Sodium Determination with Ion Selective Electrode

Method for the sodium determination by direct measurement with a sodium ion selective electrode.

Sample	50 mL Sample solution with 0.1 to 0.001 mol/L sodium
Substance	Sodium M = 22.99
Chemicals	1 mL ISA solution (4 m NH_4Cl and 1 m NH_4OH)
Titrant	
Standard	
Instruments	DL67, DL70ES, DL77 Printer (HP Desk Jet 500)
Accessories	Titration beaker ME101974 DT120 (Temp. sensor Pt100)
Indication	DX223 sodium ion selective electrode DX200 reference electrode
Chemistry	
Calculation	
Waste disposal	
Author	A. Aichert/Anke Stock

Preparation and Procedures

Milk: Dilute sample 1:10. To 50 mL of this add 1 mL ISA solution and measure.
Apple juice: To 50 mL undiluted sample add 1 mL ISA sol. and 1.5 mL NH_4OH 5% . The pH of the sample is adjusted to 7.5 with excess NH_4OH (For correct measurement with Na-ISE, the pH must be over 7).
Wine, grape juice: To 50 mL undiluted sample add 1 mL ISA sol. and 1.5 mL NH_4OH 5% . The pH of the sample is adjusted to 8 with excess NH_4OH (the pH must be over 7).
Mineral water: To 50 mL sample add 1 mL ISA solution.
Drinking water: To 50 mL sample add 1 mL ISA solution and measure. Due to the low sodium content, the standards 10-3 and 10-4 mol/L were used for calibration for this sample.
Bouillon: Place a bouillon cube in 300 mL deion.water and warm till it has disolved. Let cool and use deion.water to adjust volume to 500 mL. Dilute this solution 1:20 resp. 1:50 with deion.water. To 50 mL add 1 mL ISA sol. and measure.
Herb patè: Mix entire contents of can (125g) with 700 mL deion. water for about 5 minutes. Decant fat. Fill to 1000 mL with deion. water. To 50 mL of this add 1 mL ISA solution and measure.
Infusion solution: Dilute sample 1:100 and to 50 mL of this add 1 mL ISA solution and measure.

Remarks

Reproducibility and Recovery
Aqueous Na solutions made from NaCl were used (titrimetric standard). The Na-ISE was recalibrated daily with standards 0.1 and 0.001 g Na^+/L. It was stored in a concentrated sodium solution. To 50 mL sample add 1 mL ISA sol. then measure. The solutions were produced daily.

Conc.	Recovery	RSD of sev. series(n=6)	
mol/L	%	Method A	Method B
0.1	98-102%	0.2-0.5%	0.2-0.6%
0.01	95-100%	0.2-0.6%	0.3-0.7%
0.001	97-101%	0.2-0.8%	0.2-0.9%
0.0005	101-106%	0.2-0.4%	0.2-0.5%
0.0001	131-129%	0.2-0.9%	0.2-0.9%

Result
The differences between A and B are not decisive. Below 0.001 mol/L longer stir times (15-20 min) were necessary to get a good reproducibility. The concentration 0.001 mg/L leaves the linear range (see cal. curve). Thus, the useful concentration range was limited from 0.1 mol/L to 0.001 mol/L.
General remarks:
All measurements were performed using the simple direct measurement (method B). All measurements should be performed using the same stirring conditions. This means the same speed, stirrer type, distance form stirrer to electrode etc.

Results

Results

Sample	Dilution	Direct measurement with sodium electrode n	Mittelwert	RSD	Content (Producer spec.)
Milk, full cream, pasteurized	1:10	6	400.8 mg/L	0.20%	430-490 mg/L*
Milk, full cream, raw	1:10	6	433.8 mg/L	0.20%	430-490 mg/L*
Apple Juice	none	4	8.2 mg/L	0.64%	[30 mg/L*
Grape Juice,red	none	5	23.8 mg/L	0.66%	[30 mg/L*
Wine,red Italian	none	5	12.2 mg/L	0.63%	10-80 mg/L*
Wine, White, Swiss	none	5	8.4 mg/L	2.0%	10-80 mg/L*
Mineral Water	none	6	8.17 mg/L	0.22%	8.2 mg/L
Drinking Water, Schwerzenbach	none	5	3.81 mg/L	0.35%	--
Bouillon sodium reduced	1:20	5	468.5 mg/L	0.35%	[1.2 g/L
Bouillon normal	1:50	5	4110 mg/L	0.41%	-4 g/L
Herb patè sodium reduced	1:8	6	175.9 mg/kg	0.52%	[0.4 g/kg
Infusion Solution	1:100	5	142.5 mmol/L	0.72%	141.5 mmol/L

* Obtained from: Schweizerisches Lebensmittelbuch

Method A stirs for 5 minutes then takes 6 measurements with an interval of at least 10 seconds. The sodium concentration is calculated from the mean of these 6 measured values.
Method B measures once after a stir time of 5 minutes. The sodium concentration is calculated from this. If the sample is diluted for the measurement, the dilution factor can ve entered as a correction factor (f) for each sample. This will be used in the calculation.

Table of measured values

Titration curve

Method

```
Method A
(Direct measurement with mean value)

Title
   Method ID  . . . . . . . . . . . . . Na0A
   Title  . . . . . . . . . . . . . . . Na+ -content
   Date/time  . . . . . . . . . . . . . 15-Mar-1994  19:48
Sample
   Number samples . . . . . . . . . . . 6
   Titration stand  . . . . . . . . . . Stand 1
   Entry type . . . . . . . . . . . . . Fixed volume U
      Volume [mL]     . . . . . . . . . 50.0
   ID1  . . . . . . . . . . . . . . . .
   Molar mass M . . . . . . . . . . . . 0.0
   Equivalent number z  . . . . . . . . 1
   Temperature sensor . . . . . . . . . TEMP A
Stir
   Speed [%]  . . . . . . . . . . . . . 50
   Time [s] . . . . . . . . . . . . . . 600
Measure
   Sensor . . . . . . . . . . . . . . . Na+ -sensor
   Unit of meas.  . . . . . . . . . . . As installed
         dE [mV]  . . . . . . . . . . . 0.2
         dt [s] . . . . . . . . . . . . 5.0
         t(min) mode  . . . . . . . . . Fix
           t (min) [s]  . . . . . . . . 10.0
           t (max) [s]  . . . . . . . . 90.0
Measure
   Sensor . . . . . . . . . . . . . . . Na+ -sensor
   Unit of meas.  . . . . . . . . . . . As installed
         dE [mV]  . . . . . . . . . . . 0.2
         dt [s] . . . . . . . . . . . . 5.0
         t(min) mode  . . . . . . . . . Fix
           t (min) [s]  . . . . . . . . 10.0
           t (max) [s]  . . . . . . . . 90.0
Measure
   Sensor . . . . . . . . . . . . . . . Na+ -sensor
   Unit of meas.  . . . . . . . . . . . As installed
         dE [mV]  . . . . . . . . . . . 0.2
         dt [s] . . . . . . . . . . . . 5.0
         t(min) mode  . . . . . . . . . Fix
           t (min) [s]  . . . . . . . . 10.0
           t (max) [s]  . . . . . . . . 90.0
Measure
   Sensor . . . . . . . . . . . . . . . Na+ -sensor
   Unit of meas.  . . . . . . . . . . . As installed
         dE [mV]  . . . . . . . . . . . 0.2
         dt [s] . . . . . . . . . . . . 5.0
         t(min) mode  . . . . . . . . . Fix
           t (min) [s]  . . . . . . . . 10.0
           t (max) [s]  . . . . . . . . 90.0
Measure
   Sensor . . . . . . . . . . . . . . . Na+ -sensor
   Unit of meas.  . . . . . . . . . . . As installed
         dE [mV]  . . . . . . . . . . . 0.2
         dt [s] . . . . . . . . . . . . 5.0
         t(min) mode  . . . . . . . . . Fix
           t (min) [s]  . . . . . . . . 10.0
           t (max) [s]  . . . . . . . . 90.0
Measure
   Sensor . . . . . . . . . . . . . . . Na+ -sensor
   Unit of meas.  . . . . . . . . . . . As installed
         dE [mV]  . . . . . . . . . . . 0.2
         dt [s] . . . . . . . . . . . . 5.0
         t(min) mode  . . . . . . . . . Fix
           t (min) [s]  . . . . . . . . 10.0
           t (max) [s]  . . . . . . . . 90.0
Calculation
   Result name  . . . . . . . . . . . .
   Formula  . . . . . . . . . . . . . . R1=E[1]
   Constant . . . . . . . . . . . . . .
   Result unit  . . . . . . . . . . . . p(Na+)
   Decimal places . . . . . . . . . . . 4
Calculation
   Result name  . . . . . . . . . . . . Na+ -single
   Formula  . . . . . . . . . . . . . . R2=pw(-E[1])*1000
   Constant . . . . . . . . . . . . . .
   Result unit  . . . . . . . . . . . . mmol/L
   Decimal places . . . . . . . . . . . 4
Calculation
   Result name  . . . . . . . . . . . .
   Formula  . . . . . . . . . . . . . . R3=(C3+E[5]+E[6])/6
   Constant . . . . . . . . . . . . . . C3=E[1]+E[2]+E[3]+E[4]
   Result unit  . . . . . . . . . . . .
   Decimal places . . . . . . . . . . . 3
Calculation
   Result name  . . . . . . . . . . . . Na+ -x of 6
   Formula  . . . . . . . . . . . . . . R4=pw(-R3)*1000
   Constant . . . . . . . . . . . . . .
   Result unit  . . . . . . . . . . . . mmol/L
   Decimal places . . . . . . . . . . . 4
Statistics
   Ri (i=index) . . . . . . . . . . . . R2
   Standard deviation % . . . . . . . . Yes
   Rel. standard deviation srel . . . . Yes
   Outlier test . . . . . . . . . . . . Yes
Statistics
   Ri (i=index) . . . . . . . . . . . . R4
   Standard deviation % . . . . . . . . Yes
   Rel. standard deviation srel . . . . Yes
   Outlier test . . . . . . . . . . . . Yes
Record
   Output unit  . . . . . . . . . . . . Printer
   All results  . . . . . . . . . . . . Yes
```

```
Method B
(Simple direct measurement)

Title
   Method ID  . . . . . . . . . . . . . Na0B
   Title  . . . . . . . . . . . . . . . Na+ -content
   Date/time  . . . . . . . . . . . . . 22-Mar-1994  13:07
Sample
   Number samples . . . . . . . . . . . 6
   Titration stand  . . . . . . . . . . ST20
   Entry type . . . . . . . . . . . . . Fixed volume U
      Volume [mL]     . . . . . . . . . 50.0
   ID1  . . . . . . . . . . . . . . . .
   Molar mass M . . . . . . . . . . . . 22.99
   Equivalent number z  . . . . . . . . 1
   Temperature sensor . . . . . . . . . TEMP A
Stir
   Speed [%]  . . . . . . . . . . . . . 50
   Time [s] . . . . . . . . . . . . . . 300
Measure
   Sensor . . . . . . . . . . . . . . . Na+ -sensor
   Unit of meas.  . . . . . . . . . . . As installed
         dE [mV]  . . . . . . . . . . . 0.5
         dt [s] . . . . . . . . . . . . 5.0
         t(min) mode  . . . . . . . . . Fix
           t (min) [s]  . . . . . . . . 60.0
           t (max) [s]  . . . . . . . . 120.0
Calculation
   Result name  . . . . . . . . . . . .
   Formula  . . . . . . . . . . . . . . R=E
   Constant . . . . . . . . . . . . . .
   Result unit  . . . . . . . . . . . . p(Na+)
   Decimal places . . . . . . . . . . . 3
Calculation
   Result name  . . . . . . . . . . . . Na+ -content
   Formula  . . . . . . . . . . . . . . R2=pw(-E)*f*1000
   Constant . . . . . . . . . . . . . .
   Result unit  . . . . . . . . . . . . mg/L
   Decimal places . . . . . . . . . . . 2
Statistics
   Ri (i=index) . . . . . . . . . . . . R2
   Standard deviation % . . . . . . . . Yes
   Rel. standard deviation srel . . . . Yes
   Outlier test . . . . . . . . . . . . Yes
Statistics
   Ri (i=index) . . . . . . . . . . . . R3
   Standard deviation % . . . . . . . . Yes
   Rel. standard deviation srel . . . . Yes
   Outlier test . . . . . . . . . . . . Yes
Record
   Output unit  . . . . . . . . . . . . Printer
   All results  . . . . . . . . . . . . Yes
```