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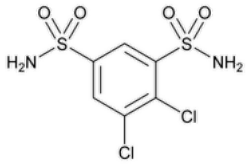
Dichlorphenamide

Change to read:



$C_6H_6Cl_2N_2O_4S_2$ ▲305.14▲ (USP 1-May-2022)

1,3-Benzenedisulfonamide, 4,5-dichloro-;

4,5-Dichloro-*m*-benzenedisulfonamide;

▲4,5-Dichlorobenzene-1,3-disulfonamide▲ (USP 1-May-2022) CAS RN®: 120-97-8; ▲UNII: VVJ6673MHY.▲ (USP 1-May-2022)

DEFINITION

Dichlorphenamide contains NLT 98.0% and NMT 101.0% of dichlorphenamide ($C_6H_6Cl_2N_2O_4S_2$), calculated on the dried basis.

IDENTIFICATION

• **A. Spectroscopic Identification Tests ⟨197⟩, *Ultraviolet-Visible Spectroscopy*:** 197U

Sample solution: 0.1 mg/mL of Dichlorphenamide in 0.4 mg/mL of sodium hydroxide solution

Analysis: To 10 mL of the *Sample solution* add 0.1 mL of hydrochloric acid.

Acceptance criteria: It exhibits absorption maxima at 295 ± 2 nm and at 286 ± 2 nm. The ratio A_{295}/A_{286} is between 0.90 and 1.00.

• **B. Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*:** 197A or 197M

ASSAY

• **Procedure**

Solution A: 2.4 g/L of monobasic sodium phosphate and 2.8 g/L of dibasic sodium phosphate in water

Solution B: Acetonitrile

Mobile phase: See *Table 1*.

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	65	35
6	65	35
23	40	60
23.1	65	35
30	65	35

Diluent: Acetonitrile and water (1:1)

Standard solution: 0.5 mg/mL of USP Dichlorphenamide RS in *Diluent*

Sample solution: 0.5 mg/mL of Dichlorphenamide in *Diluent*

Chromatographic system

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

Mode: LC

Detector: UV 280 nm

Column: 4.6-mm × 15-cm; 2.7-µm packing L1

Flow rate: 0.4 mL/min

Injection volume: 10 µL

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: NMT 2.0

Relative standard deviation: NMT 0.73%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of dichlorphenamide ($C_6H_6Cl_2N_2O_4S_2$) in the portion of Dichlorphenamide taken:

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

r_U = peak response of dichlorphenamide from the *Sample solution*

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

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

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

Acceptance criteria: 98.0%–101.0% on the dried basis

IMPURITIES

• **Residue on Ignition ⟨281⟩:** NMT 0.2%

Change to read:

• **Organic Impurities**

Solution A, Solution B, Diluent, and **Chromatographic system:** Proceed as directed in the *Assay*.

Standard solution: 0.002 mg/mL of USP Dichlorphenamide RS in *Diluent*

Sample solution: 2 mg/mL of Dichlorphenamide in *Diluent*

System suitability

Sample: *Standard solution*

Suitability requirements

Tailing factor: 0.8–1.5

Relative standard deviation: NMT 10.0%

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of each ▲individual▲ (USP 1-May-2022) impurity in the portion of Dichlorphenamide taken:

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

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

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

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

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

F = relative response factor (see *Table 2*)

Acceptance criteria: See *Table 2*. The reporting threshold is 0.05%.

Table 2

Name	Relative Retention Time	Relative Response Factor	Acceptance Criteria, NMT (%)
Dichlorphenamide	1.0	1.0	—
3,4-Dichlorobenzenesulfonamide	2.0	0.55	0.15
Any individual impurity	—	1.0	0.10
Total impurities	—	—	1.0

• **Chloride and Sulfate ⟨221⟩, *Chloride***

Standard solution: 0.28 mL of 0.020 N hydrochloric acid

Sample solution: To 100 mg of Dichlorphenamide add 25 mL of 8 N nitric acid, and warm on a steam bath to dissolve. Cool to room temperature.

Acceptance criteria: 0.20%; the *Sample solution* shows no more chloride than the *Standard solution*.

Delete the following:

▲• **Selenium ⟨291⟩**

Sample: A 100-mg specimen mixed with 100 mg of magnesium oxide

Acceptance criteria: NMT 30 ppm▲ (USP 1-May-2022)

SPECIFIC TESTS

• **Loss on Drying ⟨731⟩**

Analysis: Dry at a pressure of NMT 5 mm of mercury at 100° to constant weight.

Acceptance criteria: NMT 1.0%

ADDITIONAL REQUIREMENTS

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

• **USP Reference Standards ⟨11⟩**

USP Dichlorphenamide RS

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

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

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
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