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Draft Indian Standard

DEXTRIN FOR ADHESIVE INDUSTRY — SPECIFICATION

(First Revision of IS 6367)

(ICS 83.180)

Plastics Sectional Committee,
PCD 12

Last date for receipt of
comment is **06 December 2022**

FOREWORD

(Formal clauses to be added later)

This standard was originally published in 1971. This revision (first) has been undertaken to update the cross-referred standards in the standard.

Dextrin is a soluble carbohydrate formed by partial hydrolysis of starch by acid, enzyme or heat. Dextrin as an adhesive is used in the manufacture of bottled gum, machine labelling paste and poster gumming paste. It is also used as a binder in the manufacture of fireworks, ink tablets, grinding wheels and water colours. Dextrin is specially useful in making envelopes and cigarettes. White dextrin is generally used where colourlessness is specially required, otherwise both types of dextrin, namely, white and yellow, may be used for general purposes.

Dextrin for foundry industry is covered by IS 4269 :1981 and dextrin for textile industry is covered by IS 5448 :1980.

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 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for dextrin for adhesive industry.

2 REFERENCES

2.1 The following standards 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 indicated below:

IS No.	Title
1070 : 1992	Reagent grade water - Specification (<i>third revision</i>)
3434 : 1984	Glossary of terms for adhesives and pressure sensitive adhesive tapes (<i>first revision</i>)

3 TERNINOLOGIES

For the purpose of this standard, the definitions given in IS 3434 shall apply.

4 TYPES

4.1 There shall be two types of the material:

- a) White dextrin;
- b) Yellow dextrin.

5 REQUIREMENTS

5.1 The material shall be in the form of a fine powder, free from adventitious impurities.

5.2 The material shall also comply with the requirements given in Table 1 when tested according to methods given in column 5 of Table 1.

Table 1 Requirements for dextrin for adhesive industry

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No. of Annex A)
		White Dextrin	Yellow Dextrin	
(1)	(2)	(3)	(4)	(5)
i)	Moisture, percent by mass, <i>Max</i>	10.0	10.0	A-2
ii)	Ash, percent by mass (<i>on dry basis</i>), <i>Max</i>	0.5	0.5	A-3
iii)	Cold water solubles, percent by mass (<i>on dry basis</i>), <i>Min</i>	60	90	A-4
iv)	Acidity (ml of 0.1 N NaOH per 100 g) (<i>on dry basis</i>), <i>Max</i>	50	50	A-5
v)	Reducing sugars, percent by mass (<i>on dry basis</i>), <i>Max</i>	10	10	A-6
vi)	Dextrin, percent by mass (<i>on dry basis</i>), <i>Min</i>	53	83	A-7

6 PACKING AND MARKING

6.1 Packing

6.1.1 The material shall be packed in moisture-proof bags each containing 50 kg, unless otherwise agreed upon between the buyer and the seller.

6.2 Marking

6.2.1 The bags shall be marked with the following:

- a) The name of the material;
- b) Manufacturer's name and registered trademark, if any;
- c) Net mass of the contents;
- d) Type of the material;
- e) Code or serial number of packing; and
- f) Any other statutory requirements.

6.2.2 *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.

7 SAMPLING

7.1 Representative samples of the material for testing shall be drawn as prescribed in Annex B.

ANNEX A

(Clause 5.2 and Table 1)

METHODS OF TEST FOR DEXTRIN FOR ADHESIVE INDUSTRY

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

A-2 DETERMINATION OF MOISTURE

A-2.1 Procedure — Dry a wide-mouthed glass weighing bottle in a drying oven at 100 to 105 °C, cool in a desiccator, allow it to attain room temperature and weigh accurately. Weigh accurately about 5 g of the test sample in a tared weighing bottle. Place the weighing bottle with the sample

in the drying oven, partly remove the stopper and dry the sample at 100 to 105 °C to constant mass (± 2 mg) (about 4 h drying is sufficient).

A-2.2 Calculation

$$\text{Moisture, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 = mass in g before drying, and
 M_2 = mass in g after drying.

A-3 DETERMINATION OF ASH

A-3.1 Procedure — Weigh accurately 3 to 5 g of test sample in a tared silica dish. Gently heat the dish on a hot plate until the sample is well carbonized and then increase the heat until carbonization is complete. Transfer the dish with its contents to a muffle furnace and ash at about 800 °C to constant mass.

A-3.2 Calculation

$$\text{Ash, percent by mass (on dry basis)} = \frac{100}{(100 - A)} \times \frac{M_1}{M_2} \times 100$$

where

A = percent moisture content (as determined in A-2),
 M_1 = mass in g of the ash, and
 M_2 = mass in g of the sample taken for the test.

A-4 DETERMINATION OF COLD WATER SOLUBLES

A-4.1 Procedure — Weigh accurately 5 g of the test sample and make it into a uniform slurry with about 80 ml of water at 30 °C. Dilute to about 200 ml in a 250 ml volumetric flask. Shake it for an hour and make up the volume to 250 ml. Filter the contents discarding the first 25 ml of the filtrate and collect exactly 50 ml of the filtrate in a tared dish. Evaporate to dryness on a steam-bath and dry the residue to constant mass in a drying oven at 100 to 105 °C.

A-4.2 Calculation

$$\text{Cold water solubles, percent by mass (on dry basis)} = \frac{M \times 5 \times 100}{S \times (100 - A)} \times 100$$

where

M = mass in g of the residue,
 S = mass in g of the sample taken for the test, and
 A = percent moisture content of the sample (as determined in A-2).

A-5 DETERMINATION OF ACIDITY

A-5.1 Reagents

A-5.1.1 *Standard Sodium Hydroxide Solution* — 0.1 N.

A-5.1.2 *Phenolphthalein Indicator Solution* — 0.1 percent solution in rectified spirit (60 percent by volume).

A-5.1.3 *Neutral Distilled Water* — To 100 ml of freshly boiled and cooled distilled water, add a few drops of phenolphthalein indicator solution and then add, drop by drop, approximately 0.1 N sodium hydroxide solution carefully from a burette until a permanent faint pink colour is produced.

A-5.2 Procedure -Weigh accurately about 10 g of the test sample into a 250-ml beaker. Add about 100 ml of neutral distilled water and 2 drops of phenolphthalein indicator solution. Stir the contents well with the glass rod taking care that no drops splash out. Titrate with standard sodium hydroxide solution until solution under titration just changes to permanent pink.

A-5.3 Calculation

$$\text{Acidity, ml of 0.1 N sodium hydroxide solution required for 100 g of the sample (on dry basis)} = \frac{100000 VN}{M(100-A)}$$

where

V = volume in ml of standard sodium hydroxide solution used in the titration,
 N = normality of sodium hydroxide solution,
 M = mass in g of the sample taken, and
 A = percent moisture content of the sample (as determined in **A-2**).

A-6 DETERMINATION OF REDUCING SUGARS

A-6.1 Regents

A-6.1.1 *Lead Subacetate Solution* — Activate about 200 g of litharge by heating at 650 °C for 3 h (cooled product should be of lemon colour). Boil 430 g of neutral lead acetate, 130 g of freshly activated litharge and one liter of water for 30 min. Cool and allow to settle. Dilute the supernatant liquid to density 1.25 with freshly boiled and cooled water.

A-6.1.2 *Potassium Oxalate* — Crystalline.

A-6.1.3 *Fehling's Solution (Soxhlet Modification)* — Prepared by mixing immediately before use, equal volumes of Solution A (**A-6.1.3.1**) and Solution B (**A-6.1.3.2**).

A-6.1.3.1 *Solution A* — Dissolve 34.639 g of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water, add 0.5ml of concentrated sulphuric acid, (analytical reagent), and dilute to 500 ml in a volumetric flask. Filter the solution through prepared asbestos.

A-6.1.3.2 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, 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.

A-6.1.4 Methylene Blue Indicator Solution — Dissolve 0.2 g of methylene blue in water and dilute to 100ml.

A-6.1.5 Stock Solution of Dextrose — Weigh accurately about 10 g of anhydrous dextrose into a 1 litre volumetric 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 should not be used after 48 h.

A-6.1.6 Standard Dextrose Solution — Dilute a known aliquot of the stock solution of dextrose (**A-6.1.5**) with water containing 0.25 percent (m/v) benzoic acid to such a concentration that more than 15 ml but less than 50 ml of it shall be required to reduce all the copper in the Fehling's solution taken for titration in **A-6.2**. Note the concentration of anhydrous dextrose in this solution as milligrams per 100 ml. Prepare this solution fresh every day.

NOTE — When 10 ml of Fehling's solution (*see* also Note 2 under **A-6.2**) are taken for titration, a standard dextrose solution containing 0.11 to 0.30 percent (m/v) of anhydrous dextrose is convenient for use. With 25 ml of Fehling's solution, a standard dextrose solution containing 0.25 to 0.75 percent of anhydrous dextrose should be used.

A-6.2 Standardization of Fehling's Solution — Pour standard dextrose solution (*see* **A-6.1.6**) into a 50 ml burette (*see* Note 1). Find from Table 2 the titre (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. (For example, if the standard dextrose solution contains 167.0 mg of anhydrous dextrose per 100 ml, the corresponding titre would be 30 ml). Pipette 10ml (*see* Note 2) 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 1 ml 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 min. At the end of 2 min of boiling, add, without interrupting boiling, 1 ml 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 1 minute so that the contents of the flask boil altogether for 3 min without interruption (*see* Note 3)]. Note the titre in millilitres of standard dextrose solution. Multiply the titre (obtained by direct titration) by the number of milligrams of anhydrous dextrose in 1 ml of the standard dextrose solution to obtain the dextrose factor. Compare this factor with the dextrose factor given in Table 2 and determine correction, if any, to be applied to the dextrose factors derived from Table 2.

NOTES:

1. 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.

2. Instead of using 10 ml of Fehling's solution, a 25 ml portion may be used for standardization of dextrose solution and also for titration of test solution (A-6.3.1.2). In that case, Table 3 shall be used in place of Table 2.

3. 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.

Example:

Concentration of anhydrous dextrose in standard dextrose solution, mg/100 ml	= 167.0
Titre obtained by direct titration	= 30.1 ml
Dextrose factor for 30.1 ml of standard dextrose solution (titre in millilitres X number of milligrams of anhydrous dextrose in 1 ml 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 factors derived from Table 2	= 50.2670 - 50.11 = +0.1570

A-6.3 Procedure

A-6.3.1 Weigh accurately about 10 g of the test sample in a beaker. Mix it thoroughly with 100 ml of water. Transfer to a 250 ml volumetric flask. Add drop by drop lead subacetate solution till precipitation is complete. Make the solution up to the mark by adding water. Mix thoroughly and allow it to settle down. Filter the supernatant liquid and collect the filtrate in a dry beaker. Add dry potassium oxalate to the filtrate to remove excess of lead. Filter and collect the filtrate (as a clarified solution) in a 250 ml flask. Make it up to the mark for subsequent estimations of reducing sugar (A-6.3.2) and dextrin (A-7).

Table 2 Dextrose factors for 10 ml of Fehling's Solution
(Clause A-6.2)

<i>Titre, ml</i>	<i>Dextrose Factor¹⁾</i>	<i>Anhydrous Dextrose per 100ml of Solution, mg</i>
(1)	(2)	(3)
15	49.1	327
16	49.2	307
17	49.3	289
18	49.4	274
19	49.5	260
20	49.5	247.4

21	49.5	235.8
22	49.5	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

NOTE — 1) Milligrams of anhydrous dextrose corresponding to 10 ml of Fehling's solution.

Table 3 Dextrose factors for 25 ml of Fehling's Solution
[Clause A-6.2 (Note 2) and A-6.4]

<i>Titre,</i> ml	<i>Dextrose Factor¹⁾</i>	<i>Anhydrous Dextrose per</i> <i>100ml of Solution,</i> mg
(1)	(2)	(3)
15	120.2	801
16	120.2	751
17	120.2	707
18	120.2	668
19	120.3	633

20	120.3	601.5
21	120.3	572.9
22	120.4	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

NOTE — 1) Milligrams of anhydrous dextrose corresponding to 25 ml of Fehling's solution.

A-6.3.2 *Estimation of Reducing Sugars*

A-6.3.2.1 *Incremental method of titration* — Pour the clarified solution (**A-6.3.1**) into a 50 ml burette (*see* Note 1 under **A-6.2**). Pipette 10 ml of Fehling's solution (*see* Note 2 under **A-6.2**) into a 300 ml conical flask and run in from the burette 15 ml of the clarified 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 s, it will be possible to judge if the copper is almost 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 1 ml of methylene blue indicator solution (*see* Note 1). Continue boiling the contents of the flask for 1 to 2 min from the commencement of ebullition, and then add the clarified solution in small quantities (1 ml or less at a time) allowing the liquid to boil for about 10

s between successive additions, till the blue colour of the indicator just disappears (*see* Note 3 under **A-6.2**). In case there appears to be still much unreduced copper, after the mixture of Fehling solution with 15 ml of the clarified solution has been boiling for 15 s, add the clarified solution from the burette in large increments (more than 1 ml at a time, according to judgement) and allow the mixture to boil for 15 s after each addition. Repeat the addition of the clarified solution at intervals of 15 s until it is considered unsafe to add a large increment of the clarified solution. At this stage continue the boiling for an additional 1 to 2 min, add 1 ml of methylene blue indicator solution and complete the titration by adding the clarified solution in small quantities (less than 1 ml at a time) (*see also* 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* **A-6.3.2.2**).

A-6.3.2.2 Standard method of titration — Pipette 10 ml of Fehling's solution (*see* Note 2 under **A-6.2**) into a 300 ml conical flask and run in from the burette almost the whole of the clarified solution required to effect reduction of all the copper (determined under **A-6.3.2.1**), so that, if possible, not more than 1 ml shall be required later to complete the titration. Gently boil the contents of the flask for 2 min. At the end of 2 min of boiling, add, without interrupting boiling, 1 ml of methylene blue indicator solution. While the contents of the flask continue to boil, begin to add the clarified 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 1 min so that the contents of the flask boil altogether for 3 min without interruption (*see* Note 3 under **A-6.2**).

NOTE — The indicator is so sensitive that it is possible to determine the end point within one drop of the clarified solution in many cases. The complete decolourization 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. In case of doubt, the flame may be removed from the wire gauze for 1 or 2 s and the flask held against a sheet of white paper. (A holder of paper, suitably fixed round 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 decolorized. It is advisable not to interrupt the boiling for more than a few seconds as the indicator undergoes back oxidation rather rapidly when air is allowed free access to flask, but there is no danger of this as long as continuous stream of steam is issuing from the mouth of the flask.

A-6.4 Calculation — Refer to Table 2 (or Table 3 as appropriate) for the dextrose factor corresponding to the titre (determined as given under **A-6.3.2.2**) and apply the correction previously determined under **A-6.2**. Calculate the dextrose content of the clarified solution (*see* **A-6.3.1**) as follows:

$$\text{Anhydrous dextrose present in 1 ml of the clarified solution, mg} = \frac{\text{dextrose factor}}{\text{titre}}$$

NOTE — Tables 2 and 3 show for the standard method of titration, the values corresponding to integral millilitres of the sugar solutions; values corresponding to intermediate figures shall be obtained by interpolation

Reducing sugars, percent by mass (on dry basis)	$= \frac{mV}{10M} \times \frac{100}{100-A}$
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where

m = anhydrous dextrose in mg present in 1 ml of the clarified solution (*see A-6.4*).
V = total volume in ml of the clarified solution of the material prepared in **A-6.3.1**,
M = mass in g of the material taken for the test in **A-6.3.1**, and
A = percent moisture content of the sample as determined in **A-2**.

A-7 DETERMINATION OF DEXTRIN

A-7.1 Reagents — In addition to the reagents listed under **A-6.1**, the following reagents are required:

- a) *Hydrochloric acid* — Concentrated.
- b) *Sodium hydroxide solution* — 10 percent.

A-7.2 Procedure for Estimation of Dextrin Content

Take 50 ml of clarified solution (*see A-6.3*) in a clean 400 ml flask. Add 20 ml of hydrochloric acid and 100 ml of water. Boil gently using water cooled reflux condenser for 2.5 h. Cool to room temperature. Neutralize the solution with sodium hydroxide solution. Transfer the solution to a 500 ml volumetric flask and make up to the mark. Determine the reducing sugar in percent by mass as under **A-6**. Subtract from this, the percentage of reducing sugars obtained under **A-6.4.1** and multiply it by 0.9 to get the percentage of dextrin.

ANNEX B (Clause 7.1)

SAMPLING OF DEXTRIN FOR ADHESIVE INDUSTRY

B-1. SCALE OF SAMPLING

B-1.1 Lot — In any consignment, all the bags of material of the same type and drawn from the same batch of manufacture shall be grouped together to constitute a lot.

B-1.1.1 Tests for the determination of the conformity of a lot to the requirements of the specification shall be done for each lot separately. The number of bags to be selected at random for this purpose from a lot shall be as given below:

<i>No. of Bags in the Lot</i>	<i>No. of Bags to be Selected</i>
Up to 15	3
16 to 25	4

26 to 50	5
51 to 100	7
101 and above	10

B-2 PREPARATI ON OF TEST SAMPLES

B-2.1 From each of the bags selected according to **B-1.1.1**, small portions of the material shall be drawn with the help of a suitable sampling instrument from different parts of the bags. The total quantity of the material so drawn from each bag shall be not less than 200 g and these shall form the individual samples representative of the different bags selected.

B-2.2 From each of the individual samples formed according to **B-2.1**, approximately 150 g of the material shall be drawn and mixed together to form a composite sample. The composite sample so formed may be reduced further, if necessary, by coning and quartering so as to obtain enough material sufficient to conduct all the tests specified in this standard,

B-3 NUMBER OF TESTS

B-3.1 Tests for the determination of dextrin content shall be conducted on each of the individual samples (*see B-2.1*) separately.

B-3.2 Tests for the determination of all other characteristics mentioned in Table 1 shall be conducted on the composite sample (*see B-2.2*).

B-4 CRITERIA FOR CONFORMITY

B-4.1 The lot shall be declared as conforming to the requirements of the specification if the conditions stipulated in **B-4.1.1** and **B-4.1.2** are satisfied.

B-4.1.1 From the individual test results for dextrin content, the mean and range (difference between the maximum and minimum values of test results) shall be calculated. The value of the expression (mean — 0.6 range) shall then be found to be greater than or equal to the relevant minimum value specified in Table 1.

B-4.1.2 The test results for all the characteristics determined on the composite sample shall be found to be satisfactory.