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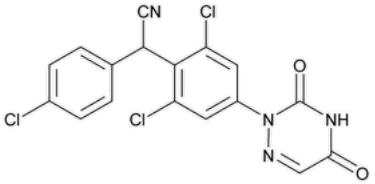
Diclazuril



$C_{17}H_9Cl_3N_4O_2$ 407.64

Benzeneacetonitrile, 2,6-dichloro-α-(4-chlorophenyl)-4-(4,5-dihydro-3,5-dioxo-1,2,4-triazin-2(3*H*)-yl)-;

(*p*-Chlorophenyl)[2,6-dichloro-4-(4,5-dihydro-3,5-dioxo-*as*-triazin-2(3*H*)-yl)phenyl]acetonitrile CAS RN®: 101831-37-2; UNII: K110K1B1VE.

DEFINITION

Diclazuril contains NLT 97.0% and NMT 101.0% of $C_{17}H_9Cl_3N_4O_2$, calculated on the dried basis.

IDENTIFICATION

Change to read:

• ▲**Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: 197K**▲ (CN 1-May-2020)

ASSAY

• **Procedure**

Buffer: Dissolve 6.3 g of ammonium formate in 800 mL of water, adjust with anhydrous formic acid to a pH of 4.0, and add 200 mL of water.

Solution A: Acetonitrile, water, and *Buffer* (3:15:2)

Solution B: Acetonitrile, water, and *Buffer* (85:5:10)

Mobile phase: See the gradient table below.

Time (min)	Solution A (%)	Solution B (%)
0	100	0
20	0	100
25	0	100
26	100	0
36	100	0

Standard solution: 0.5 mg/mL of USP Diclazuril RS in dimethylformamide

System suitability solution: 0.5 mg/mL of USP Diclazuril System Suitability Mixture RS in dimethylformamide

Sample solution: 0.5 mg/mL of Diclazuril in dimethylformamide

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: LC

Detector: UV 230 nm

Column: 4.6-mm × 10-cm; base-deactivated 3-µm packing L1

Flow rate: 1 mL/min

Column temperature: 35°

Injection size: 5 µL

System suitability

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

Suitability requirements

Resolution: NLT 1.9 between diclazuril and diclazuril ketone peaks, *System suitability solution*

Tailing factor: NMT 1.4, *Standard solution*

Relative standard deviation: NMT 2.0%, *Standard solution*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of $C_{17}H_9Cl_3N_4O_2$ in the portion of Diclazuril 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 Diclazuril RS in the *Standard solution* (mg/mL)

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

Acceptance criteria: 97.0%–101.0% on the dried basis

IMPURITIES

Inorganic Impurities

• **Residue On Ignition ⟨281⟩:** NMT 0.1%

Organic Impurities

• **Procedure 1: Residual Solvents ⟨467⟩**

Acceptance criteria: NMT 4000 ppm of *N*,*N*-dimethylformamide

• **Procedure 2**

Buffer, Solution A, Solution B, Mobile phase, Standard solution, System suitability solution, Sample solution, and **Chromatographic system:** Proceed as directed in the *Assay.*

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the area percentage of each impurity, relative to diclazuril, in the portion of Diclazuril taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times (1/F) \times 100$$

r_U = peak response of each impurity from the *Sample solution*

r_S = peak response of diclazuril from the *Standard solution*

C_S = concentration of USP Diclazuril RS in the *Standard solution* (mg/mL)

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

F = relative response factor (see *Impurity Table 1*)

Acceptance criteria

[Note—Disregard any peak observed in the blank. The reporting level for impurities is 0.05%.]

Individual impurities: See *Impurity Table 1*.

Total impurities: NMT 1.5%

Impurity Table 1

Name	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (%)
6-Carboxylic acid[a]	0.62	0.85	0.50
6-Carboxamide[b]	0.80	0.92	0.50
Diclazuril	1.00	—	—
Ketone[c]	1.03	0.52	0.10
4-Amino derivative[d]	1.09	0.81	0.50
Des-cyano derivative[e]	1.16	1.1	0.50

Name	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (%)
Trichlorodiphenyl acetonitrile[f]	1.24	0.71	0.50
Any other individual impurity	—	1.0	0.20

[a] (*RS*)-2-[3,5-Dichloro-4-[(4-chlorophenyl)cyanomethyl]phenyl]-3,5-dioxo-2,3,4,5-tetrahydro-1,2,4-triazine-6-carboxylic acid.

[b] (*RS*)-2-[3,5-Dichloro-4-[(4-chlorophenyl)cyanomethyl]phenyl]-3,5-dioxo-2,3,4,5-tetrahydro-1,2,4-triazine-6-carboxamide.

[c] 2-[3,5-Dichloro-4-(4-chlorobenzoyl)phenyl]-1,2,4-triazine-3,5(2*H*,4*H*)-dione.

[d] (*RS*)-2-(4-Amino-2,6-dichlorophenyl)-2-(4-chlorophenyl)acetonitrile.

[e] 2-[3,5-Dichloro-4-(4-chlorobenzyl)phenyl]-1,2,4-triazine-3,5(2*H*,4*H*)-dione.

[f] (*RS*)-2-(4-Chlorophenyl)-2-(2,6-dichlorophenyl)acetonitrile.

SPECIFIC TESTS

• **Loss On Drying, ⟨731⟩:** Dry a sample at between 100° and 105° under a vacuum for 4 h: it loses NMT 0.5% of its weight.

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in well-closed containers, and store at room temperature.

• **Labeling:** Label it to indicate that it is for veterinary use only.

• **USP Reference Standards ⟨11⟩**

USP Diclazuril RS

USP Diclazuril System Suitability Mixture RS

Contains diclazuril and specified impurities.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
DICLAZURIL	Documentary Standards Support	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

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