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# Corn Syrup Solids

## DEFINITION

Corn Syrup Solids (Dried Glucose Syrup) is a dried mixture 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 D-glucose, calculated on the dried basis.

## IDENTIFICATION

• **A.**

**Sample solution:** 50 mg/mL

**Analysis:** Add a few drops of the *Sample solution* to 5 mL of hot, alkaline cupric tartrate TS.

**Acceptance criteria:** A copious, red precipitate of cuprous oxide is formed (distinction from sucrose).

## 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 a 6-in open-wire 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 USP Dextrose RS

**Sample solution:** Transfer 4 g of Corn Syrup Solids to a 500-mL volumetric flask, and dilute with water to volume.

**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 will be reached in 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 a 10-mg/mL methylene blue solution. 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 boiling 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 D-glucose, calculated on the dried basis, in the portion of Corn Syrup Solids taken:

$$\text{Result} = (C_S/C_U) \times (V_S/V_U) \times [1/(0.01 \times A)] \times 100$$

$C_S$ = concentration of USP Dextrose RS in the *Standard solution* (mg/mL)

$C_U$ = concentration of Corn Syrup Solids taken to prepare the *Sample solution* (mg/mL)

$V_S$ = titrated volumes of the *Standard solution* (mL)

$V_U$ = titrated volumes of the *Sample solution* (mL)

$A$ = percentage of dry solids in Corn Syrup Solids, as determined in the test for *Total Solids*

**Acceptance criteria:** NLT 20.0% reducing sugar content (dextrose equivalent) expressed as D-glucose on the dried basis

## IMPURITIES

• **Residue on Ignition ⟨281⟩:** NMT 0.5%

• **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, and stir until completely dissolved. Cool, and add 1 g of salicylic acid preservative. [Note—Discard the solution after 1 month.]

**Sample:** 78 g

**Blank:** 200 mL of water

**Titrimetric system**

(See *Titrimetry* ⟨541⟩.)

**Mode:** Direct titration

**Titrant:** 0.005 N iodine VS

**Endpoint detection:** Visual

**Analysis:** Transfer the *Sample* to a 250-mL conical flask. Dilute with 122 mL of water, and mix to dissolve. 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 amount of sulfur dioxide ($SO_2$) in the *Sample* taken:

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

$V_S$ = *Titrant* volume consumed by the *Sample* (mL)

$V_B$ = *Titrant* volume consumed by the *Blank* (mL)

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

$F_1$ = equivalency factor, 32.0 mg/mEq

$F_2$ = conversion factor, $10^3$ µg/mg

$W$ = *Sample* weight (g)

**Acceptance criteria:** NMT 40 µg/g

• **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 solutions:** 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, dilute with water to volume, and mix. Transfer 1.0 mL of this solution to a 100-mL volumetric flask, and dilute with 5% nitric acid to volume. This solution contains 0.1 µg/mL of lead. Transfer portions of this 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 1.2 g of Corn Syrup Solids to two digestion tubes labeled *Sample solution* and temperature monitor solution, and add 0.75 mL of nitric acid to each tube. Warm both solutions slowly to 90°–95° to avoid spattering. Heat until all brown vapors have dissipated and any rust-colored tint is gone from the tube labeled *Sample solution* (20–30 min). Cool, then add 0.5 mL of 50% hydrogen peroxide dropwise to both solutions, and heat to between 90° and 95° for 5 min. Cool, then add a second 0.5-mL portion of 50% hydrogen peroxide dropwise to each solution, and heat to 90°–100° until clear (5–10 min). Cool, and transfer the solution labeled *Sample solution* to a 10-mL volumetric flask. Rinse the *Sample solution* digestion tube with 5% nitric acid, add the rinse to the volumetric flask, and dilute with 5% nitric acid to volume.

**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**

(See *Atomic Absorption Spectroscopy* ⟨852⟩.)

**Mode:** Atomic absorption spectrophotometer equipped with pyrolytically coated graphite tubes and adequate means of background correction

**Analytical wavelength:** 283.3 nm

**Injection volume:** 20 µL

**Furnace program:** See *Table 1*.

[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 |

**Analysis**

**Samples:** *Standard solutions*, *Sample solution*, *Standard blank*, *Sample blank*

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 each into the atomic absorption spectrophotometer.

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, 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* with the *Sample blank*.

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

$$\text{Result} = [V \times (C/W)] \times F$$

$V$ = volume of the *Sample solution*, 10 mL

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

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

$F$ = conversion factor, $10^{-3}$ µg/ng

**Acceptance criteria:** NMT 0.5 µg/g

## SPECIFIC TESTS

• **Microbial Enumeration Tests ⟨61⟩** and **Tests for Specified Microorganisms ⟨62⟩:** The total aerobic microbial count does not exceed $10^3$ cfu/g, and the total combined yeasts and molds count does not exceed $10^2$ cfu/g.

• **Starch**

**Sample solution:** 100 mg/mL

**Analysis:** Add 1 drop of iodine TS to 10 mL of the *Sample solution*.

**Acceptance criteria:** A yellow color indicates the absence of soluble starch.

• **Total Solids**

**Analysis:** To determine the water content, proceed as directed in *Water Determination ⟨921⟩, Method Ia*, except use a weighed amount of Corn Syrup Solids, $W_U$, for the *Test Preparation*. In *Standardization of the Reagent*, proceed as directed, except use the formula for significant amounts of water (1% or more). [NOTE—Pure methanol can make the detector overly sensitive, particularly at low ppm levels of water, causing it to deflect to dryness and slowly recover with each addition of reagent. This slows down the titration and may allow the system to actually pick up ambient moisture during the resulting long titration. Adding chloroform or a similar nonconducting solvent will retard this sensitivity and can improve the analysis.] In the *Procedure* calculating the water content in the *Test Preparation*, use $W_W = S \times F$.

Calculate the percentage of total solids in the portion of the *Test Preparation* taken:

$$\text{Result} = (W_U - W_W)/W_U \times 100$$

$W_U$ = weight of Corn Syrup Solids for the *Test Preparation* (mg)

$W_W$ = weight of water determined (mg)

**Acceptance criteria:** NLT 90.0% when the reducing sugar content is NLT 88.0%; NLT 93.0% when the reducing sugar content is 20.0%–88.0%

## ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tightly closed containers, and store in a cool, dry place.

• **Labeling:** 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 µg/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 SOLIDS | Documentary Standards Support | CE2020 Complex Excipients |

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