

ANNEX B

(Clause 6.2)

PROCEDURE AND TEST METHOD FOR ROT-PROOFING BY PENTACHLOROPHENYL LAURATE

B-1 PRESERVATIVE AGENT — The preservative agent shall be pentachlorophenyl laurate.

B-2 APPLICATION — The process shall consist of an even and thorough impregnation of the textile with either:

- a) a solvent solution of agent; or
- b) an aqueous emulsion of the agent.

This shall be followed by removal of excess and subsequent drying or thorough solvent removal. The treated textile shall be dry in handling and non-tacky.

B-2.1 Amount of Preservative Agent — The amount of preservative agent shall be as follows.

The pentachlorophenyl laurate content of the treated textile shall be not less than 1.7 percent nor more than 3.5 percent.

B-2.2 In neither case shall the free PCP content of the treated textile exceed 10 percent of the pentachlorophenyl laurate content.

B-3 DETERMINATION OF PENTACHLOROPHENYL LAURATE (PCPL) CONTENT

B-3.1 General

The method is applicable to the determination of PCPL in the absence of added pentachlorophenol. The proofing is hydrolyzed, acidified and steam distilled and the pentachlorophenol in the distillate extracted with 1,1,1 trichloroethane and complexed in 1,1,1 trichloroethane is measured on a suitable spectrophotometer at 450 nm.

B-3.2 Reagents

B-3.2.1 Ethanediol (Ethylene glycol)

B-3.2.2 1,1,1-Trichloroethane *Trichloroethane*

B-3.2.3 Pyridine (AR, GRP Grade)

B-3.2.4 Sodium Hydroxide, Pellet

B-3.2.5 Copper Sulphate Reagent Solution 50 g/l

B-3.2.6 Pentachlorophenol (Standard Reagent, Melting Point 188 °C minimum)

B-3.2.7 Hydrochloric Acid — concentrated 36 percent, (m/v) (11M)

B-3.2.8 Copper Sulphate-pyridine Reagent Solution — prepared by mixing 4 ml pyridine with 6 ml copper sulphate solution immediately before use.

B-3.3 Procedure

Weigh 2.5 g ± 0.05 g of the material, cut into small pieces of not more than 5 mm square and place in a dry 250 ml round bottomed flask (B24/29 socket). Add 30 ml of ethanediol, 4 g of sodium hydroxide (pellet form), 2.4 ml of water, in that order and a few anti bumping granules. Connect the flask with a double surface condenser, bring the contents to boiling point on a sand bath and boil them vigorously for 30 min under reflux. After this allow the contents of the flask to cool, remove the reflux condenser and add through a funnel 60 ml water followed by 20 ml hydrochloric acid. Steam distil the contents of the flask ensuring that a constant volume is maintained by applying gentle heat as necessary. Collect 300 ml of distillate in a suitable receiver, applying care to prevent loss of pentachlorophenol in the distillate by adequate cooling. Discontinue the external heating of the flask a few minutes before disconnecting the steam supply. Disconnect the condenser and fit it vertically over the distillate receiver. Wash down the condenser with 25 ml to 30 ml of trichloroethane and collect the washings in the distillate. Transfer the distillate and trichloroethane washing to a 500 ml separating funnel and shake thoroughly. Allow the layers of water and trichloroethane to separate completely before running off the trichloroethane layer into a 100 ml separating funnel. Wash the condenser and distillate receiver with a further 25 ml to 30 ml trichloroethane and add this to the aqueous solution into the 500 ml separating funnel. Repeat the extraction as given above and add the trichloroethane layer to the first trichloroethane extract in the 100 ml separating funnel. Add to the