

Status: Currently Official on 12-Feb-2025
 Official Date: Official as of 01-Jun-2023
 Document Type: Reagents
 DocId: GUID-5F9A6808-DAE9-497B-AFAB-3FA76284436C_4_en-US
 DOI: https://doi.org/10.31003/USPNF_M6948_04_01
 DOI Ref: dy4cd

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Reagents

1. DEFINITIONS

1.1 BLANK

A blank consists of the same quantities of the same reagents treated in the same manner as the specimen under test.

1.2 CONTROL

A control is a blank to which has been added the limiting quantity of the substance being tested for, or is a specified comparison solution prepared as directed in the particular test.

2. DESCRIPTION OF THE REAGENT ENTRY

See [Figure 1](#).

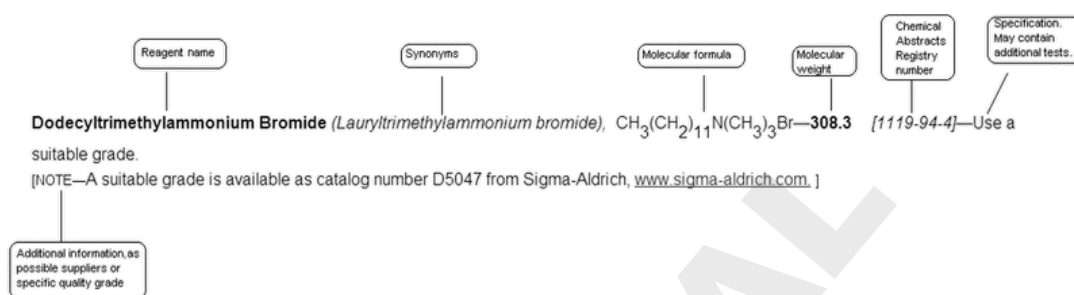


Figure 1. Components of the reagent entry.

3. VISUAL COMPARISONS

For color and turbidity comparisons, proceed as directed in [Visual Comparison \(630\)](#). The tubes used in this procedure are frequently called "Nessler tubes".

In making visual comparisons of the densities of turbid fluids, compensate for difference in color, if necessary, by viewing the turbidity through a column of water, the depth of which is determined by the volume specified in the individual reagent specification. Place the water in color-comparison tubes, and hold one of the tubes above the control tube and the other below the specimen tube.

4. RETAIN THE FILTRATE

"Retain the filtrate" is to be understood, unless otherwise indicated, that the washings of the residue are not to be added to the filtrate obtained.

5. EXPRESSION R_2O_3

The expression R_2O_3 is intended to indicate the residue on ignition from compounds precipitated upon the addition of ammonium hydroxide, such as iron oxide (Fe_2O_3) and aluminum oxide (Al_2O_3).

6. GENERAL TESTS FOR REAGENTS

The following general test methods are provided for the examination of reagents to determine their compliance with the specifications of the individual reagents and are to be used unless it is otherwise directed in such specifications.

6.1 BOILING OR DISTILLING RANGE FOR REAGENTS

Use the following procedure for determining the boiling or distilling range of reagents, unless otherwise directed in the individual specifications:

APPARATUS: Use apparatus similar to that specified for [Distilling Range \(721\)](#), [Method I](#), except that the distilling flask is to be of 250-mL capacity, to have a short neck, and to be connected to the condenser by means of a three-way connecting tube fitted with standard-taper ground joints.

PROCEDURE: Place the distilling flask in an upright position in the perforation in the asbestos board, and connect it to the condenser. Measure 100 mL of the liquid to be tested in a graduated cylinder, and transfer to the boiling flask together with some device to prevent bumping. Use the cylinder as the receiver for the distillate. Insert the thermometer, and heat so as to distill at the rate of 3–5 mL/min. Make a preliminary trial, if necessary, to determine the adjustment for the proper rate of heating. Read the thermometer when about 20 drops have distilled and thereafter at volumes of distillate of 5, 10, 40, 50, 60, 90, and 95 mL. Continue the distillation until the dry point is reached.

The *Boiling or Distilling Range* is the interval between the temperatures when 1 mL and 95 mL, respectively, have distilled.

6.2 AMINO NITROGEN TEST IN REAGENTS

Determine the percentage of loss on drying of the sample in appropriate conditions. Transfer about 500 mg of the sample to a 100-mL beaker. Add 20 mL of water. Adjust the pH potentiometrically with 0.1 N hydrochloric acid or 0.2 N sodium hydroxide to 6.0. Add 10 mL of formaldehyde solution. Titrate the solution potentiometrically with 0.2 N sodium hydroxide to a pH of 9.0. Calculate the percentage of amino nitrogen:

$$\% \text{ amino nitrogen} = \frac{V_{\text{NaOH}} \times N_{\text{NaOH}} \times 14 \times 100 \times f}{\text{mg sample} \times (1.0 - \% \text{ LOD} / 100)}$$

where f is the correction factor obtained in the standardization of 0.2 N sodium hydroxide and %LOD is the percentage of loss on drying.

Change to read:

6.3 ARSENIC IN REAGENTS

Select reagents for this test for a low arsenic content, so that a blank test results in either no stain or one that is barely perceptible.

APPARATUS: Prepare a generator by fitting a 1-hole rubber stopper into a wide-mouth bottle of about 60-mL capacity. Through the perforation insert a vertical exit tube about 12 cm in total length and 1 cm in diameter along the entire upper portion (for about 8 cm) and constricted at its lower extremity to a tube about 4 cm in length and about 5 mm in diameter. The smaller portion of the tube should extend to just slightly below the stopper. Place washed sand or a pledget of purified cotton in the upper portion to about 3 cm from the top of the tube. Moisten the sand or cotton uniformly with lead acetate TS, and remove any excess or adhering droplets of the latter from the walls of the tube. Into the upper end of this tube fit a second glass tube 12 cm in length, with an internal diameter of 2.5–3 mm, by means of a rubber stopper. Just before running the test, place a strip of mercuric bromide test paper (see *Indicator and Test Papers*) in this tube, crimping the upper end of the strip so that it will remain in position about 2 cm above the rubber stopper. Clean and dry the tube thoroughly each time it is used.

STANDARD ARSENIC SOLUTION: Use the *Standard Preparation*, prepared as directed in [▲Arsenic \(211\), Procedures, Procedure 1▲](#) (CN 1-Jun-2023) ·

TEST PREPARATION: Add 1 mL of sulfuric acid to 5 mL of a solution of the chemical substance (1 in 25), unless another quantity is directed in the individual reagent specification. Omit its addition entirely in the case of inorganic acids. Unless especially directed otherwise, add 10 mL of sulfurous acid. Evaporate the liquid in a small beaker, on a steam bath, until it is free from sulfurous acid and has been reduced to about 2 mL in volume. Dilute with water to 5 mL to obtain the *Test preparation*. Substances subjected to special treatments specified in the individual reagent specification may be used directly as the *Test preparation*. [NOTE—Solutions prepared by the dissolving of the chemical substances in dilute acids are not considered to have undergone special treatment.]

STANDARD STAIN: Place in the generator bottle 5 mL of potassium iodide TS, 2.0 mL of *Standard arsenic solution*, 5 mL of acid stannous chloride TS, and 28 mL of water. Add 1.5 g of granulated zinc (in No. 20 powder), and immediately insert the stopper containing the exit tube. Keep the generator bottle immersed in water at 25° during the period of the test to moderate the reaction so that the stain will take the form of a distinctive band to facilitate the comparison of color intensity. When evolution of hydrogen has continued for 1 h, remove the mercuric bromide test paper for comparison. This stain represents 2 µg of arsenic.

PROCEDURE: Pipet into the generator bottle 5 mL of potassium iodide TS and 5 mL of the *Test preparation*, and add 5 mL of acid stannous chloride TS. Set the apparatus aside at room temperature for a period of 10 min, then add 25 mL of water and 1.5 g of granulated zinc (in No. 20 powder), and proceed as directed for the *Standard stain*. Remove the mercuric bromide test paper, and compare the stain upon it with the *Standard stain*: the stain produced by the chemical tested does not exceed the *Standard stain* in length or in intensity of color, indicating NMT 10 parts of arsenic per million parts of the substance being tested. Because light, heat, and moisture cause the stain to fade rapidly, place the papers in clean, dry tubes, and make comparisons promptly.

INTERFERING CHEMICALS: *Antimony*, if present in the substance being tested, produces a gray stain. *Sulfites*, *sulfides*, *thiosulfates*, and other compounds that liberate hydrogen sulfide or sulfur dioxide when treated with sulfuric acid must be oxidized by means of nitric acid and then reduced by means of sulfur dioxide as directed for the *Test preparation* before they are placed in the apparatus. Certain *sulfur compounds*, as well as *phosphine*, give a bright yellow band on the test paper. If sulfur compounds are present, the lead acetate-moistened cotton or sand will darken. In that case, repeat the operation as directed for the *Test preparation* upon a fresh portion of the solution being tested, and use greater care in effecting the complete removal of the sulfurous acid. In testing hypophosphites, observe special care to oxidize completely the solution being tested as directed; otherwise the evolution of phosphine may result in a yellow stain that may be confused with the orange-yellow color produced by arsine. The stain produced by phosphine may be differentiated from that given by arsine by means of moistening it with 6 N ammonium hydroxide. A stain caused by arsine becomes dark when so treated, but a stain produced by phosphine does not materially change in color.

6.4 CHLORIDE IN REAGENTS

STANDARD CHLORIDE SOLUTION: Dissolve 165.0 mg of dried sodium chloride in water to make 1000.0 mL. This solution contains the equivalent of 0.10 mg of chlorine (Cl) in each mL.

PROCEDURE: Neutralize, if alkaline, a solution of the quantity of the reagent indicated in the test in 25 mL of water, or a solution prepared as directed in the test, with nitric acid, litmus paper being used as the indicator, and add 3 mL more of nitric acid. Filter the solution, if necessary, through a filter paper previously washed with water until the paper is free from chloride, and add 1 mL of silver nitrate TS. Mix, and allow to stand for 5 min protected from direct sunlight. Compare the turbidity, if any, with that produced in a control made with the same quantities of the same reagents as in the final test and a volume of *Standard chloride solution* equivalent to the quantity of chloride (Cl) permitted by the test. Adjust the two solutions with water to the same volume before adding the silver nitrate TS, and compare the turbidities.

In *testing barium salts*, neutralize, if alkaline, the solution containing the reagent, with nitric acid, and add only 3 drops more of nitric acid.

Conduct the remainder of the test as described previously.