

filtrate and washings to *blue litmus paper R* with *dilute hydrochloric acid R* and add 2 ml in excess. Boil until the vapour no longer darkens *lead acetate paper R* and continue boiling, if necessary, until the volume is reduced to about 10 ml. Cool, add 15 ml of *sodium carbonate solution R*, allow to stand for 5 min and filter. Dilute the filtrate to 100 ml with *water R*. To 5 ml of this solution add 2 ml of *cupri-tartaric solution R* and boil for 1 min. Allow to stand for 1 min. No red precipitate is formed.

**Chlorides** (2.4.4): maximum 0.06 per cent.

Dilute 0.8 ml of solution S to 15 ml with *water R*.

**Oxalates.** Dissolve 5.0 g in a mixture of 10 ml of *dilute sulphuric acid R* and 40 ml of *water R*. Shake the solution with 50 ml of *ether R* for 5 min. Separate the aqueous layer and shake it with 20 ml of *ether R* for 5 min. Combine the ether layers, evaporate to dryness and dissolve the residue in 15 ml of *water R*. Filter, boil the filtrate until the volume is reduced to 5 ml and add 1 ml of *dilute acetic acid R* and 1.5 ml of *calcium chloride solution R*. Allow to stand for 30 min. No precipitate is formed.

**Sulphates** (2.4.13): maximum 500 ppm.

To 3.0 ml of solution S add 3 ml of *acetic acid R* and dilute to 15 ml with *distilled water R*. Examine the solutions against the light.

**Arsenic** (2.4.2, *Method A*): maximum 2 ppm, determined on 0.5 g.

**Barium.** Dilute 10 ml of solution S to 50 ml with *distilled water R* and add 5 ml of *dilute sulphuric acid R*. Allow to stand for 5 min. Any opalescence in the solution is not more intense than that in a mixture of 10 ml of solution S and 45 ml of *distilled water R*.

**Ferric ion:** maximum 1.0 per cent.

In a ground-glass-stoppered flask, dissolve 5.00 g in a mixture of 10 ml of *hydrochloric acid R* and 100 ml of *carbon dioxide-free water R*. Add 3 g of *potassium iodide R*, close the flask and allow to stand protected from light for 5 min. Titrate with 0.1 M *sodium thiosulphate*, using 0.5 ml of *starch solution R*, added towards the end of the titration, as indicator. Carry out a blank titration. Not more than 9.0 ml of 0.1 M *sodium thiosulphate* is used.

**Heavy metals** (2.4.8): maximum 20 ppm.

Thoroughly mix 2.5 g with 0.5 g of *magnesium oxide R1* in a silica crucible. Ignite to dull redness until a homogeneous mass is obtained. Heat at  $800 \pm 50$  °C for about 1 h, allow to cool and take up the residue in 20 ml of hot *hydrochloric acid R*. Allow to cool. Transfer the liquid to a separating funnel and shake for 3 min with 3 quantities, each of 20 ml, of methyl isobutyl ketone saturated with hydrochloric acid (prepared by shaking 100 ml of freshly distilled *methyl isobutyl ketone R* with 1 ml of *hydrochloric acid R*). Allow to stand, separate the aqueous layer, reduce to half its volume by boiling, allow to cool and dilute to 25 ml with *water R*. Neutralise 10 ml of this solution to *red litmus paper R* using *dilute ammonia R1* and dilute to 20 ml with *water R*. 12 ml of the solution complies with test A. Prepare the reference solution using *lead standard solution (1 ppm Pb) R*.

**Loss on drying** (2.2.32): 5.0 per cent to 10.5 per cent, determined on 0.500 g by drying in an oven at 105 °C for 5 h.

**Microbial contamination.** Total viable aerobic count (2.6.12) not more than  $10^3$  micro-organisms per gram, determined by plate count.

## ASSAY

Dissolve 0.5 g of *sodium hydrogen carbonate R* in a mixture of 30 ml of *dilute sulphuric acid R* and 70 ml of *water R*. When the effervescence stops, dissolve 1.00 g of the substance to be examined with gentle shaking. Using 0.1 ml of *ferroin R* as indicator, titrate with 0.1 M *ammonium and cerium nitrate* until the red colour disappears.

1 ml of 0.1 M *ammonium and cerium nitrate* is equivalent to 5.585 mg of iron(II).

## STORAGE

Protected from light.

01/2008:2340  
corrected 6.0

# FERROUS SULPHATE, DRIED

## Ferrosi sulfas desiccatus

FeSO<sub>4</sub>

M<sub>r</sub> 151.9

## DEFINITION

Hydrated ferrous sulphate from which part of the water of hydration has been removed by drying.

*Content:* 86.0 per cent to 90.0 per cent.

## CHARACTERS

*Appearance:* greyish-white powder.

*Solubility:* slowly but freely soluble in water, very soluble in boiling water, practically insoluble in ethanol (96 per cent).

It is oxidised in moist air, becoming brown.

## IDENTIFICATION

A. It gives the reactions of sulphates (2.3.1).

B. It gives reaction (a) of iron (2.3.1).

C. It complies with the limits of the assay.

## TESTS

**Solution S.** Dissolve 2.00 g in a 5 per cent V/V solution of *lead-free nitric acid R* and dilute to 100.0 ml with the same acid.

**pH** (2.2.3): 3.0 to 4.0.

Dissolve 1.0 g in *carbon dioxide-free water R* and dilute to 20 ml with the same solvent.

**Chlorides** (2.4.4): maximum 300 ppm.

Dissolve 2.5 g in *water R*, add 0.5 ml of *dilute sulphuric acid R* and dilute to 50 ml with *water R*. Dilute 3.3 ml of this solution to 10 ml with *water R* and add 5 ml of *dilute nitric acid R*. Prepare the standard using a mixture of 10 ml of *chloride standard solution (5 ppm Cl) R* and 5 ml of *dilute nitric acid R*. Use 0.15 ml of *silver nitrate solution R2* in this test.

**Chromium:** maximum  $1.00 \times 10^2$  ppm.

Atomic absorption spectrometry (2.2.23, *Method I*).

*Test solution.* Solution S.

*Reference solutions.* Prepare the reference solutions using *chromium standard solution (100 ppm Cr) R*, diluted as necessary with a 5 per cent V/V solution of *lead-free nitric acid R*.

*Source:* chromium hollow-cathode lamp using a transmission band preferably of 1 nm.

*Wavelength:* 357.9 nm.

*Atomisation device:* air-acetylene flame.

**Copper:** maximum 50.0 ppm.

Atomic absorption spectrometry (2.2.23, *Method II*).

*Test solution.* Solution S.

*Reference solutions.* Prepare the reference solutions using *copper standard solution (0.1 per cent Cu) R*, diluted as necessary with a 5 per cent V/V solution of *lead-free nitric acid R*.

*Source:* copper hollow-cathode lamp using a transmission band preferably of 1 nm.

*Wavelength:* 324.7 nm.

*Atomisation device:* air-acetylene flame.

**Ferric ions:** maximum 0.50 per cent.

In a ground-glass-stoppered flask, dissolve 5.00 g in a mixture of 10 ml of *hydrochloric acid R* and 100 ml of *carbon dioxide-free water R*. Add 3 g of *potassium iodide R*, close the flask and allow to stand in the dark for 5 min. Titrate the liberated iodine with *0.1 M sodium thiosulphate*, using 0.5 ml of *starch solution R*, added towards the end of titration, as indicator. Carry out a blank test in the same conditions. Not more than 4.5 ml of *0.1 M sodium thiosulphate* is used.

**Manganese:** maximum 0.10 per cent.

Atomic absorption spectrometry (2.2.23, *Method II*).

*Test solution.* Dilute 1.0 ml of solution S to 20.0 ml with a 5 per cent V/V solution of *lead-free nitric acid R*.

*Reference solutions.* Prepare the reference solutions using *manganese standard solution (1000 ppm Mn) R*, diluted as necessary with a 5 per cent V/V solution of *lead-free nitric acid R*.

*Source:* manganese hollow-cathode lamp using a transmission band preferably of 1 nm.

*Wavelength:* 279.5 nm.

*Atomisation device:* air-acetylene flame.

**Nickel:** maximum  $1.00 \times 10^2$  ppm.

Atomic absorption spectrometry (2.2.23, *Method I*).

*Test solution.* Solution S.

*Reference solutions.* Prepare the reference solutions using *nickel standard solution (10 ppm Ni) R*, diluted as necessary with a 5 per cent V/V solution of *lead-free nitric acid R*.

*Source:* nickel hollow-cathode lamp using a transmission band preferably of 1 nm.

*Wavelength:* 232.0 nm.

*Atomisation device:* air-acetylene flame.

**Zinc:** maximum  $1.00 \times 10^2$  ppm.

Atomic absorption spectrometry (2.2.23, *Method II*).

*Test solution.* Solution S.

*Reference solutions.* Prepare the reference solutions using *zinc standard solution (100 ppm Zn) R*, diluted as necessary with a 5 per cent V/V solution of *lead-free nitric acid R*.

*Source:* zinc hollow-cathode lamp using a transmission band preferably of 1 nm.

*Wavelength:* 213.9 nm.

*Atomisation device:* air-acetylene flame.

#### ASSAY

Dissolve 2.5 g of *sodium hydrogen carbonate R* in a mixture of 150 ml of *water R* and 10 ml of *sulphuric acid R*. When the effervescence ceases, add to the solution 0.140 g of the

substance to be examined and dissolve with gentle shaking. Add 0.1 ml of *ferroin R* and titrate with *0.1 M ammonium and cerium nitrate* until the red colour disappears.

1 ml of *0.1 M ammonium and cerium nitrate* is equivalent to 15.19 mg of  $\text{FeSO}_4$ .

#### STORAGE

In an airtight container.

01/2008:0083

## FERROUS SULPHATE HEPTAHYDRATE

### Ferrosi sulfas heptahydricus

$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$   
[7720-78-7]

$M_r$  278.0

#### DEFINITION

*Content:* 98.0 per cent to 105.0 per cent.

#### CHARACTERS

*Appearance:* light green, crystalline powder or bluish-green crystals, efflorescent in air.

*Solubility:* freely soluble in water, very soluble in boiling water, practically insoluble in alcohol.

Ferrous sulphate is oxidised in moist air, becoming brown.

#### IDENTIFICATION

A. It gives the reaction of sulphates (2.3.1).

B. It gives reaction (a) of iron (2.3.1).

C. It complies with the limits of the assay.

#### TESTS

**Solution S.** Dissolve 2.5 g in *carbon dioxide-free water R*, add 0.5 ml of *dilute sulphuric acid R* and dilute to 50 ml with *water R*.

**Appearance of solution.** Solution S is not more opalescent than reference suspension II (2.2.1).

**pH** (2.2.3): 3.0 to 4.0.

Dissolve 0.5 g in *carbon dioxide-free water R* and dilute to 10 ml with the same solvent.

**Chlorides** (2.4.4): maximum 300 ppm.

Dilute 3.3 ml of solution S to 10 ml with *water R* and add 5 ml of *dilute nitric acid R*. The solution complies with the limit test for chlorides. Prepare the standard using a mixture of 10 ml of *chloride standard solution (5 ppm Cl) R* and 5 ml of *dilute nitric acid R*. Use 0.15 ml of *silver nitrate solution R2* in this test.

**Ferric ions:** maximum 0.5 per cent.

In a ground-glass-stoppered flask, dissolve 5.00 g in a mixture of 10 ml of *hydrochloric acid R* and 100 ml of *carbon dioxide-free water R*. Add 3 g of *potassium iodide R*, close the flask and allow to stand in the dark for 5 min. Titrate the liberated iodine with *0.1 M sodium thiosulphate*, using 0.5 ml of *starch solution R*, added towards the end of the titration, as indicator. Carry out a blank test in the same conditions. Not more than 4.5 ml of *0.1 M sodium thiosulphate* is used, taking into account the blank titration.

**Manganese:** maximum 0.1 per cent.

Dissolve 1.0 g in 40 ml of *water R*, add 10 ml of *nitric acid R* and boil until red fumes are evolved. Add 0.5 g of *ammonium persulphate R* and boil for 10 min. Discharge any pink colour by adding dropwise a 50 g/l solution of *sodium*