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Change to read:

0.1 N Tetrabutylammonium Hydroxide in Methanol/Isopropyl Alcohol VS

Dissolve 40 g of tetra-*n*-butylammonium iodide in 90 mL of anhydrous methanol in a glass-stoppered flask. Place in an ice bath, add 20 g of powdered silver oxide, insert the stopper in the flask, and agitate vigorously for 60 min. Centrifuge a few milliliters, and test the supernatant for iodide (see Identification Tests—General ⟨191⟩, Iodide). If the test is positive, add an additional 2 g of silver oxide, and continue to allow to stand for 30 min with intermittent agitation. When all of the iodide has reacted, filter through a fine pore size, sintered-glass funnel. Rinse the flask and the funnel with three 50-mL portions of isopropyl alcohol, adding the rinsings to the filtrate. Dilute with a mixture of three volumes of isopropyl alcohol and one volume of anhydrous methanol to 1000 mL, and flush the solution for 10 min with dry, carbon dioxide-free nitrogen. [Note—If necessary to obtain a clear solution, further small quantities of anhydrous methanol may be added.] Store in a reservoir protected from carbon dioxide and moisture, and discard after 60 days. Alternatively, the solution may be prepared by diluting a suitable volume of commercially available tetrabutylammonium hydroxide solution in methanol with a mixture of four volumes of isopropyl alcohol and one volume of anhydrous methanol.

[Note—If necessary to obtain a clear solution, further small quantities of methanol may be added.]

Standardization

▲See *Volumetric Solutions, 1. Introduction*.

See *Titrimetry* ⟨541⟩.

Standardize by one of the following procedures. [Note—Other standardization procedures may be used. See *Volumetric Solutions, 2. Preparation and Standardization, 2.3 Standardization*.]

Standardization with visual endpoint: ▲(USP 1-Dec-2022) On the day of use, dissolve about 400 mg of primary standard benzoic acid, accurately weighed, in 80 mL of dimethylformamide, add 3 drops of a 1 in 100 solution of thymol blue in dimethylformamide, and titrate to a blue endpoint with the tetrabutylammonium hydroxide solution, delivering the titrant from a buret equipped with a carbon dioxide absorption trap. Perform a blank determination, and make any necessary correction. Each milliliter of 0.1 N tetrabutylammonium hydroxide ▲$(C_4H_9)_4NOH$▲ (USP 1-Dec-2022) is equivalent to 12.21 mg of benzoic acid.

▲

$$N = \frac{\text{mg Benzoic acid}}{122.1 \times [\text{mL(sample)} - \text{mL (blank)}](C_4H_9)_4NOH}$$

Standardization with potentiometric endpoint: On the day of use, dissolve about 100 mg of primary standard benzoic acid, accurately weighed, in 80 mL of alcohol, and titrate with the tetrabutylammonium hydroxide solution to the equivalence point, delivering the titrant from a buret equipped with a carbon dioxide absorption trap. Use a combined pH electrode suitable for non-aqueous titrations. Perform a blank determination, and make any necessary correction. Each milliliter of 0.1 N tetrabutylammonium hydroxide $(C_4H_9)_4NOH$ is equivalent to 12.21 mg of benzoic acid.

$$N = \frac{\text{mg Benzoic acid}}{122.1 \times [\text{mL(sample)} - \text{mL (blank)}](C_4H_9)_4NOH}$$ ▲(USP 1-Dec-2022)

[Note—If this volumetric solution is used in a qualitative application such as pH adjustment, dissolution medium, or diluent, its standardization is not required.]

Topic/Question	Contact	Expert Committee
0.1 N TETRABUTYLAMMONIUM HYDROXIDE IN METHANOL/ISOPROPYL ALCOHOL VS	Margareth R.C. Marques Principal Scientific Liaison	HDQ Headquarters

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