

*A paper presented at the Eighty-Sixth General Meeting, held at Buffalo, N. Y., October 12, 1944, R. L. Murray presiding.*

## HISTORIC DEVELOPMENT OF CAUSTIC-CHLORINE CELLS IN AMERICA<sup>1</sup>

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### ABSTRACT

The development of the American electrolytic alkali-chlorine industry covers a period of over 100 years. Numerous difficulties, both chemical and commercial, had to be overcome. Today the industry ranks among the very foremost of the entire electrochemical group. As to the future, there are still problems of cell construction and cell operation that await solution.

It is now over sixty years since the first caustic-chlorine cell was patented in the United States. Cruickshank in England in 1800 discovered that the electric current breaks up common salt into sodium and chlorine. By 1851 a number of European patents on cells appeared. However, in 1880 Lomas published a book, "The Manual of the Alkali Trade," without mentioning electrolytic caustic, and Lunge in the same year, in his book on "Sulfuric Acid and Alkalies," devoted a bare fifteen lines to the subject. Sixteen years later, in 1896, experimental work had proceeded so far that in Lunge's Second Edition he devoted 360 pages to the chlorine industry, of which 88 pages were on the electrolytic process. Over a dozen patents were cited. Many were skeptical of the commercial value of the electrolytic process owing to the cost of current required and the lack of materials to withstand the corrosive action of the products, alkali and chlorine.

This was the situation when interest became evident in the United States. The fact that both caustic and chlorine could be made by the electrolytic process was intriguing. The first patent issued in the United States was to a Belgian, A. L. Nolf,<sup>3</sup> in 1883. His was a circular, mercury cathode cell but it was never tried out. The first patent by an American was that of A. J. Rogers<sup>4</sup> of Milwaukee, using a fused NaCl bath, but that also failed to function commercially. Several others followed but the first cell actually installed was at the S. D. Warren & Co. paper mill. This was a hypochlorite cell by Hermite and Cooper<sup>5</sup> and was installed in 1888. However, it was only partly successful and was soon abandoned.

Probably other men were experimenting, but so far as can be learned only cells which had been patented were reduced to practice, although

<sup>1</sup> Manuscript received June 6, 1944.

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<sup>3</sup> U. S. Pat. 271,906 (Feb. 6, 1883).

<sup>4</sup> U. S. Pat. 296,357 (1884).

<sup>5</sup> U. S. Pat. 381,372 (1888); Chem. News 60, 228 (1889).

not necessarily on a large, commercial scale. The first alkali-chlorine cell to reach a commercial stage in the United States was that of Ernest A. LeSueur<sup>6</sup> who, while still in school at the Massachusetts Institute of Technology but doubtless more or less familiar with what had been done abroad, believed he could design a cell which would operate successfully. He applied for a patent<sup>7</sup> in 1890. About the same time Isaiah Roberts of Brooklyn made application in 1889 and his patent<sup>8</sup> was issued in 1890 prior to that of LeSueur.<sup>9</sup> However, LeSueur was able to interest capital and the first caustic-chlorine plant was built in 1892 at Rumford Falls, Maine, and began operation in 1893. Associated with LeSueur were C. N. Waite, E. A. and H. I. Allen, Frank McDonald, H. K. Moore and J. Mercer. C. B. Barton entered the organization in 1897. These men will be remembered as pioneers in this work. A description of the Rumford Falls plant and operation was published by Charles L. Parsons.<sup>10</sup>

An interesting account of the early efforts to establish an American electrolytic alkali-chlorine industry and to overcome the natural difficulties met with was given by Charles B. Barton.<sup>11</sup> It is not an easy task to "start from scratch" in an undertaking of this kind and to bring it to successful operation—many "growing pains" and much grief were encountered. Not the least of these "growing pains" was the fact that foreign producers, finding competition arising in America, began to cut prices on imported bleach which had been selling at \$50 per ton. The price was gradually lowered, finally dropping to \$20. This was a staggering blow to the American pioneers and all but put the Rumford Falls plant out of business. But the plant continued to operate until 1898 when the Burgess Sulfito Co. at Berlin, N. H., believing the process had merit, offered to take the plant over. The stockholders of the Electrochemical Company, unwilling to invest more money, accepted the offer and the first American electrolytic caustic-chlorine plant was liquidated and passed out of the picture. The LeSueur cell, however, continues to operate successfully to this day at the Berlin plant, now owned by the Brown Co.

As the LeSueur patents had been assigned to the Electrochemical Company, the sale of the plant to Burgess prevented LeSueur from further promotion of his cell but each of the men who had been with the company followed his own ideas in designing and patenting cells. Other men who had not been connected with the Rumford plant came into prominence. Eighteen patents were granted before that of Castner<sup>12</sup> in 1894. This was followed by that of Carmichael,<sup>13</sup> of Malden, Mass., in the same year. Carmichael's cell was an elaborate affair and was tried out at the paper mill of S. D. Warren Co. but was abandoned.

Nine more patents were granted before that of J. Mercer,<sup>14</sup> a Rumford

<sup>6</sup> *Trans. Electrochem. Soc.* **63**, 187 (1933).

<sup>7</sup> British Pat. 5,983 (April, 1891); U. S. Pat. 583,330.

<sup>8</sup> U. S. Pat. 442,204 (1890).

<sup>9</sup> U. S. Pat. 450,103 (April 7, 1891).

<sup>10</sup> *J. Am. Chem. Soc.* **20**, 868-87 (1898).

<sup>11</sup> *Trans. Am. Inst. Chem. Engrs.* **13**, Part I, 1-7 (1920).

<sup>12</sup> U. S. Pat. 518,135 (1894).

<sup>13</sup> U. S. Pat. 518,710 (April 24, 1894); 637,851.

<sup>14</sup> U. S. Pat. 564,311 (1896).

man, in 1896. Mercer's cell was cylindrical with a stoneware container and was installed at the Nashua River Paper Co. at Pepperell, Mass., and also at the Merrimac Mills at Lawrence, Mass., but had a short life.

E. A. Allen and H. K. Moore were the next Rumford men to appear. In their cell<sup>15</sup> they show and claim the principle of an unsubmerged cathode, claiming priority on this feature. It will be found, however, that Faure in England patented a cell<sup>16</sup> in 1872 which disclosed an unsubmerged cathode.

The Allen-Moore cell, as shown in the patent, had a horizontal cathode as in the LeSueur cell, but was not used in that shape. It was re-designed with a vertical cathode in the next patent.<sup>17</sup> The year 1901 was prolific—21 patents appeared, including those of Bell of England and that of C. E. Acker of Niagara Falls.

By this time Moore had organized the Moore Electrolytic Co. and engaged the firm of Little & Walker to make an experimental test run on the Allen-Moore cell. Accordingly, a month's run was made. The efficiency ran from 63 to 84%. The current applied varied from 328 to 402 amperes. It is to be noted that, while graphite had been produced by Acheson and was available at this time, the anode used was amorphous carbon. A trial of the cell was also made by Eddy & Sons at Saginaw, Mich. The demonstration was successful but was not followed up by Eddy & Sons. Another trial run was made by the Penobscot Chemical Fibre Co. at Great Works, Maine. They did not adopt the cell, but A. B. Larcher, superintendent of the plant at the time, designed a cell<sup>18</sup> which was patented in 1903. Larcher made a success of it at his plant where it continues to run today; but it has not been used elsewhere. Based on the report of Little & Walker, Moore was able to enlist capital and formed the American Electrolytic Co. An agreement was made by which the new company could operate under the Moore patents; the industry took a step west and built a plant at Glen Rock, N. Y. The directors of the new company were so confident that, although they planned to operate only 40 cells at first, they bought equipment for a plant with as many as 1,000 cells capacity. This and the purchase of the salt properties, used up so much of their capital that little was left for operation. Methods of brine purification had not been worked out. The concentration of the caustic with its salt recovery had not been developed and a general ignorance of how the plant should be operated soon put the plant in such a bad shape financially that it could not fulfill its contract with Moore. The directors of the company refused to put up the necessary additional money to put the plant in working shape and thus the second attempt to establish a plant for the commercial production of caustic and chlorine passed out of the picture.

The development which had extended to Glen Rock had already gone as far as Niagara Falls. The ability to get electric power at a moderate cost was an inducement to locate plants at this point as power and salt are the main items of expense in operating alkali-chlorine diaphragm cells.

<sup>15</sup> U. S. Pat. 680,191 (Aug. 6, 1901).

<sup>16</sup> British Pat. 1,742 (1872).

<sup>17</sup> U. S. Pat. 703,289 (1902).

<sup>18</sup> U. S. Pat. 736,982 (1903).

We have already seen that Isaiah Roberts had patented a cell in 1890. A plant called the Roberts Chemical Co. was started at Niagara Falls for the production of caustic potash and hydrochloric acid. Potassium chloride was imported from Germany. A glance at the Roberts patents is sufficient to show that trouble was in store for the plant operation; and as this salt was bought on the cuff, the Germans took over the plant, changed the name to the Niagara Alkali Co., installed Siemens-Billiter cells<sup>19</sup> in 1910, and ran the plant for some time. During the World War, before the United States had gone in, the Germans became apprehensive of what would happen to them. They were successful in selling the plant to E. D. Kingsley. In 1928 the Billiter cells were replaced by Vorce cells. These cells are still in operation.

By this time other plants were built at Niagara. The Mathieson Alkali Co. at Saltville, Va., experimented with the Castner mercury cell already mentioned but, as Saltville was a poor place for such an undertaking, they built a plant at Niagara in 1897 and were the first to use graphite anodes. This plant has been very successful. It was for a long time the only mercury cell operating in the United States.

Among the earlier plants at Niagara was that of the Acker Process Co. which installed the Acker<sup>20</sup> cell in 1900, employing a fused bath of salt with a molten lead cathode. This cell was the result of earlier experimental work at Jersey City by C. E. Acker and F. M. Becket.<sup>21</sup> Owing to the dilution of the chlorine gas by the air drawn into the cell with the salt and the difficulty of keeping the cell cover tight, the ordinary style of bleach chambers could not be employed. It was found necessary to use the Hasenclever apparatus consisting of several cylinders superimposed with conveyors within the cylinders to move the lime gradually through the machine. With the impure chlorine made, the result was poor bleach. The sodium liberated by the current alloyed with the lead cathode and was recovered by passing steam through the molten alloy. By this method the caustic soda could be recovered as solid caustic, but was found to be too impure to ship in that state and had to be purified by treating in large cast iron pots in the regular manner. There was much that could have been improved about the cell and process, but the plant burned down in 1907 and was not rebuilt.

The Development and Funding Co. at Niagara was organized in 1903 to exploit a cell patented by C. P. Townsend.<sup>22</sup> In this cell the principle of unsubmerged cathode patented by Allen and Moore was "side-stepped" by filling the cathode compartment with mineral oil. The caustic solution, which filtered through the diaphragm and cathode, sank to the bottom and was drawn off and concentrated. The company changed its name to the Hooker Electrochemical Co. in 1907. The operation of the plant was taken over by A. H. Hooker who, with others, made improvements. Later, in 1913, C. W. Marsh, engineer for the plant, designed a cell<sup>23</sup> which was installed as an improvement on the Townsend cell and was later installed in a plant in Tacoma, Wash.

<sup>19</sup> Billiter U. S. Pat. 903,951 (Nov. 17, 1908). *Technische Elektrochemie*, Kurt Arndt, p. 463. Publ. F. Enke, Stuttgart (1929)

<sup>20</sup> *Trans. Electrochem. Soc.* 1, 169 (1902); U. S. Pat. 649, 565 (May 15, 1900).

<sup>21</sup> *Trans. Electrochem. Soc.* 72, 14 (1937).

<sup>22</sup> U. S. Pat. 779,383; 779,384 (1905); 972,947 (1910).

<sup>23</sup> U. S. Pat. 1,075,364 (Oct. 14, 1913).

Still later, K. E. Stuart made an exhaustive study of cells and in 1932 patented a cell<sup>24</sup> which differed materially from all others and was built in a size to carry 5,000 to 7,000 amperes.

In 1916 a plant was built at Niagara Falls for the production of aluminum from the chloride,  $\text{AlCl}_3$ . Wheeler cells were installed for the production of the chlorine, but the aluminum phase of the process never worked. The plant was eventually taken over by the Stauffer Chemical Co. who made and who still continue to make chlorine and caustic, along with other products.

During World War I another caustic-chlorine plant was built at Niagara called the Isco Chemical Co. This plant installed Nelson cells which we shall discuss presently.

The next step westward was at Wyandotte, Mich., by the Pennsylvania Salt Mfg. Co. of Philadelphia. This company was the first to make caustic soda in the United States, in 1878. It was made from Greenland cryolite by the Thomson process of roasting cryolite with lime, and had built up a flourishing business in the well known brand of "Lewis Lye." With the advent of the ammonia-soda process, the cryolite process could no longer compete.

Land underlaid with salt at Wyandotte, Mich., was purchased and an arrangement was made with George W. Bell of Liverpool, England, to install his patented mercury cell. The plant was built about 1902 or 1903, but the Bell cell was found to be a failure. An attempt was made by the operatives at the plant to reconstruct the cell, but without success.

The company considered employing LeSueur. This was not done, however, and Arthur E. Gibbs from Niagara Falls proposed a cylindrical diaphragm cell. The first one made was too small and had other drawbacks. A second larger cell was built and was started in operation December 22, 1904. The results warranted a further extension and authority was given for an installation of 50 cells. The work of extension was much delayed by continued experimentation at the plant and the company, irked by the delay, engaged the writer in May, 1905, to go to Wyandotte and take charge of the work. By this time 10 experimental cells had been built. The cells operating at that time were giving a current efficiency of about 70% with 600 to 700 amp. at a voltage of 4 to 4.5 volts. The authorized extension of 50 was completed in September, 1905. Improved results were realized. One installation followed another until a total of 2,600 cells were put in by 1913. The original efficiency of 70% was raised to 93.5% by the year and the voltage reduced to 3.54 v. In 1909 the Canadian Salt Co. at Sandwich, Canada, built a 10-ton plant adopting the Wyandotte cell as then improved and later increased its capacity as business warranted, and it continues in successful operation.

In 1913 the United Alkali Co. of England decided to use this cell at Widnes. Arrangements were made with the Pennsylvania Salt Mfg. Co. to furnish sample cells and specifications, and to educate a man to erect and run them. Audley Drake from England was selected for this work and he studied the process at Wyandotte. When he became suffi-

<sup>24</sup> Chem. & Met. Eng. 45, 354 (1938); U. S. Pat. 1,855,497 (1932).

ciently proficient, he left for England but, sailing on the S.S. "Lusitania," was lost when that boat went down (1915). D. A. Pritchard succeeded him to superintend the installation which, during World War I, was expanded to 6,000 cells.

During the time of the development of the Wyandotte cell, F. G. Wheeler was chief chemist. He had made several suggestions in connection with the building of the second cell. Resigning from the company in January, 1913, he went with the Kimberly-Clark Co. at Appleton, Wis. Having had so much experience with cells at Wyandotte, it was quite natural that he should design a cell of his own. This he did within a year after leaving Wyandotte, bringing out the Wheeler cell. This was similar in form to the Gibbs cell but avoided the claims of the Gibbs patent. Wheeler cells were also installed by the Champion Paper & Fibre Co. at Canton, N. C. Three other employees of the Pennsylvania Salt Mfg. Co. have taken out patents, by those patents have not been used.

It has not been possible to follow this history in strictly chronological order. One of our largest chemical plants is the result of research work done at Case School of Applied Science in 1887-8 by Herbert H. Dow who worked on the natural brines of the United States. Finding the largest amount of bromine in the brine from Midland, Mich., and having worked out an electrolytic method for its recovery, he started in a small way at Midland. It would take much time to follow through all the handicaps Herbert Dow had to overcome. His first work was on bromine but, as the bromine in the brine was liberated by chlorine, it was but a step to make bleaching powder and by 1903 he was making 20,000 tons of it per year.

One hurdle Dow had to take was that of the attempted block of the German bromine makers who thus tried to throttle Dow's bromine business in America. They began cutting bromine prices so that there was no profit for Dow. However, as the price at which the German bromine manufacturers were selling bromine in America was much less than the selling price in Germany, Dow bought all the bromine imported into the United States, shipped it back to Germany, and sold it there at a profit. That stalled the German producers and after that they let Dow alone.

As the Midland, Mich., brine contained a large amount of lime and magnesia, Herbert Dow did not at once try to make caustic as well as chlorine and bromine, but later, by concentrating the brine, the salt could be recovered and used in chlorine-caustic cells. Dow, with the help from his associates, A. M. Griswold, E. O. Barstow and L. E. Ward, developed a large bipolar cell<sup>25</sup> which is used for making caustic soda and the chlorine which is utilized in other processes.

In 1915 Dow gave up the manufacture of bleach in order to devote the chlorine to other chlorine products.

Two Cleveland, Ohio, men, A. W. Burwell and C. E. Baker, in 1902-3, evolved a process for recovering gold and other values from certain refractory ores in the West by the use of chlorine. They designed cells

<sup>25</sup> U. S. Pat. 1,365,875 (Aug. 2, 1921).

for the production of chlorine; a plant was built in Montana, but a flood washed out their dam and plant and it was not rebuilt.

This practically covers the pioneering period.

By this time, about 1915, the industry was on a substantial commercial basis. One other cell emanating from the Rumford plant remains to be mentioned—Frank McDonald, the last of the men from the Rumford plant, patented in 1902 a cell<sup>26</sup> which was rectangular in shape. He later took out three other patents up to 1916. He sold cells to the Warner Chemical Co. at Carteret, N. J., for the production of chlorine for carbon tetrachloride. Their experience with the cell was unsatisfactory and men at the plant working on the problem evolved the Nelson cell which was used by the Westvaco Chlorine Products Corp. and others at Charleston, W. Va., and also at Edgewood Arsenal.

McDonald also sold cells to the New York & Pennsylvania Co. at Johnsonburg, Pa. These were operated for some time but were later displaced by the Hargreaves-Bird cell, an English cell. The D. M. Bare Paper Co. at Roaring Springs, Pa., installed McDonald cells in 1904 and have continued to operate them to their satisfaction ever since. This illustrates an important feature in connection with cell work.

#### CONCLUSION

There are a few fundamental points in cell construction. Aside from these it is a matter of technical knowledge and intelligent "horse sense" in handling them. Only a comparatively few of the cells tried out have stood the test of time. Since the first patent taken out in the United States by Nolf in 1883, 61 years ago, there have been nearly 350 patents on caustic-chlorine cells. Of these, nearly 100 were by foreigners and 98 were on mercury cells. Of all these patents only about 32, less than 10%, have been tried out commercially and only 16 have stood the test of time and continue to operate.

The territory covered extends from Maine to Washington and California, and from Wisconsin to Oklahoma and Texas. Twenty-three states are represented in the list. As experimental work began in Europe before it started here, it might be expected that a number of foreign cells would have been in use in America but, of the 100 foreign caustic-chlorine patents, only 4 have been tried out here, and only 2 continue to function. As we have seen, the English Bell cell failed at Wyandotte, the Austrian Billiter cell was discontinued at the Niagara Alkali Co. The English modification of the American Castner cell is still operated at Niagara Falls by the Mathieson Alkali Co. and by the Du Pont company. A few paper mills still use the English Hargreaves-Bird cell. This last cell, however, produces carbonate of soda instead of caustic.

Many of the cells which have been patented might have succeeded if they had had a fair chance and money to back them, but a far greater number never could have become successful. There were over 40 plants operating before this present war started and the demand for chlorine by the Government has caused 7 more plants to be built. It is possible to touch only the high spots in this review. The installation of the

<sup>26</sup> U. S. Pat. 697,157 (1902).

Vorce cell at Charleston, W. Va., was written up in the March issue of 1928 of "Chemical & Metallurgical Engineering."

It may be stated as a fact that experience in operating cells in most cases indicates changes and improvements. During the ten years from the start of the cells at the Wyandotte plant under the writer, many such changes were made improving the cell in many ways.

Rubber-covered cast iron rings were replaced by cement construction. The projections on the cathode, noted in the Gibbs patent, were found unnecessary; method of feeding brine was changed. Means for improving electrical connections were devised. The result of all these changes indicated the issue of a new patent in 1918.<sup>27</sup> The writer followed with a second patent in 1932 and finally by one in 1937 showing the development of the double cathode cell<sup>28</sup> which is a radical improvement over the single cathode cell.

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**Resumen del artículo: "El Desarrollo Histórico en los Estados Unidos de Celdas Electrolíticas para Cloro y Cáustica."**

A principios del siglo XX se había experimentado mucho con diversas celdas para producir cloro y sosa cáustica, y durante la primera guerra mundial la industria se hallaba bien establecida en los E. E. U. U. En este país se han sacado unas 350 patentes de invención describiendo estas celdas, pero sólo se han probado comercialmente unos 32 tipos, y sólo la mitad de estos continúan funcionando con éxito hoy día.

El autor presenta una cronología completa de los acontecimientos principales en el desarrollo de estas celdas en los E. E. U. U.

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**CHRONOLOGY OF CHLORINE CELL DEVELOPMENT \***

This tabulation summarizes the information given in Dr. Vorce's paper. His data have been supplemented with pertinent dates and facts given by Hugh K. Moore in his chronological study of the development of synthetic hydrochloric acid presented before the American Institute of Chemical Engineers\*\* and with historical data regarding various cell installations contributed by a number of individuals whose cooperation is gratefully acknowledged.

- 1800 Cruickshank electrolyses salt.
- 1851 Charles Watt obtains British Patent 13,755 on a process of making chlorine, soda, hypochlorite, and chlorate by electrolysis of alkali chloride solutions.
- 1878 Pennsylvania Salt Company first produces caustic soda in the United States, using the Thomson process of roasting cryolite with lime.
- 1880 Morgan in British Patent 4,985 describes the electrolysis of sodium chloride in a diaphragm cell.
- 1883 First U. S. Patent on a chlorine cell issued to A. L. Nolf. This is No. 271,906 of February 6, "Process and Apparatus for Obtaining Chlorine and Sodium."
- 1884 First U. S. Patent to a U. S. citizen on a chlorine cell issued to A. J. Rogers.
- 1886 E. Mathes and Weber invent the first commercial cell for caustic soda, known as the Greisheim cell, and obtain German patent 34,888 describing it. A solid diaphragm was used.
- 1888 First cells (for hypochlorite) installed by Hermit and Cooper at S. D. Warren paper mill. Only partly successful.

\* Compiled by P. S. Brallier of the Niagara Smelting Corporation, Niagara Falls, N. Y.

\*\* Transactions. 14, 91 (1922).

<sup>27</sup> First Vorce patent U. S. #1,286,844 (Dec. 3, 1918).

<sup>28</sup> Vorce double cathode cell patent U. S. #2,078,517 (April 7, 1937).



- Ernest A. LeSueur "commences work toward a commercial solution of the wet electrolysis of common salt."
- 1889 LeSueur turns to the diaphragm type of cell after having experimented with a cell having a suspended mercury cathode.  
H. H. Dow at Midland, Mich., conducts first experiments on the electrolytic production of bromine. Chlorine was obtained as an undesired by-product. This bromine cell was the forerunner of the Dow chlorine cell.
- 1890 Isaiah Roberts applies for a U. S. patent on a chlorine cell.  
Ernest A. LeSueur applies for a U. S. patent on a chlorine cell.  
The first commercial-type LeSueur cell is built and operated on a pilot-plant scale at Newton Upper Falls, Mass.
- 1891 Roberts and LeSueur patents issue in that order.  
Castner patents an electrolytic process for manufacturing sodium and potassium.
- 1892 Ernest A. LeSueur and Charles N. Waite interest Boston capital in the formation of the Electrochemical Company, which installs LeSueur cells at Rumford Falls, Maine. E. A. and H. I. Allen, Frank McDonald, H. K. Moore, J. Mercer, and C. B. Barton are associated with LeSueur and Waite in this installation, which consists of 25 tanks taking 1200 amperes each and containing 18 bell-type cell units each.  
H. H. Dow produces bromine on a commercial scale by electrolysis of Michigan brine at Midland, Mich.  
Castner experiments with the use of the mercury cathode to produce caustic soda.
- 1893 Operation of LeSueur cells starts, making them the first to be used commercially in the U.S.A. The cells were designed to be run with the cathode either submerged or unsubmerged.
- 1894 Castner cell patent issues.
- 1895 Castner mercury cells for caustic soda are operated for the first time in America.  
LeSueur is made manager of the Electrochemical Company, and Hugh K. Moore becomes chief chemist.
- 1896 J. Mercer, from the Rumford Falls group, patents a cylindrical cell with a stone-ware container. The cell is tried at the Merrimac Mills plant of the Nassua River Paper Co. but is unsatisfactory.  
Dow starts production of bleaching powder from chlorine produced in a cell adapted from bromine manufacture.
- 1897 The Castner mercury cell for chlorine and caustic soda is installed at Niagara Falls by Mathieson Alkali Works.  
The Allen-Moore cell is invented. The pilot cells were the first to use Acheson graphite anodes, which came onto the market at this time.  
The Electrochemical Company at Rumford Falls is liquidated due to "dumping" of imported bleaching powder and caustic soda.
- 1898 The Burgess Sulfite Company, subsequently the Brown Paper Co., purchases the LeSueur cells and moves them to Berlin, N. H., where they continue to operate up to the present time. C. B. Barton is engaged.
- 1899 The Acker Process Company builds a plant at Niagara Falls, N. Y., for the production of chlorine and caustic soda, using a cell with a molten lead cathode and fused salt as electrolyte.
- 1901 E. A. Allen and Hugh K. Moore obtain U. S. patents 680,191 and 703,289. The latter specifies a vertical, unsubmerged cathode.  
Allen-Moore cells replace the Carmichael cells at the Cumberland Mills plant of the S. D. Warren Co.  
Nineteen other cell patents issue, including that of Bell, of England, and one to C. E. Acker.  
H. K. Moore organizes the Moore Electrolytic Co.  
Roberts Chemical Co. starts operation at Niagara Falls, electrolyzing muriate of potash in the Roberts cell. The chlorine produced was sold as HCl, made by combining chlorine with the by-product hydrogen to produce the first "synthetic HCl."
- 1902 Frank McDonald, from the Rumford Falls group, patents a chlorine cell. The American Electrolytic Co. installs a chlorine plant made up of Allen-Moore cells at Glen Rock, N. Y.; but is unsuccessful through over-extend-

- ing its capital investment before the initial problems had been worked out. Acheson graphite anodes were first used in a large way in this plant.
- Pennsylvania Salt Co. tries the Bell mercury cell at Wyandotte, Mich., but finds it unsatisfactory.
- A. W. Burwell and C. E. Baker design and install cells to make chlorine for gold extraction in Montana, but the plant is lost in a flood.
- Clinton P. Townsend, a patent attorney of Washington, D. C., applies for a patent on a diaphragm-type caustic-chlorine cell employing a vertical diaphragm and an oil-filled cathode compartment for separating caustic solution.
- 1903 A. B. Larcher, superintendent of Penobscot Chemical Fiber Co., Great Works, Maine, obtains a cell patent after observing the operation of the Allen-Moore cell. The Larcher cell is still in use at this plant. The Dow Chemical Co. at Midland, Mich., produces 20,000 tons per year of bleaching powder.
- 1904 A. E. Gibbs starts his second and larger vertical cylindrical cell at Pennsylvania Salt Mfg. Co., Wyandotte, Mich. McDonald cells are installed at D. M. Bare Paper Co., Roaring Springs, Pa. These cells are still in use.
- 1905 The Development and Funding Co., later becoming the Hooker Electrochemical Co., carries out pilot-plant work on the Townsend type cell in Brooklyn, N. Y. L. D. Vorce engaged by Pennsylvania Salt Mfg. Co. to expedite the installation of 50 vertical cylindrical cells at Wyandotte. The installation was completed in September. Warner Chemical Co. at Carteret, N. J., purchases McDonald cells and proceeds to make carbon tetrachloride. First J. McPhail and subsequently H. R. Nelson patented cells based on experience with and improvements on the McDonald cell.
- 1906 The Development and Funding Co. starts a 5 tons/day chlorine plant at Niagara Falls, N. Y., using 2,000-ampere Townsend cells. The chlorine was used to produce bleaching powder. Leo H. Baekeland invents a method of saturating and recirculating anolyte brine. This method was used by the Development and Funding Co. in connection with the operation of Townsend cells.
- 1907 Electro-Bleaching Gas Co. starts liquefaction of chlorine at Niagara Falls, purchasing chlorine from the Roberts Chemical Co. The Acker Process Co. plant at Niagara Falls burns down; and is not rebuilt. A. E. Gibbs receives U. S. Pat. 874,064, issued December 17, on a cylindrical cell with a submerged cathode.
- 1908 German interests take over Roberts Chemical Co. at Niagara Falls, N. Y., and change name to Niagara Alkali Co.
- 1909 Canadian Salt Co. installs 10 tons/day chlorine capacity at Windsor, Ontario, using the Gibbs cell. Pennsylvania Salt Mfg. Co. makes the first tank car shipment of liquid chlorine in the U. S. from its Wyandotte plant.
- 1910 Billiter cells, imported from Germany, are installed at the Niagara Alkali plant at Niagara Falls.
- 1912 A. H. Hooker discovers that the voltage characteristics of a cell were improved by decreasing electrode height. This principle was applied in later cells designed at the Hooker Electrochemical Co.
- 1913 Clarence W. Marsh, of the Hooker Electrochemical Co., designs a cell with a cathode screen folded into fingers to increase electrode area per unit volume. Three cells were made in a single unit, one on top of the other, in order to save floor space. The triple cell was not successful, but the folded type cathode was retained in the Hooker Type E cells, two circuits of which are still in operation at the Hooker plant at Tacoma, Wash. Pennsylvania Salt Mfg. Co. now has 2,600 Gibbs cells installed at its Wyandotte plant.

- United Alkali Co. adopts the Gibbs cell for its plant at Widnes, England. This installation was subsequently expanded to 6,000 cells.
- F. G. Wheeler designs, builds and operates a 5-ton chlorine plant for Kimberly-Clark Co. at Rothschild, Wis., using a modification of the Gibbs cell designed by him.
- 1914 Dow discards the original trap-type cell and adopts the vertical diaphragm, filter press type.
- Hooker Type F cells, similar in design to the original Townsend cell but enlarged to 5,000 amperes capacity and improved in construction, are installed at the Hooker Electrochemical Co. at Niagara Falls, in addition to existing circuits of Townsend cells and Hooker Type E cells.
- F. G. Wheeler patents a vertical cylindrical cell as the result of experience at Pennsylvania Salt Mfg. Co. with the Gibbs cell.
- 1915 Nelson cell patent issues as U. S. 1,149,210 of August 10.
- Warner-Klipstein Co. installs 560 Nelson cells at its plant at South Charleston, W. Va.
- Dow Chemical Co. discontinues the manufacture of bleaching powder in order to utilize chlorine for other products.
- 1916 Whiting mercury cells for chlorine and caustic soda are installed by the Oxford Paper Co., Rumford Falls, Me. (Replaced by Sorenson cells.)
- Wheeler cells are installed by the Champion Fibre Co. at its plant in Canton, N. C.
- Nelson cells are in use in the plant of Niagara Electrochemical Co. which became a unit of Roessler and Hasslacher Chemical Co., which in turn was eventually absorbed by E. I. du Pont de Nemours Co.
- Nelson cells are installed in the new plant of the Isco Chemical Division of Innis Spieden and Co. at Niagara Falls, N. Y., which started operation October 7.
- Wheeler cells are installed by Kimberly-Clark Co. at Kimberly, Wis.
- Wheeler cells are installed at the plant of the Niagara Smelting Corporation, Niagara Falls, N. Y., to supply chlorine for the production of aluminum by electrolysis of the fused chloride.
- 1916 Dill and Collins Paper Co. at Philadelphia installs a unit of Allen-Moore cells to produce calcium hypochloride for beaching soda pulp.
- Brown Paper Co. installs 160 Allen-Moore cells in addition to the LeSueur cells already in operation.
- Electro-Bleaching Gas Co. purchases Niagara Alkali Co. from its German owners.
- Westvaco Chlorine Products Co. is formed to take over the Warner-Klipstein chlorine unit.
- 1917 Allen-Moore cells are installed and put into operation by the Great Western Electrochemical Co. at Pittsburg, Calif.
- 1918 Nelson cells are installed at Edgewood Arsenal to a capacity of 50 tons of chlorine per day.
- An installation of Allen-Moore cells by the Chemical Warfare Service at Belle, W. Va., is completed shortly after the Armistice. This unit is subsequently acquired and operated by the Belle Alkali Co. for the production of bleaching powder.
- Dill and Collins install 120 additional Allen-Moore cells, the tower system for liquefying chlorine, and a caustic fusion system in order to supply chlorine to Edgewood Arsenal. The plant was completed about the time of the Armistice; but its products were not needed.
- L. D. Vorce receives U. S. Patent 1,286,844 issued December 3, disclosing a vertical cylindrical cell for chlorine-alkali.
- Michigan Electrochemical Co. at Menominee, Mich., installs a 5-ton chlorine unit designed by F. G. Wheeler and using his cells.
- 1920 Monsanto purchases the 120 Allen-Moore cells originally installed by Dill and Collins at Philadelphia; and makes an experimental run. The unit was then shut down for several years.
- 1921 Fields Point Manufacturing Co. installs Nelson cells at Providence, R. I.
- K. E. Stuart, A. H. Hooker, and T. L. B. Lyster develop an orifice feed system to feed brine with solid salt in suspension to chlorine cells, replacing the anolyte recirculation system invented by Baelkand.

- 1923 Pennsylvania Salt Mfg. Co. purchases the plant of the Michigan Electrochemical Co. and replaces the Wheeler cells with Gibbs cells.
- 1924 Roessler and Hasslacher Chemical Co. at Niagara Falls starts the operation of Downs cells for the production of chlorine and sodium from fused salt late in the year. By early 1925, the Nelson chlorine-caustic cells had been abandoned.
- 1926 Monsanto starts continuous operation of its Allen-Moore cells in January.
- 1927 K. E. Stuart of the Hooker Electrochemical Co. patents a cell with 5,000 to 7,000 amperes capacity.  
Westvaco Chlorine Products Co. installs 3,600 Vorce cells at South Charleston, W. Va., as an addition to their Nelson cell units.  
Belle Alkali Co. adds Vorce cells to its Allen-Moore installation.  
Solvay Process Co. installs chlorine-caustic soda cells at Syracuse, using the Allen-Moore cell.
- 1928 K. E. Stuart, with others of the Hooker Electrochemical Co., develops means of forming asbestos diaphragm on Hooker Type E cathodes. The asbestos was drawn onto the screen cathode by means of vacuum from a water suspension. This development considerably improved the current efficiency of the Type E cell because it made a more uniform diaphragm and eliminated leaks and bare cathode spots which were common with the wrapped paper diaphragm previously used.  
Hooker Type E cells are installed in the new Hooker Electrochemical Co. plant at Tacoma, Wash. The cathodes of these cells were changed to the deposited diaphragm type in 1930.  
Pennsylvania Salt Mfg. Co. installs and starts operation of Gibbs cells at their new plant in Tacoma, Wash.  
Wheeler cells are installed in a chlorine-caustic soda unit at Deep Water Point, N. J., by E. I. du Pont de Nemours Co.
- 1929 A 10-ton chlorine unit consisting of 288 Vorce cells starts operation at the Diamond Alkali plant in Painesville, Ohio.
- 1930 Niagara Alkali Co. replaces its Billiter cells with modified Gibbs cells.  
Diamond Alkali Co. installs an additional 288 Vorce cells; and on July 7 files a patent application on a vertical cylindrical diaphragm cell designed by E. W. Tucker and C. N. Windecker. Tucker had been associated with A. E. Gibbs at Pennsylvania Salt Co.  
K. E. Stuart and associates develop the Hooker Type S cell which takes full advantage of the use of deposited diaphragm, and which retains the low electrode height in accordance with the findings of A. H. Hooker.
- 1932 Diamond Alkali completes the replacement, started in 1930, of 576 Vorce cells with 864 Tucker-Windecker cells.  
U. S. Pat. 1,842,703 is issued January 26 to L. D. Vorce, describing certain improvements in his vertical cylindrical chlorine cell.
- 1934 C. N. Windecker and E. W. Tucker receive U. S. Pat. 1,957,482 issued May 8 which describes their vertical cylindrical chlorine-alkali cell.
- 1935 Canadian Industries Ltd., successor to Canadian Salt Co., completes the installation of a mercury cell alkali-chlorine unit at Cornwall, Ontario, on March 10. This mercury cell had been developed in England.
- 1935 Monsanto Chemical Co. secures licenses for installation of 240 Tucker-Windecker (Diamond Alkali) cells at its East St. Louis plant. Subsequently 160 additional cells were licensed for this plant.
- 1936 Columbia Alkali Division of Pittsburgh Plate Glass installs Vorce cells and starts production of chlorine at Barberton, Ohio.  
Hooker Type S cells are licensed for use by the Champion Paper and Fibre Company, which was the first licensee of this cell.
- 1937 L. D. Vorce receives U. S. Pat. 2,078,517 issued on April 27 and describing a vertical cylindrical cell with cathode area provided for both sides of the anode.  
A brine feed system involving automatic saturation at high temperatures and feed to cells through an accurately formed, corrosion-resistant orifice is developed at Hooker Electrochemical Co. for use in conjunction with Type S cells.  
Solvay Process Co. installs a chlorine-alkali unit at Baton Rouge, La.

- Hooker Type S cells are installed by Champion Paper and Fibre Co. at Pasadena, Texas.
- 1938 Columbia Alkali expands its Barberton plant by licensing Hooker Type S cells.  
Southern Alkali Co. starts production of electrolytic chlorine at Corpus Christi, Texas.  
Downs cells for the electrolysis of fused salt are installed in connection with the plant of the Ethyl Corporation at Baton Rouge, La.  
Michigan Alkali Co. completes installation of English mercury cells for chlorine at Wyandotte, Mich., on December 8.  
Installation of Hooker Type S cells at the Canton, N. C. plant of Champion Paper and Fibre Co. is completed.
- 1939 Canadian Industries Ltd. completes the installation of English mercury cells at Shawinigan Falls on October 9.
- 1940 Dow Chemical Co. installs a chlorine-alkali unit at Freeport, Texas.
- 1943 Mathieson Alkali installs a Defense Plant Corporation unit at Lake Charles, La., for the electrolytic production of magnesium in a cell and by a process which yields chlorine as a by-product.  
A large chlorine-alkali plant is installed for the Defense Plant Corporation by Columbia Chemicals Division of Pittsburgh Plate Glass Co. at Natrium near New Martinsville, W. Va. A Hooker Type S cell modified to operate at higher ampere loading is used.
- 1944 From 1936 to date, licenses have been issued for the installation of Hooker Type S cells in 24 plants, including Chemical Warfare Service Arsenals, and the unit at Basic Magnesium at Henderson, Nevada.

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#### DISCUSSION

L. D. VORCE: I might say that when an engineer of the United Alkali Company in England came over to visit the Wyandotte plant to see whether they could install the cell in England, I showed him all through the plant. Finally we arrived in our large cell room where we had 1,800 cells; he turned to me and said, "Mr. Vorce, I understand why the Company has made a success of this cell. It is not the cell—anyone can build a cell—its the attention which has been given to the details."

C. F. BURGESS<sup>27</sup> (*Communicated*): Mr. Vorce, who can speak with authority as a man who has graduated with honors from the pioneer period of the chlorine-caustic electrolytic cell, has summarized his observations in this interesting and unique paper.

As a historical reference I believe something more than passing mention might be given to the mercury type of cell. At Rumford Falls, often referred to as the "cradle of industrial electrochemistry in America," there was developed a mercury cell beginning in 1907 and leading to a successful installation in 1910. I refer to the Whiting electrolytic cell, a description of which was given to The Electrochemical Society and recorded in Volume 17 of the Transactions (1910).

L. D. VORCE: The Whiting cell was adopted by the Oxford Paper Company and was operated by them for some time but later Sorensen, their superintendent, redesigned the cell, improved it, and that is the cell they are using today. The Sorensen cell is a quite successful mercury cell.

<sup>27</sup> C. F. Burgess Laboratories, Inc., Chicago, Illinois.