Zinc Paste, Calamine and Clioquinol Bandage

Definition Zinc Paste, Calamine and Clioquinol Bandage consists of cotton fabric of plain weave, evenly impregnated with a suitable paste containing not less than 9.25% w/w of Zinc Oxide and containing 5.75% w/w of Calamine and 1% w/w of Clioquinol. The fabric is bleached to a good white, is reasonably free from weaving defects and contains not more than traces of leaf residue, seed coat and other impurities. It is in one continuous length. The edges are cut evenly, parallel to the warp threads, or may be serrated and are reasonably free from long loose threads. The warp and weft yarns have counts not finer than 15 tex and 25 tex respectively.

Fabric

Fibre identification The dried material obtained in the test for Weight per unit area complies with the tests for *cotton*, Appendix XX A.

Threads per 10 cm Warp, 109 to 131, Appendix XX C1, Method II; weft, 73 to 90, Appendix XX C1, Method I.

Weight per unit area Not less than 39 g m^{-2} when determined by the following method. Measure the area of a sample weighing about 10 g. Boil the sample in *water* until the soluble ingredients in the mass have completely dissolved and the insoluble ingredients have become loosened, add sufficient 2M *hydrochloric acid* to remove any adhering zinc oxide, decant the liquid through a tared sieve with a nominal mesh aperture of 106 µm, transfer the residual fabric to the sieve, wash thoroughly with *water* and dry to constant weight at 105°. Calculate the weight of the fabric, in g m^{-2}, making allowance for serrated edges if present.

Paste

Identification Extract 20 g of the bandage with two 50-ml quantities of 3M *hydrochloric acid*. Extract the combined acid extracts with two 50-ml quantities of *chloroform*. Wash the combined chloroform extracts with 20 ml of *water*, dry by shaking with *anhydrous sodium sulphate*, filter, evaporate the filtrate to dryness, wash the residue with two 10-ml quantities of *hexane* and dry the residue at a pressure not exceeding 0.7 kPa (about 5 torr). The residue complies with the following tests.

A. Dissolve 50 mg as completely as possible in 20 ml of hot *hexane*, filter and allow the filtrate to cool until crystals appear. Decant the hexane, wash the crystals with 5 ml of *hexane*, filter and dry the residue at a pressure not exceeding 0.7 kPa (about 5 torr) for 1 hour. The *infrared absorption spectrum* of the residue, Appendix II A, is concordant with the *reference spectrum* of clioquinol.

B. Fuse 50 mg with *anhydrous sodium carbonate*, dissolve the fused mass in *water* and acidify with 2M *nitric acid*. Add *silver nitrate solution*; a pale lemon coloured precipitate is produced which is insoluble in 5M *ammonia*. Add 5M *ammonia* until the solution becomes alkaline, boil gently, filter and acidify the filtrate with 2M *nitric acid*; a white precipitate is produced which darkens on exposure to light.

Weight Not less than 175 g m^{-2}, calculated from the weight and area of the sample and the weight of fabric obtained in the test for Weight per unit area.

Content of zinc oxide, ZnO Not less than 9.25% w/w when determined by the following method. Ignite 6 g of the bandage until all the carbon is removed, cool the residue, dissolve in 30 ml of 2M *nitric acid* and dilute to 250 ml with *water*. Neutralise 50 ml of this solution to *litmus paper* with 5M *ammonia solution*, add 5 ml of *ammonia buffer pH 10.9* and 100 ml of *water* and titrate with 0.05M *disodium edetate VS* using *mordant black 11 solution* as indicator. Each ml of 0.05M *disodium edetate VS* is equivalent to 4.068 mg of ZnO.

Calculate the weight of the paste taken from the Weight of paste (g m^{-2}) and the Weight per unit area of fabric (g m^{-2}) determined above.

Content of clioquinol, C_9H_5ClINO 0.90 to 1.10% w/w when determined by the following method. Extract a quantity of the bandage containing 50 mg of Clioquinol with five 35-ml quantities of 3M *hydrochloric acid*, filtering each extract through the same sintered-glass crucible (BS porosity No. 1), and dilute the combined extracts to 200 ml with 3M *hydrochloric acid* (solution A).

Boil the residual bandage with successive 50-ml quantities of *water*, filtering each through the sintered-glass crucible, until the filtrate is free from chloride and starch. Transfer the bandage and any loose threads to the sintered-glass crucible, wash with two 20-ml quantities of *chloroform* and dry at 105°. Add 8.5% to the weight of the dried material to allow for moisture regain. The difference between the weight of the bandage taken and the corrected weight of the dried, treated material represents the weight of paste taken.

To 20 ml of solution A add 5M *sodium hydroxide* until the pH is between 1 and 2 and then extract with five 20-ml quantities of *chloroform*. Filter the combined extracts and evaporate to dryness using a rotary evaporator. Add to the residue 10 ml of a hot mixture of 4 volumes of *2-methoxyethanol* and 1 volume of *water* and heat on a water bath for 5 minutes. Cool to room temperature and dilute to 20 ml with the methoxyethanol mixture. To 10 ml add 10 ml of *2-methoxyethanol* and 2 ml of a solution prepared by dissolving 0.5 g of *iron(III) chloride hexahydrate* in 80 ml of *2-methoxyethanol* and adding 0.1 ml of *hydrochloric acid* and sufficient *2-methoxyethanol* to produce 100 ml and dilute to 25 ml with *2-methoxyethanol*. Mix and measure the *absorbance* of the resulting solution at the maximum at 650 nm, Appendix II B, using in the reference cell a solution prepared in the same manner but omitting the preparation. Calculate the content of C_9H_5ClINO from the *absorbance* obtained by repeating the operation beginning at the words 'add 10 ml of *2-methoxyethanol* ...' and using 10 ml of a solution prepared in the following manner. Dissolve 0.125 g of *clioquinol BPCRS* in sufficient *2-methoxyethanol* to produce 50 ml, warming to assist solution. To 4 ml of this solution add 1 ml of *water* and sufficient of a mixture of 4 volumes of *2-methoxyethanol* and 1 volume of *water* to produce 50 ml.

Calculate the percentage content of C_9H_5ClINO in the paste.