1942

The development of an apparatus for producing a saline hypochlorite solution electrolytically for antiseptic purposes

Thomas Samuel Leary

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UMI
THE DEVELOPMENT OF AN APPARATUS FOR PRODUCING A SALINE HYPOCHLORITE SOLUTION ELECTROLYTICALLY FOR ANTISEPTIC PURPOSES

by

Thomas S. Leary

A Thesis Submitted to the Graduate Faculty for the Degree of

DOCTOR OF PHILOSOPHY

Major Subject: Chemical Engineering

Approved:

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Dean of Graduate College

Iowa State College
1942
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INTRODUCTION

With the world again engulfed in the cataclysm of a great war, it is quite timely that the research personnel of this country turn some of its efforts towards the alleviation of some of the miseries caused by this titanic struggle. It was during the last great World War that Drs. Dakin and Carrel introduced Dakin's solution and perfected its method of application for the treatment of war wounds.

Today we have the sulfa drugs which in a measure are replacing Dakin's solution for wound sterilization. One of the reasons for this replacement lies in the fact that while the sulfa drugs are expensive and difficult of preparation, once prepared they can be kept for long periods of time without losing their germicidal properties. On the other hand, Dakin's solution and other hypochlorite solutions are very unstable and lose their effectiveness if stored for any length of time even though stabilizers may be added to hinder their decomposition.

At present there are two general methods for the production of Dakin's solution. The first method is based on what is known in chemical terminology as a replacement or double decomposition reaction. This method has two modifications, that used by Dakin himself and the procedure developed by Daufresne. The second general method depends on a chlorination reaction and is the original method used by Labarraque when he made his famous Labarraque's disinfecting fluid (9).

The original method used by Dakin (9) is as follows:
The instructions which they give are as follows (26):

The second General method of the precipitation method is advocated.

*Filter the solution for extra and excess orthoion.
*Shake the precipitation to settle. *Shake the mixture well and allow the precipitate to settle.
*Shake the mixture well and allow the precipitate to settle. *Shake the mixture well and allow the precipitate to settle.
*Shake the mixture well and allow the precipitate to settle.

Procedure: Begin by adding 50 grams of ortho to 200 grams of the mixture of ortho (theophylline) and the solution of ortho.

The method usually used is that of buffered (9) which is as follows:

1. Prepare a buffer solution of 0.45 to 0.60 grams per 100 ml. of water.
2. To the buffer solution, add 40 grams of ortho.
3. Filter the precipitate to settle. *Shake the mixture thoroughly and allow to stand one half hour for ortho to crystallize.
4. Add 200 grams of the precipitate in 10 liters of tap water and add 200 grams of ortho to crystallize.
1. A solution is prepared containing 18.0 grams of dry sodium carbonate per liter (equal to 21 grams monohydrate or 48.5 grams washing soda) and a measured quantity, 4.8 grams per liter (or about 1,600 c.c.) of chlorine gas is allowed to run into the solution.

2. Ten c.c. of the solution are then titrated. If the solution is too strong it should be diluted to 0.5 per cent NaOCl with 1.4 per cent sodium carbonate. This serves to correct the unduly diminished alkalinity caused by the excess of chlorine introduced into the solution. However, the designated amount of carbonate is planned to give at a concentration of 0.5 per cent NaOCl, the minimum degree of alkalinity consistent with stability, and if chlorine has been introduced in such excess that the titer exceeds the desired amount by more than 5 or 6 c.c. of N/10 thiosulfate or if the solution fails to give a momentary flash of color with an alcoholic solution of phenolphthalein it should be discarded.

As can be seen these methods demand the services of an experienced chemist or pharmacist which makes their use prohibitive in places where such personnel is not available.

Should a method or apparatus be developed which would permit the average layman with no experience in things chemical to produce a solution of hypochlorite of the proper strength and alkalinity, it is believed that hypochlorite solutions would again come back into prominence.

An electrochemical process is the most logical approach to the solution of this problem and so it is the purpose of this research to develop an electrolytic cell which will produce a saline hypochlorite solution suitable for antiseptic use. This cell must be capable of being operated
by an inexperienced person, must have a minimum of features which might
tend to give trouble, must be versatile and rugged enough so that it can
be used in field hospitals, ambulances, and warships, as well as in base
hospitals and civilian doctors' offices.
HISTORICAL

A. History of Hypochlorite

Hypochlorite and other chlorine compounds have been used for over a century as disinfecting and deodorizing substances. Chlorine, the element, was discovered in 1774 by Scheele who called it desphlogisticated muriatic acid. Lavoisier later considered it a compound of hydrochloric acid and oxygen and as a result called it oxygenated muriatic acid, the term which was also applied to it by Berthollet. In 1810 Sir Humphrey Davy proved its elemental character and gave it the name of chlorine (18).

Berthollet (8) in 1788 first produced a hypochlorite solution with bleaching and disinfecting properties by running chlorine gas into a solution of sodium hydroxide. He did not know the composition of the resulting compound but believed it to be an addition compound of the alkali and the halogen such as \( \text{Na}_2\text{OCl}_2 \). Berzelius in 1808 likewise expressed the opinion that the substance was a mixture of a salt of an unknown acid of chlorine and sodium chloride.

In 1834 Ballard's discovery of hypochlorous acid settled the matter and proved Berthollet's substance was composed of sodium chloride and sodium hydrochlorite.

In 1792 Percy of the Javal works in Paris produced potassium hypochlorite by following the procedure of Berthollet. This solution was known as "eau de Javal" or as "Javal Water." The use of this substance as a disinfectant was advocated by Berthollet and Guyton de Morveau.
In 1820 Labarraque prepared a solution of sodium hypochlorite by the action of chlorine gas on sodium carbonate. This solution was sold under the name of "Labarraque's Disinfecting Fluid." Labarraque was pharmacist to Napoleon and later to Louis XVIII and achieved great renown on the death of the latter monarch for, as a result of the deodorizing and disinfecting value of his "Fluid," he was able to embalm the royal body, which was so thoroughly decomposed that no one could touch it until after the use of the hypochlorite solution.

Due to the instability of these solutions, they were replaced by the more stable solid "chloride of lime" which was first produced on a large scale by Tennant (18) in 1798 at his plant in Glasgow, Scotland. In 1846 Semmelweis stamped out an epidemic of puerperal fever in his clinic with this substance.

Hypochlorite was used as a deodorant and disinfectant long before the germ theory of disease was established. Since 1850 water supplies (6) have been treated with hypochlorite spasmodically and in cases of emergency although it was not known that they brought about the destruction of microorganisms. According to Vandevelde, Traube in 1894 was the first to point out that hypochlorite had a purifying action on water. It has been reported that chloride of lime was used in the treatment of sewage in London as early as 1854.

R. Koch in 1881 carried out the first investigation on the bactericidal properties of hypochlorite. This was perhaps the first demonstration that the purpose of hypochlorite in disinfection was to destroy microorganisms.
Up until the discovery in 1859 by Charles Watt (17) that hypochlorite solutions could be made by the electrolysis of a sodium chloride solution, all such solutions were made by chlorination reactions. He obtained an English patent on this process in which the following criteria were set forth:

1. Insoluble electrodes were necessary.
2. It was not necessary to use a diaphragm between the electrodes.
3. Low temperature tended to prevent the formation of sodium chlorate.
4. There must be a rapid circulation of solution from the cathode to the anode.

The first cell to be moderately successful for the preparation of hypochlorite was that of Hermite, patented in 1887. This cell consisted of a series of rotating zinc cathodic discs mounted on a shaft and were separated from each other by partitions. Thin sheets of platinum were placed between the zinc discs and used as anodes. The body of the cell was fabricated from ceramic ware with the feed coming in at the bottom and leaving through a grooved channel around the top.

In 1890 Andreoli (3) patented a process similar to that of Watt for the electrolytic preparation of hypochlorite solutions. Since that time there have been scores of modifications advocated for the electrolytic preparation of hypochlorite solutions.

At the present time electrolytic cells are in use for the preparation of solutions of hypochlorite for technical use; however, there are no cells on the market at present which are designed to produce a hypochlorite solution of the proper strength and alkalinity suitable for direct use as an antiseptic.
Eau de Javelle, eau de Labarraque and chloride of lime were not the only hypochlorite preparations used, but were merely three among many. Among the numerous preparations used were "extrait eau de Javel," "esprit de Javel," "essence de Boulogne," "Chlorozone," "hermitine," and "chloros." These need not be mentioned in detail since they represent hypochlorite preparations of varying purity and stability.

Up until the time of World War I hypochlorite preparations continued to find increasing usage as deodorants and general disinfectants. The St. Thomas Hospital had been using a sodium hypochlorite solution for the dressing of amputated stumps since 1855. Weak hypochlorite solutions had been used for the removal of sloughs and for controlling the smell of offensive wounds. Professor Delépine had been using it as a skin disinfectant in his pathological laboratories.

The great drawback in all this work was the high alkalinity of these various hypochlorite solutions which caused irritation of the tissues unless used in such weak concentrations that the desired antiseptic action was hampered. It was not until 1915 that such a solution of suitable concentration was prepared without this hindrance.

B. The Discovery of Dakin's Solution

The use of antiseptics dates back to the work of the English physician, Lister, who first showed that a wound could be rendered sterile by the use of an antiseptic agent. The agent which he used was carbolic acid or phenol as it is called in chemical terminology. Carbolic acid was found to be only one of a score of materials with antiseptic properties for as
time passed many others including mercuric chloride, iodine, hydrogen peroxide and various types of hypochlorite solutions were discovered.

Although there had been quite a bit of work done on antiseptics up to 1915, it had all been done by isolated individuals (5) with extemporized equipment. In clinical researches, the basic principles of scientific investigation were forgotten and methods employing measurements were rarely used. Any substance which possessed the property of destroying microbes in vitro, was looked upon as an antiseptic and used as such. There was no attempt to classify antiseptics as to the conditions under which they were best suited for use. Substances which coagulated proteids, which lost their bactericidal power in the presence of serum, or which were actually harmful to the tissues were used indiscriminately. Details as to optimum concentration and time of action were never sought. Every doctor had his own "pet" antiseptic which he used on all types of wounds.

As can be readily seen, antiseptics were losing their appeal to physicians, who found that they did not always stop infections as they were supposed to do. Instead of looking for the cause of such a condition in the antiseptics which they were using, many surgeons denounced the theory of antiseptics and the work of Lister was pushed into the background. As is often the case in such situations some surgeons went to the extreme of saying that rather than kill bacteria, antiseptics fostered their growth. Many others advocated their use on some types of wounds but discouraged their use on others.

It was realized (16), however, at the very early stages of the war, by all surgeons who attended the wounded either on the continent or in
England, that a very large percentage of the wounds, especially those involving extensive wounds of the extremities, were infected either at the moment of infliction or at a very short time thereafter. The antiseptic methods used by surgeons in the past were found to be unsatisfactory for this type of wound. Dr. Carrel (1) quoted before the French Académie de Médecine Professor Tuffier’s statement that of 1,000 amputations at Maison-Blanche, the operation had to be done in 80 per cent of the cases owing to complicating infection rather than to the gravity of the wound.

Many surgeons doubted that any antiseptic would be suitable for the treatment of war wounds. The principal adversary to the use of antiseptics on wounds of this type was Colonel Sir Almroth Wright, M.D., consultant physician to the British Expeditionary Force in France. Wright believed that the microbes being carried by projectiles and fragments of clothing were lodged too deep into the tissues to be reached by antiseptics and that the methods of Lister were not applicable to war wounds. To him, chemical sterilization of such a wound seemed impossible of realization and he was outspoken in his belief of this contention (26). Although the doctrines of Wright were founded upon ingenious theories and experiments in vitro rather than upon observations and experiments on actual war wounds, they were accepted by a majority of the surgeons of that time. He did, however, advocate that a single method of treatment be established and followed by all, even though an improved and more efficient technique might be later substituted for it (1).

To throw some light on this controversy, the British Medical Research Committee (16), established under the National Insurance Act, decided to
sponsor an investigation on antiseptics, in accordance with its policy of assisting the military authorities. A research group headed by Professor Lorrain Smith and including Professor Drennan of the University of Otago, Dr. Rettie, a chemical expert, and Lieutenant W. Campbell, R.A.M.C., undertook an investigation in the pathological department of the University of Edinburgh to ascertain what antiseptics fulfilled the special requirements for use in the treatment of war wounds.

Shortly afterwards a second group including Professor Cohen of the University of Leeds, Dr. Henry Dakin, director of the Baxter Laboratory of New York, who was at that time acting as bacteriologist to the hospital and laboratory established by the Rockefeller Institute at Compiègne, and Dr. Alexis Carrel, also of the Rockefeller Institute, a surgeon at that hospital, inaugurated a program headed towards the same goal. The plan which they followed called for substances, prepared by Professor Cohen in Leeds, to be tested bacteriologically by Dr. Dakin and that the most promising of these be tested clinically by Dr. Carrel in the hospital.

It is interesting to note that these two distinct research groups working independently of each other, both arrived at the same conclusion, namely, that the most effective and innocuous antiseptic for the treatment of war wounds was either hypochlorous acid or one of its salts.

Professor Smith and colleagues (19) concluded that hypochlorous acid was the most powerful antiseptic known, its effect was purely local, the decomposition products were devoid of toxicity, there was no danger from absorption, and that the flow of lymph was induced from the wound as part of the reaction of the tissue, a factor demanded by Sir Almroth Wright.
The antiseptic preparation introduced by this group consisted of a mixture of equal parts of chloride of lime and boric acid. The boric acid served to liberate free hypochlorous acid which was considered to be the real antiseptic agent. The hypochlorous acid could be used either as a gas by the addition of a small amount of water to the powdered mixture, or in solution when larger amounts of water were used. They found that a concentration of 0.5 per cent of hypochlorous acid was the most satisfactory as stronger solutions decomposed rapidly until reaching 0.5 per cent after which the decomposition was less rapid. They also recommended the use of the mixture while dry on some types of wounds. The mixture of solids was given the name of "Eupad" and the solution of free hypochlorous acid was called "Eusol."

During this investigation they tested many other antiseptics including phenol, tincture of iodine, potassium permanganate, salicylic acid, and hydrogen peroxide, all of which they found to be inferior to hypochlorous acid.

Shortly after this discovery by Professor Smith, Dr. Dakin (7) published the results of the work of his colleagues. The first step in their study was to enumerate the different factors which had to be considered in the selection of an antiseptic substance. These factors included, in addition to germicidal activity, toxicity, solubility, ability to penetrate the tissues and be absorbed, and the chemical reaction with protein and other constituents. Another distinction which differentiates these from previous workers in this field is that they considered the difference in germicidal activity of an antiseptic on microorganisms suspended in water
as against those suspended in pus, blood serum and other tissue exudate.

Among the other substances tested were phenol, hydrogen peroxide, mercuric chloride, silver nitrate, and iodine. Phenol was found to have very low germicidal power especially in the presence of serum. Hydrogen peroxide was very effective when used in a test tube but when used on wounds it was found to decompose rapidly with very little germicidal activity when in the presence of the enzyme, catalase, found in all tissues and in the blood cells. Mercuric chloride readily lost its germicidal properties in the presence of many tissue constituents and was very irritating even in dilute solutions. Silver nitrate was of greater value than mercuric chloride but when used in sufficiently high concentrations was found to be very irritating. The hypochlorite preparation of Smith when used dry was found to be quite irritating to the skin and tended to form lumps which hindered drainage.

Dr. Dakin proposed the use of a 0.5 per cent solution of sodium hypochlorite which had a small amount of boric acid added to buffer the solution and keep its alkalinity content from becoming too great. This solution has come to be known as Dakin's solution. The great objection to eau de Javal and eau de Labarraqne, as has been pointed out, was due to the high alkalinity content which tended to irritate the skin. Dr. Dakin believed that the solution had to be buffered because even though the solution were rendered neutral when prepared it would tend to become more alkaline as time progressed due to the reaction:

$$\text{NaOCl} + \text{H}_2\text{O} \rightarrow \text{NaOH} + \text{HOCI}$$

The increase in alkalinity was due to the presence of the sodium hydroxide
formed during the hydrolytic dissociation of the sodium hypochlorite. It may be again stated here that Professor Smith and Dr. Dakin had different reasons in mind for the use of boric acid in their respective preparation. The former used it to liberate free hypochlorous acid from the chloride of lime while the latter used it to buffer the solution and thereby keep down the alkalinity content.

Dr. Dakin made the following claims for his solution: It would kill staphylococci suspended in water in two hours at a concentration of hypochlorite between 1:500,000 and 1:1,000,000, while in the presence of serum the necessary concentration must be between 1:1,500 and 1:2,000. Streptococci were killed more rapidly than were staphylococci. The solution was not irritating to the skin. Its decomposition products were devoid of toxicity. Its action was local and it had a solubility action on necrosed tissue.

When free hypochlorous acid or a solution of one of its salts reacts with an organic compound containing a secondary amine group \( R_1 \text{NH} \), the first reaction almost always consists of the replacement of the hydrogen by the chlorine with the formation of a chloramine. Since all protein substances contain a large number of these groups it is natural to suppose that they would react readily with hypochlorites, a supposition which is readily proved in the laboratory. It was, therefore, believed that the antiseptic action of Dakin's solution was due to this reaction taking place in the wound.

Recent work by Charlton and Levine (6) has indicated that the disinfecting action of hypochlorite solutions is due to the presence of und dissociated hypochlorous acid.
While the credit for the discovery of Dakin's solution belongs to Dr. Dakin, its method of application is attributable to his co-worker Dr. A. Carrel. The essential part of the Carrel method consists of keeping Dakin's solution in contact with the wound continuously until the bacterial count made from a smear shows the wound to be sterile. The first step in the procedure is the mechanical removal of all foreign matter, torn and necrotic tissue. The skin around the wound is then made sterile and is covered with sterile Vaseline to protect it from the Dakin's solution. Continuous instillation of Dakin's solution into the wound so that the concentration is always kept constant and at a maximum is the ideal which is sought in this method. The solution must be changed continuously since it changes in concentration as soon as it comes in contact with the tissue. Continuous instillation is usually carried out at a constant rate, normally 5 or 6 drops per minute as regulated by a pinch cock and a drop counter. Since continuous instillation is not feasible save in the case of small, cup-shaped wounds, intermittent application is commonly used.

Pure rubber tubes, the number depending on the size of the wound, closed at one end and perforated along the sides are covered with gauze and packed into the wound. The entire wound is then protected by a covering. The tubes are connected with a reservoir of Dakin's solution and the solution is run in for a few seconds every two hours.

The dressings should be changed every day, but in cases of emergency may be allowed to go for several days. Bacterial counts show that the wounds are usually free from pus in from one to three days and are sterile in five to nine days, depending upon the nature of the wound and its con-
dition when first treated. After sterilization has taken place the wound may then be closed either by sutures or held together with adhesive tape.

Necrotic tissue and accumulated pus are usually attacked first, thus producing an exceptionally clean wound. Lymph is caused to flow in the healthy tissue which tends to protect it from action of the hypochlorite. It is only in the case of lowly vascularized tissue, such as tendons, that the solution will attack healthy tissue after the wound has become clean. Since it dissolves blood clots, care must be exercised to ward off secondary hemorrhages.

Dakin solution and the Carrel method were developed primarily for the treatment of war wounds; however, they have been found to be very valuable in the treatment of wounds resulting from industrial accidents as well as other infections. A complete bibliography concerning Dakin's solution and the Carrel method and the uses to which they have been put can be found in the Iowa Engineering Experiment Station Bulletin 111 (21).

Later work led Daufresne (3), who assisted Dr. Dakin in the biological and chemical testing of the antiseptics, to believe that certain irritation phenomena were caused by the boric acid in the original Dakin formula. As a result in the solution which was most commonly used sodium bicarbonate was substituted for the boric acid to buffer the solution. Commercial preparations of Dakin's solution frequently contain acetanilide as a stabilizing agent. Potassium permanganate is also used to stabilize it and give it a distinctive color (21).
C. The Electrolytic Hypochlorite Cell

The first evidence of the preparation of a hypochlorite solution electrolytically for antiseptic use is found in an article by Dr. Dakin (8) which stated that Dr. Heitz-Boyer and the staff of the French hospital ship, Charles Roux, had been making extensive use as an antiseptic of a hypochlorite solution of between 0.4 per cent and 0.2 per cent NaOCl prepared by the electrolysis of sea water in a simple cell using carbon electrodes. A second evidence is found (12) where electrolyzed sea water was used on hospital ships carrying infectious cases as an antiseptic to prevent secondary infections among the ships' staffs. It was stated by Carrel and Dehelly in 1918 (3) that it was possible to obtain a solution even less irritating than Dakin's solution if it were prepared by electrolytic methods. Sodium hypochlorite is prepared in an electrolytic cell according to the following well-known reactions:

1. \[ \text{NaCl} \rightarrow \text{Na}^+ + \text{Cl}^- \]
2. \[ 2\text{Cl}^- \rightarrow 2 \text{e}^- \rightarrow \text{Cl}_2 \]
3. \[ \text{Cl}_2 + 2\text{OH}^- \rightarrow \text{Cl}^- + \text{ClO}^- + \text{H}_2\text{O} \]

Sodium chloride in solution ionizes according to equation 1. The chloride ions are discharged at the anode according to equation 2. The chlorine reacts with the hydroxyl ions present in the solution to form chloride ions, hypochlorite ions and water according to equation 3. The sodium ions then react with those ions to form sodium chloride and sodium hypochlorite.

In 1928, Ralph Van Peursem (23)(24)(21), in the Chemical Engineering Department of Iowa State College, commenced work on the development of a
foolproof electrolytic cell for the production of a hypochlorite solution from aqueous sodium chloride which would fulfill the requirements as to concentration and alkalinity set down by Dr. Dakin for his hypochlorite antiseptic solution. The cell which he used consisted of a cylindrical platinum foil cathode 1 inch in diameter and 2 inches long and a flat platinum foil anode 5/8 inch wide and 4 inches long mounted in the center of the cathode. The cell body was constructed of glass tubing, 1 5/8 inches in diameter and 4 inches long, open at the top with the bottom drawn out to a small tube and bent up along the side of the cell and over in a siphon shape to maintain a constant level in the cell. The electrolyte, a saturated solution of sodium chloride, was dropped into the cell from a dropping funnel. The current was supplied by a 6 volt storage battery. This cell was not successful in that it was difficult to produce a solution of high enough concentration and low enough alkalinity to meet the requirements of Dakin's solution. A Tungar rectifier was also used as the source of power but due to the fluctuations in the current which it produced it was found necessary to stabilize it by floating a battery in parallel with the rectifier. Van Peursem's work indicated that the idea of making hypochlorite electrolytically was feasible, but that a different type cell would be needed.

In 1929, B. K. Pospishil and W. D. Harris (24)(15)(21) continued the work started by Van Peursem. They constructed a cell using nichrome electrodes which they found to decompose almost as soon as current was passed through the cell. Tantalum was likewise found to be unsatisfactory as it became brittle and decomposed rapidly in the cell. It was believed
that by using tantalum and carbon for the electrodes alternating current could be rectified in the cell. Since the heat of rectification was liberated in the cell together with the heat of electrolysis, too high temperatures were obtained in the cell and the idea was abandoned. Carbon electrodes were next tried. These were found to be more satisfactory than other material tested previously. Experiments with a test cell proved so promising that a commercial cell was designed and constructed. This cell had carbon electrodes and operated with a rate of flow of 6 cubic centimeters per minute, using a current of 1.4 amperes. Hypochlorite solution well within the desired concentration could be obtained after the first 10 minutes of operation. The alkalinity of the solution was also in the desired range. A Balkite rectifying unit, without using a storage battery, produced the direct current. It was on this cell that the first type of automatic valve was used. It was arranged in such a manner that as soon as the current was shut off the flow of electrolyte to the cell was stopped. In both the experimental and the commercial cells the salt solution was fed in at the bottom with the hypochlorite solution being drained off the top.

Further work by Hanna (11)(21) led to the discarding of carbon electrodes in favor of ones made of platinum. Electrode decomposition together with bulky electrodes caused this shift. An automatic cell which he constructed in 1930 consisted of an all glass cell, having small, thin, perforated platinum discs 5/8 inch in diameter as electrodes. In contrast to previous cells, these electrodes were sealed into the glass sides of the cell. A 5 per cent sodium chloride solution was used as the electrolyte. This was fed from a chicken feeder type reservoir, so that a constant head
could be maintained, through an automatic valve of the solenoid type, and out an orifice into the cell. The electrolyte entered the cell at the bottom, was passed up through the electrodes and out through an opening at the top. A Balkite rectifier was used to furnish the direct current which could be controlled by a radio rheostat.

This later automatic valve consisted of a piece of glass tubing with an iron nail sealed into it, placed in the center of a solenoid which was set above the orifice of delivery. The bottom end of the tube was ground to fit the orifice so that an easy moving valve was obtained. The solenoid was wired in series with the electrodes so that when current was flowing in the circuit the solenoid was actuated and the glass tube was lifted out of its ground seat and salt solution could flow through the orifice. When the current was shut off the glass tube dropped back into its seat and the flow of salt solution was cut off.

In June 1930, Paul A. Frank of Akron, Ohio, after reviewing the work already done on the antiseptic cell and realizing what a boon such an apparatus would be to the medical field, approached the authorities of Iowa State College with the idea of sponsoring further research on this cell and ultimately putting the finished cell on the market. This idea was acceptable to the College and culminated in an arrangement being made whereby Frank would be given a license to manufacture and market any cell which might culminate from the research which he would finance.

In December 1930 (2), the research was made a project of the Engineering Experiment Station, and Joseph E. Baker was hired to carry out the work. The first step in the research was the finding of more suitable materials (21) to be used in the construction of the electrodes. Various
alloys were tested and it was finally found that an alloy of 80 per cent platinum, 20 per cent iridium as the cathode, and pure rhodium as the anode gave the best results. It was likewise found that inverted cone shaped electrodes made of thin perforated sheets gave the best results.

Since a solution was desired which could be used directly on the wound without dilution it was found necessary to reduce the concentration of sodium chloride in the electrolyte to 2.5 per cent, even though more concentrated electrolytes gave solutions which were at a lower temperature and which electrolyzed much faster and used less current than those which were more dilute. It was found that 0.2 per cent of sodium bicarbonate when added to the electrolyte decreased electrode decomposition and kept the hydrogen ion concentration at about pH 9.5 to 10. It was also found that small amounts of potassium chromate, about 0.025 per cent, when added to the electrolyte produced a higher concentration of hypochlorite than when it was not used.

Various types of rectifying units were used to produce the direct current necessary to operate the cell. Up until 1935, no rectifier either of the dry plate or tube types was found suitable for use in a commercial cell. As a result when the final cell was designed it was found necessary to use a specially designed motor generator set.

The plans for the finished commercial cell were drawn up in July of 1931. The cell constructed entirely of glass had 1.5 inch diameter cone shaped electrodes, the cathode or upper electrode made of the platinum-iridium (80-20) alloy while the anode, or lower electrode, made of rhodium. These were sealed into the glass cell with platinum leads. Electrolyte was fed into the cell from the bottom through a tube which was
bent upwards in a U-bend and extended the full height of the cell. Brass
ferrules were placed around this feed tube to which were attached the wires
from the electrode leads. The body of the cell was streamlined to pro-
hibit turbulent flow of the electrolyzed solution which flowed upwards past
the electrodes and out of the cell through a short spout from which it
dropped into a receiver.

A glass trap with a solenoid-operated valve, described on page 20,
was mounted over the cell in such a position that the electrolyte flowing
through the orifice passed into the feed tube of the cell. The trap also
contained a Filtros cap which filtered the electrolyte. Above the trap,
hung from a rubber covered ring, was an inverted 2-liter flask which served
as an electrolyte reservoir.

Current was supplied to the cell from a specially designed motor
generator set having an output of 10.5 amperes at 7 volts. An ammeter,
indicating the current used in the cell, was placed in the line together
with the solenoid for the automatic valve. An electrical timer was
placed in the line leading to the alternating current motor so that the
machine could be set to produce any desired quantity of solution and would
shut itself off automatically when that amount was made. The cell pro-
duced solution at the rate of 25 c.c. per minute.

All units were mounted on a specially treated asbestos panel by nickel-
plated brackets which made a very neat and compact piece of apparatus. A
patent for the cell was applied for by Dr. O. K. Sweeney and when granted
was assigned to Iowa State College.

Frank had in the meantime organized a company known as the Electro-
lytic Antiseptic Company to manufacture and market the finished cell.
The selling price was in the neighborhood of $350.00.
When the company applied to the American Medical Association for their endorsement of the cell, they refused to give it on the grounds that the presence of small amounts of rhodium resulting from the slight decomposition of the anode in the hypochlorite solution might render it unsafe for use on wounds. This attitude was held by them in spite of the fact that the solution had been used by the Iowa State College Hospital for 2 years without any trouble and that for years some Swiss doctors had injected rhodium intravenously for the treatment of pyemia.

In order to determine whether or not the rhodium in the solution would produce any toxic results, a pathological investigation was carried out by Dr. O. H. Plant (14) of the State University of Iowa. The findings of this investigation, published in The Journal of Pharmacology and Experimental Therapeutics, showed that the amounts of rhodium present in the hypochlorite solution were too small to produce any toxic results when the solution was used in the treatment of wounds.

The American Medical Association finally, after much controversy, accepted the hypochlorite solution and included it in its list of New and Unofficial Remedies in 1936. The electrolytic cell received an enthusiastic acceptance on the market and was used by such institutions as the Rush Memorial Hospital with marvelous success. For reasons of policy in 1937, Frank decided to temporarily discontinue his promotion of the cell and was released from his agreement with Iowa State College.

From June 1935, when the development of the hypochlorite cell ceased to be a project under the Engineering Experiment Station, until 1940 little concerted work was done on this project save that of Ries (17) who, in 1936, modified the automatic control.
From previous experience with the electrolytic cell marketed by
the Electrolytic Antiseptic Company, it was found that in order for an
apparatus of this type to receive great acceptance on the market it must
have a selling price of around $150.00. Such a price would enable
physicians to have them in their private offices. It was also found that
the motor generator set needed to be cared for at times which demanded
the services of a man experienced in caring for electrical machines of
this type. Any work done from 1936 until 1940 was with the idea of finding
a rectifying unit of the dry plate type suitable for use in place of the
motor generator set.

In 1941 Walter Spencer (20) showed for the first time that a dry plate
rectifier could be used if the cell was decreased in size so that about
10 c.c. of solution would be produced per minute with a corresponding
decrease in the current required.

Work was continued by Paul McElherne (13) who designed and tested a
cell in which the electrolyte was fed into the cell through a side arm
from a trap into which the electrolyte was run from an inverted flask.
The electrolyte passed down through the electrodes and out an orifice in
the bottom of the cell. When the liquid in the reservoir and trap were
exhausted the current continued to flow through the cell until the electrodes
became dry at which time the flow of current stopped on the direct current
side of the rectifier. It was necessary to switch off the current manually
from the A.C. side of the rectifier.

With the war now in progress it is necessary that a cell be designed
as quickly as possible which can be easily manufactured at a low cost and
which will fulfill the requirements for use in this emergency. It was with this thought in mind that a cell was developed which will be described in the next section of this thesis.
EXPERIMENT

A. Batch Cell

1. Design of the cell

It is quite probable that the cell mentioned by Dr. Dakin (8) as having been used in the French hospital ship, Charles Roux, was a batch type cell. By a batch type cell is meant a cell into which a definite quantity of electrolyte is introduced, the current being allowed to flow for a definite length of time after which the entire amount of hypochlorite solution is drawn off at one time. This is in contrast to a continuous cell in which a reservoir of electrolyte is used and from which the hypochlorite solution is drawn continuously as it is produced.

Since it is the objective of this research to develop a simple electrolytic cell for the production of an antiseptic saline hypochlorite solution, it is quite natural that one would turn to the batch type of equipment for by doing so one eliminates to a great extent the use of costly control equipment.

The only reference in literature to the description of a batch type cell for the production of a hypochlorite solution for antiseptic purposes is that of Carey's (4). The cell described consisted of a platinum anode and a carbon cathode inserted through a rubber stopper into an open vessel similar to a beaker. Cooling coils made of glass through which water ran were also inserted through this same stopper. A current of 1 ampere at
12 volts was used to electrolyze a solution of 18 per cent sodium chloride. The cell produced a hypochlorite solution containing about 1 per cent sodium hypochlorite at the rate of 6 pints in 25 hours. This solution was diluted to about half its strength and used in the envelope treatment for burns.

Up until the present time there has been no work done at Iowa State College on the development of a cell of this type. All previous work has been on the continuous type of cell.

The first experimental cell consisted of a platinum screen cathode and a rhodium screen anode, 1 inch in diameter, placed horizontally with the cathode above the anode. Electrical connections were made by sealing the leads from the electrodes into 5 m.m. glass tubes which were held apart from each other by two cross pieces of tubing of the same diameter, sealed off in the center so that no connection could be made between the two legs and making it look much like a ladder with two rungs. Mercury was run into the glass legs and copper wires dipped into the mercury completing the contacts. The electrode assembly was mounted on a panel by a small piece of wood and placed high enough so that a 400 c.c. beaker could be placed beneath the electrodes and held up in place by a block. The electrodes were placed about 1/32 of an inch apart.

It was found that the hydrogen gas which was produced during electrolysis had a tendency to collect between the two electrodes and caused the current to fluctuate considerably. Agitation, produced by means of an electric motor and glass stirrer, helped somewhat to stabilize the current; however, it still varied too much to get any accurate results.

The electrodes were finally placed in a vertical position with approximately a 1/32 inch clearance between them. The current variation
ceased and it was found that agitation was not necessary. Figure 1 is a picture of the experimental cell while Figure 2 is a diagram of the cell proper as used during testing.

Current was supplied to the cell by a Battery Booster type dry plate rectifier produced by the B. L. Electric Company of St. Louis, Missouri, and sold by Sears Roebuck and Company. Its rated capacity was 6 volts at 5-10 amperes.

Test showed that it took approximately one minute of electrolysis for every 10 c.c. of solution being electrolyzed. The electrolyte was a 2.5 per cent solution of sodium chloride with 0.15 per cent of sodium bicarbonate added to buffer the resulting hypochlorite solution. When the electrolysis was started with the electrolyte at room temperature it was found that the resulting hypochlorite solution was at a temperature of about 70° C. A solution of this temperature would not only be too hot to use directly on a wound but the high temperature also increases the rate of electrode decomposition as was evidenced by the color of the solution and tends to increase the formation of chlorate at the expense of the hypochlorite already present in the solution.

It was suggested that the electrolysis be started with the temperature of the electrolyte lower than room temperature. When a solution was electrolyzed in which ice had been used in its makeup, it was found that the temperature of the hypochlorite solution produced was lowered. The use of ice was objectionable on the grounds that it was hard to handle and presented difficulties in the making up of an electrolyte of the proper strength. Further tests were run with an electrolyte which was cooled in an ice-water bath. Very satisfactory results were obtained. In
Figure 1. Experimental Model of Batch Type Cell.
Fig. 2. Batch Cell—Experimental Model.
all the work which was done on this cell the idea of its commercial use was kept in mind. As a result conditions which could be met only in the laboratory were avoided as much as possible.

An apparatus such as this lends itself readily to the design of a cell which could be put on the market for about $100.00. It can be used either on 100 volt alternating current with the rectifier, on a storage battery or on stepped down direct current. It can be made sturdy so that it can be used under the most severe conditions. It is not likely to get out of order. With the rectifier being used at one half its rated capacity there is every indication that it should have a long service life.

The tentative design of a commercial cell is shown in Figure 3. The cell as shown is made to be fastened by a clamp onto a panel and can be moved up to permit the placing of a flask in place for electrolysis and then lowered into the flask. A flaring of the leg of the cell prevents its being lowered too far and hence endangering the head of the cell. The electrodes are of the same size, shape, and material as used in the experimental cell and are held at a specific distance apart by glass-bead spacers. To impart strength and rigidity to the electrodes the leads are brought in at the center rather than at the edge as with the experimental cell. An insulated, flexible double wire cord extends from the binding posts on the panel down through the leg of the cell and is split into two branches at the Y. The separate wires are then dipped into the mercury which makes contact with the sealed in electrode leads. The branches of the Y are extended down past the lower edges of the electrodes to help protect them from injury.

The panel besides having the clamp and the binding posts fastened to
Fig. 3. Tentative Design of Commercial Cell.
(a) Detail of Cell Proper.
(b) Schematic Diagram of Entire Assembly.
it also contains the electrical timer and an ammeter to indicate the current being used. The latter is not absolutely necessary. The rectifier which is the source of power for the unit shown is mounted behind the panel.

A beaker should be used in which to carry out the electrolysis. Such a flask would eliminate any explosion hazard due to the accumulation of hydrogen as a result of the electrolysis.

A model to be used in an ambulance would be modified somewhat in that it would be necessary for it to withstand severe jars and joumings. In order to prevent a sloshing out of the solution, a soxhlet extraction flask should be used in which to carry out the electrolysis. This should also be clamped to the panel during the time of reaction. Owing to the narrowing down of the neck of the flask, open flames should be kept away from the cell while it is in operation owing to the possibility of hydrogen accumulation in the flask.

2. Operation of the cell

a. Relationship of time of electrolysis to concentration of hypochlorite solution. The first step in determining the operating characteristics of the cell was to determine the relationship between the time of electrolysis and the concentration of the resulting hypochlorite solution. The tests were carried out by electrolyzing definite quantities of electrolyte for varied lengths of time and then after determining the concentration of the resulting solutions, plotting these values against the time. Previous work had shown that it was necessary to cool the electrolyte before
electrolysis in order to keep the temperature of the finished product down and to decrease the electrode decomposition.

Table 1 shows the operation of the cell starting with electrolyte at 70°C. The electrolyte was cooled to this temperature by immersing a flask of the electrolyte in an ice-water bath. The electrolyte consisted of a 2.5 per cent sodium chloride solution with 0.15 per cent sodium bicarbonates. Reagent grade chemicals and distilled water were used in the making up of the electrolyte. The direct current was supplied to the cell by a Battery Booster type dry plate rectifier. The data given in Table 1 are plotted in the graphs of Figure 4. It can be seen that nearly straight lines are obtained and that the slope of the curves depends upon the volume of solution electrolyzed. These indicate that there is a direct relationship between the time of electrolysis and the concentration of hypochlorite.

Similar tests were run with the initial temperature of the electrolyte at 12°C. and 17°C. The data for the former run are found in Table 2 and Figure 5, while those for the latter are found in Table 3 and Figure 6. The composition of the electrolyte and the rectifier used were the same for these last two series of tests as for the first series mentioned.

A perusal of the tables shows a direct relationship between the current flowing through the cell and the temperature of the solution in it. The resistance is lowered as the temperature rises which causes an increase in the amount of current flowing through the cell.

It will be also noted that after various quantities of solution have been electrolyzed for equivalent lengths of time the temperature and concentration of the hypochlorite solutions are nearly the same.
### Table 1

The Operation of the Batch Type Cell

Initial Temperature of Electrolyte 7° C.

<table>
<thead>
<tr>
<th>Quantity, c.c.</th>
<th>Time, minutes electrolyte, solution</th>
<th>Temperature of solution, °C.</th>
<th>Temperature of Room, °C.</th>
<th>Amperes</th>
<th>Grams NaOCl per 100 c.c.</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>7.5</td>
<td>31.5</td>
<td>28</td>
<td>2.95-4.00</td>
<td>0.339</td>
</tr>
<tr>
<td>100</td>
<td>10.0</td>
<td>39.0</td>
<td>28</td>
<td>2.90-4.30</td>
<td>0.447</td>
</tr>
<tr>
<td>100</td>
<td>12.5</td>
<td>46.5</td>
<td>28</td>
<td>2.90-4.55</td>
<td>0.537</td>
</tr>
<tr>
<td>200</td>
<td>15.0</td>
<td>32.5</td>
<td>30</td>
<td>2.95-4.00</td>
<td>0.347</td>
</tr>
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<td>20.0</td>
<td>39.5</td>
<td>29</td>
<td>2.95-4.35</td>
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<tr>
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<td>25.0</td>
<td>46.5</td>
<td>31</td>
<td>2.95-4.55</td>
<td>0.548</td>
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<tr>
<td>300</td>
<td>22.5</td>
<td>33.2</td>
<td>27</td>
<td>3.00-4.10</td>
<td>0.557</td>
</tr>
<tr>
<td>300</td>
<td>30.0</td>
<td>41.0</td>
<td>30</td>
<td>3.00-4.35</td>
<td>0.467</td>
</tr>
<tr>
<td>300</td>
<td>37.5</td>
<td>46.0</td>
<td>27</td>
<td>3.00-4.60</td>
<td>0.557</td>
</tr>
</tbody>
</table>
Chart 1: Relationship between time of electrolysis and concentration of hypochlorite.

Chart 2: Initial temperature of electrolyte vs. time of electrolysis.

- Chart 1 shows the concentration of hypochlorite as a function of time for different electrolyte concentrations.
- Chart 2 indicates the initial temperature of the electrolyte over time for various electrolysis times.
<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Minutes Electrolyte Solution</th>
<th>Quantity Time of</th>
<th>Temperature Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.670</td>
<td>12</td>
<td>2.0 20-4.70</td>
<td>12</td>
</tr>
<tr>
<td>0.671</td>
<td>12</td>
<td>2.0 20-4.50</td>
<td>12</td>
</tr>
<tr>
<td>0.676</td>
<td>12</td>
<td>2.0 20-4.35</td>
<td>12</td>
</tr>
<tr>
<td>0.683</td>
<td>12</td>
<td>54.0 20-4.75</td>
<td>12</td>
</tr>
<tr>
<td>0.673</td>
<td>12</td>
<td>46.0 20-4.65</td>
<td>12</td>
</tr>
<tr>
<td>0.675</td>
<td>12</td>
<td>36.8 20-4.35</td>
<td>12</td>
</tr>
</tbody>
</table>

Table 2

---

Initial Temperature of Electrolyte 120°C

The operation of the batch type cell
Table 3

The Operation of the Batch Type Cell

Initial Temperature of Electrolyte 17° C.

<table>
<thead>
<tr>
<th>Quantity, c.c.</th>
<th>Time, minutes electrolyte</th>
<th>Temperature of Electrolyte, °C</th>
<th>Temperature of Solution, °C</th>
<th>Amperes</th>
<th>pH</th>
<th>Grams NaCl per 100 c.c.</th>
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</thead>
<tbody>
<tr>
<td>100</td>
<td>7.5</td>
<td>17</td>
<td>41.5</td>
<td>3.40-4.50</td>
<td>9.40</td>
<td>0.577</td>
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<td>100</td>
<td>10.0</td>
<td>17</td>
<td>47.5</td>
<td>3.35-4.55</td>
<td>9.50</td>
<td>0.484</td>
</tr>
<tr>
<td>100</td>
<td>12.5</td>
<td>17</td>
<td>54.0</td>
<td>3.40-4.80</td>
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<td>0.571</td>
</tr>
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<td>200</td>
<td>15.0</td>
<td>17</td>
<td>39.5</td>
<td>3.30-4.25</td>
<td>9.35</td>
<td>0.377</td>
</tr>
<tr>
<td>200</td>
<td>20.0</td>
<td>17</td>
<td>47.0</td>
<td>3.30-4.50</td>
<td>9.35</td>
<td>0.484</td>
</tr>
<tr>
<td>200</td>
<td>25.0</td>
<td>17</td>
<td>52.5</td>
<td>3.40-4.70</td>
<td>9.45</td>
<td>0.583</td>
</tr>
<tr>
<td>300</td>
<td>22.5</td>
<td>17</td>
<td>39.8</td>
<td>3.30-4.20</td>
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<td>0.377</td>
</tr>
<tr>
<td>300</td>
<td>30.0</td>
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<td>45.5</td>
<td>3.30-4.45</td>
<td>9.10</td>
<td>0.484</td>
</tr>
<tr>
<td>300</td>
<td>37.5</td>
<td>17</td>
<td>51.0</td>
<td>3.35-4.65</td>
<td>9.15</td>
<td>0.579</td>
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</tbody>
</table>
CONCENTRATION OF HYPHOCHLORITE SOLUTION FOR VARIOUS QUANTITIES OF ELECTROLYTE

CONCENTRATION OF ELECTROLYTE AND RELATIONSHIP BETWEEN TIME OF ELECTROLYTE AND INITIAL TEMPERATURE OF ELECTROLYTE-17°C.

TIME OF ELECTROLYTE (Min/Sec)

CONCENTRATION OF NaOCl (gms./100cc.)
b. Effect of the initial temperature of the salt solution on the electrolysis. Since the amount of electrolysis depends upon the current flowing and as there is a direct relationship between the temperature of the electrolyte and the amount of current flow, it is reasonable to expect that when the initial temperature of the salt solution is raised the amount of electrolysis will be increased and that for a given time of electrolysis the concentration of hypochlorite will be greater than when a cooler salt solution is used. From this it would be expected that the curves indicating the operation of the cell with rising electrolyte temperature would rise progressively for each increase in initial temperature. Figure 7 is a plot of the 200 c.c. lines from Figures 4, 5, and 6, which shows that for each increase in initial temperature there is a corresponding rise in the curves. Data for these curves are found in Table 4. There is almost a direct relationship between this increase in temperature and the upward shift of the operation curves.

c. Operation of the cell with a storage battery as the source of current. A series of tests were carried out to determine the possibility of using this cell in an ambulance with the storage battery as the source of current. A regular automobile storage battery was used in place of the rectifier. The battery was charged to capacity before starting this work to insure optimum operation. The procedure of testing and the electrolyte used were the same as in previous tests.

In order that these tests might be compared with the tests previously carried out with the rectifier, the salt solution was cooled down to 70 C. After an equivalent time of electrolysis it was found that as well as being lower in concentration the solutions from these tests were in the neighbor-
Fig. 7. Effect of Changing Initial Temperature of Electrolyte on Hypochlorite Concentration.
<table>
<thead>
<tr>
<th>Initial Temperature (°C)</th>
<th>Current, in °C</th>
<th>Temperature, in °C</th>
<th>Temperature of Room, in °C</th>
<th>Time, in minutes</th>
<th>Quantity of Electrolyte, in g</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.85-4.70</td>
<td>0.58</td>
<td>0.58</td>
<td></td>
<td>7</td>
<td>25</td>
</tr>
<tr>
<td>2.85-4.70</td>
<td>0.42</td>
<td>0.42</td>
<td></td>
<td>7</td>
<td>20</td>
</tr>
<tr>
<td>2.85-4.70</td>
<td>0.58</td>
<td>0.58</td>
<td></td>
<td>7</td>
<td>25</td>
</tr>
<tr>
<td>2.85-4.70</td>
<td>0.42</td>
<td>0.42</td>
<td></td>
<td>7</td>
<td>20</td>
</tr>
<tr>
<td>2.85-4.70</td>
<td>0.58</td>
<td>0.58</td>
<td></td>
<td>7</td>
<td>25</td>
</tr>
<tr>
<td>2.85-4.70</td>
<td>0.42</td>
<td>0.42</td>
<td></td>
<td>7</td>
<td>20</td>
</tr>
<tr>
<td>2.85-4.70</td>
<td>0.58</td>
<td>0.58</td>
<td></td>
<td>7</td>
<td>25</td>
</tr>
<tr>
<td>2.85-4.70</td>
<td>0.42</td>
<td>0.42</td>
<td></td>
<td>7</td>
<td>20</td>
</tr>
</tbody>
</table>
hood of 19 degrees cooler than were those when the rectifier was used. Since the amount of current flowing from the battery was less than that from the rectifier it was believed that this alone caused the lower temperature. It was found on plotting the data that the curve indicating the operation with the battery was not only lower than that of the rectifier but it had a decidedly different slope. This would seem to indicate that the amount of heat produced by the battery was very much less than that produced by the rectifier. This is believed to be due to the fact that the current produced by the battery is steady and unvarying while that produced by the rectifier is cyclic in character, varying from a maximum value to zero and back to maximum 120 times per second. The current registered on the ammeter, when the rectifier is used, is the average ordinate of the amperage curve. Figure 8 is a comparison of the operation of the cell with a rectifier and that with a battery. Data for the latter curve can be found in Table 5.

In previous tests with the rectifier it was found necessary to cool the electrolyte before electrolysis to keep the temperature of the resulting hypochlorite solution as low as possible. Since the battery did not produce as much heat as the rectifier it was found that cooling of the electrolyte was unnecessary. A series of tests were carried out with the initial temperature of the electrolyte at 15°C. Unlike when the rectifier was used, the current which flowed through the cell at the start of the electrolysis did not increase when the initial temperature of the electrolyte was increased. The relationship between the operations of the cell with the initial temperature of the electrolyte at 7°C and 15°C is shown in the lower two curves of Figure 9.
Fig. 6. Comparison of Operation of Rectifier and Storage Battery.

Fig. 8. Operation of Cell with Battery and Generator at 2°C.

Fig. 9. Operation of Cell with Battery at 7°C & 15°C, and with Battery and Generator at 2°C.
Table 5
Operation of the Batch Cell Using a Storage Battery as the Source of Current

<table>
<thead>
<tr>
<th>Quantity, c.c.</th>
<th>Time, minutes</th>
<th>Temperature of electrolyte, °C</th>
<th>Temperature of solution, °C</th>
<th>Room temperature, °C</th>
<th>Amperes</th>
<th>pH</th>
<th>Grams NaOCl per 100 c.c.</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>7.5</td>
<td>7</td>
<td>20.0</td>
<td>30</td>
<td>2.30-2.40</td>
<td>9.0</td>
<td>0.240</td>
</tr>
<tr>
<td>100</td>
<td>10.0</td>
<td>7</td>
<td>23.3</td>
<td>30</td>
<td>2.30-2.50</td>
<td>9.1</td>
<td>0.310</td>
</tr>
<tr>
<td>100</td>
<td>12.5</td>
<td>7</td>
<td>26.0</td>
<td>30</td>
<td>2.30-2.60</td>
<td>9.2</td>
<td>0.371</td>
</tr>
<tr>
<td>100</td>
<td>15.0</td>
<td>7</td>
<td>28.5</td>
<td>29</td>
<td>2.30-2.55</td>
<td>9.3</td>
<td>0.419</td>
</tr>
<tr>
<td>100</td>
<td>17.5</td>
<td>7</td>
<td>30.8</td>
<td>28</td>
<td>2.20-2.60</td>
<td>9.3</td>
<td>0.455</td>
</tr>
<tr>
<td>100</td>
<td>7.5</td>
<td>15</td>
<td>25.5</td>
<td>28</td>
<td>2.30-2.45</td>
<td>9.1</td>
<td>0.259</td>
</tr>
<tr>
<td>100</td>
<td>12.5</td>
<td>15</td>
<td>31.0</td>
<td>27</td>
<td>2.25-2.60</td>
<td>9.2</td>
<td>0.375</td>
</tr>
<tr>
<td>100</td>
<td>17.5</td>
<td>15</td>
<td>36.5</td>
<td>27</td>
<td>2.25-2.70</td>
<td>9.3</td>
<td>0.477</td>
</tr>
</tbody>
</table>
It was noted when the cell was operated with the battery that the current did not increase proportionately with the time as was seen when the rectifier was used. It is believed that a storage battery when producing currents of such a magnitude tends to become exhausted with a corresponding decrease in voltage.

A series of tests was next performed using an automobile battery and generator (with the motor running) as the source of current. The operation of this arrangement was more satisfactory than when the battery was used alone. The salt solution in this case was used at 27°C. The data for this series of tests are given in Table 6 and are shown as the top curve in Figure 9.

It will be seen that all three curves obtained when a battery was used as the source of current have the same general slope indicating that the amount of heat produced is the same whether the generator is used or not. The falling off of the curves at the upper ends is believed to be due to a decrease in the current produced by the battery after it has been used for a considerable length of time. It will be noted that the current passing through the battery when the generator was used in conjunction with it was much greater than when the battery was used alone. A test was made to determine if this larger current was due to the particular battery used or whether it was due to the generator. When the battery was used alone it was found that the current dropped back to the values obtained when the other battery was used. It should be noted here that the battery and generator used in conjunction with each other were in one of the Engineering Experiment Station test cars; the battery used alone was a spare
Table 6

Operation of the Batch Cell Using an Automobile Battery and Generator (with the Motor Running) as the Source of Current

<table>
<thead>
<tr>
<th>Quantity, c.c.</th>
<th>Time, minutes</th>
<th>Temperature of salt solution, °C</th>
<th>Temperature of solution, °C</th>
<th>Room temperature, °C</th>
<th>Amperes</th>
<th>pH</th>
<th>Grams NaOCl per 100 c.c.</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>7.5</td>
<td>27</td>
<td>40.8</td>
<td>27</td>
<td>3.50-3.70</td>
<td>9.2</td>
<td>0.350</td>
</tr>
<tr>
<td>100</td>
<td>10.0</td>
<td>27</td>
<td>42.5</td>
<td>29</td>
<td>3.20-3.60</td>
<td>9.3</td>
<td>0.413</td>
</tr>
<tr>
<td>100</td>
<td>12.5</td>
<td>27</td>
<td>45.5</td>
<td>29</td>
<td>3.10-3.45</td>
<td>9.4</td>
<td>0.473</td>
</tr>
<tr>
<td>100</td>
<td>15.0</td>
<td>27</td>
<td>47.5</td>
<td>28</td>
<td>3.15-3.55</td>
<td>9.5</td>
<td>0.525</td>
</tr>
</tbody>
</table>
battery loaned to the Chemical Engineering Department by the Engineering Experiment Station.

It may be said in general that the cell can be made to operate with a battery as the source of current; however, the time of electrolysis will be longer than when the rectifier is used and an automobile generator preferably of the heavy duty type should be used in conjunction with it.

d. The effect of salt concentration on the electrolysis. A suggestion was made that it might be possible to improve the operation of the cell and to cut down the time of electrolysis if a more concentrated salt solution were used than that which had been used in previous tests. Previous work had shown that for a given hypochlorite concentration, concentrated salt solutions caused the cell to operate at a lower temperature than dilute solutions. It is well to remember at this time that salt solutions containing a high percentage of sodium chloride tend to cause desiccation when applied to a wound with resulting irritation; hence if concentrated salt solutions are used for the electrolysis they must be diluted before application.

To test the operation of the cell when high salt concentrations were used and to determine whether or not it would be practical to follow the suggestion given above, several runs were made on solutions containing varying amounts of sodium chloride. The data for these runs are given in Table 7 and Figure 10.

It will be seen that the concentration of the hypochlorite solution increases as the salt concentration is increased but not in a 1:1 ratio as would be required if the salt concentration were to be kept at about 2 grams per 100 c.c. It is true that in case we were using a salt solution
## Table 7

**Effect of Varying Salt Concentrations on the Electrolysis**

<table>
<thead>
<tr>
<th>Quantity, Concentration of salt, c.c.</th>
<th>Time, minutes</th>
<th>Temperature of salt solution, °C.</th>
<th>Temperature of solution, °C.</th>
<th>Room, temperature, °C.</th>
<th>Amperes per 100 c.c.</th>
<th>Grams NaCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>25</td>
<td>20</td>
<td>7</td>
<td>39.5</td>
<td>29</td>
<td>2.95-4.35</td>
</tr>
<tr>
<td>200</td>
<td>50</td>
<td>20</td>
<td>7</td>
<td>45.0</td>
<td>32</td>
<td>3.60-5.50</td>
</tr>
<tr>
<td>200</td>
<td>75</td>
<td>20</td>
<td>7</td>
<td>46.5</td>
<td>32</td>
<td>3.90-5.70</td>
</tr>
</tbody>
</table>
Fig. 10. Effect of Salt Concentration.

Fig. 11. Effect of NaHCO₃ Concentration.
The sodium orthododecahydroborate was kept at 25 C for 1 hour. The sodium orthododecahydroborate was varied from 0 to 4.6 grams per liter in the salt solution. A series of experiments was run in which the concentration of sodium orthododecahydroborate was varied from 0 to 4.6 grams per liter in the salt solution on the operation of the batch buffer. The resulting solution and kept the pH within the desired range.

Previous work on the electrolyte cell showed that sodium orthododecahydroborate formed a highly concentrated and advantageous over the other. Although borate and hydrochloric acid was used in the original solution held the advantage over previously introduced hypochlorite solutions.

Effect of sodium orthododecahydroborate on the electrolyte solution.

Work in this direction

Tests were made to be too unstable and erratic to cause any advantages. Tests on when the same amount of ice and water were used in every case. Tests on when the same amount of ice and water were used in every case.

According to Figure 10, comparable results would be obtained. Since the electrolytes vary with the concentration of the salt solution, the electrolytes vary with the ratio of the salt solution.

When the salt solution was mentioned previously, it will be noted that such a practice would lead to erratic results by the cell. In regard to the use of ice in the makeup of the salt solution which

and decreasing the time of electrolysis.

Determining our purpose of keeping the temperature of cell operation low 100 C and then adjust it to 20 C. By doing so, however, we would be 100 C on the solution until it reached a concentration of 0.96 grams per 100 C of the solution until it reached a concentration of 0.96 grams per liter of sodium orthododecahydroborate.
Table 6

Effect of Sodium Bicarbonate on the Electrolysis

<table>
<thead>
<tr>
<th>Quantity, c.c.</th>
<th>Concentration of NaHCO₃, grams per liter</th>
<th>Time, minutes</th>
<th>Temperature of salt solution, °C.</th>
<th>Temperature of solution, °C.</th>
<th>Room temperature, °C.</th>
<th>Amperes</th>
<th>pH</th>
<th>Grams NaOCl per 100 c.c.</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>0.0</td>
<td>20</td>
<td>7</td>
<td>41.5</td>
<td>34</td>
<td>3.05-4.35</td>
<td>9.3</td>
<td>0.452</td>
</tr>
<tr>
<td>200</td>
<td>1.5</td>
<td>20</td>
<td>7</td>
<td>40.5</td>
<td>33</td>
<td>2.95-4.20</td>
<td>9.3</td>
<td>0.443</td>
</tr>
<tr>
<td>200</td>
<td>3.0</td>
<td>20</td>
<td>7</td>
<td>40.5</td>
<td>33</td>
<td>2.90-4.20</td>
<td>9.2</td>
<td>0.434</td>
</tr>
<tr>
<td>200</td>
<td>4.6</td>
<td>20</td>
<td>7</td>
<td>41.0</td>
<td>34</td>
<td>2.90-4.20</td>
<td>9.2</td>
<td>0.433</td>
</tr>
</tbody>
</table>
These data indicate that the sodium bicarbonate is not necessary for the batch cell. The hypochlorite solution produced when the sodium bicarbonate is not present in the sodium chloride solution is of the same alkalinity as when the bicarbonate is used. The sodium bicarbonate tends only to decrease the initial current in the cell and by doing so decreases the final hypochlorite concentration.

f. General observations. The batch type cell has proven itself to be a very reliable and foolproof cell. Slight changes in room temperatures (5-60 C.) do not seem to affect its operation. Since the amount of electrolysis depends on the current which is itself dependent upon the temperature of the solution, moderate deviations in temperature would bring about a change in its operation. The range in concentration permissible for Dakin's solution, however, permits these changes to be made and still have the solution fall within the proper limits of concentration. The only place where the apparatus would be subject to great temperature changes is in an ambulance and since it has been shown that the amount of heat produced in the cell when operating on a battery is small when compared with the rectifier, the flask could be jacketed to prevent heat loss from the solution and the instrument calibrated for such operation.

A test was made in which potassium chromate was added to the salt solution. The concentration of potassium chromate was 0.025 per cent. It was found that when 200 c.c. of the solution was electrolyzed for 20 minutes with an initial temperature of 70 C. the resulting solution contained a hypochlorite concentration of 0.502 grams per 100 c.c. The salt solution without the chromate when treated under the same conditions gave a solution with a hypochlorite concentration of 0.449 grams per
100 c.c. The temperatures of the resulting solutions were the same in these tests.

The hypochlorite solutions produced by the batch type cell were tested for chlorate as well as for hypochlorite. It was found that the concentration varied between 0.002-0.003 grams per 100 c.c. There seemed to be little or no correlation between the conditions of operation and the chlorate concentration. It is quite natural that the chlorate concentration should be low as the temperatures of the solutions were never over 54° C.

The cell when sold should be accompanied by an operation sheet either in the form of a graph if the timer is calibrated in minutes or it can be incorporated on the face of the timer. A sample operation sheet in the form of a graph is given in Figure 12. The data for this are given in Table 9. The data were obtained by plotting straight horizontal lines on Figures 4, 5, and 6, and dropping perpendiculars from the intersections of this line with the operating lines to the time axis. These abscissas were plotted against volume of salt solution and three lines, each one corresponding to a different initial salt solution temperature, were obtained. It is to be noted that these lines, although parallel and straight after reaching the 200 c.c. mark curve in such a manner as to pass through the 0-0 point on the axes.

In using this sheet, an operator would have only to select the volume of solution he desired, take its temperature and look up on the sheet to see the necessary time of electrolysis. By keeping the salt solution already made up in a refrigerator, which is usually handy in a doctor's office or hospital, the initial temperature of the electrolyte would always be constant and there should be uniform solutions obtained at all times.
### Table 9

Data for Operation Sheet for Batch Cell

<table>
<thead>
<tr>
<th>Quantity, c.c.</th>
<th>Grams NaOCl per 100 c.c.</th>
<th>Temperature of salt solution, °C</th>
<th>Time, minutes</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>0.475</td>
<td>7</td>
<td>10.80</td>
</tr>
<tr>
<td>100</td>
<td>0.475</td>
<td>12</td>
<td>10.10</td>
</tr>
<tr>
<td>100</td>
<td>0.475</td>
<td>17</td>
<td>9.90</td>
</tr>
<tr>
<td>200</td>
<td>0.475</td>
<td>7</td>
<td>21.40</td>
</tr>
<tr>
<td>200</td>
<td>0.475</td>
<td>12</td>
<td>20.25</td>
</tr>
<tr>
<td>200</td>
<td>0.475</td>
<td>17</td>
<td>19.50</td>
</tr>
<tr>
<td>300</td>
<td>0.475</td>
<td>7</td>
<td>31.40</td>
</tr>
<tr>
<td>300</td>
<td>0.475</td>
<td>12</td>
<td>30.25</td>
</tr>
<tr>
<td>300</td>
<td>0.475</td>
<td>17</td>
<td>29.50</td>
</tr>
</tbody>
</table>
Fig. 12. Operation Sheet For Batch Cell.

Time of Electrolysis Required to Produce a Given Volume of Hypochlorite Solution With Concentration of 0.475 Gram Per 100 cc.
It is believed by some that the proper method of treating a wound is to first apply a hypochlorite solution of slightly higher concentration than is called for by Dakin's solution and to then progressively decrease this concentration until salt solution alone is used. This technique could be carried out very satisfactorily with the batch cell, by preparing the solution in small batches and each time progressively decrease the time of electrolysis according to curves supplied with the instrument.

It has been found that there is very little or no decomposition of the electrodes during operation. Electrode decomposition is usually evidenced by the presence of a slight bluish tint in the hypochlorite solution. At no time while the cell was being tested was it possible to detect any color in the solution. The fact that the temperature is kept low in the cell accounts for this absence of electrode decomposition. This should lead to long service life for the cell and cut down its cost of operation.

3. Cost data for batch cell

A preliminary estimation of the cost of manufacturing for the batch type cell shown in Figure 3 has given the following data:

<table>
<thead>
<tr>
<th>Item</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electrodes - material and labor</td>
<td>$6.00</td>
</tr>
<tr>
<td>Rectifier</td>
<td>5.00</td>
</tr>
<tr>
<td>Time switch</td>
<td>4.25</td>
</tr>
<tr>
<td>Ammeter</td>
<td>3.85</td>
</tr>
<tr>
<td>Panel</td>
<td>2.00</td>
</tr>
<tr>
<td>Base with pads</td>
<td>3.10</td>
</tr>
<tr>
<td>Nest of beakers</td>
<td>1.25</td>
</tr>
<tr>
<td>Cell support</td>
<td>1.50</td>
</tr>
<tr>
<td>Glass and glass blowing for cell</td>
<td>5.00</td>
</tr>
<tr>
<td>Miscellaneous wiring and assembly</td>
<td>10.00</td>
</tr>
<tr>
<td></td>
<td>$41.85</td>
</tr>
</tbody>
</table>
It can be seen that a selling price of $100.00 for a cell of this type would enable the manufacturer to pay at least a 10 per cent royalty, meet overhead and development expenses, and still realize a handsome profit.

By eliminating the ammeter and the time switch the manufacturing cost could be cut to $33.85 with a correspondingly lower selling price. It would then be necessary for the operator to turn the cell off manually and to time the electrolysis with a pocket watch or clock. The removal of the time switch would remove the safety feature on the cell and might cause trouble and discontent among the operators.

Estimates of the operating cost on the cell, figuring on the use of the cell three hours a day with a selling price of $100.00, a cell life of six years, and a useful life of the rest of the apparatus of ten years, give the following information:

<table>
<thead>
<tr>
<th>Cost per liter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Distilled H₂O at 8 cents a gallon $0.0211</td>
</tr>
<tr>
<td>Sodium chloride 25 grams per liter at 4 cents a pound 0.0022</td>
</tr>
<tr>
<td>Sodium bicarbonate 1.5 grams per liter at 30 cents a pound 0.0010</td>
</tr>
<tr>
<td>Current 120 watt hours at 7.5 cents per kilowatt hour 0.0090</td>
</tr>
<tr>
<td>Cell cost $11.00 lasting 6 years 0.0030</td>
</tr>
<tr>
<td>Depreciation on original cost $100 - $11 for cell 0.0154</td>
</tr>
<tr>
<td>$0.0497</td>
</tr>
</tbody>
</table>

It should be stated that the cost for water is rather high and amounts to one third of the total cost. Zeolite softened water can be used in place of distilled water and is much less expensive.

If the sodium chloride and sodium bicarbonate were purchased already weighed up, their costs would be much greater than those given above.

The use of the apparatus for more than three hours a day would also
lower the cost per liter by decreasing the depreciation on the original cost. At the present time there is no other antiseptic that can be purchased for such a price.

B. Continuous Cell

1. Design of the cell

Although the batch type of cell has distinct advantages over a continuous cell in some cases, especially when small quantities of hypochlorite solution are needed, it is quite evident that there are places where the continuous cell could be used to an advantage. This is especially true, for example, in a hospital where large quantities of the solution are needed over a period of time.

Development work was extended on the continuous type cell, to simplify it as far as possible and to incorporate in it certain devices which would make it more foolproof. This work was begun where McElherne (13) left off. He had endeavored to feed the sodium chloride solution directly into the cell and failing in this had gone over to the idea of feeding the sodium chloride solution through a side tube from a trap.

The main trouble with feeding the sodium chloride solution directly into the cell was that the hydrogen which was formed during the electrolysis passed up into the reservoir and made it impossible to keep a steady rate of flow into the cell. As a result the head within the cell increased and the flow of hypochlorite solution from the cell was erratic, hence a variable concentration of NaOCl in the electrolyzed solution. It was found that if the salt solution delivery tube was modified so as to contain
a few holes through which the saline solution could be discharged, the passage of hydrogen gas through the delivery tube into the reservoir could be minimized.

Up until this time, the reservoir was operated like that used in the cell sold by the Electrolytic Antiseptic Company in that the solution was allowed to drain away from the delivery tube until the surface broke away from the tube and a bubble of air passed up into the reservoir with a corresponding discharge of salt solution into the cell body. It was found that when the end of the delivery tube was closed off with the exception of the holes on the sides, the surface tension around the holes was too great to permit the action to take place.

To eliminate this difficulty, an air inlet tube was introduced as is shown in Figure 14. This air inlet tube enabled the liquid to flow out of the holes, the condition being met that the distance between the tip end of the air inlet and the holes in the delivery tube were spaced far enough apart to enable the bubble of air to be sucked out of the air inlet tube. The tip end of this tube was ground off at an angle in order to decrease this distance. It was found that the aforementioned distance was about a half inch depending upon the diameter of the air inlet tube and the size of holes in the delivery tube.

In order to insure a steady flow of small bubbles of air through the air inlet tube it was found necessary to end the air inlet tube on the outside with a capillary sized opening. Since at that time there was no way of closing off the reservoir from the delivery tube nor an opening in the reservoir for refilling, it was found necessary to withdraw the entire assembly each time it became empty. It often happened, during this re-
filling process, that the air inlet tube became filled with liquid. As a result the capillary ending also was filled with liquid and when the assembly was inverted and put back into place, the air inlet system did not function properly. To eliminate this the form of ending as shown in Figure 14 was introduced. The bulbous end tended to draw liquids away from the capillary opening on the top side of the tube. Since introducing the shut-off between the reservoir and delivery tube and the opening for refilling, the necessity for removal and inversion of the assembly has been eliminated and there has been no case where the air inlet tube has been filled with liquid.

Continued work on the end of the delivery tube showed that the mere closing off of the end with the introduction of holes on the side did not solve the problem completely. It was found that small bubbles of hydrogen gas at times entered the delivery tube and over a period of time this caused a material change in the head of liquid within the cell. Upturned tips with the holes in the ends were tried. This type of end worked more satisfactorily; however, in the end it was necessary to turn to the mushroom type of end which is shown in Figure 14. This type not only eliminated the entrance of hydrogen gas into the delivery tube, but it also formed a cup around the holes and eliminated the possibility of hypochlorite solution being pumped back up into the reservoir and thereby causing erratic results in the concentration of the finished hypochlorite solution. The continuous flow of small air bubbles into the reservoir permits an almost constant head to be maintained in the cell body.

McElherne's cell had the outlet orifice at the bottom and as a result this had to be very small and very close to the electrodes in order to have
a flow of about 10 c.c. of hypochlorite solution per minute. This small hole always presented the possibility of its being partially or completely plugged and cause the rate of delivery to be lessened. It might be pointed out at this time that it is necessary that the rate of liquid flow as well as the rate of current flow through the cell be maintained at a constant value in order to obtain a solution of uniform concentration.

To eliminate as far as possible the presence of a minute sized orifice in the cell it was found necessary to draw the cell down to a smaller sized tubing just beneath the electrodes and to bring this back up along the side of the cell and bend it over again in an S shape. The end of the S was drawn into an orifice, which, because the head acting on the orifice could now be cut down to a fraction of an inch, could be much larger than that in McElherne's cell. Tests on this showed that the rate of flow from this type of outlet was variable and depended upon the amount of liquid just above the orifice in the outlet tube.

The top end of the S outlet tube was cut off and a small cup with two openings, one to fasten onto the remainder of the S tube and the other an outlet orifice was substituted for it. This cup can be seen in Figure 14. It enabled a steady rate of flow to be maintained. The orifice as shown was drawn down and ground off with a large surface around it so as to permit the formation of a drop and eliminate the possibility of surface tension drawing the hypochlorite solution up along the bottom of the cup and down the side of the tube.

With this arrangement the use of an orifice many times the size of any used in previous designs has been made possible. This eliminates to a great extent the possibility of the orifice being clogged. Should such a
thing happen, the orifice is in a very accessible position and can be easily cleared with a fine wire. In all the work which has been done with this cell there has never been an occasion when it was necessary to clear out this orifice. To eliminate the possibility of dust collecting in this cup a cap could be placed over it. It is necessary, however, that this cup be open to the atmosphere to obtain a steady rate of flow.

As was mentioned before, the first experimental setup necessitated the removal of the entire delivery assembly in order to refill the salt solution reservoir. This showed itself to be a very clumsy and messy arrangement. It was decided that a valve of some sort be interposed between the reservoir and the delivery tube. The first type of valve which came into consideration was of the well-known ground glass stopcock type.

Since this piece of apparatus is intended for use by persons unfamiliar with ordinary chemical laboratory equipment, it was believed that the stopcock might prove to be too delicate for such use. Ground glass stopcocks are bound to stick especially when in contact with saline solutions and even people very familiar with ground glass equipment often break them while trying to loosen them. The possibility of frequent breakage, together with the fact that a glass stopcock with a bore large enough to permit the air bubbles from the air delivery tube to pass through was very expensive, caused them to be rejected.

The next type of valve considered was of the ground glass plunger type as shown in Figure 14. Although this type of valve suffers the same possibility of becoming stuck it was believed that since the force necessary to dislodge such a valve would be in a straight pull rather than in a twisting motion there would be less possibility of breakage taking place
in the hands of an amateur. This type of valve was accepted and used in
the experimental model. The possibility of connecting the two sections
with a piece of rubber tubing which could be pinched off with a pinch
clamp instead of having it in one integral unit was considered but it was
discarded because each time the rubber was pinched an inching up of the
rubber on the delivery tube took place and a lowering of the head within
the cell was noted.

When the plunger type valve was first used, the shaft was stuck directly
through the rubber stopper in the top of the reservoir. Friction between
the rubber and the shaft caused the raising and lowering of the plunger to
be a difficult operation. This was eliminated by the introduction of a
piece of glass tubing of a diameter slightly larger than that of the shaft
directly into the stopper. The shaft was then inserted through the tubing
and a seal between the two was maintained by a short piece of rubber tubing.
This arrangement produced a good seal and enabled easy movement of the
plunger.

A second opening was introduced in the top of the reservoir through
which the salt solution was introduced. This opening was sealed off with
a rubber stopper. It should be emphasized here that in order to maintain a
constant head in the cell there should be no entrance of air into the
reservoir save through the air inlet tube. Entrance of air through this
tube is controlled by the level of the liquid below the tip end of this
tube.

A picture of an experimental model of the continuous cell can be
seen in Figure 15. Figure 14 is a diagram of the cell proper and Figure
15 shows the essential portions of the wire diagram.
Figure 13. Experimental Model of Continuous Cell.
Fig. 14. Continuous Cell--Experimental Model.
Fig. 5: Wiring Diagram for Continuous Cell.
The body of the cell was made of 28 mm pyrex glass tubing 3.5 inches long. The bottom of the cell was drawn down to 10 mm and a tube sealed on which was bent up along the side of the tube and sealed to the cup which was made of 28 mm tubing and was about 1 inch in height. The orifice was approximately 1/32 of an inch in diameter and would deliver a rate of flow from 9 c.c. upwards depending upon the head of liquid in the cell.

The electrodes were 7/8 inch in diameter and were inverted cone shaped with a base angle of about 30 degrees. The cathode or top electrode was made of perforated sheet platinum iridium (80-20) and the anode or bottom electrode was made of perforated sheet rhodium. The electrodes were sealed into the side walls of the cell body about 1/16 of an inch apart and were spaced by glass beads. Lead wires from the electrodes were fastened to tabs on the panel to which the cell was mounted. An opening was made just beneath the electrodes and a 10 mm pyrex tube sealed on to permit the entrance of a thermometer to measure the temperature in the cell. The thermometer was held in place by a short piece of rubber tubing.

The delivery tube was about 6 inches long and was constructed of 18 mm pyrex glass tubing. The air delivery tube was made of 5 mm pyrex glass tubing. An additional outlet was made by a 5 mm tube for connection onto a manometer which was a part of the automatic switch which will be discussed in the next section of this thesis. The bottom of the delivery tube ended in a mushroom type end which was 22 mm in diameter. Two holes allowed the salt solution to drain from the delivery tube.

The top of the delivery tube was fastened onto the bottom of the salt solution reservoir which was fitted with a plunger type ground glass valve
described on page 64 and with an opening for introducing the salt solution. The reservoir used on the experimental model held approximately 360 c.c. of salt solution. This was of course rather small and necessitated the making of short runs. A commercial model should have a capacity of at least 2 liters and would be of the same general design as the experimental cell with the omission of the thermometer.

The entire assembly was mounted on a wooden panel with the rectifier mounted behind. The rectifier was of the same type used with the batch type cell. An ammeter of the laboratory type was placed in the direct current circuit to show the amount of current flow through the cell.

2. **Automatic devices**

One of the disadvantages of the Electrolytic Antiseptic cell, manufactured and sold by the Electrolytic Antiseptic Company, was in connection with the electrical timer which shut off the current supply to the cell. The timer was designed so that it could be set for any amount of solution desired. The cell was regulated to deliver 25 c.c. of solution per minute and the timer was calibrated in terms of quantity. However, it was possible to set the timer to produce a greater quantity of solution than was present in the reservoir. Since the electrolyzed solution in the cell was forced out by the entrance of the salt solution, when the flow of salt solution from the reservoir ceased the hypochlorite solution which was already present in the cell continued to be electrolyzed for the remaining amount of time for which the timer had been set. As can be seen this was very injurious to the electrodes since the temperature of the
cell was raised depending on the length of time during which this took place which caused increased electrode decomposition.

Every effort was made in this research to develop an automatic device which would shut off the current as soon as the reservoir was empty. The idea of using a float and a mercury switch was considered. This, however, proved to be too cumbersome and was discarded.

An attempt was made to introduce auxiliary electrodes into the delivery tube with an e.m.f. of approximately one volt impressed between them. It was believed that a small current, large enough to operate a relay, would flow between these electrodes as long as they were covered with salt solution and that the current would be cut off as soon as the last of it had passed between them. The relay was to be cut into the 110 volt A.C. power supply leading to the rectifier and would cut off the power as soon as the relay was opened. When the salt solution again flowed past the electrodes, the relay would be closed and power could again flow to the rectifier. The one volt impressed between these electrodes, being less than the decomposition voltage of sodium chloride, would not produce any electrolysis.

Various types of electrodes were investigated and it was found that the maximum current which could be made to flow was in the neighborhood of 1-2 milliamperes which was too small to operate a relay. Professor Ryder of the Electrical Engineering Department suggested the use of a vacuum tube to amplify this current and permit the use of a relay. Since it was one of the objectives of this research to develop a cell as simple as possible, it was believed that such an arrangement would be too complicated and expensive and the idea was temporarily discarded. It is
believed that this type of shutoff would prove very satisfactory should it be installed. Lack of proper equipment precluded its being tested during this research.

It was found that when the plunger valve was opened allowing the salt solution to flow, filling up the delivery tube and starting flow through the cell, a partial vacuum was developed in the system which continued to hold until the last of the salt solution had drained from the delivery tube. The amount of vacuum thus drawn depended upon the length of the delivery tube. When a manometer was connected to this tube it was found that with the length of delivery tube used in the experimental cell, 6 inches, that a vacuum amounting to 1-2 mm. of mercury was obtained.

The existence of this vacuum presented a good nucleus around which to design an automatic control device. A U type manometer was constructed of 5 mm. pyrex glass tubing into which were sealed two nichrome wires, one in the bottom of the U and the other midway up on the low pressure side. The wire on the low pressure side was bent into a right angle form with the leg pointing downwards so as to produce a sharper make and break with the mercury. This arrangement functioned as a mercury switch which was placed in the 110 volt A.C. line leading to the rectifier.

The switch worked satisfactorily; however, an arc was produced each time a contact was made or broken. A small amount of transformer oil was placed over the mercury and helped somewhat to cut down the arcing. The presence of a bulb, blown on the high pressure side across from the electrode and of the transformer oil above the mercury, seemed to increase the distance traveled by the mercury meniscus. This, likewise, produced a sharper make and break of the contacts.
The arcing, although lessened by these additions continued to be troublesome. To insure long life and constancy of operation the arcing had to be eliminated since it would in time cause the mercury to oxidize which would destroy the sensitivity of the switch. It was decided that in order to do this, it would be necessary to lower the voltage across the switch. This could be brought about by the use of a suitable relay.

A Mercoid relay Type V2-3A, manufactured by the Mercoid Corporation, was obtained by a loan from the College Instrument Shop. The transformer of this relay, which cut down the voltage across the manometer switch to about 12 volts, was placed in parallel with the rectifier while the load contacts were in series with the rectifier. This relay worked very satisfactorily and the device consisting of the manometer and relay has proved to be a very satisfactory type of control device.

A cheaper relay could be used in place of the Mercoid relay. Since the amount of current flowing across the load contacts is less than 1 ampere there is little chance of the contacts burning very badly.

This automatic device permits the operator to put the desired amount of salt solution in the reservoir, open the plunger valve, and forget about the cell as the current is automatically turned on when the valve is opened. This will continue to flow until the supply of salt solution has become exhausted at which time it will be shut off. The cell can be stopped at any time merely by closing the plunger valve. With the reservoir shut off the solution which is already in the delivery tube will run out and the current will automatically be switched off when the delivery tube is emptied. This device eliminates any prolonged electrolysis as was the case in previous commercial models.
3. Operation

a. Operation of the cell at different rates of current and solution flow. The concentration of a hypochlorite solution obtained from the continuous cell depends upon two factors, the rate of solution flow through the cell and the flow of current across the electrodes. These two factors must be kept constant if the strength of the hypochlorite solution is to be uniform.

In this cell the rate of solution flow depends upon the head of liquid acting on the orifice. This was considered in the design of the electrolyte feed system and has been discussed in that section of this thesis. The question arose as to what the effect of varying amount of solution in the reservoir would have on the rate of solution flow through the cell. To clarify this a series of tests was run in which varying amounts of liquid were placed in the reservoir, the cell turned on and the rate of flow measured. The results of this series are given in Table 10.

Table 10

<table>
<thead>
<tr>
<th>Amount of liquid in reservoir at the start of electrolysis</th>
<th>Rate of flow, c.c. per minute</th>
<th>Solution temperature, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Full</td>
<td>7.7</td>
<td>50</td>
</tr>
<tr>
<td>3/4 Full</td>
<td>7.7</td>
<td>53</td>
</tr>
<tr>
<td>1/4 Full</td>
<td>7.7</td>
<td>--</td>
</tr>
</tbody>
</table>
These data show that there is no change in the rate of flow of the solution when the amount of liquid in the reservoir is varied. This enables the operator to make any quantity of solution he desires by merely measuring that amount of salt solution into the reservoir and permitting the cell to run until it shuts itself off. It should be noted here that once the plunger valve is opened, the pressure in the reservoir and delivery tube remains the same until the delivery tube runs dry or the stopper is removed from the reservoir.

It has been noticed that the hydrogen liberated during the electrolysis has a tendency to decrease the head in the cell and that the flow of solution is less when electrolysis is taking place than when it is not. This, however, does not change while the cell is in operation and constancy of flow is maintained.

The rate of current flow across the electrodes depends upon the voltage applied, the area of the electrodes, the distance between them, the concentration of the salt solution, and the temperature within the cell. The first three of these factors are incorporated in the design of the cell. The fourth can be kept constant and the fifth depends upon the rate of solution flow and the heating characteristics of the current used.

To determine the operating characteristics of the cell two series of tests were run each at a different rate of solution flow. Each test in each series had a different rate of current flow. The current was varied by introducing a rheostat in the direct current line.

The results obtained were instantaneous values in that the tests were performed on samples taken at the specific time indicated. It is seen that
these values tend to fluctuate a little depending upon the operation of
the cell at that time. To obtain values which could be taken as repre-
sentative of the entire run, averages were taken which are given in
Table II. The instantaneous values are found in the Appendix.

Table II

Operation of Continuous Cell at Various Rates
of Current Flow with Rectifier - S-7846

<table>
<thead>
<tr>
<th>Test</th>
<th>Rate, c.c. per minute</th>
<th>Amperes</th>
<th>Solution temperature, °C.</th>
<th>NaOCl grams per 100 c.c.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.3</td>
<td>1.56</td>
<td>36.4</td>
<td>0.346</td>
</tr>
<tr>
<td>2</td>
<td>5.5</td>
<td>3.05</td>
<td>53.3</td>
<td>0.493</td>
</tr>
<tr>
<td>3</td>
<td>5.3</td>
<td>4.66</td>
<td>70.6</td>
<td>0.504</td>
</tr>
<tr>
<td>4</td>
<td>7.7</td>
<td>2.10</td>
<td>37.2</td>
<td>0.534</td>
</tr>
<tr>
<td>5</td>
<td>7.7</td>
<td>3.38</td>
<td>48.9</td>
<td>0.441</td>
</tr>
<tr>
<td>6</td>
<td>7.7</td>
<td>4.70</td>
<td>61.3</td>
<td>0.484</td>
</tr>
</tbody>
</table>

Figure 16 is a plot of the data given in Table II. It can be seen
that the concentration of the hypochlorite solution for any given rate of
solution flow increases with the amount of current flowing. The relation-
ship, however, is not a direct one and the curves tend to flatten out as
the concentration of hypochlorite is increased. This tendency is very
pronounced in the curve indicating the smaller rate of flow. With in-
creasing rates of solution flow the curves tend to approach a straight line.

Up until this time a rectifier, Serial No. S-7846 sold by the Sears
Fig. 16. Operation of Continuous Cell at Various Rates of Current Flow With Rectifier S-7846.

Fig. 17. Operation of Continuous Cell at Various Rates of Current Flow With Rectifier T-1819.
heat then does the old rectifier. Figure 12 is a comparison of the
the new rectifier delivers a current which produces a greater amount of
and that after that point the concentration is reversed. It is believed that
then it does when operating with the old rectifier up to about 46 emperes
constant flow produces a solution with a lower hypochlorite concentration.
that the cell when operating with the new rectifier at a chlorine rate of
7.7 e. per minute is found in Figure 16. It is seen from these curves that a
operation curves obtained with the new rectifiers at a solution flow rate of
that a stoppage curve is obtained in both cases. A comparison of the
section of the cell is somewhat the same with each of the rectifiers in
these data are plotted in Figure 17. It can be seen that the oper-

<table>
<thead>
<tr>
<th>0.467</th>
<th>72.5</th>
<th>6.22</th>
<th>7.7</th>
<th>12</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.496</td>
<td>69.6</td>
<td>9.46</td>
<td>7.7</td>
<td>9</td>
</tr>
<tr>
<td>0.504</td>
<td>60.2</td>
<td>22.7</td>
<td>7.7</td>
<td>8</td>
</tr>
<tr>
<td>0.509</td>
<td>59.6</td>
<td>3.7</td>
<td>7.7</td>
<td>4</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>100 °F.</th>
<th>0.0 °F.</th>
<th>0.0 °F.</th>
<th>100 °F.</th>
<th>0.0 °F.</th>
<th>0.0 °F.</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.10</td>
<td>9.10</td>
<td>9.10</td>
<td>9.10</td>
<td>9.10</td>
<td>9.10</td>
</tr>
</tbody>
</table>

**Table 12**

Operation of Continuous Cell at Various Rates

The data are given in Table 12.

A series of tests was run with a solution flow of 7.7 e. per
rectifier, sorted No. 1-1979, were purchased from the Kentucky Mard and

Kochuck Company, and been used to supply the desired current. A new
Fig. 18. Comparison of Operation With the Different Rectifiers.

Fig. 19. Comparison of Cell Temperatures Obtained at Different Rates of Current Flow With the Different Rectifiers.
temperatures observed in the cell at various rates of current flow from
the two rectifiers. It can be seen that at any given rate of current flow
the new rectifier produces more heat and causes the cell to run at a
higher temperature than does the old rectifier.

b. **Operation of the cell to produce solution of hypochlorite con-
centration 0.45-0.50 grams per 100 c.c.** In order to obtain the maximum
of output from the cell it was necessary that the electrodes be connected
directly to the rectifier without a rheostat in the line. This was done
so that the full amount of current could be passed through the cell.
After a number of trials it was found that a solution could be obtained
whose hypochlorite concentration fell midway between the limits prescribed
for Dakin's solution at the rate of about 10.5 c.c. per minute. A test
run lasting 90 minutes was made. It was found that over this period with
an average rate of current flow of 4.9 amperes and an average rate of
solution flow of 10.6 c.c. per minute a solution of 0.482 grams NaOCl
per 100 c.c. was obtained at an average temperature of 60° C. This test
was made on a salt solution containing 25 grams of NaCl and 1.5 grams of
sodium bicarbonate per liter and the new rectifier was used. The pH of
the solution obtained was 8.8. The hypochlorite solution had a light
bluish tint indicating a small amount of rhodium was present. The data
for this test can be found in the Appendix as test No. 11. Analysis showed
that chlorates were not present in the solution.

c. **General observations.** It was noted that the amount of rhodium
present in the solution as was evidenced by the intensity of the bluish
tint to the solution increased as the temperature within the cell was
raised. By keeping the temperature not higher than 60° C., the amount of rhodium was kept at a minimum.

It may be noted that the pH of the hypochlorite solution produced in the continuous cell was somewhat lower than that produced in the batch type cell and also that in both types the pH varied with the concentration of the hypochlorite solutions low in hypochlorite having the lower values of pH. It is believed that the pH of the solution is regulated by the amounts of sodium bicarbonate and sodium carbonate present in the solution. Sodium bicarbonate when reacted upon by sodium hydroxide forms sodium carbonate according to the following reaction:

\[
\text{NaHCO}_3 + \text{NaOH} \rightarrow \text{Na}_2\text{CO}_3 + \text{H}_2\text{O}.
\]

Solutions containing sodium carbonate have a higher pH value than those containing sodium bicarbonate.

A test was performed in which a salt solution containing 25 grams of NaCl and 1.5 grams of NaHCO₃ per liter was electrolyzed. Before electrolysis the salt solution had a pH of 8.1 while after electrolysis it had a pH of 8.8. A second solution containing 25 grams of NaCl and 1.5 grams of Na₂CO₃ per liter was electrolyzed under the same conditions. Its pH value both before and after electrolysis was 10.2. A third solution containing 25 grams of NaCl per liter was electrolyzed. Before electrolysis this salt solution had a pH of 5.7 while after electrolysis the pH value was 9.3.

The electrolysis of the salt solution containing neither carbonate nor bicarbonate seems to indicate that some NaOH is present in the electrolyzed solution. The electrolysis of the solution containing the
tests might be made.

As was performed on the same cell in order that correlation between the water and due to the presence in these electrolytes used to make the necessary that all tests be made to remove any impurities. The difference in electrolyte species in the experimental cells and electrodes must be set in order to insure interferences. For the can be put into manufacturing limits of tolerance in the sparging of the electrolyte is necessary for a platinum cell operation. Before these cells are

than. It is believed that the presence of a titrim of platinum black on the cathode is necessary for a platinized hydrogen cathode can produced a solution having a consistent hydrogen cathode concentration.

a new cell was made a considerable period of operation was required before constructed. The solution was not used with this cell as each time constructed. It is believed to be necessary with the cathode was needed when a cell had been first staged in the buttress on the electrolyte.

Sweeney and Baker (21) believe that from the batch type cell

then that from the batch type cell would be lower expect that the pH of the solution from the continuous cell would be lower than the pH in the batch type cell. It is reasonable then to better condense for the reaction of the sodium hydroxide on the cathode. Flashed over the electrodes and the bubbling of the hydrogen gas produced

It is believed that in the continuous cell the domino flow of the while the solution is being electrolyzed.\* that some of the sodium hydroxide has been changed into sodium carbonate

with the pH shifted from 9.1 to 8.6, during electrolysis seems to indicate the presence. The electrolytes of the solution containing sodium carbonate present. The sodium carbonate indicates what pH value can be expected when Na CO₃ is
4. Cost data

Estimates on the cost of a commercial model of the continuous cell give the following:

<table>
<thead>
<tr>
<th>Item</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electrodes (material and labor)</td>
<td>$10.00</td>
</tr>
<tr>
<td>Glass and glass blowing</td>
<td>25.00</td>
</tr>
<tr>
<td>Rectifier</td>
<td>5.00</td>
</tr>
<tr>
<td>Relay (Mercoid)</td>
<td>3.60</td>
</tr>
<tr>
<td>Panel</td>
<td>2.00</td>
</tr>
<tr>
<td>Base with pads</td>
<td>2.10</td>
</tr>
<tr>
<td>Cell support</td>
<td>1.50</td>
</tr>
<tr>
<td>Reservoir support</td>
<td>1.50</td>
</tr>
<tr>
<td>Flask</td>
<td>1.00</td>
</tr>
<tr>
<td>Miscellaneous wiring and assembly</td>
<td>15.00</td>
</tr>
<tr>
<td></td>
<td>$71.70</td>
</tr>
</tbody>
</table>

This cost can be decreased by the use of a cheaper relay which can be purchased for about $3.00, or the automatic feature can be eliminated entirely with a corresponding decrease in the above cost to about $61.00.

The commercial model of the cell with the automatic feature could be retailed for about $150.00.

Estimates of the operating cost with this apparatus operating 6 hours a day, a cell life of 3 years, and a service life of the rest of the apparatus of 10 years, give the following:

<table>
<thead>
<tr>
<th>Item</th>
<th>Cost per liter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Distilled H$_2$O at 8 cents a gallon</td>
<td>$0.0211</td>
</tr>
<tr>
<td>Sodium chloride 25 grams per liter at 4 cents a pound</td>
<td>0.0022</td>
</tr>
<tr>
<td>Sodium bicarbonate 1.5 grams per liter at 30 cents a pound</td>
<td>0.0010</td>
</tr>
<tr>
<td>Current 120 watt hours per liter at 7.5 cents a kilowatt hour</td>
<td>0.0090</td>
</tr>
<tr>
<td>Cell cost - $16.00 for 3 years</td>
<td>0.0038</td>
</tr>
<tr>
<td>Depreciation on original cost - $150-$16.00 for 10 years</td>
<td>0.0102</td>
</tr>
</tbody>
</table>

As with the batch cell the cost per liter would be decreased if zeolite softened water were used; however, it would also be raised if the
sodium chloride and sodium bicarbonate were purchased in specially prepared packets. In any case the cost of the antiseptic made by the electrolytic cell is much less than any other antiseptic.

C. Analytical Procedures

1. Determination of hypochlorite with sodium thiosulfate

The determination of hypochlorites using sodium thiosulfate depends upon the liberation of iodine from an acidic solution of potassium iodide and the subsequent titration of the free iodine with sodium thiosulfate using a starch indicator. The reactions involved in this analysis are as follows:

1. \( \text{NaOCl} + \text{HCl}_2\text{H}_3\text{O}_2 + 2 \text{KI} + \text{H}_2\text{O} \rightarrow \text{NaC}_2\text{H}_3\text{O}_2 + \text{HCl} + 2 \text{KOH} + \text{I}_2 \)

2. \( \text{I}_2 + 2 \text{Na}_2\text{S}_2\text{O}_3 \rightarrow 2 \text{NaI} + \text{Na}_2\text{S}_4\text{O}_6 \)

From these reactions it is seen that the following relationship is present:

\[
\frac{\text{NaOCl}}{2} \leftrightarrow \text{Na}_2\text{S}_2\text{O}_3 \leftrightarrow \text{I}
\]

As a result the formula for calculating the concentration of sodium hypochlorite in terms of grams per 100 c.c. is as follows:

\[
\frac{\text{(c.c. of Na}_2\text{S}_2\text{O}_3)(\text{N. of Na}_2\text{S}_2\text{O}_3)\left(\frac{\text{NaOCl}}{2}\right)(100)}{\text{(c.c. of sample)(1000)}} = \text{grams NaOCl/100 c.c.}
\]

This reduces down to:

\[
\frac{\text{(c.c. of Na}_2\text{S}_2\text{O}_3)(\text{N. of Na}_2\text{S}_2\text{O}_3)(3.7227)}{\text{(c.c. of sample)}} = \text{grams NaOCl/100 c.c.}
\]
In terms of grams per 100 cc. as follows:

The formula for calculating the concentration of sodium hypochlorite

\[ \text{I} \quad \frac{2}{\text{NaOCl}} \quad \frac{2}{\text{NaClO}} \]

It is seen that the following relationship holds true:

\[ \text{NaClO} + \text{NaOH} \rightarrow \text{NaOCl} + \text{H}_2 \text{O} \]

The titration, the reaction involved in this example, is as follows:

\[ \text{NaClO} + \text{H}_2 \text{O} \rightarrow \text{NaOCl} + \text{H}_2 \text{O} \]

In the solution, no effect on the arsenite and are not introduced in sodium arsenite to sodium arsenite by the hypochlorite. Chlorates present.

This determination of hypochlorites depends upon the oxidation of

* Determination of hypochlorites by the pent method (25).

2. Determination of hypochlorites with the hypochlorites.

In this method, the chlorates present in the solution are included.

Then calculated by means of the formula given above.

From the burette reading. The concentration of sodium thiosulfate was calculated from the burette reading. The concentration of sodium thiosulfate was calculated from the burette reading. The concentration of sodium thiosulfate was calculated from the burette reading. The concentration of sodium thiosulfate was calculated from the burette reading.

A drop of starch indicator was added and the titration was continued to a few mL of starch indicator was added with distinct yellow color remained. A few mL of starch indicator was added to about \(\pm 0.0\) cc. with distinct yellow color remained. A few mL of starch indicator was added to about \(\pm 0.0\) cc. with distinct yellow color remained. A few mL of starch indicator was added to about \(\pm 0.0\) cc. with distinct yellow color remained. A few mL of starch indicator was added to about \(\pm 0.0\) cc. with distinct yellow color remained.

Finally, a test to which \(\pm 0.0\) cc. of 10 percent NaCl solution was

The procedure followed for this determination was as follows:

-5-
(c.c. of Na₂H₂AsO₃)(N. of Na₂H₂AsO₃) \(\frac{1}{2}\)(100) \(\frac{\text{NaOCl}}{\text{(c.c. of sample)}(1000)}\) = grams NaOCl/100 c.c.

This reduces to:

\(\frac{(c.c. \text{ of } \text{Na}_2\text{H}_2\text{AsO}_3)(N. \text{ of } \text{Na}_2\text{H}_2\text{AsO}_3)(3.7227)}{(c.c. \text{ of sample})} = \text{grams NaOCl/100 c.c.}\)

The procedure followed for the determination was as follows:

Five c.c. of the hypochlorite solution were pipetted into a 50 c.c. beaker. The volume was increased to about 25 c.c. with distilled water. N/10 alkaline sodium arsenite solution was titrated into the beaker with constant stirring until a drop of the solution when touched to starch iodide paper failed to produce a blue spot. The concentration of hypochlorite was then calculated by means of the formula given above.

3. Determination of chlorates

Since the chlorates are included in the thiosulfate method and not in the Fenton method, they were determined by difference. The reaction of chlorates on iodine and the subsequent titration of the iodine set free with thiosulfate take place according to the following reactions:

1. \(\text{NaClO}_3 + \text{H}_2\text{C}_2\text{O}_2 + 6\text{KI} + 3\text{H}_2\text{O} \rightarrow \text{NaC}_2\text{H}_3\text{O}_2 + \text{HCl} + 6\text{KOH} + 3\text{I}_2\)

2. \(3\text{I}_2 + 6\text{Na}_2\text{S}_2\text{O}_3 \rightarrow 3\text{Na}_2\text{S}_4\text{O}_6 + 6\text{NaI}\)

From these reactions the following relation is seen to hold:

\(\frac{\text{NaClO}_3}{6} \rightarrow \frac{\text{Na}_2\text{S}_2\text{O}_3}{\text{I}}\)
The formula for calculating the concentration of sodium chlorate by difference in terms of grams per 100 c.c. is as follows:

\[
\frac{(\text{c.c. of Na}_2\text{S}_2\text{O}_3)(N. \text{ of Na}_2\text{S}_2\text{O}_3) - (\text{c.c. of Na}_2\text{HAsO}_3)(N. \text{ of Na}_2\text{HAsO}_3)}{6} \times \frac{\text{NaClO}_3}{(\text{c.c. of sample})(1000)}
\]

= grams NaClO₃/100 c.c.

This reduces to:

\[
\frac{(\text{c.c. of Na}_2\text{S}_2\text{O}_3)(N. \text{ of Na}_2\text{S}_2\text{O}_3) - (\text{c.c. of Na}_2\text{HAsO}_3)(N. \text{ of Na}_2\text{HAsO}_3)}{1.7742} \times \frac{\text{NaClO}_3}{(\text{c.c. of sample})}
\]

= grams NaClO₃/100 c.c.

All chlorate concentrations were calculated by the difference of the two preceding methods for hypochochlorite using the above formula.

All solutions used in this analytical work were prepared and standardized according to the instructions given in the 25th edition of the Handbook of Chemistry and Physics (10).

4. Determination of pH

Determinations of pH were made with the Model 15 Coleman pH meter. The meter was adjusted for correct reading with a buffer solution of pH 4.1 which was supplied with the instrument. After checking for proper adjustment, the electrodes were washed off with distilled water and the sample contained in a 50 c.c. beaker was put into place beneath the electrodes. This sample was then discarded and a fresh sample put into place and the reading of the pH value was made. This machine is reputed to be accurate to 0.1 pH units.
RESULTS

The results of this research may be listed as follows:

1. A batch type electrolytic cell has been developed which will produce a saline hypochlorite solution suitable for use as an antiseptic.

2. This cell used a Battery Booster type dry plate rectifier as its source of direct current.

3. This cell is simple in operation and construction and can be operated by a person unskilled in chemical technique.

4. This cell produces a solution with a concentration of 0.475 grams NaOCl per 100 c.c. and a pH of 9.3 at the rate of about 10 c.c. per minute.

5. It has been found advantageous to lower the temperature of the salt solution prior to electrolysis in the batch cell in order to keep the temperature of the hypochlorite solution down and to decrease the amount of electrode decomposition.

6. Lowering the initial temperature of the salt solution increases the time of electrolysis in the batch cell.

7. This cell may be operated with a battery as its source of direct current, however, the time of electrolysis is increased over that when the rectifier is used.

8. In the batch cell the temperature of the hypochlorite solution when the battery is used is lower than when the rectifier is used so the temperature of the salt solution need not be lowered prior to electrolysis.
9. The relationship between the increase in sodium chloride concentration in the salt solution and the corresponding increase in hypochlorite concentration has been determined for the batch cell.

10. Addition of sodium bicarbonate to the salt solution decreases the concentration of the resulting hypochlorite and seems to have very little effect on the pH of the solution in the batch cell.

11. A commercial model of the batch type cell has been designed which can be sold for about $100.00.

12. The commercial model of this cell can produce a hypochlorite solution of the proper concentration for antiseptic use for less than 5 cents a liter.

13. The addition of 0.025 per cent of potassium chromate to the electrolyte increases the concentration of NaOCl from 0.449 to 0.502 grams per 100 c.c. in the batch cell.

14. A continuous type cell has been designed for the production of a saline hypochlorite solution suitable for antiseptic purposes in which the salt solution is fed directly from the reservoir into the cell body.

15. An automatic device has been designed which makes this type cell foolproof and permits its operation by an inexperienced person.

16. This cell will produce a solution with a concentration of 0.482 grams NaOCl per 100 c.c. and a pH of 8.8 at the rate of 10.6 c.c. per minute.

17. Two rectifiers were used with this cell and it was found that the rectifier sold by the Montgomery Ward and Company produced a solution lower in concentration than the rectifier sold by Sears Roebuck and Company.
18. The former rectifier caused greater heating in this cell than the latter.

19. In the continuous cell the concentration of the hypochlorite solution depends on the rate of current flow and the rate of solution flow.

20. A commercial model of the continuous cell can be sold for about $150.00.

21. This cell can produce a hypochlorite solution suitable for antiseptic purposes for less than 5 cents a liter.

22. Hypochlorite solution with a concentration of 0.45 to 0.50 grams NaOCl per 100 c.c. can be obtained in less than 5 minutes after the continuous cell has been turned on.
LITERATURE CITED


   An account is given of the work done by Dr. Carrel with Dakin's solution in the treatment of war wounds.


   A summary is given of the work performed on the antiseptic cell from 1933 until 1941.


   A letter concerning the alkalinity of the hypochlorite solution produced by the Milton Proprietary Ltd. and several quotations from 2nd edition of Carrel and Dehelly's "Treatment of Infected Wounds."


   Description of an electrolytic cell for preparing antiseptic sodium hypochlorite solution and notes on its operation.


   A discussion of the position held by antiseptic substances up until 1915.


   A short discussion of the early history of the use of hypochlorites as antiseptic agents.


   The results of the experiments performed on antiseptic substances by Dr. Dakin.

A comprehensive history of hypochlorites and their use as antiseptics.


Details concerning the preparation of Dakin's solution and of Daufresne's modification of this solution.


Instructions for the preparation and standardisation of the solutions used in the analytical work.


An account of the work done by Hanna in the development of an electrolytic cell using platinum electrodes with a solenoid operated valve.


A statement concerning the use of electrolyzed sea water as a disinfectant on hospital ships.


An account of the development of an electrolytic hypochlorite cell in which the electrolyte was fed into the cell through the side and the hypochlorite solution drained out through an orifice in the bottom of the cell.


A summary of the pathological research on the toxicity of the rhodium contained in the electrolytic hypochlorite solution produced by the antiseptic cell.


An account of the research on suitable electrodes for use in an electrolytic hypochlorite cell.

A discussion of the two research groups headed by Professor J. Lorrain Smith and Professor Cohen and of the results of their work.


An account of the development of an automatic control device to be used on an electrolytic hypochlorite cell.


Notes on the early development of hypochlorite as an antiseptic and a hypothesis on the mechanism of the antiseptic action of hypochlorite.


An account of the research performed by this group and of the development and use of Eupad and Eusol.


An account of the use of a dry plate rectifier with a hypochlorite antiseptic cell.


A summary of the work performed on the hypochlorite cell at Iowa State College and of the development of a commercial model.


Details for the analysis of hypochlorites by the Penot method.


A discussion of the first work done at Iowa State College on the electrolytic cell, a description of the cell used, and an account of the results obtained.

A summary of the work done up until 1929 on the electrolytic cell at Iowa State College.

25. Wallace and Tiernan Co., Inc. Instructions for Wallace-Tiernan Carrel-Dakin Apparatus.

Instructions for the use of the Wallace-Tiernan apparatus for making Carrel-Dakin solution by the chlorination of sodium carbonate.


An address on the treatment of war wounds and of Sir Almroth Wright's views on the efficiency and use of antiseptic substances on them.
the College Instrument Shop, and by Mr. Paul Freeman,

Anghist of the Electrical Engineering Department, Mr. J. A. Colburn of

Electrical Engineering Station, Professor J. D. Ryder and Professor R. W.

directed acknowledgment is also given to Dr. L. R. Arnold of the

need of the Department of Chemical Engineering.

This work was carried out under the direction of Dr. O. R. Sweaney.

Acknowledgments
### APPENDIX

Tests Performed on the Continuous Cell Using the Old Rectifier, No. 8-7846

<table>
<thead>
<tr>
<th>Time, minutes</th>
<th>Cell temperature, °C</th>
<th>Amperes</th>
<th>Volts</th>
<th>Rate, c.c. per minute</th>
<th>pH</th>
<th>NaOCl grams per 100 c.c.</th>
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</tr>
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Tests Performed on the Continuous Cell Using the New Rectifier, No. T-1619

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<th>NaOCl grams per 100 c.c.</th>
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<td>4.4</td>
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</table>

| **Test No. 8** | **Room temperature** |        |       |                      |    |                        |
| 5             | 47.0                 | 3.15   | 4.9   | 7.7                  | ---| 0.369                  |
| 10            | 49.2                 | 3.25   | 4.9   | 7.8                  | 8.6| 0.399                  |
| 15            | 49.8                 | 3.20   | 4.9   | 7.8                  | ---| 0.398                  |
| 20            | 50.0                 | 3.20   | 4.9   | 7.8                  | 8.6| 0.401                  |
| 25            | 50.5                 | 3.20   | 4.9   | 7.8                  | ---| 0.403                  |
| 30            | 50.5                 | 3.20   | 4.9   | 7.8                  | 8.6| 0.403                  |
| 35            | 51.5                 | 3.30   | 4.9   | 7.8                  | 8.6| 0.403                  |
| Averages*     | 50.2                 | 3.22   | 4.9   | 7.7                  | ---| 0.401                  |

<p>| <strong>Test No. 9</strong> | <strong>Room temperature 27.5° C.</strong> |        |       |                      |    |                        |
| 5             | 58.1                 | 4.3    | 5.5   | 7.7                  | ---| 0.461                  |
| 10            | 62.5                 | 4.5    | 5.5   | 7.7                  | ---| 0.476                  |
| 15            | 62.8                 | 4.5    | 5.4   | 7.7                  | ---| 0.474                  |
| 20            | 62.8                 | 4.4    | 5.4   | 7.6                  | ---| 0.480                  |
| 25            | 63.0                 | 4.4    | 5.4   | 7.7                  | ---| 0.482                  |
| 30            | 63.8                 | 4.5    | 5.4   | 7.7                  | ---| 0.484                  |
| 35            | 64.0                 | 4.4    | 5.4   | 7.7                  | ---| 0.481                  |
| Averages*     | 63.4                 | 4.48   | 5.4   | 7.7                  | ---| 0.479                  |</p>
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<th>Cell temperature, °C</th>
<th>Amperes</th>
<th>Volts</th>
<th>Rate, e.c. per minute</th>
<th>pH</th>
<th>NaOCl grams per 100 c.c.</th>
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Test Performed on Continuous Cell Using New Rectifier, No. T-1819 to Produce Solution with Hypochlorite Concentration 0.45 to 0.50 Grams per 100 c.c.

<table>
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<th>Time, minutes</th>
<th>Cell temperature, °C.</th>
<th>Amperes</th>
<th>Volts</th>
<th>Rate, c.c. per minute</th>
<th>pH</th>
<th>NaOCl grams per 100 c.c.</th>
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*Since all tests were started with the cell initially filled with salt solution the values obtained after 5 minutes of operation were lower than would be obtained in normal operation when the electrolysis would be started with the cell filled with hypochlorite solution from the previous run. As a result in taking the averages for these data, the values for the first five minutes were omitted.