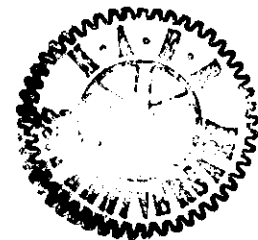


UNITED STATES NITRATE PLANT NUMBER 2
Tennessee Valley Authority
Reservation Road
Muscle Shoals
Colbert County
Alabama

HAER No. AL-46



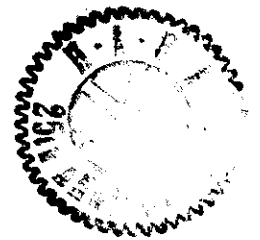
HAER
ALA
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PHOTOGRAPHS
REDUCED COPIES OF MEASURED DRAWINGS
WRITTEN HISTORICAL & DESCRIPTIVE DATA

Historic American Engineering Record
National Park Service
Department of the Interior
P.O. Box 37127
Washington, D.C. 20013-7127

HISTORIC AMERICAN ENGINEERING RECORD

UNITED STATES NITRATE PLANT NUMBER 2



HAER No. AL-46

HAER
ALA
17-MUSHO
1-

Location: Reservation Road, Muscle Shoals Alabama

Date of Construction: 1918

Designer/Engineer: Air Nitrate Corporation

Builder/Fabricator: Westinghouse, Church, & Kerr Company

Present Owner: Tennessee Valley Authority

Present Use: Environment Research Center

Significance: Production of Ammonium Nitrate

Project Information: This recording project is part of the Historic American Engineering Record (HAER), a long range program to document the engineering industrial and transportation heritage of the United States. The HAER program is administered by the Historic American Buildings Survey/Historic American Engineering Record (HABS/HAER) Division of the National Park Service, U.S. Department of the Interior. The Tennessee Valley Authority-Muscle Shoals Recording Project was cosponsored during the summer of 1994 by HAER under the general direction of Robert J. Kapsch, Chief of HABS/HAER and by the Tennessee Valley Authority with the staff of the Tennessee Valley Authority's Environmental Research Center, Muscle Shoals, Alabama. The field work, measured drawings, historical report, and photographs were prepared under the direction of Eric N. De Lony, Chief of HAER and Project Leader; Richard O'Connor, Project Historian; Jet Lowe, HAER Photographer; and Craig N. Strong, Project Architect. The recording team consisted of Tom Behrens, Field Supervisor; Balazs Krikovszky (ICOMOS) and Sergio Sanchez, Architects and Susie B. Leong, Illustrator.

Project Historian: Brian F. Coffey

Preface

Built as a war measure for the production of ammonium nitrate, a key component in high explosives, U.S.N.P. No. 2 became one of the largest plants of its kind with a capacity to produce 110,000 tons of ammonium nitrate per year. The plant and its adjoining industrial town were hurriedly constructed in a nine month period between February and November of 1918 with little regard to cost. After two brief periods of production, one toward the end of 1918, the other in the February of 1919, the 348 acre manufacturing site lay idle for the next fourteen years while Congress and private industry wrangled over bids that had less to do with the nitrate plant than they did with the hydro-power of the adjoining Wilson Dam.

The focus of this report will be on the equipment and processes of U.S.N.P. No. 2. Special attention is paid to the genesis and evolution of the chemical and electrothermal industries that culminated in the building of the plant, and the continuing technological growth that made U.S.N.P. No. 2 both a resting place for obsolete technologies, and a birthplace for new.

A Technological History of United States Nitrate Plant Number 2

Introduction

"This is a business man's war and the genius of business must be mobilized for democracy."¹

In a 1918 address in Philadelphia promoting the Third Liberty Loan, former President Taft stated the basic concept driving wartime industry in the United States. One such industry, the fixation of atmospheric nitrogen, depended entirely on recently developed commercial enterprise. In just over a decade before the war, the fixation of atmospheric nitrogen had grown from laboratory experiments to a thriving commercial industry. But with the exception of one North American company, the commercial fixation industry was centralized in western Europe, particularly in Germany, soon to become the war's most feared belligerent. As a component in the industrial process for the production of explosive-grade ammonium nitrate, the cyanamide

¹ Quoted in: C. G. Schluederberg, "The Part The United States Industries Must Perform to Enable the Allies to Win the War" Transactions of the American Electrochemical Society 33, (1919): 53.

process of nitrogen fixation had its roots in Germany's chemical fertilizer industry, and it was there that the technology crucial to America's nitrogen fixation program was stealthily acquired just prior to the war.²

Two men, Frank S. Washburn and Walter S. Landis of the American Cyanamid Company, were key figures in establishing the only industrial-scale nitrogen fixation facility in North America. In 1909, Frank Washburn, President of American Cyanamid, built a cyanamide fertilizer plant on the Canadian side of Niagara Falls, a growing center of electrothermal chemical production. American Cyanamid's efforts in the commercial development of nitrogen fixation were rewarded in 1917 when the War Department selected the company to construct and operate one of the largest nitrogen fixation plants in the world at Muscle Shoals, Alabama. The nitrogen fixation industry, according to Harry A. Curtis, Tennessee Valley Authority's revered chemist and research director, fell between "the extreme roles of fostering life and destroying it."³ Before World War I, the fixation of atmospheric nitrogen was directed towards fertilizer production. Nitrogen, whether produced organically or synthetically, is the main constituent of most fertilizers, and "fixing" the nitrogen in some other substance which could then be used as a fertilizer drove experimentation and invention to that end. Nitrogen is also a constituent of most explosives, and as the "great war" drove the need for high explosives far beyond expectation, nitrogen fixation consumed the efforts of experimenters and inventors with renewed vigor.

U.S.N.P. No. 2 grew out of a vibrant period of world-wide chemical and electrothermal experimentation and commercial chemical activity prior to World War I. The production of calcium carbide, the development of the acetylene industry and oxyacetylene welding, the production of liquid air, and the emergence of cyanamide fertilizer all matured as commercial industries in the first decade of the twentieth century. Commercially successful and historically linked, they formed the technological basis for what was to become one of the world's largest nitrogen fixation facilities. World War I was indeed a business man's war, and U.S.N.P. No. 2 is very much a part of the history of the commercial chemical industry that preceded it.

The large industrial complex that formed U.S.N.P. No. 2 was,

² Williams Haynes, American Chemical Industry, vol. 3, (New York: D. Van Nostrand Company, 1945), 83.

³ Harry A. Curtis, Fixed Nitrogen, (New York: The Chemical Catalog Company, 1932), 11.

as Curtis noted, "an assembly of several smaller plants or sections, each turning out an intermediate product, subsequently used in the process."⁴ The concept of "an assembly of smaller plants" is fundamental to understanding U.S.N.P. No. 2 and serves to organize an otherwise complicated array of processes and equipment. Existing industrial processes that already had made their mark in commercial enterprise -- calcium carbide, liquid air, calcium cyanamide, cyanamide ammonia, and nitric acid from oxidized ammonia -- were all manufactured to varying degrees in the decades preceding construction of U.S.N.P. No. 2, and were integral to the development of nitrate production at Muscle Shoals.

These constituent chemical products in turn depended upon recently developed and refined chemical processing equipment. The commercial production of calcium carbide, the first of a number of intermediate products produced at U.S.N.P. No. 2, was possible only after development of the electric furnace. Manufacture of calcium cyanamide, the next product, depended on a unique electric oven that took advantage of the exothermic nature of the nitrification of calcium carbide. The extraction of nitrogen from air grew out of recent developments in air liquefaction based on fractional distillation. Ammonia, a longtime by-product of the destructive distillation of coal, was recovered from cyanamide with a specially designed pressure-vessel known as an autoclave. Finally, the ammonia was oxidized to produce nitric acid in a unique device known as a catalyzer, an apparatus that utilized the precious metal platinum as its key component.

These technologies and their respective apparatus became commercially successful in a short period of time, from 1900 to the construction of U.S.N.P. No. 2 in 1918. Furthermore, manufacturing had refined equipment and designs to allow the rapid construction and assembly of large-scale industrial plants, crucial to wartime munitions program. This technological reliability persuaded the War Department to select the cyanamide process.

The Electric Furnace and Calcium Carbide

Electrothermal experimentation dates to the early research of Davy in 1810 and progresses through the work of Pepys,

⁴ Harry A. Curtis, Plan for the Rehabilitation of the Ammonium Nitrate Production Facilities at U.S. Nitrate Plant No. 2. Special Report # 29. (Tennessee Valley Authority, 1937) 2.

Siemens, Berthelot, Moissan and many others.⁵ The first patented production of calcium carbide is credited to the American Thomas Willson and James T. Morehead his business partner in the Willson Aluminum Company. Working on the production of aluminum which involved the intermediate production of metallic calcium, they reduced lime and carbon in an electric furnace. The final results of this experiment were illuminating both figuratively and literally. As one of the company's partners explained:

In an attempt to produce metallic calcium which should in turn react with Al_2O_3 to produce Al in a second furnace, Willson charged the furnace with a lot of lime and carbon. He did not, of course, get any metallic calcium, but the next morning, after the furnace had cooled sufficiently, the charge was thrown on a heap in the yard. An noon time, after a heavy rainstorm had set in, one of the employees, going out to lunch, lit his pipe and threw the burning match unintentionally onto the heap of stuff from the furnace. Lo and behold, the heap of black "stones" took fire! To ascertain what we had produced, Prof. Kinnicutt, of Worcester Polytechnic Institute, and Prof. F. P. Venable, of North Carolina, were called in to confirm our laboratory test. The black mass proved to be calcium carbide, CaC_2 , and the gas which had taken fire, acetylene, C_2H_2 .⁶

These events established two important principles underpinning new industries:

1. Calcium and carbon combine at high temperature to form calcium carbide.
2. Calcium carbide reacts with water to form the combustible gas acetylene.⁷

⁵ For a complete analysis of early electric furnace history and technology see: J. N. Pring, The Electric Furnace, (London: Longmans, Green and Co., 1921)

⁶ August Eimer, "Early Days in the Carbide and Ferro-Chrome Industries" Transactions of the American Electrochemical Society 51 (1927): 74-75.

⁷ If this latter reaction had not been noted, calcium carbide may well have remained just a curious product of high temperature, furnace experimentation. Acetylene, the gas that was produced at Willson Aluminum, was to become the basis for a major commercial industry, commercial gas lighting.

As calcium carbide and its by-product, acetylene gained exposure in the United States, European experimentation followed a parallel course. Frenchman Henri Moissan, an important figure in electrothermal invention and experiment, established himself as the overseas analogue to Willson. In a paper he submitted to the Academie des Sciences in 1894, he stated:

In this work, I showed that at the high temperature of the electric furnace there can exist only one compound of carbon and calcium, which is crystalline; I established the formula by analysis, and studied its properties. I showed that this substance decomposed cold water, forming absolutely pure acetylene gas - the beginning of the acetylene industry. In this connection I ought to give a word of credit for the industrial research to Wilson, an American engineer.⁸

Although Moissan was gracious enough to tip his hat to the American, he goes on to state that Willson's patent description was "without scientific value" and complained to the reader that he was the first to discover acetylene from calcium carbide.⁹ Willson and Morehead patented their electric furnace and newly discovered electrothermal process for acetylene production in the United States, and Moissan patented his work in Europe.¹⁰ Despite the lack of equipment to generate acetylene gas or devices for using acetylene, numerous carbide plants were built in Europe and the United States utilizing the electrothermal methods of Willson and Morehead. In the absence of acetylene-based lighting apparatus, many of these early plants failed commercially; nonetheless, electric furnace production of calcium carbide became firmly entrenched in this country and in Europe.¹¹

Eventually, the development and promotion of workable acetylene lighting apparatus permitted the dissemination of pioneer developments in carbide production through a number of successful commercial interests, leading most notably to the formation of Union Carbide in 1898. Other companies that shared

⁸ Henri Moissan, The Electric Furnace (Easton, PA.: The Chemical Publishing Company, 1920), 213.

⁹ Moissan, 214.

¹⁰ Haynes, *ibid.* p. 430.

¹¹ According to one author, the year 1899 saw a total of eighty-six carbide manufactories in Europe alone. See: Pring, 12-13.

in the growth of acetylene-based industry were manufactures of bicycle and automobile headlamps, rural lighting systems, and miners lanterns and helmets.¹² The rush to acetylene lighting created an "acetylene boom", and carbide manufacture became the largest consumer of energy in the electric furnace industry.¹³ In its early phase, over-production severely embarrassed carbide manufacturers, resulting in the closing of several plants.¹⁴ Nonetheless, carbide and acetylene found market niches: urban acetylene lighting plants in America grew from 4 in 1898 to 202 in 1902.¹⁵ More important, the growth of the commercial carbide and acetylene industries fostered a drive towards new, more efficient electric furnaces as industrialists began searching for ways to increase production. New furnace designs multiplied, not only for carbide production but for the smelting of metals as well. Throughout the period, starting with Willson Aluminum and culminating with U.S.N.P. No. 2, the industry improved all components of the electric furnace, from hearth to the electrodes. Willson's single electrode furnace was superseded by multiple-electrode design; arc-type furnaces waned in popularity as resistance furnaces gained favor; capacities increased, as did the amount of electrical current employed; single phase operation moved to three phase.

In conjunction with these developments came improvements in materials-handling equipment. Crushers, conveyers, and other components of raw material processing kept up with the improvements in the electric furnace. The electrode of Willson Aluminum, made by that company, was 4" square, the largest size then available; by 1918, electric furnace electrodes were measured in feet, installed in the carbide furnaces at U.S.N.P. No. 2 were 16" thick and 6 1/2' long.¹⁶ This growth in the size of electrodes paralleled growth in the industry that made them.

¹² Haynes, vol. 6, 429-435.

¹³ Jocelyn Field Thorpe, Thorpe's Dictionary of Applied Chemistry 4th ed., vol. 2. (London: Longmans, Green and Co., 1947), 216.

¹⁴ C. J. Brockman, "Twenty-Five Years of Electrochemistry" Transactions of the American Electrochemical Society 51 (1927): 87.

¹⁵ "Calcium Carbide and Acetylene" Electrochemical and Metallurgical Industry 4 (January 1906): 27.

¹⁶ C. L. Mantell, Industrial Carbon (New York: D. Van Nostrand Company, 1946), 200; Tennessee Valley Authority, Nitrate/Phosphate Activities at Muscle Shoals: 1918-1933 (Office of Agricultural and Chemical Development 1984) p.40

The typical early electrode industry was as follows: employed a small crusher for petroleum coke, a mixing pan, a small brick oven, iron molds, and a hand operated grinding and bolting outfit.¹⁷ Firms such as The National Carbon Company and Acheson Graphite Company enlarged and modernized electrode production, eventually using the electric furnace itself to turn out new and better electrodes.¹⁸ In addition to those made in the U.S., many electrodes needed for electric furnace work were imported from Germany and Sweden.¹⁹ A German method of calcining anthracite coal in an electric furnace was used by a Canadian firm to produce the first electrodes for the carbide works at American Cyanamid.²⁰

The fundamental distinction between the more common combustion-furnace and the electric furnace is that the latter produces much higher temperatures. As electrical technology evolved in the late nineteenth century to make available new methods and magnitudes of electric current production, experimentation in electrothermal chemistry naturally followed. In the context of U.S.N.P. No. 2, this unfolding of electric furnace design, experimentation, and commercial/industrial application laid the foundation for its Carbide Furnace Department.

Shortly after Willson patented his electrothermal process for producing calcium carbide, Willson Aluminum began commercial production of the product at Lynchburg, Va.²¹ The commercial version of the furnace consisted of a single electrode protruding vertically into a small hopper car filled with a charge of lime and coke. The furnace was of the "smothered arc" type, where an electric arc formed between a negative and a positive pole (electrode) was "smothered" by the charge, and melted to form calcium carbide. In the Willson furnace, the base of the hopper

¹⁷ Mantell, 195.

¹⁸ Haynes, vol. 6 , 434-435. Haynes' company history of Union Carbide provides an excellent overview of the related technologies and commercial interests surrounding carbon manufacture, acetylene production, air liquefaction, and the electrothermal industry.

¹⁹ Mantell, 193-202. Mantell's chapter "Historical Aspects of Manufactured Carbon" provides good background on the history of electrode manufacture.

²⁰ Mantell, 201.

²¹ Eimer, 75.

was made of carbon and formed the negative electrode.²² The charge melted from the bottom up and calcium carbide slowly filled the hopper. As the electrode was slowly raised more lime and coke were added.²³ Although this turn-of-the-century furnace used single-phase, direct current and only one electrode, in principle it strongly resembled those at American Cyanamid and U.S.N.P. No. 2. The latter furnace used a 3-phase alternating current and 3 large electrodes.

Probably the most important innovation in carbide production was the move from "intermittent" furnaces like that used by Willson to the "continuous" furnace. In the former, the carbide cooled inside the furnace and was forcibly removed in hardened form. In the continuous furnace, carbide was tapped in liquid form. This allowed simultaneous charging and tapping, resulting in 24 hour operation; only the replacement of partially consumed electrodes interrupted furnace operation. The first commercially successful continuous furnace, that of the German inventor, Helfenstein went into operation in 1906.²⁴ This furnace used a three-phase current supply and may very well have been the basis for the later installation at American Cyanamid.²⁵

One troubling feature of carbide production that remained throughout operations at U.S.N.P. No. 2, was furnace charging. Despite drives to mechanize all operations, the furnace department of any carbide manufactory depended on manual labor to charge requisite amounts of coke and lime. Hot, miserable, and often injurious, the job of charging the open hearth furnaces of the type used for carbide production resisted mechanization. The erratic nature of heat transferal within the furnace necessitated hand feeding to maintain uniform carbide production. At times, bursts of carbon monoxide flame would have to be smothered with more charging material.²⁶

Further improvements, however, made carbide production more efficient. Electrode holders, once just copper clasps screwed to the head of the electrode, became large water cooled, cast iron

²² Pring, 102.

²³ Pring, 102.

²⁴ Dr. Bruno Waeser, The Atmospheric Nitrogen Industry. vol.2 (Philadelphia: P. Blakiston's Son & Co., 1926) 340.

²⁵ A detailed description of this type of three-phase furnace can be found in the appendix of this report.

²⁶ Waeser, vol. 2, 341.

apparatuses. Transformers grew in complexity to provide varying current densities to handle irregularities in furnace heating. By the eve of the war, electrothermal production of calcium carbide had grown and modernized to such an extent that the furnace of Willson was viewed an historical curiosity.

Thus, the accidental fusion of calcium and carbon in a small electric furnace, and the more controlled experiments of a European scientist gave birth to new industries, industries that shaped the Carbide Department of U.S.N.P. No. 2. Simultaneously, the development of the liquid air industry in conjunction with these industries, linked the carbide and acetylene industries, and became a major industry of its own.

Liquid Air

In 1895, french chemist Henri Le Chatelier produced a gas flame that burned at a temperature of about 6,300 degrees F., hotter than any previous man-made flame. Chatelier had blended the newly available acetylene gas with oxygen, creating a flame that had immediate commercial application in the new welding industry, the previously time-consuming task of cutting steel, and, because the flame could also be used under water, in ocean-bottom salvage.²⁷ Soon after Chatelier's discovery, two other French inventors developed an appropriate torch, and commercial welding and a new oxyacetylene industry soon followed.²⁸ Crucial to the new industry -- sources of large amounts of oxygen and the large-scale production of oxygen -- depended on enlarging and improving methods for separating the two main constituents of air.

The first commercially successful apparatus for large-scale oxygen production and, in turn, nitrogen production, is attributed to Carl Von Linde, a German professor of the Polytechnic School of Munich.²⁹ The basis of Linde's oxygen production was the separation of gas by physical, rather than chemical, methods. Before Linde developed his fractional distillation process and equipment, the previous method, dating to the mid-nineteenth century, passed air through retorts filled with a heated substance called baryta, an oxide of barium. Baryta absorbed oxygen creating barium peroxide that, when heated

²⁷ Haynes, vol. 6., 5.

²⁸ Haynes, vol. 6., 5.

²⁹ J. R. Partington, L. H. Parker, The Nitrogen Industry (London: Constable & Company Limited, 1922), 6.

to an even higher temperature, gave off its oxygen.³⁰ This method, known as the "Brins Process," was controlled by the Brins Oxygen Company, later to become the British Oxygen Company, the first commercial oxygen producer.³¹ British Oxygen thrived well into the twentieth century, proving that this chemical method of oxygen production could hold its own. However, Linde's physical method eventually dominated oxygen production.

In contrast to the Brins Process, the physical method of oxygen production involved air liquefaction and its subsequent separation. Scientifically established by Joule and Thompson in 1845, the liquefaction was accomplished by compressing the air and later cooling it through expansion. The differing boiling points of nitrogen and oxygen, 196°C versus 183°C made the fractionation of the two gases, after liquefaction, relatively simple. In 1895, Carl Linde produced several liters of liquid air per hour in his laboratory in Munich. Eventually, Linde modified his apparatus to produce oxygen of high purity, and the "Linde Process" was born.³²

While Linde was beginning commercial production of oxygen, another inventor, Georges Claude, modified Linde's system of fractional distillation, developed his own method and equipment, and established in Paris in 1902 L'Air Liquide (the Liquid Air Company).³³ The "Claude Process," differed from the Linde Process in that, rather than allowing the heat of the compressed air to dissipate through simple expansion or outflow, the expanding air was forced to operate an "expansion machine," nothing more than small piston motor. In performing this "external work," even more heat was lost, leading to rapid cooling and liquefaction of the air. Claude's company furnished expertise and equipment in later liquid oxygen installations in France and Italy.³⁴

³⁰ Thorpe's Dictionary, vol. 9, 182.

³¹ Thorpe's Dictionary, vol. 9, 182.

³² Thorpe's Dictionary, vol. 9, 182.

³³ The reader will find an excellent description of both the Linde and Claude processes in: Thorpe's Dictionary vol. 7, "Liquification of Gases" pp.333-346, and Thorpe's Dictionary vol. 9, "Oxygen, Production of" pp.182-185. For an even more thorough treatment of the subject, see: Georges Claude, Liquid Air, Oxygen, Nitrogen, (Philadelphia: P. Blakiston's Son & Co., 1913).

³⁴ Waeser, vol. 2, 527.

At the time of Linde's and Claude's commercialization of their air liquefaction equipment, it was oxygen, not nitrogen, that was most in demand. Simultaneously, however, other German scientists made discoveries that caused Linde and Claude to spend equal time on nitrogen production. This potential for large-scale nitrogen production was noted by Claude:

As to nitrogen, if it does not possess as many chords to its bow, it has one which is indubitably its very own, and which enables it to dispense with all else; it is the essential basis of manures - that is, of a business which, though still in its infancy, attains already a yearly turnover of more than 500 million francs.³⁵

The profitable business to which Claude was referring was that of cyanamide fertilizer, and for L'Air Liquide, the new fertilizer industry would be a great benefit.

Cyanamide

The roots of commercial cyanamide production were set firmly in Germany in the late nineteenth century and, like the discovery of calcium carbide, calcium cyanamide was also the unintended result of commercial, industrial experimentation. The discovery of calcium cyanamide is attributable to parallel and simultaneous innovation that grew out of the constant experimentation that characterized the expanding chemical industry of the late nineteenth century and early twentieth century. In particular, Henri Moissan's attempt to nitrogenize pure calcium carbide in one of his electric furnaces in 1894 failed, but his line of reasoning, that of fixing nitrogen in calcium carbide, was on target.³⁶

Calcium cyanamide was finally emerged from attempts to produce cyanides from the alkaline earth metals. Such cyanides were used in the extraction (cyanidization) of precious metals, especially gold, from their constituent ores. German industrial chemists Rothe, Adolph Frank, and Nikodem Caro, figured prominently in these experiments where nitrogen was introduced into the electrothermal process. In trying to produce calcium cyanide in an electric furnace, Frank noted an intermediate product, calcium cyanamide that, while not a cyanide, did absorb nitrogen.

³⁵ Claude, 291.

³⁶ Waeser, vol. 1, 25.

Working along with the elder Frank was his son Albert who, like his father, observed that calcium cyanamide served no purpose in metal extraction. Yet Albert suggested to his father the possibility of using the calcium cyanamide as a nitrogenous fertilizer. With this goal in mind, Frank continued his experiments with the firm of Siemens and Halske in Berlin.³⁷ Frank sent his new nitrogenous material to academics stationed at German agricultural stations to see if calcium cyanamide could indeed compete with the long-established Chilean nitrate fertilizers.³⁸ In describing the results of this analysis, a professor from the University of Kansas brought to light important aspects of cyanamide that would have a great impact upon the world chemical industry:

The result of this experimentation has established beyond all question the fact that, under certain conditions, calcium cyanamide is a better fertilizer than the sulfate of ammonia from the gas-works, and practically equal to the saltpetre from the mines, weight for weight of the nitrogen it contains.

The exact mechanism of its action is still to be determined. It is not unlikely that the calcium cyanamide in the soil breaks down into cyanamide itself, which in turn decomposes into ammonia, which oxidizes into nitric acid, and the nitric acid so formed unites with the lime constituent of the compound to form calcium nitrate.³⁹

The research of Adolph Frank and Nikodem Caro had also generated other important discoveries that would impact the technology of the chemical industry. In 1900 Frank patented the production of ammonia by mixing water with cyanamide.⁴⁰ Caro discovered the exothermic nature of the nitrogenation process, thereby reducing significantly the cost of cyanamide production.⁴¹ In the

³⁷ Curtis, Fixed Nitrogen, 81.

³⁸ Robert Kennedy Duncan, "The Fixation of Nitrogen" extracted in pamphlet form from: The Chemistry of Commerce (Harper & Brothers, 1907): 59-61.

³⁹ Duncan, 61.

⁴⁰ Waeser, vol. 1, 27.

⁴¹ Waeser, vol. 1, 28.

relatively short span of a few years, the chemical and electrochemical research of Frank and Caro had progressed to the point where its large-scale application could proceed with confidence.

The power needed to produce cyanamide electrothermally, although less than other emerging nitrogen fixation methods, was significant. This led Frank and his "Cyanid-Gesellschaft," a cyanamide concern made up of Siemens & Halske and the Deutsche Bank, to look for sites that could provide adequate available water-power for hydroelectricity. In 1905, the Cyanid-Gesellschaft entered into an agreement with Angelo Menozzi and formed the "Societa Generale per la Cianamide" in Rome. This concern then sold the Italian and Austrian rights to the process to the "Societa Italiana per la Fabbricazione di Prodotti Azotati," which located the first commercial cyanamide plant on the Pescara River at Piano d'Orte, Italy.⁴²

Marketed under the name "Kalkstickstoff" or "Lime-Nitrogen," calcium cyanamide began to penetrate the fertilizer market. With adequate water-power the biggest factor in the location of cyanamide manufactories, France, Spain, Switzerland, Austria-Hungary, Norway, and Japan, all erected large cyanamide plants in conjunction with large hydroelectric installations.⁴³ Germany on the other hand, with few suitable water-power sites, often resorted to lignite as a fuel source. One German manufacturer of cyanamide noted with dismay, how this "originally purely German invention had fallen into the hands of foreigners."⁴⁴

And the foreigners were very successful. The plant at Piano d'Orta expanded twice and, by 1913, produced 24,000 tons of cyanamide fertilizer per year.⁴⁵ In 1907, at the Alby United Carbide Factories in Odda, Norway, a cyanamide plant was added to an existing carbide facility. Originally built for acetylene production, with a capacity of 32,000 tons of carbide a year, the new cyanamide plant developed an annual cyanamide capacity of 12,500 tons.⁴⁶

⁴² "Fixation of Atmospheric Nitrogen" Electrochemical and Metallurgical Industry 4 (August 1906): 327-328.

⁴³ Waeser, vol. 1, 25-28.; Curtis, 82.

⁴⁴ Quoted in: Waeser, vol. 1, 31.

⁴⁵ Waeser, vol. 1, 31.

⁴⁶ "A New Norwegian Calcium Carbide and Cyanamide Works" Electrochemical and Metallurgical Industry 7 (May 1909): 212-216.

These two cyanamide plants differed in the way they nitrified calcium carbide to produce cyanamide. The Italian plant passed nitrogen gas through powdered carbide in externally heated horizontal retorts. Nitrogen was produced by passing air through copper-filled retorts using principles similar to the Brins Process. These two distinct retorts operated in unison to produce nitrogen and calcium cyanamide in an interconnected system. At Odda, cyanamide was made in cylindrical ovens by an exothermic reaction of calcium carbide and nitrogen gas, the latter supplied in controlled amounts by a large liquid air plant. A carbon electrode was inserted into the oven and a charge of calcium carbide placed around it. As nitrogen was introduced into the oven, current passed through the electrode, and began a reaction that produced its own heat. Once this reaction started, the carbon electrode was removed and the nitrification of the carbide proceeded for approximately 35 hours.⁴⁷

The lime nitrogen oven, as the above device came to be known, had certain advantages over the carbide retort, advantages that had much to do with the chemical nature of the reaction of calcium carbide and nitrogen. When heated beyond a certain point, the cyanamide in the retort oven reverted to calcium carbide. This reversible reaction was called "back action" and of course, wasted the time and materials involved in the production of calcium cyanamide. Another disadvantage was that external heating led to the formation of hardened, back action carbide on the inside walls of the retort. This vitrified mass had to be hammered out, leading to excessive wear and tear on the retort. By contrast, the lime nitrogen oven used meters to control the reaction, avoiding these problems. In addition, by heating the charge from the inside out, the cyanamide shrank away from the outside wall of the oven, leaving a solid block or "ingot" of cyanamide that was easily removed. Not surprisingly, the lime nitrogen oven became the preferred method of cyanamide production.

The cyanamide boom led to additional methods of production. The "continuous" lime nitrogen oven involved passing a metal box filled with carbide through a long tunnel filled with nitrogen, the box rolling on tracks through the tunnel. Although this method was noted for its numerous mechanical problems, its acceptance in a number of cyanamide plants illustrates the

⁴⁷ Geoffrey Martin, Industrial Nitrogen Compounds and Explosives, (New York: D. Appelton and Company, 1917), 61-67.

experimental intensity of the new cyanamide industry.⁴⁸

In the first fifteen years of the twentieth century, the cyanamide process represented the most widely used method for the fixation of atmospheric nitrogen. The water-power necessary for its economic operation, as well as the required raw materials of limestone and coal were, except in a few cases, readily available across the globe. Other fixation methods, especially the "arc" process of Birkeland and Eyde, were highly dependent upon large amounts of hydroelectric power that were limited to the alpine regions of Norway, Sweden, or other topographically suited sites, and their end product, nitric acid, was not in itself a fertilizer. Cyanamide carried with it the confidence of its manufacturers that the new fertilizer could compete directly with Chilean nitrate. At the time, such confidence was warranted by the sheer number of successful cyanamide plants all over the world. Pure nitrogen, of course readily could be produced by the Claude and Linde air liquefaction systems, technologies that had developed separately but were now thrust dramatically into partnership with a new and thriving industry. Clearly, the once autonomous industries of carbide manufacture and air liquefaction had become such a good example of industrial synergy that one writer declared: "These immense industrial undertakings are undoubtedly destined to change the face of the world, and entirely alter agricultural conditions of the future."⁴⁹

Frank Washburn, Walter Landis and American Cyanamid

That Muscle Shoals, Alabama became home to one of the world's largest cyanamide plants was suggested by an announcement by a new American company in 1907.

Here contiguous to the great Birmingham coal and iron district is cheap water power, the cheapest coke in the United States, limestone of unsurpassed quality and cheapness, and abundant and efficient labor. The advantages of this situation are unusual in respect to the facts; first, that the Tennessee River is one of the network of great interior navigable rivers of the

⁴⁸ Nitrate Division, Ordnance Office of the War Department, Report on the Fixation and Utilization of Nitrogen. (Washington: GPO, 1922.) 85-86. This Government publication remains one of the best informational sources on the period that is the subject of this historical report.

⁴⁹ Martin, 61.

Mississippi Basin, reaching the greatest agricultural regions of the country; second, that two great railway systems are available for reaching every market; third, Muscle Shoals is on the border of the great phosphate of lime deposits of Tennessee, with the acid product of which lime nitrogen will be mixed universally in making the standard commercial complete fertilizers; and, fourth, the factory will be in the heart of the great cotton growing region of the United States, and close not only to the hundreds of fertilizer factories scattered throughout that section, but also to those which have clustered about the phosphate deposits.⁵⁰

The new company was the American Cyanamid Co., of New York City, and the company's president, Frank A. Washburn, had long eyed "The Shoals" as a site for hydroelectric development. The company's statement, although referring to commercial development, contained within it some critical facts that were later used by the government to justify the building of U.S.N.P. No. 2 at the same location. Unable to acquire the site for his proposed nitro-lime fertilizer plant, Washburn settled for a location on the Ontario side of Niagara Falls, already the location of a growing complex of electrochemical industries.⁵¹

Trained as a civil engineer at Cornell University and with a background in railroad transportation and dam construction, Washburn joined the W.R. Grace & Company in 1899, at the time the country's leading importer of Chilean nitrate.⁵² Soon after, Washburn married and moved to Nashville. While in the South, he became involved in hydroelectric power development, eventually becoming the president of the Alabama Power Company.⁵³ Washburn envisioned a cooperative effort between the Federal Government

⁵⁰ Quoted in: "Fertilizers from Atmospheric Nitrogen in America." Electrochemical and Metallurgical Industry 5 (July 1907): 289.

⁵¹ The potential of Muscle Shoals as a water-power site and Frank Washburn's interest therein has been fodder for much discussion. A thorough analysis is found in: Adrian G. Daniel, The Formative Period of TVA (New York: Carlton Press, 1973). See also: Haynes, vol. 2, 79-88.

⁵² Williams Haynes, Chemical Pioneers, (New York: D. Van Nostrand Company, 1939) 245-247.

⁵³ Haynes, Chemical Pioneers, 245-247.

and private capital in developing hydroelectric power at Muscle Shoals. The final product would enhance the navigability of the Tennessee through the construction of dams, locks, and canals: but more important for Washburn, it would create a large supply of cheap, hydroelectric power. With his previous exposure to the nitrate business and his knowledge of European developments in the fixation of atmospheric nitrogen, Washburn envisioned a nitrate plant as the perfect compliment to a large hydroelectric development. Accordingly, he traveled to Norway to meet with Samuel Eyde, the progenitor of the Arc process of nitrogen fixation. The preeminent historian of the American chemical industry, Williams Haynes, wrote:

The Norwegians welcomed this enthusiastic engineer who so obviously knew the hydro-electrical business. They went even further than curtesy demanded. After showing him every detail of this process and the know-how of its operation, they discussed costs fully and advised him against buying their American rights. Dr. Eyde even confessed that they had a good man in this country for three years scouting for water-power sites and had found nothing they regarded suitable. This frankness was discouraging, but Washburn was so convinced that nitrogenous fertilizers would make big power installations in the South economically feasible, that he went over to Germany to investigate the cyanamide process.⁵⁴

Washburn soon discovered that the German cyanamide process required one fourth the power needed by the Norwegian arc method. Furthermore, the two raw materials, lime and coke, were readily available in the Muscle Shoals region. Convinced of a sure thing, Washburn acquired the American rights to the Frank-Caro group's cyanamide process, and with approval from associates at Alabama Power, incorporated the American Cyanamid Co. on July 22, 1907.⁵⁵

The U.S. Government and Washburn could not agree on any private/public partnership plan for the development of Muscle Shoals. Washburn then made agreements with the Ontario Power Company at Niagara Falls, Ontario, and by 1909, North America's first cyanamide plant was in full operation.

The American Cyanamid plant at Niagara Falls depended on European technology for most of its operations. The electric

⁵⁴ Haynes, vol. 2, 80.

⁵⁵ Haynes, vol. 2, 81.

furnaces for the production of calcium carbide were of Italian design; the nitrification ovens developed by Frank and Caro produced the crude cyanamide. Nitrogen came from a chemical process similar to the Brins method discussed earlier, but at American Cyanamid this involved passing air through heated retorts packed with porous copper "briquettes," converting oxygen to copper oxides and leaving nearly pure nitrogen. Although a simple process, the copper absorbed only a limited amount of oxygen and eventually required "reducing" by forcing gas through the same retorts. The abundant natural gas reserves on the Canadian side of the falls encouraged use of this chemical method of nitrogen production. Even as the Claude and Linde systems were becoming standard equipment throughout Europe, American Cyanamide stuck with and even expanded this older process, building a coal gas plant on-site that produced as a by-product the coke used in the manufacture of calcium carbide. In a 1915 expansion and modernization, the company added a Claude liquid-air plant, bringing the cyanamide plant up to the standards of its European competitors.⁵⁶

American and European markets for cyanamid differed in important ways. American farmers were far more reluctant to accept "Cyanamid," as the fertilizer product. Furthermore, labor costs and economics of operation created different demands upon the company. American Cyanamid's chief technologist, Walter S. Landis, strove to create a plant, process and product that had distinctive, American attributes. One area where the company differed from its European counterparts was in the manufacture of calcium carbide. As Landis stated in *Metallurgical and Chemical Engineering*:

To produce day after day a high-grade calcium carbide that meets the peculiar requirements of the cyanamid industry is quite an art in itself. There are no journeymen carbide workmen in our country, and we had to start out and develop our own furnaces and methods of operation. We had to seek sources of raw materials for this manufacture, which are quite different even from those which meet the requirements of the manufacture of lighting carbide, and we have to operate our furnaces in a certain peculiar way in order to combine these raw materials into a product of such structure and grade that we can make cyanamid out of it successfully.

Landi stressed the difference between the European Nitro-

⁵⁶ "The Cyanamid Works at Niagara Falls" Engineering News 73 (January 7, 1915): 16-21.

Lime and his company's Cyanamid. This difference, he asserted, was the percentage of nitrogen in the fertilizer produced, and this percentage was based on "properly prepared" calcium carbide. Without such careful quality control in Cyanamid's carbide department, the company would not be able to offer anything better than the "low-grade" cyanamide fertilizer of the Europeans.⁵⁷ In truth, many plants in Europe, such as the cyanamide factory at Odda for example, had a product equal to American Cyanamid and were considered by many to be the most modern in the world. One of the basic operational goals of Cyanamid's Niagara plant, one also sought at European cyanamide manufactories, was the elimination of manual labor through extensive utilization of automated equipment. Such automation was also established at the Odda plant for "...handling the materials as nearly completely automatically as possible."⁵⁸ Installation of labor-saving machinery, especially for materials-handling operations, was crucial to profitable manufacturing of cyanamide. Landis described part of his company's process as follows:

The limestone and coke received in self-dumping cars pass to the storage silos by the usual conveyor equipment. The silos feed the kilns, or dryers mechanically, and the product of this operation is stored above the carbide furnaces, into which it is mechanically charged. The carbide furnaces are tapped electrically, and the chills moved by power to the storage sheds. They are brought back and dumped automatically into the crushing and milling equipment, and the finely-powdered carbide is mechanically weighed and charged into the cyanamid ovens. The crane picks up the block of cyanamid from the ovens and dumps it upon the breaking floor, where the first hand labor which actually comes into contact with the raw material or finished product is used.⁵⁹

But manual labor was still involved, especially at the electric furnaces where furnacemen using shovels still achieved the crucial mix of limestone and coke. Nonetheless, American Cyanamid's attention to efficient operation helped establish the company as America's only seller of cyanamide.

Between 1909 and 1915, American Cyanamid improved and

⁵⁷ Walter S. Landis, "Twenty-Five Years of Progress in the Cyanamid Industry" Transactions of the American Electrochemical Society 51 (1927): 68.

⁵⁸ "A New Norwegian Cyanamide Plant," 213.

⁵⁹ Landis, "Progress in The Cyanamid Industry," 70.

expanded its plant. In addition to introducing the Claude liquid air plant, the carbide furnace department modernized in 1914. In the earlier furnace installation, one based on European practice, the open-top refractory hearths were positioned with the furnace opening 3 ft. above the charging platform. This arrangement protected furnace operators from the intense heat of the furnaces, but hampered the charging operation. In the 1914 modification, the furnace opening was brought down flush with the charging floor, greatly facilitating both charging and electrode operation.⁶⁰ By 1915, American Cyanamid's plant was comparable to those in Europe.

Cyanamid's expansion occurred in conjunction with a major shift in the world cyanamide industry. Cyanamide changed from an end product to that of a raw material, necessitating the development of entirely new chemical processing apparatus. This shift coincided with the advent of war and, like the cyanamide industry that preceded it, Germany was the source of the new technology.

The Autoclaving of Ammonia

Nitrogen's essential role in both fertilizers and explosives inextricably links the history of fertilizer and wartime chemicals. The cyanamide industry thrived just as the world powers prepared for an unprecedented use of high explosives. Referring back to the natural breakdown of cyanamide in the soil quoted earlier, cyanamide's capacity to produce ammonia, and subsequently, nitric acid, is what changed the complexion of the cyanamide industry. Frank and Caro had early on established the reaction and it was not long before a suitable apparatus and system for producing ammonia from cyanamide were developed. The German patent for this chemical process established what one writer declared "the first solution to the problem of synthetically producing ammonia from the nitrogen of the air and the hydrogen of water."⁶¹

The key to transforming cyanamide into ammonia was an apparatus known as an autoclave. Use of closed vessels capable of withstanding high temperatures and pressures dates from the seventeenth century, and had become common in a number of chemical processes by the turn of the century.⁶² The first

⁶⁰ "The Cyanamid Works at Niagara Falls," 19.

⁶¹ Waeser, vol. 2, 389.

⁶² Thorpe's Dictionary, vol. 1, 550.

cyanamide autoclaves were rudimentary pieces of equipment used to produce ammonia for the production of ammonium sulfate, yet another fertilizer product. Although some considered it "...irrational to convert a finished fertilizer, such as calcium cyanamide, into another fertilizer," the cheap cost of steam in Germany made the process economically feasible.⁶³

To produce ammonium sulfate, crushed cyanamide was packed into elongated pressure vessels and "overheated steam" entering the autoclave from the bottom converted the cyanamide to ammonia gas. The gas was then released through a valve at the top of the vessel, into piping that lead to an awaiting tank of sulfuric acid.⁶⁴ As the cyanamide industry reached its highest rates of production, observers suggested the installation of ammonia/ammonium sulfate plants at existing cyanamide plants to utilize the over-production of what was becoming a bloated cyanamide supply.⁶⁵ To a large extent this occurred, and the dissemination of cyanamide ammonia production naturally followed the path of the earlier cyanamide plants, with new autoclaving facilities established in Belgium, Norway, France, Switzerland, Italy, and Japan.⁶⁶

In the years just prior to the outbreak of World War I, refinements and modifications to the process of autoclaving cyanamide to produce ammonia resulted in an extremely pure product. Industrial chemists discovered early on that using crude cyanamide in the autoclave resulted in an ammonia gas that contained impurities in the form of acetylene and cyanamide compounds (dicyandiamides). To increase the efficiency of ammonia production, caustic soda ash and lime were added to the charge of cyanamide, and the use of such alkaline additives became standard practice in the manufacture of cyanamide ammonia.⁶⁷ The autoclaves themselves grew in size and mechanical complexity, with agitators, valves, and sludge removal components becoming increasingly efficient. What came to be known as

⁶³ Waeser, vol. 2, 387.

⁶⁴ "Fixation of Atmospheric Nitrogen" Electrochemical and Metallurgical Industry 5 (March 1907): 77-79.

⁶⁵ "Fixation of Atmospheric Nitrogen," (see note 70 above).

⁶⁶ Walter S. Landis, "The Production of Ammonia From Cyanamid" Transactions of the American Institute of Chemical Engineers 8 (1916): 270.; Waeser, vol. 2, 389-390.

⁶⁷ Landis, "The Production of Ammonia From Cyanamid" 267.; Waeser, vol. 2, 389-390.

"mother liquor," a solution derived from the autoclave sludge, was used as a liquid vehicle for the agitation process. The spent sludge often ended up in "unsightly heaps" adjacent to the plants.⁶⁸

The hydration of cyanamide to produce ammonia soon became an industry almost as large as the cyanamide industry itself.

The Catalytic Oxidation of Ammonia

"While under present conditions the transformation of ammonia into nitric acid with the assistance of platinum and air has no great value, yet the time will come when it may be of commercial importance."⁶⁹

So declared, in 1839, the German technical chemist Kuhlman after producing nitric acid vapors by passing a mixture of air and ammonia through a heated glass tube filled with spongy platinum. Kuhlman's experiment and subsequent observation that such a method of producing nitric acid from ammonia gas could be marketed commercially, were indeed prophetic. After a number of other experiments using the same catalyst, one man in particular, Wilhelm Ostwald, performed the extensive research and developed the specialized equipment that made Kuhlman's assertion a reality. By 1901, Ostwald had developed an effective catalyzer; by 1908, he had built two small plants for the catalytic oxidation of ammonia.⁷⁰ Until this time the production of nitric acid had been wholly dependent upon Chilean nitrates; now it could be produced with ammonia gas.

The timing of this technological development could not have been more serendipitous. Germany and the rest of Central Europe manufactured almost all of their nitric acid by treating Chilean nitrates with sulfuric acid (oil of vitriol) in large retorts. With the approach of World War I, the supply of nitric acid, as the basis for most high explosives, was becoming a major concern. Catalytic oxidation of ammonia assured ample domestic supplies of nitric acid especially since ammonia was readily available through the autoclaving of cyanamide.

Ostwald's first plant, located at Gerthe, Germany, converted

⁶⁸ Waeser, vol. 2, 389-390.

⁶⁹ Quoted in: Walter S. Landis, "The Oxidation of Ammonia" Chemical and Metallurgical Engineering 20 (May 1, 1919): 470.

⁷⁰ Landis, "Oxidation of Ammonia," 470.

by-product ammonia gas from coke ovens.⁷¹ As noted earlier, European cyanamide manufactures had begun to autoclave their product to produce ammonia around the same time as Ostwald's work in catalyzation. As a consequence, in 1911, a French concern began the production of nitric acid using the Ostwald process in a Belgian plant which used cyanamide supplied from Odda in Norway.⁷² In 1913, the Nitrogen Products & Carbide Company, Ltd., of London, purchased the rights to the Ostwald process and made plans to build a plant at Dagenham, England.⁷³ These developments were noted in a curious and highly informational letter sent to *Metallurgical and Chemical Engineering*:

Sir: - The article in the August issue on this subject is of great interest to me. It is true that the process of making nitric acid from ammonia produced from calcium cyanamid is new for England as stated there. But exactly this same process has been and is in successful commercial operation in a plant in Belgium though nothing has yet been published about it. The following notes on the process should, therefore, be of interest. Naturally certain important details are being kept secret and must be withheld from publication.

If we mix calcium cyanamid with water we get ammonia.
$$\text{CaCN}_2 + 3\text{H}_2\text{O} = \text{CaCO}_3 + 2\text{NH}_3.$$
To mix cyanamid with plain water is impractical for industrial purposes for the reason that not all of the nitrogen can thereby be extracted in form of ammonia. In order to obtain all the nitrogen as ammonia, we must use alkali and carry on the process along the following lines which have been developed in many years of hard work and experimental research.

If we mix the correct quantities of CaCN_2 , H_2O , Na_2CO_3 and NaCl in one dish and mix well, acetylene will be evolved. This is due to the fact that cyanamid contains 0.1 to 0.3 per cent of carbide of calcium. After the carbide has thus been removed the mixture is transferred to an autoclave and the steam turned on and the pressure raised to 6 or 8 atmospheres. All the nitrogen will then distill in the form of ammonia gas.

⁷¹ Waeser, vol. 2, 611.

⁷² Waeser, vol. 2, 611.

⁷³ Waeser, vol. 2, 611.

If the operation in the autoclave is carried out properly, the ammonia and steam come out entirely clean without carrying any cyanamid dust. Nevertheless, the practice is to pass the ammonia and steam through a cleaning apparatus for the purpose of catching any dust which might be carried along.

After that operation ammonia and steam are passed together into a column which cools and condenses the steam and lets pure ammonia gas enter into the gasometer.

Ammonia obtained by this process is very pure, the purity being 99.8 per cent or more.

The entire process is easily carried out and is a large source of ammonia which may be transformed into many chemical substances. We can transform ammonia nitrogen into nitric nitrogen from which we can get very easily ammonium sulfate or liquid ammonia, but the most interesting transformation process is the transformation into nitric acid and ammonium nitrate.

If we let the ammonia pass through tubes containing a catalytic agent (platinum) we can transform ammonia H_3N into NO_2 and with further oxidation in oxidation towers into nitric acid of 36 deg. B. This acid we can transform with the same ammonia into nitrate of ammonium and we can continue concentrating further to highest concentrations.

The process is exceedingly interesting and very simple and in the writer's opinion it has a great future. A. B.⁷⁴

This letter, written in 1913, illustrates the birth of an industrial process that was to be applied at U.S.N.P. No. 2. It is at this technological intersection of cyanamide production, autoclaving of cyanamide to produce ammonia, and the oxidation of ammonia through catalytic action, that Walter S. Landis of American Cyanamid played a major role in bringing the process to America.

⁷⁴ Letter, "The Ostwald Process for Making Nitric Acid from Ammonia and the Production of Ammonia from Calcium Cyanamid." Reprinted in: Metallurgical and Chemical Engineering 11 (September 1913): 476.

As chief technologist for American Cyanamid, Landis was primarily responsible for communicating European autoclave and catalyzer technology to the United States. In the first part of 1914, as representative of American Cyanamid, Landis secured contracts for German ammonia-oxidation and autoclave equipment to build "the largest single ammonia plant then projected or operating in the cyanamide industry."⁷⁵ Landis' trip coincided with the beginning of a major shift in the nitrogen fixation industry from the production of fertilizer to that of nitrates for munitions. Williams Haynes, in his thorough study, offers an engaging footnote regarding this tenuous transfer of technology. In a letter to Haynes, Landis stated:

My problem was to inspect and accept this equipment and see what could be learned as to progress in the art of converting ammonia to nitric acid. Advent of the war earlier than expected, completely disrupted our plans, but I was able to bring out some of the autoclaves used to convert cyanamide to ammonia, with the auxiliary equipment and the drawings of the plant. At that time Germany had already made active plans for an enormous expansion of cyanamide production, which by the end of 1915 reached a total of about 400,000 tons, or about 15 times the prewar production.⁷⁶

This intriguing scenario, set in the politically volatile period just after the declaration of war, illustrates how important Landis' personal connections were. That this equipment, crucial to the manufacture of munitions, was given away in a seemingly friendly exchange between two industrialists, suggests the void that existed between private and government interests. The German Government was able to seize most of the equipment Landis had come for, but not before some of the vital pieces and important information was brought to the United States.

Toward the end of 1915, as the war in Europe raised U.S. anxiety over the supply of nitrates, American Cyanamid continued development of the commercial production of nitric acid from cyanamide ammonia. Landis was charged with setting-up an

⁷⁵ Landis, "The Production of Ammonia From Cyanamid," 268; Haynes, 2. pp. 82-83. At this time, Britain, concerned with the need for an ample supply of fixed nitrogen in case of war, prompted American Cyanamid to launch this investigation. Furthermore, the ammonia oxidation that had been occurring in recent years prompted them to seek a manufacturer of cyanamide.

⁷⁶ Haynes, vol. 2, 83.

experimental ammonia oxidation plant at the cyanamide works at Niagara Falls.⁷⁷ With only a few German-made cyanamide autoclaves for the production of ammonia, American Cyanamid contracted with E. I. du Pont de Nemours & Co., at the time the country's largest explosives manufacturer, to install and operate the equipment using American Cyanamid's product as a raw material.⁷⁸ Du Pont went into immediate production, generating "several tons of ammonia gas per day,"⁷⁹ augmenting its supply of ammonia for the production of ammonium nitrate. The nitric acid needed for the production of ammonium nitrate was based solely on imports of Chilean nitrate which, when treated with sulfuric acid, yielded nitric acid. Landis' as well as other chemist's experiments in the catalytic oxidation of ammonia forged the way to independence from Chilean nitrate.

On his junket to Germany, Landis had not been impressed by their catalyzer design, commenting that he had "not seen an apparatus function for more than a few hours at a time."⁸⁰ In addition, Landis stated that he "found little or no information on the converting of the nitric oxide obtained from a catalyzer into nitric acid."⁸¹ He did note the use of "towers of chemical stoneware, tourils and such equipment..." but called what he saw "small-scale equipment."⁸² Such equipment was crucial to the commercial production of nitric acid, and it was not long before what Landis called "small-scale" was greatly enlarged.⁸³

⁷⁷ Landis, "Oxidation of Ammonia," 473.

⁷⁸ Waeser, vol. 1, 62.; Haynes, vol. 2, 83-84.

⁷⁹ Landis, "The Production of Ammonia," 268.

⁸⁰ Landis, "Production of Ammonia," 268.

⁸¹ Landis, "The Oxidation of Ammonia," 473.

⁸² Landis, "Oxidation of Ammonia," 473.

⁸³ The "tourils" that Landis had noticed were part of an absorption tower for the production of nitric acid from ammonia gas. The development and refinement of the "packed tower" was closely associated with the fixed nitrogen industry. Up to the time of World War I, little theoretical work was done on the theory of absorption and tower packing materials were based on their cheapness (broken bricks, glass, granite, coke etc.). With the drive toward independence from Chilean nitrate, the absorption of nitrous gasses by water in a packed tower saw vast improvements. One of the most common absorption tower fillings, and one used at both U.S.N.P. No. 1 and 2, was known as the Raschig Ring. These

Upon establishing his experimental plants at Du Pont and at American Cyanamid, and following the principles and methods established by Ostwald, Landis explored many variations on the basic theme of exposing a mixture of air and ammonia to a heated platinum catalyst. The end result of this catalytic reaction was nitric oxide, which could then be turned into nitric acid, an ingredient that could be used in the production of both fertilizers and explosives. Many platinum screens and gauzes, alone or in sequence, were heated to varying degrees while differing proportions of air and ammonia gas were introduced into the catalyzer. Eventually, Landis achieved good results with an electrically heated catalyst within a 4 foot aluminum tower. The ammonia gas entered the catalyzer from the top, passed through the heated platinum, and exited as a hot gas composed primarily of nitrogen oxides.⁸⁴

With the German autoclaves in operation, Landis set about finding a manufacturer who could make the autoclaves in the United States. Accordingly, W. M. Kellogg of New York developed the first American cyanamide autoclaves based on the German design, while redesigning the equipment and "adapting it to American standards and manufacturing conditions."⁸⁵

While Du Pont used their autoclaves in the production of explosives, American Cyanamid, although knowing full well of the likelihood of such an application, sought to use the new autoclaving equipment in fertilizer production. Rather than producing ammonium sulfate, however, their attention was drawn to a small fertilizer concern in New Jersey that was producing a new phosphatic fertilizer by treating phosphate rock from Florida with ammonia. An agreement was made with the Amalgamated Phosphate Company of Warners, New Jersey, and American Cyanamid installed autoclaves under agreements similar to Du Pont, having the company use cyanamide from Niagara to produce ammonia for their ammonium phosphate fertilizer.⁸⁶ Amalgamated Phosphate Corp. was acquired by American Cyanamid in 1916 and became Ammo-

corkscrew-shaped stoneware rings were randomly packed in the tower and provided both a large wetting surface and the necessary turbulence for absorption. See: H. W. Webb, Absorption of Nitrous Gases. (London: Edward Arnold & Co., 1923).

⁸⁴ Landis, "The Oxidation of Ammonia," 474.

⁸⁵ Landis, "Production of Ammonia," 274.

⁸⁶ Haynes, vol. 2, 83-84.

Phos Corp.⁸⁷ American Cyanamid had experimented with ammonium phosphate fertilizer since 1910 so the acquisition was a logical one.

The Ammo-Phos plant at Warners became for American Cyanamid a logical venue to introduce on a commercial scale, its then experimental process of oxidizing cyanamide ammonia with platinum catalyst. Following an intense period of experimentation, the company built the first American plant for the production and oxidation of cyanamide ammonia at Warners, N.J. in 1916. Platinum gauze catalyzers of Landis's own design were installed and production of nitric acid, with its accompanying absorption equipment, commenced in July of that year.⁸⁸

Explosives and World War I

The President of the United States is hereby authorized and empowered to make, or cause to be made, such investigation as in his judgement is necessary to determine the best, cheapest, and most available means for the production of nitrates and other products for munitions of war and useful in the manufacture of fertilizers and other useful products by water power or any other power as in his judgment is the best and cheapest to use... June 3, 1916

In any study of United States Nitrate Plant Number 2, the historical compass points to the National Defense Act of 1916. It is here that much of what has been called "the Muscle Shoals Controversy" is rooted. Yet, when placed within the overall context of U.S. wartime spending for production of munitions, U.S.N.P. No. 2 was not as controversial as some have asserted. In the relatively brief 19 months of American involvement in World War I, the government built 53 new plants for making and loading explosives and propellants some successful, others not so.⁸⁹ The government plant at Perryville, Maryland for example, produced ammonium nitrate using the experimental "Brunner-Mond" process of double decomposition.⁹⁰ In addition, the government also engaged commercial manufacturers of ammonium nitrate, the

⁸⁷ Waeser, vol. 2, 267-268.

⁸⁸ Landis, "Oxidation of Ammonia," 475.

⁸⁹ Benedict Crowell, America's Munitions, (Washington: Government Printing Office, 1919), 105.

⁹⁰ Crowell, 105.

only producers in this country before the war. Companies such as Trojan Powder Company launched large-scale production of ammonium nitrate by expanding their existing facilities. Atlas Powder Company planned and operated the large government facility at Perryville. Before and during the war, commercial industry supplied the key ingredient of high explosives.

The one aspect of these plants and the companies that ran them that made the War Department anxious was their dependence on Chilean nitrate as a source of nitric acid. Controlled in large part by American interests, to get Chilean nitrate from South America to the United States required ships to run a potential gauntlet of German submarines. One of these subs, the Deutschland, had made a unique appearance in New York Harbor just prior to the war and further German nautical activities such as the submarine blockade of Britain were ample reason for apprehension regarding our dependence on Chilean nitrate.

Government's response was both belated and needlessly time consuming, considering what commercial industry had already done. The fixation of atmospheric nitrogen quickly became the obsession of the government. As Williams Haynes noted: "Synthetic production of nitrogen for explosives required answers to two correlated but distinct questions: Which process of nitrogen fixation? What most advantageous location?"⁹¹

In 1916, at the request of General William Crozier, Chief of Ordnance, Charles L. Parsons, Chief Chemist for the Bureau of Mines made a hasty but thorough inspection of European nitrogen fixation plants and technology. Without dismissing entirely other fixation methods then in use or already developed, Parsons recommended that the government construct experimental plants based on the Haber and Bucher processes,⁹² German processes recently adopted by the General Chemical Company of New York and the Nitrogen Products Company of Greene, Rhode Island.⁹³ Both

⁹¹ Haynes, vol. 2, 89-100.

⁹² For a detailed summary of these Government sponsored investigations, see: Margaret J. Clarke, "The Federal Government and the Fixed Nitrogen Industry, 1915-1926" (Ph.D. diss., Oregon State University, 1977.; House Committee on Military Affairs, Muscle Shoals Propositions: Hearings 67th Cong., 2nd sess. 1922. "Statement of William Parsons," 1115-1145.

⁹³ The Haber process was a method of synthetically producing ammonia by uniting nitrogen and hydrogen under intense pressure. The nitrogen required would be produced by fractional distillation of air by either the Claude or Linde systems. The Bucher process

companies offered their technology to the government and generated proposals for Parsons' to bring to General Crozier.⁹⁴

Other offers came from two larger chemical concerns, E. I. du Pont de Nemours Powder Company and American Cyanamid. In March of 1916, Pierre S. du Pont wrote the Secretary of War detailing his company's research into the fixation of atmospheric nitrogen and its recently purchased patents for the Birkeland-Eyde process.⁹⁵ The company had "...sent a corps of its experts to Europe to thoroughly investigate the various processes there employed."⁹⁶ As this "arc" method for producing nitric acid from the air required large amounts of electrical energy, du Pont's proposal to the Secretary of War echoed many of the same sentiments regarding government construction of large hydroelectric plants that Washburn had raised in 1906. Less than a month after du Pont's letter, Washburn published and circulated a pamphlet entitled "The Facts In The Nitrogen Case Now Before Congress."⁹⁷ In this dense little treatise on nitrogen fixation, du Pont's fixation plans, and his own company's expertise in that area, Washburn mustered economic and technical evidence to support his process. While du Pont had the upper hand when it came to the manufacture of explosives, the stipulation that any wartime nitrogen plant must be used for fertilizer in times of peace gave Washburn a decided edge. Furthermore, Ordnance Chief Crozier had contacted Washburn as early as 1915 regarding a government cyanamide plant and considered him a friendly

was also known as the cyanide process and involved liberating ammonia from sodium cyanide. The latter process was incarnated as U.S.C.P. No. 4 (United States Chemical Plant Number Four) in Saltville, Va. For details on these and all Government fixation plants see: "Report on the Fixation and Utilization of Nitrogen" (note 52. above). See also this report's Bibliography for more titles on nitrogen fixation.

⁹⁴ Clarke, 72.

⁹⁵ Letter, reprinted in: "Proposed Plant for the Fixation of Atmospheric Nitrogen" Metallurgical and Chemical Engineering 14 (April 1, 1916): 362-363.

⁹⁶ "Proposed Plant," 363.

⁹⁷ "Fixation of Atmospheric Nitrogen Before Congress" Metallurgical and Chemical Engineering 14 (April 15, 1916):418-420.

consultant in all matters of nitrogen fixation.⁹⁸

For plants using just nitrogen fixation processes, sites throughout the South were preferred, particularly those with hydroelectric power. The selection of General Chemical's Haber process reduced the importance of hydroelectric power. General Crozier, representing the Nitrate Supply Committee of the Ordnance Department, presented the following recommendation:

I think that the examinations made indicate the limit of choice to the four localities, viz: Chattanooga, Knoxville, Sheffield, and North Birmingham, and after fully considering all the aspects of the matter it is my opinion that the site at North Chattanooga, Tenn., having been adjudged first from the fertilizer standpoint and next from the compromise standpoint, is best fitted for the construction now contemplated, and I recommend its adoption and the purchase of a tract of 500 to 600 acres.⁹⁹

Nonetheless, President Wilson chose the Sheffield, Alabama, as the site for the government's first nitrogen fixation plant and in July of 1917, General Chemical Company and the newly formed Division T, the Nitrate Division of the Ordnance Department, began work on United States Nitrate Plant Number 1.

Among the nine recommendations set forth by the Nitrate Supply Committee was one that attempted to keep the government's expenditures on atmospheric nitrogen production limited to testing and experimentation and to avoid any commitments to large hydroelectric projects.¹⁰⁰

In his recollection of this prewar period, Parsons insisted that Plant No. 1 "was not built for war emergencies" and existed

⁹⁸ Clarke, 46. Crozier was one of the few government officials that had recognized the potential military crisis of a terminated supply of Chilean nitrate.

⁹⁹ Muscle Shoals Propositions, 1121.

¹⁰⁰ Reprinted in "Nitrogen Fixation by the Haber Method" Chemical and Metallurgical Engineering 22. (June 9, 1920): 1073; Recommendation number eight stated the following: That the decision as to more extensive installation of nitrogen fixation processes and water-power development in connection with them be postponed until the plants above recommended are in operation or until further need arises.

only as a trial plant.¹⁰¹ It was in the midst of its construction, however, that "further need" arose, and the government decided to build both a cyanamide plant and an adjoining hydroelectric dam and powerhouse.¹⁰² Only one man and one company could provide the technology for producing ammonium nitrate from cyanamide: Frank Washburn and American Cyanamid.

As in his attempt to locate American Cyanamid at Muscle Shoals in 1907, Washburn again confronted the U.S. Government as to some compromise over the use of American Cyanamid's patented process and the development of hydroelectric power on the Tennessee. The agreement they reached became a source of great debate, confusion, and eventual frustration for both parties in the years following the war. For a brief period of time, between November, 1917 and February of 1918, they accomplished a vast and unique technological feat. United States Nitrate Plant Number 2 represented the peak of the cyanamide industry's brief but dramatic technological evolution from a producer of fertilizers to a supplier of nitrates for munitions.

Building U.S.N.P. No. 2

Private companies constructed U.S.N.P. No. 2 under the direction of American Cyanamid's subsidiary, Air Nitrates Corporation. Westinghouse, Church, Kerr & Company, Air Nitrates Corporation's largest sub-contractor, a well-established engineering and construction firm based in New York, specializing in power plant and railroad terminal construction.¹⁰³ The contract with WCK called for them to,

...in the shortest time possible, do the necessary engineering involved in the designing, drafting, and laying out of the work based on the general drawings, specifications, and directions furnished by the corporation [Air Nitrates Corp.] from time to time, and furnish labor, materials tools, machinery, equipment,

¹⁰¹ "Statement of Dr. Charles L. Parsons," 1129.

¹⁰² For an alternative viewpoint as to the need for U.S.N.P. No. 2, see:

¹⁰³ Brian F. Coffey, "Boston's South Terminal Station: An Electric Traction History" Railroad History 170 (Spring 1994); One of their largest achievements was the engineering and construction of Boston's South Terminal Station which was, at the time of its construction, the largest and most modern railroad terminal in the world.

facilities, and supplies, and drainage, sewerage, and water system, buildings, foundations roads, and ways; such machinery and equipment, if any, as the corporation may request the contractor to furnish and install; and such other thereto, as the corporation may specifically request in writing.¹⁰⁴

In listing WCK's construction schedule for the above contract, the immensity of the plant is more clearly understood.

Railroad tracks to serve site; roads from Florence and Sheffield to be put in shape to stand traffic and reconstruct so-called River Road to provide access to plant; provide temporary office buildings; provide temporary water supply from spring located near river bank; provide camp and commissary, including water supply and sewer system, to house and feed workmen, also an emergency hospital; provide temporary utilities for light and power, water supply, compressed air, steam, machine shop, garage etc.; construct manufacturing plant with its utilities; construct an industrial village with all its utilities to house the permanent operatives.¹⁰⁵

WCK built 73 nitrate plant buildings, 2,165 temporary and cantonment buildings and 190 structures in the industrial village.¹⁰⁶

Chemical Construction Corporation, incorporated in 1914, was one of the few American companies with experience in building acid-absorption equipment.¹⁰⁷ In conjunction with Sullivan Machinery Company and Duriron Castings Company, Chemical Construction would

...design, erect, and build a complete nitric acid recovery plant consisting of 12 units, each unit to have a capacity sufficient to treat 2,500 cubic feet per minute of gas containing 8 per cent NO. This work was to include one gas cooling plant comprising gas flue connections, steam boilers, gas coolers, and

¹⁰⁴ Report on the Fixation and Utilization of Nitrogen, 277.

¹⁰⁵ Report on the Fixation and Utilization of Nitrogen, 281.

¹⁰⁶ Westinghouse, Church, Kerr & Company, "U.S. Nitrate Plant No. 2" Promotional Pamphlet (1919?).

¹⁰⁷ Haynes, vol. 6, 6.

oxidation towers; and a nitric acid gas absorption plant, with acid storage, weighing tanks, exhausters, and acid pumping equipment.¹⁰⁸

Air Reduction Company was incorporated in November, 1915 as America's first supplier of Claude Process air liquefaction equipment. With direct connections to L'Air Liquide in France, Air Reduction supplied the equipment and expertise for setting-up what was to be the largest liquid air plant in the world, producing nitrogen at a rate of one half million cubic feet per hour.

Another important sub-contractor was M. W. Kellogg of New York. As builders of American Cyanamid's autoclaves, this firm manufactured and assisted in the installation of 56 such pressure vessels along all the associated piping that ran throughout the plant. Their previous work with American Cyanamid's Warners plant in New Jersey was invaluable.

American Cyanamid's most recent ventures into the production of cyanamide ammonia were crucial to rapid manufacture and installation of autoclave equipment at U.S.N.P. No. 2. A series of 1916 blueprints of the autoclave installation at Ammo-Phos were used in the production of Air Nitrate Corporation's construction drawings. E. I. du Pont even supplied blueprints of their autoclaves to help with the illustration of special autoclave parts.¹⁰⁹ In addition, blueprints of American Cyanamid's Niagara Falls plant dating to 1913 were used to develop construction drawings illustrating everything from railroad track layouts to electric furnace designs.

The construction of Nitrate Plant No. 2 survived severe weather and, more ominously, a severe outbreak of influenza.¹¹⁰

¹⁰⁸ Report on Fixation and Utilization of Nitrogen, 281.

¹⁰⁹ All blueprints and drawings of U.S.N.P. No. 2 were made available to the HAER recording team. Although many of these drawings have been microfilmed, a large amount have been kept in storage at the original plant. During the recording project, plans were developed to transfer all original plant drawings to the East Point, Georgia, National Archives Center.

¹¹⁰ The severe cold of January, 1918 and the influenza epidemic that occurred in the fall of the same year are part of the lore of the Muscle Shoals area. For more details on these natural disasters see: Daniel Schaffer, The Moral Materialism of War: Muscle Shoals, Alabama 1917-1918. (Tennessee Valley Authority, Office of Natural Resources and Economic Development, Cultural

Despite these setbacks, only eight months and eight days after construction began, manufacturing commenced at U.S.N.P. No. 2. A report of the War Department's Nitrate Division chronicled the start-up as follows:

The actual operation of the nitrate plant may said to have commenced at the time the electric current was first turned on in the carbide furnaces, which took place at 12:02 p.m., October 26, 1918. On October 23, 1918, the first air compressor was started in the liquid air plant. The first nitric acid was produced a 1:30 a.m., November 19, 1918, and the first ammonium nitrate at 3:20 a.m., November 25. Thus one month elapsed from the beginning of the carbide manufacture to the time of production of the finished ammonium nitrate, and the first ammonium nitrate was produced just two weeks after the armistice.¹¹¹

Construction continued even as operations began. But the signing of the armistice had put yet another burden upon the government: specifically, what to do with an operating but partially-completed nitrate plant, and more generally, what long term plan should be developed for a multi-million dollar chemical plant and hydroelectric facility. On November 19, 1918, government and company officials decided to complete the plant and test its efficiency. This test was carried out by employees of the Air Nitrates Corporation under the supervision of the Nitrate Division of the War Department from January 11 through 25, 1919. According to the results of the test and the statements of the plant's operators, "all departments of the plant performed satisfactorily" and the equipment showed to have a capacity "equal to and in many cases in excess of its requirements."¹¹² The total amount of ammonium nitrate produced was said to be 1,700 tons.

The War Department felt obligated to bring U.S.N.P. No. 2 to high operating standards. Using the test results and recommendations for operational improvements, "redesigns and rearrangements were laid out, such as would render United States Nitrate Plant No. 2 effective as a military establishment."¹¹³ This work continued through the summer of 1920. The question of

Resources Program, 1984).

¹¹¹ Report on the Fixation and Utilization of Nitrogen, 282.

¹¹² Report on the Fixation and Utilization of Nitrogen, 284.

¹¹³ Report on the Fixation and Utilization of Nitrogen, 287.

what to do with U.S.N.P. No. 2 launched a long and contentious debate.

Postwar and Beyond

The technological history of U.S.N.P. No. 2 entered a second and equally dynamic phase at the cessation of World War I. The cyanamide industry was never to regain its pre-war prominence. Dr. Parsons now a consulting chemist to the hearings of the sixty-seventh congress, testified to the depressing reality of the commercial cyanamide industry:

The CHAIRMAN. Have you any opinion about the cyanamid process yourself?

Doctor PARSONS. I have been through a number of plants; all there are in this country and several in Europe. I made a very careful study of the process and its operation.

The CHAIRMAN. Do you think that it is likely to be superseded by new inventions or new processes?

Doctor Parsons I think it is practically already superseded by the Haber process and its modifications, and on that I can give you some very definite data. The cyanamid plant in Canada is practically shut down. It is producing no cyanamid, or very little cyanamid, for fertilizer. The cyanamid plants in Italy are closed. The cyanamid plants in France are closed. The cyanamid plants in Dalmatia and Silesia are closed. The cyanamid plants in Norway and Sweden are closed. The large cyanamid plant at Odda, Norway, which I had the pleasure of visiting in 1916, and which is fully equal to the Muscle Shoals plant in quality and one of the best constructed plants I ever investigated. It is not quite one-half the size of the Muscle Shoals plant, is now closed, and has been sold at auction within the last month for \$10,000, or rather 50,000 kronen.¹¹⁴

One other thing I would like to call your attention to is a statement made to me by Mr. Barton in the summer of 1920, which I quoted in

¹¹⁴ Muscle Shoals Propositions, 1121.

the record because it is somewhat of a prophecy which I fully believed in at that time. Mr. Barton was the promoter in England and of the Norwegian and Swedish plants, and occupied in North Europe very much the same position to the cyanamid companies that Mr. Washburn occupied in America. Mr. Barton stated to me, "Doctor Parsons, as sure as we sit here, the cyanamid process is a thing of the past, being sure to be replaced by the synthetic ammonia process."¹¹⁵

It should also be noted that the cyanamide produced at U.S.N.P. No. 2 was not a fertilizer, but only an intermediate product in the production of ammonia. Even American Cyanamid acknowledged the impracticability of using U.S.N.P. No. 2 as a cyanamide fertilizer plant: "As it stands to-day, the plant can not make any material that is used in the fertilizer industry."¹¹⁶ Furthermore, the American market for "cyanamid" had always been a hard one to develop and the product was "not received with favor by American users, in spite of its good fertilizing qualities."¹¹⁷ Although a separate plant for the post-treatment (oiling or granulating) of the crude cyanamide could have been added to the plant, the enormous capacity of the plant's furnaces and lime-nitrogen ovens would make any such additions and production inefficient. The entire annual demand for the United States could have been produced in two of the plant's twelve electric furnaces.¹¹⁸

By-products of cyanamide were, at the time of U.S.N.P. No. 2, only in the experimental stages, but some chemists saw an opportunity to utilize at least part of the plant for cyanamide production. Urea, used in treating celluloid film, dicyandiamide, a constituent of organic dyes, and the nitrogenous materials guanidine and nitroguanidine were all seen as hopeful cyanamide by-products that might be made at the plant. Ammonia from cyanamide could be used to make ammonium sulfate as in Europe and ammonium phosphate could also be made utilizing some of the plants equipment. Even ammonium nitrate, the plants fundamental product, was considered a possible fertilizer if properly oiled and treated.

¹¹⁵ Muscle Shoals Propositions, 1123.

¹¹⁶ Muscle Shoals Propositions, 463.

¹¹⁷ Quoted in: Waeser, vol. 1, 274.

¹¹⁸ Muscle Shoals Propositions, 463.

Yet the fundamental question as to the disposition of U.S.N.P. No. 2 was bound less by technological issues than it was by political ones. The War Act of 1916 allegedly had settled the question. The plant would be operated by the government for the production of munitions in times of war and fertilizers in times of peace. The first bill regarding this governmental responsibility was introduced by Senator Wadsworth in November of 1919. The bill never passed, yet its wording had a prophetic quality.

An act to provide further for the national defense; to establish a self-sustaining Federal agency for the manufacture, production, and development of fixed-nitrogen production; and for other purposes.¹¹⁹

It was not until 1933 that such a "self-sustaining Federal agency" would eventually fulfill the goals as set forth in Senator Wadsworth's bill. Until then, however, U.S.N.P. No. 2 waited quietly, a huge industrial soldier silenced by the temporary peace of a warring society.

Conclusion

Mr. Miller. Now, the cyanamid process, you say, is an obsolescent process, but it will make ammonium nitrate?

Dr. Parsons. Yes sir; and that is why it was used.

Mr. Miller. As between a surety and a failure, or as between a process that all agree will manufacture what we are after, although it costs more, as compared with a process that we do not know how to follow out, it seems to me that in time of war the better judgement is to follow the system that has been proved, and I suppose that was the policy that the government followed.

Dr. Parsons. That is exactly the policy upon which plant No. 2 was based, and to which I think no one has taken exception. It was a process by which you can get ammonium nitrate, no matter what the cost might be.¹²⁰

¹¹⁹ Report on the Fixation and Utilization of Nitrogen, 291.

¹²⁰ Muscle Shoals Propositions, 1129-1130.

The above exchange between John F. Miller of the Committee on Military Affairs and Charles Parsons, consulting chemist, reduced the tumultuous period of government inquiry into nitrogen fixation down to its basic conclusion: U.S.N.P. No. 2 was built because the cyanamide route to ammonium nitrate worked. Experimentation with other methods of nitrogen fixation, especially the Haber process of producing ammonia, were too late to produce the needed product. In times of war, dependability, not innovation, is the crucial factor.

World War I marked a turning point for the American fixed nitrogen industry. Perhaps its greatest influence was to establish the Federal Government as a new participant in fixed nitrogen research.¹²¹ But until World War I, it was private industry that sponsored both the theoretical and applied chemical research that led to the commercial success and wartime strength of nitrogen fixation. This could not be more evident than in the case of the global cyanamide industry. At the height of the war, the existing cyanamide industry coupled with the latest developments in the oxidation of cyanamide ammonia supplied a needed alternative to the once-dominant Chilean nitrate. Germany, the birthplace of both technologies, depended more than any nation upon the commercial cyanamide industry both as a source of fertilizer and as a source of ammonia. In the United States, although the Haber process of producing synthetic ammonia eventually superseded the cyanamide process as a route to producing nitric acid, the success of the cyanamide industry and the technological development associated with it was part of the same drive towards innovation that characterized the period covered in this study. In such a period of rapid technological development, obsolescence is inevitable. Yet obsolescence is not the result of technological failure, it is the result of technological change.

¹²¹ The earliest Governmental work on nitrogen fixation was associated with the Bureau of Soils in 1915. In 1919, the Government established the Fixed Nitrogen Research Laboratory in Washington D.C.

APPENDIX

This section describes the operations and equipment of U.S.N.P. No. 2. The information and data is based on a number of sources, including documents of the War Department's Nitrate Division, consisting of a description of the plant and a report of the two weeks test run that occurred in the spring of 1919.¹²² In 1938, J. F. Carle of the Tennessee Valley Authority produced a highly detailed description of the layout, process, and equipment of U.S.N.P. No. 2.¹²³ This latter TVA report remains the most detailed analysis of the plant. In addition, trade and industry journals of the time published exceptional articles, describing the plant, process and equipment.¹²⁴ This appendix attempts to synthesize these descriptions to provide a complete description of the plant. For more details consult the noted sources, all of which are available at the library of the Tennessee Valley Authority's Environmental Research Center, Muscle Shoals, Alabama.

The Plant

The government reservation on which U.S.N.P. No. 2 was situated embraced a 2300 acre tract of land in Muscle Shoals, Colbert County, Alabama. This land is located on the south bank of the Tennessee River. The plant itself was comprised of sixteen buildings relating directly to production and numerous auxiliary buildings for storage, washrooms, machine shops etc. The plant buildings were oriented north to south and the process of manufacture followed generally this orientation. The plant buildings were constructed with steel framing and concrete foundations and floors. Large monitor roofs on each building

¹²² War Department Office of the Chief of Ordnance Nitrate Division, Brief Description of United States Nitrate Plant No. 2, May, 1919 (TVA Technical Report 118195); War Department Office of the Chief of Ordnance Nitrate Division, Report of Two Weeks' Operating Test of U.S. Nitrate Plant No. 2, March 31, 1919 (TVA Special Report S-8). See also: Nitrate Division, Ordnance Office War Department, Report on the Fixation and Utilization of Nitrogen, (Washington: Government Printing Office, 1922)

¹²³ J. F. Carle, U.S. Nitrate Plant No. 2, Muscle Shoals, Alabama. (Tennessee Valley Authority, Special Report # 7. 19)

¹²⁴ For example, see: Andrew M. Fairlie, "Muscle Shoals Nitrate Plant" Chemical and Metallurgical Engineering 20. No 1 January 1, 1919. pp.8-17.

allowed light to enter through an expanse of windows. Building walls were constructed of a handsome clay building tile supported by structural steel columns spaced 25' apart, generally, and roof trusses spanning 50'.¹²⁵

The production of ammonium nitrate at U.S.N.P. No. 2 was based on "fixing" atmospheric nitrogen in the form of calcium cyanamide, producing ammonia as a by-product from the cyanamide, oxidizing the ammonia to form nitric oxide, absorbing the oxides in water to produce nitric acid, reintroducing ammonia into the nitric acid to form ammonium nitrate liquor, then evaporating or crystallizing the liquor to form ammonium nitrate. The plant can be analyzed in a number of ways. The most basic division would separate it into two parts: the dry end and the wet end. The former would include the buildings, equipment, materials and machinery for processing the raw materials, limestone, coke, air and water, to produce calcium cyanamide. The second or "wet" processes utilized equipment to convert the cyanamide into ammonia, eventually leading to the production of ammonium nitrate. The buildings that related directly to the process and their approximate dimensions were as follows:

Lime Kiln Building: 200' x 240'
Coal Mill Building: 50' x 120'
Raw Material Building: 50' x 100'
Carbide Furnace Building: 90' x 1,050'
Carbide Cooling Shed: 50' x 950'
Carbide Mill Room: 150' x 120'
Liquid Air Building: 100' x 575'
Cyanamide Oven Building: 250' x 520'
Cyanamide Mill Room: 150' x 120'

¹²⁵ It has been surmised by industrial historians that this structural design, known as "blow-out" construction, was to allow any accidental explosions to dissipate their energy through the hollow-tiled walls, leaving the buildings, and hopefully plant employees, to remain in repairable condition. Such construction was used at the U.S. Ordnance Corps' Picatinny Arsenal in Dover, New Jersey and visitors to both sites have noted similarities between their design and construction.

Hydrator and Silo Building: 106' x 136'
Process Steam Plant: 46' x 120'
Autoclave Building: 80' x 250'
Catalyzer Building (6): 50' x 180'
Nitric Acid Building: 100' x 600'
Neutralizer Building: 90' x 150'
Ammonium Nitrate Building (5): 60' x 120'

In addition to the above, the government established a limestone quarry operation in adjacent Franklin Co., Alabama. When completed, this quarry operation consisted of twenty-one permanent buildings and the requisite grinding and crushing units.

Power for the long-term operation of the plant was to be supplied by a large hydroelectric facility on the Tennessee River. As wartime demand for explosives reached critical levels in 1918, an auxiliary steam power plant of 60,000 K.W. capacity was erected on the reservation in order to provide immediate power. In addition, the government paid for the installation of an additional 30,000 K.W. steam generator and an 80-mile transmission line from an existing steam power plant of the Alabama Power Company in Gorgas, Alabama.

Waco Quarry

In the production of calcium carbide at U.S.N.P. No. 2, the wartime manufacturing requirements of the plant called for a supply of crushed limestone amounting to approximately 1,200 tons a day. The first contract for this supply of limestone was arranged by the Air Nitrates Corporation with the firm of Foster & Creighton, operators of a limestone quarry and crushing plant located about 26 miles south of the plant near the small town of Rockwood, Alabama. This agreement with Foster & Creighton was dated January 16, 1918. The stone required was to be crushed to 1 $\frac{1}{2}$ " to 2" in size.

In late March of the same year, the government, seeking a larger and better supply of limestone, commenced a geologic survey of the region. This survey uncovered an adequate site known as "Hill Property," a deposit of oolitic limestone which was to become Waco quarry. The site, located 6 miles east of Russellville, Alabama, comprised 440 acres of private land.

Crushing and processing equipment for the Waco site was ordered and stored at the nitrate plant until authorization to purchase the land was given on January 4, 1919.

Meanwhile, the Rockwood quarry produced far less limestone than expected and was taken over by the government after two weeks of operation. Additional equipment was added that brought production up to levels adequate for testing operations at the nitrate plant.

Operability of the Waco quarry was not confirmed until the summer of 1921. By contract with the firm of Arthur McKee Company of Cleveland, Ohio, the equipment stored at the nitrate plant was installed and the quarry was given a test run on June 14. The test was successful and the quarry and its equipment was put in idle stand-by.

Raw Material Department

The operations of this department consisted of preparing the raw materials for use in the carbide department. Limestone and coke were brought into the plant over the west main line of the plant railroad system and unloaded from a trestle at the north end of the manufacturing area. The limestone and coke were either stored or placed directly into the process by means of a system of discharging bins and belt conveyors.

OPERATION

Limestone, arriving by bucket elevators and belt conveyors, was dumped into a large silo measuring 14' by 175'. The limestone was discharged from the bottom of the silo and was burned (calcined) in seven rotary lime kilns, 125' long by 8' in diameter. Each kiln had a capacity of 100 tons per day of lime, and six kilns supplied the plant, leaving one in reserve. The kilns were fired with powdered coal, giving a maximum temperature of 1,600° to 2,000°F. The kilns were pitched to induce the movement of the limestone from charging end to discharging end and revolved at a rate of 1 minute and 30 seconds per revolution. At the end of each kiln was a rotary cooler, 50' long by 5' in diameter. The lime passed through the coolers and was conveyed to bucket elevators which brought the limestone to another set of belt conveyors that led to the lime silos.

Coke was prepared in a coke dryer building. It was crushed by two roll crushing units to $\frac{3}{4}$ " size, conveyed and deposited in silos, and then fed into and dried in four rotary dryers, 5' 6" in diameter by 40'. Dried coke discharged from the

end of the dryer into a dust chamber where coke dust settled and was removed while the dried coke was conveyed to the dried coke silos.

The eight silos into which the prepared limestone and coke were deposited (four silos for each) were made of monolithic reinforced concrete and held approximately 700 tons of lime and 425 tons of coke. The charge for the carbide furnaces was proportioned under these bins and the mixture was delivered by belt conveyors to hoppers at each carbide furnace.

It should be noted here that the coal used to fire the kilns and dryers required its own coal mill building equipped with its own dryers, crushers, and mills.

* * *

Production efficiency of the lime kilns was very high during the two week test period, with a rate of 104.6 percent of rated capacity. The kilns were estimated to be able to produce at full capacity nearly 735 tons of lime per day. However, complaints arose regarding the limestone acquired from the Rockwood quarry. This stone was said to be soft, and to disintegrate into a fine dust by the time it exited the kiln. Although the dust was used in the furnaces, extra labor was needed to screen and remove the excess amounts from the cooler.

The coke dryer performed at or beyond its rated capacity.

Estimated labor requirements for 100 percent capacity operation of the Raw Material Department included superintendents, clerks, and shift foremen assigned to both the coal and coke milling departments. The following direct labor was estimated as being required for each department operating six kilns 24 hours per day (three shifts):

COAL MILL

3 Dryer Operators
3 Pulverizer Operators
3 Conveyor Operators
3 Laborers

LIME KILNS

3 Sub-foremen
9 Kiln Operators (2 kilns to one man)
9 Conveyor Operators
8 Laborers

COKE MILL (labor requirement based on supply of coke for 100 percent operation of ten carbide furnaces)

3 Dryer Operators
2 Crusher Operators
2 Conveyor Operators
2 Laborers

In addition to these workers, foremen, engineers, firemen, and laborers amounting to a total of seven additional employees were estimated as required to handle the raw materials as they entered the plant.

Carbide Furnace Department

The Carbide Furnace Room consisted of twelve carbide furnaces, each of a capacity of about 48 tons per day. The building was divided into two floors, a charging floor and a tapping floor. The tops of the open hearth furnaces were flush with the charging floor and the furnace hearth extended below towards the tapping floor, the bottom of the furnace being supported on 3' concrete piers. There was a rectangular stack over each furnace for carrying out of the building the dust and carbon monoxide gas that resulted from the furnace operation.

A narrow gauge rail system was set into the tapping floor in conjunction with a system of winches, chill cars, and battery-operated electric locomotives, all of which served to convey the molten carbide to the Carbide Cooling Shed.

The furnaces were of the arc-resistance type, 22'- 6" long, 13' -1" wide and 9'- 0" deep. The electric current came to the building at 12,000 volts, three phase, and was stepped down in water-cooled transformers of 2775 K.V.A. capacity, three of which were situated at each furnace. Three electrodes were used in each furnace, one for each phase. Each electrode consisted of three 16" x 16" x 84" carbons bound together with 1" mesh woven wire and cemented together and coated over the outside with a mixture of retort cement and asbestos. Each electrode was fitted with a water-cooled copper head secured by means of copper pins extending transversely through the carbons. The electrodes were assembled in an electrode shop located at one end of the Carbide Furnace Room. The electrode shop was fitted up with four hand-fired ovens for baking the covering onto the electrodes, two planers, two 30" radial drills and one wet grinder.

The electrodes, each weighing with their cooling heads 3,000 pounds, were conveyed to and from the electrode shop by means of special 4-ton cranes that traveled upon a system of monorails

which ran above the furnaces. Each furnace has three monorails, one monorail for each phase. The electrodes were suspended above the furnaces on steel beam supports spaced 4' apart and were lowered and raised by a system of cables, sheaves, counterweights, and electrically-powered winches with a solenoid control. The amount of current that flowed through each phase was regulated by raising or lowering the electrode. This regulation was accomplished by means of hand regulators which operated the winches or automatically through a switchboard.

OPERATION

The bottom of the furnace was first covered with a layer of spent electrodes (stubs obtained from the heads of the electrode holders) on top of which was placed a layer of coke about 6" thick. The current was turned on and the electrodes were lowered until in contact with the coke, which completed the circuit. The load was permitted to build up gradually to the desired amount at which point the furnace was hand fed with a coke and lime mix as the electrodes slowly rose. The charge was continually built up in order to keep the molten bath of carbide below the surface of the charge. Thus, by keeping the surface "dark," the electrode heads and connections were subjected to as little heat as possible. After approximately 40 minutes, the taphole, which had been filled with cement prior to furnace operation, was burnt through with a portable graphite electrode. The molten bath of carbide was allowed to flow into an awaiting chill car. When the car is full, the taphole was plugged with a shovelful of carbide, which was thrown into the taphole and packed firmly into place with an iron rammer.

The chill car was pulled from the tapholes by an electric locomotive over a set of scales from which it was taken into the cooling building. The body of the car was lifted off the truck and set on the floor to cool for about 40 hours.

* * *

During the two-week test, only two of the twelve carbide furnaces were in operation. Nonetheless, production estimates and furnace capacities could be generated; 1472.06 tons of what was called "crude carbide" was produced. The carbide content of this product was estimated at 81.6 percent. The amount of 100 percent calcium carbide produced came to 1201.62 tons. Despite some downtime for power trouble, the furnaces produced beyond their rated capacity, operating at one period for 24 hours a day. For the period of operation, the capacity of the furnaces was rated at 111.76 percent.

Each furnace required five second-class furnace men, one

stationed at each corner of the furnace to shovel in the charge and one to bring lime and coke via wheelbarrow from the adjacent silos. It should be noted here that, although the charge would be a proportioned mixture of both lime and coke, achieving the desired product required adding small amounts of either pure lime or pure coke. This latter procedure explains the fifth furnace (wheelbarrow) man.

The graphite tapping pencil was connected to the phase No. 1 electrode which formed a circuit with the phase 2 electrode. At times during the test run, the flexible cable attached to this tapping pencil would short-circuit when coming into contact with the tapping platform, leading to the recommendation of asbestos paper wrapping for the cable. Furthermore, unlike American Cyanamid's Canadian plant, the tap hole on the U.S.N.P. No. 2 furnaces was inconveniently high, necessitating an improvised steel platform. This arrangement caused consternation amongst the tappers as they worried about falling on the steps of this platform while in the act of dodging molten carbide.

In the electrode shop, spent carbons would have to be removed from their bolt and cotter-pin holders. This became a hot and frustrating task as the pin would swell under such intense heat, leading to the plea for a "quick-clamp" connection. As mentioned, the stubs of the spent electrodes would be used to line the bottom of the furnace, serving together as the circuit completion electrode for the suspended electrodes above.

The two weeks test run report contained many recommendations for improving operations but was clear to point out that the furnaces "easily exceeded their rated capacity regularly, and their general may be said to have been successful."

The labor estimated as being required for the 100 percent operation of the Carbide Furnace Department was as follows (based on one shift):

- 1 General foreman
- 1 Sub-foreman
- 3 First-class furnace man
- 50 Second-class furnace men
- 10 First-class tappers
- 10 Second-class tappers
- 3 Locomotive drivers
- 3 Locomotive driver's helpers
- 2 Beltmen
- 2 Beltman's helpers
- 1 Weigher
- 1 Weigher's helper
- 2 Sweepers

1 Clerk

The estimated crew required for the care and maintenance of the electrodes in the electrode shop was based on six eight hour shifts per week producing sixty electrodes:

1 Foreman
2 Drillers
2 Planers
4 Pipe fitters
4 Assemblers
4 Assembler's helpers
2 Cranemen
2 Laborers
1 Sweeper

Carbide Mill Department

Before calcium carbide could be charged into the lime nitrogen ovens, it had to be ground to a fine powder and have fluorspar added. The Carbide Mill Building was equipped with three 30" by 42" Buchanan jaw crushers, three 10' diameter by 48" Hardinge ball mills, and three 7' diameter by 24' Smidth tube mills, all of which served to reduce the carbide to a powder and produce an intimate mixture of fluorspar and carbide.

OPERATION

When the carbide in the chills had cooled, a travelling crane picked up the chill and brought it to one of three concrete tipple blocks, one tipple located in front of each crusher. The chill was lowered and was hooked onto lugs projecting from the top of the tipple. The chill was quickly released and the carbide would fall about 10' onto an inclined breaking floor, which led directly to one of the jaw crushers. The material was allowed to feed to the crushers gradually, the larger lumps being worked forward to the mouth of the crusher by workmen with steel bars. The jaw crushers crushed the carbide to 2" size. Bucket elevators dumped the crushed carbide into bins located above the feed ends of the Hardinge ball mills.

The Hardinge ball mills were each loaded with 25 tons of chilled steel balls ranging in size from 3" to 5". The mill revolved at 16 revolutions per minute and reduced the carbide to a powder. Screw conveyors brought the powdered carbide to a large silo that fed each tube mill.

The Smidth tube mills were packed with 32 tons of chilled steel cylinders (cylpebs) which further pulverized the carbide

into a fine powder. These mills revolved at the rate of 24 revolutions per minute. The fluorspar (3 percent of the mixture) was added into the tube mill with the carbide and originated from its own silo adjacent to an array of screw conveyors that fed the mills.

To prevent the formation of acetylene from moist air and carbide and possible explosions of acetylene mixed with air, the ball mills and tube mills were filled with nitrogen gas throughout the milling process. In addition, the screw conveyors and bucket elevators were enclosed and fed with nitrogen gas.

* * *

The crushers crushed a total of 1395.51 tons of carbide during the test and preformed at nearly their rated capacity. The mills, however, varied greatly in their performance. The Hardinge ball mills, for example, were rated to turn out ground carbide at the rate of 16 tons per hour, but the rate of the test turned out to be 10.52 tons per hour. Pushing the mills any further resulted in "large quantities of unground lumps of carbide, which caused great trouble subsequently in the conveyors and Smidth mills. Mr. Hardinge himself had come down to the plant and "ordered that six or seven inch balls be substituted for the three, four and five inch balls" then in use. The ball change, so Mr. Hardinge said, would result in capacities of 16 to 20 tons per hour. By contrast, the Smidth mills performed excellently, their only problem being having to be fed "light" because of the poor performance of the ball mills. Running light resulted in the tendency of the mills to heat up and conduct that heat to the bearings, but one of the three mills was equipped with water-cooled bearings, handled the excess heat, and led to the recommendation of adding the same bearings to the other two mills.

The crew (1 shift) estimated for operating the Carbide Mill Room at 100 percent capacity was made up of the following:

- 1 General Foreman
- 1 Crusher Operator
- 1 Hardinge Mill Operator
- 1 Smidth Mill Operator
- 1 Smidth Mill Helper
- 1 Conveyor Man
- 1 Laborer
- 1 Sub-foreman
- 1 Locomotive Driver
- 2 Cranemen
- 2 Chill Car Operators
- 4 Crusher Laborers

Nitrogen Department Liquid Air Building

Atmospheric nitrogen for the nitrogenation of carbide, as well as for the Carbide Mill Room, was secured by the fractional distillation of liquid air. The Claude process for producing nitrogen was used, under patents controlled by Air Reduction Company, Inc., of New York. The production of nitrogen was performed in a separate building located well to the east of the plant. The building housed the single largest assembly of liquid air apparatus in the world: sixteen air purification towers, fifteen air compressors, thirty Claude liquid air columns, all of which produced nitrogen at the rate of 17,657 cubic feet per hour.

OPERATION

Air was drawn into the building through a 36" spiral riveted pipe that extended north and south from the building 1,600' in each direction, with valves so arranged that the air might be drawn through either the north or the south branch in order to secure that containing the least amount of acetylene gas and carbon monoxide. Quantities of both these gasses were produced in connection with the operation of other parts of the plant, the direction of the wind determined to which intake these would be blown. Both gases were detrimental to the nitrogen-making equipment. For the purpose of purification, the air passed through sixteen caustic towers, 30' high and 9 $\frac{1}{2}$ ' in diameter, each tower being packed with earthenware spiral rings. The first eight towers contained a solution, more or less spent, of caustic soda, and the others contained fresh solution which was pumped to the top of the towers and ran down over the spiral tile. The spent solution was returned to the caustic building in the autoclave group for recausticizing, the solution from the second eight towers being transferred to the first eight and a new solution put in the second eight towers, as required.

After purification, the air was drawn through separators to eliminate particles of caustic and moisture. From the separators the air went to the compressors. The air was compressed to about 500 pounds pressure per square inch, leaving the compressors at about 150°F. It then entered a cooler where it was reduced to room temperature, passed through an oil separator where practically all of the oil was removed, and then went to the Claude liquid air columns.

The air was cooled in one of the two heat exchangers in each liquid air column. This exchanger was constructed like a steam condenser, the out-going nitrogen and oxygen passing around the

outside of the tubes and thereby cooling the incoming air to about -100°C . One exchanger was used until it was necessary to thaw out the accumulated ice when the other exchanger was put into service. From the exchanger a small portion of the air, about 10 percent, went to the liquefier, the remaining 90 percent passing through an expansion engine where the work of expansion produced a proportionate extraction of heat. The air liquefied in the liquefier mixed with the exhaust from the expansion engine, formed a heavy vapor which rose through tubes surrounded by liquid oxygen which, by proper regulation, lowered the temperature to the liquefaction point of oxygen, condensing oxygen with considerable nitrogen from this heavy vapor. The remaining cold gas, high in nitrogen, passed up through serpentine coils surrounded by liquid oxygen and was there liquefied, this liquid continued to the top of the rectifying column where it spilled out on the rectifying trays. These rectifying trays were so constructed that the gases rich in oxygen from the first mixture of liquid air and exhaust from the expansion engine, were forced to bubble up through the pure nitrogen, a heat exchange took place whereby the nitrogen was vaporized and the oxygen condensed, to run down and give up its cold by liquefying the nitrogen in the serpentine coils and condensing the oxygen from the first mixture of liquid air and expanded air as spoken of above. Nitrogen gases came off at the top of the rectifying column with an average purity of about 99.8 percent. The liquid oxygen that accumulated in the bottom of the apparatus was mostly vaporized in the process of giving up its cold, that not required for this purpose was drawn off as a liquid and thrown away. The regulation of temperature for the separation of oxygen and nitrogen had to be kept within 13 degrees Centigrade, oxygen liquefying at -182° and nitrogen at -195°C .

* * *

The nitrogen plant saw few problems in its operation and produced nitrogen gas at or beyond its rated capacity. The test report concluded that "...the operation of the Plant from day to day was so uniform that the daily log sheets call for no comment." The suggested daily labor force for 100 percent operation was as follows:

1 Superintendent		
1 Clerk		
3 Shift Foremen (1 per shift)		
15 Column Operators (per shift)		
3 Compressor Operators	"	"
2 Soda Tower Operators	"	"
1 Laborer	"	"

1 Switchboard Operator " "
1 Mechanic " "

Lime Nitrogen Department Cyanamide Oven Building

The Lime Nitrogen Building or Oven Room was divided into five long bays, the four outer bays containing the ovens and the center bay reserved for storage and for the manufacture of paper cylinders used in charging the ovens. In the four outside bays were sixteen rows of ovens, 96 in a row, making a total of 1538 ovens. The rows of ovens were in pairs, two double rows in each oven bay, and were charged with carbide by four charging cars, one car for each bay. Each oven bay was also served by a 5-ton travelling crane. On the south end of the building was the cooling shed and the Lime Nitrogen Mill Room.

The lime nitrogen ovens each consisted of an outside shell of steel set on a reinforced concrete disc. The shell was lined with 2" of infusorial earth and 8" of fire brick. The inside measurement of the oven was 34" in diameter and about 60" high. The oven was covered with a hollow iron disc filled with infusorial earth. Over this cover was the cover proper, which was made tight by means of an inverted flange, dipping into a sand seal. Electrical connections were provided in the center of the top and bottom of the oven. In the top was a small vent which allowed a slight circulation through the oven, assuring that there was no concentration of inert gases. Each oven was provided with two inlets for the nitrogen gas, one located at the bottom of the oven, and the other on the side of the oven near the bottom. The necessary temperature for starting the reaction between the carbide and the nitrogen was obtained through the medium of a narrow carbon electrode or "pencil" 12 millimeters in diameter and 78" long.

OPERATION

Powdered carbide was conveyed from the Carbide Mill Room into a bank of eight receiving silos in the Lime Nitrogen Building. Each of these silos had four discharge mouths through which carbide was loaded into hoppers. The hoppers then unloaded the carbide into four charging cans located on the electrically-powered charging car which ran on tracks over the row of ovens. The car then ran to a bank of four ovens, the cans were lowered to the ovens, and approximately 1,600 pounds of pulverized carbide discharged through the bottom of each can and into its corresponding oven.

Before this charging procedure, a heavy paper cylinder 29"

diameter and a 3" paper tube in the center of the cylinder were placed in the oven. Both the cylinder and tube were 5' long. The electrode pencil would be inserted into the tube. When ready for operation, the charge of carbide was carefully discharged into the space between the tube and the inside of the cylinder. After charging, the covers were placed on the ovens, the electrode was inserted into the center tube, the nitrogen was admitted, and the current was turned on at approximately 250 amperes and 100 volts. The paper tube surrounding the electrode would burn away after about 20 minutes at which point the current was reduced to 135 amperes and 50 volts. From 8 to 12 hours after starting, the reaction in the oven became exothermic and the electrode was removed. The nitrification of the carbide took approximately 40 hours.

To remove what was now a cyanamide pig or "nitro," a pig hoisting clamp was lowered and secured around the pig, and the pig was then lifted out of the oven by a crane and put onto the discharging car. Once four pigs were on the car, they were carried to the cooling shed and transferred to an iron cooling platform that held nine pigs. After cooling, the platform was set on a tippie by a crane. A mechanical arm then pushed the pigs off the tippie, one at a time, onto a breaking floor from which the lime nitrogen tumbled directly to the Lime Nitrogen Mill Building.

The Lime Nitrogen Mill Department was a duplicate of the Carbide Mill Department, except that milling in an atmosphere of nitrogen was not necessary and that flint pebbles were used instead of steel cylinders in the Smidth tube mills.

* * *

For the most part, operations during the test were satisfactory, with some minor trouble with automatic measuring machines and the machine for gluing the paper cylinders and tubes. One apparatus that was a distinct failure was the charging car. The report accompanying the test declared the following:

"The charging car as it stands is about the worst piece of machinery in this group and gives trouble constantly due to a poor design for lowering and hoisting the cars when filling the ovens."

Immediate replacement of the car with a more reliable design was recommended.

The estimated direct labor requirement for 100 percent operation of the Lime Nitrogen Oven Room included the following:

5 Papermen
5 Chargers
5 Cranemen
5 Operators
5 Conveyormen
5 Laborers

In the Lime Nitrogen Mill Room, Hardinge ball mills ran below rated capacity for lack of an ample supply of material to mill. This was due to the feeding system which could not keep up with the appetite of the mills. The smaller than rated amount of cyanamide fed into the Hardinge mill created "a terrible noise due to the balls hitting against the sides." The Smith mills performed well, and after problems in the hydrating building that stifled their output were cleared up, they produced beyond their rated capacity.

The direct labor requirements for the Mill Room and Cooling Building included personnel to fill the following positions:

Sub-foreman
Craneman
Helper
Mill Operators
Conveyor Man
Operator's Helper
Jib Craneman
Jib Craneman's Helper
Silo Helper
Sweeper

Hydrating Building

The Hydrating Building consisted of a 150 ton feed bin with three discharge openings, each opening feeding a dual system of screw conveyors that led into a hydrator. The hydrators were each 36' long and 43" in diameter. Through each hydrator ran a shaft, along the length of which were paddles set every 3¹/₂" and 90° apart. The paddles were 16" long with 5" by 1" blades set at an angle of 45° so as to mix and move the material along through the length of hydrator trough to the discharge end. The actual hydration of the cyanamide occurred by means of a spraying apparatus located under the discharge from the screw conveyor. A vent for each hydrator extended upwards above each trough, the two outside vents angling towards the middle where all three met to form a central stack which passed through the roof of the building.

Storage for milled cyanamide was provided for in an adjacent

building consisting of 9 monolithic concrete silos, of a total capacity of about 5,000 tons. A system of conveyors and bucket elevators brought the milled cyanamide from the Mill Building and supplied either the feed bins or the storage silos. Cyanamide from the storage silos could be reintroduced into the conveyor system, thereby allowing the stored cyanamide to supply the feed bins.

OPERATION

The milled cyanamide was fed into the hydrators and a fine spray of water struck the material, setting off a reaction whereby the residual carbide in the cyanamide released acetylene gas. This reaction continued as the paddles, turning at 50 revolutions per minute, mixed and moved the cyanamide along the trough. The reaction produced heat, which vaporized much of the water that passed, along with the acetylene gas, through the vents and outside the building. A system of screw conveyors and bucket elevators moved the hydrated cyanamide to a trestle that linked the Hydrating Building to the Autoclave Building. Through this enclosed trestle ran another system of screw conveyors that brought the cyanamide into the Autoclave Building.

* * *

Operations in the Hydrating Building were discontinuous due to the sporadic demands for cyanamide in the Autoclave Department. Complaints arose regarding the screw conveyors as they would often become jammed with excess cyanamide. The recommendation was made to replace them with belt conveyors. Likewise, the single-chain bucket elevators were said to have given trouble due to "...chains breaking and piling up the buckets and chain in the boot of the elevator." Double-chain buckets were recommended.

Direct labor positions were not listed.

Autoclave Building and Ammonia Gas Department

In the Autoclave Building, fifty-six autoclaves divided into seven units of eight autoclaves each, extended through two operating floors and were supported on the bottom floor by an interconnected network of steel framing. Each autoclave was 21' high and 6' in diameter. On the top operating floor, the autoclaves extended about 3' above the floor surface. On the middle floor, the bottom of the autoclave was about 6' above the floor surface. Cyanamide was stored in fourteen hoppers and a system of screw conveyors brought the cyanamide to the autoclaves where it was fed into them through a canvas spout. In addition

to the above were scale hoppers for measuring the cyanamide charge, and a storage bin for the soda ash.

Adjacent to the Autoclave Building were two filter buildings and a caustic soda building. The Filter Building contained two sets of rotary filters, ten filters to a set. The filters were fed by means of sludge troughs that ran from the Autoclave Building. Process tanks, centrifugal pumps, filtrate storage tanks, and filtrate measuring tanks rounded out the equipment in the Filter Building. The main feature of the Caustic Building was a 230 foot screw conveyor that brought soda ash into the Autoclave Building. In the Caustic Building were also located caustic make-up tanks where the caustic solution for use in the Liquid Air Building was prepared from soda ash, lime and water.

Located outside, between the Autoclave Building and the process boiler house, were seven sets of ammonia gas processing equipment, each set consisting of an ammonia column, a dephlegmator, and a condenser. The ammonia column stood ground-level on a concrete pedestal while the dephlegmator and condenser were elevated on a platform which stood about 18' above the ground. A system of catwalks connected each set of equipment and three stairways descending down from the Autoclave Building adjoined the catwalk.

Description of Autoclaves

The autoclaves were pressure vessels made of 1-1/4" cast steel. They were 21' high by 6' in diameter with a top and bottom neck riveted on to receive the bearings for the vertical agitator shaft. On the top of the autoclave was a cast 8" charging neck, a 5" ammonia outlet, an 8" acetylene outlet, two 1/2" outlets for pressure and temperature gauges and an outlet for two 3" blowoffs. On the bottom was an 8" outlet for slurry and on the side near the bottom were three steam inlets and a compressed air inlet. The vertical agitator shaft had three sets of arms and a drag on the bottom. It was operated through the bottom by a set of motor-driven gears.

OPERATION

The operation of the autoclaves was controlled from the top operating floor. When ready to charge the autoclave, the operator signaled the Filter Building and about 2,000 gallons of "mother liquor" (sodium hydroxide solution) would be discharged into the autoclave and run to a depth of about 10'. The cyanamide spout was then placed over the autoclave and approximately 8,000 pounds of cyanamide and 300 pounds of soda ash were introduced. While the charge was being introduced into

the mother liquor the vents at the top of the autoclave were kept open and an exhaust fan removed any acetylene gas that formed. These vents were then sealed and steam was introduced at the bottom of the autoclave and was kept on until the pressure reached 60 pounds. At this temperature and pressure the reaction started and the steam was turned off.

The reaction then proceeded by itself, generating ammonia and steam and creating pressure that had to be relieved by means of safety valves. These valves could be connected with the ammonia main or the atmosphere by the operator. With the relief valve open the pressure rose to about 225 pounds in 15 to 20 minutes. As the gas was discharged into the ammonia main the pressure dropped off and the reaction was entirely complete in about an hour. The operator, always listening to the distinctive whistle made by the flow of gas, could tell if any foam or liquid from the charge was leaving the autoclave. If this occurred, the valves were cut down permitting, if necessary, ammonia to escape into the air through the safety valve rather than permitting foam or liquid to enter the ammonia main. When the pressure in the autoclave was reduced to that of the main, the outlet was closed and the operation was repeated a second and a third time, in order to free the mother liquor of the ammonia and to assure that the reaction was complete. The remaining gas was finally blown out of the autoclaves by steam. The time of the autoclave cycle was about eight hours and the approximate capacity was one ton of ammonia gas per cycle per autoclave. About 97 percent of the nitrogen content in the cyanamide was recovered as ammonia in this operation. The wet ammonia gas from the autoclaves contained about 75 percent steam and 25 percent ammonia.

After all ammonia had been exhausted from the autoclaves, the spent charge was drawn off to a sludge tank in one of the filter buildings. This sheet iron tank extended the full length of ten rotary vacuum filters, and had an agitator running through its entire length. The sludge was then fed from this tank to the filters, which had a 6' drum with a 3' face covered with canvas. The vacuum for the filters was maintained by three duplex air pumps. After filtering and washing with hot water, the residue was scraped off the filter drum and fell into a screw conveyor that discharged into a square, hopper-bottomed tank where water was added in such amounts as to permit handling by centrifugal pumps which passed the sludge to a dump. The sludge consisted mostly of calcium carbonate and graphite.

The filtrate was taken up by a vacuum from each filter and was stored in steel storage tanks that were equipped with cooling coils to bring the temperature of the solution as low as possible. From these storage tanks the liquid, which constituted the mother liquor, passed into measuring tanks where it was

accurately measured for delivery to the autoclaves.

AMMONIA GAS PROCESSING

Live steam from the Process Steam Plant was admitted at the bottom of the ammonia column and passed up through the first plate through holes covered with boiling cups. The temperature of the water on the first plate was around boiling point, driving off from the water all the ammonia that reached that point. In the fourth compartment from the bottom of the column entered steam and ammonia from the autoclaves. This gaseous mixture bubbled up through the remaining plates where some of the moisture condensed, the rising gas mixture consequently containing more and more ammonia.

The gas left the top of the ammonia column at about 170°F. and passed into a dephlegmator, which was a vertical, water-cooled tube shaped apparatus that eliminated moisture by condensation. The gas flowing out of the bottom of the dephlegmator would flow into the top of the second piece of apparatus, identical with the dephlegmator but called, in this instance, a condenser, where all the moisture was finally eliminated. The condensation from these two condensers flowed back into the ammonia column. This liquor flowing back into the column was richer in ammonia than the liquor in the column and was, therefore, not in equilibrium with the vapor in the column. An interchange of heat took place, steam being condensed and ammonia distilled which enriched the vapor passing into the condensers. This process of distillation without additional expenditure of heat is called "dephlegmation."

The ammonia gas from each unit entered a 28" ammonia main that ran to the intake end of the Catalyzer Building where the main divided, one branch going into the neutralizer building and the other connecting with two gas holders, each which had a capacity of 60,000 cubic feet.

* * *

The two week test of this department, dubbed "the wet end," was carried on by employees of the Air Nitrates Corporation. One basic issue that became an object of concern was that there was no accurate way to measure the amount of ammonia produced. Air Nitrates Corporation did present an estimate based on a ratio of steam used and ammonia produced arrived at from "their long experience and careful determinations" made at their similar facility in New Jersey, but the Ordnance Department officers in charge of the test declared:

It is hard to understand on what basis this is figured, as we have no facilities for measuring the steam used in the autoclaves, either at this Plant or at the "Warners Plant of the American Cyanamid Company" where these elaborate tests are "supposed" to have been conducted.

Certain valves on the autoclaves proved unsatisfactory as well as the acetylene piping line, which became plugged with cyanamide dust. The canvas feed nozzle also proved to be a problem as it became saturated with moisture.

Nonetheless, the autoclaves performed beyond their rated capacity and each consumed nearly 100 tons of cyanamide per day.

Direct labor positions were not listed.

Nitric Acid Department

This department consisted of facilities and equipment for the catalytic oxidation of ammonia gas, the cooling of nitric oxide gas, the absorption of nitric oxide gas in water, and the neutralization of nitric acid with ammonia. In addition, gas holders for ammonia gas, and storage and settling tanks for nitric acid and ammonium nitrate liquor were located adjacent to the processing buildings. Ammonia gas was held in two large gas holders, each 60' in diameter, single lift, water-sealed, and of a capacity of 60,000 cubic feet. These holders acted as pressure regulators. There were six catalyzer buildings, each containing on one end a meter room for mixing air and gas. The balance of the building housed 116 catalyzers arranged in four rows, 29 catalyzers to each row. The Cooling Building contained twenty-four high temperature coolers, twelve low temperature coolers, and twelve oxidation towers, each low temperature cooler handling the output of two high temperature coolers. The Absorption Tower Building housed twelve units of absorption towers, two 60' towers to each unit. In the Neutralizing Building there were four smaller absorption towers with two mixing tanks, four condensers, four neutralizing tanks, four settling tanks, one receiving tank and three filter presses. There were also eight settling tanks located outside the building.

The main absorption towers were 31'- 6" x 34'- 2" square by 60'- 0" high and were divided into four compartments. The towers were built of acid-proof brick and were packed with spiral tiles to provide the greatest possible surface area and to increase the turbulence of descending liquid, two important elements in the absorption process. The pumping of liquid was accomplished by a system of air lifts and acid wells, the wells being sunk to a

depth of about 100'. There were ten wells for each set of two towers amounting to a total of 120 wells.

OPERATION

Ammonia gas from the gas holders was mixed with air in a steel tank 36' high 9' in diameter. This tank was packed with 6" spiral rings to induce the thorough mixing of the air and ammonia gas. The composition of this mixture was about 10 percent ammonia and 90 percent air. The mixture flowed from the mixing tank under slight pressure to the catalyzers. The catalyzer consisted of a rectangular aluminum body over the bottom of which was the platinum catalyst, a mesh platinum gauze that measured 13" by 27". The gauze was electrically heated to a temperature of 750 to 800°C. The nitric oxide gasses from the catalyst flowed down through a ribbed cast iron spout that cooled the gas before it passed through a 7" circular outlet into a brick acid-proof duct under the floor which discharged into a flue in the adjoining cooling building.

The nitric oxide gas entered the cooling building and passed into a high temperature cooler, which was essentially a steam boiler. The hot nitric oxide gas was further cooled as it generated steam inside the boiler. The gas exhausted into an aluminum duct and then passed into the low temperature cooler. This cooler was constructed of acid-proof masonry and had five compartments, the gas passing from one to another through the series of five. The cooler was packed with duriron and stoneware tubes through which cooling water circulated. The gas was cooled to approximately 30°C. In the low temperature cooler, nitric acid began to form, and part of the nitric oxide became oxidized to nitrogen peroxide. The nitric acid was drained into a well of the absorption tower system. The gas, which was now a mixture of nitrogen oxide and nitrogen dioxide, passed to the oxidation towers. Each oxidation tower was divided into four compartments. The gas entered the tower and was circulated through the compartments, passing through holes at the bottom of one compartment and up and through holes on the top of the next. The gas then passed to the absorption tower building.

The gas from the oxidation towers passed into the first absorption tower and proceeded to circulate through all eight of the compartments of the two tower unit in a fashion similar to that of the smaller oxidation towers. The absorbing liquid flowed through these compartments counter-currently. Water was sprayed from the top of the last compartment of the unit and percolated downward, absorbing the gas to produce a dilute nitric acid. This dilute nitric acid was then pumped to the top of the

next compartment and descended, as did the water, absorbing gas to create an increasingly stronger solution of nitric acid. The acid continued this circulation through all the compartments of the unit.

From the absorption tower the acid passed to aluminum brick-lined weighing tanks, twelve in number, each covered with an acid-proof brick dome. From these tanks the acid passed to storage tanks and then, when needed, passed to the Neutralizer Building.

Ammonia from the Autoclave Building was led directly into the neutralizer through four pipe inlets located at the bottom of the tank. Ammonia gas exited the pipes, bubbled through the nitric acid in the tank, and through a process of neutralization, formed a solution of ammonium nitrate. This type of reaction generates heat, forming ammonia gas and water vapor which, in this case, was sent to a cooler of similar design of the low temperature cooler previously mentioned. In this cooler, the moisture was condensed and the remaining ammonia gas was sent to an absorption tower while the condensate was sent back to the neutralizer. In the absorption tower, nitric acid descended while ammonia gas ascended, forming a partially neutralized solution which was mixed in a mixing tank and then used for fresh solution in the neutralizing tank. The ammonium nitrate solution went from the neutralizer to storage/settling tanks. Sediment that formed in the tank was periodically filtered in Sperry filter presses. This sediment was generated by the temporary corrosive action of acid on the new iron and aluminum piping in the absorption and neutralizing systems. It was felt that after six months of operation all the piping would be purged of its soluble constituents and the filter presses would no longer be necessary. Ammonium nitrate solution was pumped, when needed, to storage tanks in the ammonium nitrate houses.

* * *

During the two week test, two units of equipment were operated in the oxidation and acid section of the plant, each unit consisting of fifty catalyzers, one high temperature cooler, one low temperature cooler, one oxidation tower, and two absorption towers. Two basic problems arose in the Catalyzer Building during the test: an inadequate and fluctuating power supply, and acetylene and phosphine in the ammonia gas. The power problem resulted in low gauze temperatures, necessitating the raising of the ammonia content of the ammonia gas/air mixture. The acetylene problem originated with the operation of the autoclaves and

...On several occasions, there were sudden shots of very bad

gas which blackened the screens and caused familiar phosphine spots to form.

Adjustments were made in the operation of the autoclaves and the catalyzers to help alleviate these problems. Recommendations were made to establish a more stable supply of power and requests were made to install an emergency switch that would allow all power to the catalyzers to be shut off "...in case of a heavy shot of gas or a too high ammonia mix," each of which could cause an explosion.

It was found that during operation of the high temperature coolers and catalyzers, unhealthful conditions relating to excess heat and improper ventilation resulted. This led to the suggestion of lagging the flues and catalyzers with asbestos and providing fans to cool the buildings.

Filters for the ammonia gas line were recommended as the small cloth filters on each catalyzer became easily fouled, a situation that could lead to damaging the expensive platinum gauze.

The coolers were said to be too small, "...causing the absorption tower to become heated to a high temperature, thus lowering the efficiency of absorption."

No direct labor positions were listed.

Ammonium Nitrate House

Each of the five ammonium nitrate houses included a storage tank, ten evaporating pans and twenty crystallizing kettles (two kettles to each pan). The evaporating pans were enameled on the inside and fitted with steam coils for evaporation and air pipes for agitation. Each pan was 11'- 0" long, 6'- 6" wide, and 2'- 2" deep. The crystallizing or graining kettles were fitted with an agitator and were water-cooled by means of a jacket that surrounded the kettle. This jacket could also be steam-heated. The kettles measured 6'- 0" in diameter by 2'- 2" deep.

OPERATION

Steam from the Process Steam Plant was let into the coils and nitrate liquor was then discharged into the evaporating pan. Air was fed through the air pipes and was released through holes, which agitated the liquor while evaporation ensued. It took about five hours to evaporate the liquor to the proper strength.

From the evaporating pan the concentrated solution was run into the cast iron crystallizer or grainer. It was necessary that the liquor was not rapidly reduced in temperature, so the kettles were first heated by running steam into the steam/water jackets. Agitators revolved, which kept the material agitated while it was gradually being cooled. At the discretion of the operator, the steam was turned off and water was let in, which further cooled the liquor while ammonium nitrate salts began to form. The grainers discharged ammonium nitrate onto a portable belt conveyor which conveyed the material to a storage and loading room at the end of the building.

* * *

Most of the comments regarding this department, as well as the nitric acid section, centered on the incompetency, according to Ordnance Department officers, of the Air Nitrates Corporation operators. Data and statistics on ammonium nitrate produced was said to be indeterminable due to "...the continued careless operation throughout the test." Aside from this human element of operation, problems with the drives on the crystallizing kettles and other mechanical flaws were noted.

No direct labor positions were listed.

Perhaps the concluding remarks of this part of the test report best illustrate what was one the characteristic features (flaws) of the "wet end" of U.S.N.P. No. 2

...To those who were on the ground witnessing the operation of this plant day after day, it is obvious that the operating management of the Air Nitrates Corporation is entirely indifferent to the actual cost per pound for the production of ammonium nitrate.

It has been found, in taking the operator's reports during this test and endeavoring to determine the final quantities, that the management of this particular group has never really known what they were doing from a commercial point of view as regards the operation of the equipment.

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