

NITRITE METHOD 1

Using Cleve's Acid

PRINCIPLE OF METHOD

The well known Griess test, as modified by Ilosvay, depends on the diazotisation of sulphanilic acid by nitrous acid. The compound thus formed is then coupled with 1-naphthylamine-7-sulphonic acid to produce a red azo dye, the colour of which is measured by comparison with a series of Lovibond permanent colour glass standards.

REAGENTS REQUIRED

Various formulae for the reagent have been suggested and experiment has shown that the colours produced by these vary slightly; it is therefore important to employ a reagent corresponding with that used for standardizing the colours. The following formulae have been adopted.

1. **Sulphanilic Acid Solution.** Dissolve 0.5g. of sulphanilic acid ($\text{NH}_2\text{C}_6\text{H}_4\text{SO}_3\text{H}$) in 30ml. of glacial acetic acid, (CH_3COOH) and add 120ml. of deionised water. Filter, if necessary. Store in the dark.
2. **Naphthylamine Sulphonic Acid.** Dissolve 0.2g. of Cleve's acid (1-naphthylamine-7-sulphonic acid ($\text{H}_2\text{N}\cdot\text{C}_{10}\text{H}_6\cdot\text{SO}_3\text{H}$)) in 30ml. of glacial acetic acid, add 120ml. of deionised water and filter, if necessary. Store in dark.

Should either of these reagent solutions become coloured on storage, shake with a small quantity of zinc dust and refilter.

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

THE STANDARD LOVIBOND NESSLERISER DISC NJ

The disc covers the range 0.05 μg . to 1 μg . of nitrogen (N) present as nitrite, in the following steps:-0.05, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.8, 1.0 μg ., equivalent to 0.001 to 0.02mg./l. if using a 50ml. sample.

To convert from nitrogen to nitrite ion (NO_2) multiply the result by 3.286.

METHOD

1. Fill one of the Nessleriser cylinders to the 50ml. mark with deionised water and place in the left hand compartment of the Nessleriser.
2. Fill the other Nessleriser cylinder to the mark with the solution under examination, at 20°C. Add 2ml. of reagent 1 and 2ml. of reagent 2, mix, and allow to stand for 30 ± 5 minutes, then place in the right hand compartment of the instrument.
3. Stand the Nessleriser before a standard source of white light such as the Lovibond Daylight 2000 Unit or, failing this, North daylight and compare the colour produced in the test solution with the colours in the disc, rotating the disc until a colour match is obtained. 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 water under examination and diluting to 50ml. with deionised water before adding the reagent.
4. The sample when tested must be at a temperature of 20°C.
5. The concentration as N = $\frac{\text{Disc Reading}}{\text{Volume of Sample}}$ mg./l.

NOTE

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

REVISION HISTORY

Date	Change Note	Issue
06/11/02	36/460	2
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