SRI LANKA STANDARD 845 : 1989

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SPECIFICATION FOR

GELATINE (FOOD GRADE)

SRI LANKA STANDARDS INSTITUTION

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SLS 845:1989
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Gr. 12

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

SRI LANKA STANDARD

SPECIFICATION FOR GELATINE (FOOD GRADE)

FOREWORD

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1989-05-12, after the draft, finalized by the Drafting Committee on Food Additives, had been approved by the Agricultural and Food Products Divisional Committee.

This specification is subject to the restrictions imposed under the Food Act No. 26 of 1980 and the regulations framed thereunder.

All standard values used throughout this specification are given in SI units.

For the purpose of deciding whether a particular requirement of this specification 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 places retained in the rounded off value shall be the same as that of the specified value in this specification.

In the preparation of this specification the assistance obtained from the publications of the Bureau of Indian Standards and the British Standards Institution is gratefully acknowledged.

1 SCOPE

This specification prescribes the requirements and methods of sampling and test for gelatine (food grade) which is also known as edible gelatine.

2 REFERENCES

CS 102 Presentation of numerical values.
SLS 311 Method for determination of lead.
SLS 312 Method for determination of arsenic.
SLS 428 Random sampling methods.
SLS 516 Microbiological test methods
Part 1 : Aerobic plate count.
Part 3 : Detection and enumeration of coliforms, faecal coliforms and *E.Coli*
Part 5 : Detection of *Salmonella*.

3 DEFINITION

For the purpose of this specification the follcwing definition shall apply:

3.1 **gelatine** : A purified product obtained by partial hydrolysis of collagen, derived from the skin, white connective tissues and bones.

4 REQUIREMENTS

4.1 General

4.1.1 Gelatine shall be pale yellcwish, translucent and in the form of sheets, flakes, shreds or coarse to fine powder. It shall be stable in air when dry.

4.1.2 It shall have a characteristic odour.

4.1.3 It shall not contain any added colouring matter.

4.1.4 It shall be free from dirt and/or any other extraneous matter.

4.2 Solubility

4.2.1 It shall be practically insoluble in cold water but, shall swell and soften when immersed in it. It shall gradually absorb 5 to 10 times its own mass of water. It shall be soluble in hot water; mixture of hot water and glycerin; and in acetic acid (approximately 5 mol/l).

4.2.2 It shall be practically insoluble in alcohol (95 per cent V/V), chloroform, diethyl ether and volatile oils.

4.3 Transparency

A two per cent (m/m) solution in hot water maintained at a temperature of 0°C for six hours shall form a transparent or translucent jelly.

4.4 Gel strength

The material shall satisfy the requirements of the test prescribed in Appendix A.

4.5 Chemical requirements

Gelatine shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Column 4 of the table.

TABLE 1 -Chemical requirements for gelatine

Sl. No. (1)	Characteristic (2)	Requirement (3)	Method of test (4)
i)	Moisture, per cent by mass, max.	15.0	Appendix B
ii)	Total ash, per cent by mass, max.	3.0	Appendix C
iii)	Sulfur dioxide, mg/kg, max.	1000	Appendix D
iv)	Nitrogen (on dry basis), per cent by mass, min.	15.0	Appendix E
v)	pH value, at 27 ± 2 °C.	4.0 to 6.3	Appendix F

4.6 Microbiological limits

The material shall conform to the limits given in Table 2 when tested in accordance with the methods prescribed in Column 4 of the table.

TABLE 2 - Microbiological limits

Sl. No. (1)	Test organism (2)	Limit (3)	Method of test (4)
i)	Aerobic plate count	Not more than 5000 per g	)
ii)	*E.coli*	Absent in 1 g	) Appendix G
iii)	*Salmonella*	Absent in 25 g	)

4.7 Trace metal limits

The material shall conform to the limits given in Table 3 when tested in accordance with the methods prescribed in Column 4 of the table.

TABLE 3 -Trace metal limits

Sl. No. (1)	Characteristic (2)	Limit (3)	Method of test (4)
i)	Zinc, mg/kg, max.	100	Appendix H
ii)	Arsenic, mg/kg, max.	2	Appendix J
iii)	Lead, mg/kg, max.	5	Appendix K
iv)	Copper, mg/kg, max.	30	Appendix L

5 PACKAGING AND MARKING

5.1 Packaging

5.1.1 *Bulk packaging*

Gelatine shall be hygienically packed in suitable drums or bags which shall withstand breakages during handling and transport.

5.1.2 *Retail packaging*

Gelatine shall be hygienically packed in polyethylene or other suitable moisture proof packing material. These packages may be enclosed in a suitable container.

5.2 Marking

5.2.1 *Bulk packages*

Marking on drums or bags shall be as agreed to between the purchaser and the supplier.

5.2.2 *Retail packages*

Each package or container shall be legibly and indelibly marked or labelled with the following

a) Name of the product including the words `Food grade' or `Edible';
b) Brand name or trade mark, if any;
c) Net mass, in grams;
d) Name and address of the manufacturer and distributor (including the country of origin);
e) Batch or code number;
f) Date of minimum durability, indicated by the words `BEST BEFORE' ; and
g) Instructions for storage.

6 SAMPLING

6.1 Lot

All containers containing edible gelatine of one batch of manufacture or supply shall constitute a lot.

6.2 General requirements of sampling

6.2.1 The sample shall be collected from unopened and undamaged containers.

6.2.2 The samples for microbiological analysis shall be drawn first.

6.2.3 Precautions shall be taken to protect samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

6.2.4 The sampling instrument shall be clean and dry when used. When taking samples for microbiological examination the sampling instrument shall be sterilized.

6.2.5 The samples shall be placed in clean, dry and moisture proof containers. The samples for microbiological examination shall be kept in sterilized containers.

6.2.6 The sample containers shall be sealed air tight, after filling and marked with necessary details of sampling.

6.2.7 The samples shall be stored in such a manner that there will be no deterioration of quality of the material.

6.3 Scale of sampling

6.3.1 The samples shall be tested from each lot for ascertaining its conformity to the requirements of this specification.

6.3.2 *Sampling from bulk containers*
The number of bulk containers to be selected from a lot shall be in accordance with Table 4.

TABLE 4 - Scale of sampling for bulk containers

Number of bulk containers in the lot (1)	Number of bulk containers to be selected (2)
Up to 50	2
51 to 100	3
101 to 150	4
151 and above	5

6.3.3 *Sampling from retail containers*

6.3.3.1 The number of retail containers to be selected from a lot shall be in accordance with Table 5.

TABLE 5 - Scale of sampling retail containers

Number of retail containers in the lot (1)	Number of retail containers to be selected (2)
Up to 300	10
301 to 500	12
501 to 1 000	14
1 001 to 3 000	16
3 001 to 10 000	20
10 001 and above	25

6.3.3.2 If containers are packed in cartons, 10 per cent of the cartons subject to a minimum of 5, shall be selected first. Approximately an equal number of retail containers shall be taken from each carton selected, to get a sample of size as given in Table 5.

6.3.4 The containers shall be selected at random. In order to ensure randomness of selection random tables as given in SLS 428 shall be used.

6.4 Preparation of samples

6.4.1 *From bulk containers*

6.4.1.1 Samples for microbiological examination

Approximately equal quantities of material shall be taken from top, middle and bottom portions of each container selected as in 6.3.2 using an appropriate sampling instrument. The material thus obtained from each container shall be transferred to separate sample containers. The minimum size of a sample shall be 100 g.

6.4.1.2 Individual samples

Approximately equal quantities of material shall be taken from top, middle and bottom portions of each container selected as in 6.3.2 using an appropriate sampling instrument. The material taken from each container shall be transferred to separate sample containers. The minimum size of a sample shall be 50 g.

6.4.1.3 Preparation of composite sample

Approximately equal quantities of material shall be taken from top, middle and bottom portions of each container selected as in 6.3.2 using an appropriate sampling instrument, mixed together and reduced to get a composite sample of 250 g. The composite sample shall be transferred to a sample container.

NOTE - In case of sampling from bulk containers, samples shall be prepared at the place of inspection.

6.4.2 *From retail containers*

6.4.2.1 Samples for microbiological examination

A sufficient number of containers, subject to a minimum of 3 containers, shall be selected from the containers selected as in 6.3.3 to get a sample of 100 g.

6.4.2.2 Individual samples

Approximately equal quantities of the material shall be taken from each container selected as in 6.3.3 after drawing the samples for microbiological testing. The material taken from each container shall be transferred to separate sample containers. The minimum size of a sample shall be 2 g.

6.4.2.3 Preparation of composite sample

The remaining material of the containers after preparing individual samples shall be mixed together to form a composite sample of 250 g. If the material is not sufficient to form a composite sample of 250 g, a sufficient number of containers shall be taken from the lot to form a composite sample of 250 g.

6.5 **Number of tests**

6.5.1 Each container selected as in 6.3.2 shall be inspected for packaging and marking requirements.

6.5.2 The sample prepared as in 6.4.1.1 shall be tested for requirements given in 4.6. In the case of retail containers each container selected as in 6.4.2.1 shall be tested for requirements given in 4.6.

6.5.3 The individual samples prepared as in 6.4.1.2 and 6.4.2.2 shall be tested for requirements given in 4.1.

6.5.4 The composite samples prepared as in 6.4.1.3 and 6.4.2.3 shall be tested for requirements given in 4.2, 4.3, 4.4, 4.5 and 4.7.

7 METHODS OF TEST

Tests shall be carried out as prescribed in the appropriate appendices of this specification.

8 CRITERIA FOR CONFORMITY

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

8.1 Each container inspected as in 6.5.1 satisfies the relevant requirements.

8.2 The sample for microbiological testing when tested as in 6.5.2 satisfies the relevant requirements.

8.3 Each individual sample when tested as in 6.5.3 satisfies the relevant requirements.

8.4 The composite sample when tested as in 6.5.4 satisfies the relevant requirements.

APPENDIX A
DETERMINATION OF GEL STRENGTH

A.1 PROCEDURE

Weigh, to the nearest 0.01 g, about 1 g of the sample and transfer with 99 ml of water in to a 200-ml flask. Allow to stand for 15 minutes, and place the flask in a water bath at 60 °C. Swirl occasionally until it dissolves completely. Transfer 10 ml of the solution to a test tube having an internal diameter of 12-mm and place the tube in an ice bath. The level of the solution shall be below the level of the ice and water. Place the bath containing the tube in a refrigerator, and maintain it at 0°C for 6 hours. No movement of gel shall be observed when the tube is removed from the bath and inverted.

APPENDIX B
DETERMINATION OF MOISTURE

B.1 APPARATUS

B.1.1 *Metal dish with a lid*, of about 70-mm in diameter, about 15-mm in height and about 25 g in mass.

B.1.2 *Desiccator*

B.2 PROCEDURE

Weigh, to the nearest milligram, about 1 g of the sample (do not powder sheet gelatine while preparing the sample) in the metal dish (B.1.1). Add 10 ml of water and allow to soak. Heat on a water bath to form a homogeneous solution. Continue heating until most of the water has evaporated. Dry at 105 $\pm$ 5 °C for 2 hours. Cool in a desiccator and weigh. Repeat the process of drying, cooling and weighing at 30 minute intervals until the difference between two successive weighings does not exceed 1 mg.

B.3 CALCULATION

$$\text{Moisture, per cent by mass} = \frac{m_1 - m_2}{m_1 - m_0} \times 100$$

where,

m_1 is the mass, in g, of the dish with the sample before drying;
m_2 is the mass, in g, of the dish with the sample after drying; and
m_0 is the mass, in g, of the empty dish.

APPENDIX C
DETERMINATION OF TOTAL ASH

C.1 APPARATUS

C.1.1 *Platinum or silica dish*

C.1.2 *Furnace*, maintained at 500 ± 5 °C.

C.1.3 *Desiccator*

C.2 PROCEDURE

Weigh, to the nearest milligram, about 5 g of the sample in a tared platinum or silica dish (C.1.1). Heat over a low flame until all the organic matter has been charred. Complete ashing by placing the dish in the furnace (C.1.2). When all the carbon has been burnt cool the dish in a desiccator and weigh.

Repeat the process of heating, cooling and weighing at 30 minute intervals until the difference between two successive weighings does not exceed 0.01 g. Record the lowest mass.

C.3 CALCULATION

$$\text{Total ash, per cent by mass} = \frac{m_2 - m_2}{m_1 - m_0} \times 100$$

where,
m_0 is the mass, in g, of the empty dish;
m_1 is the mass, in g, of the sample with the dish, and
m_2 is the mass, in g, of ash with the dish.

APPENDIX D
DETERMINATION OF SULFUR DIOXIDE

D.1 REAGENTS

D.1.1 *Iodine solution*, standard volumetric solution, 0.05 mol/l.

Dissolve 20 g of potassium iodide (A.R. grade) in 40 ml of water in a glass stoppered 1 litre flask. Weigh about 12.7 g of iodine (A.R. grade) and transfer it by means of a small dry funnel into the prepared potassium iodide solution. Insert the glass stopper into the flask and shake until all the iodine has dissolved. Make upto the mark with distilled water. Standardize the iodine solution against a standard 0.05 mol/l solution of arsenic (III) oxide or against a standard 0.01 mol/l solution of sodium thiosulfate.

D.1.2 *Concentrated hydrochloric acid*, rel. den = 1.18.

D.1.3 *Starch solution*, 1 percent (m/V).

D.2 PROCEDURE

Set up the apparatus as in Fig. 1. Transfer 25 g to 100 g of sample using 200 ml of recently boiled and cooled distilled water in to the distillation flask. Connect up the apparatus. Place a sufficient quantity of recently boiled and cooled distilled water in the beaker. Add 0.25 ml of starch solution (D.1.3). Add iodine solution (D.1.1) dropwise from the burette while stirring until a pale blue colour is produced. Place the beaker so that end of the condenser dips into the solution. Add through the tap funnel 20 ml of hydrochloric acid (D.1.2). Heat the flask over the naked flame of a burner (See note) so that the liquid boils in not more than 2 1/2 minutes. Add iodine solution (D.1.1) from the burette into the receiving beaker so that the pale blue colour is maintained throughout the titration. Continue the titration until the colour due to 0.1 ml of iodine persists for at least 1 minute. This shall be reached within a boiling time of 10 minute.

Interference from sulfides may be prevented by adding 0.2 g of copper acetate prior to the addition of hydrochloric acid.

1 ml of 0.05 mol/l iodine is equivalent to 0.0016 g of sulfur dioxide.

NOTE - Care shall be taken to prevent charring the sample at the bottom of the distillation flask.

D.3 CALCULATION

$$\text{Sulfur dioxide, mg/kg} = \frac{V \times 0.0016 \times 10^6}{m}$$

where,

V is the volume, in ml, of 0.05 mol/l iodine required for the titration; and

m is the mass, in g, of the sample taken for the test.

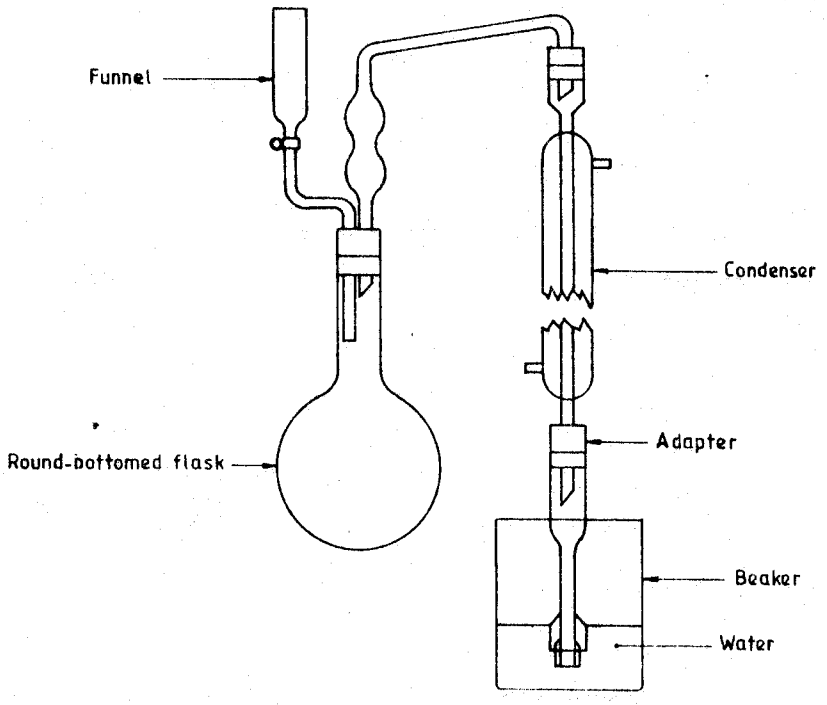

FIGURE 1 - Apparatus for determination of sulfur dioxide

APPENDIX E
DETERMINATION OF NITROGEN

E.1 APPARATUS

E.1.1 **Apparatus** as shown in Fig. 2.

E.1.2 *Kjeldahl flask*, 500-ml capacity.

E.2 REAGENTS

E.2.1 *Anhydrous sodium sulfate*

E.2.2 *Copper sulfate*

E.2.3 *Concentrated sulfuric acid*, rel. den. = 1.84.

E.2.4 *Sodium hydroxide solution*

Dissolve about 255 g of sodium hydroxide in 500 ml of water.

E.2.5 *Sulfuric acid solution*, standard volumetric solution, $C(H_2SO_4)$ = 0.05 mol/l.

E.2.6 *Methyl red indicator solution*

Dissolve 1 g of methyl red in 200 ml of 95 per cent ethyl alcohol.

E.2.7 *Sodium hydroxide solution*, standard volumetric solution, C(NaOH) = 0.1 mol/l.

E.3 PROCEDURE

E.3.1 Weigh, to the nearest milligram, about 0.3 g of the sample and transfer carefully into the kjeldahl flask so that the particles of the sample do not stick onto the neck of the flask. Add about 10 g of anhydrous sodium sulfate (E.2.1), about 0.2 g to 0.3 g of copper sulfate (E.2.2) and 20 ml of sulfuric acid (E.2.3). Place the flask in an inclined position. Heat below the boiling point of the acid until frothing ceases. Heat until the acid boils vigorously and digest till the mixture become clear and pale green or colourless. Cool the contents of the flask. Transfer quantitatively to the round-bottom flask using water. (The total quantity of water used shall be about 200 ml.) Add few pieces of pumice stones. Assemble the apparatus (E.1.1) taking care that the dip-tube extends below the surface of the sulfuric acid (E.2.5) contained in the conical flask. Add about 50 ml of sodium hydroxide (E.2.4) carefully through the side of the flask so that it does not mix at once with the acid solution but forms a layer below the acid layer. Mix the contents of the flask by shaking.

Distill until all the ammonia has passed into the sulfuric acid (E.2.5) containing few drops of methyl red indicator (E.2.6). Detach the flask from the condenser and rinse the condenser thoroughly using water into the conical flask. Wash the dip-tube carefully into the conical flask making sure all traces of the condensate are transferred. When all the washings have drained into the conical flask, titrate against the sodium hydroxide solution (E.2.7).

E.3.2 Carry out a blank determination using all reagents in the same quantities but without the sample.

E.4 CALCULATION

$$\text{Nitrogen (on dry basis), per cent by mass} = \frac{1\cdot 0\,(V_1 - V_2)\,c}{m\,(100 - W)}$$

where,

V_1 is the volume, in ml, of the standard sodium hydroxide solution used in the blank determination (E.3.2);

V_2 is the volume, in ml, of the sodium hydroxide solution used in the determination (E.3.1) of the;

c is the concentration, in mol/l, of the sodium hydroxide solution;

m is the mass, in g, of the sample taken for the test; and

W is the moisture, per cent by mass, of the material (A.2.1).

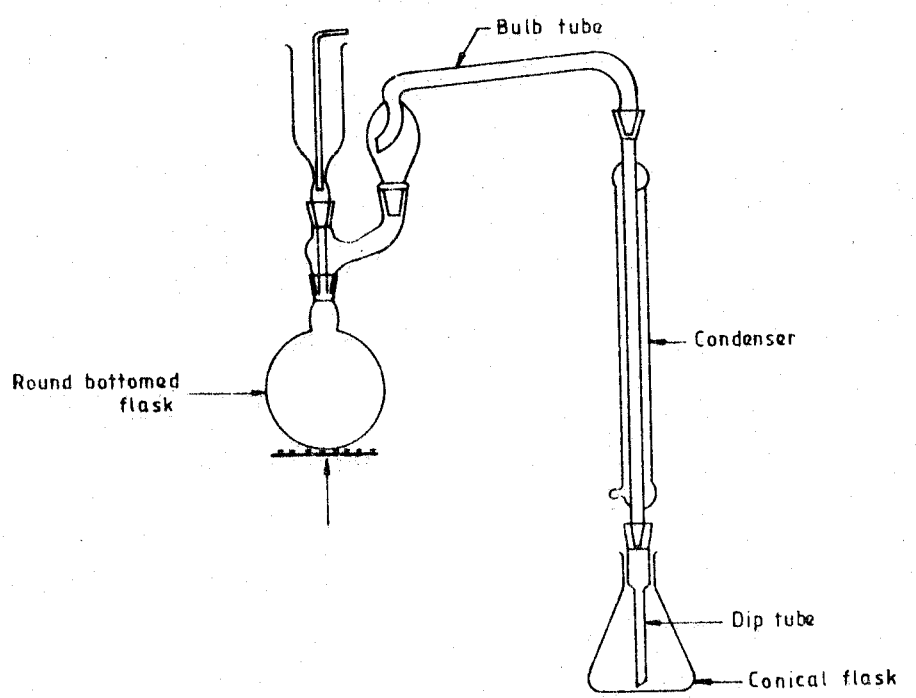

FIGURE 2 - Apparatus for determination of nitrogen

APPENDIX F
DETERMINATION OF pH

F.1 APPARATUS

F.1.1 *Stoppered flask*, of chemically resistant glass, of capacity 100-ml.

F.2 PROCEDURE

Dissolve 1.00 g of powdered gelatine in a small quantity of warm, recently boiled distilled water, in the flask (F.1.1). Dilute to 100 ml with the same water. Swirl and allow to cool to room temperature. Determine the pH value of the solution using a pH meter or other acceptable method. Care shall be taken throughout the operation to minimize absorption of carbon dioxide from the air. Record the pH.

APPENDIX G
MICROBIOLOGICAL EXAMINATION

G.1 PREPARATION OF TEST SAMPLE

Mix contents of retail units (smaller than 100 g) to make up at least 100 g, in a sterile container.

G.1.1 Rehydration of product

Weigh, about 10 g of gelatine into a sterile container aseptically. Transfer this test portion into 90 ml of sterile water in a flask or bottle at room temperature, taking care to avoid gelatine particles being held by condensed water inside the neck of the container.

Swirl the suspension vigorously to wet the gelatine particles uniformly, immerse the flask in a water bath at 45 $\pm$ 1 ^{o}C and ensure that the level of suspension is below the level of water in the bath for a period not exceeding 20 minutes and until solution is complete. Agitate the suspension vigorously several times while it is in the water bath.

When solution is complete, swirl thoroughly and dispense the solution immediately after removing it from the water bath and not later than 5 minutes. Prepare serial decimal dilutions using 0.1 per cent peptone diluent.

NOTES

1 If difficulty is experienced in dispersing the gelatine owing to particles adhering to the walls of the container, the use of sufficient glass beads, 5-mm in diameter, to cover the bottom of container is recommended.

2 With some gelatine samples the first decimal dilution may set rapidly. In these cases prewarmed glass petri dishes may be used.

G.2 TESTS

G.2.1 Aerobic Plate Count

Proceed as described in SLS 516 : Part 1.

G.2.2 Enumeration of *E.coli*

Proceed as described in SLS 516 : Part 3.

G.2.3 *Salmonella*

Pre-enrichment

Weigh, about 25 g into a sterile container. Transfer this test portion into 300 ml of nutrient broth using the procedure given in Clause G.1.1 for rehydration. If pH is outside the range of 6.0 to 7.0, adjust the pH with sterile 1M NaOH or 1M Hcl so that it will fall within the range. Incubate at 37 $\pm$ 1 ^{o}C for 24 hours.

Proceed as described in SLS 516 : Part 5.

APPENDIX H
DETERMINATION OF ZINC

H.1 REAGENTS

H.1.1 *Hydrochloric acid*, approximately 5 mol/l.

Dilute 420 ml of concentrated hydrochloric acid (rel. den.= 1.18) to 1000 ml with water.

H.1.2 *Dithizone stock solution*, 1 g/l solution of diphenyl dithiocarbozone in chloroform. Filter and store in a refrigerator.

H.1.3 *Sodium citrate*, 300 g/l aqueous solution and free of zinc.

Remove the zinc by making the solution slightly alkaline with ammonia (rel. den. = 0.88 to 0.89) and extracting with successive portions of the dithizone stock solution (H.1.2) until the last extract remains green and the aqueous layer becomes slightly yellow. Remove the excess of dithizone by adding 2 ml of 30 g/l solution of citric acid. Extract with successive portions of chloroform (H.1.9) until the last extract is colourless.

H.1.4 *Bromothymol blue indicator solution*

Warm 0.1 g of bromothymol blue with 3.2 ml of 0.05 mol/l sodium hydroxide solution and 5 ml of 90 per cent (V/V) ethanol until it is completely dissolved. Dilute the solution to 250 ml with 20 percent (V/V) ethanol. The commercially available indicator may also be used.

H.1.5 *Ammonium hydroxide*, 10 mol/l solution

Dilute 550 ml of ammonia solution (rel. den. = 0.88 to 0.89) to 1000 ml with water.

H.1.6 *Dithiocarbamate stock solution*

Dilute 1 ml of redistilled carbon disulfide to 10 ml with dry chloroform. Add this mixture slowly, with stirring and cooling, to 3 ml of redistilled diethyl amine. Dilute to 10 ml with dry chloroform.

Alternatively, dissolve 1 g of crystalline diethyl ammonium diethyl dithiocarbamate in 100 ml of redistilled chloroform. Store in a glass-stoppered amber-coloured bottle. Prepare freshly once a week.

H.1.7 *Dithiocarbamate extraction solution*

Dilute 5 ml of the dithiocarbamate stock solution (H.1.6) to 100 ml with chloroform. Store in a glass-stoppered bottle. Prepare freshly before use.

H.1.8 *Alkaline citrate solution*

Mix 50 ml of the sodium citrate solution (H.1.3), 100 ml of aqueous sodium hydroxide solution having a concentration of 100 g/l and 100 ml of water.

H.1.9 *Chloroform*, redistilled, and containing 1 per cent (V/V) of ethanol.

H.1.10 *Bromine water*, saturated solution.

H.1.11 *Sodium metabisulfite*, 50 g/l aqueous solution.

H.1.12 *Borax solution*, ($Na_2B_4O_7.10H_2O$), 30 g/l aqueous solution.

Extract with successive portions of the dithizone stock solution (H.1.2) until the last extract remains green. Remove excess dithizone by extracting with successive portions of chloroform until the last extract is colourless.

H.1.13 *Dithizone titration solution*

H.1.13.1 Add 2 ml of ammonium hydroxide solution (H.1.5) to 50 ml of water.

H.1.13.2 Extract 40 ml of the dithizone stock solution (H.1.2) with two 50 ml portions of dilute ammonia solution (H.1.13.1). Discard the chloroform layer. Acidify the extracts with dilute hydrochloric acid (1 volume of concentrated hydrochloric acid with 3 volumes of water). Shake with 100 ml of chloroform, allow to separate and discard the upper aqueous layer. Wash the lower chloroform layer with two successive 10 ml portions of water and filter through a dry filter paper. Prepare freshly before use.

H.1.14 *Stock zinc solution*

Dissolve 1.00 g of pure zinc in 5 ml of concentrated hydrochloric acid (rel. den. = 1.18) and 10 ml of water and dilute to 1000 ml.

H.1.15 *Standard zinc solution*, 1 ml containing 10 µg of zinc.

Mix 5 ml of the stock zinc solution (H.1.14) and 5 ml of 1 mol/l hydrochloric acid solution. Dilute to 500 ml with water. Prepare freshly before use.

H.2 APPARATUS

All glassware used shall be of zinc-free borosilicate glass and shall be thoroughly washed with concentrated sulfuric acid (rel. den. = 1.84), rinsed thoroughly with water and dried.

H.2.1 *Volumetric flask*, 50-ml.

H.2.2 *Platinum or silica dishes*, having an internal diameter of at least 95-mm and height of 40-mm.

H.2.3 *Separating funnels*, 100-ml and 25-ml.

H.2.4 *Conical flask*, 100-ml.

H.2.5 *Muffle furnace*, maintained at 450 ± 10 °C.

H.2.6 *Spectrophotometer*.

H.3 PROCEDURE

H.3.1 Preparation of test solution

Weigh, to the nearest milligram, about 2 g of the sample in the dish (H.2.2). Heat gently over a low bunsen flame until the sample has completely charred and all visible fumes have been evolved. Place in the muffle furnace (H.2.5), until the carbon has been burnt off and a greyish-white ash has been formed. Remove from the muffle furnace. Cool, add 5 ml of water and 10 ml of the hydrochloric acid solution (H.1.1) to the ash in the dish. Boil gently for 5 minutes. Transfer the solution to the volumetric flask (H.2.1) rinsing with two successive 10 ml portions of water, through a 90 mm 'ashless' filter paper. Rinse the paper twice with 5 ml portions of water. Allow the clear solution to cool to room temperature and make up to the mark with water.

H.3.2 Zinc extraction

Take an appropriate aliquot (about 5 ml) of the test solution (H.3.1) to a beaker. It shall contain not more than 50 ug of zinc. Add 5 ml of sodium citrate solution (H.1.3) and 0.2 ml of bromothymol blue indicator solution (H.1.4), followed by the ammonia solution (H.1.5) until the colour of the solution just changes to the full blue colour of the indicator (pH 7.5 to 8.0).

If a precipitate forms, add hydrochloric acid to dissolve it, followed by more sodium citrate solution and ammonia solution as before.

Transfer the solution to 100-ml separating funnel (H.2.3), add 15 ml of the dithiocarbamate extraction solution (H.1.7), shake vigorously for 30 seconds to 40 seconds, and allow to separate. Run off the lower chloroform layer into a second separating funnel. Extract the aqueous layer with two successive 5 ml portions of the dithiocarbamate extraction solution (H.1.7) shaking for 30 seconds to 40 seconds. Allow to separate. Add each lower chloroform layer to the second separating funnel. Discard the aqueous layer. To the combined chloroform extracts, add 15 ml of the citrate solution (H.1.8), shake for 30 seconds to 40 seconds. Allow to separate. Run the lower chloroform layer into another separating funnel. Add 10 ml of citrate solution, shake and allow complete separation. Discard the lower chloroform layer. Combine the aqueous solutions, add 5 ml of chloroform, shake for about 10 seconds, allow to separate, run off the chloroform layer and discard it. Wash the aqueous layer with 2 ml of chloroform without mixing. Discard the washing. Transfer the aqueous solution to the conical flask (H.2.4). Rinse with a small amount of water. Add hydrochloric acid until the colour of the indicator just changes from blue to yellow and add 2 ml in excess. Add 10 ml of bromine water (H.1.10), boil carefully until the solution is nearly colourless. Add 0.5 ml of the sodium metabisulfite (H.1.11) and cool to room temperature.

H.3.3 **Determination of zinc**

Transfer the solution from the conical flask to the 250-ml separating funnel (H.2.3). Rinse the flask with few millilitres of water. Add 0.2 ml of bromothymol blue indicator (H.1.4), 2 ml of sodium citrate solution (H.1.3) and ammonia solution (H.1.5), drop by drop, until the colour of the solution just changes to the full blue colour of the indicator. Add 10 ml of borax solution (H.1.12) giving a pH of 8.7 to 9.3. Add the dithizone titration solution (H.1.13) from a burette, first in 1 ml portions together with about 3 ml of chloroform (H.1.9), shaking vigorously for 30 seconds between additions. Allow to separate and discard the lower red chloroform layer and then in progressively smaller portions until a 0.1 ml portion with about 2 ml of chloroform results in a purplish colour in the solvent layer. This end point is confirmed by removing the solvent layer and add a further 0.1 ml portion of the dithizone titration solution. Shake and allow to separate. The solvent layer shall remain green.

H.3.4 **Blank test**

Carry out a blank test following the entire procedure but omitting the sample.

H.3.5 **Standardization of dithizone titration solution**

After titration of the blank solution, run off the lower chloroform layer and remove the excess of dithizone from the aqueous layer by shaking with two successive 5 ml portions of the chloroform (H.1.9). Allow to separate. Discard the chloroform layers. Add 5 ml of the dilute standard zinc solution to the aqueous layer (H.1.15) and titrate with the dithizone titration solution (H.1.13) as given in H.3.3. Remove the excess of dithizone from the titrated liquid as before by extracting with two successive portions of chloroform. Add 25 ml of the dilute standard zinc solution (H.1.15) and titrate as before.

H.4 **CALCULATION**

The difference between the two titrations is the volume of dithizone titration solution required by 200 ug of zinc. Hence, calculate the number of micrograms of zinc equivalent to 1 ml of dithizone titration solution.

$$\text{Zinc content, milligrams per kilogram} = V_1 \times m_1 \frac{50}{V_2} \times \frac{1}{m_0}$$

where,

V_1 is the volume, in ml, of dilute dithizone standard solution required for titration of the test solution;

m_1 is the mass, in µg of zinc equivalent to 1 ml of dithizone titration solution;

V_2 is the volume, in ml, of the test solution ; and

m_0 is the mass, in g, of the sample.

APPENDIX J
DETERMINATION OF ARSENIC

J.1 PROCEDURE

J.1.1 Preparation of the test solution

Weigh, to the nearest milligram, about 2 g of the sample, place in a beaker. Add 20 ml of water and 10 ml of concentrated hydrochloric acid (rel. den. = 1.18). Heat the beaker on a steam bath for 1 hour. Allow to cool to room temperature. Transfer the contents and washings of the beaker to a wide-mouthed bottle.

J.1.2 If the sample contains large amounts of sulfur it shall be treated as follows.

To 2.00 g of the sample add 10 ml of water. Allow to stand for 1 hour. Warm to dissolve. Add 10 ml of hydrochloric acid [32 per cent (m/m) solution] and bromine solution in slightly excess. Add 2 ml of stannated hydrochloric acid and heat under a reflux condenser for one hour. Cool. Add 10 ml of water and 10 ml of hydrochloric acid.

J.1.3 Proceed as given in Method 2.1 (Modified Gutzeit method) SLS 312 : 1976.

APPENDIX K
DETERMINATION OF LEAD

K.1 PROCEDURE

K.1.1 Preparation of the test solution

Weigh, to the nearest milligram, about 3 g of the sample in a platinum or silica dish. Heat gently over a low bunsen flame until the sample has completely charred and all visible fumes have been evolved. Place in a muffle furnace, controlled at 450 °C, until the carbon has been burnt off and a whitish-grey ash has been formed. Remove from the furnace and cool. Add 5 ml of water and 10 ml of hydrochloric acid solution (5 mol/l) and heat on a water bath for 5 minutes. Filter through to a fluted 90-mm `ashless' filter paper and filter into a 150-ml beaker. Wash with two successive 10 ml portions of water. Rinse the paper twice with 5 ml portions of water. Allow the clear solution to cool to room temperature.

K.1.2 Proceed as given in Method A of SLS 311 : 1975.

APPENDIX L
DETERMINATION OF COPPER

L.1 REAGENTS

L.1.1 *Stock copper solution*

Dissolve 0.3926 g of copper sulfate pentahydrate, ($CuSO_4.5H_2O$) in 1 mol/l solution of sulfuric acid. Dilute to 1000 ml using 1 mol/l solution of sulfuric acid at 20°C.

L.1.2 *Standard copper solution,* 1 ml containing 2 µg of copper.

Dilute 10.0 ml of the stock copper solution (L.1.1) to 500 ml with sulfuric acid at 20 °C. Prepare freshly before use.

L.1.3 *Dithiocarbamate solution*

1 g/l solution of diethyl ammonium diethyl dithiocarbamate in carbon tetrachloride.

L.1.4 *Disodium ethylenediamine-NNN^{1}N^1 tetra-acetate dihydrate (EDTA) - citrate solution.*

Dissolve 20 g of ammonium citrate and 5 g of EDTA in water and dilute to 100 ml. Extract with successive 15 ml portions of the dithiocarbamate solution (L.1.3) until a colourless extract is obtained.

L.1.5 *Thymol blue solution*

Dissolve 0.1 g of thymol blue with 4.3 ml of 0.05 mol/l sodium hydroxide in 5 ml of ethanol 90 per cent (V/V). Warm to dissolve and dilute to 250 ml with ethanol 20 per cent (V/V).

L.1.6 *Ammonia solution,* 6 mol/l.

Prepare by using concentrated ammonium hydroxide, NH_4OH, (rel. den = 0.88 to 0.89).

L.1.7 *Hydrochloric acid,* approximately 5 mol/l solution.

Dilute 420 ml of concentrated hydrochloric acid (rel. den. = 1.18) to 1000 ml with water.

L.2 APPARATUS

As given in H.2.

L.3 PROCEDURE

L.3.1 Preparation of calibration curve

Add 2.0 ml, 5.0 ml, 10.0 ml, 15.0 ml, 20.0 ml, and 25.0 ml of the standard copper solution (L.1.2) into six 100-ml separating funnels (H.2.3). Leave another separating funnel without adding the copper solution. Treat the solution in each separating funnel as follows:

Dilute to 25 ml with water. Add 10 ml of the EDTA - citrate solution (L.1.4) and 5 drops of the thymol blue solution (L.1.5). Add sufficient amount of 6 mol/l ammonia solution (L.1.6) to give a bluish green colour when cold.

Add 15 ml of dithiocarbamate solution (L.1.3), shake vigorously for 2 minutes and allow to separate. Place a plug of cotton wool in the stem and collect the carbon tetrachloride layer. Immediately measure the absorbance of each extract in a 10-mm cell against the blank extract, at 436-nm.

Plot the absorbance against the copper content (µg/ml).

L.3.2 Preparation of the test solution

Weigh, to the nearest milligram, about 2 g of the sample in the dish (H.2.2). Heat gently over a low bunsen flame until the sample has completely charred and all visible fumes have been evolved.

Place in the furnace (H.2.5) until the carbon has been burnt off.

Remove the dish from the furnace and cool. Add 5 ml of water and 20 ml of the hydrochloric acid (L.1.7) and boil gently for 5 minutes. Filter the solution through a 110-mm 'ashless' filter paper into the volumetric flask (H.2.1) and wash the dish with two 5 ml portions of water. Finally wash the filter paper with two further 5 ml portions of water. Cool to room temperature and dilute to the mark.

L.3.3 Determination

Pipette 25 ml of prepared solution into a 100-ml separating funnel. It shall contain not more than 50 µg of copper. Proceed as described in L.3.1 commencing with 'addition of 10 ml of the EDTA-citrate solution.'

L.3.4 Blank test

Carry out a blank test following the entire procedure but omitting the sample.

L.3.5 Expression of results

Read the amount of copper present corresponding to the respective absorbances of the blank and test solutions from the calibration curve. Under the test conditions μg of copper is equivalent to milligrams per kilogram of copper.

AMD 276

Draft Amendment No. 1 approved on 2001-03-20
to SLS 845 : 1989

SPECIFICATION FOR GELATINE

Page 4

Clause 4.1.1

"In line 1, insert 'colourless or' between the third and fourth word".

Clause 4.1.2

Amend to read as follows.

It shall have a very slight odour and taste but not objectionable which is characteristic and bouillon-like.

Clause 4.2.2

"In line 2, insert 'fixed and' between the fourth and fifth word".

Clause 5.2.2 f

Amend to read as "Date of expiry"

Clause 5.2.2

Insert the following clause as 5.2.2. h

"The words animal origin"

NE/-
2001-01-17
Doc. AMD.1

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