42.1.15

AOAC Official Method 971.27
Sodium Chloride in Canned Vegetables

Method III
(Potentiometric Method)
First Action 1971
Final Action 1976

A. Principle

Product is dispersed with H_2O and acidified; soluble chlorides are titrated potentiometrically with $AgNO_3$. Applicable to levels ≥0.03% NaCl. For convenience in calculations, weights or volumes and molarity are specified so that 1 mL $AgNO_3$ = 0.1% NaCl. If balance permitting rapid weighing of specified weight is not available, convenient weight test sample and molarity $AgNO_3$ solution may be used.

B. Apparatus

(**a**) *Balance.*—Capacity, ≥200 g, taring range, ≥100 g, readability, ≤0.01 g. Mettler No. P1200 (replacement Model PM2000) (Mettler-Toledo GmbH, PO Box VI-400, Sales International, CH-B606 Greifenseel, Switzerland), or equivalent, is convenient.

(**b**) *Electrodes.*—Ag billet combination electrode (Beckman No. 39261, or equivalent), or separate indicating Ag (Beckman 39261, Orion 94-17 [replaced by 94-17-BN], Fisher 13-639-122, or equivalent), and glass reference (Beckman 39419, Orion 90-02 [replaced by 90-02-00], Fisher 9-313-216, or equivalent) electrodes. Before initial use and before each day's use, if necessary, clean Ag billet electrode tip with scouring powder or other suitable material and rinse thoroughly with H_2O. (Hot H_2O may be required with some kinds of laboratory samples.) Clean other electrodes as recommended by manufacturer. Reclean as frequently as necessary to prevent drifting of end point reading. With some test samples, periodically rinse electrodes with H_2O and wipe with tissue to prevent accumulation of film. It is unnecessary to coat Ag billet electrodes with AgCl.

(**c**) *Magnetic stirrer.*—Operating through variable transformer to permit range of speed which, once set, is constant.

(**d**) *pH meter.*—Preferably direct reading, with scale divisions 1 mV or less; range at least ±700 mV, e.g., digital type.

C. Reagents

(**a**) *Nitric acid, dilute.*—1 + 49. Dilute 20 mL HNO_3 to 1 L with H_2O.

(**b**) *Silver nitrate standard solution.*—0.0856M. Dissolve 14.541 g $AgNO_3$ in H_2O and dilute to 1 L in volumetric flask. Standardize as in **D**, and adjust to exact molarity specified so that with indicated test sample weight, 1 mL = 0.1% NaCl. Store in Pyrex container out of direct sunlight. Solution is stable in room light.

(**c**) *Sodium chloride standard solution.*—0.0856M. Dissolve in H_2O 5.000 g NaCl (if assay is <100.0% NaCl, divide 5.000 g by % NaCl/100 to obtain corrected weight), previously dried 2 h at 110°C, and dilute to 1 L in volumetric flask.

(**d**) *Water.*—Distilled or deionized, halogen-free by following test: Add 1 mL ca 0.1M $AgNO_3$ and 5 mL HNO_3 (1 + 4) to 100 mL of the H_2O. No more than slight turbidity should be produced.

D. Standardization

Pipet 25 mL NaCl standard solution into 250 mL beaker, dilute to ca 50 mL with H_2O, and add 50 mL HNO_3 (1 + 49). Insert electrodes, start magnetic stirrer, and stir throughout titration at constant rate producing vigorous agitation without splashing. Titrate with $AgNO_3$ standard solution, adjusting increments with rate of voltage change so that accurate plot of mV against mL $AgNO_3$ solution can be prepared. Add total of 50 mL $AgNO_3$ solution to obtain complete curve.

Determine inflection point by drawing 2 straight lines with 45° slope with respect to axes and tangent to titration curve at the 2 points of greatest curvature. Inflection point is at intersection of titration curve with line drawn parallel to, and midway between, other 2 lines. From volume $AgNO_3$ solution used, calculate molarity and adjust to 0.0856M. Restandardize occasionally. Use inflection point as end point in titrating test portions. Recheck end point potential occasionally, and redetermine when either individual electrode, combination electrode, or pH meter is replaced by preparing new titration curve.

For greatest accuracy, when series of determinations on same food is performed, determine and use end point from titration curve of that food rather than using end point obtained with NaCl standard solution.

E. Preparation of Sample

(**a**) *Clear liquids with low viscosity.*—(Fruit juices, clear soups, wines, etc.) Use directly.

(**b**) *Comminuted products.*—(Tomato juice, tomato catsup, strained vegetables, etc.) Thoroughly shake unopened container to incorporate any sediment. Transfer entire contents to large glass or porcelain dish and mix thoroughly, continuing stirring ≥1 min. Transfer to glass-stoppered container, and shake or stir thoroughly each time before removing test portions for analysis.

(**c**) *General method for heterogeneous (fish, meat, etc.), low moisture (cereal products, etc.), and hard-to-disperse, homogeneous (cheese, peanut butter, etc.) foods.*—Weigh 50.0 g test sample into 1 L (qt) container of high-speed blender and add 450 g H_2O. Cover, start blender at low speed by use of variable transformer for initial dispersion, and blend thoroughly at high speed (1–2 min is usually adequate). Equivalent of 5 g test sample is conveniently dispensed through 50 mL pipet with tip cut off. Thoroughly mix test sample suspension immediately before pipetting aliquot for analysis so that solid material is uniformly suspended.

(**d**) *Other types of foods.*—Prepare test sample by method (**a**), (**b**), (**c**), or other suitable method.

To preserve test samples or sample suspensions for future analysis, add 0.5 mL ca 37% HCHO solution/100 g sample or sample suspension, mix well, and store at room temperature. Correct for dilution by HCHO solution by multiplying % NaCl by 1.005.

F. Determination

(**a**) *For products containing less than 5 per cent salt.*—Place 5.00 g (or 5.00 mL if concentration is to be expressed on weight/volume basis) prepared test sample from (**a**) or (**b**) or 50.0 g from (**c**) into tared 250 mL beaker; add H_2O to ca 50 mL if (**a**) or (**b**) is used. (Use boiling H_2O with samples such as butter to melt fat.) Add 50 mL HNO_3 (1 + 49). Titrate as in **D**, using 10 mL buret if salt content is ≤1%.

$$\text{NaCl, \%} = \text{mL 0.0856M } AgNO_3/10$$

(**b**) *For products containing 5 or more per cent salt.*—Place 5.00 g (or 5.00 mL if concentration is to be expressed on w/v basis) prepared test sample from (**a**) or (**b**) into 100 mL volumetric flask and dilute to volume with H_2O. Mix, and transfer aliquot containing

50–250 mg NaCl to 250 mL beaker. If test sample is prepared by (**c**), transfer weighed aliquot containing 50–250 mg NaCl to tared 250 mL beaker. Proceed as in **D**, beginning "...dilute to ca 50 mL with H_2O,...".

$$\text{NaCl, \%} = F \times \text{mL 0.0856M } AgNO_3/10$$

where F = dilution factor = 100/mL aliquot titrated if test sample is prepared by (**a**) or (**b**) or 50/g aliquot titrated if prepared by (**c**).

(**c**) *General case.*—Accurately weigh approximately test sample weight stated. (If % NaCl ≥5%, weigh <5 g sample rather than diluting to 100 mL, if more convenient.) Use ca 0.1M $AgNO_3$ solution, accurately standardized as in **D**, without adjusting to specific molarity, and titrate as in **D**.

$$\text{NaCl, \%} =$$
$$\text{mL } AgNO_3 \times \text{M } AgNO_3 \times 0.05844 \times 100/\text{g test sample}$$

If test sample is overtitrated, add NaCl standard solution, and complete titration. Correct for volume of standard solution added.

References: *JAOAC* **54,** 471(1971); **57,** 1209(1974).

CAS-7647-14-5 (sodium chloride)