



## **Bureau of Mining Regulation and Reclamation**

### GUIDANCE DOCUMENT

#### NEVADA MODIFIED NET ACID GENERATION TEST PROCEDURE

21 November 2024

The Nevada Division of Environmental Protection, Bureau of Mining Regulation and Reclamation (Division) has determined the Nevada Modified Net Acid Generation (NV-NAG) Test Procedure to be an acceptable method for assisting in the determination of potential acid generation (PAG) from mined materials. Sulfate-bearing minerals (e.g., barite, gypsum, alunite and other similar-type minerals) have been observed to exhibit “false positive” PAG results on repeated occasions when tested under the commonly used and industry-accepted Nevada Modified Sobek Procedure (NMSP). This phenomenon has been verified in the literature.

When the presence of sulfate-bearing minerals is suspect and an accurate laboratory PAG determination is necessary, the Division will require samples to be characterized using the NV-NAG test procedure, mineralogical characterization, and NMSP testing of suspect samples by a Nevada-approved/certified laboratory. In addition, the operator is required to provide sample mineralogy, Nevada Modified NV-NAG, and NMSP results to the Division. For additional guidance regarding material characterization requirements, please see the “Waste Rock, Overburden, and Ore Evaluation” guidance document which can be found on the Division website.

The Division may require the NV-NAG be performed when a request for HCT termination is submitted, provided that certain criteria are met. The Division will consider an HCT termination request following a minimum 20 weeks of HCT testing. If the NMSP ANP/AGP ratio of the initial HCT feed material was  $\leq 1.2$  and the weekly HCT data generally indicate neutral leachate conditions, the Permittee may be required to complete the NV-NAG as part of the termination request. The NV-NAG shall be performed on the original HCT feed sample. Beginning 1 August 2020, the Division will only accept NV-NAG test results from laboratories approved by the State of Nevada Bureau of Safe Drinking Water, Laboratory Certification Branch for the NV-NAG method.

Please note: The majority of the Nevada laboratories certified under the “Mining Approval Program” have been approved for a similar version of this method since approximately 2017. The Nevada Modified Net Acid Generation Test Procedure standardizes sample grind size consistent with the NMSP and mandates the sequential addition as the minimum test protocol. The 1 August 2020 requirement corresponds to the laboratory certification renewal date and mandates the implementation date of modifications to existing certifications.

#### **Procedure Summary**

The NV-NAG test involves the addition of 250 milliliters (ml) of 15% hydrogen peroxide ( $H_2O_2$ ) to 2.5 grams (g) of pulverized (95% minus 150 mesh) sample. Allow sample to react until ‘boiling’ or effervescence ceases. This may require the sample to be left overnight, at the longest. The entire sample is heated until gently bubbling for approximately 1-2 hours to remove excess  $H_2O_2$  and encourage release of inherent neutralizing capacity. Once the sample has cooled to room temperature, measure the pH of the solution (NAG pH).

A NAG pH less than 4.5 standard units (S.U.) will require repeating of the NV-NAG test on the filtered or centrifuged solid residue of the sample, repeat this process until the NAG pH is greater than or equal to 4.5 S.U. or after five sequential additions. Once the NAG pH is greater than or equal

to 4.5 S.U., titrate the solution to 7.0 S.U., and add all NAG acidities to determine total NAG acidity in kg H<sub>2</sub>SO<sub>4</sub> per metric tonne.

Solution assays of sequential NAG solutions may be carried out by filtering solutions from each stage using No. 40 Whatman or finer filter paper or centrifuging, topping up the filtered solutions to the original 250 ml with deionized water to account for loss by evaporation, and analysis of solutions for metals (and sulfur [S]) by Inductively Coupled Plasma, Optical Emission Spectroscopy/Mass Spectroscopy (ICP-OES/MS).

### **NAG pH only option**

The NAG pH only option of the NV-NAG test procedure can be used when the NV-NAG test procedure is not required to address the presence of sulfate minerals that can cause a ‘false positive’ PAG results in the NMSP. The NAG pH only option will need to be performed by a Nevada-certified laboratory for the NV-NAG test, but will not include the measurement of total NAG. These screening level NAG pH data can be used to select samples for further characterization, as appropriate. However, the full NV-NAG test procedure will need to be completed for instances where an accurate laboratory PAG determination is needed and cannot be obtained from the NMSP due to the interference of sulfate minerals.

### **Principles**

The NAG test involves the addition of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) to a prepared sample of mine rock or process residue to oxidize reactive sulfide minerals, followed by measurement of the pH of the reaction solution to provide a total acid-producing potential for samples in which oxidation of sulfide sulfur (S) is incomplete (typically when pyritic S is greater than 0.7 to 1% S). Finally, if any net acidity is produced during the procedure, it is titrated with sodium hydroxide (NaOH).

### **Comments**

A temperature rise is commonly observed in NAG testing of sulfidic samples as a result of catalytic decomposition of peroxide by metal ions released during sulfide oxidation.

The pH of the 15% hydrogen peroxide solution should be checked prior to use to ensure it is greater than or equal to 4.5. To adjust the pH use a NaOH solution made up by adding 1 g NaOH to 100 mL of deionized water. A target pH of 4.5 but not greater than 6.0 is desired. Record the pH of the H<sub>2</sub>O<sub>2</sub> reagent for each batch.

### **Chemicals**

1. Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>) 15%.
2. Hydrochloric acid (HCl) solution, 0.1 Normality (N), certified grade (Fisher So-A-54 or equivalent).
3. Sodium hydroxide pellets (Fisher S320-10 or equivalent).
4. 0.5 N Sodium hydroxide (NaOH): Dissolve 20.0 grams (g) of NaOH pellets in carbon dioxide (CO<sub>2</sub>) free water and dilute to 1.0 liter. Protect from CO<sub>2</sub> in the air with ascarite tube. Standardize solution by placing 50 milliliters (ml) of certified 0.1 N HCl in a beaker and titrating with the prepared 0.5 N NaOH until a pH of 7.00 S.U. is obtained.

Calculate the Normality (N<sub>2</sub>) of the NaOH using the following equation:

$$N_2 = (N_1 V_1) / V_2, \text{ where}$$

$V_1$  = Volume of HCl used, ml;

$N_1$  = Normality of HCl used;

$V_2$  = Volume of NaOH used, ml;

$N_2$  = Calculated Normality of NaOH.

5. 0.1 N Sodium hydroxide (NaOH): Dilute 400 ml of deionized water with 100 ml of 0.5 N NaOH;
6. pH 1.68 buffer solution;
7. pH 4.0 buffer solution;
8. pH 7.0 buffer solution;
9. Deionized water;

### Equipment

1. Burette;
2. Hot Plate;
3. pH meter equipped with combination electrode;
4. Balance, accurate to 0.01 gram;
5. No. 40 Whatman or finer filter paper or tubes and centrifuge;
6. 500 ml conical flask.

### Procedure

1. Prepare the sample to 95% minus 150 mesh;
2. Check the pH of the  $H_2O_2$  to ensure the pH is between 4.5 and 6 S.U. If the pH is less than 4.5 S.U., add diluted NaOH until the pH is greater than 4.5 S.U. This step must be performed to negate a false positive result of the sample being identified as acid generating;
3. In a 500 ml flask combine  $2.5 \text{ g} \pm 0.1 \text{ g}$  of pulverized sample with 250 ml of 15%  $H_2O_2$  (equilibrated to room temp). Allow sample to react until 'boiling' or effervescence ceases. This may require the sample to be left up to 12 hours, or overnight at the longest;
4. Place sample on a hot plate at medium heat and gently boil for 1 to 2 hours or until the effervescing ceases. This allows removal of excess  $H_2O_2$  and encourages release of inherent neutralizing capacity. **The sample must not be allowed to boil dry**; add deionized water if necessary;
5. Allow the sample to cool to room temperature and record the final pH (NAG pH);
6. Centrifuge or filter the solid from the liquid and retain (To avoid potential interference from filter paper, the Division recommends centrifuging or rinsing the solid residue from the filter paper);
7. Add deionized water to the liquid to bring it back to 250 ml;
8. If NAG pH is greater than 4.5, titrate the solution to pH 7.0 with 0.10 M of NaOH and record the volume. Report the titrated acidity as the NAG Acidity (NV-NAG). If NAG pH is greater than 7.0 S.U. then report the volume of NaOH and the NAG for the sequential addition as zero;

9. If NAG pH is less than 4.5, titrate the solution to pH 4.5 with the appropriate molarity of NaOH (see table below) and record the volume;

| NAG pH            | NaOH Concentration |
|-------------------|--------------------|
| >2 standard units | 0.10 M             |
| <2 standard units | 0.50 M             |

10. Following titration to pH 4.5:

- Verify the H<sub>2</sub>O<sub>2</sub> is within acceptable pH range;
- In a 500 ml flask combine solid residue from Step 6, with 250 ml of 15% H<sub>2</sub>O<sub>2</sub> (equilibrated to room temp). Allow sample to react until 'boiling' or effervescence ceases. This may require the sample to be left overnight, 12 hours at the longest;
- Place sample on a hot plate at medium heat and gently boil for 1 to 2 hours or until the effervescing ceases. This allows removal of excess H<sub>2</sub>O<sub>2</sub> and encourages release of inherent neutralizing capacity. **The sample must not be allowed to boil dry;** add deionized water if necessary;
- Allow the sample to cool to room temperature and record the final pH (NAG pH);
- Centrifuge or filter the solid from the liquid and retain (To avoid potential interference from filter paper, the Division recommends centrifuging or rinsing the solid residue from the filter paper);
- Add deionized water to the liquid to bring it back to 250 ml;
- Record volume of NaOH for each titration separately. Add all of the titrated NAG acidity to get a total NAG Acidity in kg H<sub>2</sub>SO<sub>4</sub> per metric tonne\* sample. Repeat from Step 8 as needed or for a maximum of five iterations.

### Calculations

1. Calculate NV-NAG using the following equation:

$$NV - NAG \text{ (in kg H}_2\text{SO}_4 \text{ / tonne)} = \frac{49(V)(N)}{W}$$

V = Volume of base NaOH titrated, in ml

N = Normality of the base NaOH, in moles per liter

W = Weight of the sample, in grams

Please note the calculation may need to be completed for each normality then be added together.

\* 1 metric tonne equals 1,000 kg.

2. Convert NV-NAG in kg H<sub>2</sub>SO<sub>4</sub> per metric tonne to kg H<sub>2</sub>SO<sub>4</sub> per short ton using the following equation:

$$\frac{NV - NAG(\text{kg H}_2\text{SO}_4/\text{tonne})}{1.102} = NV - NAG \left( \frac{\text{kg H}_2\text{SO}_4}{\text{ton}} \right)$$

5. Convert NV-NAG in kg H<sub>2</sub>SO<sub>4</sub> per short ton to kg CaCO<sub>3</sub> per short ton using the following equation:

$$\text{NV - NAG (kg H}_2\text{SO}_4 \text{ / ton)} \times 1.0206 = \text{NV - NAG (kg CaCO}_3 \text{ / ton)}$$

### Reporting Requirements (See Table below)

1. Report NAG pH from Step 5, and each at sequential addition, as appropriate;
2. Report NaOH volume (ml) used in Step 9 for each sequential addition;
3. Report NaOH normality used in Step 9 for each sequential addition;
4. Report NV-NAG for each sequential addition;
5. Report NV-NAG in kg H<sub>2</sub>SO<sub>4</sub> per short ton and kg CaCO<sub>3</sub> per short ton.

### References:

AMIRA International Ltd. (2002). ARD Test Handbook, Prediction and Kinetic Control of Acid Mine Drainage. Environmental Geochemistry International Pty. Ltd. and Ian Wark Institute, University of South Australia.

Prestia, A., R.J. Bowell, R. Warrender, C. Brough, and K. Fallowfield. *Application of ABA Methods to Barite Projects in Nevada*. Conference Paper, International Applied Geochemistry Symposium, April 2015.

Warwick A. Stewart, Stuart D. Miller, and Roger Smart, *Advances in Acid Rock Drainage (ARD) Characterisation of Mine Wastes*, Paper presented at the 7<sup>th</sup> International Conference on Acid Rock Drainage (ICARD), March 26-30, 2006, St. Louis MO. R.I. Barnhisel (ed.) Published by the American Society of Mining and Reclamation (ASMR), 3134 Montavesta Road, Lexington, KY 40502.

Sobek et al, (1978), Field and Laboratory Methods Applicable to Overburden and Minesoils, EPA 600/2-78-054, 203 pages.

### Revisions:

Revision 00: 20 July 2021.

Revision 01: Updated 31 January 2021.

Revision 02: Updated 16 November 2021, added centrifuging option and divide by 1.102 to convert from metric tonne to short tons (ton).

Revision 03: Updated 31 January 2022, provided additional clarification throughout the document for centrifuging and filter paper is not to be digested with the solid. Modification of reporting requirements number 4 from total volume of NaOH titrated to NV-NAG for each sequential addition and addition of the Calculations Table at the end of the Procedure. Further clarified the method for sequential additions, including for when the test results in 7.0 S.U. for NAG pH.

Revision 04: Updated 20 September 2022, changed units for normality in Calculation Table from M/L to N.

Revision 05: Updated 22 July 2024, modified to the maximum of 5 sequential additions, added option for only NAG pH.

## NEVADA MODIFIED NET ACID GENERATION TEST PROCEDURE CALCULATION TABLES

**Table 1: Nevada Modified Net Acid Generation Test Calculation Table with Formulas**

| Parameter  | I.D.                                    | Units                                  | Formula   |
|--|---|--|---|
| Sample Weight  | W                                       | g                                      |   |
| <b>FIRST ADDITION</b>                                  |   |  |   |
| NAG pH-S1  | NAG-pH-S1                               | S.U.                                   |   |
| NaOH Normality-S1                                      | N-S1                                    | N                                      |   |
| Volume NaOH-S1   | V-S1                                    | mL                                     |   |
| NAG-S1   | <b>NAG-S1</b>                           | kg H <sub>2</sub> SO <sub>4</sub> /Ton | =49(N-S1)(V-S1)/(W*1.102)   |
| <b>SECOND ADDITION</b>                                 |   |  |   |
| NAG pH-S2  | NAG-pH-S2                               | S.U.                                   |   |
| NaOH Normality-S2                                      | N-S2                                    | N                                      |   |
| Volume NaOH-S2   | V-S2                                    | mL                                     |   |
| NAG-S2   | <b>NAG-S2</b>                           | kg H <sub>2</sub> SO <sub>4</sub> /Ton | =49(N-S2)(V-S2)/(W*1.102)   |
| <b>THIRD ADDITION</b>                                  |   |  |   |
| NAG pH-S3  | NAG-pH-S3                               | S.U.                                   |   |
| NaOH Normality-S3                                      | N-S3                                    | N                                      |   |
| Volume NaOH-S3   | V-S3                                    | mL                                     |   |
| NAG-S3   | <b>NAG-S3</b>                           | kg H <sub>2</sub> SO <sub>4</sub> /Ton | =49(N-S3)(V-S3)/(W*1.102)   |
| <b>FOURTH ADDITION</b>                                 |   |  |   |
| NAG pH-S4  | NAG-pH-S4                               | S.U.                                   |   |
| NaOH Normality-S4                                      | N-S4                                    | N                                      |   |
| Volume NaOH-S4   | V-S4                                    | mL                                     |   |
| NAG-S4   | <b>NAG-S4</b>                           | kg H <sub>2</sub> SO <sub>4</sub> /Ton | =49(N-S4)(V-S4)/(W*1.102)   |
| <b>FIFTH ADDITION</b>                                  |   |  |   |
| NAG pH-S5  | NAG-pH-S5                               | S.U.                                   |   |
| NaOH Normality-S5                                      | N-S5                                    | N                                      |   |
| Volume NaOH-S5   | V-S5                                    | mL                                     |   |
| NAG-S5   | <b>NAG-S5</b>                           | kg H <sub>2</sub> SO <sub>4</sub> /Ton | =49(N-S5)(V-S5)/(W*1.102)   |
|  |   |  |   |
| <b>Total NAG in kg H<sub>2</sub>SO<sub>4</sub>/Ton</b> | <b>NAG(H<sub>2</sub>SO<sub>4</sub>)</b> | kg H <sub>2</sub> SO <sub>4</sub> /Ton | = <b>NAG-S1</b> + <b>NAG-S2</b> + <b>NAG-S3</b> + <b>NAG-S4</b> + <b>NAG-S5</b> |
| <b>Total NAG in kg CaCO<sub>3</sub>/Ton</b>            | NAG(CaCO <sub>3</sub> )                 | kg CaCO <sub>3</sub> /Ton              | = <b>NAG(H<sub>2</sub>SO<sub>4</sub>)</b> * 1.0206                              |

### Reminders/Requirements

- Many sequential additions may be needed per the method, to a maximum of 5, and the table above illustrates the required reporting parameters for each addition.