CHLORIDE TESTING
BENCHNOTES

Salt Determination for Soups and Sauces in mEq/L using the Digital Chloridometer

Principle: *The salt content of soups and sauces is important in determining shelf life and taste.*

Procedure - Reagents

A) Acid Reagent

1. Acid reagent is available from Labconco or it may be made in-house.
2. Weigh out 9.0 grams of polyvinyl alcohol into a 250 ml beaker. Add enough water to make a slurry.
3. Heat to boiling about 500 ml distilled water. When the water begins to boil, add the slurry. Rinse the slurry beaker with a small amount of water and add to the rest.
4. Stir to dissolve and continue to heat until the solution boils again. Remove from heat and cool.
5. When cool, skim the top of the beaker, then pour the solution into a 1000 ml volumetric flask.
6. Add 6.3 grams concentrated reagent grade Nitric acid and mix.
7. Add 105 grams reagent grade Glacial Acetic acid.
8. Add 5.8 milligrams Sodium Chloride (0.0058 grams). Fill to the mark with distilled water.

B) Sodium Chloride Standard Solution

1. From dried reagent grade NaCl and distilled water, make a series of standard solutions in the following concentrations: 5%, 1.0%, 2.0%, 3.0%, 4.0%, 5.0%.

C) Calibration and Blank Adjustment

1. Clean the electrodes with silver polish and inspect for wear. If electrode is one half its original size, pull down more silver wire.
2. Rinse electrode with distilled water and buff dry.
3. Set instrument on high and blank adjust wheel to zero.
4. In three separate sample vials place 4 ml acid reagent. Raise sample to electrodes and record results.
5. Take the average of the three runs and place this number into the blank adjustment wheel.

D) Determination of slope for conversion to mEq/L

1. Take 10 grams of the standard solution in part B1 and add 240 grams distilled water.
2. Take 1.0 ml diluted 0.5% standard and place in a clean sample vial. Add 4 ml acid reagent and analyze. Repeat this step three more times and take the average.
3. Repeat steps D1-D2 for the rest of the standards.
4. Take the average of the standard and divide them by their corresponding concentrations in % NaCl.
5. Take the averages of all the standards in step D4 and average these. This is the Chloridometer slope value.

6. Using the slope value (step D5) convert mEq/L to % NaCl by dividing the sample reading by the slope value. NOTE: It is a good idea to check the slope every morning. To do this, make a stock solution of 1:25 of the 1.0% salt standard. It should correspond to the slope value.

E) Sample Testing

1. Thoroughly blend the sample.
2. Weigh out 10 grams of sample and add to 250 grams water. Mix well.
3. Take 1 ml dilute sample and place in 4 ml acid reagent.
4. Run on the Chloridometer.
5. Convert mEq/L to % salt by using the slope table or by manual calculations.

F) Running High Salt Concentrations

1. Taking 5.0 grams sample, add 245 grams water.
2. Take 1 ml dilute sample and place in 4 ml acid reagent.
3. Run on Chloridometer and multiply the result by 2.

Reference

Labconco Digital Chloridometer Manual
Campbell Research and Development Technical Service

This method is for reference only. It is not a citable document. It is based on data we believe to be reliable. It is offered in good faith but without guarantee.

CHLORIDE TESTING

BENCHNOTES

Determination of Low Concentrations of Chloride in Surface and Ground Water Using a Digital Chloridometer

Principle: *The determination of chloride in water allows workers to monitor the water treatment and purification process.*

Procedure

A) Reagent Preparation

1. Concentrated acid stock: In a 500 ml volumetric flask, add 250 ml distilled water to 12 ml concentrated nitric acid (0.4 N HNO_3) and 200 ml glacial acetic acid and 8 mg NaCl. Add distilled water to bring up to 500 ml.

2. Gelatin Reagent: Heat one liter of distilled water. Slowly empty one vial of gelatin reagent and mix. Reagent is good for six months if stored under refrigeration.

B) Instrument Set Up

1. Using a Digital Chloridometer, thoroughly clean all four electrodes with silver polish, rinse with distilled water and buff dry.

2. Set range switch on the low position.

3. Fill four vials with 3 ml distilled water and 1 ml concentrated acid stock. Add four drops of gelatin reagent and titrate.

4. Take the average of the four blanks and place this number in the blank adjustment switch.

C) Sample Testing

In a vial place 3 ml sample along with 1 ml concentrated acid stock with 4 drops gelatin. Titrate this sample in the normal manner and divide the final readout by 300.

Reference

Stallard, R.F. *Major Element Geochemistry of the Amazon River System,* Ph.D. Thesis, June 1980, Massachusetts Institution of Technology-Woods Hole Oceanographic Institution Joint Program in Oceanography, WHOI-80-29, 335, 366 p.

CHLORIDE TESTING

BENCHNOTES

Determination of Salt in Cheese using the Digital Chloridometer

Principle: *The concentration of NaCl in cheese affects the product's shelf life and taste.*

Procedure

A) Sample Preparation

1. Predetermine the moisture content of the cheese sample.
2. In a dry Erlenmeyer flask, weigh out 15-16 grams shredded cheese. Record the weight to the nearest milligram.
3. Zero the same flask and add 85 grams 0.1 N NaOH. Record to the second decimal.
4. Cap flask with a tight fitting foam rubber stopper and place in a shaker for 40 minutes at 65° C.
5. Repeat steps 1-3 and include one set of duplicates and a control.

B) Chloridometer Set Up

1. Clean the silver electrodes with silver polish. Inspect the electrode for wear. If the diameter is 1/2 the original thickness, replace it.
2. Rinse the electrode with distilled water.
3. Turn the range setting switch to LOW and the titration switch to STANDBY.

C) Sample Testing

1. To a glass sample vial, add 4 ml acid reagent and 4 drops of gelatin. (If using Chloridometer Acid Reagent, the gelatin can be omitted.)
2. Turn the titration switch to AUTO.
3. Place sample on the pedestal and lift to engage the stirrer. (The solution is now zeroed and ready for sample testing.)
4. Using a 100 µl pipette, take an aliquot of chloride standard, gently wipe the tip and deliver it into the zeroed vial. Engage the stirrer. Compare the result with the standard known concentration. It should be ±2.0 mEq/l. It is best to run the standard an average of three times.
5. Allow the sample to sit for five minutes after the shaker has turned off.
6. Remove the test sample, tilt it slightly and insert the pipette tip below the fat layer.
7. Pipette a 100 µl sample into a sample vial with 4 ml acid reagent along with 4 drops gelatin that have been zeroed.
8. Run samples in duplicate and average the numbers.

D) Calculations

1. Multiply the sample weight by its fraction of moisture to get the weight of water in the cheese.
2. Add the cheese water weight to the weight of the NaOH to get the total dilution weight.
3. Multiply the total moisture weight by the average count.
4. Multiply that result by a constant 0.000585.
5. Divide the result from step 4 by the sample weight to get the percent salt.
6. Divide the percent salt by the fraction of moisture to get the percent salt in moisture.

Example

15.234 grams sample, 38.0% moisture, 85.12 grams NaOH, averaged instrument count of 465

$$\frac{[(0.380 \times 15.234) + 85.12] \times 465 \times .000585}{15.234} = 1.62\ \%\ \text{NaCl}$$

$$1.62\% \div 0.38 = 4.27\%\ \text{salt in moisture}$$

Reference

Wisconsin Dairies, Baraboo, Wisconsin

CHLORIDE TESTING

BENCHNOTES

Sweat Analysis: Chloride by Coulometric-Amperometric Titration with a Digital Chloridometer

Principle: *The sweat chloride concentration can be determined from a sample after dilution and elution by titration using a Digital Chloridometer. Constant direct current is passed between a pair of silver electrodes, causing release of silver ions at a steady rate into the titration solution. When all the chloride has combined with silver ions, the appearance of free silver is indicated to the electrodes. The amount of silver needed to precipitate the chloride in the sample is proportional to the elapsed time.*

Apparatus

Digital Chloridometer and titration vials

Reagents

1. Double Strength Acid Dilution

300 ml Glacial Acetic Acid
19.2 ml Concentrated Nitric Acid
To 1200 ml distilled deionized Type I water (DI-H_2O), add the above amount of acid and mix. Store in a glass bottle. Stable 6 months at room temperature.

2. Gelatin Reagent

3.1 g Gelatin Mixture (6.2 g vial)
Add 3.1 g of gelatin mixture to 500 ml of hot DI-H_2O. Heat gently with continuous stirring until the solution is clear. Aliquot into 13 x 100 mm tubes, cover with Parafilm* or cap. This reagent is stable for 6 months at 4° C. Use a fresh tube with every run. Liquefy gelatin by immersing in warm water and mixing gently.
(*Parafilm® is a registered trademark of American National Can.)

Standard

1.00 mEq/l chloride standard

Store in a tightly stoppered glass bottle at 4° C. The standard is stable for 3 months or according to manufacturer's recommendations. Bring to room temperature before use.

Control

30 mEq/l chloride control

This is made as a 30 mEq/l chloride standard. It should be made up separately from the standards used to calibrate the Chloridometer. Store in a tightly stoppered glass bottle at 4° C. The standard is stable for 3 months or according to manufacturer's recommendations. Bring to room temperature before use.

6-28-A-5/96-100-R2

CHLORIDE TESTING

BENCH NOTES

Determination of Salt in Canned Tuna using the Digital Chloridometer

Principals of Operation

- The combination of silver ions and chloride ions is a quantitative reaction that results in an insoluble silver chloride precipitate. The Digital Chloridometer performs this reaction by the electrolytic generation of silver ions at a constant rate.
- When an amount of silver equivalent to the chloride content of a solution is reached, the current between a pair of indicator electrodes increases and is detected by the sensing circuitry.
- When the indicator current reaches a preset intensity, the instrument automatically stops both the counter and generation of silver ions.
- Since silver ions are generated at a constant rate, the amount used to precipitate the chloride ions is proportional to the elapsed time. The Digital Chloridometer displays this relative time in units of milliequivalents chloride per liter (mEq/L).
- Comparison of sample readings to known standard readings allows calculation of salt content of samples.

Apparatus

- Labconco Digital Chloridometer, catalog #442-5000 or #442-5100
- Sample Vials, 20 x 40 mm, catalog #586-0007 (200 provided with Digital Chloridometer)
- Automatic pipettor

Reagents

- Gelatin Reagent (Gelatin, Thymol Blue, Thymol; 60:1:1): Dissolve 0.62 gram reagent in 100 ml hot distilled water. Store in refrigerator when not in use.
- Acid Solution: Dilute 10 ml concentrated nitric acid and 150 ml glacial acetic acid to one liter with distilled water. **CAUTION:** Acid/water combination generates heat. Add acid to approximately 500 ml distilled water before diluting to volume with additional water.
- Salt Solution (Stock Standard): Dissolve 1.00 gram dry sodium chloride in 500 ml of distilled water (use a volumetric flask).
- Salt Solution (Cannery Standard, 0.00025 gram NaCl/ml): Pipet 25 ml of the Stock Standard into a 200 ml volumetric flask and dilute to volume with distilled water.

Sample Preparation

a. Blend fish in a Waring Blender with an equal weight of distilled water to produce a 1:1 slurry.

b. Transfer 10.0 grams of slurry to a 250 ml Erlenmeyer flask. Add 190 ml distilled water. Stopper and shake vigorously for 30 seconds.

c. Allow the mixture to settle.

Analytical Procedures

1. Before each set of analyses, titrate two 2.0 ml aliquots of Cannery Standard with 3.0 ml acid reagent and 4 drops of gelatin reagent to condition the electrodes.

2. Prepare four blank vials, each containing 2.0 ml distilled water, 3.0 ml acid reagent and 4 drops gelatin reagent.

3. Prepare two standard vials, each containing 2.0 ml of Cannery Standard, 3.0 ml acid reagent and 4 drops gelatin reagent.

4. Prepare sample vials, each containing 2.0 ml of a different extract, 3.0 ml acid reagent and 4 drops gelatin reagent.

5. Set **range** selector at **high, titration** switch to **auto,** and **blank adjust** at 00.0.

6. Titrate both conditioning solutions (step 1). Place one vial at a time in the holder and slide it all the way up. After the counter stops, repeat with the second solution. Disregard these readings.

7. Rinse electrodes with distilled water after each titration.

8. Titrate each blank solution, recording each reading. Average the four readings to obtain a blank correction factor. Enter the average blank value in the **blank adjust** switches.

9. Titrate both standards and all sample solutions, recording readings for each.

Calculation

The salt content of a sample is calculated by comparing the sample reading with the average reading of the two standards.

$$\% \text{ salt in fish} = \frac{\text{Sample Reading}}{\text{Average Standard Reading}}$$

Precautions

- Leave electrodes immersed in a vial of distilled water when the instrument is not in use.

- To avoid uncontrolled background errors, be certain that the reagent volume is constant and that the vials are clean.

- Clean electrodes daily.

References

- *Digital Chloridometer Instruction Manual,* 1978.
- *Aminco Solid-State Automatic Chloride Titrator and Direct Milliequivalent Readout Instruction,* No. 951, 1968.

Acknowledgements

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Procedure

1. Clean the electrodes on the Chloridometer according to the manufacturer's recommendations. Pull down additional silver wire when the electrode becomes thinned or shortened.
2. Remove the 1.00 mEq/l standard and the 30 mEq/l control from the refrigerator and allow them to come to room temperature.
3. Rinse several 1 ml volumetric pipettes with DI-H_2O. Also rinse the flask containing the DI-H_2O before using.
4. Label a 13 dram vial containing a piece of 2" x 2" gauze the "Gauze Blank" vial.
5. Prepare the "Control" vial. Wearing powder-free gloves, weigh a 13 dram vial labeled "Control". Record the weight (W1) on the worksheet. Add 100 µl of the 30 mEq/l control to the vial. Reweigh the vial and record the weight (W2) on the worksheet. Subtract the difference (i.e. W2-W1). This difference should be approximately 100 mg.
6. Using an automatic pippettor, add 8 ml of DI-H_2O to the patient vials, the Gauze Blank vial and the Control vial. Recap the vials and shake them for one minute. Allow the vials to sit at room temperature for at least 15 minutes. At the end of the 15 minutes, use a pipette to press the elute from the gauze and remove the gauze from the vials. Use the elute as the test solution in step 7.
7. Set the chloride titration vials as follows in duplicate using a preset 3 ml pipettor for double strength acid dilution. Use 1 ml volumetric pipettes to deliver the test solutions and DI-H_2O.

	Blank	Gauze Blank	Standard	Test Control
Double Strength Acid	3 ml	3 ml	3 ml	3 ml
DI-H_2O	1 ml	—	—	—
1 mEq/l Chloride Standard	—	—	1 ml	—
Gelatin Reagent	4 drops	4 drops	4 drops	4 drops
Test Solution from Vials	—	1 ml	—	1 ml

The solution's acidity is indicated by the presence of a pink color when 4 drops of gelatin are added to the diluted sample. Do not titrate unless pink color is present.

8. Set the Chloridometer up as follows:
   Blank adjust: 00.0
   Range: Low
   Titration: Standby
9. Rinse the electrode assembly of the Chloridometer with DI-H_2O. Wipe the assembly with an absorbent tissue.
10. Insert the chloride titration vial on the cup holder and place the vial under the electrode assembly. Press the titration switch to START. Hold down switch until counter starts counting from zero. The stirring rod should be able to rotate freely and appear to be a blur when performing.
11. Wait until the counter stops and record the titration time (in seconds) on the worksheet.

Calculations

1. Average the readings of duplicates for each vial. Then perform the following calculation:

$$\frac{\text{Test-gauze Blk}}{\text{Std. - Blk}} \times 8 + \text{wt. of sweat in gm} \times \frac{\text{Conc. Std.}}{(1.0)} = \text{mEq/l Cl in sweat}$$

Example

	Duplicates	Avg.
Std.	381/385	383
Blk.	32/33	33
Gauze Blk.	40/39	40
gm of sweat	.10035	40

$$\frac{\text{Test-Gauze Blk}}{\text{Std. - Blk.}} \times \frac{8 + \text{gm of sweat}}{\text{gm of sweat}} = \text{mEq/l}$$

$$\frac{161-40}{383-33} \times \frac{8.10035}{.10035} = \text{mEq/l}$$

$$\frac{121}{350} \times \frac{8.10035}{.10035} = 28 \text{ mEq/l}$$

2. Results should be rounded off to the nearest whole number and the calculations should be reviewed by the supervisor.
3. Compare the bilateral sweat chloride values. They should agree within two standards.
4. Check that the control is within acceptable range, approximately 25-32 mEq/l.

Linearity

To 160 mEq/l. Any patient with a result greater than 160 mEq/l should be reported as greater than 160 mEq/l. Any result less than 1 mEq/l should be reported as less than 1 mEq/l.

Common Conversion Factors

1 PPM = 1mg/l
1 PPM Cl = .0001% Cl 1% NaCl = 0.61% Cl
1 PPM NaCl = .0001% Cl 1% Cl = 1.39% NaCl

mEq Cl/l to % NaCl

$$\% \text{ NaCl} = \frac{\text{(reading) (5.85) (final sample volumes, liters)}}{\text{(sample volume, liters) (density g/l)}}$$

mEq Cl/l to mg NaCl/l = (reading) (58.5)
mEq Cl/l to ppm Cl = (reading) (35.5)

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