



Thiosulfate in Lean Amine Solutions

UOP Method 818-20

Scope

This method is for determining thiosulfate in lean amine solutions. Thiosulfate is calculated and reported as either apparent H₂S as grains per gallon or as sodium thiosulfate in g/100 mL. The lower limit of detection is approximately 0.04 g/100 mL as apparent H₂S.

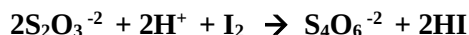
References

ASTM D4052, "Standard Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter" www.astm.org

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.

The following reaction takes place during this process



Apparatus

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

Balance, capacity 500 g, capable of weighing to 0.01 g

Beakers, 100- and 250-mL, VWR, Cat. Nos. 89001-058 and -062, respectively.

Boiling chips, VWR, Cat. No. 26397-409

Buret, pyrex, Class A 50-mL, VWR, Cat. No. 17452-404

Cylinders, graduated, Class B, 25- and 250-mL, VWR, Cat. Nos. 10545-970 and 10546-056

Flask, Erlenmeyer, 250-mL, VWR, Cat. No. 10536-914

Flask, volumetric, Class A 100-mL, VWR, Cat. No. 89001-964

Flask, volumetric, Class A 1000-mL, VWR, Cat. No. 89001-966 (2 required)

Fume hood

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

Pipets, volumetric, Class A 1-, 5- and 10-mL, VWR, Cat. Nos. 10546-006, -014, and -024, respectively.

Pipet filler

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

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 or distilled water.

Hydrochloric acid, concentrated, reagent, VWR, Cat. No. JT9535-0

Iodine solution, 0.1N, VWR, Cat. No. RC400032

Lead acetate paper, VWR, Cat. No. EM1.09511.0003

Methyl orange indicator solution, 0.1 wt-% concentration, VWR, Cat. No. BDH7610-2

Sodium thiosulfate solution, 0.2N, VWR, Cat. No. RC7960-1

Starch solution 0.5% w/v, VWR, Cat. No. 97026-746

Water, deionized or distilled.

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.

The following steps must be performed in an efficient fume hood. Hydrogen sulfide if present will be produced in Steps 1 to 3.

1. Pipet 10 mL of the lean amine sample into a 250-mL Erlenmeyer flask containing boiling chips.
2. Dilute to a volume of 75-100 mL with deionized water.
3. Place the flask and contents on a hot plate and carefully boil at a slow rate. Make additions of deionized water, as required, to keep the volume greater than 25 mL.
4. Test the vapors occasionally 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.
5. Cool the flask and contents to room temperature and adjust the volume to 100 mL with deionized water.
6. Place a magnetic stirring bar in the flask and place on magnetic stirrer. Pipet 1-mL of concentrated hydrochloric acid into the flask and turn on the magnetic stirrer.
7. Add a measured excess of the 0.1N 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.
8. 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.
 9. If the acidified sample was colored, add starch immediately and titrate with the standard sodium thiosulfate solution until the blue color disappears.
 10. 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.
 11. If necessary, determine the relative density of the lean amine sample, see *Calculations*.

Calculations

Calculate the amount of thiosulfate contained in the amine sample using Equation 1.

$$\text{Thiosulfate, as apparent H}_2\text{S, grains/gal} = \frac{995(\text{AN}_I - \text{BN}_T)}{V} \quad (1)$$

where:

A = standard iodine solution added, mL

B = standard sodium thiosulfate solution added, mL

N_I = normality of the iodine solution

N_T = normality of the sodium thiosulfate solution

V = sample volume, mL

995 = Conversion factor g/ml to grains/gal as shown below

995 = (34.08 g/mol)(15.43 gr/g)(3.785 L/gal)(1 mol H₂S/2 mol Na₂S₂O₃)

The thiosulfate concentration may also be expressed as follows:

$$\text{Sodium thiosulfate, g/100 mL} = 0.0159 T \quad (2)$$

$$\text{Sodium thiosulfate, mass-\%} = \frac{0.0159 T}{S} \quad (3)$$

where:

S = relative density of lean amine sample, at 20°C, per ASTM D4052 or equivalent

T = thiosulfate, as apparent H₂S, grains/gal

0.0159 = Conversion factor grains/gal to g/100ml as shown below

0.0159 = (1 g/15.43 gr)(1 gal/3.785 L)(1 L/10 100-mL portions)(158.11 g Na₂S₂O₃ *2) / (34.08 g H₂S)

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 or 0.04g/100ml.

Time for Analysis

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

Suggested Supplier

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