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 <70°C (preferably 60°C), under pressure ≤50 mm Hg (6.7 kPa). Bleed oven with current of air (dried by passing through anhydrous $CaSO_4$, P_2O_5, or other efficient desiccant) during drying to remove H_2O 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.

B. Drying at Atmospheric Pressure —Procedure 1960

(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, 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. For large-grain sugars, increase temperature to 105–110°C in final heating periods to expel last traces of occluded H_2O. Report loss in weight as H_2O.

C. Drying on Pumice Stone —Final Action —Surplus 1989*

(Applicable to massecuites, molasses, and other liquid and semi-liquid products.)

See **31.007**, 14th Ed.

D. Drying on Quartz Sand —Final Action

(Applicable to massecuites, molasses, and other liquid and semi-liquid products.)

Digest pure quartz sand that passes No. 40 but not No. 60 sieve with 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 min 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), bleeding 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 H_2O.

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: *JAOAC* **8**, 255(1925).

44.1.04

AOAC Official Method 932.14
Solids in Syrups
First Action 1932
Final Action

A. By Means of Spindle

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

(a) *Direct.*—Density of juices, syrups, etc., is conveniently determined with Brix or Baumé hydrometer, preferably former as scale graduations agree closely with percent total solids. Table for comparison of degrees Brix (percent by weight of pure sucrose in pure solutions), degrees Baumé (modulus 145), specific gravity at 20/4°C, and specific gravity at 20/20°C is given in **942.33** (*see* Appendix C).

Use Brix spindle graduated in tenths 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 H_2O, or dissolve weighed portion and dilute to known volume with H_2O. In first instance, percent total solids is calculated by following formula:

$$\text{Solids in undiluted material, } \% = \frac{WS}{w}$$

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

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

$$\text{Solids in undiluted material, } \% = \frac{VDS}{w}$$

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 H_2O, and multiplying Brix of solution by 2.

B. By Means of Pycnometer

(a) *Specific gravity (in vacuo or in air).*—Determine specific gravity of solution at 20/4°C, 20/20°C in vacuo, or 20/20°C in air as in **945.06C** (*see* 26.1.06), using either pycnometers 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 up to lower end of 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 H_2O almost to graduation mark, running it down side of neck to prevent mixing with molasses. Let stand several h or overnight for bubbles to escape. Place flask in constant temperature H_2O bath, preferably at 20°C, and leave until it reaches bath temperature.