

**QUANTITATIVE ANALYSIS OF  
COMBINED AND FREE AMMONIA IN AMMONIUM SALTS IN CLEANERS****Application**

This test method is used to determine the percent of combined and free ammonia in ammonium salts found in cleaners. The procedure is intended to be a simplified version of the applicable ASTM standard (e.g., D 501 or D 800) as well as being more appropriate for dealing with the many formulations of cleaners that are commonly encountered in plant operations.

**Apparatus Required****Hot Plate****Distillation Apparatus****Distilling Flask**

1,000 mL; similar to a Kjeldahl or a boiling flask with ground glass joints.

**Buret**

50 mL

**Flask**

500 mL

**Boiling Chips**

The above apparatus is available from any reputable laboratory equipment supply house.

**Materials Required****Distilled or Deionized Water**

10 ppm max total dissolved solids (ASTM D 1888)

**Hydrochloric Acid**

Concentrated, Reagent grade; CAUTION: Corrosive

| Date       | Action  | Revisions                                  |
|------------|---------|--------------------------------------------|
| 2000 08 31 | Revised | Editorial – no technical change A. Cockman |
| 2000 03 23 |         | Editorial – no technical change A. Cockman |
| 1991 09 24 |         |                                            |

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**Sodium Hydroxide**

Reagent grade; CAUTION: Corrosive

**Red Litmus Paper**

**Solutions Required**

**Sodium Hydroxide Solutions**

- 50 % concentration
- 0.5 N - Refer to FLTM AZ 102-01

**Methyl Red Indicator**

Refer to FLTM AZ 102-01

**Conditioning and Test Conditions**

All test values indicated herein are based on material conditioned in a controlled atmosphere of 23 +/- 2 °C and 50 +/- 5 % relative humidity for not less than 24 h prior to testing and tested under the same conditions unless otherwise specified.

**Procedure**

**I. Combined Ammonia**

1. Take a 1 to 2 g aliquot of the prepared 1 % solution in a 1 000 mL distilling flask.
2. Add 250 mL of distilled water to the Kjeldahl flask.
3. Add boiling chips to this flask.
4. Buret 40 mL of 0.5 N hydrochloric acid into a 500 mL receiving flask.
5. Add five drops of methyl red indicator to this receiving flask.
6. Connect the receiving flask to the condenser, making sure that the glass outlet tube is immersed in the acid solution.
7. Add 50 mL of a 50 % sodium hydroxide solution to the distilling flask.

Note: Be sure that the sodium hydroxide does not come in contact with the ground glass joints.

8. Immediately connect the distilling flask to the water condenser which has ground glass joints by means of a glass connecting arm which is also equipped with ground glass joints.