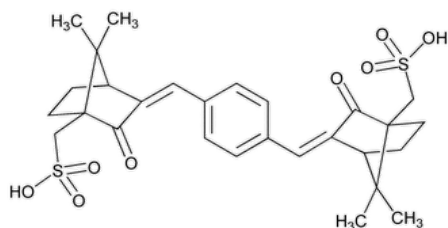


Status: Currently Official on 14-Feb-2025  
 Official Date: Official as of 01-May-2020  
 Document Type: USP Monographs  
 DocId: GUID-F330AF2E-C1D4-4F33-908D-9ED8FC938EAB\_2\_en-US  
 DOI: [https://doi.org/10.31003/USPNF\\_M3063\\_02\\_01](https://doi.org/10.31003/USPNF_M3063_02_01)  
 DOI Ref: f6khm

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## Ecamsule Solution



$C_{28}H_{34}O_8S_2$  562.69

Bicyclo[2.2.1]heptane-1-methanesulfonic acid, 3,3'-(1,4-phenylenedimethyldiene)bis[7,7-dimethyl-2-oxo]-.

(±)-(3E,3'E)-3,3'-(p-Phenylenedimethyldiene)bis[2-oxo-10-bornanesulfonic acid] CAS RN®: 92761-26-7; UNII: M94R1PM439.

» Ecamsule Solution is an aqueous solution of  $C_{28}H_{34}O_8S_2$ . It contains not less than 30.0 percent and not more than 34.0 percent, by weight, of ecamsule ( $C_{28}H_{34}O_8S_2$ ).

**Packaging and storage**—Preserve in tight containers. Protect from light, and store at room temperature.

### USP REFERENCE STANDARDS (11).—

[USP Ecamsule Solution RS](#)

[USP Ecamsule Related Compound A RS](#)

1,4-Phenylenedimethanol.

$C_8H_{10}O_2$  138.16

[USP Ecamsule Related Compound B RS](#)

4-(Hydroxymethyl)benzoic acid.

$C_8H_8O_3$  152.15

[USP Ecamsule Related Compound C RS](#)

Terephthalic acid.

$C_8H_6O_4$  166.13

[USP Ecamsule Related Compound D RS](#)

((1S,4R)-7,7-Dimethyl-2-oxobicyclo[2.2.1]heptan-1-yl)methanesulfonic acid.

$C_{10}H_{16}O_4S$  232.30

[USP Ecamsule Related Compound E RS](#)

Sodium ((1S,4R,E)-3-(4-(hydroxymethyl)benzylidene)-7,7-dimethyl-2-oxobicyclo[2.2.1]heptan-1-yl)methanesulfonate.

$C_{18}H_{21}NaO_5S$  372.41

[USP Ecamsule Related Compound F RS](#)

4-((E)-((1S,4SR)-7,7-Dimethyl-3-oxo-4-(sulfomethyl)bicyclo[2.2.1]heptan-2-ylidene)methyl)benzoic acid.

$C_{18}H_{20}O_6S$  364.41

[USP Ecamsule Related Compound G RS](#)

4-((E)-((1S,4SR)-7,7-Dimethyl-3-oxo-4-(sulfomethyl)bicyclo[2.2.1]heptan-2-ylidene)methyl)benzaldehyde, sodium salt.

$C_{18}H_{19}NaO_5S$  370.40

[USP Ecamsule Triethanolamine RS](#)

Bicyclo[2.2.1]heptane-1-methanesulfonic acid, 3,3'-(1,4-phenylenedimethyldiene)bis[7,7-dimethyl-2-oxo]-, triethanolamine salt (1:2).

$C_{28}H_{34}O_8S_2 \cdot (C_6H_{15}NO_3)_2$  861.07

**Labeling**—The label states that this article is not intended for direct administration to humans or animals.

### Identification—

#### Change to read:

**A:** [▲Spectroscopic Identification Tests \(197\), Infrared Spectroscopy: 197A](#) (CN 1-May-2020) —Place a drop of Ecamsule Solution on a diamond sampling surface and dry it with a stream of warm air. The IR absorption spectrum conforms to that of [USP Ecamsule Solution RS](#), similarly obtained.

#### Change to read:

**B:** [▲SPECTROSCOPIC IDENTIFICATION TESTS \(197\), Ultraviolet-Visible Spectroscopy: 197U](#) (CN 1-May-2020) —

**Solution**—Transfer 0.25 g of Ecamsule Solution to a 100-mL volumetric flask, and dilute with water to volume. Further dilute 2 mL of this solution with water to 100 mL.

The **Solution** exhibits absorption maximum between 342 and 346 nm.

**Limit of chloride**—Dissolve about 10 g of Ecamsule Solution, accurately weighed, in 70 mL of water. Titrate this solution with 0.01 N silver nitrate, determine the endpoint potentiometrically (see [Titrimetry \(541\)](#)), and calculate the percentage of chloride in the portion of  $C_{28}H_{34}O_8S_2$  taken by the formula:

$$100(35.5)(VN/W)(100/A)$$

in which 35.5 is the atomic weight, in g per mole, of chloride; V is the volume, in mL, of silver nitrate used for titration; N is the concentration, in normality, of silver nitrate; W is the weight, in mg, of Ecamsule Solution taken for determination; and A is the assay, in percent, of Ecamsule Solution: not more than 0.3% of chloride is found.

**Limit of sodium—**

*Diluent*—Transfer 5 mL of nitric acid in a 1000-mL volumetric flask containing about 500 mL of water, and dilute with water to volume.

*Test solution*—Transfer about 1 g of Ecamsule Solution, accurately weighed, to a 100-mL volumetric flask, and dilute with *Diluent* to volume.

*Standard solutions*—Dilute quantitatively, and stepwise if necessary, a commercially available sodium atomic absorption standard solution containing 1000 µg of sodium per mL with *Diluent* to obtain solutions having known concentrations of 1, 5, 10, and 20 µg per mL, respectively.

*Procedure* (see [ATOMIC ABSORPTION SPECTROSCOPY \(852\)](#))—Concomitantly determine the absorbances of the *Standard solutions* and the *Test solution* at the sodium emission line of 330 nm or 589 nm with a suitable atomic absorption spectrophotometer equipped with a sodium lamp and an air–acetylene flame, using *Diluent* as the blank. Determine the concentration of sodium, in µg per mL, in the *Test solution* using the calibration graph. Calculate the percentage of sodium in the portion of  $C_{28}H_{34}O_8S_2$  taken by the formula:

$$100 \times 10^{-6}(CV/W)(100/A)$$

in which C is the concentration, in µg per mL, of sodium in the *Test solution*, the multiplier of  $10^{-6}$  is for conversion of µg per mL to g per mL; V is the volume, in mL, of *Test solution*; W is the weight, in g, of Ecamsule Solution taken for determination; and A is the assay, in percent, of Ecamsule Solution: not more than 0.3% of sodium is found.

**Related compounds—**

*Test for related compounds A to F—*

*Solvent A*—Prepare a mixture of acetonitrile and 85% phosphoric acid (1000:1).

*Solvent B*—Prepare a mixture of water and 85% phosphoric acid (1000:1).

*Standard solution*—Dissolve an accurately weighed quantity of [USP Ecamsule Related Compound A RS](#), [USP Ecamsule Related Compound B RS](#), [USP Ecamsule Related Compound C RS](#), [USP Ecamsule Related Compound D RS](#), [USP Ecamsule Related Compound E RS](#), and [USP Ecamsule Related Compound F RS](#) in water, sonicating if necessary, to obtain a solution having known concentrations as found in [Table 1](#).

*Test solution*—Transfer about 100 mg, accurately weighed, of Ecamsule Solution to a 50-mL volumetric flask, and dilute with water to volume.

*Chromatographic system* (see [CHROMATOGRAPHY \(621\)](#))— The liquid chromatograph is equipped with either a programmable variable wavelength detector or two separate detectors capable of monitoring at 200 nm and 300 nm and a 4.6-mm × 15-cm column that contains 5-µm packing L1. The flow rate is about 1 mL per minute. The chromatograph is programmed as follows.

| Time (minutes) | Solution A (%) | Solution B (%) | Elution         |
|----------------|----------------|----------------|-----------------|
| 0–25           | 20             | 80             | isocratic       |
| 25–27          | 20→80          | 80→20          | linear gradient |
| 27–47          | 80             | 20             | isocratic       |
| 47–50          | 80→20          | 20→80          | linear gradient |
| 50–65          | 20             | 80             | equilibration   |

Chromatograph the *Standard solution*, and record the peak areas as directed for *Procedure*: the relative standard deviations of the ecamsule related compound peaks for replicate injections are not more than 10.0%; and the resolution, *R*, between all adjacent peak pairs of ecamsule related compounds is not less than 1.5 measured at 200 nm.

*Procedure*—Separately inject equal volumes (about 10 µL) of the *Standard solution* and the *Test solution* into the chromatograph, allow the chromatogram to run for about 20 minutes for the *Standard solution* and 60 minutes for the *Test solution*, record the chromatograms at 200 nm from 0 to 8 minutes and at 300 nm after 8 minutes, and measure the peak areas. Calculate the percentage of ecamsule related compounds A, B, C, D, and F in the portion of  $C_{28}H_{34}O_8S_2$  taken by the formula:

$$100[100C_s/(C_u \times A)](r_u/r_s)$$

in which  $C_s$  is the concentration, in mg per mL, of the ecamsule related compound in the *Standard solution*;  $C_u$  is the concentration, in mg per mL, of the *Test solution*; A is the assay, in percent, obtained from the Assay; and  $r_u$  and  $r_s$  are the peak areas of the ecamsule related