

# AGRICULTURAL EXPERIMENT STATION

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## THE PREPARATION, TESTING, AND USE OF CHLORINE DISINFECTANTS

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### INTRODUCTION

The ever-increasing demand for biological cleanliness in the handling of dairy products has created an unprecedented use for chlorine disinfectants on the farm and in dairy plants. The following directions for the preparation and testing of chlorine solutions have been found satisfactory under laboratory and field conditions. They are presented in a condensed form with the object of eliminating some of the uncertainty in the very important process of sterilizing dairy equipment.

Three methods of preparing strong stock solutions of chlorine are described, the choice of which depends upon the amount of disinfectant used. In general it is inadvisable to prepare more stock solution than can be used within two months.

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## PREPARATION OF CHLORINE DISINFECTANTS

### CHLORIDE OF LIME METHOD

1. Dissolve the contents of a 12-ounce can of fresh chloride of lime (bleaching powder) in 2 gallons of water, using an earthenware vessel. Stir vigorously several times during the first 3 to 6 hours, then allow the solution to stand undisturbed over night.

2. Siphon the clear supernatant liquid into 1-gallon jugs. A funnel, fitted with a filter pad of absorbent cotton, should be placed in the mouth of the jug to keep out all solid particles of the sediment. Place tightly-fitting stoppers in the jugs and store in a cool place. If clear-glass vessels are used they should be placed in the dark. Effective filtering of the solution and the use of tightly-fitting stoppers greatly enhance the keeping quality of the stock solution.

This stock solution contains calcium hypochlorite as the germicide. Its strength is entirely dependent upon the available chlorine content of the chloride of lime. Since the small 12-ounce packages obtainable in drug stores are frequently low in available chlorine, due to prolonged storage, it is inadvisable to use this method of preparing disinfectant unless facilities are available for testing the final product. However, this method has the advantage of simplicity and affords a very convenient means of preparing a small amount of disinfectant at a cost of about 25 cents per gallon. The chief disadvantage of this procedure lies in the uncertainty of the available chlorine content of the small packages of chloride of lime. Since the small purchase does not warrant direct shipment from the manufacturer, it is necessary to accept a product which may or may not be fresh. If the bleaching powder contains the maximum amount of available chlorine, about 35 per cent, very satisfactory results may be obtained. The use of this method is recommended only where small quantities of chlorine are used.

### SODIUM HYPOCHLORITE FROM CHLORIDE OF LIME

1. Dissolve 20 pounds of fresh chloride of lime in 8 gallons of warm water (not over 120° F.). This solution should be prepared in a 20- to 30-gallon stone jar. (Do not use a metal container.)

2. In any convenient vessel dissolve 10 pounds of soda ash (or 27 pounds of sal soda) in 6 gallons of water.

3. Mix the two solutions by pouring the soda solution into the stone jar containing the chloride of lime. Stir the mixture two or three times at convenient intervals during the remainder of the day, then allow it to stand undisturbed over night.

4. Siphon the clear supernatant liquid into gallon jugs. A funnel and cotton pad should be placed in the mouth of the jug to keep out any particles of sediment. The jugs should be tightly stoppered and stored in a cool place. Clear-glass jugs should be kept in the dark.

This stock solution contains sodium hypochlorite, which is more desirable as a disinfectant than the calcium hypochlorite. Since a larger amount of the chloride of lime is employed in this formula, an order may be placed with a local druggist for a direct shipment

from the manufacturer, stipulating a guarantee of 35 per cent available chlorine at time of shipment.

This method of preparing the stock solution is advantageous for dairy plants using from 3 to 5 gallons per month. At the current prices the cost of ingredients is about 15 to 20 cents per gallon for a solution testing approximately 4 per cent available chlorine.

When the demand exceeds 5 gallons a month, it is recommended that the stock solution be prepared from chlorine gas in accordance with the following method.

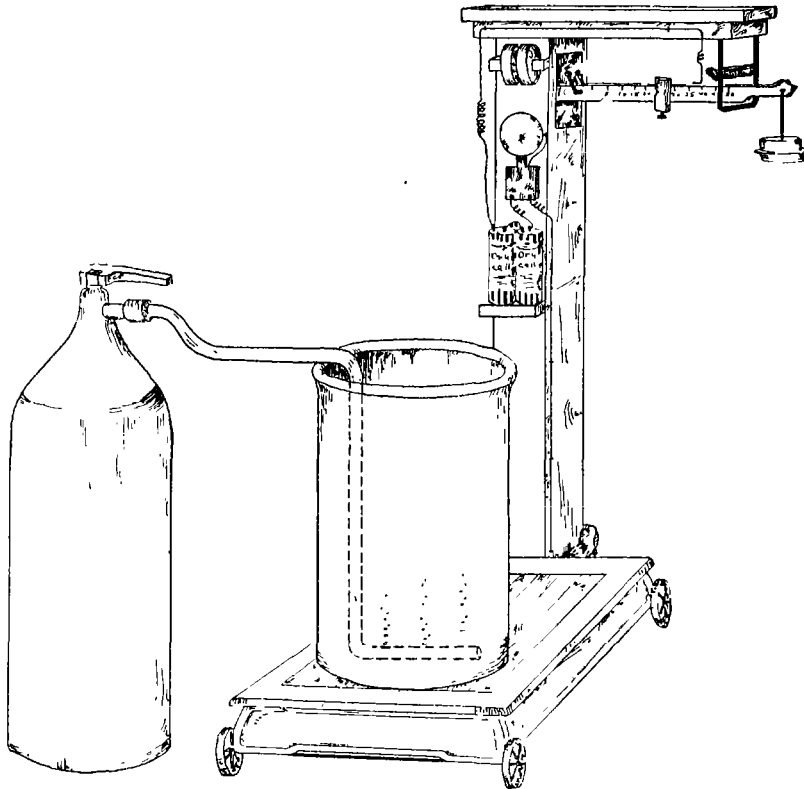


FIG. 1.—Outfit for making sodium hypochlorite disinfectant from chlorine gas.

**SODIUM HYPOCHLORITE FROM CHLORINE GAS**

**Equipment**

The arrangement of equipment for making sodium hypochlorite from chlorine gas is illustrated in figure 1.

A 1/2-inch lead pipe is tightly fastened to the yoke connection supplied with the chlorine tank. This pipe is bent so as to conduct the gas to the bottom of the jar, where it escapes through pin holes punched at 1-inch intervals in the pipe. The end of the pipe should be closed, and the pipe suspended so its weight does not rest on the jar.

The illustration shows the connections for an electric bell which signals to the operator when chlorination is complete. One wire from the bell is grounded to a metal part of the scales. The circuit is completed through another wire suspended in the arc in which the scale beam swings. This is a very convenient, though a non-essential part of the equipment.

The stone jar used should hold 20 to 30 gallons and be of such dimensions as to provide at least an 18-inch column of water over the lead pipe in the bottom. Ordinary platform scales are sufficiently sensitive for this work. The tank of liquid chlorine may be purchased through any local druggist or chemical supply house.

#### Chlorination

1. With the equipment arranged as illustrated, place 100 pounds of water in the jar.
2. Add slowly 6.75 pounds of 76 per cent commercial grade caustic soda and 2.75 pounds of soda ash (washing powder). Allow the mixture to stand at least 2 hours to insure complete solution.
3. After carefully balancing the scales, advance the beam weight the equivalent of 3.75 pounds. Admit chlorine gas slowly until the scales again reach a balance (until the bell signals). Chlorination should require from 1½ to 2 hours. After chlorination place the stock solution in tightly-stoppered glass or earthenware jugs and store in a cool dark place.

This is a simple, convenient, and economical method of preparing a stock solution of sodium hypochlorite. At current prices, the cost of ingredients for a solution testing 3.5 to 4 per cent available chlorine is about 10 cents per gallon. This method is especially recommended for dairy plants using large amounts of disinfectant, either within the plant and subsidiary plants or in supplying chlorine to patrons as an agency for improving the quality of the raw products.

#### SIMPLIFIED TEST FOR AVAILABLE CHLORINE

One of the most serious objections to the use of chlorine disinfectants is the probability of unknowingly employing weak solutions. Effective sterilization of dairy equipment is of too much importance to be controlled by guesswork. The highly volatile character of many chlorine compounds renders it inadvisable to use "rule of the thumb" methods in diluting stock solutions. Tests should be made to show the available chlorine content of the stock solution, and the proper dilution calculated on this basis. Similarly, the final chlorine rinse water should be tested at frequent intervals to prevent the use of solutions from which the chlorine has disappeared.

The following method of testing both the stock and the final chlorine rinse water is designed to render this important test available to those who should be using it daily.<sup>2</sup>

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2. Fay, A. C. A simplified test for chlorine disinfectants. Ice Cream Trade Jour. Vol. 27, No. 7, pp. 33-34; July, 1931.

**PREPARATION OF INGREDIENTS**

**Standard Sodium Thiosulphate Solution.**—Dissolve 9 grams of sodium thiosulphate in approximately 6 ounces of boiling water. Place the solution in a 1-pint milk bottle and dilute to 1 pint with water that has been previously boiled. Keep this solution tightly covered. If a yellow precipitate of sulphur appears after long standing, the solution should be discarded and a new one prepared. (Sodium thiosulphate may be bought at almost any drug store either in the form of the C. P. chemical or as "Granular Hypo" prepared by Eastman Kodak Company. Babcock cream scales and the accompanying 9-gram weight are sufficiently sensitive for weighing this salt.)

**Acid-iodide Powder Mixture.**—Thoroughly mix potassium iodide crystals and boric acid powder in the proportion of 1:2 by weight ( $\frac{1}{2}$  pound to 1 pound). Place this in a wide-mouth brown glass bottle and keep covered.

**PERFORMING THE TEST**

**Testing the Chlorine Rinse Water.**—In the practical use of chlorine disinfectants the operator usually is not concerned with an exact determination of the available chlorine content of the rinse water. He is usually satisfied with the assurance that the solutions employed are maintained above an arbitrary minimum strength which is known to give satisfactory results. This test affords that assurance by the following very simple procedure:

1. Place 1 teaspoonful of the acid-iodide powder mixture in a clean quart milk bottle.
2. Using a pint milk bottle, pour in 1 pint of the chlorine rinse to be tested and shake until the brownish-yellow color is evenly distributed.
3. With a Babcock milk pipette add slowly 17.6 cc. of the standard sodium thiosulphate solution.

If the brownish-yellow color disappears after the addition of 17.6 cc. of the standard thiosulphate solution, the chlorine rinse water contains less than 100 parts per million (p.p.m.) of available chlorine. If the color is still evident, there are more than 100 p.p.m. of available chlorine.

By using 1 quart of chlorine rinse water instead of the pint, more or less than 50 p.p.m. may be detected. Similarly, by using  $\frac{1}{2}$  pint of chlorine rinse water in the test, more or less than 200 p.p.m. may be detected. These are the concentrations most commonly employed in dairy practice.

The test as previously described shows only roughly the presence of more or less than 50, 100, or 200 p.p.m. available chlorine. Although this is sufficient for practical use, it is frequently desirable to know the chlorine content within narrower limits.

Using a teaspoonful of the powder mixture dissolved in 1 pint of chlorine rinse water to be tested, add the standard sodium thiosul-

phate drop by drop from a burette (or 10 cc. pipette graduated to 1/10 cc.) until the color disappears. Multiply the cc. of standard solution used by the factor 5.74 to give the parts per million of available chlorine,

**Testing Stock Solution.**—Proper dilution of the stock solution necessitates a knowledge of its available chlorine content.

1. Dilute 1 cc. of the stock solution with approximately ½ pint of water.
2. Add 1 teaspoonful of the powder mixture and shake.
3. Admit the standard sodium thiosulphate solution drop by drop until the color disappears.
4. Multiply the cc. of standard solution used by the factor 0.27 to give the per cent available chlorine in the stock solution.

**Practicability of the Tests.**—These methods of testing chlorine stock and rinse solutions are designed primarily for those who are actually engaged in the use of chemical disinfectants in dairy plants. By substituting Babcock cream scales, 17.6 cc. milk pipettes, and milk bottles already found in a dairy plant for more accurate analytical balances, burettes, and volumetric flasks, these very important and necessary tests may be applied in any dairy plant. Tests performed in this manner have been found to yield results within 5 per cent of the more accurately controlled laboratory procedure. This variation is well within the limits of practical application.

**THE USE OF CHLORINE DISINFECTANTS**

**DILUTIONS RECOMMENDED**

For submerging well-cleaned bottles, pails, separator parts, etc., in which the time of action may exceed 60 seconds, the chlorine rinse solution should be maintained at or above 50 p.p.m. If less time is available the solution should contain at least 100 p.p.m. A chlorine solution to be pumped through the pipelines, vats, etc., in a plant, should be of such strength that it will test at least 50 p.p.m. as delivered from the last piece of equipment. For effective use in spraying vertical coolers, sides of vats, and other equipment in which the time of contact is only a few seconds, chlorine solutions should test at least 200 p.p.m. available chlorine.

**Calculation of Dilutions.**—Intelligent use of chlorine solutions demands the elimination of guesswork in diluting the stock solution. By substituting the proper values in the following formula it is possible to calculate the amount of stock solution to be employed in making any volume of any desired strength of chlorine rinse water.

$$\frac{\text{Strength of rinse desired (expressed in p. p. m.)}}{\text{Strength of stock solution (expressed in per cent)}} \times \frac{\text{Gallons of rinse desired}}{\text{Factor, 2.64}} = \text{cc. of stock to be used.}$$

CHLORINE DISINFECTANTS

EXAMPLE: To prepare 50 gallons of rinse containing 100 p.p.m. from a stock solution testing 4 per cent.

$$\frac{100 \text{ (p.p.m.)}}{4 \text{ (per cent)}} \times \frac{50 \text{ (gal.)}}{\text{Factor, 2.64}} = 473 \text{ cc. of stock to be used.}$$

If it is desirable to make larger volumes of rinse water, it may be convenient to calculate the amount of stock solution in terms of units larger than cubic centimeters. The factor 2.64 may be replaced by any one of the following factors to express the answer in terms of the units indicated: Gallons, 10,000; quarts, 2,500; pints, 1,250; and fluid ounces, 78.

PRECAUTIONS TO BE OBSERVED

**Discard Cloudy Rinse Water.**—Only perfectly clear chlorine rinse water should be used. When the solution becomes clouded with milk carried over from wash vats it should be discarded even though it may still contain more than the minimum required amount of available chlorine. Unpublished experimental results have shown that the minimum lethal strength of available chlorine is higher in solutions clouded with milk than in clear solutions.

**Use Cold Solutions.**—The common practice of using hot chlorine rinse water is more likely to be detrimental than helpful. Although it is true that the higher temperature increases the rate of reaction between bacterial protoplasm and disinfectant, it likewise accelerates the rate of reaction with other organic matter. It is true that a perfectly clear, hot, chlorine rinse solution is more effective than a cold one of similar strength, and that the heating of such a clear solution to temperatures below boiling does not induce excessive loss of chlorine. If, on the other hand, a small amount of organic matter is added, the solution loses its chlorine at a tremendous rate. The presence of organic matter in a cold solution lessens its value, but the effect is so greatly accelerated at high temperatures that it discourages the use of warm chlorine rinse water.

**Use Well-cleaned Equipment.**—The most important prerequisite to satisfactory results with chlorine disinfectants in dairy practice is thorough cleaning of utensils before sterilization. The sole purpose of using the chlorine should be to destroy microorganisms. Chlorine is not a cleaning agent, although when liberated it is a very reactive substance. If there is any organic matter present other than bacteria, the disinfecting power of the solution will be dissipated in nonessential reactions. Since under practical conditions it is almost impossible to exclude every trace of organic matter, an excess of chlorine must be allowed for these reactions. The extent to which allowance must be made for this purpose, even in very well-cleaned utensils, is surprising. It has been shown, for example, that 0.1 p.p.m. available chlorine kills most bacteria under laboratory conditions which are carefully controlled so as to eliminate even the slightest trace of organic matter. Under the practical conditione

in a dairy with well-cleaned utensils, it is advisable to use about 50 p.p.m. available chlorine in rinse solutions in order to insure satisfactory results. It is evident that this commonly recommended strength is 500 times more concentrated than is actually required to kill the individual cell. This very large safety factor is required to take care of the extraneous reactions with the relatively small amount of organic matter unavoidably left in a well-cleaned utensil. It is useless to employ chlorine sterilizers in visibly dirty vessels. The importance of thorough cleaning before applying chlorine solutions cannot be overemphasized.

**Dry Equipment Thoroughly.**—Under practical dairy conditions it is not feasible to completely sterilize utensils either by steam or chemicals. Nearly always a few organisms will survive the sterilization process employed. One of the most important aspects of any practical sterilization practice is to prevent the growth of surviving organisms. If the vessel is left perfectly dry, there will be no growth of the viable cells during the time the vessel is not in use. Utensils that have been sterilized in a chlorine disinfectant should be subsequently rinsed with enough hot water or steam so that the vessel will dry from its own heat.

#### SUMMARY

Methods of preparing home-made hypochlorite disinfectants at a cost of 10 to 25 cents per gallon are given. Detailed procedures are presented for making calcium or sodium hypochlorite from bleaching powder, as well as directions for preparing the sodium hypochlorite from chlorine gas.

A simple, practical test for available chlorine, which may be used in any small dairy, is described. A roughly quantitative procedure has been designed to determine easily and quickly if a chlorine rinse solution contains an adequate concentration of available chlorine. The procedure as outlined for testing the stock solution for per cent available chlorine, as well as the final rinse water for parts per million available chlorine, should eliminate much of the uncertainty involved in the use of empirical methods for diluting chlorine disinfectants.

A formula is given which makes possible the calculation of the proper dilution of any stock chlorine solution so as to yield any desired amount of chlorine rinse water of any desired strength. Dilutions are suggested for the more common sterilization procedures in dairy practice.

The use of clear rinse solutions, free from organic matter, is stressed as one of the most essential factors in the intelligent use of chlorine disinfectants. The accelerating effect of heat on the loss of chlorine in the presence of small amounts of organic matter suggests caution in the use of hot chlorine rinse waters. The futility of attempting sterilization of unclean utensils with chlorine, and also the importance of drying the equipment after sterilization, are discussed.

