

ALKALINITY OF ACETONE

UOP Method 808-84

SCOPE

This method is for determining the alkalinity of acetone, which is calculated as ammonia (NH_3), from 500 mass-ppm down to a lower detection limit of 3.

OUTLINE OF METHOD

The acetone sample is added to water previously pH adjusted to a methyl red endpoint. Alkalinity is determined by titration with 0.005 *M* sulfuric acid and reported as mass-percent NH_3 .

APPARATUS

Buret, 10-mL, 0.05-mL Subdivisions

Cylinder, graduated, 50-mL

Flask, Erlenmeyer, 250-mL

Flask, volumetric, 100-mL

Pipet, 50-mL

Stirring apparatus, Sargent-Welch Scientific, Cat. No. S-76491-10, or equivalent, with Teflon coated stirring bar

REAGENTS AND MATERIALS

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. Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of ASTM D 1193.

Isopropyl alcohol, 99% minimum purity, Sargent-Welch Scientific, Cat. No. SC14354-500ML, or equivalent.

Methyl red indicator

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Methyl red solution. Dissolve 0.2 g of methyl red indicator in isopropyl alcohol. Dilute to 100 mL with the alcohol.

Sodium hydroxide standard solution, aqueous, 0.01 *M*. Prepare from the solid or a concentrate. Standardize by any acceptable procedure, e.g., potassium hydrogen phthalate.

Sodium hydroxide, 50 mass-% aqueous solution

Sulfuric acid, 0.005-*M*. Prepare from a concentrate or the 96% acid. Standardize by any acceptable procedure, e.g., THAM, or against the previously standardized 0.01-*M* sodium hydroxide.

Sulfuric acid, concentrated, 96%

PROCEDURE

Measure 50 mL of water into a 250-mL Erlenmeyer flask. While stirring, add 2 drops of methyl red solution. If the water is acidic, as indicated by a red color, adjust the pH by adding 0.01-*M* sodium hydroxide dropwise from a buret until a slight yellow coloration persists and then back titrate to a faint red (pink) coloration with 0.005-*M* sulfuric acid. If on addition of the indicator the solution is colored yellow, add 0.005-*M* sulfuric acid until the first pink coloration.

After pH adjustment is complete, pipet 50 mL of acetone sample into the 250-mL Erlenmeyer flask. Start the stirrer and observe the color of the solution. No color change indicates that the alkalinity is zero. If the solution is alkaline, titrate with 0.005-*M* sulfuric acid to a faint pink coloration and record the volume of acid used.

The relative density of the sample can be determined using ASTM D 4052 or other accepted techniques.

CALCULATIONS

Calculate the alkalinity of the acetone sample as follows:

$$\text{NH}_3, \text{ mass-ppm} = \frac{0.00068 \text{ VM } 10^6}{D}$$

where:

D = relative density of the sample at the sampling temperature

M = molarity of the sulfuric acid

V = volume of 0.005-*M* sulfuric acid used to titrate the sample, mL

0.00068 = 0.017(2)/50, millimolecular weight of NH₃ multiplied by the number of protons in H₂SO₄, divided by the sample volume

10⁶ = conversion factor to calculate mass-ppm

PRECISION

Based on 6 replicate determinations, the estimated standard deviation (esd) for alkalinity at the 10 mass-ppm level was calculated to be 0.13 mass-ppm. Duplicate results by the same operator should not differ by more than 0.48 mass-ppm (95% probability) at the stated level.

TIME FOR ANALYSIS

The elapsed time for a single analysis is 1.0 hour. The labor requirement is 0.25 hour.

REFERENCE

ASTM Method D 4052, www.astm.org

SUGGESTED SUPPLIER

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