Bangladesh Standard
Specification For
Soft Drink Powder
(Draft for Second Revision)

1. Scope

1.1 This Standard describes the requirements and methods of sampling and test for sweetened soft drink powder.

2. References

2.1 The Bangladesh Standards and international standards listed in Annex – A are necessary adjuncts to this standard. For references, the latest edition of the referenced document (including any amendments) applies.

3. Terminology

3.1 Soft Drink Powder – Soft Drink Powder means the dry product in a form of crystals or powder which, when reconstituted with potable water in accordance with the manufacturer's instructions, yields a non-alcoholic, sweetened, flavoured (with or without essences) beverage intended for direct consumption. It may contain sugars or permitted sweeteners, food acids, permitted food additives, fruit solids or fruit powders, and other ingredients consistent with good manufacturing practice.

3.2 Reconstituted drink –A product obtained by reconstitution of flavoured drink in solid form as crystals, powder or tablets, using potable water in accordance with the direction for use as provided on the product label for direct human consumption.

4. Ingredients

4.1 All ingredients shall conform to the relevant Bangladesh Standard specifications, where available. All materials shall be food grade, halal and free from foreign matter, harmful microorganisms, insect infestation, objectionable flavours and odours, food additives and processing aids.

4.2 Basic Ingredients

Soft drink powder shall be prepared from the following essential ingredients:

4.2.1 Sweeteners – It may contain one or more sweeteners such as:

a) Nutritive Sweeteners – Sugar, palm sugar, cane sugar, invert sugar, sorbitol, liquid glucose, lactose, dextrin, dextrose, dextrose monohydrate, fructose, maltose, icing sugar

b) Non-nutritive Sweeteners – As permitted under the food category 14.1.4 and 14.1.4.3 in latest version of Codex General Standard for Food additives (CODEX STAN 192) or permitted by national legislations.

c) When combinations of non-nutritive sweeteners are used, the sum of the proportions of individual sweeteners, expressed as percentages of their respective maximum permitted levels, shall not exceed 100%.

4.2.2 Acidulants - As permitted under the food category 14.1.4 and 14.1.4.3 in latest version of Codex General Standard for Food additives (CODEX STAN 192) or permitted by national legislations.

4.2.3 Flavouring Substances - As permitted under the food category 14.1.4 and 14.1.4.3 in latest version of Codex General Standard for Food additives (CODEX STAN 192) or permitted by national legislations.

Note: Food category 14.1.4.3 in the Codex STAN 192 General Standards for Food Additives (GSFA) is the category for concentrates (liquid or solid) for water-based flavoured drinks**,** and this category explicitly includes powdered concentrates / powder mixes for preparation of beverages (i.e. soft-drink powders). Products classified under food category 14.1.4.3 are considered a subcategory of 14.1.4 (Water-based flavoured drinks, including “sport”, “energy” or “electrolyte” drinks and particulated drinks). So, the general provisions applicable to category 14.1.4 also extend to 14.1.4.3, unless specific exceptions are stated.

4.3 Optional ingredients

4.3.1 Food additives- In the preparation of soft drink powder, the addition of colourings, preservatives, humectants, emulsifying stabilizing or thickening agents, anti-caking agents, buffering agents, antioxidants, carbonating agents, foaming agents, flavour enhancers, cloudifying agents, stabilizing agents required shall be according to the relevant food category 14.1.4 and 14.1.4.3 of the updated version of CXS 192 or permitted by national legislations.

4.3.2 Vitamins and minerals: Vitamins and minerals, as added nutrients (conforming to the latest available version of CXG 55:2005 Guidelines for Vitamin and mineral Food Supplements)

4.3.3 Fruit Juice Powder

4.3.4 Edible Salt (conforming to the latest available version of BDS 1236)

5. Requirements

5.1 Hygienic Requirement – During processing, handling, storage and transportation, effective measures must be taken to prevent cross contamination with chemicals, microbial or physical contaminants.

5.1.1 The product shall be processed and packed under strict hygienic conditions in premises maintained in accordance with BDS 822.

5.2 Requirements for raw materials - All raw materials used in the manufacture of soft drink powder shall be clean and fit for human consumption and shall comply with the requirements of the rules issued under the National regulations.

5.3 Flavour and Odour

5.3.1 The product, before and after reconstitution to drinking strength, shall have a pleasant characteristic flavour. When soft drink powder is dissolved in potable water in accordance with the instruction given on the label by the manufacturer, the reconstituted drink product shall have taste and aroma characteristic of the flavour to which it is claimed or implied on the label. It shall be free from off odours and off-flavours.

5.4 Requirements for Dry Product

5.4.1 The product shall be

a. in granular / crystalline powdered or tablet form, homogeneous and free flowing
b. free from any dirt and extraneous matter
c. non-toxic and fit for human consumption.
d. dissolved completely in potable water upon shaking or stirring

5.4.2 The product shall also comply with the requirements specified in Table 1 and shall not exceed the limits for heavy metals given in table 2.

Table -1 : Requirements for Soft Drink Powder

Sl.No.	Characteristic	Requirement	Method of Test Ref. to Annex of this Standard
(1)	(2)	(3)	(4)
i.	Moisture content, percent by mass, Max.	1.0	Annex - C
ii.	Sulphuted ash content, percent by mass, Max.	2.0	Annex - D
iii.	Acid insoluble ash content, percent by mass, Max.	0.05	Annex-B of BDS 2033
iv.	Solubility (at ambient temperature), percent by mass, Min.	95.0	Annex - E
v.	Acidity (calculated as anhydrous citric acid), percent by mass, Max.	10	Annex - F

Table- 2 : Limits for Heavy Metal

Sl. No.	Characteristic	Limits	Test Method
(1)	(2)	(3)	(4)
i.	Cadmium (as Cd,) mg/kg, Max.	0.1	AOAC 999.11
ii.	Lead (as Pb), mg/kg, Max.	0.2	AOAC 994.02
iii.	Arsenic (as As) mg/kg, Max.	0.1	AOAC 986.15
iv.	Tin (as Sn), mg/kg, Max.	25.0	AOAC 985.16

5.4.3 The product shall also comply with the Microbiological requirements specified in Table-3

Table -3 : Microbiological Requirements

Sl. No	Characteristic	Limit	Test Method
(1)	(2)	(3)	(4)
i.	Total plate count, per g Max.	100	ISO 4833-2
ii.	Total coliform count, per g	Nill	ISO 4832
iii.	Mould count, per g Max.	10	ISO 21527-2

5.5 Requirements for reconstituted product

5.5.1 The product, after reconstitution to drinking strength according to the manufacturer's instructions, shall conform to the following requirements:

5.5.1.1 Sugar content

If nutritive sugar is used, the sugar content (when calculated as sucrose) shall be not less than 5 percent by weight of the reconstituted product, when tested as specified in Annex–G.

5.5.1.2 Vitamin C content

The Vitamin C content shall be not less than 30mg/100ml of the reconstituted product, when tested as specified in Annex-H.

NOTE: The requirement shall only apply to products implied or claimed to be fortified with Vitamin C.

5.6 Legal requirement – The product shall in all other aspects comply with the requirements of the legislations enforced in the country.

6. Packing And Marking

6.1 Packing – The product, if wrapped, shall be in food grade, plain or printed waxed paper, foil or cellulose film or other food grade thermoplastic films. In case of printed packaging material, the printing ink shall be non-toxic and shall not come in direct contact with the product. The wrapped or unwrapped material shall be bulk packed in clean, food grade, reasonably air-tight and sound containers that have no effect on product properties. Such containers shall be made of tinplate, glass, plastics, moisture proof paper, cellulose film or any other suitable packing material.

6.2 Marking

6.2.1 The containers or sachet shall be marked with the following particulars:
a) Name of the product, declared as Soft drink powder;
b) Manufacturers'/Importer name and full address with recognized trade mark brand name, if any;
c) Batch / code number;
d) Date of manufacture;
e) Date of Expiry, (In case of imported product, the remaining expiry date shall not be less than two-third of the original period;
f) Net content;
g) Maximum retail price (MRP);
h) Where nutritional claims are made or implied, the nutritional information declaring the total quantity of nutrient per 100 gram of the product as sold as well as per serving of the reconstituted product;
i) List of ingredients in descending order of proportion;
j) Information for storage, where necessary;
k) Name of the non-nutritive sweetener used;
l) When non-nutritive sweeteners are added as substitutes for sugars, the statement, "with non-nutritive sweetener(s)" or "sugar-free", shall be included in conjunction with or in close proximity to the product name;
n) If Aspartame used, must declare "Not for Phenylketonurics".

6.2.2 Directions for reconstitution of the product to drinking strength and storage instructions shall appear either on the label of the container, sachet or on an accompanying leaflet. In addition to general directions being given, the reconstitution rate shall be given as grams per 100 ml of the reconstitute product.

6.2.3 The containers may also be marked with the BSTI Certification Mark.

NOTE – The use of BSTI Certification Mark is governed by the provisions of Bangladesh Standards and Testing Institution Act, 2018 and the Rules and Regulations made thereunder. Details of conditions, under which a license for the use of BSTI Certification Mark may be granted to manufacturers or processors, may be obtained from the Bangladesh Standards and Testing Institution.

7. Sampling

Representative samples of the material shall be drawn as described in Appendix B.

8. Conformity To Standard

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

8.1 Each container examined as in B 5.1 satisfies the relevant requirements.
8.2 The composite sample tested as in B 5. 2 satisfies the relevant requirements.

Annex A
(Clause 2)
List of Relevant Reference Standards

BDS NO.	Title
BDS 138	Refined Sugar
BDS 490	Hard Candy
BDS 1000	Soft candy
BDS 1010	Methods of sampling and test for processed fruits and vegetables
BDS 1236	Iodized salt
BDS 1240	Packaged Drinking Water
BDS 2033	Isabgol Husk
BDS 2043	Energy Drink
CXS 192	General Standard for Food Additives

Annex - B
(Clause-7)
Method of Sampling

B-1 Lot

All containers of one size, containing soft drink powder belonging to one batch of manufacture shall constitute a lot.

B-2 General Requirements of Sampling

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

B-2.1 The sample shall be taken in a protected place not exposed to damp air and dust.

B-2.2 The sampling instrument shall be clean and dry when used.

B-2.3 The samples shall be placed in suitable clean and dry containers. Each container shall be sealed air-tight after filling and marked with necessary details of sampling.

B-3 Scale of Sampling

B-3.1 Samples from each lot shall be tested for ascertaining conformity of the lot to the requirements of this standard.

B-3.2 The number of containers to be selected from the lot shall be in accordance with columns 2 and 3 of Table 3.

Table 3 Scale of Sampling

Number of containers in the lot	Number of containers to be selected	
	Container size 500 g, more	Container size less than 500g
(1)	(2)	(3)
Up to 15	03	05
16 to 25	04	06
26 to 50	05	08
51 to 150	07	10
151 to 300	10	15
301 to 1000	15	20
1001 and above	20	25

B.3.3 Containers shall be selected at random. In order to ensure randomness of selection, random number tables as given in BDS 1010 (Methods of sampling and test for processed fruits and vegetables) shall be used.

B.4 Preparation of a Composite Sample

B.4.1 A small quantity of material shall be drawn from different parts of each container selected as in B.3.2 using an appropriate sampling instrument.

B-4.2 The material so obtained shall be mixed together and reduced using a suitable method, if necessary, to produce a composite sample of not less than 150 g.

B-5 Number of Tests

B-5.1 Each container selected as in B-3.2 shall be examined for packing and marking requirements.

B-5.2 The composite sample obtained as in B.4 shall be examined for requirements given in 4.3, 4.4 and 4.5

Annex - C
[(Table 1, Item (I)]
Determination of Moisture

C-0 Either of the two methods, namely Air oven Method and Vacuum Oven Method, may be employed, but in case of dispute, the Vacuum Oven Method shall be adopted.

C-1 AIR OVEN METHOD

C-1.1 Apparatus- The following apparatus is required;

(a) Dish 5.0 cm in diameter and 2.5 cm high, made of aluminium and having a close-fitting cover provided with a knob.

(b) Electric oven- with automatic regulation of temperature by a suitable thermostatic arrangement. There should be a good draught of air in the over by natural convection.

C-1.2 Procedure- Spread in an oven layer, with a glass rod on nickel spatula, about 10g of the sugar in the previously dried and tared dish. Cover the dish immediately and weigh it accurately. Remove the cover and place the dish in the electric oven and dry the material for 2 hours at 110° ±1°C. Remove the dish from the oven, cover it and cool in a desiccator and weigh.

Heat the dish again in the electric oven at 110° ± 1°C for 30 minutes, cool and weigh, Repeat this process until the loss in weight between two successive weighings does not exceed one milligram. Record the lowest weight.

C-1.3 Calculation- Calculate as follows:

Moisture content of the material, percent by mass = $\frac{100w}{W}$

where

w= loss in weight in g of the material on drying, and
W= weight in g of the material taken for the test.

C-2 VACUUM OVEN METHOD

C-2.1 Apparatus - The following apparatus is required :

(a) Dish 5.0 cm in diameter and 2.5cm high, made of aluminium and having a close fitting cover provided with a knob.

(b) Vacuum oven- fitted with necessary accessories as to provide slow current of dry air through the oven to ensure removal of water vapours.

C-2.2 Procedure- Spread in an oven layer, with a glass rod or nickel spatula, about 10g of the sugar into the dried and tared dish. Cover the dish and weigh it accurately. Remove the cover and place the dish in the vacuum over for 5 hours at 60° ± 1°C under a pressure not exceeding 50 mm of mercury, using a slow current of dry air to facilitate removal of water vapour. Remove the dish from the oven, cover it, cool in a desiccator and weigh. Heat the dish again in the vacuum oven for one hour, cool, and weigh, Repeat this process until the loss in weight between two successive weightings does not exceed one milligram. Record the lowest weight.

C-2.3 Calculation- Calculate as given under C-1.3

Annex - D
[Table 1, Item (ii)]
Determination of Sulphated Ash

D-1 Reagent

D-1.1 Concentrated Sulphuric acid- sp.gr. 1.84

D-2 Preparation of Sample-Mince as quickly as possible with a sharp-edged knife or grind in an dry pestle and mortar 150g of the sample on a clean porcelain Slab. Mince thoroughly to secure a uniform sample. Store the minced sample immediately in an air-tight glass container and use this wherever the use of prepared sample is indicated.

D-3 Procedure-Accurately weigh about 5g of the prepared sample (see D-2) in to a 9 cm diameter platinum of silica dish. Add a few drops (about 1.5 ml of concentrated sulphuric acid to the material in the dish. Gently heat the dish on a hot plate until the material is well carbonized and then increase the heat until the evolution of sulphuric acid fumes ceases. Ash the carbonized matter in a muffle furnace at 600±20°C. Cool the ash and moisten it with a few drop of concentrated sulphuric acid, heat strongly on a hot plate until sulphuric acid fumes ceases to be evolved and finally ash in the muffle furnace at 600 ± 20°C for about 2 hours.

Cool in a desiccator and weigh. Heat again in the muffle furnace for 30 minutes at 600±20°C. Cool in a desiccator and weigh. Repeat the process of heating in the muffle furnace for 30 minutes cooling and weighing till the difference between two successive weighings is less than 10mg. Record the lowest mass.

D-4 Calculation

D-4.1 Ash, sulphated percent by mass = $\frac{100\,M_1}{M_2}$

where

M_1 = mass in g of the ash, and
M_2 = mass in g of the prepared sample taken for the test.

Annex - E
(Table-I, Item-iv)
Method of Test for Solubility

E-1 PROCEDURE

E-1.1 Reconstitution and separation of insoluble matter.

E.1.1.1 Weigh to the nearest milligram, about 5 g of the sample into a dried, tared 50 ml centrifuge tube. Add 10 ml of water and mix well to form a uniform paste, free from lumps, using a glass rod. Add a further 25 ml of water to the centrifuge tube using a portion of it to wash the glass rod completely free of the powder. Close the tube and shake vigorously for 3 minutes. Centrifuge the tube for 15 minutes at 400 ± 100 revolutions per minute.

E.1.2 Determination of dissolved solids in supernatant solution

E-1.2.1 Pipette out 5 ml of the supernatant solution and transfer to a dried and tared glass, aluminium or stainless-steel dish provided with a cover. Weigh the dish with the contents and place on a boiling water-bath for 15 minutes and transfer the dish to an air-over maintained at 103 ± 1°C. Dry for 3 hours. Cool the dish in a desiccator and weigh again.

E-1.3 Determination of residue

E-1.3.1 Decant off the remaining solution as completely as possible without disturbing the sediment at the bottom of the tube. Weigh the centrifuge tube with the wet sediment. Dry the contents of the tube by first placing the tube in a boiling water-bath and later in an electric oven at 103 ± 1°C for 3 hours. Cool in a desiccator and weigh. Dry the tube further for 1 hour, cool and weigh. Repeat drying at hourly intervals, cooling and weighing till the difference between two successive weighings does not exceed 1 milligram.

E-2 Calculation

$$\text{Solubility, percent by mass} = 100 - \frac{100(m_4 - m_2) - Y(m_3 - m_4)}{m_1}$$

Where,

m_1 = mass, in g, of the material taken for test ;
m_2 = mass, in g, of the centrifuge tube ;
m_3 = mass, in g, of centrifuge tube with residue before drying ;
m_4 = mass, in g, of the centrifuge tube with residue after drying ; and
Y = solids in supernatant liquid, in g per ml (sec E-1.2)

Annex - F
(Table -1, Item-v)
Method of Test for Acidity (as Anhydrous Citric Acid)

F-1 Reagents

F-1.1 Standard sodium hydroxide solution, approximately 0.1 M.

F-1.2 Phenolphthalein indicator solution. Dissolve 0.5 g of phenolphthalein in 200 ml of 50 percent ethyl alcohol by volume.

F.2 Procedure

F-2.1 Weigh, to the nearest milligram, about 10 g of the powder in a suitable dish or basin. Transfer the contents to a conical flask with 100 ml to 150 ml of recently boiled and cooled distilled water. Add 1 ml of phenolphthalein indicator solution and titrate against the standard sodium hydroxide solution. For observing the colour change at the end point, use another portion of the sample diluted to the same proportion in a similar flask.

F-3 Calculation

F-3.1 Acidity (as anhydrous citric acid), percent by mass = $\frac{6.404VM}{m}$

Where,

V= volume, in ml, of standard sodium hydroxide, required for titration;
M = molarity of the standard sodium hydroxide solution; and
m = mass, in g, of the sample taken for test.

Annex - G
[Clause 5.5.1.1]
Determination of Sucrose

G-1 APPARATUS-The following apparatus is required.

G-1.1 Analytical balance.

G-1.2 Normal weight- 26.000g (brass weight) as weighed in air at 20°C.

G-1.3 Basin- made of nickel or german silver, large enough to hold twice the normal weight of sugar.

G-1.4 Volumetric flasks- four; One of capacity 200 ml; one of capacity 110 ml with 100 ml and 110 ml marks; and two of capacity 100 ml each; calibrated at 20°C.

G-1.5 Conical flask of capacity 250 ml.

G-1.6 Long- stemmed funnel- with a stem long enough to extend below the neck of the Volumetric flask (see G-1.4)

G-I.7 Stemless funnel- capable of holding 200 ml.

G-1.8 Glass Cylinder- capable of holding 200 ml.

G-1.9 Watch glass- large enough to cover the stemless funnel

G-1.10 Pipette- 50ml, Calibrated at 20°C

G-1.11 Thermometer- having a range 0° to 100° C and graduated at every 0.1° C

G-1.12 Saccharimeter- graduated in international sugar scale and provided with a 200 mm tube. The Saccharimeter should be stored in a cabinet, the inside of which is maintained at 20°C. Where this is not possible necessary corrections shall be applied based on the characteristics of the particular instrument.

G-2 **REAGENTS**- The following reagents are required.

G-2.1 Anhydrous lead subacetate- finely powdered.

G-2.2 Deleading solution- Dissolve 7g of disodium hydrogen phosphate ($Na_2\ HPO_4\ 12H_2O$) and 3g of potassium oxalate ($K_2C_2O_4\ H_2O$) in water and make up the volume to 100ml.

G-2.3 Sodium chloride solution- Dissolve 231. 50g of sodium chloride in water and make up the volume to one litre in a volumetric flask.

G-2.4 Hydrochloric acid- sp. gr. 1.1029, at 20° ± 4°C

G-3 **Test Temperature**- The polarization of the material shall be determined at 20° C, as far as possible.

G-4 **PROCEDURE**

G-4. 1 Preparation of the solution- Weigh accurately in the basin twice the normal weight (see G-1.2) of the powdered material on the analytical balance. Transfer this quantity to the 200 ml volumetric flask with the aid of the long-stemmed funnel. Rinse the basin and the funnel with water, and transfer the washings to the volumetric flask taking care that the volume in the flask does not exceed 160 ml. Bring the sugar into solution by gently swirling the flask and make up the volume to 200ml with water. Clarify the solution by adding anhydrous lead subacetate in small quantities until nearly all the impurities have been precipitated. If necessary add 1-2 ml of alumina cream to clarify the turbidity. Place the stemless funnel over the glass cylinder and fix in the funnel a fluted cone of dry filter paper, large enough to hold 200 ml of the liquid. Pour the whole of the defecated solution on to the fluted cone of dry filter paper and cover it immediately with the watch glass. Collect the filtrate in the cylinder.

G-4.2 Deleading the filtrate- Take 100 ml of the filtrate in a 110 ml volumetric flask. Add the deleading solution (see G-2.2) in small quantities at a time to the whole of the filtrate until the excess of lead is completely precipitated. Make up the volume to 110 ml with water and filter.

G-4.3 Polarization- Pipette two portions of 50 ml each of the clear filtrate (see G-4.2) separately into a 100 ml volumetric flask and a 250 ml. conical flask and treat them separately as follows:

a) Polarization before inversion- To the filtrate contained in the 100 ml volumetric flask, add 1.10 ml sodium chloride solution and make up to volume with water.

Polarize this solution in the saccharimeter using 200 mm tube enclosed in a water jacketed tube at the test temperature (see G-3). Note the reading (p) of the saccharimeter for the solution before inversion.

b) Polarization after inversion- To the filtrate in the conical flask, add 10 ml of hydrochloric acid solution. Mix the contents of the flask rotating it. Insert a thermometer in the conical flask containing the reaction mixture. Immerse the conical flask in a hot water bath and heat the contents to 60°C. Agitate the solution continuously for 3 minutes and allow it to remain in the bath for a total time of 10 minutes. Cool the contents of the conical flask to 20°C. Transfer quantitatively the same to a 100 ml volumetric flask. Make up the volume to 100ml. Polarize this solution in the saccharimeter at the test temperature (see G-3). Note the reading (Pt) of the saccharimeter for the solution after inversion.

G-5 Calculation- Calculate as follows:

Sucrose content of the material, percent by mass. = $\dfrac{100\ (p - p')}{132.56 + 0.0794\ (m - 13) - 0.53\ (t - 20)}$

where

P = reading of the saccharimeter observed at t° C before inversion (see G-4.3 (a), calculated to normal weight basis.

P' = reading of the saccharimeter observed at t° C after inversion (see G-4.3 (b), calculated to normal weight basis.

m = in g of the moisture free material per 100 ml of the solution (see G-4.3 (b), and

t = temperature in degrees celsius at which the polarization is carried out.

Annex - H
(Clause - 5.5.1.2)
Determination of Vitamin C Content

H-1 Principle

H-1.1 Vitamin C is extracted and the titration is performed in the presence of HPO_3CH_3COOH or HPO_3-CH_3 COOH-H_2SO_4 solution to maintain proper acidity for reaction and to avoid autoxidation of acid at high p^H. Ascorbic acid reduces oxidation-reduction indicator dye 2, 6-dichloroindophenol to a colourless solution. At the end point excess unreduced dye is rose pink in acid solution.

H-2 Reagents

H-2.1 Extracting solutions

H-2.1.1 Metaphosphoric acid-acetic acid solution

Dissolve, with shaking, 15 g of HPO_3 pellets or freshly pulverized Strick HPO_3 in 40ml acetic acid and 200 ml water; dilute to approximately 500 ml and filter rapidly through a fluted paper into a glass- stoppered bottle. (HPO_3 slowly changes to $H_3\ PO_4$, but if stored in a refrigerator, the solution remains satisfactory for 7 to 10 days).

H-2.1.2 Metaphosphoric acid-acetic acid-sulphuric acid solution. Proceed as in H-2.1.1 adding 0.15 M sulphuric acid in place of water.

H-2.2 Ascorbic acid standard solution (1 mg/ml)

Weigh, to the nearest 0.01 mg approximately 50mg USP ascorbic acid reference standard, that has been stored in a desiccator away from direct sunlight. Transfer to a 50 ml volumetric flask. Dilute to volume immediately before use with HPO_3-CH_3 COOH solution (see H 2.1.1).

H-2.3 Indophenol standard solution

Dissolve 50 mg, of 2,6 dichloroindophenol sodium salt, that has been stored in a desiccator over soda lime, in 50ml water to which has been added 42mg sodium bicarbonate; shake vigorously and when the dye dissolves, dilute to 200 ml with water. Filter through fluted paper into an amber glass-stoppered bottle. Keep stoppered, out of direct sunlight and store in a refrigerator. (Decomposition products that make the end point indistinct occur in some batches of dry indopenol and also develop with time in stock solution. Add 5.0 ml extracting solution containing excess ascorbic acid to 15 ml dry reagent. If reduced solution is not practically colourless, discard, and prepare new stock solution. If dry dye is at fault obtain new supply).

Transfer three, 2.0 ml portions of ascorbic acid standard solution to each of three 50 ml Erlenmeyer flasks containing 5.0 ml HPO_3 -CH_3 COOH solution (see H 2.1.1). Titrate rapidly with indophenol solution from a 50 ml burette until light but distinct rose-pink persists for more than 5 s. (Each titration should require approximately 15 ml indophenol solution and titrations should check within 0.1 ml).

Similarly titrate three blanks composed of 7.0 ml HPO_3-CH_3 COOH solution (H-2.1.1), and a volume of water approximately equal to the volume of indophenol solution used in direct titrations. After subtracting average blanks (usually approximately 0.1 ml) from standardization titration, calculate and express concentration of indophenol solution as milligrams of ascorbic acid equivalent 1.0 ml reagent, standardize indophenol solution daily with freshly prepared ascorbic acid standard solutions.

H-2.4 Thyme blue p^H indicator (0.04 percent)
Dissolve 0.1 g, indicator by grinding in an agate mortar with 10.75 ml, 0.02 M sodium hydroxide and dilute to 250ml with water. Transition range : 1.2 (red) to 2.8 (yellow)

H-3 Preliminary test for appreciable quantities of basic substances

Grind a portion of the sample and add approximately 25 ml of HPO_3-CH_3 COOH solution (H-2.1.1). Test the p^H by placing a drop of thymol blue p^H indicator on the pestle. A p^H value higher than 1.2 indicates the presence of appreciable amounts of basic substances.

H-4 Preparation of sample assay solution

Reconstitute the product according to the manufacturer's instructions and take an amount of solution containing approximately 100 mg ascorbic acid (designate this volume Vo). If appreciable amounts of basic substances are present, adjust p^H to approximately 1.2 with solution H-2.1.2. Dilute with solution H-2.1.1 to a measured volume containing 10 mg to 100 mg ascorbic acid per 100 ml of solution. Designate this volume V_3.

H-5 Procedure

Titrate three sample portions of the solution prepared as in H-4 containing approximately 2 mg ascorbic acid and make blank determinations for correction of titrations as in H-2.3, using proper volumes of HPO_3-CH_3 COOH solution (H-2.1.1) and water. If approximately 2 mg ascorbic acid is contained in a portion of the sample assay solution of less than 7 ml, add HPO_3-CH_3 COOH solution to obtain 7 ml for titration. Designate this volume as V_4

NOTE : Products containing ferrous (Fe), stannous (Sn) and cuprous (Cu) give values in excess of their actual ascorbic acid content by this method. Following are simple tests to determine whether these reducing ion are present in such amounts so as to invalidate the test.

Add 2 drops of 0.05 aqueous methylene blue solution to 10ml of freshly prepared sample assay solution and HPO_3-CH_3COOH reagent, and mix. Disappearance of the methylene blue colour in 5 s to 10 s indicates presence of interfering substances.

Stannous does not answer this test and may be tested as follows: To 10 ml sample solution, add 10 ml HCl (1 + 3), 5 drops of 0.05 aqueous indigocarine solution and mix. Disappearance of colour in 5 s to 10 s indicates the presence Sn or other interfering substances.

H-6 Calculation

Content of vitamin C (ascorbic acid) in mg = $(V_1 - V_2) \times \frac{m_1}{V_0} \times \frac{V_3}{V_4} \times 100$

per 100 ml of sample solution,

Where,

Vo = volume, in ml, of prepared sample solution containing 100 mg ascorbic acid
V_1 = volume, in ml of standard indophenol solution required for the titration ;
V_2 = volume, in ml of standard indophenol solution required for blank titration ;
V_3 = volume, in ml, of initial assay solution
V_4 = volume, in ml, of solution used for titration ; and
m_1 = mass in mg, of ascorbic acid equivalent to 1.0ml indophenol standard solution.