

THIOSULFATE IN LEAN AMINE SOLUTIONS

UOP Method 818-81

SCOPE

This method is for determining thiosulfate in lean amine solutions. Thiosulfate is calculated and reported as either apparent H₂S in grains/gal or as sodium thiosulfate in g/100 mL. The lower limit of detection is approximately 25 grains/gal.

OUTLINE OF METHOD

The amine sample is diluted with water, boiled to eliminate hydrogen sulfide and acidified. The thiosulfate in the acidified solution is treated with an excess of standard iodine solution. The excess iodine is back-titrated to the starch indicator endpoint with standard sodium thiosulfate solution.

APPARATUS

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

Beaker, 100- and 250-mL

Boiling chips

Buret, 50-mL

Cylinder, graduated, 25- and 250-mL

Flask, Erlenmeyer, 250-mL

Flask, volumetric, one-liter (2 required)

Flask, volumetric, 100-mL

Hot plate

Pipets, volumetric, 1-, 5- and 10-mL

Stirrer, magnetic, with Teflon-covered stirring bar

Stirring rod, glass, 150-mm length

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REAGENTS AND MATERIALS

Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications exist. References to water, unless otherwise indicated, mean distilled or deionized water.

Hydrochloric acid, concentrated

Iodine solution, 0.05-*M*. To prepare, dissolve 25 g of potassium iodide in 200 mL of water. Warm slightly to dissolve. Cool and add 12.69 g iodine crystals. Stir the solution to dissolve the reagents and transfer quantitatively to a one-liter volumetric flask. Dilute to volume with water. Store in an amber bottle. Standardize against the 0.1-*M* sodium thiosulfate. If samples are analyzed infrequently, the iodine solution must be standardized prior to use.

Lead acetate paper

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

Salicylic acid

Sodium thiosulfate solution, 0.1-*M*. To prepare, dissolve 24.82 g of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in 200 mL of warm water. Quantitatively transfer to a one-liter volumetric flask, cool, and dilute to volume with water. Standardize in any acceptable fashion such as by the iodine or arsenious oxide technique.

Starch, arrowroot, Leco Corp., Cat. No. 051-061, or equivalent

Starch solution. Stir with a glass rod while slowly adding 0.25 g of starch to 10 mL of water in a 100-mL beaker. Quantitatively transfer the resulting solution to a 100-mL volumetric flask containing 60-70 mL of hot, 100 C, water. Swirl the flask to mix. Add 0.12 g of salicylic acid, predissolved in 5 mL of warm water, to the flask as a preservative. Cool the flask and contents to room temperature and dilute to volume with water. A fresh solution must be prepared if a mold appears or when a definite blue color is not obtained with iodine solution.

PROCEDURE

Arrange the apparatus in an efficient hood. Hydrogen sulfide is an extremely poisonous gas. Pipet 10 mL of the lean amine sample into a 250-mL Erlenmeyer flask containing boiling chips. Dilute to a volume of 75-100 mL with water. Place the flask and contents on a hot plate and carefully boil at a slow rate. Make additions of water, as required, to keep the volume greater than 25 mL. Occasionally test the vapors with moist lead acetate paper until no darkening of the paper is observed, thereby indicating that the hydrogen sulfide has been removed. Continue boiling the solution 15 minutes longer.

Cool the flask and contents to room temperature and adjust the volume to 100 mL with water. Place a magnetic stirring bar in the flask and place on magnetic stirrer. Pipet one milliliter of concentrated hydrochloric acid into the flask and turn on the magnetic stirrer. Add a measured excess of the standardized iodine solution to the flask from either a buret or pipet. The volume of iodine solution added will vary depending upon the thiosulfate content of the amine solution. It should be enough to require at least a 10 mL back titration with the sodium thiosulfate solution.

If the acidified sample is not darkly colored before the addition of the iodine solution, titrate with the standard sodium thiosulfate solution until the sample-solution is almost colorless, then add 2 mL of starch indicator solution and continue the titration until the sample-solution is colorless. If the acidified sample was colored, add starch immediately and titrate with the standard sodium thiosulfate solution until the blue color disappears. Test the sample-solution with methyl orange indicator to verify that the solution was sufficiently acid. If the solution is not acid (red colored), repeat the analysis but this time add 25% more concentrated hydrochloric acid.

CALCULATIONS

Calculate the amount of thiosulfate contained in the amine sample as follows:

$$\text{Thiosulfate, as apparent H}_2\text{S, grains/gal} = \frac{995(2A M_I - B M_T)}{V}$$

where:

- A** = standard iodine solution added, mL
- B** = standard sodium thiosulfate solution added, mL
- M_I** = molarity of the iodine solution
- M_T** = molarity of the sodium thiosulfate solution
- V** = sample volume, mL

The thiosulfate concentration may also be expressed as follows:

$$\text{Sodium thiosulfate, g/100 mL} = 0.0159 T$$

$$\text{Sodium thiosulfate, wt-\%} = \frac{0.0159 T}{S}$$

where:

- S** = specific gravity of lean amine sample, g/mL
- T** = thiosulfate, as apparent H₂S, grains/gal

PRECISION

Insufficient data are available to calculate an esd with at least 4 degrees of freedom. It is estimated that duplicate analyses should agree within 25 grains/gallon.

TIME FOR ANALYSIS

The elapsed time for one analysis is approximately 1 hour. The labor requirement is 1 hour.

REFERENCE

Girdler Method SM-3, The Girdler Corp., Louisville, KY

SUGGESTED SUPPLIER

Leco Corp., 300 Lakeview Ave., St. Joseph, MI 49085