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Factory Tests for Dairy Products

By

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COOPERATIVE EXTENSION SERVICE IN AGRICULTURE AND HOME ECONOMICS OF THE STATE OF IDAHO UNIVERSITY OF IDAHO EXTENSION DIVISION AND UNITED STATES DEPARTMENT OF AGRICULTURE COOPERATING

DAIRY SECTION

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THIS BULLETIN makes available to the operators of dairy manufacturing plants the latest laboratory methods of analysis of practical use in production and quality control of dairy products.

The tests described have been selected because they are the most commonly needed and applicable to factory control work. The procedures recommended are either the official methods or the most reliable, accepted methods.

Equipment necessary for performing the various tests can be purchased from any dairy supply company.

Babcock Test for Butterfat

The Babcock test was devised by Dr. S. M. Babcock, Chief Chemist of the Wisconsin Agricultural Experiment Station and is based upon chemical action and centrifugal force.

Sampling Milk and Cream

The accuracy of the Babcock Test depends upon the care used in sampling.

A. Milk. Normal fresh milk should be sampled immediately after it has been poured from one container to another several times. A uniform sample may be obtained after thorough stirring. If a cream layer has formed, the milk should be warmed to 60° to 70° F. and agitated in such manner as to prevent churning.

B. Cream. Cream is thicker and more viscous than milk and is therefore more difficult to sample. The cream should be thoroughly stirred and immediately sampled. A slotted cream sampler is recommended.

C. Abnormal Milk and Cream.

1. Sour Milk. It is almost impossible to accurately sample sour milk. When a sample is to be taken, the milk should be thoroughly stirred and sampled immediately.

2. Sour Cream. An accurate sample may be obtained after the cream is thoroughly stirred.

3. Churned Milk or Cream. Milk or cream containing particles of butter should be warmed to 105° to 115° F. When butter granules have melted, the product should be poured back and forth several

times and sampled as rapidly as possible. It is very difficult to obtain duplicate samples that will check.

4. Frozen Milk and Cream. An accurate sample cannot be taken from the frozen material. Place the milk and cream in a water bath at 80° to 85° F. until it thaws. Mix well and sample as usual. If some of the fat oils off during melting, the milk or cream should be poured back and forth several times from one vessel to another and sampled immediately.

D. Composite Sample. A composite milk sample is one which, when properly prepared, represents two or more lots of milk. A proportionate part of each patron's milk is taken each day with a sampling tube or dipper and placed in an airtight bottle containing a preservative (corrosive sublimate). Composite samples should be stored at 40° F., but measured for testing at 70° F. Samples should be tested for fat about every week or 10 days.

Table 1.—Determining the percentage of butterfat in dairy products by the Babcock Method[1]

Product	Babcock bottle recommended	Amount of sample used	Amount H_2SO_4[6] added ml.	Centrifuge (minutes)[5] 1st run	2nd run	3rd run	Temperature of water bath °F.	Remain in water bath (min.)	Glymol added	Read
Milk	8% milk	17.5 ml.	17.5	5	2	1	135 to 140	5	Not used	Lower extreme of lower meniscus to upper extreme of upper meniscus
Cream	6½" 9g. 50% 9" 9g. 50% 9" 18g. 50%	9 gm.[2] 9 gm. 18 gm.	8 to 12[5]	5	2	1	135 to 140	5	Few drops	Bottom of lower meniscus to line between fat and glymol
Skim milk	Skim milk	17.5 ml.	18 to 20	10	2	1	135 to 140	5	Not used	Lowest to highest point of the fat column
Buttermilk[4]										
Whey	Skim milk	17.5 ml.	13 to 17	10	2	1	135 to 140	5	Not used	Lowest to highest point of the fat column
Cheese	6½" 9g. 50%	9 gm.[5]	17.5	5	2	1	135 to 140	5	Few drops	Bottom of lower meniscus to line between fat and glymol
Butter	6½" 9g. 50%	4.5 gm.[7]	14	5	2	1	135 to 140	5	Few drops	Read as directed for cream. Multiply reading by 2

[1]All glassware used must have been inspected, tested, and approved by the College of Agriculture of the University of Idaho. Such glassware is stamped with the letters "S.G.I."

[2]Cream is weighed—Balance should have a sensitivity not in excess of 30 milligrams at full load.

[3]An alternate method of centrifuging is as follows: After the cream and acid have been thoroughly mixed and all lumps have completely disappeared, add a few milliliters (not less than 5ml.) of hot, soft water; whirl 5 minutes, add hot, soft water to near top of graduated mark on the neck, and whirl 1 minute.

[4]Use one of the modified Babcock methods.

[5]Obtain a number of small portions from various parts of the cheese. Cut fine, grate or grind the portions so that the cheese can be well mixed. Work quickly and protect the cheese from the air as much as possible to avoid loss of moisture. After cheese is weighed into the cream bottle, add 12 to 15 ml. of water at a temperature of 160° to 170° F., mix well with the cheese. Add the acid in several portions, shaking the bottle after each addition of acid. Leave the bottle standing until all cheese particles are completely dissolved.

[6]Commercial sulphuric acid having a specific gravity of 1.82 to 1.83 at 68° F. should be used in making the test.

[7]Weigh a 4.5 gm. sample into a 9 gm. 50 per cent cream test bottle previously balanced on an accurate scale. Add 14 ml. of warm water and mix thoroughly.

[8]After first run add sufficient soft water at 135° to 140° F. to bring the level of the contents to within ½ inch of the lower end of the graduated neck. After second run fill to nearly the top of the graduated column with soft water at 135° to 140° F.

Table 2.—Modifications of the Babcock test for determining the percentage of butterfat in dairy products.

Product	Babcock bottle recommended[1]	Amount of sample used	Reagents added ml.	Digestion		Centrifuge minutes			Temperature of water bath °F	Time in water bath mins.	Glymol	Read[9]
				Temperature °F.	Time minutes	1st run	2nd run	3rd run				
Ice cream	8% milk	9 gm.	13 ml. glacial acetic acid 9 ml. sulfuric acid[2]	170	5	5	2	1	135-140	5	None	S.A.M. x 2
Ice cream	8% milk	9 gm.	5 ml. reagent A[3] 30 ml. reagent B	175 to 180	15	5	3	1	135-140	15	Few drops	S.A.C. x 2
Ice cream	8% milk	9 gm.	2 ml. ammonium hydroxide[4] 3 ml. N. butyl alcohol 17.5 ml. dil. sulfuric acid	not used	not used	5	2	2	130	5	Few drops	S.A.C. x 2
Ice cream	8% milk	9 gm.	15 ml. of Minnesota reagent[5]	180	10 to 15	1	½		120	5	None	S.A.M. x 2
Ice cream	8% milk	9 gm.	2.5 ml. reagent A[6] 9 ml. reagent B	212	15 to 30	5	2	1	130-140	5	None	S.A.M. x 2
Sweetened Condensed milk	8% milk	9 gm. 50% dil.	Use Swope reagents as for ice cream	not used	not used	5	2	2	130	5	Few drops	S.A.M. x 4
Evaporated milk	20%9 gm ice cream	6 gm.	Swope reagents as for ice cream	not used	not used	5	2	2	130	5	Few drops	S.A.C.x1.5
Evaporated milk	20%9 gm. ice cream	9 gm.	10 ml. Minnesota reagent[5]	180	5	1	½		130	5	None	S.A.M.
Powdered milk	8% milk	18 gm.[7]	14 to 16 ml. com. sulfuric acid	not used	not used	5	3	1	135-145	5	None	S.A.M. x 8
Buttermilk	Milk or skim milk	9 gm.	10 ml. Minnesota reagent	180	5	1	½		130	5	None	S.A.M. x 2
Buttermilk	Milk or skim milk	9 gm.	2 ml. of N. butyl alcohol 7-10 ml. of sulfuric acid[8]	not used	not used	6	2	2	135-140	5	None	S.A.M. x 2
Chocolate milk	8% milk	18 gm.	2 ml. ammonium hydroxide 3 ml. N. butyl alcohol 15 to 17.5 ml. dil. H_2SO_4[4]	not used	not used	5	2	2	130	5	None	S.A.M.

[1]All glassware used must have been inspected, tested, and approved by the College of Agriculture of the University of Idaho. Such glassware is stamped with letters "S.G.I."

[2]Add the glacial acetic acid and agitate thoroughly. Then add sulfuric acid (Sp. Gr. 1.83) and shake thoroughly. Place bottles in the water bath and digest for 5 minutes. Complete test as for milk.

[3]Reagent A. 9 parts N-butyl alcohol and 1 part by volume of C.P. ammonium hydroxide (Crowe).

Reagent B. Equal parts by volume of sulfuric acid (Sp. Gr. 1.82-1.83) and ethyl alcohol (95%) (Crowe). Weigh out ice cream mix, then add reagent A. Mix thoroughly. Add reagent B. Mix thoroughly. Shake bottle at least 3 times during digestion.

[4]Ammonium hydroxide (28 to 29% NH_3)—Normal butyl alcohol (b/p. 117° C.). Dilute by volume by adding 3½ parts of acid to 1 part of water. Cool. Shake after addition of each reagent (Swope).

[5]100 gm. of sodium carbonate (mono-hydrate) and 200 gm. of sodium salicylate are made up to 1000 ml. with water. To the above, add 30 ml. of 50% sodium hydroxide and 100 ml. of N-butyl alcohol. Shake bottles once or twice during the digestion period.

[6]Reagent A. 75 ml. of C.P. ammonium hyroxide, 35 ml. of N-butyl alcohol and 15 ml. of 95 per cent ethyl alcohol (Overman-Garrett).

Reagent B. 200 gm. of tri-sodium phosphate (commercial grade) and 150 gm. of sodium acetate (commercial grade) make up to 1 liter. (Overman-Garrett.)

Mix thoroughly after addition of each reagent. Digest in boiling water bath until fat separates and forms a clear layer on top of the liquid.

[7]20 gm. of milk powder dissolved in 140 gm. of water at 125° F.

[8]Place 2 ml. of N-butyl alcohol in the test bottle. Add 9 gm. of buttermilk. Mix thoroughly. When testing, use both skim milk and whole milk test bottles, as there is often too much fat to be read in a skim milk bottle.

[9]S.A.M. = Same as milk. S.A.C. = Same as cream.

Table 3.—Determination of titratable acidity

Product	Amount of sample	Dilution	Amount of indicator[1]	Procedure	Read
Milk	9 ml.	None	3 to 4 drops	Add slowly 0.1N sodium hyroxide[2] until first definite and relative permanent shade of pink has been reached[3]	Ml. of 0.1N sodium hydroxide times 0.1 equals "percent lactic acid"
Skim milk	9 ml.	None	3 to 4 drops	Same as for milk	Same as for milk
Buttermilk	9 ml.	None	3 to 4 drops	Same as for milk	Same as for milk
Whey	9 ml.	None	3 to 4 drops	Same as for milk	Same as for milk
Cream	9 gm.[4]	None	6 drops	Same as for milk	Same as for milk
Cream	9 ml.	9 ml.[5]	6 drops	Same as for milk	Same as for milk
Condensed milk products	9 ml.	9 ml.[5]	4 drops	Same as for milk	Same as for milk
Ice cream mixes	9 ml.	9 ml.[5]	8 drops	Same as for milk	Same as for milk

[1]1 per cent alcoholic solution of phenolphthalein.

[2]Preparation of 0.1N sodium hydroxide solution.

a. Prepare a stock solution of NaOH by dissolving 400 grams in 400 ml. of recently boiled distilled water.
b. Allow solution to cool and store in a stoppered bottle for several days.
c. Decant the clear supernatant liquid into another clean bottle.
d. 7 ml. of the clear concentrated solution made up to 1000 ml. with recently boiled distilled water should yield a solution that is approximately 0.1N.
e. To standardize the solution to exactly 0.1N weigh out 10.2071 gm. potassium acid phthalate and make up to 500 ml. with recently boiled distilled water.
f. Pipette 50 ml. of the potassium acid phthalate solution into a beaker. Add 0.5 ml. of indicator (1 per cent alcoholic solution of phenolphthalene) and titrate to first definite and relative permanent shade of pink.
g. Normality of unknown solution $= \frac{0.1 \times 50}{\text{ml. of NaOH solution}}$

[3]In the titration it will be observed that there is a tendency for the pink color to fade. This fading involves the precipitation of tri-calcium phosphate. Carry the titration to the first definite and relatively permanent shade of pink.

[4]Weigh 9 gm. into white cup.

[5]Rinse the pipette with one filling of distilled water.

Determination of pH

Although the acidity of milk and milk products is usually measured and expressed as titratable acidity, the most accurate method of measuring is the determination of the hydrogen ion concentration, or pH.

A common type of equipment for determining the pH of milk and milk products employs the quinhydrone method, which consists of a portable potentiometer, a calomel half-cell, one or more dry cells, and some gold-plated platinum electrodes. A sample of the product to be tested is mixed with a small quantity of quinhydrone, placed in a short glass tube with an electrode which is connected with the calomel half-cell, and the potential read in millivolts. This reading is converted to pH units from a table, after making a correction for temperature.

In recent years, considerable interest has been shown by manufacturers of butter in the pH of butter sera. It is believed that the pH of the butter sera is an index of the keeping quality of the butter.

The most satisfactory method of following the rate and degree of acidity development in cheese is by the determination of pH values at various intervals.

Directions are usually supplied by the manufacturers of pH measuring devices, and it is recommended that in making pH determinations on milk and milk products, the specific instructions of the manufacturer of the measuring instrument used be followed.

Methylene Blue Reduction Test

The apparatus required is either thoroughly boiled or sterilized just before use. The methylene blue solution is prepared by dissolving a standard tablet in 50 ml. of boiling distilled water and then adding 150 ml. of cold distilled water. One ml. of the methylene blue solution is added to 10 ml. of milk in a test tube and thoroughly distributed, the test tube being closed with a cork or rubber stopper. The temperature is quickly brought to 37° C. (98.6° F.) in a water bath, and the tube incubated at this temperature. The time required for the disappearance of the color is then noted. The frequency of the observations is determined by the number of groups into which it is desired to divide the samples. The following grouping is commonly used:

Class 1. Good milk, not decolorized in 5½ hours.
Class 2. Milk of fair average quality, decolorized in less than 5½ hours, but not less than 2 hours.
Class 3. Unsatisfactory milk, decolorized in less than 2 hours, but not less than 20 minutes.
Class 4. Very unsatisfactory milk, decolorized in 20 minutes or less.

The methylene blue test does not determine the number of organisms in a sample of milk, but simply measures one type of their activity and, accordingly, no close agreement can be expected between reduction time and bacterial counts.

The Resazurin Test

1. Measure 0.1 ml. of 0.05 per cent Resazurin dye solution into a sterile test tube.
2. Add 10 ml. of the milk sample to be tested. Incorporate thoroughly.
3. Incubate the samples for one hour at 98° F. in a covered water bath.
4. Read and record results as quickly as possible after the incubation period.

The color of the milk at the end of one hour's incubation ranges from blue, which is good milk, through purple pink, slight pink, pink, vivid pink, to white, which is very poor milk.

Tests for Sediment

Milk

1. Thoroughly stir the milk if a pint sample is to be taken from can; no additional agitation is necessary if the sample is to be taken from the milk freshly dumped into the weigh vat. (A temperature of from 70° to 80° F. facilitates filtration and helps to uniformly distribute the fat.)

2. Force or draw through a lintine disc by means of sediment tester. (It is good practice to rinse the tester with water after samples containing appreciable sediment.)

3. Dry and classify the soiled lintine disc.

Cream

1. If the cream is sweet and free from defects, a 2-ounce sample of cream may be diluted with 8 ounces of hot water (180° F.) and passed through a sediment tester using a lintine disc.

2. For medium sour cream, place a 2-ounce sample of cream in a pint graniteware cup, add 8 ounces of hot (180° F.) baking soda solution (8 teaspoonfuls per gallon of water). Stir thoroughly and run through the sediment tester. Rinse bottle and cup with baking soda solution and run through tester.

3. For very sour cream, place a 2-ounce sample in a cup, add a 9 gm. acid dipper of 1 per cent lye solution (10 gm. lye in 1 quart distilled water), stir, allow to stand 2 minutes, add 8 ounces of hot distilled water (180° F.), mix thoroughly, and pass through the tester. Rinse the sample bottle with a little cold water to insure its complete transfer to the cup, and rinse the cup with a little hot water to transfer it completely to the tester.

Butter

Obtain a representative sample of butter and warm to approximately 85° to 90° F. Stir the sample constantly during the warming period.

1. Place a 100-gm. (¼ lb.) sample of butter in an enamel or graniteware container or glass beaker.

2. Add 8 ounces of hot filtered water (180° F.).

3. Heat the mixture in a water bath to a temperature of 165° F., stirring thoroughly to insure complete melting and mixing of the butter, and run through the sediment tester, which previously has been thoroughly rinsed with filtered water.

4. Rinse out sediment tester with at least 4 ounces of hot water (180° F.).

5. Dry and classify the soiled lintine disc.

Cheese

1. Place cheese to be tested on clean parchment paper. Remove the rind with a knife and cut into strips of suitable size to feed into a food chopper.

2. Force the cheese through a food chopper, after having removed the cutting knife so that the cheese is forced only through the usual perforated end plate.

3. Collect the crushed cheese in a clean 200 ml. beaker.

4. Weigh 100 gm. of the cheese into a clean quart milk bottle.

5. Add 200 ml. of the filtered solvent solution (150 gm. of sodium citrate dissolved and diluted in distilled water to 1200 ml.) and place in water bath at 140° F.

6. Stir with a mechanical agitator (1000 r.p.m.) for 15 minutes.

7. Add 100 ml. portion of distilled water and 100 ml. of the solvent solution, and agitate until cheese is thoroughly dissolved.

8. Divide the contents into two equal portions and filter each part through a single sediment disc.

9. Rinse the sediment tester with filtered water to insure that all the sediment is on the filter pad.

Since 100 gm. of cheese represents about one quart of milk, then each sediment disc would represent the sediment from one pint of milk, and can, therefore, be compared with standards used for judging sediment in whole milk.

Specific Gravity of Milk

1. Place in a lactometer cylinder sufficient milk at 60° F. to float a lactometer.
2. Gently insert an accurate Quevenne lactometer[1] into the milk and allow it to reach equilibrium.
3. Read the scale at the point where the top of the curved surface of the milk comes in contact with the stem of the lactometer.

If the milk is at a temperature other than 60° F. when the reading is taken, a correction must be made. Add 0.1 lactometer degree for each degree of temperature above 60° F. Subtract 0.1 lactometer degree for each degree of temperature below 60° F. These corrections apply only when milk is between 50° and 70° F.

The specific gravity is obtained by placing 1.0 before the corrected lactometer reading. The Quevenne lactometer reads from 15 to 40, corresponding to specific gravity of 1.015 and 1.040.

If a New York Board of Health lactometer is used, it can be converted to Quevenne degrees by multiplying N.Y.B. of H. reading by 0.29.

The approximate total solids content may be determined by the following formula:

$$\text{Per cent total solids} = \frac{\text{Que. lact. reading}}{4} + (1.2 \text{ per cent of fat} + 0.14).$$

[1]The lactometer should be checked for accuracy before use by placing it in an 8 per cent sucrose solution (8 gm. of sucrose in 92 gm. of distilled water). If the lactometer is accurate, it will read 31.06 at 60° F.

Sharp and Hart (Cornell University) advise heating the milk to 113° F., cooling to 86° F., and determining the specific gravity at 86° F. They have devised the following equation for calculating the percent of total solids in milk:

$$\text{Per cent total solids} = \left(\text{Fat} \times 1.2537 + \left\{0.2680 \times \frac{\text{Lact.}}{\text{Sp. Gr.}}\right\}\right.$$

Moisture Test for Butter

1. Take a sample of butter from different parts of the churn and put into a clean, dry cup.

2. Mix butter thoroughly by means of a dry spatula until it becomes salvy and glossy and there is no loose moisture in the cup.

3. Slightly warm a clean aluminum moisture cup over an alcohol flame or electric hot plate, place it on the right hand pan of a moisture scale and balance the scale, leaving the slide weights on the percentage beams on the zero mark.

4. Place a 10-gram weight on the left hand scale pan and weigh 10 gm. of butter into the cup.

5. Drive off the moisture by holding cup over a low flame or electric hot plate by means of a pair of tongs. Keep cup in rotary motion to prevent burning. Do not touch the wick of the burner or the bottom of the cup will become smoked and an incorrect test will result. Moisture is all gone when foaming ceases and the contents become brown, and before they begin to smoke. Cool.

6. Place cup back on the moisture scale, and balance scale by means of weights on the percentage beams. Read the percentage of moisture directly from the beams.

Salt in Butter

1. Dissolve 2.906 gm. of silver nitrate in water and make up to 1000 ml.

2. Weigh out 10 gm. of butter or rinse the residue from the determination of moisture in butter into a 250 ml. cylinder with hot water (preferably condensed steam).

3. Add water until the bottom of the fat column is up to 250 ml. mark. Mix thoroughly. Allow fat to rise.

4. Remove 25 ml. of the liquid under the fat with a pipette and run into a white porcelain cup and add a few drops of a 10 per cent solution of potassium chromate.

5. Add silver nitrate solution from a burette until one drop produces a brick red color which does not disappear on stirring. Each ml. of silver nitrate used equals 0.1 per cent salt.

The Kohman Method of Butter Analysis

Take a representative sample of butter. Warm it slightly and macerate it with a spatula. Weigh 10 gm. of butter into a moisture cup on the right hand scale pan, leaving the balance weight on the

lower beam of the moisture scale on the zero mark and the weight on the upper beam on the 10 per cent mark and using a 10 gm. weight. Now drive off the moisture as in the ordinary moisture test and weigh, after the cup has cooled somewhat, by using only the lower beam on the scale. Read the moisture test directly from the lower beam.

Return the balance weight on the lower beam to the zero mark and remove the 10 gm. weight from the pan. Fill the cup half full of high test gasoline or petroleum ether, keeping away from the flame; stir with a clean glass rod and allow to settle for 2 minutes. Pour off the supernatant liquid into the sink, fill the cup again half full of the gasoline, allow to settle and pour off again. Wipe the outside of the cup with a clean, dry cloth and slowly evaporate the residue to a dry powder on a hot plate, tipping and turning the cup slightly to prevent spurting of the material from the cup. Allow the cup to cool, place it on the same scale pan and balance the scale by using the balance weight on the upper beam. Subtracting the reading from 10 gives the per cent of curd plus salt. The percentage of fat is found by subtracting the per cent of moisture and the per cent of curd and salt from 100.

The percent of salt is detemined by titrating the curd and salt dissolved in a little distilled water with a silver nitrate solution containing 29.06 gm. of silver nitrate per liter of solution, using a 10 per cent potassium chromate solution as an indicator. If a silver nitrate solution containing 2.906 gm. of silver nitrate per liter of solution is used, dissolve the salt and curd in 250 ml. of distilled water and titrate 25 ml. of the solution. The end point is the appearance of a brick red color. Each ml. of silver nitrate solution used represents 0.1 per cent salt in the butter. The per cent curd is found by subtraction.

Preparation and Standardization of Chemical Solutions

General Suggestions for the Care of Standard Solutions

Keep all solutions tightly stoppered to prevent evaporation. Avoid unnecessary exposure to light. Silver nitrate and sodium thiosulphate solutions are decomposed easily by light and therefore must be kept in a dark brown bottle and stored in a dark place. Keep the solution at a reasonably uniform temperature (60° to 70° F.). Check or re-standardize all standard solutions at least once a month.

0.1N Sodium Hydroxide Solution

Detailed directions are given in footnotes 2, Table 3, page 6.

0.1N HC1 and 0.1N H_2SO_4 Solutions

1. 2.8 ml. of H_2SO_4 (sp. gr. 1.84) made up to 1 liter of solution will be approximately O.1N.
2. 9 ml. of HC1 (sp. gr. 1.18) made up to 1 liter will be approximately 0.1N.

Pipette 50 ml. of the acid solution to be standardized into a 250 ml. flask or beaker. Add 0.5 ml. of phenolphthalein indicator solution. Add standard NaOH solution from a burette until a faint pink color is produced. Repeat the titration until closely agreeing results are secured. Calculate the normality of the two acid solutions using the following formula:

$$\text{Normality of unknown solution} = \frac{\text{Normality of Standard NaOH} \times \text{ml. of NaOH solution used}}{\text{ml. of acid solution being standardized}}$$

Standard Sodium Thiosulphate Solution

1. Weigh out 25 grams of $Na_2S_2O_3 \cdot 5H_2O$ and make up to 1 liter with CO_2 free water (boil).
2. Let this solution stand in a dark place for a week or 10 days before standardizing.

Weigh out exactly 1.6258 gm. of KIO_3HIO_3 and make up to 500 ml. with distilled water. Dissolve 5 gm. of KI in the least quantity of water necessary, using a 500 ml. Erlenmeyer flask. Prepare some dilute HCl using one part of HCl to two parts of distilled water. Add 10 ml. of the dilute HCl to the KI solution, then add 50 ml. of the standard KIO_3HIO_3 solution. Make up to about 250 ml. with distilled water. Add the sodium thiosulphate solution from a burette drop by drop until the solution becomes colorless. To make the end point more distinct, 2 ml. of a 2 per cent soluble starch solution may be added as the end point is approached. Calculate the normality of the solution using the following formula:

$$\text{Normality of sodium thiosulphate solution} = \frac{0.10 \times \text{ml. of biniodated sol. used}}{\text{ml. of thiosulphate solution required}}$$

Standard Silver Nitrate Solutions

To prepare 1 liter each of silver nitrate solutions containing:

A. 29.062 gm. of $AgNO_3$ per liter.
B. 17.260 gm. of $AgNO_3$ per liter.

Note: The above solution should be prepared with recently boiled distilled water, filtered and stored in a dark place.

Prepare standard salt solutions from pure dried NaCl to contain:

A. 2.9230 gm. of NaCl in 500 ml. of solution.
B. 5.9395 gm. of NaCl in 1000 ml. of solution.

Prepare a 10 per cent solution of potassium chromate.

To standardize silver nitrate solution A., pipette 25 ml. of the salt solution (2.9230 gm. of NaCl in 500 ml. of solution) into a beaker. Add a few drops of potassium chromate indicator. Add the silver nitrate solution A. (29.062 gm. per liter) from a burette until a faint, blood red tinge is produced. Repeat the titration until closely agreeing results are secured. Calculate the normality of the solution, using the following formula:

$$\text{Normality of } AgNO_3 \text{ solution} = \frac{0.1 \times \text{ml. of NaCl sol. used}}{\text{ml. of the } AgNO_3 \text{ sol. being standardized}}$$

To standardize silver nitrate solution B., pipette 25 ml. of salt solution B. (5.939 gm. of NaCl in 1000 ml. of solution) into a beaker. Add 2 ml. of potassium chromate indicator. Add the standard silver nitrate solution B. from a burette until a faint blood red tinge is produced. Repeat the titration several times until closely agreeing results are secured. Calculate the normality using the same formula as for silver nitrate solution A.

Soluble Starch Solution

Weigh out 2 gm. of soluble starch and suspend in 25 ml. of cold water. Place 75 ml. of distilled water in an evaporating dish and heat to boiling. Add in small installments with constant stirring the 25 ml. starch suspension. Let cool and make to 100 ml.

Indicator Solutions

Phenolphthalein

Weigh out 0.5 gm. of the indicator and dissolve in 50 ml. of 95 per cent alcohol.

Methyl Orange

Weigh out 0.25 gm. of the indicator and dissolve in 500 ml. of distilled water.

Bromthymol Blue

Weigh out 0.2 gm. of indicator and dissolve in 200 ml. of distilled water.

Preparation of Glymol

The reagents used are Diamond paraffin oil, obtained from Standard Oil Company of New Jersey or allied companies, and National Oil red C, an oil soluble technical red analine dye in powder form. It can be obtained from the National Analine and Chemical Company, 140 Rector Street, New York, New York.

Weigh out 1 ounce of the dye. If caked or lumpy, pulverize it. Dissolve the ounce of dye in a quart of Diamond paraffin oil. Warming the oil and allowing it to stand with frequent stirring while dissolving hastens the process. When dissolved, add Diamond paraffin oil to make the volume up to 5 gallons. Stir well.

Sulfuric Acid Dichromate Cleaning Mixture

1.	Commercial sodium dichromate (powder)	8 gm.
2.	Water	300 ml.
3.	Commercial sulfuric acid	460 ml.

Dissolve the sodium dichromate in warm water. Cool this solution and add it slowly to the concentrated sulfuric acid, agitating the mixture constantly. This procedure can best be carried out by using a large Pyrex flask which is kept in a bath of cold water. The flask should have a capacity of at least twice the volume of the mixture being prepared in it.

Checking Strength of Washing Solutions

Routine Titration Method for Plant Control[1]

The apparatus and reagents needed are a volumetric flask of 100 ml.; a burette of 50 ml. graduated in tenths with a burette stand; an Erlenmeyer flask of 100 ml.; a pipette of 1 ml.; sulfuric acid, tenth normal; methyl orange indicator (0.05 per cent solution in distilled water).

From one lot of the washing compound used, obtain a fair sample of the powdered or crystalline material and make up in the laboratory the strength solution which is required in the principal soaking tank of the bottle-washing machine. If, for example, a 5 per cent solution is required, weigh out carefully 5 gm. of the washing compound and transfer to the 100 ml. flask. Dissolve the material in a small amount of distilled water, cool to room temperature, fill to the mark with distilled water and mix well. With the pipette, transfer 1 ml. of the solution to the Erlenmeyer flask. Add a little distilled water and a few drops of methyl orange indicator. Add slowly tenth normal sulfuric acid from the burette until the yellow changes to a slight pink color. The number of milliters of tenth normal sulfuric acid used in the foregoing titration is the index of the number of cubic centimeters of tenth normal sulfuric acid needed to neutralize 1 ml. of the washing compound solution in the principal tank of the bottle-washing machine.

Testing Chlorine Solutions

Testing Stock Chlorine Disinfectant Solutions for Their Available Chlorine

Pipette a 50 ml. sample of each of the chlorine solutions to be tested into a glass beaker. Add approximately 50 ml. of distilled water to each sample. Add 5 ml. of 20 per cent solution of potassium iodide solution and 10 ml. of glacial acetic acid. Add 0.1N sodium thiosulphate solution from a burette, drop by drop, until the solution becomes colorless. Repeat the titration until closely agreeing results are secured. Calculate the available chlorine in the sample tested using the following formula:

$$\text{ml. of } 0.1N\ Na_2S_2O_3 \times 70.9 = \text{available chlorine in P.P.M.}$$

Testing the Strength of Chlorine Disinfectants in Powdered Form

Weigh out 10 gm. of the sample to be tested. Dissolve the weighed sample in distilled water and make up to 200 ml. in a volumetric flask. Pipette out 1 ml. of the solution. Calculate the per cent chlorine in the sample tested using the following formula:

$$\frac{\text{ml. of } 0.1Na_2S_2O_3 \times .003545 \times 100}{\text{weight of sample}} = \text{Per cent chlorine}$$

[1]This method is especially applicable when the bottle-washing solutions are made up principally of caustic soda or a mixture of caustic soda and soda ash and when for plant routine purposes the relative strength only of solutions in the different soaking compartments is required. There is no simple plant method for determining the ingredients in complex washing compounds.

Correction of Brine to Proper Degree of Alkalinity

Filter the brine sample or allow the sediment to settle. Transfer 50 ml. of the brine to a 250 ml. Erlenmeyer flask and add a few drops of phenolphthalein solution. If the brine is acid, titrate with 0.1N sodium hydroxide to a faint pink color; if the brine is strongly alkaline as indicated by a heavy red color, titrate with 0.1N hydrochloric acid to a faint pink.

Using the following table, correct the brine solution to the proper degrees of alkalinity:

Ml. acid or alkali to neutralize 50 ml. of brine	Acid Brine Lbs. of NaOH per 100 gal. required to bring brine to proper alkalinity	Alkaline Brine ml. of conc. HCl sp. gr. 1.185 required to bring 100 gal. of brine to proper alkalinity
1	0.0667	65.7
2	0.1334	131.4
3	0.2001	197.1
4	0.2668	262.8
5	0.3335	328.5
6	0.4002	394.2
7	0.4669	459.9
8	0.5336	525.6
9	0.6003	591.3
10	0.6670	657.0