Limits:

- *impurity A*: not more than 1.7 times the area of the corresponding peak in the chromatogram obtained with reference solution (a) (0.5 per cent),
- *impurity C*: not more than twice the area of the corresponding peak in the chromatogram obtained with reference solution (a) (0.5 per cent); if the area of the peak due to impurity C in the chromatogram obtained with the test solution is greater than the area of the corresponding peak in the chromatogram obtained with reference solution (a) (0.25 per cent), dilute the test solution to obtain an area equal to or smaller than the area of the peak in the chromatogram obtained with reference solution (a); calculate the content of impurity C taking into account the dilution factor;
- *impurity D*: not more than the area of the principal peak in the chromatogram obtained with reference solution (c) (0.3 per cent),
- *any other impurity*: not more than the area of the peak due to impurity A in the chromatogram obtained with reference solution (a) (0.3 per cent),
- *total*: not more than 6.7 times the area of the peak due to impurity A in the chromatogram obtained with reference solution (a) (2.0 per cent),
- *disregard limit*: 0.17 times the area of the peak due to impurity A in the chromatogram obtained with reference solution (a) (0.05 per cent).

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

Dilute 5.0 ml of solution S to 20.0 ml with *methanol R*. 12 ml of the solution complies with limit test B. Prepare the standard using lead standard solution (1 ppm Pb) obtained by diluting *lead standard solution (100 ppm Pb) R* with *methanol R*.

ASSAY

Liquid chromatography (*2.2.29*) as described in the test for related substances with the following modification.

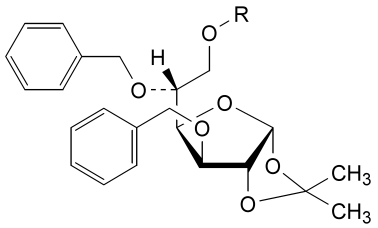
Injection: test solution (b) and reference solution (b).

Calculate the sum of the percentage contents of the α-anomer and the β-anomer of tribenoside.

STORAGE

Under nitrogen, in an airtight container.

IMPURITIES



A. R = CH_2-C_6H_5: 3,5,6-tri-*O*-benzyl-1,2-*O*-(1-methylethylidene)-α-D-glucofuranose,

B. R = H: 3,5-di-*O*-benzyl-1,2-*O*-(1-methylethylidene)-α-D-glucofuranose,

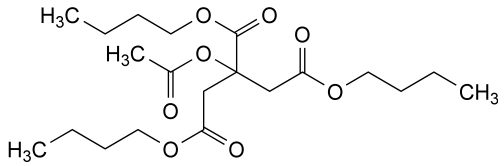
C. C_6H_5-CHO: benzaldehyde,

D. C_6H_5-CH_2-O-CH_2-C_6H_5: dibenzyl ether.

01/2008:1770

TRIBUTYL ACETYLCITRATE

Tributylis acetylcitras



$C_{20}H_{34}O_8$ M_r 402.5

[77-90-7]

DEFINITION

Tributyl 2-(acetyloxy)propane-1,2,3-tricarboxylate.

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

CHARACTERS

Appearance: clear, oily liquid.

Solubility: not miscible with water, miscible with alcohol and with methylene chloride.

IDENTIFICATION

Infrared absorption spectrophotometry (*2.2.24*).

Comparison: *Ph. Eur. reference spectrum of tributyl acetylcitrate.*

TESTS

Appearance. The substance to be examined is clear (*2.2.1*) and not more intensely coloured than reference solution BY_6 (*2.2.2, Method II*).

Acidity. Dilute 10 g with 10 ml of previously neutralised *alcohol R*, add 0.5 ml of *bromothymol blue solution R2*. Not more than 0.3 ml of *0.1 M sodium hydroxide* is required to change the colour of the indicator to blue.

Refractive index (*2.2.6*): 1.442 to 1.445.

Related substances. Gas chromatography (*2.2.28*).

Test solution. Dissolve 1.0 g of the substance to be examined in *methylene chloride R* and dilute to 20.0 ml with the same solvent.

Reference solution (a). Dissolve 50 mg of the substance to be examined and 50 mg of *tributyl citrate R* in *methylene chloride R* and dilute to 20.0 ml with the same solvent.

Reference solution (b). Dilute 1.0 ml of the test solution to 20.0 ml with *methylene chloride R*. Dilute 1.0 ml of this solution to 25.0 ml with *methylene chloride R*.

Column:

- *material*: fused silica,
- *size*: *l* = 30 m, Ø = 0.53 mm,
- *stationary phase*: *poly[(cyanopropyl)(methyl)][(phenyl)(methyl)]siloxane R* (film thickness 1.0 μm).

Carrier gas: *helium for chromatography R*.

Linear velocity: 36 cm/s.

Split ratio: 1:20.

Temperature:

- *column*: 200 °C,
- *injection port and detector*: 250 °C.

Detection: flame ionisation.

Injection: 1 μl.

Run time: twice the retention time of tributyl acetylcitrate.

Relative retention with reference to tributyl acetylcitrate (retention time = abou 26 min): impurity B = about 0.83; impurity A = about 0.87.

System suitability: reference solution (a):

– *resolution*: minimum 2.0 between the peaks due to impurity A and tributyl acetylcitrate.

Limits:

– *impurities A, B*: for each impurity, not more than the area of the principal peak in the chromatogram obtained with reference solution (b) (0.2 per cent),

– *any other impurity*: for each impurity, not more than 0.5 times the area of the principal peak in the chromatogram obtained with reference solution (b) (0.1 per cent),

– *total*: not more than 2.5 times the area of the principal peak in the chromatogram obtained with reference solution (b) (0.5 per cent),

– *disregard limit*: 0.25 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.

2.0 g complies with limit test F. Prepare the standard using 2 ml of *lead standard solution (10 ppm Pb) R*.

Water (*2.5.12*): maximum 0.25 per cent, determined on 2.00 g.

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

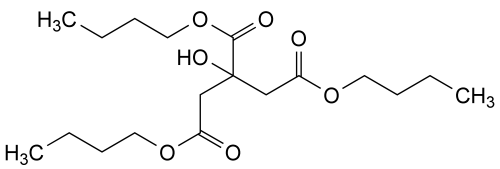
ASSAY

Introduce 1.500 g into a 250 ml borosilicate glass flask. Add 25 ml of *2-propanol R*, 50 ml of *water R*, 25.0 ml of *1 M sodium hydroxide* and a few glass beads. Heat under a reflux condenser for 1 h. Allow to cool. Add 1 ml of *phenolphthalein solution R1* and titrate with *1 M hydrochloric acid*. Carry out a blank titration.

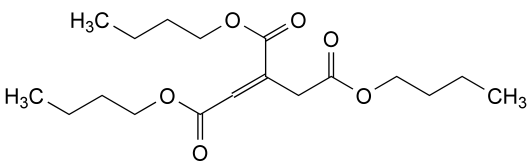
1 ml of *1 M sodium hydroxide* is equivalent to 100.6 mg of $C_{20}H_{34}O_8$.

IMPURITIES

Specified impurities: A, B.



A. tributyl 2-hydroxypropane-1,2,3-tricarboxylate (tributyl citrate),



B. tributyl propene-1,2,3-tricarboxylate (tributyl aconitate).

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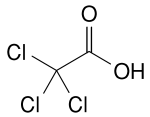
corrected 6.0

TRICHLOROACETIC ACID

Acidum trichloraceticum



$C_2HCl_3O_2$ M_r 163.4
[76-03-9]

DEFINITION

2,2,2-Trichloroacetic acid.

Content: 98.0 per cent to 100.5 per cent.

CHARACTERS

Appearance: white or almost white, crystalline mass or colourless crystals, very deliquescent.

Solubility: very soluble in water, in alcohol and in methylene chloride.

IDENTIFICATION

First identification: A.

Second identification: B, C.

A. Infrared absorption spectrophotometry (*2.2.24*).

Comparison: *Ph. Eur. reference spectrum of trichloroacetic acid.*

B. To 0.5 ml of solution S (see Tests) add 2 ml of *pyridine R* and 5 ml of *strong sodium hydroxide solution R*. Shake vigorously and heat in a water-bath at 60-70 °C for 5 min. The upper layer shows an intense red colour.

C. Solution S is strongly acidic (*2.2.4*).

TESTS

Solution S. Dissolve 2.5 g in *water R* and dilute to 25 ml with the same solvent.

Appearance of solution. Solution S is clear (*2.2.1*) and not more intensely coloured than reference solution BY_7 (*2.2.2, Method II*).

Chlorides (*2.4.4*): maximum 100 ppm.

Dilute 5 ml of solution S to 15 ml with *water R*. The solution complies with the limit test for chlorides.

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

ASSAY

Dissolve 0.150 g in 20 ml of *water R*. Titrate with *0.1 M sodium hydroxide*, determining the end-point potentiometrically (*2.2.20*).

1 ml of *0.1 M sodium hydroxide* is equivalent to 16.34 mg of $C_2HCl_3O_2$.

STORAGE

In an airtight container.