Sulphates and selenates (*2.4.13*): maximum 300 ppm (determined as sulphates).

Dissolve 0.5 g in 10 ml of *distilled water R*. Add 0.5 ml of *hydrochloric acid R1* and dilute to 15 ml with *distilled water R*.

Iron: maximum 50 ppm.

To 2 ml of solution S add 2 ml of a 200 g/l solution of *sulphosalicylic acid R*, 5 ml of *concentrated ammonia R* and dilute to 10 ml with *water R*. The solution is not more intensely coloured than a reference solution prepared in the same manner using 1 ml of *iron standard solution (10 ppm Fe) R*.

ASSAY

Dissolve 0.120 g in 50 ml of *water R*, add 7 ml of *glacial acetic acid R*, 25.0 ml of *0.1 M sodium thiosulphate* and 0.5 g of *potassium iodide R*. Titrate immediately with *0.05 M iodine solution* using *starch solution R* as indicator.

1 ml of *0.1 M sodium thiosulphate* is equivalent to 6.575 mg of $Na_2SeO_3,5H_2O$.

STORAGE

In an airtight container.

01/2008:0983

SODIUM STARCH GLYCOLATE (TYPE A)

Carboxymethylamylum natricum A

DEFINITION

Sodium salt of a cross-linked partly *O*-carboxymethylated potato starch.

Content: 2.8 per cent to 4.2 per cent of Na (A_r 22.99) (substance washed with ethanol (80 per cent *V/V*) and dried).

CHARACTERS

Appearance: white or almost white, fine, free-flowing powder, very hygroscopic.

Solubility: practically insoluble in methylene chloride. It gives a translucent suspension in water.

Examined under a microscope it is seen to consist of: granules, irregularly shaped, ovoid or pear-shaped, 30-100 µm in size, or rounded, 10-35 µm in size; compound granules consisting of 2-4 components occur occasionally; the granules have an eccentric hilum and clearly visible concentric striations; between crossed nicol prisms, the granules show a distinct black cross intersecting at the hilum; small crystals are visible at the surface of the granules. The granules show considerable swelling in contact with water.

IDENTIFICATION

A. pH (see Tests).

B. Prepare with shaking and without heating a mixture of 4.0 g of the substance to be examined and 20 ml of *carbon dioxide-free water R*. The mixture has the appearance of a gel. Add 100 ml of *carbon dioxide-free water R* and shake. A suspension forms that settles after standing.

C. To an acidified solution, add *iodinated potassium iodide solution R1*. The solution becomes blue or violet.

D. Solution S2 (see Tests) gives reaction (a) of sodium (*2.3.1*).

TESTS

Solution S1. Centrifuge the suspension obtained in identification test B at 2500 *g* for 10 min. Collect carefully the supernatant liquid.

Solution S2. Place 2.5 g in a silica or platinum crucible and add 2 ml of a 500 g/l solution of *sulphuric acid R*. Heat on a water-bath, then cautiously over a naked flame, raising the temperature progressively, then incinerate in a muffle furnace at 600 ± 25 °C. Continue heating until all black particles have disappeared. Allow to cool, add a few drops of *dilute sulphuric acid R*, heat and incinerate as above. Allow to cool, add a few drops of *ammonium carbonate solution R*, evaporate to dryness and incinerate cautiously. Allow to cool and dissolve the residue in 50 ml of *water R*.

Appearance of solution S1. Solution S1 is clear (*2.2.1*) and colourless (*2.2.2, Method II*).

pH (*2.2.3*): 5.5 to 7.5.

Disperse 1.0 g in 30 ml of *water R*.

Sodium glycolate: maximum 2.0 per cent. *Carry out the test protected from light.*

Test solution. Place 0.20 g in a beaker. Add 5 ml of *acetic acid R* and 5 ml of *water R*. Stir until dissolution is complete (about 10 min). Add 50 ml of *acetone R* and 1 g of *sodium chloride R*. Filter through a fast filter paper impregnated with *acetone R*, rinse the beaker and filter with *acetone R*. Combine the filtrate and washings and dilute to 100.0 ml with *acetone R*. Allow to stand for 24 h without shaking. Use the clear supernatant liquid.

Reference solution. Dissolve 0.310 g of *glycollic acid R*, previously dried *in vacuo* over *diphosphorus pentoxide R* at room temperature overnight, in *water R* and dilute to 500.0 ml with the same solvent. To 5.0 ml of this solution add 5 ml of *acetic acid R* and allow to stand for about 30 min. Add 50 ml of *acetone R* and 1 g of *sodium chloride R*. Filter through a fast filter paper impregnated with *acetone R*, rinse the beaker and filter with *acetone R*. Combine the filtrate and washings and dilute to 100.0 ml with *acetone R*. Allow to stand for 24 h without shaking. Use the clear supernatant liquid.

Heat 2.0 ml of the test solution on a water-bath for 20 min. Cool to room temperature and add 20.0 ml of *2,7-dihydroxynaphthalene solution R*. Shake and heat in a water-bath for 20 min. Cool under running water, transfer to a volumetric flask and dilute to 25.0 ml with *sulphuric acid R*, maintaining the flask under running water. Within 10 min, measure the absorbance at 540 nm (*2.2.25*) using *water R* as the compensation liquid. The absorbance of the solution prepared with the test solution is not greater than that of a solution prepared at the same time and in the same manner with 2.0 ml of the reference solution.

Sodium chloride: maximum 7.0 per cent.

Place 0.500 g in a beaker and suspend in 100 ml of *water R*. Add 1 ml of *nitric acid R*. Titrate with *0.1 M silver nitrate*, determining the end-point potentiometrically (*2.2.20*), using a silver indicator electrode.

1 ml of *0.1 M silver nitrate* is equivalent to 5.844 mg of NaCl.

Iron (*2.4.9*): maximum 20 ppm determined on 10 ml of solution S2.

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

1.0 g complies with test D. Prepare the reference solution using 2 ml of *lead standard solution (10 ppm Pb) R*.

Loss on drying (*2.2.32*): maximum 10.0 per cent, determined on 1.000 g by drying in an oven at 130 °C for 1.5 h.

Microbial contamination. It complies with the test for *Escherichia coli* and *Salmonella* (*2.6.13*).

ASSAY

Shake 1.000 g with 20 ml of *ethanol (80 per cent V/V) R*, stir for 10 min and filter. Repeat the operation until chloride has been completely extracted and verify the absence of chloride using *silver nitrate solution R2*. Dry the residue at 105 °C to constant mass. To 0.700 g of the dried residue, add 80 ml of *glacial acetic acid R* and heat under a reflux condenser for 2 h. Cool the solution to room temperature. Titrate with *0.1 M perchloric acid*, determining the end-point potentiometrically (*2.2.20*). Carry out a blank titration.

1 ml of *0.1 M perchloric acid* is equivalent to 2.299 mg of Na.

STORAGE

In an airtight container, protected from light.

01/2008:0984

SODIUM STARCH GLYCOLATE (TYPE B)

Carboxymethylamylum natricum B

DEFINITION

Sodium salt of a cross-linked partly *O*-carboxymethylated potato starch.

Content: 2.0 per cent to 3.4 per cent of Na (A_r 22.99) (substance washed with ethanol (80 per cent *V/V*) and dried).

CHARACTERS

Appearance: white or almost white, fine, free-flowing powder, very hygroscopic.

Solubility: practically insoluble in methylene chloride. It gives a translucent suspension in water.

Examined under a microscope it is seen to consist of: granules, irregularly shaped, ovoid or pear shaped, 30-100 µm in size, or rounded, 10-35 µm in size; compound granules consisting of 2-4 components occur occasionally; the granules have an eccentric hilum and clearly visible concentric striations; between crossed nicol prisms, the granules show a distinct black cross intersecting at the hilum; small crystals are visible at the surface of the granules. The granules show considerable swelling in contact with water.

IDENTIFICATION

A. pH (see Tests).

B. Prepare with shaking and without heating a mixture of 4.0 g of the substance to be examined and 20 ml of *carbon dioxide-free water R*. The mixture has the appearance of a gel. Add 100 ml of *carbon dioxide-free water R* and shake. A suspension forms that settles after standing.

C. To an acidified solution, add *iodinated potassium iodide solution R1*. The solution becomes blue or violet.

D. Solution S2 (see Tests) gives reaction (a) of sodium (*2.3.1*).

TESTS

Solution S1. Centrifuge the suspension obtained in identification test B at 2500 *g* for 10 min. Collect carefully the supernatant liquid.

Solution S2. Place 2.5 g in a silica or platinum crucible and add 2 ml of a 500 g/l solution of *sulphuric acid R*. Heat on a water-bath, then cautiously over a naked flame, raising the temperature progressively, and then incinerate in a muffle furnace at 600 ± 25 °C. Continue heating until all black particles have disappeared. Allow to cool, add a few drops of *dilute sulphuric acid R* and heat and incinerate as above. Allow to cool, add a few drops of *ammonium carbonate solution R*, evaporate to dryness and incinerate cautiously. Allow to cool and dissolve the residue in 50 ml of *water R*.

Appearance of solution S1. Solution S1 is clear (*2.2.1*) and colourless (*2.2.2, Method II*).

pH (*2.2.3*): 3.0 to 5.0.

Disperse 1.0 g in 30 ml of *water R*.

Sodium glycolate: maximum 2.0 per cent. *Carry out the test protected from light.*

Test solution. Place 0.20 g in a beaker. Add 5 ml of *acetic acid R* and 5 ml of *water R*. Stir until dissolution is complete (about 10 min). Add 50 ml of *acetone R* and 1 g of *sodium chloride R*. Filter through a fast filter paper impregnated with *acetone R*, rinse the beaker and filter with *acetone R*. Combine the filtrate and washings and dilute to 100.0 ml with *acetone R*. Allow to stand for 24 h without shaking. Use the clear supernatant liquid.

Reference solution. Dissolve 0.310 g of *glycollic acid R*, previously dried *in vacuo* over *diphosphorus pentoxide R* at room temperature overnight, in *water R* and dilute to 500.0 ml with the same solvent. To 5.0 ml of this solution add 5 ml of *acetic acid R* and allow to stand for about 30 min. Add 50 ml of *acetone R* and 1 g of *sodium chloride R*. Filter through a fast filter paper impregnated with *acetone R*, rinse the beaker and filter with *acetone R*. Combine the filtrate and washings and dilute to 100.0 ml with *acetone R*. Allow to stand for 24 h without shaking. Use the clear supernatant liquid.

Heat 2.0 ml of the test solution on a water-bath for 20 min. Cool to room temperature and add 20.0 ml of *2,7-dihydroxynaphthalene solution R*. Shake and heat in a water-bath for 20 min. Cool under running water, transfer quantitatively to a volumetric flask and dilute to 25.0 ml with *sulphuric acid R*, maintaining the flasks under running water. Within 10 min, measure the absorbance at 540 nm (*2.2.25*) using *water R* as the compensation liquid. The absorbance of the solution prepared with the test solution is not greater than that of a solution prepared at the same time and in the same manner with 2.0 ml of the reference solution.

Sodium chloride: maximum 7.0 per cent.

Place 0.500 g in a beaker and suspend in 100 ml of *water R*. Add 1 ml of *nitric acid R*. Titrate with *0.1 M silver nitrate*, determining the end-point potentiometrically (*2.2.20*) using a silver indicator electrode.

1 ml of *0.1 M silver nitrate* is equivalent to 5.844 mg of NaCl.

Iron (*2.4.9*): maximum 20 ppm determined on 10 ml of solution S2.

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

1.0 g complies with test D. Prepare the reference solution using 2 ml of *lead standard solution (10 ppm Pb) R*.

Loss on drying (*2.2.32*): maximum 10.0 per cent, determined on 1.000 g by drying in an oven at 130 °C for 1.5 h.

Microbial contamination. It complies with the test for *Escherichia coli* and *Salmonella* (*2.6.13*).