A STUDY OF DEACON'S PROCESS

AND

AN INVESTIGATION OF THE INHERENT POSSIBILITIES
IN THE OXIDATION OF HYDROCHLORIC ACID BY
ATMOSPHERIC OXYGEN IN THE PRESENCE
OF CERTAIN CATALYSTS AND
TEMPERATURES

By

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Submitted in partial fulfillment of the
requirements for the degree of Master of
Science in the school of Arts and Sciences
in the University of Alabama.

University, Alabama
1931
ACKNOWLEDGMENT

The writer wishes to take this opportunity of acknowledging his indebtedness to Dr. Jack P. Montgomery for his aid and direction in the problem herein considered and to Dr. George D. Palmer appreciation is expressed for his suggestion and help.

Wherever a publication or book has been used as a reference it has been acknowledged by a footnote and in the appended bibliography.
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INTRODUCTION

Chlorine is finding an increasing number of uses annually, and production of the element is steadily increasing.

It is of significance to note that, even in these times of economic stress when nearly all industries have reduced production to a minimum, the output of chlorine is greater than ever before in its history.¹

Consequently, the economical production of chlorine becomes a problem worthy of serious consideration.

At present the annual production of the element in America is more than thirty thousand tons, two-thirds of which is used in the bleaching of paper pulp, ten per cent in water purification, and about seven per cent in laundry bleaching. Until recent years it was used in combination with lime as bleaching powder. However, there is now practically no demand for this product, and the element is being liquefied and handled in steel cylinders.²

¹ Birmingham News, July 2, 1931, "Market Letter".
² Holmes, H. N., General Chemistry, pp. 166-167.
Chlorine is also used in the chlorination of organic compounds. It is interesting to note that in such a reaction only one atom in each molecule of chlorine combines with the compound being treated, the other atom uniting with hydrogen to form hydrochloric acid.\(^1\) It is possible that a way to utilize the acid thus formed may be found in the results of this study.

In the past many chemists have been attracted by the inherent possibilities in the oxidation of hydrochloric acid by atmospheric oxygen as a means of evolving chlorine cheaply. It was along this line of thought that such men as Oxland, Vogel, Thibierge, and Binks proceeded.\(^2\) It remained for Henry Deacon, however, to establish a process that could be classified as a technically satisfactory industry. He presented his process to the chemical industry in 1868\(^3\).

The general idea seems to be that there is no better catalyst for the process than the one used by Deacon. As an indication of this thinking the following statement is taken in whole from Thorpe's Dictionary of Applied Chemistry,\(^4\)

\(^1\) Holmes, H. N., General Chemistry, pp. 166-167.
\(^4\) Ibid.
"From a diagram by Hurter, showing the affinity of technically available elements for oxygen, chlorine, and hydrogen, it appears that no other metal than copper forms two oxides and two chlorides in which the combination is of so loose a character.

"It is therefore almost a certainty that a cheaper and more efficient catalyst cannot be found, and that the Deacon process is the best for the direct conversion of gaseous hydrogen chloride into free chlorine."

However, it is the purpose of this study to follow closely the principles of Deacon's process but using different catalysts and different temperatures, if needed, in the hope of finding a practical substitute for the salts of copper.
CHAPTER I

A REVIEW OF DEACON'S PROCESS

Since, in many instances, the steps involved in this investigation are similar to those employed by Henry Deacon space is allowed for a review of the process that bears his name.

Ludwig Mond says of Deacon, "because of his process, its simplicity and beauty, the name of Henry Deacon will ever be famous in the annals of industrial chemistry". There is little doubt but that Deacon was successful in offering an entirely practical method for obtaining chlorine that would serve in the manufacture of bleaching powder, and at the same time was doubtless the most economical process known.

To put it briefly, the process was founded on the fact that a mixture of hydrogen chloride and oxygen in contact with certain catalysts, and at a temperature just below red heat would react to form chlorine and water.

Deacon found the salts of copper to be the most effective catalysts in his work. In such a reaction cupric chloride is used. The cupric

2. Ibid.
chloride breaks up into cuprous chloride and chlorine. The cuprous chloride combines with oxygen to form oxychloride, and this again acts upon the hydrogen chloride, yielding water and reforming cupric chloride. The reaction can then start again.¹

However, there seems to be a bit of confusion as to what the theoretical yield of chlorine should be. According to the explanation given above, all of the hydrogen chloride would be converted into chlorine and the proportionate amount of water, but in practice, as Thorpe says, "under the most favorable conditions only a little more than half of the hydrogen chloride is decomposed."²

A seemingly conflicting statement is made by Kendall.³ He says,

"When the mixture of gases is passed over the heated mass, steam, chlorine, and about twenty per cent of unchanged oxygen and hydrogen chloride issue at the other end. Longer heating does not alter the proportion of the materials transformed.

"That eighty per cent is changed and twenty per cent unchanged is due to the fact that the reaction is reversible. No more than twenty per cent of the hydrogen chloride remains unchanged because it seems that an equilibrium exists such that the ratio remains the same."

² Ibid.
From the last three paragraphs it will be seen that certain chemists are not in accord as to just what yield is possible by Deacon's process. Thorpe, as stated above, suggests that theoretically one hundred per cent of the available chlorine would be liberated. Kendall, on the other hand, in his Inorganic Chemistry, makes the statement that it is impossible to obtain more than eighty per cent of the gas from a given quantity of hydrogen chloride.

Perhaps some chemist has already made an investigation of which we are not aware, but if not it would seem that there is much to be learned concerning the exact reaction conditions involved in Deacon's process. Until then we are forced to accept the yield found in actual practice as being indicative of the ultimate possibilities so long as the same catalysts and temperatures are employed.

Deacon found that pure oxygen gave a better yield of chlorine than when used in atmospheric mixture, but the additional efficiency was not great enough to justify the added expense. For this reason his process was developed in such a manner as to use only atmospheric oxygen.

Deacon found by experiment that the yield of chlorine was best when four parts of air by volume combined with one of hydrogen chloride. An excess
of air was found to be less injurious than a lack of it, in which case decomposition fell off rapidly.

Experiment also proved that the process worked more efficiently when the gaseous hydrogen chloride was dried before entering the decomposing chambers. Sulphuric acid in concentrated form was used for this purpose.

After the gas was dried it was heated to about five hundred degrees before passing into the decomposer. The heating apparatus was a furnace sixteen feet by sixteen feet, in which were twenty-six vertical pipes, twelve inches wide and nine feet high, "connected like the 'breeches pipes' in the blast-heating stoves, formerly used in iron works".¹

The decomposer had no heat of its own, but was supplied by the waste heat from the furnace. It was an upright cast-iron cylinder, twelve to fifteen feet wide. It contained a cylindrical ring of broken bricks, supported by two cylindrical cast-iron rings of shutters placed concentrically one within the other. The gases entered from the outside at the circumference, and passed across the decomposing mass into the inner space, and were led away through a pipe. The annular space between the shutters was

three feet wide, and was divided into six compart-
ments, one of which was emptied every ten days. This
was accomplished by means of discharging doors below
the cylinders. Fresh decomposing material was then
thrown in from above. This material consisted of
broken bricks dipped in a solution of cupric chloride.
This mass lost its decomposing properties after about
ten weeks and had to be replaced.

The temperature in the decomposer was held
within the range between four hundred eighty and five
hundred degrees.

After the gases had left the decomposer they
consisted of a mixture of hydrogen chloride, free
chlorine and steam. In the average case about one-
half of the hydrogen chloride was oxidized, sometimes
the yield would be a little better.

The hot exit gases were cooled by passing through
a long string of earthenware pipes and freed from
hydrogen chloride by washing with water in ordinary
acid condensers, consisting of stone towers filled
with coke, combined or not combined with acid cisterns,
Woulff's jars, etc.
Other Contact Substances that have been Used in the Process by Deacon.

Lamy, in 1873, made an investigation of the mutual action of hydrogen chloride and oxygen in the presence of certain metallic compounds. They were copper, iron, manganese and chromium. He found the copper salts, as used by Deacon, to be more effective than the others.¹

In 1855, Thibierge, proposed the use of ferric chloride, or chromic oxide. These have not had any practical success.

Nickel oxide was proposed by Ludwig Mond, 1886. He passed hydrogen chloride over heated nickel oxide. The product was then exposed to dry and heated air, and nickel oxide was again formed and chlorine given off.

Bone-black has been used as a possible substitute, only in an experimental capacity, for the copper salts now employed.¹

C. T. Kingzett worked in this capacity with magnesium chloride, and obtained very satisfactory results.²

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CHAPTER II
DESCRIPTION OF EXPERIMENTAL PROCEDURE

The first step was to design laboratory apparatus that could be used to oxidize hydrogen chloride according to Deacon's process. Briefly, it consisted of a hydrogen chloride generator, a quartz combustion tube, and an electric furnace controlled by a calibrated rheostat. An arrangement was included whereby the exit gases from the combustion tube could be collected for analysis.

The writer, in order to familiarize himself with the workings of the process, carried out several experiments, following closely the methods of Deacon, using the same catalysts and temperatures.

After these preliminaries the work proceeded with the idea in mind of using a number of catalytic substances over a wide range of temperatures in the hope of finding one that could be successfully substituted for the copper salts formerly employed.

In the beginning a small combustion boat was filled with the catalyst to be used and then placed in the quartz tube.氢化氢chloride, mixed with atmospheric oxygen, was passed over the mass and the gases issuing at the other end passed directly into a solution of potassium iodide containing
starch. Any chlorine liberated would replace the iodine, forming potassium chloride and setting the iodine free. The reaction between the iodine and starch, indicated by the resulting purple color, would indirectly show the presence of chlorine.

The starch test was necessary only insofar as it furnished a more sensitive indicating reaction than if the color of the free iodine itself had been depended upon. Of course, the starch was needed in solution only when it was desirable to know positively whether or not a certain substance did or did not serve in any degree as a catalytic agent.

Cupric chloride was used first. A small combustion boat was impregnated with it and heated to a temperature of about four hundred fifty degrees. Immediately after the mixture of air and hydrogen chloride came in contact with the cupric chloride the starch-potassium iodide solution showed a dark purple color, marking the presence of chlorine.

The same results were obtained with other metallic compounds. Among them were, salts of iron, manganese, and magnesium. Oxides of nickel and chromium were employed with positive results. Bone-black was found to be effective, also.
To increase the yield of chlorine, two combustion boats, instead of one, filled with the catalytic substance being used was placed in the quartz tube, and an immediate deepening of the iodine color (the use of starch having been discontinued at this point) indicated an increased production of chlorine.

From this point on the work was carried on quantitatively. The technique employed will not be entered into here, but is placed in the chapter presenting ANALYTICAL PROCEDURE AND QUANTITATIVE RESULTS.

After increasing the amount of the catalytic material being used, and obtaining increased yields of chlorine, a study of the other variables entering into the process was made.

Of course, it will be recognized that the chlorine yield must have been very small, perhaps not more than five per cent when the first little combustion boat with its charge of cupric chloride was placed in the quartz tube and heated to the known optimum temperature. And, since records show that as much as half of the available chlorine might be obtained under favorable conditions by Deacon's process, it was evident that several factors would have to be
altered and controlled.

It was thought that if a thorough study were made in the beginning, time would be saved, because it would stand to reason that if a new catalytic substance should be encountered there would be certain things affecting it in common with those already tried.

Experiment has shown that the presence of moisture has an adverse effect on the yield of chlorine, bringing about a reduction of from three to six per cent, depending on the amount of water vapor permitted to enter the apparatus. For this reason the water factor was controlled by inserting sulphuric acid drying bottles into the line of apparatus to thoroughly dry the gases before they passed over the catalytic mass.

A method whereby the amount of hydrogen chloride mixing with the atmospheric oxygen could be approximated had to be brought into play. This was accomplished by means of a large bottle of air fitted with a separatory funnel in such a manner that water could be used to force the air from the bottle into a flask to be mixed with the hydrogen chloride before entering the furnace. A detailed discussion of the apparatus used will be found in the chapter given to its description.
Tables found in another part of this paper will show a direct relationship in Deacon's process between the amount of air used and the yield of chlorine. An excess of air was found to give better results than an amount that would theoretically be enough to carry the chemical reaction to an end.

After using combustion boats to carry the catalyst it was suggested that perhaps the amount of catalytic surface exposed had a direct bearing on the yield of chlorine. This was soon found to be true, and methods for increasing the exposed surface to a maximum were sought.

Impregnating asbestos with the catalyst was hit upon. The tube would then be filled with the mass, and it was in this manner that a yield of chlorine was effected that most nearly approached in per cent the commercial results, using, of course, cupric chloride.

Then asbestos alone was tried. At the usual temperatures there was no reaction, but when raised to bright red heat it was found that the oxidation of hydrochloric acid was effected. The per cent of decomposition would not go higher than seventeen. However, this was to be considered significant. And from then on the work dealt primarily with the
increasing of the yield of chlorine, using asbestos as the catalyst.

In the meantime silica gel was tried in the quartz tube and at a temperature of about eight hundred fifty degrees it served as a catalyst, though rather inefficiently. It is impossible to say just what per cent yield would be possible from silica gel since it was impossible to get good results from it in this work, due to its fine texture. When the tube was filled with silica gel the gases found passage impossible without pressure, and if less were used the gases could pass directly over the mass without making contact. The yield obtained under such handicaps was never more than seven per cent.

The idea then was suggested that it might be productive of good results if silica gel were mixed with the asbestos. Such proved to be true and analysis showed an increased yield, about forty per cent under the best conditions.

Other silicon bearing compounds were tried, and sand is perhaps the only one worthy of note. Sand with asbestos was found to be even more efficient than asbestos with silica gel. It was with sand that the yield was increased to nearly sixty-two per cent. Some calculations gave a higher percentage yield, but
not enough of them to be reliable.

The type and size sand seems to have a marked effect. Calculations were made that determined the effect of the size of the sand particles. It was found that if the sand were smaller than that which would stop on a number thirty-five mesh that its efficiency decreased from the optimum. And the same was true of sand that would stop on size twenty mesh. So it would seem that the surface factor is an important one.

The yield of chlorine was best, too, when the sand and asbestos were mixed in the ratio of fifteen to one by weight respectively.

It was an interesting fact to note that only Italian asbestos gives very satisfactory results. The asbestos used in this work was obtained from The J. T. Baker Chemical Company, Phillipsburg, New Jersey. Its chief characteristic is its short fibre, in direct contrast to the long fibered variety found in Canada and Russia.
CHAPTER THREE

DESCRIPTION OF APPARATUS USED IN THE EXPERIMENTAL PROCEDURE

On the opposite page will be found a photograph of the apparatus used in this study. The hydrogen chloride generator (1) is connected with the flask (2), as is the large bottle fitted with the separatory funnel (3), in such a manner that air from the large bottle and hydrogen chloride from the generator may be brought together for mixing before entering the drying bottle (4). The drying bottle contains sulphuric acid and insures the thorough dehydration of the gases before entering the quartz tube (5). The tube is supplied with heat from the electric furnace (6), which is controlled by the calibrated rheostat (7). On leaving the furnace the gases enter a solution of potassium iodide in bottles (8) and (9).
As has been stated before in this paper, the chlorine liberated in the process employed was caused to pass into a solution of potassium iodide. The chlorine combined with the potassium forming potassium chloride and setting iodine free;

\[ \text{Cl}_2 + 2\text{KI} \rightarrow 2\text{KCl} + \text{I}_2 \]

In order to calculate the quantity of chlorine produced in any given experiment it was only necessary to determine the amount of liberated iodine and then by employing the principles of equivalent weights it was possible to arrive at the quantity of chlorine.

In determining the amount of free iodine a standardized solution of sodium thiosulphate was used in titration;

\[ \text{Na}_2\text{S}_2\text{O}_3 + \text{I}_2 \rightarrow \text{Na}_2\text{S}_4\text{O}_6 + 2\text{NaI} \]

The sodium thiosulphate solution was standardized against a known solution of iodine.

The hydrogen chloride used in this work was prepared by dropping hydrochloric acid on concentrated sulphuric acid. The apparatus consisted of a separatory funnel connected with a large flask as shown in the photograph.
The amount of hydrogen chloride liberated was determined by adding silver nitrate in excess to an aliquot portion of the residue in the generator flask and then weighing the precipitated silver chloride. The chemical factor of chlorine in silver chloride, .2474, was multiplied by the weight of silver chloride. This gave in grams the chlorine remaining in the generator. Subtracting this figure from the original amount of chlorine in the acid used indicated the amount passing through the furnace.

After employing the procedure for some time it was observed that when the physical factors were controlled carefully there was a very small difference between the amounts of silver chloride precipitated in each case. A mean of ten determinations was taken as representing the hydrochloric acid used in each experiment and was treated as a constant in the analytical procedure.

The quantitative results presented in the form of tabulations represent, in the main, the experimentation after the optimum temperature had been discovered. This was found to be about 850° C, bright red heat. It might be stated here that with the catalysts employed, in no instance were significant yields of chlorine obtained below 800° C.
Also, numerous experiments were made at higher temperatures and in every case it was seen that there was no advantage in working with temperatures above 850° C. In these experiments the same combination of catalysts was used as shown in the tables given in the following pages.

The following tables represent the results of calculations based on the analytical procedure explained in detail in the first part of this chapter. In all cases, the amount of chlorine in hydrogen-chloride passing over the catalytic mass is taken as a constant, two and fifty-one hundredths grams. The yield of freed chlorine is divided by this constant to obtain the per cent yield.

Table One

Catalyst - asbestos

Temperature of furnace 850° C.

<table>
<thead>
<tr>
<th>Chlorine in gaseous hydrogen chloride passing over catalytic material</th>
<th>Free chlorine obtained in grams</th>
<th>Per cent yield of chlorine</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 2.51 grams</td>
<td>.4262</td>
<td>16.98</td>
</tr>
<tr>
<td>2. 2.51 grams</td>
<td>.3871</td>
<td>15.42</td>
</tr>
<tr>
<td>3. 2.51 grams</td>
<td>.4023</td>
<td>16.02</td>
</tr>
<tr>
<td>4. 2.51 grams</td>
<td>.3045</td>
<td>12.13</td>
</tr>
<tr>
<td>5. 2.51 grams</td>
<td>.3672</td>
<td>14.62</td>
</tr>
</tbody>
</table>
Table Two

Catalyst - sand that will stop on number 35 screen mesh.

Temperature of furnace 850⁰ C.

<table>
<thead>
<tr>
<th>Chlorine passing over catalytic mass</th>
<th>Yield of chlorine in grams</th>
<th>Per cent yield of chlorine</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.51 grams</td>
<td>1. 0.2915</td>
<td>11.61</td>
</tr>
<tr>
<td></td>
<td>2. 0.3206</td>
<td>12.77</td>
</tr>
<tr>
<td></td>
<td>3. 0.2610</td>
<td>10.39</td>
</tr>
<tr>
<td></td>
<td>4. 0.3016</td>
<td>12.01</td>
</tr>
<tr>
<td></td>
<td>5. 0.2780</td>
<td>11.08</td>
</tr>
</tbody>
</table>

Table Three

Catalyst - asbestos and silica gel in the proportion of 12 - 1 by weight.

<table>
<thead>
<tr>
<th>Chlorine passing over catalytic mass</th>
<th>Yield of chlorine in grams</th>
<th>Per cent yield of chlorine</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.51 grams</td>
<td>1. 1.0121</td>
<td>40.32</td>
</tr>
<tr>
<td></td>
<td>2. 1.0926</td>
<td>43.52</td>
</tr>
<tr>
<td></td>
<td>3. 0.9877</td>
<td>39.35</td>
</tr>
<tr>
<td></td>
<td>4. 0.9368</td>
<td>37.32</td>
</tr>
<tr>
<td></td>
<td>5. 1.0062</td>
<td>40.08</td>
</tr>
<tr>
<td></td>
<td>6. 0.9012</td>
<td>35.90</td>
</tr>
<tr>
<td></td>
<td>7. 0.9678</td>
<td>38.55</td>
</tr>
</tbody>
</table>
Table Four

Catalyst - sand and Italian asbestos in the proportion of 15 - 1 by weight. The size sand was that which would stop on size 35 screen mesh.

Temperature of furnace 850°C.

<table>
<thead>
<tr>
<th>Amount of chlorine passing over catalytic mass</th>
<th>Yield of chlorine in grams</th>
<th>Per cent yield of chlorine</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.51 grams</td>
<td>1.4909</td>
<td>59.39</td>
</tr>
<tr>
<td>2.</td>
<td>1.4216</td>
<td>56.63</td>
</tr>
<tr>
<td>3.</td>
<td>1.3804</td>
<td>54.95</td>
</tr>
<tr>
<td>4.</td>
<td>1.5090</td>
<td>60.11</td>
</tr>
<tr>
<td>5.</td>
<td>1.4116</td>
<td>56.23</td>
</tr>
<tr>
<td>6.</td>
<td>1.5391</td>
<td>61.31</td>
</tr>
<tr>
<td>7.</td>
<td>1.3985</td>
<td>55.71</td>
</tr>
<tr>
<td>8.</td>
<td>1.4981</td>
<td>59.68</td>
</tr>
<tr>
<td>9.</td>
<td>1.4783</td>
<td>58.88</td>
</tr>
<tr>
<td>10.</td>
<td>1.5002</td>
<td>59.76</td>
</tr>
</tbody>
</table>
Table Five

Catalyst - same as in table four.

Temperature of furnace 850° C.

In this case water vapor was allowed to enter the furnace, and it is the purpose of this table to show the effect on the yield of chlorine.

<table>
<thead>
<tr>
<th>Chlorine passing over catalytic mass.</th>
<th>Yield of chlorine in grams</th>
<th>Per cent yield of chlorine</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.51 grams</td>
<td>1.  1.3460</td>
<td>53.62</td>
</tr>
<tr>
<td></td>
<td>2.  1.3391</td>
<td>53.35</td>
</tr>
<tr>
<td></td>
<td>3.  1.4097</td>
<td>56.16</td>
</tr>
<tr>
<td></td>
<td>4.  1.3105</td>
<td>52.21</td>
</tr>
<tr>
<td></td>
<td>5.  1.3012</td>
<td>51.80</td>
</tr>
<tr>
<td></td>
<td>6.  1.2916</td>
<td>51.45</td>
</tr>
<tr>
<td></td>
<td>7.  1.3581</td>
<td>54.10</td>
</tr>
<tr>
<td></td>
<td>8.  1.2816</td>
<td>51.05</td>
</tr>
<tr>
<td></td>
<td>9.  1.4012</td>
<td>55.82</td>
</tr>
<tr>
<td></td>
<td>10. 1.3178</td>
<td>52.50</td>
</tr>
</tbody>
</table>
CONCLUSIONS

From the results of this investigation it would seem that the chief advantage of the apparatus employed, when compared to that used by Deacon, is its compactness.

Perhaps a study might well be made to determine if direct chlorination of organic substances is possible when the same apparatus and catalytic substances dealt with in this study are used.
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