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Mosquito nets — Specification

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Foreword

Uganda National Bureau of Standards (UNBS) is a parastatal under the Ministry of Tourism, Trade and Industry established by the Act of Parliament of 1983, of the Laws of Uganda. UNBS is mandated to co-ordinate the elaboration of standards and is

(a) a member of International Organisation for Standardisation (ISO) and

(b) a contact point for the WHO/FAO Codex Alimentarius Commission on Food Standards, and

(c) the National Enquiry Point on TBT/SPS Agreements of the World Trade Organisation (WTO).

The work of preparing Uganda Standards is carried out through Technical Committees. A Technical Committee is established to deliberate on standards in a given field or area and consists of representatives of consumers, traders, academicians, manufacturers, government and other stakeholders.

Draft Uganda Standards adopted by the Technical Committee are widely circulated to stakeholders and the general public for comments. The committee reviews the comments before recommending the draft standards for approval and declaration as Uganda Standards by the National Standards Council. The Technical Committee on the Textile and Apparels (TC 7) elaborated this Uganda Standard and the following institutions were represented.

Committee membership

The following organisations were represented on the Textile and Apparels Standards Technical Committee, UNBS/TC 7, during the development of this standard:

- Kyambogo University
- Busitema University
- Ministry of Health (National Malaria Control Programme)
- Nett shoppe Uganda Limited
- Sumitomo Chemical
- Vestergaard
- Uganda Industrial Research Institute
- Uganda National Bureau of Standards

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Mosquito nets — Specification

1 Scope

This Draft Uganda Standard prescribes requirements for long lasting insecticidal Mosquito nets intended for malaria vector control.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1833, *Textiles — Binary fibre mixtures — Quantitative chemical analysis*

ISO 2060, *Textiles — Yarn from packages — Determination of linear density (mass per unit length) by skein method*

ISO 2960, *Textiles — Determination of bursting strength and bursting distension — Diaphragm methods*

ISO 3758 *Textiles* — Care labelling code using symbols

ISO 3801, *Textiles — Woven fabrics — Determination of mass per unit length and mass per unit area*

ISO 5077, *Textiles — Determination of dimensional change in washing and drying*

ISO 6330, *Textiles — Domestic washing and drying procedure for textile testing*

ISO 6941, *Textile fabrics — Burning behaviour — Measurement of flame spread properties of vertically orientated specimens*

ISO 8388 *Textiles* — Knitted fabrics. Types. Vocabulary

US 417, *Glossary of terms for defects in fabrics – Woven fabrics*

US 418, *Glossary of terms for defects in fabrics – Knitted fabrics*

3 Terms and definitions

For the purpose of this standard the definitions given in US 417 and US 418 and the following definitions shall apply.

3.1 Long Lasting Insecticidal Nets (LLIN)
factory treated mosquito net made with a netting material that has insecticide incorporated within the yarn or wound around the fibres. The LLIN must have the capacity to retain its effective biological activity without re-treatment for at least twenty WHO standard washes under laboratory conditions and three years of recommended use under field conditions hence fulfilling the WHOPES 2 test.

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3.2 Combination nets

are finished nets composed of different netting materials

4 Symbols (and abbreviated terms)

The abbreviations below are found throughout this standard.

- WHO World Health Organization
- WHOPES World Health Organization Pesticides Evaluation Scheme
- PBO Piperonyl butoxide

5 Requirements

5.1 General

5.1.1 Textile netting materials

The textile materials used for the manufacture of mosquito netting and any other textile products intended for malaria vector control shall fulfil the requirements as specified in tables 1 to 5 below.

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Table 1 — Specification for netting materials

Parameter	Specifications Polyester	Polyethylene	Test method (ISO)
Fibre	100 % polyester	100 % polyethylene	ISO 1833
Denier	≥ 100	≥ 100	ISO 2060
Filaments	Multi-filament ≥ 36	Mono/multi-filament	
Mesh size	24 holes/cm^2 Min.	6.25 holes/cm^2, min	ISO 1833
Weight (g/m^2)	40 g/m^2.(min)	40 g/m^2.(min)	ISO 3801
Bursting strength	350 Kpa.(min)	350 Kpa.(min)	ISO 2960
Dimensional stability	Threshold/range of 5 % Max	Threshold/range of 5 % pa Max.	ISO 6330 ISO 3801 ISO 5077
Flammability (Fire safety)*	Not inflammable (class 1)	Not inflammable (class 1)	16 CFR Part 1610
Weave / netting type	Warp knitted	Raschel[1)]	ISO 8388
Colour fastness to: Light Washing	4 or better 4 or better	4 or better 4 or better	ISO 105
Seam	sewn using 100% polyester thread	sewn using 100% polyester thread	

1) Raschel knit is a warp knitted fabric that produces fabric similar to tricot but usually of coarser texture and with openwork patterns

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Table 2 – Specification for netting materials for combination nets

Parameter	Requirement	Test Method
Fibre	Roof: 100 Polyethylene Side: 100 Polyester	ISO 1833 or ASTM D276
Denier	100D	ASTM D 1907-1 / ISO 2060 - Option 1
Filaments	Roof: Monofilament Sides: Multifilament	Counting
Mesh size	Roof: Min. 20 holes/cm^2 Side: Min. 24 holes/cm^2	ISO 1833
Weight (gm^2)	Roof: Min. 30 g/m^2 Sides: Min. 36 g/m^2	ISO 3801
Bursting strength	Roof: Min. 400 kPa Sides: Min. 350 kPa	ISO 13938 - 1, head area = 7.3 cm^2
Dimensional stability of netting to wash	Max. 5 %, either shrinkage or expansion	ISO 3759, ISO 6330, 8A / Flat drying, ISO 5077.
Flammability (Fire safety)*	Class I	16 CFR Part 1610
Weave / netting type	Warp knitted	ISO 8388
Colour fastness to light	4 or better	ISO 105, B02, Test method 2
Seam	sewn using 100% polyester thread	Visual check

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Active Ingredients of the combination Net

SL NO	Ingredient	Dosage	
		g/Kg	Mg/m^2
Roof	Deltamethrin	3.00 - 5.00	105 - 175
	PBO	18.75 - 31.25	656.25 - 1093.75
Sides	Deltamethrin	2.1 - 3.5	63.0 - 105

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5.1.2 Defects

5.1.2.1 The mosquito nets shall be free from any non-acceptable defects.

5.1.2.2 Defects allowed shall only be classified into major and minor defects as per the relevant Uganda standards

5.2 Sizes and dimensions for mosquito nets

5.2.1 Sizes

The mosquito nets shall be of any of the following sizes:

(a) X-small;

(b) Small;

(c) Medium;

(d) Large; and

(e) X- large.

5.2.2 Dimensions

The dimensions of the mosquito nets shall be as given in tables 5 and 6 for the respective nets.

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Table 3 — Rectangular net dimensions

Net Size	Width, min, cm	Length, min, cm	Height, min, cm
X small	70	180	170
Small	100	180	170
Medium	130	180	170
Large	160	180	170
X- Large	190	180	170
NOTE For manufacturers, sizes not conforming with the above dimensions will be considered special orders The text/column in red to be deleted, it is not relevant			

Table 4

— Conical nets dimensions

Net Size	Height, Min Cm	Circumference diameter, Min cm	Ring diameter, Min cm
Single fitted conical	220	850	56
Double fitted conical	250	1050	56
Extra double fitted	250	1250	65

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5.3 Active ingredients

For treated nets, the active ingredients of nets shall comply with requirements of table 5 when tested in accordance with annexes A, B and C

Table 5: Active ingredients of nets

SL NO	Ingredient	Dosage	
		g/Kg	Mg/m2
1	Deltamethrin	, 1.05-1.75	, 41.25- 68.75
2	Permethrin	17-23	510-1150
3	Alpha –cypermethrin	3.75-8.4	150-250

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5.4 Manufacture and workmanship

5.4.1 General requirements

5.4.1.1 The mosquito net shall be rectangular or conical in shape unless otherwise agreed between the purchaser and the supplier.

5.41.2 The hem shall be firm and capable of withstanding the normal conditions of use.

5.5 Reinforcement at bottom

The bottom edge of the walls shall be stitched, hemmed, given a finished edge or be reinforced.

Note A sheeting border is sometimes required to improve the lifespan of nets.

5.6 Net attachments or tying tapes

Rectangular nets shall be equipped with non-rusting metal rings or fabric loops . Each ring/loops shall have a corresponding string for suspensions. Conical nets shall be equipped with a loop/non-rusting ring with sufficient strength.

5.7 Top support ring

Conical nets shall have a top support ring made of non-rusting metal and which has sufficient strength to withdraw the net when hanged at the required position.

6 Packing and labelling

6.1 Packing

The packaging material shall be leak proof and shall be sufficiently large to wrap completely the mosquito net bundles. The individual nets' packaging materials should be biodegradable.

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6.2 Labelling

6.2.1 General

The labelling shall include

(a) a permanent label attached to the net (tag); and

(b) a pictorial leaflet relating to insecticide treatment and care of nets.

6.2.2 Label tag

The label tag attached to the net shall contain the following information:

(a) type of netting material;

(b) net dimensions in centimetres (height, width and length for rectangular nets, net diameter and height for conical nets and surface area);

(c) country of origin;

(d) name and physical address of the manufacturer and/or importer/distributor;

(e) status of treatment that is, insecticide used, formulation and dosage;

(f) the efficacy of the chemical shall be indicated in term of number of washes;

(g) lot/ batch number; and

(h) pictograms or simple instructions in accordance with ISO 3758 giving warning to avoid frequent washing, high temperature or dry cleaning (solvents), bleaching and exposure to sun light.

6.2.3 Pictorial leaflet

The pictorial leaflet shall contain information relating to insecticide treatment and the care of nets. The following information shall appear:

(a) size in centimetres and surface area in square metres;

(b) washing instructions in pictograms (temperature for washing, no bleach, no ironing, no dry cleaning and no tumble;

(c) instructions not to leave the net in the sun; and

(d) a warning that frequent washing removes the insecticide.

7 Sampling

7.1 Lot

The quantity of mosquito nets delivered to a consignee against the same dispatch note shall constitute a lot.

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7.2 Scale of sampling

7.2.1 Samples shall be tested for each lot for ascertaining its conformity to the requirements of this standard.

7.2.2 The number of pieces to be selected from a lot shall be in accordance with Table 7.

Table 7 – Scale of sampling

Number of pieces in the lot	Number of pieces to be selected
Up to 8	2
9 to 15	3
16 to 25	4
26 to 50	5
51 and above	7

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Annex A
(normative)

Determination of Deltamethrin content in Insecticide treated Mosquito nets by High performance Liquid Chromatography

A.1 Sampling

A sample is defined as one finished bed net taken randomly from a batch of bed net. Sub sample by cutting 18 pieces of size 100cm^2 from a net either randomly from the whole net or at recommended position as in appendix 1. Cut 18 pieces into half, one portion is used for Deltamethrin content analysis and the other is used for washing test if required.

The portion used for Deltamethrin content analysis is cut into pieces of less than 2cm x 2cm each. Mix well. Sample shall be separately packed in aluminium foil and kept out of direct sunlight at room temperature or lower.

A.2 Identification tests.

Use the HPLC method below. The retention time of Deltamethrin in the sample solution should not deviate by more than 15 s from that of the calibration solution if column oven is available.

A.3 Active ingredient

A.3.1 Outline of Method

The sample is extracted in a mixture of iso-octane and 1,4-dioxane.

The Deltamethrin content is determined by normal phase high performance liquid chromatography using dipropyl phthalate as internal standard and detection at 236 nm.

A.3.2 Scope

This method is used for Deltamethrin determination in net sample, before and after washing.

A.3.3 Reagents

A.3.3.1 Iso Octane, HPLC grade

A.3.3.2 1,4 Dioxan, HPLC grade. Add 0.15% (v/v) water before use

A.3.3.3 Deltamethrin, neat standard,

A.3.3.4 Dipropyl Phthalate,

A.3.3.5 Water ,HPLC grade or high

A.3.3.6 Extraction solvent (ES): iso octane + 1,4 dioxane=80+20

A.3.3.7 Mobile Phase (MP):iso octan + 1,4 dioxane = 95 + 5

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A.3.3.8 Internal Standard solution (IS)0.5 mg/ml of dipropyl phthalate in extraction solvent.

A.3.3.9 Deltamethrin standard solution 0.6 mg/ml(DS)

Weight 0.03 g(to the nearest of 0.01` mg) of Deltamethrin neat standard, quantitatively transfer to 50 volumetric flask, dissolve completely with extraction solvent , keep regulated by water bath at 20°C, fill up to mark with extraction solvent.

A.3.4 Apparatus

A.3.4.1 Shaker

A.3.4.2 Ultrasonic bath

A.3.4.3 HPLC, equipment with pump, auto-injector , column oven (optional) and UV detector, Guard Column (Supelguard Si). Analytical Column Supelco Si 5 µm, 150 x 4.6 or Lichrosorb Si60, 5 µm, 150 x 4.6

A.4 Procedure

A.4.1 Operating conditions

A.4.1.1 Mobile Phase: iso octan + 1,4 dioxane =95 + 5

A.4.1.2 Flow rate 1.3ml/min, isocratic

A.4.1.3 Guard Column Superguard Si

A.4.1.4 Analytical Column Licherosorb Si60, 5µm, 150 x 4.6 40°C if column oven is available, or room temperature

A.4.1.5 Inject Volume 5µl

A.4.1.6 Wavelength 236 nm

A.4.1.7 Run time 6-8 minute

A.4.2 Preparation of calibration curve

A.4.2.1 Into a series of clean 20ml PTFE liner screw cap vials, add accordingly as table below. Filter through 0.45µm syringe filter before use.

Code	IS	DS	ES	Delta(mg)	Total Volume
C1	1ml	0.5ml	13.5 ml	0.30	15 ml
C2	1ml	0.7ml	13.2 ml	0.42	15 ml
C3	1ml	0.9ml	12.8 ml	0.54	15 ml
C4	1ml	1.1ml	12.4 ml	0.66	15 ml
C5	1ml	1.3ml	12.1 ml	0.78	15 ml

A.4.2.2 All standards should be kept in the refrigerator if not in use in WELL-TIGHTEN, paraffin film sealed cap.

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A.4.3 Calibration

A.4.3.1 Daily calibrate HPLC system with full series of 05 standards, beginning with the lowest level standard. The correlation coefficient should be less than 0.99 over this range.

A.4.4 Preparation of sample

A.4.4.1 Weigh (to the nearest 0.1 mg) into an Erlenmeyer or a screw cap neutral glass bottle (50ml) sufficient sample to contain about 0.5 mg of Deltamethrin. For 75D, 100D and 150D netting sample, suitable weight (w g) is 0.3 g, 0.4 g and 0.6 g respectively. Add by pipette 1.0 ml internal standard solution. Add 14 ml extract solvent. Replace the cap closely.

Put the bottles into the ultrasonic bath, setting temperature 80°C, running time 15 min. Vigorously shake the bottle using the shaker in 30 min at room temperature, speed of shaking is at level of 150-200 beats per minute.

Using a syringe membrane filter with pore size of 0.45 µm or finer, filter c.a 1ml of extract solution into clean amber. Sample shall be injected within 24 hours since extraction, for longer waiting time the vial should be kept in a refrigerator.

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A.4.5 Determination

A.4.5.1 Inject sample with HPLC configured as in (a)

$$\text{Deltamethrin content(g/kg)} = \frac{a}{w}$$

a=Deltamethrin reading from the analysis in mg

w=mass of sample taken (g)

Note: Conventional unit of active ingredient content (deltamethrin) for netting is mg/m^2. The conversion from g/kg to mg/m^2 is made as follows:

Deltamethrin content (mg/m^2) = Deltamethrin content (g/kg) x D

D=weight per square meter

D of typical polyester netting

75 Denier netting, D=30g/m2

100 Denier netting, D=40g/m^2

150 Denier netting, D=60g/m^2

A.4.6 Method Validation Summary

A.4.6.1 Precision

Analyze 05 replicates RSD is 1.8%

A.4.6.2 Accuracy

Recovery of 04 Laboratory Synthetic samples is in the range of 97% to 101%

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Averaged at 99.3%

A.4.6.3 Linearity

Over the range of Deltamethrin from 0.30 t0 0.78 mg, the correlation coefficient R^2 is observed not less than 0.995.

Determination of release /Retention Index

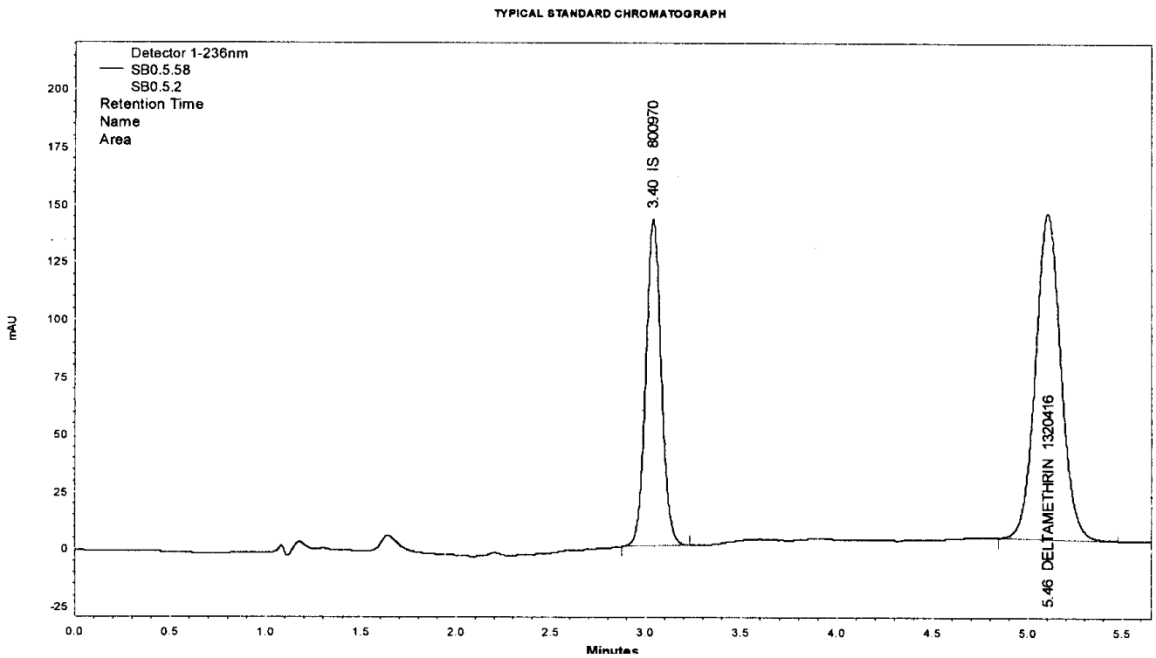

Figure 1:Chromatogram of calibration solution

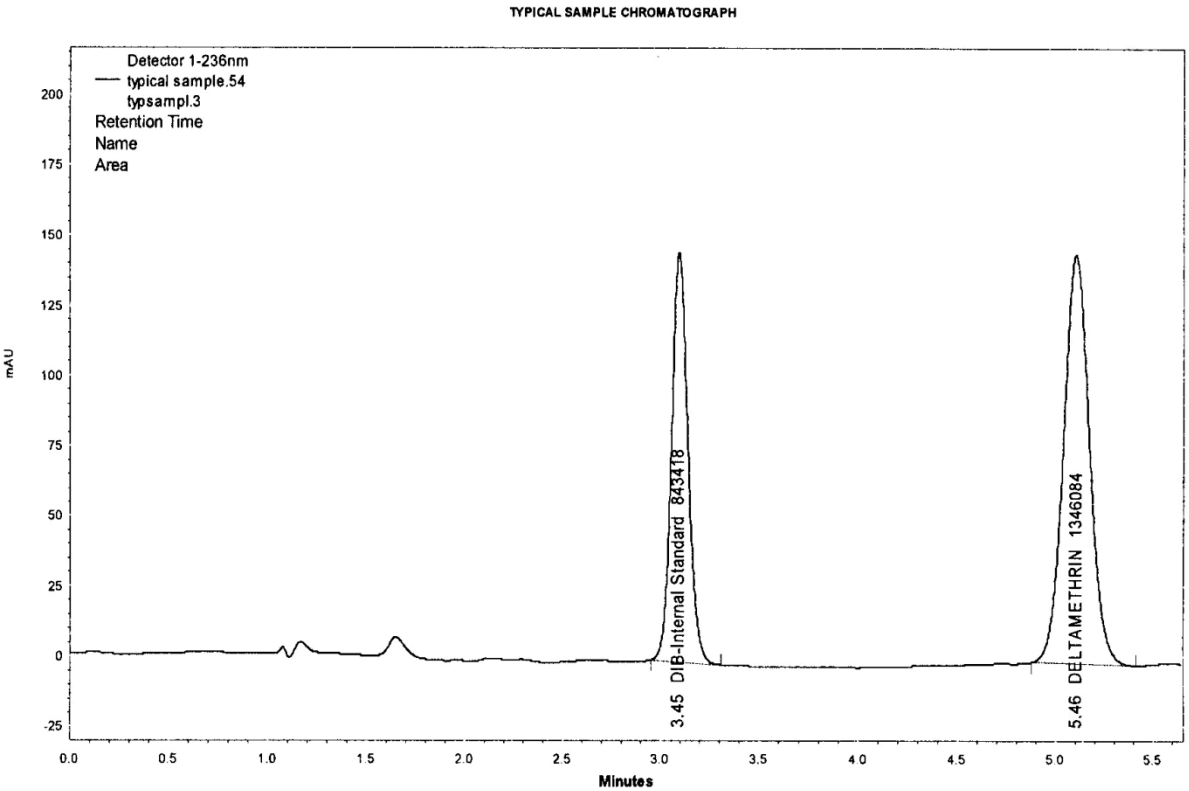

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Figure 2: Chromatogram of sample

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Annex B
(normative)

Determination of Permethrin content in Insecticide treated mosquito nets

B.1 Sampling:

Take at least 100g

B.1.2 Identity tests

B.1.3 GLC. Equipped with a split/splittless and a flame ionization detector. Capillary column fused silica,30mx0.25 (i.d) mm, film thickness:0.25 µm, coated with cross linked dimethyl (DB-1 or equivalent) and electric integrator or data system

B.1.4 Infrared. Extract the sample with suitable solvent. Filter and evaporate the solvent. Proceed as for 331/TC/m/2.2

B.1.5 Permethrin . The content of permethrin in test samples are determined by capillary GC using flame ionization detection and triphenyl phosphate as internal standard, and trans-isomer ratio is calculated from the chromatogram obtained

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B.2 Reagents

B.2.1 Heptane analytical grade

B.2.2 **Internal standard solution**. Dissolve triphenyl phosphate (1.0g) in heptane (150 ml). Ensure that a sufficient quantity of this solution is prepared for all samples and calibration standards to be analysed.

B.2.3 **Calibration solution**. Homogenise the permethrin working standard. When the permethrin working standard is waxy solid or partly waxy solid homogenize it by warming it to melting and stirring. Prepare calibration solutions in duplicate. Weigh (to the nearest 0.1 mg) 72 to 88 mg(s mg) of permethrin working standards into a vial or stoppered flask (200 ml). Add by pipette internal standard solution (10 ml) and dissolve completely. Add by measuring cylinder heptanes (90 ml) and mix well (Solutions C_A and C_B)

B.3 Procedure

B.3.1 Preparation of sample solution.

Clean scissors with acetone before use. Cut the sample with the scissors into 5-10mm square. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1mg) sufficient sample to contain 36 to 44mg (w mg) of permethrin into a vial or stoppered flask (100ml). Add by pipette internal standard solution (5 ml) and by measuring cylinder heptane(45 ml). Place the vial or stoppered flask in a water bath(85-90ºC) for 45 minutes. Shake the vial or stoppered flask once or twice during extraction. Filter a portion of each sample solution through a filter paper prior to analysis (Solutions S_A and S_B)

B.3.2 Calculation of permethrin content.

B.3.2.1 Calculate the mean value of each pair of response factors bracketing the two injections of a sample and use this value for calculating the permethrin contents of the bracketed sample injections.

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$$fi = \frac{lrxsxP}{Hsx2}$$

Content of permethrin $= \frac{fxHw}{lqxw} g/kg$

Where:

fi = individual response factor

f= mean response factor

Hs=total peak area of permethrin (cis-permethrin + trans-permethrin) in the calibration solution

Hw= total peak area of permethrin (cis-permethrin + trans -permethrin) in the sample solution

Ir= peak area of the internal standard in the calibration solution

Iq=peak area of the internal standard in the sample solution

s=mass of permethrin working standard in the calibration solution (mg)

w=mass of sample taken (mg)

P=purity of permethrin working standard (g/kg)

B.3.2.2 Precision

B.3.2.1 Repeatability **r** = 1.6 g/kg at 20.3 g/kg active ingredient content

= 1.3 g/kg at 20.0 g/kg active ingredient content

= 0.9 g/kg at 18.7 g/kg active ingredient content

B.3.2.2 Reproducibility **R** = 1.9 g/kg at 20.3 g/kg active ingredient content

=1.5 g/kg at 20.0 g/kg active ingredient content

=1.5 g/kg at 18.7 g/kg active ingredient content

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B.4 Determination of Surface concentration and release index

B.4.1 Reagents

Internal standard solution for calibration solution.

Dissolve triphenyl phosphate (0.1 g) in acetone (200ml) to prepare a stock solution. Transfer by pipette the stock solution (5 ml) to a volumetric flask (50ml). Make up to volume with acetone and mix well. Ensure that a sufficient quantity of this solution is prepared for all calibration standard to be analysed.

Calibration solution. Homogenise the permethrin working standard. When the permethrin working standard is always solid or partly solid homogenise it by warming it to melting and by stirring. Prepare calibration solutions in duplicate. Weigh (to the nearest 0.1 mg) 90 to 110 mg (s mg) of permethrin working standard into a volumetric flask (100 ml) and make up to volume with acetone and mix well. Transfer by pipette this solution (1 ml to a volumetric flask (20 ml), make up to volume with acetone and mix well. Transfer by pipette this solution (5 ml) to a vial (20 ml), add by pipette internal standard solution for calibration solution (5 ml) and mix well (Solution C_A and C_B).

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B.4.2 Apparatus as for 331/TC/m/3 except:

Constant temperature oven capable of controlling temperature with the range of ±2°C is recommended.

Rotary evaporator

B.4.3 Procedure as for 331/TC/m/3 except:

B.4.3.1 Gas chromatographic conditions (typical):

B.4.3.2 Injection system

B.4.3.3 Slit flow approximately 10ml/min

B.4.4 Linearity check.

Check the linearity of the detector response by injecting 1 µl of solution s with permethrin concentrations 0.1, 1 and 2.5 times that of the calibration solution before conducting analysis.

B.4.5 System equilibration.

Prepare two calibration solutions. Inject 1 µl portions of the first one until the response factors obtained for two consecutive injections differ by less than 2.0%. Then inject a 1 µl portion of the second solution. The response factor for this solution should not deviate by more than 2.0% from that for the first calibration solution, otherwise prepare new calibration solutions.

B.4.6 Preparation of sample solution

Clean scissors and tweezers with acetone before use. Prepare sample solutions in tripricate for each sample [Note 1]) Cut ca .5 cm x 5 cm net samples with the scissors. Weigh accurately , to the nearest 0.1 mg, of each sample (w mg). Transfer it with the tweezers into a vial (20 ml). Add by pipette internal standard solution for sample solution (10 ml). Cap the vial and shake the solution by hand for 1 minute [Note 2.] Take out ht netting with the tweezers and discard the solution.

Let the netting dry at room temperature for ca. 10 minutes. Transfer it into a vial (20ml) with the tweezers. Cap the vial and place it in a temperature -controlled oven set at 70Oc. Heat the sample for 2 hours [Note 3)] . After heating , remove the vial from the oven and let it equilibrate to room temperature. Add by pipette internal standard solution for sample solution (10ml) into the vial. Cap the vial tweezers. Let the netting dry at room temperature for ca. 10 minutes. Transfer the sample solution from the vial into the same round-bottom flask. Evaporate the solution in vacuo to dryness. Add by pipette acetone (2ml) into the flask to dissolve the residue (Solutions for "surface concentration at pot-wash 1", S_{A1},S_{BI} and S_{C1}).

Transfer the dried netting into a vial (20ml) with the tweezers. Repeat heating, internal standard solution adding, evaporation and dissolving procedures as above. (Solutions for "surface concentration at Post -wash 2", S_{A2}, S_{B2} and S_{C2}). Again, let the netting dry at room temperature for ca. 10 minutes. Transfer the dried netting into a vial (20ml) with the tweezers. Repeat heating, internal standard solution adding, evaporation and dissolving procedures as above. (Solutions for surface concentration at post-wash 3", S_{A3},S_{B3}, and S_{C3})

Note 1: Analytical error is larger than average content determinations

Triplicate determinations, therefore ,are recommended

Note 2: The shaking speed is about 30 times per 10 seconds

Note 3: Put vials in a covered cardboard box while heating so that the vials are not exposed to direct streams of warm air.

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B.4.7 Determination .

Inject in duplicate 1 µl portions of each sample solution bracketing them by injections if the calibration solutions as follows; calibration solution CA, sample solution SA1, sample solution SA1, calibration solution CB, sample solution SB1, sample solution SB1, calibration solution CA and so on measure the relevant peak areas.

B.4.8 Calculation of surface concentration.

Calculate the mean value of each pair of response factors bracketing the two injections of a sample and use this value for calculating the surface concentration of the bracketed sample injections.

$$fi = \frac{lrxsxP}{Hs}$$

Surface concentration= $\frac{fxHw}{lqxwx2}$ µg/g

Where:

fi=individual response factor

f=mean response factor

Hs=total peak area of permethrin (cis-permethrin + trans-permethrin) in the calibration solution

Hw=total peak area of [ermethrin (cis-permethrin + trans-permethrin) in the sample solution

Ir= peak area of the internal standard in the calibration solution

Iq=peak area of the internal standard in the sample solution

s=mass of permethrin working standard taken (mg)

w=mass of sample taken (mg)

P=purity of permenthrin working standard (g/kg)

B.4.9 Calculation of release index.

Calculate the mean value of the two injections of sample solutions SA3, SB3, and SC3 by the equations described in (f) , and the release index for each piece of the netting.

Release index = $\frac{C}{B}$

Where:

B= mean value of surface concentration at post-wash 2 (µg/g)

C=mean value of surface concentration at post-wash 3 (µg /g)

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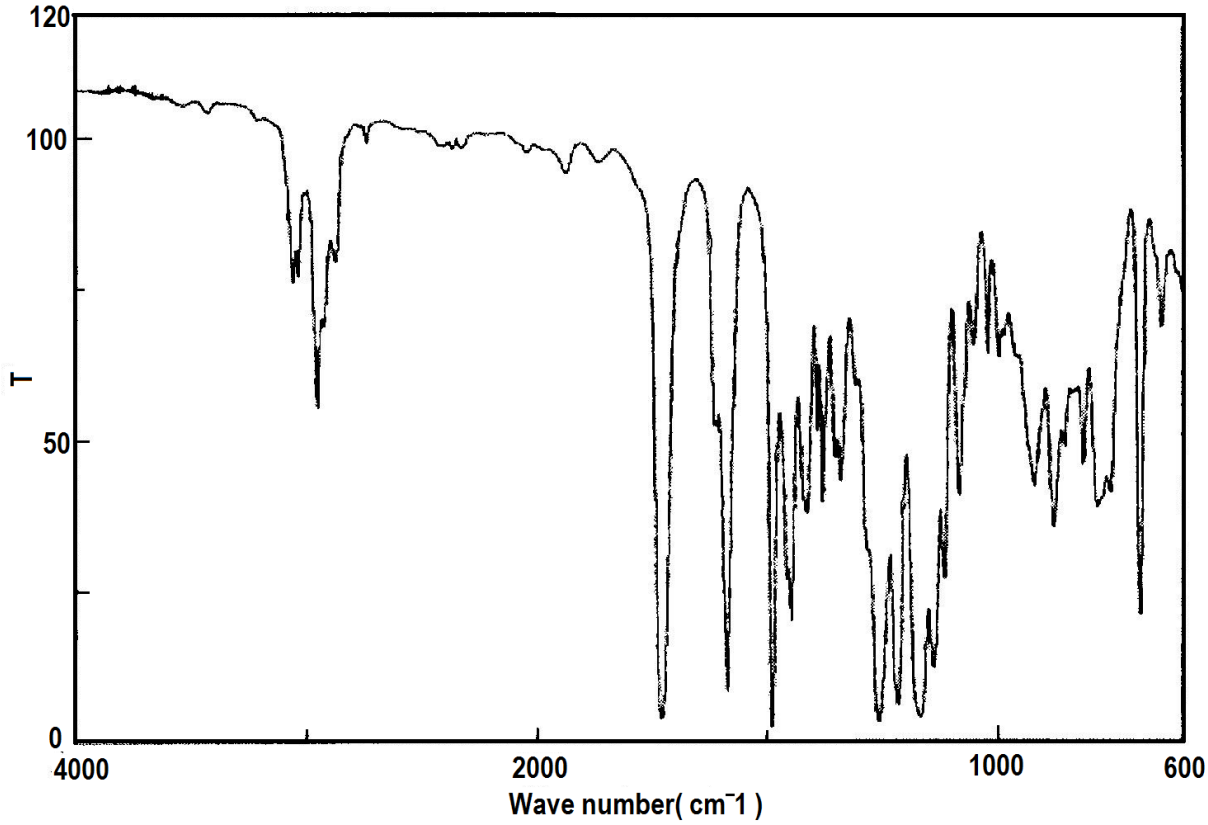

Fig. 1 Infrared Spectrum of permethrin

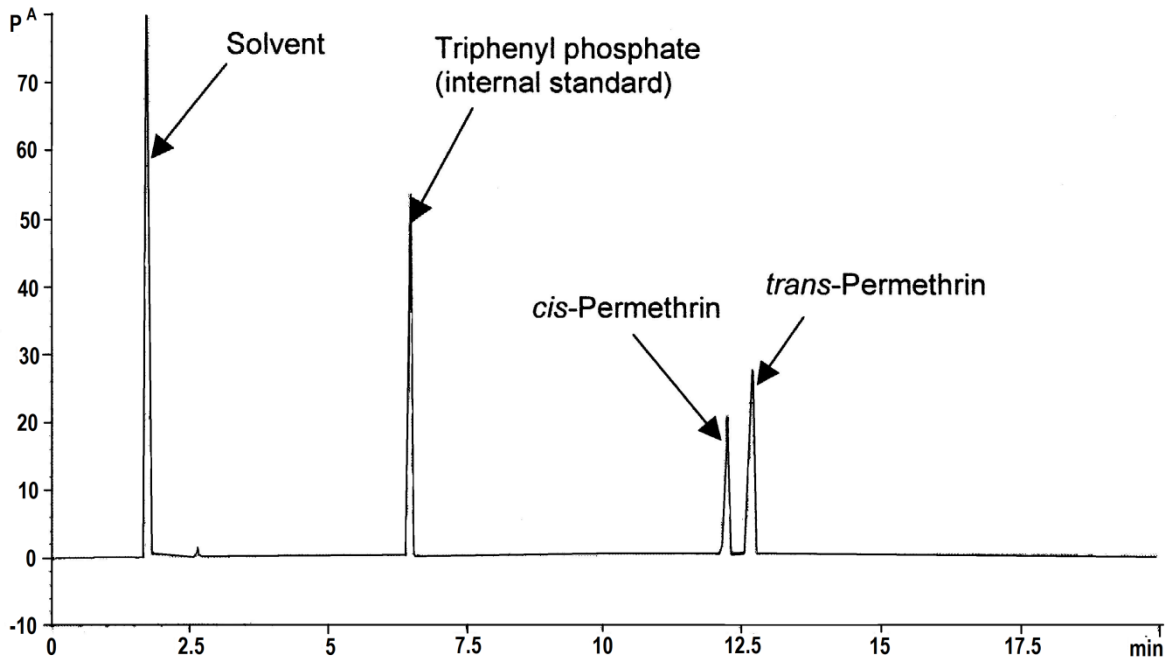

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Fig 2 Example of gas chromatogram of permethrin TC

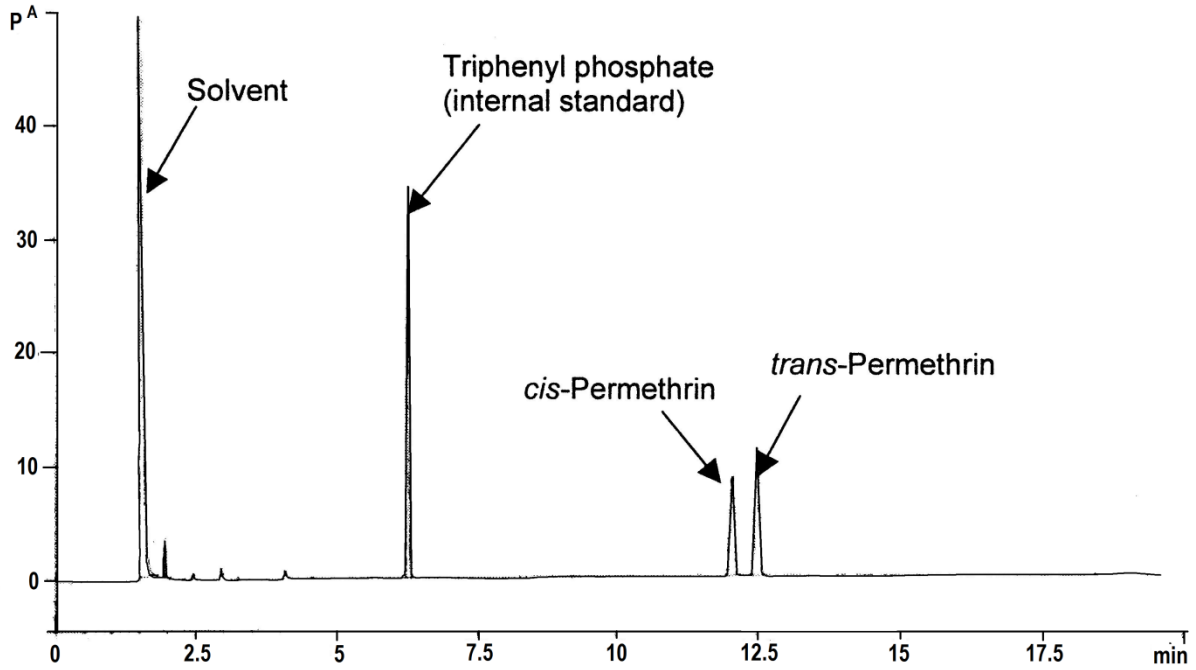

Fig 3 Example of gas chromatogram of permethrin LN

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Annex C
(normative)

Determination of Alphacypermethrin content in Insecticide treated mosquito nets

C.1 Sampling

Take net specimens

C.2 Identity test

GLC capable of operating over a range 100 to 300ºC, fitted with a flame ionization detet ector

C.3 Alphacypermethrin

C.3.1 As for alphacypermethrin 454/TC/(M)3

C.3.2 Outline of method: As for alphacypermethrin is dissolved in tetrahydrofuran and determined by capillary gas chromatography in split injection mode using flame ionization and internal standardization

C.4 Reagents:

C.4.1 Tetrahydrofuran

C.4.2 Aphacypermethrin standard of known purity,Di(2-ethylhexyl)phthalate(dioctyl phthalate, DOP), internal standard,purity atleast 980 g/kg and giving no peaks with similar retention times to alphcypermethrin

C.4.3 Citric acid 5% solution. Dissolve citric acid(25g) in water (500ml)

C.4.4 Internal standard solution. Disolve dioctyl phthalate (5.0g) intetrahydrofuran (500ml). Ensure sufficient quantity of this solution is prepared for all samples and calibration solutions to be analysed

C.5 Calibration Solutions

C.5.1 Weigh 50 mg alpha cypermethrin (to the nearest 0.1 mg) in a volumetric flask (25ml). Fill to the mark with THF. Place the flask in an ultrasonic bath for 10 min. After temperature equilibrium pipette 1.50 ml, 4.50ml of this solution into three volumetric flaks (50 ml). Add 0.5 ml of internal standard solution (dioctyl phthalate, 1% in acetone) and fill up each to the mark with THF.

These solutions are used as calibration solutions A (C_A)' B (C_B) and C (C_C). Transfer 200 µl out of each flask into separate GC vials. Add one drop of citric acid in each case and seal the vials. Place the vials into the sample tray (cooled down to 15º C) of the GC apparatus.

Note: Citric acid is added to stop epimerization of alphacypermethrin in solution

C.5.2 Description of the calibration solutions:

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C_A :concentration of approximately 3.0 mg alphacypermethrin in 50ml THF

C_B:concentration of approximately 6.0 mg alphacypermethrin in 50 ml THF

C_C:concentration of approximately 9.0 mg alphacypermethrin in 50 ml THF

C.5.3 Apparatus

C.5.3.1 Gas chromatograph with flame ionization detector (e.g. HP6890 Plus)

C.5.3.2 Automatic sample injector (e.g. HP series 7683) equipped with a sample which is cooled down to 15ºC

C.5.3.1 Capillary column fused silica (e.g. DB-1) 30mx 0.32 mm, film thickness of 0.25µm.

C.5.3.2 Electronic data evaluation system (e.g. HP ChemStation)

C.5.3.3 Ultrasonic bath

C.5.3.4 Electronic balance

C.5.3.5 Laboratory glassware

C.6 Procedure

C.6.1 Operating conditions. As per alphacypermethrin 454/TC/(M)3

C.6.1.1 Preparation of the samples

C.6.1.2 Determination via the water/detergent extraction process

C.6.1.2.1 **First step:** Quantification of the adherent contents of alphacypermethrin on surface fibers

a) Procedure done by manufacturer

Place 2 g impregnated net in a beaker (1l).Add soap (commercially available bar soap or as flakes, 2g/l) and water (500ml). The washing is done for 10 min. at 30º C on a shaker with 155 movements/min. Directly after the washing, the wash liquor is acidified with 10 ml/l acetic acid 30% in order to prevent hydrolysis of the extracted alphacypermethrin (note:there is no difference in hydrolysis in neutral water compared to wash liquor after 10 min. at 30 ºC). Transfer 150 ml of the washing liquid into a separation funnel (500 ml). The extraction procedure is done twice with 100ml ethyl acetate each by shaking twice for 30 seconds. Both portions of ethyl acetate are combined together in a flask (250 ml). Ethyl acetate is evaporated in a rotary evaporator. The flask is stoppered and sent to the analytical laboratory.

b) Procedure done by analytical laboratory

Fill up the flask with THF(50ml). Add 0.5 ml of internal standard solution (dioctyl phthalate, 1% in acetone). Transfer 200 µl of sample out of the flask into a separate GC vial. Add 1 drop of citric acid into the vial.

C.6.1.2.2 Second step: Quantification of the total contents of alphacypermethrin

Weigh (to the nearest 0.1 mg) 1 g of the impregnated net into a volumetric flask (100 ml) and add THF (50 ml). Place this flask in a refluxing apparatus. Heat up the flask in the apparatus and reflux at approximately 90º C (oil bath temperature). Sampling will be done after 5 min. refluxing. Add 0.5 ml of internal standard solution (dioctyl phthalate, 1 % in acetone). Transfer 200 µl of the sample into separate GC vial. Add 1 drop of citric acid into the vial.

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C.6.1.3 Determination via the n-hexane extraction process

C.6.1.3.1 First step: Quantification of the adherent contents of alphacypermethrin on the surface of the fibers

a) Weigh (to the nearest adherent drops of n-hexane) out of the flask into another volumetric flask (100 ml) and add THF (50 ml). Place this flask in a refluxing apparatus. Heat up the flask in the apparatus and reflux at approximately 90º C. Sampling will be done after 5 min refluxing. Add 0.5 ml of internal standard solution (dioctyl phthalate, 1 % in acetone) . Transfer 200 µl of the sample into a separate GC vial. Add I drop of citric acid into the vial.

C.6.1.3.2 Second step:Quantification of the total contents of alpha-cypermethrin

Transfer the net(without adherent drops of n-hexane) out of the flask in to another volumetric flask (100ml) and add THF (5Oml). Place this flask in a refluxing apparatus. Heat up the flask in the apparatus and reflux at approximately 90ºC. Sampling will be done after v5 min refluxing. Add 0.5ml of internal standard solution (dioctyl phthalate, 1% in actone). Transfer 200µl of the same into a separate GC vial. Add 1 drop of citric acid into the vial

C.6.2.3 **System calibration**

As for alphacypermethrin 454/TC/(M3)

C.6.1.4 Determination

Each calibration solution C_i and each sample solution S_J is injected twice. The following sequence is advised:

C_A , $C_{A,,}$ C_B, C_B,C_C, $C_{C,}$ S_A, S_A,S_B, S_B,S_C,$S_{C,...}$

C.6.1.5 Calculation

As for alphacypermethrin 454/TC/(M)3 and see test of linearity

Concentration = $\frac{fxHw}{lqxw}$

Where: f=response factor

Hw=total peak area of alphacypermethrin (cis +II) in the sample solution

Lq=peak area of internal standard in the sample solution

w=mass of sample taken

C.6.1.6 Surface concentration and Release index

a) Determination

The determination of alphacypermethrin content is done via the n-hexane extraction process from fibers

Each calibration solution Cj and each sample solution Sj ai injected twice. The following sequence is advised: CA, CA,CB,CB,CB,CC,CC,SA1,SA1,SA2,SA2,SA3,SA3,SB1,SB1,...

S_A=refers to first rinse; S_A mean value of $S_{A1,}$ $SA_{2,}$$S_{A3}$

S_B=refers to second rinse; S_B mean value of $S_{B1,}$$S_{B2,}$$S_{B3}$

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S_c=refers to third rinse;S_C mean value of $S_{C1,}$ $S_{C2,}S_{C3}$

Where $_{1,2,3}$ are net samples

a) Calculation of the release index

Calculate the mean value of sample solutions S_C and S_B by the equations described in 6.2.5 and the release index for each piece of netting.

Release index=$1-(S_{C}/_{S}S_{B})$, where

S_B =refers to second rinse; S_B mean $S_{B1,}S_{B2,}S_{B3}$

S_C=refers to third rinse; S_C mean value of $S_{C1,}$ $S_{C2,}S_{C3}$

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