CONTAMINANTS

• **Articles of Botanical Origin** ⟨561⟩, *Pesticide Residue Analysis*: Meets the requirements

SPECIFIC TESTS

• **Loss on Drying** ⟨731⟩
Sample: Use a powdered sample. If the Aloe is not powdered, crush it in a mortar until it passes through a no. 40 sieve, and mix the ground material before weighing the sample.
Analysis: Dry the *Sample* at 105° for 5 h.
Acceptance criteria: NMT 12.0%

• **Articles of Botanical Origin** ⟨561⟩, *Methods of Analysis, Total Ash*
Acceptance criteria: NMT 4.0%

• **Alcohol-Insoluble Substances**
Sample: 1 g of powdered Aloe
Analysis: Add the *Sample* to 50 mL of alcohol in a flask. Heat the mixture to boiling, and maintain at incipient boiling for 15 min, replacing any loss due to evaporation. Remove from the heat, and shake the mixture at intervals for 1 h. Pass through a small dried and tared filter paper or a dried and tared filtering crucible, and wash the residue on the filter with alcohol until the last washing is colorless. Dry the residue at 105° to constant weight.
Acceptance criteria: The weight of the residue is NMT 10.0% of the weight of Aloe taken.

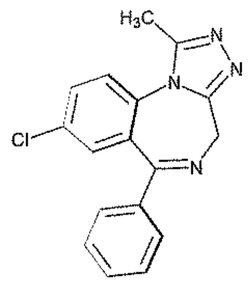
• **Botanical Characteristics**
Curaçao aloe: Brownish-black, opaque masses. Its fractured surface is uneven, waxy, and somewhat resinous.
Cape aloe: Dusky to dark brown irregular masses, the surfaces of which are often covered with a yellowish powder. Its fracture is smooth and glassy.
Powdered Aloe: Yellow, yellowish brown to olive-brown in color. When mounted in olive oil, it appears as greenish-yellow to reddish-brown irregular fragments, the hues of which depend to some extent upon the thickness of the fragments.

ADDITIONAL REQUIREMENTS

• **USP Reference Standards** ⟨11⟩
USP Aloin RS

USP Monographs

Alprazolam



$C_{17}H_{13}ClN_4$ 308.76
4*H*-[1,2,4]Triazolo[4,3-α][1,4]benzodiazepine, 8-chloro-1-methyl-6-phenyl-;
8-Chloro-1-methyl-6-phenyl-4*H*-*s*-triazolo[4,3-α][1,4]benzodiazepine [28981-97-7].

DEFINITION

Alprazolam contains NLT 98.0% and NMT 102.0% of $C_{17}H_{13}ClN_4$.
[**Caution**—Care should be taken to prevent inhaling particles of Alprazolam and exposing the skin to it.]

IDENTIFICATION

• **A. Infrared Absorption** ⟨197M⟩
• **B.** The retention time of the major peak from the *Sample solution* corresponds to that from the *Standard solution*, as obtained in the *Assay*.

ASSAY

• **Procedure**
Diluent: Acetonitrile and water (1:1)
Buffer: 1.4 g/L of monobasic potassium phosphate in water
Mobile phase: Acetonitrile and *Buffer* (1:1)
Standard solution: 25 µg/mL of USP Alprazolam RS in *Diluent*. [Note—The solution is stable for 48 h at room temperature when stored in closed containers.]
Sample solution: 25 µg/mL of Alprazolam in *Diluent*. Sonicate for about 1 min. [Note—The solution is stable for 48 h at room temperature when stored in closed containers.]
Chromatographic system
(See *Chromatography* ⟨621⟩, *System Suitability*.)
Mode: LC
Detector: UV 231 nm
Column: 4.6-mm × 25-cm; packing L1
Column temperature: 40°
Flow rate: 1 mL/min
Injection size: 20 µL
System suitability
Sample: *Standard solution*
Suitability requirements
Tailing factor: NMT 2.0
Relative standard deviation: NMT 2.0%
Analysis
Samples: *Standard solution* and *Sample solution*
Calculate the percentage of alprazolam ($C_{17}H_{13}ClN_4$) in the portion of Alprazolam taken:

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

r_U = peak area from the *Sample solution*
r_S = peak area from the *Standard solution*
C_S = concentration of USP Alprazolam RS in the *Standard solution* (mg/mL)
C_U = concentration of Alprazolam in the *Sample solution* (mg/mL)
Acceptance criteria: 98.0%–102.0%

IMPURITIES

• **Residue on Ignition** ⟨281⟩: NMT 0.5%

Delete the following:

•• **Heavy Metals,** *Method II* ⟨231⟩: 20 ppm •• (Official 1-Jan-2018)

• **Organic Impurities**
Diluent, Buffer, Mobile phase, and **Chromatographic system:** Proceed as directed in the *Assay*.
System suitability solution: 20 µg/mL each of USP Alprazolam RS, USP Alprazolam Related Compound A RS, and USP 2-Amino-5-chlorobenzophenone RS in *Diluent*
Standard solution: 0.25 µg/mL of USP Alprazolam RS in *Diluent*. [Note—When stored in closed containers, the solution is stable for 48 h at room temperature.]
Sample solution: 250 µg/mL of Alprazolam in *Diluent*. Sonicate for about 1 min. [Note—When stored in closed containers, the *Sample solution* is stable for 24 h at room temperature.]
System suitability
Samples: *Standard solution* and *System suitability solution*
[Note—For relative retention times, see *Table 1*.]
Suitability requirements
Resolution: NLT 2.0 between alprazolam related compound A and alprazolam, *System suitability solution*
Relative standard deviation: NMT 5.0%, *Standard solution*