Dry 2.5 g test portion, 920.175(a) (see 44.1.01), in flat dish (Ni, Pt, or Al with tight-fit cover), 2 h at 75°C (preferably 60°C), under pressure ≤ 50 mm Hg (6.7 kPa). Blot excess with air-tight cover through anhydrous CaSO₄, P₂O₅, or other efficient desiccant) during drying to remove H₂O vapor. Remove dish from oven, cover, cool in desiccator, and weigh. Redry 1 h and repeat process until change in weight between successive dryings at 1 h intervals is ≤ 2 mg.

D. Drying at Atmospheric Pressure

---Procedure 1980---

(Applicable to cane and beet, raw and refined sugars.)

Dry ca 5 g test portion, 920.175(a) (see 44.1.01), in flat dish (Ni, Pt, or Al with tight-fit cover), 3 h at 100°C. Remove dish from oven, cool in desiccator, and weigh. Redry 1 h and repeat process until change in weight between successive dryings at 1 h intervals is ≤ 2 mg. For large-grain sugars, increase temperature to 105–110°C in final heating period to expel last traces of occluded H₂O. Report loss in weight as H₂O.

C. Drying on Punicea Stone

---Surplus 1989---

(Applicable to massessures, molasses, and other liquid and semiliquid products.)

See 31.007, 14th Ed.

D. Drying on Quartz Sand

---Final Action---

(Applicable to massessures, molasses, and other liquid and semiliquid products.)

Digest pure quartz sand that passes No. 40 but not No. 60 sieve with 1 M HCl, wash acid-free, dry, and ignite. Preserve in stoppered bottle. Place 25–30 g prepared sand and short stirring rod in dish ca 55 mm diameter and 40 mm deep, fitted with cover. Dry thoroughly, cover dish, cool in desiccator, and weigh immediately. Add enough diluted product of known weight to yield ca 1 g dry matter and mix thoroughly with sand. Heat on steam bath 15–20 min, stirring at 2–3 mm intervals, or until mass becomes too stiff to manipulate readily. Dry at ≤ 70°C (preferably 60°C), under pressure ≤ 50 mm Hg (6.7 kPa), blending with dry air as in A. Make trial weighings at 2 h intervals toward end of drying period (ca 18 h) until change in weight is ≤ 2 mg.

For materials containing no fructose or other readily decomposable substance, dry 8–10 h at atmospheric pressure in oven at 100°C, cool in desiccator, and weigh, repeating heating and weighing until loss in 1 h heating is ≤ 2 mg. Report loss in weight as H₂O.

As dry sand, as well as dried sample, absorbs appreciable moisture on standing over most desiccating agents, make all weighings as quickly as possible after cooling in desiccator.

Reference: AOAC 8, 255(1925).

44.1.04

AOAC Official Method 920.14

Solids in Syrups

First Action 1932

Final Action

A. By Means of Spindle

(Accurate only when applied to pure sucrose solutions, and extensively used for approximate results with liquid sugar products containing invert sugar and other nonsucrose solids.)

(a) Direct.—Density of jellies, syrups, etc., is conveniently determined with Brix or Baumé refractometers, preferably with arc scale graduations agreeing closely with percent total solids. Table for comparison of degrees Brix (percent by weight of pure sucrose in pure solution), degrees Baumé (modulus 145), specific gravity at 20°C, and specific gravity at 20/20°C is given in 942.33 (see Appendix C).

Use Brix spindle graduated in tenths of 1 and of appropriate range, and cylinder of sufficient diameter (12 mm larger than spindle bulb) to permit spindle to come to rest without touching sides. Solution should be at room temperature. If this varies > 1°C from temperature at which spindle was graduated (20°C), apply correction according to 900.03 (see Appendix C). Before taking reading, let solution stand in cylinder until all air bubbles escape and all fatty or waxy materials come to top and are skimmed off. (Air bubbles may be conveniently removed by applying vacuum to cylinder by means of tube passing through stopper inserted in top of cylinder.) Lower spindle slowly into syrup, do not let syrup on spindle reach above syrup level.

(b) Double dilution.—If syrup is too dense to determine density directly, dilute weighed portion with weighed amount of H₂O to, or divide weighed portion and dilute to known volume with H₂O. In first instance, percent total solids is calculated by following formula:

\[
\text{Solids in undiluted material, } % = \frac{W_S}{W}
\]

where \( S = \) percent solids in undiluted material; \( W = \) weight diluted material; and \( w = \) weight syrup taken for dilution.

When dilution is made to definite volume, use following formula:

\[
\text{Solids in undiluted material, } % = \frac{VD_S}{W}
\]

where \( V = \) volume diluted solution at given temperature; \( D = \) specific gravity of diluted solution at same temperature; \( S = \) percent solids in diluted solution at same temperature; and \( w = \) weight syrup taken for dilution.

Calculation is simplified by mixing equal weights sugar product and H₂O, and multiplying Brix of solution by 2.

B. By Means of Pycometer

(a) Specific gravity (in vacuo or in air).—Determine specific gravity of solution at 20/20°C, 20/20°C vacuo, or 20/20°C in air in 945.06C (see 26.1.06), using either pycometers described in 945.06A(b) (see 26.1.06) or other suitable type. Apply air buoyancy correction to specific gravity in air and determine percent by weight of solids as sucrose from appropriate table, 942.33 (see Appendix C) or 962.37 (see Appendix C). When density of substance is too high for direct determination, dilute and then calculate sucrose content of original material as in A(b).

(b) Specific gravity of molasses.—Use special calibrated 100 mL volumetric flask with neck ca 8 mm id. Weigh empty flask and then fill with molasses, using long stem funnel reaching below graduation mark, until level of molasses is 1–2 mm lower than neck of flask. (Flow of molasses may be stopped by inserting glass rod of suitable size into funnel so as to close stem opening.) Carefully remove funnel to prevent molasses from coming in contact with neck, and weigh flask and molasses. Add H₂O almost to graduation mark, run off down side of neck to prevent mixing with molasses. Let stand several hr overnight for bubbles to escape. Place flask in constant temperature bath, preferably at 20°C, and leave until it reaches bath temperature.