Limit:

– *hydrazine*: any spot corresponding to salicylaldehyde azine in the chromatogram obtained with the test solution is not more intense than the spot in the chromatogram obtained with the reference solution (1 ppm).

Monomers: maximum 0.1 per cent.

Dissolve 10.0 g in 30 ml of *methanol R* and add slowly 20.0 ml of *iodine bromide solution R*. Allow to stand for 30 min protected from light with repeated shaking. Add 10 ml of a 100 g/l solution of *potassium iodide R* and titrate with *0.1 M sodium thiosulphate* until a yellow colour is obtained. Continue titration dropwise until the solution becomes colourless. Carry out a blank titration. Not more than 1.8 ml of *0.1 M sodium thiosulphate* is used.

Impurity A. Liquid chromatography (*2.2.29*).

Test solution. Dissolve 100 mg of the substance to be examined in *water R* and dilute to 50.0 ml with the same solvent.

Reference solution. Dissolve 100 mg of *2-pyrrolidone R* in *water R* and dilute to 100 ml with the same solvent. Dilute 1.0 ml to 100.0 ml with *water R*.

Precolumn:

– *size*: *l* = 0.025 m, Ø = 4 mm,

– *stationary phase*: *end-capped octadecylsilyl silica gel for chromatography R* (5 µm).

Column:

– *size*: *l* = 0.25 m, Ø = 4 mm,

– *stationary phase*: spherical *aminohexadecylsilyl silica gel for chromatography R* (5 µm),

– *temperature*: 30 °C.

Mobile phase: *water R*, adjusted to pH 2.4 with *phosphoric acid R*.

Flow rate: 1 ml/min.

Detection: spectrophotometer at 205 nm. A detector is placed between the precolumn and the analytical column. A second detector is placed after the analytical column.

Injection: 10 µl. When impurity A has left the precolumn (after about 1.2 min) switch the flow directly from the pump to the analytical column. Before the next chromatogram is run, wash the precolumn by reversed flow.

Limit:

– *impurity A*: not more than the area of the principal peak obtained with the reference solution (0.5 per cent).

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

12 ml of solution S complies with limit test A. Prepare the standard using *lead standard solution (2 ppm Pb) R*.

Loss on drying (*2.2.32*): maximum 5.0 per cent, determined on 0.500 g by drying in an oven at 100-105 °C.

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

Viscosity, expressed as *K*-value. Dilute 5.0 ml of solution S to 50.0 ml with *water R*. Allow to stand for 1 h and determine the viscosity (*2.2.9*) of the solution at 25 ± 0.1 °C using viscometer No. 1 with a minimum flow time of 100 s. Calculate the *K*-value from the expression:

$$\frac{1.5log\eta - 1}{0.15 + 0.003c} + \frac{\sqrt{300c\,log\eta + (c + 1.5c\,log\eta)^2}}{0.15c + 0.003c^2}$$

c = percentage concentration (g/100 ml) of the substance to be examined, calculated with reference to the dried substance,

η = viscosity of the solution relative to that of water.

ASSAY

Ethenyl acetate. Determine the saponification value (*2.5.6*) on 2.00 g of the substance to be examined. Multiply the result obtained by 0.1534 to obtain the percentage content of the ethenyl acetate component.

Nitrogen. Carry out the determination of nitrogen (*2.5.9*) using 30.0 mg of the substance to be examined and 1 g of a mixture of 3 parts of *copper sulphate R* and 997 parts of *dipotassium sulphate R*, heating until a clear, light green solution is obtained and then for a further 45 min.

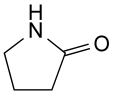
STORAGE

In an airtight container.

LABELLING

The label states the *K*-value.

IMPURITIES



A. pyrrolidin-2-one (2-pyrrolidone).

01/2005:0893

COPPER SULPHATE, ANHYDROUS

Cupri sulfas anhydricus

$CuSO_4$ M_r 159.6

DEFINITION

Anhydrous copper sulphate contains not less than 99.0 per cent and not more than the equivalent of 101.0 per cent of $CuSO_4$, calculated with reference to the dried substance.

CHARACTERS

A greenish-grey powder, very hygroscopic, freely soluble in water, slightly soluble in methanol, practically insoluble in alcohol.

IDENTIFICATION

A. Add several drops of *dilute ammonia R2* to 1 ml of solution S (see Tests). A blue precipitate is formed on further addition of *dilute ammonia R2*, the precipitate dissolves and a dark blue colour is produced.

B. It complies with the test for loss on drying (see Tests).

C. Dilute 1 ml of solution S to 5 ml with *water R*. The solution gives reaction (a) of sulphates (*2.3.1*).

TESTS

Solution S. Dissolve 1.6 g in *water R* and dilute to 50 ml with the same solvent.

Appearance of solution. Solution S is clear (*2.2.1*).

Chlorides (*2.4.4*). Dilute 10 ml of solution S to 15 ml with *water R*. The solution complies with the limit test for chlor-ides (150 ppm). Examine the tubes laterally against a black background.

Iron. Not more than 150 ppm of Fe, determined by atomic absorption spectrometry (*2.2.23, Method I*).

Test solution. Dissolve 0.32 g in 10 ml of *water R*, add 2.5 ml of *lead-free nitric acid R* and dilute to 25.0 ml with *water R*.

Reference solutions. Prepare the reference solutions using *iron standard solution (20 ppm Fe) R*, adding 2.5 ml of *lead-free nitric acid R* and diluting to 25.0 ml with *water R*.

Measure the absorbance at 248.3 nm using an iron hollow-cathode lamp as a source of radiation and an air-butane flame.

Lead. Not more than 80 ppm of Pb, determined by atomic absorption spectrometry (*2.2.23, Method I*).

Test solution. Dissolve 1.6 g in 10 ml of *water R*, add 2.5 ml of *lead-free nitric acid R* and dilute to 25.0 ml with *water R*.

Reference solutions. Prepare the reference solutions using *lead standard solution (100 ppm Pb) R*, adding 2.5 ml of *lead-free nitric acid R* and diluting to 25.0 ml with *water R*.

Measure the absorbance at 217.0 nm using a lead hollow-cathode lamp as a source of radiation and an air-butane flame.

Loss on drying (*2.2.32*). Not more than 1.0 per cent, determined on 0.500 g by drying in an oven at 250 °C.

ASSAY

Dissolve 0.125 g in 50 ml of *water R*. Add 2 ml of *sulphuric acid R* and 3 g of *potassium iodide R*. Titrate with *0.1 M sodium thiosulphate*, using 1 ml of *starch solution R*, added towards the end of the titration, as indicator.

1 ml of *0.1 M sodium thiosulphate* is equivalent to 15.96 mg of $CuSO_4$.

STORAGE

Store in an airtight container.

01/2005:0894

COPPER SULPHATE PENTAHYDRATE

Cupri sulfas pentahydricus

$CuSO_4,5H_2O$ M_r 249.7

DEFINITION

Copper sulphate pentahydrate contains not less than 99.0 per cent and not more than the equivalent of 101.0 per cent of $CuSO_4,5H_2O$.

CHARACTERS

A blue, crystalline powder or transparent, blue crystals, freely soluble in water, soluble in methanol, practically insoluble in alcohol.

IDENTIFICATION

A. Add several drops of *dilute ammonia R2* to 1 ml of solution S (see Tests). A blue precipitate is formed on further addition of *dilute ammonia R2*, the precipate dissolves and a dark blue colour is produced.

B. It complies with the test for loss on drying (see Tests).

C. Dilute 1 ml of solution S to 5 ml with *water R*. The solution gives reaction (a) of sulphates (*2.3.1*).

TESTS

Solution S. Dissolve 5 g in *water R* and dilute to 100 ml with the same solvent.

Appearance of solution. Solution S is clear (*2.2.1*).

Chlorides (*2.4.4*). Dilute 10 ml of solution S to 15 ml with *water R*. The solution complies with the limit test for chlorides (100 ppm). Examine the tubes laterally against a black background.

Iron. Not more than 100 ppm of Fe, determined by atomic absorption spectrometry (*2.2.23, Method I*).

Test solution. Dissolve 0.5 g in 10 ml of *water R*, add 2.5 ml of *lead-free nitric acid R* and dilute to 25.0 ml with *water R*.

Reference solutions. Prepare the reference solutions using *iron standard solution (20 ppm Fe) R*, adding 2.5 ml of *lead-free nitric acid R* and diluting to 25.0 ml with *water R*.

Measure the absorbance at 248.3 nm using an iron hollow-cathode lamp as a source of radiation and an air-butane flame.

Lead. Not more than 50 ppm of Pb, determined by atomic absorption spectrometry (*2.2.23, Method I*).

Test solution. Dissolve 2.5 g in 10 ml of *water R*, add 2.5 ml of *lead-free nitric acid R* and dilute to 25.0 ml with *water R*.

Reference solutions. Prepare the reference solutions using *lead standard solution (100 ppm Pb) R*, adding 2.5 ml of *lead-free nitric acid R* and diluting to 25.0 ml with *water R*.

Measure the absorbance at 217.0 nm using a lead hollow-cathode lamp as a source of radiation and an air-butane flame.

Loss on drying (*2.2.32*): 35.0 per cent to 36.5 per cent, determined on 0.500 g by drying in an oven at 250 °C.

ASSAY

Dissolve 0.200 g in 50 ml of *water R*. Add 2 ml of *sulphuric acid R* and 3 g of *potassium iodide R*. Titrate with *0.1 M sodium thiosulphate*, adding 1 ml of *starch solution R* towards the end of the titration.

1 ml *0.1 M sodium thiosulphate* is equivalent to 24.97 mg of $CuSO_4,5H_2O$.

01/2005:1304

CORIANDER

Coriandri fructus

DEFINITION

Coriander consists of the dried cremocarp of *Coriandrum sativum* L. It contains not less than 3 ml/kg of essential oil, calculated with reference to the dried drug.

CHARACTERS

The cremocarp is brown or light brown and is more or less spherical, about 1.5 mm to 5 mm in diameter, or oval form 2 mm to 6 mm long.

It has the macroscopic and microscopic characters described under identification tests A and B.

IDENTIFICATION

A. The mericarps are usually tightly connected. The cremocarp is glabrous and has ten wavy, slightly raised primary ridges and eight straight, more prominent