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Change to read:

0.1 N Tetrabutylammonium Hydroxide VS

▲▲ (USP 1-Dec-2022)

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 ▲▲ (USP 1-Dec-2022) [toluene](#), adding the rinsings to the filtrate. Dilute with a mixture of three volumes of ▲▲ (USP 1-Dec-2022) [toluene](#) 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 ▲▲ (USP 1-Dec-2022) [toluene](#) 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 mL of 0.1 N tetrabutylammonium hydroxide ▲▲ (C₄H₉)₄NOH ▲▲ (USP 1-Dec-2022) is equivalent to 12.21 mg of benzoic acid.

$$\text{▲▲ N} = \frac{\text{mg Benzoic acid}}{122.1 \times [\text{ml (sample)} - \text{ml (blank)}] (\text{C}_4\text{H}_9)_4\text{NOH}}$$

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 mL of 0.1 N tetrabutylammonium hydroxide (C₄H₉)₄NOH is equivalent to 12.21 mg of benzoic acid.

$$\text{N} = \frac{\text{mg Benzoic acid}}{122.1 \times [\text{ml (sample)} - \text{ml (blank)}] (\text{C}_4\text{H}_9)_4\text{NOH}} \text{▲▲ (USP 1-Dec-2022)}$$

[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 VS	Margareth R.C. Marques Principal Scientific Liaison	HDQ Headquarters

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