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Glucosamine and Methylsulfonylmethane Tablets

DEFINITION

Glucosamine and Methylsulfonylmethane Tablets are prepared from either Glucosamine Hydrochloride, Glucosamine Sulfate Sodium Chloride, Glucosamine Sulfate Potassium Chloride, or a mixture of any of them, with Methylsulfonylmethane. Tablets contain NLT 90.0% and NMT 120.0% of the labeled amount of glucosamine ($C_6H_{13}NO_5$) and NLT 90.0% and NMT 110.0% of the labeled amount of methylsulfonylmethane ($C_2H_6O_2S$).

IDENTIFICATION

- **A. PRESENCE OF GLUCOSAMINE:** The retention times of the major peaks of the *Sample solution* correspond to those of the *Standard solution*, as obtained in the *Content of Glucosamine*.
- **B. PRESENCE OF METHYLSULFONYLMETHANE:** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Content of Methylsulfonylmethane*.

STRENGTH

• CONTENT OF GLUCOSAMINE

Diluent: Transfer 29 μ L of acetic acid and 5 mL of acetonitrile to a 100-mL volumetric flask containing 50 mL of water, and dilute with water to volume.

Borate buffer: 0.2 M (76.3 g/L of sodium borate in water) adjusted with hydrochloric acid TS to a pH of 9.5. [NOTE—Buffer must be stored at room temperature. It must be warmed to dissolve if crystallization occurs.]

Acetate buffer: 6.80 g/L of sodium acetate trihydrate in water adjusted with dilute acetic acid to a pH of 5.9

Derivatizing reagent: In a 14-mL polypropylene culture tube dissolve 50 mg of o-phthalaldehyde in 1.25 mL of anhydrous methanol. Add 50 μ L of 3-mercaptopropionic acid and 11.2 mL of *Borate buffer*, and mix gently. Allow to stand in the dark for 30 min before use. [NOTE—Reagent strength is maintained by adding 10 μ L of 3-mercaptopropionic acid every 2 days. Storage should be in the dark, at room temperature, and can be used for NMT 2 weeks.]

Mobile phase: Methanol and *Acetate buffer* (1:9)

Standard solution: 1.0 mg/mL of [USP Glucosamine Hydrochloride RS](#) in water. Allow to stand at room temperature for 1 h.

Sample solution: Transfer an equivalent to 25 mg of glucosamine from NLT 20 Tablets, finely powdered, to a 25-mL volumetric flask, and dilute with *Diluent* to volume. Mix on a vortex mixer to suspend the powder in solution. Sonicate in a 65° water bath for 20 min. Remove from the bath, stir for 5 min with the aid of a magnetic stirrer, and centrifuge.

Chromatographic system

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

Mode: LC

Detector: UV 340 nm

Column: 3.0-mm $\times$ 5-cm; packing L1

Flow rate: 1 mL/min

Injection size: 10 μ L

System suitability

Samples: Five individual aliquots of the *Standard solution* derivatized as directed for *Analysis*. Each derivatized aliquot is injected only once. [NOTE—The relative retention times for the β -anomer and the α -anomer are 1.0 and 1.8, respectively. The retention time for the β -anomer is NLT 4 min.]

Suitability requirements

Relative standard deviation: NMT 2.0% for five replicate injections

Analysis

Samples: *Standard solution* and *Sample solution*

Transfer 100 μ L of the *Derivatizing reagent* and 100 μ L of the *Standard solution* or the *Sample solution* to a vial containing 400 μ L of *Borate buffer*. Allow the derivatization to proceed for 1 min. Inject the derivatized solutions immediately after the derivatization reaction.

Calculate the percentage of the labeled amount of glucosamine ($C_6H_{13}NO_5$) in the portion of Tablets taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times (M_{r1}/M_{r2}) \times 100$$

r_U = peak response of the β -anomer from the derivatized *Sample solution*

r_S = peak response of the β -anomer from the derivatized *Standard solution*

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

C_U = nominal concentration of glucosamine in the *Sample solution* (mg/mL)

M_{r1} = molecular weight of glucosamine, 179.17

M_{r2} = molecular weight of glucosamine hydrochloride, 215.63

Acceptance criteria: 90.0%–120.0% of the labeled claim

• **CONTENT OF METHYLSULFONYLMETHANE**

Diluent: Transfer 950 mL of methanol to a 1-L volumetric flask. Add 0.60 mL of diethylene glycol methyl ether, and dilute with methanol to volume.

Standard solution: 0.4 mg/mL of [USP Methylsulfonylmethane RS](#) in *Diluent*. Sonicate at 50° for 1 min, and allow to cool to room temperature.

Sample solution: Finely powder NLT 20 Tablets. Dissolve a portion of the finely powdered material, equivalent to 1 Tablet, in *Diluent*, and sonicate for 15 min at 50°. Allow to cool to room temperature, dilute with *Diluent* to volume, and mix. Quantitatively dilute with *Diluent* to obtain a final concentration of 0.4 mg/mL of methylsulfonylmethane. Transfer 1 mL of the suspension to a 1.5-mL microcentrifuge tube, and centrifuge for 20 s. Use the supernatant.

Chromatographic system

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

Mode: GC

Detector: Flame ionization

Column: 0.53-mm $\times$ 30-m capillary; 5- μ m phase G2 coating

Temperature

Column: 120°

Injector: 250°

Detector: 250°

Carrier gas: Helium

Flow rate: 5 mL/min

Injection size: 1 μ L

Injector type: Split ratio, 2:1

System suitability

Sample: *Standard solution*

Suitability requirements

Relative standard deviation: NMT 2.0% for the peak response ratio of methylsulfonylmethane to diethylene glycol methyl ether from replicate injections

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of the labeled amount of methylsulfonylmethane ($C_2H_6O_2S$) in the portion of Tablets taken:

$$\text{Result} = (R_U/R_S) \times (C_S/C_U) \times 100$$

R_U = peak response ratio of methylsulfonylmethane to diethylene glycol methyl ether from the *Sample solution*

R_S = peak response ratio of methylsulfonylmethane to diethylene glycol methyl ether from the *Standard solution*

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

C_U = nominal concentration of methylsulfonylmethane in the *Sample solution* (mg/mL)

Acceptance criteria: 90.0%–110.0% of the label claim