SILICA METHOD 2

Using 4-Amino-3-Hydroxynaphthalene-1-Sulphonic Acid

PRINCIPLE OF THE METHOD

Silicic acid reacts with ammonium molybdate in acid solution to form silicomolybdate, and this is reduced with 4-amino-3-hydroxynaphthalene-1-sulphonic acid to produce molybdenum blue. Tartaric acid is added to suppress interference by phosphates. The intensity of the blue colour, which is proportional to the concentration of Silica, is measured by comparison with Lovibond permanent colour glass standards.

REAGENTS REQUIRED

1. **Sulphuric Acid/Molybdate Reagent**. To 75g. of ammonium molybdate ($(NH_4)_6Mo_7O_{24}.4H_2O$) dissolved in silica-free deionised water, add 322ml. of 5M sulphuric acid and make up to 1 litre.

2. **Tartaric Acid Reagent**. Dissolve 10g. of tartaric acid ($C_4H_6O_6$) in 100ml.of silica-free water.

3. **4-Amino-3-Hydroxynaphthalene-1-Sulphonic Acid Reagent.**

   (a) Dissolve 90g. of sodium metabisulphite ($Na_2S_2O_5$) in 800ml. of silica-free deionised water.

   (b) Dissolve 14g. of sodium sulphite hydrated ($Na_2SO_37H_2O$) in approximately 100ml. of silica-free deionised water.

To solution (b) add 1.5g. of 4-amino-3-hydroxynaphthalene-1-sulphonic acid ($NH_2C_{10}H_4$ (OH) (SO_3H)) mix until dissolved and add to solution (a). Make up total volume to 1 litre.

All chemicals used in the preparation of reagents should be of analytical reagent quality.

THE STANDARD LOVIBOND COMPARATOR DISC 3/140 and NESSLERISER DISC NV

Disc 3/140 covers the range 0.1 to 1.0mg./l. Silica ($SiO_{2)}$, in steps of 0.1 omitting the 0.9mg./l. and is used with 40mm. cells.

Disc NV covers the range 0.2 to 1.0mg./l. Silica SiO_2, in steps of 0.1mg./l. and is used with 50ml. Nessler cylinders.

METHOD

Comparator Disc 3/140

1. Adjust the temperature of the sample to 20°C to 30°C (see note 1) then fill a 40mm. moulded cell to the 20ml. mark.

2. Add 2ml. of sulphuric acid/molybdate (Reagent 1) and mix. After 5 minutes add 4ml. of tartaric acid (Reagent 2) and 1ml. of 4-amino-3-hydroxynaphthalene-1-sulphonic acid (Reagent 3).

3. Prepare a reagent "blank" using deionised water instead of test sample with the amounts of reagents as given above.

4. Allow to stand for twenty minutes, making quite certain that the temperature does not fall below 20°C. Place this 40mm. cell in the right-hand compartment of the Comparator and a similar cell containing the reagent "blank" in the left-hand compartment.

5. Match the colour of the solution against the colours in the disc using a standard source of white light such as the

Lovibond Daylight 2000 Unit, or failing this, North daylight (not fluorescent lighting). The figure displayed in the bottom right hand corner of the Comparator is the Silica concentration in mg./l. as SiO_2.

Nessleriser Disc NV

1. Follow the method as for disc 3/140 as described above, but use a 50ml. sample in a 50ml. Nessler cylinder instead of a 20ml. sample in 40mm. cell.

2. The resulting colour is matched against the disc NV in a Nessleriser 2150.

NOTES

1. It is important that the test should be carried out at between 20°C and 30°C, since it has been found that a temperature under 20°C the reaction does not proceed to completion and low results are obtained.

2. It must be emphasized that the readings obtained by means of the Lovibond Nessleriser and disc are only accurate provided that Nessler cylinders are used which conform to the specification employed when the discs were calibrated, namely that the 50ml. calibration mark is at a height of 113 ± 3mm. measured internally.

REVISION HISTORY

Date	Change Note	Issue
20/06/02	36/460	2
03/05/05	CA243	3