

**SRI LANKA STANDARD 915 : 1991**

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**SPECIFICATION FOR**  
**BENZOIC ACID, POTASSIUM BENZOATE AND**  
**SODIUM BENZOATE (FOOD GRADE)**

**SRI LANKA STANDARDS INSTITUTION**



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SODIUM BENZOATE (FOOD GRADE)

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Gr.11

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SRI LANKA STANDARD  
SPECIFICATION FOR BENZOIC ACID, POTASSIUM BENZOATE AND  
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**FOREWORD**

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1991-04-02, after the draft, finalized by the Drafting Committee on Food Additives had been approved by the Agricultural and Food Products Divisional Committee.

Benzoic acid, potassium benzoate and sodium benzoate are used as antimicrobial preservatives in the food industry. This specification is therefore formulated for the benefit of quality control exercises which are carried out to ensure the identity and purity of the products.

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

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 Food and Agriculture Organization and the Bureau of Indian Standards is gratefully acknowledged.

**1 SCOPE**

This specification prescribes the requirements, methods of sampling and test for benzoic acid (benzenecarboxylic acid, phenylcarboxylic acid), potassium benzoate (potassium salt of benzenecarboxylic acid, potassium salt of phenylcarboxylic acid) and sodium benzoate (sodium salt of benzenecarboxylic acid, sodium salt of phenylcarboxylic acid) of food grade.

**2 REFERENCES**

- CS 102 Presentation of numerical values.
- SLS 312 Determination of arsenic.
- SLS 428 Random sampling methods.
- SLS 467 Labelling of pre-packaged foods.

### 3 REQUIREMENTS

#### 3.1 General requirements

##### 3.1.1 Description

3.1.1.1 Benzoic acid shall be in the form of a white crystalline solid having a faint characteristic odour.

3.1.1.2 Potassium benzoate shall be in the form of a white crystalline powder.

3.1.1.3 Sodium benzoate shall be in the form of a white, almost odourless, crystalline powder or granules.

##### 3.1.2 Solubility

3.1.2.1 Benzoic acid shall be slightly soluble in water and freely soluble in chloroform and ethanol.

3.1.2.2 Potassium benzoate shall be freely soluble in water and soluble in ethanol.

3.1.2.3 Sodium benzoate shall be freely soluble in water and sparingly soluble in ethanol.

##### 3.1.3 Identification

###### 3.1.3.1 Benzoic acid

###### 3.1.3.1.a Melting range

The melting range of benzoic acid shall be between 121.5 °C to 125 °C (see Note) tested in accordance with the method described in A.1.1.

###### NOTE

The "melting range 121.5 °C to 123.5 °C" means that the corrected temperature at which the material is observed to form droplets should be at least 121.5 °C and the material should be completely melted at the corrected temperature of 123.5 °C.

###### 3.1.3.1.b Sublimation

Benzoic acid crystals shall be formed in the colder parts of the test tube without leaving a residue at the bottom, when tested in accordance with the method described in A.1.2.

3.1.3.1.c Reaction with ferric chloride

A buff coloured precipitate shall be formed when tested in accordance with the method described in A.1.3.

3.1.3.2 Potassium benzoate and sodium benzoate

3.1.3.2.a Melting range

The melting range of the precipitate obtained with dilute hydrochloric acid shall be between 121.5 °C to 123.5 °C (See Note under 3.1.3.1.a) when tested in accordance with the method described in A.2.1.

3.1.3.2.b Reaction with ferric chloride

A buff coloured precipitate shall be formed when tested in accordance with the method described in A.2.2.

3.1.3.2.c Reaction with sodium hydrogen tartrate

In the case of potassium benzoate a white crystalline precipitate shall be formed when tested in accordance with the method described in A.2.3.

3.1.3.2.d Reaction with uranyl zinc acetate

In the case of sodium benzoate a yellow crystalline precipitate shall be formed when tested in accordance with the method described in A.2.4.

3.2 Chemical requirements

Benzoic acid, potassium benzoate and sodium benzoate shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Column 6 of the table.

TABLE 1 - Chemical requirements for benzoic acid, potassium benzoate and sodium benzoate

Sl. No.	Characteristic	Requirement			Method of test
		Benzoic acid	Potassium benzoate	Sodium benzoate	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Benzoic acid, on dry basis, as $C_6H_5COOH$ , per cent by mass, min.	99.5	—	—	B.1
ii)	Potassium benzoate, on dry basis, as $C_6H_5COOK$ , per cent by mass, min.	—	99.0	—	B.2
iii)	Sodium benzoate, on dry basis, as $C_6H_5COONa$ , per cent by mass, min.	—	—	99.0	B.3
iv)	Loss on drying, per cent by mass, max.	0.5	—	1.5	Appendix C
v)	Sulfated ash, per cent by mass, max.	0.1	—	—	Appendix D
vi)	Readily carbonizable substances	To pass the test	To pass the test	To pass the test	Appendix E
vii)	Readily oxidizable substances	To pass the test	To pass the test	To pass the test	Appendix F
viii)	Chlorinated organic compounds, as Cl, per cent by mass, max.	0.07	0.07	0.07	Appendix G
ix)	Arsenic, mg/kg, max.	3	3	3	Appendix H
x)	Heavy metals, as lead, mg/kg, max.	10	10	10	Appendix J
xi)	Acidity and alkalinity	—	To pass the test	To pass the test	Appendix K

#### 4 PACKAGING AND MARKING

##### 4.1 Packaging

4.1.1 Benzoic acid, potassium benzoate and sodium benzoate shall be filled in air-tight, amber coloured containers which shall not contaminate the contents with metals or other impurities.



## 4.2.1 Marking

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

- (a) Name of the product, including the words "Food Grade";
- (b) Brand name or trade name, if any;
- (c) Net mass, in grams or in kilograms;
- (d) Name and address of the manufacturer and distributor, including the country of origin;
- (e) Batch or code number; and
- (f) Date of manufacture.

## NOTE

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

4.2.2 General guidelines for marking and labelling as given in SLS 647 shall be followed.

## 5 SAMPLING

## 5.1 Lot

In any consignment all the containers of the same size containing benzoic acid, potassium benzoate or sodium benzoate belonging to one batch of manufacture or supply shall constitute a lot.

## 5.2 Scale of sampling

5.2.1 Samples shall be tested from each lot for ascertaining its conformity to the requirements of this specification.

5.2.2 The number of containers to be selected from a lot shall be in accordance with Table 2.

TABLE 2 - Scale of sampling

Number of containers in the lot (1)	Number of containers to be selected (2)
Up to 15	2
16 to 25	3
26 to 50	5
51 to 100	8
101 and above	13

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

### 5.3 Number of tests

5.3.1 Each container selected as in 5.2.2 shall be inspected for packaging and marking requirements.

5.3.2 A sufficient quantity of material shall be drawn from each container selected as in 5.2.2 and mixed to form a composite sample. The composite sample thus obtained shall be tested for the requirements given in 3.1 and 3.2.

#### NOTE

*Test for the requirements given in 3.1 should be carried out only in case of dispute.*

## 6 METHODS OF TEST

Tests shall be carried out as prescribed in Appendices A to K of this specification.

## 7 CRITERIA FOR CONFORMITY

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

7.1 Each container inspected as in 5.3.1 satisfies the relevant requirements.

7.2 The test results on composite sample when tested as in 5.3.2 satisfy the relevant requirements.

### APPENDIX A IDENTIFICATION TESTS

#### A.1 BENZOIC ACID

##### A.1.1 *Determination of melting range*

##### A.1.1.1 *Apparatus*

A.1.1.1.1 Capillary tube, 0.10 mm to 0.15 mm of wall thickness and 0.9 mm to 1.1 mm of internal diameter.

A.1.1.1.2 Standard mercury glass thermometer

A.1.1.1.3 Melting point apparatus, provided with an appropriate liquid like paraffin or silicone oil and fitted with an auxiliary thermometer.

### A.1.1.2 Procedure

Dry the sample under the conditions specified for loss on drying (see Appendix C) before determining the melting range. If a temperature is not specified dry the sample for 24 hours in a desiccator.

Transfer the dried powder to a dry capillary tube (A.1.1.1.1) and pack the powder by gently tapping the tube on a hard surface so as to form a tightly packed column of 2 mm to 4 mm in height. Attach the capillary tube with its contents to a standard thermometer (A.1.1.1.2) so that the closed end is at the level of the middle of the bulb. Heat in the melting point apparatus (A.1.1.1.3) and regulate the rise of temperature during the first period to 3 °C per minute. When the temperature reaches 116.5 °C, adjust the rise of temperature of the apparatus to 1 °C to 2 °C per minute. Take the reading of the thermometer at which the material is observed to form droplets against the side of the tube and the temperature at which it is completely melted, as indicated by the formation of a definite meniscus.

### A.1.1.3 Temperature correction

Apply the emergent-stem correction to the temperature readings, as follows:

Before starting the determination of the melting range, attach the auxiliary thermometer so that the bulb touches the standard thermometer at a point midway between the graduation for the expected melting range and the surface of the heating material. When the material has melted, read the temperature on the auxiliary thermometer.

Calculate the correction to be added to the temperature reading of the standard thermometer from the following formula:

$$0.00016 N (T - t)$$

where,

N is the number of degrees of the scale of the standard thermometer between the surface of the heating material and the level of the mercury;

T is the temperature, in °C, of the standard thermometer; and

t is the temperature, in °C, of the auxiliary thermometer.

**A.1.2 Sublimation**

Place a small amount of sample in a dry test tube. Wrap a moistened filter paper around the test tube commencing from a height of about 40 mm from the bottom of the tube. Heat the tube over a low flame. Observe the formation of crystals.

**A.1.3 Reaction with ferric chloride**

**A.1.3.1 Reagents**

**A.1.3.1.1 Calcium carbonate**

**A.1.3.1.2 Ferric chloride, 90 g/l solution.**

**A.1.3.2 Procedure**

Warm gently about 0.1 g of the sample with 0.1 g of calcium carbonate (A.1.3.1.1) and 5 ml of water. Filter and add ferric chloride solution (A.1.3.1.2) to the filtrate. Observe the colour of the precipitate.

**A.2 POTASSIUM BENZOATE AND SODIUM BENZOATE**

**A.2.1 Determination of melting range.**

**A.2.1.1 Apparatus, listed under A.1.1.1**

**A.2.1.2 Reagent**

**A.2.1.2.1 Hydrochloric acid, dilute**

Prepare by diluting 236 ml of concentrated acid (rel. den. = 1.18) with sufficient water to make 1000 ml.

**A.2.1.3 Procedure**

Prepare 20 g/l solution of potassium benzoate or sodium benzoate and acidify with dilute hydrochloric acid (A.2.1.2.1). Filter and collect the precipitate on to the filter paper. Wash the precipitate with distilled water until free from chloride. Dry the precipitate as given in Appendix C.

Proceed as given in A.1.1.2.

### A.2.2 Reaction with ferric chloride

To a 10 per cent (m/m) solution of the sample in water add ferric chloride solution. Observe the colour of the precipitate.

### A.2.3 Reaction with sodium hydrogen tartrate

Acidify a 10 per cent (m/m) solution of the potassium benzoate in water with dilute hydrochloric acid and filter. Make the filtrate neutral with sodium hydroxide (approximately 1 mol/l). Add a saturated solution of sodium hydrogen tartrate and ethanol (1:1) and shake. Observe the formation of the precipitate.

### A.2.4 Reaction with uranyl zinc acetate

#### A.2.4.1 Reagents

A.2.4.1.1 Acetic acid, approximately 6 per cent (V/V).

A.2.4.1.2 uranyl zinc acetate solution

Dissolve 10 g of uranyl acetate,  $(\text{CH}_3\text{COO})_2\text{UO}_2 \cdot 2\text{H}_2\text{O}$ , with 50 ml of water and 5 ml of acetic acid (approximately 5 mol/l). Dissolve 30g of zinc acetate by heating with 30 ml of water and 3 ml of acetic acid. Mix the two solutions, allow to cool and filter.

#### A.2.4.2 Procedure

Acidify a solution of the sodium benzoate with dilute acetic acid (A.2.4.1.1) and filter if necessary. Add uranyl zinc acetate solution (A.2.4.1.2). Observe the formation of the precipitate.

## APPENDIX B DETERMINATION OF BENZOIC ACID, POTASSIUM BENZOATE AND SODIUM BENZOATE

### B.1 BENZOIC ACID

#### B.1.1 Reagents

B.1.1.1 *Ethanol*, previously neutralized using phenolphthalein.

B.1.1.2 *Sodium hydroxide*,  $c(\text{NaOH}) = 0.5 \text{ mol/l}$  solution.

### B.1.1.3 *Phenolphthalein*

Dissolve 0.2 g of phenolphthalein in 60 ml of 90 per cent (V/V) ethanol and add sufficient water to make 100 ml.

### B.1.2 Procedure

Weigh, to the nearest milligram, about 2.5 g of the sample, previously dried for 3 hours over sulfuric acid. Dissolve in 15 ml of warm ethanol (B.1.1.1). Add 20 ml of water and titrate with sodium hydroxide (B.1.1.2) using phenolphthalein as an indicator.

### B.1.3 Calculation

One millilitre of 0.5 mol/l solution of sodium hydroxide is equivalent to 61.06 mg of benzoic acid.

Benzoic acid, as  $C_6H_5O_2$ , per cent by mass =  $\frac{12.21 \times c \times V}{m}$

where,

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

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

V is the volume, in ml, of the sodium hydroxide solution required for the titration.

## B.2 POTASSIUM BENZOATE

### B.2.1 Reagents

B.2.1.1 *Hydrochloric acid*,  $c(HCl) = 0.1$  mol/l solution.

B.2.1.2 *Phenolphthalein indicator*

Dissolve 0.2 g of phenolphthalein in 60 ml of 90 per cent (V/V) ethanol and add sufficient water to make 100 ml.

B.2.1.3 *Ether*

B.2.1.4 *Bromophenol blue*

Dissolve 0.1 g of bromophenol blue in 100 ml of dilute ethanol (1:2 ratio) and filter if necessary.

B.2.1.5 *Hydrochloric acid*,  $c(HCl) = 0.5$  mol/l solution.

**B.2.2 Procedure**

Weigh, to the nearest 0.1 mg, about 2.5 to 3 g of the sample, previously dried at 105 °C to a constant mass. Dissolve in 50 g of water. Neutralize the solution, if necessary, with hydrochloric acid (B.2.1.1) using phenolphthalein (B.2.1.2) as an indicator. Add 50 ml of ether and a few drops of bromophenol blue (B.2.1.4) and titrate with hydrochloric acid (B.2.1.5), shaking constantly, until the colour of the indicator begins to change.

Separate the lower layer, wash the ethereal layer with 10 ml of water. Add the washings and an additional 20 ml of ether to the separated aqueous layer. Complete the titration with the hydrochloric acid (B.2.1.5) shaking constantly.

**B.2.3 Calculation**

One millilitre of 0.5 mol/l solution of hydrochloric acid is equivalent to 80.11 mg of potassium benzoate.

Potassium benzoate, as  $C_6H_5COOK$ ,  
per cent by mass =  $\frac{16.2 \times c \times v}{m}$

where,

- m is the mass, in g, of the sample taken for the test;
- c is the concentration, in mol/l, of the sodium hydroxide solution; and
- V is the volume, in ml, of the sodium hydroxide solution required for the titration.

**B.3 SODIUM BENZOATE**

**B.3.1 Reagents, listed under B.2.1**

**B.3.2 Procedure**

Weigh, to the nearest milligram, about 3 g of the sample previously dried for 4 hours at 105 °C and dissolve in 50 ml of water. Proceed as described in B.2.2 commencing from "Neutralize the solution, if necessary .....

### B.3.3 Calculation

One millilitre of 0.5 mol/l solution of hydrochloric acid is equivalent to 72.05 mg of sodium benzoate.

Sodium benzoate, as  $C_6H_5NaO_2$ , per cent by mass =  $\frac{14.41 \times c \times V}{m}$

where,

$m$  is the mass, in g, of the sample taken for the test;  
 $c$  is the concentration in mol/l, of the sodium hydroxide solution;  
 and  
 $V$  is the volume, in ml, of the sodium hydroxide solution required for the titration.

## APPENDIX C DETERMINATION OF LOSS ON DRYING

### C.1 BENZOIC ACID

#### C.1.1 Apparatus

*Desiccator*, desiccant concentrated sulfuric acid (rel. den = 1.84)

#### C.1.2 Procedure

Weigh, to the nearest milligram, about 2 g of benzoic acid in a weighing bottle. Distribute the sample as evenly as practicable. Place the bottle containing the sample (uncovered) in the desiccator (C.1.1) with sulfuric acid as a desiccant for 3 hours. Close the bottle and weigh. Repeat the process of drying, weighing at 30 min intervals until the difference between two successive weighings does not exceed 1 mg.

#### C.1.3 Calculation

Loss on drying, per cent by mass =  $\frac{m_1 - m_2}{m_1 - m_0} \times 100$

where,

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



## C.2 SODIUM BEZOATE

## C.2.1 Apparatus

C.2.1 Weighing bottle, glass - stoppered, shallow

C.2.2 Oven, maintained at  $105 \pm 1^\circ\text{C}$ .

## C.2.3 Desiccator

## C.2 Procedure

Weigh, to the nearest milligram, about 2 g of powdered sodium benzoate in the tared weighing bottle (C.2.1) Distribute the sample as evenly as practicable to a depth of about 5 mm. Place the bottle containing the sample (uncovered) in the oven (C.2.2) for 4 hours. Close the bottle, allow it to come to the room temperature in a desiccator and weigh. Repeat the process of drying, cooling and weighing at 30 min. intervals until the difference between two successive weighings does not exceed 1 mg.

## C.2.3 Calculation

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

where,

$m_0$  is the mass, in g, of the empty bottle;

$m_1$  is the mass, in g, of the bottle with the sample before drying;  
and;

$m_2$  is the mass, in g, of the bottle with the sample after drying.

APPENDIX D  
DETERMINATION OF SULFATED ASH

## D.1 REAGENT

D.1.1 Sulfuric acid, dilute, approximately 2 mol/l solution

## D.2 APPARATUS

D.2.1 *Platinum dish*, 50-ml to 100-ml in capacity

D.2.2 *Hot plate or infra-red heat lamp*

D.2.3 *Muffle furnace*, maintained at  $800 \pm 25^\circ\text{C}$ .

D.2.4 *Desiccator*

## D.3 PROCEDURE

Weigh, to the nearest milligram, about 2 g of the sample in to a tared platinum dish (D.2.1) or other suitable container. Add sufficient amount of dilute sulfuric acid (D.1.1) to moisten the entire sample. Heat using a hot plate or an infra-red heat lamp until the sample is dry and thoroughly charred. Then continue heating until completely volatilized or nearly all the carbon has been oxidized. Cool and moisten the residue with 0.1 ml to 0.2 ml of the sulfuric acid. Heat in the same manner until the remainder of the sample and any excess sulfuric acid are volatilized. Finally ignite in the muffle furnace (D.2.3) for 15 minutes, or longer, if necessary, to complete ignition. Cool in a desiccator and weigh.

### NOTE

*In order to promote volatilization of sulfuric acid, it is advisable to add few pieces of ammonium carbonate just before completing ignition.*

## D.4 CALCULATION

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

where,

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

APPENDIX E  
DETERMINATION OF READILY CARBONIZABLE SUBSTANCES

## E.1 REAGENTS

E.1.1 Sulfuric acid, concentrated, rel. den. = 1.84

E.1.2 Matching fluid

E.1.2.1 *Cobalt chloride solution*

Weigh, to the nearest milligram about 65 g of cobalt chloride hexahydrate ( $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ). Dissolve this in a sufficient amount of dilute hydrochloric acid prepared by mixing 25 ml of concentrated hydrochloric acid (rel.den. = 1.18) with 975 ml of water. Pipette 5 ml of this solution in to a 250- ml iodine flask, add 5 ml of 3 per cent (V/V) hydrogen peroxide and 15 ml of 20 per cent (V/V) sodium hydroxide solution. Boil for 10 minutes, cool and add 2 g of potassium iodide and 20 ml of 25 per cent (V/V) sulfuric acid. When the precipitate is dissolved titrate the liberated iodine with 0.1 mol/l solution of sodium thiosulfate, using starch as an indicator.

One millilitre of 0.1 mol/l solution of sodium thiosulfate is equivalent to 23.79 mg of cobalt chloride hexahydrate. Adjust the final volume of the solution by adding a sufficient amount of dilute hydrochloric acid until 1 ml of the solution contains 59.5 mg of cobalt chloride hexahydrate.

E.1.2.2 *Cupric sulfate solution*

Weigh, to the nearest milligram, about 65 g of cupric sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ). Dissolve this in a sufficient quantity of dilute hydrochloric acid prepared by mixing 25 ml of concentrated hydrochloric acid (rel.den.=1.18) and 975 ml of water. Pipette 10 ml of this solution into a 250-ml iodine flask, add 40 ml of water, 4 ml of acetic acid and 3 g of potassium iodide. Titrate the liberated iodine with 0.1 mol/l solution of sodium thiosulfate using starch as an indicator.

One millilitre of 0.1 mol/l solution of sodium thiosulfate is equivalent to 24.97 mg of cupric sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ). Adjust the final volume of the solution by adding a sufficient amount of dilute hydrochloric acid until 1 ml of the solution contains 62.4 mg of cupric sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ).

### E.1.2.3 *Ferric chloride solution*

Weigh, to the nearest milligram, about 55 g of ferric chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ). Dissolve this in a sufficient quantity of dilute hydrochloric acid prepared by mixing 25 ml of concentrated hydrochloric acid (rel.den.=1.18) and 975 ml of water. Pipette 10 ml of this solution into a 250-ml iodine flask, add 15 ml of water, 5 ml of hydrochloric acid and 3 g of potassium iodide. Allow the mixture to stand for 15 min. Dilute with 100 ml of water and titrate the liberated iodine with 0.1 mol/l solution of sodium thiosulfate using starch as an indicator. Carryout a blank determination using same quantities of reagents in a similar manner and make any necessary corrections.

One millilitre of 0.1 mol/l solution of sodium thiosulfate is equivalent to 27.03 mg of ferric chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ). Adjust the final volume of the solution by adding a sufficient amount of dilute hydrochloric acid until 1 ml of the solution contains 45.0 mg of ferric chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ).

### E.1.2.4 *Preparation of matching fluid*

Pipette out 0.2 ml of cobalt chloride solution (E.1.2.1), 0.3 ml of ferric chloride solution (E.1.2.2), 0.1 ml of cupric sulfate solution (E.1.2.3), 4.4 ml of water and mix well in a glass container.

## E.2 PROCEDURE

Weigh, to the nearest milligram, about 0.5 g of the sample and dissolve in 5 ml of sulfuric acid (E.1.1) in a glass container. Allow the solution to stand for 15 minutes and compare the colour of the solution with the matching fluid (E.1.2) against a background of white porcelain.

The sample solution should not have a deeper colour than the matching fluid.

## APPENDIX F DETERMINATION OF READILY OXIDIZABLE SUBSTANCES

### F.1 REAGENTS

F.1.1 *Sulfuric acid*, concentrated, rel. den. = 1.84

F.1.2 *Potassium permanganate*,  $c(\text{KMnO}_4) = 0.02$  mol/l solution.

## F.2 PROCEDURE

Add 1.5 ml of sulfuric acid (F.1.1) to 100 ml of water. Heat to boil and add potassium permanganate (F.1.2) in drops, until the pink colour persists for 30 seconds. Weigh, to the nearest milligram, about 1 g of the sample and dissolve in the above hot solution. Titrate with the potassium permanganate solution (F.1.2) to a pink colour, which persists for 15 seconds.

The required volume of 0.02 mol/l potassium permanganate should be not more than 0.5 ml.

## APPENDIX G DETERMINATION OF CHLORINATED ORGANIC COMPOUNDS

### G.1 APPRATUS

Crucible, silica or platinum.

### G.2 REAGENTS

G.2.1 Nitric acid, concentrated, rel. den. = 1.40

G.2.2 Calcium carbonate

G.2.3 Nitric acid, dilute, prepared by diluting 105 ml of nitric acid (70 per cent V/V) with water to make 1000 ml.

G.2.4 Silver nitrate, 0.01 mol/l solution.

G.2.5 Hydrochloric acid, 0.01 mol/l solution.

### G.3 PROCEDURE

Weigh, to the nearest milligram, about 0.25 g of the sample and dissolve in 10 ml of water. (In the case of benzoic acid use 10 ml of 0.1 mol/l sodium hydroxide solution instead of water). Acidify with nitric acid (G.2.1), filter off the precipitate using a ashless filter paper. Add 0.5 g of calcium carbonate (G.2.2) to the precipitate, transfer the filter paper with the contents into the crucible (G.1) and mix the contents using a glass rod. Remove the material adhering to the glass rod using a small piece of ashless filter paper and put this piece of filter paper into the crucible. Dry the contents and ignite. Dissolve the residue in 20 ml of dilute nitric acid (G.1.3) and filter. Mix the filtrate with 0.5 ml of silver nitrate solution (G.1.4).

The turbidity of the solution should be not more than that obtained by a similar volume of water with the addition of 0.5 ml of silver nitrate (G.1.4) and 0.5 ml of hydrochloric acid (G.1.5).

APPENDIX H  
DETERMINATION OF ARSENIC

H.1 REAGENTS

- H.1.1 *Sulfuric acid* concentrated, rel. den = 1.84  
H.1.2 *Hydrogen peroxide*, 30 per cent (V/V) solution.

H.2 PROCEDURE

Weigh, 1.0 g of the sample and transfer into a flask. Add 10 ml of sulfuric acid (H.1.1) and few glass beads, and digest on a hot plate in a fume hood until charring begins. After the sample has been initially decomposed by the acid, add hydrogen peroxide (H.1.2) dropwise with caution and reheat between each addition (First few drops must be added very slowly with sufficient mixing). Discontinue heating if foaming becomes excessive. Maintain the oxidizing conditions during the digestion by adding small quantities of peroxide whenever the mixture turns brown or dark in colour.

Continue the digestion until the organic matter is destroyed and the solution becomes colourless. Cool, add 10 ml of water cautiously, evaporate and cool. Add 10 ml of water, mix and wash the sides of the flask with few millilitres of water. Transfer to the gutzeit bottle with a small quantity of water and dilute the solution to 35 ml.

Proceed as given in Method 2.1 (modified Gutzeit Method) of SLS 312 : 1976.

APPENDIX J  
DETERMINATION OF HEAVY METALS

J.1 REAGENTS

- J.1.1 *Nitric acid*, concentrated, rel. den. = 1.40.  
J.1.2 *Sodium carbonate*  
J.1.3 *Acetic acid*, dilute, 60 g/l solution.  
J.1.4 *Hydrochloric acid*, dilute, prepared by diluting 236 ml of concentrated hydrochloric acid (rel. den. = 1.18) with water to make 1000 ml.

J.1.5 Ammonia solution, dilute, prepared by diluting 400 ml of ammonium hydroxyde (rel.den.=0.91) with water to make 1000 ml.

#### J.1.6 Lead nitrate stock solution

Dissolve 159.8 mg of lead nitrate,  $Pb(NO_3)_2$ , in 100 ml of water containing 1 ml of nitric acid. Dilute with water to 1000 ml and mix well. This solution should be prepared and stored in a glass container which is free from lead salts.

J.1.7 Standard lead solution, containing 10  $\mu$ g of lead ( $Pb^{2+}$ ) in 1 ml of solution.

Dilute 10.0 ml of lead nitrate stock solution, accurately measured, with water to 100.0 ml. Prepare freshly before use.

#### J.1.8 Hydrogen sulfide solution, saturated

Store in a small, dark, amber-coloured bottle, filled nearly to the top. It is unsuitable unless it possesses a strong odour of hydrogen sulfide. Store in a cool, dark place.

### J.2 PREPARATION OF SAMPLE

#### J.2.1 Benzoic acid

Weigh, to the nearest milligram, about 2 g of the sample and volatilize over a low flame. Add 2 ml of nitric acid (J.1.1) and about 10 mg of sodium carbonate (J.1.2) and evaporate to dryness on a steam bath. Dissolve the residue in a mixture of 1 ml of dilute acetic acid (J.1.3) and 24 ml of water.

#### J.2.2 Potassium benzoate and sodium benzoate

Weigh, to the nearest milligram, about 4 g of the sample and dissolve in 40 ml of water. Add 10 ml of dilute hydrochloric acid (J.1.4) dropwise with vigorous stirring. Then filter the solution.

### J.3 PROCEDURE

#### J.3.1 Preparation of control solution (Solution A)

Pipette 2.0 ml of standard lead solution (J.1.7) into 50-ml nessler tube and add water to make 25 ml. Adjust the pH value to lie between 3.0 and 4.0 using short-range pH indicator paper, by the addition of dilute acetic acid solution (J.1.3) or ammonia solution (J.1.5). Dilute with water to 40 ml and mix the solution.

#### J.3.2 Preparation of test solution (Solution B)

Take 25.0 ml of the sample solution (J.2.1 or J.2.2) into a 50- ml nessler tube, similar to the one used for the Solution A. Adjust the pH value to lie between 3.0 and 4.0 using short-range pH indicator paper, by the addition of dilute acetic acid solution (J.1.3) or ammonia solution (J.1.5). Dilute to 40 ml with water and mix the solution.

Add 10 ml of freshly prepared hydrogen sulfide solution (J.1.8), to each tube, mix, allow to stand for 5 minutes and observe downwards over a white surface.

The colour of the Solution B, should be not darker than that of the Solution A.

### APPENDIX K DETERMINATION OF ACIDITY AND ALKALINITY

#### K.1 REAGENTS

K.1.1 Sodium hydroxide,  $c(\text{NaOH}) = 0.1 \text{ mol/l}$  solution.

K.1.2 Hydrochloric acid,  $c(\text{HCl}) = 0.1 \text{ mol/l}$  solution.

K.1.3 Phenolphthalein indicator.

#### K.2 PROCEDURE

Weigh, to the nearest milligram, about 2 g of the sample and dissolve in 20 ml of freshly boiled water. Neutralize this solution with either the sodium hydroxide solution (K.1.1) or the hydrochloric acid solution (K.1.2) using phenolphthalein as an indicator.

The required volume of sodium hydroxide solution or hydrochloric acid solution for the titration should be not more than 0.5 ml.



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