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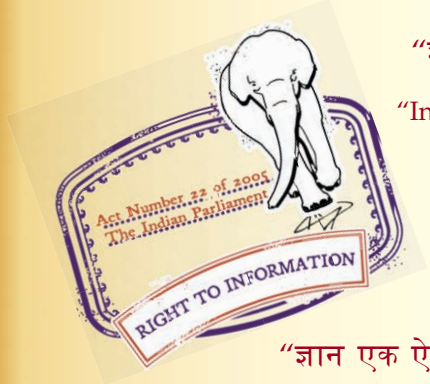
IS 1485 (1993): Macaroni, Spaghetti, Vermicelli and Egg Noodles [FAD 16: Foodgrains, Starches and Ready to Eat Foods]



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मैकरोनी, मोटी सैबई, बर्मिसेली एवं एग नूडल – विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

MACARONI, SPAGHETTI, VERMICELLI AND EGG NOODLES — SPECIFICATION

(Second Revision)

UDC 664.694

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

July 1993

Price Group 5

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Foodgrains and Foodgrain Industries Sectional Committee had been approved by the Food and Agriculture Division Council.

Macaroni, spaghetti, vermicelli and egg noodles belong to a class of food products generally known as 'Macaroni Products'. The Italians call them 'Pasta Alimentare' (Alimentary Paste), while German terminology is 'Teigwaren' (Paste Goods). The Macaroni industry is well developed in Italy, USA, France, Switzerland and other western countries. Although vermicelli (*SEMIAN*) has been known and produced in India for a long time, the production of macaroni and spaghetti is of comparative recent origin in this country. The principal raw materials are *SUJI* (semolina) or *MAIDA* obtained preferably from hard wheat, such as durum. The minor ingredients which may be added are edible groundnut flour, tapioca flour, soya flour, milk powder casein, gluten, vegetables and spices. Besides, they may also be enriched with vitamins and minerals. In the case of egg noodles, egg in any form may be added.

The manufacturing process for macaroni, spaghetti, vermicelli and egg noodles consists of making the dough from *SUJI* or *MAIDA* with or without other ingredients with cold or lukewarm water, kneading it and then extruding it through an extrusion press fitted with a die of the desired shape. The extruded product cut to a given length is then dried to a definite moisture content under controlled conditions of temperature and humidity. The dried product is suitably packed depending on the market requirements.

This standard was first published in 1959. It had been revised in 1976 with a view to update the requirements specified in the standard as manufacture of macaroni, spaghetti, vermicelli and egg noodles had stabilized and the forms and types of macaroni and spaghetti had become rationalized. Requirements for various characteristics had been reviewed and that of total ash modified. Suitable modification had also been effected in the 'cooking test'.

This standard is now being revised to include both instant and egg noodles which have become very popular in the country. The revision also incorporates Amendment No. 1 to the Standard. The list of ingredients also has been expanded.

In the preparation of this standard, due consideration has been given to the provisions stipulated under the *Prevention of Food Adulteration Act*, 1954 and the Rules framed thereunder and the *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977. However, this standard is subject to the restrictions imposed under these Rules, wherever applicable.

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 analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MACARONI, SPAGHETTI, VERMICELLI AND EGG NOODLES — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for macaroni, spaghetti, vermicelli and egg noodles.

2 REFERENCES

2.1 The Indian Standards listed in Annex A are necessary adjuncts to this standard.

3 REQUIREMENTS

3.1 Ingredients

3.1.1 *Essential Ingredients*

The following materials shall be used in the manufacture of macaroni, spaghetti, vermicelli and egg noodles.

3.1.1.1 *Maida or suji — see* IS 1009 : 1979 or IS 1010 : 1968 respectively. These shall be first passed through suitable sieves before use.

3.1.1.2 *Water — see* IS 4251 : 1967.

3.1.1.3 *Egg (only in the case of egg noodles)* — These may be in any form namely liquid eggs, frozen eggs, dried eggs or egg yolk.

3.1.2 *Optional Ingredients*

In addition to the essential ingredients specified under **3.1.1**, any of the following ingredients may be used in manufacturer of macaroni, spaghetti, vermicelli and egg noodles.

3.1.2.1 *Milk powder (whole and skim) — see* IS 1165 : 1986.

3.1.2.2 *Edible casein — see* IS 1167 : 1965.

3.1.2.3 *Edible tapioca flour — see* IS 1318 : 1969.

3.1.2.4 *Edible oilseeds flour — see* relevant Indian Standards.

3.1.2.5 *Spices — see* relevant Indian Standards.

3.1.2.6 *Gluten*

3.1.2.7 *Soya flour — see* IS 7835 : 1975, IS 7836 : 1975 or IS 7837 : 1975.

3.1.2.8 Di-sodium phosphate (Na_2PHO_4 $7H_2O$) —0·5-1·0 percent (only in quick cooking products).

3.1.2.9 Vegetable or vegetable products and fruit or fruit products (preserved, dehydrated or pulp).

3.2 Description

3.2.1 *Form and Dimensions*

The forms and dimensions of macaroni, spaghetti, vermicelli and egg noodles should be as given below.

3.2.1.1 *Macaroni*

The material may be of two types, namely, long goods or cut goods:

a) The long goods should be in the form of tubular rods, smooth or corrugated. These should have the outer diameter ranging between 3 and 5 mm and of wall thickness of about 1 mm.

b) The cut goods should be obtained by extrusion and may be in the form of elbows, tubes, shells, alphabets, numerals stars, wheels, rings, rice, melon seeds as agreed to between the purchaser and the supplier.

3.2.1.2 *Spaghetti*

The material should be in the form of solid rods of diameter 1 mm.

3.2.1.3 *Vermicelli*

The material should be in the form of solid rods of diameter between 0·5 and 1·25 mm.

3.2.1.4 *Egg noodles*

The material shall be in the form of ribbons, and shall have not less than 5·5 percent by mass of egg solids or egg yolk solids when tested by the method given in Annex B.

3.2.2 *Other Physical Characteristics*

Macaroni, spaghetti, vermicelli and egg noodles shall be of good characteristic colours, flavour and odour and shall be free from rancidity, mustiness, bitterness or any other undesirable taste or odour. It shall also be free from impurities, any foreign matter, cracks, flaws, mould, insect infestation or other spoilage. The material shall retain its shape and show no signs of disintegration and shall swell appreciably when plunged into vigorously-boiling water and boiled for 10 minutes.

NOTE — In case of instant or quick cooking noodles, the material shall be boiled for the time declared in the cooking instructions.

The material shall be smooth to the touch and shall not contain any added colouring matter.

3.3 Enrichment

Subject to the agreement between the purchaser and the supplier, the material may be fortified with one or more of the following:

a) *Mineral* — Calcium, phosphorus and iron.

b) *Vitamins* — A, B complex and D.

3.4 The material shall be manufactured in premises maintained under hygienic conditions *see* IS 2491 : 1972.

3.5 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Macaroni, Spaghetti, Vermicelli and Egg Noodles

(*Clause* 3.5)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Moisture, percent by mass, *Max*	11	C
ii)	Total ash (on dry basis), percent by mass, *Max*	0·7	D
iii)	Acid insoluble ash (on dry basis), percent by mass, *Max*	0·05	E
iv)	Total protein (N×5·7) (on dry basis), percent by mass, *Min*	10	F
v)	Cooking test: Total solids in gruel, percent by mass, *Max*	8	G
vi)	Free acidity (ml of 1N, NaOH solution per 100 g of product), *Max*	4	H

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in cardboard cartons with a lining of moisture-proof material or in suitable plastic film or moisture-proof paper bags, sealed to prevent ingress of moisture. Tinplate containers may also be used. The material coming into contact with the food product shall be of food grade quality. The size of the container is subject to agreement between the purchaser and the supplier.

4.2 Marking

Each container shall be suitably marked so as to give the following information:

a) Name of the material;

b) If enriched with vitamins: (i) the word 'vitaminized' placed in brackets, below (a); and (ii) the details of enrichment and quantities added;

c) If spices have been added, the word 'spiced' to be given on the label;

d) If egg has been added, it shall be declared on the label;

e) All ingredients shall be declared on the label;

f) Name and address of the manufacture;

g) Batch or code number;

h) Net mass;

j) Date of manufacture;

k) Expiry date;

m) Cooking instructions; and

n) Any other details required under the *Standards of Weights and Measures (Packaged Commodities) Rules, 1977 and PFA Rules.*

4.2.1 The container may also be marked with the Standard Mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn and conformity of the material to the requirements of this specification shall be determined by the method prescribed in Annex F of IS 1158 : 1973.

6 TESTS

6.1 Tests shall be carried out as prescribed under **3.2.2** and appropriate appendices specified in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results.

ANNEX A

(*Clause* 2.1)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*	*IS No.*	*Title*
265 : 1987	Hydrochloric acid (*third revision*)	1167 : 1965	Casein (edible quality) (*revised*)
460 (Part 1) : 1985	Test sieves : Part 1 Wire cloth test sieves (*third revision*)	1318 : 1969	Edible tapioca flour (*first revision*)
1009 : 1979	*MAIDA* for general purposes (*second revision*)	2491 : 1972	Code for hygienic conditions for food processing units (*first revision*)
1010 : 1968	*SUJI* or *RAWA* (semolina) (*first revision*)	4251 : 1967	Quality tolerances for water for processed food industry
1070 : 1992	Reagent grade water (*third revision*)	7835 : 1975	Edible medium-fat soya flour
1158 : 1973	Corn flakes (*first revision*)	7836 : 1975	Edible low-fat soya flour
1165 : 1986	Milk-powder (*third revision*)	7837 : 1975	Edible full-fat soya flour

ANNEX B

(*Clause* 3.2.1.4)

DETERMINATION OF EGG SOLIDS IN MACARONI PRODUCTS

B-0 PRINCIPLE

Egg solid content of egg noodles is estimated closely from cholesterol content of unsaponifiable matter. When analyzing egg-free products or material containing less than 0·23 percent unsaponifiable matter, add 10 mg cholesterol before analyzing and correct results accordingly. The added cholesterol should have melting point not less than 147°, and its purity should be checked by submitting 20 mg to the determination as given below.

B-1 APPARATUS

B-1.1 Cold Baths

Prepare one bath with crushed ice and one with salt-ice mixture.

B-1.2 Mohr Pipettes

One graduated to 0·01 ml and one to 0·1 ml.

B-1.3 Filteration Bell Jar

Size to accomodate 300-ml Erlenmeyer flask connected to vacuum source by 2-way stopcock

B-1.4 Filtering Device

For filtering at 0°C, prepare filter tube of Knorr type. Tube should be about 20 mm internal diameter, body about 11 cm long, and stem about 10 cm long, provided with usual nickel or Monel metal disk. Insert this tube through bottom of container of such size that crushed ice can be packed around body of tube to height of 7-8 cm. Fit tube into 500-ml suction flask. In tube prepare mat of asbestos 6-8 mm thick, packed fairly tight, and cover with layer of sand about 12 mm deep. Digest asbestos with acid and alkali. Purify sand by passing through 60-mesh sieve, then treat with warm hydrochloric acid until extract is practically colourless. Wash, dry, and ignite sand.

B-1.5 Separating Funnels

Separating funnels, one 250-ml and one 500-ml. Wash free of grease. Funnels must be ether-tight, with stopcocks lubricated only with water.

B-1.6 Sintered Glass Filter — 30-50 ml capacity.

B-2 REAGENTS

B-2.1 Bromine Solution

Weigh to 0·1 g a narrow-mouthed, glass-stoppered 25-ml flask containing 5 ml carbon tetrachloride. Add 4-5 g bromine, weigh again and dilute with carbon tetrachloride to calculated final concentration of $0{\cdot}22 \pm 0{\cdot}02$ g bromine per ml. (This reagent should not be more than 2 days old.) Store under refrigeration.

B-2.2 Acetic Acid Solution

Pipette or burette 200 ml glacial acetic acid into 250-ml glass-stoppered volumetric flask; dilute to mark with water, mix cautiously, dilute to mark, and mix again.

B-2.3 Sodium Hypochlorite Solution

Dissolve 88 g reagent grade sodium hydroxide in 200 ml water in a wide-mouthed 3-litre flask. Add about 1 500 g crushed ice and pass in chlorine until 71 g is absorbed. Dilute to 2 litre and then store in dark bottles under refrigeration. Solution should be alkaline to phenolphthalein. Before using, check concentration of available chlorine as follows: Pipettee 5 ml solution into 100 ml water containing 2 g potassium iodide. Add 5 ml hydrochloric acid (1:1) and titrate with 0·1 N $Na_2S_2O_3$. Solution should be equivalent in available chlorine to 0·95 – 1·05 N NaOCl.

B-2.4 Sodium Formate Solution

Dissolve 50 g reagent grade sodium formate and dilute 100 ml.

B-2.5 Hydrochloric Acid

Approximately 6 N. Mix 500 ml concentrated hydrochloric acid with water and dilute to 1 litre.

B-2.6 Potassium Iodide Solution

20 percent. Dissolve 20 g potassium iodide and dilute to 100 ml. This solution must be colorless when acidified with hydrochloric acid.

B-2.7 Soluble Starch Solution — 1 percent.

B-2.8 Sodium Thiosulphate Solution — 0·02 N.

B-2.9 Sodium Sulphate Anhydrous

Powdered to pass through 60-mesh sieve.

B-2.10 Potassium Hydroxide Solution

a) 100 percent — Dissolve 10 g potassium hydroxide in 10 ml water

b) 1 percent — Dissolve 10 g potassium hydroxide in water and make upto 1 litre.

B-2.11 Ammonium Molybdate Solution

Dissolve 5 g ammonium molybdate and make to 100 ml.

B-2.12 Ether

a) Anhydrous, reagent grade

b) Dry and peroxide free — (check for presence of peroxides, just prior to use). Rinse glass-stoppered cylinder with ether, place 10 ml ether in rinsed cylinder, and add 1 ml freshly prepared 10 percent solution of cadmium potassium iodide. Let stand protected from light for one hour, shaking occasionally, no color should develop in either liquid layer. To dry ether, shake with excess of anhydrous calcium chloride and filter.

B-3 PROCEDURE

B-3.1 Determination of Unsaponifiable Matter

Weigh 10-g sample into 500-ml Erlenmeyer flask and add, with shaking, 30 ml hydrochloric acid (1 : 1). Heat on a steam bath for 30 minutes with occasional shaking. Cool under cold-water tap and add carefully, with shaking, 30 g, potassium hydroxide pellets. Add pellets at such rate that liquid may boil, but not so fast as to cause splattering. While flask is still hot, place on steam bath, cover with small watch-glass, and heat for 3 hours with occasional swirling. Cool, add 30 ml of 95 percent alcohol and 500 ml water and mix well. (Samples may be allowed to stand overnight at this point).

Add 100 ml ether, swirl mixture vigorously for one minute, and transfer to 500-ml separating funnel, washing flask with 50-ml and 25-ml portions of ether. Wash flask with 50 ml dilute potassium hydroxide solution. Pour washings slowly into funnel while gently swirling liquid, and continue swirling for 10 to 15 seconds.

Let liquid separate (about 10 minutes) and slowly draw off soap solution into 250-ml separating funnel, but do not draw off any emulsion or insoluble matter at interface. If emulsion forms and does not break to give sharp interface within 10 minutes, pour 5 ml alcohol into funnel and let stand until emulsion breaks. Rinse down sides of 500-ml funnel with 10 ml dilute potassium solution and draw this off into smaller funnel. Add 50 ml ether to smaller funnel and shake vigorously.

After liquids have separated, discard lower layer. Add ether layer to solution in larger funnel and rinse smaller funnel with 10 ml ether. Wash ether solution as before with 100 ml

dilute potassium hydroxide solution, still retaining any insoluble matter or emulsion in funnel. In the same manner wash ether solution with two additional 100-ml portions of dilute potassium hydroxide solution, testing portions of last washings for soap by acidifying with dilute hydrochloric acid (1 : 4). Acidified washings should be clear or only faintly turbid. If necessary, repeat washings with dilute potassium hydroxide solution until acidified washings are clear.

Wash ether solution by swirling with successive portions : 50 ml water, 50 ml water containing 0·5 ml 0·1 N hydrochloric acid, and two more 50-ml portions water. Draw off as much water as possible without loss of ether solution.

Filter ether solution into dry 500-ml suction flask through 50-g layer of sodium sulphate on sintered glass filter, using no suction for first several millilitres and then gentle suction for remainder.

Rinse separate funnel and filter with successive 10-, 5-, 5-, and 5-ml portions dry ether. Rinse stem of funnel, add glass beads to flask, and evaporate on steam bath to about 20-ml volume (current of air directed across neck of flask will increase rate of evaporation). Heat 50-ml glass-stoppered Erlenmeyer flask at 100-105 °C for 1 hour, cool in air for 30 minutes, and weigh. Transfer ether solution in suction flask quantitatively to weighed Erlenmeyer flask. Evaporate ether and dry flask at 100°C. Weigh flask again and determine unsaponifiable matter by difference.

Carry out blank determination using all reagents in above method without sample, and deduct this value from weight of unsaponifiable matter. Calculate unsaponifiable matter.

B-3.2 Determination of Egg Solids

Wash down sides of flask containing unsaponifiable matter with 2·0 ml anhydrous ether delivered from Mohr pipette. Pack flask in ice bath up to neck and let stand for at least 10 minutes. From another Mohr pipette add 0·20 ml cold bromine solution, swirl, stopper replace in ice bath for 10 minutes.

During bromination, cool acetic acid solution in ice-salt bath until temperature is −5°C and in same bath cool a 25-ml cylinder. Measure, 15 ml acetic acid solution into cold cylinder and add to reaction flask. Swirl reaction mixture for 3 minutes while holding in ice bath, and let stand in ice bath for 10 minutes. With slight suction pour mixture down stirring rod into cold filter tube, leaving rod in tube. Wash down sides of flask with 5 ml cold acetic acid solution and replace in ice bath. When liquid in funnel recedes below surface of sand, add acetic acid from reaction flask. Again repeat washing with 5 ml cold acetic acid solution and suck free of excess liquid. Wash flask and tube with ice water, filling filter tube about five times. Drain flask and apply suction to filter to remove all excess water, discarding filtrate and washings.

Remove crushed ice from around filter tube and transfer tube to top of filtration bell jar. Place 300-ml Erlenmeyer flask under filter tube so that stem projects well into the neck of the flask. Wash filter with 10 ml alcohol, 10-, 5-, and 5-ml portions of ether, and finally with 10 ml alcohol, stirring sand gently with each portion of solvent, and let mixture stand for about 1 minute before applying suction. Wash stem of filter with few ml ether, remove 300-ml flask, and to filtrate add 1 ml potassium hydroxide solution. Mix and wash down sides of flask with 5 ml ether. Evaporate on steam bath, using stream of clean air to remove all vapors.

Add 40 ml hot water to residue, mix and neutralize with 6 N hydrochloric acid, using 1 drop methyl red indicator. Add 10 g NaCl, 3 g $NaH_2PO_4H_2O$, and 20 ml N_2OCL solution. Bring to vigorous boil, remove from heat, and at once, with care, add 5 ml sodium formate solution. Cool and dilute to about 150 ml. Add 5 ml potassium iodide solution, 1-2 drops ammonium molybdate solution, and 25 ml 6 N hydrochloric acid. Immediately titrate rapidly with thiosulfate, using starch indicator. Correct titre by blank value obtained on reagents, starting with addition of potassium hydroxide to alcohol ether solution.

B-4 CALCULATIONS

B-4.1 Cholesterol (mg) $= 0{\cdot}55 + 0{\cdot}688$ (ml $0{\cdot}02\,N\,Na_2S_2O_3$)

B-4.2 Percent sterol calculated as cholesterol in sample (moisture-free basis) $= \dfrac{\text{mg cholesterol}}{(100 - \text{percent moisture})} = C$

B-4.3 Percent commercial egg-yolk solids (moisture-free basis) $= \dfrac{(C - 0{\cdot}024)\,100}{(2{\cdot}88 - 0{\cdot}024)} = 35\,(C - 0{\cdot}024)$

B-4.4 Percent commercial whole-egg solids (moisture-free basis) $= \dfrac{(C - 0{\cdot}024)\,100}{(2{\cdot}11 - 0{\cdot}024)} = 48\,(C - 0{\cdot}024)$

ANNEX C

[*Table* 1, *Item* (i)]

DETERMINATION OF MOISTURE

C-1 PROCEDURE

C-1.1 Preparation of Sample

Grind in a pestle and mortar about 30 g of the material so that at least 90 percent passes though 425-micron IS Sieve [*see* IS 460 (Part 1) : 1985]. Transfer this prepared sample to a well stoppered glass bottle for use as indicated in **C-1.2** and **F-3.1**.

C-1.2 Weigh accurately about 5 g of the prepared sample in a suitable moisture dish, made of porcelain, silica or platinum, previously dried in an air-oven and weighed. Place the dish in an air-oven maintained at 105 ± 2°C for five hours. Cool the dish in a desiccator and weigh the dish with the lid on. Heat again at 105 ± 2°C in the air-oven for 30 minutes. Cool the dish in the desiccator and weigh. Repeat this process of heating for 30 minutes, cooling and weighing till the difference in mass between two successive weighings is less than one milligram. Note the lowest mass.

NOTE — Preserve the dish containing this dried material for the determination of total ash (*see* **D-1.1**).

C-2 CALCULATION

C-2.1 Moisture, percent by mass $= \frac{100\,(M_1 - M)}{M_2 - M}$

where

M_1 = mass, in g, of the dish with the material before drying;

M = mass, in g, of the empty dish; and

M_2 = mass, in g, of the dish with the material after drying.

ANNEX D

[*Table* 1, *Item* (ii)]

DETERMINATION OF TOTAL ASH

D-1 PROCEDURE

D-1.1 Ignite the dried material (*see* **C-1.2**) in the dish with the flame of a suitable burner for about one hour. Complete the ignition by keeping in a muffle furnace at 600 ± 20°C until grey ash results. Cool in a desiccator and weigh. Heat again at 600 ± 20°C in the muffle furnace for 30 miuutes. Cool in the desiccator and weigh. Repeat the process of heating for 30 minutes, cooling and weighing till the difference in mass between the two successive weighings is less than one milligram. Note the lowest mass.

NOTE — Preserve the dish containing the ash for the determination of acid insoluble ash (*see* **E-2.1**).

D-2 CALCULATION

D-2.1 Total ash (on dry basis), percent by mass $= \frac{100\,(M_2 - M)}{M_1 - M}$

where

M_2 = mass, in g, of the dish with the ash;

M = mass, in g, of the empty dish; and

M_1 = mass, in g, of the dish with the dried material (*see* M_2 in **C-2.1**).

ANNEX E

[*Table* 1, *Item* (iii)]

DETERMINATION OF ACID INSOLUBLE ASH

E-1 REAGENTS

E-1.1 Dilute Hydrochloric Acid — approximately 5 N, prepared from concentrated hydrochloric acid (*see* IS 265 : 1962).

E-2 PROCEDURE

E-2.1 To the ash contained in the dish (*see* **D-1.1**) add 25 ml of dilute hydrochloric acid, cover with a watch-glass and heat on a waterbath for 10 minutes. Allow to cool and filter the contents of the dish through Whatman filter paper No. 42 or its equivalent. Wash the filter paper with water until the washing are free from the acid and return it to the dish. Keep it in an air-oven maintained at 105 ± 2°C for about three hours. Ignite in a muffle furnace at 600 ± 20°C for one hour. Cool the dish in a desiccator and weigh. Heat again at 600 ± 20°C in the muffle furnace for 30 minutes. Cool the dish in the desiccator and weigh. Repeat the process of heating for 30 minutes, cooling and weighing till the difference in mass between two successive weighings is less than one milligram. Note the lowest mass.

E-3 CALCULATION

E-3.1 Acid insoluble ash (on dry basis), percent by mass $= \frac{100\,(M_2 - M)}{M_1 - M}$

where

M_2 = mass, in g, of the dish with the acid insoluble ash;

M = mass, in g, of the empty dish; and

M_1 = mass, in g, of the dish with the dried material (*see* M_2 in **C-2.1**).

ANNEX F

[*Table* 1, *Item* (iv)]

DETERMINATION OF TOTAL PROTEIN

F-1 APPARATUS

F-1.1 A recommended apparatus, as assembled, is shown in Fig. 1.

F-1.1.1 *Description*

The assembly consists of a round bottom flask *A* of 1 000 ml capacity fitted with a rubber stopper through which passes one end of the connecting bulb tube *B*. The other end of the bulb tube is connected to the condenser *C* which is attached by means of a rubber tube to a dip tube *D* which dips into a known quantity of standard sulphuric acid contained in a beaker *E* of 250 ml capacity.

F-1.2 Kjeldahl Flask — of capacity 500 ml

F-2 REAGENTS

F-2.1 Anhydrous Sodium Sulphate

F-2.2 Copper Sulphate

F-2.3 Concentrated Sulphuric Acid — sp gr 1·84

F-2.4 Sodium Hydroxide Solution — Dissolve about 225 g of sodium hydroxide in 500 ml of water.

F-2.5 Standard Sulphuric Acid — 0·1 N.

F-2.6 Methyl Red Indicator Solution — Dissolve one gram of methyl red in 200 ml of rectified spirit (95 percent by volume).

F-2.7 Standard Sodium Hydroxide Solution — 0·1 N.

F-3 PROCEDURE

F-3.1 Transfer carefully about one gram of the prepared sample (*see* **C-1.1**) accurately weighed, to the Kjeldahl flask, taking precautions to see that particles of the material do not stick on to the neck of the flask. Add about 10 g of anhydrous sodium sulphate, about 0·2 to 0·3 g of copper sulphate and 20 ml of concentrated sulphuric acid. Place the flask in an inclined position. Heat below the boiling point of the acid until frothing ceases. Increase heat until acid boils vigorously and digest for 30 minutes after the mixture becomes clear and

pale green or colourless. Cool the contents of the flask. Transfer quantitatively to the round bottom flask, with water, the total quantity of water used being about 200 ml. Add with shaking a few pieces of pumice stone to prevent bumping. Add about 50 ml of the sodium hydroxide solution (which is sufficient to make the solution alkaline) 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. Assemble the apparatus taking care that the dip tube extends below the surface of the standard sulphuric acid contained in the beaker. Mix the contents of the flask by shaking and distil until all ammonia has passed over into the standard sulphuric acid. Shut off the burner and immediately detach the flask from the condenser. Rinse the condenser thoroughly with water into the beaker. Wash the dip tube carefully so that all traces of the condensate are transferred to the beaker. When all the washings have drained into the beaker, add two or three drops of methyl red indicator solution and titrate with the standard sodium hydroxide solution.

F-3.2 Carry out a blank determination using all reagents in the same quantities but without the material to be tested.

F-4 CALCUATION

F-4.1 Total protein (on dry basis), percent by mass $= \frac{798\,(B-A)\,N}{M_1\,(100-M)}$

where

B = volume, in ml, of the standard sodium hydroxide solution used to neutralize acid in the blank determination;

A = volume, in ml, of the standard sodium hydroxide solution used to neutralize the excess of acid in the test with the material;

N = normality of the standard sodium hydroxide solution;

M_1 = mass, in g, of the prepared material taken for the test; and

M = moisture, percent by mass of the material (*see* **C-2.1**).

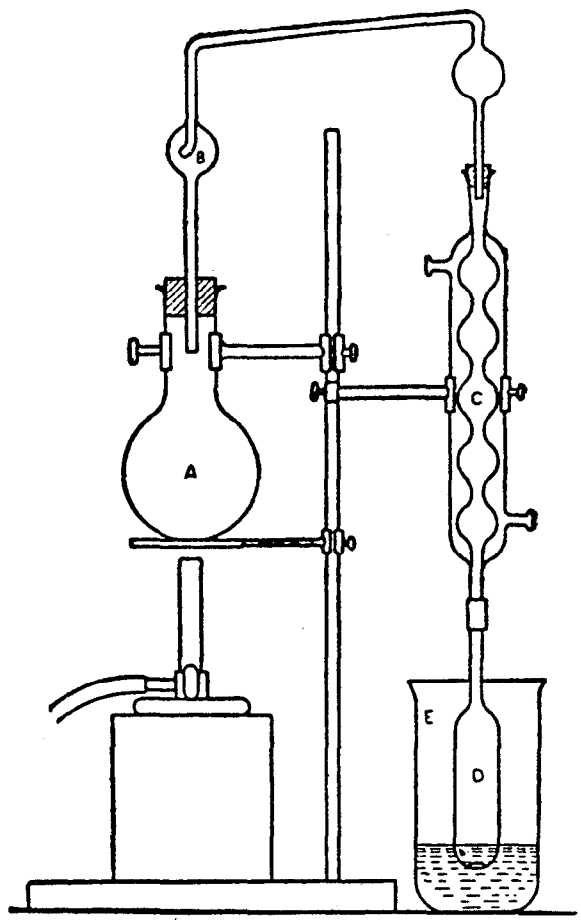

FIG. 1 APPARATUS FOR DETERMINATION OF PROTEIN

ANNEX G

[*Table* 1, *Item* (v)]

DETERMINATION OF TOTAL SOLIDS IN GRUEL

G-1 APPARATUS

G-1.1 Lipless Beaker — tall-form, of capacity 500 ml.

G-2 PROCEDURE

G-2.1 Take 250 ml water in the lipless beaker and heat over hot-plate or any suitable burner till water boils. Introduce 25 g of the material (previously broken into about 10 mm lengths in case of long goods) and stir thoroughly with a glass rod. Cook for 10 minutes with occasional stirring. At the end of 10 minutes allow the material to drain for five minutes. Measure the volume of gruel collected. Pipette out 20 ml of the gruel, after stirring well to give an even distribution of the solid content, into a tared petri dish and evaporate to dryness on a water-bath. Transfer the petri dish to a hot air-oven maintained at 105 ± 2°C and dry to constant mass.

G-3 CALCULATION

G-3.1 Total solids in gruel, percent by mass $= \frac{(M_2 - M_1)V}{5}$

where

M_2 = mass, in g, of petri dish with total solids,

M_1 = mass, in g, of empty petri dish, and

V = volume of gruel in ml.

ANNEX H

[*Table* 1, *Item* (vi)]

DETERMINATION OF ACIDITY

H-1 REAGENTS

H-1.1 Sodium Hydroxide — 0·1 N solution.

H-1.2 Phenolpthalein Indicator Solution

H-2 PROCEDURE

H-2.1 Grind 10 grams of the product and add 100 millilitres of distilled water (start the test from within an hour from the grinding). Leave for one hour, stirring 3 times for two minutes each time, at approximately equal intervals. Titrate with 0·1 N solution of NaOH using phenolpthalein as an indicator.

H-3 CALCULATION

H-3.1 Free acidity (ml of 1 N NaOH) $= \frac{10\,000 \times V \times N}{M}$

where

V = volume, in ml, of standard sodium hydroxide solution used in the titration;

N = normality of sodium hydroxide solution; and

M = mass, in g, of the sample taken.

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(Second Revision)

(*Page* 1, *clause* 3.2.1.3, *line* 2) — Insert 'or of slightly curved or bend product' between the words 'rods' and 'of'.

(FAD 16)

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