SRI LANKA STANDARD 586:1982
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METHODS OF TEST FOR SUGAR CONFECTIONERY

BUREAU OF CEYLON STANDARDS

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SRI LANKA STANDARD
METHODS OF TEST FOR SUGAR CONFECTIONERY

FOREWORD

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Bureau of Ceylon Standards on 1982-11-24, after the draft, finalized by the Drafting Committee on Sugar Confectionery had been approved by the Agricultural and Food Products Divisional Committee.

All standard values given in this specification are in SI units.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with CS 102. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

In the preparation of this standard, the assistance obtained from the publications of the Indian Standards Institution is gratefully acknowledged.

1 SCOPE

This standard prescribes the methods of test for sugar confectionery.

2 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water shall be employed in tests.

NOTE - "Pure chemicals" shall mean chemicals that do not contain impurities which affect test results.

3 DETERMINATION OF MOISTURE CONTENT

3.1 Procedure

3.1.1 *Preparation of sample*

Mince as quickly as possible with a sharp-edged knife or grind in a dry pestle and mortar 150 g 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.

3.1.2 Weigh to the nearest milligram about 5 g of the prepared sample (See **3.1**) in a tared weighing bottle having a diameter of about 40 mm and a height of about 25 mm. Distribute the material as evenly as practicable over the bottom of the bottle by gentle sidewise movements. Place the bottle in a vacuum oven, remove the cover of the bottle and dry the material for six hours at 80 ± 1 °C at a pressure not exceeding 5 mm Hg. Allow the bottle to cool to room temperature and weigh.

3.2 Calculation

3.2.1 Moisture, per cent by mass $= \frac{100 (m - m_1)}{m}$

where

m = mass in g of the prepared sample taken for the experiment; and

m_1 = mass in g of the material after drying for six hours.

4 DETERMINATION OF SULPHATED ASH

4.1 Reagent

4.1.1 *Concentrated sulphuric acid,* sp. gr. 1.84 .

4.2 Procedure

Weigh to the nearest milligram about 5 g of the prepared sample (See **3.1.1**) into a 9-cm diameter platinum or 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 drops of concentrated sulphuric acid, heat strongly on a hot plate until sulphuric acid fumes cease to be evolved and finally ash in the muffle furnace at 600 ± 20 °C for 2 hours. Cool in a desiccator and weigh. Heat again in the muffle furnace for 30 minutes at 600 ± 20 °C. 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 10 mg. Record the lowest weight.

4.3 Calculation

4.3.1 Ash, sulphated, per cent by weight $= \frac{100\, m_1}{m_2}$

where

m_1 = mass in g, of the ash; and

m_2 = mass in g, of the prepared sample taken for the test.

5 DETERMINATION OF ACID INSOLUBLE ASH

5.1 Reagent

5.1.1 *Dilute hydrochloric acid,* approximately 5 N (prepared from concentrated hydrochloric acid).

5.2 Procedure

Weigh to the nearest milligram about 20 g of the prepared sample (See 3.1.1) in a tared, clean and dry porcelain dish. Ignite the material in the dish with the flame of a Mekar burner for about one hour. Complete the ignition by keeping in a muffle furnace at 600 ± 20 °C until grey ash results. Cool in a desiccator. To the ash, add 25 ml of the dilute hydrochloric acid, cover with a watch-glass and heat on a water-bath for 10 minutes. Allow to cool and filter the contents of the dish through Whatman filter No. 42 or its equivalent. Wash the filter with water until the washings are free from chlorides. Return the filter and the residue to the dish. Keep it in an air-oven maintained at 105 ± 2 °C for about 3 hours. Ignite in the muffle furnace at 600 ± 20 °C for one hour. Cool the dish in a desiccator and weigh. Heat again for 30 minutes in the muffle furnace, cool and weigh. Repeat this process of heating for 30 minutes, cooling and weighing till the difference between two successive weighings is less than one milligram. Note the lowest weight.

5.3 Calculation

5.3.1 Acid insoluble ash, per cent by mass = $\frac{100 (m_2 - m)}{m_1 - m}$

where

m_2 = mass in g, of the porcelain dish with the acid insoluble ash;

m = mass in g, of the empty porcelain dish; and

m_1 = mass in g, of the porcelain dish with the prepared sample taken for the test.

6 DETERMINATION OF REDUCING SUGARS

6.1 Reagents

6.1.1 *Stock solution of dextrose*

Weigh accurately 10 g of anhydrous dextrose into a 1-litre graduated flask and dissolve it in water. Add to this solution, 2.5 g of benzoic acid, shake to dissolve the benzoic acid and make up the volume to the mark with water (This solution shall not be used after 48 hours).

6.1.2 *Standard dextrose solution*

Dilute a known aliquot of the stock solution of dextrose (See **6.1.1**) with water containing 0.25 per cent (m/v) of benzoic acid to such a concentration that more than 15 ml but less than 50 ml of it will be required to reduce all the copper in the Fehling's solution taken for titration. Note the concentration of anhydrous dextrose in this solution as milligrams per 100 ml (See Note), prepare this solution afresh every day.

NOTE - When 10 ml (See **6.3.1.1***) of Fehling's solution are taken for titration a standard dextrose solution containing 0.11 per cent to 0.30 per cent (m/v) of anhydrous dextrose is convenient for use.*

6.1.3 *Methylene blue indicator solution*

Dissolve 0.2 g of methylene blue in water and dilute to 100 ml.

6.1.4 *Petroleum ether,* re-distilled below 60 °C.

6.1.5 *Fehling's solution (Soxhlet modification)*

Prepared by mixing immediately before use, equal volumes of solution A and solution B.

6.1.5.1 Solution A

Dissolve 34.639 g of copper sulphate ($CuSO_4.5H_2O$) in water, add 0.5 ml of concentrated sulphuric acid of sp.gr. 1.84 and dilute to 500 ml in a graduated flask. Filter the solution through prepared asbestos.

6.1.5.2 Solution B

Dissolve 173 g of Rochelle salt (potassium sodium tartrate ($KNaC_4H_4O_6.4H_2O$) and 50 g of sodium hydroxide, analytical reagent in water, dilute to 500 ml in a graduated flask and allow the solution to stand for two days. Filter the solution through prepared asbestos.

Table 1 - Dextrose factor for 10 ml of Fehling's solution

(See 6.1.5.3)

Titre ml (1)	Dextrose factor* (2)	Dextrose content in mg per 100 ml of solution (3)
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	143.9
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

**Milligrams of anhydrous dextrose corresponding to 10 ml of Fehling's solution.*

6.1.5.3 Standardization of Fehling's solution

Pour standard dextrose solution (See 6.1.2) into a 50-ml burette (See Note 3 under 6.2.3). Find the titre (i.e. the volume of standard dextrose solution required to reduce all the copper in 10 ml of Fehling's solution) corresponding to the concentration of standard dextrose solution from Table 1. (If, for example: the standard dextrose solution contains 167.0 mg of anhydrous dextrose per 100 ml, the corresponding titre would be 30 ml). Pipette 10 ml (See 6.3.1.1) of Fehling's solution into a 300-ml 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 not more than one millilitre will be required later to complete the titration. Heat the flask containing the mixture over a wire gauze. Gently boil the contents of the flask for 2 minutes. At the end of 2 minutes of boiling, add without interrupting boiling, one millilitre of methylene blue indicator solution. While the contents of the flask continue to boil, begin to add standard dextrose solution (one or two drops at a time) from the burette till the blue colour of the indicator just disappears. (The titration should be completed within one minute, so that the contents of the flask boil altogether for 3 minutes without interruption (See Note 2 under 6.2.3). Note the titre (i.e. the total volume in millilitres of standard dextrose solution used for the reduction of all the copper in 10 ml of Fehling's solution). Multiply the titre (obtained by direct titration) by the number of milligrams of anhydrous dextrose in one millilitre of the standard dextrose solution to obtain the dextrose factor. Compare this factor with the dextrose factor given in Table 1 and determine correction, if any, to be applied to the dextrose factor derived from Table 1.

EXAMPLE :

Concentration in mg/100 ml of anhydrous dextrose in standard dextrose solution	=	167.0
Titre in ml obtained by direct titration	=	30.1
Dextrose factor for 30.1 ml of standard dextrose solution	=	Titre in ml x number of mg of anhydrous dextrose in one millilitre of standard dextrose solution
	=	30.1 x 1.670
	=	50.2670
Dextrose factor for 30.1 ml of standard dextrose solution from Table 2 (calculated by interpolation)	=	50.11
Correction to be applied to the dextrose factor derived from Table 2	=	50.2670 - 50.11
	=	+0.1570

6.2 Procedure

6.2.1 *Preparation of solution*

Weigh to the nearest milligram about 3 g to 4 g of the prepared sample (See 3.1.1) in Soxhlet extraction thimble and extract the fat in a Soxhlet apparatus using petroleum ether. Take out carefully the thimble along with the fat-free material from the Soxhlet apparatus and dry the same to be free from the petroleum ether. Dissolve carefully the entire fat-free sample in a small quantity of water in a beaker. If necessary, add water to the thimble and dissolve the adhering material. Collect the washings into the beaker. Warm to a temperature of 50 °C to 60 °C. Cool it. Filter through a Whatman filter paper No. 40 or its equivalent, collecting the filtrate in a 100-ml graduated flask. Wash the filter paper and the insoluble starch residue, if any, on the filter paper carefully. Collect the washings in the graduated flask. Make up to the mark with water.

6.2.2 *Incremental method of titration*

Pour the prepared solution (See 6.2.1) into a 50-ml burette (See Note 3 below 6.2.3). Pipette 10 ml of Fehling's solution into a 300-ml conical flask and run in from the burette 15 ml of the prepared solution. Without further dilution, heat the contents of the flask over a wire gauze, and boil. (After the liquid has been boiling for about 15 seconds it will be possible to judge if almost all the copper is reduced by the bright red colour imparted to the boiling liquid by the suspended cuprous oxide). When it is judged that nearly all the copper is reduced, add one millilitre of methylene blue indicator solution (See Note 1 below). Continue boiling the contents of the flask for one to two minutes from the commencement of ebullition, and then add the prepared solution in small quantities (one millilitre or less at a time), allowing the liquid to boil for about 10 seconds between successive additions, till the blue colour of the indicator just disappears (See Note 2 below 6.2.3). In case there still appears to be much unreduced copper after the mixture of Fehling's solution with 15 ml of the prepared solution has been boiling for 15 seconds, add the prepared solution from the burette, in larger increments (more than one millilitre at a time according to judgement), and allow the mixture to boil for 15 seconds after each addition. Repeat the addition of the prepared solution at intervals of 15 seconds until it is considered unsafe to add a large increment of the prepared solution. At this stage, continue the boiling for an additional one to two minutes, add one millilitre of methylene blue indicator solution and complete the titration by adding the prepared solution in small quantities (less than one millilitre at a time) (See also Note 2 below).

NOTES

1 It is advisable not to add the indicator until the end point has been nearly reached because the indicator retains its full colour until the end point is almost reached and thus gives no warning to the operator to go slowly.

2 When the operator has had a fair amount of experience with the method, a sufficiently accurate result may often be obtained by a single estimation by the incremental method of titration. For the utmost degree of accuracy of which the method is capable, a second titration should be carried out by the standard method of titration. (See 6.2.3).

6.2.3 *Standard method of titration*

Pipette 10 ml of Fehling's solution into a 300-ml conical flask and run in from the burette almost the whole of the prepared solution required to effect reduction of all the copper (determined under 6.2.2) so that if possible, not more than one millilitre will be required later to complete the titration. Gently boil the contents of the flask for 2 minutes. At the end of 2 minutes of boiling; add without interrupting boiling, one millilitre of methylene blue indicator solution. While the contents of the flask continue to boil, begin to add the prepared solution (one or two drops at a time) from the burette till the blue colour of the indicator just disappears (See Note 1). (The titration should be completed within one minute so that the contents of the flask boil altogether for 3 minutes without interruption (See Note 2).

In case of doubt, the flame may be removed from the wire gauze for one or two seconds and the flask held against a sheet of white paper. (A holder of paper, suitably fixed around the neck of the flask, is very convenient for this purpose as it can be left round the neck of the flask, without risk of overbalancing it). The top edge of the liquid would appear bluish if the indicator is not completely decolourized. It is inadvisable to interrupt the boiling for more than a few seconds as the indicator undergoes back oxidation rather rapidly when air is allowed free access into the flask, but there is no danger of this as long as a continuous stream of steam is issuing from the mouth of the flask.

NOTES

1 The indicator is so sensitive that is possible to determine the end point within one drop of the prepared solution in many cases. The complete decolouration of the methylene blue is usually indicated by the whole reaction liquid, in which the cuprous oxide is continuously churned up, becoming bright red or orange in colour.

2 It should be observed that with both incremental and standard methods of titration, the flask containing the reaction mixture is left on the wire gauze over the flame throughout the titration, except when it may be removed for a few seconds to ascertain if the end point is reached.

3 In adding sugar solution to the reaction mixture, the burette may be held in hand over the flask. The burette may be fitted with a small outlet tube bent twice at right angles, so that the body of the burette can be kept out of the steam while adding sugar solution. Burettes with glass taps are unsuitable for this work, as the taps become heated by the steam and are liable to jam.

6.3 Calculation

6.3.1 Refer to Table 2 for the dextrose factor corresponding to the titre (determined as given under **6.2.3**) and apply the correction previously determined under **6.1.5.3**. Calculate the dextrose content of the prepared solution (See **6.2.1**) as follows:

$$\text{Milligrams of anhydrous dextrose present in one millilitre of the prepared solution} = m = \frac{\text{Dextrose factor}}{\text{Titre}}$$

6.3.1.1 Instead of using 10 ml of Fehling's solution, a 25 ml portion may also be substituted throughout the procedure (including *standardization of Fehling's solution* under **6.1.5.3**). In this case, the standard dextrose solution, used in standardizing the Fehling's solution and the prepared solution of the material (See **6.2.1**) shall contain 0.25 per cent to 0.75 per cent (m/v) of anhydrous dextrose, and Table 2 shall be used for all calculations.

6.3.2 Reducing sugars, per cent by mass = $\frac{m}{M} \times 100$

where,

m = milligrams of anhydrous dextrose in 1 ml of the solution of the material (See **6.3.1**) ; and

M = mass in g of the prepared sample used for making 100 ml of solution (See **6.2.1**).

7 DETERMINATION OF SUCROSE

7.1 Reagents

7.1.1 *Concentrated hydrochloric acid,* sp. gr. 1.16, of analytical reagent grade.

7.1.2 *Fehling's solution,(Soxhlet modification)*, prepared by mixing immediately before use, equal volumes of solution A and solution B.

7.1.2.1 Solution A

Dissolve 34.639 g of copper sulphate ($CuSO_4.5H_2O$) in water, add 0.5 ml of concentrated sulphuric acid of sp. gr. 1.84, and dilute to 500 ml in a graduated flask. Filter the solution through prepared asbestos.

7.1.2.2 Solution B

Dissolve 173 g of Rochelle salt {potassium sodium tartrate ($KNaC_4H_4O_6.4H_2O$)} and 50 g of sodium hydroxide, analytical reagent in water, dilute to 500 ml in a graduated flask and allow the solution to stand for two days. Filter this solution through prepared asbestos.

TABLE 2 - **Dextrose factor for 25 ml of Fehling's solution**

(See 6.1.5.3, 6.3.1 and 6.3.1.1)

Titre ml (1)	Dextrose factor* (2)	Dextrose content in mg per 100 ml of solution (3)
15	120.2	810
16	120.2	751
17	120.2	707
18	120.2	668
19	120.3	638
20	120.3	601.5
21	120.3	572.9
22	120.3	547.3
23	120.4	523.6
24	120.5	501.9
25	120.5	482.0
26	120.6	463.7
27	120.6	446.8
28	120.7	431.1
29	120.7	416.4
30	120.8	402.7
31	120.8	389.7
32	120.8	377.6
33	120.9	366.3
34	120.9	355.6
35	121.0	345.6
36	121.0	336.3
37	121.1	327.4
38	121.2	318.8
39	121.2	310.7
40	121.2	303.1
41	121.3	295.9
42	121.4	289.0
43	121.4	282.4
44	121.5	276.1
45	121.5	270.1
46	121.6	264.3
47	121.6	258.8
48	121.7	253.5
49	121.7	248.4
50	121.8	243.6

**Milligrams of anhydrous dextrose corresponding to 25 ml of Fehling's solution.*

NOTE - Table 1 and 2 show, for the standard method of titration, the values corresponding to integral millilitres of the sugar solutions, intermediate values being obtained by interpolation.

7.2 Procedure

Take 10 ml of the prepared solution (See **6.2.1**) in a conical flask and add 1.5 ml of the concentrated hydrochloric acid and about 10 ml of water. Heat the flask at 60 °C to 70 °C for 10 minutes in a water-bath. Cool immediately and neutralize with 30 per cent sodium hydroxide (m/v) and transfer quantitatively the neutralized inverted solution to a graduated flask and make up the volume to 100 ml.

Determine the reducing sugars in the inverted solution as described in **6**.

7.3 Calculation

7.3.1 Sucrose, per cent by mass = (Q - R) 0.95

where,

Q = Total sugars (after inverting); and

R = Reducing sugars (before inverting) (See **6.3**)

8 DETERMINATION OF FAT

8.1 Apparatus

8.1.1 *Buchner funnel,* 9-cm size

8.1.2 *Soxhlet apparatus,* with 250-ml flat bottom soxhlet flask.

8.2 Reagents

8.2.1 *Hydrochloric acid,* sp. gr. 1.16.

8.2.2 *Filter-aid,* suitable type.

8.2.3 *Petroleum ether,* re-distilled below 60 °C.

8.2.4 *Sodium sulphate,* anhydrous.

8.3 Procedure

Weigh to the nearest milligram 20 g to 30 g of the prepared sample (See **3.1.1**) into a 400-ml beaker and add 30 ml of water and 25 ml of hydrochloric acid. Heat for 30 minutes on a steam-bath with frequent stirring. Add 5 g of filter-aid and 50 ml of ice-cold water and chill for 30 minutes in ice-cold water. Fit a piece of heavy linen into the buchner funnel and moisten with water. Apply gentle suction and pour over it a suspension of 3 g of filter-aid in 30 ml of water. Filter the hydrolyzed mixture by gentle suction, rinsing the beaker three times with ice-cold water, taking

care to leave a layer of liquid on the filter. Finally wash three times with ice-cold water and suck dry. Transfer the filter-cake from the funnel to the original beaker, using a small piece of filter paper to transfer any material adhering to the funnel. Wash the funnel with petroleum ether into the beaker and evaporate the other on the steam-bath. Break up the cake with a glass rod and allow it to remain on the steam-bath until the contents are so dry as to enable pulverizing easily. Place in an oven at 100 ± 2 °C for one hour. Add 15 g powdered anhydrous sodium sulphate and mix well.

8.3.1 Transfer the mixture to the fat extraction thimble of the Soxhlet apparatus. Wash the beaker with 50 ml of petroleum ether and transfer the washings to the thimble. Extract the fat with petroleum ether so that at least 300 ml have been circulated. Transfer the extract to a tared dish and evaporate the petroleum ether on the steam-bath. Dry the fat till the difference in weight between two successive weighings is not more than one milligram.

NOTE - In case of confectionery not containing milk, dry the material at 100 ± 2 °C for 4 hours and extract the fat in a Soxhlet apparatus as described in 8.3.1.

8.4 Calculation

8.4.1 Fat, per cent by mass (on dry basis) $= \frac{m_1}{m} \times 100 \left(\frac{100}{100 - M} \right)$

where,

m_1 = mass, in g, of extracted fat;

m = mass, in g, of the prepared sample taken for the test; and

M = percentage of moisture in the material as determined in 3.2.1.

9 DETERMINATION OF SULPHUR DIOXIDE

9.1 Apparatus

9.1.1 The apparatus, as assembled, is shown in Figure 1.

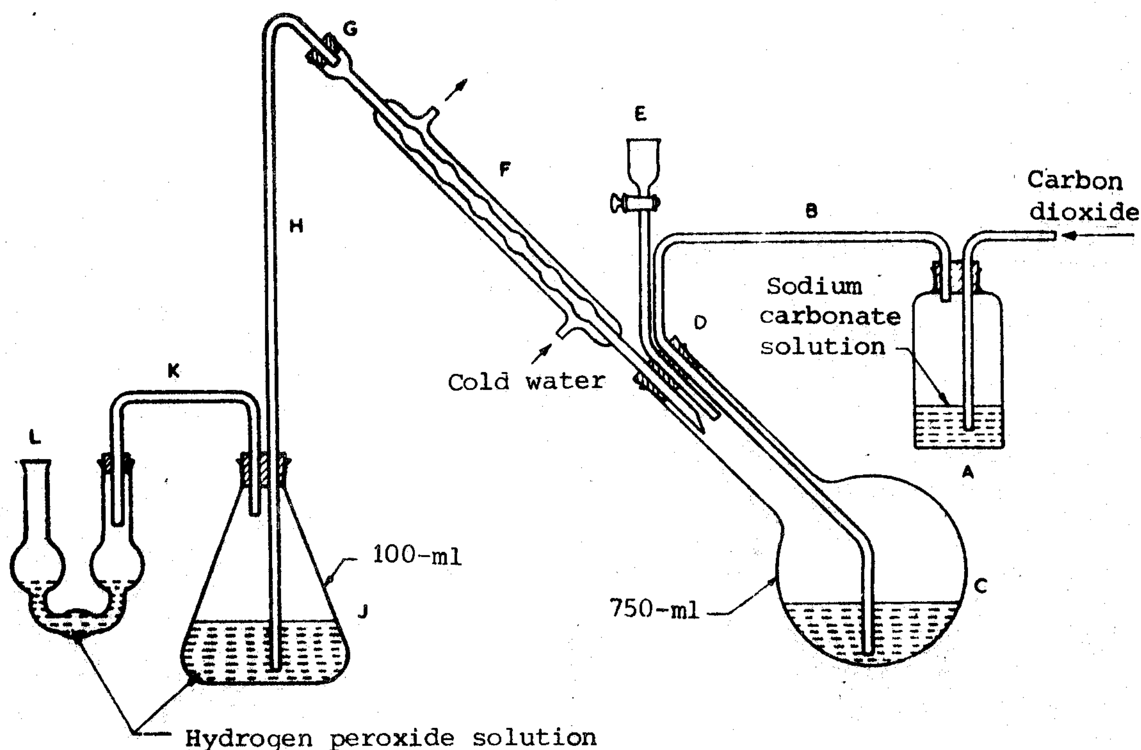

FIGURE 1 - Assembly of apparatus for the determination of sulphur dioxide

9.1.2 The apparatus consists of the round bottom flask C of capacity 750-ml fitted with the three-hole stopper D. The rubber stopper D is fitted with the delivery tube B, the dropping funnel E, and the sloping, water-cooled, reflux condenser F, the lower end of which is cut off at an angle. The free end of delivery tube B is connected to the wash bottle A containing sodium carbonate solution. The upper end of the reflux condenser F is connected to the delivery tube H by the rubber stopper G. The free end of the delivery tube H nearly reaches the bottom of the 100-ml erlenmeyer flask J containing 25 ml of hydrogen peroxide solution. The erlenmeyer flask J is provided with a two-hole rubber stopper; through one hole passes the delivery tube H and through the other, the tube K. The free end of the tube K is connected to the peligot tube L containing 5 ml of hydrogen peroxide solution.

9.2 Reagents

9.2.1 *Sodium carbonate solution,* 10 per cent (m/v), aqueous.

9.2.2 *Bromophenol blue indicator solution*

Dissolve 0.1 g of bromophenol blue in 3.0 ml of 0.05 N sodium hydroxide solution and 5 ml of ethyl alcohol (90 per cent by volume) by gently warming. Make up the volume of the solution with ethyl alcohol (20 per cent v/v) to 250 ml in a volumetric flask.

9.2.3 *Hydrogen peroxide solution*

Dilute a 30 per cent (m/v) hydrogen peroxide solution with about twice its volume of water and neutralize the free sulphuric acid that may be present in the hydrogen peroxide solution with barium hydroxide solution, using bromophenol blue indicator solution. Allow the precipitate of barium sulphate to settle, and filter. Determine the concentration of hydrogen peroxide in the filtrate by titrating with standard potassium permanganate solution. Dilute the filtrate with cold water so as to obtain a 3 per cent(m/v) solution of hydrogen peroxide.

9.2.4 *Concentrated hydrochloric acid,* sp. gr. 1.16.

9.2.5 *Carbon dioxide gas,* from a cylinder.

9.2.6 *Standard sodium hydroxide solution,* approximately 0.1 N standardized at the time of the experiment using bromophenol blue indicator solution.

9.3 Procedure

9.3.1 Assemble the apparatus as shown in Figure 1. Introduce into the flask C, 300 ml of water and 20 ml of concentrated hydrochloric acid through the dropping funnel E. Run a steady current of cold water through the condenser F. Boil the mixture contained in the flask G for a short time to expel air from the system in a current of carbon dioxide gas previously passed through the wash bottle A. Weigh accurately about 100 g of the material and mix with the minimum quantity of water so as to make the diluted material easily flow down to the dropping funnel. Introduce the diluted material into the flask C through the dropping funnel E. Wash the dropping funnel with a small quantity of water and run the washing into the flask C. Again boil the mixture contained in the flask C in a slow current of carbon dioxide gas (passed previously through the wash bottle A) for one 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 over residual traces of sulphur dioxide retained in the condenser). When the delivery tube H, just above the erlenmeyer flask J, becomes hot to the touch, remove 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 solution and titrate with standard sodium hydroxide solution. (bromophenol blue is unaffected by carbon dioxide and gives a distinct change of colour in cold hydrogen peroxide solution).

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

9.4 Calculation

9.4.1 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 the material;

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

N = normality of standard sodium hydroxide solution; and

M = mass in g of the material taken for the test.

10 DETERMINATION OF GELATINE CONTENT

10.1 Reagents

10.1.1 *Catalyst mixture*

Mix intimately 400 g of sodium sulphate, 16 g of hydrated copper sulphate and 3 g of selenium dioxide.

10.1.2 *Screened methyl red indicator*

Dissolve 0.016 g of methyl red and 0.083 g of bromocresol green in 100 ml of alcohol.

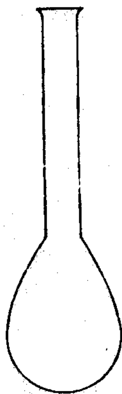

FIGURE 2 -

Macro Kjeldahl digestion flask

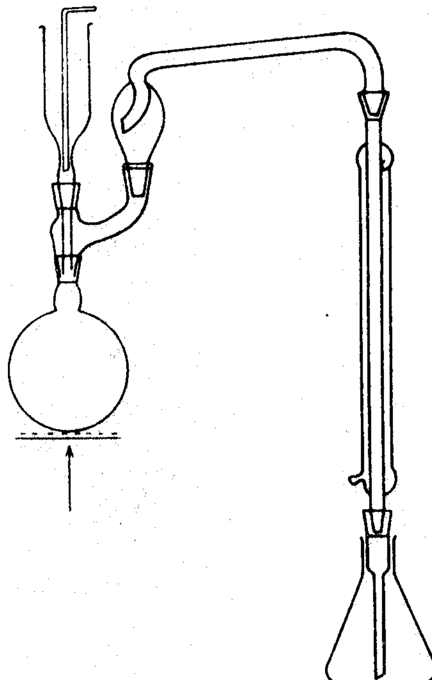

FIGURE 3 - Macro Kjeldahl distillation apparatus

10.2 Procedure

Weigh out a suitable quantity of the material and transfer it to a dry 500-ml to 800-ml kjeldahl digestion flask (Fig. 2) (See Notes 1,2,and 3 below). Add 8 g of catalyst mixture and 20 ml to 25 ml of concentrated nitrogen free sulphuric acid and mix by swirling. Heat the flask fitted with a loose pear-stopper in an inclined position in a fume-cupboard. (See Note 4 below). Apply heat gently at first, but when the initial frothing has subsided, increase the gas supply gradually until the liquid boils at a moderate rate. Swirl and shake the flask from time to time in order to wash down any charred material adhering to the flask. Continue the heating for 1 hour after the liquid has become clear. Allow the flask to cool. Dilute the mixture with not more than 200 ml of tap water and transfer it to a 1-litre distillation flask (Fig. 3). Wash the mixture with several small volumes of tap water until the total volume is about 400 ml. To the 500-ml receiving flask add 50 ml of 2 per cent boric acid solution and a few drops of screened methyl red indicator. Add one large piece of granulated zinc to the distillation flask and connect the apparatus to the delivery tube dipping below the boric acid solution. Ensure that all the joints are tight. Add through the tap funnel 75 ml of 50 per cent sodium hydroxide solution, close the funnel and confirm that the liquid is alkaline after mixing. Boil the alkaline liquid in the flask, taking care to prevent undue frothing in the early stages, and distil over about 300 ml. Open the tap funnel before turning off the gas, wash down the delivery tube into the receiver and titrate the cold distillate with 0.1 N sulphuric acid. A blank should be carried out from time to time. When using reagents of analytical purity grade the blank titration is usually 0.3 ml to 0.5 ml of 0.1 N acid.

NOTES

1 Weigh out the sample on a folded filter paper supported on a watchglass, roll the paper and its contents and drop it directly into the bottom of the flask. A similar filter paper should be included in the blank.

2 Use an amount of sample containing 0.03 g to 0.04 g of nitrogen.

3 Avoid the material clinging to the neck of the flask.

4 The flask should be supported so that it can be shaken easily. If a special stand is not available, place the flask on a sheet of asbestos with a hole and loosely support the neck in a retort stand ring (not a clamp).

5 The mixture will usually become solid if it is allowed to stand. This can be prevented if the liquid is carefully diluted with a small volume of tap water immediately after cooling.

6 Alkalinity is usually shown by the liquid turning from light blue to dark blue owing to the effect on the copper catalyst.

7 Ensure that the rate of flow of the condenser water is sufficient to keep the distillate cold.

10.3 Calculation

$$\text{Total nitrogen} = \frac{A - B \times 0.001\ 4 \times 100}{m}$$

$$\text{Gelatine content} = \text{Total nitrogen} \times 5.55$$

where,

A = volume in ml, of 0.1 N sulphuric acid needed for titration;

B = volume in ml, of 0.1 N sulphuric acid needed for the blank titration; and

m = mass in g, of the material taken for the test.

1 ml of 0.1 N sulphuric acid = 0.0014 g of nitrogen.

Blank Page

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