APPLICATION NOTE

Iodide in raw and processed milk by direct ion selective electrode determination

Water Analysis Instruments,
Thermo Fisher Scientific

Key words
Milk, raw milk, processed milk, homogenized milk, dairy, iodine, iodide, ion selective electrode, ISE, Orion 9453BN, Orion 9653BNWP, measure, determination.

Goal

This application note describes a simple ion selective electrode (ISE) method for the determination of iodide in raw and processed milk.

Introduction

Iodide in milk is measured directly using an Orion iodide ion selective electrode (ISE). The only sample preparation required is to bring the sample to room temperature and to add a small volume of ionic strength adjustor (ISA) to the sample before testing. The use of a nickel nitrate ISA allows for superior electrode stability. The specified electrode rinse routine allows for stable electrode response throughout the testing, for skim and whole milk, both raw and homogenized[1].

Recommended Equipment

- Thermo Scientific™ Orion™ pH/ISE meter
- Orion iodide ISE
- Automatic temperature compensation probe (ATC)
- Automatic stirrer (optional)
- Automatic or glass pipettes
- 1 L and 100 mL volumetric flasks
- 100 mL beakers

Required Solutions

- 0.1M iodide standard
- Optimum results D reference filling solution
- Reagent grade water (RGW) that is free of iodide

User prepared solutions:

- 1M Nickel nitrate ISA
- Electrode rinse solution 1 (0.3% EDTA/SDS)
- Electrode rinse solution 2 (50% acetone-water mix)

User Prepared Solutions

1. Prepare 100 mg/L iodide standard by pipetting 7.88 mL of 0.1M iodide standard (Orion 945306) into a 1000 mL volumetric flask and diluting to the mark with RGW.
2. Prepare 1000 ug/L iodide standard by pipetting 10 mL of 100 mg/L iodide standard into a 1000 mL volumetric flask and diluting to the mark with RGW.

ThermoFisher SCIENTIFIC

3. Prepare 500 ug/L iodide standard by pipetting 5 mL of 100 mg/L iodide standard into a 1000 mL volumetric flask and diluting to the mark with RGW.
4. Prepare 100 ug/L iodide standard by pipetting 1.0 mL of 100 mg/L iodide standard into a 1000 mL volumetric flask and diluting to the mark with RGW.
5. Prepare 1M nickel nitrate ISA: Place 29.08 g of nickel (II) nitrate hexahydrate ($Ni(NO_3)_2$-$6H_2O$) into a 100 mL volumetric flask with 80 mL of RGW. Mix until dissolved. Add RGW to make 100 mL.
6. Prepare Electrode Rinse Solution 1 (0.3% EDTA/SDS): Place 3 g of disodium EDTA and 10 g of sodium dodecyl sulfate into a 1 L container. Add RGW to make 1 L. Stir until dissolved. Place 50 mL into a beaker for rinsing after milk samples.
7. Prepare Electrode Rinse Solution 2 (50% acetone-water mix): Mix equal parts acetone and RGW. Place 50 mL into a beaker for rinsing after milk samples.

Electrode Setup

See the electrode user guide for preparation of the electrode.

Meter Setup

Connect the iodide combination ISE (9653BNWP) to the BNC connector on the meter. Connect the ATC to the ATC/CON connector on the meter. Connect the stirrer to the meter. Power up the meter, go into Setup, Mode and Settings. Set measure mode to ISE, Read Type to Continuous, Resolution to 3, and Measure Unit to ppb. *Note: if not using the motorized 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, 500, and 1000 ug/L), measure 50 mL of standard into a clean dry beaker and add 1 mL of the nickel nitrate ISA.
2. Prior to calibration, equilibrate the ISE in the 100 mg/L standard for 15 minutes.
3. Perform a two point calibration using 100 ug/L and 1000 ug/L iodide standards.
   a. Rinse the electrode, ATC, and stirrer with RGW before and after each standard.
   b. Make sure the stirrer is on during the measurements.
   c. Note the temperature of the standards. If not at room temperature (RT), bring to RT (±1 ºC) before proceeding with the calibration.
4. After the calibration, the calibration slope will be displayed. It should be between -54 and -60 mV/decade.
5. If the slope is good, return to Measure mode and read the 500 ug/L mid-level standard to verify calibration.
6. If the slope or calibration verification 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, collect 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 1 mL of nickel nitrate 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 RGW and gently shake or dab electrode with a lab tissue to remove excess water drops.
2. Place all probes in sample, turn on stirrer, and wait for a stable reading.
   - Note the sample temperature. If it is not at RT (±1 ºC), bring to RT before proceeding.
3. When a stable reading is achieved, “ready” will be displayed. This is the sample result in ppb (ug/L).
4. After the measurement, rinse with RGW, then place the ISE into Electrode Rinse Solution 1 for 10 seconds. Rinse with RGW, and dip briefly (< 5 seconds) into Electrode Rinse Solution 2. Rinse well with RGW.

Quality Control (QC)

Recommended QC procedures may include: mid-level calibration verification, QC sample analysis, sample duplicates, and/or matrix spikes.

thermo scientific

Performance

J. Melichercik et. al.[2] reported method performance as follows:

1. Recoveries for milk samples spiked at 50, 100, 200, 400 and 1000 ug/L ranged from 87 to 114%.
2. Within run repeatability (as standard deviation) was ±12 ug/L.
3. Overall agreement between ISE and high performance liquid chromatography (HPLC) results was r^2 = 0.85 and 0.84 for raw and processed milk, respectively.

See Reference 2 for more performance details.

References

1. Determination of Iodide in Milk Using the Iodide Specific Ion Electrode and its Application to Market Milk Samples. Lacroix, D.E. and Wong, N.P. Journal of Food Protection, 43:9, 1980, 672-674.
2. Comparison of Ion-Specific Electrode and High Performance Liquid Chromatography Methods for the Determination of Iodide in Milk. Melichercik, J. et al. Journal of Dairy Science, Volume 89, Issue 3, 2006, 934.937.

Hints and Tips for Iodide 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.

Daily Care	Weekly Care	As Needed
• Store overnight in RGW • Top up the reference filling solution • Using the special rinse routine, rinse well before, between, and after each sample and standard	• Place a drop of liquid dish detergent on a moist cloth or tissue and gently rub over the sensing element. Rinse well with RGW • Flush and replace the reference filling solution	• Gently polish the sensor, if the cleaning procedure does not improve performance • See User Manual for maintenance details.

To purchase Thermo Scientific laboratory products, please contact your local equipment distributor and reference the part numbers listed below:

Product	Description	Cat. No.
Meters	Thermo Scientific™ Orion Star™ A214 pH/ISE benchtop meter	STARA2140
	Thermo Scientific™ Orion™ Dual Star™ two channel pH/ISE benchtop meter	2115000
	Thermo Scientific™ Orion™ Versa Star Pro™ 40 pH/ISE benchtop meter	VSTAR40A
Electrode	Thermo Scientific Orion iodide Ion Selective Electrode (ISE)	9653BNWP
	Thermo Scientific Orion ATC probe, stainless steel	927007
Solutions	Thermo Scientific Orion 0.1M iodide standard	945306
	Thermo Scientific Orion Optimum Results D fill solution (refills)	900063
Accessories	Thermo Scientific Orion stirrer	096019
Reagent Grade 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/water**

Water and Lab Products

Australia: (613) 9757-4300 In Australia: (1300) 735-295 **China:** (86) 21-6865-4588
Germany: (49) 6184-90-6321 **India:** (91) 22-6716-2261/2247 **Japan:** (81) 045-453-9175
North America: 1-978-232-6000 Toll Free: 1-800-225-1480 **Singapore:** (65) 6778-6876

ThermoFisher SCIENTIFIC