

TURBIDIMETRIC SULFATE DETERMINATION IN EFFLUENTS

Principle of method

In acidic solutions sulphate ions are precipitated with barium chloride to give barium sulphate crystals of uniform size. Light absorbed with a spectrophotometer at 420 nm is measured.

Reagents

Barium chloride crystals $BaCl_2 \cdot 2H_2O$.: To remain between -20+30 mesh sieves.

Conditioning reagent.: 75 g NaCl is dissolved in 300 mL of water, and 30 mL of concentrated HCl (36% w/w), 100 mL of 95% propan-2-ol and 50 mL of glycerol are added and mixed well.

Standard sulphate solution.: 0.1479 g of anhydrous sodium sulphate is dissolved in water and made up to 1 litre. $1.0 \text{ mL} \equiv 100 \mu\text{g SO}_4^{2-}$.

Instrumentation

UV-VIS Spectrophotometer or nephelometer

Determination of sulphate

Appropriate quantity of the sample (containing not more than 4 mg of sulphate), filtered if necessary, is measured into 250-mL conical flask and made up to 100 mL with water. 5.0 mL conditioning reagent is added (use magnetic stirrer at a constant speed). 0.5 g barium chloride crystals are added and continue to stir exactly one minute at a constant speed. After one minute, measure the optical density by means of a spectrophotometer at a wavelength of 380 to 425 nm using 40 to 50 mm cells with water in the reference cell. Take readings at intervals of 30 seconds over a period of 4 minutes, and record the maximum reading.

If absorption due to either colloidal matter or colour observed with the blank is high compared to the absorption observed after barium chloride addition, the method is unsuitable.

Temperature, mixing rate and standing time should be kept as constant as possible in both standards and samples. The calibration curve is generally nonlinear, and points of interest should be checked with each series of samples.

A nephelometer preferably may be substituted for the spectrophotometer.

Calibration Graph

Measure appropriate amounts of sulphate standard solution, covering the range 0 to 4.0 mg of SO_4^{2-} , into a series of 100-mL beakers and proceed as for the test solution. Measure the absorbances of solutions, and construct a graph relating the absorbances to the microgrammes of SO_4^{2-} .

Evaluation of the data

From a previously prepared calibration graph, read the number of milli(micro)grammes of sulphate equivalent to the observed absorbances of the blank and test solutions and calculate the amount of sulphate in the sample