Application Note 014

Measuring Salt Content in Canned Vegetables Using Manual Titration

Water Analysis Instruments, Thermo Fisher Scientific

Key Words

Salt analysis, manual titration, canned vegetables, food analysis, titration of chloride.

Goal

The following application note describes how to reliably measure the salt content in canned vegetables by manual titration to mV endpoint using a Thermo Scientific™ Orion™ Chloride Ion Selective Electrode (ISE) or a Thermo Scientific Orion Silver Billet Electrode.

Introduction

Salt analysis is a routine task in food analysis, and titration of chloride (Cl^-) is a common technique to determine the sodium chloride (NaCl)/salt content in food. In this note, Cl^- in ground peeled tomatoes is manually titrated by silver nitrate ($AgNO_3$) to a fixed millivolt reading on a Thermo Scientific Orion chloride ion selective electrode (ISE) or by a Thermo Scientific Orion Silver Billet electrode. This method produces results comparable to autotitration or ion chromatography (IC) methods, yet is simple and inexpensive.

Recommended Equipment

- Thermo Scientific™ Orion Star™ A211 pH meter, Star A214 pH/ISE meter or equivalent
- Orion 9617BNWP Chloride ISE or Orion 9780SC Silver Billet electrode with 91CBNC cable
- Orion Stirrer Probe (Cat. No. 096019)
- Swing Arm Stand (Cat. No. 090043)
- Electrode Polishing Strip (Cat. No. 948201)
- 10 mL burette, burette clamp, ring stand

Required Reagents and Solutions

- 0.1M Silver Nitrate Solution
- 0.1M NaCl (sodium chloride) Standard (Cat. No. 941706)
- Optimum Result B Filling Solution (Cat. No. 900062) for chloride ion selective electrode; or, silver chloride pH filling solution (Cat No. 900011) for silver billet electrode
- Ionic Strength Adjuster (ISA) (Cat. No. 940011)
- Deionized water (DI) of conductivity <2 µS/cm

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Solutions Preparation

Prepare or purchase $AgNO_3$ titrant solution of 0.1M. To prepare: weigh approximately 8.5g $AgNO_3$ and dissolve in 500 mL DI. Store in a brown or opaque bottle. Standardize as indicated below.

Meter Setup

Connect Orion electrode and the stirrer probe to the meter. In Setup, select the mV mode, set read type to continuous, and set the stirrer speed to 3. Refer to the *Orion meter user guide* for more details.

Electrode setup, storage, soaking, and rinsing

See the electrode user guide or instruction sheet.

Electrode Performance Check

See *Standardization of Titrant* section, below. Alternately, analyze a QC sample.

Titration setup

Secure the burette on the clamp. Fill the burette with $AgNO_3$ solution (titrant) and adjust the level to the zero mark.

Standardization of Titrant

Add 3.0 mL of 0.1M NaCl standard, 50 mL of DI, and 1 mL ISA to a 100 mL beaker. Rinse the electrode and stirrer with DI. Immerse the electrode and stirrer at least one inch below the liquid level in the beaker. Tap to release bubbles trapped on the surface of the electrode. Turn on the stirrer.

Adding $AgNO_3$ titrant from the burette, titrate at moderate rate until the mV reading is near 240 mV. (Note: mV reading will start low and increase as titrant is added). Allow time for the reading to stabilize before adding more titrant. As endpoint is approached, the reading will take longer to stabilize. Start to add titrant slowly until the reading just exceeds 260 mV (or 290 mV for silver billet). This is the endpoint of the titration. See an example titration curve on the following page. Record the volume of titrant used (V_t). Repeat the standardization procedure if required by user's protocol. Calculate the titrant concentration (T) as indicated on the following page.

Sample Preparation and Preservation

Liquid samples: Measure with a pipet (mL) or with a scale (gram, g). Record exact amount. Transfer to a 150 mL beaker and add DI water to the 50 mL mark on the beaker. Add 1 mL ISA.

Solid samples: Weigh about 50 g of sample. Record exact weight to 0.01 g. Place into blender or food processor. Add 450 mL of DI water. Blend until smooth and liquid. Using a wide-mouth pipet or graduated cylinder, transfer an amount of the well-mixed sample to a 150 mL beaker. Add DI water to 100 mL mark on the beaker. Add 2 mL ISA.

% salt	g or mL liquid food	mL of pureed solid food
0.1	12.5	125
0.25	5.0	50
0.50	5.0	50
0.75	20*	20
1.0	20*	20
5.0	4*	4
10.0	2*	2

*Prepare like for a solid sample (50g sample plus 450 mL DI), then use this volume of prepared sample.

Sample titration

Prepare samples as detailed above. Prepare one to three replicates of each sample as desired. Titrate the sample, as described above. Record the volume of titrant used (V_s). Calculate the salt (sodium chloride) content of the sample using the equations on the following page. After each titration, rinse the electrode and stirrer with DI, wipe with a lint-free wiper, and rinse again with DI. Tap to remove excess water drops.

Quality Control (QC)

Recommended QC procedures may include: titrant standardization, analysis of chloride standard or QC sample, duplicates, matrix spike, and/or blank titration.

Accuracy can be improved by performing a blank titration. Place 50 mL of DI into a 150 mL beaker. (If solid samples are titrated, use 100 mL of DI). Add 1 mL ISA for each 50 mL DI. Titrate. Subtract the volume required to reach endpoint in the blank titration (Vb) from the sample titration volume. See *Calculations* on the following page.

Results

Salt Analysis in Ground Peeled Tomatoes by Manual Titration

Sample	Weight (g)	Endpoint Volume* (mL)	ppm Salt (sodium chloride) in sample	% Salt (sodium chloride) in sample	Sodium (mg per 66 g serving)
Sample 1	4.33	3.83	5060	0.506 %	131
Sample 2	4.15	3.68	5070	0.507 %	132
Sample 3	4.10	3.68	5130	0.513 %	133
* The endpoint volume of the blank (0.10 mL) has been subtracted		Mean	5090	0.509 %	132
		RSD	0.8 %		
		Spike Sample Recovery	97.2 %		

Raw Data

Comparison of Manual Titration with Other Methods

Method	ppm Salt (sodium chloride) in sample	% Salt (sodium chloride) in sample	Sodium per serving (mg per 66 g serving)
Manual Titration	5090	0.509 %	132
Autotitration	5020	0.502 %	130
Ion Chromatography	4860	0.486 %	126
		RSD	2.4 %

Calculations

1. **Titrant concentration (T)**

   $T = V_{Cl} \times M_{Cl} \div V_t$

   Example: 3.00 mL × 0.100 M ÷ 3.06 mL = 0.0980 M titrant

   T = concentration of silver nitrate titrant (M, mol/L)
   V_{Cl} = volume of sodium chloride standard (mL)
   M_{Cl} = molarity of the sodium chloride standard (M, mol/L)
   V_t = volume of titrant used at the end point for titrant standardization (mL)

2. **% Salt (sodium chloride) concentration in the sample (C%) as % NaCl***

   $C\% = 5.844 \times T \times (V_s - V_b) \div (W \times DF)$

   Example: 5.884 × 0.0980M × (3.93 – 0.10 mL) ÷ (51.5 g × 0.103) = 0.414% salt

   C% = concentration of sodium chloride in the sample (%)
   V_s = volume of titrant used at the end point for sample titration (mL)
   V_b = volume of titrant used at the end point for blank titration (mL), if blank titration performed.
   W = weight of sample (g)
   DF = dilution factor; mL of sample titrated ÷ total mL of pureed sample, e.g. 51.5 g sample ÷ (450 mL + 51.5) = 0.103.

3. **ppm Salt (sodium chloride) concentration in the sample (C_{ppm}) as ppm NaCl***

   $C_{ppm} = 58440 \times T \times (V_s - V_b) \div (W \text{ or } V \times DF)$

   Example: 58440 × 0.0980M × (3.93 – 0.10 mL) ÷ 5.30 mL = 4140 ppm or mg/L salt

   C_{ppm} = concentration of salt (sodium chloride) in the sample (ppm, mg/kg, or mg/L)
   V = volume of sample titrated

4. **Sodium* per serving ($C_{serving}$) as mg sodium per serving of food**

   $C_{serving} = C_{ppm} \times 0.3934 \times S \div 1000$

   Example: 4140 ppm × 0.3934 × 66 mL ÷ 1000 = 107 mg Na per 66 mL serving

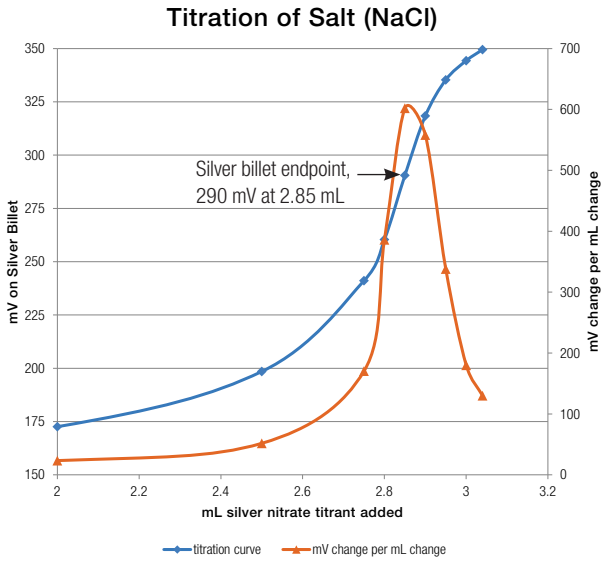
   Or, $C_{serving} = C\% \times 3.934 \times S$

   Example: 0.414% × 3.934 × 66 g = 107 mg Na per 66g serving

   $C_{serving}$ = sodium per serving of food (mg per serving of food)
   S = weight or volume of one serving (g or mL)

*assumes that the sodium and chloride in the sample come from salt, NaCl

Example of a Titration Curve for Salt by Silver Billet Electrode



mL Titrant	mV	mV change per mL change
2.00	172.6	
2.50	198.5	51.8
2.75	241.1	170.4
2.80	260.4	386.0
2.85	290.5	602.0
2.90	318.4	558.0
3.00	344.3	180.0
3.04	349.5	130.0

References

1. Sodium Chloride in Canned Vegetables. AOAC Official Method 971.27 – 1976. www.aoac.org

2. EPRI FGD chemistry and analytical methods handbook rev 1, volume 2, Method 4, O4-1—O4-27. www.epri.com

Visit www.thermoscientific.com/water for additional information on Thermo Scientific Orion products, including laboratory and field meters, sensors and solutions for pH, ion concentration (ISE), conductivity and dissolved oxygen analysis plus spectrophotometry, colorimetry and turbidity products.

To purchase an Orion chloride electrode, ISE meter and other related products, please contact your local equipment distributor and reference the part numbers listed below.

Product	Description	Part Number
Meters	Thermo Scientific Orion Star A214 pH/ISE Benchtop Meter	STARA2140
	Thermo Scientific Orion Star A211 pH Benchtop Meter	STARA2110
	Thermo Scientific Orion VERSA STAR pH/ISE Benchtop Meter	VSTAR40A
Electrodes	Thermo Scientific Orion Chloride Ion Selective Electrode	9617BNWP
	Thermo Scientific Orion Silver Billet Electrode	9780SC
Solutions	Thermo Scientific Orion NaCl Standard, 0.1M	941706
	Thermo Scientific Orion Optimum Results B Filling Solution	900062
	Thermo Scientific Orion Electrode Filling Solution	900011
	Thermo Scientific Orion ISE Ionic Strength Adjuster (ISA)	940011
Accessories	Thermo Scientific Orion Stirrer Probe	096019
	Thermo Scientific Orion Swing Arm Stand	090043
	Thermo Scientific Orion Electrode Polishing Strip	948201

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