

**PAKISTAN STANDARD SPECIFICATION  
FOR  
BISCUITS**



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**PAKISTAN STANDARDS AND QUALITY CONTROL AUTHORITY  
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0. **FOREWORD**

0.1 This Pakistan Standard Specification was adopted by the Pakistan Standards & Quality Control Authority on **03-03-2021** after the finalized by the Cereal Pulses & Their Products Technical Committee had been approved by the National Standards Committee for Agriculture & Food Products.

0.2 In order to keep abreast of the Progress in Industry the Pakistan Standards are subject to periodical review suggestions for improvement shall always be welcomed and put to the relevant committee for its consideration.

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# SAARC STANDARD

## SARS 0006:2017

First edition  
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### BISCUITS — SPECIFICATION

ICS 67.060

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## FOREWORD

The South Asian Regional Standards Organization (SARSO) is a Specialized Body of South Asian Association for Regional Cooperation (SAARC) aimed to achieve and enhance coordination and cooperation among SAARC Member States in the fields of standardization and conformity assessment and to develop harmonized Standards for the South Asian region to facilitate intra-regional trade and to have access in the global market. The Member States of SAARC are Afghanistan, Bangladesh, Bhutan, India, Maldives, Nepal, Pakistan and Sri Lanka. The Agreement on the establishment of SARSO entered into force with effect from 25 August 2011 after ratification by all Member States of SAARC.

The National Standards Bodies of the SAARC Member States participate in the development of SAARC Standards (SARS) through the Sectoral Technical Committees (STCs). The SARS are developed through consensus and are drafted in accordance with the editorial rules of the SARSO Directives, Part 2.

The SARSO Secretariat is the guardian of the authoritative versions of the SAARC Standards and is responsible for keeping master texts of SAARC Standards, both in hard and soft form. The Member States are responsible for making SAARC Standards available for sale, distribution, etc at the national level. In accordance with the 'SAARC Agreement on Implementation of Regional Standards', the approval of a SAARC Standard implies that Member States have an obligation to give it the status of a National Standard.

This SAARC Standard was considered by the Technical Management Board and approved by the Governing Board of SARSO on the recommendation of the STC-01: Food and Agriculture Products.

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## INTRODUCTION

The term 'Biscuit' covers a large variety of sweet, savoury, salted, filled and coated biscuits. It is difficult to classify biscuits based on chemical composition and processing methodologies due to overlap. However, varietal differences can be distinguished well by their sensory attributes.

This SAARC Standard mainly lays down essential requirements to which biscuits of the different types should conform.

This SAARC Standard does not cover the requirements for wafer biscuits, sugar-free biscuits and diet biscuits.

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, as per the relevant national Standards of the respective Member State. The number of significant places retained in the rounded off value should be the same as that of the specified value in the relevant referred Standard.

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SAARC Standard

## BISCUITS - SPECIFICATION

### 1 SCOPE

1.1 This Standard specifies the requirements, methods of sampling and test for biscuits baked from dough containing essential ingredients (see 3) with or without the addition of optional ingredients (see 4).

1.2 This Standard does not cover wafer biscuits, sugar-free biscuits, diet biscuits and Special purpose biscuits including High Energy biscuits.

### 2 NORMATIVE REFERENCES

The Standards referred in the text contain provisions which through reference in this text constitute provisions of this Standard. At the time of publication, the editions indicated were valid. All Standards are subject to revision and parties to agreements based on this Standard are encouraged to investigate the possibility of applying the most recent editions of the Standards referred in the text.

ISO 4833-1:2013 *Microbiology of the food chain -- Horizontal method for the enumeration of microorganisms - Part 1: Colony count at 30 degrees C by the pour plate technique*

ISO 21527-2:2008 *Microbiology of food and animal feeding stuffs -- Horizontal method for the enumeration of yeasts and moulds -- Part 2: Colony count technique in products with water activity less than or equal to 0.95*

ISO 4831:2006 *Microbiology of food and animal feeding stuffs -- Horizontal method for the detection and enumeration of coliforms -- Most probable number technique*

ISO 7251:2005 *Microbiology of food and animal feeding stuffs -- Horizontal method for the detection and enumeration of presumptive Escherichia coli -- Most probable number technique*

ISO 6888-1:1999 *Microbiology of food and animal feeding stuffs -- Horizontal method for the enumeration of coagulase-positive staphylococci (Staphylococcus aureus and other species) -- Part 1: Technique using Baird-Parker agar medium*

ISO 6579:2002 *Microbiology of food and animal feeding stuffs -- Horizontal method for the detection of Salmonella spp.*

### 3 ESSENTIAL INGREDIENTS

The following materials shall be used in the preparation of biscuit dough.

#### 3.1 Wheat Flour

#### 3.2 Fat or Shortening

The following materials, singly or in combination shall be used:

Hydrogenated vegetable oil, bakery shortening, refined edible vegetable oils, butter oil, butter, Ghee and margarine.

#### 3.3 Potable Water as per WHO Guidelines for drinking water quality.



### 3.4 Leavening Agents

- Baking powder
- Ammonium bicarbonate
- Sodium bicarbonate
- Ammonium carbonate
- Active baker's yeast (*Saccharomyces cerevisiae*)
- Sodium acid pyrophosphate
- Any other approved aerating agent

## 4 OPTIONAL INGREDIENTS

4.1 In addition to the essential ingredients specified under clause 3, any of the following ingredients may be used in the preparation of biscuits.

### 4.1.1 Cereals and Cereal Products

- Whole wheat meal
- Wheat flour
- Semolina
- Wheat bran, edible
- Wheat germ
- Maize germ
- Barley powder/flour
- Oats/Oat flour, edible
- Gram flour
- Rice/rice flour
- Malt flour
- Any other edible cereal

### 4.1.2 Oil Seed Products

- Soya bean flour, full fat, medium fat or solvent extracted low fat
- Peanuts
- Peanut butter
- Edible groundnut flour. High protein mixes for use as food supplement. Other edible oil seed flours, protein isolates and concentrates.

### 4.1.3 Edible Starches/modified starches

- Tapioca
- Sweet potato
- Arrowroot starch
- Maize starch
- Tapioca starch
- Rice starch
- Potato starch
- Other starches

### 4.1.4 Milk and Milk Products

- Casein
- Milk powder

- Skimmed milk powder
- Butter milk and its solids
- Liquid milk
- Condensed milk
- Cheese
- Whey solids
- Malted milk food
- Dairy cream

#### 4.1.5 Sugars

- Sugars (sucrose)
- Glucose/glucose syrup
- Fructose/Fructose Syrup
- Dextrose/Dextrose Syrup
- Jaggery and Khandsari
- Molasses (edible), cane
- Lactose
- Malt extract
- Invert syrup
- Golden syrup/treacle
- Honey
- Sugar alcohol
- Any other permitted sugar products

#### 4.1.6 Nuts, Fruits and Fruit Products

- Desiccated coconut
- Dry fruits
- Edible nuts
- Pectin
- Fruit jams
- Dehydrated fruits

#### 4.1.7 Spices and Condiments

- Ginger
- Chilli
- Pepper
- Saffron
- Carom seeds (Ajowan)
- Cardamom
- Cumin
- Cinnamon
- Other permitted spices under food regulations of SAARC Member States.

#### 4.1.8 Miscellaneous

- Coffee products
- Cocoa products
- Chocolate
- Edible vegetables/Vegetable products
- Soluble starch phosphate

- Edible salt
- Caramel
- Egg and egg products
- Dietary fibers
- Oligofructose

#### 4.1.9 Food Processing Aids and Food Additives

**4.1.9.1** Flavours, flavouring agents, flavour improvers and fixers, as permitted under respective National Food Regulations of Member States.

**4.1.9.2** Colouring matter and preservatives, as permitted, under respective National Food Regulations of Member States.

**4.1.9.3** Antioxidants, emulsifying and stabilizing agents, as permitted under respective National Food Regulations of Member States.

**4.1.9.4 Enzymes and gluten conditioners** - Proteolytic enzymes and amylases, sodium bisulphite and sodium metabisulphite, L-cysteine limited by GMP.

**4.1.9.5 Flour improvers**, as permitted under food regulations of SAARC Member States.

#### 4.1.10 Nutrients

- Vitamins
- Proteins, protein isolates and concentrates
- High protein mixes for use as food supplement
- Minerals
- Amino acids
- Potassium iodide

#### 4.1.11 Mould Inhibitors/Acidity Regulators

- Acetic acid or lactic acid
- Vinegar or any other permitted acidity regulators under food regulations of SAARC Member States.

*NOTE: All ingredients and additives should comply to the relevant National Standards of Member States or relevant Codex Standards in case of non availability of National Standards*

## 5 TYPES OF BISCUITS

On the basis of sensory attributes, the different varieties can be broadly classified as given below:

Type I	Sweet
Type II	Semi-sweet
Type III	Crackers
Type IV	Cookies
Type V	Savoury
Type VI	Salty
Type VII	Specialty Biscuits

### 5.1 Sweet

This is the most common variety of biscuits where fat and sugar levels are generally high. This variety generally provides a comparatively shorter bite, sweet taste.

## 5.2 Semi-sweet

Semi-sweet type of products has comparatively low level of sweetness, in general. The product is harder in texture and generally low in fat content.

## 5.3 Crackers

Crackers are biscuits which in general show a typical flaky inner layers. These products can be fermented or non-fermented types, oil-dipped/sprayed or not and they may or may not be sprinkled with salt, sugar or flavours.

## 5.4 Cookies

Cookies are a group of products which are the shortest in bite compared to general/common sweet varieties. These may be further enriched by incorporation of nuts, dry fruits, chocolates, etc. This product has a relatively high sugar and fat content as compared to the sweet variety.

## 5.5 Savoury

Savoury biscuits are biscuits which may contain spices and/or salt. The salt and/or spices may also be sprayed on the biscuits with/without oil.

## 5.6 Salty

Salted biscuits are biscuits which are sprinkled with salt or spread with oil mixed with salt.

## 5.7 Specialty Biscuits

There are several other products termed as specialty biscuits which include different varieties of biscuits, such as filled biscuits or coated biscuits, cream sandwich biscuits.

### 5.7.1 Filled Biscuits

Filled biscuits shall be biscuits sandwiched or center filled with a filling of either cream, jam, jelly, marshmallow, chocolates, caramel, figs, raisins, cheese or like with sweet type shells, semi-sweet type shells, cracker shells or even cookie type shells.

*NOTE: For the purpose of this Standard 'cream' means basically a homogeneous preparation of hydrogenated vegetable oil or bakery shortening, icing sugar, pulverized sugar, permitted flavours and permitted food colours with or without other ingredients in small proportions.*

### 5.7.2 Coated Biscuits

Coated biscuits shall be biscuits as such or with filling in between (see 5.7.1), but coated with chocolate or caramel or other suitable enrobing materials. This group will also include products with whole meal.

## 6 REQUIREMENTS

### 6.1 General Requirements

The biscuits shall be properly baked so that they are crisp and have uniform texture and appearance. The design, if any, on the biscuits should be clear. They shall have an agreeable flavour typical of well-baked biscuits of different types and shall be free from any soapy or bitter after-taste. The biscuits shall be free from fungal growth, insect infestation, objectionable taste and odour. The biscuits shall also be free from any other harmful foreign matter.

*NOTE: The appearance, taste, odour and texture are determined by organoleptic tests.*

6.2 Biscuits shall be manufactured under hygienic conditions as detailed in the respective National Standards. In case such National Standards are not available, the Codex Alimentarius Commission Standards on the hygienic conditions shall be followed.

6.3 Biscuits shall also conform to the requirements given in Table 1.

**Table 1- Requirements for Biscuits  
(Clause 6.3)**

SI.No.	Characteristic	Requirement	Method of test, Ref. to
(1)	(2)	(3)	(4)
i)	Moisture, percent by mass, Max.	5.0*	Annex B
ii)	Acid insoluble ash, (on dry basis), percent by mass, Max.	0.05	Annex C
iii)	Acidity of extracted fat (as oleic acid), percent by mass. Max.	1.0	Annex D
iv)	Lead (as Pb), mg/kg, Max.	2.0	Annex E AOAC 999.11 (as alternate method)
v)	Arsenic (as As), mg/kg Max.	1.1	AOAC 986.15
*In case of filled biscuits, maximum of 7.0 percent by mass may be allowed and for cookies maximum of 6.0 percent by mass may be allowed.			

6.4 Sandwiched, filled and/or coated biscuits shall comply with the microbiological limits given in Table 2, when tested according to the methods given in column 7 of the Table 2.

**Table 2 - Microbiological Limits  
(Clause 6.4)**

Sl. No.	Test organism	n	C	Limit		Method of test
				m	M	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
1	Aerobic plate count	5	2	10 <sup>3</sup>	10 <sup>4</sup>	ISO 4833-1 : 2013
2	Yeast & Mould count, per g	5	2	10 <sup>2</sup>	10 <sup>3</sup>	ISO 21527-2 : 2008
3	Coliforms, per g (MPN)	5	2	10	10 <sup>2</sup>	ISO 4831 : 2006
4	<i>E. coli</i> , per g (MPN)	5	0	0	-	ISO 7251 : 2005
5	<i>Staphylococcus aureus</i> , per g	5	2	10	10 <sup>2</sup>	ISO 6888-1 : 1999
6	<i>Salmonella</i> , per 25 g	5	0	0	-	ISO 6579 : 2002

where

$n$  is the number of samples to be tested;

$c$  is the maximum allowable number of samples yielding values between  $m$  and  $M$ ;

$m$  is the limit below which the count is acceptable for any sample; and

$M$  is the limit above which the count is unacceptable for any sample.

*NOTE: Adoption of the ISO Standards referred in column 7 as SAARC Standards is under consideration by SARSO. Till the time these SAARC Standards are not published, Member States may also use their National Standards on these respective subjects in case these are identical with ISO Standards.*

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## 7 PACKAGING

Biscuits shall be packaged in clean, sound containers, made of tin plate, cardboard, paper or other material agreed upon between the purchaser and the vendor, in such a way as to protect them from breakage, contamination, absorption of moisture and seepage of fat from the biscuits into the packing materials and should not impart any objectionable odour and taste to the biscuits. The biscuits shall not come in direct contact with the packaging materials other than grease proof or sulphite paper, cellulose film, aluminium foil laminates food grade plastics conforming to the relevant National Standard or any other non-toxic packing material which may be covered with a moisture-proof film, waxed paper or moisture-proof laminates, the inner layer coming in contact with the biscuits shall be of food-grade quality, or coated paper. The biscuits in tins should not come into direct contact with unlacquered metal walls.

## 8 LABELLING AND MARKING

8.1 The following particulars shall be clearly and indelibly marked on the label of each container/packet:

- a) name of the product;
- b) trade name, if any;
- c) name and address of the manufacturer including the country of origin;
- d) batch/lot or code number;
- e) net mass in grams or kilograms;
- f) list of ingredients, in descending order of their composition by mass;
- g) the statement with respect to addition of permitted colours and flavours;
- h) any Permitted Food Additive's Name, Class and name of International Numbering System (INS) number;
- i) month and year of manufacture;
- j) the words 'Best before ..... ' (month and year to be indicated); and
- k) any other requirements as specified under the respective national regulations.

### 8.2 Certification Marking

The product may also be marked with the Standard Mark of conformity.

## 9 SAMPLING

The method of drawing the representative samples of the biscuits and the criteria for conformity shall be as specified in Annex A.

**Annex A**  
**(Normative)**  
**(Clause 8)**

**METHODS OF SAMPLING**

**A-1 General Requirements of Sampling**

In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

**A-1.1** Sample shall be taken in a protected place which is free of odour and not exposed to damp air, dust or soot.

**A-1.2** Precautions shall be taken to protect the samples, the lots being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**A-1.3** The samples shall be placed in air tight, clean and dry glass, metallic containers suitably lacquered or lined and stored in such a manner that the material is not unduly affected.

**A-1.4** Each container containing the samples shall be marked with full details of sampling, such as, date and time of sampling, batch or code number, name of the manufacturer and other relevant particulars.

**A-1.5** The samples shall be stored at room temperature.

**A-1.6** Sampling shall be done by a person agreed to between the purchaser and the vendor, and if desired by them, in the presence of the purchaser or their representative and the vendor.

**A-1.7** Unless otherwise agreed to between the purchaser and the vendor, sampling shall be done at the bakery.

**A-1.8** Samples shall be tested within as short a time as possible after their drawl, preferably within 24 hours of sampling.

**A-2 Lot Inspection**

**A-2.1 Scale of Sampling**

**A-2.2.1 Lot**

All the containers in a single consignment belonging to the same batch of manufacture shall constitute a lot.

**A-2.2.2** For ascertaining the conformity of the material to the requirements of the specification, samples shall be tested from each lot separately.

**A-2.2.3** The number of packages/containers to be sampled from a lot shall depend upon the size of the lot and shall be in accordance with Table 3.



**Table 3 - Number of Containers to be selected  
(Clauses A-2.2.3 and A-2.3.1)**

Lot Size	Sample Size
(1)	(2)
Up to 50	3
51 to 150	4
151 to 300	5
301 to 500	6
501 to 1 000	8
1 001 and above	10

**A-2.2.4** The containers shall be selected from the lot at random following a suitable procedure to ensure randomness.

### **A-2.3 Test Samples and Referee Samples**

**A-2.3.1** From each lot, draw the number of containers of biscuits as given in column 2 of Table 3. These containers shall be opened and mixed. From each selected container, about 600 g of biscuits shall be taken from different packets/portions. This quantity of 600 g shall be, after proper mixing, divided into two equal parts of 300 g biscuits each. The first part of 300 g shall be divided into three equal parts of 100 g each. One of them shall be for the purchaser, another for the vendor and the third for the referee. These biscuits shall be packed in air-tight dry containers and labeled with the particulars as given in A-1.4. Each of these containers of 100 g shall constitute individual test sample. These individual test samples shall be separated into three identical sets of test samples in such a way that each set has a sample representing each selected container.

**A-2.3.2** The second part of 300 g shall be suitably powdered and divided into two equal parts of 150 g each. While powdering the biscuits, the following precautions shall be observed:

- a) a sample of plain biscuits shall be ground as quickly as possible;
- b) the cream, chocolate, jam, jelly or any other filling between biscuits should be removed by gently scraping before powdering the sample;
- c) as far as possible, the coating and fillings should be removed before powdering the biscuits except for microbiological requirements; and
- d) as the biscuits are highly hygroscopic the preparation of the sample should be done very quickly, preferably in a closed and dry place.

**A-2.3.3** Out of these two portions of 150 g each, the first portion shall be divided into three equal parts of 50 g each. These parts shall be transferred immediately to clean, air tight and dry containers which are then sealed and labeled with the particulars as given in A-1.4. Each of these sample containers of 50 g shall constitute individual test sample to be tested for moisture. These individual samples shall be separated into identical sets of test samples in such a way that each set has a sample representing each selected container. One of these sets shall be marked for the purchaser, another for the vendor, and the third for the referee.

**A-2.3.4** Out of the second portion of 150 g, approximately equal quantity of material shall be taken from each container selected from the lot. It shall be thoroughly mixed so as to constitute the composite sample of not less than 450 g. This shall be divided into three equal parts. These parts shall be transferred to clean, air tight and dry containers which are then sealed and labeled with all the particulars given in A-1.4. One of them shall be for the purchaser, another for the vendor and the third for the referee.

**A-2.3.5** Referee samples shall consist of a set of individual test samples (see A-2.3.1), a set of individual moisture samples (see A-2.3.3) and the composite sample (see A-2.2.4) and shall bear the seals of the purchaser and the vendor. These shall be kept at a place agreed to between the two so as to be used in case of a dispute.

#### **A-2.4 Number of Tests and Criteria for Conformity**

**A-2.4.1** The general requirements shall be tested on each of the individual test samples (see A-2.3.1).

**A-2.4.2** The moisture content shall be tested on individual moisture samples (see A-2.3.3).

**A-2.4.3** Tests for the determination of remaining characteristics, such as, acid insoluble ash and acidity of extracted fat shall be conducted on the composite sample (see A-2.3.4).

**A-2.4.4** The lot shall be declared as conforming to the requirements of the relevant material specification if all the test results on individual and composite samples meet the relevant specification requirements.

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**Annex B**  
**(Normative)**  
**[Table 1, Item (i)]**

**DETERMINATION OF MOISTURE**

**B-1 General**

Two methods for the determination of moisture content in biscuits have been given. Method I, namely, the moisture meter method may be used as a routine method after calibrating it with the oven method given under Method II.

**B-2 Method I**

This method involves the use of electrical moisture meters for rapid estimation of moisture in the biscuit samples. Various moisture meters are available. The conversion tables for their use are available with the equipment, but the instruments shall be calibrated under the conditions of actual use in comparison with the oven method given under Method II.

**B-3 Method II**

**B-3.1 Apparatus**

**B-3.1.1** Moisture Dish, made of porcelain, silica, glass or aluminium.

**B-3.1.2** Oven, Electric, maintained at  $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$ .

**B-3.1.3** Desiccator

**B-3.2 Procedure**

Weigh accurately about 5 g of the prepared sample in the moisture dish, previously dried in the oven and weighed. Place the dish in the oven maintained at  $105^{\circ}\text{C} \pm 2^{\circ}\text{C}$  for 4 h. Cool in the desiccator and weigh. Repeat the process of drying, cooling and weighing at 30 min interval until the difference between the two consecutive weighings is less than one milligram. Record the lowest mass.

**B-3.3 Calculation**

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

where

$M$  is mass, in g, of the empty dish;

$M_1$  is mass, in g, of the dish with the material before drying; and

$M_2$  is mass, in g, of the dish with the material after drying to constant mass.

**Annex C**  
**(Normative) [Table**  
**1, Sl. No. (ii)]**

**DETERMINATION OF ACID INSOLUBLE ASH**

**C-1 Apparatus**

- C-1.1 Dish** – silica or porcelain.
- C-1.2 Muffle Furnace** – maintained at  $600^{\circ}\text{C} \pm 20^{\circ}\text{C}$ .
- C-1.3 Water-Bath**
- C-1.4 Desiccator**

**C-2 Reagent**

- C-2.1 Dilute Hydrochloric Acid** – approximately 5 N, prepared from concentrated hydrochloric acid.

**C-3 Procedure**

Weigh accurately about 20 g of biscuit powder in the dish and ash in the muffle furnace at  $600^{\circ}\text{C} \pm 20^{\circ}\text{C}$  until light grey ash is obtained. Remove the dish from the furnace and allow it to cool at room temperature. Add 25 ml of the hydrochloric acid to the dish, cover with a watch-glass and heat on the water-bath for 10 min. Mix the contents with the tip of a glass rod and filter through Whatman filter paper No. 42 or its equivalent. Wash the filter paper with water until the washings are free from acid when tested with a blue litmus paper. Return the washed filter paper to the dish for ashing in the muffle furnace as above. Cool the dish in a desiccator and weigh. Again ignite the dish for half an hour in the furnace, cool and weigh. Repeat this operation until the difference between successive weighings is less than 1 mg. Filter 25 ml of the hydrochloric acid through a blank filter paper, wash, ash and weigh it as in the case of acid insoluble ash. Subtract its mass from the mass of insoluble ash of the sample.

**C-4 CALCULATION**

**C-4.1** Acid insoluble ash, percent by mass = 
$$\frac{100 (M_1 - M)}{M_2}$$

where

$M$  is mass, in g, of the empty dish in which the sample is taken for ashing;  
 $M_1$  is mass, in g, of the dish containing acid insoluble ash (see Note) and  
 $M_2$  is mass, in g, of the sample.

*NOTE: Correct the acid Insoluble ash mass for the blank of filter paper, if any*

**C-4.2** Acid insoluble ash, percent by mass (on dry basis) = 
$$\frac{A \times 100}{100 - M}$$

where

$A$  is acid insoluble ash, percent by mass (see C-4.1); and  
 $M$  is percentage of moisture in the biscuit (see B-3.3).

**Annex D**  
**(Normative) [Table**  
**1, Sl. No. (iii)]**

**DETERMINATION OF ACIDITY OF EXTRACTED FAT**

**D-1 Apparatus**

**D-1.1 Soxhlet Apparatus** – with a 250 ml flat bottom flask.

**D-2 Reagents**

**D-2.1 Phenolphthalein Reagent** [1.0 Percent in Ethanol (95 Percent)]

**D-2.2 0.1 M Potassium Hydroxide Solution**

**D-2.3 Ether-Ethanol (95 Percent) Stock Solution**

**D-2.4 Petroleum Ether (Boiling Point 40°C - 80°C)** – Equal volumes of ethanol-ether which has been neutralized to phenolphthalein with 0.1 M potassium hydroxide.

**D-3 Procedure**

**D-3.1** Weigh accurately a mass of biscuit powder containing more than 3.0 g of fat and transfer it to the thimble and plug it from the top with extracted cotton and filter paper.

*NOTE: In case of filled and coated biscuits, the mass of the biscuits includes the filled and coated material also.*

**D-3.2** Dry the thimble with the contents for 15 to 30 min at 100°C in an oven. Extract the fat with petroleum ether (see D-2.4) in the Soxhlet apparatus for 3 to 4 h and evaporate off the solvent in the flask on a water-bath. Remove the traces of the residual solvent by keeping the flask in the hot air oven for about half an hour. Cool the flask. Weigh accurately about 3.0 g of extracted fat in a tared 250 ml flat-bottomed flask and add 50 ml of mixture of equal volume of alcohol and ethanol (see D-2.3). If the test specimen does not dissolve in the cold, connect the flask with a suitable condenser and warm slowly with frequent shaking, until the specimen dissolves. Add 1 ml of phenolphthalein reagent (see D-2.1) and titrate the contents to a distinct pink colour with the potassium hydroxide solution taken in a 10 ml micro burette. If the contents of flask become cloudy, during titration, add another 50 ml of the reagent (see D-2.1) and continue titration. Make a blank titration of the 50 ml reagent. Subtract from the titre of the fat, the blank titre.

**D-4 Calculation**

Acidity of extracted fat (as oleic acid), percent by mass =  $\frac{1.41 \times V}{M}$

where

$V$  is volume of 0.1 M potassium hydroxide solution used in titration after subtracting the blank; and  
 $M$  is mass, in g, of extracted fat taken for the titration.

**Annex E**  
**(Normative) [Table**  
**1, Sl. No. (iv)]**

**METHOD FOR DETERMINATION OF LEAD BY ATOMIC ABSORPTION**  
**SPECTROPHOTOMETER**

**E-1 Outline of the Method**

The sample is brought into the solution by suitable treatment with acids or acid combinations, diluted with distilled water, filtered and suitable dilutions are made for aspiration into the air acetylene flame. The Standard solution is made in the same way for calibration. The most sensitive lead line is 217.0 nm, however, other lines suitable for higher concentration can also be used.

**E-2 Interference**

**E-2.1** No cationic interference has been reported for the air acetylene flame. A number of anionic interferences have been reported. Phosphates, carbonates, iodides, fluorides and acetates suppress lead absorbance significantly at concentrations ten times greater than lead. These interferences can be largely overcome by addition of EDTA solution so as to give 0.1 molar concentration of EDTA in the final solution for aspiration.

**E-2.2** At 217.0 nm wavelength, non-atomic species in the flame absorb strongly. Where the sample has a high concentration of dissolved solids, it is necessary to use background correction with the help of hydrogen continuum lamp.

**E-3 Apparatus**

**E-3.1** Atomic absorption spectrophotometer provided with background corrector and having following parameters:

- |                                 |   |   |
|---------------------------------|---|---|
| a) Lamp current                 | - | depending on the lamp and instrument used |
| b) Support                      | - | air                                       |
| c) Fuel                         | - | acetylene                                 |
| d) Flame stoichiometry          | - | oxidizing                                 |
| e) Wavelength and working range |   |   |

Wavelength (nm)	Band Pass (nm)	Working range, µg/ml
217.0	1.0	5 to 20
283.3	0.2	10 to 40
261.4	0.2	200 to 800
202.2	0.2	250 to 1 000
205.3	0.2	2 000 to 8 000

*NOTE: In case of multi-element hollow cathode lamp containing copper, the second sensitive line for lead, that is, 283.3 nm may be taken to avoid the interference of copper.*

**E-4 Reagents**

**E-4.1 Pure Lead Metal** – 99.99 percent.

**E-4.2 Concentrated Nitric Acid** – see National Standards of the Member States or any international/other National Standards

**E-4.3 Concentrated Hydrochloric Acid** – see National Standards of the Member States or any international/other National standards

**E-4.4 Standard Lead Solution** – Dissolve 1.0 g of lead in 1:1 nitric acid, dilute to one litre with distilled water to give 1000 µg/ml of lead.

## E-5 Sample Preparation

**E-5.1** The sample of biscuits is brought into solution by wet digestion with nitric acid/perchloric acid or dry ashing at 450°C and dissolving in nitric acid. Filtrate is made up to suitable known volume.

### E-5.2 Procedure

Optimize the response of instrument by adjustment of burner height and flame. Aspirate water to get zero absorption, when stable response is observed, aspirate standards (at least 4) and note down absorption. Aspirate sample to get absorption of the sample. Prepare calibration curve by plotting the net absorption value of the standard against concentration in µg/ml of lead. Locate the point of the sample absorption and calculate the concentration of lead in the sample.

### E-5.3 Calculation

$$\text{Lead as Pb, (mg/kg)} = \frac{C \times V}{M}$$

where

C is concentration of lead in µg/ml in final solution,  
V is volume in ml of final solution, and  
M is mass in g of the sample in final solution.

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