



Diethanolamine Content in Solutions by Color Indicator Titration

UOP Method 824-20

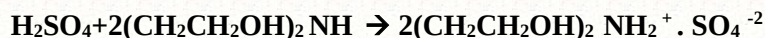
Scope

This method is used for determination of diethanolamine (DEA) content in a fresh & regenerated solutions. DEA is most commonly 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 1 mass%.

The sample should be free from any contamination of caustic materials like sodium or potassium hydroxide and their carbonates and also from basic material like ammonia, which also form salts with sulfuric acid and will impart a positive interference in this method.

Outline of method

The alkanolamine sample is diluted with water and titrated with a standard sulfuric acid solution to the methyl red indicator endpoint. The following reaction takes place during titration. Diethanolamine is a weak base and reaction involves protonation with dilute sulfuric acid and formation of Diethanol ammonium sulfate salt.



Apparatus

References to catalogue numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Beaker, 250-mL, VWR, Cat. Nos. 89001-062,

Buret, 50-mL, with PTFE stopcock, Class A, VWR, Cat. No. 76003-588

Cylinder, graduated, 100-mL, glass, 1.0-mL subdivisions, Class B VWR, Cat. No. 10545-958

Hot plate, Dylatherm, VWR, Cat. No. 12620-978

Pipets, volumetric, Class A, TC and TD, 1-, 5- mL, VWR, Cat. Nos. 02-555-331, and -335, respectively

Pipet filler, rubber, VWR, Cat. No. 10834-142

Stirrer, magnetic, Dylastir, VWR, Cat. No. 12620-974

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. SAFETY DATA SHEETS (SDS) OR EXPERIMENTAL SAFETY DATA SHEETS (ESDS) 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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Stir bar, Teflon-covered VWR, Cat No. 58948-150

Stirring rod, glass, 150-mm length, VWR, Cat. No. 59060-047

Reagents and Materials

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used. References to water mean deionized water.

Methyl red indicator solution, 0.1% (W/V) aqueous solution, VWR, Cat. No. RC5060-32

Sulfuric acid solution, aqueous, 0.1-N standardized, VWR, Cat. No. EM1.09074.1000

Water, deionized

Procedure

The analyst is expected to be familiar with general laboratory practices, the technique of titration, and the equipment being used. Dispose of used supplies and samples in an environmentally safe manner according to applicable regulations.

Because of the potential hazards resulting from the liberation of gaseous H_2S during this analysis, arrange the apparatus and activity in an efficient hood.

1. Pipet one milliliter of the amine solution sample into a beaker containing 100 mL of water and a magnetic stirring bar.
2. Remove any remaining sample from the pipet into the water by blowing into the pipet by pipet filler. With viscous samples, rinse the pipet into the beaker with water.
3. Add 3 drops of methyl red indicator solution to the sample solution and turn on the magnetic stirrer. Titrate the sample solution with standard 0.1 N sulfuric acid to a definite pink endpoint.
4. Place the beaker on the hot plate and heat to boiling for one minute. If the solution remains pink, use the first endpoint.
5. 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 H_2S , 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 H_2S and CO_2 tends to make the methyl red endpoint less definite.

Calculations

Calculate the concentration of diethanolamine in the sample solution using Equation 1.

$$\text{Diethanolamine, g/100 ml} = \frac{105.14 \times A \times N}{V} \quad (1)$$

where:

$$105.14 = \frac{105.14 \times 100}{1000} \quad (1A)$$

105.14 = Molar mass of Diethanolamine

1000 = factor to convert liter to milliliter

100 = factor to convert to 100 ml

A = volume of standard sulfuric acid solution, mL

N = Normality of sulfuric acid solution

V = sample volume, mL

$$\text{Diethanolamine, mass--\%} = \frac{10.51 \times A \times N}{S \times V} \quad (2)$$

where:

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

A = previously defined

N = previously defined

V = 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.

Suggested supplier

VWR International, 1310 Goshen Parkway, West Chester, PA 19380, USA, 1-610-431-1700,
www.vwr.com