

CRD-C 403-71

METHOD OF TEST FOR DETERMINATION OF SULFATE ION
IN SOILS AND WATER

1. Scope

1.1 This method of test covers determination of sulfate ion content in soil and water samples.

1.2 The analytical procedure for water samples and for the extracts from soil samples is based on ASTM Designation: D 516, "Methods of Test for Sulfate Ion in Industrial Water and Industrial Waste Water." The conductivity method for determining water-soluble sulfate in soil samples is based on that used at the U. S. Bureau of Reclamation.

WATER

2. The determination of sulfate ion in water samples shall be as prescribed in CRD-C 408.

SOIL

3. Scope

3.1 This section describes two methods for the quantitative determination of the water-soluble sulfate content of soil samples.

4. Apparatus

4.1 The apparatus shall consist of the following:

4.1.1 Balances, Weights, Glassware, and Laboratory Containers shall be of the types specified for chemical determinations and analytical work. See CRD-C 209.

4.1.2 Mortar.- A mortar approximately 8 in. in diameter and pestle.

4.1.3 Sieve.- No. 50 (297-micron) square-hole, woven wire cloth sieve conforming to the "Standard Specifications for Sieves for Testing Purposes" (CRD-C 102).

4.1.4 Agitator.- To be used in extracting soluble sulfates from the sample. Any device that is capable of tumbling or shaking a 500-ml, or larger, container will be satisfactory.

4.1.5 Conductivity Bridge.- A

bridge having a range of 100 to 100,000-ohms resistance. L&N Portable Bridge No. 4-960 has been found to be satisfactory.

4.1.6 Conductivity Cell.- Tubes graduated from 0 to 12 cm, equipped with platinum electrodes. The No. 9-369 conductivity cell, manufactured by Eimer and Amend, has been found satisfactory.

5. Sample Preparation

5.1 The sample shall be representative of the soil to be tested and shall weigh at least 1 lb. A portion weighing approximately 1 lb shall be selected by quartering and shall be air-dried at room temperature and humidity for at least 24 hr, or until the sample can be easily sieved over a No. 50 sieve. The coarser fractions are then crushed (Note 1) in the mortar to pass the No. 50 sieve. The crushed and sieved sample is then thoroughly mixed and repeatedly quartered to a suitable size for final sampling and is then dried to constant weight at 60 ± 10 C (Note 2). The prepared samples shall be stored in airtight containers

Note 1.- The sample shall be crushed by impact, not grinding, as rapidly as possible to reduce the error that may be introduced by oxidation of sulfides.

Note 2.- The amount needed for final sampling will depend upon the method used to determine soluble sulfates. Approximately 5 g is needed for the acid-soluble method and 75 g for the conductivity method. The sample shall be dried to constant weight just prior to the determination.

6. Determination of Soluble Sulfate,
Acid Soluble Method

6.1 Procedure.- Disperse a 1-g sample in a beaker with 25 ml of water using a swirling motion. While still swirling, add 5 ml of HCl all at once. Dilute immediately to 50 ml and digest, just short of boiling, for 15 min. Filter and wash the residue thoroughly with hot water. Dilute the filtrate to approximately 200 ml and continue as directed in Paragraph 9(c) to (f) of CRD-C 408.

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6.2 Calculation.- Calculate the percentage of SO₄ in the soil to the nearest 0.1 as follows:

P = W x 41.15

where:

- P = SO₄, %
W = BaSO₄, g, and
41.15 = molecular ratio of SO₄ to BaSO₄ (0.4115) x 100.

6.3 Report.- If the result obtained by the above procedure is less than 0.2 percent, report it as such. If the result is 0.2 percent or more, the SO₄ composition of the soil must be determined on a water extract.

6.4 Extraction Procedure.-

6.4.1 The ratio of water to soil to use for the extract should be such that all the SO₄ present in the sample would be dissolved at the end of the agitation period. This ratio may be calculated by assuming:

6.4.1.1 All the SO₄ is present in the form of gypsum, and

6.4.1.2 The solubility of gypsum is 0.2 percent.

The ratio will, therefore, be

r = 9P

where:

- r = ratio of water to soil, ml per g,
9 = the molecular ratio of gypsum to SO₄ (172 per 96) divided by the solubility of gypsum, and
P = percent SO₄ in the soil found by the acid soluble method.

6.4.2 The calculated quantities of soil and water are added to the agitator container (Note 3) and the mixture is agitated continuously for 6 hr. Transfer an aliquot of the clear leach water (filtered if necessary) to a beaker and dilute to approximately 200 ml. Add HCl until acid to methyl red and add 10 ml excess of HCl(1:9). Determine the SO₄ content by continuing with the procedure described in Paragraph 9(c) to (f) of CRD-C 408.

Note 3.- The volume of water may be as much as, but no more than, 80 percent of the volume of the container.

6.5 Calculate and report the per-

centage SO₄ to the nearest 0.1 as follows:

P = 41.15 W r/V

where:

- P = SO₄, %
W = BaSO₄, g,
r = ratio of water to soil, ml per g, and
V = volume of aliquot, ml.

7. Determination of Soluble Sulfate, Conductivity Method

7.1 Extraction Procedure.- The prepared sample is agitated continuously for at least 6 hr. The ratio of water to soil will depend upon the suspected sulfate content. A ratio of 10 to 1 is suggested for the first trial. The solids shall be allowed to settle out of the liquid phase for at least 30 min before taking an aliquot of the extract for resistance measurement. This aliquot need not be filtered.

7.2 Resistance Measurements.- Measure the resistance of the extract and determine the conductivity cell constant by following the procedures described in Method CRD-C 12.

7.3 Calculations.- Compare the resistance of the extract with the calculated minimum resistance of leach water obtained from a soil containing 0.1 percent SO₄. The equation for calculating the minimum resistance is derived using the theoretical relation between conductance and the sulfate content of a solution of a pure sulfate compound, which is:

(L x 10^5) / 12 me/L = (K x 10^5) / (12 x R) = 8333 (K/R) me/L

The relation between the SO₄ content of the soil and the resistance of the extract is:

P = (8333 x K x 4.8 x V) / (R x 1000 x M) = (40 x K x r) / R

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where

M = weight of the sample, g,
 4.8 = equivalent weight of SO₄
 (0.048), g, x 100, and
 1000 = factor for converting l to ml,
 and all other symbols are as
 previously defined.

The minimum resistance of an extract from a soil containing 0.1 percent SO₄ would, therefore, be:

$$R = \frac{40 \times K \times r}{P} = \frac{40 \times K \times r}{0.1}$$

$$= 400 \times K \times r$$

7.4 Report.- Resistance values for extracts that are greater than the calculated minimum indicate that the soil contains less than 0.1 percent SO₄ and should be reported as such. Resistance values that are less than the calculated minimum indicate SO₄ concentrations in the soil greater than 0.1 percent and the potential presence of gypsum. Soils containing gypsum must be subjected to further leaching to insure complete solution of the gypsum, and then the actual SO₄ content of the leach water must be determined.

7.5 Determination of the Presence of Gypsum.- The potential presence of gypsum is ascertained by comparing the resistance of the extract with

the resistance of a saturated gypsum solution. The latter resistance is calculated as follows:

$$R = \frac{K}{2250 \times 10^6} = 444 K$$

where:

2250 x 10⁶ = the specific conductance of a saturated gypsum solution.

If the resistance of the extract is equal to or less than 444 K, it may be saturated with gypsum, and undissolved S O₄ may be present. The extract should be discarded and a new extract made using a higher water to soil ratio. If the resistance of the extract is greater than 444 K, gypsum is not present or has been completely dissolved.

7.6 Determination of SO₄ Content.- After determining that gypsum is not present or has been completely dissolved, transfer an aliquot of the last extract made (filtered if necessary) to a beaker and follow the applicable portions of the procedures described in Paragraph 6.4.

7.7 Alternate Method.- As an alternate to making trial leaches, if the S O₄ concentration of the soil is greater than 0.1 percent, the procedures described in Paragraph 6.1 may be used.