



Oznaczanie siarczanów
Determination of Sulphate

MS *Spektrum*

The sulphate present in the sample is dissolved in water and precipitated out of solution using an excess of Barium chloride. The precipitate is removed by centrifugation and is then dissolved in Ammonium Ethylenediaminetetraacetate. The Ba²⁺ complex formed is then aspirated directly into the flame and the sulphate content of the sample calculated from the Barium concentration recorded. Samples must be free of anions that produce insoluble Barium salts other than sulphate.

Equipment Required:

1. A BWB Flame Photometer.
2. An accurate balance weighing to +/-0.0005g.
3. Centrifuge

Reagents:

1. Sodium sulphate (Reagent)
2. Concentrated hydrochloric acid
3. Barium chloride solution (0.1% W/V)
4. Ammonium Ethylenediaminetetraacetate solution: Dissolve 5g of Ethylenediaminetetraacetic acid in 100ml deionised water, add 50ml of ammonia solution (S.G. 0.88) and dilute to 500ml with deionised water.

Preparation of Stock Standard:

Dissolve 0.6713g Sodium sulphate (Reagent) in deionised water and dilute to 100mls. This is 2000 ppm SO₄²⁻ stock standard.

Method:

1. Accurately weigh a sample containing not more than 0.2g of sulphate. Dissolve in deionised water and transfer the solution to a 100ml volu-

metric flask.

2. Add 10ml of concentrated HCl. Mix thoroughly and dilute to the mark with deionised water.
3. Pipette 5ml of the sample solution into a 10ml calibrated centrifuge tube and add 5ml 0.1% BaCl₂. Mix the solution well and spin in a centrifuge for three minutes at 4000 rpm.
4. Discard the supernatant liquid.
5. Re-suspend the pellet in 10ml of deionised water and spin again for 3 minutes at 4000rpm.
6. Discard the supernatant liquid.
7. Add Ammonium Ethylenediaminetetraacetate solution and stir until the pellet completely dissolves. Use gentle heat if necessary.
8. Dilute exactly up to the 10ml calibration mark with the Ammonium Ethylenediaminetetraacetate solution.
9. Subject a 5ml portion of the stock sulphate standard to identical treatment (i.e. Stages 1-9). This yields a final solution containing 1000 ppm SO₄²⁻
10. Using the Ammonium Ethylenediaminetetraacetate solution as diluent prepare additional standards of 600, 300 and 100 ppm SO₄²⁻ from the stock standard.
11. Using the Barium channel, aspirate the Standards and Blank, entering their values when prompted.
12. Aspirate the sample into the flame and take reading from the Barium channel.

Calculation:

To obtain the mass of sulphate in the original sample in mg divide the sample sulphate concentration from the graph by 5.



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