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Title: Determination of Salt		
Revision: 03	Replaces: CLG-SLT.02	Effective: 07/27/2009

Contents

A. INTRODUCTION

1. Theory

The sodium chloride content is determined by the well-known Volhard method. The sample is treated with $AgNO_3$ then wet-ashed, and the excess $AgNO_3$ is back titrated with KSCN.

The $AgNO_3$ solution must be added first, followed by the concentrated HNO_3. *This order of addition is critical to ensure complete precipitation of the chlorides.* If HNO_3 is added first, loss of chloride by volatilization as HCl could occur because HCl has a higher vapor pressure than HNO_3.

The volume of $AgNO_3$ solution added must be in excess of that required to react with the chlorides in the sample.

The concentrated solution of $KMnO_4$ is added to oxidize any organic matter not disposed of by the HNO_3. Should too much $KMnO_4$ be accidentally added, the addition of small quantities of sugar or a small piece of filter paper will cause color removal.

Following boiling, cooling, and dilution, back-titrate the excess $AgNO_3$ with KSCN solution, employing ferric ammonium sulfate solution as an indicator.

The $FeNH_4(SO_4)_2$ reacts with an excess of thiocyanate, forming the salmon colored complex, ferric thiocyanate $FeSCN^{++}$, indicating the end point.

NOTE: After all the silver has been back-titrated, an excess of thiocyanate may react with the precipitated AgCl because the solubility product of AgSCN is 1/100 that of AgCl.

$^{s}AgSCN$	1.0×10^{-12}
$^{s}AgCl$	1.1×10^{-10}

The addition of nitrobenzene or diethyl ether overcomes this difficulty by coating the precipitated AgCl, thereby withdrawing it from the action of the thiocyanate solution. If results are rounded to 0.1%, precipitate coating is not needed.

2. Applicability

This method is applicable to the determination of salt in processed meat products at a level ≥ 0.08%.

B. EQUIPMENT

Note: Equivalent apparatus may be substituted

1. Apparatus
   a. Burette - Class A, Kimax
   b. 300 mL Erlenmeyer flasks - Pyrex
   c. 25 mL pipettes - Class A, Pyrex
   d. Boiling chips - Carborundum #12 granules, Cat. No. 133-B, Hengar Co.
   e. Volumetric flask - 1 L, Class A, Kimax
   f. Drying oven - FREAS oven Model 625, Cat. No. 51221139, Precision Scientific

2. Instrumentation

   None

C. REAGENTS AND SOLUTIONS

Note: Equivalent reagents or solutions may be substituted

1. Reagents
   a. Ferric alum indicator-Saturated aqueous solution of reagent grade $FeNH_4(SO_4)_2 \cdot 12 H_2O$ - ACS Grade, Cat. No. 3070, RICCA Chemical Company
   b. Nitric Acid - reagent grade (HNO_3) ACS Grade, Cat. No. 9601, J.T. Baker
   c. Potassium Permanganate ($KMnO_4$) - ACS Grade, Cat. No. 7056, Mallinckrodt
   d. Diethyl Ether - reagent grade, Cat No. 9244, J.T. Baker
   e. Lactose - ACS Grade, Cat. No. 2248, J.T. Baker
   f. Silver Nitrate ($AgNO_3$) - ACS Grade, Cat. No. 2169, Mallinckrodt
   g. Potassium Thiocyanate - ACS Grade, Cat. No. 7168, Mallinckrodt
   h. Potassium Chloride - ACS Grade, Cat. No. 6858, Mallinckrodt
   i. Potassium Chromate - ACS Grade, Cat. No. 6870, Mallinckrodt
   j. Water, distilled or deionized

2. Solutions
   a. Silver Nitrate (0.1000N ± 0.0005N):

      Dissolve 17.04 g of $AgNO_3$ in water in a 1 L volumetric flask. Dilute to volume with water.

b. Potassium thiocyanate (0.1000N ± 0.0005):

Dissolve 9.72 g of reagent grade KSCN in water in a 1 L volumetric flask. Dilute to volume with water.

c. 1:1 Nitric Acid

Add 100 mL of HNO_3 to 100mL of water and mix carefully.

d. 5 % Potassium permanganate solution

Add 100 g of $KMNO_4$ to a 2 L graduated cylinder or volumetric flask. Dilute to volume with water. Mix well. Store in actinic glassware or otherwise protect from light.

e. Potassium Chromate-5 % solution

Add 5 g of K_2CrO_4 to 100 mL volumetric flask. Dilute to volume with water and mix.

3. Standardization of $AgNO_3$ and KSCN

a. Standardize the $AgNO_3$ solution as follows

i. Weigh 0.2500 ± 0.0500 g of KCl that has been dried at 101 °±1 °C for 1 hour ± 10 min into a 250 mL Erlenmeyer flask and dissolve in 40 mL of water.

ii. Add approximately 1 mL of K_2CrO_4 indicator.

iii. Titrate with the $AgNO_3$ solution to a permanent light brown (salmon colored) endpoint.

b. Standardize the KSCN solution as follows

i. Pipette 25 mL of standard $AgNO_3$ solution into a 300 mL Erlenmeyer flask.

ii. Add approximately 80 mL of water.

iii. Add 15 mL of a 1:1 HNO_3.

iv. Add approximately 2 mL of the ferric alum indicator.

v. Titrate with KSCN solution to a permanent light brown (salmon colored) end point. The ratio of the volume of KSCN to the volume of $AgNO_3$ should be 1:1.

D. STANDARDS

None

E. SAMPLE PREPARATION

Process samples until homogeneous.

F. ANALYTICAL PROCEDURE

1. Determination

   a. Weigh 2.5-3.0 g of finely comminuted and thoroughly mixed sample into a 300 mL Erlenmeyer flask. Run a reagent blank and a previously analyzed sample as a recovery with each set of samples.

      Note: For country ham and cured products, weigh out 1.0 to 1.5 g. For "seasoning" samples weigh 10 g into a 100 mL volumetric flask and dilute to volume with water. Pipet a 1.0 mL aliquot into a 300 mL Erlenmeyer flask and record the weight as 0.10 g.

   b. Add 25.0 mL of 0.1000 ± 0.0005 N $AgNO_3$ solution, swirl flask until sample and solution are in intimate contact, and then add 15 mL of conc. HNO_3.

   c. Add sufficient boiling chips and boil until meat digests. Add a small amount of lactose to the reagent blank.

      Note: Solution will turn from a cloudy white color to yellow.

   d. Add $KMnO_4$ solution in small portions while boiling to turn solution dark brown.. Continue boiling until color disappears. Continue adding small portions of $KMnO_4$ until solution retains dark color for several minutes before clearing. Wash sides of flask with water..

      NOTE: If solution retains color and will not become colorless, add a small amount of lactose until color disappears.

   e. Add approximately 25 mL of water**;** boil for approximately 5 min, cool to room temperature in the fume hood, rinse the neck of the flask and dilute to approximately 150 mL with water.

   f. Add approximately 5 mL of diethyl ether (optional), approximately 2 mL of the ferric alum indicator, and swirl to coagulate the precipitated AgCl. (If results are rounded to 0. 1 %, the diethyl ether need not be added.)

   g. Titrate the excess $AgNO_3$ with KSCN solution to a permanent, salmon colored, end point.

      NOTE: If titration with KSCN is less than 2 mL, repeat with a smaller sample weight.

G. CALCULATIONS

1. Procedure

$$\text{Percent NaCl} = \frac{[25.0\ \text{mL} - (\text{mL KSCN})(R)](\text{N } AgNO_3)(5.85)}{\text{Sample Weight}}$$

$$\text{where R = ratio of } \frac{\text{mL } AgNO_3}{\text{mL KSCN}}$$

As determined in C. 3. Standardization of $AgNO_3$ and KSCN

H. SAFETY INFORMATION AND PRECAUTIONS

1. Required Protective Equipment — Safety glasses, heat-resistant gloves, and lab coat.

2. Hazards

Procedure Step	*Hazard*	*Recommended Safe Procedures*
Ferric alum $FeNH_4(SO_4)_2 \cdot 12H_2O$.	May irritate skin, eyes, or respiratory system	Prepare and use in a fume hood.
Nitric Acid - reagent grade (HNO_3)	Will cause severe burns to all body tissue. May be fatal if swallowed or inhaled. Will react with water or steam to produce heat and toxic and corrosive fumes.	Prepare solutions in a fume hood. Store out of direct sunlight. Regulate contact with heat, water, and incompatible materials.
Potassium Permanganate ($KMnO_4$)	Oxidizer, contact with reducing agents or combustibles may cause ignition or extremely violent combustion. Causes burns to all tissue. Toxic metal fumes may form when heated to decomposition.	

Diethyl Ether	Flammable liquid and vapor. After long standing or after exposure to air or light it may form explosive peroxides that are sensitive to mechanical impact and static discharge. Harmful if swallowed, inhaled, or absorbed through the skin. Inhalation of vapors may cause dizziness and unconciousness. May explode on contact with nitric acid.	Use in fume hood. Protect from exposure to air. Do not evaporate to near dryness.
Silver Nitrate ($AgNO_3$)	Corrosive, causes burns to all tissue. May be fatal if swallowed. Oxidizer, contact with reducing agents or combustibles may cause ignition. Reacts with ammonia to form explosive residues.	
Potassium Thiocyanate	Harmful if swallowed or inhaled. Causes irritation to the skin, eyes, and respiratory tract.	
Potassium Chromate	May be harmful if absorbed through skin or swallowed. May cause eye and skin burns. May cause respiratory tract irritation.	
Potassium Chloride	May cause irritation to the skin, eyes, and respiratory tract.	

3. Disposal Procedures

Procedure Step	*Recommended Safe Procedures*
Acid digestion mixture	The acid supernatant after siphoning off can be neutralized and disposed of if the amount of silver in the solution is low enough to meet local ordinances, otherwise the liquid and solid will have to be lab packed for disposal.

I. QUALITY ASSURANCE PLAN

1. Performance Standard

Analyte	*Analytical Range*	*Repeatability Standard Deviation*	*Reproducibility Standard Deviation*
Salt	[1]	<0.16	<0.16[2]

[1]Limit may vary due to sample and aliquot sizes and sample type.

[2]One standard deviation based on historical data.

2. Critical Control Points and Specifications

	Record	*Acceptable Control*
a.	Standard silver nitrate	Record all standardization calculations in "Standards Book" 0.1000 ± 0.0005 N
b.	Standard potassium thiocyanate	Same as above.
c.	Sample size	2.5 - 3.0 g (Smaller samples sizes may be taken for high salt content samples.) Repeat any analysis if KSCN titration is less than 2 mL.
d.	Addition of silver nitrate and nitric acid	Silver nitrate must be added first.

3. Readiness To Perform

   a. Familiarization

      i. Phase I: Standards-Not applicable unless performing reagent standardizations.

      ii. Phase II: Samples previously analyzed with known salt content. Two sets performed on two different days. Six samples each set analyzed in duplicate.

      Note: Phases I and II may be performed concurrently.

      iii. Phase III: 15 check samples for analyst qualification:

         a. Samples submitted by the Quality Assurance Manager (QAM), Accredited Laboratory Program, or supervisor.

         b. Authorization from Quality Assurance Manager (QAM) and supervisor is required to commence official analysis.

b. Acceptability criteria.

Refer to I. 1.

4. Intralaboratory Check Samples

a. System, minimum contents.

Frequency: 1 per week, per analyst, if samples are analyzed.

b. Acceptability criteria.

Refer to I. 1.

If unacceptable values are obtained, then:

i. Stop all official analyses by that analyst.

ii. Take corrective action.

5. Sample Acceptability and Stability

a. Matrix: Fresh and processed meat and poultry products.

b. Sample receipt size, minimum: 1 lb

c. Condition upon receipt: Sealed from air, and unspoiled.

d. Sample storage:

i. Time and Condition -24 months frozen or 1-3 weeks refrigerated.

6. Sample Set

a. Reagent blank

b. Recovery

c. Samples

J. WORKSHEET

Start: **Method: CLG SLT.___** **Analyst / AC:** *

Complete: **Signature:**

Enter data from analysis in the cells below

Enter mLs of $AgNO_3$

Enter mLs of KSCN

Enter Normality of $AgNO_3$

Ratio (R)

$$\frac{\text{mls of } AgNO_3}{\text{mls of KSCN}} = \text{Ratio (R)}$$

ILN #	Serial #	*Sample Weight (g)*	*Titrate start (mL)*	*Titrate stop (mL)*	mLs KSCN =	**% Salt**
Blank						0

Balance		cleaned			
Solutions					Unique ID #
$AgNO_3$ standard solution					
KSCN standard solution					
Conc. HNO_3					
5% Potassium permangenate					
Ferric Ammonium Sulfate					
Brinkman dispensette FSIS ILN#					mLs delivered

$$\% \text{ Salt} = \frac{[25.00 - (\text{mLs KSCN})(R)]\ (\underline{N}\ AgNO_3)\ (5.85)}{\text{Sample weight (g)}}$$

Data Transfer checked by:

QC data checked and entered by:

K. APPENDIX

1. Reference

   Official Methods of Analysis of the Association of Official Analytical Chemists, 15th Edition: 935.47, 941.18.

L. APPROVALS AND AUTHORITIES

1. Approvals on file.

2. Issuing Authority: Director, Laboratory Quality Assurance Division.