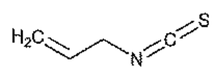
Chromatographic system (see *Chromatography* ⟨621⟩)—The liquid chromatograph is equipped with a 254-nm detector and a 4-mm × 30-cm column that contains packing L1. The flow rate is about 1.5 mL per minute. Chromatograph the *Standard preparation,* and record the peak responses as directed for *Procedure:* the relative retention times are about 0.6 for hypoxanthine and 1.0 for allopurinol; the resolution, *R,* between the analyte and internal standard is not less than 5; and the relative standard deviation for replicate injections is not more than 3.0%.

Procedure—Separately inject equal volumes (about 15 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the quantity, in mg, of allopurinol ($C_5H_4N_4O$) in the portion of Tablets taken by the formula:

$$2.5C(R_U / R_S)$$

in which *C* is the concentration, in µg per mL, of USP Allopurinol RS in the *Standard preparation;* and R_U and R_S are the peak response ratios of allopurinol to hypoxanthine obtained from the *Assay preparation* and the *Standard preparation,* respectively.

Allyl Isothiocyanate



C_4H_5NS 99.15
3-Isothiocyanato-1-propene;
Isothiocyanic acid allyl ester [57-06-7].

DEFINITION
Allyl Isothiocyanate contains NLT 93.0% and NMT 105.0% of allyl isothiocyanate (C_4H_5NS).
[**CAUTION**—Allyl Isothiocyanate is a potent lachrymator, with a pungent, irritating odor. Care should be taken to protect the eyes, to prevent inhalation of fumes, and to avoid tasting.]

IDENTIFICATION
• **A. INFRARED ABSORPTION ⟨197F⟩:** The spectrum exhibits pronounced peaks at about 700, 950, 980, 1300, 1340, 1350, 1410, 1420, 1650, 2100, and 2200 cm^{-1}.

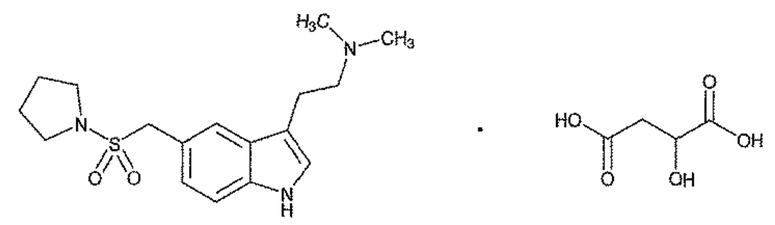
ASSAY
• **PROCEDURE**
Sample solution: Transfer 4 mL into a 100-mL volumetric flask, and dilute with alcohol to volume.
Analysis: Transfer 5.0 mL of the *Sample solution* to a 100-mL conical flask, and add 50.0 mL of 0.1 N silver nitrate VS and 5 mL of ammonia TS. Connect the flask to a reflux condenser, heat on a water bath for 1 h, and allow to cool to room temperature. Disconnect the flask from the condenser, transfer the contents of the conical flask to a 100-mL volumetric flask with the aid of water, and dilute with water to volume. Pass through a dry filter, discarding the first 10 mL of the filtrate. To 50.0 mL of the subsequent filtrate add 5 mL of nitric acid and 2 mL of ferric ammonium sulfate TS, and titrate the excess silver nitrate with 0.1 N ammonium thiocyanate VS. Perform a blank determination, using 5 mL of alcohol in place of the *Sample solution*, and make any necessary correction. Each mL of 0.1 N silver nitrate is equivalent to 4.958 mg of allyl isothiocyanate (C_4H_5NS).
Acceptance criteria: 93.0%–105.0%

IMPURITIES
• **LIMIT OF PHENOLS**
Sample solution: Dilute a 1-mL sample with 5 mL of alcohol.
Analysis: Add 1 drop of ferric chloride TS to the *Sample solution.*
Acceptance criteria: A blue color is not produced immediately.

SPECIFIC TESTS
• **SPECIFIC GRAVITY ⟨841⟩:** 1.013–1.020
• **REFRACTIVE INDEX ⟨831⟩:** 1.527–1.531, determined at 20°
• **DISTILLING RANGE,** *Method I* **⟨721⟩:** 148°–154°

ADDITIONAL REQUIREMENTS
• **PACKAGING AND STORAGE:** Preserve in tight containers.

Almotriptan Malate



$C_{17}H_{25}N_3O_2S \cdot C_4H_6O_5$ 469.55
Pyrrolidine, 1-[[[3-[2-(dimethylamino)ethyl]-1*H*-indol-5-yl]methyl]sulfonyl]-, hydroxybutanedioate (1:1);
1-[({3-[2-(Dimethylamino)ethyl]indol-5-yl}methyl) sulfonyl]pyrrolidine malate (1:1) [181183-52-8].

DEFINITION
Almotriptan Malate contains NLT 98.0% and NMT 102.0% of almotriptan malate ($C_{17}H_{25}N_3O_2S \cdot C_4H_6O_5$), calculated on the anhydrous and solvent-free basis.

IDENTIFICATION
• **A. INFRARED ABSORPTION ⟨197K⟩**
• **B.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY

Change to read:

• **PROCEDURE**
Buffer: 2.72 g/L of monobasic potassium phosphate in water. Adjust with phosphoric acid to a pH of 3.5.
Mobile phase: Methanol and *Buffer* (40:60)
System suitability solution: 0.14 mg/mL each of USP Almotriptan Malate RS and USP Almotriptan Related Compound B RS in *Mobile phase.* Sonication may be used to promote dissolution.
Standard solution: 0.14 mg/mL of USP Almotriptan Malate RS in *Mobile phase.* Sonication may be used to promote dissolution.
Sample solution: 0.14 mg/mL of Almotriptan Malate in *Mobile phase.* Sonication may be used to promote dissolution.
Chromatographic system
(See *Chromatography* ⟨621⟩, *System Suitability.*)
Mode: LC
Detector: UV 230 nm
Column: 4.6-mm × 15-cm; 5-µm packing L10
Flow rate: 1 mL/min
Injection volume: 10 µL
Run time: NLT (IRA 1-May-2017) 2 times the retention time of almotriptan