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# **Corn Syrup**

CAS RN®: 8029-43-4.

## **DEFINITION**

Corn Syrup is an aqueous solution of saccharides obtained by partial hydrolysis of edible corn starch by food grade acids and/or enzymes. It contains NLT 20.0% reducing sugar content (dextrose equivalent) expressed as p-glucose, calculated on the dried basis.

#### IDENTIFICATION

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Analysis: Add a few drops of a solution of Syrup (1 in 20) to 5 mL of hot, alkaline cupric tartrate TS.

Acceptance criteria: A copious, red precipitate of cuprous oxide is formed.

#### **ASSAY**

Reducing Sugars (Dextrose Equivalent)

**Apparatus:** Mount a ring support on a ring stand 1–2 in above a gas burner, and mount a second ring 6–7 in above the first. Place 6-in openwire gauze on the lower ring to support a 250-mL conical flask, and place a 4-in watch glass with a center hole on the upper ring to deflect heat. Attach a 25-mL buret to the ring stand so that the tip just passes through the watch glass centered above the flask. Place an indirectly lighted white surface behind the assembly for observing the endpoint.

**Standard solution:** 6 mg/mL of <u>USP Dextrose RS</u> **Sample solution:** 10 mg/mL of Corn Syrup

Analysis: Transfer 25.0-mL portions of alkaline cupric tartrate TS to each of two flasks, and boil. Immediately place one flask on the wire gauze of the *Apparatus*, and adjust the burner so that the boiling point is reached in about 2 min. Titrate with the *Standard solution* to within 0.5 mL of the anticipated endpoint. Heat the flask, with swirling, boil moderately for 2 min, and add 2 drops of methylene blue solution (1 in 100). Immediately add 2 drops of the *Standard solution* from the buret, and bring to a boil. Allow the cuprous oxide to settle slightly, and observe the color of the supernatant. Complete the titration within 1 min by adding the *Standard solution* dropwise, and boil after each addition to the disappearance of the blue color, as determined by viewing against a white background in daylight or under equivalent illumination. If more than 0.5 mL of the titrant is required after the addition of the indicator, repeat the titration, adding the necessary volume of titrant before adding the indicator. Bring the contents of the second flask to a boil, and similarly titrate with the *Sample solution*. Calculate the percentage of reducing sugars as p-glucose, calculated on the dried basis, in the portion of Corn Syrup taken:

Result = 
$$(C_c/C_{ij}) \times (V_c/V_{ij}) \times [1/(0.01 \times A)] \times 100$$

C<sub>s</sub> = concentration of <u>USP Dextrose RS</u> in the Standard solution (mg/mL)

 $C_{ij}$  = concentration of Corn Syrup in the Sample solution (mg/mL)

V<sub>s</sub> = titrated volume of the Standard solution (mL)

 $V_{ij}$  = titrated volume of the Sample solution (mL)

A = percentage of dry solids in Corn Syrup measured by the refractive index

Acceptance criteria: NLT 20.0% reducing sugar content on the dried basis

# **IMPURITIES**

Residue on Ignition (281)

Sample: 20 g

Acceptance criteria: NMT 0.5%

https://trungtamthuoc.com/

#### LIMIT OF LEAD

[Note—For the preparation of all aqueous solutions and for the rinsing of glassware before use, use water that has been passed through a strong-acid, strong-base, mixed-bed ion-exchange resin. For digestion, use acid-cleaned, high-density polyethylene, polypropylene, polytef, or quartz tubes. Select all reagents to have as low a content of lead as practicable, and store all reagent solutions in borosilicate glass containers. Cleanse glassware before use by soaking in warm 8 N nitric acid for 30 min and rinsing with deionized water. Store final diluted solutions in acid-cleaned plastic or polytef tubes or bottles.]

Modifier solution: 200 mg/mL of magnesium nitrate. Just before use, transfer 1.0 mL of this solution to a 10-mL volumetric flask, and dilute with 5% nitric acid to volume.

Standard stock solution: Transfer 10.0 mL of lead nitrate stock solution TS to a 100-mL volumetric flask, add 40 mL of water and 5 mL of nitric acid, and dilute with water to volume. Transfer 1.0 mL of this solution to a second 100-mL volumetric flask, dilute with 5% nitric acid to volume, and mix. This solution contains 0.1 µg/mL of lead.

Standard solutions: Transfer portions of Standard stock solution to four suitable containers, and dilute with 5% nitric acid to obtain Standard solutions having lead concentrations of 100, 50, 25, and 10 ng/mL, respectively.

Sample solution: [Note—Perform this procedure in a fume hood.] Transfer 1.5 g of Corn Syrup to a digestion tube, and add 0.75 mL of nitric acid to the tube. Warm the solution slowly (to avoid spattering) to 90°-95°. Heat until all brown vapors have dissipated and any rust-colored tint has disappeared from the tube (20-30 min). Cool, add 0.5 mL of 50% hydrogen peroxide, dropwise, to the solution, heat to  $90^{\circ}-95^{\circ}$  for 5 min, and cool. Add a second 0.5-mL portion of 50% hydrogen peroxide dropwise to the solution, and heat to 90°-100° until clear (5-10 min). Cool, and transfer the solution to a 10-mL volumetric flask. Rinse the digestion tube with 5% nitric acid, add the rinse to the volumetric flask, dilute with 5% nitric acid to volume, and mix.

Standard blank: 5% Nitric acid

Sample blank: Transfer 1.5 g of water to a digestion tube, and proceed as directed for the Sample solution, beginning with "add 0.75 mL of nitric acid".

#### Instrumental conditions

Mode: Graphite furnace atomic absorption with pyrolytically coated graphite tubes and adequate means of background correction

Lamp: Lead hollow-cathode

Analytical wavelength: Lead emission line of 283.3 nm

Furnace program: See <u>Table 1</u>. [Note—The temperature program may be modified to obtain optimum furnace temperatures.]

Table 1

Step	Dry	Ash	Purge	Atomize
Temperature (°)	200	750	Cool down, and purge the air from the furnace 20	1800
Ramp time(s)	20	40	_	0
Hold time(s)	30	40	60	10
Argon flow rate (mL/min)	300	300	300	Argon gas flow stopped

Injection volume: 20 µL

## **Analysis**

[Note—Use peak area measurements for all quantitations.]

Samples: Add 5 µL of the Modifier solution to 20 µL each of the Standard solutions, the Sample solution, the Standard blank, and the Sample blank, and mix.

Separately inject equal volumes (about 20 µL) of the Samples into the instrument for analysis. Using the Standard blank to set the instrument to zero, determine the integrated absorbances of the Standard solutions. Plot the integrated absorbances of the Standard solutions versus their contents of lead, in ng/mL, and draw the line best fitting the four points to determine the calibration curve. Similarly determine the integrated absorbances of the Sample solution and the Sample blank. Correct the absorbance value of the Sample solution by subtracting from it the absorbance value obtained from the Sample blank.

Calculate the concentration of lead in the portion of Corn Syrup taken:

Result =  $(C_I \times V/W) \times F$ 

C, = concentration of lead in the Sample solution, as determined from the calibration curve (ng/mL)

V = volume of the Sample solution, 10 mL

W = weight of Corn Syrup taken to prepare the Sample solution (g)

F = conversion factor, 10<sup>-3</sup> μg/ng

Acceptance criteria: NMT 0.5 µg/g

· LIMIT OF SULFUR DIOXIDE

**Starch indicator solution:** Mix 10 g of soluble starch with 50 mL of cold water, transfer to 1000 mL of boiling water, stir until completely dissolved, cool, and add 1 g of salicylic acid preservative. [Note—Discard this solution after 1 month.]

Sample: 100 g

Blank: 200 mL of water
Titrimetric system
(See <u>Titrimetry (541)</u>.)
Mode: Direct titration
Titrant: 0.005 N iodine VS

**Endpoint detection:** Visual

**Analysis:** Transfer the *Sample* to a 250-mL conical flask, add 100 mL of water, and mix. Cool to 5°-10°. While stirring with a magnetic stirrer, add 10 mL of cold (5°-10°) 1.5 N sodium hydroxide. Stir for an additional 20 s, and add 10 mL of *Starch indicator solution*. Add 10 mL of cold (5°-10°) 2.0 N sulfuric acid, and titrate immediately with *Titrant* until a light blue color persists for 1 min. Perform a blank determination, and make any necessary correction.

Calculate the concentration, in ppm (µg/g), of sulfur dioxide (SO<sub>2</sub>) in the Sample taken:

Result = 
$$[(V_S - V_B) \times N \times F_1/W] \times F_2$$

V<sub>s</sub> = Titrant volume consumed by the Sample (mL)

 $V_{\rm p}$  = Titrant volume consumed by the Blank (mL)

N = actual normality of the Titrant (mEq/mL)

 $F_{\star}$  = equivalency factor, 32.0 mg/mEq

W = Sample weight (g)

 $F_2$  = conversion factor,  $10^3 \, \mu g/mg$ 

Acceptance criteria: NMT 40 µg/g

• ABSENCE OF SOLUBLE STARCH

**Analysis:** Dissolve 1 g in 10 mL of water, and add 1 drop of iodine TS. **Acceptance criteria:** A yellow color indicates the absence of soluble starch.

# **SPECIFIC TESTS**

- MICROBIAL ENUMERATION TESTS (61) and Tests for Specified Microorganisms (62): The total aerobic microbial count does not exceed 10<sup>3</sup> cfu/g, and the total combined molds and yeasts count does not exceed 10<sup>2</sup> cfu/g.
- Total Solids

### Instrumental conditions

(See Refractive Index (831).)

**Mode:** Refractometer equipped with a jacket for water circulation or some other mechanism for maintaining the sample at 20 ± 0.1° or 45 ± 0.1°

Before proceeding with measurements, ensure that the sample and the prism have reached the equilibrium temperature and that the instrument has been properly checked and calibrated against a standard provided by the manufacturer.

**Analysis:** Measure the refractive index of Corn Syrup, and convert the value to approximate percent solids value using <u>Table 2</u> and <u>Table 3</u>. [Note—<u>Table 2</u> covers the approximate total solids levels of these products in commerce. If the ash or dextrose equivalent of the sample

differs from that of the product in <u>Table 2</u>, use <u>Table 3</u> for the ash and dextrose equivalent correction.]

Table 2. Reference for Converting the Refractive Index to Approximate Percent Solids

Dextrose Equivalent (DE)	Dry Substance (DS) (%)	Refractive Index at 20°	Refractive Index at 45°	Degrees Baumé at 140° F (60° C) + 1
	76.0	1.4888	1.4837	40.98
	77.0	1.4915	1.4864	41.49
	78.0	1.4943	1.4892	42.00
20 DE Com ourum 0 20	79.0	1.4971	1.4919	42.51
28 DE Corn syrup—0.3% - ash	80.0	1.4999	1.4947	43.01
	78.6	1.4933	1.4882	41.99
	79.6	1.4960	1.4909	42.49
	80.6	1.4988	1.4936	42.99
24 DE Himb madhasa	81.6	1.5015	1.4964	43.49
34 DE High-maltose corn syrup=0.3% ash	82.6	1.5043	1.4992	43.99
	78.4	1.4938	1.4887	42.01
	79.4	1.4965	1.4914	42.52
	80.4	1.4993	1.4941	43.02
06 DE 0 0.0%	81.4	1.5021	1.4969	43.52
36 DE Corn syrup—0.3% - ash	82.4	1.5049	1.4997	44.02
	78.9	1.4934	1.4883	42.00
	79.9	1.4961	1.4910	42.51
	80.9	1.4988	1.4937	43.01
40.05.11.1	81.9	1.5016	1.4964	43.51
43 DE High-maltose corn syrup—0.3% ash	82.9	1.5044	1.4992	44.01
	78.7	1.4933	1.4882	42.01
	79.7	1.4960	1.4909	42.51
	80.7	1.4988	1.4936	43.02
40.05.0	81.7	1.5015	1.4964	43.52
43 DE Corn syrup—0.3% - ash	82.7	1.5043	1.4992	44.01
43 DE Corn syrup (ion-exchanged)-0.03%	78.8	1.4935	1.4884	41.99

Dextrose Equivalent (DE)	Dry Substance (DS) (%)	Refractive Index at 20°	Refractive Index at 45°	Degrees Baumé at 140° F (60° C) + 1
ash	79.8	1.4962	1.4911	42.50
	80.8	1.4990	1.4938	43.00
	81.8	1.5018	1.4966	43.50
	82.8	1.5045	1.4994	43.99
	80.5	1.4962	1.4911	42.64
	81.5	1.4989	1.4938	43.14
	82.5	1.5016	1.4965	43.64
50.55.0	83.5	1.5044	1.4992	44.13
53 DE Corn syrup—0.3% ash	84.5	1.5072	1.5020	44.63
	81.0	1.4955	1.4904	42.53
	82.0	1.4982	1.4931	43.02
	83.0	1.5009	1.4958	43.52
62 DE Composition 0.2%	84.0	1.5037	1.4985	44.01
63 DE Corn syrup—0.3% ash	85.0	1.5064	1.5012	44.50
	81.3	1.4963	1.4912	42.60
	82.3	1.4990	1.4939	43.10
	83.3	1.5017	1.4965	43.59
63 DE Corn syrup (ion-exchanged)—0.03%	84.3	1.5044	1.4993	44.09
ash	85.3	1.5072	1.5020	44.58
	81.0	1.4949	1.4898	42.36
	82.0	1.4975	1.4924	42.86
	83.0	1.5002	1.4951	43.36
66 DE Corn syrup—0.3%	84.0	1.5029	1.4978	43.85
ash	85.0	1.5056	1.5005	44.35
95 DE Corn syrup-0.3% ash	69.0	1.4598	1.4550	35.46
αοιι	70.0	1.4621	1.4573	35.96
	71.0	1.4644	1.4596	36.46
	72.0	1.4668	1.4619	36.96

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Dextrose Equivalent (DE)	Dry Substance (DS) (%)	Refractive Index at 20°	Refractive Index at 45°	Degrees Baumé at 140° F (60° C) + 1
	73.0	1.4692	1.4643	37.45
	69.0	1.4597	1.4549	35.39
	70.0	1.4620	1.4572	35.89
	71.0	1.4644	1.4595	36.39
95 DE Corn syrup (ion-exchanged)—0.03%	72.0	1.4667	1.4619	36.89
ash	73.0	1.4691	1.4642	37.38

Table 3. Ash and Dextrose Equivalent (DE) Corrections for Corn Syrup: Changes in Refractive Index for an Increase in Dry Substance (DS)

Dry Substance (DS) (%)	1% Ash	1 Dextrose Equivalent (DE)
2	0.000000	-0.000001
4	0.000000	-0.000003
6	0.000001	-0.000005
8	0.000002	-0.000007
10	0.000003	-0.000010
12	0.000004	-0.000012
14	0.000006	-0.000015
16	0.00008	-0.000017
18	0.000010	-0.000020
20	0.000013	-0.000023
22	0.000016	-0.000026
24	0.000019	-0.000029
26	0.000022	-0.000033
28	0.000026	-0.000036
30	0.000030	-0.000040
32	0.000034	-0.000044
34	0.000039	-0.000048
36	0.000044	-0.000052
38	0.000049	-0.000057

Dry Substance (DS)	1% Ash	1 Dextrose Equivalent (DE)
(%)		
40	0.000055	-0.000061
42	0.000061	-0.000066
44	0.000068	-0.000071
46	0.000074	-0.000076
48	0.000082	-0.000081
50	0.000089	-0.000087
52	0.000097	-0.000093
54	0.000105	-0.000099
56	0.000114	-0.000105
58	0.000123	-0.000112
60	0.000133	-0.000118
62	0.000143	-0.000125
64	0.000153	-0.000132
66	0.000164	-0.000140
68	0.000175	-0.000147
70	0.000187	-0.000155
72	0.000199	-0.000163
74	0.000212	-0.000172
76	0.000225	-0.000181
78	0.000239	-0.000190
80	0.000253	-0.000199
82	0.000268	-0.000208
84	0.000283	-0.000218

Acceptance criteria: The total solids value is NLT 70.0%.

## **ADDITIONAL REQUIREMENTS**

- PACKAGING AND STORAGE: Preserve in tight containers. No storage requirements specified.
- Label it to indicate its nominal dextrose equivalent. Label it also to indicate the presence of sulfur dioxide if the residual concentration is greater than 10 ppm ( $\mu$ g/g).
- USP REFERENCE STANDARDS (11)

  USP Dextrose RS

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
CORN SYRUP	<u>Documentary Standards Support</u>	CE2020 Complex Excipients

Chromatographic Database Information: Chromatographic Database

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