Colorado Procedure – Laboratory 2103-13

Standard Method of Test for

Determining the Sulfate Ion Content in Water or Water-Soluble Sulfate Ion Content in Soil

1. SCOPE

1.1 This method establishes a procedure for determining the amount of sulfate ions present in water or determining the water soluble sulfate ion content in soil effecting CDOT projects.

1.2 Method A describes the procedure for determining the concentration of sulfate ions present in water. Method B describes the concentration of water soluble sulfate ions present in soil.

2. APPARATUS

2.1 Colorimeter - Hach models: Pocket Colorimeter II, DR 820, DR 850, DR 950 or equivalent.

2.1.1 Sample Cells – As recommended by the colorimeter manufacturer.

2.2 Glassware – 500 ml Erlenmeyer Flasks, 500 ml graduated cylinder.

2.3 Pipet - graduated 10 ml with bulb.

2.4 Filter Papers - #42 Whatman

2.5 Timer - readable to 1 second.

2.6 Drying apparatus - Any suitable device capable of drying samples at a temperature not exceeding 60°C (140°F).

2.7 Misc. Equipment - Funnel, Rubber Stoppers.

2.8 Barium Chloride - Pre-measured packet.

2.9 Sieves - #4 and #40.

2.10 Digestion heat source – Any suitable device that will maintain the temperature of the solution @ 140°F (+/- 5°F) throughout the digestion period.

2.11 Distilled water

Note 1: Clean all glassware with distilled water prior to each use.

METHOD A
(Sulfate Ion Content in Water)

3. PROCEDURE

3.1 Use a pipet to place 10 ml of sample into 2 sample cells. One will serve as the blank and the other as the reacted sample. If the sample exhibits turbidity or color then place a filter paper in the funnel and filter the sample until a clear sample is obtained.

3.2 Set the timer for 5 minutes. Add reagent to one of the cells. Cap the cell and shake vigorously. Place the sample in a location where it will be undisturbed and start the timer.

3.3 Within 5 minutes after the timer goes off place the blank into the colorimeter and cover. Zero the meter according to the manufacturer's recommendations.

3.4 Remove the blank and replace it with the reacted test sample and cover. Take the reading in accordance with manufacturer recommendations.

3.5 Record reading. If the reading exceeds the limits of the meter or reports the meter's maximum reading (refer to the manufacturer's manual) the sample will need to be further diluted with distilled water. Discard test and repeat steps 3.1 through 3.5 with new diluted sample. **See Example 1, Method A**

4. CALCULATION

4.1 Correct the reading obtained in Subsection 3.5 using the Standard Sulfate Solution correction curve (see Section 7). To find the correction locate the reading on the bottom scale, follow a vertical line to the curve and then a horizontal line to the vertical scale.

Note 2: Some colorimeter models may have a user defined correction curve stored, automatically correcting the reading. When using this feature, follow the manufacturer's procedure to produce the correction curve.

4.2 Multiply the corrected reading by the total dilution**. See example 1, Method A.** This will give the parts per million

METHOD B
(Water Soluble Sulfate Ion Content of Soil)

5. PROCEDURE

5.1 Obtain sample according to CP 24 or as required by the Pipe Material Selection Policy.

5.2 Dry the sample to constant weight at a temperature not exceeding 60°C (140°F).

5.3 Process the material over a #4 sieve being careful to dislodge any material adhering to the aggregate particles and avoid breaking down the natural size of the particles..

5.4 Process the minus #4 material using a rubber coated pestle until it passes the #40 sieve being careful to dislodge any material adhering to the aggregate particles and avoid breaking down the natural size of the particles. Repeat until no additional minus #40 material is produced.

5.5 Prior to obtaining a test sample, ensure sample is mixed so that uniformity of the sample is achieved. Obtain a representative 25g test sample and place it in a clean, 500 ml flask.

5.6 Add 250 ml of distilled water to the flask. Mix thoroughly, by shaking, using sufficient effort so that no material is left on the bottom of the flask (this is the 1st 10:1 dilution). Seal the flask with a rubber stopper and let the sample sit undisturbed for a minimum of 16 hours. Maintain the temperature of the solution @ 140°F (+/- 5°F).

5.7 After completion of the soaking period if the sample exhibits turbidity (cloudiness) filter the solution through a #42 Whatman filter paper until a clear sample is attained.

5.8 Use a pipet to place 25 ml of the solution into a 500 ml clean flask and add 225 ml of distilled water and mix thoroughly. (This is the 2nd 10:1 dilution, 10:1 + 10:1 =100:1 dilution ratio). * If the sample doesn't require further dilution, this 100:1 dilution would be the final dilution ratio used in the calculation.

5.9 Use a pipet to place 10 ml of solution into 2 sample cells. Add reagent into one of the cells. Cap the cell and shake vigorously. Place the sample in a location where it will be undisturbed and let stand for a minimum of 5 minutes but not more than 10 minutes.

5.10 Place the blank into the colorimeter, cover, and zero the meter. Replace the blank with the reacted sample, cover, and take the reading according to manufacturer's instructions.

5.11 Record reading. If the reading exceeds the machines capabilities the sample will need to be further diluted. **See Example 1, Method B.**

6. CALCULATION

6.1 Correct the reading obtained in Subsection 5.11 using the Standard Sulfate Solution correction curve (see Section 7). To find the correction locate the reading on the bottom scale, follow a vertical line to the curve and then a horizontal line to the vertical scale.

Note 3: Some colorimeter models may have a user defined correction curve stored, automatically correcting the reading. When using this feature, follow the manufacturer's procedure to produce the correction curve.

6.2 Multiply corrected reading by the total dilution; **See Example 1, Method B.** This will give the parts per million.

6.3 Divide the parts-per-million (ppm) obtained in Subsection 6.2 by 10,000 to obtain the percent sulfate in soil by mass.

Standard Sulfate Solution Correction Curve

7. GENERATING CORRECTION CURVE

7.1 Using reagent grade sulfate standard solution prepare samples at 10 ppm increments across the range of the colorimeter in 250 ml beakers. See Example 2.

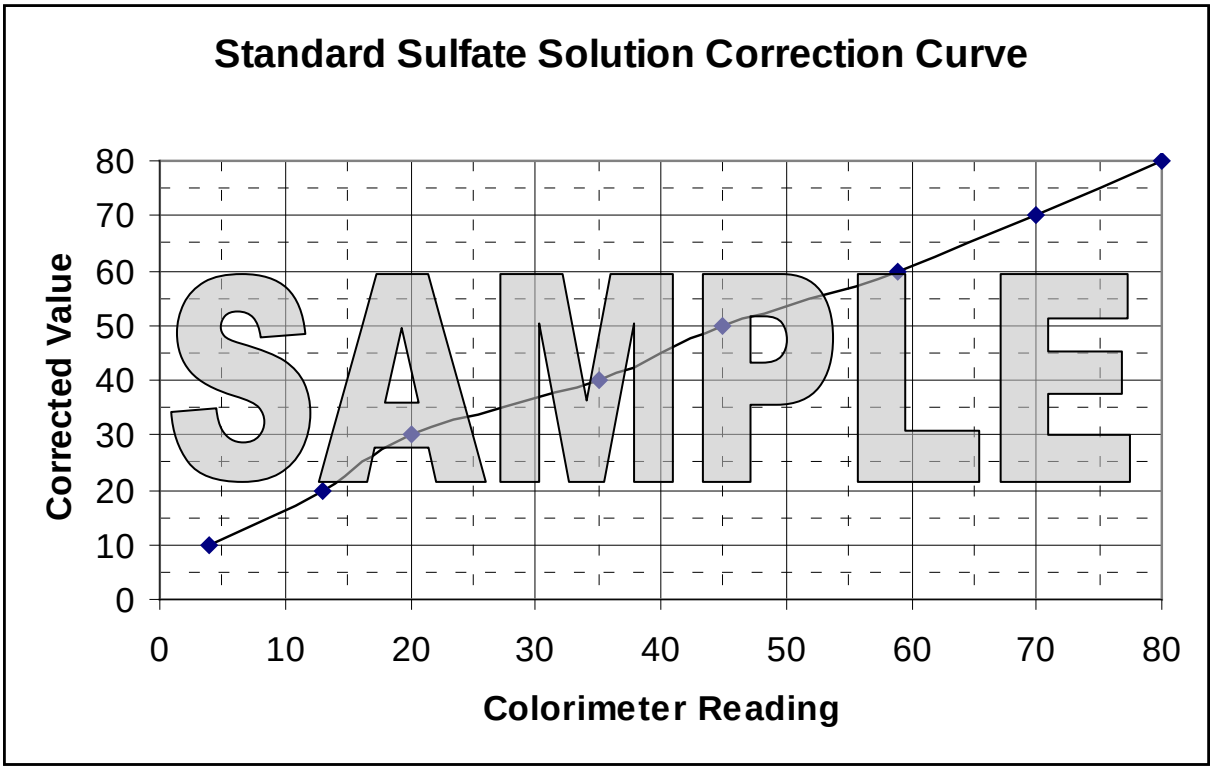
7.2 For each dilution fill 2 sample cells to the 10 ml line. One will be the blank and the other will be the reacted sample. Add reagent to one of the cells. Shake the cell vigorously and let stand undisturbed for 5 minutes but not more than 10 minutes.

7.3 Place the blank into the colorimeter, cover, and zero the meter. Replace the blank with the reacted sample, cover, and take the reading. Record the reading.

7.4 Once all the readings are obtained graph the results and draw the correction curve. See example.

7.5 Correction curves shall be run upon receipt of a new lot of reagent or every 6 months (whichever comes first), or when results are in question.

Sample Correction Curve



Example Curve

Curves are specific to each colorimeter and each lot of reagent.

Water Soluble Sulfates Worksheet

Project No. _____ **Contract ID** _____

Sample I.D._____ **Sample Location** _____

Soil Description_____ **Tested By**: _____

Sample Date _____ **Receive Date** _____ **Test Date** _____

Saturation Date _____

Saturation Time _____

Test Start Time _____

A) Number of Dilutions _____

B) Final Dilution _____

C) Reading _____

D) Corrected Reading _____

E) Sulfate Concentration _____
E = (B x D) (ppm or %)

Simplified Procedure

1) Dry soil (140°F / 60°C) and process through the #4 sieve.
2) Process a representative sample through a #40 sieve.
3) Place a 25g representative sample into clean container.
4) Add 250 ml of distilled water and shake well (This is a 10:1 dilution).
5) Let the container stand undisturbed for a minimum of 16 hrs maintaining the solution @ 140°F (+/- 5°F).
6) Pipet 25 ml of the 10:1 dilution and deposit into a clean 500 ml flask (do not disturb sediment). If sample exhibits turbidity then filter until clear.
7) Dilute test sample to 250 ml by adding 225 ml of distilled water (This is the second 10:1 dilution multiplied by the first 10:1 dilution makes this a 100:1 dilution and the final dilution if the sample doesn't require further dilution).
8) Pipet 10 ml of sample into the sample cells (1 blank and 1 reaction cell).
9) Add reagent to 1 cell, shake well and let stand a minimum of 5 min. but not more than 10 min.
10) Place blank into the colorimeter and zero the meter.
11) Replace blank with reacted cell and take the reading.
12) Record the reading and correct it by using the Standard Sulfate Solution Correction Curve.
13) Corrected reading multiplied by the final dilution = ppm, divide ppm by 10,000 to get % sulfate by mass.
14) If the reading exceeds the limits of the meter or the maximum value of the meter is displayed, discard the test samples and the blank. Clean the sample cells. Dilute the sample further by taking 25 ml from the 10:1 test sample (step 4) and dilute to 500 ml. (200:1 final dilution). Repeat steps 8 – 12. Continue dilutions until a reading is obtained.

Example 1

Method A: Sulfate Ion Content in Water

If the Sulfate level is too high for the Colorimeter to read and the sample requires a larger dilution to get a reading:

Dilution examples:
10ml sample diluted to 50ml = 5:1 dilution
10ml sample diluted to 100ml = 10:1 dilution
10ml sample diluted to 200ml = 20:1 dilution
10ml sample diluted to 300ml = 30:1 dilution
10ml sample diluted to 400ml = 40:1 dilution etc.

Once a reading is obtained, correct it using the Standard Sulfate Solution Correction Curve.

Multiply the corrected reading by the total dilution used.

Example:
The colorimeter reported a reading of 25. Using the correction curve the corrected reading is 24.
The sample was diluted to **200ml**, the total dilution would be **20:1**, so take the **corrected reading** and multiply it by **20** to get the sample's sulfate ion content in parts per million (ppm).
24 x 20 = 480 ppm.

Method B: Water Soluble Sulfate Ion Content in Soil

If the Sulfate level is too high for the Colorimeter to read and the sample requires a larger dilution to get a reading:

Dilution examples:
***Using the 10:1 diluted sample from subsection 5.6**

25ml sample diluted to 250ml = 10:1
25ml sample diluted to 300ml =12:1
25ml sample diluted to 350ml =14:1
25ml sample diluted to 400ml = 16:1
25ml sample diluted to 450ml = 18:1
25ml sample diluted to 500ml = 20:1 etc.

or

25ml sample diluted to 250ml = 10:1
12.5ml sample diluted to 250ml =20:1
6.25ml sample diluted to 250ml =40:1
3.125ml sample diluted to 250ml =80:1

Determining the final dilution ratio, multiply the **10:1** dilution from subsection **5.6** by the second dilution ratio.

Example:
The colorimeter reported a reading of 37. Using the correction curve the corrected reading is 39.
25ml of the 10:1 diluted sample from subsection 5.6 was diluted to 500ml (20:1) multiply 10 x 20 =200.
The final dilution is 200:1.

Multiply your corrected reading from the Standard Sulfate Solution Correction Curve by the final dilution ratio to get the sample's sulfate ion content in parts per million (ppm).
39 x 200 = 7,800 ppm.

Divide the ppm by 10,000 to obtain the percent Sulfate in Soil by mass.
7,800 ppm / 10,000 = 0.78 %.

Example 2

Generating a Correction Curve

Apparatus:

9 - 250ml beakers
1,000 ppm Sulfate Standard Solution:
Distilled water

Place 100 ml of distilled water in a 250 ml beaker – This is the 0 ppm sample.

Place 1 ml Sulfate Standard Solution in a 250 ml beaker and dilute to 100 ml - This is the 10 ppm.

Place 2 ml Sulfate Standard Solution in a 250 ml beaker and dilute to 100 ml - This is the 20 ppm sample.

Place 3 ml Sulfate Standard Solution in a 250 ml beaker and dilute to 100 ml - This is the 30 ppm sample.

Place 4 ml Sulfate Standard Solution in a 250 ml beaker and dilute to 100 ml - This is the 40 ppm sample.

Place 5 ml Sulfate Standard Solution in a 250 ml beaker and dilute to 100 ml - This is the 50 ppm sample.

Place 6 ml Sulfate Standard Solution in a 250 ml beaker and dilute to 100 ml - This is the 60 ppm sample.

Place 7 ml Sulfate Standard Solution in a 250 ml beaker and dilute to 100 ml - This is the 70 ppm sample.

Place 8 ml Sulfate Standard Solution in a 250 ml beaker and dilute to 100 ml - This is the 80 ppm sample.

Plot the colorimeter reading on the horizontal axis vs. the known sample ppm on the vertical axis. Starting on the 0 ppm point use a straight line to connect each of the points.

Example: A point is a known 10 ppm solution but the colorimeter shows a reading of 8. Plot a point at 10 on the horizontal axis and 8 on the vertical axis. Repeat this for each of the 9 samples

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