

Standard Test Method for Ethylene Oxide Content of Polyethoxylated Nonionic Surfactants¹

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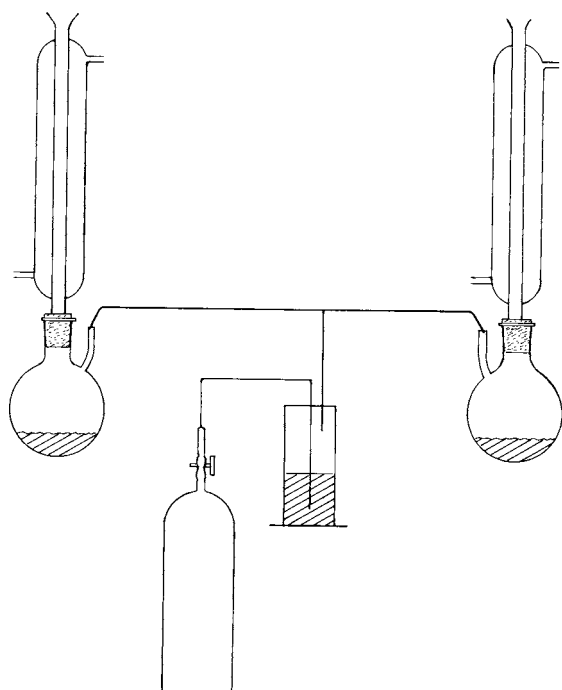


FIG. 1 Apparatus

5.3 Carbon Dioxide or Nitrogen, cylinder gas.

5.4 Lubricant for ground-glass joints.

5.5 Dibutyl Phthalate.

5.6 Glass Beads.

5.7 Hydriodic Acid (55 to 58 %) (suitable for methoxyl determinations)—The hydriodic acid (HI) should not contain hypophosphorus acid (H_3PO_2) stabilizer. The free iodine content should be less than 10 mL of 0.1 N sodium thiosulfate solution/5 mL of HI.

5.8 Hydrochloric Acid (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

5.9 Methanol.

5.10 Potassium Dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$), primary standard.

5.11 Potassium Iodide Solution (200 g/L)—Dissolve 200 g of potassium iodide (KI) in distilled water and dilute to 1000 mL.

5.12 Sodium Thiosulfate, Standard Solution (0.1 N)—Dissolve 24.8 g of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) in water and dilute to 1000 mL. Standardize by accurately weighing 0.16 to 0.22 g of finely ground and dried $\text{K}_2\text{Cr}_2\text{O}_7$ into a 500-mL iodine flask. Dissolve in 25 mL of water, add 5 mL of HCl and 15 mL of KI solution and swirl to mix. Allow to stand for 5 min, then add 100 mL of distilled water. Titrate with standard $\text{Na}_2\text{S}_2\text{O}_3$ solution, shaking continuously until the yellow color has almost disappeared. Add a small amount of Paragon indicator⁵ or 1 to 2 mL of starch indicator solution and continue the titration, adding the $\text{Na}_2\text{S}_2\text{O}_3$ solution slowly until the blue color has just disappeared. Calculate the normality of the $\text{Na}_2\text{S}_2\text{O}_3$ solution as follows:

$$N = C / (0.04904 \times D) \quad (1)$$

⁵ Paragon indicator for iodine titrations may be purchased from Eastern Chemical Corp., 34 Spring St., Newark, NJ 07104.

where:

N = normality of the $\text{Na}_2\text{S}_2\text{O}_3$ solution,

C = grams of $\text{K}_2\text{Cr}_2\text{O}_7$ used, and

D = millilitres of $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration of the solution.

5.13 Starch Indicator Solution—Make a homogeneous paste of 10 g of soluble starch in water. To this add with rapid stirring 1 L of boiling water and then cool. As a preservative, add 1.25 g of salicylic acid per litre. Store the indicator in a refrigerator at 4 to 10°C. Prepare fresh indicator when the titration end point from blue to colorless fails to be sharp.

6. Procedure

6.1 Assemble the apparatus as shown in Fig. 1. Lubricate the ground-glass joints with a very thin film of lubricant. The dual apparatus is designed to permit a sample and a blank to be run simultaneously; however, it should not be necessary to make more than two blank determinations each day.

6.2 Accurately weigh a sample of such size that 4 to 5 meq of iodine will be liberated (about 0.15 g of a nonionic surfactant that contains 60 % ethylene oxide) into a 100-mL round bottom flask. Pipet exactly 5.0 mL of HI into the flask, and add a glass bead. Connect the vertical condenser to the flask using a thin seal of lubricant at the outer edge. Prepare a blank by pipetting 5.0 mL of HI into a second flask containing a glass bead. Connect the condenser as described above, and allow water to circulate through the condensers.

6.3 Attach the tubes from the carbon dioxide manifold to the sidearms on the flasks. Open the valve on the gas cylinder and adjust the flow to a rate of 1 to 5 bubbles per second through the bubbler, blanketing the solutions in the reaction flasks. The flow of carbon dioxide should continue for 10 min before heat is applied to the reaction flasks. After 10 min turn on the heat, and reflux the contents gently for 90 min.

6.4 After the 90-min boiling period, wash the walls of each condenser with 15 mL of 20 % KI solution to dissolve any free iodine in the condensers. Wash the condensers with two 10-mL portions of water and disconnect. Wash the tips of the condensers with water, adding the washings to the flasks. Transfer the contents of the reaction flasks to Erlenmeyer flasks by washing with water.

NOTE 4—Rinse any iodine that adheres to the condensers into the flasks with a small amount of methanol. On rare occasions some samples leave a tarry residue as a button. Add methanol to dissolve the residue, and wash into the Erlenmeyer flask.

6.5 Titrate with standard $\text{Na}_2\text{S}_2\text{O}_3$ solution, shaking continuously until the yellow color has almost disappeared. Add a small amount of Paragon indicator or 1 to 2 mL of starch indicator solution and continue the titration, adding the $\text{Na}_2\text{S}_2\text{O}_3$ solution slowly until the blue color has just disappeared.

7. Calculation

7.1 Calculate the ethylene oxide content as follows:

$$\text{Ethylene oxide, weight \%} = [(A - B) \times N \times 2.203] / W \quad (2)$$

where: