

TITRIMETRIC DETERMINATION OF CO₂ IN ETHANOLAMINES

UOP Method 829-82

SCOPE

This method is for the titrimetric determination of carbon dioxide in ethanolamine solutions used for scrubbing gas streams such as those found in hydrogen plants that contain only carbon dioxide as the acidic contaminant. The presence of hydrogen sulfide or sulfur dioxide will produce high results, since these components will be included in the titration.

OUTLINE OF METHOD

The amine sample is dissolved in anhydrous methanol. The amine/methanol solution is titrated with a standard methanolic sodium hydroxide solution using thymolphthalein as the indicator, and the CO₂ content calculated.

APPARATUS

Balance, torsion or top-loading type, 500 g capacity, capable of weighing to 0.01 g

Beaker, 250-mL

Bottle, dropping, 30-mL capacity

Buret, 50-mL, with Teflon, stopcock, 0.1-mL subdivisions (2 required)

Cylinder, graduated, 100-mL, glass, 1.0-mL subdivisions

Flask, Erlenmeyer, 500-mL

Flask, volumetric, 100-mL and one-liter

Pipet, volumetric transfer, 10-mL

Stirrer, magnetic, with Teflon-coated stirring bar, Sargent-Welch Scientific Co., Cat. No. S-76490, or equivalent

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REAGENTS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available, unless otherwise specified. References to water mean deionized or distilled water.

Bromothymol blue indicator. Dissolve 0.1 g of bromothymol blue in 100 mL of 20% methanol in water.

Methanol, absolute, 99.8% minimum purity

Sodium hydroxide solution, methanolic, 0.5-*M*. Dissolve 21g of sodium hydroxide in 100 mL of water. Quantitatively transfer to a one-liter volumetric flask, cool and dilute to volume with absolute methanol. Standardize the methanolic sodium hydroxide with 0.5-*M* standard sulfuric acid in 100 mL of absolute methanol containing 5 drops of thymolphthalein indicator. Before adding the 0.5-*M* sulfuric acid to the methanol, titrate the methanol with methanolic sodium hydroxide to a light blue color.

Sulfuric acid solution, aqueous, 0.5-*M*. Standardize with tris(hydroxymethyl)aminomethane and use bromothymol blue for the indicator. Titrate to a yellow color.

Thymolphthalein indicator. Dissolve 10 g of thymolphthalein in absolute methanol, transfer to a 100-mL volumetric flask and dilute to volume with absolute methanol.

Tris(hydroxymethyl)aminomethane, ultrapure, acidimetric standard, Sargent-Welch Scientific Co., Cat. No., SC15540-025 GM, or equivalent

PROCEDURE

To a 500-mL Erlenmeyer flask containing a magnetic stirring bar add 100 mL of absolute methanol and 5 drops of thymolphthalein indicator. Place the flask on the magnetic stirrer and start the stirrer. Titrate the methanol with 0.5-*M* methanolic sodium hydroxide to a light blue color. Read the volume added to the nearest 0.1mL. This titration represents the methanol blank. Pipet 10 mL of amine sample (see *NOTE*) into the 500-mL Erlenmeyer flask containing the above solution. Titrate the amine and methanol solution with 0.5-*M* methanolic sodium hydroxide to a light blue color. Read the total volume of titrant used to the nearest 0.1 mL.

CALCULATIONS

Calculate the volume of carbon dioxide in the sample solution at standard conditions (15.6 C and 101.3 kPa) as follows:

$$\text{Cubic feet of carbon dioxide/gal of amine solution} = \frac{3.2(A - B)M}{V}$$

where:

A = total volume of standard methanolic sodium hydroxide added, mL

B = volume of standard methanolic sodium hydroxide for blank, mL

M = molarity of methanolic sodium hydroxide solution

V = sample volume, mL

NOTE

Analysis of samples containing alkaline carbonates or acidic compounds resulting from amine decomposition will be incorrect since these components will effect the titration. To compensate for these effects, the sample can be heated overnight under reflux conditions to remove the majority of the CO₂. Titration of the refluxed sample can then be used along with the titration of the original sample to obtain an approximate CO₂ content.

PRECISION

An estimated standard deviation is not reported since insufficient data are available at present to permit this calculation with at least 4 degrees of freedom.

TIME FOR ANALYSIS

The elapsed time for one analysis is 1.0 hour. The labor requirement is 0.5 hour.

REFERENCE

Method SM-100, The Girdler Corp., Louisville, KY

SUGGESTED SUPPLIER

Sargent-Welch Scientific Co., 7300 N. Linder Ave., Skokie, IL 60077