Sodium in milk by direct Ion Selective Electrode (ISE) determination

Key words

Milk, Whole Milk, Processed Milk, Homogenized Milk, Pasteurized Milk, Dairy, Sodium, Ion Selective Electrode, ISE, Orion 8611BN, measure, determination.

Goal

This application note describes a simple, direct ion selective electrode (ISE) method for the determination of sodium in whole or skim milk. The only sample preparation required is to bring the sample to room temperature and to add a volume of ionic strength adjustor (ISA) solution to the sample before testing.

Introduction

The Indian Council of Medical Research has reported that "milk adulterants have hazardous health effects" that can cause immediate gastrointestinal complications and far more serious long-term effects.[1]

Examples of adulterants that may be added to milk include: sodium hypochlorite (to reduce the microbial count), sodium carbonate, sodium bicarbonate, sodium citrate, sodium hydroxide (to reduce high acidity due to milk spoilage), and/or sodium chloride (to thicken diluted milk).[2]

Recommended equipment

- Thermo Scientific™ Orion Star™ A214 pH/ISE meter pH and sodium kit, Cat. No. STARA2148, (which includes the ROSS Ultra pH electrode, Cat. No. 8102BNUWP, ROSS sodium ion selective electrode (ISE), Cat. No. 8611BNWP, Orion ATC temperature probe, Cat. No. 927007MD, Orion stirrer probe, Cat. No. 096019, sodium standards, reagents, and solutions);
- 5 mL pipette;
- 100 mL graduated cylinders;
- 1-L (1000 mL) volumetric flask;

- 100-mL beakers; plastic squeeze bottle (1L) for rinsing.
- If not using the automatic stirrer, use a stir bar and magnetic stirrer instead.

Required solutions

- 1000 ppm Sodium standard (Cat. No. 841108);
- 100 ppm Sodium standard (Cat. No. 941107);
- Sodium ISA (Cat. No. 841111);
- Sodium electrode storage solution (Cat. No. 841101);
- Sodium electrode fill solution (Cat. No. 90010);
- Sodium electrode reconditioning solution (Cat. No. 841113);
- Reagent grade water (RGW) that is free of sodium.
- User prepared solutions

User prepared solutions

Sodium rinse solution: Add 10 mL of sodium ISA (Cat. No. 841111) to a 1L squeeze bottle and fill with RGW. Don't rinse the electrode with RGW. Rinse with this sodium rinse solution.

ThermoFisher SCIENTIFIC

Electrode setup

See the electrode user guide for preparation of the electrode. Be sure to use the sodium electrode reconditioning solution to recondition the electrode before the first use, on a weekly basis, and whenever the slope of the electrode becomes too low or the response becomes slow. Note: sodium reconditioning solution contains a hazardous chemical. Observe all necessary safety precautions.

Meter setup

Connect the prepared sodium ISE to the BNC connector on the meter. Connect the ATC to the ATC/CON connector on the meter. Connect the stirrer probe to the meter. Power up the meter, go into Setup, pH/ISE Channel, Mode and Settings. Set Measure Mode to ISE, Read Type to Continuous, Resolution to 3, and Measure Unit to mg/L or ppm. Note: if not using the motorized automatic Orion stirrer, use a magnetic stirrer and a stir bar to stir each sample and standard. Stirring is required for the fastest response and best method performance.

Calibration

1. Prepare standards for testing as follows: for each standard (100 and 1000 mg/L), use a graduated cylinder to measure 50 mL of standard into a clean dry beaker. Add 5 mL of sodium ISA with a pipet.

2. Prior to calibration, rinse the ISE with sodium rinse solution and gently shake or dab electrode with a soft lab tissue to remove excess water drops.

3. Then place the ISE, ATC, and stirrer in the 100 mg/L standard and stir for 1 minute.

4. Go into Calibration mode and perform a two point calibration using 100 mg/L and 1000 mg/L sodium standards.

   – Make sure the stirrer is on during the measurements.

   – Note the temperature of the standards. If not at room temperature (RT), bring to RT (e.g., +/- 2°C) before proceeding with the calibration.

   – Once in Calibration mode, press “start” and wait for a stable reading.

   – When the reading stabilizes, select “edit”, enter the value “100” and accept. Press “next”.

   – Rinse the electrode, ATC, and stirrer with sodium rinse solution. Place the electrode in the 1000 mg/L standard.

   – Press “start” and stir for at least 1 minute.

   – After at least 1 minute and when the reading stabilizes, select “edit”, enter the value “1000” and accept. Press “cal done”.

   – After the calibration, the calibration slope will be displayed. It should be between 54 and 60 mV/decade.

   – If the slope is good, return to Measure mode.

   – If the slope is not acceptable, see the Electrode Performance Check section below.

Electrode performance check

If the calibration slope is not between 54 to 60 mV, check slope according to the Checking Electrode Operation (Slope) procedure described in the electrode user manual. If results drift, determine the response time by collecting readings at 1 minute intervals until the results stabilize. If electrode slope is low or electrode drifts (takes too long to stabilize), perform weekly maintenance as described at the end of this note. For more information, consult the troubleshooting section of the electrode manual.

Sample preparation

Measure 50 mL of milk into a 100 mL beaker. Add 5 mL of sodium ISA solution to the beaker. Allow the sample to come to RT. The sample is ready for analysis.

Analysis

1. Rinse electrode, ATC probe and stirrer with sodium ISE rinsing solution and gently shake or dab electrode with a soft lab tissue to remove excess water drops.

2. Place probes and stirrer in sample, turn on stirrer, and wait for a stable reading. This usually takes about 2 minutes.

   – Note the sample temperature. If it is not at RT (e.g., +/- 2 degree C), bring to RT before proceeding.

3. When a stable reading is achieved, “ready” will be displayed. This is the sample result in ppm (mg/L).

4. After the measurement, rinse completely with sodium rinse solution and proceed to the next sample.

Calculation of Sodium per serving

To convert results in ppm (mg/L) to mg of sodium per serving, use the following equation:

mg Sodium per serving = results in mg/L × mLs per serving size ÷1000

Example: 387 mg/L x 240 mL per serving ÷ 1000 = 92 mg sodium per serving of 240 mL

Quality Control (QC)

Recommended QC procedures may include: calibration and calibration verification, sample duplicates, slope, and drift.

Performance

Three milk samples were analyzed:

1. Refrigerated pasteurized (PST), homogenized whole milk with added vitamins C & D
2. Boxed shelf-stable ultra heat treated (UHT) whole milk with added vitamin D
3. A certified reference material (CRM) skim milk powder

Sodium in milk was analzyed in the applications lab by direct ISE testing and by three external labs performing Flame Atomic Absorption Spectrometry (FAAS) or Inductively Coupled Plasma (ICP) testing. Sodium results by ISE agreed well with sodium results by FAAS and ICP. One external lab (Lab 2 FAAS) gave outlier results. See Figure 1.

Sodium in the CRM was analyzed by direct ISE testing and ICP. Results by both methods showed good agreement with the CRM certified value. See figure 2.

Sodium results in milk and the CRM by direct ISE testing gave good reproducibility. Duplicate sample results and results between two different Orion sodium electrodes agreed within 2%.

Conclusions

Sodium in milk can be determined quickly and simply by direct ISE method. The only sample preparation required is to bring the sample to room temperature and add a volume of ionic strength adjustor (ISA) solution to the sample before testing. Each test takes only 2 or 3 minutes. Results are accurate and reproducible. Results of direct ISE testing compared well with results from FAAS and ICP testing. Direct ISE testing of the CRM milk powder gave results that agreed well with the certified value. Sample duplicate results agreed well. Duplicate electrode results agreed well.

Figure 1: Sodium in Whole Milk

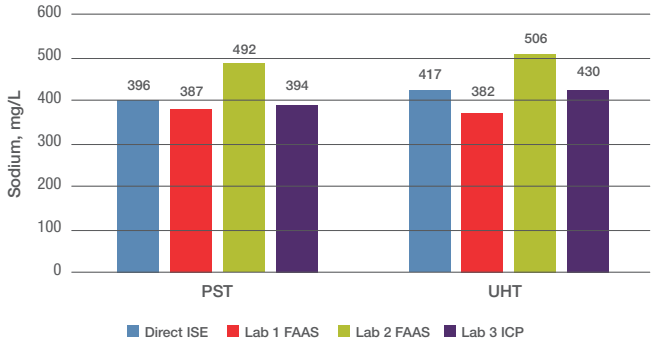
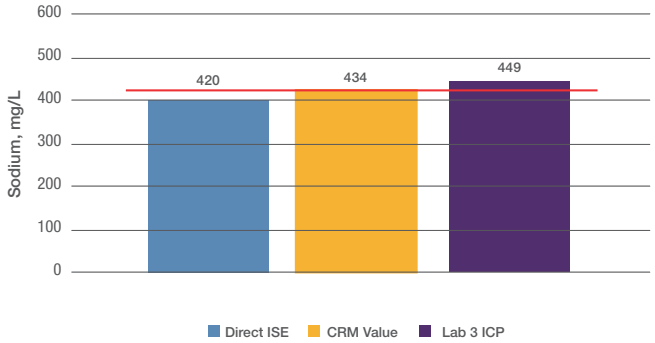


Figure 2: Sodium in CRM



Hints and tips for sodium in milk

- Refer to the electrode user guide for details on cleaning, storage, and maintenance recommendations to keep the electrode performing well. Main points for electrode care are summarized below in Figure 3.
- Do not use Orion 841109 Sodium Known Addition 1000 ppm standard to prepare a 100 ppm standard. The concentration of sodium will not be as expected.
- If it is desired to prepare (rather than purchase) 100 ppm sodium standard, follow these instructions. Use a 100 mL graduated cylinder to measure 100 mL of 1000 ppm Sodium standard (Orion 841108). Add the measured standard to a 1000 mL (1L) volumetric flask or 1L graduated cylinder. Dilute to 1L with RGW. Mix well.
- Orion pH/ISE meters other than the Orion Star A214 meter may be used for this testing. See the last page for some choices of Orion meters.

Electrode handling and care

Refer to the meter and electrode user manuals for details on cleaning, storage, and maintenance recommendations to keep the meter and electrode performing well.

Daily care

- Add sodium electrode fill solution to the bottom of the fill hole and leave the fill hole open during measurement.
- Rinse the electrode thoroughly with sodium rinse solution before and after measurements. Don't use RGW to rinse.
- At the end of the testing session, rinse the electrode with sodium rinse solution and gently wipe the ISE with a soft lab tissue to remove any fats or oils.
- Cover the fill hole. Store overnight in sodium electrode storage solution.

Weekly or biweekly care

- Drain and replace the fill solution of the electrode.
- Change the electrode storage solution.
- Recondition the electrode as described below.

As needed

- If the slope becomes too low or the electrode response becomes too slow, recondition the electrode as described below. This is a very important maintenance activity which is needed to guarantee the continued good performance of the sodium electrode. Do not skip this activity.
- See User Manual for other maintenance details.

Electrode reconditioning

- Sodium reconditioning is a very important maintenance activity which is needed to guarantee the continued good performance of the sodium ISE. Do not skip this activity.
- Sodium reconditioning solution contains a hazardous chemical. Observe all necessary safety precautions. Place a small amount of sodium reconditioning solution in a plastic beaker. Place the sodium ISE in the electrode holder. Immerse just the glass bulb of the sodium ISE into the reconditioning solution for 1 minute. Remove from solution and rinse completely with sodium ISE rinse solution. Flush and refill the electrode with new sodium electrode fill solution. Soak in sodium storage solution for 15 minutes before returning to use. When handling sodium reconditioning solution, observe all necessary safety precautions and dispose of the used reconditioning solution properly.

References

1. Milk Adulteration and its Control. Dairy Knowledge Portal. https://www.dairyknowledge.in/content/milk-adulteration-and-its-control-0.
2. "Detection of several common adulterants in raw milk by MID-infrared spectroscopy and one-class and multi-class multivariate strategies". Food Chemistry, Volume 230, 1 September 2017, pages 68 - 75.

thermo scientific

Ordering information

Description	Cat. No.
Meter	
Thermo Scientific Orion Star A214 pH/ISE Benchtop Meter pH and Sodium Kit	STARA2148
Thermo Scientific Orion Dual Star Two Channel pH/ISE Benchtop Meter pH and Sodium Kit	2115205
Thermo Scientific Orion Versa Star Pro 40 pH/ISE Benchtop Meter Only	VSTAR40A
Electrodes	
Thermo Scientific Orion Sodium Ion Selective Electrode (ISE)	8611BNWP
Thermo Scientific Orion ATC probe, stainless steel	927007
Accessories	
Thermo Scientific Orion Stirrer Probe	096019
Solutions	
Thermo Scientific Orion 1000 ppm Sodium Standard	841108
Thermo Scientific Orion 100 ppm Sodium Standard	941107
Thermo Scientific Orion Sodium Ionic Strength Adjustor (ISA)	841111
Thermo Scientific Orion Sodium Electrode Storage Solution	841101
Thermo Scientific Orion Sodium Electrode Reconditioning Solution	841113
Thermo Scientific Orion Sodium Electrode Fill Solution	900010
Laboratory reagent water	
Thermo Scientific Barnstead Smart2Pure 12 UV Water Purification System	50129890*

*Please contact your local Thermo Scientific representative for support on ordering the best water purification system for your application.

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

ThermoFisher SCIENTIFIC