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MEMORANDUM FOR PRR (Contractor Publication)

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Joseph C. Schumacher (WECCO), "History of Establishing a Source of Potassium and Ammonium Perchlorate for Use in Solid Propellant Rockets"

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HISTORY OF ESTABLISHING A SOURCE OF POTASSIUM AND AMMONIUM PERCHLORATES FOR USE IN SOLID PROPELLANT ROCKETS

Joseph C Schumacher

35th AIAA/ASME/SAE/ASEE Joint Propulsion Conference and Exhibit
20–24 June 1999
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HISTORY OF ESTABLISHING A SOURCE OF POTASSIUM AND AMMONIUM PERCHLORATES FOR USE IN SOLID PROPELLANT ROCKETS

Joseph C Schumacher*

Abstract

An Air Corps Jet Propulsion Project (ACJP) at the California Institute of Technology and Western Electrochemical Company Inc. (WECCO) agreed in December 1942 to establish a domestic source of supply of perchlorates in Los Angeles, California for use in solid rocket propellants. This project was designated GALCIT Project No.1. WECCO, with financial aid from ACJP, designed and constructed a perchlorate pilot plant in Los Angeles. Experimental quantities of potassium and ammonium perchlorates were produced as required by ACJP. The GALCIT project was successfully completed in November 1943. In 1943 WECCO designed and constructed a large-scale plant in Los Angeles to produce potassium perchlorate at the rate of 100 tons/month. Start-up began in January 1944. Expansion of the Los Angeles plant to 200 tons/month was started late in 1944. WECCO designed another potassium perchlorate plant to be constructed in Henderson, Nevada in 1945. This new plant was designed to produce potassium perchlorate at a rate of 1200 tons/month. Start-up of the Henderson plant began in the first week in July 1945. WECCO began production of ammonium perchlorate (AP) in a small plant in Henderson, Nevada in 1947. WECCO designed a new AP plant with production capacity of 50 tons/day. This new AP plant was constructed in Henderson, Nevada and began production in 1953

Introduction

Robert L. Geisler, a member of the American Institute of Aeronautics and Astronautics (AIAA) Solid Rocket Technical Committee's History Subcommittee wrote to me in July 1998 stating that the history subcommittee arranges a history session at each annual propulsion meeting. He also stated that the committee has been trying to trace the origins of the introduction of each major ingredient in solid propellant as a function of time. Mr. Geisler stated that they have a pretty good handle on the various rubber binders used and on the beginning of the use of aluminum powder fuel. They found that their knowledge of how potassium and then ammonium perchlorate evolved as propellant oxidizers is minimal. This is the place they needed my help. Even the oldest member on the history subcommittee has only a vague idea that use of KP and AP, made by American Potash in Henderson, Nevada under government support, came into use sometime in the early to mid 1950s. The committee would like the additional details that only I can provide, such as, who first conceived of using it in solid propellants, when the first mixes were made and by whom, and what was the first sizable rocket to fly with the new material. They also wanted to know how the materials became available in this country and when and at what level as well as who made this happen.

As I was the apparent principal architect of that aspect of the history, Professor Winfred A. Foster, chairman of the history subcommittee of AIAA's Solid Rocket Technical Committee, formally invited me to present a paper on the history of establishing a source of perchlorates for use in solid rocket propellants. I accepted Professor Foster's invitation and this paper represents my endeavor to fulfill that commitment.

Early History of Perchlorates

The history of Perchlorates[1] can be traced back to 1816 when Count Frederick Von Stadion synthesized potassium perchlorate in his laboratory in Germany. He identified the sparingly water soluble salt as potassium "oxychlorate" ($KClO_4$). In the course of identifying the composition and structure of the new salt, Von Stadion synthesized perchloric acid ($HClO_4$). He also prepared $KClO_4$ by electrolyzing a saturated aqueous solution of potassium chlorate ($KClO_3$) between platinum electrodes. Von Stadion recognized the potential importance of electrochemistry as an effective method of chemical synthesis.

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Member AIAA, Member Emeritus, The Electrochemical Society, Inc., Member Emeritus, American Chemical Society

G.S. Serullas reported his discovery of ammonium perchlorate (NH_4ClO_4) and other salts of $HClO_4$ in 1831.G.S. Serullas can be credited with popularizing the name "perchlorate" to replace the name "oxychlorate" used earlier by Count Von Stadion and others.

During the next 60 years continuing basic research by many investigators provided a great treasure of knowledge of the physical and chemical characteristics of $HClO_4$ and its salts. This research effort was driven to a great extent by scientific curiosity, but there was also a desire to find practical new uses for perchlorates in certain applications including analytical chemistry, pyrotechnics, and explosives.

The natural occurrence of $KClO_4$ in Chilean nitrate fertilizer as reported by H. Beckhurts in 1886 and by B. Sjollema in 1896 is evidence that $KClO_4$ was synthesized by a natural process probably in ancient times long before F. Von Stadion did so in 1816.

Other than H. Kolbe's verification[2] of F. Von Stadion's early work on electrolysis of chlorate to perchlorate very little work was done on electrolytic methods of production and use until about 1890. In 1890 O.F. Carlson, founder and president of Stockholms Superfosfat Fabriks AB (Fosfatbolaget), applied for a patent covering electrolytic cells without diaphragms for production of sodium chlorate ($NaClO_3$). The formation of perchlorate in these cells was observed. Research to find conditions favorable to formation of perchlorate in these cells was intensified in the laboratory and in the pilot plant that Fosfatbolaget built in 1892. A commercial plant was constructed in 1893 in Mansbo, Sweden. Small quantities of sodium perchlorate were produced in 1898 with varying results. Sometimes a bath of $NaClO_3$ could be oxidized to $NaClO_4$ in 5 to 6 days. Other times the process lasted many weeks. Production was running smoothly in 1904. Sodium perchlorate is chemically converted into either ammonium perchlorate (AP) or potassium perchlorate (KP).

Ammonium perchlorate was produced in small quantities in Mansbo, Sweden in 1895. This product was intended for use as an ingredient in a new type of explosive for which O. F. Carlson was granted a patent in 1897. The well-known explosive "Carlsonit" was developed later and was covered by patents in many European countries and the United States.

France, Germany, Switzerland and the United States began producing perchlorates following Sweden's pioneering work. Sodium, potassium and ammonium perchlorates were produced in Chedde, France in 1901. Electrochemie Turgi began production of perchlorates in Switzerland in 1907. Oldbury Electro-Chemical of Niagara Falls, New York started production of sodium perchlorate in 1908 and KP in 1910. Total world production of perchlorates was estimated to be in the range of 2000-3000 metric tons/year at the beginning of WW I in 1914. During WWI Chemical Works Griesheim, located in Bitterfeld, Germany, produced about 20,000 metric tons/year of perchlorates for various military uses. Fosfatbolaget constructed a new plant in Trollhatten, Sweden in 1915 with annual production capacity of 750 metric tons of AP and 475 metric tons of KP. Although demand for perchlorates declined after WWI, that trend reversed as WWII approached and new uses for perchlorates were developed. Cardox Corporation designed and constructed a potassium perchlorate production plant in Claremore, Oklahoma in 1941. Cardox designed and constructed a larger potassium perchlorate plant in Claremore in 1943 for the Defense Plant Corporation (DPC). Cardox operated these plants to supply potassium perchlorate for own requirements for use in a new type of coal mining device.

Western Electrochemical Company, Inc. (WECCO) constructed and operated a pilot plant in 1940 to produce experimental quantities of potassium chlorate ($KClO_3$) (Fig. 1). WECCO constructed and 4,5 operated a commercial $KClO_3$ plant[3] in 1941 (Fig. 2). I designed both plants that were contructed in Los Angeles, California. Product from these two plants was sold to the match industry and to the U.S. Army for use in small arms ammunition. WECCO constructed a pilot plant I designed in 1943 in Los Angeles, California to produce KP and AP.

Vick Chemical Company constructed and operated a small AP plant in1943 in Greensboro, North Carolina. The Cardox plants and the Vick plant were operated for a short time and were then shut down, and dismantled.

Perchlorates for Solid Rocket Motors

I was working in my office at WECCO in Los Angeles, California on a quiet Saturday afternoon in December 1942 when I received a telephone call from a man who identified himself as Jack Parsons of the Guggenheim Aeronautics Laboratory at the California Institute of Technology in Pasadena, California (GALCIT). Mr. Parsons told me that GALCIT was working on a new classified military project that if successful would require very large quantities of perchlorates. He said he knew that WECCO was in commercial production of potassium chlorate and wanted to know if we would possibly be interested and capable of producing KP and other perchlorates for

GALCIT's research project. He went on to say that GALCIT was interested in developing a reliable domestic source of perchlorates. I told him that since producing perchlorates was part of our original business plan, we would be very interested in exploring the possibilities with GALCIT. Jack Parsons then invited me to attend a meeting in their office on Colorado Blvd. in Pasadena for further discussion on the following Monday morning.

I met Jack Parsons and others including Prof. Theodore Von Karman and Dr. Frank Molina in that meeting. After much discussion and many questions, it was decided that I should submit a proposal to design, construct and operate a small pilot plant to produce experimental quantities (100 to 200 lbs) of KP or AP as required by the GALCIT project.

Approximately one week later, as I recall, I submitted WECCO's proposal to the GALCIT group. WECCO proposed to perform the defined task during the next 6 to 12 months for the sum of $20,000. After much discussion and many questions our proposal was verbally accepted. Professor Von Karman's presence, his questions and comments in these meetings, were very helpful to me and revealed his keen interest in this project.

The Agreement

Late in December 1942 the Air Corps Jet Propulsion Research Project (ACJP) and WECCO signed an agreement[4] to establish a source of supply of perchlorates in Los Angeles, California. WECCO designed and constructed a small pilot plant to produce experimental quantities of KP and/or AP as required by ACJP. The project was designated GALCIT Project No. 1.

The GALCIT project pilot plant was constructed during the next few months in space provided by WECCO within the building adjacent to their existing potassium chlorate plant. The pilot plant consisted of electrolytic cells and accessory equipment to produce sodium chlorate crystals, electrolytic cells and accessory equipment to produce a purified solution of sodium perchlorate, and process equipment to chemically convert sodium perchlorate into potassium or ammonium perchlorate (Figs. 3-4).

The pilot plant operated successfully for about four months. During this time several hundred pounds of KP and AP were produced and delivered to ACJP. The pilot plant was shut down after all objectives were accomplished. Soon afterward GALCIT decided to concentrate on KP as the preferred oxidizer for composite solid rocket propellants. However, interest in AP was maintained for future development.

GALCIT Project No.1 gave ACJP special perchlorates for their research with solid rocket propellants and gave WECCO valuable experience required to design a large-scale plant for KP production.

Los Angeles Potassium Perchlorate Plant

GALCIT Project No.1 was a prelude to the formation of Aerojet Engineering Corporation and large-scale commercial development of composite solid propellant rocket motors. The first application of these solid rocket motors was for military aircraft Jet-Assisted-Take-Off devices called "JATOs". The selection of KP as the oxidizer in JATO solid propellant created a new demand for KP that grew very rapidly. In mid 1943 DPC contracted with WECCO to construct and operate a manufacturing plant to produce KP at a rate of approximately 100 tons/month. As the oxidizer in solid rocket propellants, our product had to meet designated quality specifications.

The new plant[5] was constructed in a group of existing old industrial buildings in the city of Los Angeles, California (Figs. 6-10). Start-up of the plant began in January 1944. Product from the new plant was well received by manufacturers of solid rocket motors and demand continued to grow. Construction was begun later in 1944 to double the capacity of the plant to 200 tons/month.

New Opportunities in Henderson, Nevada

On a purely exploratory visit to the Basic Magnesium Inc. (BMI) magnesium metal manufacturing plant in Henderson, Nevada in June 1944 I had the good fortune to meet Dr. Harley Lee, Technical Director of BMI operations. Dr. Lee welcomed me and said my visit was very timely. He then related a brief history of their very large-scale electrochemical and electrothermal facilities and told me that since their goals for production of magnesium metal had been met, the plant was scheduled to be shut down. Dr. Lee went on to say that they were seeking possible alternative uses for their facilities. He arranged an impromptu meeting with his staff to provide an opportunity for exchanging ideas. After that meeting I was given a comprehensive conducted tour of certain sections of the plant. I was truly amazed especially to see 10 magnesium metal plant units in a row each designed to produce 15 tons of magnesium metal per day. All 10 units appeared to be of identical design and construction except for the electrical rectifier sections. Some were equipped with mercury arc rectifiers and others with motor generator sets. I was deeply impressed with the high quality of the design, engineering and construction of these fine facilities and can recall visualizing at least one of those metal units converted into a chlorate and perchlorate manufacturing plant.

I returned to Los Angeles and immediately began to formulate a preliminary plan to convert one of the 10 magnesium metal production units into a chlorate-perchlorate manufacturing plant as I had envisioned it. Preliminary calculations indicated that one of the 10 units probably could be modified and converted to annual production of 15,000 tons of KP. I presented my preliminary plan to my business associate Kenneth Walsh and to the board of directors of WECCO. We received the board's approval to proceed with preparation of a definitive plan and a proposal to be presented to DPC and the Military Services for consideration.

Our proposal was presented to DPC in the latter part of 1944. We received notice of approval to proceed with detailed engineering and construction of a KP manufacturing facility for production at a rate of 1,200 tons/month. WECCO selected Bechtel-McCone of Los Angeles to engineer and construct the new plant.[6] Not long afterwards in my first meeting with him, Frank Case, General Manager of BMI, gave me the choice. I selected Metal Unit No. 4 as the location for the new perchlorate plant. I chose Unit No.4 because it was equipped with Westinghouse mercury arc rectifiers and was centrally located in the complex. Modification of Unit 4 and construction of the new perchlorate plant[6,7] began in March 1945 and start-up began early in July 1945 (Figs. 11-13).

Personnel

It was very fortunate that there was a large pool of experienced engineering, production and management personnel available to draw on when staffing the new Henderson perchlorate plant. These people had been employed by BMI and lived nearby in the town of Henderson, Nevada. Most of their families enjoyed living in that desert area which was an important factor contributing to the success of this project. J. Ray Coulter, an engineer with long experience in mining and metallurgy and the No. 3 man in BMI's management organization, was selected to be General Manager for WECCO's Henderson management organization. Mr. Coulter selected all of the 75 employees required to staff the operation. Fred D. Gibson, Sr., one of the original BMI group and WECCO's assistant General Manager, later replaced J Ray Coulter as General Manager of Nevada Operations. All of the original staff contributed significantly to the success of our Henderson, Nevada operations.

End of WWII – Plant Shutdown

WECCO received a telegram from the U. S. Government on August 13, 1945, the day after VJ Day, ordering us to shutdown the Henderson perchlorate plant. The plant had been in operation for about one month with barely enough time to demonstrate good performance. We were faced with an enormous problem (i.e., survival). It became necessary to immediately develop a plan to deal with the critical situation we faced.[8]

WECCO made a working arrangement with Reconstruction Finance (RFC) to recover all of the in-process chlorate and perchlorate products generated during the short period of start-up operations and to place the Henderson plant in a stand-by condition. WECCO made another working arrangement with RFC to continue operating the Los Angeles plant, with some modifications, to make it possible to produce sodium chlorate, potassium chlorate, and to test the post war market for these products. From August 15 to December 31, 1945, much to our surprise and relief, the market for our products increased substantially. In fact demand for our products exceeded production capacity of the Los Angeles plant.

Post War Realignment and Consolidation

Previous experience following WWI showed that small domestic chlorate plants could not survive in a world market because of competition from large-scale, low-cost producers located in Europe. Therefore we knew that we had to obtain control of the plant we had constructed in Henderson, Nevada or establish a new one of at least the same size or we would eventually be forced out of business. Accordingly we worked out a plan to lease the Henderson plant facilities with an option to buy and submitted this plan as a proposal to the Reconstruction Finance Corporation (RFC) for consideration. When RFC accepted our proposal early in 1946, we took immediate action to implement our plan to modify the plant to produce and market sodium chlorate, potassium chlorate, sodium perchlorate, and potassium perchlorate. In the reorganization that followed all of WECCO's manufacturing operations were transferred to Henderson, Nevada. Research and Development operations were relocated in a new laboratory in Culver City, California; executive and Marketing offices were relocated in downtown Los Angeles~~ and arrangements were made with DPC to~~

Production and sales of our products reached high levels in the late 1940s and our business began to show definite signs of survival and financial success.

AP Becomes The Preferred Oxidizer for Composite Solid Rocket Propellants

A new chapter in the history of WECCO began in 1950 when Aerojet, Thiokol and the US Navy informed us that military demand for AP could possibly grow to 50 tons/day or more in the near future. This renewed interest in AP caused us to begin an R&D project in our Culver City laboratory to design and develop a continuous process to produce AP at a rate of 50 tons/day. Details of the new process are described in a U.S. Patent No. 2,739,873 issued March 27, 1956.[9] One problem we were very concerned about in designing and engineering this new plant was safety. We were unable to find sufficient reliable information about safe handling and storage of large quantities of 50 tons or more of AP. We found it necessary to conduct experiments to determine sensitivity of AP to shock, heat and moisture as well as certain other basic characteristics. Results obtained from our research project[10] provided a sound basis for the design of the new large-scale plant.

Process Design and Engineering for a New AP Plant

WECCO signed a contract (NOAS51-1008) with the Bureau of Aeronautics of the U.S. Navy in 1950 to provide design criteria and process design for a new AP manufacturing plant to be constructed adjacent to our plant site in Henderson, Nevada. The new plant was designed for daily production capacity of 50 tons of AP crystals.

WECCO furnished basic design criteria and basic process design for the new AP plant. The Bechtel Corporation of Los Angeles and San Francisco furnished detailed process design and engineering. Haddock Engineers, Ltd. started construction of the new plant in 1952 on a plant site near WECCO's electrolytic sodium perchlorate plant and other electrolytic plants. Start-up of the new AP plant began in 1953. Production of AP at design capacity of 50 tons/day was successfully demonstrated within several months of operation. Thereafter the level of operation fluctuated depending on sales and market demand.

Major New Use for Sodium Chlorate Develops

Early in the 1950s Solvay Process Division of Allied Chemical Corp. introduced a new process for large-scale on-site generation of chlorine dioxide (ClO_2) for use in bleaching wood pulp. This new bleaching process became very successful. Its widespread use in the United States and Canada created a major new and expanding market for sodium chlorate (the starting material in the process of producing ClO_2). This new market development gradually brought 100% utilization of WECCO's sodium chlorate plant capacity and discussions with Art Chadwick and Harold Merrit of Solvay Process Division of Allied Chemical about a merger of Solvay Division, American Potash and Chemical (AP& CC) and WECCO. Although the merger did not materialize, our discussions paved the way for other merger discussions with Peter Colefax, President of AP&CC. These discussions resulted in AP&CC's acquisition of about 48% of WECCO's stock in 1954 and 100% in 1955. After the merger, I became Vice President, Research of AP&CC, the surviving corporation.

More Expansion of AP Production Capacity and Competition

A new round of expansion AP production capacity was initiated in 1957 to meet projected requirements for new military solid rocket programs, including the ICBM and POLARIS missiles. As a result, the number of solid rocket propellant motor manufacturers increased from two to seven and the number of producers of AP increased from one to four. At this time AP&CC purchased the Henderson, Nevada AP plant constructed in 1952. Unfortunately, demand for AP did not increase as projected. In fact demand increased barely enough to exceed the capacity of AP&CC's Henderson plant. The net result was a large over-capacity for production of AP and the shutdown of Hooker and Foote's (HEF) joint venture plant located in Columbus, Mississippi and Penn Salt's plant in Portland, Oregon. There were now only two major domestic producers of AP: Kerr-McGee Chemical Corporation, the surviving company of the merger of Kerr-McGee Corporation and AP&CC in 1967, and Pacific Engineering and Production Company of Nevada which was founded by Fred D. Gibson, Sr. and other former employees of WECCO.

More Solid Rocket Motors

Late in the1960s it appeared that demand for AP had peaked and would probably hold steady except in time of national emergencies. Then the Space Shuttle came along and changed the outlook again. The real stars of the space shuttle propulsion system are two solid rocket boosters which stand 115 feet tall and measure 12 feet in diameter These boosters provide more than three fourths of the thrust necessary to lift the space shuttle during the 120 seconds they are fired. The three main engines burning liquid hydrogen and liquid oxygen produce more than 1.1 million pounds of thrust at lift off. I am told that at full power this is the equivalent of 23 Hoover Dams. The two solid rocket boosters together provide an additional 5.8 million pounds of thrust needed to lift the 4.5-million pound space shuttle off the ground. Each booster is loaded with more than 1 million pounds of solid rocket propellant containing about 70% of AP. Therefore each flight of the space shuttle consumes about 1.5 million pounds of AP. I expect that AP will continue to be an important part of civilian and military programs involved with exploration and colonization of space for many years into the future. I know of no reason why AP production cannot keep pace with future demand whatever that may be.

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10. Schumacher, Joseph C., and Stern David R., "Large-Scale Continuous Production of Ammonium Perchlorate," *Chemical Engineering Progress*, Vol. 53 No. 9, 1957, pp. 428-432.

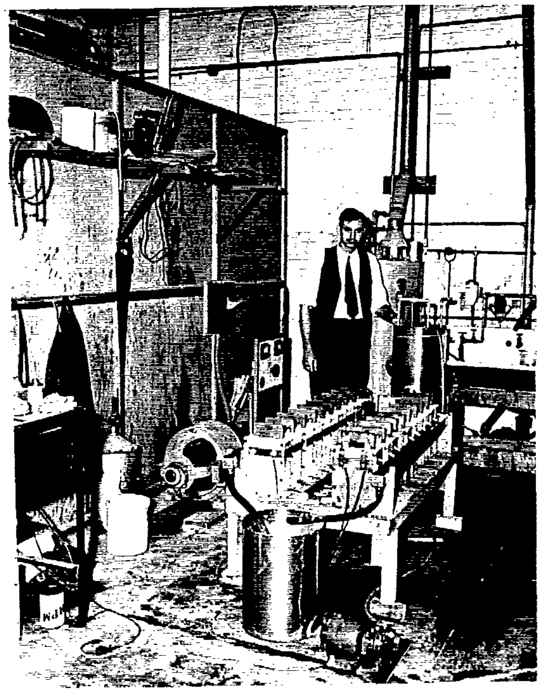

Figure 1. $KClO_3$ Pilot Plant

Figure 2. Los Angeles $KClO_3$ Production Plant 800 Amp Electrolytic Cells

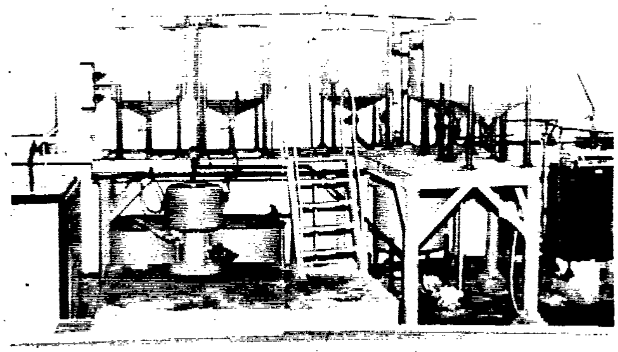

Figure 3. Los Angeles Potassium and Ammonium Perchlorate Pilot Plant

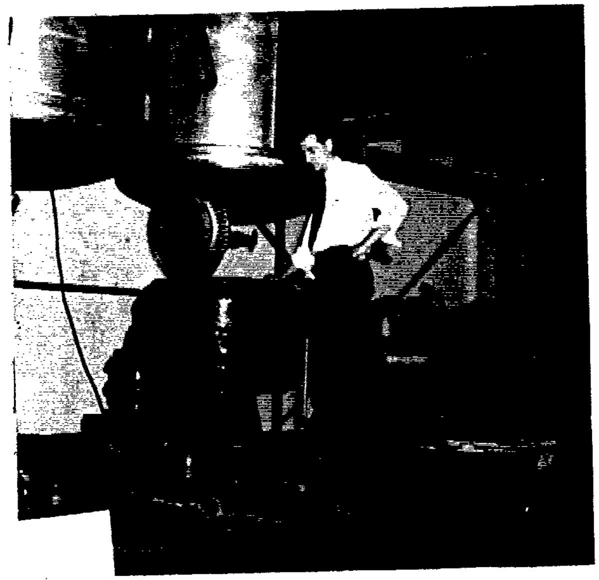

Figure 5. Los Angeles $KClO_3$ Plant Crystallizers and Centrifuge

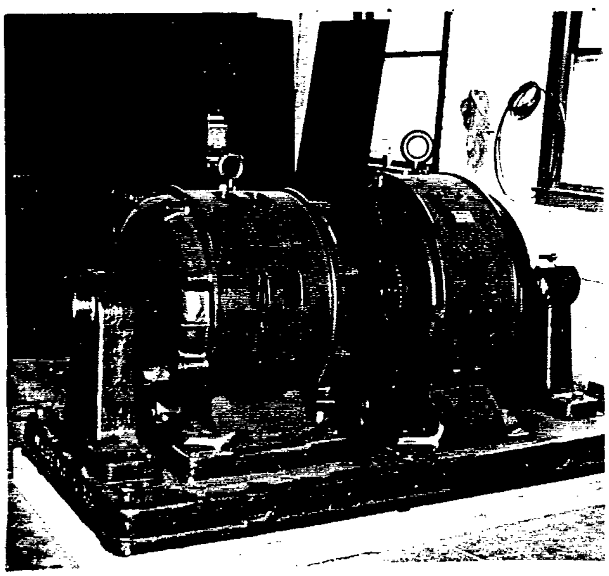

Figure 4. Los Angeles $KClO_3$ Plant Motor-Generator Set

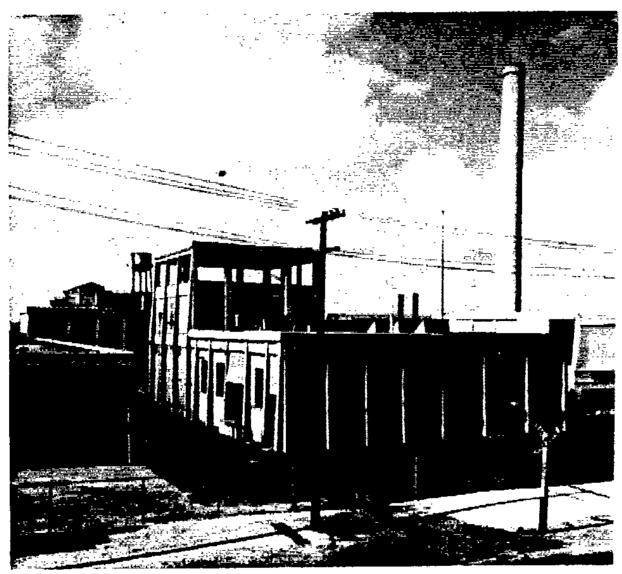

Figure 6. Los Angeles Potassium Perchlorate Plant

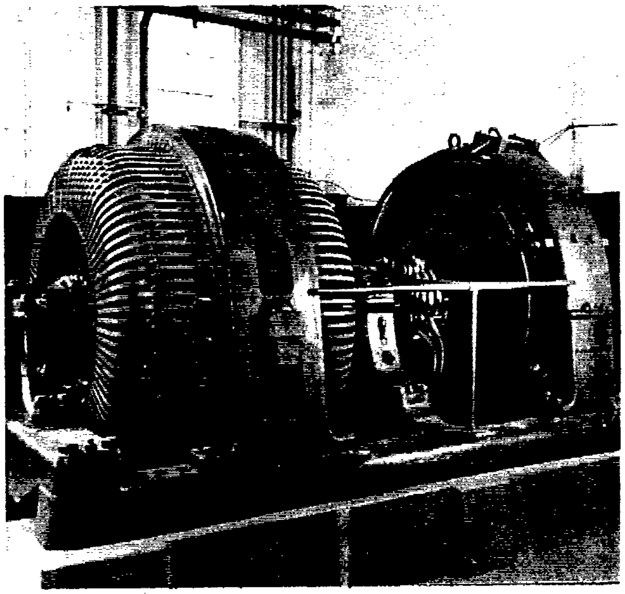

Figure 7. Los Angeles $KClO_4$ Plant
Motor-Generator Set

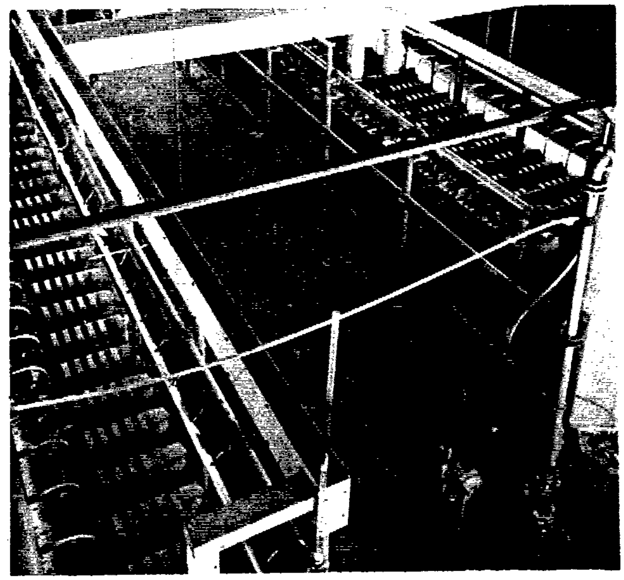

Figure 9. Los Angeles $KClO_4$ Plant
Sodium Perchlorate Cells 2500 Amps

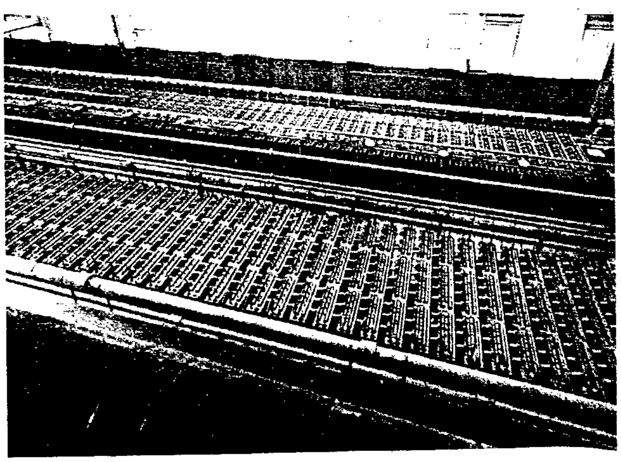

Figure 8. Los Angeles $KClO_4$ Plant
$NaClO_3$ Cells – 2500 Amps

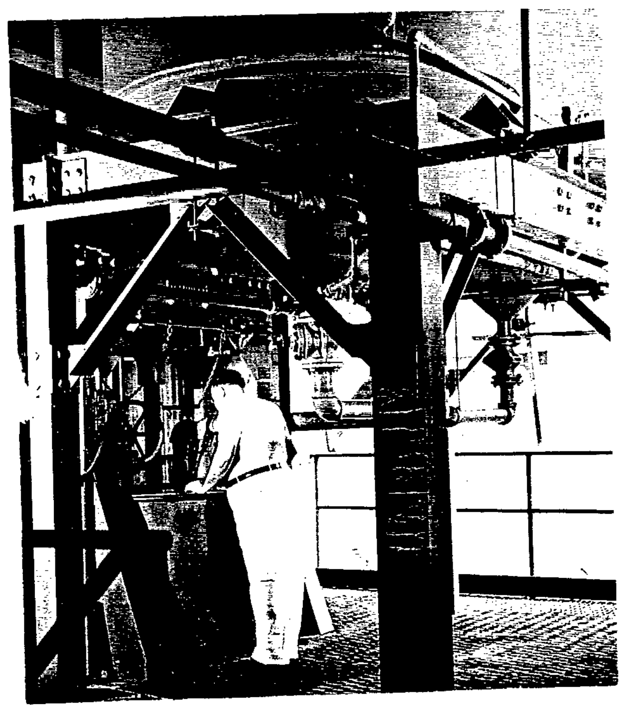

Figure 10. Los Angeles $KClO_4$ Plant
Processing Equipment

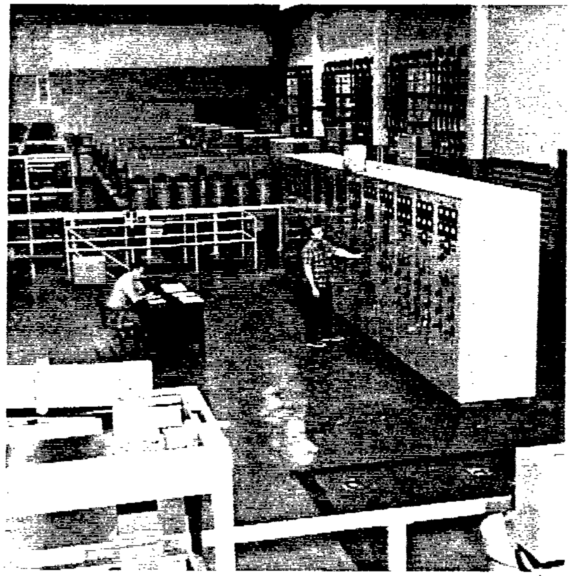
Figure 11. Mercury Arc Rectifiers and Controls
Henderson, Nevada Plant


Figure 12. WECCO Henderson, Nevada $KClO_4$ Plant
5000 Amp Electrolytic Chlorate and Perchlorate
Cell Room

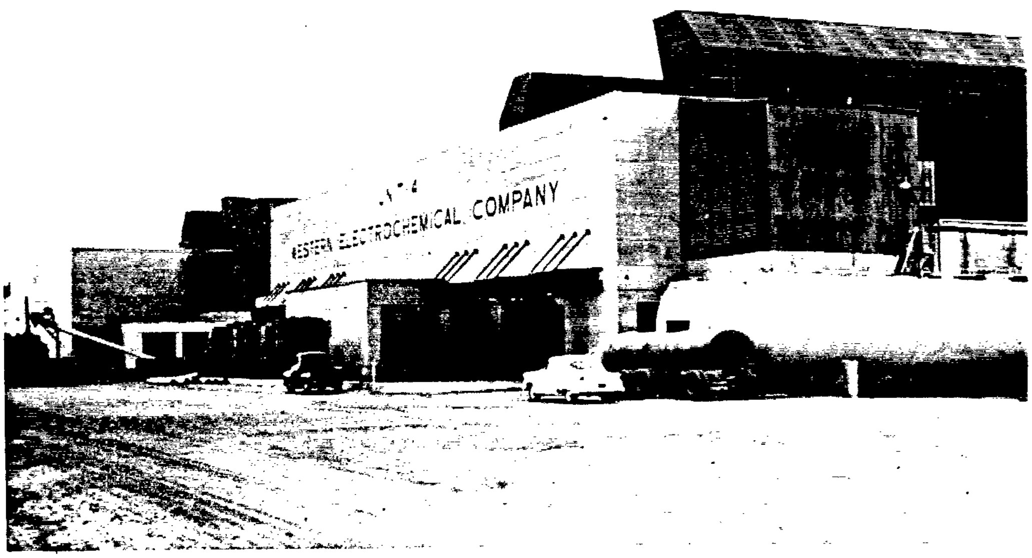

Figure 13. WECCO Henderson, Nevada Perchlorate
Plant Transformer Substation