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## PRODUCTION OF POTASSIUM PERCHLORATE \*

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### ABSTRACT

For several years an electrolytic process for the production of potassium perchlorate has been operated at Claremore, Oklahoma, by the Cardox Corporation of Chicago. This paper describes the process and the electrolytic cells. Sodium chloride is oxidized to sodium chlorate in one set of cells, and the sodium chlorate is oxidized to sodium perchlorate in a second set of cells. The sodium perchlorate is then reacted with potassium chloride to form potassium perchlorate and sodium chloride, and the latter salt is recycled to the chlorate cells. All operations are conducted batchwise. A graphite anode-steel cathode cell is used for the conversion of chloride to chlorate, and a platinum-clad anode-steel cathode cell is used to change chlorate to perchlorate. Construction and operating data are presented for both cells.

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### INTRODUCTION

Since early 1942 potassium perchlorate has been produced at Claremore, Oklahoma, by the Cardox Corp. of Chicago, using an electrolytic process. Although the two types of cells involved operate upon sodium salts, the basic raw material is potassium chloride. Fresh sodium chloride is introduced only to replace mechanical losses.

This paper will describe the cyclic process and the two cells developed to implement the production of potassium perchlorate in this plant.

### THE PROCESS

Briefly, the process consists of the following steps:

1. Electrolysis of sodium chloride to sodium chlorate in appropriate cells.
2. Concentration of the chlorate cell liquor to separate sodium chloride, followed by cooling to crystallize sodium chloride.
3. Electrolysis of sodium chlorate solution to produce sodium perchlorate in solution.

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formed by the spalling of the cell anodes are settled and left behind when the solution is decanted off. The evaporator is operated to achieve a final boiling temperature of  $54.4^{\circ}\text{C}$  ( $130^{\circ}\text{F}$ ) at the 26 in. (660 mm.) of Hg vacuum maintained in the unit. Some sodium chlorate is precipitated along with the sodium chloride during the concentration since the solubility limit of both salts is exceeded at this temperature. At the conclusion of the evaporation a plug cock on the bottom discharge line is opened gradually to raise the pressure, stir up the settled salts, and redissolve the precipitated sodium chloride. A final temperature of  $113^{\circ}\text{C}$  ( $235^{\circ}\text{F}$ ) is attained as the pressure approaches atmospheric, and the sodium chlorate is completely soluble at this higher temperature.

The evaporator slurry is then run directly to a decanting tank (5) where the mother liquor is separated and led to a Swenson-Walker crystallizer (6). The wet, unwashed  $\text{NaCl}$  crystals are dissolved in the decanting tank in fresh water or in perchlorate mother liquor, and the resultant solution is pumped to the chlorate cell feed tank (1) for re-entry into the process. As the chlorate solution enters the crystallizer at about  $93^{\circ}\text{C}$  ( $200^{\circ}\text{F}$ ) it contains approximately 60%  $\text{NaClO}_3$ , 4%  $\text{NaCl}$ , and 36%  $\text{H}_2\text{O}$ . In the crystallizer the solution is cooled to an average temperature of  $30^{\circ}\text{C}$  ( $86^{\circ}\text{F}$ ) with cooling water. Sodium chlorate precipitates, but sodium chloride remains in solution. The small quantity of  $\text{KClO}_4$  in the liquor also precipitates at this point.

Separation of the  $\text{NaClO}_3$  crystals is effected in a Bird solid-bowl continuous centrifuge (7) whose capacity is 1,500 lb. (680 kg.) of damp  $\text{NaClO}_3$  per hour. Wash water is used in small quantities sufficient to displace the mother liquor only; too much washing will dissolve  $\text{NaClO}_3$  rather than any solid  $\text{NaCl}$ . Very little solid  $\text{NaCl}$  is present in the chlorate crystals, the  $\text{NaCl}$  content of the mother liquor being the source of the final  $\text{NaCl}$  content which averages 0.2%, with a maximum of less than 1.0%, on a dry basis. Mother liquor plus the wash water from the centrifuge is recirculated to the storage tank (3) from which the evaporator is fed; it contains about 400 g./L of  $\text{NaClO}_3$  and 100 of  $\text{NaCl}$ .

The approximately 2 g./L of sodium dichromate carried in the chlorate cell liquor to inhibit cathodic reduction and reduce corrosion of the steel cell tanks remain in the liquors throughout the above portion of the process, and also serve to protect the steel equipment used subsequent to the electrolysis step. The dichromate is recycled with the  $\text{NaCl}$  solution from the evaporation step to the chlorate cells with very little replacement required. A concentration of about one gram per liter of  $\text{Na}_2\text{Cr}_2\text{O}_7$  is maintained in the perchlorate cells to decrease cathodic reduction and protect the steel equipment from corrosion.

Sodium chlorate made by the above operations is dissolved in a dissolving tank (8) to obtain a concentrated solution for feed to the perchlorate cells. Steam heating coils supply the considerable heat required to dissolve the  $\text{NaClO}_3$ , which has a negative heat of solution of some 5.6 kilogram-calories per gram-mole. The concentrated sodium chlorate solution, containing from 650 to 700 g./L  $\text{NaClO}_3$ , is run into the perchlorate cells (10) where the chlorate is oxidized to per-

chlorate. A detailed description of the cell and its operation will be given later. From the cells the perchlorate solution, whose composition approximates 800 g./L  $\text{NaClO}_4$  and 20 of  $\text{NaClO}_3$ , is pumped to a storage tank (11).

Potassium perchlorate is made by allowing the sodium perchlorate solution to react metathetically with a potassium chloride solution in a Swenson-Walker crystallizer (13). The potassium chloride solution is prepared at a concentration of 270 to 300 g./L  $\text{KCl}$  by dissolving solid  $\text{KCl}$  crystals in hot water (12). One solution is fed to one end of the crystallizer, and the other is run into the opposite end. After the solutions have been mixed, cooling water is circulated through the shell and a final average temperature of  $20^\circ\text{C}$  ( $68^\circ\text{F}$ ) is reached. The quantity of potassium chloride added is slightly less than the stoichiometric requirement to depress the potassium ion content of the mother liquor which is subsequently returned to the chlorate cells.

A Bird solid bowl continuous centrifuge (14) is used to separate the  $\text{KClO}_4$  crystals from the slurry leaving the crystallizer, and the crystals are washed with water to remove mother liquor and any solid sodium and potassium chlorides present in the cake. The  $\text{KClO}_4$  crystals are dried in a Link-Belt Roto-Louvre dryer (15) with air heated by passage through steam coils, elevated to a small bin, and packaged. The final product contains a maximum of 0.25%  $\text{NaCl}$ , 0.3%  $\text{KClO}_3$ , and 0.15%  $\text{H}_2\text{O}$ .

Mother liquor plus wash water from the centrifuge contains 155 g./L  $\text{NaCl}$ , and is concentrated in a single-effect vacuum evaporator (18) to a concentration of 280 g./L before being returned to the feed tank for the chlorate cells. As the concentrated liquor cools in this storage tank, some of the  $\text{KClO}_4$  content precipitates.

#### CHLORATE CELL

The cell used for the electrolysis of chloride to chlorate depends upon the use of vertical graphite anodes and steel cathodes. Similar cells were investigated extensively by Groggins, McLaren, Pitman, Davis, and Turer.<sup>1</sup>

A diagram of the Cardox chlorate cell construction is shown in Fig. 2. The cell body is an uninsulated sheet steel rectangular tank strengthened by 1.5 in. (3.8 cm.) angle irons around the top and down each corner, extensions of the corner angle irons serving as the legs. Two vertical sheet steel cathodes divide the cell into three sections lengthwise. They are welded to the end walls of the tank and extend from about 3 in. (7.62 cm.) above the floor to within about 4 in. (10.16 cm.) of the top of the cell body to allow the cell liquor to circulate in a downward-upward path around them. Two rows of 5 each graphite electrodes hang between the cathode sheets, and between the two anode rows is positioned a steel cooling chamber about the same height as the cathodes and 3 in. (7.62 cm.) wide, also welded to the cell end walls. The graphite electrodes are 7 in. (17.8 cm.) wide,

<sup>1</sup> P. H. Groggins, A. L. Pitman, J. McLaren and F. H. Davis, *Chem. & Met. Eng.* **44**, 302 (1937); A. L. Pitman, J. McLaren, F. H. Davis and P. H. Groggins, *Ibid.* **45**, 692 (1938); P. H. Groggins, A. L. Pitman and F. H. Davis, *Ibid.* **47**, 468 (1940); J. McLaren, J. Turer, A. L. Pitman, F. H. Davis and P. H. Groggins, *Trans. Electrochem. Soc.* **79**, 93 (1941).

1 in. (2.54 cm.) thick and 40 in. (101.6 cm.) long, and rest at the bottom on concrete blocks about 2.5 in. (6.33 cm.) wide, 4 in. (10.16 cm.) long and 3.5 in. (8.9 cm.) high with notches on the upper surface to receive the anodes. They are positioned at the top by 5 rectangular porcelain insulators, each slotted to receive two opposing anodes; the insulators rest in the cell cover. The anodes extend about 4 in. (10.16 cm.) above the cover, and copper bus bars running along each side of the two rows are bolted to them by two bolts per anode.

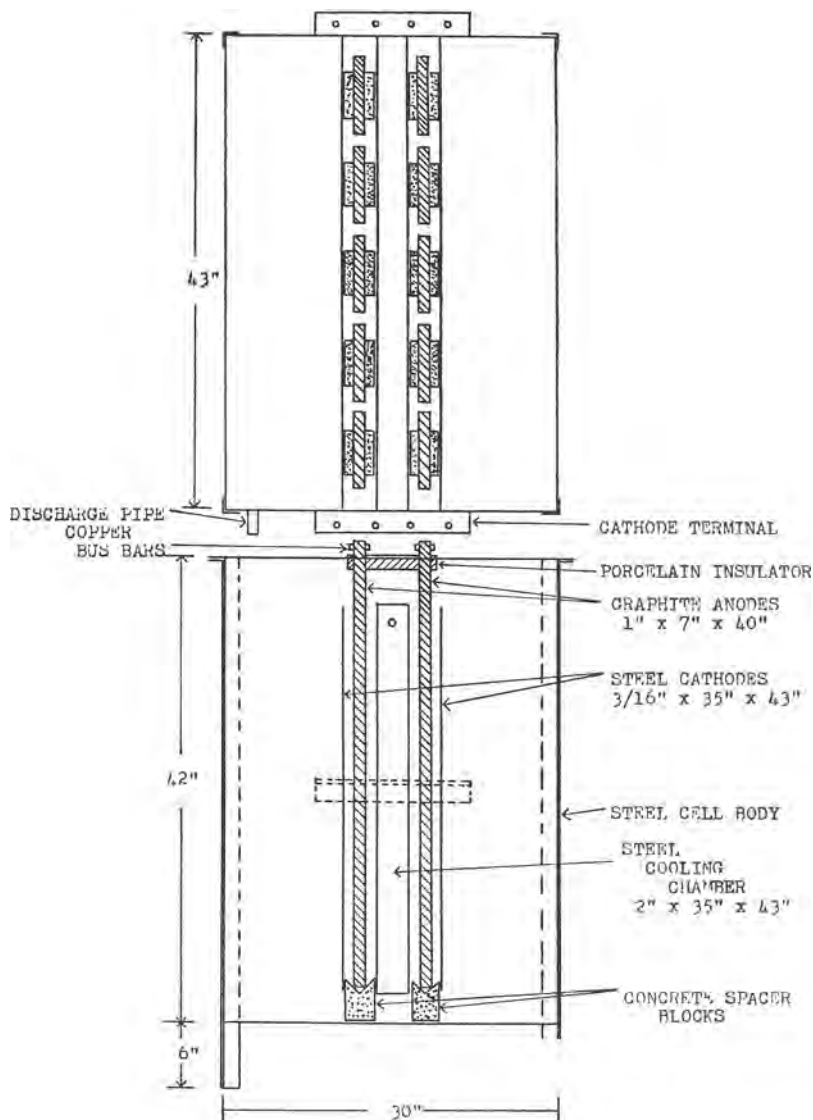


FIG. 2. Cross-section of the Cardox chlorate cell.

Cooling water flows through the cells in parallel, entering and leaving the cooling chamber by rubber hoses attached to nipples welded into the cell walls. Short horizontal lengths of 2 in. (5.08 cm.) angle irons welded to the end walls serve as contacts by which power cables are connected from the anode bus bars of one cell to the cathodes of the next. The sheet steel cell body acts somewhat as a cathode, although the cathodic current density on the greater area of the walls is so small as to afford little more than cathodic protection to the cell body.

Hydrogen discharged at the cathodes causes a circulation of the cell liquor up the central section and down the outer sections. It is vented through the roof from each cell by means of a plastic tube leading from the cell cover. No segregation of anodic and cathodic gases is made.

The cells are operated batchwise, each 200 gal. (757 L) per cell charge of sodium chloride solution being electrolyzed to a final concentration of 85 g./L NaCl before the cell is emptied. Temperature is maintained at an average of 35° C (95° F) by the cooling water circulating through the cooling chamber, and by heat losses to the air through the cell walls. To decrease anode loss the temperature is kept as low as possible with the cooling water available.

Cell voltage ranges from 3.1 to 3.5 v. depending upon the age of the graphite electrodes, and 1,600 amp. are carried by each cell. Current density averages 50 amp./sq. ft. (5.4 amp./dm.<sup>2</sup>). Anodes have an average life of one year and losses, including stub losses, are about 20 lb./ton NaClO<sub>3</sub> (10 g./kg.). Current efficiency has been maintained at 85% to 90% for several years. The total power applied to the bank of chlorate cells is 500 kw. Production per cell is some 2 lb. (0.908 kg.) of NaClO<sub>3</sub> per hour.

The pH of the cell liquor is kept at 6.8 by additions of a 1:1 or 2:1 hydrochloric acid solution every 24 hours. A glass electrode is used to measure the pH of samples.

Table I summarizes the pertinent data on construction and operating characteristics of the chlorate cell.

TABLE I

*Characteristics of Cardox Chlorate Cell*

Dimensions, cell body	height, 42 in. (106.7 cm.); length, 43 in. (109.2 cm.); width, 30 in. (76.2 cm.)
Capacity	200 gal. (757 L)
Anode	10 graphite bars, 1 x 7 x 40 in. (2.54 x 17.8 x 101.7 cm.)
Life	1 year
Consumption	20 lb./ton NaClO <sub>3</sub> (10 g./kg.)
Cathode	2 steel sheets, 43 x 35 x $\frac{1}{8}$ in. (109.2 x 89 x 0.48 cm.)
Voltage	3.1 to 3.5
Current, amperage	1,600
Current efficiency	85% to 90%
Current density, anodic	50 amp./sq. ft. (5.4 amp./dm. <sup>2</sup> )
Temperature, average	35° C (95° F)
pH	6.8
Production per cell	2 lb. NaClO <sub>3</sub> /hr. (0.91 kg./hr.)
Cell feed	250 g./L NaCl
Cell effluent	450-550 g./L NaClO <sub>3</sub> , 85 g./L NaCl

### PERCHLORATE CELL

Fig. 3 is a diagram of the Cardox perchlorate cell showing construction and dimensional features. The cell body, similar to that used for the chlorate cell, is constructed of sheet steel and angle irons. A steel cooling chamber, 30 in. (76.2 cm.) long, 27 in. (68.5 cm.) high, and 2 in. (5.08 cm.) wide, placed vertically in the center of the length of the cell is welded to the ends of the cell, and contains inlet and outlet nipples for connection to the cooling water circulation system. The cathodes are four vertical 3 in. (7.62 cm.) diameter steel tubes, 33 in. (83.8 cm.) long, depending from the cell cover, and are spaced two on each side of the cooling chamber. Each cathode pipe has 8 to 10 holes, 0.5 in. (1.27 cm.) in diameter, drilled through the pipe wall

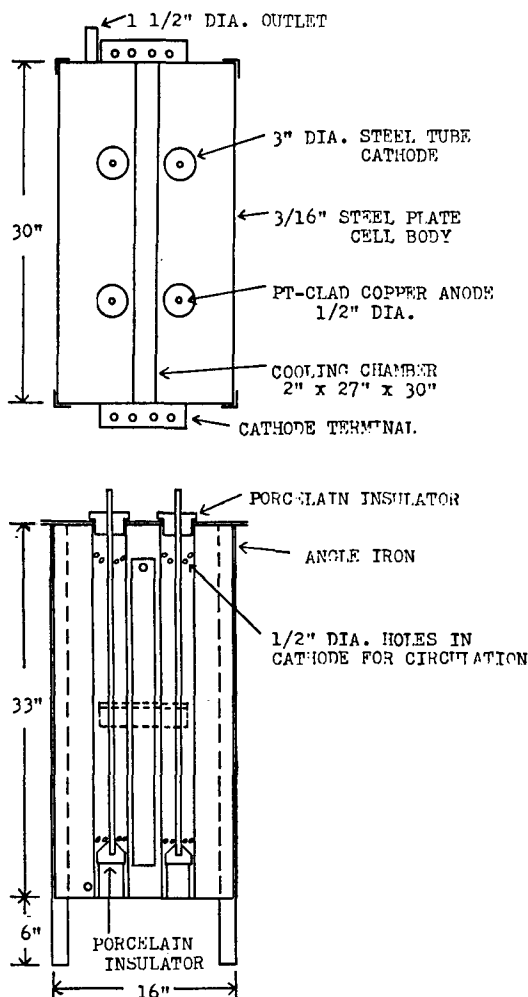


FIG. 3. Cross-section of the Cardox perchlorate cell.

near each end to permit the entrance of solution at the bottom and exit of hydrogen and liquor at the top. Porcelain insulator-spacers in the bottom and top of the cathode tube hold the anode. The bottom spacer rests on steel clips fastened on the inner wall at the bottom of the tube, and the top spacer has a flange at its upper edge which rests on the top edge of the tube.

Anodes are 0.5 in. (1.27 cm.) copper rods clad with a 0.02 in. (0.05 cm.) thick platinum sheet. They are made by the American Platinum Works, Newark, N. J. A similar construction is described by Burman.<sup>2</sup> The 30 in. (76.2 cm.) long anodes are centered in the cathode tubes by the two porcelain spacers. Insulated, stranded copper cables connect the four anodes in parallel to a steel angle iron welded to the wall of the next cell.

Each cell is filled to within 2 in. (5.08 cm.) of the cover with 50 gal. (189 L) of a solution of  $\text{NaClO}_3$  nearly saturated at 45° C (113° F); its concentration is 650 to 700 g./L  $\text{NaClO}_3$ . The cells are operated batchwise, and each portion of liquor is electrolyzed to a final chlorate content of 20 g./L  $\text{NaClO}_3$  before being discharged to a storage tank. Hydrogen formed at the cathode leaves the tube through the upper row of small holes and is vented through the roof from the cell cover.

Operating temperature ranges from 45° to 55° C (113° to 131° F), and the pH is alkaline, at times reaching a maximum of 10.5. The pH is not controlled as it is in the chlorate cell.

Some chlorine is evolved early in the cycle, and at the end of the electrolysis traces of ozone are found in the exit gas.

Voltage of the cell is 6.2 to 6.8 and is sensitive to temperature and chlorate concentration. It rises at the end of the run as the chlorate concentration becomes lower. A current of 500 amp., 125 amp. per electrode, is maintained. Current density is 480 amp./sq. ft. (52 amp./dm.<sup>2</sup>), and a current efficiency of 93% to 97% has been obtained for several years. Production per cell is 2 lb./hr. (0.91 kg./hr.).

TABLE II

*Characteristics of Cardox Perchlorate Cell*

Dimensions, cell body	height, 33 in. (83.8 cm.); length, 30 in. (76.2 cm.); width, 16 in. (40.6 cm.)
Capacity	50 gal. (188.5 L)
Anode	4 Pt-clad Cu rods, 0.5 in. (1.27 cm.) dia. x 30 in. (76.2 cm.) long
Cathode	4 steel tubes, 3 in. (7.6 cm.) dia. x 33 in. (83.8 cm.) long
Voltage	6.2 to 6.8
Current, amperage	500/cell, 125/electrode
Current density, anodic	480 amp./sq. ft. (52 amp./dm. <sup>2</sup> )
Current efficiency	93% to 97%
Temperature, average	45° to 55° C (113° to 131° F)
pH	alkaline, less than 10.5
Production per cell	2 lb. $\text{KClO}_4$ /hr. (0.9 kg./hr.)
Cell feed	650 to 700 g./L $\text{NaClO}_3$
Cell effluent	800 g./L $\text{NaClO}_4$ 20 g./L $\text{NaClO}_3$

<sup>2</sup> L. C. Burman, *Chem. & Met. Eng.* **48**, No. 3, 89 (1941).



Operation of the cell is greatly affected by the chloride content of the cell liquor. At concentrations of more than 2% (dry basis) no perchlorate is formed. The effect is abrupt, and it appears that a critical chloride concentration is involved. No explanation of this effect can be offered.

The potassium ion content of the  $\text{NaClO}_3$  solution fed to the perchlorate cells causes precipitation of some  $\text{KClO}_4$  in the cells as the  $\text{NaClO}_3$  is converted to  $\text{NaClO}_4$ . About 2 lb. (0.91 kg.) of  $\text{KClO}_4$  forms per day per cell. This equals about 5% of the total perchlorate present in the final cell liquor. It is washed from the cells to a collecting sump and returned to the process when the batch is finished.

Table II lists the construction and operating characteristics of the perchlorate cell.

#### ACKNOWLEDGMENT

Appreciation is expressed to the officials of Cardox Corporation for permitting the publication of the above information, and to the many technical men of Cardox Corporation whose joint contributions resulted in the development of the cells and the process.

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#### Resumen del artículo: "La Producción de Perclorato Potásico."

Se describe la producción comercial de perclorato potásico por la Cardox Corporation (Fig. 1) por un procedimiento discontinuo.

Se pasa un electrólito con 200 a 250 gm. por litro de  $\text{NaCl}$  por celdas (Fig. 2) que lo convierten en gran parte en clorato sódico. Emplean ánodos de grafito y cátodos y recipiente de acero. Luego se separa el clorato por evaporación y enfriamiento, y se disuelve hasta 700 gm. por litro en agua y se oxida a perclorato en otras celdas (Fig. 3) con ánodos platinados y cátodos de acero.

Entonces se mezcla la solución de  $\text{NaClO}_4$  con otra de  $\text{KCl}$ , precipitándose cristales de  $\text{KClO}_4$ . El líquido restante se concentra por evaporación y se devuelve a las celdas que producen clorato.

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#### DISCUSSION

MILTON JANES<sup>3</sup>: I was interested in your comments about chlorine dioxide explosions. We encountered a chlorine dioxide explosion while extracting an anode with water in a Soxhlet apparatus. Chlorine dioxide was formed by interaction of hydrochloric acid and chlorate and a sharp explosion occurred blowing the condenser out of the top of the extractor. We had occasion to make a few test runs with a batch process similar to what you have described and lessened the possibility of chlorine dioxide explosions in the cell by taking advantage of dynamic evaporation which occurred in the electrolytic cell as a result of hydrogen evolution. Thus instead of adding concentrated acid we employed acid considerably diluted with water or with brine, and thus avoided local high hydrochloric acid concentration in the cell and consequently any degree of chlorine dioxide production.

<sup>3</sup> National Carbon Company, Inc., Cleveland, Ohio.

The acid that we finally concentrated on was about a one to one. It has been found other places that chlorine dioxide, to be evolved with an acid, has to have a reducing agent there. The chlorine ion itself acts as a reducing agent but you have to have a pretty high concentration of acid. The one to one is not bad but we are dealing with laborers or gentlemen of labor, I might say, who were previously farmers, and many points escaped them. If they fell asleep once, they had twice as much acid the next time and of course the local high concentration would result in some disturbances.

RALPH M. HUNTER<sup>4</sup>: I would like to ask any one of the four previous authors,—what is the matter with magnetite? There is a scarcity of magnetite mentioned in the papers.

MILTON JANES: I am not going to answer the question. I would just like to qualify by saying that I recognize that in Germany graphite was scarce and expensive and yet there was magnetite, and in the U. S. magnetite is, let us say, not available, and yet it was overwhelmingly used in Germany. Now, if it were available at a decent price, would it be an attractive anode?

C. L. MANTELL<sup>5</sup>: Well, did you ever try to make it?

C. A. HAMPEL: Replying to Dr. Mantell, it is difficult to make. It would be a very good anodic material but is unavailable in this country. About four years ago, I tried to get a piece of it for experimental work. I wrote to all the gentlemen at St. Mary's, Pennsylvania, that I knew and they reported to have experimented with it at one time but with no results, and I wrote to someone else, I believe it was Dr. M. B. Geiger of Oldbury Electro Chemical Company, in hopes that he might have used magnetite as an anodic material and could give me a little sample. He didn't know where it could be obtained and didn't know of it in this country. During the war, of course, there was very little commerce between the United States and Germany so they didn't have that source of supply.

MILTON JANES: In a report by Mr. Hunter on German chlorate practice<sup>6</sup> a figure is given for magnetite anode weight loss of 0.02 pounds per pound of chlorate produced. Essentially the same figure for magnetite weight loss is reported by Mr. R. B. McMullin.<sup>7</sup> This amounts to 40 pounds per ton of chlorate produced, as large if not larger than the weight loss with graphite anodes. To be sure, volume loss would be less for the magnetite anodes because of the considerable contrast in densities of the two anode materials. Furthermore, we have understood that a Swedish firm producing chlorate using magnetite anodes apparently did not remove the iron resulting from solution of the anodes, and making a virtue of necessity called their product "Golden Brown" chlorate.

J. C. SCHUMACHER<sup>8</sup>: We were very much interested in what the Germans had done with magnetite, and were able to secure some samples of the type and quality they used. We found that the cell voltage was higher for magnetite than for graphite, and on going into the matter further, we concluded that probably one of the reasons the Germans hadn't used graphite was that they had never developed a suitable graphite for this type of work. I understand that they have gone over to some graphite, and that they probably would use more graphite if they had a suitable quality available.

NELSON C. WHITE<sup>9</sup>: We had in our possession one small piece of magnetite which came from Sweden years and years ago. We have done a small amount of experimental work in a very small cell with that one anode, and at the risk of calling everybody wrong who has spoken today, we didn't get any indication

<sup>4</sup>Dow Chemical Co., Midland, Mich.

<sup>5</sup>Starrett-Lehigh Bldg., New York, N. Y.

<sup>6</sup>Office of Technical Services Report PB-204 (1946) "The Electrochemical Industry, Bitterfeld Area."

<sup>7</sup>FIAT Final Report N. 732 (1946), "Electrochemical Operations at I. G. Farbenindustrie A. G., Bitterfeld."

<sup>8</sup>Western Electrochemical Co., Los Angeles, Calif.

<sup>9</sup>International Minerals & Chem. Corp., Carlsbad, N. Mex.

of color from iron and I would say the loss was infinitesimal. However it is like the cardox platinum loss, we haven't operated long enough to know what the loss is going to be per unit of chlorate produced, but we would very much like to find somebody, in spite of Dr. Mantell, that could make some anodes so that we could try them out on a decent sized scale.

W. C. GARDINER<sup>10</sup>: The Carborundum Company, Research Department, made a magnetite anode several years ago which I tested as a chlorine anode but it gave a yellow coloration to the brine and had a high overvoltage so that I was no longer interested. I mention it now as a source of magnetite. If anybody wishes to test it, I suggest that they get in touch with the Carborundum Company.

R. M. HUNTER: At the end of the war the chlorate cells at Bitterfeld were unanimously equipped with 144 magnetite electrodes per cell. Each electrode was about 2" x 4½" x 48" and although there was very little evidence of wear, I was informed that the consumption was 0.02 lbs. magnetite per pound of chlorate. The dense uniform appearance of the electrodes would seem to contradict this figure, but it was stated to be true.

A. C. LOONAM<sup>11</sup>: I would like to ask Dr. Hunter how they got a decent electrical conductivity on the magnetite anodes.

R. M. HUNTER: Speaking from memory on a detail on which I did not take notes, I recall that the 2" by 4" fused magnetite anodes were cast around a copper bar approximately 1¼" in diameter. The magnetite extended several inches above the liquor level and I believe the copper bar was fastened to an assembly frame, which conducted the current, by a turnscREW.

A. C. LOONAM: Dr. Fink can probably tell more about this than I, but it is my understanding that when the big electrolytic copper plant at Chuquicamata, Chile, was started, magnetite anodes were installed. These were hollow and plated on the inside with copper to improve their electrical conductivity. However, the gas given off got behind the magnetite and caused it to spall off. As a result, these anodes were abandoned and replaced, first by Duriron and later by the Chiles anode developed by Dr. Fink.

<sup>10</sup> Mathieson Alkali Works, Niagara Falls, N. Y.

<sup>11</sup> 70 East 45th St., New York, N. Y.