

- 5.3 Phenylarsine oxide 0.0250 N: commercially available.
- 5.4 Starch indicator: commercially available.
- 5.5 Procedure for standardization (see Residual Chlorine-iodometric titration Method 330.3, section 5.15).

6.0 Procedure

6.1 Unprecipitated sample

- 6.1.1 Place a known amount of standard iodine solution (5.2) into a 500 mL flask. The amount should be estimated to be in excess of the amount of sulfide expected.
- 6.1.2 Add distilled water, if necessary, to bring the volume to approximately 20 mL
- 6.1.3 Add 2 mL of 6 N HCl (5.1).
- 6.1.4 Pipet 200 mL of sample into the flask, keeping the tip of the pipet below the surface of the sample.
- 6.1.5 If the iodine color disappears, add more iodine until the color remains. Record the total number of milliliters of standard iodine used in performing steps 6.1.1 and 6.1.5.
- 6.1.6 Titrate with the reducing solution (0.0250 N sodium thiosulfate or 0.0250 N phenylarsine oxide solution (5.3)) using a starch indicator (5.4) until the blue color disappears. Record the number of milliliters used.

6.2 Precipitated samples

- 6.2.1 Add the reagents to the sample in the original bottle. Perform steps 6.1.1, 6.1.3, 6.1.5, and 6.1.6.

6.3 Dewatered samples

- 6.3.1 Return the glass fibre filter paper which contains the sample to the original bottle. Add 200 mL distilled water. Perform steps 6.1.1, 6.1.3, 6.1.5, and 6.1.6.
- 6.3.2 The calculations (7) should be based on the volume of original sample put through the filter.

7.0 Calculations

- 7.1 One mL of 0.0250 N standard iodine solution (5.2) reacts with 0.4 mg of sulfide present in the titration vessel.
- 7.2 Use the formula

$$\text{mg/L sulfide} = \frac{400(A - B)}{\text{mL sample}}$$

where:

A = mL of 0.0250 N standard iodine solution (5.2)

B = mL of 0.0250 N standard reducing sodium thiosulfate or phenylarsine oxide) solution (5.3).

8.0 Precision and Accuracy

8.1 Precision and accuracy for this method have not been determined.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 505, Method 428D, (1975).