

SILICA METHOD 1

Using Ammonium Molybdate

PRINCIPLE OF THE METHOD

The colour standards are designed to match the colours produced by the addition of ammonium molybdate and sulphuric acid to solutions containing silicon. In this test the concentration of the sulphuric acid present in the mixture is important and the presence of either too little or too much free acid results in diminution of the intensity of the yellow colour due to silicon. It is essential, therefore, when carrying out the test to adhere strictly to the conditions described below, under which the colour glasses have been standardised. In particular, the test **must** be carried out within the stated temperature range.

REAGENTS REQUIRED

EITHER

1. A 10% w/v Aqueous Solution of Ammonium Molybdate ((NH₄)₆Mo₇O₂₄·4H₂O) (Analytical Reagent Grade).
2. 1M Sulphuric Acid (H₂SO₄).
3. Mix 1 volume of the Ammonium Molybdate Solution with 2 volumes of the Sulphuric Acid

OR

- 1 Carefully add 6ml. conc. sulphuric acid to about 60ml. water; cool and add 4.0g. ammonium molybdate, stir to dissolve. Make up to 120ml., mix well . Transfer 100ml. to an amber glass bottle.

THE STANDARD LOVIBOND COMPARATOR DISC 3/13 and NESSLERISER DISCS NN AND NN SPECIAL

Disc 3/13 covers the range from 2.5 to 25.0 (in steps of 2.5 up to 20mg./l.) of silicon, as silica SiO₂, and is designed for use with a 40mm. cell.

Disc NN covers the range 1, 2, 4, 6, 8, 10, 12, 16 and 20mg./l. sample of silicon as Silica (SiO₂), based on a 50ml. sample.

Disc NN Special contains only two standards, 2 and 3mg./l. of SiO₂. This disc has been prepared for use in the control of 'jet-boost' water for aircraft.

METHOD

Comparator

1. Fill one of the 40mm. glass cells with deionised water and place in the left-hand compartment of the Comparator. If the solution under test is not colourless, the cell should be filled with the solution instead of with deionised water.
2. In a suitable flask place 25ml. of the solution under examination, at 25° to 35°C*, add 3ml. of the reagent, mix, allow to stand for 10 minutes and then pour into the other 40mm. cell and place in the right-hand compartment of the instrument.
3. Hold the Comparator facing a standard source of white light such as the Lovibond Daylight 2000 unit or, failing this, North daylight and compare the colour produced with the colours in the standard disc, rotating the latter until a colour match is obtained.

4. Should the colour in the test solution be deeper than the standard colour glasses, a fresh test should be carried out using a smaller quantity of the solution under examination and diluting to 25ml. with deionised water before adding the reagents, due correction being made to the result obtained. The value displayed is the concentration of Silica in mg./l..

Nessleriser

1. Fill one of the Nessler cylinders to the 50ml. mark with deionised water and place in the left-hand compartment of the Nessleriser. If the solution under test is not colourless, the Nessler cylinder should be filled with the solution instead of deionised water.
2. Fill the other Nessler cylinder to the 50ml. mark with the solution under examination at 25° to 30°C*, add 6ml. of the reagent, mix, allow to stand for ten minutes and then place in the right-hand compartment of the instrument.
3. Stand the Nessleriser before a standard source of white light as described for the Comparator above – and compare the colour produced with the colours in the standard disc, rotating the disc until a colour match is obtained. The value displayed is the concentration of Silica in mg./l..
4. Should the colour in the test solution be deeper than the standard colour glasses, a fresh test should be carried out using a smaller quantity of the solution under examination and diluting to 50ml. with deionised water before adding the reagents. Due correction must be made for the dilution when calculating the result.

* It is important that the test should be carried out at the temperature stated.

NOTES

1. Most colourless salts, even when present in relatively large quantities, are without influence upon the colour produced in the test, provided the concentration of free acid is not unduly disturbed. Phosphates, however, must be absent, since they respond to the test and yield a yellow colour similar to that produced by silica.

British Standard 1427:1962 gives a method to prevent interference from phosphate in the concentrations usually present in water. This uses trisodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$) 30% w/v in water. After adding the ammonium molybdate reagent and standing for 10 minutes as in the standard method, add to both the blank and the test 1ml. citrate in the case of the Comparator test and 2ml. in the case of the Nessleriser test. Mix and match.

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 $113 \pm 3\text{mm}$. measured internally.

REVISION HISTORY

Date	Change Note	Issue
20/6/02	36/460	2
13/04/05	CA243	3
13/11/06	JC109	4