OIV-MA-AS322-06 Copper

Type IV method

1. Principle

The method is based on the use of atomic absorption spectrophotometry.

2. Apparatus

2.1. Platinum dish.

2.2. Atomic absorption spectrophotometer.

2.3. Copper hollow cathode lamp.

2.4. Gas supplies: air–acetylene or nitrous oxide/acetylene.

3. Reagents

3.1. Metallic copper.

3.2. Nitric acid (ρ_{20} = 1.38 g/mL), 65%.

3.3. Nitric acid (3.2), diluted 1/2 (*v/v*) with water.

3.4. Solution containing 1g of copper per L.

Use of a standard commercial copper solution is preferred. Alternatively this solution may be prepared by weighing 1.000 g of metallic copper and transferring it without loss to a 1000 mL volumetric flask. Add just enough dilute nitric acid to dissolve the metal, add 10 mL of concentrated nitric acid and make up to the mark with double distilled water.

3.5. Solution containing copper at 100 mg/L

Transfer 10 mL, of the 1 g/L solution 3.4. into a 100 mL volumetric flask, and make up to the mark with double-distilled water.

3.6. Double-distilled water

4. Procedure

1. Preparation of sample and determination of copper

Place 20 mL sample in a 100 mL volumetric flask and make up to 100 mL with double-distilled water. Modify the dilution if necessary to obtain a response within the dynamic range of the detector.

Measure the absorbance at 324.8 nm. Set the zero with double distilled water.

4.2. Constructing a standard curve

Pipette 0.5, 1 and 2 mL of copper solution into each of three 100 mL volumetric flasks and make to the volume with double distilled water; the solutions contain 0.5, 1 and 2 mg of copper per liter respectively. Measure the absorbance of standard solutions and the sample prepared in and repeat each measurement. Plot a graph showing the variation in absorbance as a function of the copper concentration in the standard solutions.

5. Expression of results

5.1. Method of calculation

Using the measured absorbance of the samples read off the concentration C in mg/L from the calibration curve.

If F is the dilution factor, the concentration of the copper present is given in milligrams per liter by:

$$F \times C$$

It is quoted to two decimal places.

Notes:

a) Select a sample dilution appropriate to the sensitivity of the apparatus to be used and the concentration of the copper present in the sample.

b) Proceed as follows when very low copper concentrations are expected in the sample to be analyzed: Place 100 mL of the sample in a platinum dish and evaporate on a water bath at 100 □C until it becomes syrupy. Add 2.5 mL of concentrated nitric acid drop wise, covering the bottom of the dish completely. Carefully ash the residue on an electric hotplate or over a low flame; then place the dish in a muffle furnace set at 500□ ± 25□C and leave for about one hour. After coo1ing, moisten the ash with 1 mL of concentrated nitric acid while crushing it with a glass rod; allow the mixture to evaporate and ash again as before. Place the dish in the muffle furnace again for 15 min; repeat the treatment with nitric acid at least three times. Dissolve the ash by adding 1 mL of concentrated nitric acid and 2 mL of double distilled water to the dish and transfer to a 10 mL flask. Wash the dish three times using 2 mL of double distilled water each time. Finally, make to volume with double distilled water. Proceed to analyze the sample as in 4.1 but use 10 mL of solution. Take into account the change in dilution factor when calculating the results.