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SPECIFICATION FOR CANNED FISH CURRY

SRI LANKA STANDARDS INSTITUTION

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SLS 1106 : 1995

Gr. 12

SRI LANKA STANDARDS INSTITUTION
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Sri Lanka.

Sri Lanka Standards are subject to periodical revision in order to accommodate the progress made by industry. Suggestions for improvement will be recorded and brought to the notice of the Committees to which the revisions are entrusted.

This standard does not purport to include all the necessary provisions of a contract.

Sri Lanka Standard
SPECIFICATION FOR CANNED FISH CURRY

FOREWORD

This standard was approved by the Sectoral Committee on Agriculture and Food Technology - 2 and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 1995-11-23.

During the formulation of this specification due consideration has been given to the relevant provisions made under the Sri Lanka Food Act No. 26 of 1980. Specific requirements given in this specification, wherever applicable, are in accordance with the relevant regulations. However, general provisions made under the Sri Lanka Food Act have not been included in this specification and therefore, the attention of the user of this specification is drawn to the general provisions made in the regulations framed under the Food Act.

Guidelines for the determination of a compliance of a lot with the requirements of this standard based on statistical sampling and inspection are given in Appendix A.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or an analysis, shall be rounded off in accordance with CS 102. The number of significant figures to be retained in the rounded off value shall be the same as that of the specified value in this standard.

In the preparation of this standard, the assistance derived from the following publications is gratefully acknowledged :

(i) SLS 591 Canned Fish
(ii) Official Methods of Analysis of the Association of official Analytical Chemists (AOAC), 1990, 15th edition.

1 SCOPE

This specification prescribes the requirements and methods of test for canned fish curry.

2 REFERENCES

ISO	3972	International Standard determination of sensitivity of taste and detection thresholds
IS	8140	Indian Standard guide for selection of panel for sensory evaluation of foods and beverages
CS	102	Presentation of numerical values
CS	124	Test sieves
SLS	302	Determination of zinc
SLS	311	Determination of lead
SLS	312	Determination of arsenic
SLS	315	Determination of tin
SLS	345	Determination of mercury
SLS	428	Random sampling methods
SLS	467	Labelling of prepackaged foods
SLS	516	Microbiological test methods Part 10 Commercial sterility of canned foods
SLS	591	Canned fish
SLS	614	Potable water
SLS	902	Code of practice for canning of fish
SLS	974	Code of hygienic practice for fresh fish
SLS	975	Code of hygienic practice for frozen fish

3 DEFINITIONS

For the purpose of this specification, the following definitions shall apply :

3.1 canned fish curry : Edible dressed fish (whole fish) or chunks of edible dressed fish processed in a curry and packed in hermetically sealed containers and then processed by heat treatment to preserve it. The product is such the fish as well as the curry could be consumed directly without any further processing.

3.2 container : A can made of lacquered tin plate or aluminium.

3.3 count : The number of units of fish present in the container.

3.4 washed drained mass : The mass of the contents of the container after washing and filtering as prescribed in B.2.1.

3.5 net mass : The gross mass of the unopened container less the mass of the empty container.

4 PRESENTATION

4.1 Form of Pack

4.1.1 *chunks*

A mixture of chunks of dressed fish in which the original muscle structure is retained. Pieces of a chunk may be added if necessary, to fill a container.

4.1.2 *whole fish*

The whole dressed fish shall be filled in the cans in such a manner that they remain in the same direction and there shall be no cross filling.

5 REQUIREMENTS

5.1 Raw materials

The product shall be prepared from clean, wholesome and sound fish, which may be either fresh or frozen.

5.2 Hygiene

The product shall be prepared in accordance with SLS 902, SLS 974 and SLS 975.

5.3 Packing media

The product shall be packed in a curry/gravy. The curry/gravy shall be prepared with salt, seasoning ingredients, spices, tomato, onions, chillies, mustard, garlic, potable water conforming SLS 614 and any other suitable ingredient.

5.4 Final product

5.4.1 The can shall not show any signs of swelling, seam defects, corrosion or other deformations when observed externally. Insignificant corrosion, deformation due to bad handling shall not be considered as visual defects. The interior surface of the can shall not show any signs of deterioration.

5.4.2 The can shall give a negative pressure when punctured. If round cans are used, the vacuum shall be not less than 13.0 Kpa.

NOTE

When determining the vaccum, canned products vaccum gauge, piercing type (approved by British Food Manufacturing Industries Research Association, U.K. or equivalent) may be used.

5.4.3 The contents of the can on opening shall not display any appreciable disintegration. Excessive separation of muscle fibres resulting in a fluffy suspension shall be considered as disintegration. Fish units from which portions have separated out would be treated as disintegrated units. The percentage of such portions separated as units or wholly disintegrated fish units calculated on the basis of the washed drained mass, when tested in accordance with Appendix B shall not exceed 20 per cent based on the average of five cans.

5.4.4 The contents of the can including the curry/gravy on opening shall have the colour, odour and flavour/taste characteristic of typical curry fish.

5.4.5 The product shall be free from any type of poisonous and deleterious substances.

5.4.6 The washed drained mass of the contents of each can shall be not less than 65 per cent by mass of the water capacity of the can when tested in accordance with Appendix B.

5.4.7 The product shall be firm in texture. The bones shall be soft and yielding. It shall be free from pieces of detached or loose skin and scales.

5.4.8 The product shall be free from foreign materials and grit. The product shall be, to the extent possible in good manufacturing practice, free from parasites.

5.4.9 The product shall be assessed for sensory quality characteristics (see 5.4.4, 5.4.7 and 5.4.8) as prescribed in Appendix C.

5.4.10 The product when prepared using Tuna species included in Appendix D.7 of SLS 591 : 1982 should not contain more than 100 mg/kg histamine when determind in accordance with Appendix D.

5.4.11 The product shall satisfy the test for commercial sterility when tested in accordance with Part 10 of SLS 516.

5.4.12 The product shall not exceed the limits for heavy metals given in Table 1 when tested in accordance with Appendix E.

TABLE 1 - Limits for heavy metals

Sl.No. (1)	Heavy metal (2)	Limit, mg/kg (3)
i	Arsenic (as As)	01
ii	Lead (as Pb)	05
iii	Zinc (as Zn)	50
iv	Tin (as Sn)	250
v	Mercury (as Hg)	0.5

6 PACKAGING AND MARKING

6.1 Packaging

The product shall be packed in suitable cans and hermetically sealed. If the cans are lacquered, the lacquer used shall be of such quality that it does not impart any foreign unpleasant taste and/or smell to the contents of the can and does not peel off during processing and storage of the product. The lacquer shall not be soluble in the curry.

Labels or label adhesives or pastes which are hygroscopic and contain acids or mineral salts which are liable to promote rusting of container shall be avoided.

6.2 Marking

6.2.1 The marking and labelling of the containers may be done either by printing or lithographing on the containers themselves or attaching labels printed on paper.

6.2.2 The following shall be marked legibly and indelibly on the container or on the label :

a) Name of the product;
b) Brand name or trade name if any ;
c) Net mass, in grams;
d) Name and address of the manufacturer or distributor;
e) Batch or code number or code marking ;
f) Date of expiry ;
g) Washed drained mass, in grams; and
h) A complete list of ingredients used.

6.2.3 The marking and labelling shall be in accordance with SLS 467.

NOTE

Attention is drawn to certification marking facilities offered by the Sri Lanka Standards Institution. See the inside back cover of this standard.

7 METHODS OF TEST

Tests shall be carried out as prescribed in Part 10 of SLS 516 and Appendices B to E of this specification.

APPENDIX A
COMPLIANCE OF A LOT

The sampling scheme given in this appendix should be applied where compliance of a lot to the requirements of this standard is to be assessed based on statistical sampling and inspection.

Where compliance with this standard is to be assured based on manufacturers' control systems coupled with type testing and check tests or any other procedure, appropriate schemes of sampling and inspection should be adopted.

A.1 LOT

In any consignment all the cans of curry fish of same presentation and belonging to one batch of manufacture or supply shall constitute a lot.

A.2 DEFECTIVE CAN

A can failing to satisfy any one or more of the requirements inspected visually.

A.3 SCALE OF SAMPLING

A.3.1 Samples shall be tested from each lot for ascertaining conformity of material to the requirements of this specification.

A.3.2 The number of cases to be selected from the lot shall be in accordance with Column 2 of Table 2.

TABLE 2 - Scale of sampling

Number of cases in the lot (1)	Number of cases to be selected (2)	Number of cans in the sub sample (3)
Upto 50	5	5
51 to 150	10	8
151 to 1 000	20	15
1 001 and above	30	20

A.3.3 An equal number of cans shall be drawn from each case selected as in A.3.2 to form a sample of 200 cans. If the number of cases in the lot is not sufficient to obtain 200 cans, more cases shall be drawn to get the required number of cans.

A.3.4 The cases and cans shall be selected at random. In order to ensure randomness of selection, random number tables as given in SLS 428 shall be used.

A.4 NUMBER OF TESTS

A.4.1 All the cans of the sample selected as in A.3.3 shall be examined visually for the requirements specified in 5.4.1.

A.4.2 If no defective cans are found on visual examination (A.4.1), eight cans shall be drawn randomly and tested for commercial sterility (5.4.11).

A.4.3 If one or two defective cans are found on visual examination (A.4.1), the whole lot shall be sorted for removal of defective cans. If sorting reveals 1 per cent or less defective cans, 20 of sorted sound cans shall be drawn from the lot and tested for commercial sterility (5.4.11).

A.4.4 If the cans tested as in A.4.2 or A.4.3 satisfy the given requirement, a sub sample of sound cans of size as given in Column 3 of Table 2 shall be drawn from the sample selected as in A.3.3.

A.4.4.1 Each can of the sub sample shall be examined for labelling and marking requirements.

A.4.4.2 Tests for requirement 5.4.2 and 5.4.5 shall be conducted on each can in the sub sample. Having passed these tests each can shall be tested for requirements 5.4.6, 5.4.3 and 5.4.9

A.4.4.3 After testing as in A.4.4.2 the contents of all the cans shall be mixed together to form a composite sample. The composite sample so formed shall be tested for limits specified in Table 1.

A.5 CRITERIA FOR CONFORMITY

A lot shall be declared as conforming to the requirements of this specification if the following conditions are satisfied:

A.5.1 From the sample of cans examined visually as in A.4.1

a) No defective cans are found in the sample of 200 cans; or
b) One or two defective cans are found in the sample of 200 cans and 1 per cent or less defective cans are found in the lot on sorting.

NOTES

1. If 3 or more defective cans are found, the lot shall be rejected.

2. If sorting reveals more than 1 per cent defective cans, the lot shall be rejected.

A.5.2 Each can tested for commercial sterility as in A.4.2 or A.4.3 as the case may be, satisfies the relevant requirement.

A.5.3 Each can examined as in A.4.4.1 satisfies the labelling and marking requirements.

A.5.4 Each can tested as in A.4.4.2 satisfies the relevant requirements.

A.5.5 The test results on the composite sample tested as in A.4.4.3 comply with relevant limits.

APPENDIX B
DETERMINATION OF WASHED DRAINED MASS

B.1 APPARATUS

Sieve, of aperture 2.00-mm, conforming to CS 124.

B.2 PROCEDURE

B.2.1 Carefully weigh, the clean and dry sieve and transfer the contents of the can to the sieve. Wash first the covering curry/gravy and then the full contents with hot tap water (approximately 40 ^{0}C), using a wash bottle on the tared circular sieve.

Wash the contents of the sieve with hot water until free of adhering curry/gravy. Where necessary separate optional ingredients (spices, vegetables, fruits) with pincers. Allow to drain for 5 minutes, measured from the time the washing procedure has finished. The difference between the two masses is the washed drained mass. Calculate the washed drained mass as a percentage of the water capacity of the can (See B.2.2). Retain the residue on the sieve as well as the drained liquid.

B.2.2 Determine the water capacity of the can by the procedure given in B.2.3 to B.2.6.

B.2.3 Cut out the lid without removing or altering the height of the double seam.

B.2.4 Wash the can with distilled water, dry and weigh the empty can.

B.2.5 Fill the container with distilled water to 4 mm vertical distance below the top level of the container and weigh.

B.2.6 Substract the mass in B.2.4 from the mass in B.2.5. The difference shall be considered to be the mass of water required to fill the container.

APPENDIX C
EVALUATION OF SENSORY QUALITY CHARACTERISTICS

C.1 GENERAL

The basic purpose of this evaluation is to ensure a certain degree of consumer acceptability and satisfaction in respect of sensory quality of the product while conforming to other requirements stipulated in this specification.

The assessors for the sensory evaluation panel should be selected after screening tests. It is important that the selected assessors undergo familiarization training on evaluation of canned fish curry in accordance with this scheme. During the familiarization training it is necessary that the assessors identify and differentiate each sensory attribute and use the appropriate description provided in these guidelines, when scoring. It is also necessary that the assessors are exposed to a wide range of qualities of canned fish curry so that they are familiar with full range of the scales for different sensory attributes. During evaluation sessions it is recommended that not more than six cans presented to an assessor, at a time.

It is recommended that decision for acceptance/rejection be based on the assessment of at least three (03) assessors.

C.2 OUTLINE OF THE PROCEDURE

C.2.1 Each can is assessed for different sensory attributes (appearance, odour, flavour, texture) using the scoring system provided in C.5.

C.2.2 A weighted score for the overall sensory quality is then calculated for each can with maximum score of 20.

C.2.3 The average weighted score for each can is calculated based on the scores given by the assessors. Each can is attributed as non-defective/defective based on this score.

C.2.4 Decision on rejection or acceptance of the sample is taken based on the number of defective/non-defective units in the sample.

C.3 GENERAL TEST CONDITIONS

C.3.1 Test room

The test should be carried out in a room suitably arranged for sensory evaluation. The testing area should be free from foreign and undesirable odours. The test room should have adequate light.

C.3.2 **Assessors**

The assessors should be selected after a screening test. The assessors should be familiar with the product and trained prior to the test, to evaluate canned fish curry according to these guidelines. Guidelines for selection of assessors are given in ISO 3972 and IS 8140.

C.4 **PROCEDURE**

C.4.1 **Materials required**

C.4.1.1 *Can* opener

C.4.1.2 *White curry dishes (diameter of about 100 mm)*

C.4.1.3 *Stainless steel spoons*

C.4.1.4 *Disposable containers*

C.4.1.5 *Score sheet*

C.4.2 **Preparation of sample**

C.4.2.1 Code number each can in the sample.

C.4.2.2 Arrange the cans with the code numbers leaving sufficient space in between. White dishes with respective code numbers should be placed parallel to each can.

C.4.2.3 Open the cans using a can opener, transfer the contents with the curry carefully into the coded dishes. Care should be taken when transferring the contents, so that the fish units will not be disintegrated. Carefully spread the fish units without damaging. A spoon should be provided for each dish.

C.4.3 **Evaluation of sample**

C.4.3.1 Each can should be evaluated for the following sensory attributes in the order indicated using the scales given in C.5.

a) Appearance (curry/gravy, overall colour, physical defects)
b) Odour
c) Flavour (combination of taste and odour of the fish and curry/gravy)
d) Texture

Each assessor should independently assign points for each sensory attribute using a scale of 0-5.

C.4.3.2 When assessing for appearance, observations on presentation form of pack, curry/gravy should be made.

C.4.3.3 When assessing for odour the following points should be taken into consideration.

a) Evaluate the odour within 1 minute to 5 minutes after opening the can.
b) Both the curry / gravy and fish units should be taken into consideration.
c) From each can select fish units in the ratio of 1:3. Take pieces from these selected fish units. Using fingers gently crush and smell the fish. Report the score based on the overall assessment.
d) If fish units tested are found to have an objectionable odour, test the other units also for the overall assessment.

C.4.3.4 When assessing for flavour, the following points should be taken into consideration.

a) The flavour is the total perception of both taste and odour when the food is in the mouth.
b) From each can select fish units in the ratio of 1:3.
c) Take a representative portion of the fish into mouth, bite well and assess its flavour. Also taste the curry/gravy. Report the score based on the overall assessment.

C.4.3.5 When assessing for texture, the following points should be taken into consideration.

a) From each can select fish units in the ratio of 1:3.
b) Take fish unit on a separate plate. In the case of whole fish, hold the ventral side upward and using the spoon separate the fish unit into two fillets. Observe the texture as it appears.
c) Break a piece from one fillet. Using fingers gently press and feel for its texture. Observe the texture by crushing.
d) Look for any bones present in the unit, press them and feel for the texture.
e) Report the score based on the overall assessment.
f) If fish units tested are found to have an unsatisfactory textures test the other units also for the overall assessment.

C.5 SCALES FOR ASSESSMENT

C.5.1 Appearance

The overall colour of the fish curry/gravy and characteristic shape of the fish/units, defects should be considered. Physical defects include the presence of scales, hard scutes, bones, bruises, fins, viscera, parts of head, foreign material.

Description	Score
Thick, red colour gravy with vegetable seasoning. Characteristic metallic grey or silvery grey of fish skin (in whole fish). Free from scales, hard scutes, bones, parts of head, viscera,foreign material (in whole fish). Characteristic brownish colour of fish meat with original muscle structure (in fish units). Free from scales, hard scutes	5
Practically free* from scales, hard scutes, bones, parts of head, fins, viscera, foreign material. Characteristic red colour, watery gravy with less vegetable seasongings.	4
Reasonable amount** of scales, hard scutes, bones parts of head, fins, viscera, foreign material.	3
Considerable amount*** of scales, hard scutes, broken and cracked fish, parts of head, fins, viscera. Absence of characteristic red colour in the gravy.	2 1
Severe discoloration, excessive amount of scales, hard scutes, bones, bruises, broken and cracked fish, parts of head, fins, viscera, foreign material. Absence of vegetable seasonigs.Pieces look very dry.	0

** Present in negligible amounts which may not be noticed by average consumer.*

*** May be noticeable but not offensive to the majority of consumers.*

**** Present to an extent which will be offensive to the majority of consumers.*

C.5.2 Odour

The overall curry/gravy and whole fish and fish units should be considerded.

Description	Score
Fresh, mild, characteristic smell of good quality fish curry. Desirable appetizing odours.	5
Slightly pronounced or strong smell of fish curry Generally less appetizing.	4
Lack of fish curry smell or slight* acidic and/or other off odours, or slight scorched or caramelized odours.	3
Slight** but objectionable odours ie. metallic, rancid, putrid, ammoniacal, sulfide, musty or any other foreign odours.	2 1
Distinct objectionable odours, metallic, rancid, putrid, very strong dried fish odours.	0

** May not be noticed by majority of consumers, even if noticed may not be offensive.*

*** Noticeable and may offend the consumer.*

C.5.3 Flavour

Description	Score
Fresh characteristic flavour of good quality curry fish which are desirable. Spicy and salty	5
Definite loss of flavour but not off flavours, Lack of spicyness, saltines	4
Absolutely no flavour or slight* off flavours, metallic, acidic, woody, grasssy flavours, typical burnt flavours. Flat, bland.	3
Objectionable metalllic, rusty, rancid, bitter or rubberlike flavours or slight putrid ad sulfide flavours or distinct foreign flavours	2 1
Inedible	0

** Not offensive to the majority of consumers.*

C.5.4 Texture

The thickness of the curry/gravy and texture of fish units should be considered.

Description	Score
Flesh well-bound, intact, firm. Free from mushiness. Bones soft and yielding.Thick, viscous	5
Lack of firmness in the flesh. Bones are not too soft and yielding. Thickness of the curry is less	4
Flesh loosely bound, slightly* soft and mushy. Hard bones (not easily friable using thumb and forefinger). Watery, dilute.	3
Flesh crumbly, pasty or pulpy**	2 1
Excessively mushy, crumbly, pulpy	0

** Not offensive to the majority if the consumers.*

*** To an extent that it is offensive to the consumer.*

C.6 CALCULATION AND INTERPRITATION OF RESULTS

C.6.1 Calculate the weighted sensory scores and total weighted score, using the raw scores assigned by each assessor, for each can as indicated in Table 3.

TABLE 3 - Calculation of the weighted score and total weighted score for different sensory attributes

Sl. No. (1)	Characteristic (2)	Raw score assigned on a scale of 0-5 (3)	Weighted score obtained (4)
i)	Appearance	A	A x 0.8
ii)	Odour	B	B x 1.2
iii)	Flavour	C	C x 1.2
iv)	Texture	D	D x 0.8
Total weighted sensory score			(A x 0.8)+(Bx1.2)+(C x 1.2)+(DxO.8)

C.6.2 Using the total weighted scores (S_1, S_2, S_3, etc.) calculate the average weighted sensory score ($\bar{S}$), for each can.

C.6.3 Attribute each can as non-defective or defective based on the average weighted sensory score ($\bar{S}$).

$\bar{s} > 12$ ——> non-defective
$\bar{s} < 12$ ——> defective

Report the total number of non-defective and defective cans in the sample.

C.6.4 Accept or reject the sample of cans based on the acceptance numbers specified in the sampling plan given in Table 4.

C.7 SCALE OF SAMPLING AND CRITERIA

The following scale of sampling is recommended to be used in the evaluation of sensory quality characteristics in the specification of canned fish curry.

C.7.1 Number of cans to be selected from a lot should be in accordance with Table 4.

TABLE 4 – Acceptance and rejection limits

Number of cases in the lot (1)	Number of cans to be selected (2)	Cumulative sample size (3)	Acceptance number (4)	Rejection number (5)
Up to 50	1st stage sample 5	5	0	2
	2nd stage sample 5	10	1	2
51 to 150	1st stage sample 8	8	0	3
	2nd stage sample 8	16	3	4
151 to 1000	1st stage sample 15	15	0	4
	2nd stage sample 25	40	4	5
1001 and above	1st stage sample 20	20	1	5
	2nd stage sample 40	60	6	7

C.7.2 A sample of cans as given in first stage sample of Column 2 of Table 4 should be selected randomly and are examined by assessors.

C.7.3 If the number of defective cans in first stage sample is between corresponding acceptance number and rejection number as given in Column 4 and Column 5, a sample of cans as given in second stage sample of Column 2 of Table 4 should be selected and examined by assessors.

C.7.4 A lot should be considered as conforming to the sensory characteristics if the following condition is satisfied:

a) The number of defective cans in first stage sample when examined as in C.7.2 is less than or equal to the corresponding acceptance number given in Column 4 of Table 4; or
b) The number of defective cans in cumulative sample (number of defective cans in first stage sample and second stage sample) is less than or equal to the corresponding acceptance number given in Column 4 of Table 4.

APPENDIX D
DETERMINATION OF HISTAMINE CONTENT

Rinse all plastic and glass containers with 25 per cent (V/V) hydrochloric acid and water before use.

D.1 APPARATUS

D.1.1 **Chromatographic tube**, length 200-mm, inner diameter 7-mm, polypropylene tube, flow rate controlled at more than 3 ml/minute.

D.1.2 **Photofluorometer**, with excitation at 350 nm and measuring emission at 444 nm.

D.2 REAGENTS

D.2.1 **Ion exchange resin**, Bio-Rad AG 1-x8, 50 - 100 mesh or Dowex 1-x8, 50 - 100 mesh.

Add 15 ml of 2 mol/l NaOH per 1 g resin, to a beaker to convert to OH form. Swirl the mixture and allow to stand for less than 30 minutes. Decant the liquid and repeat with additional base. Wash the resin thoroly with water, slurry into fluted paper (S & S No. 588, or equivalant) and wash again with water. Prepare resin fresh weekly and store under water.

Place glass wool plug in base of tube (D.1.1), and slurry in enough resin to form 8 cm bed. Maintain water level above to of resin bed at all times. Wash column with 10 ml of water before applying each extract.

NOTE

Do not regenerate resin in packed column; rather, use batch regeneration in beaker when necessary.

D.2.2 Phosphoric acid, standardized, c (H_3PO_4) = 1.78 mol/l

Dilute 121.8 ml of 85 per cent (V/V) Phosphoric acid to one litre. Standardize 5.00 ml by titrating with 1.00 mol/l sodium hydroxide using phenolphalin as the indicator.

D.2.3 Ortho - Phthalic dicarboxaldehyde (OPT) solution, 0.1 per cent

Dissolve 100 mg OPT in 100 ml methyl hydroxide. Store in amber bottle in a refrigerator. Prepare fresh weekly.

D.2.4 Histamine standard solutions

D.2.4.1 Stock solution, 1 mg/ml

Weigh accurately about 169.1 mg histamine.2HCl into a 100-ml volumetric flask. Dissolve and dilute to volume with 0.1 mol/l hydrochloric acid. Prepare fresh weekly and store in refrigerator.

D.2.4.2 Intermediate solution, 10 µg/ml

Pipette 1 ml of stock solution (D.2.4.1) into 100-ml volumetric flask. Dilute to volume with 0.1 mol/l hydrochloric acid. Prepare freshly weekly and store in refrigerator.

D.2.4.3 Working solutions, 0.5 µg/5 ml, 1.0 µg/5 ml and 1.5 µg/5 ml

Pipette 1 ml, 2 ml and 3 ml of intermidiate solution into 100 ml volumetric flasks separately. Dilute to volume with 0.1 mol/l hydrochloric acid. Prepare fresh daily and store in refrigerator.

D.3 PROCEDURE

D.3.1 Preparation of standard curve

Pipette in duplicate 5 ml aliquots of working standard solution (D.2.4.3) into 50-ml glass or polypropylene Erlenmeyer flasks. Pipette in 10 ml of 0.1 mol/l hydrochloric acid to each flask and mix. Pipette in 3 ml of 1 mol/l sodium hydroxide and mix. Within 5 minutes, pipette in 1 ml of OPT solution (D.2.3) and mix immediately. After exactly 4 minutes, pipette in 3 ml of phosphoric acid (D.2.2) and mix immediately.

NOTE

It is important to mix thoroughly after each addition and at least once during OPT reaction. Run 6 to 10 OPT reactions simultaneously by adding reagents to Erlenmeyers in set order.

Prepare blank by substituting 5 ml of 0.1 mol/l hydrochloric acid for histamine solution. Within 90 minutes, record fluorescence intensity (I) of working standard solution with water in reference cell, using extinction warelength of 350 nm and emission wavelength of 444 nm.

Plot I (corrected for blank) against µg histamine/5 ml aliquot.

D.3.2 Determination

D.3.2.1 Transfer 10 g of the prepared sample to semimicro container of a high speed blender, add about 50 ml methyl alcohol. Blend for 2 minutes. Transfer to a 100 - ml glass-stoppered volumetric flask, rinsing lid and blender jar with methyl alcohol and adding rinsings to the flask.

Heat in a water bath at 60 °C for 15 minutes. Cool to 25 °C, . Dilute to volume with methyl alcohol. Filter through a folded paper.

D.3.2.2 Pass 4 ml to 5 ml of water through the column (D.1.1) and discard eluate. Pipette 1 ml of extract (D.3.2.1) onto column and add 4 ml to 5 ml of water. Immediately initiate column flow into a 50-ml volumetric flask containing 5.00 ml of 1.00 mol/l hydrochloric acid. When liquid level is approximately 2 mm above the resin, add about 5 ml of water and elute. Follow with water in larger portions until about 35 ml has eluted. Stop column flow. Dilute to volume with water. Stopper and mix. Refrigerate the elute.

Pipette 5 ml of the eluate into 50 ml erlenmeyer flasks. Pipette in 10 ml of 0.1 mol/l hydrochloric acid. Pipette in 3 ml of 1 mol/l sodium hydroxide and mix. Proceed as in D.3.1.

D.3.2.3 If the sample contains more than 15 mg histamine per 100 g of fish, pipette 1 ml of sample - OPT mixture into 10-ml beaker containing exactly 2 ml of blank - OPT mixture. Mix thoroughly. Read fluorescence of new solution. Dilute and mix aliquots with blank - OPT mixture as needed to obtain a measurable reading.

NOTE

This approximation indicated proper dilution of elute required prior to second OPT reaction needed for reliable quantitation of sample. Alternatively, use sensitivity range control of fluorometer, if available, to estimate dilution. Use these approximations to prepare appropriate dilutions of aliquot of elute with 0.1 mol/l hydrochloric acid, proceeding as in D.3.1 commencing with the addition of 3 ml of 1 mol/l sodium hydroxide.

D.4 CALCULATION

Plot of (I) against µg histamine/5 ml of solution should be a straight line passing through the origin with a slope
$m = [(I_a/1.5) + I_b + 2I_c]/3$

D.4.1 Histamine content
mg/100 g of fish $= (10)(F)\ (1/m)(I_s)$

where

I_s is the fluorescence of the sample
I_a is the fluorescence of the 1.5 µg histamine standard solution;
I_b is the fluorescence of the 1.0 µg histamine standard solution;
I_c is the fluorescence of the 0.5 µg histamine standard solution; and
F is the dilution factor

$$F = \frac{\text{eluate (ml)} + \text{hydrochloric acid (ml)}}{\text{eluate (ml)}}$$

F = 1 for undiluted eluate

D.4.2 If calibration plot is not linear, use the standard curve directly for quantitation. Each subdivision of abscissa should be 0.1 µg histamine /5 ml solution.

Histamine content
mg/100 g of fish $= (10)(F)(W)$

where,

F is the dilution factor, and
W is the µg of histamine /5 ml solution as determined from the curve.

APPENDIX E
DETERMINATION OF HEAVY METALS

E.1 DETERMINATION OF ARSENIC

E.1.1 Reagents and apparatus

E.1.1.1 Nitric acid, rel. den = 1.38

E.1.1.2 Sulfuric acid, rel. den. = 1.84

E.1.1.3 Ammonium oxalate solution, aquous, saturated.

E.1.1.4 Volumetric flask, of capacity 100-ml or 50-ml.

E.1.2 Procedure

E.1.2.1 Mix the solid and liquid portions of the sample and macerate using a mechanical grinder.

E.1.2.2 Place a suitable quantity (usually 5 g to 10 g) of the prepared sample (E.1.2.1) into a Kjeldahl flask. Add 20 ml of nitric acid (E.1.1.1) and upto 20 ml of water (depending on the water content of the sample). Boil so that the volume is reduced to about to 20 ml. Cool and add 10 ml of sulfuric acid (E.1.1.2). Boil again and add futher small quantities of nitric acid until the liquid turns brown. Continue heating until the nitrous vapours are eliminated and there is a concentration of white fumes above the decomposition liquid. Cool and add 10 ml of ammonium oxalate (E.1.1.3). Boil until copious white fumes are again produced. The liquid should be colourless or pale yellow. Cool and transfer quantitatively into a volumetric flask (E.1.1.4). Dilute to mark with water.

E.1.2.3 Proceed as given in SLS 312.

E.2 DETERMINATION OF LEAD

E.2.1 Carry out the same procedure for preparation of the sample as in E.1.2.2 with the following alterations:

a) The quantity of prepared sample used should contain more than 40 ug of lead.
b) The volume of sulfuric acid used should be not more than 5 ml.
c) The contants should be transfered to a 50-ml volumetric flask.

E.2.2 Proceed as given in SLS 311.

E.3 DETERMINATION OF ZNIC

E.3.1 Carry out the same procedure for preparation of the sample as in E.1.2.2. Dry ashing of the sample at 550 °C or ignition of the sample at 450 °C overnight may also be used for preparation of the sample.

E.3.2 Proceed as in SLS 302.

E.4 DETERMINATION OF TIN

E.4.1 Reagents

E.4.1.1 Nitric acid, rel. den. = 1.42

E.4.1.2 Hydrocholric acid, rel. den. = 1.19

E.4.1.3 Potassium chloride, 10 mg/ml solution

Dissolve 1.91 g of potassium chloride in water and dilute to 100 ml.

E.4.2 Preparation of the sample

Mix the solid and liquid portions of the sample and mascerate using a mechanical grinder. Weigh, to the nearest 0.001 g, about 5 g to 10 g of the prepared sample into an Erlenmeyer flask. Dry in an oven at 120 ^{0}C. Add 30 ml of nitric acid (E.4.1.1) and within 15 minutes heat gently in a fumehood to initiate digestion, avoiding excessive frothing. (Do not add nitric acid to sample unless there is time to complete this stage of digestion in the same day.) Gently boil until 3 ml to 6 ml of the digest remains or until the sample just begins to dry on bottom. Do not allow the sample to char. Remove the flask from heat. Without delay, add 25 ml of hydrochloric acid (E.4.1.2) and heat gently for about 15 minutes until bumping from evolution of chlorine stops. Increase heat and boil until 10 ml to 15 ml remains. Using a similar flask with 15 ml of water estimate the volume. Add about 40 ml of water. Swirl and pour into a 100-ml volumetric flask rinsing once with about 10 ml of water. Pippet 1.0 ml of potassium chloride (E.4.1.3) into a volumetric flask. Cool and dilute to volume with water. Add additional water to compensate for volume of fat, if any, in the flask. Mix well. Filter 30 ml to 50 ml through a dry, medium porosity filter paper into a dry polypropylene or polyethylene screw-cap bottle.

E.4.3 Proceed as given in SLS 315.

E.5 DETERMINATION OF MERCURY

E.5.1 Apparatus

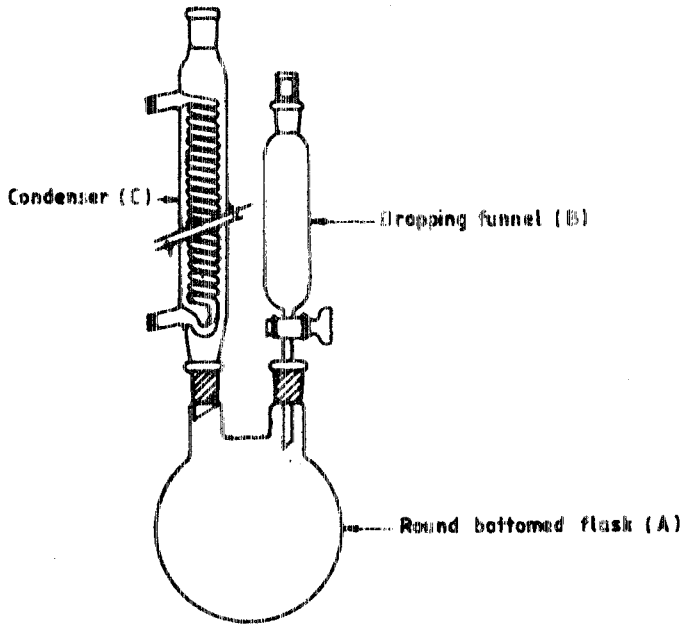

Fig. 1 - Decomposition apparatus

E.5.2 Reagents

E.5.2.1 Nitric acid, rel. den. = 1.42

E.5.2.2 Sulfuric acid, rel. den. = 1.84

E.5.2.3 Urea, 400 g/l solution

E.5.2.4 Nitric acid, 5 per cent (V/V) solution

E.5.3 Preparation of the sample

Mix the solid and liquid portions of the sample and macerate using a mechanical grinder. Weigh, to the nearest 0.01 g, about 5 g of the prepared sample to the round bottom flask A of the decomposition apparatus. Add 5 ml to 10 ml of water.

Place few glass beads in the flask A, and connect to the apparatus (E.5.1). By means of the dropping funnel B add drop by drop, 5 ml of nitric acid (E.5.2.1). Start a fast flow of water through the condenser C and heat the flask with a low flame.

Allow the reaction to proceed very gently so as to avoid any loss of mercury. Continue decomposition under reflux for about 30 minutes until the liquid has a uniform appearance. If the mixture turns brown add several drops of nitric acid (E.5.2.1) through the dropping funnel B until the colour is discharged. Allow to cool.

Carefully add 10 ml of a mixture of equal parts of nitric acid (E.5.2.1) and sulfuric acid (E.5.2.2). Heat with a low flame. Add nitric acid (E.5.2.1) drop by drop if the digest turns brown. Continue heating until nitrous vapours are eliminated and there is a concentration of white fumes above the decomposition liquid.

Control heating so that the white fumes do not rise more than half way up the condenser C . The liquid should be colourless or pale yellow. Allow to cool. Add 5 ml of urea solution (E.5.2.3) through the side neck and boil under reflux for 30 minutes. Allow to cool.

Disconnect the apparatus and transfer the contents of the flask A into a 100-ml volumetric flask. Rince the condencer C twice with 15 ml to 20 ml of nitric acid (E.5.2.4) collecting the rinsings in the flask A and transfering them to the volumetric flask. Carefully rinse the device twice with 10 ml to 20 ml of water and add the rinsings to the volumetric flask. Dilute to mark with water.

E.5.4 Proceed as given in SLS 345.

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