DIOCTYL SODIUM SULFOSUCCINATE


SYNONYMS
DSS, docusate sodium, INS No. 480

DEFINITION

Chemical names
Sodium 1,4-bis-(2-ethylhexyl)-sulfosuccinate; butanedioic acid, sulfo-1,4-bis-(2-ethylhexyl) ester sodium salt; dioctyl sodium sulfosuccinate

C.A.S. number
577-11-7

Chemical formula
C_{20}H_{37}NaO_7S

Structural formula

\[
\begin{align*}
\text{COO} & \quad \text{CH}_2 & \quad \text{CH(CH}_2)_2\text{CH}_3 \\
\text{CH}_2 \\
\text{CH} & \quad \text{SO}_2\text{Na} \\
\text{COO} & \quad \text{CH}_2 & \quad \text{CH(CH}_2)_2\text{CH}_3 \\
& & \quad \text{C}_2\text{H}_5
\end{align*}
\]

Formula weight
444.56

Assay
Not less than 98.5% on the dried basis

DESCRIPTION
White, wax-like, plastic solid, having a characteristic odour suggestive of octanol, but free from odour of other solvents

FUNCTIONAL USES
Emulsifying agent, wetting agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)
Sparingly soluble in water; freely soluble in ethanol and glycerol

Infrared absorption

pH (Vol. 4)
5.8 - 6.9 (1 in 100 soln)

PURITY
Loss on drying (Vol. 4) Not more than 2% (105° for 2 h)

Clarity of solution Dissolve 25 g of the sample in 94 ml of ethanol. The solution does not develop a haze within 24 h

Sulfated ash (Vol. 4) Between 15.5% and 16.2%. Test 1 g of the sample (Method I)

Bis-(2-ethylhexyl)-maleate Not more than 0.4% See description under TESTS

Lead (Vol. 4) Not more than 2 mg/kg Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, “Instrumental Methods.”

TESTS

PURITY TESTS

Bis-(2-ethylhexyl)-maleate Reagents
- Supporting electrolyte: Dissolve 21.2 g of anhydrous lithium perchlorate (LiClO₄) in 175 ml of water in a 250-ml beaker. Adjust the pH of this solution to 3.0 by the dropwise addition of glacial acetic acid (usually 1 or 2 drops is sufficient), using a suitable pH meter. Quantitatively transfer the solution into a 200-ml volumetric flask, dilute to volume with water, and mix.

- Standard solution: Transfer 100-110 mg of bis-(2-ethyl-hexyl)-maleate (a suitable grade of bis-(2-ethylhexyl)-maleate is available as OT-35 from American Cyanamid Company, Pine Chemicals Department, Pearl River, New York 10965, USA), accurately weighed, into a 100-ml volumetric flask. Record the exact weight, to the nearest 0.1 mg, as Wₐ. Dilute to volume with water, and mix.

- Sample stock solution: Transfer 12.5 g of the sample, accurately weighed, into a 150-ml beaker. Record the exact weight, to the nearest 10 mg as Wₛ. Add 80-90 ml of isopropanol, and stir with a glass stirring rod until the sample is dissolved. Quantitatively transfer this solution, with the aid of isopropanol, into a 250-ml volumetric flask. Dilute to volume with isopropanol, and mix.

- Test solution A: Pipet 50.0 ml of the "Sample stock solution" and 20.0 ml of the "Supporting electrolyte" into a 100-ml volumetric flask. Dilute to within 15 mm of the graduated volume line with isopropanol, stopper, shake to facilitate solution, and set aside for 2 min. Dilute to volume with isopropanol, and mix. A completely clear solution should be obtained.

- Test solution B: Pipet 50.0 ml of the "Sample stock solution", 10.0 ml of the "Standard solution", and 20.0 ml of the "Supporting electrolyte" into a 100 ml volumetric flask, and complete the preparation as described for "Test Solution A".

- Blank: Pipet 20.0 ml of the "Supporting electrolyte" into a 100-ml
volumetric flask, dilute to volume with isopropanol, and mix.

Procedure
Rinse a polarographic H-cell several times with small portions of "Test solution A", then fill the cell half-full with the solution, place a paper tissue in the top of the sample side of the cell and pass a moderate stream of nitrogen through the solution for 15 min. (NOTE: The nitrogen should first be saturated by passing it through a suitable scrubber containing isopropanol). After 15 min, divert the nitrogen stream over the surface of the solution, and remove the tissue from the cell. Set the polarizing voltage of a suitable, previously calibrated polarograph (Metrohm Polacord E-261 or equivalent) at -1.3 volts. Adjust the current sensitivity to the lowest range (most sensitive) at which the current oscillations will remain on scale, and record the polarogram, scanning a voltage range of -0.9 to -1.5 volts at this sensitivity and using a saturated calomel electrode as the reference electrode. Record the average oscillations, in mm, at -1.3 volts as A, and those at -1.0 volt as B. (NOTE: If a manual polarograph is used, record the average oscillations of the solutions at -1.3 volts and -1.0 volt respectively). Repeat the entire procedure using Test solution B, recording the average oscillations at -1.3 volts as D, and those at -1.0 volt as E. Similarly, repeat the entire procedure using the "Blank", recording the average oscillations at -1.3 volts as G, and those at -1.0 volt as H.

Calculation
Make the following preliminary calculations (in mA):

\[ C' = (A - B) \times S_1 \]

where
C is net diffusion current of "Test solution A", and
S_1 is current sensitivity used for "Test solution A"

\[ F = (D - E) \times S_2 \]

where
F is net diffusion current of "Test solution B", and
S_2 is current sensitivity used for "Test solution B"

\[ I = (G - H) \times S_3 \]

where
I is net current introduced by the "Blank", and
S_3 is current sensitivity used for the "Blank"

\[ J = F - C' \]

where
J is diffusion current due to added maleate, added to Test Solution B from the Standard Solution

\[ K = C' - I \]
METHOD OF ASSAY

Solutions
- Sample solution: Transfer about 3.8 g of the sample, previously dried at 105°C for 2 h and accurately weighed, into a 500-ml volumetric flask, dissolve in chloroform. Dilute to volume with the same solvent, and mix.
- Tetra-n-butylammonium iodide solution: Transfer 1.250 g of tetra-n-butylammonium iodide to a 500-ml volumetric flask, dilute to volume with water, and mix.
- Salt solution: Dissolve 100 g of anhydrous sodium sulfate and 10 g of sodium carbonate in sufficient water to make 1000 ml

Procedure
Pipet 10.0 ml of the "Sample solution" into a 250-ml flask, and add 40 ml of chloroform, 50 ml of "Salt solution", and 10 drops of bromophenol blue TS. Titrate with "Tetra-n-butylammonium iodide solution" to the first appearance of a blue colour in the chloroform layer after vigorous shaking. Calculate the percentage of C₂₀H₃₇NaO₇S by the formula:

\[
\frac{V \times 1.250 \times 444.6 \times 10}{W \times 369.4}
\]

where
V = the volume, in ml of tetra-n-butylammonium iodide solution required
444.6 = formula weight of diocetyl sodium sulfusuccinate
W = the weight, in g, of the sample taken
369.4 = formula weight of tetra-n-butylammonium iodide

Finally, calculate the percentage of bis-(2-ethylhexyl)-maleate in the original sample taken by the formula:

\[
\frac{K \times 50W_A}{J \times W_S}
\]

where
K is the diffusion current due to maleate present in Test Solution A.