Anionics

The Procedure is based on Epton’s method, also known as the two-phase titration where the anionic surfactant is titrated with a cationic surfactant (1 mol cationic = 1 mol anionic) in the presence of methylene blue as indicator.

**Reagents**

- Methylene Blue
- Sodium Sulfate
- Fumic Sulfuric Acid
- Chloroform
- Deionized Water

**Equipment (See Figure 3)**

- 25-mL graduated glass cylinder with stopper
- 10-mL graduated buret; scale is read to the nearest 0.01 mL
- Double Buret Holder

**Methylene Blue Indicator: 1 liter**

1. Transfer to 1-liter volumetric flask with a magnetic stirring bar inside
   a. 50 grams of sodium sulfate
   b. 0.03 grams of Methylene Blue
2. Add about 600-mL of deionize water
3. Mix thoroughly on a magnetic plate located inside the hood
4. Slowly, while mixing contents, add 6-mL of Fumic Sulfuric acid

*Hint: a very clean 10-mL pipette is filled, directly from bottle, to the 6-mL mark using a pipette filler that lets evacuate, fill pipette, and dispense liquid easily. (The use of 1,5, or 3-mL Ostwald-Folin pipettes is recommended)*

5. Sliding a magnet outside flask wall, lift magnetic-stirring bar inside flask to its top and fill to 1-liter mark with deionize water. Release stirring bar and mix well
**Titrant**

Prepare a solution of cationic surfactant; e.g., Hyamine, TEGO, of molar equivalence of "unknown" sample to be determined.

*Hint: the operator should guess-estimate the surfactant concentration in test sample. For accuracy, the volume of titrant dispensed should not be larger than 10-mL; thus titrant molarity should be adjusted accordingly to “unknown” sample molarity*

**Titrant Calibration**

Prepare a standard solution with a pure, standard, Sodium Dodecyl Sulfate (SDS). The SDS molarity is prepared to theoretically match molarity of cationic solution to be used for titrating unknown sample, which concentration has been guest-estimated.

Determine Molarity of cationic titrant according to **Titration Procedures**

**Sample Preparation**

1. Weight at least three aliquots of different volumes into clean, dried 25-mL graduated cylinders labeled 1 to 3 with water resistant markers.

   **Optional Hints for speeding determination**
   1- Gauge a volume, from micro-liters to milliliters, using pipettes. For precise measure, discharge volume measured into a 25-mL cylinder located on an accurate balance. This method will eliminate steps of sample dilutions that increase experimental errors; If sample is of (a) high-surfactant concentration, weigh the equivalent of µL (b) low-surfactant concentration, weight the equivalent of mL.

   2- Write value of weighted volume on cylinder immediately after pulling away from balance. Before titration transfer sample number and its value to labnotebook

   3- Before cleaning cylinders with water for next titration, whip markings with a paper towel slightly soaked with iso-propyl alcohol (IPA)

2. Add to sample in 25-mL cylinder
   3-mL of Chloroform
   5-mL of Methylene Blue indicator

3. Insert stopper and mix cylinder contents: See 0% image
4. Follow directions on **Titration Procedure**
**Titration Procedure**

Figure 1 is to illustrate steps for finding same-color end point. After each addition of cationic-surfactant solution, contents are shaken and let equilibrate before further addition. The titration should be conducted to for following more or less images disclosed.

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**Percentage of completion using Hyamine volume added. [Hyamine]=0.001 M**

<table>
<thead>
<tr>
<th>Percentage</th>
<th>Image</th>
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<tbody>
<tr>
<td>0%</td>
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</tr>
<tr>
<td>27%</td>
<td><img src="image2.png" alt="Image" /></td>
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<td>54%</td>
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<td>108%</td>
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Two Phase Epton’s Titration
End-Point: equal color

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*Figure 1. Pictures to illustrate 2-Phase titration*
Test Case Example

Find the concentration of a nominal 0.5% surfactant concentration where

- Molecular Weight = 450
- Hyamine Molarity (after SDS standardization) = 0.0095

Procedure

- Titrate 3 different aliquot (1, 1.5, 2.0 gram)
- Plot values
- Calculate concentration by

  \[
  \% \text{ Anionic Surfactant} = \text{Slope} \times \text{Titrant concentration (molar)} \times \frac{\text{MW}}{10}
  \]

  \[
  \% \text{ Anionic Surfactant} = 1.0197 \times 0.0095 \times 450 / 10
  \]

  \[
  = 0.434
  \]

Figure 2. Example of data handling
Equipment Images

Double Buret Holder for 10mL

Pipette Fillers

Graduated Cylinder with stopper

Figure 3: Images to illustrate Equipment
For fumic sulfuric acid use Ostwald-Folin pipettes (5 ml and 1 ml, or 3ml)

Note: Centrifuging may help to break emulsions, and will help to detect the end point.