- *stationary phase*: spherical *graphitised carbon for chromatography R1* (5 µm) with a specific surface area of 120 m^2/g and a pore size of 25 nm.

Mobile phase: dissolve 50.0 mg of *ferric sulfate pentahydrate R* in 50 mL of *0.5 M sulfuric acid* and add 750 mL of *water R*; adjust to pH 1.5 with *0.5 M sulfuric acid* or *1 M sodium hydroxide*, add 20 mL of *ethylene glycol R* and dilute to 1000 mL with *water R*.

Flow rate: 1 mL/min.

Detection: spectrophotometer at 273 nm.

Injection: 20 µL; filter the solutions and inject immediately.

Run time: 4 times the retention time of the iron complex of impurity A.

Retention time: iron complex of impurity A = about 5 min; iron complex of edetic acid = about 10 min.

System suitability: reference solution:

- *resolution*: minimum 7 between the peaks due to the iron complex of impurity A and the iron complex of edetic acid;
- *signal-to-noise ratio*: minimum 50 for the peak due to impurity A.

Limit:

- *impurity A*: not more than the area of the corresponding peak in the chromatogram obtained with the reference solution (0.1 per cent).

Disodium edetate: maximum 1.0 per cent.

Dissolve 5.0 g in 250 mL of *water R*. Add 10 mL of *ammonium chloride buffer solution pH 10.0 R* and about 50 mg of *mordant black 11 triturate R*. Not more than 1.5 mL of *0.1 M magnesium chloride* is required to change the colour of the indicator to violet.

Chlorides: maximum 0.1 per cent.

Dissolve 0.7 g in *water R* and dilute to 20 mL with the same solvent. Add 30 mL of *dilute nitric acid R*, allow to stand for 30 min and filter. Dilute 10 mL of the filtrate to 50 mL with *water R*. Use this solution as the test solution. Prepare the reference solution using 0.40 mL of *0.01 M hydrochloric acid*, add 6 mL of *dilute nitric acid R* and dilute to 50 mL with *water R*. Filter both solutions if necessary. Add 1 mL of *silver nitrate solution R2* to the test solution and to the reference solution and mix. After standing for 5 min protected from light, any opalescence in the test solution is not more intense than that in the reference solution.

Iron (*2.4.9*): maximum 80 ppm.

Dilute 2.5 mL of solution S to 10 mL with *water R*. Add 0.25 g of *calcium chloride R* to the test solution and the standard before the addition of the *thioglycollic acid R*.

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

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

Water (*2.5.12*): 5.0 per cent to 13.0 per cent, determined on 0.200 g.

ASSAY

Dissolve 0.500 g in *water R* and dilute to 200 mL with the same solvent. To 20.0 mL of this solution, add 80 mL of *water R* and adjust to pH 2 with *dilute nitric acid R*. Titrate with *0.01 M bismuth nitrate*, using 0.1 mL of a 1 g/L solution of *xylenol orange R* as indicator. The colour of the solution changes from yellow to red.

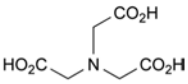
1 mL of *0.01 M bismuth nitrate* is equivalent to 3.74 mg of $C_{10}H_{12}CaN_2Na_2O_8$.

STORAGE

In an airtight container, protected from light.

IMPURITIES

Specified impurities: A.



A. nitrilotriacetic acid.

01/2008:1471
corrected 6.0

SODIUM CAPRYLATE

Natrii caprylas

$C_8H_{15}NaO_2$ M_r 166.2
[1984-06-1]

DEFINITION

Sodium octanoate.

Content: 99.0 per cent to 101.0 per cent (anhydrous substance).

CHARACTERS

Appearance: white or almost white, crystalline powder.

Solubility: very soluble or freely soluble in water, freely soluble in acetic acid, sparingly soluble in ethanol (96 per cent), practically insoluble in acetone.

IDENTIFICATION

A. Examine the chromatograms obtained in the test for related substances.

Results: the principal peak in the chromatogram obtained with the test solution is similar in retention time and size to the principal peak in the chromatogram obtained with reference solution (a).

B. To 0.2 mL of solution S (see Tests) add 0.3 mL of *water R*. The solution gives reaction (b) of sodium (*2.3.1*).

TESTS

Solution S. Dissolve 2.5 g in *carbon dioxide-free water R* and dilute to 25 mL with the same solvent.

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

pH (*2.2.3*): 8.0 to 10.5 for solution S.

Related substances. Gas chromatography (*2.2.28*): use the normalisation procedure.

Test solution. Dissolve 0.116 g in *water R* and dilute to 5 mL with the same solvent. Add 1 mL of a 2.8 per cent *V/V* solution of *sulfuric acid R* and shake with 10 mL of *ethyl acetate R*. Separate the organic layer and dry over *anhydrous sodium sulfate R*.

Reference solution (a). Dissolve 0.10 g of *caprylic acid CRS* in *ethyl acetate R* and dilute to 10 mL with the same solvent.

Reference solution (b). Dilute 1 mL of the test solution to 100 mL with *ethyl acetate R*. Dilute 5 mL of this solution to 50 mL with *ethyl acetate R*.

Column:

- *material*: fused silica;
- *size*: *l* = 30 m, Ø = 0.25 mm;
- *stationary phase*: *macrogol 20 000 2-nitroterephthalate R* (film thickness 0.25 µm).

Carrier gas: *helium for chromatography R.*

Flow rate: 1.5 mL/min.

Split ratio: 1:100.

Temperature:

	Time (min)	Temperature (°C)
Column	0 - 1	100
	1 - 25	100 → 220
	25 - 35	220
Injection port		250
Detector		250

Detection: flame ionisation.

Injection: 1 μL.

System suitability: reference solution (b):

- *signal-to-noise ratio*: minimum 5 for the principal peak.

Limits:

- *any impurity*: for each impurity, maximum 0.3 per cent;
- *total*: maximum 0.5 per cent;
- *disregard limit*: 0.5 times the area of the principal peak in the chromatogram obtained with reference solution (b) (0.05 per cent).

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

Dissolve 2.0 g in *glacial acetic acid R* and dilute to 10 mL with the same acid. Add 10 mL of *ethanol (96 per cent) R*. 12 mL of the solution complies with test B. Prepare the reference solution using 1 mL of *lead standard solution (10 ppm Pb) R* and 9 mL of a mixture of equal volumes of *glacial acetic acid R* and *ethanol (96 per cent) R*.

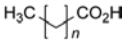
Water (*2.5.12*): maximum 3.0 per cent, determined on 1.000 g.

ASSAY

Dissolve 0.150 g in 50 mL of *anhydrous acetic acid 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 16.62 mg of $C_8H_{15}NaO_2$.

IMPURITIES

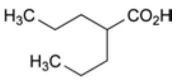


A. *n* = 4: hexanoic acid,

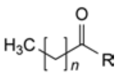
B. *n* = 5: heptanoic acid,

C. *n* = 7: nonanoic acid,

D. *n* = 8: decanoic acid,



E. 2-propylpentanoic acid (valproic acid),

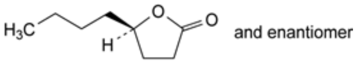


F. R = OCH_3, *n* = 6: methyl octanoate,

G. R = OC_2H_5, *n* = 6: ethyl octanoate,

H. R = OCH_3, *n* = 8: methyl decanoate,

I. R = CH_3, *n* = 8: undecan-2-one,



J. (*RS*)-5-butyltetrahydrofuran-2-one (γ-hydroxyoctanoic acid lactone).

01/2008:0773

SODIUM CARBONATE, ANHYDROUS

Natrii carbonas anhydricus

Na_2CO_3 M_r 106.0
[497-19-8]

DEFINITION

Content: 99.5 per cent to 100.5 per cent (dried substance).

CHARACTERS

Appearance: white or almost white, slightly granular powder, hygroscopic.

Solubility: freely soluble in water, practically insoluble in ethanol (96 per cent).

IDENTIFICATION

A. Dissolve 1 g in *water R* and dilute to 10 mL with the same solvent. The solution is strongly alkaline (*2.2.4*).

B. The solution prepared for identification test A gives the reaction of carbonates (*2.3.1*).

C. The solution prepared for identification test A gives reaction (a) of sodium (*2.3.1*).

D. Loss on drying (see Tests).

TESTS

Solution S. Dissolve 2.0 g in portions in a mixture of 5 mL of *hydrochloric acid R* and 25 mL of *distilled water R*. Heat to boiling and cool. Add *dilute sodium hydroxide solution R* until the solution is neutral and dilute to 50 mL with *distilled water R*.

Appearance of solution. The solution is clear (*2.2.1*) and not more intensely coloured than reference solution Y_6 (*2.2.2, Method I*).

Dissolve 2.0 g in 10 mL of *water R*.

Alkali hydroxides and bicarbonates. Dissolve 0.4 g in 20 mL of *water R*. Add 20 mL of *barium chloride solution R1* and filter. To 10 mL of the filtrate add 0.1 mL of *phenolphthalein solution R*. The solution does not become red. Boil the remainder of the filtrate for 2 min. The solution remains clear (*2.2.1*).

Chlorides (*2.4.4*): maximum 125 ppm.

Dissolve 0.4 g in *water R*, add 4 mL of *dilute nitric acid R* and dilute to 15 mL with *water R*.

Sulfates (*2.4.13*): maximum 250 ppm, determined on solution S.

Arsenic (*2.4.2, Method A*): maximum 5 ppm, determined on 5 mL of solution S.

Iron (*2.4.9*): maximum 50 ppm.

Dilute 5 mL of solution S to 10 mL with *water R*.

Heavy metals (*2.4.8*): maximum 50 ppm.

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

Loss on drying (*2.2.32*): maximum 1.0 per cent, determined on 1.000 g by drying at 300 ± 15 °C.

ASSAY

Dissolve 1.000 g in 25 mL of *water R*. Add 0.2 mL of *methyl orange solution R* as indicator. Titrate with *1 M hydrochloric acid* until the colour changes from yellow to red.

1 mL of *1 M hydrochloric acid* is equivalent to 52.99 mg of Na_2CO_3.

STORAGE

In an airtight container.