44.5.14 - Sugars and Sugar Products / Maple, Sap, Maple Syrup, Maple Syrup Products

AOAC Official Method 964.21
Formaldehyde in Maple Syrup

Spectrophotometric Method
First Action 1964
Final Action 1965

(Not suitable for beet or cane sugars.)

A. Apparatus

(**a**) *Distillation apparatus.*—30 mL micro Kjeldahl flask, with standard taper 19/38 joint and 10 cm H_2O-cooled West condenser with standard taper 19/38 inner joint bent at 90° angle. Insulate condenser from standard taper joint to condenser H_2O jacket. Install adjustable flask heater equipped with conical porcelain coil element and hand-controllable rheostat placed on underside (Cenco, Inc., 2600 Kostner Ave, Chicago, IL 60623, USA, No. 16530-1, or equivalent).

(**b**) *Spectrophotometer.*—Suitable for measuring *A* at 415 nm; with matched 1 cm cells or matched test tubes.

B. Reagents

(**a**) *Nash's Reagent "B".*—Dissolve 150 g NH_4CH_3COO, 3 mL CH_3COOH, and 2 mL acetylacetone in 200-300 mL H_2O in 1 L flask and dilute to volume.

(**b**) *Formaldehyde.*—Approximately 37% by weight. Assay by **898.01B** (*see* 7.8.04).

C. Preparation of Standard Solutions

(**a**) *Stock solution.*—1000 µg/g. Weigh 5.35 g 37.4% CH_2O solution (for other concentrates, g CH_2O solution required = 200/% CH_2O) into 2 L volumetric flask containing H_2O and dilute to volume with H_2O.

(**b**) *Solution B.*—50 µg/g . Pipet 10 mL stock solution into 200 mL volumetric flask and dilute to volume with H_2O.

(**c**) *Solution C.*—100 µg/g. Pipet 10 mL stock solution into 100 mL volumetric flask and dilute to volume with H_2O.

(**d**) *Solution D.*—200 µg/g. Pipet 10 mL stock solution into 50 mL volumetric flask and dilute to volume with H_2O.

(**e**) *Formaldehyde standard solutions.*—Prepare 1, 2, and 4 µg/g standard solutions by pipetting 10 mL *Solutions B, C,* and *D,* respectively, into 500 g syrup and stirring mechanically 15 min.

D. Determination

Weigh 20 ± 0.20 g syrup into tared 30 mL micro Kjeldahl flask and insulate flask neck with asbestos cord as in **A**(**a**) from base of standard taper joint down to body of flask. (This insulation is temporary; repeat after each washing and weighing.) Add 2 drops antifoam agent, and connect West condenser. Mount apparatus, adjusting slope of condenser at 45° angle, with flask bottom centered in conical cavity and at such height that syrup level in flask is even with top plate of heater, without touching hot element at any point; *see* Figure **964.21**. Heat flask with heater, previously adjusted with control knob in such position that exactly 3 mL H_2O is distilled from syrup in 12-14 min. Collect 3 mL distillate in 5 mL graduate with funnel top. Using transfer pipets, place 1 mL distillate in 13 mm id test tube, and add 1 mL H_2O and 2 mL Nash's reagent. Heat 30 min in H_2O bath at 37 ± 1°C to develop color. Transfer colored solution to 1 cm cell and measure *A* at 415 nm against H_2O.

E. Blanks

To determine *A* due to reagents, substitute 1 mL H_2O (from same source as used in determination) for 1 mL test distillate. Subtract *A* of blank from that for sample to obtain *A* due to CH_2O. Or, as simpler operation, measure *A* of distillate with instrument adjusted to 0 *A* for blank.

Obtain concentration of CH_2O in syrup from *A*, using standard curve.

F. Preparation of Standard Curve

Construct standard curve by plotting *A* obtained for syrups containing 1, 2, and 4 ppm CH_2O against concentration of added CH_2O in ppm.

Straight line relationship is obtained for standard curve. Project this line to *Y* axis (*A*); *Y* intercept indicates blank for syrup. Since syrup used to construct curve from *A* values may be atypical, draw and use parallel curve with 0 intercept. Correct ppm values obtained from this curve for average syrup blank (ca 0.9 ppm).

References:
JAOAC **47,** 548(1964); **56,** 132(1973).

CAS-50-00-0 (formaldehyde)