

QUANTITATIVE ANALYSIS OF NITRITE (AS NO₂) IN CLEANERS**Application**

This method describes two test procedures for determining the percent nitrite in a cleaner. The procedures are: distillation method by conversion to NH₃ (preferred) and the permanganate method.

Apparatus Required

Analytical Balance - Having a precision of +/- 0.1 mg

Hot Plate

Distillation Apparatus

Titration Apparatus

Kjeldahl Distillation Flask - 500 mL

Erlenmeyer Flask - 500 mL

Graduated Cylinder - 100 mL

Materials Required

Mossy Zinc

Reagent grade

Ferrous Sulfate

Reagent grade

Sodium Hydroxide

Reagent grade. CAUTION: Corrosive

Distilled or Deionized Water

10 ppm max total dissolved solids (ASTM D 1888)

Date	Action	Revisions
2001 01 22	Revised	Editorial – no technical change A. Cockman
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Page 1 of 8

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Kerosene, Silicone or Strontium Chloride

Refer to Note "A" in the Procedure. CAUTION: Kerosene has a low flash point; strontium chloride is toxic.

Potassium Permanganate

Reagent grade. CAUTION: Dangerous fire and explosion hazard.

Solutions Required

Hydrochloric Acid Solution - 0.5 N

Sodium Hydroxide Solution - 30 % conc and 0.5 N

Methyl Red

Refer to FLTM AZ 102-01

Potassium Permanganate - 0.1 N (KMnO_4)

Ferrous Sulfate - 0.1 N (FeSO_4)

Sulfuric Acid - 50 %

Conditioning and Test Conditions

All test values indicated herein are based on material conditioned in a controlled atmosphere of 23 $\pm$ 2 $^{\circ}\text{C}$ and 50 $\pm$ 5 % relative humidity for not less than 24 h prior to testing and tested under the same conditions unless otherwise specified.

Procedure for Distillation Method

The Distillation Method by Conversion to NH_3 is preferred.

1. Place a 0.5 to 1.0 g aliquot of the prepared 1 % solution of cleaner into a 500 mL Kjeldahl distillation flask.
2. A receiving flask containing methyl red indicator and 25 mL of 0.5 N HCl must be in place prior to starting the distillation.
3. The end of the delivery tube must be immersed in the HCl solution during the distillation.
4. Dilute the sample in the distilling flask to 250 mL with distilled water.
5. Add to this 5.0 g of mossy zinc and 2.0 g of ferrous sulfate.

6. Add 50 mL of a 30 % sodium hydroxide solution being careful not to touch the sides or lip of the flask.
7. Agitate this mixture by swirling.
8. Distillation should then be made for 3/4 to 1 hour until approximately half of the volume of liquid in the distilling flask has been recovered in the receiving flask.
9. Wash the condenser and tube with distilled water into the receiving flask.
10. Titrate the excess 0.5 N HCl with 0.5 N NaOH.
11. Calculate the amount of NO₂ as follows:

$$\% \text{ NO}_2 = \frac{A - B}{C} \times 0.023 \times 100$$

Where: A = mL of 0.5 N HCl
 B = mL of 0.5 N NaOH
 C = Weight of sample, grams

Note "A": On occasion, excessive foaming may take place. Addition of kerosene, silicone or strontium chloride to the distillation flask normally suppresses this foaming tendency.

General Notes on the Distillation Method

Nitrite, nitrate, ammonia and ammonium are quantitatively detected by this method. If two or more of these species are present, the result must be interpreted as total nitrogen. This may be useful information, since the contribution from ammonia, ammonium and nitrite can be determined by alternate methods.

Some organic compounds also partly distill or give off ammonia on distillation with strong alkali. Mono, di-, and tri- ethanolamine, EDTA and other nitrogen-bearing compounds will contribute to the result but not in a quantitative manner. The presence of these nitrogen-bearing compounds is therefore undesirable.

For samples containing a low concentration of nitrite, better accuracy can be obtained by analyzing a larger sample.

If the observed titration is less than 0.5 mL, the distillation must be repeated with a larger sample. This is necessary to ensure that nitrite is actually present in the sample being analyzed.