

	TRACE-PM 10.16 Tests for Ions	
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	Document Manager: Cheryl Lozen	Approved By: Jeffrey Nye

10.16 Tests for Ions

10.16.1 Test for Ions

This procedure represents various tests that may be performed to screen or identify the presence of a particular ion. These tests are not included in any of the procedures which have been previously described in this manual.

The following is a list of tests described:

- Test A - Ammonia (NH₃)

10.16.2 Safety Considerations

Standard Laboratory Precautions

10.16.3 Preparations

10.16.3.1

Materials for Test A1

- Hexane
- Hydrochloric acid (HCl)
- Pasteur Pipet
- Centrifuge

10.16.3.2

Materials for Test A2

- 10% Hydrochloric Acid
- Pasteur Pipet
- Acetone
- Centrifuge

10.16.3.3

Materials for Test A3

- Macherey-Nagel Ammonium Test Paper Cat. #90722.

Supplied by:

Gallard-Schlesinger, Industries, Inc.
584 Mineola Ave., Carle Place, N.Y.
(516) 333-5600

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10.16.3.4

Materials for Test A4

- Distilled water
- Filter Paper
- Nessler's Reagent

Preparation of Nessler's Reagent:

Dissolve 8.15 grams HgCl_2 in 90ml hot distilled H_2O . With stirring, add to it a hot solution containing 9.96 grams KI in 30ml distilled H_2O . Filter the red HgI_2 ppt with suction and wash twice using 30ml of distilled H_2O each time. Stir the moist product into 8ml of hot distilled H_2O containing 9.79 grams of KI. Heat on hot water bath for 20 minutes. Stir occasionally, allowing about a fourth of the liquid to evaporate. Centrifuge or decant and discard any residue. Put the centrate in an evaporating dish and store over calcium chloride in a desiccator. This will give a moist crystalline mass of tetraiodomercurate (II) dihydrate, $\text{K}_2 \text{HgI}_4 \cdot 2 \text{H}_2\text{O}$. The moist crystalline mass can be dried further by pressing between sheets of filter paper and more desiccation over CaCl_2 . The yellow crystalline product will last for years and is used to prepare Nessler's solution by dissolving a few yellow crystals in about 1ml of 3M KOH just before testing for NH_3 or the ammonium ion. As long as blanks and controls react appropriately the crystalline product can be used to prepare Nessler's solution.

10.16.4 Instrumentation

- Fourier Transform Infrared Spectrometer (FTIR)
- Scanning Electron Microscope with Energy Dispersive X-Ray System (SEM/EDS)

10.16.5 Minimum Standards & Controls

All tests must be performed with blanks to check for contamination and controls to check for correct response of known samples.

10.16.6 Procedure or Analysis

10.16.6.1

Test A1 (ammonia).

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10.16.6.1.1

Place 2ml of hexane in a culture tube, then add 3-4 drops of HCl.

10.16.6.1.2

Shake and centrifuge.

10.16.6.1.3

Remove hexane layer and place into another culture tube.

10.16.6.1.4

Bubble suspected NH_3 gas into the lower part of the tube containing the hexane. Use a syringe with long needle or pipet with a bulb.

10.16.6.1.5

Centrifuge and pour off the hexane. A precipitate should be NH_4Cl .

10.16.6.1.6

Perform one or a combination of the following for identification: FTIR or SEM/EDS.

10.16.6.2

Alternate Test A2 (ammonia)

10.16.6.2.1

Place 1 ml of 10% HCl in culture tube.

10.16.6.2.2

Bubble air sample containing suspected NH_3 into the liquid. Use a syringe with long needle or pipet with a bulb.

10.16.6.2.3

Add acetone \approx 2-3 ml until a precipitate forms.

10.16.6.2.4

Centrifuge and remove liquid.

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10.16.6.2.5

Add additional acetone and pipet out the suspended white residue into a beaker. This residue should be NH_4Cl .

10.16.6.2.6

Perform one or a combination of the following for identification: FTIR or SEM/EDX.

10.16.6.3

Alternate Test A3 (ammonia)

10.16.6.3.1

A commercial ammonium test paper for the detection of ammonium and ammonia is available.

10.16.6.3.2

Follow instructions provided with the test paper.

10.16.6.4

Alternate Test A4 (Ammonium ion or Ammonia vapors)

10.16.6.4.1

Liquid samples

Dilute 1-3 drops of test solution with 0.5 to 2ml distilled water. Add 2-3 drops of Nessler's solution to the diluted sample. A yellow to brown color of OH-Hg-NH-HgI is a positive test.

10.16.6.4.2

Vapor samples--ammonia vapors

10.16.6.4.2.1

Put a sample of the liquid to be tested in a test tube, ensuring that the walls of the test tube above the liquid stay dry. Wet a strip of filter paper with Nessler's solution and suspend the so wetted filter paper above the liquid in the test tube with a cork stopper. Ammonia vapors coming from the liquid will cause the Nessler's reagent on the filter paper to turn brown.

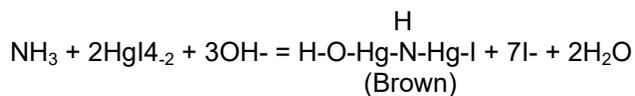
10.16.6.4.2.2

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If the sample is debris in a can or jar, suspend a strip of filter paper wet with Nessler's solution over the debris in the container using the lid to secure the strip above the debris. Allow 1-5 minutes for the vapors to react with the Nessler's reagent on the filter paper. Ammonia vapors coming from the debris will cause the Nessler's reagent on the filter paper to turn brown

10.16.6.4.3

Analytical Reaction of Nessler's Test for Ammonia



10.16.7 References

Nordman, Joseph. Qualitative Testing and Inorganic Chemistry; John Wiley & Sons: New York, NY, 1957; pp 328-330.