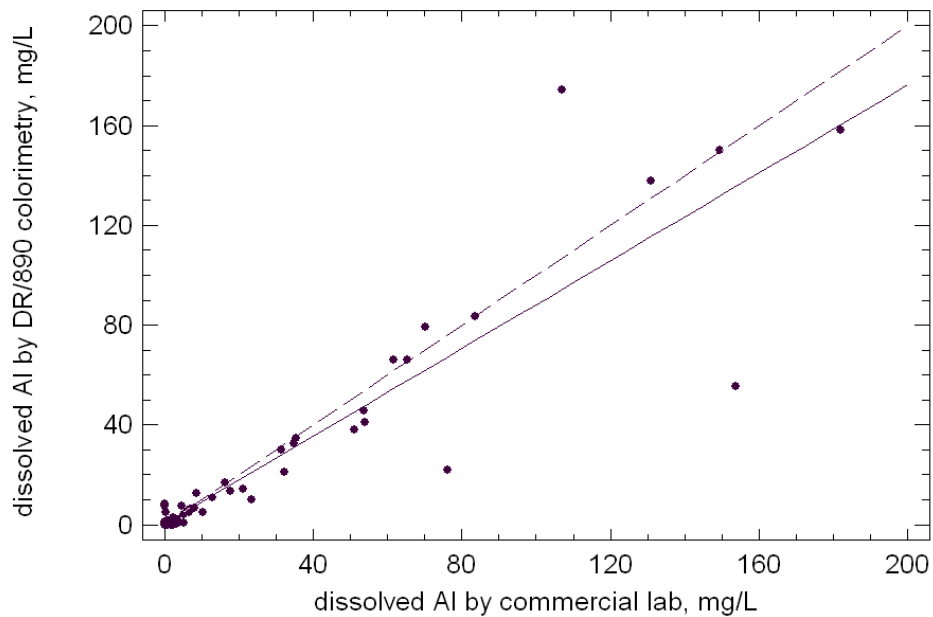


Figure 6. Scatter plot of dissolved Al with regression line (solid) and line of perfect correlation (dashed), N=66.

Manganese results were somewhat similar to the Al test comparison. The field colorimetric test generally produced lower values than did analysis by the ICP (Fig. 8, $R^2 = 0.85$). Only

results of the comparison of dissolved Mn are presented here because total MN may assume to be equivalent to dissolved Mn at the pH levels typically encountered in Midwestern mine drainage ($\text{pH} < 8$).

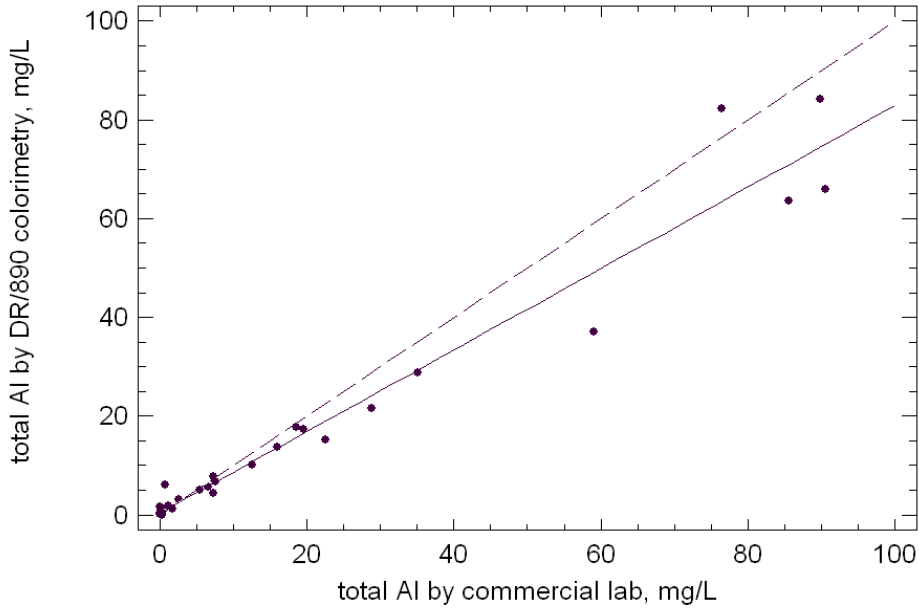


Figure 7. Scatter plot of total Al with regression line (solid) and line of perfect correlation (dashed), $N=30$.

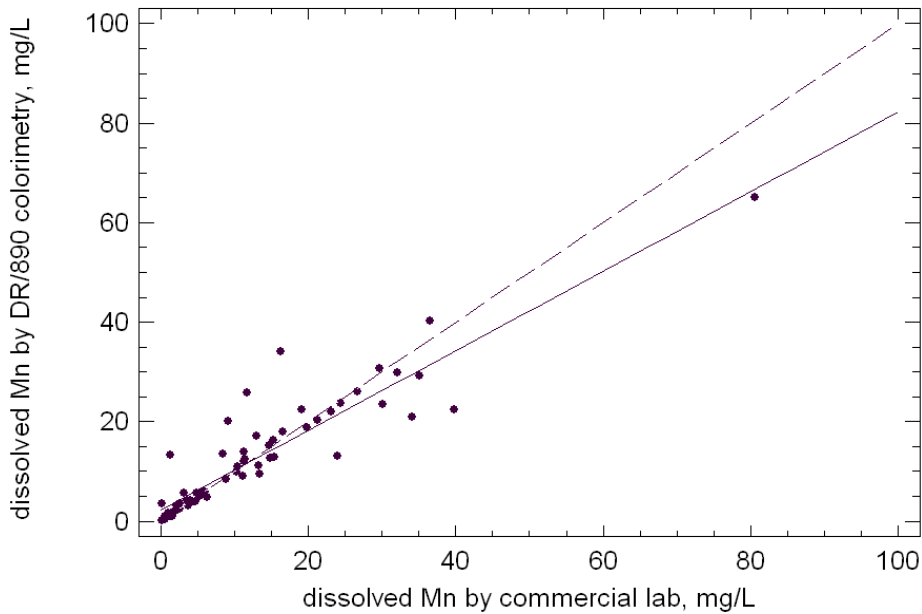


Figure 8. Scatter plot of dissolved Mn with regression line (solid) and line of perfect correlation (dashed), $N=67$.

Sulfate tests differ considerably from the lab results (Fig. 9, $R^2 = 0.62$). Again, dilution errors are suggested. The test measures turbidity and based on our limited assessment does not appear to be very precise.

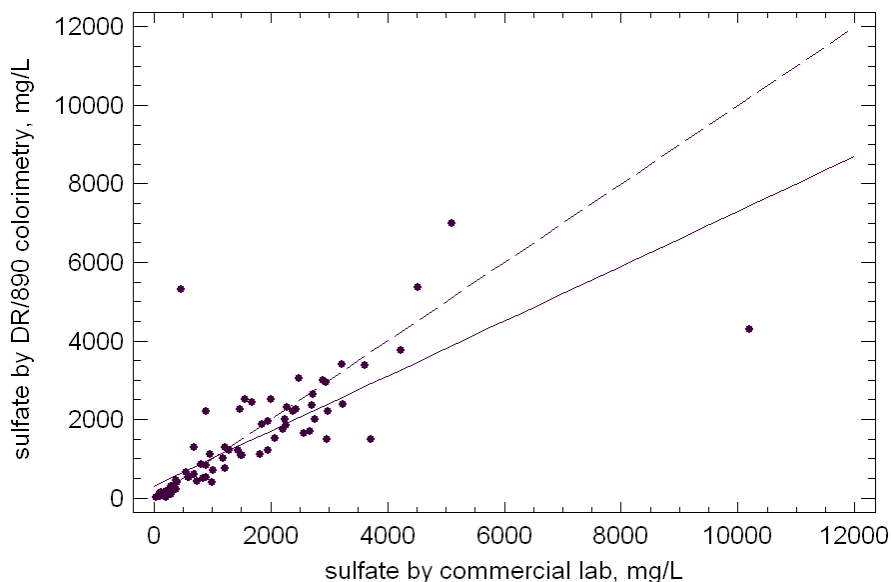


Figure 9. Scatter plot of sulfate with regression line (solid) and line of perfect correlation (dashed), N=87.

Additional evaluation of the SulfaVer® test using test standards is warranted. Nevertheless, this test is a good screening tool for evaluation of AMD sites because the presence of elevated sulfate is a key indicator of mining influence.

Measurements produced by field total alkalinity methods differed considerably from laboratory results (Fig. 10). Field results are considerably higher than laboratory results ($R^2 = 0.91$). This is expected because, over time, hydrolysis of metal solutes (Fe and Al) will produce acidity and lower test values. How long a sample is held before analysis is an important consideration. Standard Methods (1995) recommends a maximum storage time of 24 hours and suggests that alkalinity be determined immediately after sample collection. Consequently, in-the-field-testing for total alkalinity might be preferable to laboratory testing especially if the latter were delayed beyond 24 hours of sample collection.

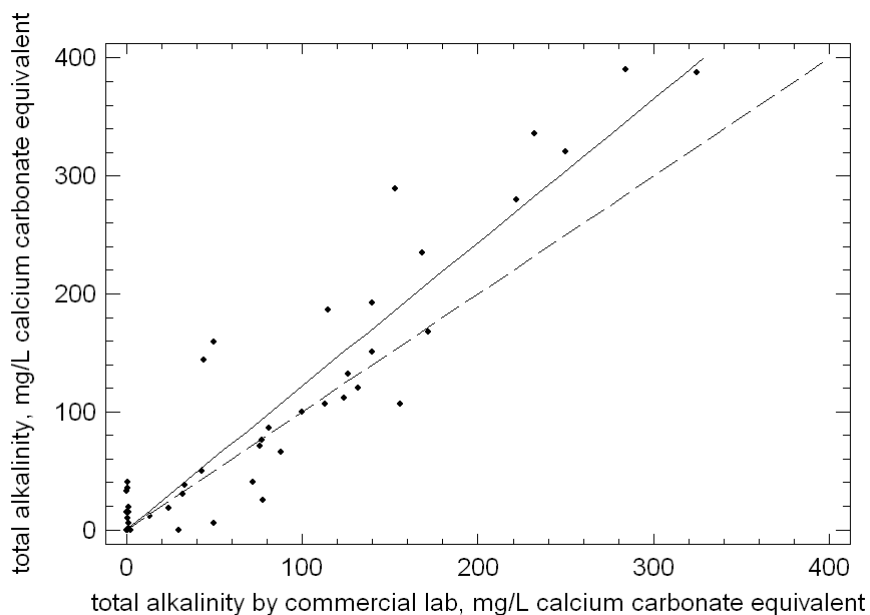


Figure 10. Scatter plot of total alkalinity with regression line (solid) and line of perfect correlation (dashed), N=87.

Concluding Remarks

Colorimetric analysis of dissolved metals - Fe^{2+} iron, total iron ($\text{Fe}^{2+} + \text{Fe}^{3+}$), Mn, and Al — generally gave results comparable to those from analysis by ICP. However, only the iron tests would be suitable for mine drainage treatment system design. This study indicated that field colorimetric tests for total and Fe^{2+} iron can provide a precise and accurate analysis of these key mine drainage parameters. In addition, field colorimetry is a simple, low-cost field method for analysis of the mine drainage parameters Al and Mn. Note however that colorimetric tests for Al and Mn may give values somewhat lower than ICP tests. Colorimetric analysis of sulfate produced results considerably different from more precise and accurate laboratory gravimetric methods or ion chromatography. However, use of the SulfaVer[®] turbidity method run on a portable colorimeter is suggested as a screening tool. Careful dilution is the key to accurate field analysis of AMD with high contaminant levels. Routine verification of test results by using a duplicate sample as well as an additional evaluation using test standards is recommended.

Field measurement of total alkalinity has value. This is especially true for those situations where the drainage is known or suspected to contain high metals and alkalinity, a condition that may be encountered in the discharge from alkalinity-enhancing components of active and passive

treatment systems. Field alkalinity titration is also more generally recommended when it would be unlikely that a laboratory could perform the titration within 24 hours of sample collection.

No cost comparisons between the field and laboratory test methods were made for this study. However, reagent costs for all field tests described in this study are less than \$1.00 per test. This compares to between \$5.00 and \$30.00 (US) per test expected for commercial laboratory analysis. However, for cases where the need is to support regulatory compliance and research efforts—situations where accuracy and precision are critical—there is no substitute for laboratory methods. Note however that the field determination of Fe²⁺ iron is the accepted standard practice, and, as a practical matter in most situations, this would also be true for total alkalinity.

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