

LAB. MANUAL 5



MANUAL OF METHODS OF ANALYSIS OF FOODS

FRUIT AND VEGETABLE PRODUCTS



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MANUAL FOR ANALYSIS OF FRUIT AND VEGETABLE PRODUCTS

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MANUAL FOR ANALYSIS OF FRUIT AND VEGETABLE PRODUCTS

Standards for processed fruits and vegetables are laid down in section 2.3 of Food Safety and Standards (Food Product Standards and Food Additives) Regulations, 2011 and include thermally processed fruits and vegetables, fruit and vegetable juices, soups (canned/bottled/flexibly packed), soup powders, dehydrated vegetables, fruit jam/ jelly/ marmalade/ fruit cheese, squashes, sherbets, chutneys, pickels etc. the standards for dried fruits and nuts are given in section 2.3.47 (raisins, pistachio nuts etc.)

1. THERMALLY PROCESSED FRUITS AND VEGETABLES (CANNED / BOTTLED / FLEXIBLY PACKAGED)

1.1 Physical examination

Note the external condition of the can such as rusty spots, body dents, scratches, leakage around seams, condition of the ends etc. The external condition of the can is described in terms such as:

- a) FLAT (both ends concave)
- b) FLIPPER (a mechanical shock producing distortion in one end or both ends)
- c) SPRINGER (one end is distorted while other end is flat) and
- d) SWELL (both ends convex)

1.2 Determination of Vacuum

Place the pointed end of the vacuum gauge in the middle of the top plate of the can and press firmly to pierce the can. Note down the vacuum in millimeters of mercury (Ref: - I S I Hand book of Food Analysis (Part 1) – 1980 page 2)

1.3 Fill of Container

1.3.1 Principle

This method determines the percent total volume of a container occupied by the

contained food. It is designed primarily for cans but can be used for wide mouth glass containers also

1.3.2 Apparatus

Head space Gauge – One can be conveniently made from a straight edge a small ruler.

1.3.3 Procedure

Open the container (use a can opener for cans and remove lid for jars) and measure the distance from the container top to the food using the headspace gauge. This is usually done at the center but if the food surface is uneven, then make several measurements at different points and average them Pour out the food over a sieve to determine drained weight Wash, dry and weigh the container.

Fill the container with water to within 5 mm of the top (using the head space gauge). Weigh the container and water.

Next draw off water from the container until the water is at the same level as measured for the food. Again weigh the container and water. (Note that the water temperature should be the same during both weighings)

1.3.4 Calculation

$$\text{The \% fill of the container} = \frac{W2 - T}{W1 - T} \times 100$$

Where

T = Weight of empty container

W1 = Container plus water, first weight

W 2 = Container plus water, second weight

(Ref: - F.A.O Manuals of Food Quality Control 14 / 8 page 184)

1.4 Drained weight:

1.4.1 Principle:

The sample is drained on a standard mesh sieve. The weight of the material remaining on the sieve is expressed as percentage of the can contents.

1.4.2 Apparatus:

Sieve with square openings, 2.8 mm x 2.8 mm (No. 6 B.S.). Use a sieve of 20 cm if the total weight of contents is under 1.5 kg and of 30 cms if the weight is more than 1.5 kg.

* Table for sieve size				
Method	Capacity of container	Sieve Diameter	Mesh opening	Draining time min
Canned fruits and vegetable	a. less than 1.5 kg	20	2.8 X 2.8	2
	b. 1.5 kg or more	30	2.8 X 2.8	2
Canned tomatoes	a. less than 1.5 kg	20	11.2 X 11.2	2
	b. 1.5 kg or more	30	11.2 X 11.2	2
Washed drained wt.		20	0.3 X 0.3	5

1.4.3 Procedure:

Weigh the full can; open and pour the entire contents on a circular sieve; without shifting the product incline the sieve to facilitate drainage. In the case of products with a cavity such as peach halves, invert if necessary so that the liquid can drain through the cavity but otherwise the product should not be disturbed. Drain for two minutes; weigh the drained solids and the empty can

$$\% \text{ drained weight} = \frac{\text{Drained weight}}{\text{Net weight of contents}} \times 100$$

Net wt. of contents = Gross weight – tare weight of the can.

For canned tomatoes use a sieve with square openings of 11.2 mm x 11.2 mm.

For products in sauce, use a sieve with square openings of 0.3 mm x 0.3 mm;

Wash the contents on the sieve with water until free of adhering substances; spread on sieve and drain for 5 minutes, dry the underside of the sieve and weigh.

(Ref: - F.A.O Manuals of Food Quality Control 14 / 8 page 183 / Codex Alimentarius Commission Recommended method 36 / 7 – 1970, and method 44 – 1972)

1.5 Internal condition of the can:

Examine the internal surface for any corrosion, pitting, scratching, defects in lacquering, leakages, discolouration, detinning etc.

1.6 Determination of soluble solids

1.6.1 Principle:-

Measurement of the refractive index of the test solution at 20 °C, using a refractometer, and use of tables correlating refractive index with soluble solids content (expressed as Sucrose), or direct reading of the soluble solids content on the refractometer.

1.6.2 Apparatus

- a) **Refractometer** - indicating the refractive index by means of a scale graduated in 0.001, in order to allow readings to be estimated to 0.0002.

Refractometer - indicates the percentage by mass of Sucrose by means of a scale graduated in 0.5 %, in order to allow readings to be estimated to 0.25 %. This refractometer shall be adjusted so that at 20 ° C it registers for distilled water a soluble solid (Sucrose) content of zero.

- b) **Water circulating apparatus** - to maintain the temperature of the prisms of the refractometer constant to within 0.5 °C in the neighbourhood of 20 °C which is the reference temperature. If the temperature of circulating water is different from 20 °C use temperature correction as per table on page no.
- c) **Beaker**- capacity 250 ml

1.6.3 Procedure:

1.6.3.1 Preparation of test solution

(a) Clear liquid products

Thoroughly mix the sample and use it directly for determination.

(b) Semi thick products (purees etc)

Thoroughly mix the sample. Press a part of the sample through a gauge/muslin cloth folded in four, rejecting the first drops of the liquid and reserving the remainder of the liquid for the determination

(c) Thick products (jams, Jellies etc)

Weigh into the tared beaker to the nearest 0.01 gm, a suitable quantity (upto 40 gm) of the sample and add 100 – 150 ml of distilled water. Heat the contents of the beaker to boiling and allow to boil gently for 2- 3 minutes, stirring with a glass rod. Cool the contents and mix thoroughly. After 20 minutes weigh to the nearest 0.01gm, then filter through a fluted filter paper or a Buchner funnel into a dry vessel. Reserve the filtrate for determination

1.6.3.2 Determination

Adjust the water circulation in order to operate at the required temperature and allow it to flow to bring the prisms of the refractometer to the same temperature which shall remain constant to within 0.5°C during the determination.

Put a small quantity of the test solution (2- 3 drops are sufficient) on the fixed prism of the refractometer and immediately adjust the movable prism. Suitably illuminate the field of view. Bring the line dividing the light and dark parts of the surface in the field of view to the crossing of the threads and read the value of refractive index.

Determine percent sugar from the table

If the determination has been carried out at a temperature other than 20°C ± 0.5°C the following corrections are required

(a) For the scale indicating refractive index apply the formula

$$n_D^{20} = n_D^t + 0,0013(t - 20)$$

where

n_D^{20} is the refractive index at 20 °C;

n_D^t is the refractive index at the temperature of measurement;

t is the temperature of measurement, in degrees Celsius.

(b) For the scale indicating percentage by mass or Sucrose correct the result according to the table 1

1.6.3.3 Calculation

(a) Refractometer with refractive index scale

Read from table 2 the percentage mass of sucrose corresponding to the value of refractive index corrected for temperature if necessary. In the case of liquid or semi thick products the soluble solid content is equal to the number found. If the determination has been carried out on a diluted sample the soluble solid content is equal to

$$\frac{P \times m1}{m0}$$

Where,

P is the percentage by mass of soluble solids in the diluted solution m0 is the mass, in gm of the sample before dilution m 1 is the mass in gm of the sample after dilution

Take the result as the arithmetic mean of two determinations. Express the result to one decimal place

(b) Refractometer with sugar scale

In the case of liquid or semi thick products the soluble solid content, as a percentage by mass of the sucrose is equal to the value read, corrected for temperature if necessary. If the determination has been made on a diluted solution calculate the soluble solids as shown above

Take the result as the arithmetic mean of two determinations.

(Ref: - I.S 13815: 1993 / I.S.O 2173: 1978 Fruit and Vegetable Products Determination of Soluble solid Content - Refractometer method)

IS 13815 : 1993
ISO 2173 : 1978

TABLE 1 – Correction of readings of the refractometer with scale indicating sucrose for a temperature different from $20 \pm 0,5^{\circ}\text{C}$

Temperature $^{\circ}\text{C}$	Scale reading for soluble solids content, % (m/m)									
	5	10	15	20	25	30	40	50	60	70
Corrections to be subtracted										
15	0,29	0,31	0,33	0,34	0,34	0,35	0,37	0,38	0,39	0,40
16	0,24	0,25	0,26	0,27	0,28	0,28	0,30	0,30	0,31	0,32
17	0,18	0,19	0,20	0,21	0,21	0,21	0,22	0,23	0,23	0,24
18	0,13	0,13	0,14	0,14	0,14	0,14	0,15	0,15	0,16	0,16
19	0,06	0,06	0,07	0,07	0,07	0,07	0,08	0,08	0,08	0,08
Corrections to be added										
21	0,07	0,07	0,07	0,07	0,08	0,08	0,08	0,08	0,08	0,08
22	0,13	0,14	0,14	0,15	0,15	0,15	0,15	0,16	0,16	0,16
23	0,20	0,21	0,22	0,22	0,23	0,23	0,23	0,24	0,24	0,24
24	0,27	0,28	0,29	0,30	0,30	0,31	0,31	0,31	0,32	0,32
25	0,35	0,36	0,37	0,38	0,38	0,39	0,40	0,40	0,40	0,40

TABLE 2 – Refractive index and corresponding percentage by mass of soluble solids (sucrose)

Refractive index	Soluble solids (sucrose) content	Refractive index	Soluble solids (sucrose) content	Refractive index	Soluble solids (sucrose) content	Refractive index	Soluble solids (sucrose) content
n_D^{20}	% (m/m)	n_D^{20}	% (m/m)	n_D^{20}	% (m/m)	n_D^{20}	% (m/m)
1,333 0	0	1,367 2	22	1,407 6	44	1,455 8	66
1,334 4	1	1,368 9	23	1,409 6	45	1,458 2	67
1,335 9	2	1,370 6	24			1,460 6	68
1,337 3	3	1,372 3	25	1,411 7	46	1,463 0	69
1,338 8	4			1,413 7	47	1,465 4	70
1,340 3	5	1,374 0	26	1,415 8	48		
		1,375 8	27	1,417 9	49	1,467 9	71
1,341 8	6	1,377 5	28	1,420 1	50	1,470 3	72
1,343 3	7	1,379 3	29			1,472 8	73
1,344 8	8	1,381 1	30	1,422 2	51	1,475 3	74
1,346 3	9			1,424 3	52	1,477 8	75
1,347 8	10	1,382 9	31	1,426 5	53		
		1,384 7	32	1,428 6	54	1,480 3	76
1,349 4	11	1,386 5	33	1,430 8	55	1,482 9	77
1,350 9	12	1,388 3	34			1,485 4	78
1,352 5	13	1,390 2	35	1,433 0	56	1,488 0	79
1,354 1	14			1,435 2	57	1,490 6	80
1,355 7	15	1,392 0	36	1,437 4	58		
		1,393 9	37	1,439 7	59	1,493 3	81
1,357 3	16	1,395 8	38	1,441 9	60	1,495 9	82
1,358 9	17	1,397 8	39			1,498 5	83
1,360 5	18	1,399 7	40	1,444 2	61	1,501 2	84
1,362 2	19			1,446 5	62	1,503 9	85
1,363 8	20	1,401 6	41	1,448 8	63		
		1,403 6	42	1,451 1	64		
1,365 5	21	1,405 6	43	1,453 5	65		

1.7 Determination of Sodium Chloride (salt content) in brine:

1.7.1 Principle:

Direct titration of NaCl in brine with standard silver nitrate solution based on the Mohr method is adequate for routine analysis.

1.7.2 Reagents:

- i) 5% (w/w) aqueous potassium chromate solution.
- ii) 0.1N aqueous silver nitrate solution.
- iii) N Sodium Hydroxide

1.7.3 Procedure:

Take 5 to 10 gm of liquid portion from the drained weight determination. If it is acidic, neutralize it with standard Sodium Hydroxide using phenolphthalein as indicator. Add 1 ml of 5% aqueous potassium chromate solution and titrate with 0.1N AgNO₃ solution to produce red-brown end point.

$$\text{NaCl \%} = \frac{\text{Titre value} \times \text{Normality of AgNO}_3 \times 58.4 \times 100}{\text{Weight of the sample} \times 1000}$$

(Ref: - I S I Handbook of Food Analysis (Part VIII) – 1984 page 5)

1.8 Metallic contaminants:

Follow procedures given in the Manual of Methods of Analysis - Metals

1.9 Pesticide residues:

Follow procedures given in the Manual on Pesticide residues.

1.10 Microbiological examination:

Follow procedures given in the relevant Manual.

2.0 Thermally processed fruit and vegetable juices fruit beverages / fruit drinks and fruit nectars (canned / bottled) / flexibly/ aseptically packaged and non-thermally processed fruit beverages / drinks.

2.1 Total Solids

(a) Insoluble matter absent (applicable to jellies and syrups also)

Digest pure quartz sand that passes No 40 but not No 60 sieve with HCl, wash acid free, dry and ignite. Preserve in stoppered bottle. Place 26- 30 gm sand and a short stirring rod in the dish about 55 mm in diameter and 40 mm deep, fitted with cover. Dry the dish thoroughly cool in a dessicator and weigh immediately. Add enough sample to yield about 1 gm dry matter and mix thoroughly with sand. Heat on steam bath 15 – 20 minutes, stirring at 2 – 3 minutes interval or until mass becomes too stiff to manipulate readily.

Dry at less than 70 ° C under pressure of about 50 mm Hg in vaccum oven passing a current of dry air (dried over CaSO₄ or P₂O₅). Make trial weighings at 2 hours interval after 18 hrs until change in weight is equal to 2 mg.

$$\text{Calculation: \% Total Solids} = \frac{(W3-W1) \times 100}{(W2-W1)}$$

(b) Insoluble matter present (applicable to jams, marmalades and preserves also)

Accurately weigh into a large flat bottomed dish sufficient sample that will give 2- 3 gm dry matter. If necessary to secure thin layer of material, add few ml water and mix thoroughly. Dry at 70 ° C under pressure less than 100 mm Hg until consecutive weighing's made at 2 hr intervals vary less than 3mg.

(Ref: - A.O.A.C 17th edition, 2000, Official method 920.151 Solids (Total) in Fruits and Fruit Products).

2.2 Total soluble solids:

Follow method given in Clause 1.6 above

2.3 Determination of pH Value: -

pH is the measurement of H⁺ ion activity; It measures active acidity. pH may be determined by measuring the electrode potential between glass and reference electrodes; pH meter is standardised using standard pH buffers.

Use homogenized sample for the determination of pH.

Preparation of sample for pH measurement:

Liquids

Immerse the standardized electrode tip into the solution and stir the sample gently by means of a rod or "flea" to give a constant pH value.

Non-homogeneous products

If it is useful to know the pH of different components of the sample or differences between the pH at several points of the test portion, separate these as best as possible, homogenize and read them separately. For a bulk pH measurement, homogenize a representative aliquot to give a moist homogeneous mixture. Treat bulk as for moist homogeneous products (7.1.4).

Dry products

Have a standard practice for the dilution of dried materials - particularly when comparing the pH of sub-samples of the same product, as pH may change with the extent of dilution.

For example homogenize with an equal volume of distilled or deionized water. Immerse the electrode in the sample and mix gently until a constant pH reading is obtained.

Moist homogeneous products

Homogenise the sample, immerse or embed the electrode and ensure that there is adequate contact between probe and sample. Read when the meter reading is stable. Do three separate measurements on the test sample - the extreme readings should not differ by more than 0.15 pH units. Take as the result the arithmetic mean of the three readings.

2.4 Determination of acidity (Applicable to jams , jellies also)

Titration acidity can be expressed conveniently in gms acid per 100 gm or per 100 ml as appropriate, by using the factor appropriate to the acid as follows:

1 ml of 0.1 N NaOH equals

Malic acid - 0.0067 gms

Oxalic acid - 0.0045 gms

Citric acid monohydrate - 0.0070 gms

Citric acid anhydrous - 0.0064 gms

Tartaric acid - 0.0075 gms

Lactic acid – 0.0090 gms

Acetic acid – 0.0060 gms

Oleic acid – 0.00282 gms

(a) Colourless or slightly coloured solutions

Take 10 gm well mixed juice, dilute to 250 ml with neutralised or recently boiled water. Titrate with 0.1 N NaOH using 0.3 ml phenolphthalein for each 100 ml of the solution to pink end point persisting for 30 seconds.

Report acidity as ml 0.1 N NaOH per 100 gm or 100 ml as required.

(b) Highly coloured solutions**Sample preparation:**

- 1) Dilute known weight of sample with neutralized water and titrate to just before end point with 0.1 N alkali using 0.3 ml phenolphthalein for each

100 ml solution being titrated. Transfer measured volume (2- 3 ml) of solution into about 20 ml of neutral water in small beaker. (in this extra dilution fruit juice becomes so pale that phenolphthalein colour is easily seen). If test shows that end point is not reached, pour extra diluted solution back into original solution, add more alkali and continue titration to end point. By comparing dilutions in small beakers differences produced by a few drops of 0.1 N alkali can be easily observed.

- 2) In case of jams and jellies mix sample thoroughly. Weigh 300 gm mixed sample into a 2 litre flask and dissolve in water heating on steam bath if necessary. Apply as little heat as possible to minimize inversion of sucrose. Cool dilute to volume, mix thoroughly by shaking and use aliquots for various determinations. If insoluble material is present mix thoroughly and filter first.

Electromeric method can be used to raise the pH slowly to 8.15 to remove colour interference.

(Ref: - A.O.A.C 17th edn, 2000, Official method 942.15 Acidity (Titrable) of fruit products read with A.O.A.C official method 920. 149 Preparation of test sample).

Potentiometric method can also be used for the same. Slowly add 0.1N alkali till its pH raises to 8.1. Calculate acidity as predominant acid present in the sample by the formula:

$$\text{Acidity} = \frac{\text{Eq.wt. of acid} \times N \text{ NaOH} \times 100}{\text{Sample weight/vol. of alcohol} \times 1000}$$

2.5 Determination of Volatile acids

2.5.1 Apparatus

(a) Steam Distillation Apparatus as shown below

2.5.2 Procedure

Add about 600 ml boiled water to outer chamber of still. Dissolve 10 gm test sample in water, dilute to 25 ml. Pour this into inner chamber and stopper. Boil water 3 minutes with side arm opening. Close and distill about 300 ml into Erlenmeyer flask. Add 0.5 ml phenolphthalein to distillate and titrate rapidly with 0.1 N NaOH until pink colour persists 15 seconds

Express results as gm acetic acid per 100 ml or gm

$$1 \text{ ml } .1 \text{ N NaOH} = 0.0060 \text{ gm acetic acid} \times 10 \text{ (10 gm test sample taken)}$$

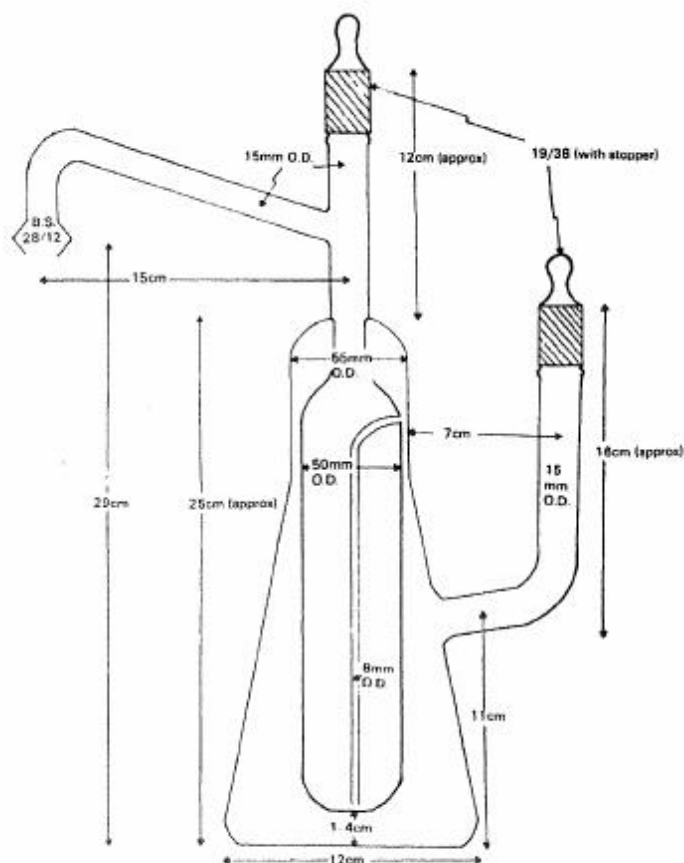


Figure 960.16—Steam distillation flask.

(A.O.A.C 17th edn, 2000, Figure 960.16)

(Ref :- A.O.A.C 17th edn ,2000 Official method 925.34 Acidity (Volatile) of Fruit

Products, Steam Distillation method read with A.O.A.C official method 964.08 acidity (Total Volatile) of wines

2.6 Determination of total Sugars:

The presence of added sucrose can be detected by determining sugars before and after inversion by copper- reduction methods.

(Standardization of Fehling's solution: Prepare standard dextrose solution into a 50ml. burette. Find the titre (volume of dextrose solution required to reduce all the copper in 10 ml. of Fehling solution) corresponding to the standard dextrose solution (Refer table below). Pipette 10 ml of Fehling's solution into a 300 ml of conical flask and run in from the burette almost the whole of the standard dextrose solution required to effect reduction of all the copper, so that more than one millilitre will be required later to complete the titration. Heat the flask containing mixture over wire gauze. Gently boil the contents of the flask for 2 minutes. At the end of two minutes of boiling add without interrupting boiling, one ml. of methylene blue indicator solution. While the contents of the flask begins to boil, begin to add standard dextrose solution (one or two drops at a time) from the burette till blue color of indicator disappears [The titration should be completed within one minute so that the contents of the flask boil together for 3 minutes without interpretation. Note the titre (that is total volume in ml. of std. dextrose solution used for the reduction of all the copper in 10 ml. of Fehling's solution). Multiply the titre (obtd. by direct titration) by the number of milligrams of anhydrous dextrose in one millilitre of standard dextrose solution to obtain the dextrose factor. Compare this factor with the dextrose factor and determine correction.

Dextrose factors for 10 ml. of Fehling's Solution		
Titre (ml)	Dextrose factor	Dextrose content per 100 ml of solution (mg)
15	49.1	327
16	49.2	307
17	49.3	289
18	49.3	274
19	49.4	260
20	49.5	247.4

21	49.5	235.8
22	49.6	225.5
23	49.7	216.1
24	49.8	207.4
25	49.8	199.3
26	49.9	191.8
27	49.9	184.9
28	50.0	178.5
29	50.0	172.5
30	50.1	167.0
31	50.2	161.8
32	50.2	156.9
33	50.3	152.4
34	50.3	148.0
35	50.4	148.9
36	50.4	140.0
37	50.5	136.4
38	50.5	132.9
39	50.6	129.6
40	50.6	126.5
41	50.7	123.6
42	50.7	120.8
43	50.8	118.1
44	50.8	115.5
45	50.9	113.0
46	50.9	110.6
47	51.0	108.4
48	51.0	106.2
49	51.0	104.1
50	51.1	102.2
Miligrams of anhydrous dextrose corresponding to 10 ml of Fehlings solution		

Reference: Table 2: IS 6287:1985, Methods for sampling and analysis for sugar confectionery, Pg.11

Transfer test sample representing about 2- 2.5 gm sugar to 200 ml volumetric flask, dilute to about 100 ml and add excess of saturated neutral Lead acetate solution (about 2 ml is usually enough). Mix, dilute to volume and filter, discarding the first few ml filtrate. Add dry Pot. or Sod. Oxalate to precipitate excess lead used in clarification, mix and filter, discarding the first few ml filtrate.

Note: Use of Potassium Ferrocyanide and Zinc acetate is preferable instead of Lead acetate and Sodium oxalate, due to safety issues.

Take 25 ml filtrate or aliquot containing (if possible) 50 – 200 mg reducing sugars and titrate with mixed Fehling A and B solution using Lane and Eynon Volumetric method.

(1) Fehling A: Dissolve 69.28-g copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in distilled water. Dilute to 1000 ml. Filter and store in amber coloured bottle.

(2) Fehling B: Dissolve 346 g Rochelle salt (potassium sodium tartrate) ($\text{K Na C}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$) and 100 g NaOH in distilled water. Dilute to 1000 ml. Filter and store in amber coloured bottle.

For inversion at room temperature, transfer 50 ml aliquot clarified and de-leaded solution to a 100 ml volumetric flask, add 10 ml HCl (1+ 1) and let stand at room temperature for 24 hours. (For inversion, the sample with HCl can be heated at 70° C for 1 hr. This saves time and makes the whole process shorter). Neutralise exactly with conc. NaOH solution using phenolphthalein and dilute to 100 ml. Titrate against mixed Fehling A and B solution (25 ml of Fehlings Solution can be considered for the purpose) and determine total sugar as invert sugar (Calculate added sugar by deducting reducing sugars from total sugars).

Reducing and total reducing sugar can be calculated as,

$$\text{Reducing sugar (\%)} = \frac{\text{mg. of invert sugar} \times \text{vol. made up} \times 100}{\text{TR} \times \text{Wt. of sample} \times 1000}$$

$$\text{Total reducing sugar (\%)} = \frac{\text{mg. of invert sugar} \times \text{final vol. made up} \times \text{original volume} \times 100}{\text{TR} \times \text{Wt. of sample} \times \text{aliquot taken for inversion} \times 1000}$$

$$\text{Total sugar (as sucrose) (\%)} = (\text{Total reducing sugar} - \text{Reducing sugar}) \times 0.95 + \text{Reducing sugar}$$

Added sugar = Total sugars – Reducing sugars

(Ref :- A.O.A.C 17th edn, 2000 Official Method 925.35 Sucrose in Fruits and Fruit Products read with A.O.A.C Official method 923.09 Lane and Eynon general volumetric method)

*HPLC method can also be used for sugar profiling.

(Ref: - A.O.A.C 984.17: 'Method for the determination of Sugars in foods', *Jr. Agri. and Food Chemistry*, 19(3):551-54, (1971)(Modified) Brobst, K.M.

"Gas-Liquid Chromatography of Trimethylsilyl Derivatives, *Methods in Carbohydrate Chemistry*," 6:3-8, Academic Press, New York, NY, (1972). (Modified))

2.8 Determination of Vitamin C (Ascorbic Acid):

The ascorbic acid content in fruits and vegetables can be estimated by macerating the sample with stabilising agents such as 20 % metaphosphoric acid.

2.8.1 Principle:

2, 6 -dichlorophenol indophenol is reduced to a colourless form by ascorbic acid. The reaction is specific for ascorbic acid at pH 1 to 3.5. The dye is blue in alkaline solution and pink in acid.

2.8.2 Reagents:

(1) Standard Indophenol Solution – Dissolve 0.05 gm 2, 6 dichlorophenol indophenol in 50 ml. water, to which 42 mg. sodium carbonate is added, and make upto 200 ml. with water and filter. Sodium carbonate is added for stability purpose. The dye solution keeps for a few weeks if stored in refrigerator. Prepare fresh if possible and standardize before use.

Blank correction: Dissolve 50 mg 2,6-dichloroindophenol Na salt that has been stored in desiccator over soda lime, in 50 mL H₂O to which has been added 42 mg NaHCO₃; shake vigorously, and when dye dis solves, dilute to 200 mL with H₂O. Filter through fluted paper into amber glass-stoppered bottle. Keep stoppered, out of direct sunlight, and

store in refrigerator. (Decomposition products that make end point in distinct occur in some batches of dry indophenol and also develop with time in stock solution. Add 5.0 mL extracting solution containing excess ascorbic acid to 15 mL dye reagent. If reduced solution is not practically colorless, discard, and prepare new stock solution. If dry dye is at fault, obtain new supply.)

Transfer three 2.0 mL aliquots ascorbic acid standard solution to each of three 50 mL Erlenmeyers containing 5.0 mL $\text{HPO}_3\text{-CH}_3\text{COOH}$ solution, B(a)(1). Titrate rapidly with indophenol solution from 50 mL burette until light but distinct rose pink persists ³⁵ s. (Each titration should require ca 15 mL indophenol solution, and titrations should check within 0.1 mL). Similarly titrate 3 blanks composed of 7.0 mL $\text{HPO}_3\text{-CH}_3\text{COOH}$ solution, B(a)(1), plus volume H_2O ca equal to volume indophenol solution used in direct titrations. After subtracting average blanks (usually ca 0.1 mL) from standardization titrations, calculate and express concentration of indophenol solution as mg ascorbic acid equivalent to 1.0 mL reagent. Standardize indophenol solution daily with freshly prepared ascorbic acid standard solution.

(2) Standard Ascorbic acid solution – Dissolve 0.05 gm pure ascorbic acid in 60 ml of 20 % metaphosphoric acid (HPO_3) and dilute with water to exactly 250 ml in a volumetric flask.

(3) Metaphosphoric acid - 20 %

(4) Acetone

2.8.3 Standardisation of Dye:

Pipette 10 ml of standard Ascorbic acid solution in a small flask and titrate with indophenol solution until a faint pink colour persists for 15 seconds. Express the concentration as mg Ascorbic acid equivalent to 1 ml of dye solution i.e 10 ml of

Ascorbic acid solution = 0.002 gm ascorbic acid

If 0.002 gm ascorbic acid requires V ml dye solution to neutralize it then 1 ml dye solution = $0.002 / V$ gm ascorbic acid.

2.8.4 Procedure

Pipette 50 ml of unconcentrated juice (or the equivalent of concentrated juice) into a 100 ml volumetric flask, add 25 ml of 20 % metaphosphoric acid as stabilizing agent and dilute to volume. Pipette 10 ml in a small flask and add 2.5 ml acetone. Titrate with indophenol solution until a faint pink colour persists for 15 seconds.

2.8.5 Calculation

Vitamin of Vitamin C per 100g/ml = Titer value x Dye factor X Vol made up X 100

Where, Aliquot x : is wt. or vol. of sample

mg Ascorbic acid /g, tablet, ml, etc. = $(X - B) \times (F/E) \times (V/Y)$

Where, X = average ml for test solution titration,

B = average ml for test blank titration,

F = mg ascorbic acid equivalent to one ml iodophenol standard solution,

E = no. of g, tablets, ml, etc. assayed

V= volume initial test solution and

Y= volume test solution titrated

Note:-

Acetone may be omitted if sulphur dioxide is known to be absent. Its function is to form the acetone bisulphate complex with sulphur dioxide which otherwise interferes with the titration. Sometime a small proportion of the ascorbic acid in foods becomes reversibly oxidized during aging and forms dehydroascorbic acid. If this is suspected, first estimate the ascorbic acid as above, then through another portion of the solution pass a stream of Hydrogen sulphide for 10 minutes. Stopper the flask and allow it to

stand overnight in a refrigerator. Then remove hydrogen sulphide by bubbling nitrogen through the mixture and titrated as before. The difference between the two titrations gives a measure of the dehydroascorbic acid. One international unit of vitamin C = 50 µg ascorbic acid.

(Ref :- F.A.O Manuals of Food Quality Control 14 / 8, page 194 / Pearson's Composition and Analysis of Foods 9th edn,1991, page 264 and AOAC Official Method 967.21 Ascorbic acid in Vitamin preparation and juices)

2.9 Determination of Ethanol Content

2.9.1 Principle

Note:

- This test method covers only the product which does not contain ethanol as an ingredient.
- The method is not applicable to products containing more than 5 % (*m/m*) of ethanol.

Separation of ethanol by distillation followed by oxidation by Potassium dichromate in a sulphuric acid medium and determination of excess dichromate by Ferrous ammonium sulphate in the presence of Ferrous 1, 10 phenanthroline as indicator

2.9.2 Reagents

- a) Concentrated Sulphuric acid – 1.84 gm / ml.
- b) Dilute sulphuric acid –1.49 gm / ml (1+ 1).
- c) Calcium hydroxide suspension obtained by shaking 110 – 112 gm of Calcium oxide in 1 litre water.
- d) Potassium Dichromate solution containing 42.572 gm of $K_2Cr_2O_7$ per litre. 1 ml of this solution is equivalent to 0.01 gm ethanol.
- e) Potassium Permanganate solution containing 1.372 gm of $KMnO_4$ per litre.

10 ml of this solution is equivalent to 1 ml of Ammonium ferrous sulphate solution.

f) Ammonium Ferrous Sulphate solution $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)$

Dissolve 170.2 gm ammonium ferrous sulphate hexahydrate in water. Add 20 ml of concentrated sulphuric acid and dilute to 1 litre with water. Stabilize by the addition of Aluminium chips. 2 ml of this solution is equivalent to 1 ml Potassium Dichromate solution.

g) Ferrous – 1, 10 phenanthroline solution – Dissolve 0.695 gm of Ferrous sulphate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) in 100 ml water. Add 1.485 gm of 1,10 phenanthroline monohydrate and heat to aid solution.

This solution keeps well

2.9.3 Apparatus

- (a) Distillation apparatus comprising a 500 ml flask surmounted by a fractionation column and a condenser ending in a slightly tapered extension piece long enough to reach the bottom of a 100 ml volumetric flask Any other steam distillation may be used provided it satisfies the following criteria

No loss of ethanol greater than 0.02 % shall occur in the course of a distillation, i.e 200 ml of a 10 % (v/v) ethanol / water mixture distilled five times in succession shall contain at least 9.9 % of ethanol after the last distillation

- (b) Heating apparatus which does not cause even slight decomposition of the extractable material contained in the flask.

- (c) Volumetric flask – 100 ml capacity.

- (d) Pipettes – 5 ml, 10 ml and 20 ml capacity.

- (e) Burettes – 50 ml capacity.

- (f) Wide necked flasks –250 ml capacity with ground glass stoppers, clean dry free from grease and airtight(absolute tightness can be ensured with the aid of a poly

tetra fluorethylene sleeve).

(g) Blender

(h) Balance capable of weighing to the nearest 0.01 gm

2.9.4 Preparation of Sample

(a) Solid or thick products (puree, marmalade or jam, fruits, vegetables)

Blend mechanically whole of the sample. Take care that the temperature of the product does not rise. Take sufficient quantity to carry out two parallel determinations.

(b) Liquid products (juices, pulps and syrups)

Thoroughly mix the sample. Take sufficient quantity to enable two parallel determinations.

2.9.5 Procedure

Weigh to the nearest 0.01gm sufficient quantity of sample (in case of liquids volume) so that the quantity of ethanol collected in 100 ml of distillate is less than 1gm. Dilute the test portion with about 50 ml water and transfer quantitatively to the flask of the distillation apparatus. Rinse the vessel used to take the test portion with not more than 120 ml water and transfer it to the flask.

Make the product slightly alkaline (pH 8 ± 0.2) with the Calcium hydroxide suspension shaken before use Add glass beads or porcelain to control the rate of boiling. Pour 10 ml water in a 100 ml volumetric flask and insert the tapered extension of the distillation apparatus so that it is immersed in the liquid.

Distill the diluted test portion (previously made alkaline) in such a way that the distillate when it reaches the volumetric flask is at a relatively low temperature (15 –

20°C). Collect about 80 – 85 ml of distillate. Stop the distillation, avoid back suction of the distillate and rinse the condenser and extension with a few ml of water. Shake the volumetric flask to mix the contents. If necessary immerse the flask in cold water at 15 – 20°C for a few minutes. Dilute the Contents of the volumetric flask to the mark with water and shake.

2.9.6 Oxidation

Pour 20 ml (v1) of Potassium dichromate solution accurately measured and 20 ml of dilute sulphuric acid into a 250 ml flask with a ground glass stopper and shake. Add 10 ml (v0) of distillate accurately measured. Stopper the flask, moistening the stopper with a drop of sulphuric acid. Shake the flask and wait for at least 30 minutes shaking the flask from time to time. The resultant mixture should in no case assume the green coloration of the Chromium cation as this would indicate that the ethanol content of the test portion was too high. If this occurs, recommence the oxidation taking a smaller portion of the distillate (e.g 5 ml). If necessary recommence both distillation and oxidation taking a smaller test portion. Take account of any such changes in the calculations.

If the test portion contains too little ethanol, a smaller amount of Potassium dichromate solution may be used i.e 10 ml or 5 ml of the solution diluted with 10 or 15 ml distilled water respectively. Take account of any such changes in the calculations.

2.9.7 Titration

Titrate the excess of dichromate, using the ammonium ferrous sulphate solution. The excess of dichromate should be at least equal to 20 % of the quantity used for the blank test. Shake the flask after each addition. When the colour changes to greenish blue, add 4 drops of ferrous 1, 10 phenanthroline solution. Continue the addition of ammonium ferrous sulphate solution until the colour of the medium changes from greenish blue to brown. If the end point is passed, return to it precisely by adding Potassium permanganate solution.

Deduct from the volume of ammonium ferrous sulphate solution used, one tenth of the volume of potassium permanganate solution added. Let (v₂) be the volume remaining after this deduction which represents the exact volume of ammonium ferrous sulphate equivalent to the excess potassium dichromate

Carry out two determinations on the same sample

2.9.8 Blank test

Carry out a blank test under the same conditions as for the titration replacing the volume (v₀) of the distillate by the same volume of distilled water. Let (v₃) be the volume of ammonium ferrous sulphate solution used

2.9.9 Calculation

(a) Solid Products

$$\text{Ethanol content \% weight} = \frac{0.01 V_1 \times V_3 - V_2 \times 100 \times 100}{V_3 - V_0 \quad m}$$

Where,

m = weight in gm of the test sample

V₀ = volume in ml of distillate taken for the titration

V₁ = volume in ml of Potassium dichromate solution used for the oxidation

V₂ = volume in ml of ammonium ferrous sulphate solution used for the back titration of the dichromate

V₃ = volume in ml of ammonium ferrous sulphate used in blank test

(b) Liquid Products

$$\text{Ethanol content \% weight} = \frac{0.01 V_1 \times V_3 - V_2 \times 100 \times 100}{V_3 - V_0 \quad V_4}$$

Where,

V₀, V₁, V₂, V₃ have the same meaning as above and

V₄ = volume in ml of the test portion

Note:-

In case of products having essential oils the distillate is turbid with drops of essential oil floating on the surface. The method has to be modified. As follows:

Collect the distillate in a 100 ml volumetric flask and allow it to stand for two hrs. Dilute to the mark with water, the interface between the two phases (essential oil and water) being at the level of the mark. Allow to stand for a further 1hr to 2 hrs. Discard the small quantity of essential oil collected on the surface either by suction with a fine pipette or by filtration through paper in a covered funnel

Transfer the still turbid filtrate to a 150 ml flask and add 10 gm of polystyrene granules (granule size 1 mm to 2 mm). Shake the stoppered flask for 15 minutes and then filter the mixture through gauge in a covered funnel. The liquid should then have become clear and have lost its odour completely. Proceed with the determination on this liquid.

(Ref: - I.S 15096: 2002 / I.S.O 2448: 1998 Fruit and Vegetable Products Determination of Ethanol Content)

2.10 Determination of Mineral Impurities**2.10.1 Principle**

Separation of organic matter by floatation and of heavy impurities by sedimentation, incineration of the sediment at approximately 600 °C and weighing of the residue obtained.

2.10.2 Regents:

- a) Sodium Chloride - Approx 15 % (w/v) solution
- b) Silver Nitrate - approx 17 gm / litre

2.10.3 Apparatus

- a) Blender
- b) Beakers 800 and 2000 ml capacity
- c) Ashless filter paper
- d) Incineration dishes – porcelain or platinum
- e) Muffle Furnace capable of being maintained at $600 \pm 10^{\circ}\text{C}$
- f) Dessicator
- g) Balance

2.10.4 Preparation of test sample

(a) General case

Thoroughly mix the entire laboratory sample, using if necessary the blender. Allow frozen or deep frozen products to thaw in a closed vessel and add the liquid formed during the process to the product before mixing.

(b) Dried products

Weigh 100 gm of the product, transfer to a 800 ml beaker and add 400 ml water. Bring to boil then leave overnight at room temperature to allow the product to rehydrate

2.10.5 Amount of sample to be taken for test

Weigh 500 gm of test sample in general case. If the sample is less than 500gm weigh all of it. For dried products take the entire test sample

2.10.6 Determination

(a) Separation of sediment

Transfer test sample into a 2 litre beaker. Add water until the beaker is almost full and mix by agitating if necessary using a stirring rod. Leave for about 10 minutes and pour the upper layer and water into a second 2 litre beaker. Again fill the first

beaker with water, mix, agitate and leave for 10 minutes. Fill the second beaker with water, mix, agitate and leave for 10 minutes. Then pour the upper layer from second beaker into another 2 litre beaker and the upper layer from the first beaker into second beaker. Repeat these operations carefully, pouring the upper layer of the third beaker into the sink, until all the floating fruit pulp has been discarded. Combine all the sediments in the first beaker. Eliminate seeds or fruit pulp which may have settled by treating the sediments with warm sodium chloride solution. Remove the sodium chloride by washing with warm water, verifying the absence of chloride ions by testing the washings with silver nitrate solution. Quantitatively transfer the remaining residue to ashless filter paper placed in a funnel and rinse with water.

(b) Incineration

Heat an empty dish in the muffle furnace, cool in a dessicator and weigh accurately to the nearest 0.0002 gm.

Transfer the filter paper with the residue to the incineration dish. Heat the dish over a burner for a few minutes, then transfer to muffle furnace maintained at $600 \pm 10^\circ\text{C}$ and incinerate for 1 hour. Cool in a dessicator and weigh to the nearest 0.0002 gm. Carry out at least two determinations on the same sample.

2.10.7 Calculation:

$$\text{Mineral impurities \% by mass} = \frac{(m_2 - m_1) \times 100}{m_0}$$

Where,

m_0 = mass in gm of test portion

m_1 = mass in gm of empty dish

m_2 = mass in gm of dish and incinerated residue

(Ref: - I.S 13816: 1993 / I.S.O 762:1982 Fruit and Vegetable Products –Determination of Mineral Impurities Content)

2.11 Determination of Fruit content

2.11.1 Principle

By the addition of Formaldehyde one H ion is liberated per molecule of amino acid. It is titrated with alkali. The secondary amino group of histidine does not react, those of proline and hydroxy proline react to about 75 %. Tertiary nitrogen and guanidine – groups undergo no reaction.

2.11.2 Apparatus

pH meter

2.11. 3 Reagents:

- (a) Sodium Hydroxide 0. 25 N
- (b) Formaldehyde solution – Pure formalin of at least 35 % is brought exactly to pH 8.1 with dilute sodium hydroxide as determined by means of a pH meter.
- (c) Hydrogen Peroxide – pure 30 %

2.11.4 Procedure:

25 ml of fruit juice (for lemon juice 10 ml + 10 ml distilled water or the corresponding amount of concentrate diluted to this volume are neutralised in a beaker with 0.25 N NaOH to pH 8.1 on the pH meter. 10 ml of formaldehyde solution is then added. After about 1 minute the solution is titrated potentiometrically to pH 8.1 with 0.25 N Sodium Hydroxide.

If more than 20 ml of 0.25 N Sodium Hydroxide is required the titration is to be repeated using 15 ml of formaldehyde solution instead of 10 ml. When sulphur dioxide is present the sample is treated with a few drops of 30 % hydrogen peroxide before neutralization.

2.11.5 Calculation

The amount of alkali used in titration expressed as ml 0.1 N alkali and referred to 100 ml fruit juice or 100 gm concentrate is equal to the formol number of the sample under test. Calculate to whole numbers (without decimals)

Interpretation –

As fruits ripen, the formol number of the juice tends to decrease, as a rule. Conversely on storage of the juice a slight increase may be noticed. Various factors can lead to a lowering of the formol number of a fruit juice e. g. treatment with ion exchangers or addition of ascorbic acid

In the literature the formol number may also be found defined as ml N alkali for each 100 ml sample which corresponds to values 10 times smaller than those given by the preceding method of calculation

For orange juice the % fruit is $\frac{1.05 F}{1.4}$

Where, F is the formal number

(Ref :- F.A.O Manuals of Food Quality Control 14 / 8 page 189 / Pearson's Composition and Analysis of Foods 9th edn, 1991, Page 271-72)

2.12 Total soluble solids free of added salt (in Vegetable juices and Tomato juice)

2.12.1 Procedure

Determine salt titrimetrically as stated above. Determine total solids and insoluble solids to arrive at total soluble solids. Deduct salt content from total soluble solids or deduct salt content from total soluble solids as determined in 1.6.

3.0 Thermally Processed Fruit Nectars / Fruit Drinks / Beverages / Fruit Pulp / Vegetable juice / pulp / puree (Canned/ Bottled / Poly packed)

3.1 Total soluble solids - Follow method given in clause 1.6

3.2 Min Fruit Content – Follow method given in clause 2.11

3.3 Ethanol Content – Follow method given in clause 2.9

3.4 Essential Oil Content – Follow method given in clause 2.7

3.5 Total solids exclusive of added sugar –

Determine added sugar as per method in clause 2.6. Determine total soluble solids (total solids – insoluble solids) and deduct the amount of added sugar.

3.6 Microbiological analysis of thermally processed food

<u>Parameters</u>	<u>Method</u>
Aerobic Plate Count (30 deg /72 hrs)	IS : 5402 : 2012
Coliform Count	IS: 5401 (Part 1) - 2002
Yeast Count (25 deg/ 120hrs)	IS: 5403-1999
Mould Count (25 deg/ 120hrs)	IS : 5403-1999
Detection of E. coli	IS : 5887 (Part 1) - 1976
Detection of S. aureus	IS: 5887 (Part 2) 1976
Detection of Shigella spp	IS: 5887 (Part 7) 1999
Detection of Clostridium botulinum	IS: 5887 (Part 4) 1999
Detection of Vibrio cholerae	IS: 5887 (Part 5) - 1976

4.0 Soup Powders

4.1 Determination of Moisture Content

4.1.1 Procedure

Weigh accurately about 5 gm of well mixed sample in a previously dried and tared moisture dish (about 75 mm wide and 25 mm deep). Place the dish in an air oven maintained at $105 \pm 2^{\circ}\text{C}$ and dry at least for 2 hours. Cool in a dessicator and weigh.

Repeat the process of heating, cooling and weighing until the difference between two successive weighings is less than 1 mg Record the lowest weight.

4.1.2 Calculation

$$\text{Moisture Percent by weight} = 100 \frac{(M_1 - M_2)}{M_1 - M}$$

Where, M_1 = weight in gm of dish with material before drying

M_2 = weight in gm of dish with the dried material

M = weight in gm of empty dish

(Ref: - I.S.I Handbook of Food Analysis (Part VIII) – 1984 page 12 /Determination of Moisture in Dehydrated Vegetables)

5.0 Tamarind Pulp / Puree / Concentrate

5.1 Determination of T.S.S - Follow method given in clause 1.6

5.2Determination of Titrable acidity – Follow method given in clause 2.4

5.3 Determination of Ash Insoluble in Hydrochloric acid

5.3.1 Reagents

- a) Hydrochloric acid - 10 % solution
- b) Silver Nitrate – approx 17 gm per litre

5.3.2 Apparatus

- (a) Blender
- (b) Muffle Furnace
- (c) Boiling water bath
- (d) Drying oven - capable of being maintained at $103 \pm 2^\circ \text{C}$
- (e) Dessicator

- (f) Dishes – silica or platinum
- (g) Ashless filter paper
- (h) Analytical Balance

5.3.3 Procedure

Weigh to the nearest 0.01 gm, 5 – 10 gm test sample according to the water content of the product in a previously dried and tared dish. Place the dish and its contents on a boiling water bath and evaporate the water present in the product. Dry in the oven. After drying carbonize and then completely incinerate in the muffle furnace maintained at 525 ± 25 °C till a grey ash is obtained. Allow to cool in a dessicator. Add 10 – 25 ml of dilute hydrochloric acid solution (4 + 1), cover with a watch glass and heat on a boiling water bath for 15 minutes.

Transfer the residue to an ashless filter paper placed in a funnel. Rinse the dish with hot water and transfer the contents of the dish to the filter paper. Wash the filter paper and its contents until there is no trace of chloride ions in the liquid flowing from the funnel (test with silver nitrate solution). Prepare a new dish or clean the first dish, heat in the muffle furnace to the incineration temperature, allow to cool in a dessicator and weigh to the nearest 0.0002 gm. Place the filter paper and residue in the dish, dry in oven and incinerate in the muffle furnace for 30 minutes. Cool and weigh to the nearest 0.0002 gm. Carry out two determinations on the same test sample

5.3.4 Calculation

Ash insoluble in Hydrochloric acid

$$\text{Percent by mass} = \frac{m_2 - m_1}{m_0 - m_1} \times 100$$

Where

m_0 = mass in gm of dish and test portion

m_1 = mass in gm of empty dish

m_2 = mass in gm of dish and acid insoluble ash

Carry out at least two determinations on the same test sample and take the arithmetic mean of the two results

(Ref :- I.S 13846 : 1993 / I.S.O 763 ; 1982 Fruit and Vegetable Products -
Determination of ash insoluble in dilute Hydrochloric Acid)

6.0 Fruit Bar / Toffee

6.1 Determination of Moisture – Follow method given in clause 4.1

6.2 Determination of Total soluble solids - Follow method given in clause 1.6

6.3 Determination of Fruit Content –

6.3.1 Principle

By the addition of Formaldehyde one H ion is liberated per molecule of amino acid. It is titrated with alkali. The secondary amino group of histidine does not react, those of proline and hydroxy proline react to about 75 %. Tertiary nitrogen and guanidine – groups undergo no reaction.

6.3.2 Apparatus

pH meter

6.3.3 Reagents:

- a) Sodium Hydroxide 0.25 N
- b) Formaldehyde solution – Pure formalin of at least 35 % is brought exactly to pH 8.1 with dilute sodium hydroxide as determined by means of a pH meter.
- c) Hydrogen Peroxide – pure 30 %

6.3.4 Procedure:

25 ml of fruit juice (for lemon juice 10 ml + 10 ml distilled water or the corresponding amount of concentrate diluted to this volume are neutralised in a beaker with 0.25 N NaOH to pH 8.1 on the pH meter. 10 ml of formaldehyde solution is then

added. After about 1 minute the solution is titrated potentiometrically to pH 8.1 with 0.25 N Sodium Hydroxide.

If more than 20 ml of 0.25 N Sodium Hydroxide is required the titration is to be repeated using 15 ml of formaldehyde solution instead of 10 ml. When sulphur dioxide is present the sample is treated with a few drops of 30 % hydrogen peroxide before neutralization.

6.3.5 Calculation

The amount of alkali used in titration expressed as ml 0.1 N alkali and referred to 100 ml fruit juice or 100 gm concentrate is equal to the formol number of the sample under test. Calculate to whole numbers (without decimals)

Interpretation –

As fruits ripen, the formol number of the juice tends to decrease, as a rule conversely on storage of the juice a slight increase may be noticed. Various factors can lead to a lowering of the formol number of a fruit juice e. g treatment with ion exchangers or addition of ascorbic acid

In the literature the formol number may also be found defined as ml N alkali for each 100 ml sample which corresponds to values 10 times smaller than those given by the preceding method of calculation

For orange juice the % fruit is $\frac{1.05 F}{1.4}$

1.4

Where, F is the formol number

(Re:- F.A.O Manuals of Food Quality Control 14 / 8 page 189 / Pearson's Composition and Analysis of Foods 9th edn, 1991, Page 271-72)

7.0 Fruit / Vegetable Cereal flakes

7.1 Determination of Moisture – Follow method given in clause 4.1

7.2 Determination of acid insoluble ash – Follow method given in clause 5.3

7.3 Determination of starch content - Follow method given in Methods of analysis for Cereal and Cereal products

8.0 Squashes, Crushes, Fruit syrups / sherbets, Synthetic syrups, Ginger cocktail

8.1 Determination of total soluble solids – Follow method given in clause 1.6

8.2 Determination of titrable acidity – Follow method given in clause 2.4

8.3 Minimum Fruit Juice / Puree Content - Method not available

9.0 Murabba

9.1 Determination of Total soluble solids – Follow method given in clause 1.6

9.2 Determination of Fruit Content –

9.2.1 Principle

By the addition of Formaldehyde one H ion is liberated per molecule of amino acid. It is titrated with alkali. The secondary amino group of histidine does not react, those of proline and hydroxy proline react to about 75 %. Tertiary nitrogen and guanidine – groups undergo no reaction.

9.2.2 Apparatus

a) pH meter

9.2.3 Reagents:

(a) Sodium Hydroxide 0. 25 N

(b) Formaldehyde solution – Pure formalin of at least 35 % is brought exactly to pH 8.1 with dilute sodium hydroxide as determined by means of a pH meter.

(c) Hydrogen Peroxide – pure 30 %

9.2.4 Procedure:

25 ml of fruit juice (for lemon juice 10 ml + 10 ml distilled water or the corresponding amount of concentrate diluted to this volume are neutralised in a beaker with 0.25 N NaOH to pH 8.1 on the pH meter. 10 ml of formaldehyde solution is then added. After about 1 minute the solution is titrated potentiometrically to pH 8.1 with 0.25 N Sodium Hydroxide.

If more than 20 ml of 0.25 N Sodium Hydroxide is required the titration is to be repeated using 15 ml of formaldehyde solution instead of 10 ml. When sulphur dioxide is present the sample is treated with a few drops of 30 % hydrogen peroxide before neutralization.

9.2.5 Calculation

The amount of alkali used in titration expressed as ml 0.1 N alkali and referred to 100 ml fruit juice or 100 gm concentrate is equal to the formol number of the sample under test. Calculate to whole numbers (without decimals)

Interpretation –

As fruits ripen, the formol number of the juice tends to decrease, as a rule. Conversely on storage of the juice a slight increase may be noticed. Various factors can lead to a lowering of the formol number of a fruit juice e. g treatment with ion exchangers or addition of ascorbic acid

In the literature the formol number may also be found defined as ml N alkali for each 100 ml sample which corresponds to values 10 times smaller than those given by the preceding method of calculation

For orange juice the % fruit is $\frac{1.05 F}{1.4}$

1.4

Where, F is the formol number

(Ref :- F.A.O Manuals of Food Quality Control 14 / 8 page 189 / Pearson's Composition and Analysis of Foods 9th edn, 1991, Page 271-72)

10.0 Candied, Crystallised and Glazed Fruit / Vegetable/ Rhizome / Fruit Peel

10.1 Determination of Total Sugars –

Grind about 50 gm sample in a blender or pestle and mortar, transfer to a 500 ml beaker and add 400 ml water. Neutralise with 1N NaOH using phenolphthalein indicator. Boil gently with occasional stirring. Cool and transfer to 500 ml volumetric flask. Make upto volume and filter. Pipette 100ml aliquot into another 500 ml volumetric flask Add 2 ml of neutral lead acetate. Let it stand for 10 minutes, then precipitate the excess of lead with Pot. Oxalate solution. Make upto mark and filter. Determine reducing sugar by Lane and Eynon volumetric method. Determine total sugars by inverting 25 ml of the clarified solution in a 250 ml volumetric flask with HCl in a water bath at 70 °C, cool, neutralise with NaOH and make upto volume. Determine total sugar by Lane and Eynon Volumetric method.

(Ref: - Manual of Analysis of Fruit and Vegetable Products S. Rangana, 1977 Page 10-11)

11.0 Mango Chutney

11.1 Determination of Total soluble solids – Follow method given in clause 1.6

11.2 Determination of pH – Determine with a pH meter after standardizing the pH meter with a buffer of pH 4 or refer 'Method No.2.3-Determination of pH'

11.3 Determination of Total Ash –

Weigh 5 – 10 gm sample in a silica or platinum dish (7-8 cm dia), dry on a water bath, ignite on a burner and ash in a muffle furnace at 525- 550°C for 4-6 hrs till a white ash is obtained. Cool the dish and weigh. Keep in muffle furnace again for 1 hr, cool and weigh. Note the lowest weight and calculate total ash.

Calculation:

$$\% \text{ Total ash} = (W3-W1)*100 / (W2-W1)$$

Where,

W1 = Weight of empty dish,

W2= Weight of dish containing sample,

W3 =Weight of dish containing ash

(Ref: - Manual of Analysis of Fruit and Vegetable Products S. Rangana, 1977 page8)

11.4 Determination of Ash Insoluble in HCl – Follow method given in clause 5.3

11.5 Determination of Fruit Content – Refer Method No.2.11

12.0 Tomato Ketchup / Tomato Sauce / Culinary Pastes

12.1 Determination of Total soluble solids (salt free basis) – Follow methods given in clause 1.7 and 1.6

12.2 Determination of Titrable acidity – Follow method given in clause 2.4

13.0 Soyabean Sauce

13.1 Determination of total solids (salt free basis) – Follow methods given in clause 1.6 and 1.7

13.2 Determination of acidity – Follow method given in clause 2.4

14.0 Brewed and Synthetic Vinegar

14.1 Definitions

14.1.1 Vinegar – is the liquid produced from a suitable raw material containing starch or sugar or starch and sugar by the process of double fermentation, alcoholic and acetous and which contains at least 4 % W / V acetic acid.

14.1.2 Malt Vinegar – is the vinegar produced without intermediate distillation by the process of double fermentation, alcoholic and acetous, from malted barley, with or without the addition of cereal grain, the starch of which has been converted to sugars solely by the diastase of the malted barley.

14.1.3 Grain Vinegar - is the vinegar produced without intermediate distillation by the process of double fermentation, alcoholic and acetous from any cereal grain the starch of which has been converted to sugars by a process other than solely by the diastase of malted barley.

14.1.4 Spirit Vinegar – is the vinegar made by the acetous fermentation of the alcoholic distillate from the product of alcoholic fermentation of suitable raw material solutions containing sugars. Thus the term ‘spirit vinegar’ must not be applied to any product obtained by the acetous fermentation of alcohol.

(Ref: - Pearsons Composition and Analysis of Foods 9th edn , page 460)

14.2 Preparation of sample – Mix thoroughly and filter through rapid filter paper

(Ref A.O.A.C 17th edn, Official method 935.35 (b) Vinegars)

14.3 Determination of Total Solids

Measure 10 ml product into weighed 50 mm diameter flat bottomed Pt dish, evaporate on boiling water bath 30 minutes, and dry exactly 2.5 hours in an oven at a temperature of boiling water (100 °C). Cool in a dessicator and weigh. The volatile acids which tend to remain partially in the total solids can be removed by three evaporations with water

(Ref A.O.A.C 17th edn, 2000 Official method 935.35 (c) Vinegars / Pearson's Composition and Analysis of Foods 9th edn, 1991 page 461)

14.4 Determination of Total Ash

Measure 25 ml product into weighed Pt dish, evaporate to dryness on water bath, and heat in furnace 30 minutes at 500 – 550°C, Break up the charred mass in Pt dish, add hot water, filter through ashless paper and wash thoroughly with water. Return paper and contents to dish, dry and heat 30 minutes at about 525° C or until all carbon is burnt off. Add filtrate, evaporate to dryness and heat 15 minutes at 525°C. Cool in a dessicator and weigh (weight x). Reheat 5 minutes at 525°C and cool for 1 hr in dessicator. Put 1 or 2 dishes in dessicator at a time. Place weight x on the balance pan before removing dish from dessicator and weigh rapidly to nearest mg. Calculate total ash from last weight.

(Ref: - A.O.A.C 17th edn, Official method 930. 35 (d) Vinegars)

14.5 Determination of Acidity

Dilute 10 ml sample with recently boiled and cooled water until it appears only slightly coloured and titrate with 0.5 N alkali using phenolphthlein

1 ml 0.5 N alkali =0.0300 gm acetic acid

(Ref: - A.O.A.C 17th edn, 2000 Official Method 930. 35 (J) Vinegars)

14.6 Test for presence of Mineral Acid

Mix 2 ml sample with 2 ml alcohol and add 2 drops of methyl orange. A red colour indicates low pH due to added mineral acid. Other indicators that may be employed are methyl violet and metanil yellow. The pH of the products containing 4 % acetic acid seldom falls below 2.9 for malt vinegar and 2.5 for artificial product.

(Ref: - Pearson's Composition and Analysis of Foods 9th edn, 1991 page 462)

14.7 Test for presence of Caramel

14.7.1 Caramel is detected by Fiehe's reaction.

14.7.2 Reagents:

- (i) 1% Resorcinol in conc. HCl.
- (ii) Diethyl ether.

14.7.3 Procedure:

Extract 100 ml of vinegar with 50 ml of diethyl ether in a separating funnel; transfer the ether layer to a porcelain dish and evaporate at room temperature. To the residue add 3 drops of resorcinol. Appearance of rose- pink colour shows the presence of caramel

(Ref: - Pearson's Composition and Analysis of Foods 9th edn, 1991, page 462)

14.8 Determination of Phosphorous (in Malt Vinegar)

14.8.1 Reagents

- (1) Vanadate - molybdate composite reagent - Dissolve 20 gm ammonium molybdate in 400 warm water (50°C) and cool.

Dissolve 1.0 gm ammonium vanadate in 300 ml boiling distilled water, cool and add 140 ml conc Nitric acid gradually with stirring. Then add the molybdate solution gradually to the acid vanadate solution with stirring and dilute to 1 litre with water.

- (2) Standard Phosphate solution - Prepare a stock solution containing 3.834 gm Potassium dihydrogen phosphate (KH_2PO_4) per litre.

Dilute 25 ml to 250 ml (1 ml = 0.2 mg P_2O_5).

14.8.2 Preparation of Standard Curve

To a series of 100 ml volumetric flasks add 0,2.5, 5, 10, 20, 30, 40, and 50 ml of the standard phosphate solution (= 0- 10 mg P_2O_5) and dilute each to 50- 60 ml with water. Add a few drops of ammonia solution (0.88) and make just acid with nitric acid (1:2). Add 25 ml of vanadate – molybdate reagent, dilute to the mark and mix. Allow to stand for 10 minutes and measure the optical density in a 2.5 or 10 mm cell at 470 nm.

14.8.3 Procedure

Boil the ash with 10 ml of 5 M HCl and wash the solution into a 100 ml flask with water, filtering if necessary. Neutralise by drop wise addition of 0.88 ammonia (the volume of the solution at this stage should be 50 – 60 ml).

Make just acid with dilute nitric acid , add 25 ml of vanadate – molybdate solution, make upto mark, and measure optical density after allowing to stand for 10 minutes.

(Ref :- Pearson's Composition and Analysis of Foods 9th edn,1991, page 37)

14.9 Determination of Nitrogen

14.9.1 Apparatus:

- (a) Kjeldahl digestion flask - 500 or 800 ml.
- (b) Kjeldahl distillation apparatus, - same digestion flask fitted with rubber stopper through which passes lower end of efficient rubber bulb or trap to prevent mechanical carry over of NaOH during distillation.
- (c) Conical flask, 250 ml.
- (d) Burette 50 ml.

14.9.2 Reagents

- (a) Concentrated Sulphuric acid - sp gr 1.84
- (b) Sodium Hydroxide solution - 45%. Dissolve 450 gm of Sodium Hydroxide in 1000 ml water.
- (c) Standard Sulphuric acid solution – 0.1 N
- (d) Standard Sodium Hydroxide solution – 0.1 N
- (e) Methyl Red Indicator solution - Dissolve 0.5 gm methyl red in 100 ml of alcohol

14.9.3 Procedure

Take 25 – 50 ml of the sample and transfer to a 500 or 800 ml Kjeldahl flask taking care to see that no portion of the sample clings to the neck of the flask. Add 0.7 gm of Mercuric oxide, 15 gm of Potassium Sulphate and 40 ml of concentrated sulphuric acid. Add two to three glass beads. Place the flask in an inclined position on the stand in the digestion chamber and digest. Heat the flask gently at low flame until the initial frothing ceases and the mixture boils steadily at a moderate rate. During heating rotate the flask several times. Continue heating for about an hour or more until the colour of the digest is pale blue. Cool the digest and add slowly 200 ml of water. Cool, add a piece of granulated Zinc or anti bump granules and carefully pour down the side of the flask sufficient Sod. Hydroxide solution (450gm / litre) to make the contents strongly alkaline (about 110 ml) before mixing the acid and alkaline layer. Connect the flask to a distillation apparatus incorporating an efficient flash head and condenser. To the condenser fit a delivery tube which dips just below the surface of the pipetted volume of standard acid contained in a conical flask receiver. Mix the contents of the digestion flask and boil until 150 ml have distilled into the receiver. Add 5 drops of methyl red indicator and titrate with 0.1 N Sod. Hydroxide solution. Carry out a blank titration.

1 ml of 0.1 N H_2SO_4 = 0.0014gm N.

(Ref :- Pearsons Composition and Analysis of Foods 9th edn, page 17)

14.10 Formol Titration for differentiation between brewed and synthetic vinegar

In general, brewed vinegars give definite formol titration but artificial products do not. This method is a rapid sorting test but is not specific for differentiating between brewed and non brewed vinegars as distilled vinegars do not give a formol titration

14.10.1 Procedure

Add phenolphthalein to 10 ml sample, nearly neutralize the acid with 0.5M sodium hydroxide and then make exactly neutral with 0.05 M sod Hydroxide. Add 5 ml formalin (previously made exactly neutral to phenolphthalein, mix well and after standing for 5 min, titrate the acidity produced with 0.05 M or 0.1 M sod hydroxide

Most brewed vinegars give a formol titration equivalent to 0.5 – 3.0 ml of 0.1 M NaOH on a 10 ml sample. Artificial products and distilled vinegars give a negligible formol titration.

(Ref: - Pearson's Composition and Analysis of Foods 9th edn,1991 page 463)

14.11 Examination of Distillate

Distil 60 ml sample from a 350 ml flask fitted with a small tap funnel. When 45 ml distillate has come over add 15 ml water to the flask down the tap funnel and distil a further 15 ml to give a total volume of distillate of 60 ml.

14.11.1 Oxidation value is the no. of ml of 0.002M KMnO₄ used by 100 ml sample in 30 minutes under standard conditions.

To a 250 ml glass stoppered bottle add 5 ml of distillates from a malt or wine vinegar (or 10 ml from a dilute spirit vinegar or artificial product) 10 ml dilute H₂SO₄ (1:3) and exactly 15 ml of 0.02M KMnO₄. Allow to stand at 18°C for 30 minutes; add 5

ml of 10% KI solution. Titrate the liberated iodine with 0.02M $\text{Na}_2\text{S}_2\text{O}_3$ using starch indicator (a ml). Carry out the blank (b ml).

If 10 ml is used oxidation value = 20 (b – a)

If 5 ml is used oxidation value = 40 (b – a)

Alcohol and acetyl methyl carbinol are the principle contributors to the oxidation value.

(Ref: - Pearson's Composition and Analysis of Foods 9th edn, page 464).

See also A.O.A.C 17th edn, Official method 944.10 Permanganate oxidation number).

14.11.2 Alkaline Oxidation Value – is the number of parts by weight of oxygen required to oxidise 100 000 parts of sample under standard conditions

Procedure:

To a 250 ml glass stoppered bottle add 2 ml of distillate, 100 ml water 10ml of 10 % sodium hydroxide solution and exactly 10 ml of 0.02M Potassium Permanganate. Allow to stand for 30 minutes and then acidify with 10 ml of dilute sulphuric acid (1 + 3). Add 0.5 gm Potassium Iodide and titrate the liberated iodine with 0.02 M Sodium thiosulphate using starch near the end point(a ml). Carry out a blank at the same time (titration b).

$$\text{Alkaline Oxidation Value} = 8 (b - a)$$

(Ref: - Pearson's Composition and Analysis of Foods 9th edn, 1991, page 464)

15.0 Carbonated Fruit Beverage / Fruit Drink

15.1 Determination of Total Soluble solids – Follow method given in clause 1.6

15.2 Determination of pH – Follow method given in clause 2.3

15.3 Determination of Fruit content – Refer Method No.2.11

16.0 JAM, JELLY AND MARMALADE

16.1 Total Soluble Solids: - Follow method given in clause 1.6

16.2 Acidity: - Follow method given in clause 2.4

Take 10 gms. of sample; mix thoroughly with about 50 ml water and titrate with 0.1N NaOH using phenolphthalein indicator. Report acidity as citric acid and as malic acid if apple predominates. With highly coloured jams such as blackberry and black current, titrate potentiometrically to pH 8.1.

16.3 Fruit Content: Refer Method No.2.11

16.4 Preservatives: Refer to the Manual on Food Additives

16.5 Added Colouring Matter: Refer to the manual on food additives

17.0 DEHYDRATED FRUITS / DEHYDRATED VEGETABLES:**17.1 Preparation of sample:**

Observe the sample closely for mould, insect, larvae, extraneous matter etc. Take about 25 to 50 gms of sample, grind quickly to pass through a 30 mesh sieve.

17.2 Determination of Moisture: - Follow method given in clause 4.1

17.3 Determination of Total Ash – Follow method given in clause 14.4. Weigh accurately about 5 gm sample for determination of ash.

17.4 Determination of Acid insoluble ash – Follow method given in clause 5.3. Use the ash obtained in 17.3 for determining acid insoluble ash.

17.5 Test for presence of Peroxidase**17.51 Reagents**

- (a) Guaiacol solution – 1 % prepared by dissolving 1 gm of 0.9 ml guaiacol in 50 ml ethyl alcohol and adding 50 ml water
- (b) Hydrogen peroxide – 1 %. Dilute 1 part of 3% Hydrogen Peroxide with 2 parts of water.

17. 5.2 Procedure

Take 25 gm of the material and coarsely powder it. Place 5 gm on a white porcelain saucer or evaporating dish. Add enough guaiacol solution to wet all the cut surfaces, then immediately add a similar amount of Hydrogen peroxide solution. At the end of three minutes note whether a reddish brown colour has developed. If none is observed the test is negative. Neglect any colour that may be developed after 3 minutes.

(Ref: - I.S.I Handbook of Food Analysis (Part VIII) – 1984 page 13)

17.6 Determination of Rehydration ratio:

17.61 Procedure

Cook (simmer) in a beaker one part of dehydrated vegetable in 10 parts of one percent sodium chloride solution for 20 minutes and then allow it to cool at room temperature for 45 minutes. The time taken for cooking shall be the time taken from the start of boiling (simmering). Drain off excess solution by covering the beaker with watch glass with convex surface and inverting the container for 5 minutes. Weigh cooled material.

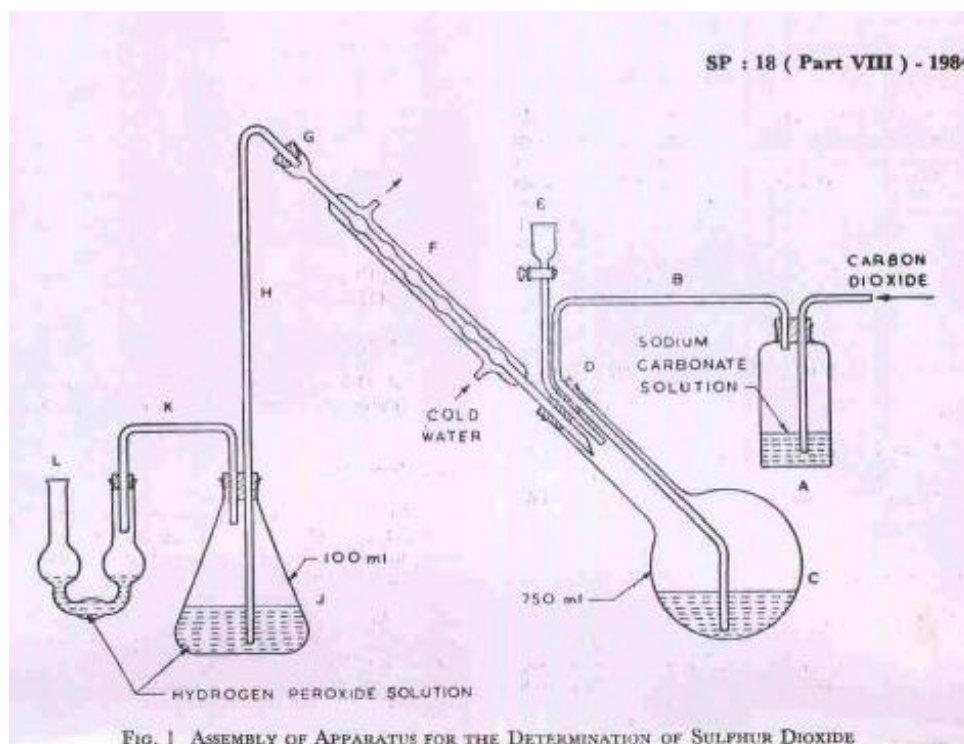
17.62 Calculation

$$\text{Rehydration Ratio} = \frac{\text{Weight of Reconstituted sample}}{\text{Weight of dehydrated sample}}$$

(Ref: - I.S.I Handbook of Food Analysis (Part VIII) – 1984 page 13)

17.7 Determination of Sulphur Dioxide

17.71 Apparatus – The apparatus assembled as shown below may be used



17.7. 2 Reagents

- (1) Sodium Carbonate solution – 10 % (w / v)
- (2) Bromophenol indicator solution – Dissolve 0.1 gm of bromophenol blue in 3 ml of 0.05N sodium hydroxide solution and 5 ml of ethyl alcohol (90%) by warming gently. Make upto 250 ml in a volumetric flask with 20 % ethyl alcohol.
- (3) Hydrogen Peroxide solution – Dilute a 30 % Hydrogen peroxide solution with twice its volume of water and neutralize the free sulphuric acid that may be present in the H_2O_2 with barium hydroxide solution using bromophenol blue indicator. Allow the ppt of barium sulphate to settle filter and determine the concentration of H_2O_2 in the filtrate by titrating with standard potassium permanganate. Dilute the filtrate with cold water so as to obtain a 3 % solution of hydrogen peroxide.

- (4) Concentrated Hydrochloric acid – sp gr 1.16
- (5) Carbon dioxide gas from a cylinder
- (6) Standard sodium hydroxide solution – 0.1 N standardised at the time of the experiment with bromophenol blue indicator.

17.7.3 Procedure

Weigh 25 ml of Hydrogen peroxide solution in Erlenmeyer flask(J) and 5 ml in Peligot tube (L), Assemble the apparatus as shown above. Introduce into the flask (C) 300 ml water and 20 ml of concentrated Hydrochloric acid through the dropping funnel (E). Run a steady current of cold water through the condenser (F). To expel air from the system boil the mixture contained in the flask (C) for a short time in a current of Carbon dioxide gas previously passed through the wash bottle (A).

Weigh accurately about 25 gm of sample and dissolve in the minimum quantity of water. Introduce this solution into the flask (C) through the dropping funnel (E). Wash the dropping funnel with a small quantity of water and run the washings into the flask (C). Distill the mixture contained in the flask (C) in a slow current of Carbon Dioxide gas passed previously through the wash bottle (A) for 1 hour. Just before the end of the distillation stop the flow of water in the condenser (This causes the condenser to become hot and drives off the residual traces of sulphur dioxide retained in the condenser). When the delivery tube (H) just above the Erlenmeyer flask (J) becomes hot to touch disconnect the stopper (G) immediately. Wash the delivery tube (H) and the contents of the Peligot tube (L) with water into the Erlenmeyer flask (J). Cool the contents of the Erlenmeyer flask to room temperature, add a few drops of bromophenol blue indicator and titrate with standard sodium hydroxide solution (Bromophenol blue is unaffected by carbon dioxide and gives a distinct colour change in cold hydrogen peroxide solution).

Carry out a blank determination using 20 ml of concentrated hydrochloric acid diluted with 300 ml of water

17.74 Calculation

$$\text{Sulphur Dioxide mg / kg} = \frac{32\,000 (V - v) N}{M}$$

Where,

V = volume in ml of standard sodium hydroxide solution required for the test with sample

v = volume of standard sodium hydroxide solution required for the blank determination

N = Normality of standard sodium hydroxide solution

M = mass in gm of the sample taken for test

(Ref: - I.S.I. Hand book of Food Analysis (Part VIII) – 1984 page 12)

18.0 PICKLES

18.1 Determination of Drained weight – Follow method given in clause 1.4

18.2 Determination of Sodium Chloride in brine - Follow method given in Clause 1.7

18.3 Determination of titrable acidity - Follow method given in clause 2.4

18.4 Acidity of Vinegar: - Follow method given in clause 14.5

19.0 TABLE OLIVES

19.1 Determination of Sodium chloride - Follow method given in clause 1.7

19.2 Determination of pH of brine - Follow method given in clause 2.3

19.3 Determination of acidity of Brine - Follow method given in clause 2.4

20.0 DRIED FRUITS AND NUTS

20.1 Determination of moisture –

Weigh accurately about 5 gm sample into a dried tared metal dish (about 7.5– 8 mm diameter provided with closely fitting lid) containing about 2 gm of finely divided glass fibre filter. Moisten with hot water, mix thoroughly, evaporate to almost dryness on steam bath and dry for 6 hours in a vacuum oven at $70 \pm 1^{\circ}\text{C}$ under pressure equal to 100 mm Hg. Admit a slow current of dried air (dried by passing through H_2SO_4). Replace cover, cool dish in dessicator and weigh.

(Ref: - A.O.A.C 17th edn, 2000 Official Method 934.06 Moisture in dried fruits.)

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*The methods mentioned in the manual needs to be verified/ validated before they are put in use by the laboratory.



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