

DIETHANOLAMINE IN SOLUTIONS BY COLOR INDICATOR TITRATION

UOP Method 824-81

SCOPE

This control method is used for the analysis of diethanolamine (DEA) in solutions used to remove acid gases, such as hydrogen sulfide and carbon dioxide from hydrocarbon streams. The method utilizes an acid-base titration that determines basic constituents, i.e., free amine and amine associated with weak acids. The value obtained is a measure of the potential efficiency of the solution to remove the above gases. The lower limit of detection is less than 1 wt-%.

OUTLINE OF METHOD

The amine sample is diluted with water and titrated with a standard sulfuric acid solution to the methyl red indicator endpoint.

APPARATUS

Beaker, 250-mL

Buret, 50-mL, with Teflon stopcock, 0.1-mL subdivisions

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

Hot plate, adjustable head, Sargent-Welch Scientific Co., Cat. No. S-41000 A, or equivalent

Pipet, serological, calibrated for blow-out, one-milliliter capacity, Sargent-Welch Scientific Co., Cat. No. S-69625-C, or equivalent

Pipet manipulator, Sargent-Welch Scientific Co., Cat. No. S-69793, or equivalent

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

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.

Methyl red indicator solution, 0.1 wt-% concentration. Dissolve 0.1 g of methyl red in 100 g of water.

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

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Sulfuric acid solution, aqueous, 0.05-M, standardized

PROCEDURE

Because of the potential hazards resulting from the liberation of gaseous H₂S during this analysis, arrange the apparatus in an efficient hood. Pipet one milliliter of the amine solution sample into a beaker containing 100 mL of water and a magnetic stirring bar. Remove any remaining sample from the pipet into the water by blowing into the pipet. With viscous samples, rinse the pipet into the beaker with water. Add 3 drops of methyl red indicator solution to the sample solution and turn on the magnetic stirrer. Titrate the sample solution with standard 0.05 M sulfuric acid to a definite pink endpoint. Place the beaker on the hot plate and heat to boiling for one minute. If the solution remains pink, use the first endpoint. If the solution turns yellow, cool the solution and titrate again to a definite pink endpoint. Take the final reading for the total volume of titrant used.

In the titration of samples containing H₂S, if the color of the methyl red indicator is destroyed, more indicator should be added during the titration. A lean (regenerated), as opposed to rich, amine solution should be analyzed whenever possible since the presence of H₂S and CO₂ tends to make the methyl red endpoint less definite.

CALCULATIONS

Calculate the concentration of diethanolamine in the sample solution as follows:

$$\text{Diethanolamine, g/100 ml} = \frac{21.0 \text{ AM}}{V}$$

where:

A = standard sulfuric acid solution, mL

M = molarity of sulfuric acid solution

V = sample volume, mL

$$\text{Diethanolamine, wt - \%} = \frac{21.0 \text{ AM}}{SV}$$

where:

S = specific gravity of sample, 60/60 F (15.6/15.6 C)

A, **M** and **V** are as previously defined

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. It is estimated that duplicate analyses should agree within 0.1 g/100 mL or 0.1 wt-%.

TIME FOR ANALYSES

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

REFERENCE

Method SM-1D, The Girdler Corp., Louisville, KY

SUGGESTED SUPPLIER

Sargent-Welch Scientific Co., 7300 N. Linder Ave., Skokie, IL 60076