IMPURITIES

A. 1-(4-chlorophenyl)-5-[6-(3-cyanoguanidino)hexyl]-biguanide,

\[
\begin{align*}
\text{N} & \quad \text{N} \\
\text{Cl} & \quad \text{H} \\
\text{N} & \quad \text{N}
\end{align*}
\]

B. \[\text{[6-[5-(4-chlorophenyl)guanidino]hexyl]amino} \text{imino-methyl} \text{urea,}
\]

\[
\begin{align*}
\text{N} & \quad \text{N} \\
\text{O} & \quad \text{N} \\
\text{H} & \quad \text{N}
\end{align*}
\]

C. \[1,1^\prime\text{-[hexane-1,6-diylbis[imino(iminocarbonyl)]bis[3-(4-chlorophenyl)urea].}
\]

D. \[1,1^\prime\text{-[[[4-chlorophenyl]amino][iminomethyl][imino]-methylene][bis[imino(hexane-1,6-diyl)]]bis[5-(4-chlorophenyl)biguanide].}
\]

DEFINITION

1,1,1-Trichloro-2-methylpropan-2-ol.

Content: 98.0 per cent to 101.0 per cent (anhydrous substance).

CHARACTERS

Appearance: white or almost white, crystalline powder or colourless crystals, sublimes readily.

Solubility: slightly soluble in water, very soluble in ethanol (96 per cent), soluble in glycerol (85 per cent).

mp: about 95 °C (without previous drying).

IDENTIFICATION

A. Add about 20 mg to a mixture of 1 ml of pyridine R and 2 ml of strong sodium hydroxide solution R. Heat in a water-bath and shake. Allow to stand. The pyridine layer becomes red.

B. Add about 20 mg to 5 ml of ammoniacal silver nitrate solution R and warm slightly. A black precipitate is formed.

C. To about 20 mg add 3 ml of 1 M sodium hydroxide and shake to dissolve. Add 5 ml of water R and then, slowly, 2 ml of iodinated potassium iodide solution R. A yellowish precipitate is formed.

D. Water (see Tests).

TESTS

Solution S. Dissolve 5 g in ethanol (96 per cent) R and dilute to 10 ml with the same solvent.

Appearance of solution. Solution S is not more opalescent than reference suspension II (2.2.1) and not more intensely coloured than reference solution BY5 (2.2.2, Method II).

Acidity. To 4 ml of solution S add 15 ml of ethanol (96 per cent) R and 0.1 ml of bromothymol blue solution R1. Not more than 1.0 ml of 0.01 M sodium hydroxide is required to change the colour of the indicator to blue.

Chlorides (2.4.4): maximum 300 ppm.

Dissolve 0.17 g in 5 ml of ethanol (96 per cent) R and dilute to 15 ml with water R. When preparing the standard, replace the 5 ml of water R by 5 ml of ethanol (96 per cent) R.

Water (2.5.12): maximum 1.0 per cent, determined on 2.00 g.

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

ASSAY

Dissolve 0.100 g in 20 ml of ethanol (96 per cent) R. Add 10 ml of dilute sodium hydroxide solution R, heat in a water-bath for 5 min and cool. Add 20 ml of dilute nitric acid R, 25.0 ml of 0.1 M silver nitrate and 2 ml of dibutyl phthalate R and shake vigorously. Add 2 ml of ferric ammonium sulphate solution R2 and titrate with 0.1 M ammonium thiocyanate until an orange colour is obtained.

1 ml of 0.1 M silver nitrate is equivalent to 5.92 mg of C₄H₇Cl₃O.

STORAGE

In an airtight container.

CHLOROBUTANOL, ANHYDROUS

Chlorobutanolum anhydricum

C₄H₇Cl₃O

[57-15-8]  

M, 177.5

01/2008:0382  
corrected 6.0