TECHNICAL NOTE

Determination of salt content in prepared food by automatic titration

Introduction

Salt (sodium chloride) content in food is determined with a Thermo Scientific™ Orion Star™ titrator using the T6 Salt preprogrammed method. To determine salt content, the sample is titrated to the equivalence point using silver nitrate titrant [1,2]. The sample preparation and titration conditions described here are optimized to use a minimum amount of the titrant, show good precision, and finish the titration quickly.

Materials

- 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™ 9780SC Silver Billet Combination Electrode (or equivalent) with Orion cable (Cat. No. 91CBNC)
- Thermo Scientific™ Orion Star™ T900 Series Light Blocking Tubing Kit (Cat. No. START-TB2)
- Pipette: 10 mL graduated
- Beakers: 100 mL, 150 mL, and 1,000 mL

Required reagents and solutions

- Titrant: purchased or prepared silver nitrate, 0.1 M (0.1 N)
- Standardizing solution (optional): Thermo Scientific™ Orion™ Ion Selective Electrode Calibration Standard, Potassium Chloride, 0.1 M (Cat. No. 921906)
- 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.

Titrator setup

Install the Orion Star T900 Series Light Blocking Tubing Kit to help protect the integrity of the silver nitrate titrant, if desired. Connect the electrode and the stirrer probe to the titrator. If not previously done, import the T6a and T6b Salt preprogrammed methods into the titrator from the "Methods" screen. Rinse and fill the burette with titrant. See the user manual for details on setting up the titrator.

If bubbles are visible in the tubing, dispense titrant (from the "Burette" screen) until the bubbles are expelled. Tap the tubing to dislodge bubbles. Consider standardizing the titrant before titrating samples. See "Titrant" section on page 4.

Table 1

T6a Salt by weight method: preprogrammed parameters	
Electrode	**Parameter**
Electrode type	ISE
ISE type	Silver (Ag+)
Electrode name	Edit as desired
Titrant	**Parameter**
Titrant name	AgNO3
Titrant ID	Edit as desired
Conc. input mode	Standardization
Nominal concentration	0.1 M
Standardize tech	Equivalence Pt.
Number of endpoints	1
Results units	M
Standardize reaction ratio	1
Standard name	KCl
Standard amount	Fixed volume, 2 mL
Standard concentration	0.1 M
Pre-dose titrant volume	0 mL
Max. total titrant volume	5 mL
Stand. Process control	Routine
Pre-stir duration	5 sec
Stir speed	Medium
Titration	**Parameter**
Titration technique	Equivalence Pt.
Number of endpoints	1
Display units	mV
Titration type	Direct
Blank required	No
Result units	% w/w
Reaction ratio	1
Sample MW	58.44
Sample amount	Variable weight
Pre-dose titrant volume	0 mL
Max. total titrant volume	10 mL
Titration process control	Routine
Pre-stir duration	5 sec
Stir speed	Medium
Sample ID	Manual

Table 2

T6b Salt by volume method: preprogrammed parameters	
Electrode	**Parameter**
Electrode type	ISE
ISE type	Silver (Ag+)
Electrode name	Edit as desired
Titrant	**Parameter**
Titrant name	AgNO3
Titrant ID	Edit as desired
Conc. input mode	Standardization
Nominal concentration	0.1 M
Standardize tech	Equivalence Pt.
Number of endpoints	1
Results units	M
Standardize reaction ratio	1
Standard name	KCl
Standard amount	Fixed volume, 2 mL
Standard concentration	0.1 M
Pre-dose titrant volume	0 mL
Max. total titrant volume	5 mL
Stand. Process control	Routine
Pre-stir duration	5 sec
Stir speed	Medium
Titration	**Parameter**
Titration technique	Equivalence Pt.
Number of endpoints	1
Display units	mV
Titration type	Direct
Blank required	No
Result units	% w/v
Reaction ratio	1
Sample MW	58.44
Sample amount	Variable volume
Pre-dose titrant volume	0 mL
Max. total titrant volume	10 mL
Titration process control	Routine
Pre-stir duration	5 sec
Stir speed	Medium
Sample ID	Manual

Electrode preparation

Remove the electrode from storage. Add electrode fill solution up to the bottom of the fill hole. Leave the fill hole open during testing. Rinse thoroughly with RGW before and between titrations. At the end of the day, clean any foreign materials from the silver sensor by wiping with a moistened lint-free wiper. Thoroughly rinse the electrode with RGW, and store in tap water with the ceramic junction submerged. Cover the fill hole.

Sample preparation

Weight-based salt results

For thick liquid (e.g., ketchup) semi-solid, or solid samples, prepare the samples as follows.

1. If the sample has >1% salt, dilute the sample as follows:
    a. Measure 25.0 g of sample into a beaker. Add 475 mL of RGW. Mix well for 2 minutes, then let any solids settle out. This is the diluted sample.

    b. Pipet a portion from the top layer of the diluted sample for testing as indicated in Table 3 (second column).

    c. Place that portion of the diluted sample in a 100 mL or 150 mL beaker. Add RGW to about the 60 mL mark on the beaker.

    d. Proceed with the titration as indicated in the “Sample titration” section.

    e. When prompted, enter the diluted sample weight as indicated in Table 3 (last column).

2. If the sample has ≤1% salt, dilute the sample as follows:
    a. Measure 50.0 g of sample into a beaker. Add 450 mL of RGW. Mix well for 2 minutes, then let any solids settle out. This is the diluted sample.

    b. Pipet a portion from the top layer of the diluted sample for testing as indicated in Table 3 (second column)

    c. Place that portion of the diluted sample in a 100 mL or 150 mL beaker. Add RGW to about the 60 mL mark on the beaker.

    d. Proceed with the titration as indicated in the “Sample titration” section.

    e. When prompted, enter the diluted sample weight as indicated in Table 3 (last column).

Table 3

Thick liquid, semi-solid, and solid samples—weight-based results (% w/w)		
Salt content*	**Volume taken for titration**	**Enter weight as**
15%	1.0 mL diluted sample	0.05 g
10%	2.0 mL diluted sample	0.10 g
5%	3.0 mL diluted sample	0.15 g
1%	10.0 mL diluted sample	0.5 g
0.5%	20.0 mL diluted sample	1.0 g

* For other salt content values, follow instructions for closest % value or the next lowest value either one works.

Table 4

Thin liquid samples—volume-based results (% w/v)		
Salt content*	**Volume taken for titration**	**Enter volume as**
15%	1.0 mL diluted sample	0.05 mL
10%	2.0 mL diluted sample	0.10 mL
5%	3.0 mL diluted sample	0.15 mL
1%	1.0 mL (not diluted)	1.0 mL
0.5%	2.0 mL (not diluted)	2.0 mL

* For other salt content values, follow instructions for closest % value or the next lowest either one works.

Volume-based salt results

For thin liquid samples (e.g., clear soups, soy sauce), prepare the samples as follows.

1. If the sample has >1% salt, dilute the sample as follows:
   a. Measure 25.0 mL of sample into a beaker. Add 475 mL of RGW. Mix well to dilute the sample.

   b. Pipet a portion of the diluted sample for testing as indicated in Table 4 (second column)

   c. Place that portion of the diluted sample in a 100 mL or 150 mL beaker. Add RGW to about the 60 mL mark on the beaker.

   d. Proceed with the titration as indicated in the ”Sample titration” section.

   e. When prompted, enter the diluted sample volume as pipetted and indicated in Table 4 (last column).

2. If the sample has ≤1% salt, measure the sample directly.
   a. Pipet the volume indicated in Table 4 (second column) into a 100 mL or 150 mL beaker.

   b. Add RGW to about the 60 mL mark on the beaker.

   c. Proceed with the titration as indicated in the “Sample titration” section.

   d. When prompted, enter the sample volume as pipetted, as indicated in Table 4 (last column).

Sample titration

For thick liquid (e.g., ketchup), semi-solid, or solid samples, data are reported on a % w/w basis:

1. From the “Home” screen or the “Methods” screen, select the option to run the saved method “T6a Salt by weight”.

2. Rinse the electrode, stirrer, and dispenser with RGW. Place the electrode, stirrer, and dispenser into the prepared sample in the beaker. Ensure that the dispenser tip is inserted below the surface of the sample. Ensure that the electrode ceramic junction is immersed.

3. Start the titration. Enter the sample weight and sample ID when prompted.

4. Results are reported as salt in % w/w. For other results units, see “Method modifications” below.

For thin liquid samples (e.g., clear soups, soy sauce), data are reported on a % w/v basis:

1. From the “Home” screen or the “Methods” screen, select the option to run the saved method “T6b Salt by volume”.

2. Rinse the electrode, stirrer, and dispenser with RGW. Place the electrode, stirrer, and dispenser into the prepared sample in the beaker. Ensure that the dispenser tip is inserted below the surface of the sample. Ensure that the electrode ceramic junction is immersed.

3. Start the titration. Enter the sample volume and sample ID when prompted.

4. Results are reported as salt in % w/v. For other results units, see “Method modifications” below.

Results

Table 5

Salt content of foods			
Sample	**Average (n = 3)**	**Relative Standard Deviation**	**Analysis time**
Ketchup	2.798% w/w	0.33%	01:53 min
Salsa	1.651% w/w	0.45%	02:25 min
Soy sauce	16.73% w/v	0.59%	02:58 min
Marinara sauce	0.382% w/w	1.32%	02:13 min

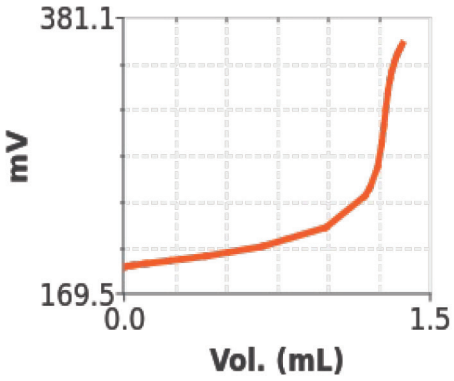

Figure 1. Salt content in salsa.

Range

This preprogrammed titration method covers a range of about 20 to 0.5% w/w salt (sodium chloride), when using 0.1 M (0.1 N) silver titrant and the prescribed volume of sample.

Method modifications

- For salt by weight results in units other than % w/w:
- Edit the method (salt by weight), edit “Titration”, and change the result units to mg/100 mg, g/100 g, or ppm by weight.
- For salt by volume results in units other than % w/v:
- Edit the method (salt by volume), edit “Titration”, and change the results units to mg/100 mL, g/100 mL, or ppm.

Titrant

Over time, standard titrant solutions age and can change in concentration. For higher accuracy, determine the exact concentration by standardizing the titrant. It is common to standardize on a weekly basis, but other standardization frequencies may be suitable.

1. Standardizing titrant
   a. Pipet 2.0 mL of the standardizing solution, 0.1 M (0.1 N) potassium chloride standard, into a clean 100 mL beaker. Add about 60 mL of RGW to the beaker.

   b. Select “T6a Salt”. At the titration pre-check screen, select the “Standardize” option.

   c. Start the titration.

   d. Run 3 or more cycles for the most accurate results.

   e. The new standardized titrant concentration will automatically be saved and used for subsequent salt content titration methods.

2. Certified standardized titrant solutions
   a. Some customers may prefer not to standardize their titrant, instead choosing to purchase and use certified standardized titration solutions. In this case, edit the “Titrant” section of the method. Choose manual entry and enter the certified concentration \ and titrant ID (i.e., lot number) if desired.

   b. Silver nitrate titrant is light sensitive. Install the Orion Star T900 light-blocking tubing kit on the tubing of the titrator as an extra measure to protect the integrity of the titrant.

Titrator 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. The main points for care are summarized below.

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Daily care

- If bubbles are visible in the titrator tubing, dispense titrant until bubbles have been expelled. Tap tubing to dislodge bubbles that stick.
- Add electrode fill solution up to the bottom of the fill hole, and leave the fill hole open during measurement.
- Rinse electrode well with RGW before and between titrations.
- Clean any foreign material from the silver billet sensor by wiping gently with a moistened lint-free wiper.
- Storage: Thoroughly rinse the electrode with RGW, and store in tap water with the ceramic junction submerged. Cover the fill hole.

Weekly or biweekly care

- Drain and replace the fill solution of the electrode.
- Change the storage solution of the electrode.
- Consider standardizing the titrant on a weekly basis, or more frequently, as desired.

As needed

- For slow or drifting electrode response, soak electrodes for 15 minutes in warm 1% laboratory detergent while stirring. Rinse well with RGW afterward.
- If the response is still slow or drifting, use Thermo Scientific™ Orion™ pH cleaning solution C per instructions.
- See the electrode user manuals for maintenance details.

Ordering information

Product	Description	Cat. No.
Titrator kit	Orion Star T930 Ion Titrator Chloride/Salt Kit with Silver Billet Electrode and Electrode Cable	START9301
Titrator	Orion Star T930 Ion Titrator, without electrode	START9300
	Orion Star T940 All-in-One Titrator, without electrode	START9400
Electrode and cable	Orion Silver Billet Combination Electrode	9780SC
	Orion Screw Cap Cable (used with Orion Silver Billet Combination Electrode, 9780SC)	91CBNC
Reagent-grade water	Barnstead Smart2Pure 12 UV Water Purification System	50129890*
Accessories	100 and 150 mL beakers 1,000 mL beakers 10 mL graduated pipet	Contact your sales representative for purchase
Reagents	0.1 M (0.1 N) silver nitrate titrant	
	Orion 0.1 M potassium chloride standard solution	921906

* 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**

References

1. Sodium Chloride in Canned Vegetables, Method 971.27. Official Methods of Analysis (OMA). AOAC International, 2275 Research Blvd, Ste 300, Rockville, MD 20850-3250.
2. Ward, R.E. and Carpenter, C.E. (2010). Traditional Methods for Mineral Analysis. In: S. Nielsen, ed., Food Analysis, 4th ed. New York: Springer, pp. 206-207.

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Find out more at **thermofisher.com/titrator**