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TERMINATION REPORT RELATING TO THE
SHEER-KORMAN PROCESS

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July 1950

Burns and Roe, Inc.
New York, New York

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T E R M I N A T I O N R E P O R T

CONTRACT AT(30-1)-438

RELATING TO

THE SHEER-KORMAN PROCESS

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ABSTRACT

Contract No. AT (30-1) - 438 authorized the Contractors to design, construct and operate a pilot plant for the production of beryllium chloride, based on the Sheer-Korman Process.

A development laboratory was constructed at the pilot plant site to determine factors required for the construction of a commercial plant and to carry out an experimental anode fabrication program.

Subcontractors with special knowledge in their fields were selected to work with the Contractor Group and to design and construct certain components of the pilot plant.

Successful experimental anodes were developed at the Maspeth laboratory by an extrusion process, from beryl ore and bituminous coal.

Process demonstrations were made, a small pilot plant was designed, constructed and operated, and research and development were conducted on a laboratory scale. Chloridization of beryl ore and bituminous coal anodes was demonstrated. Some progress was made in development of a collection system, including collection by cold electrostatic precipitation.

The contract was terminated when the Commission decided that final design of a pilot plant was undesirable or impossible during the contract period. The data obtained were not sufficient to permit an economic evaluation of the process.

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INTRODUCTION

The Sheer-Korman Process is a process for producing chlorides of metals from ore by the use of a high erosion arc. The process depends on the peculiar property of such an arc of vaporizing and ionizing the material contained in the anode. The reduced metals thus vaporized and ionized by the intensely concentrated energy of the arc are united with the chlorine of a surrounding atmosphere. The vapors and fumes thus formed are then to be separated, preferably by fractional condensation or other appropriate means.

Experimental work on a laboratory scale preceding the contract led to the belief that:

1. Under favorable conditions, somewhere near 100% of the metal contained in the ore can be converted to chlorine compounds.
2. The erosion rate for beryl ore based on arc power exceeded 100 grams per kwh.
3. The products could be separated to the required degree of purity.
4. Additional information was required for the construction of a plant to produce beryllium chloride on a commercial scale, which included:
 - a. The possibility of producing commercial anodes which would maintain the arc at a suitable erosion rate.
 - b. The chloride conversion rate attainable under commercial conditions in percent of metal assay in the original ore.

- c. The erosion rate attainable under commercial conditions in grams of metallic chloride per kwh of the total power consumed.
- d. The purity of the final product produced.

It was recognized that additional technical problems to be solved were: the construction of the arc chamber, feeding the electrodes, handling and feeding the chlorine, controlling moisture, collecting the chlorides and recovery of waste gases.

Contract No. AT(30-1)-438 was signed on February 24, 1949 authorizing the participants, comprised of the Inventor Group and Burns and Roe, Inc., to design, construct and operate a pilot plant for the production of beryllium chloride based on the Sheer-Korman Process. A laboratory was constructed at the plant site to develop information and experience to determine the factors required for the construction of a commercial plant on a pilot plant scale. In order to develop suitable anodes from beryl ore, this laboratory was equipped to process raw materials, manufacture and bake anodes, sample and analyze materials in process, conduct tests and perform trial burning of finished anodes in a commercial carbon arc unit.

To guard against beryllium poisoning, health procedures were instituted which included showers and complete change of clothing for workers daily, weekly check-up on physical fitness and periodic medical examinations. Protective hoods and a ventilating and filtering system were installed on all toxic

equipment.

A specification was prepared for the various pilot plant components based upon description of the process, calculations and data furnished by the Inventors.

Subcontracts were let with Combustion Engineering-Superheater, Inc., and General Precision Laboratory, Inc., for the design, construction and furnishing of an arc chamber and its related mechanism. A subcontract was let with Research Corporation for precipitators, condensers and other units of the collection system required to handle products of the process.

Experimental anodes were developed from beryl ore, bituminous coal and various mixing agents by an extrusion and baking method. Anodes extruded using bentonite with water and molasses as addition agents produced reasonably successful results. The best of these anodes were those extruded with molasses.

Shelled anodes resembling commercial carbon electrodes were also fabricated which provided continuous burning in an arc unit although erosion rates obtained were lower than those with full core anodes.

Development work was carried on with commercial carbon arc units with the arc enclosed in metallic chambers. Arcs were operated in both air and chlorine atmospheres as a means of trial testing of anodes produced in the extrusion laboratory.

Experiments were made on magnetic stabilization of arcs enclosed by metal chambers.

Demonstration runs were made to determine process efficiency and product purity from small scale pilot plant equipment set up in the laboratory, the first of these being accomplished to meet a deadline of April 4, 1950.

The results of Run #1 demonstrated on a semi-commercial scale, the feasibility of operating a high intensity arc in a chlorine atmosphere using an anode made of beryl ore and bituminous coal. Chlorination of the ore and some degree of selective condensation and product collection was achieved. The presence of moisture hampered results, reaffirming the importance of moisture control.

As part of the research program, further demonstrations followed on a smaller scale using a transite arc chamber and glass apparatus collection system. In general, the problems of moisture control and the limited duration of the arcing period with full core anodes prevented the obtaining of satisfactory results. Continuous arc operation was short, of only one to three minutes duration. However, product gases, although partially hydrolyzed, were successfully precipitated in a Cottrell.

Several anode erosion rates were measured in a chlorine atmosphere as 115 to 150 grams of anode per kwh using extruded anodes composed of beryl ore, bituminous coal and molasses.

PART I - INVENTION AND EARLY DEVELOPMENT OF THE SHEER-KORMAN
PROCESS

A. Arc Experiments

The first experiments leading to the Sheer-Korman process were made in 1939 in order to develop a practical process for the treatment of siliceous aluminum ores which do not lend themselves readily to commercial processes. At that time high grade bauxite was scarce and there was considerable interest in finding a process that could reduce kaolin on an economically competitive basis.

The objective of Korman and Sheer's early work was to investigate qualitatively the possibility of the arc chlorination of clay to form a mixture of anhydrous chlorides. They regarded this step as the key to the development of such a new process-if it could be carried out cheaply enough-since they believed that the mixture of anhydrous chloride products could be easily purified by available techniques.

The apparatus used in the first experiments is shown in the diagram A included in the appendix.

The first step taken was to examine the effect of bringing a mixture of ore and carbon into contact with the flame of an arc. A graphite cup was packed with this mixture. An arc was struck between the rim of the cup and a carbon electrode in a jar filled with chlorine. Substantially, no reduction took place.

Next, efforts were made to introduce the ore physically into the arc. This proved to be extremely difficult since the flame of the arc seemed to avoid all physical contact with solid materials.

Following this, efforts were directed toward the objective of fabricating the ore-carbon mixture. This was done in order to avoid the physical dispersion of the material by the arc flame, which had been experienced when this mixture was in powdered form. Proper quantities of clay were thus mixed with carbon in the form of lamp black and powdered graphite with a suitable binder and squeezed into a mold. The binders used were in turn molasses, oil, tar, water glass etc.

A typical water glass formula was:

Kaolin	475 gms.
Carbon	100 "
Water glass (38%)	5 oz.
Water	3 oz.

The briquets formed from the above most successful formula were about 1" long, 3/8 "in diameter, and of uniform consistency throughout, without any carbon shell.

An arc was struck between carbon electrodes and the briquet was thrust into the flame. Again the flame was found to avoid the solid material. When thrust into the corona of

the flame, the briquet was observed to melt. No substantial reaction was observed to take place.

In the next experiment, the briquet so formed was used as the anode of the arc. Since the crater of the arc is known to be the hottest portion of the arc, it was believed that this would promote the desired reaction. Great difficulty was experienced in maintaining the arc discharge and it was evident that this was due to the high resistance of the briquet. In general, during the normal operation of the arc, there was still substantially no reduction of the ore to metal chlorides. There were, however, sporadic moments during some condition of abnormal operation of the arc when a much faster erosion took place. The reasons for this more rapid erosion were not then understood.

A patent search was made showing that it had already been proposed to use an arc for ore reduction in a reducing atmosphere, as for example, in the patent to Austin Smith #1,752,936 which proposed four ways of introducing the ore into the arc:

1. As a powder;
2. Through a hollow electrode;
3. As pencil projected in from the side;
4. To incorporate it in the electrode, not stating which.

All of these suggestions had been tried, without practical effect, in the tests already described.

The prior patents did not suggest making the ore-containing electrode the anode of the arc, nor did they teach how to make an anode of ore that would have sufficient conductivity to maintain the arc. The patents did not account for the sporadic results so far observed. In fact, these patents did not disclose any means of attaining practical results.

Patent counsel advised that the fact that the results obtained by Sheer and Korman from the proposals of Austin Smith were so meagre, provided a probable explanation for the reason that process has never come into practical use. He stated, however, that if these sporadic effects of high erosion should be investigated and if this abnormal condition, whatever it was, could be maintained, it might well furnish the basis for a new and valuable process.

At this time, in the middle of 1940, a room was rented at the Electrical Testing Laboratories, East End Avenue, at 79th Street, New York City, to conduct this further research. A new and improved apparatus was built. However, construction was consistent with methods used in previous experiments.

The briquet was held in a socket made from graphite rod. This assembly served as the positive electrode and was

free to move in a vertical direction. A gas seal was achieved by means of a rubber diaphragm clamped to the lower part of the briquet holder and to the bottom of the arc chamber. The arc chamber was constructed of transite having a pyrex glass observation window. The cathode consisted of a graphite rod fixed in a vertical position and held in a graphite fixture cemented to the top of the box. Means for introducing chlorine through an annular opening surrounding the cathode were also provided.

The arc was struck by raising the anode holder manually until the briquet made contact with the cathode, and then lowering it sufficiently to establish the arc.

The briquets employed were formed of mixtures of the ore with from 15 to 20% carbon to render them conductive. A suitable binder was used and mixtures were compressed into briquets 3/8" in diameter and 1" long. The briquets were then baked at approximately 1200°F to bond the materials together.

Typical of the results obtained from arc operation using a clay and carbon briquet of 3/8" diameter were:

Voltage across terminals:	20 - 60
Current in amperes:	16 - 40
Length of arc:	1/8" - 3/4"
Erosion rate:	20 g/kwh
Color of arc:	blue white
Atmosphere around arc:	clear

The observed erosion rate was disappointing - too slow to be of commercial value.

Then a new phenomenon was encountered. As the anode was lowered, inadvertently far below its normal position while the arc was burning, the arc was not extinguished as is the case with carbon electrodes. Instead, the color of the flame changed to a reddish orange, while at the same time a dense white condensate appeared in the chamber in the form of a cloud. This was assumed to be the same phenomenon already observed periodically, but heretofore not isolated. The condensate so formed was found to be water soluble, indicating it to be aluminum chloride.

Experiment showed that this new phenomenon occurred if the arc was first formed in a normal manner and then drawn out to abnormal length, e. g. 2-1/2 to 3 inches. When this was done, the same phenomenon always occurred, that is, the arc assumed the reddish color and the white product was spewed out from the anode as a cloud, and the anode was consumed rapidly.

It was surprising that a higher erosion was obtained, because when the arc was elongated this way, the current was reduced.

There was another phenomenon that characterized this

long arc. The arc crater instead of eroding away the briquet over its entire surface, bored a hole down the axis of the briquet, leaving a wall around that end of the arc. This new phenomenon continued until the crater eroded completely through and made contact with the carbon anode holder. The arc, thereupon, again took on the blue white color of a carbon arc, the discharge of the white vapor ceased and the erosion rate fell away. Diagram B in the appendix illustrated this phenomenon.

The arc was burned for a considerable time in this condition, but the phenomenon did not reoccur. Once the arc travelled to the carbon holder, instead of the briquet, the high erosion rate was gone. Thereafter the ore, although present at high temperature in contact with and completely surrounding the arc, nevertheless ceased to take active part in the "mechanism" of the arc. This was quite surprising, as the temperature of the walls of the briquet channel due to the contact of the arc flame was probably not more than 10% below that of the arc crater itself. No temperature measurements of this part of the anode were taken, however.

Comparitive results of the elongated arc operation are shown by the following average figures:

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	<u>Volts</u>	<u>Current in Amperes</u>	<u>Color</u>	<u>Estimated Erosion g/kwh</u>
Long arc on briquet	110-150	7-10	Red	320+
Short arc on briquet	40- 60	20-24	White	20
Long arc on graphite	20- 40	20-40	White	20

The explanation of why greater results were obtained by lesser current in the long arc is believed to be as follows:

In any arc, the anode is subjected to an intense bombardment of electrons entering the anode from the arc stream. The velocity and hence the kinetic energy that these electrons acquire before they strike is determined by the electric field through which they pass, particularly closely adjacent to the anode crater.

In the normal arc, the energy given out by the electrons impinging on the anode face is converted into heat, raising the temperature of the crater face to about 4000 °K, at which the entire energy, except that conducted away by the electrodes themselves, is dissipated in the form of radiant energy. In such an arc, the calculated average value of the electron velocity is below that required to ionize the substance of the electrode, and the temperature never rises high enough to cause any appreciable vaporization of the anode material. The evaporation is too slow to be of importance and the solid carbon and ore cannot mingle freely with the chlorine and with each other. This produces a condition in which the reaction is very slow.

When the arc is elongated, the electron stream which previously had covered the anode face suddenly contracts and covers only a smaller part of that area, at the center. Even though the current is decreased by this phenomenon the voltage is increased. This greater potential could cause an increased velocity in the impinging electrons, and the electron energy is concentrated on a very much smaller area.

If a sufficient number of electrons achieved a velocity greater than that required to ionize the elements existing in the ore, we might expect a good fraction of ionization, and an accompanying increase in the vaporization. Such ionizations could weaken chemical bonds, so that dissociation and reduction of the ore material would be greatly facilitated. These phenomena could occur substantially only at the anode crater of the arc and only when the material to be dissociated is a part of the anode. Furthermore, once dissociation takes place the products themselves resulting from the dissociation are highly volatile at the crater temperature, especially when ionized. They would, therefore, evaporate much more quickly from the reaction zone. This would seem to further facilitate evaporation of the ore. Thus there would be created a much greater quantity of vapor of the ore and ore materials in vapor form to mingle with the chlorine, thereby greatly facilitating the reaction.

The presence of the reaction products in comparatively large quantity in the highly luminous area immediately in front of the anode crater was assumed to be responsible for the change in the coloration of the arc, the reddish color being characteristic of the aluminum spectrum.

The reason for the sudden contraction of the arc and the consequent sudden increase in current density and energy concentration, has not been completely explained. This condition accompanies or causes the sudden appearance of vapor of the ore and ore materials and involves complicated factors of magnetic field and energy distribution within the arc.

The change in color and the formation of large quantities of a cloud of aluminum chloride in conjunction with the rapid erosion of the anode, indicated that the reaction rate was probably high because the increased voltage and increased current density of the long arc had increased the ionization and vaporization.

A series of experiments testing this theory established that the elongated arc to which reference has been made could achieve a rate of erosion which was impossible with the arc operated in a normal manner.

Tests were then made to determine the applicability of the new process to other ores, including kyanite, bauxite, asbestos, serpentine, feldspar and so forth. In all cases

similar results were achieved and high erosions were obtained. Therefore, it appears that both theory and practice justified the conclusion that it was applicable to any refractory aluminum ore and possibly to any refractory ore.

B. Collection System

Attention was next given to the collection of the products. It had been observed that the chlorides were always formed as a highly dispersed smoke or fume. Attempts were made to produce a system of collection by various means. These included dissolving the product in water and analyzing the solution, use of a cooled baffle system with a variety of plate compositions, and employment of filters and bags. Wet collection and baffles were completely ineffective. Filters and bags were only partially successful. The control of the pore openings in the filter was a difficult problem. Either the fume was too fine to be caught, or it would collect until the pores were clogged and could not be freed quickly enough to be practical. It was hoped that this work was carried far enough to indicate that the problem could be solved. The solution in practical form was felt to be more properly left to a more extensive stage of development.

C. Vacuum Work

The work had now been carried into the war period. Chlorine had become a critical material and this fact was one

of the chief objections to the exploitation of the chlorine process at that time, a situation which might last for several years.

It was, therefore, determined to direct the experimentation to another phase of the process in which chlorine was not required. The theory which had been evolved for the process indicated that the break-down of the ore compounds was accomplished by the arc itself, independent of the chlorine. A series of experiments in neutral atmospheres and in vacuum, were thereupon initiated. The arc was struck between a carbon cathode and a briquet anode of similar composition to that already described, the arc being drawn out to produce the effects which were peculiar to the new phenomena. The apparatus used in these experiments is shown in diagram C in the appendix.

The results obtained at low pressures were far more interesting than those obtained at normal pressures of neutral gases. The anode eroded in a manner similar to the anode in the elongated chlorine arc, although not quite as efficiently. It gave forth a grayish powder, which upon examination was found to contain small metallic crystals. In the best experiments more than 40% of the metal content of the ore was reduced directly to the metal by the elongated arc operating in vacuum.

A patent application was filed in 1942 on this vacuum process, and some discussions were held as to its exploitation.

At this point, however, the experimenters were absorbed by the war machine. All their time was consumed by the war effort and for this reason further laboratory activity had to be suspended.

D. Resumption of the Chlorine Process at
Phelps Dodge Corporation

When Korman and Sheer were released from their war time assignments, the Phelps Dodge Corporation was induced to support the continuation of the laboratory investigations.

These tests were directed to the applicability of the process for the production of beryllium from beryl ore. This company's interest in this process centered in the possibility of large scale production of cheap beryllium copper alloy. It was felt that the Sheer-Korman process offered the possibility of producing cheap beryllium, which was necessary to produce a competitive alloy. Dr. Korman was engaged by that company to work full time on the process at their plant.

Initially, a demonstration was set up for application of the arc chlorination process to the production of chloride from beryl. After laboratory-scale chlorination was successfully accomplished, investigation was undertaken to determine the method for producing beryl anodes by procedures and materials adaptable to commercial practice. A period of four months was devoted to this phase alone.

The result of this study on anodes developed a procedure which succeeded in giving results superior to the developments that had theretofore been obtained.

In these tests it was discovered that as compared between the short arc and the long, using beryl as the ore, the rate of wastage of the anode was not a measure of the rate of reduction of the ore. This was due to the fact that most of the material which came from the anode with the short arc consisted of unreacted fragments of briquet thrown off and therefore did not represent a true erosion. With the elongated arc, however, practically all of the material thrown off by the arc was obtained in the form of a chloride with very little unreacted ore. This dispersal effect obtained in the experiments with short arcs was observed with beryl ore. It was not further investigated because the elongated arc gave the same results with the beryl ore that had been obtained with all other ores so far attempted.

In order to demonstrate that the products of the beryl disintegration could be obtained in the vapor phase and carried out of the chamber, the apparatus was modified and the walls of the chamber were constructed of fused quartz and insulated, so that the reaction products were produced and withdrawn in the vapor phase. They were partially collected by bag filters.

Some further work was carried out on the separation of the reaction products, and it was concluded that the silicon of the ore could be quantitatively separated in a comparatively simple manner as silicon tetrachloride. Further consideration and study caused the conclusion to be drawn that the products could be separated entirely by fractional distillation, but facilities were not available to determine the practical form of the necessary apparatus.

Upon completion of these experiments at the Phelps Dodge Corporation it was felt that the work had progressed as far as it could be effectively carried out with the apparatus available. The work so far done was considered to be of special significance because it established the validity of the basic phenomenon that is, the effect of a high erosion arc on refractory ores, and it pointed the direction in which further investigation must be carried out in the next larger scale of operations.

In planning the next step, it appeared that the application of the elongated arc for the erosion of an ore-bearing anode might present considerable difficulty on a large scale.

The phenomenon leading to the high erosion rate was, therefore, again examined. The following physical effects had been observed with the elongated arc:

1. The crater assumed a small area of the anode, leaving the remainder of the anode untouched;

2. Instead of traversing the cross-section of the anode or spreading out over its face, as in the case of the short arc, this smaller crater was maintained in size, but continued to bore rapidly in depth towards the bottom of the anode;

3. This erosion was accompanied by evidence of a marked ionization of the vapor of the ore material, as indicated by spectrographic analysis.

In examining these phenomena it became clear that the dominant factor in the elongated arc was that the current which had previously occupied the entire face of the anode, was now concentrated in an area about $1/4$ that size. Thus, although the total current decreased, the current density per unit of area greatly increased, assuming that the area of the hole bored in the briquet is the area of the actual crater. Thus, increased current density was an accompaniment of the higher voltage and was apparently directly connected with the ionization.

The conclusion was reached that the greater erosion is the result of the increased energy density per unit area of the crater. This comparison is shown as follows:

	<u>Voltage</u>	<u>Current</u> <u>in amperes</u>	<u>Crater area</u> <u>in cm²</u>	<u>Amperes</u> <u>/cm²</u>	<u>Percentage of the</u> <u>Ore chlorinated</u>
Short Arc	70	10 - 13	0.283	40	5 - 45%
Long Arc	130	8	0.073	110	39 - 80%

E. Theory of Arc "Mechanism"

The probable effect of the concentration of energy by elongation of the arc was to deliver energy by electron bombardment to the anode at a rate greater than could be dissipated laterally from the crater. Under these circumstances, the necessary result was that the energy delivered to the crater accumulated with consequent rise in temperature until ionization and sufficient vaporization took place to restore the balance. No facilities were available for making quantitative measurements of either of these factors, but there remained no doubt of their presence.

It is desirable at this point to consider the consequences of increasing the rate of vaporization of anode material by this process.

It is recognized that in the case of a normal carbon arc the extent of vaporization of anode material is so small that it does not materially influence the behavior of the arc discharge. Only sufficient vaporization occurs to furnish the few positive ions necessary to maintain quasi-neutrality in the arc flame. For the size of current involved this requirement is easily satisfied by a few vapor molecules which,

when ionized, drift through the arc flame toward the cathode very slowly because of their large mass.

Because of the slow drift velocity, a small positive ion concentration is readily able to neutralize practically all of the negative electron space charge, creating a quasi-neutral condition in the flame.

In the normal arc the electrons leaving the discharge to enter the anode effectively provide a thin layer of negative space charge in this region. In order for the arc to operate, electrons reaching this region must be provided somehow with sufficient acceleration to penetrate this negative space charge against its repulsive forces. Accordingly the voltage distribution of the arc discharge seems to adjust itself, so that a relatively high voltage exists across this layer, high enough to produce the required acceleration. This is known as the "anode fall of potential" or "anode drop". The electrons here attain their highest velocity and it is here that the positive ions are formed due to collisions of electrons having more than ionizing velocity with the material of the surface of the anode and with such vapor molecules as may exist in the anode region. Such positive ions tend to neutralize the negative space charge and keep the anode drop at a moderate value.

It is this phenomenon which is affected by elongating the arc. First, by increasing the energy density at the crater, much more anode material vaporizes. This vapor mechanically displaces positive ions from the region of the anode drop, blowing them away rapidly. This results in a decrease of the positive ion concentration in this region and a corresponding increase in the negative space charge. An increased ionization of the displacing vapor is produced, since more electrons now have entered the region. These electrons simultaneously attain a greater than ionizing velocity there.

The effect, therefore, of increasing the length of the arc is apparently to increase the rate of energy transfer to a given mass of anode, to the point where there is a marked increase of the vaporization and ionization of the ore. Both of these effects greatly accelerate the reaction, which is now predominately a vapor phase reaction.

How far the ultimate reactions occur in separate stages and how far in a single stage has not yet been determined. As has been previously stated, when the high erosion arc is operated at low gas pressures, the ore compounds are broken into elements. Although this takes place in several stages, the overall reaction when chlorine is employed is typically represented by

This reaction is believed to take place while the ore, carbon, and chlorine are mingled in vapor phase, so that they can mix most thoroughly and react most rapidly upon each other on this account. Moreover, chlorine (and the other halogens) possesses the ability to form negative ions, and in the high erosion arc the arc materials had a high degree of positive ionization. Thus all these factors if present would combine to speed up the conversion of ore material into chlorides.

It was recognized that there were practical difficulties in working a process in which only the ore of the electrode reacted, and in which no apparent reaction occurred with the walls of the tunnel formed through the briquet by the elongated arc. The conclusion was reached that the next step was to find how to obtain the new high erosion arc in a manner to cover the entire electrode face, and thus reduce the electrode as a whole.

The change from low current arc phenomena to the elongated high erosion arc was not a gradual one. It appeared quite suddenly. Up to a certain point the entire anode face was kept in incandescence by the arc without appreciable reduction. Then the phenomenon changed and the arc jumped to the small area and the high erosion began.

The above theory contended that this new phenomenon

was dependent on exceeding a critical current density, and that the elongated arc attained that critical density by concentrating on an area small enough to insure it. If this theory was correct, and if this critical density were maintained sufficient to insure the ionization and vaporization of anode material, it appeared that the high ore reduction rate could be achieved over the entire face without the need of elongating the arc.

In another field, an arc had been produced for searchlight purposes, known in Europe as the Beck arc. In this country it is known as the high intensity arc because of the high intensity of the light source created, particularly when vapors given off by the anode are confined adjacent to the anode crater. This concentration of light is particularly adaptable to searchlights since a maximum of light intensity can be concentrated in the focus of an optical system.

This searchlight work seemed to confirm the Sheer and Korman conclusion that if the critical current density for any particular anode were maintained, the new type of arc could be made to cover the entire anode surface and yet maintain a rapid vaporization of the anode material. This is, that it would be possible to maintain the new phenomenon over the entire anode face.

Because of this conclusion, considerable study was given to this searchlight work. It had many characteristics

in common with the high erosion arc phenomenon, so that the research upon it brought out considerations for further development of the new process. These are:

1. A very high current density above a certain critical value for the electrode employed can be made to maintain the new phenomena over the entire cross-section area of the anode. This current density is comparable to that maintained by Sheer and Korman over a localized area.

2. A rapid ebullition of the anode material takes place, to the extent that the electrical discharge near the anode occurs entirely in the vapor of the anode. This is independent of the nature of the gas in the surrounding medium, in contradistinction to the ordinary arcs where the atmosphere surrounding the arc carries the current of the discharge.

This rapid erosion of the anode was a necessary evil in the high intensity searchlight arc. The highly luminous vapor could not be obtained without it, but the erosion itself was a waste, and added greatly to the cost and trouble of maintenance.

3. The vapor issues forth from the crater as a high velocity jet, so that there appears a separate and distinct flame, called the "anode flame" or "tail flame". This flame or vapor jet is easily distinguished from the

regular cathode discharge flame, called the "negative flame" or "contracted arc stream", which exists in all arcs. The "tail flame" is highly unidirectional, so that the effluent vapors are not dispersed in direction. It was evident that the collection of products is greatly facilitated thereby.

The source of the large kinetic energy of the anode flame of such an arc is considered to arise from the fact that as a result of vaporization, the volume of the anode material expands, as Finkelberg estimates, some 100,000 times, thereby blowing the vapor away from the anode with great force. At the same time the magnetic field of the arc itself gives the flame highly directional effect. The conditions, if fully utilized, would then be ideal for a rapid throughput of material.

In the high intensity arc the rapid vapor stream blows the positive ions, which tend to decrease the anode drop, away from the anode as fast as they formed. This increases the resultant negative space charge and consequently the anode drop. Its effect in this respect is thought to be like that produced at lower current densities by elongating the arc, but to a much higher order of magnitude. The anode drop in the high intensity arc is, therefore, unusually high.

This high anode drop accelerates the electrons to a high velocity. It is by the impact of these electrons upon

the anode crater that electrical energy is transferred to the material of the anode. It therefore follows that the above "mechanism" results in a redistribution of the energy input, so that a high percentage of it goes into erosion of the anode.

Electron collisions may transfer energy in two ways: (1) by elastic impact and (2) by inelastic impact. The former transfers the kinetic energy of the electron to the molecule, raising its temperature, while the latter ionizes it, rendering it less stable chemically.

The inventors of the high intensity searchlight arc encountered difficulty in reducing the erosion and limiting the dispersal of the arc flame to secure an intense light concentration at low cost. This suggested a method whereby the erosion could be made a maximum and the flame could be expanded and utilized in ore reduction.

With such a high erosion arc there could be obtained a high speed jet of highly ionized vapor of the ore. Anode products which would readily mix with the chlorine in an ionized condition would have the maximum opportunity for reaction in this vapor stream. These were believed to be the phenomena that had been obtained by Sheer and Korman in the elongated arc, but on a smaller scale such that they were not fully recognized at once.

It was concluded that the high erosion arc possessed these certain factors in common with the high intensity arc,

1. The effluent vapor is highly charged.
2. The condensate is in the form of an extremely fine smoke of charged particles.

These factors were believed to afford the following advantages:

1. Collection by electrical precipitation is facilitated.
2. It is possible to prevent the fouling of the arc chamber by charging the walls with an electrical potential.
3. Highly comminuted products can be obtained.

The work on the high intensity arc had established that with the common arc the voltage decreases as the current increases, whereas the new arc phenomenon characteristic of both the high intensity and the high erosion arc produces precisely the opposite result. An increase of current therefore, requires an increase of voltage.

It has, moreover, been established by Dr. Finkelburg that the voltage characteristic of an arc is discontinuous at the point where the arc jumps from the common arc phenomena to the high intensity phenomena. The same is true, accordingly of the high erosion phenomena, which in this respect is identical.

This jump in voltage characteristic, therefore, marks the line separating the new arc phenomena from the common arc phenomena. This rise in voltage in the high erosion arc is closely associated with the anode "mechanism" and appearance of the tail flame.

In the high erosion arc, since the ionization and vaporization occur only at the anode face, the anode must be made to embody the ore material in as high a percentage as possible. These refractory ore materials have relatively high resistance even at high temperatures. Notwithstanding this fact, the anode must be so fabricated that it can carry the large currents required to produce the high erosion effects.

With the smallest current intensities just sufficient to produce the high erosion effect, considerable tolerance in the resistivity of the anode is possible, and it was within this range of currents that the original work was done. The importance of this problem increased as the scale of operations increased, and in fact, the extent to which the scale of operations can be increased in a single unit may in part be determined by this fact. It was, therefore, realized that the problem of fabrication of an anode capable of carrying high currents must be solved, as a preliminary step to work on a larger scale. The work done so far had been almost exclusively with an electrode uniform throughout without any carbon shell,

but these conclusions led to the consideration of the need for employing a cored electrode having a carbon shell.

F. Contract Negotiations

At this point further progress required the building of a larger scale plant.

Toward the close of the work at Phelps Dodge Corporation, these experiments came to the attention of two groups, with whom discussions were conducted on continued support for the process on a larger scale. The first group was the New York Office of the Atomic Energy Commission, and the second group was represented by the financial interests of Mr. David Rockefeller. Mr. Belmore and Mr. Roboff, representing the Atomic Energy Commission, visited the Phelps Dodge laboratory in December, 1946, and were informed of the details of the work up to that time. Later, another visit was made, in which preliminary consideration was given to a development contract. A subsequent meeting was held at the New York Office, in which the Commission stated that it was interested in determining if the process could be shown to produce high purity beryllium chloride from beryllium oxide.

The Rockefeller group offered to conduct the tests on a larger scale than heretofore, to accomplish this specific objective. This arrangement was agreeable to the New York Office of the Atomic Energy Commission, who agreed to act without

obligation as observers in this phase of the research.

Accordingly, the inventors entered into an agreement with Mr. David Rockefeller, wherein it was agreed that the research would be conducted in the laboratories of the Laboratory for Electronics, Inc., in Boston. This organization is headed by Mr. Sims McGrath, brother-in-law of Mr. David Rockefeller. Mr. McGrath supervised the work. The actual laboratory work was conducted by a Dr. Lawrence B. Woolaver, employed by the Laboratory for Electronics for this purpose.

The objectives were:

1. To study procedures for the fabrication of BeO anodes.
2. To operate the arc at higher current density, and attempt to study the high erosion arc as a means of conducting this process.
3. To do research on the collection system, in an effort to collect 100% of the products.
4. To produce beryllium chloride having a purity meeting the Atomic Energy Commission's specification, from oxide furnished by the Commission.

After an initial period for construction, this work was begun. The anode problem was attacked along two lines:

1. The fabrication of anodes without a shell in a manner worked out previously by Dr. Korman and Mr. Sheer.

It appeared that anodes made by methods then known could not carry sufficient current to erode the entire anode face, unless so much carbon was included that the production rate in grms. per kw. hr. would be appreciably reduced.

2. The fabrication of anodes having a conductive shell of graphite. In view of the mechanical difficulties and equipment required for fabricating this type of anode, the assistance of the National Carbon Company was obtained for this phase of the work. The percentage of carbon in the cores of these anodes could now be reduced, since the conductivity requirements were fulfilled by the graphite shell. These cored carbons produced high intensity arcs of a satisfactory character.

In the Boston laboratory, however, the limitations of the available power facilities prevented operation at desirable current values, except for a short time. It was found that with the smallest electrodes of this character which the National Carbon Company's equipment could provide, namely 11 mm. in diameter, required about 150 amps to get the full value of the high intensity effect with a satisfactory tail flame. This represented about 50% overload upon the power facilities. Accordingly the apparatus was run at these current values only for short periods of time, generally about five minutes.

In these short runs, however, it was found that the anodes could be fed through the brushes continuously and

that if sufficient precaution was taken to exclude air and moisture, fairly complete chloridization could be achieved. A number of experiments were conducted with currents less than 150 amps. These also showed the presence of the high erosion phenomena.

In order to investigate the high intensity effect more thoroughly and in order to test some larger electrodes (5/8" diameter), Sheer and Korman made a trip to the Cleveland laboratories of the National Carbon Company. The equipment there was capable of supplying more than 300 amps, and although these anodes were run in air, they showed a high erosion effect in stable operation for comparatively long periods of time. The vapor stream and ionization effects were clearly observed; the latter was manifest by the intensely green colorization of the tail flame, which was more than a foot long. This color is characteristic of the beryllium spectrum.

The erosion rates achieved, both in Boston and in Cleveland were only moderate, possibly because of the extremely thick shell of graphite. These shells were fabricated from graphite tubes in stock in order to expedite the preparation of a sample of beryllium chloride. It was the consensus of opinion that a much thinner shell would improve the erosion rate considerably, since the graphite shell was thicker than necessary to produce the conductivity required. Also the extra

carbon of the anode caused a needless waste of power in its vaporization. It was decided at this point to defer erosion studies until such time as greater power facilities might be installed at the Boston laboratory and the collection system and purity problems had been solved.

It was felt that the study of the collection system could be made with currents available at the laboratory even though the high erosion effect might not be so pronounced. Thus a current of about 60 amperes was employed. This was about twice that which had been employed at the Phelps Dodge laboratory. Many types of collection systems were employed, but, as had been found previously, no mechanical system was successful.

It was not until a small Cottrell precipitator was built that collection was possible. From the results obtained it appeared that the efficiency of collection approached 100%, until the capacity of the Cottrell was reached. It was during these tests also that the high charge of the products was demonstrated. From this point on, the Inventors considered the Cottrell to represent the best method of collection.

Some work was done also on selective precipitation, by operating the collecting electrode of the Cottrell at different temperatures. This was done by heating the collecting

electrode by means of an external electric circuit. It was found that an operating temperature could be maintained where beryllium chloride collected, but aluminum chloride and ferric chloride did not collect at all. (These tests were observed by Mr. Roboff of the Atomic Energy Commission).

Using this equipment, samples, made by the arc process from BeO anodes and collected by means of a Cottrell maintained at 150°C, were delivered to the Commission for analysis, without further purification. Since the reports on the analysis showed that the specifications for purity were adequately met, no further attempt for subsequent purification was made. It was found that when using the chloridization process and the electrostatic method of collection, the resultant product contained 20% less contaminant than the beryllium oxide used to fabricate the anodes.

The Rockefeller group then agreed that the process had been demonstrated to be successful on a laboratory scale, but for other reasons declined to proceed on the basis outlined in the preliminary agreement with the Inventors. Counter-proposals were made for the erection of a plant, but these did not meet with the satisfaction of the Inventor Group and were declined.

The Inventor Group was advised by the Commission of their interest in a Government contract for a pilot plant.

The Government further advised that it would greatly facilitate the making of such a contract if one of the participants was a financially responsible engineering firm acceptable to the Commission. For this purpose the firm of Burns and Roe, Inc., was joined with the Inventor Group as a participant in the contract.

PART II - CONTRACT WORK

A. Start of Program

1. Organizing of Work

The Atomic Energy Commission authorized the Contractors to design, construct and operate a pilot plant for the production of beryllium chloride based on the Sheer-Korman Process. An organization was set up by Burns and Roe, Inc., to handle all phases of the proposed project and a library research program was undertaken to gather pertinent fundamental physical and chemical data.

From preliminary information, specifications were prepared for the operational equipment of the process and its necessary power supply. Cost estimates were made, the pilot plant site was accepted, preliminary contacts were made with prospective vendors and measures were taken for maintaining security regulations.

Contract No. AT(30-1)-438 was signed on February 24, 1949 between the United States of America, authorized by the Atomic Energy Commission, and the participant group comprised of the Inventors, Charles Sheer, Samuel Korman, Philip H. Sellew, and Willis B. Rice, and Burns and Roe, Inc. To cover work prior to the formal signing of the contract, the effective date was set as November 9, 1948.

Following the receipt of necessary security clearance for personnel, information and files of the Commission on the previous work of the Inventors were turned over to Burns and Roe, Inc. A program was approved by the Commission for the execution of the work.

During the early planning period, it was realized that certain development work should be undertaken preliminary to the design of the pilot plant. Approval was requested to proceed with the acquisition and installation of the necessary equipment to carry out this work, including the fabrication of anodes. The Commission provided additional funds for this purpose.

It was decided to set up an experimental laboratory to process anodes, and also to determine factors required for the construction of a commercial plant. While the laboratory was being constructed, experimental work was initiated to further investigate the fabrication of anodes by the pellet method which had already been used by the Inventors.

2. Pellet Fabrication

Messrs. Sheer and Korman resumed the fabrication of pellets from mixtures of beryl ore and carbon in a restricted portion of the laboratories of Columbia University. They succeeded in making firm pellets having relatively low resistivity, as described in report BAR-6.

This work was, however, discontinued at the University because of the absence of satisfactory hoods and other

measures to avoid danger from beryllium poisoning. The pelletizing equipment was then transferred to Maspeth and incorporated with the facilities there. After the redirection of the contract some pelleting work was resumed at Columbia.

While the laboratory at Maspeth was under construction, it was important that this anode investigation be continued in some manner that would not imperil the staff with beryllium poisoning. This could be done by using instead of beryl, a substance having similar mechanical properties, namely sand. The use of sand enabled an investigation of the effect of such variables as particle size, baking temperature and kerosene binder on the mechanical properties and resistivity of finished anodes. Work was expedited by elimination of the necessity for protective hoods and safety measures against toxic contamination.

3. Laboratory Set-up at Site

The design and construction of this laboratory was started in July of 1949 to provide means for processing the raw materials, manufacturing and baking anodes, sampling and analyzing of materials in process, testing and burning of finished anodes in an arc. Equipment was selected, purchased and installed for the crushing, grinding, classifying of ores and carbonaceous materials, and the mixing, extruding and baking of anodes.

The continuous extrusion method of anode fabrication was selected as more economical than the intermittent or batch method of fabrication. In this connection, the extrusion equipment of various manufacturers was investigated. Finally, after successful green extrusions had been made using mixtures of bauxite or sand and lamp black, with kerosene as an extrusion agent, a double worm extruder was selected and purchased for installation at the Maspeth laboratory.

For trial burning of anodes, a commercial type carbon arc unit was selected and purchased. Large ventilated hoods and a complete dust collection system were installed in the anode laboratory to enclose all equipment for control of toxic dust and fumes. A complete analytical laboratory was installed for sampling and for analysis and control of the process work.

Conferences were held with the Commission's Health Department for the institution of health procedures, checking of the operation of the ventilating system and medical examinations for personnel. Shower facilities and mechanical laundering equipment were installed and workers were subjected to a strict shower routine with a complete change of clothing daily, while working with toxic materials. A weekly check was made on weight, height, lung capacity, and

general fitness of all laboratory workers, and health records were maintained.

B. Design of the Pilot Plant

1. Design Considerations

An examination of the basic data and results of preliminary experiments performed by the Inventors showed that the problems of applying the Sheer-Korman Process as developed on a research scale to that of a commercial pilot plant were complicated because of the need for more specific information. Knowledge was required concerning process thermodynamics and kinetics; the composition and properties of gases and products and their corrosive effects on materials of construction; the type of enclosure or chamber to house the arc; and methods of condensation and collection of products.

Some of these problems were answered, at least in part, by theoretical calculations based on the incomplete stoichiometric and thermal data available in the literature. To answer the remaining problems, subcontractors with special knowledge in their fields were selected to work with the Contractor Group on particular components of the pilot plant. In addition, various units of the process were designed and set up on a small scale in the laboratory at the Maspeth site to provide data from actual experiments for use in design of the larger units by the engineers of Burns and Roe, Inc., and their subcontractors.

Work was undertaken at the National Carbon Company, Cleveland, Ohio, and consultations were held with Dr. Finkelburg, a German scientist noted for his high intensity arc searchlight work, to develop data on arc operation and flame characteristics.

To expedite the work of the subcontractors, a specification was prepared during the early development of the project from descriptions of the process, calculations and data furnished by the Inventors. It was understood that changes in the requirements and design would be necessary eventually in light of information yet to be obtained, and that the specification would be revised to include the additional information and data available before the start of construction of equipment by the subcontractors.

Discussions were held with the Commission on the sizing of the pilot plant required to demonstrate the commercial feasibility of the process. It was decided to construct the pilot plant with sufficient flexibility to handle beryl and lower grade ores. It was realized that it would be necessary to extrapolate the experimental data at least ten-fold and possibly more.

In order to supply the subcontractors with additional design data, calculations were made using a representative analysis of beryl ore and published data on vapor

pressures for the various chlorides involved. These calculations, reported in BAR-10, were based on an assumed "mechanism" of separation as defined by the Inventors, i. e. fractional condensation from a perfect gas with no formation of solid or liquid solutions, eutectics or azeotropes.

Also, from available thermodynamic data for the various reactions, it was possible to calculate the enthalpy of the products as a function of temperature, and so permit the design of a heat recovery and gas cooling system for the arc chamber.

2. Subcontractor Activities

a. Combustion Engineering-Superheater, Inc.

Immediately after the signing of the subcontract with Combustion Engineering-Superheater, Inc., a sub-subcontract was let by Combustion Engineering-Superheater, Inc. with General Precision Laboratory, Inc. It was agreed by all parties and the Commission that General Precision Laboratory, Inc. would handle the arc mechanism and its related items and Combustion Engineering-Superheater, Inc. would handle the remainder of the subcontract.

After the formal contract signing and upon receipt of security clearance for personnel, Combustion Engineering-Superheater, Inc. and General Precision Laboratory, Inc. were made familiar with the process and given

all of the information available to Burns and Roe, Inc., at that time. The subcontractors were also given copies of Specification ABR-10 to be used as the starting point for all design and re-search problems.

A meeting was held in the offices of Burns and Roe, Inc. with the subcontractors to review this specification and other data, and to discuss all questions and suggestions regarding the design of the mechanism. The meeting was attended by representatives of the Inventor Group, Combustion Engineering-Superheater, Inc., General Precision Laboratory, Inc. and Burns and Roe, Inc. The following problems were discussed at the meeting:

1. Combustion of product gas produced in the arc chamber.
2. Temperature and chemical changes expected during movement of gas through chamber.
3. Corrosion characteristics of product gas at various temperatures.
4. Materials available for use in contact with product gas at various temperatures.
5. Thermo-physical properties of the product gas at the temperatures existing in the reaction chamber, such as conductivity, viscosity, specific heat, density, degree of dissociation, non-luminous and luminous radiation properties.
6. Size, shape, and temperature distribution of arc flame.

7. Anode fabrication, feed method, characteristics and erosion rates.
8. Chlorine requirements

Some of the above questions were answered by the Inventor Group based on the experience they had with their test models. The remainder of the problems had to be answered tentatively to the satisfaction and best of knowledge of all those present.

Subsequently, conferences were held to discuss these problems again, so that by October 26, 1949, General Precision Laboratory, Inc. was able to submit their Sketch No. 1 - RM-172. This sketch consisted of the arc mechanism as envisioned by General Precision Laboratory based upon Specification ABR-10. Combustion Engineering-Superheater, Inc. then superimposed on this sketch a design of a furnace enclosure to contain the arc unit.

At this point, a number of physical design problems became apparent. They were as follows:

1. How to provide a fluid-cooled metal enclosure while at the same time preventing direct impingement of flame on the walls.
2. How to provide openings for the electrode holders while at the same time providing for a wide angle of adjustment of trim angle and sealing of the openings.
3. How to seal the electrode openings without making electrical contact with the electrode holders.

4. How to cool the seals required at the openings for the electrodes.
5. How to provide an access opening or "manhole".
6. How to employ relatively cool fluid for cooling without condensing corrosive products on the metal.
7. How to provide a lining material that would insure a high temperature surface in contact with the furnace gas (to prevent condensation) while at the same time transmitting sufficient heat so as not to reach a temperature that would result in rapid lining deterioration.
8. How to insure against inward-leakage of air and moisture, and outward-leakage of product gas, including the problem of maintaining anhydrous conditions during start-up and down periods.
9. How to cool the compartment containing the negative electrode adjusting mechanism.
10. How to estimate the amount of cooling surface required at a given assumed temperature.
11. How to design a cooled lining to operate at the assumed temperature.
12. How to control anode feed and positioning for the varying conditions during start-up, normal operation and restriking of arc.

Problems 1, 2, 3, 4, 5, 8, 9 and 12 were left for solution by Combustion Engineering-Superheater, Inc. and General Precision Laboratory, Inc. However, to aid the subcontractors in their work, it was decided that the other

problems which related to thermal and radiation characteristics of the products should be discussed with an outside consultant capable of suggesting solutions or procedures for solving these problems.

One of the most outstanding authorities on this type of analysis is Professor H. C. Hottel of the Massachusetts Institute of Technology, Cambridge, Massachusetts. Therefore, it was decided to retain his services for this purpose.

On December 20, 1949, representatives of Combustion Engineering-Superheater, Inc. and Burns and Roe, Inc., held a conference with Professor Hottel at the Massachusetts Institute of Technology to discuss thermal and radiation problems. Professor Hottel suggested that the best procedure would be to obtain some data on a high intensity carbon arc and then find a way to correlate this with the process arc. He suggested the use of a disk type thermocouple which he had developed for previous experiments of his own, and later sent a sample of this thermocouple to Burns and Roe, Inc.

Combustion Engineering-Superheater, Inc. made several thermocouples of varying size similar to the sample furnished by Professor Hottel. These thermocouples were taken to the National Carbon Company laboratory at Cleveland, Ohio where a high intensity carbon arc apparatus

was available. Experiments were conducted by use of the disk thermocouples to determine the radiation characteristics of the arc crater and its tail flame.

On February 17, 1950, representatives of Combustion Engineering-Superheater, Inc., General Precision Laboratory, Inc. and Burns and Roe, Inc., again held a conference with Professor Hottel at the Massachusetts Institute of Technology. At this meeting, the results of the radiation test at Cleveland were shown to Professor Hottel and an additional point on the H-T curve shown in BAR-10 was added to help extrapolate in a high temperature region.

It was then agreed that Professor Hottel and Combustion Engineering-Superheater, Inc. would calculate independently, the transfer rates and heat resistances necessary to maintain the proper wall temperature in the chamber and to absorb the required amount of heat energy. As a means of correlating these results with the radiation characteristics of a process arc, Professor Hottel recommended that similar measurements be made on the arc located at the Maspeth laboratory. This laboratory project was placed on the agenda for the immediate future.

In the meantime, the research and development work being performed in the Maspeth laboratory showed that the manufacture of continuous electrodes would require a long,

intensive study of this problem alone. However, experimental electrodes two feet long were manufactured in the laboratory. In order to accelerate the project it was decided to design the arc unit and its surrounding chamber to use these electrodes.

The change to two foot electrodes introduced the following additional design problems for Combustion Engineering-Superheater, Inc. and General Precision Laboratory, Inc.:

1. How to provide for removal of unburned electrode stubs at the rate of up to 5 per minute, with stubs several inches long.
2. How to coordinate flow of chlorine and regulation of furnace pressure when interruptions to arc occur so frequently.
3. How to provide a suitable sealed opening for a third electrode striking mechanism which would now be required.
4. How to purge the system to permit frequent renewal of the striker electrode

In order to prevent the electrode stubs from clogging the arc area, Combustion Engineering-Superheater, Inc. and General Precision Laboratory, Inc. decided to invert the entire unit, thereby permitting the stubs to fall clear and into a hopper at the bottom of the chamber. The water wall surface of the chamber was also redesigned so that the chamber could be dismantled easily. The design was such that by opening only a few joints, the arc unit and top portion of the

chamber could be lifted off the main body of the chamber.

General Precision Laboratory, by means of experiments in their own laboratory, determined that the effect of inverting the arc on its performance was practically negligible.

In anticipation of the materials required for the final design by Combustion Engineering-Superheater, Inc. and General Precision Laboratory, Inc., a corrosion test program was initiated at Combustion Engineering-Superheater's shops in Chattanooga, Tennessee. These tests were intended to determine the best materials of construction for all portions of the arc and its enclosing chamber.

From the early part of the active subcontract work, General Precision Laboratory, Inc. conducted small scale corrosion tests on a limited number of materials which they hoped would be useful in the construction of the arc unit. These tests gave results which only indicated materials to be used and it was expected that Combustion Engineering-Superheater's test would prove which materials were best for the pilot plant unit.

When it became necessary to freeze the design of the arc chamber and the arc mechanism to permit the subcontractors to finalize their design to meet project schedules, the Contractor Group decided to attain maximum flexibility by

basing the design on an erosion rate considerably below that originally specified by the Inventors. Therefore, in order to keep the quantity of products up to a reasonable flow for measurement and collection, the arc chamber design was modified to include three arcs instead of one. This created several additional problems for the subcontractors but they proceeded to change the design to suit the new conditions.

On March 3, 1950, the United States Atomic Energy Commission forwarded a letter to the Contractors redirecting the work under their contract. Therefore, on March 7, 1950, Burns and Roe, Inc. sent letters to their subcontractors discontinuing the work involved. The subcontractors, however, were retained on a consultant capacity under the terms of their subcontract relating to preliminary design work.

At the time of the discontinuation of the contract, Combustion Engineering-Superheater, Inc. and General Precision Laboratory, Inc. were about to begin the final design for the pilot plant unit, although several problems still existed and a number of calculated risks would be necessary. The necessity for this decision was based upon the relatively close completion date and the anxiety of the Commission for quick results. However, the contract redirection stopped the work at this point.

General Precision Laboratory, Inc. had a "breadboard" layout of the arc feeding device and had worked out a positive position control for the electrode. They were in the process of designing the three electrode unit and the striker linkage for the new layout.

Combustion Engineering-Superheater, Inc. was beginning the corrosion tests in Chattanooga, was designing the arc chamber for the three arc system, and had made a thorough study of chlorine control systems. They had devised a scheme for purging, sealing and cooling the electrode cylinders and had decided upon the standard auxiliary equipment which they would furnish with the chamber. This auxiliary equipment is standard with Combustion Engineering-Superheater's small forced circulation package boiler.

In view of the above progress, Burns and Roe, Inc., Combustion Engineering-Superheater, Inc. and General Precision Laboratory, Inc. were in the process of writing a new tentative specification for electro-chemical arc equipment when subcontract work was halted.

b. Research Corporation

The facilities of Research Corporation at Bound Brook, New Jersey were investigated in connection with the design and fabrication of certain components of the product collection system, including condensers and electrostatic

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precipitators. Negotiations began in June, 1949 and a contract was signed in October, 1949.

The basic problem involved the separate condensation of five groups of chlorides present as vapors in a gaseous mixture from the reactor, their separation from the gas stream and subsequent removal from the system in the anhydrous state. Specifications required that the operation be carried out in a manner so as to recover the beryllium chloride in a high state of purity and the aluminum chloride and silicon tetrachloride fractions in a state of purity that would permit commercial disposal.

Various exploratory studies and investigations were made in connection with the pilot plant design and in December 1949 Burns and Roe, Inc. requested a quotation on a laboratory size precipitator to be used with the small scale pilot plant set-up, giving Research Corporation the required data. A unit was subsequently purchased and installed at Maspeth.

In February, 1950, at the time of redirection of the contract and suspension of subcontractors activities, Research Corporation was engaged in a study of the general problem but work on equipment for the large pilot plant had not advanced beyond the exploratory stage. No drawings had been made or formal reports submitted on the progress of the work.

PART II - CONTRACT WORK

C. Development Work

The general plan of development work was adopted in consultation with the Inventor Group, and received their approval before being put into effect. This general plan was then reduced to specific procedures (see Appendix I). The work carried out under these procedures was reviewed by members of the Inventor Group as it progressed. Later, after the redirection of the contract by the Commission, concentrating the contract scope upon the research work, a research director was appointed. Thereafter, the work was carried out directly under his supervision with the guidance of the Inventors.

1. Anode Program

a. General Considerations

For this process it had been decided to form extruded anodes, as they were deemed to be more suitable for continuous use. Extrusion tests were made at the plants of commercial companies having wide extrusion experience, until the construction of the laboratory had sufficiently advanced to enable the carrying out of a complete program of processing ores and carbon, extruding, baking and trial burning of anodes.

Work on anode fabrication was based upon recommendations resulting from the work of the Inventors at Columbia University, as defined in report BAR-6. Some of the factors, however, could still be determined more simply by the

pellet method than by the extrusion process, so that both methods were used at the Maspeth laboratory, in the study of anode fabrication. A search was made for a suitable carbon to match the "Coke No. 1" (from an undetermined source) used together with beryl ore and kerosene by the Inventors in fabrication of successful pellets in their earlier work.

The problems encountered included not only the mechanical fabricating of rods of given dimensions and composition but of producing them with characteristics to permit satisfactory baking and successful burning in an arc. Variables considered were particle size of ore and carbon, carbon source, aqueous and non-aqueous extrusion agents, curing the extruded rods, baking cycle, mixing technique, and moulds for baking.

A tabulation of the factors involved included the following:

1. Acquisition of carbon materials and a study of their use as carbon sources for anodes.
2. Acquisition of various beryl ores and a study of their effect on the anodes produced.
3. Determination of satisfactory (type and composition of) extrusion agent to be used with beryl ore and carbon mixes.
4. Effect of (variables of) particle size distribution of the beryl ore and carbon used.
5. Determination of the effect of baking time and temperature on the properties of the anode.

6. Effect of the variation of carbon composition on the anodes produced.
7. Effect of baking in structurally tight porous moulds and in loose graphite tubes.

b. Extrusion

After proper technique and suitable mixtures were developed, beryl ore rods were successfully fabricated by the extrusion process, which could then be baked to form experimental anodes. Important factors in the extrusion process were:

1. Operation

The mechanical operation of the particular type of extruder proved difficult to control with the hard abrasive ores and carbons used. To avoid binding and damage to expensive worms, liners and dies, it was necessary to start at low speed and feed the mixture slowly until extrusion commenced, and once extrusion started, to control its rate within torque limitations of the equipment.

Low viscosity extrusion agents such as kerosene and water tended to "bleed", producing a small piece of very wet rod followed by binding and ultimate stopping of extrusion due to the dry hard nature of the mixture.

When cold extrusion was desired, cooling water was essential, otherwise heating occurred which produced rods with blistered surfaces.

To permit hot extrusions, oil heaters a circulating pump and controls for the extruder jackets were installed to enable extrusion of materials at temperatures up to 450°F. To start hot extrusions, a dry mix with an agent solid at room remperature but fluid at higher temperature, was fed into the extruder and allowed to warm up, and then was extruded slowly. It was essential that the die be heated as well as the extruder itself to prevent binding and damage.

For cold extrusions one or two repasses of material through the extruder frequently helped in producing a denser and more compact rod with a smoother surface. In this respect, the results were similar to tests later carried out on a deaerating type extruder at the Chattanooga plant of the American Lava Corporation, under the supervision of Mr. Sheer.

A flat type die would not work with the materials used, but dies with long or short tapers were equally satisfactory. A specially designed main extruder worm with tapered flights extending into the die cone offered no advantage over a normal type worm.

ii. Mixes

Work was carried out with a series of mixtures of ore and carbon with a variety of extrusion agents. Experimentation with various cokes and coals soon proved bituminous coal to be the most suitable carbon source for anodes.

Ore to coal ratios in mixtures ranged from 5;1 to 2-1/2:1 and in general extrusions made with the higher coal content were better. They presented greater difficulty in baking, but resulted in stronger anodes with greater electrical conductivity after successful baking technique was developed.

The addition of some graphite to extrusion mixtures as a partial substitute for bituminous coal was not found to be beneficial.

Hot extrusions were attempted with a mixture of pitch and beryl ore using pitch as a carbon source and at the same time taking advantage of its plastic qualities. Coal tar pitch with a melting point of 190°F was used for these extrusions. Difficulty was encountered since very careful control was required of die and oil temperatures and frequently the operation caused excessive torque and eventual damage to the extruder worm. Rods produced were short, soft and rough surfaced.

Extrusion results emphasized the importance of the lubricating properties, bonding and viscosity of extrusion agents. Substances such as fuel oil, kerosene and water alone do not possess sufficient bonding qualities seemingly necessary to produce good extrusions. Pitch, motor oil, molasses, other oils, and water with bentonite possess desirable properties of bonding and lubrication

so far as extrusions are concerned.

The time of mixing depended upon the extrusion agent used. Investigations of the highly viscous oils indicated that long periods of mixing were essential prior to extrusion. In many instances, a two hour mixing period was required and in extreme cases, the mix had to be allowed to stand overnight.

These results indicated a critical composition range for the various extrusion agents and mixes. Below the critical range, the mix was too dry for extrusion and above the range, rods were produced that were too wet to retain their extruded shape. Most of the mixes, in particular: peanut oil, molasses, sugar solutions, and water with bentonite added tended to improve their hardness when exposed to the air, or if heated to a temperature between 120° and 150°C for a number of hours.

The most successful rods produced during the anode program were extruded from a preparation of molasses, beryl ore and coal. The ore and coal were tumbled in a ball mill for twenty-four hours in order to obtain an intimate mixture of the solids. Very little reduction in particle size occurred since only a few balls were used in the mill. This mix was then combined with the molasses in a mixer for about fifteen minutes prior to extrusion. The preparation of the mix was as follows:

40 ounces of beryl ore (-60 mesh),
slagging temperature 2550°F.

10 ounces of bituminous coal (-325
mesh) from East Gulf, West Virginia
(16% V.C.M.)

275 ml. of molasses prepared by
mixing 5 parts of Brer Rabbit
molasses with 1 part water.

111. Addition Agents

Investigation proved that in order to obtain extrusions of anodes composed of beryl ore and carbon, it was advisable to find a suitable addition agent. Kerosene was specified as first choice by the Inventors because of their previous experience in pellet anode fabrication, but did not prove satisfactory for extrusion.

A number of extrusion agents were used with varying degrees of success. In this search, a ratio of 5:1 ore to bituminous coal mix, was maintained and various agents were investigated with the results outlined below.

Kerosene

Since previous pellet fabrication by the Inventors had produced excellent results with kerosene, a great deal of time was expended in attempts to obtain successful extrusions with this agent. Kerosene was used up to 14.7%, wet basis. When the mix contained smaller amounts of kerosene, no rods could be extruded due to a solid block in the

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die cone. When the mix was high in kerosene, a very wet extrusion of very short duration occurred, indicating that the ore did not retain the agent sufficiently to allow continuous flow. No composition was found that provided proper binding and lubrication.

Motor Oil SAE #10

Mix content ranged from 9% to 17.3%, wet basis. Extrusions of smooth, shiny rods, 6" in length were produced.

Fuel Oil #3

Mix content ranged from 4% to 12.2%, wet basis. Results were unsuccessful since fuel oil produced too dry or too wet extrusions as in the case of kerosene.

Motor Oil SAE #30

Mix content ranged from 7% to 18.4%, wet basis. No extrusion could be obtained below a 15% mixture. Above this point, a soft, dull, smooth rod was produced. At 15%, a hard cone was left in the die and the worms failed to clean themselves even when running without the die head.

Manila Coconut Oil

Mix content ranged from 5.9% to 15.6%, wet basis. Below 14%, material was too dry and caused binding in the machine. At about 15.5%, continuous, rapid extrusions of soft, dull rods were obtained.

Peanut Oil - Refined

Mix content ranged from 4% to 16.4%, wet basis. Below 14.9%, short, smooth rods were produced similar to SAE #10 rods. However, at 16.4%, the rods produced were smooth surfaced and comparatively

hard. Repassing some of the rods through the extruder aided in producing smoother surfaced rods.

Carnauba Wax

The wax was mixed with bituminous coal as a carbon source and ranged from 50% to 100% of the total carbon content. It in turn was mixed with ore in the respective proportions of 1:5. The critical percentage of extrusion with carnauba was 60% to 65%. Above this percentage, the rods produced were too soft for collection and below this amount, the mix was too dry for extrusion. This required hot extrusion at 270°F exit oil temperature. However, the range of possible extrusion proved too narrow and difficult to produce good rods.

Molasses

Mix content was 21.1%, wet basis. Rods produced from molasses were smooth surfaced, dull in appearance and mechanically strong. These rods were the most successful obtained during the anode program.

Sugar Solution

44.4% sugar solution mixed in 25% wet basis proportion with ore and carbon produced extruded rods that were too soft and not collectable. However, mixes of 50% sugar solution mixed to 11% wet basis proportion, produced smooth surfaced rods of fair mechanical strength.

Pitch

Previous attempts to utilize pitch as a combination carbon source and agent had proved unsuccessful. Combining the pitch with bituminous coal, lamp black or

graphite, maintaining ore to carbon ratios of 5:1, was also found to be unsuccessful, even though extruded jacket temperatures were raised to 400°F to induce the pitch to flow and lubricate the extrusion.

Water and Bentonite

Water, when used alone as an agent, behaved similarly to kerosene, failing to bind the mix. However, when 3% to 4% of the mix was made up with bentonite, good extrusions were obtained, second only to those obtained using molasses as an agent.

Activating the bentonite-water mixture prior to use, made no improvement in the successful anodes produced at Maspeth. In tests performed at the American Lava Company, Chattanooga, Tenn., extrusions with activated bentonite resulted in excellent green rods. These rods could not be baked into satisfactory anodes, but this was attributed mainly to the more refractory nature of the ore used.

c. Baking of Extruded Rods

Tests were carried out with the extruded rods to determine the effect of baking conditions upon their properties. The properties were determined by resistivity measurements, physical appearance, tests for hardness, and analysis for moisture, carbon and volatile combustible material (VCM). Extruded rods were placed in graphite tubes sealed with graphite plugs and baked in an electric furnace which had been brought up to a predetermined temperature and purged with nitrogen.

The performance of the electric furnace to be used was tested by measuring anode temperatures at various points and determining the time for the baking tube to heat up. With the furnace set at 2350°F, the temperatures of a full charge of nine rods baked simultaneously did not vary by more than 75°F for correspondingly similar positions traversing the furnace, but did vary as much as 250°F between the front and center of a two foot rod.

Baking periods lasted about six to seven minutes until the maximum temperature of 2350°F was attained. Volatile material was evolved very rapidly, bursting into flame upon contact with the air. The use of a nitrogen "Blanket" was found to reduce this to some extent. Rapid evolution of volatile material frequently caused deterioration of the anodes. However, anodes were produced which were mechanically satisfactory for burning in the commercial arc unit with some degree of success. These were obtained from rods extruded with agents such as peanut oil, coconut oil, motor oil, carnauba wax, sugar solutions and molasses.

The better anodes were produced from mixes with molasses, sugar solutions, and water with bentonite as addition agents. Curing at 120° to 150°C for a few hours, preliminary to baking, was found to give smooth surfaced anodes of very good strength in the case of the molasses and sugar

solutions mixes. Elimination of the curing step produced anodes with similarly good strength, but with some surface cracking. Baking of the rods extruded with water and bentonite, without preliminary curing, produced anodes of good strength and smooth surface appearance.

The best anodes were those extruded with molasses, then cured preliminary to baking, followed by baking in split moulds made of porous graphite. This resulted in straight uniform rods much improved in physical properties. The half shell moulds served to maintain a straight contour during the baking operation, thus eliminating the tendency to distort which was encountered when baking rods in loose graphite tubes.

d. Trial Pelleting

Pelleting of anodes was resumed both at Columbia University and at Maspeth. Mixtures of kerosene, beryl ore and bituminous coal were used, as well as kerosene, beryl ore, and samples of "Coke #1" in an attempt to determine the best composition as well as the best mixing and baking technique for later adaptation to extruded anodes. Up to this time, although usable extruded rods had been produced which gave fairly good erosion rates, nevertheless they did not match in an overall sense mechanical and physical properties of the relatively small pellet samples produced by the Inventors in the early pelleting work.

At the same time, extrusion work continued along lines parallel to the pelleting, making use of lessons learned in variations of ores, carbon source, mixing technique, baking time and temperature.

A major intent during this phase of the program, was to reproduce, from available materials, pellets similar in characteristic to those fabricated by the Inventors both prior to the inception of the contract, and during the early pelleting work at Columbia in the spring of 1949. All this early work was accomplished with the carbon source designated as "Coke #1" of which only sample quantities then remained.

The following results are based on the resistivity, mechanical strength and hardness of the pellets. It was assumed that for a given ore to carbon ratio the lower the resistivity, the better the pellet.

i Carbon Sources

In studying this variable, nine different petroleum cokes, one gas plant coke, coal tar pitch and a number of bituminous coals from West Virginia and Pennsylvania were tested.

From experience gained, it appeared that several grades of bituminous coal with analyses approaching that of the original "Coke #1", produced pellets of the same

general characteristics. Another important variable to be considered, however, was the particle size and slagging temperature of the beryl ores used.

ii. Ore Sources

An investigation was made of the slagging temperatures of the several beryl ores available and the results showed that this temperature varied from 2350°F to some temperature above 2600°F. The sintering temperature of these ores is approximately 250°F below the slagging value and the best bond characteristics for anode pellets is believed to require a temperature somewhere between the sintering and slagging points.

iii. Mixing

It was found that mixing was extremely important in obtaining proper pellets. Difficulties in pelleting some of the rougher mixes were reduced by tumbling the dry mixtures of carbon and ore in a ball mill, with a few glass balls added, for four to eight hours prior to addition of the agent. This method seemed to provide a more intimate and homogeneous mix and improve the pellets.

The resistivity of pellets made with normal mixing methods averaged 0.58 ohm-cms and those made from tumbled mixes 0.23 ohm-cms. Ore of particle size distribution from -60 mesh to +325 mesh produced better pellets than those of smaller size distribution.

Calcining the ore also improved the pellet properties. The pressure required to produce mechanically sound green pellets was found to be higher as the particle size increased.

iv. Baking

The use of relatively high baking temperatures of 2300° to 2700°F produced better pellets than if the same ore and carbon source were baked at low temperatures. Improvement above 2300°F, however, was probably not sufficient to warrant higher temperatures. Baking temperatures were also found to be dependent upon the BeO composition of the beryl ore. The higher the BeO composition, the higher the baking temperature required. When heated rapidly to elevated temperatures, the 16% to 21% VCM bituminous coal pyrolyzed with release of volatile matter with somewhat less violence than observed with higher VCM carbon sources.

The result strived for in this baking process was the formation of a carbon matrix to provide both electrical conductivity and mechanical strength. In general, the more rapid the heating (by use of higher baking temperatures), the stronger and more conductive the matrix.

e. Shelled Anodes

Problems encountered in the fabrication of full core anodes of sufficient strength and uniformity to

be tested and utilized in the commercial arc unit led to the investigation of anodes with outer shells of carbon or graphite and with cores of beryl ore and carbon mixtures.

Anodes were constructed along the lines of commercial anodes, and lent themselves readily to operation in the arc apparatus. This method of fabrication eliminated the mechanical weakness, rough surface and distorted shape inherent in the full core anodes fabricated during the initial stage of the development. At least two basic methods for producing shelled anodes were investigated, one using extruded rods and the other using tamped cores.

The first method employed the usual baking procedure. Extruded rods containing pitch, fused readily into graphite tubes during baking, even during short baking periods of thirty seconds duration at a temperature of 2300°F. Rods extruded with peanut oil, coconut oil and motor oil were also found to seize into the graphite tubes during baking.

In the second method, mixtures of pitch and ore were tamped into a graphite tube and given a short baking. Mixtures of bituminous coal and ore were also used but with a longer baking cycle.

2. Arc Burning

a. Equipment

In order to provide information for the ultimate design of a pilot plant, it was considered advisable to procure a commercial carbon arc unit, normally used for motion picture projection. The equipment was rated for arc current of 300 amperes with a good possibility of modification for currents up to 600 amperes. This unit could be readily adapted for extruded anodes, and afforded the advantage of automatic feed and rotation of the anode, plus striking and control of the arc through an adjustable cathode arm.

A metal chamber and ejector were designed and fabricated to enclose the arc and draw off for disposal the products of arc erosion, to permit trial burning and testing of extruded and baked anodes in air.

Initial arc operation was performed in air to study arc characteristics with the various compositions of anodes used. Subsequently, in connection with process demonstration work, a second similar commercial type unit without housing or supports was obtained to be built into the equipment designed for operation in a chlorine atmosphere. Modifications were also made to enable use of currents between 300 and 600 amperes, although need for these higher values never materialized during the contract period.

b. Trim Angle

Investigators of carbon arc operation for lighting purposes such as Finkelburg and Jones had established empirically an optimum angle of 127° between anode and cathode. This design was adhered to in the construction of the arc chamber.

Experimental work with the arc unit at the Maspeth laboratory demonstrated that better and more stable arc operation could be obtained with the cathode facing the anode, its centerline at the bottom edge of the anode. For the same resistor bank tap setting, operation under these conditions produced a higher current and a lower voltage across the arc than was obtained for the 127° angle. The operating temperature of the air arc chamber was also found to be lower with the electrodes facing each other.

c. Magnetic Stabilization

When an arc is operated with the cathode set at an angle to the anode, a magnetic field is required to aid in stabilizing the arc.

The use of metal chambers to contain the arc complicated the magnetic flux pattern. Experimentation was necessary with the several chambers used to determine the proper orientation of magnets to achieve stabilization of the arc, when anode and cathode were set at an angle. Operation of

the arc with the two electrodes opposed and in line was also investigated at the Maspeth laboratory. A stable arc was maintained, even though enclosed in a metal chamber, without the use of a compensating magnetic field.

d. Anode Contacts

The commercial type of arc unit employs a rotating chuck which feeds the anode through tight vise-like brushes with semi-circular contact surfaces. In general, this type of contact subjects anodes to physical stress that proved unsatisfactory for full core anodes which were mechanically weak, rough surfaced or distorted.

Near the close of the contract period, anodes extruded with molasses and baked in half shell porous moulds showed promise of meeting physical requirements for use with these standard type contacts.

It was found impractical to use pellets with the commercial arc contact because of their short length. Moreover attempts to cement pellets together to form a longer anode were only partially satisfactory since strong bonding with conductive material and proper alignment was difficult. No actual burning experience was obtained with cemented pellet anodes.

To minimize the power loss in the anode, graphite contacts were designed to reduce the protrusion of the

anode beyond the brush contact. In a commercial unit, this is normally accomplished by the use of water cooled silver contacts, but this was impractical for use with chlorine since it would have required the enclosure of such contacts in the arc chamber. In this position they would be exposed to the atmosphere of chlorine and product chlorides, which are corrosive under moist conditions, and at sufficiently high temperatures in a dry state.

For the higher intensity arcs, it was found desirable to use less expensive graphite contacts located inside the arc chamber with a protrusion of the anode of approximately one centimeter. With this set-up, the tendency was reduced for the protruded section of the anode to melt rather than to erode at its tip. Experience was also gained in water cooling of arc contacts and for this purpose special silver, bronze, and copper contacts were fabricated.

In a continued effort to reduce the stresses imposed upon the anodes by contact pressure, special "floating" contacts were constructed consisting of a fixed base and an upper portion which rested on the anode by gravity. The anode was fed between the two grooved sections which adjusted themselves to imperfections and curvature of the rod. This type of contact enabled the use of mechanically weak rods although it did not reduce arcing at the contact because of the rough

anode surface and variation in the rod diameter.

Near the end of the contract period, a packed graphite contact was developed consisting of a graphite box packed with loose graphite under pressure through which the anode was fed. An adjustable cap at the top of the box permitted variation of contact pressure. This contact was tested in air with the commercial arc unit and proved very satisfactory since it afforded good surface contact to anodes, including those of poorer surface characteristics. Arcing at the contacts was eliminated and once used, the unburned portion of one anode could be removed and replaced by a new anode without disturbing the graphite packing forming the brush contact.

In general no apparent differences were found between brushes made of bronze, copper, silver, graphite or carbon. Metallic brushes are adaptable for water cooling but would present a problem if used inside a chamber containing chlorine gas or chlorides.

In connection with a transite arc chamber, as used with the research pilot plant in the closing month of the contract, experiments were carried out using various compositions of carbon and graphite brush materials for the contacts. The object was to devise a more abrasive brush contact which would tend to abrade the rough surface of the anode, thereby improving contact and eliminating brush arcing. This work was not completed although results obtained were promising.

e. Full Core Anodes

Of the anodes produced by extrusion, both bentonite and molasses rods were found to be reasonably satisfactory for experimental purposes. In the beginning the arc burning was conducted in air rather than in chlorine. Difficulties encountered were arcing at the brushes, and slagging and drooping of that portion of the anode projecting beyond the end of the brush contacts. The overhang in the commercial arc burning apparatus was 2-1/2 inches with low current contacts and 1/2 inch with higher current water cooled contacts. Operational experience with available full core anodes in the air arc chamber with a protrusion of approximately 1/2 inch proved that slagging and drooping of the anode, although somewhat reduced by a shorter projection, still prevented continuous arc burning in air.

Improvement was obtained when anodes were hand fed into an arc in chlorine, using arc brush design based on previous Inventor experience, rather than normal commercial contacts. The problem of arcing at the brush contacts was mitigated sufficiently to permit erosion of several lengths of anodes for periods up to three minutes, eroding approximately one foot lengths in a run without slagging or drooping. This operation, with selected anodes of suitable strength and resistivity, was greatly facilitated by application of a coating of Aquadag to the anode surfaces prior to use.

f. Shelled Anodes

Shelled anodes resembled commercial carbon electrodes and burned satisfactorily in the arc unit. Difficulties encountered due to a tendency of the core to separate from the shell during burning were overcome by adjustment of the core composition. The most favorable composition was found to be five parts of ore to one part of bituminous coal. This ratio falls within the stoichiometric range for the reaction of carbon with the oxygen in the ore to form CO or CO₂.

It was necessary to machine the graphite shells to diameters of either 13.6 mm or 16 mm in order to permit use with standard commercial contacts. The results of burning these shelled anodes were as follows:

16 mm Anodes

Shelled anodes containing cores extruded with peanut oil, coconut oil and motor oil burned satisfactorily. Experimental anodes fabricated by tamping a pure beryl ore into the 16 mm shells did not burn satisfactorily.

13.6 mm Anodes

A mixture of beryl ore and pitch in a ratio of 5:1 was tamped into 13.6 mm shells and the resultant rod flash baked at 2350°F. A second group of rods was fabricated in a similar manner from a 5:1 mixture of beryl ore and bituminous coal, baked at 2300°F. Both groups of anodes of these compositions burned satisfactorily.

Satisfactory results were obtained using anodes with separate feed of shell and core. The shell served to

maintain a stable arc and permitted vaporization of the core. For this purpose, extruded rods were made with molasses and sugar solutions and baked at 2300°F for seven minutes. The resultant 11 mm anodes were used as cores with 16 mm graphite shells. These dual anodes were inserted in the arc unit in the normal manner except that operation was performed with separate feed of the shell and core. Burning was quite satisfactory, the erosion rate being 27 to 90 grams of ore/kw-hr compared to 8 to 20 grams/kw-hr for the 13.6 mm shelled anodes described above. The erosion rate of the 16 mm shell was 1/4 to 1/7 that of the core. Tablets produced from a 5:1 mixture of beryl ore and graphite and fed separately through the shell, gave comparable erosion rates.

3. Arc Reaction Chamber

A study of information and data pertaining to the Sheer-Korman Process disclosed many problems that had to be solved in order to construct a proper chamber to enclose and control an arc, and to withstand corrosive characteristics of chlorine and product gases at high temperatures. It was expected that several of these problems would be solved by subcontractors who are specialists in their particular fields. At the same time it was realized that certain components of the process equipment would have to be fabricated on a small scale basis followed by considerable experimentation and development work, to obtain data

permitting the design and completion of the final pilot plant.

The more immediate problems requiring solutions to permit the subcontractors to proceed with their design included:

1. Determination of suitable materials of construction applicable to the walls of the chamber.
2. Development of an insulating and sealing sleeve through which electrodes would be introduced into the chamber.
3. The effect of arc radiation on the surrounding walls and the necessity for cooling.
4. The size and shape of the arc tail flame to permit design of the method of take-off of arc products.
5. The effect of particle charges and chloride condensation on cooling surfaces.
6. The need for magnetic stabilization of the arc enclosed by a metal chamber.

In order to investigate temperature distribution and flame characteristics of an arc operating in air, a steel chamber was constructed to enclose the arc. A water jet aspirator was connected to the take-off section of the chamber to withdraw the products of the arc. No attempt was made at collection, instead, when burning toxic materials, products were washed down the sewer. Thermocouples were cemented to the outside wall of the metal chamber and temperatures were recorded during arc operation. At a point on the outside of

the wall from 3 to 3-1/2 inches from the arc, the temperature recorded was 1310°F. This could be reduced by 300°F by passing air at a velocity of 1000' per minute over the outside wall.

The arc flame pattern was observed by means of a periscope under various operating conditions, with the air arc chamber. It was found that unless stabilization of the arc was maintained by magnets, local overheating due to the flame impinging on the wall would tend to melt the metal. Various refractories such as graphite plates, graphite coated with Sauereisen, cast Sauereisen, cast plastic fire brick material, and slices from insulating fire bricks were used around the anode in an attempt to find a material that would withstand the intense heat. However, for short protrusions, none of these refractories were found to be satisfactory from a temperature and sealing viewpoint.

To study the behavior of the arc in a chlorine atmosphere, a nickel arc chamber was constructed to withstand the corrosive action of the chlorine and the product chlorides at high temperatures. A jacket for water cooling was included. This unit was incorporated into the laboratory pilot plant and used in a first demonstration run. No satisfactory method of completely sealing the anode aperture had been devised by the time tests were initiated in this device. Indications were that chlorine attacked the nickel to some extent, possibly due to the inleakage of humid air.

The use of a metal chamber necessitated methods of insulating the anode and cathode from the walls. A ceramic sleeve was used around the cathode and various types of anode plates were tried. These were: water-cooled nickel plate, refractory coated graphite plate, and several types of cast and carved refractory plates. One of the more durable refractory plates was used. Difficulties were encountered during the first run due to flame impingement, which eroded through the plate in the area of the anode. The anode opening also became enlarged. Loss of product resulted.

A transite arc chamber was then fabricated in connection with the research program to study the burning of anodes which were fed manually through machined spring-loaded graphite contacts. The design of this arc chamber was such that either normal length 11 mm full core anodes or pellets could be burned. Removable sight glass windows were installed on each side of the arcing positions in order to observe the flame characteristics. To minimize the presence of water in the chamber, the anode was fed through a close fitting graphite sleeve. In order to make the transite impervious, the outer surface was coated with water glass. Subsequent operation seemed to indicate that this was not adequate. The transite chamber, however, was found to withstand the corrosive action of chlorine and products in the high temperature zones adjacent

to the arc. Operation with this equipment again pointed out the need for suitable seals around the electrodes. Also improvement was needed in the sight glass arrangement to eliminate deposits which obscured vision of the arc.

4. Collection System

The method of separation of product visualized was one of selective condensation incorporated with electrostatic precipitation. Of the three major products : BeCl_2 , AlCl_3 and SiCl_4 ; beryllium chloride has the highest boiling point. By regulating the exit gas temperatures from various condensers of the process equipment it appeared to be possible to condense selectively and to collect the several components. The success of separation by this procedure appeared to depend on the correct choice of temperature and absence of moisture.

The equipment set up for demonstration Run No. 1 included, consecutively after the nickel chamber, the following: a nickel pipe condenser, then a nickel Cottrell - both externally heated; a cold pipe condenser; a carbon tetrachloride absorption column. Results with this equipment indicated that collection might be possible without a Cottrell.

In order to establish design data for the separation of chlorine from carbon dioxide, an experimental glass laboratory column 4" in diameter and 3' high and packed

with 1/4" Berl saddles was constructed. Various gas compositions and ratios of gas/liquid flow were employed. It was found that a good separation could be achieved and that chlorine could be stripped readily from its solution in CCl_4 to yield a recovery of about 95%.

Operation of the first demonstration run with this absorption column indicated that it can be used satisfactorily for absorbing chlorine in the exit gases.

Experience with the small scale pilot plant pointed up the difficulties involved in using an oil seal around the high tension electrode in a commercial type Cottrell. This oil had to be inert to the action of chlorine and the products. Chlorinated transformer oil such as Pyranol was satisfactory. However, after completing the pilot plant run it was found that this oil vaporized in the heated Cottrell and condensed in the aluminum chloride condenser.

For further study of the problems involved in a collection system, small scale condensers and Cottrells made of fused silica were connected to the transite arc chamber, as part of the research program. A fused silica transition tube, externally heated, led from the arc box to a fused silica Cottrell also heated. The electrostatic feature of the Cottrell was obtained by coating a portion of the interior surface with a conductive film of Aquadag and

using a nickel rod equipped with a scraper disc as the high tension electrode. A pyrex water cooled condenser led from the hot Cottrell to a Cottrell similarly constructed but not heated. A cold trap for condensing SiCl_4 followed the Cottrell. Several pyrex glass tubes filled with silica gel and P_2O_5 were placed between the cold trap and air aspirator to prevent moisture back-up by drying the air which infiltrates.

Initial operation of this small scale second process demonstration unit indicated that high temperature and humid air inleakage to the system through the arc chamber caused reaction with the Cl_2 , resulting in excessive corrosion of the nickel electrode in the Cottrell. Since time was limited and data on erosion rates were desired, the equipment was revised to contain only the arc chamber, transition piece and cold Cottrell.

5. Process Demonstration

In February 1950 efforts were concentrated on procuring preliminary data on process efficiency and product purity from a small scale pilot plant by a deadline set by the Commission as April 4, 1950. Design and construction of the large pilot plant was halted pending the availability of more basic design data and experience from laboratory scale operations.

To afford this demonstration of the Sheer-Korman Process, a small scale pilot plant unit already under

fabrication was somewhat modified and assembled in the laboratory to consist of a water cooled nickel reaction chamber for use with chlorine, a commercial arc unit, heated nickel pipe condenser, heated nickel Cottrell, air cooled iron pipe condenser, chlorine absorption column and an air aspirator for exhausting the system. This unit which was being constructed to determine design factors for a large plant, was used to attempt a small scale process evaluation by determination of the energy required for burning of the ore anodes, the amount of chlorine required, the efficiency of conversion of the ore and the erosion rate of the anodes at various arc currents.

At the Commission's further request, an attempt was also to be made, if possible, to achieve determination of the efficiency of the chloride separation process, the purity of BeCl_2 collected, and a rough estimate of the economic advantage or disadvantage of the process.

For the initial process demonstration designated as Run No. 1, it was decided to use shelled anodes since these were the only anodes at this stage of development which permitted the maintenance of a stable arc and which would serve to provide reasonable continuity of operation. The total accumulated burning time of anodes during the initial run was two hours and fifty-seven minutes. The erosion rate for shelled anodes, in chlorine, was determined to be of the same order of magnitude as that already obtained in air.

The results of Run No. 1 demonstrated the feasibility of operating a high intensity arc in a chlorine atmosphere using an anode made of beryl ore and bituminous coal. Chlorination of the ore and some degree of selective condensation and product collection was achieved. The importance of the control of moisture was emphasized, and the need for better seals around the electrodes was realized. Moisture also affects the products and their separation. The high recovery of heat from the arc chamber (an average of 60% to 70%) tended to confirm an exothermic reaction, although no accurate determination of heat losses could be made.

In continuation of experimental process demonstration, work on a smaller scale followed. A transite arc chamber and glass apparatus collection system were set up to provide evaluation of the Sheer-Korman Process on a research scale in contrast to the semi-production scale contemplated in the small scale pilot plant unit. The design of this equipment was based to a large extent on the results obtained by the Inventors in previous experiments at Boston. The system devised for the collection of products from the transite arc reaction chamber was composed of fused silica, including heated and cold Cottrell units with Aquadag coatings on interior surfaces and nickel electrodes, pyrex water cooled condensers, cold traps, drying tubes and an air aspirator.

A long graphite sleeve provided for anode feed and some measure of sealing during operation, and machined graphite spring-loaded brush contacts served to supply electric current through the anode. Chlorine was introduced around the cathode which was connected by a flexible cable to the electric supply.

In operation, the problems of moisture control and the limited duration of the arcing period, prevented the obtaining of conclusive results. All trial runs with this equipment were made under the guidance and with the participation of one of the Inventors, whose previous experience, including preheat runs for the elimination of moisture and study of arc phenomena, was utilized in an attempt to provide successful demonstrations.

A series of investigations was made of brush materials in an effort to eliminate arcing at the contacts and breaking of the anodes during operation. All runs were made with anodes extruded with beryl ore, bituminous coal and molasses. From operating experience, it was found that these were the only full core anodes available which permitted any sustained arc operation.

Excessive corrosion of the nickel electrode of the hot Cottrell and cracking of the fused silica vessels made continued operation with this equipment impractical.

Operational runs indicated that the cold trap maintained at -35°C , was relatively ineffective and collected little or no silicon tetrachloride. These results were based on very short arc burning periods.

More favorable results were obtained with an abbreviated pyrex glass collection system apparatus consisting of transition piece and cold Cottrell in which the product gases were successfully precipitated. Products were partially hydrolyzed since the maintenance of proper moisture control was not possible in the limited period of time available, near the termination of the contract. Several anode erosion rates were measured as 115 to 150 grams of anode per kwh, based on continuous arc operation of one to three minutes duration, the longest obtained with full core anodes in a chlorine atmosphere.

6. Analytical Control

To provide for analysis of process samples, a complete analytical control laboratory was constructed at the plant site - equipped with reagent chemicals; drying ovens; weighing balances; electric heating devices; glass blowing apparatus; laboratory sink, stands, clamp, chemical fume hood, storage racks and utilities.

Chemical control of raw materials and continuous sampling and analysis of anode composition and products collected were carried on throughout in the Maspeth laboratory. Experiments were set up to test the methods of

analysis of various anions (chlorides and oxides) and a standardized procedure developed. Standardized procedures were also developed for analyzing chlorine, carbon dioxide, silicon tetrachloride dissolved in carbon tetrachloride, and for gaseous mixtures containing these components.

Standard coal and pitch analyses were made on the carbon materials to determine their carbon, VCM, moisture, and ash content. Extruded and baked anodes were analyzed to determine their carbon content. Baked anodes were examined under a microscope to determine existence of carbon matrix, surface characteristics, uniformity of mixture, and description of particle size. In addition, a Moh scale was used to determine anode hardness. Resistivity measurements were made on anodes to determine their electrical properties. The laboratory also carried out sieve analyses of ore and carbon to determine size distribution.

7. Materials of Construction

Information on the materials of construction satisfactory for use in contact with the Sheer-Korman product gases was found to be inadequate. There were only casual references to suitable materials in sections of reports which Burns and Roe engineers were permitted to read at the Atomic Energy Commission library. For the most part, the standard references in technical literature had to be used as a guide, even though insufficient.

A program was started at the Maspeth laboratory to test materials of construction. A small nickel-plated steel vessel was designed and fabricated for testing corrosion resistance of various materials in an anhydrous atmosphere of metallic chloride at elevated temperatures. However, work was not completed.

Since nickel was deemed to be reasonably resistant to the expected corrosive conditions it was desirable to be able to coat various steel, stainless steel, copper or brass equipment with this metal. Standard electroplating methods are usually unsatisfactory for odd-shaped pieces containing sharp edges or corners, nor are they readily applicable to internal plating of fabricated units. Therefore, experiments were performed at the Maspeth laboratory with the process of nickel plating by chemical reduction, as developed by Brenner and Riddell but never extensively used. Tests on samples indicated the feasibility of producing adherent low-porosity coatings which gave complete coverage of all surfaces, including welds.

To meet construction schedules, materials for certain items of equipment had to be selected before corrosion tests could be made. Thus it was necessary to abide by the following sources of information:

1. Literature in the Atomic Energy

Commission library revealed that manufacturers dealing with the same conditions used Nichrome V and fused silicon carbide retorts for the sublimation of both beryllium and aluminum chlorides, as well as for ore chlorination in the presence of carbon. Mention was made also of the use of chloride condensers made of nickel and maintained at temperatures around 300°C. Other than referring qualitatively to nickel chloride contamination that resulted from the use of the above material, no precise data were offered on the effect of temperature, moisture and other chlorides.

2. The Chemical Engineering "Materials of Construction" symposium dated November 1946 indicated that nickel would satisfactorily withstand attack by dry chlorine at temperatures up to 1000°F.

3. Various sources of information revealed that iron should not be exposed to dry chlorine at temperatures above 400°F. Above that the corrosion rate was stated to increase rapidly and become infinite at 483°F, at which point the iron ignited.

4. Other sources of information indicated that reactions occurred between silica and chlorides of beryllium and aluminum at rates varying with temperature.

Therefore, in the choice of materials of construction to resist chlorine and chloride corrosion, it was decided to use steel at temperatures below 350°F, nickel between 350°F. and 850°F., and glass at room temperature.

D. Redirection and Termination of Contract

The original schedule advanced for the completion of design and delivery at the site of the various components of the pilot plant was April 1, 1950. Installation was to be completed and start-up of operations was to take place on or before June 30, 1950.

As the contract work progressed, it became apparent that there were many problems to be solved and considerable development work to be done on a laboratory and small pilot plant scale to permit design and construction of the various units of the plant. Despite this realization, all efforts were directed toward meeting the schedule dates and the Commission was kept informed at all times of the calculated risks which were necessary in the design of relatively expensive equipment on the basis of the preliminary data available.

In a meeting on February 24, 1950, the Contractors were informed that the Commission had determined that it was in the best interest of the Government that there be a redirection of the work under the contract. This was confirmed in a letter from the Commission dated March 3, 1950, directing that work be

discontinued on the large pilot plant and that the small scale pilot plant unit then being fabricated and assembled in the laboratory at Maspeth be redesignated for contract purposes as the "pilot plant".

It was directed that efforts be concentrated on procuring preliminary data on process efficiency and product purity from the small scale pilot plant on or before a deadline of April 4, 1950. The redirection suspended, without termination, the work of the subcontractors for the design and supply of parts or equipment for the projected large pilot plant but at the same time gave authorization for their use as consultants in the design of the small scale pilot plant. Demonstration Run No. 1 on the small scale laboratory pilot plant was made in accordance with this redirection and Report NYO-1036 was submitted on April 3, 1950 on its results, as discussed in more detail elsewhere in this report.

In a further letter of direction dated March 21, 1950, the Commission set up a research program and weekly progress meetings to plan specific experiments and evaluate experimental results as they pertained to the contract. In the execution of this program, the Commission ordered the discontinuance of operation of the small scale pilot plant following the completion of Run No. 1, and shifted emphasis to a smaller scale research pilot plant unit.

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Mr. A. J. Bendler, Chemical Engineering Consultant associated with Burns and Roe, Inc., was selected, with the Commission's approval, as director of the research program, reporting to the Contract Group and to the Commission's representative through the weekly progress meetings. He also assumed direction of the anode fabrication program which had been underway since the start of activities at the Maspeth laboratory.

The research unit was constructed, assembled and operated under the active supervision and with participation of the Inventors, in an effort to provide evaluation of the Sheer-Korman Process. During this phase of development, plans and procedures for research and development work were submitted and reviewed by the Commission's representative at the weekly progress meetings prior to inception of the various programs.

In keeping with the reduced scale of activities, on April 13, 1950, the Commission directed that personnel assigned and scope of work at the Maspeth plant be reduced in order to limit monthly expenditures to \$10,000 by Burns and Roe, Inc., for materials, services and reimbursements to the Inventor Group.

On June 2, 1950, at the weekly progress meeting, Mr. Anscher, representing the Commission, gave notice of the termination of the contract effective as of the close of business June 2, 1950. He stated that authority for continuation of the

contract could only be based on work of a developmental or construction nature requiring the Contractor to be in a position to build a pilot plant, and that it was obvious that the work then in progress under the contract had not yet provided sufficient data to make possible the design and construction of a large pilot plant. It was agreed that information and data to be gained during the remaining contract period would not be sufficient to permit the construction of a pilot plant and, therefore, in the Commission's opinion, did not warrant continuation.

After formal termination of the contract, the Contractor Group brought all work to an immediate close and began preparation of a termination report. Burns and Roe, Inc., proceeded with termination close-out activities at the Maspeth plant, under the supervision and direction of the Commission's representatives, including preparation of physical inventories and pricing of items and equipment, dismantling, decontamination, shipping out and disposal of laboratory equipment and handling of other termination matters necessary to meet contract obligations.

E. Summary and Conclusions

1. Anode Electrodes

Satisfactory experimental anode electrodes were fabricated of appreciable length composed of beryl ore and

carbon. The beryl ore was carefully sized and calcined and mixed with finely sized non-calcined bituminous coal having a composition within a prescribed range.

It is known that a specific combination of closely controlled steps is required, including choice of proper extrusion aid and baking temperature and technique. The problem has been intensively investigated and certain critical factors have been exposed and evaluated. Carbon sources and detailed processing steps are now more clearly understood.

Extension of these results is necessary to obtain an anode with an improvement in strength, conductivity, homogeneity and smoothness of surface for commercial application. This is a fundamental requirement to afford continuous operation of the process and to provide a basis for determination of production economy.

While it is believed that the major tributary problems involved in anode fabrication have been recognized, further laboratory development is necessary to establish a complete specification for anode preparation and to enable comparison of the different alternates on economic grounds.

2. Arc Operation

Both full core and shelled anodes were eroded in an arc in a chlorine atmosphere. The shelled anodes provided continuity of arc operation on a pilot plant scale, while the full core anodes were only sufficient for laboratory demonstration.

Development work was centered on improving the physical characteristics of the full core anodes, as well as the design and materials of the contact brushes, to permit more continuous operation.

The highest erosion rates were obtained with carefully selected full core anodes. These were coated after baking to provide a smoother surface and better contact, and then were hand fed into the arc through specially designed graphite brush contacts. Erosion was measured during several continuous arc periods of one to three minutes duration, during which rates were obtained which exceeded 100 grams of ore per kilowatt hour.

3. Collection System

The design of a multistage collection system was not completed during this contract period. Many schemes were proposed and components of several systems were tested.

It was found that products from the chlorine arc could be collected by electrostatic precipitation. With the reaction chamber discharging directly into the Cottrell unit product particles were swept through if the collecting unit was not energized. Energizing the unit caused product particles to adhere to the walls, resulting in what appeared to be complete recovery.

Where process apparatus was set up with heated transition sections and heated condensing surfaces between

the arc chamber and precipitation units, product particles adhered to these surfaces. There was evidence of some degree of separation of product components. However, attempts to determine analytically the efficiency of chloridization, and to achieve selective stage condensation of products were hampered by the presence of moisture.

4. Moisture Control

The problem of controlling moisture to permit derivation of an anhydrous product from the Sheer-Korman Process received primary consideration during design and operation of process apparatus. The necessity of such control was recognized from the outset. However, difficulties encountered can be emphasized by the fact that none of the designs were completely successful.

Attention was given to the following steps in efforts to remove moisture and prevent infiltration of moisture-laden air:

- a. Preheating apparatus.
- b. Purging with dry gas.
- c. Use of drying tubes in chlorine supply system.
- d. Pressurizing and mechanical sealing.
- e. Drying of hooded area surrounding apparatus.

It is felt that further development along these lines should enable a satisfactory design within practical economic limits.

APPENDIX-I

List of Laboratory Procedures and Completion Reports

Collection System

<u>No.</u>	<u>Procedure</u>	<u>Completion Report</u>	<u>Title</u>
A1-1	11/22/49	12/19/49	Nickel Plating by Chemical Reduction
A1-2	1/3/50	1/5/50	Nickel Plating of Stainless Steel, Copper and Brass
A2	1/4/50	4/11/50	Corrosion by Gaseous Aluminum Chloride
A3	3/11/50	3/11/50	Melting Point of Solder

Electrode Fabrication

B1	10/20/49	12/8/49	Effect of Sand Particle Size and Baking Temperature on Resistance and Strength of Bituminous Coal Electrodes
B1-1	11/9/49	"	Effect of Sand Particle Size and Baking Temperature on Resistance and Strength of Bituminous Coal Electrodes
B3	11/7/49	"	Optimum Baking Schedule for Best Bond and Resistivity of Sand - Coke 1 Pellets
B1-2	11/14/49	12/2/49	Evaluation of Ottawa Sand No. 71 for Use Directly in Slugs
B2	11/4/49	12/7/49	Baking Furnace Calibration
B2-1	11/14/49	12/8/49	Baking Furnace Calibration - Longitudinal Temperature Gradient
B2-2	11/14/49	"	Baking Furnace Calibration - Cooling Effect of Nitrogen Purge

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Electrode Fabrication (Con't)

<u>No.</u>	<u>Procedure</u>	<u>Completion Report</u>	<u>Title</u>
B2-3	11/28/49	2/1/50	Furnace Calibration - Elimination of Temperature Gradient
B4	11/10/49	Standard	Resistivity Determination
B4-1	2/2/50	Procedure 2/16/50	Resistivity of Elevated Temperatures
B5	11/16/49	12/12/49	Evaporation of Kerosene from Extruded Rods
B5-1	12/2/49	"	Evaporation of Kerosene from Extruded Rod Under Reduced Pressure
B6	11/18/49	4/12/50	Determining Extrusion Properties of Ottawa Sand No. 71
B7	12/12/49	2/24/50	Pellet Evaluation of Carbon Materials Using Sand No. 71
B8	12/16/49	1/5/50	Baking of Extruded Pitch Rod
B9	no date	1/30/50	Evaluation of Wetting Agents for Cold Extrusions
B9-1	1/9/50	2/24/50	Baking Evaluation of Wetting Agents for Extrusion
B10	1/10/50	1/11/50	Extrusion of Sand Bituminous Coal Rods
B10-1	1/10/50	1/23/50	Baking of Sand Bituminous Coal, Extruded Rods and Analysis
B11	1/11/50	2/23/50	Extrusion and Baking of Ore-Bituminous Coal-Peanut Oil Rods
B12	1/17/50	1/30/50	Extrusion of Ore (102), Lampblack (4), Pitch (65) P Blends
B12-1	1/26/50	3/14/50	Extrusion of Ore (102), Lampblack (4), Pitch (65) P Blends

Electrode Fabrication (Con't)

<u>No.</u>	<u>Procedure</u>	<u>Completion Report</u>	<u>Title</u>
B12-2	1/17/50	1/30/50	Baking of Ore (102), BR Graphite (16) Pitch (65) P-Extrusions
B12-3	1/30/50	2/8/50	Extrusion of Ore
B12-4	2/2/50	3/14/50	Baking of Ore (102) BR Graphite (16) Pitch (65) P-Extrusions
B12-5	2/2/50	3/14/50	Baking of Ore (102) BR-Bituminous Coal (76) CPA - Pitch (65) P-Extrusions
B13	1/24/50 1/26/50	1/30/50	Determination of Softening Characteristics of Ore-Carbon- Pitch Mixes
B14	1/30/50	3/15/50	Comparison Test of Hot Extrusions
B15	2/6/50	2/21/50	Extrusion of Ore, Carnauba Wax, Bituminous Coal Mixes
B16	2/7/50	Standard Procedure	Standardized Baking Procedure for Extruded Rods
B16-1	2/16/50	Standard Procedure	Standardized Tests on Baked Rods
B17	2/10/50	3/14/50	Extrusion of Ore (102) BR, Bituminous Coal (76) CPA, Molasses (104)
B18	2/10/50	3/14/50	Extrusion of Ore (102) BR, Bituminous Coal (76) CPA, Sugar (105) Solutions
B19	2/10/50	3/21/50	Effect of Low Temperature Curing of Extruded Rods before High Temperature Baking
B20	2/13/50	3/15/50	Extrusion of Ore(102) BAR-Pitch (65) P Mixes of Varying Ratios

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Electrode Fabrication (Con't)

<u>No.</u>	<u>Procedure</u>	<u>Completion Report</u>	<u>Title</u>
B21	2/13/50	3/16/50	Ore (102) BAR - Pitch (65) P Dry Filled Shelled Rods
B21-1	2/21/50	3/16/50	Bauxite Ore (110) - Pitch (65) P Dry Filled Shelled Rods
B22	2/21/50	3/16/50	Extrusion of Ore (102) BAR - Bituminous Coal (76) CPA Mixes of Varying Ratios
B23	2/21/50	5/1/50	Extrusion of Ore (102) BAR - Bentonite - Bituminous Coal (76) CPA Mixes of Varying Ratios
B24	2/24/50	3/16/50	Oxide - Pitch - Shelled Rods
B25	2/24/50	Standard Procedure	Sizing of Shelled Rods to Specified Diameter
B26	3/2/50	4/10/50	Preparation of Lower VCM Content Pitches from Coal Tar Pitch (18) 310°F m. p.
B26-1	4/4/50	6/2/50	Extrusions of Rods Made with 20% VCM Pitch
B27	3/13/50	3/16/50	Preparation of Shelled, Tamped Sand Coal Anodes
B28	3/16/50	3/16/50	Performance Tests of New Extruder Die
B29	5/1/50	6/2/50	Extruded Beryl Anodes with 13.5% Carbon, Using Molasses
B30	--	3/15/50	Report on Extruder Tests at Rutgers Univ.
B31	3/31/50	4/24/50	Effect of Baking Time and Temperature on Extruded Rods Made with Peanut Oil
B32	3/30/50	6/2/50	Continuous Baking of Extruded Cores

Electrode Fabrication (Con't)

<u>No.</u>	<u>Procedure</u>	<u>Completion Report</u>	<u>Title</u>
B33	4/4/50	4/12/50	Effect of Coal Concentration on Extruded Rods
B34	4/4/50	4/10/50	Investigate the Effect of Water as a Wetting Agent on Extruded Rods
B35	6/2/50	6/2/50	Molasses Extrusions
B36	4/13/50	4/20/50	Silicone Fluids as Extrusion Aids
--	--	12/16/49	Kingston Trip on Air Separator for Ore
--	--	12/19/49	Extrusion Tests at Norristown, Pa.

Analytical Procedures and Analyses

C1	11/15/49	Standard Procedure	Procedure for Ro-Tap Analyses - Dry
C2	11/15/49	Standard Procedure	Procedure for Ro-Tap Analysis - Wet
C3	12/23/49	12/23/49	Analysis of Chlorine in Chlorine Carbontetrachloride Solutions
C3-1	12/23/49	1/10/50	Analysis of Chlorine in Chlorine Carbontetrachloride Solutions
C4	12/23/49	Standard Procedure	Determination of Chlorine in Chlorine Carbontetrachloride Solutions
C5	12/28/49	12/28/49	Analysis of Mixtures Containing Silicon, Iron, Aluminum, Beryllium and Nickel
C6	12/29/49	Standard Procedure	Outline of Analysis of Mixtures Containing Silicon, Iron, Aluminum, Beryllium and Nickel

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Analytical Procedures and Analyses(Con't)

<u>No.</u>	<u>Procedure</u>	<u>Completion Report</u>	<u>Title</u>
C6-1	3/3/50	Standard Procedure	Method for Analysis of Mixtures Containing Silicon, Iron, Aluminum, Beryllium and Nickel
C6-2	3/27/50	Standard Procedure	Method for Analysis of Mixtures Containing Silicon, Iron, Nickel, Aluminum and Beryllium
C7	1/3/50	Standard Procedure	Determination of Moisture in Process Materials
C8	1/3/50	Standard Procedure	Determination of Moisture, Volatile and Combustible Matter in Carbon Materials
C9	1/3/50	Standard Procedure	Determination of Moisture, Volatile and Combustible Matter, and Carbon Content in Process Materials
C10	1/18/50	Standard Procedure	Determination of Chlorine and Carbon Dioxide in Exit Gases of Chlorine Recovery Systems
C11	1/30/50	Standard Procedure	Analysis of Shell Electrodes
C12	2/15/50	Standard Procedure	Microscopical Analysis of Electrodes
C13	2/27/50	5/9/50	Silicon Tetrachloride - Absorption Tower Tests
C14	3/6/50	Standard Procedure	Method of Analysis of Solutions Containing Carbon Tetrachloride, Silicon Tetrachloride, Chlorine and Carbon Dioxide
C14-1	3/25/50	Standard Procedure	Method of Analysis of Solutions and Gases Containing Carbon Tetrachloride, Silicon Tetrachloride and Chlorine

Analytical Procedures and Analyses (Con't)

<u>No.</u>	<u>Procedure</u>	<u>Completion Report</u>	<u>Title</u>
C15	3/6/50	Standard Procedure	Method for Analysis of Gas Mixtures Containing Carbon Tetrachloride, Silicon Tetrachloride, Chlorine and Carbon Dioxide
C16	3/27/50	Standard Procedure	Short Method for Determination of R_2O_3 And BeO in $BeCl_2$
C16-1	4/19/50	Standard Procedure	Revised Short Method for Determination of R_2O_3 and BeO in $BeCl_2$
C17	3/30/50	Standard Procedure	Determination of Total Chloride in Suspected Beryllium Chloride Sample

Arc Lamp

D1	12/1/49	12/19/49	Current, Voltage, Gap Characteris- tics of Commercial 13.6 mm. Anode
D1-1	no date	12/21/49	Current, Voltage, Gap Characteris- tics of Commercial 11 mm. Anode No. 3.
D1-2	12/19/49	4/18/50	Current, Voltage, Gap Characteris- tics of Special 11 mm. Anode No. 4
D2	12/15/49	no date	Air Arc Chamber Operation
D3	12/29/49	1/16/50	Effect of Stabilizing Magnet on Arc
D3-1	1/24/50	4/28/50	Effect of Stabilizing Magnet on Arc
D4	1/10/50	1/11/50	Air Arc Chamber Operation with Sand-Bituminous Coal - Peanut Oil Rods
D5	1/11/50	1/18/50	Arc Burning of Ore-Bituminous Coal Rods in Air Box

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Arc Lamp (Con't)

<u>No.</u>	<u>Procedure</u>	<u>Completion Report</u>	<u>Title</u>
D5-1	1/19/50	1/24/50	Arc Burning of Ore - Bituminous Coal Extrusions made by Procedures B9 and B11
D5-2	1/25/50	2/21/50	Arc Burning of Ore-Bituminous Coal Pitch Rods from Procedure B12
D6	1/23/50	2/24/50	Arc Burning of Ore-Bituminous Coal Rods with Graphite Shell
D6-1	1/25/50	2/21/50	Arc Burning of Ore-Bituminous Coal Pitch Rods from Procedure B12 with Graphite Shell
D7	2/2/50	2/2/50	Arc Burning of Sand Rods with graphite shells made in Norristown, Pa. and Passing of Effluent Gases Through Cottrell Precipitator
D8	2/2/50	2/20/50	Air Arc Operation of Graphite Shells Packed with Ore
		3/20/50	Supplement
D9	3/1/50	4/7/50	Air Arc Chamber Burning of Beryl Pitch Dry Filled Shelled Anodes
D10	3/1/50	4/7/50	" " " "
D10-1	4/7/50	3/15/50	Erosion Rates of Extruded & Tamped Rods in Air
D11	3/1/50	3/13/50	Arc Stabilization in Chlorine Chamber
D12	3/13/50	3/16/50	Burning of Shelled Sand Anodes in Air Chamber
D13	3/16/50	3/16/50	Heat Run on Chlorine Chamber
D14	3/30/50	4/5/50	Air Arc Operation with Continuous Feed of Core Through Shell

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Arc Lamp (Con't)

<u>No.</u>	<u>Procedure</u>	<u>Completion Report</u>	<u>Title</u>
D15	3/31/50	4/6/50	Temperature Gradients on Air Arc Chamber
D15-1	4/25/50	4/4/50	Temperature Gradients on Air Arc Chamber with Air Cooling
D16	4/5/50	4/25/50	Determination of the Maximum Current Tolerated for a Five Inch Protrusion of Anode
D17	4/5/50	4/5/50	Performance of Arc with Axes of Anode and Cathode Parallel
D18	4/10/50	5/1/50	Arc Mechanism
D19	4/25/50	4/25/50	Erosion Rate of Anode Core and Shell for Parallel Axes

Chlorine Recovery

E1	12/2/49	2/3/50	Absorption of Chlorine in Carbon Tetrachloride
E1-1	12/2/49	1/4/50	Absorption of Chlorine in Carbon Tetrachloride Calibration of Orifices
E1-2	1/4/50	2/3/50	Checking Chlorine Scrubbing Train
E2	2/7/50	2/7/50	Test for Characteristics of Jet Exhauster

APPENDIX-II

List of Interim Reports

<u>No.</u>	<u>Date</u>	<u>Title</u>
BAR-PAT-1	6/23/49	Status Report on Patent Search
BAR-1	4/1/49	Progress Report for February 24, 1949 to April 1, 1949
BAR-2	5/1/49	Progress Report for April 1949
BAR-3	6/1/49	Progress Report for May 1949
BAR-4	7/1/49	Progress Report for June 1949
BAR-5	8/1/49	Progress Report for July 1949
BAR-6	8/31/49	Laboratory Investigation of the Factors Affecting the Fabrication of Anodes for the Sheer-Korman Process
BAR-7	9/1/49	Progress Report for August 1949
BAR-8	10/1/49	Progress Report for September 1949
BAR-9	11/1/49	Progress Report for October 1949
BAR-10	12/1/49	Theoretical Study of the Thermal Requirements of the Collection System for Metallic Chlorides in the Sheer-Korman Process
BAR-11	12/1/49	Progress Report for November 1949
BAR-12	1/1/50	Progress Report for December 1949
BAR-13	2/1/50	Progress Report for January 1950
NYO-1035	3/1/50	Progress Report for February 1950
NYO-1036		Small Scale Pilot Plant - Run No. 1 Sheer-Korman Process

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List of Interim Reports (Con't)

<u>No.</u>	<u>Date</u>	<u>Title</u>
NYO-1037	4/1/50	Progress Report for March 1950
NYO-1038	5/1/50	Progress Report for April 1950
NYO-1039	6/1/50	Progress Report for May 1950
-	April 1950	Combustion Engineering-Superheater, Inc. Interim Report - Project BAR
-	4/26/50	Combustion Engineering-Superheater, Inc. Measurement of Thermal Radiation near a High Current Electric Arc
-	5/5/50	Research Corporation - Summary of Contract Work

APPENDIX III

List of Sketches and Drawings

Electrical Drawings

<u>Dwg. No.</u>	<u>Title</u>
3001	240v D.C.-110v A.C.- 440v A.C. Cable Plan
3001-1	"
3001-2	"
3001-3	"
3001-4	"
3001-5	"
3001-6	"
3001-7	"
3002	Arrangement & Cable Layout of Resistors
3002-1	"
SK1301	Arrangement & Cable Layout for Resistors
3003	Plan View of Bldg. 43A
3004	Elevation of North East Wall Bldg. 43A
3005	Elevations of North East & North West Corridor Wall Bldg 43A
3006	Elevations of West & North West Walls Bldg. 43A
3007	Elevations of South & East Walls Bldg 43A
3008	Precipitator
3009	Elevation of North wall West End Bldg. 48

Structural Sketches

<u>Dwg. No.</u>	<u>Title</u>
Sk-25-1	Passageway to Bldg. 48
Sk-25-2	Passageway to Bldg. 48
Sk-26	Isometric Arrangement of Chlorine Shed
Sk-30	Caustic & Service Water Piping for Chlorine Shed
Sk-31	"
Sk-32	Steam & Condensate - Sprinkler & Drain Piping
Sk-33	"
Sk-34	Street plan for routing Chlorine Gas

Structural Drawings

4001	Proposed Passageway between bldg. 48 & etc.
4002	Foundation Plan of proposed passageway
4003	Loading Platform in Bldg. 48
4004	Monorail over Loading Platform
4005	Chlorine Storage Shed
4006	" "
4007	" "
4008	" "
4009	" "
4010	" "
4011	" "

Laboratory Sketches

<u>Dwg. No.</u>	<u>Title</u>
Sk-3	Supporting Frame for Ball mill
Sk-4	Discharge hopper for mill
Sk-5	Typical closing gate for hoppers & buckets
Sk-6	Retaining split gland for stuffing box
Sk-7	Charging hopper for mill
Sk-8A	Bucket 25
Sk-8	Gas Chamber
Sk-8-1	"
Sk-8-2	"
Sk-8-3	"
Sk-8-5	"
Sk-8-6	"
Sk-8-7	"
Sk-8-8	"
Sk-8-9	"
Sk-8-10	"
Sk-8-11	"
Sk-8-12	"
Sk-9	Closing Gate for dust collector
Sk-10	Dust Collector Bucket
Sk-11	Supporting Mounts for Ball Mill Motor & G. R.

Laboratory Sketches (Con't)

<u>Dwg. No.</u>	<u>Title</u>
Sk-13	Air Box
Sk-13-1	Air Box
Sk-15-1	Funnel for Air Classifier
Sk-15-2	Air Classifier Discharge Gate
Sk-15-3	" "
Sk-16	Ball Mill Lifting Jack
Sk-16-2	" "
Sk-17-1	Assembly of Chlorides Condenser
Sk-17-2	Chlorides Condenser - Details
Sk-17-3	" "
Sk-17-4	" " (12/29/49)
Sk-17-4	" " (1/11/50)
Sk-17-5	Chloride Condenser Plate
Sk-17-6	"
Sk-17-7	"
Sk-17-8	Assembly of Chlorides Condenser
Sk-17-9	Chloride Plate Condenser - Frame for End Pl
Sk-17-10	Chloride Plate Condenser - Frame for Inter. Pl.
Sk-17-11	Chloride Cond. Meth. of Assembly
Sk-17-11A	" "
Sk-17-12	" "

Laboratory Sketches (Con't)

<u>DWG. No.</u>	<u>Title</u>
Sk-17-13	Chloride Plate Condenser End Plates
Sk-18-3	Vacuum Cleaning System
Sk-18-4	Vacuum Cleaning System Bldg. #48
Sk-19-3	Chloride Generator & Condenser System
Sk-19-2	" "
Sk-19-1	" "
Sk-20	Sentry Furnace Graphite Boat
Sk-21	Storage Cabinet
Sk-24	Corrosion Testing Pot
Sk-29	Lifting Mechanism for Bucket
Sk-37-2	Elbow Connecting Transition piece & Condenser
Sk-37-1	Transition piece between Arc Chamber & Cond.
Sk-38	Transition piece from Condenser to Cottrell Precip.
Sk-39	Prep. for equipment Run #2
Sk-40-0	Small scale Pilot Plant Sheer-Korman Process
Sk-40-1	Equip. Prep. for Run #2
Sk-40	" "
Sk-40-2	" "
Sk-41	Heating Jacket for Precipitator
Sk-43	Alkali Tank for Chlorine Absorption

Laboratory Sketches (Con't)

<u>Dwg. No.</u>	<u>Title</u>
Sk-46	Extrusion die
Sk-47-1	Chloride Plate Oil Condenser end Plates
Sk-47-2	" "
Sk-47-3	" "
Sk-47-4	" "
Sk-48	Nickel Condenser
Sk-48-1	Chloride Plate Oil Condenser 2" frame for Inter. Plates
Sk-49	Retort for Chloride Analysis
Sk-50	Vacuum Cleaner Inst. Collector Adapter for Dirt Bucket
Sk-51	Alterations to Dust Hopper
Sk-52	Conn. Pipe from Arc Chamber to Precipitator
Sk-52-1	Precipitator Cylinder
Sk-53	Air Cooled Condenser
Sk-48-2	Chloride Plate Condenser - Intermediate Plates
Sk-48-3	" " - Frame for End Plates
Sk-8-0	Gas Chamber
Sk-8-01	"
Sk-8-02	"

Laboratory Drawings

<u>Dwg. No.</u>	<u>Title</u>
1202	Preliminary Sketches of equipment
1204	Small scale test set up for laboratory
1205	Small scale test set up for laboratory
1206	Waste gas disposal system
1207	" "
1208	Hood for Ball mill
1209	Hood canopy for mill
1210	Canopy Hood for Extrusion Machine
1211	Hood Canopy for Mixing & Storage Bin
1212	Enclosed Hood for Crusher and Storage Bin
1213	Plot Plan of area to be vacuum cleaned
1401	Dust Removal Ducts
1401-1	Prelim. layout dust removal ducts
1402	Supporting frames for resistor banks
1450	Hood Laboratory Bench
1451	Hood for Ro-Tap working bench etc.
1452	Hood for Mixer
1453	Hood for Electric Furnace & Extruder
1451-1	Isometric of 1451
1455	Darkroom- Building 42
1456	Sand Box

Laboratory Drawings(Con't)

<u>Dwg. No.</u>	<u>Title</u>
1457	Experimental Hood
1458	Electric Furnace
1459	Ball Mill Hood
<u>Layout Drawings</u>	
1203	Preliminary Laboratory equipment layout
2002	New York Naval Shipyard - Plan of fence
2003	New York Naval Shipyard - fence details So. Ave.
2004	" " " " - Plan of new fence
2005	" " " " - Struct. & Plumbing demolition dr
2006	" " " " "
2007	" " " " "
2008	" - Plan of 43A
2009	" - Plan of 42
2100	Plot Plan of Maspeth Annex
2101	New York Naval Shipyard - Maspeth Annex
2102	" " " Section Map
2103	Plot Plan of Bldg. 43

APPENDIX - IV

List of Data Books

Collection System

<u>Book</u>	<u>Pages</u>
A-1	A-1 to A-75
A-2	A-101 to A-175

Electrode Fabrication

B-1	B-1 to B-75
B-2	B-101 to B-174
B-3	B-202 to B-275
B-4	B-301 to B-376
B-5	B-401 to B-475
B-6	B-501 to B-575

Analytical Procedures & Analyses

C-1	C-1 to C-75
C-2	C-101 to C-175
C-3	C-201 to C-275

Arc Operation

D-1	D-1 to D-75
D-2	D-101 to D-175

Chlorine Recovery

E-1	E-1 to E-75
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Pellet Anodes

F-1	F-1 to F-75
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APPENDIX V

COPIES OF INVENTORS' SKETCHES

Diagram A - Apparatus used in first experiments - refer to page 10.

Diagram B - High Erosion Arc Phenomenon - refer to page 16.

Diagram C - Apparatus used in Vacuum experiments - refer to page 21.

Diagram A

Copy of Inventors' Sketch

POSITIVE & NEGATIVE TERMINALS
TO BE CONNECTED TO
220 V. D.C. SUPPLY

Diagram B

Copy of Inventors' Sketch

Diagram C

Copy of Inventors' Sketch