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June 7, 1993

QUALITATIVE ANALYSIS FOR SULFATE IN THE SOIL

PROCEDURE:

To 1 gm of dry soil sample, add an equivalent amount of anhydrous sodium carbonate in a 100 ml beaker. Add about 30 ml of distilled deionized water and boil the contents for ten minutes. Cool this solution and filter the Na_2CO_3 extract through a Whatman qualitative filter paper. To 1 ml of the extract in a test tube add excess dilute HCl (until the effervescence ceases) and to this solution add 1 ml of 0.1M $BaCl_2$ solution. A milky white precipitate insoluble in conc HCl confirms the presence of sulfate.

Note: (i)A yellow coloration of the extract indicates the presence of chromate.

(ii)A blue coloration indicates the presence of copper.

Centrifugation of the above mentioned precipitate would indicate the concentration of SO_4^{2-} in the soil A very thin precipitate (faint coating on the walls of the test tube) would indicate a low concentration of sulfate. Under these circumstances (10-400 ppm) EPA Methods SW-846, 9035, or 9036 may be used. Should heavy precipitation be observed, SO_4^{2-} can be determined gravimetrically. (Please see attached procedure).

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ANALYSIS OF A SOLUBLE SULFATE IN SOIL

The analysis of a soluble sulfate is based upon precipitation with barium ion

$$Ba^{2+} + SO_4^{2-} \text{ -----} \rightarrow BaSO_4(s)$$

The barium sulfate is collected on a suitable filter, washed with water, and strongly ignited.

Superficially, this method appears straightforward. In fact, however, it is subject to numerous interferences due chiefly to the tendency of barium sulfate to occlude foreign anions and cations. Table 1 summarizes the more common interferences affecting this analysis. Purification by reprecipitation is not feasible because there is no practical solvent for barium sulfate. It is therefore necessary to eliminate the principal interferences by preliminary treatment of the sample and then to precipitate the barium sulfate from hot, dilute solutions. Even so, the excellent agreement often observed between theoretical and experimental results is due in considerable measure to a cancellation of errors.

PROCEDURE

Dry the soil for at least 1 hour at 105 to 110 degrees C, allow it to cool to room temperature in a desiccator. Weigh (to the nearest 0.1 mg) individual 0.5 to 0.7 g samples into 400 ml beakers. Dissolve each in 200 ml of distilled water to which 4 ml of 6-F hydrochloric acid have been added.

For each sample, dissolve 1.3 g of barium chloride dihydrate in 100 ml of distilled water, and filter if necessary. Heat nearly to boiling before quickly adding, with vigorous stirring, to hot solutions of the sample.

Digest the precipitated barium sulfate for 1 to 2 hours (see Note 1). Decant the hot supernatant through a fine ashless paper (see Note 2). Wash the precipitate three times with hot water, decanting the washings through the filter. Finally, transfer the precipitate to the paper. Place papers and contents in marked porcelain crucibles that have been ignited to constant weight, gently char off the papers. Ignite to constant weight at 900 degrees C. Report the percentage of sulfate in the sample.

NOTES

1. The digested precipitates can be allowed to stand until the following laboratory period without harm.

2. Use of Schleicher and Schuell No. 589 Blue Ribbon or Whatman No. 42 paper is recommended. If desired, the precipitate can be collected in a Gooch crucible or a porcelain filtering crucible. Complete removal of $BaSO_4$ from the latter type crucible is difficult, however.

TABLE 1

INTERFERENCES ATTENDING THE GRAVIMETRIC DETERMINATION OF SULFATE AS $BaSO_4$

Effect upon Analysis	Nature of Interference
Low results	1. Excessive amounts of mineral acid present. (Solubility of Ba SO_4 is appreciably greater in strong acid media.)
	2. Coprecipitation of sulfuric acid. (Note that this is a source of error in a gravimetric determination of sulfate but not of barium since this H_2SO_4 is driven off during ignition.)
	3. Coprecipitation of alkali metal and calcium ions. (Sulfates of these ions weigh less than the equivalent amount of $BaSO_4$ which should have formed.)
	4. Coprecipitation of ammonium ion. (Ammonium sulfate is volatilized upon ignition of the precipitate.)
	5. Coprecipitation of iron as a basic iron (III) sulfate.
	6. Partial reduction of $BaSO_4$ to BaS if filter paper is charred too rapidly.
	7. Presence of trivalent chromium. [May not achieve complete precipitation of $BaSO_4$ owing to formation of soluble complex sulfate of chromium (III).]
High results	1. Absence of mineral acid. (The slightly soluble carbonate or phosphate of barium can precipitate under these conditions.)
	2. Coprecipitation of barium chloride.
	3. Coprecipitation of anions, particularly nitrate and chlorate as barium salts.