

भारतीय मानक
Indian Standard

IS 1162 : 2021

केन मोलासेस — विशिष्टि
(पहला पुनरीक्षण)

Cane Molasses — Specification
(*First Revision*)

ICS 67.180.10

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

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Sugar Industry Sectional Committee, FAD 02

FOREWORD

This Indian Standard (First Revision) was adopted by Bureau of Indian Standards, after draft finalized by the Sugar Industry Sectional Committee had been approved by the Food and Agriculture Division Council.

This standard was first published in 1958, and first revision of this standard is undertaken to align it with the latest technology and industry practice. Cane molasses is an important by-product in the manufacture of cane sugar. It is a dark coloured viscous syrupy liquid obtained in this country as a by-product both by the vacuum pan process and the Khandsari process of manufacture of cane sugar. The bulk of the supply of molasses is, however, obtained from the vacuum pan process. The requirements prescribed in this standard are, therefore, related to the material produced from the vacuum pan process.

The composition of molasses depends on such factors as the quality and variety of cane as well as the manorial process, climatic and soil conditions, the extent of milling and the mode of processing of the juice. The method of processing of the juice has, no doubt, a greater influence than other factors. Because of the fact that, in India, molasses is essentially obtained as a by-product during the manufacture of Direct-consumption Plantation white sugar by the vacuum pan process, the composition of molasses produced in this country differs from that obtained in other sugar producing countries where it is obtained during the production of raw sugar. Molasses is used in the fermentation industries for production of ethyl alcohol, rum, yeast, and acetone butanol, certain organic acids, etc. The largest utilization of molasses in India at present is, however, in the alcohol industry. As such, this standard has been drawn up mainly keeping in view the requirements of the alcohol industry.

Molasses has often to be stored in sugar factories for an appreciable length of time. The storage conditions available at many factories are not as good as they should be. Under these conditions, there is a gradual reduction of the percentage of its sugar content. After giving due consideration to these factors for controlling the sale of molasses, it has been decided that there shall be three grades of molasses with varying requirements as specified in Table 1.

In view of these, the determination of total reducing matter when compared to that of fermentable sugars and because of the fact that about 95 percent of the total sugars in molasses are fermentable, it has been decided to prescribe requirements for total reducing matter (*see* Table 1) as against fermentable sugars.

Taking into consideration the views of producers, consumers, testing authorities and technologists, it is felt that it should be related to the manufacturing and trade practices followed in the country in this field. Furthermore, due weightage had to be given to the need for international co-ordination among standards prevailing in different countries of the world in this field.

The *Food Safety and Standards Act*, 2006 and the Rules framed thereunder and the Legal Metrology (*Packaged Commodities*) Rules, 2011. However, this standard is subject to the restrictions imposed under these, whenever applicable.

The composition of the Committee responsible for the formulation of this standard is given at Annex E.

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 IS 2 : 1960 'Rules for rounding off numeral values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in the relevant referred standard.

Indian Standard

CANE MOLASSES — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of test for cane molasses obtained in the manufacture of direct consumption white sugar by the vacuum pan process and meant primarily for use in the alcohol industry.

2 REFERENCES

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

<i>IS No.</i>	<i>Title</i>
266 : 1993	Sulphuric acid (<i>third revision</i>)
376 : 1986	Specification — Sodium hydroxide, analytical reagent (<i>third revision</i>)

3 TERMINOLOGY

For the purpose of this standard, molasses shall mean the mother liquor left over, after the recovery of sugar in the vacuum pan process.

4 GRADES

There shall be three grades of molasses, namely, Grade 1, Grade 2 and Grade 3.

5 SAMPLING

Representative samples of the material shall be drawn as prescribed in Annex A.

6 REQUIREMENTS

6.1 The material shall be dark coloured, viscous, syrupy liquid having a characteristic odour.

6.2 The material shall comply with the requirements given in Table 1.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the, use of water as it reagent is intended.

NOTE — ‘Pure’ chemicals shall mean analytical grade chemicals that do not contain impurities which affect the result of analysis.

Table 1 Requirement for Molasses
(Clause 6.2)

Sl No.	Characteristics	Requirement for			Method of Test (Ref to Annex)
		Grade 1	Grade 2	Grade 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Density in degrees Brix at 27-5° <i>Min</i>	85	80	80	B
ii)	Ash sulphated, percent by mass (calculated for 100° Brix), <i>Max</i>	14.0	17.5	17.5	C
iii)	Total reducing matter as invert sugar, percent by mass, <i>Min</i>	50	44	40	D

8 PACKING AND MARKING

8.1 Bulk Delivery

The material shall be delivered in bulk in tankers.

8.2 Delivery in Containers

The material shall be packed in clean, dry and leak-proof containers. Unless otherwise agreed between the purchaser and the vendor such containers shall be made of tin plate, black or galvanized iron sheets and may be of capacity between 175 and 225 litres or 18 litres.

8.3 Marking

8.3.1 The following information shall be marked legibly and indelibly on each container:

- Name of the material,
- Grade of the material,
- Name of the supplier,
- Net quantity, and
- Any other requirements as given under the *Legal Metrology (Packaged Commodity) Rules, 2011* and *Food Safety and Standards (Food Products Standards and Food Additives) Regulations, 2011*.

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8.3.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity

assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder and the products may be marked with the Standard Mark.

ANNEX A

(Clause 5)

SAMPLING OF CANE MOLASSES

A-1 GENERAL REQUIREMENTS

A-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

A-1.1 The sampling instrument shall be clean and dry when used.

A-1.2 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.

A-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling so as to bring all portions into uniform distribution.

A-1.4 The samples shall be placed in suitable, clean, dry and air-tight metal or glass containers, on which the material has no action.

A-1.5 The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

A-1.6 Each sample container shall be sealed air-tight with a glass stopper after filling, and marked with full details of the sample and the date of sampling.

A-1.7 Sampling can consist of a weighted metal cylinder, with removable top, to which a rod is attached (see Fig. 1). The cylinder is fastened to a suitable rod. For taking sample it is lowered in the tank to the required depth and this top is removed with the help of rod and the can then filled.

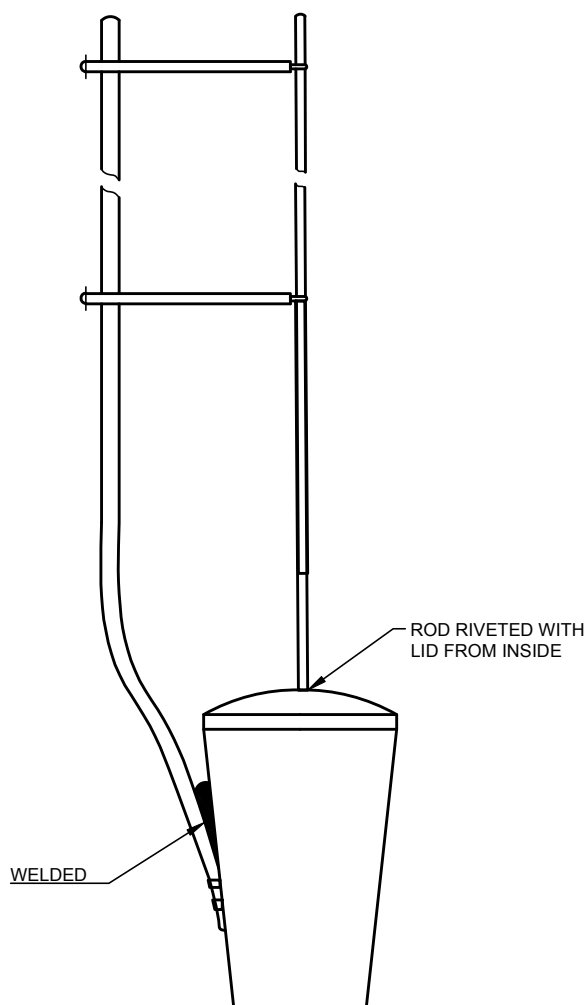


FIG. 1 SAMPLING CAN FOR TANK/TANKER

A-2 SCALE OF SAMPLING

A-2.1 Supply in Tank/Tanker

Samples shall be drawn from each tank/tanker as prescribed in A-3.1.

A-2.2 Supply in Containers

A-2.2.1 All the containers in a single consignment shall constitute one lot.

A-2.2.2 Gross Sample

For the purpose of drawing samples for test, a number of containers shall be selected at random from a lot. This number of containers in relation to the size of the lot, or the scale of sampling, shall be subject to an agreement between the purchaser and the vendor.

As a guide to such an agreement, a scale of the size of the sample is suggested in Table 2.

Table 2 Minimum Number of Containers to be Selected for Sampling from Sampling from Various Sizes of Lots

(Clause A-2.2.2)

Lot Size	Sample Size	Lot Size	Sample Size
2 to 8	2	217 to 343	7
9-27	3	344-512	8
28-64	4	513-729	9
65-125	5	730-1000	10
126-216	6	1001-1331	11

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A-3 PROCEDURE

A-3.1 Sampling from a Tank/Tanker — Take equal portions of the material from different layers of the tank/tanker to obtain a quantity not less than 2.5 liters and mix thoroughly in a suitable container, which is dry and clean, to obtain a composite sample for the tank/tanker. Divide this composite sample in three equal portions of not less than 750 ml in dried bottles or other containers, seal air-tight and label with all the particulars of sampling given under A-1.6.

A-3.2 Sampling from Containers

A-3.2.1 Mixing

Thoroughly mix the contents of all the containers in the gross sample whether they are drums or cans or others, by shaking or stirring, or both, or by rolling so as to bring all portions into uniform distribution.

A-3.2.2 Drawing of Samples

Draw by inserting the sampling device or any other convenient method of sampling, equal portions of the well-mixed material from each container in the gross sample so as to obtain a quantity not less than

2.5 liters. Divide this composite sample into three equal portions of not less than 750 ml in dried bottles or other containers, seal air-tight and label with all the particulars of sampling given under A-1.6.

A-3.3 Sample for Purchaser and Vendor

One sample shall be sent to the purchaser and another to the vendor.

A-3.4 Referee Sample

The third sample bearing the seals of the purchaser and the vendor shall constitute the referee sample to be used in case of dispute between the purchaser and the vendor. It shall be kept at a place agreed to between the purchaser and the vendor.

A-3.5 Time Limit for Analysis

All samples shall be tested as early as possible from the date of sampling.

A-3.6 Criterion for Acceptance

If, on testing, the sample is found to conform to the requirements specified in the standard, the consignment shall be accepted.

ANNEX B

[Table 1, item (i)]

DETERMINATION OF DENSITY IN BRIX

B-1 APPARATUS

B-1.1 Hydrometer — Calibrated at 27.5 °C and fitted with a thermometer.

B-1.2 Immersion Vessel — Suitable for the hydrometer used.

B-2 PROCEDURE

B-2.1 Weigh 200 g of the material. Add 1 400 g of cold water. Mix well till a homogenous solution results.

Determine the Brix using Brix hydrometer or suitable range noting simultaneously the temperature of the solution. Apply the temperature corrections to the readings by using Table 3.

B-3 CALCULATION

Multiply the corrected Brix reading (*see B-2.1*) by 8. This gives the density or the molasses in degrees Brix at 27.5 °C.

**Table 3 Temperature Corrections to Readings of Brix Hydrometers
Calibrated At 27.5 °C Observed Degrees Brix
(Clause B-2.1)**

Temperature °C														
	0	5	10	15	20	25	30	35	40	45	50	55	60	70
Subtract from the Readings														
10	0.75	0.82	0.89	0.96	1.02	1.08	1.14	1.20	1.24	1.28	1.31	1.33	1.35	1.37
11	0.74	0.79	0.86	0.92	0.98	1.03	1.09	1.14	1.18	1.21	1.24	1.26	1.28	1.30
12	0.72	0.76	0.82	0.88	0.93	0.98	1.04	1.08	1.12	1.14	1.17	1.19	1.20	1.22
13	0.69	0.73	0.78	0.84	0.88	0.93	0.98	1.02	1.06	1.07	1.10	1.52	1.13	1.15
14	0.66	0.70	0.74	0.79	0.83	0.88	0.92	0.96	0.99	1.00	1.03	1.05	1.06	1.07
15	0.63	0.66	0.70	0.74	0.78	0.82	0.86	0.90	0.92	0.93	0.95	0.97	0.98	0.99
16	0.60	0.62	0.66	0.69	0.73	0.76	0.80	0.83	0.86	0.86	0.88	0.90	0.91	0.92
17	0.56	0.58	0.61	0.64	0.68	0.70	0.74	0.77	0.79	0.79	0.81	0.83	0.83	0.84
18	0.52	0.54	0.56	0.59	0.62	0.64	0.68	0.70	0.72	0.72	0.74	0.75	0.75	0.76
19	0.48	0.49	0.51	0.54	0.56	0.58	0.61	0.63	0.65	0.65	0.67	0.68	0.68	0.68
20	0.43	0.44	0.46	0.48	0.50	0.52	0.54	0.56	0.58	0.58	0.59	0.60	0.60	0.60
21	0.38	0.39	0.41	0.42	0.44	0.46	0.47	0.49	0.51	0.51	0.51	0.52	0.52	0.52
22	0.33	0.34	0.35	0.36	0.38	0.40	0.40	0.42	0.43	0.43	0.43	0.44	0.44	0.44
23	0.28	0.28	0.29	0.30	0.32	0.33	0.33	0.33	0.36	0.36	0.36	0.36	0.36	0.36
24	0.22	0.22	0.23	0.24	0.25	0.26	0.26	0.27	0.28	0.28	0.28	0.28	0.28	0.28
25	0.16	0.16	0.17	0.18	0.18	0.19	0.19	0.20	0.20	0.20	0.20	0.20	0.20	0.20
26	0.10	0.10	0.11	0.11	0.11	0.12	0.12	0.12	0.12	0.12	0.12	0.12	0.12	0.12
27	0.3	0.03	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04
28	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04
29	0.11	0.11	0.11	0.11	0.11	0.11	0.12	0.12	0.12	0.12	0.12	0.12	0.12	0.12
30	0.18	0.18	0.18	0.18	0.18	0.19	0.19	0.20	0.20	0.20	0.20	0.20	0.20	0.20
31	0.25	0.25	0.25	0.26	0.26	0.27	0.27	0.28	0.28	0.28	0.28	0.28	0.28	0.29
32	0.32	0.33	0.33	0.34	0.34	0.35	0.35	0.36	0.36	0.36	0.36	0.36	0.36	0.37
33	0.40	0.41	0.41	0.42	0.42	0.43	0.43	0.44	0.44	0.44	0.44	0.44	0.44	0.45
34	0.48	0.49	0.49	0.50	0.50	0.51	0.52	0.52	0.52	0.53	0.53	0.53	0.53	0.53
35	0.56	0.57	0.57	0.58	0.59	0.60	0.61	0.61	0.61	0.62	0.62	0.62	0.62	0.62
36	0.64	0.65	0.65	0.67	0.68	0.69	0.70	0.70	0.70	0.71	0.70	0.70	0.70	0.70
37	0.72	0.74	0.74	0.76	0.77	0.78	0.79	0.79	0.79	0.80	0.79	0.79	0.79	0.79
38	0.81	0.83	0.83	0.85	0.86	0.87	0.88	0.88	0.88	0.89	0.88	0.88	0.88	0.88
39	0.90	0.92	0.92	0.94	0.95	0.96	0.97	0.97	0.97	0.98	0.97	0.97	0.97	0.97
40	0.99	1.01	1.01	1.03	1.04	1.05	1.06	1.06	1.06	1.07	1.06	1.05	1.05	1.05
45	1.48	1.50	1.50	1.52	1.53	1.53	1.53	1.53	1.52	1.52	1.51	1.50	1.50	1.48
50	2.03	2.04	2.04	2.05	2.06	2.05	2.04	2.03	2.01	2.00	1.99	1.97	1.96	1.92
55	2.62	2.63	2.63	2.64	2.62	2.60	2.58	2.55	2.52	2.50	2.48	2.45	2.43	2.37
60	3.26	3.28	3.27	3.25	3.22	3.18	3.13	3.09	3.04	3.02	2.98	2.94	2.90	2.83
65	3.95	3.98	3.95	3.90	3.86	3.79	3.71	3.66	3.58	3.56	3.50	3.44	3.38	3.30
70	4.70	4.74	4.70	4.60	4.54	4.44	4.32	4.24	4.14	4.12	4.03	3.96	3.87	3.78
75	5.50	5.56	4.48	5.34	5.26	5.12	4.95	4.86	4.72	4.70	4.57	4.50	4.37	4.27
80	6.35	6.41	6.32	6.12	6.02	5.84	5.62	5.51	5.32	5.30	5.14	5.04	4.88	4.77

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ANNEX C

[Table 1, Item (ii)]

DETERMINATION OF SULPHATED ASH

C-1 REAGENT

Concentrated Sulphuric Acid, sp grade 1.84 (conforming to analytical reagent grade of IS 266).

C-2 PROCEDURE

C-2.1 Weigh accurately about 5 g of the material into a suitable tared platinum dish or quartz crucible. Gradually add 2 ml of concentrated sulphuric acid to the material in the dish. Place the dish or crucible on a hot air oven. When the charred mass does not increase in volume any longer, continue the heating over a small flame, care being taken to avoid spurting, until the fumes of Sulphur trioxide or Sulphur dioxide are not evolved. Increase heat till a pinkish grey ash is obtained (if available a muffle furnace may be used

at a temperature of 550° to 600 °C.) Cool the ash and moisten it with a few drops of concentrated sulphuric acid, heat strongly on a hot plate until fumes of Sulphur trioxide cease to be evolved and finally ash to constant mass.

C-3 CALCULATION

Ash, sulphated, percent by mass (calculated for 100 Brix) = $\frac{10\,000 \times w}{W X}$

where

w = Mass in g, of the ash;

W = Mass in g, of the material taken for the test; and

X = Density of molasses in degrees Brix (**B-3**).

ANNEX D

[Table 1, Item (iii)]

DETERMINATION OF TOTAL REDUCING MATTER

D-1 REAGENTS

D-1.1 Stock Solution of Invert Sugar

Weigh accurately 9.500 0 g of pure sucrose on a watch glass and transfer it to a one liter volumetric flask with 100 ml water. Add 5 ml of concentrated hydrochloric acid (sp gr 1.19). Allow this to stand for 3 days at 20° to 25 °C and then make up to volume with water. This is stable for several months.

D-1.2 Standard Solution of Invert Sugar

Pipette 50 ml of the stock solution of invert sugar (**D-1.1**) in a 250 ml volumetric flask. Neutralize carefully with sodium hydroxide of about one percent (w/v) and make up to the volume.

D-1.3 Methylene Blue Indicator Solution

Dissolve 1.0 g of methylene blue in water and dilute to 100 ml.

D-1.4 Fehling Solution (Soxhlet Modification)

Prepare by mixing immediately before use, equal volumes of solution A, prepared as described under **D-1.4.1**, and solution B, prepared as described under **B-1.4.2**.

D-1.4.1 Solution A

Dissolve 34.639 g of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and 0.5 ml of concentrated sulphuric acid of sp gr 1.84 (conforming to analytical reagent grade of IS 266) and dilute to 500 ml in a volumetric flask. Filter the solution through prepared asbestos.

D-1.4.2 Solution B

Dissolve 173 g of Rochelle salt [potassium sodium tartrate ($\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$)] and 50 g of sodium hydroxide analytical reagent (conforming to IS 376) in water. Dilute to 500 ml in a volumetric flask and allow the solution to stand for two days. Filter this solution through prepared asbestos.

D-1.4.3 Standardization of Fehling Solution Pour standard invert sugar solution (see D-1.2) into a 50 ml burette. Pipette 10 ml of Fehling solution into a 300 ml flask and run in from the burette almost the whole of the standard invert sugar solution required to effect reduction of the copper. 10 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 content of the flask for 2 minutes. At the end of 2 minutes of boiling, add, without interrupting boiling, 3 to 5 drops of methylene

blue indicator solution. While the contents of the flask continue to boil, begin to add standard invert sugar 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. 10 that the contents of the flask boil altogether for 3 minutes without interruption (*see* Note 4, P 18) Note the titre (that is, the total volume in millilitre of standard invert sugar solution used for the reduction of all the copper in 10 ml of Fehling solution): Multiply the titre (obtained direct titration) by the number of milligrams or invert sugar in one millilitre of the standard invert sugar solution to obtain the invert sugar factor. Compare this factor, with the invert sugar factor given in Table 4 and determine correction, if any, to be applied to the invert sugar factors derived from Table 4.

This Table shows, for the standard method of titration, the values corresponding to integral milliliters of the sugar solutions, the values corresponding to intermediate figures being obtained by interpolation.

Table 4 Invert Sugar Factor for 10 ml of Fehling's Solution

(Clause D-1.4.3 and D-3.1)

Titre in Milliliters	Invert Sugar Factor	Milligrams of Invert Sugar per 100 Milliliters of solution
(1)	(2)	(3)
15	50.5	336
16	50.6	316
17	50.7	298
18	50.8	282
19	50.8	267
20	50.9	254.5
21	51.0	242.9
22	51.0	231.8
23	51.1	222.2
24	51.2	213.3
25	51.2	204.8
26	51.3	197.4
27	51.4	190.4
28	51.4	183.7
29	51.5	177.6
30	51.5	171.7
31	51.6	166.3

Table 4 (Concluded)

Titre in Milliliters	Invert Sugar Factor	Milligrams of Invert Sugar per 100 Milliliters of solution
(1)	(2)	(3)
32	51.6	161.2
33	51.7	156.6
34	51.7	152.2
35	51.8	147.9
36	51.8	143.9
37	51.9	140.2
38	51.9	136.6
39	52.0	133.3
40	52.0	130.1
41	52.1	127.1
42	52.1	124.2
43	52.2	121.4
44	52.2	118.7

* Milligrams of invert sugar corresponding to 10 ml of Fehling's solution.

Example:	
Concentration of invert sugar in standard invert sugar solution as mg/100 ml	= 200 mg
Titre obtained by direct titration	= 26.2
Invert sugar factor for 26.2 ml of standard invert sugar solution	= Titre in ml × number of mg of invert sugar in 1 ml of standard = 26.22 = 52.4
Invert sugar factor for 26.2 ml from Table 4 (calculated by interpolation)	= 51.32
Correction to be applied to the invert sugar factors derived from Table 4	= 52.4-51.32 = + 1.08

D-1.5 Neutral Lead Acetate

Dissolve 100 g of lead acetate $[\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}]$ in distilled water and dilute to one litre.

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D-1.6 Sodium Phosphate-Potassium Oxalate Solution

Dissolve 70 g of disodium hydrogen phosphate, dodecahydrate ($\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$) and 30 g of potassium oxalate ($\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$) in water and dilute to one litre.

D-1.7 Sodium Hydroxide Solution

Approximately 6 N, prepared by dissolving sodium hydroxide analytical reagent (conforming to IS 376).

D-1.8 sp gr 1.029 at 20 °C.

D-2 PROCEDURE

D-2.1 Preparation of Solution — Weigh accurately about 12.5 g of molasses and transfer to a 250 ml volumetric flask. Add 25 ml of the lead acetate solution. Make up to volume, mix and filter. Reject the first few drops of the filtrate. To 100 ml of the clear filtrate in a 500 ml volumetric flask. Add 10 ml of the sodium phosphate-potassium oxalate mixture. Make up to volume with water, shake and filter. Reject the first few drops of the filtrate and use the clear filtrate for preparation of invert solution (*see* D-2.1.1).

D-2.1.1 Preparation of Invert Solution

Take 50 ml of the filtrate (*see* D-2.1) in a 100 ml volumetric flask, add 25 ml of water and 10 ml of concentrated hydrochloric acid. Heat on a water-bath at 70 °C and, regulate heat in such a way that the temperature is maintained at 70 °C. Insert a thermometer and heat with constant agitation until the thermometer in the flask indicates 67 °C. When the temperature of the thermometer reaches at 67 °C leave the flask in the water bath for exactly 5 min, during which time the temperature should gradually rise to about 69.5 °C. Plunge the flask at once into water at 20 °C. When the content have cooled to about 35 °C, remove the thermometer from the flask, rinse it and add 10 ml of 6 N sodium hydroxide solution for neutralization of acid, leave the flask in water bath at 20 °C for about 30 min and then make up exactly to volume with water. Mix the solution well.

D-2.2 Incremental Method of Titration

Pour the prepared invert solution (*see* D-2.1.1) into a 50 ml burette (*see* Note 5). 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 (*see* D-2.1). Without further dilution heat the contents of the flask over a wire gauze and boil. After the liquid has been boiled for about 15 seconds it will be possible to judge if the copper is almost all 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 3 to 5 drops of methylene blue indicator solution (*see* Note 1). 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 milliliter 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 4). In case there appears to be still much unreduced copper, after the mixture of Fehling's solution with 15 ml of the prepared solution has been boiling for a quarter of a minute, add the prepared solution from the burette in larger increments (more than one milliliter at a time, according to judgement), and allow the mixture to boil for a quarter of a minute after each addition. Repeat the addition of the prepared solution at intervals of 15 seconds until it is considered unsafe to add large increment of the prepared solution. At this stage continue the boiling for an additional one to two minutes, add 3 to 4 drops of methylene blue indicator solution and complete the titration by adding the prepared solution in small quantities (less than one milliliter at a time) (*see* Note 2).

NOTES

1 It is advisable not to add the indicator until the neighborhood of the end point has been 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, but 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* D-2.3).

D-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 or all the copper (determined under D-2.2) so that, if possible, not more than one milliliter shall be required later to complete the titration. Gently boil the contents of the flask for 2 min. After the 2 min of boiling add one milliliter of methylene blue indicator solution, without interrupting boiling. While the contents of the flask continue to boil, begin to add the solution (one or two drops at a time) from the burette till the blue colour of the indicator just disappear (*see* Note 3). The titration should be completed within one minute and the content of the flask boil altogether for 3 min without interruption (*see* Note 4).

NOTES

3 The indicator is so sensitive that it is possible to determine the end point within one drop of the prepared solution in many cases. The complete decolorization or the methylene blue is usually indicated by the whole reaction liquid which the cuprous oxide is continuously churned up becoming bright red or orange in colour. In case of doubt, the same may be removed from the wire gauze for one or two seconds and the flask held against a sheet of white paper. (A bolder or paper, suitably fixed round the neck of the flask, is very convenient (or this purpose it can be left round the neck of the flask without

risk of over balancing it.) The top edge of the liquid would appear bluish if the indicator is not completely decolorized. 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.

4 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.

5 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.

D-3 CALCULATION

D-3.1 Refer to Table 4 for the invert sugar factor corresponding to the titre (determined as given under **D-2.3**) and apply the correction previously determined.

Milligrams of invert sugar present in 1 ml of the prepared solution = $\frac{\text{Invert Sugar Factor}}{\text{Titre}}$

D-3.2 Reducing matter, percent by mass = $\frac{250 C}{W}$

where

C = milligrams of the invert sugar in 1 ml of the prepared solution (*see D-3.1*), and

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

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ANNEX E

(Foreword)

COMMITTEE COMPOSITION

Sugar Industry Sectional Committee, FAD 02

<i>Organization</i>	<i>Representative(s)</i>
National Sugar Insititure, Kanpur	SHRI NARENDRA MOHAN (Chairman) SHRI ASHUTOSH BAJPAI (<i>Alternate</i>)
Army Service Core (ASC), New Delhi	LT COL B. B. SAHU
CONCERT, Chennai	SHRI R. SANTHANAM SHRI M. SOMASUNDARAM (<i>Alternate</i>)
Consumer Guidance Society of India, Mumbai	SHRI SITARAM DIXIT DR M. S. KAMAT (<i>Alternate</i>)
Food Corporation of India, New Delhi	SHRI DEEPAK KUMAR PANWAR SHRI RAKESH KUMAR RANJAN (<i>Alternate</i>)
Food Safety Standards Authority of India, New Delhi	MS APOORVA SRIVASTAVA (<i>Technical Officer</i>)
Global Cane Sugar Ltd, New Delhi	DR G. S. C. RAO MR ANIL SRIVASTAVA (<i>Alternate</i>)
Indian Institute of Sugarcane Research, Lucknow	DR A. D. PATHAK DR A. K. SHARMA (<i>Alternate</i>)
Indian Institute of Toxicology Research, Lucknow	DR YOGESHWER SHUKLA
Indian Sugar Mills Association, New Delhi	SHRI G. K. THAKUR SHRI PANKAJ RASTOGI (<i>Alternate</i>)
Indian Sugar Exim Corporation, New Delhi	MR RAJIV AGGARWAL MR RAJEEV KURUP (<i>Alternate</i>)
Ministry of Consumer Affairs, Food & Public Distribution, New Delhi	SHRI SURESH CHANDRA
National Co-operative Development Corporation, New Delhi	SHRI K. P. VAISH SHRI N. K. SHARDA (<i>Alternate</i>)
National Fed of Co-operative Development Corporation, New Delhi	MR MANOHAR GOPAL JOSHI
The Sugar Technologists Association of India, New Delhi	SHRI SANJAY AWASTHI SHRI ANURAG GOYAL (<i>Alternate</i>)
Triveni Engineering & Industries Ltd, Muzaffarnagar, UP	RAJESH SINGH P. K. KHADELWAL (<i>Alternate</i>)
BIS Directorate General	SHRI P. RAJESH, SCIENTIST 'E' AND HEAD (FAD) [REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)]

Member Secretary

SHRI RAJPAL
SCIENTIST 'C' (FAD), BIS

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