APPENDIX B

RESPONSE TO PEER REVIEW COMMENTS ON DRAFT KINETIC TEST PROCEDURE

* Note: The version of the test method used for peer review preceded the Final Draft version;. Comments from this peer review were considered and incorporated in revisions to the draft method where appropriate.

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RESPONSE TO PEER REVIEW COMMENTS ON DRAFT KINETIC TEST PROCEDURE

In late January 2003, the draft Kinetic Test Procedure was sent to six reviewers identified by the project team as experts in the fields of either kinetic test procedures, the mining industry, or geochemisty.

REVIEWERS

Arthur W. Rose, P.G.	Penn State University
Kim Lapakko	MN Department of Natural Resources
WW. White, P.G.	U.S. Bureau of Land Management
Robert Kleinman	Department of Energy
Terry Brown	Western Research Institute
Gwen Geidel	University of S. Carolina (review comments not yet received)

To date, comments have been received from 5 of the six reviewers and are summarized below. In addition to asking reviewers to provide general comments, the Project Team asked that reviewers consider and address the following specific aspects of the draft method:

1) Document's Scientific Merit: Reviewers are asked to comment on the scientific soundness of the draft method.
   a. The method contains procedures for assembling samples based on particle size distribution, including removal of small particle-size portions. These procedures are intended to provide results that can be interpreted against a standard, and to eliminate or reduce channeling and exposure variability. Do you have any comments related to this aspect of the method?
   b. Are the parameters for water (alkalinity, acidity, dissolved metals (iron, manganese, aluminum, magnesium, calcium), sulfate and conductivity) and soil characteristics (percent sulfur, neutralization potential, and particle size distribution) sufficient to provide useful information regarding mine drainage quality? Are any of these parameters unnecessary, or are there additional parameters that should be tested?
   c. Do the weathering procedures described in the method provide a useful standardized assessment of sample exposure?
   d. Section 10 includes information regarding the method performance (relative percent difference, RPD, between duplicate samples) that can be expected by laboratories using duplicate samples. This information also is intended to provide quality control specifications that can be applied for verification of data. Do you believe that the RPD between duplicate samples exposed to identical weathering procedures is sufficient for evaluating method performance. Can you recommend any additional or alternative cost effective and feasible quality control procedures?

2) Document Quality and Clarity: Peer reviewers are asked to critique the document for its clarity given its potential scientific and technical applications. More specifically, reviewers are asked to address the following questions:
   a. Are sufficient details provided for setting up and implementing the method procedures?
   b. Are data equations presented in a technically clear and appropriate manner?
   c. Does the method accurately present the equations necessary to obtain useable data results?

3) Do you believe that this draft method has the potential to serve as an effective and standardized tool in predicting the quality of mine drainage? Do you have recommendations that may enhance method procedures towards obtaining this goal?

4) Do you believe that the draft method has sufficient detail to allow its application in laboratories that typically perform mine drainage analyses? Do you believe that the detail provided allows sufficient

flexibility for method users to adjust for cost and laboratory conditions, while still meeting the method performance criteria?

5) What, in your opinion, are the most influential variables in creating the results that are obtained using the weathering procedures in this draft method?

1. Scientific Merit:

a. The method contains procedures for assembling samples based on particle size distribution, including removal of small particle-size portions. These procedures are intended to provide results that can be interpreted against a standard, and to eliminate or reduce channeling and exposure variability. Do you have any comments related to this aspect of the method?

1a.1	**Comment:** The removal of the small fraction could and probably will significantly affect the acid forming potential of some materials. For example, I have studied materials containing acid forming materials almost entirely in the small particle-size fractions. How does this method account for that? Acid-Base Accounting? ***Response***: *We agree and have extensively considered the fact that removal of the small fraction particle size will affect the acid forming potential of certain rock material. To perform laboratory-scale kinetic leaching evaluations of these materials, however, it is not possible to completely represent field conditions. For example, rocks are crushed in order to fit rock samples into the leaching columns, resulting in an increase in the percentage of fine particles. In addition, method development activities demonstrated numerous problems caused by fine particles in terms of gas/water introduction and leachate collection from the columns over time. To address these practical concerns and provide a standardized procedure, the method specifies a particle size distribution for all samples. Text has been added (Section 8.1.3 of revised method) to acknowledge that "Once samples have been collected and crushed, sample particle size distribution that occurred in the field is lost. This distribution is intended to provide standardized conditions and collection of leachate."*
1a.2	**Comment:** Section 3.1.1 What column size is used for particles <0.5 cm? If particles are crushed to <3/8", why not just specify this size? Or give a minimum typical size based on 3/8" material? If the material is smaller, does it make any difference if the column is this large? Do you need to mix units here (cm vs. in)? ***Response:*** *This section also states that "For smaller particles, a factor greater than four should be used. Scaling factors that consider the ratio of column dimensions and particle size are presented in Murr et al. 1977." For clarification, the following sentence has been added: "This method specifies a maximum sample particle size of 3/8-inch (see Table 2 in Section 8.13) and uses 2-inch diameter columns."*
1a.3	**Comment:** Section 7.1.5--last sentence seems to give guidance and doesn't. We know that small particles might have a significant impact--telling the user to further evaluate it without giving guidance on how to do it is not fair--the method created the problem and has to resolve it. ***Response:*** *This section has been removed from the method. Section 8.1.2 (Section 7.1.2 of the method reviewed by the commenter) covers this concern, stating that "Samples should be analyzed for neutralization potential and percent total sulfur. Methods for analysis of these parameters are included in Table q. If the overall change in particle size, NP, percent sulfur, or other parameters will be determined, there analyses also may be performed on the sample after the last leachate has been collected and the sample removed from the column." Please also see response Comment 1a.1.*
1a.4	**Comment:** Section 7.2 Why are fines eliminated from testing? This fraction of the sample will generally have the highest specific surface area and the largest extent of iron sulfide and carbonate mineral liberation. Consequently, this fraction may have the largest impact on drainage quality and should be subjected to testing. Alternatively, the rationale for excluding this fraction must be clearly stated. ***Response:*** *See response to Comment 1a.1.*

1a.5	**Comment:** Part 7.1.5 – I am not sure I understand the basis for this determination. The leaching method is needed because acid-base accounting doesn't provide results that will describe results in the field. Yet, the evaluation of the fine fraction (the most reactive component) should be evaluated using acid-base accounting before consideration for additional evaluation. In addition, the products from the fine fraction may interact with the products from the coarse fraction. If the fine fraction is removed from the system, how do you account for any interactions? ***Response:*** *See response to Comments 1a.1 and 1a.3.*
1a.6	**Comment:** Section 6.1.3 I am skeptical of this reconstruction step. If the sample crushes easily and most pyrite is in the fines, then the test results may be much lower than in nature. I think you have to put the whole sample into the column, in its natural particle sizes. Otherwise, the result can be markedly different from nature. ***Response:*** *See response to Comment 1a.1.*
1a.7	**Comment:** Section 6.1.5 The fines are not added to the column? Again, I don't like this. ***Response:*** *See response to Comment 1a.1.*
1a.8	**Comment:** Part 7.1.4 – The exclusion of the < 35 mesh size fraction from the column is a concern. Both potential acidity and neutralization potential associated with the fine size fractions are eliminated. Since the smaller size fractions can be considered more reactive (more soluble, etc.), the removal of this material from leaching column may impact the resulting leachate chemistry. In addition, the interaction of the large and small size fractions will not be evaluated by the weathering process. ***Response****: See response to Comment 1a.1.*
1a.9	**Comment:** In terms of technical merit, I am particularly unhappy with the procedure of removing the fines, and making an arbitrary mix of the coarser fractions. This has got to result in acidity, etc. that are not representative of the field material, in some cases. I can see that doing a size analysis can allow you to estimate an approximate surface area, and that you might have some problems with clogging by fines, but the procedure seems very artificial and perhaps misleading in some of the most critical cases, where lots of fines are present in nature, or the rock is very inhomogenous physically. ***Response:*** *See response to Comments 1a.1 and 1a.3.*

b. Are the parameters for water (alkalinity, acidity, dissolved metals (iron, manganese, aluminum, magnesium, calcium), sulfate and conductivity) and soil characteristics (percent sulfur, neutralization potential, and particle size distribution) sufficient to provide useful information regarding mine drainage quality? Are any of these parameters unnecessary, or are there additional parameters that should be tested?

1b.1	**Comment:** Section 6.3.1.2 If the leachate is filtered, CO_2 will be lost. The procedure in this section needs more work. Probably the leachate should be stirred gently, its pH and conductivity measured, then part put into a cooled container, and the rest filtered. Preferably the alkalinity should be measured immediately. ***Response:*** *The following note is included in the method (at Section 8.5, Leachate Collection): "The procedure used to collect leachate must minimize carbon dioxide degassing (e.g., insert the drainage tube into the bottom of the sample collection container throughout collection; seal the container immediately following collection; refrigerate the sample if analysis is not performed immediately; keep sample container submerged in ice if collection drainage is slow)." Instructions to filter the leachate to remove fine particles have been removed. Instead, the leachate is analyzed immediately for determination of pH and conductivity, and additional aliquots are prepared for further analysis. Aliquots are filtered only if they are to be analyzed for dissolved parameters (method Section 8.5.1.2).*

1b.2	**Comment:** Table 2. What does "Alkalinity 2030" mean? The procedures list seems to end with an imcomplete method (I-). ***Response:*** *Thank you. "2030" has been removed, and replaced with "(to pH 4.5)". Additionally, the USGS Method cited has been revised to I-2030.*
1b.3	**Comment:** Section 2.0 Production of metals was mentioned in section 1, paragraph 2, sentence 3. Consequently, methods for determining solid-phase metal concentrations should be included. ***Response:*** *Table 1 provides a list of methods that can be used to determine the neutralization potential and total sulfur content of soil samples, both of which are to be determined during initial sample characterization (see method Section 8.1.2). Analytical procedures for determining metal concentrations in soil samples are outside the scope of this method.*
1b.4	**Comment:** Section 3.0 The purpose of the method is to characterize water quality. What aspects of water quality are of interest, pH, acidity, metals, other? ***Response:*** *Recommended target analytes, along with appropriate corresponding analytical methods, are listed in Table 3 of the method (Analytes and Appropriate Methods). Note, this is Table 2 in the method reviewed by the commenter. Additionally, Section 8.5.2 (Section 7.4.2 of reviewed method) notes that leachate is analyzed immediately upon collection for specific conductance, alkalinity, and pH.*
1b.5	**Comment:** Section 7.3.1 "...until the conductivity of the flush water stabilizes..." Guidance should be supplied as to what constitutes stabilization. For example "...until the conductivity of the flush water changes by no more than XX ΦS..." ***Response:*** *This sentence has been revised to: "Continue to add, drain, and analyze reagent water in this manner until the conductivity of the water stabilizes (relative standard deviation between conductivity measurements ≤ 20%)."*
1b.6	**Comment:** Section 7.4.1.2 In concert with the following comment, is there certainty that solids in the leachate sample do not include precipitates? Although I lack experience with the method, given the large size of particles in the column it seems unlikely that they would be lost during leachate collection. ***Response:*** *Appendices addressing carbonate dissolution, pyrite oxidation, and mineral solubility of calcite and gypsum have been added to the method.*
1b.7	**Comment:** Section 7.4.1.3 What is the degree of certainty that this method of collection eliminates precipitation of calcite? That is, has this aspect been evaluated empirically? ***Response:*** *See response to Comment 1b.6.*
1b.8	**Comment:** Table 3 Why is pH excluded from results? Is this controlled by the gas mixture rather than mineral dissolution reactions? ***Response:*** *pH quality control results were determined during the multi-laboratory study and are provided in Table 4 of the September 2008 method revision.*
1b.9	**Comment:** The parameters for water and soil characteristics appear to be sufficient. However, HCO_3^{-1} might be included since this constituent maybe important in certain situations. ***Response:*** *Alkalinity accounts for the bicarbonate anions in a sample. Alkalinity can be converted to bicarbonate by dividing alkalinity by 0.8202 (see Study and Interpretation of the Chemical Characteristics of Natural Water, US Geol. Survey Water-Supply Paper 2254, p. 57)*

1b.10	**Comment:** Section 3.2 Can gas sorption determinations of surface area be conducted on particle sizes this large? ***Response:*** *Yes. These determinations were performed by the Materials Research Laboratory at Penn State University during development and validation of this method.*
1b.11	**Comment:** Section 3.2 The total surface area determination described applies to the entire solid matrix. With respect to pyrite and carbonate mineral reactions, the surface areas of these minerals is of importance. It is unlikely the total surface area will accurately reflect the surface areas of these minerals. Consequently, the merits of conducting gas sorption analysis on the solids matrix for the purpose of normalizing rates for surface area are unclear. ***Response:*** *Gas sorption analysis is optional.*
1b.12	**Comment:** Section 3.2 Determining the extent of carbonate and iron sulfide mineral liberation may be more appropriate for normalizing dissolution rates of these minerals. ***Response:*** *Appendices have been added to the method to provide example calculations for determining carbonate dissolution rates, pyrite oxidation rates, and mineral solubility of calcite and gypsum.*
1b.13	**Comment:** Section 6.1 Should lithologic logs be retained to document the representativeness of samples? ***Response:*** *Yes, lithologic logs are routinely retained by regulatory agencies and permittees as part of regulatory requirements.*
1b.14	**Comment:** Section 9.4.2 As mentioned previously, expression of results in terms of total surface area may be misleading because this does not necessarily reflect the available mineral surface areas that contribute to reactions of interest. ***Response:*** *This equation has been removed from the method. Instead, results are calculated in units of mass weathered per week and mass produced per sample weight. Method appendices also have been added to provide example calculations for determining carbonate dissolution rates, pyrite oxidation rates, and mineral solubility of calcite and gypsum.*
1b.15	**Comment:** Part 7.1.2 – Total S may be an appropriate parameter for calculating potential acidity for eastern mines. However, many sites/materials contain SO_4^{-2} and SO_4^{-2} minerals in addition to pyrite. In such cases pyrite levels should be determined and used in lieu of total S. ***Response:*** *Appendices have been added to the method to provide example calculations for determining carbonate dissolution rates, pyrite oxidation rates, and mineral solubility of calcite and gypsum.*
1b.16	**Comment:** The use of total S levels to calculate acid potential may work for some mines but many of the mines in the west and some in the east contain significant SO_4^{-2}'s, SO_4^{-2} minerals etc. Pyrite determinations are needed to calculate potential acidity at some mine sites/materials. ***Response:*** *See response to Comment 1b.15.*
1b.17	**Comment:** Section 9.1 Will NP be presented in units of mg/kg or percent, or as more commonly presented in units of parts per thousand? ***Response:*** *The units reported for NP of the rock sample will depend on the reporting requirements specified by the data user. A footnote also has been added to provide a reference for procedures that can be used to determine NP. (See method Section 2.0.)*

1b.18	**Comment:** Section 9.3 May also want to record cumulative mass release for critical analytes ***Response:*** *The equations reviewed by the commenter have been removed from the method. Instead, results are calculated in units of mass weathered per week and mass produced per sample weight.*
1b.19	**Comment:** Part 7.4.2 – Sample solution for SO_4^{-2} determinations should be filtered using a 0.45 :m filter. ***Response:*** *This sentence has been revised to state that: "If the leachate will be analyzed for dissolved parameters (SO_4^{-2}, metals), the leachate must be filtered through a 0.45 µm filter prior to analysis." (See method Section 8.5.1.2.)*
1b.20	**Comment:** Section 7.1.2 The sample characterization is extremely limited, and additional analyses should be considered. To conduct a 16-week test on a sample on which so little is known limits the value of the results. It is possible that there is substantial data on specific strata to counter this deficiency to some degree. If so, the rationale for such limited solid-phase characterization should be stated. For example, if metal release is of interest, determination of metal concentrations in the initial sample would be beneficial. Whole rock and trace metal chemistry analyses can be conducted for less than $100 per sample. The mineral phases in which carbonate occurs may also prove to be valuable. ***Response:*** *A note has been added to this section, stating that additional parameters may be measured if required or requested by the data user.*
1b.21	**Comment:** All the test parameters are needed (alkalinity, acidity, pH, conductivity, Fe, Mn, Al, Ca, Mg, SO4). Also should specify the temperature and the amounts of sample and solution. ***Response:*** *We agree that the test parameters listed (see method Table 3) are needed. Specific requirements regarding sample preservation (e.g., temperature) and volumes are noted in the analytical method selected for measuring each parameter, and are not within the scope of this method. Section 9.1 (Section 8.1 of the method reviewed) also states that "All quality control measures described in the reference analytical methods for leachate analysis (Table 3) and sample characterization (Table 1) should be used.*
1b.22	**Comment:** Section 2.2 You need to clearly present this as two alternative methods for surface area: 1) Calculation from particle size, 2) Gas adsorption. I don't see any description in the procedure for getting the surface area from the size distribution. ***Response:*** *A footnote has been added to this section, to provide two references to procedures that can be used to determine particle surface area using gas adsorption. (See method Section 3.2.)*
1b.23	**Comment:** Section 7.1.2 What grain size sample is submitted for NP and S determinations? This seems to require a grinding step. ***Response:*** *Grain size specifications are provided in the methods listed for determination of these parameters (see Method Table 1). Typically, a split of the sample is ground to -60 mesh.*

c. Do the weathering procedures described in the method provide a useful standardized assessment of sample exposure?

1c.1	**Comment:** Section 2.0 Why is the 8 to 16 week period selected? Is this duration adequate to empirically determine if coal mine wastes will produce acid? ***Response:*** *This sentence has been revised to state that the sample are exposed to simulated weathering conditions over a period of at least 12 weeks. During method development and validation in several laboratories, soils, and studies, it was determined that analyte production typically peaked at approximately 10 –12 weeks.*
1c.2	Section 6.3 Appropriate ranges should be provided for "…dry, and cool conditions." ***Response****: The last sentence of this Section states that "Sample shipment, storage, and preservation procedures are described in ASTM D5079."*
1c.3	**Comment:** The weathering procedures described may provide a useful standardization assessment for a single sample exposure if the weathering procedure is written specifying specific procedures. Seemingly, simple changes in the procedure will result in significant differences in results. After spending three (3) years with OSM attempting to develop QA/QC programs for laboratories in the Western United States, I found that laboratory evaluations of pH, EC etc. from a saturated paste or 1:1 mixture were often different likely as a result of equilibration time (12 to 24 hours), extraction method (differences in vacuum, etc.); all seemingly insignificant differences in method. ***Response:*** *We agree. The quality control specifications provided in Section 11.0 (Method Performance) of the method were generated from pooled data generated in seven laboratories performing the method on replicate soil samples. These specifications reflect the variability that should be expected across and within laboratories.*
1c.4	**Comment:** Part 9.4.2 – Surface area determinations may be difficult to duplicate. QA/QC would be a large problem between laboratories. I am not sure how important it is to show the data on a surface area basis. ***Response:*** *Surface area determinations are optional.*
1c.5	**Comment:** Part 7.2.2 – Any type of filling and packing (tapping) will cause differential compaction and particle size segregation. ***Response:*** *We agree. This section (Section 8.2.2 in September 2008 revision) has been modified to state "Using a wide-bore or powder funnel, add approximately 2,000 grams of the reconstructed sample to the column, being careful to ensure uniform distribution with little to no packing."*
1c.6	**Comment:** Sections 7.3.2 and 7.3.3.2 Does the column sit unsaturated for 7 days or 6 days? Logically, it is the latter. ***Response:*** *This section (Section 8.4.2 in September 2008 revision) has been modified to clarify that the column is allowed to sit for a period of 6 days during the humidified air cycle.*

1c.7	**Comment:** Section 3.2 Not clear. It appears that the gas mixture is used in 2 ways: 1) bubbled into reagent water reservoir, and 2) bubbled into column. Is the 10% CO_2 defined by the gas mixer or by the outflow gas composition? If the flow rate is appreciable, I would think that inflow composition would be OK, since the amt. Of water is finite. Does one need a gas analyzer to measure the outflow gas composition? Specifications on instrument and allowable error in percentage? ***Response:*** *Section 4.2.1.1 (Section 5.2.1.1 of September 2008 revision) provides instructions for monitoring gas flow, including daily monitoring using flow meter, gas meters, or tube indicators) to ensure that the CO_2 in the outflow is at least 10%. A requirement to use a portable CO_2 meter, capable of measuring CO_2 to 10% (within $\pm$ 0.5%), to take daily readings of the CO_2 released from each column also has been added (see method Section 8.3.2.2).*
1c.8	**Comment:** Section 4.2.1.2 Tubing clamps seem pretty crude for controlling flow. What flow rate is desired? Why not a flow meter (bubbler)? ***Response:*** *This section (Section 5.1.4 of September 2008 revision) has been modified to include details regarding tubing and clamps that are recommended for controlling flow. Information also has been provided regarding recommended flow meters (rotometers) capable of controlling flow at approximately 1 L/minute. (See method Section 5.2.1.3.)*
1c.9	**Comment:** Section 4.1.2 It is not clear that glass beads of the size indicated would produce "uniform introduction of both water and gases." For example, the criterion stated could result in glass beads of 1-inch diameter in a 2-inch diameter column. I have no data or literature to cite that would indicate this would not produce uniform flow, but intuition suggests that it will be of little benefit. Please provide a citation regarding this criterion. ***Response:*** *Specifications for plastic (polypropylene) beads of ½-inch diameter or HDPE 5/16-inch diameter, have been included. (See method Section 5.1.5.2.)*
1c.10	**Comment:** Glass wool is suggested as a possible filter media. USBM initially used glass wool as filter media in its first humidity-cell tests of metal-mine waste rock. Unfortunately, after 25 weeks of testing it was discovered that glass wool contributed alkalinity to the effluent (an increase of at least 2 pH units). After considerable leach testing of various filter media material, we found that polypropylene felt was non-reactive and provided a very satisfactory filter material. The polypropylene product we used was purchased from National Filter Media Corp. (NFMC) and the product serial number is S/226 075 020 (my NFMC contact in Salt Lake City is Mark Rydalch 2 801-363-6736). Also see my comments on Section 8.4. ***Response:*** *Thank you. In response to this and other comments, a note has been added to Section 5 of the method to state that "Glass wool has been shown to neutralize acid and elevate pH in experimental work at both the Minnesota Department of Natural Resources and the US Bureau of Mines. It should not be used in this type of testing unless it is tested and shown to be unreactive. Aquarium filter media is provided as an alternative liner material.*
1c.11	**Comment:** Glass wool has been shown to neutralize acid and elevate pH in experimental work at both the Minnesota Department of Natural Resources and the US Bureau of Mines. It should not be used in this type of testing (ASTM 2000, note 5, p. 259) unless it is tested and shown to be unreactive. ***Response:*** *See response to Comment 1c.10.*

1c.12	**Comment:** Section 4.2 It appears that maintaining the appropriate gas mixture may be important for obtaining reproducible results. Values presented in Table 3 indicate that RPD for air-CO_2 mixtures is always greater than that for air-only. One very plausible explanation for this is that the air-CO_2 mixture is not effectively controlled. ***Response:*** *We agree that maintenance of gas flow and mixture components is important for obtaining reproducible results. The method performance specifications provided in Section 11.0 of the September 2008 revision are based on data generated in seven laboratories, and consider the variability that can occur within and between laboratories. Section 4.2.1.1 (Section 5.2.1.1 of September 2008 revision) provides instructions for monitoring gas flow, including daily monitoring using flow meter, gas meters, or tube indicators) to ensure that the CO_2 in the outflow is at least 10%. A requirement to use a portable CO_2 meter, capable of measuring CO_2 to 10% (within $\pm$0.5%), to take daily readings of the CO_2 released from each column also has been added (see method Section 8.3.2.2).*
1c.13	**Comment:** If variation in the air-CO_2 mixture affects results, the use of a certified gas mixture appears to be a reasonable approach to eliminating this problem. I am not familiar with the other methods of maintaining a constant ratio of air to CO_2. If their use is to be permitted, testing of the control method (if not done already) would be prudent. ***Response:*** *See response to Comment 1c.12.*
1c.14	**Comment:** Section 4.2.1.2 The MN DNR experienced problems maintaining constant flow with tubing clamps. The ability of these clamps to maintain adequately constant flow for this method should be checked if this has not been done already. ***Response:*** *See response to Comment 1c.12.*
1c.15	**Comment:** The application of humidified air to the ASTM 5744 cell, during the early stages of development, at times resulted in addition of water to the cell if flow rates were excessive. Establishing a range of acceptable gas flow rates into the column would help control this variability. ***Response:*** *See response to Comment 1c.12.*
1c.16	**Comment:** Section 5.0 The gas mixture described in 5.1.1 and 5.1.2 is different than the alternative described in 5.1.3 (approximate O_2:CO_2:N_2 ratios of 2:1:7 vs 1:1:8). Has the influence of this variation been examined? Is there a commercially available mixture that more closely approximates that of 90% air-10%CO_2? Given the variability in results for the air-CO_2 mixture, providing a consistent mixture (applied at a consistent flow to the column) should be considered. ***Response:*** *The primary concern is to provide 10% CO_2 to mimic field conditions. Both of the gas mixtures cited will provide 10% CO_2. Additionally, either mixture will provide sufficient oxygen for oxidation of pyrite.*
1c.17	**Comment:** Part 7.3.1 – Tapping the column and the use of the wire to adjust saturation will result in differential compaction and particle segregation. I don't think either is needed. ***Response:*** *We agree. Both have been removed from the method. (Also see response to Comment 1c.5.)*

1c.18	**Comment:** Sections 7.2 and 7.3: I am concerned that no specific sample mass or leach volume has been prescribed. I recognize that these would change depending upon the size of column selected (i.e., 2, 4, or 6-inch ID) and target sample masses could be identified/prescribed for each size. If the objective is to completely flush reaction products from the column during each weekly leach so that mass release rates can be determined (especially from replicates of the same lithology having the same percent S and Ca + Mg carbonates) then a fixed mass and volume would prevent adding additional variables into the test. ***Response:*** *The method has been revised to specify a column inner diameter of 2 inches, column height of 2.5 feet, and a sample weight of approximately 1800 – 2000 grams. Instructions to measure and record the volume of water added to and collected from each column, with each weekly saturation/drainage cycle, also have been added. (See method Section 8.5.1.1.)*
1c.19	**Comment:** Section 7.3.2 The extent of water retained may affect dissolution reactions. Can the column be weighed before and after the rinse to determine water retention? Retention can be approximated by the difference of water added and water removed. However, this does not account for potential water addition during the humidified air cycle. Such addition was observed during early developmental stages of the ASTM 5744 Modified Humidity Cell test (also mentioned in comments under section 4.2.1.2; see also 7.4.1.1). If this occurs in the column method, it will affect the accuracy of the approximation mentioned in the third sentence of this comment. ***Response:*** *We agree and acknowledge that it water will be retained during each saturation/leachate collection cycle. Instructions have been added to measure and record the volume of water with each weekly cycle (see method Section 8.5.1.1). An example form for use in recording this and other weekly monitoring information also has been added (see Section 13.0).*
1c.20	**Comment:** Section 7.4.1 The first sentence indicates "...the total volume of water/leacheate is drainage from the column..." To what does "total volume" refer? The amount added? All water in the column? ***Response:*** *The word "total" has been deleted from this sentence. Also see response to Comment 1c.19.*
1c.21	**Comment:** Section 7.4.1: I question that it is possible to recover the total leach volume from the rock column. As much as 150 mL of a 500 mL leach was typically retained in USBM humidity cell tests after each leach cycle. We did notice that recovered leach volumes began to increase after multiple leach cycles as the retained interstitial water volume stabilized over time. However, we never achieved 100% recovery of the 500 mL leach volume (I recall something like 400+mL at best). ***Response:*** *See response to Comment 1c.19.*
1c.22	**Comment:** Section 7.4.1.1 Recommend recording total leach volume minus recovered leach volume. This will help you document volume of interstitial water remaining in the sample at the beginning of the humidified air cycle. Also recommend weighing column at the end of the humidified air cycle. (In the early stages of method development, water addition during the humdified air cycle at times was so great that the bed became saturated and water ponded at the top. As the technique and technicians improved, the water additions during the humidified air cycle were generally less than 5 mL.) ***Response:*** *Instructions to measure and record the volume of water added and leach volume removed with each weekly cycle has been added to the method. (See response to Comment 1c.19.) Although weighing columns before and after leachate collection would provide useful information, it would be impractical and perhaps problematic.*

1c.23	**Comment:** Table 3 As mentioned previously, the greater variability in results using the air-CO_2 mixture may indicate problems in controlling this mixture. The variability of this mixture and its effect on results should be examined, if this has not yet been done. ***Response:*** *The effect of CO_2 was evaluated in two laboratories during initial method development. Results are provided in method Reference 12.21. See response to 1c.12 regarding method requirements for measurement of the CO_2 component in gas mixtures.*
1c.24	**Comment:** The procedure does not indicate how many weeks the test should extend for, and how the results for multi-week tests should be reported. It is common that the first few weeks are much different from later weeks. At least 8 weeks needed? Perhaps the data for the week of maximum acidity and an average of the last few weeks should be reported. ***Response:*** *The method summary has been revised to state that the sample are exposed to simulated weathering conditions over a period of at least 12 weeks. During method development and validation in several laboratories, soils, and studies, it was determined that analyte production typically peaked at approximately 1 –12 weeks. See response to Comment 1c.1.*
1c.25	**Comment:** Section 4.1 Note that using clear columns and storing columns in the light may enhance biological growth, for example, algae. For a 16-week test, this is unlikely to be extensive. For tests of longer duration, algal growth may be of concern. ***Response:*** *Method developers considered this concern, but thought the ability to observe sample conditions throughout the weathering and leaching procedures was more important. See method Section 5.1.*
1c.26	**Comment:** Section 3.1.2 Can introduced gas pass up thru the glass wool, geotextile (size?), etc? I would expect it to be trapped once the system is saturated, and result in a gas bubble underneath that wants to push up. Some of your later discussion implies that this happens. Why not place the gas inflow above the glass wool etc.? ***Response****: Some of the laboratories involved in the method development and validation studies experienced and resolved this problem by controlling the flow of air into the column. As a result, the method text has been revised to clarify that once the column has been saturated, the gas mixture is introduced through the gas inlet port at the bottom of the column until a slight positive pressure is reached (i.e., a small outflow is produced through the air vent in the top of the column). Gas flow can be controlled and maintained at approximately 1.0 L.minute using a combined flow regulator and meter (e.g., Omega FL-3817-V Series Rotometer or equivalent." See method Section 8.4.3.1.*
1c.27	**Comment:** Section 7.3.3.1: It strikes me that it would be difficult to prevent displacing some of the leach water with the gas during its introduction into the column, and consequently having some of it escape from the air exit port (this fluid loss would mess up your mass release calculations). Using the gas-saturated reagent water leach alternative mentioned in the note following this section seems much more simple and trouble free. Suggest replacing the verbiage in Section 7.3.3.1 with the note information. ***Response****: The alternative procedure that was included in the method reviewed by the commenter has been deleted, and the method has been revised to provide a standardized procedure that involves cycles of 24-hour periods of saturation with reagent water, followed by 6-day exposures to a humidified gas mixture. Also, see response to Comment 1c.26.*

1c.28	**Comment:** Section 7.3.3.1 It seems there is a potential for water to be forced through the air/gas vent if gas is introduced to the column when it is filled with water. Given this possibility, it seems that saturating reagent water with the gas mixture prior to flooding the column should be prescribed. Furthermore, sections 4.2 and 4.2.2 leave the impression that gas will be introduced into the reagent water reservoir. Perhaps experience indicates gas can be added to the water-filled column without water loss and, therefore, either alternative is acceptable. ***Response***: *See response to Comments 1c.26 and 1c.27.*
1c.29	**Comment:** Section 3.3.1 Acidification of the entire sample may provide an alternative for dissolving any $CaCO_3$ precipitated during sample collection. How long does sample collection take? Could $CaCO_3$ precipitate during this period even though flow is introduced to the bottom of the collection vessel and the vessel is covered immediately after sample collection? ***Response:*** *Acidification is not necessary. Alkalinity is measured promptly following sample collection, minimizing effects from $CaCO_3$ precipitation.*

d. Section 10 includes information regarding the method performance (relative percent difference, RPD, between duplicate samples) that can be expected by laboratories using duplicate samples. This information also is intended to provide quality control specifications that can be applied for verification of data. Do you believe that the RPD between duplicate samples exposed to identical weathering procedures is sufficient for evaluating method performance. Can you recommend any additional or alternative cost effective and feasible quality control procedures?

1d.1	**Comment:** Tables 3 and 4. What is a relative percent difference? Why not just percent difference? Or is this percent difference from the mean or some more complicated statistical function? Need to define this more exactly. ***Response:*** *An equation has been added to the method to define relative percent difference. See Equation 1, Section 9.2.3.*
1d.2	**Comment:** Section 8.3 Replicate Samples: Recommend including statement or note emphasizing importance of using same sample mass and leach volume for each replicate to avoid introducing additional variables into the test. ***Response:*** *This section (Section 9.2 of the September 2008 revision) has been modified to clarify that duplicate samples are run using identical sample masses and leaching volumes.*
1d.3	**Comment:** Title of Table 4 (which needs to be renumbered) Highest resulting percent difference (RPD) between duplicate samples during method validation in two laboratories also, i would argue again that what has been done so far has not been validation--testing would better describe it. ***Response:*** *Since receipt of this comment, the method has been validated in a multi-laboratory study involving eight laboratories; the table has been updated to reflect results in seven laboratories that did not submit outlying results. The table has also been renumbered and given the following title: Expected method precision (as RPDs) based on Interlaboratory Study Results.*
1d.4	**Comment:** Part 8.3 – The use of at least triplicate samples rather than duplicate samples is necessary for the method performance determinations. Statistical analysis based on duplicate samples is a stretch. Method performance must be based on triplicate samples at a minimum. If two results differ significantly, what does the statistical result tell you? Not much. You still have no idea about performance. ***Response***: *Duplicate sample are typically used in U.S. EPA and ASTM analytical methods. We agree, however, that the use of triplicate (or four, five….) samples would provide more statistical power. Method users are not limited to use of duplicates.*

1d.5	**Comment:** Part 8.4 – Control Sample – USGS in Denver has developed a hard rock standard sample. It could be done on a regional basis (coal type) with expiration dates. ***Response:*** *The Brush Creek Shale that was used in method development and validation is a control sample that was developed in cooperation with USGS in Denver. Requirements to run a standard material have been removed from the method.*
1d.6	**Comment:** Section 8.2 How will standard reference results be used to "evaluate the validity of field sample results"? ***Response:*** *Use of standard reference materials has been removed from the method. See response to Comment 1d.5.*
1d.7	**Comment:** Section 8.4 Control Sample: Recommend using inert material (e.g., well characterized quartz chips of requisite particle size); this will allow you to check for unexpected contributions from the test apparatus (this is how USBM discovered that glass wool filter media contaminated the humidity-cell effluent with alkalinity and raised pH from 7 to 9+). ***Response:*** *Blanks have been added to the method (see method Section 9.2).*
1d.8	**Comment:** In general, there are difficulties in providing some degree of standardization using performance based criteria. It appears that some standardization (prescription) could be applied that would benefit the reproducibility of results. It appears that additional prescriptive aspects of testing might be provided without placing undue burden on test practitioners. These discussions have probably taken place, and this issue should be addressed again after additional interlaboratory method validation. ***Response:*** *Results and information generated during the interlaboratory method validation study have been used to standardize numerous aspects of the method (see response to Comments 1c.18, 1c.26, and 1c.27, for example). The method's performance-based criteria is consistent with U.S. EPA, ASTM, Environment Canada, and numerous other agencies and organizations approaches to providing standardized methods that allow flexibility for adjusting to costs, product availability, technology improvements, and data user requirements. See http://www.epa.gov/epawaste/hazard/testmethods/pbms.htm for information regarding U.S. EPA's Performance-Based Measurement System.*
1d.9	**Comment:** At this stage of test development it is difficult to assess how effective the "performance-based" criteria will be in providing comparable results within and among testing laboratories. Ultimately this must be assessed in terms of environmental mine waste management decisions made based on test results. For example, drainage pH values predicted by different laboratories may be 2.9 and 3.4. This could be viewed as a large difference. However, if the decision on how the material tested will be handled at the operation is the same, irrespective of the pH difference, then the difference is inconsequential in the practical sense. ***Response****: Laboratory variability, based on results of the interlaboratory method validation study, was assessed and used to establish the method performance criteria that are provided in Table 4 of the method. Also see response to Comment 1d.9.*
1d.10	**Comment:** Section 6.2 How many splits are collected? ***Response:*** *The following sentence has been added: "To demonstrate the accuracy of results, it is recommended that at least two identical homogeneous sample aliquots are prepared from each bulk sample. See method Sections 7.3 and 8.1.3.*
1d.11	**Comment:** Section 10.0 You should not define RPD again and again--you already defined it on the previous page--now just use the acronym, or don't use it at all except in the tables. Last sentence in the text, delete the word "through" ***Response:*** *Thank you. Text has been revised as recommended.*

2. Document Quality and Clarity: Peer reviewers are asked to critique the document for its clarity given its potential scientific and technical applications. More specifically, reviewers are asked to address the following questions:

a. Are sufficient details provided for setting up and implementing the method procedures?

2a.1	**Comment:** I see a lot of features that need to be clarified and tightened up. Has this been read by people from an analytical lab? Some of the lab procedures seem rather crude or potentially unsatisfactory. Also, the authors need to decide if they are specifying a distinct procedure, or just recommending improvements to existing methods. All the qualifications seem to leave a lot of room for individual initiative in the specifics of the method. ***Response:*** *The method has been reviewed by and tested in up to eight individual laboratories; two of these laboratories participated in both the method development and validation studies, and provided significant input regarding method implementation details (e.g., apparatus, materials, reagents, gas mixtures, column preparation, and gas introduction). We are not aware of what existing methods the commenter is referring to in this comment; this method is intended as a stand-alone distinct procedure. However, additional details have been added throughout the method since receipt of reviewers comments and completion of laboratory testing.*
2a.2	**Comment:** Sufficient details are provided to setup and implement the method. However, it is recommended that allowances for manipulating or changing the procedure be limited. ***Response:*** *See response to Comment 1d.9.*
2a.3	**Comment:** Section 4.1.4 Do you really mean natural rubber tubing? I would think plastic tubing would be preferable, such as Tygon. Or is this really a rubber? Does natural mean it comes from rubber trees vs. a chemical plant where most rubber now is made? ***Response:*** *Thick wall, rubber latex tubing is used for tubing that will require clamping. Specifications for this tubing are provided in method Section 5.1.4.2.*
2a.4	**Comment:** Section 4.1.5 It seems like removing the tubing to drain is pretty messy. Why not put in a 3-way stopcock? ***Response:*** *The tubing is disconnected from the water source, but is not removed from the column port. A clamp is used to stop water flow. See method Figure 1.*
2a.5	**Comment:** Section 6.1.1 What do the sieve sizes mean (4,10,16,35,60)? Are these mesh openings/in, or sieve numbers? If the latter, what scale? What size sample should be sieved (lbs)? ***Response:*** *The sieve sizes are listed as "No. 4, 10, 16, etc.", which is standard notation. Table 2 lists these as sieve numbers, and notes that sieves of their equivalent mesh size can be used. The following sentence also has been added to the method: "Approximately 2 kg is needed for each column." See method Section 8.1.1.*
2a.6	**Comment:** Section 7.2.2 Is resolution of the sample weight to 0.05 g necessary? This represents an error of (0.05/1800) = 0.0028%. If weighing to 0.05 g is not a problem, I have nothing against the requirement. However, it is highly unlikely that other measurements in the test will be near this degree of resolution and lesser requirement for weight accuracy could be adopted. ***Response:*** *This sentence has been removed, and a note has been added to state that "The total weight of the sample added to the column must be recorded to the nearest 1.0 gram, for use in results calculations." See text box in method Section 8.2.2.*
2a.7	**Comment:** Section 5.2 Some standards for water purity should be provided, e.g. ASTM 2000. ***Response****: This section has been revised to state that reagent water is prepared by distillation, deionizatin, reverse osmosis, or other technique that removes potential interferences (e.g., metals and organics).*

2a.8	**Comment:** Section 7.1.2 The sample should be dried and weighed following test completion. ***Response:*** *Although we agree that drying and weighing the sample after test completion could provide useful information, it is not considered necessary to produce method results.*
2a.9	**Comment:** Section 7.1.2 Portions of fresh and leached samples should be archived for potential future examination. ***Response:*** *Although we agree that it may be desirable to archive leachate samples for potential future evaluation, it is not considered necessary.*
2a.10	**Comment:** Section 6.1 What is the minimum size of sample (lbs.)? ***Response:*** *The method has been revised to specify a column inner diameter of 2 inches, column height of 2.5 feet, and a sample weight of approximately 1800 – 2000 grams.*

b. Are data equations presented in a technically clear and appropriate manner?

2b.1	**Comment:** Data equations provided are clear. However, there is no advantage in showing acid generation on a particle surface area basis. Surface area is not an easy and/or precise determination. ***Response:*** *This equation has been removed from the method. Instead, results are calculated in units of mass weathered per week and mass produced per sample weight.*
2b.2	**Comment:** Sections 9.3 and 9.4.2--equations have words and unitsallruntogetherlike this, insert spaces. ***Response:*** *Thank you. Equations have been clarified.*

c. Does the method accurately present the equations necessary to obtain useable data results?

2c.1	**Comment:** The equations provide useable data, however I see no advantage in showing the data on a surface area basis. ***Response:*** *This equation has been removed from the method. Instead, results are calculated in units of mass weathered per week and mass produced per sample weight.*
2c.2	**Comment:** Section 8.3.2 This equation is said to be a rate, but it lacks a time input. Why not just include division by days in the equation? ***Response:*** *This equation has been removed from the method. Instead, results are calculated in units of mass weathered per week and mass produced per sample weight.*

3. Do you believe that this draft method has the potential to serve as an effective and standardized tool in predicting the quality of mine drainage? Do you have recommendations that may enhance method procedures towards obtaining this goal?

3.1	**Comment:** Somewhere there needs to be a discussion of how the results of this test are to be used. If the test gives acidity <0 after 8 weeks, is the site ok? How does one use the data on mg/cm^2 and mg/cm^2day? ***Response:*** *Section 10.0 of the method has been expanded to include instructions and guidance regarding use of method results. Appendices also have been added to provide example calculations for determining carbonate dissolution and pyrite oxidation rates, and for estimating mineral solubility of calcite and gypsum.*

3.2	**Comment:** How will test data be used for prediction? Will it be assumed that column data will simulate that in the field? Will drainage quality or rates be used in conjunction with modeling? ***Response:*** *See response to Comment 3.1.*
3.3	**Comment:** How is variability in the overburden composition accounted for? Are several samples from each stratum tested? ***Response:*** *The number and location of samples that should be collected will depend on the site and rock structure to be evaluated. Section 7.1 of the method instructs users to "Collect representative bulk samples using air-rotary, drilling, core drilling, or extraction ....." The section also provides several references of standardized sample collection procedures.*

4(a). Do you believe that the draft method has sufficient detail to allow its application in laboratories that typically perform mine drainage analyses?

4a.1	**Comment:** You have made some nice progress in defining a method and I particularly like the fact that you address potential problems in spots, such as the use of the bulb pipette. The description is in general, detailed enough to allow other labs to adopt it. ***Response:*** *Thank you.*
4a.2	**Comment:** The description is in general, detailed enough to allow other labs to adopt it. ***Response:*** *Thank you. Additional detail has been added based on observations and results during the interlaboratory method validation study.*
4a.3	**Comment:** The draft method (without the ability for individual laboratories to modify the method) provides sufficient detail to allow its successful application. ***Response:*** *See response to Comment 1d.9.*

4(b). Do you believe that the detail provided allows sufficient flexibility for method users to adjust for cost and laboratory conditions, while still meeting the method performance criteria?

4b.1	**Comment:** I strongly recommend that a standard method be established with little allowance for deviations. The method must compare "apples with apples" and not "apples with oranges". ***Response:*** *See response to Comment 1d.9.*
4b.2	**Comment:** Few, if any, deviations from the method can be tolerated. Small deviations could result in great differences between laboratories. ***Response:*** *See response to Comment 1d.9.*
4b.3	**Comment:** I do not believe that the flexibility for a laboratory to modify the method will result in meeting the method performance criteria. Each laboratory may reach a high level of precision, but comparison between laboratories will be less than acceptable. ***Response:*** *See response to Comment 1d.9.*
4b.4	**Comment:** Section 7.3 The possibility of multiple T and moisture and timing regimes seems to negate the advantage of a standard procedure. ***Response:*** *See response to Comment 1d.9.*

5. What, in your opinion, are the most influential variables in creating the results that are obtained using the weathering procedures in this draft method?

5.1	**Comment:** The most influential variables in creating the results obtained using the weathering procedures in this draft method are: 1. Particle size – Removing the small particle size may decrease variability but at the same time a significant source of acidity and neutralization potential may be eliminated for some materials/sites. 2. Method flexibility may significantly impact the ability to compare data between laboratories. ***Response:*** *See response to Comments 1a.1 and 1d.9.*

GENERAL

G.1	**Comment:** Another major question is whether this procedure, or any kinetic procedure, really provides results similar to runoff from a field site. This would seem to be another major research project needed before this method can be considered reliable. ***Response:*** *Penn State University's Mineral Research Institute applied this method to rock samples in a highly acidic rock in major highway cut and fill in north central Pennsylvania and demonstrated that the results compared well to the results being produced in the field (see method Reference 12.7). Section 3.0 of the method acknowledges, however, that "the method is limited by the extent to which the sample and simulated weathering conditions approximate actual site conditions." The method further acknowledges that "It is not possible to collect a sample from the field for evaluation in the laboratory without disrupting the in-situ particle size distribution through collection mechanisms and crushing." Because it is not possible to precisely mimic field conditions, particularly weathering conditions over time, the method is designed to generate data based on standardized conditions, and those conditions must be considered by each data user. Section 3.0 also states that when assessing method results, "the user should consider sample collection and storage procedures, the changes made to the sample between collection and preparation (e.g., sample crushing and reconstruction), and the similarity of the simulated weathering to actual site conditions (e.g., percent humidity, partial pressures of gases, and saturation/drying cycles).*
G.2	**Comment:** Section 7.1.2 As a general comment, there are at least two benefits to conducting weathering tests under controlled conditions. First, a body of empirical data will be generated to understand the behavior of particular lithologic units. Second, a scientific foundation for the behavior of lithologic units can be developed. In order for the second benefit to be derived, tests must be conducted on well-characterized samples. Is the level of scientific understanding at a stage where the second benefit is of no value? ***Response****: Section 8.1.2 of the method discusses sample characterization. The extent of characterization is optional and depends on data user needs. The U.S. Geological Survey did extensive characterization of samples that were used in the interlaboratory study during validation of this method. Results were presented at the 2005 Geological Society of America conference in Philadelphia, PA .*

G.3	**Comment:** Part 7.3 – The statement that these procedures are recommended for evaluation of overburden in non-arid regions or areas where there may be variably saturated conditions does not make much sense. Acid mine drainage whether occurring in the east or west have variable saturated conditions. ***Response:*** *We agree that saturation cycles occurs in both arid and non-arid regions of the U.S., however, the statement is intended to distinguish between western arid coal regions and the more humid eastern coal regions where variable saturated conditions are more prevalent.*
G.4	**Comment:** Paragraph 2, sentences 1 and 2. These sentences appear to be at odds with one another. The first says that acid-base accounting (ABA) is applicable "to strata that have an appreciable amount of either pyrite or carbonates." The second indicates that "mines with either a scarcity or an abundance of these materials fall into a "gray" area that is difficult to predict." Thus the first says that ABA is applicable if there is an "appreciable amount" of pyrite or carbonate and the second indicates that an abundance of these materials leads to an inconclusive result. This should be clarified. ***Response****: This paragraph has been revised to clarify that ABA is limited to strata that have an appreciable net acid-base balance, and that mines with near equal amounts of acid and alkaline production potential fall into a gray area that is difficult to predict. See second paragraph of method Section 1.0.*
G.5	**Comment:** I strongly urge the team to consider a field validation test. What you have done so far has not validated the method at all. It doesn't matter if 20 people agree that 2+2=5 if that is not the right answer. Comparing the replicability of the method is a good and necessary step, but so is evaluating whether your improvements in procedures that enhance consistency and reaction rates improve or detract from accurate predictions. Using samples from problematic sites that have already been reclaimed, test the variables (such as adding carbon dioxide, and removing the small particles, both of which could dramatically affect the validity of the prediction) to see if you are making the method more or less useful. If not, in my opinion, you are debating whether it is more appropriate to do a waltz or a highland fling on top of the pin. ***Response:*** *See response to G.1.*
G.6	**Comment:** Tests of this nature include the period between rinses, during which most dissolution occurs, and the rinse phase, during which reaction products are removed. A brief description of the test's intent and rationale regarding these phases may benefit the uninitiated (and assist those reviewing the method). It appears that simulation of field conditions is of interest in order to maintain a reaction environment similar to that in the field. The CO_2-air mixture is added to the column and to the rinse water (leachant) to maintain a condition that is apparently expected in the field. It may be helpful to state this, as well as the reasons gas phase composition is of importance to the reactions of interest. Alternatively, citations addressing this aspect could be provided. It is unclear if the rinse phase is intended to remove the majority of reaction products. Clarification would, once again, be helpful. ***Response****: Although dissolution and chemical reactions that occur in the columns is critical to understanding the geochemistry of samples, particularly when exposed to weathering conditions, such details are outside the scope of the method. However, useful information regarding carbonate dissolution, pyrite oxidation, and the mineral solubility of calcite and gypsum have been added as method appendices. .*

MISCELLANEOUS

M.1	**Comment:** How do you define "mine discharges at a mine site", i.e. does "mine discharges" mean that only the quality of discharge from the adit is being predicted? ***Response:*** *The method is primarily designed to predict mine discharge quality of post mining discharges at surface mines. However, the method could also be applied to active mines, including adit and other active mine discharges.*
M.2	**Comment:** You intersperse metric and standard units. You need to be consistent at least. Given the fact that the method, if adopted, will undoubtedly be used internationally, I would suggest including both (one in parentheses) each time. ***Response:*** *Method developers have intentionally used English units when referring to construction materials, and metric units when referring to laboratory measurements.*
M.3	**Comment:** Para. 2.0 line 4. A bracket appears for no reason, and is not closed. ***Response:*** *Thank you. This has been corrected.*
M.4	**Comment:** Section 1.0, second paragraph, second line Strata that are dominated by either pyrite or... next line substitute both for "those" --same idea--having a scarcity or an abundance of just one is not problematic. ***Response:*** *See response to Comment G.4.*
M.5	**Comment:** lines 9-11--...are needed to predict mine drainage pollution...also have for predicting the efficiencies.... next paragraph--last sentence doesn't make sense as written please rewrite and avoid the phrase method performance--it sounds like a drama lesson. ***Response:*** *The Scope and Application section of the method has been reviewed and revised for clarification. The term "method performance" is used across all program offices with the U.S. Environmental Protection Agency. See response to Comment 1d.8.*
M.6	**Comment:** Section 2.0 Delete described in this Method--The procedures include: ***Response:*** *The commenter's recommended edit has been incorporated.*
M.7	**Comment:** Section 3.0 First paragraph, second sentence: Consider as an alternative, "…quality that will be produced by the weathering of an overburden sample…" ***Response:*** *This sentence has been revised to state that "The purpose of this method – to characterize the water quality of mine site drainage – is limited primarily by the extent to which the sample and simulated weathering conditions approximate actual site conditions.*
M.8	**Comment:** Section 3.1.2 ...requirement for determining the particle size distribution...third line, eliminate first "and" ***Response:*** *Thank you. The correction has been incorporated.*
M.9	**Comment:** Section 3.2 First line, delete determination of; 6th line, to consider assessing the effective.... ***Response****: The recommended edit has been incorporated.*
M.10	**Comment:** Section 3.3 Eliminate dash ***Response:*** *Thank you. The dash has been eliminated.*

M.11	**Comment:** Section 4.1, 5th line Transparent material is recommended so that sample conditions can be monitored during.... ***Response:***
M.12	**Comment:** Section 4.2.1 ...and throughout rest of document, eliminate semi-colon after 20-25 degrees C ***Response:*** *The semi-colon is an artifact of different versions of Microsoft Word/Word Perfect, and has been eliminated from the September 2008 pdf version of the method.*
M.13	**Comment:** Section 5.1.2 Insert that in last sentence--to ensure that all oil is removed. ***Response:*** *The commenter's recommended edit has been incorporated.*
M.14	**Comment:** Section 6.1 Insert commas after et al. in both locations ***Response:*** *Commas have been inserted.*
M.15	**Comment:** Section 6.2 Remove comma before et al., 1988 ***Response:*** *Comma has been removed.*
M.16	**Comment:** Section 7.1.1 ...to ensure that no particles are sized greater than....Also, insert space between No. and 4 ***Response:*** *The commenter's recommended edits have been incorporated.*
M.17	**Comment:** Section 7.1.2 It looks like there is an unnecessary space before Sample; 3rd line, delete "an assessment of"; 5th line--on the sample after the last leachate sample.... ***Response:*** *The commenter's recommended edits have been incorporated.*
M.18	**Comment:** Table 1 Note Should Approved be capitalized? ***Response:*** *Approved has been revised to lower case in the title of this table.*
M.19	**Comment:** Section 7.1.2 Last sentence. Consider as alternative wording, "...performed on the sample following the test (i.e., the..." ***Response:*** *This sentence has been revised to state that "…these analyses also may be performed on the sample after the last leachate sample has been collected and the sample has been removed from the column."*
M.20	**Comment:** Section 7.1.3 ....portions, separate samples... ***Response:*** *Thank you. Correction has been incorporated. (See method Section 8.1.3.)*
M.21	**Comment:** Section 7.1.4 table referred to is untitled and unnumbered--it should be Table 2 ands referred to as such. ***Response:*** *The edit recommended by the commenter has been incorporated. (See Table 2, method Section 8.1.3.)*
M.22	**Comment:** Section 7.2.3 Remove semi-colons ***Response:*** *Semi-colons have been removed.*
M.23	**Comment:** Section 7.4.1.3 For the last sentence consider, "...in ice if drainage is slow.) ***Response:*** *The edit recommended by the commenter has been incorporated. (See method Section 8.5.1.)*

M.24	**Comment:** Section 7.4.1.3: Redundant - this is the same verbiage as the Note in Section 7.4.1. Recommend eliminating one or the other. ***Response:*** *The redundant text has been removed from this section, and is provided only as a note. See method Section 8.5.1.*
M.25	**Comment:** Section 7.3.3.2 A sentence needs to be inserted after the first sentence saying that the columns should be drained and then allowed to remain undisturbed for 6 (or 7) days. ***Response:*** *This section (method Section 8.4.3) has been revised to state that "Following this 24-hour saturation period, drain the column and collect the leachate, then repeat the humidified air cycle (Section 8.4.2). Section 8.4.2 explains that "The column is allowed to sit for a period of 6 days during the humidified air cycle. This cycle is repeated after each saturation cycle (Section 8.4.3)."*
M.26	**Comment:** Section 7.4.2 Looks like an unnecessary space before Recommended. Reference to Table 2 should now be Table 3. ***Response:*** *Thank you. Corrections have been incorporated.*
M.27	**Comment:** Table 4 Should the gas mix for the coal refuse be "Saturated air" rather than "Saturate H_2O"? ***Response:*** *This table has been removed from the method; it has been replaced with information based on inter-laboratory study data.*
M.28	**Comment:** Many of the references are incomplete. ***Response:*** *Method reference citations have been completed, where information is available.*
M.29	**Comment:** References: ASTM. 2000. D5744-96, Standard test method for accelerated weathering of solid materials using a modified humidity cell. *In* Annual book of ASTM Standards, 11.04. American Society for Testing and Materials, West Conschohocken, PA (257-269). ***Response:*** *This reference is provided as indicated by the commenter.*