IMPURITIES

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

$$\begin{array}{c|c}
H \\
O & N \\
NH_2 & NH
\end{array}$$

B. [[[6-[5-(4-chlorophenyl)guanidino]hexyl]amino]iminomethyl]urea,

C. 1,1'-[hexane-1,6-diylbis[imino(iminocarbonyl)]]bis[3-(4-chlorophenyl)urea],

D. 1,1'-[[[[(4-chlorophenyl)amino]iminomethyl]imino]-methylene]bis[imino(hexane-1,6-diyl)]]bis[5-(4-chlorophenyl)biguanide].

01/2008:0382 corrected 6.0

CHLOROBUTANOL, ANHYDROUS

Chlorobutanolum anhydricum

 ${\rm C_4H_7Cl_3O} \\ [57\text{-}15\text{-}8]$

 M_{\star} 177.5

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 $C_4H_7Cl_3O$.

STORAGE

In an airtight container.