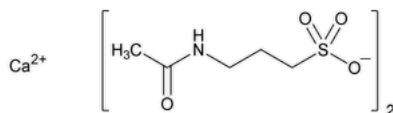


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Acamprosate Calcium



$C_{10}H_{20}CaN_2O_8S_2$ 400.48

1-Propanesulfonic acid, 3-(acetylamino)-, calcium salt (2:1);

Calcium 3-(acetylamino)propane-1-sulfonate CAS RN®: 77337-73-6.

DEFINITION

Acamprosate Calcium contains NLT 98.0% and NMT 102.0% of acamprosate calcium ($C_{10}H_{20}CaN_2O_8S_2$), calculated on the dried basis.

IDENTIFICATION

Change to read:

- **A.** [▲ SPECTROSCOPIC IDENTIFICATION TESTS \(197\), Infrared Spectroscopy: 197K](#)▲ (CN 1-MAY-2020)
- **B.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the Assay.
- **C.** [IDENTIFICATION TESTS—GENERAL \(191\), Chemical Identification Tests, Calcium](#): Meets the requirements

ASSAY

PROCEDURE

Mobile phase: Add 5.0 mL of [triethylamine](#) per 1 L of [water](#) and adjust with [phosphoric acid](#) to a pH of 4.0.

System suitability solution: 10 mg/mL of [USP Acamprosate Calcium RS](#) and 0.005 mg/mL each of [USP Acamprosate Related Compound B RS](#) and [glacial acetic acid](#) in [water](#). Sonication may be used to aid in dissolution.

Standard solution: 0.3 mg/mL of [USP Acamprosate Calcium RS](#) in [water](#). Sonication may be used to aid in dissolution.

Sample solution: 0.3 mg/mL of Acamprosate Calcium in [water](#). Sonication may be used to aid in dissolution.

Chromatographic system

(See [Chromatography \(621\), System Suitability](#).)

Mode: LC

Detector: UV 210 nm

Column: 4.6-mm × 25-cm; 5-μm packing [L1](#)

Flow rate: 0.7 mL/min

Injection volume: 20 μL

Run time: NLT 2 times the retention time of the acamprosate peak

System suitability

Samples: *System suitability solution* and *Standard solution*

[NOTE—See [Table 1](#) for the relative retention times.]

Suitability requirements

Resolution: NLT 1.5 between acetic acid and acamprosate related compound B; NLT 1.3 between acamprosate related compound B and acamprosate, *System suitability solution*

Tailing factor: NMT 2.0, *Standard solution*

Relative standard deviation: NMT 0.73%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of acamprosate calcium ($C_{10}H_{20}CaN_2O_8S_2$) in the portion of Acamprosate Calcium taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

C_S = concentration of [USP Acamprosate Calcium RS](#) in the *Standard solution* (mg/mL)

C_U = concentration of Acamprosate Calcium in the *Sample solution* (mg/mL)

Acceptance criteria: 98.0%–102.0% on the dried basis

IMPURITIES

Change to read:

• LIMIT OF ACAMPROSATE RELATED COMPOUND A

Solution A: 5 g/L of [fluorescamine](#) in [acetonitrile](#). Use within 24 h of preparation.

Buffer: 13.8 g/L of [monobasic sodium phosphate](#) prepared as follows. Transfer a suitable amount of [monobasic sodium phosphate](#) to a volumetric flask. Dissolve in 90% of the final flask volume of [water](#). Adjust with [10 N sodium hydroxide TS](#) or [phosphoric acid](#) to a pH of 6.5. Dilute with water to volume.

Mobile phase: [Acetonitrile](#), [methanol](#), and *Buffer* (10:10:80)

Diluent: 24.6 g/L of [boric acid](#) prepared as follows. Transfer a suitable amount of [boric acid](#) to an appropriate volumetric flask. Dissolve in 90% of the final flask volume of [water](#). Adjust with [10 N sodium hydroxide TS](#) to a pH of 10.4. Dilute with [water](#) to volume.

Standard stock solution A: 250 µg/mL of [USP Acamprosate Related Compound A RS](#) in [water](#)

Standard stock solution B: 1 µg/mL of [USP Acamprosate Related Compound A RS](#) from *Standard stock solution A* in *Diluent*

Standard solution: Transfer 3.0 mL of *Standard stock solution B* to an appropriate container. Add 0.15 mL of *Solution A* and shake vigorously for 30 s. Heat in a water bath at 50° for 30 min. Cool under a stream of cold water, centrifuge, and pass the supernatant through a suitable membrane filter.

Sample stock solution A: 20 mg/mL of Acamprosate Calcium in water

Sample stock solution B: 2000 µg/mL of Acamprosate Calcium from *Sample stock solution A* in *Diluent*

Sample solution: Transfer 3.0 mL of *Sample stock solution B* to an appropriate container. Add 0.15 mL of *Solution A* and shake for 30 s. Heat in a water bath at 50° for 30 min. Cool under a stream of cold water, centrifuge, and pass the supernatant through a suitable membrane filter.

Chromatographic system

(See [Chromatography \(621\)](#), *System Suitability*.)

Mode: LC

Detector: UV 261 nm

Column: 4.6-mm × 15-cm; 3- or 5-µm packing [L1](#)

Flow rate: 1 mL/min

Injection volume: 20 µL

Run time: NLT 2 times the retention time of acamprosate related compound A

System suitability

Sample: *Standard solution*

[NOTE—The relative retention times for fluorescamine and acamprosate related compound A are about 0.5 and 1.0, respectively. Acamprosate calcium is not detected by this chromatographic system.]

Suitability requirements

Resolution: NLT 2.0 between fluorescamine and acamprosate related compound A

Relative standard deviation: NMT 5.0% for acamprosate related compound A

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of acamprosate related compound A in the portion of Acamprosate Calcium taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response from the *Sample solution*

r_S = peak response from the *Standard solution*

C_S = concentration of [USP Acamprosate Related Compound A RS](#) (ERR 1-Dec-2018) in the *Standard solution* (µg/mL)

C_U = concentration of Acamprosate Calcium in the *Sample solution* (µg/mL)

Acceptance criteria: NMT 0.05%

• ORGANIC IMPURITIES

Mobile phase: Add 5.0 mL of [triethylamine](#) per 1 L of [water](#) and adjust with [phosphoric acid](#) to a pH of 4.0.

System suitability solution: 10 mg/mL of [USP Acamprosate Calcium RS](#) and 0.005 mg/mL each of [USP Acamprosate Related Compound B RS](#) and [glacial acetic acid](#) in [water](#). Sonication may be used to aid in dissolution.

Standard solution: 0.005 mg/mL of [USP Acamprosate Calcium RS](#) in [water](#). Sonication may be used to aid in dissolution.

Sample solution: 10 mg/mL of Acamprosate Calcium in [water](#). Sonication may be used to aid in dissolution.

Chromatographic system

(See [Chromatography \(621\)](#), *System Suitability*.)

Mode: LC