Temperature:

	Time (min)	Temperature (°C)
Column	0 - 20	150 → 250
	20 - 40	250
Injection port		250
Detector		250

Detection: flame ionisation.

Injection: 1 μL of the test solution and reference solutions (a) and (c).

System suitability: reference solution (c):

- *resolution*: minimum 5.0 between the peaks due to cetyl alcohol and stearyl alcohol.

Calculate the percentage content of $C_{16}H_{34}O$.

01/2008:1906

CETYL PALMITATE

Cetylis palmitas

DEFINITION

Mixture of C_{14}-C_{18} esters of lauric (dodecanoic), myristic (tetradecanoic), palmitic (hexadecanoic) and stearic (octadecanoic) acids ('Cetyl esters wax').

Content (expressed as hexadecyl hexadecanoate): 10.0 per cent to 20.0 per cent for Cetyl palmitate 15, 60.0 per cent to 70.0 per cent for Cetyl palmitate 65 and minimum 90.0 per cent for Cetyl palmitate 95.

CHARACTERS

Appearance: white or almost white, waxy plates, flakes or powder.

Solubility: practically insoluble in water, soluble in boiling anhydrous ethanol and in methylene chloride, slightly soluble in light petroleum, practically insoluble in anhydrous ethanol.

mp: about 45 °C for Cetyl palmitate 15 and Cetyl palmitate 65 and about 52 °C for Cetyl palmitate 95.

IDENTIFICATION

A. It complies with the limits of the assay and the chromatogram obtained with the test solution shows the typical main peak(s).

B. Saponification value (see Tests).

TESTS

Appearance of solution. The solution is not more intensely coloured than reference solution Y_6 (*2.2.2, Method II*).

Dissolve 4.0 g in *methylene chloride R* and dilute to 20 mL with the same solvent.

Acid value (*2.5.1*): maximum 4.0.

Dissolve 10.0 g in 50 mL of the solvent mixture described by heating under reflux on a water-bath for 5 min.

Hydroxyl value (*2.5.3, Method A*): maximum 20.0.

Iodine value (*2.5.4, Method A*): maximum 2.0.

Saponification value (*2.5.6*): 105 to 120.

Heat under reflux for 2 h.

Alkaline impurities. Dissolve 2.0 g 'with gentle heating' in a mixture of 1.5 mL of *ethanol (96 per cent) R* and 3 mL of *toluene R*. Add 0.05 mL of a 0.4 g/L solution of *bromophenol blue R* in *ethanol (96 per cent) R*. Not more than 0.4 mL of *0.01 M hydrochloric acid* is required to change the colour of the solution to yellow.

Nickel (*2.4.31*): maximum 1 ppm.

Water (*2.5.12*): maximum 0.3 per cent, determined on 1.0 g using a mixture of equal volumes of *anhydrous methanol R* and *methylene chloride R* as solvent.

Total ash (*2.4.16*): maximum 0.2 per cent, determined on 1.0 g.

ASSAY

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

Test solution. Dissolve 20.0 mg of the substance to be examined in *hexane R* and dilute to 20.0 mL with the same solvent.

Reference solution (a). Dissolve 20.0 mg of *cetyl palmitate 95 CRS* in *hexane R* and dilute to 20.0 mL with the same solvent.

Reference solution (b). Dissolve 20.0 mg of *cetyl palmitate 15 CRS* in *hexane R* and dilute to 20.0 mL with the same solvent.

Column:

- *material*: stainless steel;
- *size*: *l* = 10 m, Ø = 0.53 mm;
- *stationary phase*: *poly(dimethyl)siloxane R* (film thickness 2.65 μm).

Carrier gas: *helium for chromatography R*.

Flow rate: 6.5 mL/min.

Split ratio: 1:10.

Temperature:

	Time (min)	Temperature (°C)
Column	0 - 10	100 → 300
	10 - 15	300
Injection port		350
Detector		350

Detection: flame ionisation.

Injection: 1 μL.

Relative retention with reference to cetyl palmitate (retention time = about 9 min): cetyl alcohol = about 0.3; palmitic acid = about 0.4; lauric ester = about 0.8; myristic ester = about 0.9; stearic ester = about 1.1.

System suitability: reference solution (b):

- *resolution*: minimum of 1.5 between the peaks due to cetyl palmitate and cetyl stearate.

STORAGE

At a temperature not exceeding 25 °C.

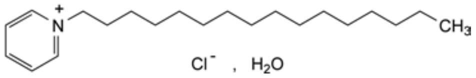
LABELLING

The label states the type of cetyl palmitate.

01/2008:0379
corrected 6.0

CETYLPYRIDINIUM CHLORIDE

Cetylpyridinii chloridum



$C_{21}H_{38}ClN,H_2O$ M_r 358.0
[6004-24-6]

DEFINITION

Cetylpyridinium chloride contains not less than 96.0 per cent and not more than the equivalent of 101.0 per cent of 1-hexadecylpyridinium chloride, calculated with reference to the anhydrous substance.

CHARACTERS

A white or almost white powder, slightly soapy to the touch, soluble in water and in alcohol. An aqueous solution froths copiously when shaken.

IDENTIFICATION

First identification: B, D.

Second identification: A, C, D.

A. Dissolve 0.10 g in *water R* and dilute to 100.0 mL with the same solvent. Dilute 5.0 mL of this solution to 100.0 mL with *water R*. Examined between 240 nm and 300 nm (*2.2.25*), the solution shows an absorption maximum at 259 nm and 2 shoulders at about 254 nm and at about 265 nm. The specific absorbance at the maximum is 126 to 134, calculated with reference to the anhydrous substance.

B. Examine by infrared absorption spectrophotometry (*2.2.24*), comparing with the spectrum obtained with *cetylpyridinium chloride CRS*. Examine the substances in the solid state.

C. To 5 mL of *dilute sodium hydroxide solution R* add 0.1 mL of *bromophenol blue solution R1* and 5 mL of *chloroform R* and shake. The chloroform layer is colourless. Add 0.1 mL of solution S (see Tests) and shake. The chloroform layer becomes blue.

D. Solution S gives reaction (a) of chlorides (*2.3.1*).

TESTS

Solution S. Dissolve 1.0 g in *carbon dioxide-free water R* and dilute to 100 mL with the same solvent.

Appearance of solution. Solution S is not more opalescent than reference suspension II (*2.2.1*) and is colourless (*2.2.2, Method II*).

Acidity. To 50 mL of solution S add 0.1 mL of *phenolphthalein solution R*. Not more than 2.5 mL of *0.02 M sodium hydroxide* is required to change the colour of the indicator.

Amines and amine salts. Dissolve 5.0 g with heating in 20 mL of a mixture of 3 volumes of *1 M hydrochloric acid* and 97 volumes of *methanol R* and add 100 mL of *2-propanol R*. Pass a stream of *nitrogen R* slowly through the solution. Gradually add 12.0 mL of *0.1 M tetrabutylammonium hydroxide* and record the potentiometric titration curve (*2.2.20*). If the curve shows 2 points of inflexion, the volume of titrant added between the two points is not greater than 5.0 mL. If the curve shows no point of inflexion, the substance to be examined does not comply with the test. If the curve shows one point of inflexion, repeat the test but add 3.0 mL of a 25.0 g/L solution of *dimethyldecylamine R* in *2-propanol R* before the titration. If the titration curve after the addition of 12.0 mL of the titrant shows only one point of inflexion, the substance to be examined does not comply with the test.

Water (*2.5.12*): 4.5 per cent to 5.5 per cent, determined on 0.300 g by the semi-micro determination of water.

Sulfated ash (*2.4.14*). Not more than 0.2 per cent, determined on 1.0 g.

ASSAY

Dissolve 2.00 g in *water R* and dilute to 100.0 mL with the same solvent. Transfer 25.0 mL of the solution to a separating funnel, add 25 mL of *chloroform R*, 10 mL of *0.1 M sodium hydroxide* and 10.0 mL of a freshly prepared 50 g/L solution of *potassium iodide R*. Shake well, allow to separate and discard the chloroform layer. Shake the aqueous layer with three quantities, each of 10 mL, of *chloroform R* and discard the chloroform layers. To the aqueous layer add 40 mL of *hydrochloric acid R*, allow to cool and titrate with *0.05 M potassium iodate* until the deep-brown colour is almost discharged. Add 2 mL of *chloroform R* and continue the titration, shaking vigorously, until the chloroform layer no longer changes colour. Carry out a blank titration on a mixture of 10.0 mL of the freshly prepared 50 g/L solution of *potassium iodide R*, 20 mL of *water R* and 40 mL of *hydrochloric acid R*.

1 mL of *0.05 M potassium iodate* is equivalent to 34.0 mg of $C_{21}H_{38}ClN$.

01/2009:0313
corrected 7.0

CHARCOAL, ACTIVATED

Carbo activatus

DEFINITION

Obtained from vegetable matter by suitable carbonisation processes intended to confer a high adsorption power.

CHARACTERS

Appearance: black, light powder free from grittiness.

Solubility: practically insoluble in all usual solvents.

IDENTIFICATION

A. When heated to redness it burns slowly without a flame.

B. Adsorption power (see Tests).

TESTS

Solution S. To 2.0 g in a conical flask with a ground-glass neck add 50 mL of *dilute hydrochloric acid R*. Boil gently under a reflux condenser for 1 h, filter and wash the filter with *dilute hydrochloric acid R*. Evaporate the combined filtrate and washings to dryness on a water-bath, dissolve the residue in *0.1 M hydrochloric acid* and dilute to 50.0 mL with the same acid.

Acidity or alkalinity. To 2.0 g add 40 mL of *water R* and boil for 5 min. Cool, restore to the original mass with *carbon dioxide-free water R* and filter. Reject the first 20 mL of the filtrate. To 10 mL of the filtrate add 0.25 mL of *bromothymol blue solution R1* and 0.25 mL of *0.02 M sodium hydroxide*. The solution is blue. Not more than 0.75 mL of *0.02 M hydrochloric acid* is required to change the colour of the indicator to yellow.

Acid-soluble substances: maximum 3 per cent.

To 1.0 g add 25 mL of *dilute nitric acid R* and boil for 5 min. Filter whilst hot through a sintered-glass filter (10) (*2.1.2*) and wash with 10 mL of hot *water R*. Evaporate the combined filtrate and washings to dryness on a water-bath, add to the residue 1 mL of *hydrochloric acid R*, evaporate to dryness again and dry the residue to constant mass at 100-105 °C. The residue weighs a maximum of 30 mg.

Alkali-soluble coloured substances. To 0.25 g add 10 mL of *dilute sodium hydroxide solution R* and boil for 1 min. Cool, filter and dilute the filtrate to 10 mL with *water R*. The solution is not more intensely coloured than reference solution GY_4 (*2.2.2, Method II*).

Ethanol (96 per cent) soluble substances: maximum 0.5 per cent.

To 2.0 g add 50 mL of *ethanol (96 per cent) R* and boil under a reflux condenser for 10 min. Filter immediately, cool, and dilute to 50 mL with *ethanol (96 per cent) R*. The filtrate is not more intensely coloured than reference solution Y_6 or BY_6 (*2.2.2, Method II*). Evaporate 40 mL of the filtrate to dryness and dry to constant mass at 100-105 °C. The residue weighs a maximum of 8 mg.

Fluorescent substances. In an intermittent-extraction apparatus, treat 10.0 g with 100 mL of *cyclohexane R1* for 2 h. Collect the liquid and dilute to 100 mL with *cyclohexane R1*. Examine in ultraviolet light at 365 nm. The fluorescence of the solution is not more intense than that of a solution of 83 μg of *quinine R* in 1000 mL of *0.005 M sulfuric acid* examined under the same conditions.

Sulfides. To 1.0 g in a conical flask add 5 mL of *hydrochloric acid R1* and 20 mL of *water R*. Heat to boiling. The fumes released do not turn *lead acetate paper R* brown.