MT 19X WASH RESISTANCE INDEX OF LN

SCOPE

This method is intended for determination of the wash resistance index of long-lasting insecticidal net (LN) (Note 1). It is suitable for coated and incorporated types of LN. The wash resistance index is defined as the proportion of the content of the active ingredient on/in the LN after washing compared to before washing using the procedure in this method.

OUTLINE OF METHOD

Suitable pieces of the net are subjected to successive washing-rinsing-heating cycles and the total active ingredient content is determined in washed samples after a number of washings (Note 2). The wash resistance index is determined by the decrease of the total content of the active ingredient after several cycles using the appropriate analytical method.

REAGENTS

CIPAC washing agent (Note 3)
Deionised water

APPARATUS

Oven flameproof and thermostatically controlled to the required temperature (± 2°C) (Note 4)
Water bath capable of maintaining a specified temperature (30°C ± 2°C) and large enough to allow several 1 L capped bottles to be immersed in an upright position in the water to the 500 ml mark
1 L screw capped glass bottles conforming to ISO 4796 and DIN 168 (outside diameter 101 mm, height 225 mm) (see Figure 1)
Glass bottles 100 to 125 ml fitted with screw-caps and fitted with polyethylene inserts
Scissors
Tweezers
Stopwatch
Refrigerator

PROCEDURE

(a) Sampling. Cut 6 pieces of 25 × 25 cm from the net or netting (Note 2 and 5). Subject three of the pieces to 4 washing-rinsing-heating cycles as described below in paragraphs b-c-d. Analyse the remaining 3 pieces for their content of

active ingredient without any washing. If desired, label the pieces using a suitable ink for easier identification.

(b) Washing. Add 2.5 ml of the stock solution of the CIPAC washing agent (Note 3) to 500 ml of de-ionised water at 30°C ± 2°C in a 1 L glass bottle. Insert a piece of net and cap the bottle. Invert the bottle 10 times (Note 6). Place the bottle in the water bath in an upright position free from vibration (Note 7) and not under direct sunlight. After 10 minutes, remove the net sample using tweezers, and remove any remaining adherent drops of wash fluid by gentle shaking.

(c) Rinsing. Insert the washed net sample into a 1 L glass bottle containing 500 ml of de-ionised water at 30°C ± 2°C, cap and invert 10 times (Note 6). Place the bottle in the water bath in an upright position free from vibration (Note 7) and not under direct sunlight. After 10 minutes, remove the net sample using tweezers, replace the deionised water, insert the sample into the 1 L bottle and repeat this rinsing step once more.

(d) Heating. Using tweezers pull out the net sample from rinsing fluid and carefully remove any remaining adherent water drops by gentle shaking. Allow the sample to dry for 30 minutes at room temperature on a line and keep protected from direct sunlight. Fold the sample carefully once or twice in each direction, place it loosely rolled in a glass bottle, cap and store it in an oven at 40°C ± 2°C for 22 hours ± 2 hours before starting the next washing cycle (Note 8 and 9). Repeat the washing-rinsing-heating process (b-c-d) 3 more times. At the end each of the 3 pieces of net has been washed 4 times.

(e) Assay. Analyse each of the 6 net samples using the appropriate CIPAC method for determination of total active ingredient content (Note 10). Express results as g active ingredient per kg net material.

(f) Calculation. Calculate the wash resistance index, w, after the 4 washing-rinsing-heating cycles using the following equation (Note 11):

$$w = 100 \times \sqrt[4]{(t_4/t_0)}$$

where:

- t_4 = total active ingredient content (in g/kg) after 4 washing cycles. This value is calculated by averaging the total active ingredient content of the 3 pieces (Note 12)
- t_0 = total active ingredient content (in g/kg) before washing (no washing). This value is calculated by averaging the total active ingredient content of the 3 pieces
- w = wash resistance index, expressed as percentage (Note 13)

(g) Reporting. Report the wash resistance index to the nearest 1 % and specify the following parameters:
- temperature at which the heating procedure was performed if it was deviated from the standard (40°C ± 2°C)
- assay method used

Note 1 Definition of wash resistance index
Long-lasting insecticidal nets (LN) are usually washed several times during their normal use in geographic areas where they are utilized to control vector borne diseases. It is important that the concentration of the insecticide on/in the fibre of the net remains sufficiently high after multiple washings to keep its effectiveness against insects. The term "wash resistance index" is defined as the remaining amount of the total content of one or more active ingredient(s) of a long-lasting insecticidal net after washing.

Note 2 In some cases, finished nets are composed of two or more different netting in their manufacture (combination LNs). Each netting has a different specification which may be for different fibres, and/or active ingredient(s) with or without synergists. In such a situation each of the different netting types shall be evaluated independently following the method described above in paragraphs a-b-c-d.

Note 3 The stock solution of the CIPAC washing agent is prepared as follows: The bottle of polyoxyethylene glycol (25) monostearate (CAS number 9004-99-3 or 37231-60-0) is heated to about 50°C to melt and to reduce its viscosity. The bottle is turned through 180 degrees a few times to ensure homogeneity. Into a suitable glass flask containing 80 ml of de-ionised water weight 12.0 g of sodium oleate (CAS number 143-19-1) plus 8.0 g of polyoxyethylene glycol (25) monostearate. Heat this mixture at ca 50°C turning through 180 degrees frequently or stirring by a magnetic stirrer until the mixture becomes clear and homogeneous. This CIPAC washing agent can be used for up to 4 weeks if the flask is kept sealed in the dark in a refrigerator.

Note 4 The oven should be kept at 40°C ± 2°C unless a lower temperature is stated in the specification of the particular net in order to avoid excessive “sweating” of the netting material.

Note 5 Samples should be taken according to Figure 2 or on a convenient diagonal across the width of bulk material. Do not use material within 10 cm of the seams or edges.

Note 6 The expression “invert the bottle” implies that the bottle is turned by hand through 180 degrees, and is then brought back to its original position, the whole operation being completed in approximately 2 sec.

Note 7 A water bath should be chosen which is as free as possible from vibration, which may affect the wash resistance index (the total active

ingredient content unwashed). If the water bath without stirrers is used, the temperature should be checked frequently to ensure that the water bath is maintaining the proper temperature. Alternatively a thermostated oven at 30°C ± 2°C can be used.

Note 8 The storage at 40°C ± 2°C is intended to allow migration of the active ingredient from the inner parts of the fibre of the long-lasting insecticidal net to the surface. This process is essential for both LNs (incorporated or coated LN) to establish a physico-chemical equilibrium of active ingredient between the surface of the net and the inner part of the coating or the fibre before the next washing. The average storage time until the next washing step is approximately 22 hours. Lower heating temperatures may be necessary and justifiable for certain net/active ingredient combinations.

Note 9 When longer storage times are necessary, store the sample folded and loosely rolled in a glass bottle at low temperature (from 0°C to 5°C) while protecting it from direct sunlight. Net samples having finished their scheduled washing cycles can also be stored in a glass bottle in a refrigerator at 4°C (± 3°C) awaiting analysis.

Note 10 Unwashed samples and wash samples must be analysed simultaneously to reduce the analytical error.

Note 11 This equation is based on the free migration model (see e.g. Report of the eleventh WHOPES working group meeting 10 – 13 December 2007, ref: WHO/HTM/NTD/WHOPES/2008.1). If this model is inappropriate for describing the wash resistance of a specific LN, an alternative model may be justified.

Note 12 If the variation in active ingredient content calculated is significantly high, this value is to be re-calculated by averaging the total active ingredient content of a minimum of 3 pieces.

Note 13 Example for illustration: After 4 washing-rinsing-heating cycles the average active ingredient content is reduced to 78 % compared to the value prior to washing. The calculated wash resistance index in this case would be 94 %. This implies that the average active ingredient content after – hypothetically – <u>one</u> washing-rinsing-heating cycle would be reduced by 6 %.

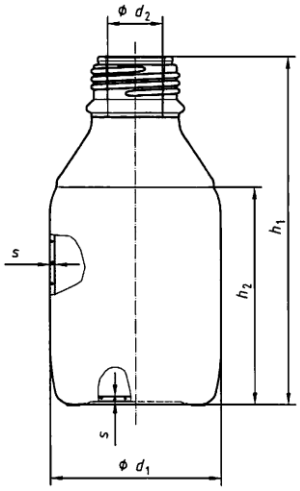

Figure 1 1 L screw capped glass bottle (diameter 101 mm, height 225 mm)

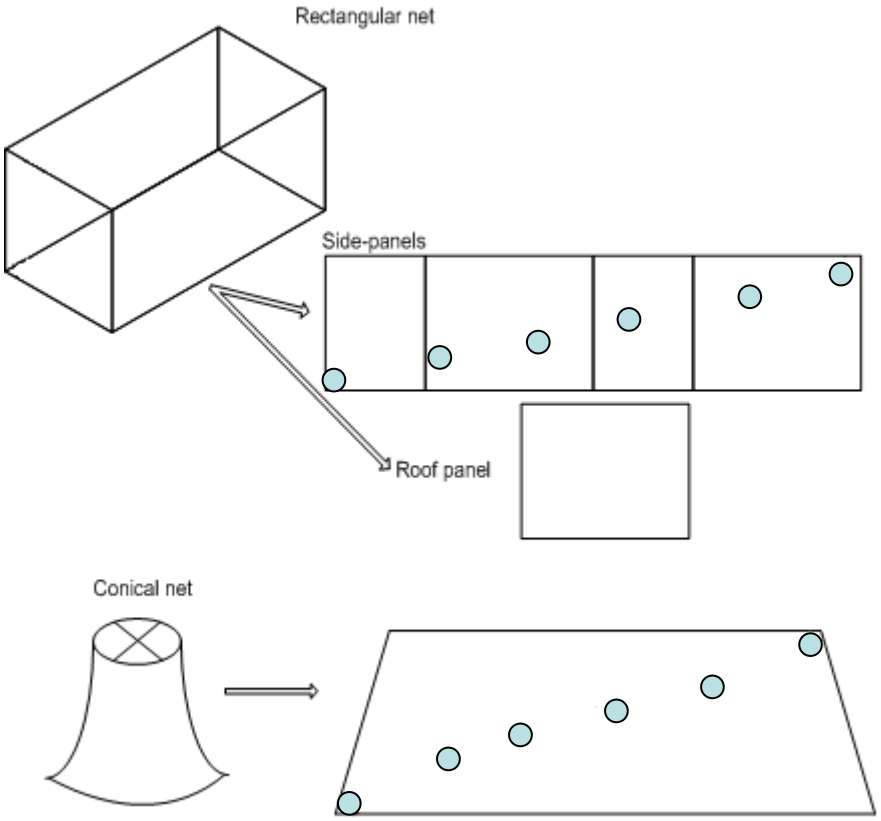

Figure 2 Recommended positions from which 6 pieces of netting should be taken from a finished LN to form a representative sample