

Determination of Sodium in Canned Vegetables by Automatic Multiple Known Addition Titration

Introduction

Sodium in canned vegetables is determined using the preprogrammed method, Na Veg. A sodium ion selective electrode (ISE) is used to sense the potential due to sodium concentration and determine the amount in the sample. Small amounts of a sodium standard are added to the sample, and the sample concentration is determined from the changes in potential after each addition. This analytical technique is known as the multiple known addition (MKA) or known addition procedure (KAP). MKA can be used to enhance measurements usually performed by direct calibration, providing faster results and more conveniently. In this way, automation of non-titratable species is possible. The use of KAP analysis can also minimize errors due to matrix effects.

By the process of adding small, known amounts of standard to the sample in the beaker, MKA performs the calibration and calculates sample concentration directly in the sample matrix. Because electrode slope and offset (E0) are determined in the sample matrix during analysis, there is no need for independent calibration (and periodic recalibration). MKA may also minimize errors due to the sample matrix that can affect the direct testing method.

The Orion T900 series titrator adds the small, known additions of standards until the results calculated after each addition agree within the precision level selected by the method. The final result is reported. A spike recovery test is calculated for every sample. Along with sodium results, the determined slope of the electrode, the precision of the MKA result, and the spike recovery of the standard is calculated and reported for each test cycle.



Orion Star T930 ISE Titrator with 20 mL burette

Recommended Equipment

Thermo Scientific™ Orion Star™ T930 Ion Selective Electrode (ISE) Titrator or T940 All-in-One Titrator or equivalent with a 20 mL burette.

Thermo Scientific™ Orion™ ROSS™ 8611BNWP Sodium Ion Selective Electrode

Thermo Scientific™ Orion™ START-CP3 Orion pint bottle dispenser cap

Thermo Scientific™ Orion™ 810017 electrode storage sleeve and base

Pipette: 50 mL (or 50 mL graduated cylinder)

Graduated cylinder: 500 mL

Beakers: 100 mL; 600, 800, or 1000 mL

Squeeze wash bottle: 500 or 1000 mL

Required Solutions

MKA Standard Titrant: Thermo Scientific™ Orion™ 650700 Sodium Known Addition Kit (1M NaCl standard with ISA, and Sodium ISA), or 1000 mg/L sodium KAP standard (Orion 841109) for lower sodium levels

ISE Fill solution: Thermo Scientific™ Orion™ 900010 Sodium Electrode Fill Solution.

Sodium Electrode Reconditioning Solution: Thermo Scientific™ Orion™ 841113

Sodium Electrode Storage Solution: Thermo Scientific™ Orion™ 841101

Reagent grade water (RGW).

Use suitable Personal Protective Equipment (PPE) as recommended by the Safety Data Sheets (SDS) for the chemicals utilized during this procedure.

Electrode and Titrator Setup

First time electrode use: Before using the sodium electrode for the first time, fill the electrode with sodium electrode filling solution, and *recondition the electrode glass bulb* using the sodium reconditioning solution (Orion 841113). Follow the protocol as described in the sodium ISE user guide in the section titled Electrode Reconditioning. Use suitable PPE and observe the necessary safety precautions when using the reconditioning solution. *After reconditioning, soak* in sodium electrode storage solution for two hours before the first use.

Electrode storage sleeve: Remove the red cap with the o-ring from the Orion 810017 electrode storage sleeve. It will not be needed for routine storage. Insert the sleeve into the base. Place about 10 mL of Orion 841101 sodium electrode storage solution into the sleeve. Insert the sodium ISE into the sleeve for soaking and storage. The glass bulb and Sure-Flow should be submerged in the solution.

Titrator Setup: Connect the electrode and the stirrer probe to the titrator. If not previously done, import the Na Veg preprogrammed method into the titrator from the “Methods screen”. Open the sodium KAP analysis standard bottle (Orion 650700) and put on the Orion pint bottle dispenser cap (Orion START-CP3). Rinse and fill the burette with titrant. See the titrator user manual for details on setting up the titrator. Note: Do not use the sodium known addition standard (Orion 841109) that comes with the sodium electrode unless testing for low level sodium as described later in this application note.

If bubbles are visible in the tubing, dispense titrant (from the “Burette” screen) until the bubbles have been expelled. Tap the tubing to help dislodge bubbles.

Table 1

T9 Na Veg Preprogrammed Parameters	
Electrode	Parameter
Electrode Type	ISE-MKA
ISE Type	Sodium (Na ⁺)
Electrode Name	edit as desired
Titration	Parameter
Titration Name	Sodium (Na ⁺)
Titration ID	edit as desired
Titration Concentration	1 M
Titration	Parameter
Result Units	mg/100g
Sample MW	22.99 g/mol
Sample Amount	Variable Weight
Total Solution Volume	55 mL
Max Total Titrant Volume	20 mL
Blanc Conc. Required	No
MKA Process Control	Routine
Precision Level	2%
Prestir	10 seconds
Stir Speed	Fast
Sample ID	Manual

Solutions Preparation

Sodium Electrode Rinse Solution: For rinsing sodium electrodes between measurements: Make 1L (1000 ml) of rinse solution by adding 10 mL of sodium ISA (Orion 841111) to a 1L squeeze bottle and filling it with RGW. Alternately, make 500 mL of rinse solution by adding 5 mL of sodium ISA (Orion 841111) to a 500 mL squeeze bottle and filling it with RGW.

Daily Conditioning Solution for sodium electrode:

Place 50 mL of RGW into a 100 mL beaker and add 5 mL of sodium ISA. Prepare fresh each day.

Electrode Preparation

At the start of the day:

- Remove the electrode from storage solution. Add Orion 900010 sodium electrode fill solution up to the bottom of the fill hole. Leave the fill hole open during testing.
- Rinse thoroughly with sodium electrode rinse solution before and between titrations.
- Condition the electrode by soaking 5 minutes in 50 mL RGW mixed with 5 mL of sodium ISA.

At the end of the day:

- Thoroughly rinse the electrode with sodium electrode rinse solution and place in the storage sleeve with sodium electrode storage solution.
- Cover the fill hole when in storage.
- Place the tip of the dispenser in a small beaker of water to prevent salt from crusting on the end and clogging the dispenser.

Sample Preparation for Canned Vegetables

1. Open the can and pour the entire contents with all the liquids into a blender. Blend until homogenized.
2. Weigh 25.0 g of sample into a large beaker (e.g., 800 mL) beaker. (If the sample weight is not exactly 25.0 g, record the exact weight). Add 475 mL of RGW. The final volume in the beaker will be approximately 500 mL total.
3. Mix well for 2 minutes, then let solids settle (e.g., 5 minutes).
4. Decant the diluted sample by pouring off the top liquid layer into a new beaker, leaving the solids in the original beaker.
5. Using a graduated cylinder or 50 mL pipet, measure 50 mL of the decanted sample into a 100 mL beaker for analysis. Note, this is equivalent to 1/10th of the starting sample weight.
6. Add 5.0 mL of sodium ISA into the beaker. The sample is now ready for testing.
7. Proceed with the titration as indicated below in the Sample Titration section.
8. When prompted, enter the sample weight as 2.5 grams. (If the original sample weight was not 25.0 g, enter the sample weight as 1/10th of the weight recorded in step 2 above).

Table 2

Routine sample preparation					
Sample Concentration (mg/100g)	Titration Concentration (M)	Sample Size (g)	RGW added	mL of sample/mL of ISA	Sample weight to enter
100 - 1000	1	25.0	475 mL	50/5	2.5

Sample Titration

1. From the “Home” screen or the “Methods” screen, select option to run the saved method “Na Veg”.
2. Rinse the electrode, stirrer, and dispenser with sodium electrode rinse solution.
3. Place the electrode and stirrer into the prepared sample in the beaker. Ensure that the electrode junction is immersed.
4. Position the dispenser tip just above but not touching the surface of the sample.
5. Start the titration. Enter the sample weight and sample ID when prompted
6. Results are reported as sodium, mg /100g.
7. If desired, for higher accuracy, run 2 or more cycles and report the average concentration (as calculated and logged by the titrator).

Range

This preprogrammed titration method covers a range of about 100 to 1000 mg sodium /100g, when using 1M sodium known addition procedure (KAP) standard titrant and the prescribed preparation of sample. For higher or lower concentration samples, see the Modifications section below.

Figure 1: Sodium in Corn - cycle 3 results

Cycle Number	3	<p>Precision is the agreement between the calculated result after each known addition (in %RSD).</p> <p>The spike recovery is the calculated recovery of the first addition based on the final reported result.</p> <p>The electrode slope is determined in the beaker based on the multiple known additions of standard added.</p>
Excluded	No	
Sample Amount	2.500 gram	
Sample Concentration	142.4 mg/100g	
Precision Level	0.8 %	
Spike Recovery	100.1 %	
Electrode Slope	59.2 mV/dec	
Temperature	25.0 C	
Titration Duration (Minutes)	00:02:01	

Table 3

Results				
Sample	Average	RSD (n = 3 cycles)	Average Analysis Time	Direct ISE Result
Mixed Vegetables	218 mg/100 g	2.1 %	02:31 min	208 mg/100g
Corn, 50% less sodium	143 mg/100g	0.4%	02:01 min	137 mg/100 g
Green Beans	330 mg/100g	2.4%	02:01 min	319 mg/100g
Peas	272 mg/100g	2.3%	02:15 min	278 mg/100g

Modifications

For sample concentrations that are higher or lower than the stated range, modify the protocol as follows:

- For samples of concentrations between 1000 - 5000 mg sodium/100g, weigh 5.0 grams of sample into a large beaker and add 495 mL of RGW. Prepare sample as usual. When the titrator prompts for sample weight, enter 0.5g.
- For samples between 50 - 100 mg sodium/100g, weigh 50 grams of sample into a large beaker and add 450 mL of RGW. Prepare sample as usual. When the titrator prompts for sample weight, enter 5.0 g.
- For samples between 5 – 50 mg sodium/100g:
 - Switch titrant to the 1000 mg/L sodium KAP standard (Orion 841109) that comes with the sodium electrode.
 - Copy the Na Veg method to create a low level version. Change the titrant concentration to 0.04350M (1000 mg/L) and save. Load this method.
 - Prepare the sample as usual (as directed in the Sample Preparation section on page 3).

Table 4

Modifications for out of range samples					
Sample Conc (mg/100g)	Titant Conc (M)	Sample size (g)	RGW added mL	mL of sample/ mL of ISA	Sample weight to enter
1000-5000	1	5.0	495	50/5	0.5
100-1000	1	25	475	50/5	2.5
50-100	1	50	450	50/5	5.0
10-50	0.04350 M (1000 mg/L)	25	475	50/5	2.5
5-10	0.04350 M (1000 mg/L)	50	450	50/5	5.0

Titration and electrode care

Refer to the titrator and electrode user manuals for details on cleaning, storage, and maintenance recommendations to keep the titrator and electrode performing well. Main points for care are summarized below.



Orion Star T930 ISE Titrator with 20 mL burette

Daily care

- Dispense 1 mL of titrant to clear the dispenser.
- If bubbles are visible in the titrator tubing, dispense titrant until bubbles have been expelled. Tap tubing to dislodge bubbles that stick.
- Add Orion sodium electrode fill solution up to the bottom of the fill hole and leave the fill hole open during measurement.
- Rinse electrode well with sodium electrode rinse solution before and between titrations.
- Conditioning: Soak 5 minutes in RGW/ISA conditioning solution prior to the first MKA titration.
- Storage: Thoroughly rinse the electrode with sodium electrode rinse solution and store in storage solution. Cover the fill hole.
- Storage: Place the tip of the dispenser in a small beaker of water.

Weekly or biweekly care

- Drain and replace the fill solution of the electrode.
- Change the electrode storage solution in the electrode storage sleeve.

As needed

- For slow or drifty electrode response, recondition the sodium electrode glass bulb. Follow the instructions in the electrode user guide, Electrode Reconditioning section. Soak time can be from 30 seconds to a maximum of 5 minutes
- See the electrode user manuals for maintenance details.

Ordering Information

Product	Description	Cat. No.
Titration Kit	Orion Star T930 ion titrator sodium MKA kit with 8611BNWP ROSS sodium electrode	START9303
Titrator	Orion Star T930 Ion Titrator without electrode	START9300
	Orion Star T940 All-in-One Titrator without electrode	START9400
Electrode Only	Orion ROSS™ Sodium Ion Selective Electrode	8611BNWP
Orion Solutions	Orion sodium known addition kit (3 x 475 mL of 1M sodium standard and 1 x 475 mL ISA).	650700
	Orion sodium electrode fill solution	900010
	Orion sodium electrode reconditioning solution	841113
	Orion sodium electrode storage solution	841101
	Orion known addition standard, 1000 ppm with ISA	841109
Accessories	Orion pint bottle dispenser cap for T9000 series	START-CP3
	Orion electrode storage sleeve	810017
	Pipette: 50 mL (or 50 mL graduated cylinder)	
	Graduated cylinder: 500 mL	
	Beakers: 100 ml; 600, 800, or 1000 mL	
	Squeeze wash bottle: 500 or 1000 mL	
Reagent Grade Water	Thermo Scientific™ Barnstead™ Smart2Pure™ Pro UV Water Purification System	50129890*

* Please contact a sales representative for support on ordering the best water purification system for your application, or find out more at thermofisher.com/purewater

Find out more at thermofisher.com/titrator

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