

DETERMINATION OF SULPHIDE IN WATER

This method is applicable in essentially all waters including most industrial waste waters.

Equipment Required

1. EDT DR359TX Ion Meter or pH meter with mV scale
2. EDT directION Sulphide Combination ion selective electrode(3225)

Reagents

1. Sulphide anti-oxidant buffer, (SAOB)
2. Sodium sulphide standards

Standard Preparation:

Sulphide Anti-Oxidant Buffer

To approximately 600 ml of distilled water in a 1000 ml beaker, add 200 ml of 10M NaOH (or 80g reagent grade pellets), 35 grams of ascorbic acid and 67 grams of disodium EDTA. Stir until everything dissolves and transfer the solution to a 1000 ml volumetric flask. Dilute to the mark with distilled water.

Freshly prepared SAOB, when stored in a tightly stoppered bottle, has a shelf life of approximately two weeks, if opened frequently. When oxidised, the solution turns dark brown and should be discarded. Light brown solutions are still usable.

Sodium Sulphide Standards

Sulphide standardising solutions are prepared from reagent grade sodium sulphide hydrate, $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$. It is advised that all sulphide solution preparation and measurement should be performed in a hood, to avoid breathing noxious fumes.

Precise standards cannot be prepared by weighing the salt because of the large and variable hydration of water. Instead, prepare saturated Na_2S solution by adding approximately 100 gms of the $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ hydrate to approximately 100 ml of water, shake well, stopper securely, and allow to stand, at least overnight.

To prepare a stock sulphide solution, pipette 1 ml of the saturated solution described above into 50 ml of SAOB and dilute to 100 ml volume.

Standardisation

The concentration of stock sulphide solution must be determined by electrode titration before constructing a calibration curve. Use a titrant of known concentration of either 0.1M lead perchlorate or 0.1M cadmium nitrate.

Take 50 ml of stock sulphide solution, add this to 25 ml SAOB and 25 ml H_2O , and titrate using the sulphide electrode, and either of the titrants mentioned previously

Calculation

The concentration of stock sulphide solution in mg/litre (C) is given by:

$$C = (\text{ml of } 0.1\text{M lead or cadmium solution}) \times 64.$$

Each day, prepare four calibration standards using 100 ml volumetric flasks as follows:

"A" 5.00 ml of "sulphide stock" 45 ml of SAOB (use graduated cylinder) water to volume

"B" 1.00 ml of "sulphide stock" 50 ml of SAOB water to volume

"C" 2.00 ml of calibration standard "A" 50 ml of SAOB . water to volume

"D" 1.00 ml of calibration standard "A" 50 ml of SAOB water to volume

The concentration of the calibrating standard is calculated from the concentration of the sulphide stock, as determined by titration.

If the stock concentration is C mg/litre, then the calibration standards have the following concentrations:

$$\text{"A"} = 0.05C$$

$$\text{"B"} = 0.01C$$

$$\text{"C"} = 0.001C$$

$$\text{"D"} = 0.0005C$$

Sample Preparation.

Samples are treated prior to analysis with sulphide anti-oxidant buffer.

Samples should be taken with a minimum of aeration to avoid air oxidation of sulphide or loss of volatile hydrogen sulphide from the sample. Samples can be preserved by adding 0.2 ml (4 drops) of 2M zinc acetate (equivalent to 128 mg/litre S⁼) and 0.05 ml (1 drop) of 6M sodium hydroxide to a 100 ml bottle, filling it completely with the sample, and stoppering it with no air bubbles trapped under the stopper. If the concentration of sulphide is greater than approximately 100 mg/litre, the amounts of both reagents should be increased.

Samples which have been "preserved" with zinc can be measured without special treatment, since SAOB contains EDTA to redissolve the zinc and free the sulphide. The entire sample is used for analysis, and since the results will be given as mg. sulphide per litre, the sample volume must be known.

Method

Prepare a calibration curve by immersing the electrode pair in each of the calibration standards, beginning with the weakest one, and record the stable millivolt potential reading developed by each one. Construct a graph using this data, using semilog paper and placing the sulphide concentration scale on the logarithmic scale and the mV reading on the linear scale.

When using the DR359TX the meter will automatically set the standard curve and read the sample concentration in direct units.

Pipette the sample into an equal volume of SAOB, stir thoroughly without vortex, and allow to stand for 3-5 minutes. Place the electrodes in the solution, record the stable electrode potential and determine the sulphide concentration of the sample from the calibration curve.

Between samples, rinse the electrodes with deionised water, blot dry and immerse them in a "blank" solution of 50 ml SAOB plus 50 ml deionised water.

Expected Range

Samples containing 0.1 to 3200 mg/litre of sulphide may be analysed by this method. The concentration range may be extended by dilution of an appropriate aliquot.

Reference

Annual Book of ASTM Standards, Part 31.