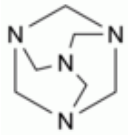
Edition: BP 2025 (Ph. Eur. 11.6 update)

Methenamine

General Notices

(Ph. Eur. monograph 1545)



$C_6H_{12}N_4$ 140.2 *100-97-0*

Action and use

Anti-infective.

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DEFINITION

1,3,5,7-Tetraazotricyclo[3.3.1.1^{3,7}]decane.

Content

99.0 per cent to 100.5 per cent (dried substance).

CHARACTERS

Appearance

White or almost white, crystalline powder or colourless crystals.

Solubility

Freely soluble in water, soluble in ethanol (96 per cent) and in methylene chloride.

IDENTIFICATION

First identification: A.

Second identification: B, C, D.

A. Infrared absorption spectrophotometry (*2.2.24*).

Comparison *methenamine CRS*.

B. To 1 mL of solution S (see Tests) add 1 mL of *sulfuric acid R* and immediately heat to boiling. Allow to cool. To 1 mL of the solution add 4 mL of *water R* and 5 mL of *acetylacetone reagent R1*. Heat on a water-bath for 5 min. An intense yellow colour develops.
C. To 1 mL of solution S add 1 mL of *dilute sulfuric acid R* and immediately heat to boiling. The solution gives the reaction of ammonium salts and salts of volatile bases (*2.3.1*).
D. Dissolve 10 mg in 5 mL of *water R* and acidify with *dilute hydrochloric acid R*. Add 1 mL of *potassium iodobismuthate solution R*. An orange precipitate is formed immediately.

TESTS

Solution S

Dissolve 10.0 g in *carbon dioxide-free water R* prepared from *distilled water R* and dilute to 100 mL with the same solvent.

Appearance of solution

Solution S is clear (*2.2.1*) and colourless (*2.2.2, Method II*).

Acidity or alkalinity

To 5 mL of solution S add 0.1 mL of *phenolphthalein solution R*. Not more than 0.2 mL of *0.1 M hydrochloric acid* or *0.1 M sodium hydroxide* is required to change the colour of the indicator.

Free formaldehyde

Maximum 50 ppm.

Dissolve 0.8 g in *water R* and dilute to 8 mL with the same solvent. Add 2 mL of *ammoniacal silver nitrate solution R*. After 5 min, any grey colour in the solution is not more intense than that in a standard prepared at the same time and in the same manner with a mixture of 8 mL of freshly prepared *formaldehyde standard solution (5 ppm CH_2O) R* and 2 mL of *ammoniacal silver nitrate solution R*.

Chlorides (*2.4.4*)

Maximum 100 ppm.

Dilute 5 mL of solution S to 15 mL with *water R*.

Sulfates (*2.4.13*)

Maximum 100 ppm, determined on solution S.

Ammonium (*2.4.1*)

Maximum 50 ppm.

Dilute 2 mL of freshly prepared solution S to 13 mL with *water R*. Add 2 mL of *dilute sodium hydroxide solution R*.

Loss on drying (*2.2.32*)

Maximum 2.0 per cent, determined on 1.000 g by drying in a desiccator.

ASSAY

Dissolve 0.100 g in 30 mL of *methanol R*. Titrate with *0.1 M perchloric acid*, determining the end-point potentiometrically (*2.2.20*).

1 mL of *0.1 M perchloric acid* is equivalent to 14.02 mg of $C_6H_{12}N_4$.

STORAGE

Protected from light.

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