

Percent Active Sulfonate Content by Cationic Titration (Hyamine)

Procedure No. 817/4.9/T1409A

1.0 SCOPE

- 1.1 This method is used to determine the percentage of sulfonate in "HYBASE" products.
- 1.2 This test method may involve hazardous materials, procedures, or equipment. It is the responsibility of the users to refer to the appropriate MSDS information before proceeding with this method to determine the appropriate health and safety precautions prior to use.

2.0 REFERENCED DOCUMENTS

- 2.1 "Determination of Anionic - Active Detergents by Two-Phase Titration," work done by V.W. Reid, G.F. Longman, and E. Heinerth for the CIA (Commission Internationale of Analysis), 4 January 1967.
- 2.2 Gretna Analytical Method 1028, Revision dated 01/20/93.

3.0 SUMMARY OF METHOD

- 3.1 A sample is dissolved in 1:1 Heptane: Isopropyl Alcohol to a known volume. An aliquot of this mixture is then combined with Chloroform, Isopropyl Alcohol and an acid indicator solution, then, titrated to the Hyamine end point.

4.0 APPARATUS

- 4.1 Volumetric Flask, 100 ml
- 4.2 Graduated cylinder, 100 ml
- 4.3 Hot Plate
- 4.4 Pipet, 10 ml
- 4.5 Erlenmeyer flash, 250 ml
- 4.6, Burette (50ml) graduated in 1/10 ml
- 4.7 Standard laboratory protective wear (See Section 1.2)

5.0 Reagents (See Section 1.2)

- 5.1 Sodium Hydroxide (NaOH), N/10
- 5.2 Phenolphthalein, 1% solution in IPA

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- 5.3 Heptane, reagent grade
- 5.4 Isopropyl Alcohol (IPA), reagent grade
- 5.5 Methylene Chloride (CH₂Cl₂), reagent grade
- 5.6 Ethyl Alcohol, Grade 3A
- 5.7 Sulfuric Acid, concentrated (95-98)
- 5.8 Sodium Sulfate, reagent grade
- 5.9 Dimidium Bromide (British Drug House) *
- 5.10 Disulphine Blue (British Drug House's Erioglaurine) *
- 5.11 Sodium Lauryl Sulfate, specially pure (British Drug House) *
- 5.12 Hyamine 1622 (British Drug House) *

* Available in U.S. from Gallard-Schlesinger Chemical Mfg. Co.
Carle Plao
Long Island, NY
Telephone: (516) 333-5600

6.0 PREPARATION OF STANDARD SOLUTIONS.

- 6.1 Mixed Indication stock solution (concentrate)

0.5 0.005g Dimidium Bromide

0.25 0.005g Disulphine Blue

Weigh into separate 100ml beakers and dissolve each in approximately 30ml hot 10% ethanol. Transfer quantitatively to a 250ml, volumetric flask, dilute to volume with 10% ethanol and mix by inverting flask several times.

- 6.2 Acid Indicator Solution

Dissolve 25g of sodium sulfate in about 500 ml of deionized water. Transfer the solution to a 1 liter volumetric flask. Add 10.00 -10.20 g of concentrated sulfuric acid and a 10 ml aliquot (by pipet) of the mixer indicator stock solution. Dilute to volume with deionized water and mix well,

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6.3 Standard Sodium Lauryl Sulfate Solution

Dry the sodium lauryl sulfate in a vacuum oven @ 60°C for 2 hours. Cool in a desiccator. Accurately weigh to four decimal places, 1.9 - 2.1g sodium lauryl sulfate and dissolve in deionized water. Transfer quantitatively to a 1 liter volumetric flask, dilute to volume with deionized water and mix well.

6.4 0.003 M Hyamine Solution

Dissolve 5.20g Hyamine-1622 in 1 gallon deionized water.

Note: These solutions are stable for at least six months and probably much longer.

7.0 STANDARDIZATION OF 0.003 M HYAMINE SOLUTION

7.1 Pipet 10 ml of the standard sodium lauryl sulfate solution into a 100 ml stoppered mixing cylinder. Add 10 ml methylene chloride and 40 ml acid indicator solution. Titrate with 0.003 M Hyamine solution as described in section 8.0.

7.2 Calculate the molarity of the Hyamine solution to six decimal places (four significant figures) by:

$$\text{M Hyamine} = \frac{W \times 10}{\text{mls Hyamine} \times 288.38}$$

Where W = Grams of sodium-lauryl sulfate used to Prepare the std. SLS solution,

8.0 DESCRIPTION OF HYAMINE TITRATION

8.1 Start the titration by adding about ½ of the expected volume of Hyamine solution, dispensed from a 50 ml buret with 0.1 ml divisions. Stopper the cylinder and shake vigorously for 10-15 seconds. Observe the solvent phase. There is no need to wait for complete separation, The lower solvent layer is colored pink prior to the end point and can be observed when only a few drops have separated. Proceed with the titration by adding 1 ml of Hyamine solution at a time. Shake vigorously after each addition and observe the solvent layer. As the end point is approached, reduce Hyamine additions to 0.1 ml increments, The end point is taken when the pink color is completely discharged from the solvent layer which is then gray. With excess Hyamine (< 0.1 ml) the lower layer is a definite blue.

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8.2 Summary of the Color Chances

	<u>Emulsion while Shaking</u>	<u>Separated Lower Layer</u>
Early	Cream or Pinkish	Pink
Approaching end point	Light Yellow	Pink
Very near end point	Greenish Yellow	Pink
At end point	Greenish Yellow	Gray
Over-titration	Green or Blue	Blue

As experience is gained (after a few runs) the titration can be completed within 5 minutes

9.0 PROCEDURE

- 9.1 Weigh a 1.0-1.5 gram sample to the nearest 0.0001 gram into a 100ml Volumetric Flask
- 9.2 Add 10-15 ml of 1:1 Heptane: Isopropyl Alcohol and heat carefully on a hot plate to effect solubility of sulfonate in solvent mixture.
- 9.3 Build to volume using 1:1 Heptane: Isopropyl Alcohol. Stopper, and mix well
- 9.4 Pipet a 10 ml aliquot of this mixture into a 100 ml stoppered mixing cylinder.
- 9.5 Add 10 mls Methylene Chloride and 40 mls of acid indicator solution to the stoppered mixing cylinder. Titrate using 0.003 M Hyamine Solution to the grey end point.

10.0 CALCULATIONS

$$\% \text{ Active} = \frac{(\text{ml Hyamine}) (\text{M Hyamine}) (\text{100 ml dilution}) (\text{Eq wt}) (100)}{(\text{wt of sample}) (\text{10 ml Aliquot}) (1000)}$$

Note: With materials of 25-40% active, reasonable titration of 15-35 mls are obtained using a 10 ml aliquot. For higher active materials use a 5 ml aliquot.