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TECHNICAL  
INSPECTION PROCEDURES

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DETERMINATION OF SALT CONTENT  
IN  
PROCESSED FOODS

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FOR USE OF USDA PROCESSED FOODS INSPECTORS

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DECEMBER 1963

TECHNICAL INSPECTION PROCEDURES  
 DETERMINATION OF SALT CONTENT  
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## **FOREWORD**

This instruction will give inspectors a more thorough understanding of analyses for determination of salt content in processed foods.

Follow this instruction when analyzing a processed product for salt content unless specifications or standards cite another method. Slight deviations in preparatory procedures are sometimes necessary for a particular commodity. Be guided by the Inspector's Instructions for the product for such preparatory steps.

## **!! SAFETY FIRST !!**

### For Your Protection

Silver Nitrate ( $\text{AgNO}_3$ ) solutions are employed in titrations for salt determination. To prepare this reagent it is often necessary for you to work with concentrated or crystalline  $\text{AgNO}_3$ . Both are extremely caustic and must be treated as such. Wear protective clothing and gloves so that it cannot possibly contact your skin or eyes. Work only in an adequately ventilated room, or under a ventilated hood, to avoid inhalation of the vapors. If you inadvertently come in direct contact with concentrated or crystalline  $\text{AgNO}_3$ , immediately wash the area with copious amounts of running water.

Dilute (0.1N)  $\text{AgNO}_3$  is not corrosive but will discolor the skin on contact.

Be equally cautious when handling concentrated Nitric Acid. If it should contact your skin, quickly cover with large amounts of sodium bicarbonate or wash with running water.

See File Code 111-A-10 and 111-A-46 for more information concerning the safe handling of corrosive chemicals.

### Glassware and Equipment

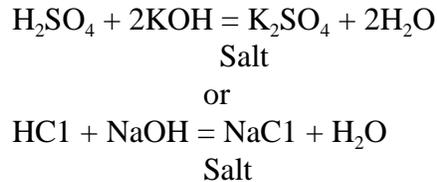
Wash flasks and beakers immediately after each titration or use. Otherwise they will become stained with a residue which is most difficult to remove. Burettes will pick up a dark stain with prolonged use of  $\text{AgNO}_3$  solution. It is not practical to wash a burette after each test or between a series of titrations. However, a good washing at the end of each day will aid in keeping staining to a minimum.

## INSPECTOR'S INSTRUCTION FOR SALT DETERMINATION

- SEPTEMBER 1963 -

### TERMINOLOGY

Chemically speaking, a salt is any of a class of compounds formed when the Hydrogen portion of an acid is partly or wholly replaced by a metal or a metallic radical, as for example -



"Salt" as used in this instruction and in most food standards means "common salt" or sodium chloride (NaCl). It is also customarily referred to as "table salt" although table salt generally contains a few additives and is not chemically pure.

In the analyses set forth in this instruction the total chloride content (that which occurs naturally plus any added by the processor) is ascertained and results are expressed in terms of sodium chloride. These results frequently include several other chloride salts such as potassium chloride and calcium chloride. Because these other salts are usually present in extremely small quantities, the error is insignificant when total chlorides are expressed as sodium chloride.

### NECESSITY FOR SALT DETERMINATION

The amount of salt present in certain foods may be significant because of one or more of the following:

1. Effect upon flavor of the product;
2. Part of the processing technique, such as in cucumber pickles;
3. Economic consideration, either intentional or non-intentional on the part of a processor. For example, the undeclared addition of salt to certain concentrated products, such as tomato puree or paste, will make them seem more highly concentrated than they actually are, when tested by the refractometric method. The salt raises the refractive index of the product and, if not subtracted from the reading, erroneously high results are obtained insofar as tomato solids are concerned; and

4. Dietary aspects, such as low sodium diets. In the case of products for sodium restricted diets the methods and procedures in this instruction are not sufficiently precise to check the sodium level. Most dietary foods have a sodium content of less than 10 mg. per 100 g. and levels of 50 mg. per 100 g. are uncommon, except possibly in tuna or salmon. Therefore, special methods, which are capable of separating levels of 1 mg. magnitude, are used on products declared for dietary (sodium restricted) use.

## METHODOLOGY AND REAGENTS

Various methods are available for determining the salt content of food products. However, the most practical and common methods used involve titration - either with a visual indicator or by a potentiometer.

Of the titration methods the following are in common usage for salt determinations:

1. Visual Indicator Method
  - (a) Mohr method
  - (b) Volhard method
2. Potentiometer (pH Meter) Method

Regardless of the method used, the reaction depends upon precipitation of all the chloride as  $\text{AgCl}$  and detecting the end point either by a color change or observation of electromotive force (meter reading).

The preparation and standardization of reagents are very important in whichever method is used. The following methods and procedures will be helpful in preparing and standardizing the reagents applicable to the particular method.

### $\text{AgNO}_3$

$\text{AgNO}_3$  is unstable in the presence of light. It should be kept in a dark brown glass container and stored in a cool, dark place. As with all reagents, once a portion of the  $\text{AgNO}_3$  has been transferred to another container, unused quantities must not be returned to the master container because of the possibility of strength loss or adulteration. Because it tends to weaken with time, the normality of  $\text{AgNO}_3$  should be periodically checked against a known saline solution as indicated herein.

1. Preparation

Dissolve 16.99 grams of C.P.  $\text{AgNO}_3$  in distilled water and make up to 1 liter. This should give a solution that is very slightly stronger than 1/10 normal. Any opalescence in the final solution is due to traces of a chloride, or to unclean glassware.

## 2. Standardization

Measure out exactly 5.845 grams of reagent grade NaCl. Make up to 1000 ml. volume with distilled water in a volumetric flask. This will produce a salt solution of exactly 0.1 Normality. Take a measured amount of the salt solution (25 to 40 ml.), add potassium chromate indicator, and titrate to the end point. The addition of calcium carbonate is not necessary (no acidity present) but may assist in detecting the end point. For those not thoroughly familiar with the proper end point, a procedure as follows will be very helpful. Place 40-50 ml. of distilled water in a titration flask. Add 2 ml. of potassium chromate indicator and 0.5 grams calcium carbonate. To this solution add 1 drop of N/10 AgNO<sub>3</sub>. A slight yellow-orange color should develop which represents the proper silver-chromate end point.

Calculate the normality of the AgNO<sub>3</sub> solution by the formula -  $V_1N_1 = V_2N_2$

### Example

35.00 ml. of a standard (exactly N/10) NaCl solution requires 34.20 ml. of AgNO<sub>3</sub> to reach the end point. Using the above cited formula -

$$(35.00)(0.10) = 34.20(X)$$

$$3.50 = 34.2X$$

$$0.1023 = X$$

Therefore the Normality of the AgNO<sub>3</sub> reagent is 0.1023.

In using the reagent AgNO<sub>3</sub> it is convenient to multiply the actual titer by a factor to convert to equivalent N/10 strength. In the above example the factor is 1.023.

### Example

40.21 ml. of 0.1023 N AgNO<sub>3</sub> used in titration. Convert to exactly N/10 AgNO<sub>3</sub> by -

$$(40.21)(1.023) \text{ or } 41.13 \text{ ml. equivalent N/10 AgNO}_3.$$

### 1/ NH<sub>4</sub>CNS (Ammonium Thiocyanate)

This reagent is needed for the Volhard Method and forms a pale rose to brown color when slight excess is added to a standard AgNO<sub>3</sub> solution which is acidified with nitric acid and in the presence of ferric alum indicator.

## 1. Preparation

Dissolve 7.6 grams of reagent grade (C.P.) Ammonium Thiocyanate in about 500 ml. of distilled water. Transfer to 1000 ml. flask and make up to mark.

## 2. Standardization

To a 250 ml. titration flask add 40 ml. of N/10  $\text{AgNO}_3$ , 5 ml. of  $\text{HNO}_3$  (1 + 1) and 1 ml. of ferric alum indicator. Titrate with the  $\text{NH}_4\text{CNS}$  solution (about 40 ml.) to pale rose, or tinge of brown end point. Calculate the Normality of the thiocyanate solution by the formula  $V_1N_1 = V_2N_2$  similar to the example under  $\text{AgNO}_3$ . In this case the exact Normality of the  $\text{AgNO}_3$  solution must be known.

### 1/ $\text{HNO}_3$ (1 + 1)

Prepare by combining equal volumes of concentrated  $\text{HNO}_3$  and distilled water. As with all acids, add the acid to the water. For safety, add the acid rather slowly.

### 1/ $\text{HNO}_3$ (2 percent)

Prepare by combining concentrated  $\text{HNO}_3$  and distilled water in the ratio of 3 ml.  $\text{HNO}_3$  per 197 ml. water.

### Distilled Water

As a matter of good laboratory technique always use distilled water. Tap water from some sources contains substantial amounts of chloride and would contribute to erroneously high results:

### $\text{CaCO}_3$ (Calcium Carbonate)

Finely ground, reagent grade.

### $\text{K}_2\text{CrO}_4$ (Potassium Chromate Indicator)

Dissolve 5 grams of  $\text{K}_2\text{CrO}_4$  in water and make up to 100 ml.

### 1/ $\text{NH}_4\text{CNS}$ (Ammonium Thiocyanate)

0.10N - Adjust and standardize by titrating against N/10  $\text{AgNO}_3$ .

### 1/ Ferric Indicator

Saturated solution of  $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}$

### 1/ Filter Aid

Celite or diatomaceous earth.

### 1/ Volhard Method Only.

Two alternate visual indicator methods are available for determination of salt in foods:

A. Mohr Method

This determination is generally simpler, easier, and faster than Volhard's method. It is the official A.O.A.C. method for determining salt content of most foods and is particularly suited for products which are not highly colored so as to interfere with the end point.

1. Equipment and Reagents:

Standard titration burette with stand (See Plate No. 1)

Erlenmeyer flask (See Plate No. 2)

Pipette, if results expressed in terms of grams per 100 ml.  
(See Plate No. 3)

Analytical Balance, if results expressed in terms of percent by weight. (See Plate No. 4)

Distilled Water

$\text{AgNO}_3$  (Silver Nitrate), reagent grade of appropriate Normality. (Generally N/10)

$\text{CaCO}_3$  (Calcium Carbonate) for acid products.

$\text{K}_2\text{CrO}_4$  (Potassium Chromate) indicator

See Page 7 for illustrations - Plates 1 through 4

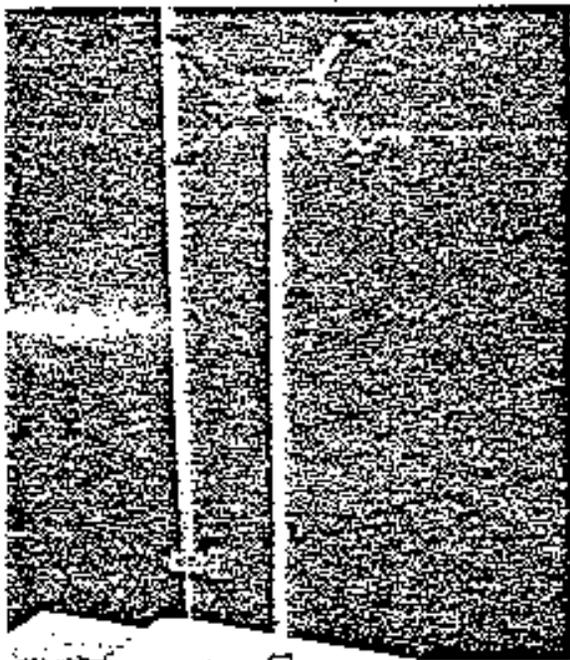


PLATE NO. 1 - Standard Titration Burette with stand.

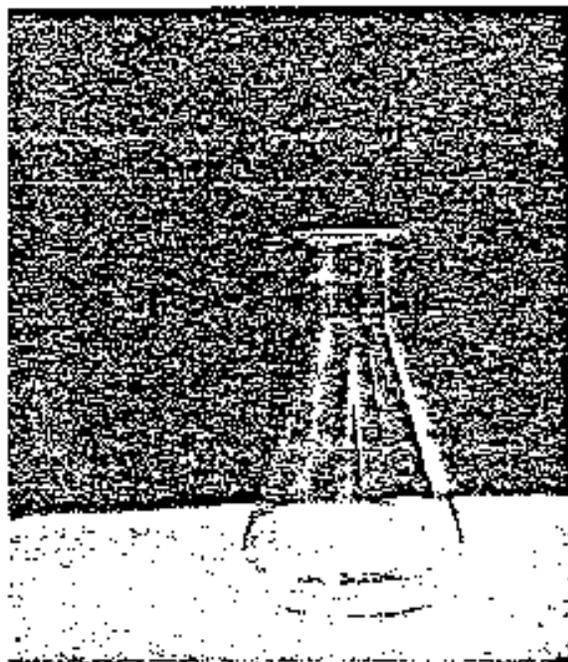


PLATE NO. 2 - Erlenmeyer Flask (250 ml.).

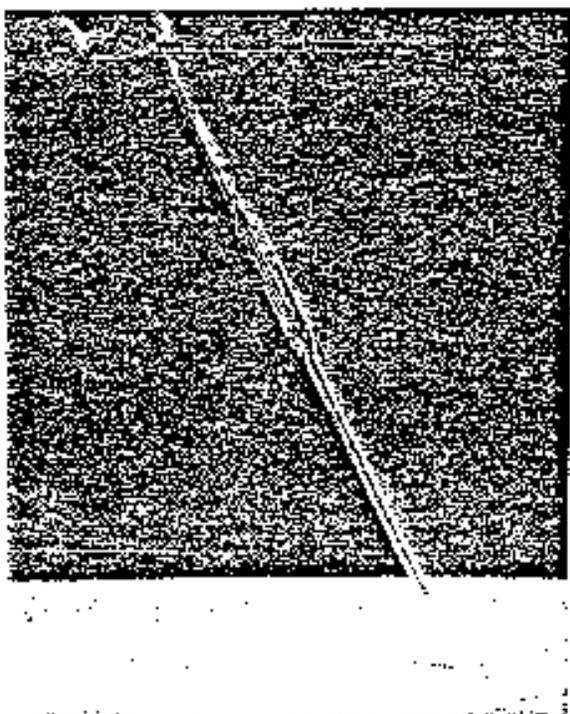


PLATE NO. 3 - Pipette (10 ml.).

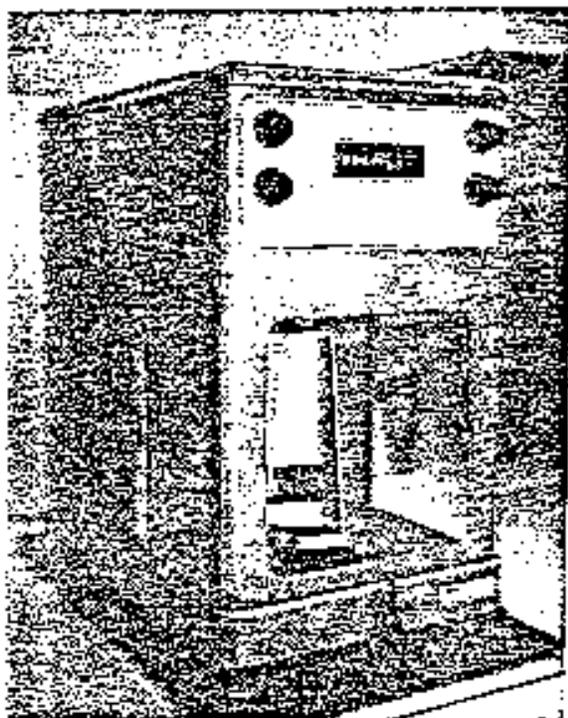


PLATE NO. 4 - Analytical Balance.

## 2. Explanation of Chemical Reaction

The addition of standard  $\text{AgNO}_3$  to a sample solution, using  $\text{K}_2\text{CrO}_4$  as the visual indicator, yields an insoluble precipitate which is proportional to the amount of total chlorides in the solution. To reduce experimental error, a sample of adequate size should be used so that at least 20 ml. of  $\text{AgNO}_3$  are required to precipitate all the chlorides. As mentioned earlier, any naturally occurring acidity must first be neutralized (using  $\text{CaCO}_3$ ); otherwise no visual end point may be attained. The red-colored silver chromate complex, formed by the combination of  $\text{AgNO}_3$  and  $\text{K}_2\text{CrO}_4$ , is soluble in acid and loses its color. The salt content of the sample may be calculated from the volume of standard  $\text{AgNO}_3$  used to reach the end point. The chemical reaction is:  $\text{AgNO}_3 + \text{NaCl} \rightarrow \text{NaNO}_3 + \text{AgCl}$ .

When all the  $\text{AgCl}$  (silver chloride) has been precipitated, a yellow-orange color appears which denotes the end point. This color is the result of the formation of a second precipitate ( $\text{Ag}_2\text{CrO}_4$ ) as shown by the following equation  $2\text{AgNO}_3 + \text{K}_2\text{CrO}_4 \rightarrow \text{Ag}_2\text{CrO}_4 + 2\text{KNO}_3$ .

Certain dyes of the fluorescein series may also be used as indicators. They may be used in slightly acid solutions (pH 4.4 or higher) and give more accurate results with the more dilute solutions (0.05N or less). Dichlorofluorescein is the most widely used of these days.

## 3. Procedure

- a. Fill the clean burette with N/10  $\text{AgNO}_3$ . Always read the burette scale with reference to the bottom of the meniscus of the  $\text{AgNO}_3$ .
- b. Put the prescribed amount of sample into the Erlenmeyer flask.
- c. Dilute with about 25 ml. of distilled water. This increases the volume of solution in the flask so that better agitation can be obtained and the end point easier to detect.
- d. Neutralize any naturally occurring acidity, such as in pickles and sauerkraut. Addition of about 0.5 grams of powdered  $\text{CaCO}_3$  (calcium carbonate) is usually sufficient and the simplest method of neutralization. This need not be weighed and can be estimated by using the tip of a spatula.
- e. Add approximately 2 ml. (4 to 5 drops) of  $\text{K}_2\text{CrO}_4$  indicator.
- f. Titrate with  $\text{AgNO}_3$  to the characteristic yellow-orange end point of the chromate indicator (See Plate No. 5). Inspection Aid No. 48, obtainable from the Area Officer-in-Charge, contains a color photograph of the end point color for salt titrations using  $\text{K}_2\text{CrO}_4$  as the indicator.



PLATE NO. 5 - Titration technique utilized for the Visual Indicator Method of salt determination.

#### 4. Calculations

After determining the amount of  $\text{AgNO}_3$  (to the nearest one-tenth ml.) necessary to exactly reach the end point, calculate the salt content according to the following formula:

$$S = \frac{(T) (N) (0.05845) (100)}{V}$$

S = Salt Content

T = Ml. of  $\text{AgNO}_3$  required to reach the end point

N = Normality of the  $\text{AgNO}_3$

V = Sample size - either in ml. or grams depending upon the terms in which the results are expressed.

0.05845 = the factor necessary to convert the titration to NaCl and represents the number of grams of NaCl which will completely react with one ml. of Normal AgNO<sub>3</sub>.

100 = the factor necessary to convert the results into percent by weight or volume.

### Example

10 ml. of pickle juice requires 41.7 ml. of exactly N/10 AgNO<sub>3</sub> to reach the proper end point. The percent salt is calculated as follows:

$$S = \frac{(41.7) (0.10) (0.05845) (100)}{10} = 2.44$$

Therefore Salt content is 2.44 grams per 100 ml. of pickle juice.

A handy mathematical shortcut may be used when many samples are being checked, if the size of sample and normality of AgNO<sub>3</sub> remain constant. Merely establish a mathematical constant by pre-calculating the following portion of the formula:  $\frac{(N) (0.05845) (100)}{V}$

V

The following example will illustrate the use of the above formula:

You have several samples of pickle juice to titrate for salt. You know from previous experience that 0.1N AgNO<sub>3</sub> used in conjunction with a sample size of 10 ml. of pickle juice provides a convenient working range with the burette. Therefore, you decide to keep them constant as follows:

$$N = 0.1$$

$$V = 10 \text{ ml.}$$

The formula is precalculated:

$$\frac{(0.1) (0.05845) (100)}{10} = 0.05845$$

Therefore the factor of 0.05845 is multiplied by the amount of AgNO<sub>3</sub> used during each titration to obtain the amount of salt in the samples.

Assume the first sample required 28.1 ml. of N/10 AgNO<sub>3</sub>; then the conversion is merely (28.1) (0.05845) or 1.65 grams per 100 ml. of product. All subsequent samples would be calculated in a like manner.

This method is more complicated and time consuming than Mohr's method. However, the end point is easier to discern, especially in intensely-colored products.

1. Equipment and Reagents

Those specified for Mohr's method, except for  $K_2CrO_4$  indicator and  $CaCO_3$ .

$HNO_3$  (1 + 1) -- Netric Acid

$HNO_3$  (2 percent) -- Dilute Nitric Acid

Filtering aid (such as Celite or diatomaceous earth)

Ferric Alum indicator

$NH_4CNS$  (Ammonium Thiocyanate) of same normality as  $AgNO_3$  used, preferably 0.1N.

Filter paper, Buchner funnel and vacuum source

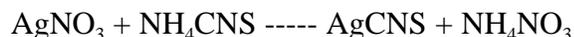
Beaker of appropriate size (normally 250 ml.)

2. Chemical Reaction

The Volhard method is considerably more detailed than the Mohr method but is used to good advantage on products which have a color or other characteristics that interfere with detecting the chromate end point of the Mohr method. Examples are Worcestershire sauce and peanut butter - products which do not lend themselves to the Mohr end point.

In the Volhard procedure an excess of standard  $AgNO_3$  is added to the prepared sample and all of the chlorides are precipitated. The solution is then filtered to remove the precipitate and all suspended matter. The beaker in which the sample is treated as well as the filter must be thoroughly washed with dilute  $HNO_3$  to remove all the  $AgNO_3$ . The filtrate, which must be acidified with  $HNO_3$ , is back titrated with  $NH_4CNS$  to the point at which all of the excess  $AgNO_3$  has reacted with the thiocyanate solution. The end point (brown tinge) is detected by using ferric alum as an indicator. By subtracting the volume of thiocyanate from that of the silver nitrate the volume of silver nitrate used in the reaction can be readily ascertained and the results converted to  $NaCl$ .

The chemical reactions in the procedure are as follows:



In this equation the excess  $AgNO_3$  reacts with  $NH_4CNS$  to form an insoluble precipitate (in acid solution) of  $AgCNS$ . As soon as all of the  $Ag$  ion has reacted with the  $CNS$  ion any excess  $NH_4CNS$  reacts with the ferric alum indicator to form a brown complex which imparts a reddish-

brown tinge to the solution. This tinge of brown color represents the end point. The reaction with the ferric alum indicator to form a reddish-brown color complex is as follows:



(brown tinge)

In the Volhard method it is necessary to filter off the precipitated silver chloride since it is more soluble than silver thiocyanate and would react with  $\text{NH}_4\text{CNS}$  resulting in a higher back titration which in turn would mean less  $\text{AgNO}_3$  and consequently low results. Filtering also removes suspended matters that might be in the product being tested and thereby "Clean up" the filtrate for improved back titration.

### 3. Procedure

- a. Place measured sample in Erlenmeyer flask
- b. Add 5 ml. of  $\text{HNO}_3$  (1 + 1)
- c. Add 50 ml. distilled water and about 1/2 gram of filtering aid
- d. Add an excess (usually 30-40 ml.) of N/10 of  $\text{AgNO}_3$
- e. Stopper flask and shake vigorously
- f. Allow to stand for a few minutes
- g. Filter through rapid filter paper in Buchner funnel (See Plate No. 6). Wash with dilute  $\text{HNO}_3$  (2 percent) and place filtrate in beaker
- h. Add 5 ml. of Fe Alum indicator
- i. Titrate filtrate to a permanent, faint brownish tinge using standard N/10  $\text{NH}_4\text{CNS}$ .

### 4. Calculations

Subtract the amount of N/10  $\text{NH}_4\text{CNS}$  used in step i, above, from the amount of N/10  $\text{AgNO}_3$  used in step d., above. The remainder represents "T" in the mathematical formula described earlier under the Mohr method. Use the same formula to calculate salt for Volhard's method of titration.



PLATE NO. 6 - Filtering the treated sample, a necessary step in Volhard's method.

Example

To a 10.00 gram sample of product 40.00 ml. N/10 AgNO<sub>3</sub> is added.  
The filtrate requires 10.2 ml. N/10 NH<sub>4</sub>CNS to reach the end point.

$$\text{N/10 AgNO}_3 \text{ in reaction} = 40.0 - 10.2 = 29.8$$

$$\text{S} = \frac{(29.8) (0.10) (0.05845) (100)}{10}$$

$$\text{S} = 1.74 \text{ Percent}$$

## POTENTIOMETER (pH METER) METHOD

This method is superior to visual titration methods whenever there are interfering substances in the product that make it difficult to visually observe the correct end point. For example, the intense red color of a tomato product masks the yellow-orange end point of potassium chromate. In peanut butter the potentiometer method has been very effective because of accuracy and speed.

There are many makes and models of potentiometers in use. They may vary somewhat in location of electrodes and controls but the operating principle is the same (See Plate No. 7). This instruction, together with the operating manual for any particular potentiometer, should be sufficient to determine salt content of foods.

A pH meter is primarily designed for measurement of acidity but, with the proper substitution of electrodes, it easily accommodates salt determinations.

### A. Discussion

The electrochemical reaction between the electrodes and the sample is a form of electrolysis. The calomel electrode contains potassium chloride (KCL); the sample, containing sodium chloride acts as the electrolyte; and the silver electrode contains silver (Ag) ions. The millivolt scale on the meter reflects continuing changes in electromotive force (EMF) between the two electrodes as  $\text{AgNO}_3$  is added from the burette. After all the NaCl in the sample is chemically combined with  $\text{AgNO}_3$  (added from the burette) and there is a very slight excess of  $\text{AgNO}_3$ , the meter will read 0.28 millivolts. This meter reading corresponds with the greatest change in EMF per added volume of  $\text{AgNO}_3$ .

### B. Equipment and Reagents

$\text{AgNO}_3$  N/10

Distilled Water

Burette and Stand

250 ml. Beaker

Glass electrode pH meter or potentiometer. Any meter capable of reading voltage changes of 0.01 volts, or less, may be used. Any of the standard makes of pH meters are suitable.

Calomel electrode for above meter (See Plate No. 8)

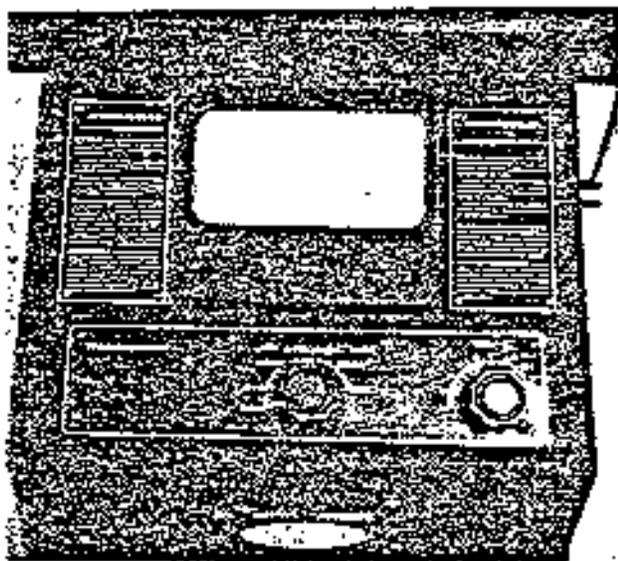


PLATE NO. 7 - A typical potentiometer, often used for measurement of acid and salt contents of foods.

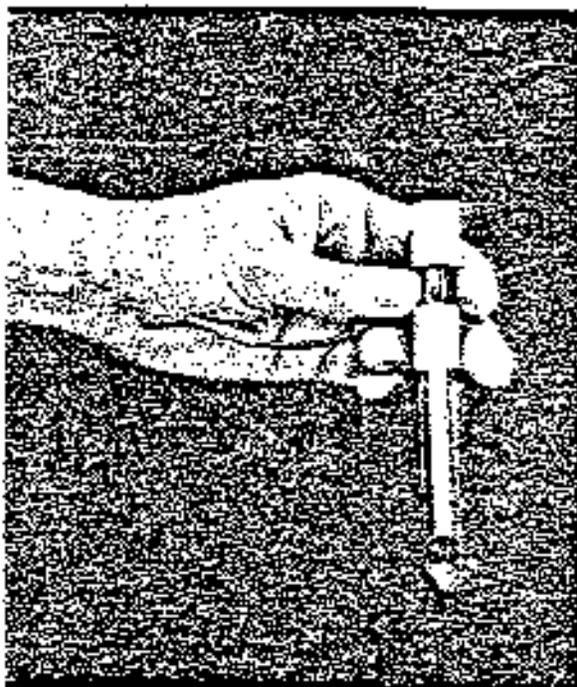


PLATE NO. 8 - Calomel electrode.

Silver electrode for above meter (See Plate No. 9)

Magnetic Stirrer (See Plate No. 10)

#### C. Electrode Attachment

Using a jack adapter, connect the calomel electrode to the proper terminal on the meter (See Plate No. 11). Only one terminal will accept the jack adapter. Connect the silver electrode to the other terminal on the meter.

#### D. Procedure

1. Place sample in beaker. Sample size should be sufficient to require 20-40 ml. N/10  $\text{AgNO}_3$ . On most food products a 5 to 10 gram sample is sufficient.
2. Add sufficient distilled water so that the electrodes will be submerged to a depth of about 1 inch.
3. Determine temperature of sample/water mixture. Set temperature knob on meter.
4. Submerge both electrodes in sample solution to a depth of about one inch. Add more water, if necessary.

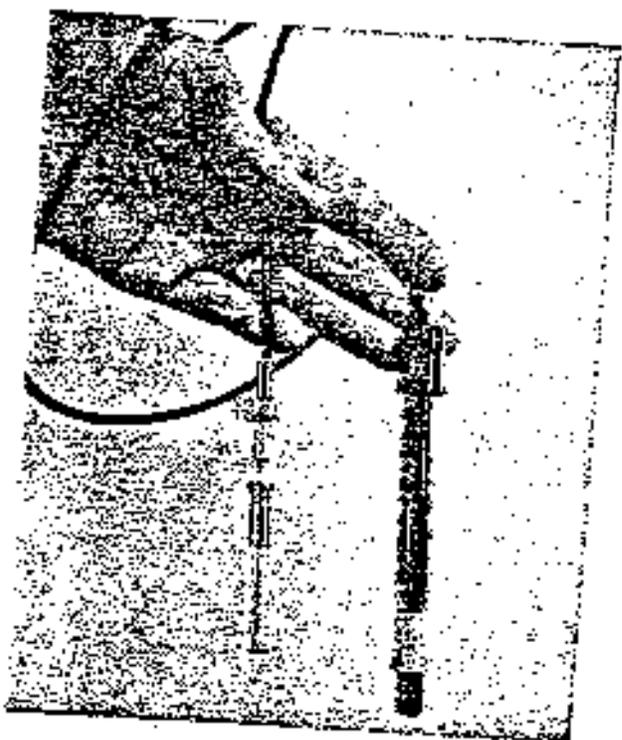


PLATE NO. 9 - Silver electrode with jack adapter.

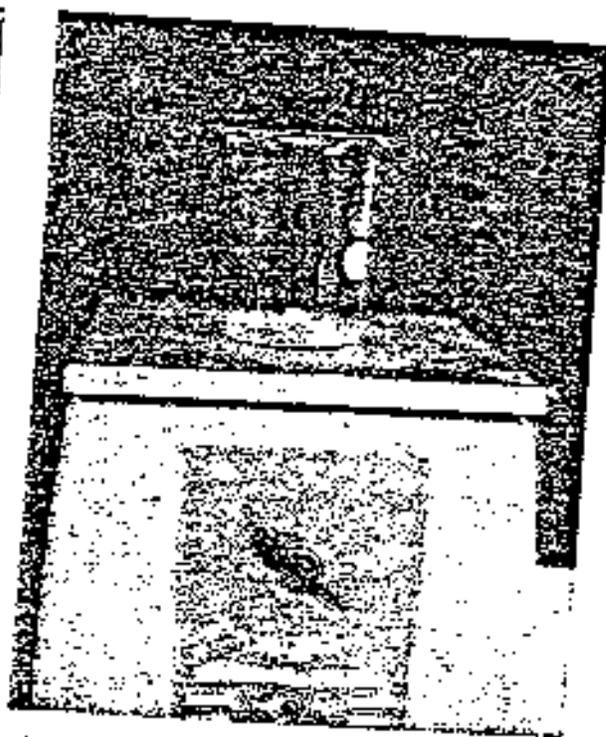


PLATE NO. 10 - Magnetic stirrer. Note the magnetized agitator inside the beaker.

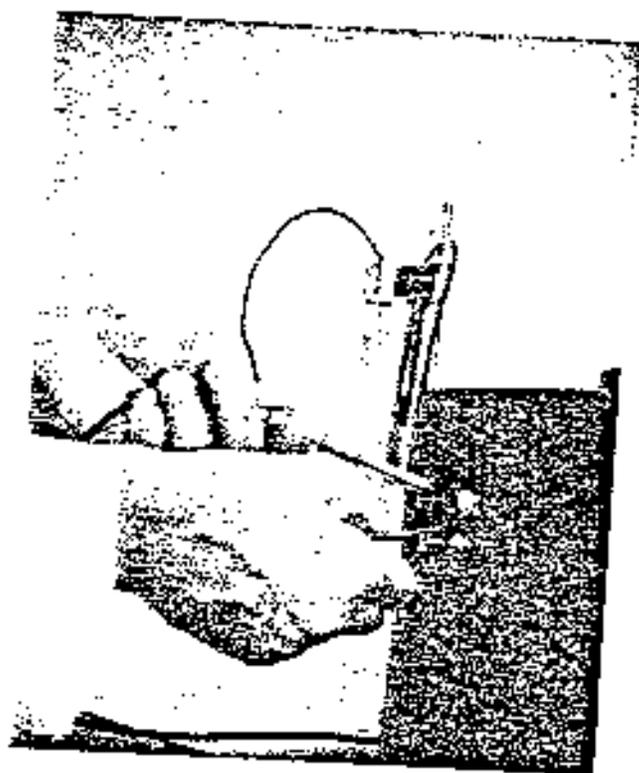


PLATE NO. 11 - Connecting the calomel and silver electrodes to the terminals at rear of potentiometer.

5. Turn potentiometer switch to "on" position. Turn Meter Range switch to "zero". Bring meter needle to 0.00 millivolt reading, by means of the Standardization knob (this reading is normally found at the right-end of the meter scale).
6. Turn Range switch to proper millivolt scale (if meter has a multi-range scale).
7. Add  $\text{AgNO}_3$  to sample from burette stirring vigorously with magnetic stirrer. The  $\text{AgNO}_3$  may be added rapidly until a meter reading of from 0.15 to 0.20 is obtained, then slowly until 0.28 (the end point) is attained.
8. Record the amount of  $\text{AgNO}_3$  used.
9. Turn potentiometer switch to "off" position and remove electrode from sample. Rinse electrodes with distilled water and wipe dry. The surfaces of the electrodes must be thoroughly cleaned after each determination and should not be allowed to stand in a solution after the analyses.

E. Calculations

The same mathematical formula is used as in the Mohr and Volhard method, using the amount of  $\text{AgNO}_3$  used in steps 7 and 8 for the factor T.