

44.1.15

AOAC Official Method 923.09 Invert Sugar in Sugars and Syrups Lane-Eynon General Volumetric Method First Action 1923 Final Action

A. Reagents

Soxhlet modification of Fehling solution.—Prepare by mixing equal volumes of (a) and (b) immediately before use.

(a) *Copper sulfate solution.*—Dissolve 34.639 g $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in H_2O , dilute to 500 mL, and filter through glass wool or paper. Determine Cu content of solution [preferably by electrolysis, 929.09F (see 44.1.17)] and so adjust that it contains 440.9 mg Cu/25 mL.

(b) *Alkaline tartrate solution.*—Dissolve 173 g potassium sodium tartrate $4\text{H}_2\text{O}$ (Rochelle salt) and 50 g NaOH in H_2O , dilute to 500 mL, let stand 2 days, and filter through prepared Fiberglas or porous (0.45 μm) filter with Fiberglas prefilter.

(c) *Invert sugar standard solution.*—1%. To solution of 9.5 g pure sucrose, add 5 mL HCl and dilute with H_2O to ca 100 mL. Store several days at room temperature (ca 7 days at 12–15 °C or 3 days at 20–25 °C); then dilute to 1 L. (Acidified 1% invert sugar solution is stable for several months.) Neutralize aliquot with ca 1M NaOH and dilute to desired concentration immediately before use.

B. Standardization

Accurately pipet 10 or 25 mL mixed Soxhlet reagent, or 5 or 12.5 mL each of Soxhlet solutions, A(a) and (b), into 300–400 mL Erlenmeyer. (Amount of Cu taken differs slightly between 2 methods of pipetting, and method used must be consistent in standardization and determination.) Prepare standard solution of pure sugar of such concentration that >15 mL and <50 mL is required to reduce all the Cu. Dispense from buret with offset tip to keep tube out of steam. To determine mg sugar required to completely reduce Cu at different concentrations, consult Table 930.44 (see Appendix C) and Table 930.45 (see Appendix C). Add sugar solution within 0.5–1.0 mL of total required, heat cold mixture to bp on wire gauze over burner, and maintain moderate boiling 2

min (coarse grains of C or other suitable inert material may be used to prevent bumping). Without removing flame add 1 mL 0.2% aqueous methylene blue solution (or 3–4 drops 1% solution) and complete titration within total boiling time of ca 3 min by small additions (2–3 drops) of sugar solution to decoloration of indicator. (Maintain continuous evolution of steam to prevent reoxidation of Cu or indicator.) After complete reduction of Cu, methylene blue is reduced to colorless compound and solution resumes orange Cu_2O color which it had before addition of indicator.

Multiply titer by mg/mL standard solution to obtain total sugar required to reduce the Cu. Compare with tabulated value in Table 930.44 (see Appendix C) or Table 930.45 (see Appendix C) to determine correction, if any, to be applied to table. Small deviations from tabulated values may arise from variations in technique or composition of reagents. If only approximate results (within 1%) are required, standardization may be omitted, provided specifications of analysis are rigidly observed.

C. Determination

(a) *Incremental method.*—If approximate concentration of sugar in test sample is unknown, proceed by incremental method of titration. To 10 or 25 mL mixed Soxhlet solution, add 15 mL sugar solution and heat to bp over wire gauze. Boil ca 15 s and rapidly add further amounts of sugar solution until only faintest perceptible blue remains. Then add 1 mL 0.2% aqueous methylene blue solution (or 3–4 drops 1% solution) and complete titration by adding sugar solution dropwise. (Error resulting from this titration will generally be 1%.)

(b) *Standard method.*—For higher precision repeat titration, adding almost entire sugar solution required to reduce all Cu and proceed as in B. From Table 930.44 (see Appendix C) or Table 930.45 (see Appendix C) find total reducing sugar corresponding to titer and apply correction previously determined. Calculate as follows: total reducing sugar required = $100/\text{titer} = \text{mg sugar in } 100 \text{ mL}$.

References: *J. Soc. Chem. Ind.* **42**, 32T(1923).

JAOAC **9**, 35(1926); **12**, 38(1929).

CAS-8013-17-0 (invert sugar)