

**Content:** 99.5 per cent to 100.5 per cent (anhydrous substance).

#### CHARACTERS

**Appearance:** white or almost white, crystalline powder, colourless crystals or granules.

**Solubility:** very soluble in water, freely soluble in ethanol (96 per cent).

mp: about 153 °C, with decomposition.

#### IDENTIFICATION

**First identification:** B, E.

**Second identification:** A, C, D, E.

A. Dissolve 1 g in 10 ml of *water R*. The solution is strongly acidic (2.2.4).

B. Infrared absorption spectrophotometry (2.2.24).

**Preparation:** dry the substance to be examined and the reference substance at 100-105 °C for 2 h.

**Comparison:** *anhydrous citric acid CRS*.

C. Add about 5 mg to a mixture of 1 ml of *acetic anhydride R* and 3 ml of *pyridine R*. A red colour develops.

D. Dissolve 0.5 g in 5 ml of *water R*, neutralise using 1 M *sodium hydroxide* (about 7 ml), add 10 ml of *calcium chloride solution R* and heat to boiling. A white precipitate is formed.

E. Water (see Tests).

#### TESTS

**Appearance of solution.** The solution is clear (2.2.1) and not more intensely coloured than reference solution Y<sub>7</sub>, BY<sub>7</sub> or GY<sub>7</sub> (2.2.2, *Method II*).

Dissolve 2.0 g in *water R* and dilute to 10 ml with the same solvent.

**Readily carbonisable substances.** To 1.0 g in a cleaned test tube add 10 ml of *sulphuric acid R* and immediately heat the mixture in a water-bath at 90 ± 1 °C for 60 min. Cool rapidly immediately afterwards. The solution is not more intensely coloured than a mixture of 1 ml of red primary solution and 9 ml of yellow primary solution (2.2.2, *Method I*).

**Oxalic acid:** maximum 360 ppm, calculated as anhydrous oxalic acid.

Dissolve 0.80 g in 4 ml of *water R*. Add 3 ml of *hydrochloric acid R* and 1 g of *zinc R* in granules. Boil for 1 min.

Allow to stand for 2 min. Transfer the supernatant liquid to a test-tube containing 0.25 ml of a 10 g/l solution of *phenylhydrazine hydrochloride R* and heat to boiling.

Cool rapidly, transfer to a graduated cylinder and add an equal volume of *hydrochloric acid R* and 0.25 ml of a 50 g/l solution of *potassium ferricyanide R*. Shake and allow to stand for 30 min. Any pink colour in the solution is not more intense than that in a standard prepared at the same time in the same manner using 4 ml of a 0.1 g/l solution of *oxalic acid R*.

**Sulphates** (2.4.13): maximum 150 ppm.

Dissolve 2.0 g in *distilled water R* and dilute to 30 ml with the same solvent.

**Aluminium** (2.4.17): maximum 0.2 ppm, if intended for use in the manufacture of dialysis solutions.

**Prescribed solution.** Dissolve 20 g in 100 ml of *water R* and add 10 ml of *acetate buffer solution pH 6.0 R*.

**Reference solution.** Mix 2 ml of *aluminium standard solution (2 ppm Al) R*, 10 ml of *acetate buffer solution pH 6.0 R* and 98 ml of *water R*.

**Blank solution.** Mix 10 ml of *acetate buffer solution pH 6.0 R* and 100 ml of *water R*.

**Heavy metals** (2.4.8): maximum 10 ppm.

Dissolve 5.0 g in several portions in 39 ml of *dilute sodium hydroxide solution R* and dilute to 50 ml with *distilled water R*. 12 ml of the solution complies with test A.

Prepare the reference solution using *lead standard solution (1 ppm Pb) R*.

**Water** (2.5.12): maximum 1.0 per cent, determined on 2.000 g.

**Sulphated ash** (2.4.14): maximum 0.1 per cent, determined on 1.0 g.

**Bacterial endotoxins** (2.6.14): less than 0.5 IU/mg, if intended for use in the manufacture of parenteral dosage forms without a further appropriate procedure for the removal of bacterial endotoxins.

#### ASSAY

Dissolve 0.550 g in 50 ml of *water R*. Titrate with 1 M *sodium hydroxide*, using 0.5 ml of *phenolphthalein solution R* as indicator.

1 ml of 1 M *sodium hydroxide* is equivalent to 64.03 mg of C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>.

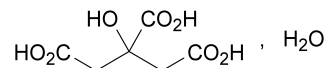
#### LABELLING

The label states, where applicable, that the substance is intended for use in the manufacture of dialysis solutions.

01/2008:0456  
corrected 6.0

## CITRIC ACID MONOHYDRATE

### Acidum citricum monohydricum



C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>·H<sub>2</sub>O  
[5949-29-1]

M<sub>r</sub> 210.1

#### DEFINITION

2-Hydroxypropane-1,2,3-tricarboxylic acid monohydrate.

**Content:** 99.5 per cent to 100.5 per cent (anhydrous substance).

#### CHARACTERS

**Appearance:** white or almost white, crystalline powder, colourless crystals or granules, efflorescent.

**Solubility:** very soluble in water, freely soluble in ethanol (96 per cent).

#### IDENTIFICATION

**First identification:** B, E.

**Second identification:** A, C, D, E.

A. Dissolve 1 g in 10 ml of *water R*. The solution is strongly acidic (2.2.4).

B. Infrared absorption spectrophotometry (2.2.24).

**Preparation:** dry the substance to be examined and the reference substance at 100-105 °C for 2 h.

**Comparison:** *citric acid monohydrate CRS*.

C. Add about 5 mg to a mixture of 1 ml of *acetic anhydride R* and 3 ml of *pyridine R*. A red colour develops.

D. Dissolve 0.5 g in 5 ml of *water R*, neutralise using 1 M *sodium hydroxide* (about 7 ml), add 10 ml of *calcium chloride solution R* and heat to boiling. A white precipitate is formed.

E. Water (see Tests).

## TESTS

**Appearance of solution.** The solution is clear (2.2.1) and not more intensely coloured than reference solution Y<sub>7</sub>, BY<sub>7</sub> or GY<sub>7</sub> (2.2.2, Method II).

Dissolve 2.0 g in *water R* and dilute to 10 ml with the same solvent.

**Readily carbonisable substances.** To 1.0 g in a cleaned test tube add 10 ml of *sulphuric acid R* and immediately heat the mixture in a water-bath at 90 ± 1 °C for 60 min. Cool rapidly immediately afterwards. The solution is not more intensely coloured than a mixture of 1 ml of red primary solution and 9 ml of yellow primary solution (2.2.2, Method I).

**Oxalic acid:** maximum 360 ppm, calculated as anhydrous oxalic acid.

Dissolve 0.80 g in 4 ml of *water R*. Add 3 ml of *hydrochloric acid R* and 1 g of *zinc R* in granules. Boil for 1 min. Allow to stand for 2 min. Transfer the supernatant liquid to a test-tube containing 0.25 ml of a 10 g/l solution of *phenylhydrazine hydrochloride R* and heat to boiling. Cool rapidly, transfer to a graduated cylinder and add an equal volume of *hydrochloric acid R* and 0.25 ml of a 50 g/l solution of *potassium ferricyanide R*. Shake and allow to stand for 30 min. Any pink colour in the solution is not more intense than that in a standard prepared at the same time in the same manner using 4 ml of a 0.1 g/l solution of *oxalic acid R*.

**Sulphates (2.4.13):** maximum 150 ppm.

Dissolve 2.0 g in *distilled water R* and dilute to 30 ml with the same solvent.

**Aluminium (2.4.17):** maximum 0.2 ppm, if intended for use in the manufacture of dialysis solutions.

**Prescribed solution.** Dissolve 20 g in 100 ml of *water R* and add 10 ml of *acetate buffer solution pH 6.0 R*.

**Reference solution.** Mix 2 ml of *aluminium standard solution (2 ppm Al) R*, 10 ml of *acetate buffer solution pH 6.0 R* and 98 ml of *water R*.

**Blank solution.** Mix 10 ml of *acetate buffer solution pH 6.0 R* and 100 ml of *water R*.

**Heavy metals (2.4.8):** maximum 10 ppm.

Dissolve 5.0 g in several portions in 39 ml of *dilute sodium hydroxide solution R* and dilute to 50 ml with *distilled water R*. 12 ml of the solution complies with test A. Prepare the reference solution using *lead standard solution (1 ppm Pb) R*.

**Water (2.5.12):** 7.5 per cent to 9.0 per cent, determined on 0.500 g.

**Sulphated ash (2.4.14):** maximum 0.1 per cent, determined on 1.0 g.

**Bacterial endotoxins (2.6.14):** less than 0.5 IU/mg, if intended for use in the manufacture of parenteral dosage forms without a further appropriate procedure for the removal of bacterial endotoxins.

## ASSAY

Dissolve 0.550 g in 50 ml of *water R*. Titrate with 1 M *sodium hydroxide*, using 0.5 ml of *phenolphthalein solution R* as indicator.

1 ml of 1 M *sodium hydroxide* is equivalent to 64.03 mg of C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>.

## STORAGE

In an airtight container.

## LABELLING

The label states, where applicable, that the substance is intended for use in the manufacture of dialysis solutions.

01/2008:1609

## CITRONELLA OIL

## Citronellae aetheroleum

## DEFINITION

Oil obtained by steam distillation from the fresh or partially dried aerial parts of *Cymbopogon winterianus* Jowitt.

## CHARACTERS

Pale yellow to brown-yellow liquid, with a very strong odour of citronellal.

## IDENTIFICATION

**First identification:** B.

**Second identification:** A.

A. Thin-layer chromatography (2.2.27).

**Test solution.** Dilute 0.1 g of citronella oil in 10.0 ml of *alcohol R*.

**Reference solution.** Dilute 20 µl of *citronellal R* in 10.0 ml of *alcohol R*.

**Plate:** TLC silica gel plate R.

**Mobile phase:** *ethyl acetate R*, *toluene R* (10:90 V/V).

**Application:** 5 µl, as bands.

**Development:** over a path of 15 cm.

**Drying:** in air.

**Detection:** spray with *anisaldehyde solution R* and heat at 100-105 °C for 10 min. Examine in ultraviolet light at 365 nm.

**Result:** see below the sequence of the zones present in the chromatograms obtained with the reference and test solutions. Furthermore, other zones are present in the chromatogram obtained with the test solution.

Top of the plate	
Citronellal: a violet zone	A zone similar in colour to the citronellal zone An orange zone (citronellol-geraniol)
Reference solution	Test solution

B. Examine the chromatograms obtained in the test for chromatographic profile.

**Results:** the characteristic peaks in the chromatogram obtained with the test solution are similar in retention time to those in the chromatogram obtained with the reference solution. Neral and geraniol may be absent in the chromatogram obtained with the test solution.

## TESTS

**Relative density (2.2.5):** 0.881 to 0.895.

**Refractive index (2.2.6):** 1.463 to 1.475.

**Optical rotation (2.2.7):** -4° to +1.5°.

**Chromatographic profile.** Gas chromatography (2.2.28): use the normalisation procedure.

**Test solution.** The substance to be examined.