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H. FOSTER BAIN, DIRECTOR

THE CHLORIDE VOLATILIZATION PROCESS OF ORE TREATMENT

BY

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With an Introductory Chapter by

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PREFACE.

At its tenth regular session in 1913 the Utah Legislature provided for the establishment of a metallurgical research department in connection with the State school of mines of the University of Utah. As stated in the act providing for this department (Laws of Utah, 1913, ch. 102, sec. 2, pp. 199–200), the purposes of this research department have been to conduct experiments and research, either alone or in cooperation with the Federal Bureau of Mines and other agencies, with a view of finding ways and methods of profitably treating low-grade ores and of obtaining other information that shall have for its object the benefit of the mining industry and the utilization and conservation of the mineral resources of the State, and to publish and distribute bulletins and articles relating to the department and its work.

This act became effective in July, 1913. In January, 1914, the university made a working agreement with the Federal Bureau of Mines. By the terms of this agreement the work of the metallurgical research department is under the direction of metallurgists of the Bureau of Mines assigned to duty at the university and Salt Lake City. From January, 1914, to July, 1916, D. A. Lyon, metallurgist, was in charge of the work, assisted by O. C. Ralston and other members of the metallurgical staff of the bureau who were on duty at Salt Lake City.

As a part of the cooperative agreement the University of Utah provides six metallurgical fellowships. The fellowships are awarded to graduates of colleges, preferably of mining schools, who have shown special aptitude for research investigations. Their employment extends over the entire 12 months.

Persons desiring further information regarding the cooperation between the university and the bureau are referred to Bulletin 157, Bureau of Mines, pages 9 to 11.

H. Foster Bain,
Director.

THE CHLORIDE VOLATILIZATION PROCESS OF ORE TREATMENT.

By Thomas Varley, E. P. Barrett, C. C. Stevenson, and R. H. Bradford.

INTRODUCTION.

The art of treating ores by the chloride volatilization process is still in the experimental stage. The process has not been sufficiently developed along metallurgical lines to warrant a definite statement as to the exact place it will occupy in the industry. The basic theory of the process has received the attention of prominent metallurgists for 20 years, and considerable research and experimental work have been carried on.

Much of the experimental work done has never been published; if the accumulated results were made known, they would greatly aid the further development of the process. This bulletin aims to bring the salient features of the process to the attention of metallurgists for the purpose of furnishing information to and receiving comments from interested parties with the ultimate hope that the process will be a commercial success.

If commercially utilized, the process will fill a long-felt want in metallurgy, especially in the treatment of oxidized and semioxidized or "carbonate" ores of copper, lead, and silver. Such ores are difficult to treat by gravity concentration or by flotation: in the former their tendency to slime upon crushing and their being of lower specific gravity than the sulphide minerals cause serious losses; in the latter much has been done in sulphidizing oxidized ores and subsequently recovering the artificial sulphides by flotation. Difficulties in proper sulphidizing and the low recoveries obtained have not balanced the cost of the treatment in many plants and in very few has it proved successful.

Evidently there is a big void to fill in the treatment of these ores. No radical changes in present methods are forecast, but it is obvious that chloride volatilization can have a distinct place as a method of treatment for ores that are not readily amenable to present methods. In many plants it might replace concentration methods, especially where part of the mineral content in the ores exists in forms other than sulphides.

In addition to the minerals named above, gold in ores has been readily volatilized. Zinc does not form a volatile chloride, if an oxidizing condition is maintained in the roasting operations. Some excellent results have been obtained with carbonate-zinc concentrates containing silver and lead. Practically all the silver and lead were volatilized, but very little of the zinc. Experiments have proved that the process is probably one of the best for making a clean-cut separation of lead and zinc.

Not much work has been done on sulphide ores, because the need is pressing for a volatilizing method for treating oxidized and carbonate ores. Experiments on sulphides have been, however, encouraging. When the sulphur content is above 5 per cent a preliminary roast is usually necessary before chloride roasting and volatilization.

The effect of sulphur is discussed by Croasdale in his chapter on the history of the process. He and other investigators declare that sulphur is a consumer of salt, and when it is present in an ore in excessive amounts a proportionately larger amount of salt is required to effect the chloridization and volatilization of the metals present.

The process would doubtless be favorable for use in regions not well supplied with water. All the apparatus required for the construction of a plant are practically standard mechanical devices and machinery. The most important raw materials in the operation of a plant are the fuel and the salt or other haloid. In mining districts remote from railroads these materials, of course, would be the largest factor in the cost of running a plant. Nevertheless, the cost would often be more than offset by the freight charges on the concentrates and other supplies necessary for a plant using almost any other kind of process. These conditions will be discussed in some detail in describing the operations of plants that have been built.

The experiments described in this bulletin cover part of the investigations carried on by the United States Bureau of Mines, in cooperation with the department of metallurgical research of the University of Utah, on ores from all parts of the United States. Large quantities of different types of ores not amenable to any other process were available, and the method developed gives promise of great commercial importance.

DEFINITIONS.

Chloride volatilization.—Chloride volatilization may be defined as the process of separating or isolating certain metals from worthless gangue or other minerals by means of alkali or alkaline earth halides—such as salt and calcium chloride—which are added to the prepared ores in proper amounts, treated in a suitably designed furnace at temperatures high enough to form and to vaporize or volatilize the chlorides of the metals present. The exact chemical reactions are not definitely known. Vaporous chlorides of certain metals in the ores are formed and are drawn away from the heated charge into properly provided containers, whence they are recovered by some suitable means.

Chloridation.—Chloridation is the process of transforming certain metals in ores from their original mineralogical form to the chloride form by mixing the prepared ores with certain salts, then heating under proper conditions to temperatures not high enough to cause volatilization.

Treater.—Wherever "treater" is mentioned it definitely refers to the Cottrell electrical precipitator.

Fume.—The volatilized metal chlorides driven from the ores and collected in the treaters are called fume.

Calcine.—By calcine is meant the material or residue remaining after the extraction of the valuable metals by volatilization. All this material may be waste or it may include some valuable minerals not volatilized.

ACKNOWLEDGMENTS.

The writers of this bulletin acknowledge their indebtedness to Mr. Stuart Croasdale for his valuable article on the early history of the volatilization process.

They wish to acknowledge also the valuable services tendered by R. E. Head, microscopist, Intermountain experiment station, Salt Lake City, for help in the determination of the physical properties of the ores tested and the assistance given in preparing the photographs; to O. C. Ralston and Arthur E. Wells for their work on the process as pioneers in the Bureau of Mines, and their useful contributions; and to J. C. Morgan, former assistant chemist, Bureau of Mines, for his aid in the development of the process.

The following research fellows of the metallurgical research department of the University of Utah have made special studies on the subject: Clyde E. Williams, L. G. Gerhardt, J. D. MacKenzie, and Virgil Miller. C. E. Ohrn, mechanic, University of Utah, designed and built many mechanical appliances for use in the experiments. In addition, thanks are here extended to Dr. J. F. Merrill, dean of the School of Mines and Engineering of the University of Utah, for his interest in the work and for aid given in supplying funds for the purchase of the necessary equipment for laboratories.

The bureau wishes to express its appreciation of the many courtesies shown to its investigators during the plant tests at the Yellow Pine Mining Co., Goodsprings, Nev. Morris P. Kirk, general manager, provided the equipment, labor, and technical assistance necessary to make the experiments successful.

INVESTIGATIONS OF THE PROCESS BY THE BUREAU OF MINES.

In 1916 the Bureau of Mines, through A. E. Wells, superintendent of the experiment station, Salt Lake City, and O. C. Ralston, metallurgist at that station, was convinced that further work on the process was justified, especially as the invention of the Cottrell electrical precipitator provided suitable means of recovering the fumes evolved in volatilization. The lack of such a satisfactory device had resulted in the failure of some phases of the process in the earlier trials by Croasdale and others. Since 1916 the work has gradually grown until at present it is the major investigation carried on by the Intermountain experiment station of the bureau, located at Salt Lake City.

In order to obtain all possible data on the early history of the process the writers asked Stuart Croasdale to contribute to this bulletin a chapter covering his work, which they take much pleasure in publishing. Mr. Croasdale gives the history of the process up to the time the Bureau of Mines undertook its investigation.

The contributions of the authors cover numerous experiments conducted on a large variety of ores, each of which is described in detail.

The bibliography of patents includes not only the technical features of the process but also many mechanical devices which are more or less directly connected with the process.

In addition to the early work done by Croasdale, Ben Howe conducted somewhat similar experiments on ores from the Gwalia Consolidated Mining Co.'s properties in Western Australia. An abstract of a report on his work is also included. (See p. 24.)

The application of the Pohlé-Croasdale process was also attempted at a plant of the Rigby Mining & Reduction Co., Mayer, Yavapai County, Ariz., in 1906. For a description of the plant see page 28. A plant was also built at Fairbank, Ariz., in 1918, to try out this process. Walter A. Schmidt briefly describes that plant on pages 29 to 30.

DEVELOPMENT OF THE VOLATILIZATION PROCESS.

By STUART CROASDALE

EARLY HISTORY.

The volatilization of metals as chlorides and their recovery as fume is a natural and logical development from chloridizing roasting, which in turn is a natural development from wet processes using salt; a brief history of the use of chlorine in metallurgy should therefore be of interest.

Chlorine, as a metallurgical reagent for extracting metals from ores, has been used almost exclusively for the recovery of gold and silver. It has been used to some extent in the treatment of low-grade copper ores, but never in a major metallurgical process for the recovery of copper. The connection of chlorine with the metallurgy of other base metals has been subsidiary.

High-grade gold and silver ores and high-grade base-metal ores containing small or large amounts of gold and silver are usually smelted to recover the metals. Chlorine has been associated only with hydrometallurgical processes (including amalgamation) usually for low-grade ores but sometimes for high-grade siliceous ores remote from other facilities for treatment.

The earliest hydrometallurgical process for treating gold and silver ores was amalgamation. For the oxidized ores near the surface—where the gold and silver were in metallic condition or the silver occurred as a haloid—this process was simple, but when deeper mining reached sulphide ores another method had to be devised to put these metals into a condition to be amalgamated.

Consequently the patio process for the treatment of silver ores was invented or introduced in Mexico by Bartholome Medina, a miner of Pachuca, in 1557.[1] According to a report by Luis Berrio de Montalvo, addressed to the Viceroy of Mexico and printed in Mexico in 1643, Medina derived his information from Spain that silver could be obtained from its ores by means of mercury and salt. A similar process was evidently known in Europe before this time, because in a treatise by Biringuccio, published in Venice in 1540, under the title

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[1] Percy, John, Metallurgy of silver and gold, London, 1880, pp. 576–656.

"De la Pyrotechnica," it is asserted that silver can be obtained from its ores in the wet way by the action of sulphate of iron, verdigris, mercuric chloride, salt, and vinegar; this invention probably records the introduction of chlorine into metallurgy. The process was especially adapted to the arid regions of Mexico and South America, where fuel and water were scarce and means of transport expensive.

In 1609 the Cazo or Caldron process was invented by a priest, Alonzo Barba, of Peru, for treating surface ores, particularly those containing chloride of silver. The process was conducted in a vessel made wholly of copper or having a copper bottom, the reagents being the copper of the vessel and a boiling solution of common salt. The silver chloride of the ore was reduced to a metallic state by the copper of the vessel and then the silver was taken up by the mercury. The cuprous chloride formed was taken up by the salt solution. As the metals in the ores changed to sulphides, arsenides, and antimonides with depth, this process had to be abandoned.

The Krohnke process, in which cuprous chloride was added to the hot salt solution to decompose these minerals, was introduced in Chile in 1860. The silver was recovered by means of zinc or lead used in the form of amalgam.

CHLORIDIZING ROASTING.

In Europe, where fuel, mechanical appliances, and better operating facilities were available, more complicated processes were developed to treat these ores. Chloridizing roasting of silver ores was first introduced in Vienna by Born, and combined with the Cazo process in 1786 at Chemnitz in Hungary. The Cazo process was soon superseded by the barrel amalgamation process, which was first installed on a large scale in 1790 at Halsbrucker Hütte, near Freiberg, Saxony. Metallurgical works had been established at this point since 1710, and chloridizing roasting with barrel amalgamation was used not only for the ores but also for metallurgical products such as matte, blister copper, and speiss.

The chloridized ore or furnace product, still containing an excess of salt, was rotated in a wooden barrel with water and scrap iron until the silver was reduced to a metallic state. Mercury was then added to recover the silver as amalgam. Owing to the base metals present which were also chloridized in roasting and reduced by the scrap iron, the bullion obtained was very low grade. Attempts to "destroy" the base metals, as it was then called, by roasting at a higher temperature, resulted in considerable loss of silver through volatilization with the base metal chlorides, and the chloridizing roast was eventually used only on the low-grade ores containing the least amount of volatile base metals.

This led to the invention of the Augustin process and its introduction at the Gottesbelchnung Hütte near Mansfeld, Germany, in 1843. Later, in the same year, the process was introduced at the Freiberg works. In this process, ores were roasted with salt and then leached with a saturated solution of common salt. The silver chloride passed into solution, from which the silver was precipitated by metallic copper. The process was introduced at both Mansfeld and Freiberg to recover the silver from the copper mattes, but owing to the imperfect extraction of the silver by the brine solution, it was soon abandoned and was superseded by the Ziervogel process in 1848, which was applied more particularly to mattes and furnace products than to the ores themselves. In this process no salt was used. The iron and copper were converted into oxides and the silver into sulphate by careful oxidizing roasting. The silver was then leached out by warm water and precipitated on copper.

In 1848 Dr. John Percy, of London, suggested the use of sodium or calcium thiosulphate—incorrectly called hyposulphite—as a solvent for the chloride of silver after ores had been subjected to a chloridizing roast. The first practical application of this suggestion was made in 1856 by Von Patera on the rich silver ores of the Joachimsthal district, Bohemia. To reduce losses by volatilization, Von Patera introduced steam into the furnace during the chloridizing roasting. This process was first introduced in America by Ottokar Hofmann, in 1868, at La Dura, Sonora, Mexico; and at Melrose, near Alameda, Calif., by G. Küstel, in 1874; it was gradually adopted at other mines in Mexico and the United States, reaching the zenith of its application during the succeeding 25 years, until the demonetization of silver by the Government of the United States in 1893 closed every leaching plant in the country that had been treating silver ores.

Up to this time and for a number of years afterwards, gold in ores that had been subjected to a chloridizing roast was supposed to be converted first into the chloride which decomposed into chlorine and metallic gold at a temperature below 300° C. Hence if gold occurred in silver ores, it was converted into metallic gold by chloridizing roasting, and was not recovered by any of the solvents used for the extraction of silver. (See also p. 45.)

Plattner proposed converting the gold into chloride by means of chlorine gas and extracting it with water. This process was introduced in Silesia, Germany, in 1851. The ore was either roasted "dead" or, if it contained silver, it was chloridized by roasting with salt. The silver was first leached out by brine or "hyposulphite" solutions, then chlorine gas was applied to the ore in the vats, and the gold was recovered by subsequent leaching with water.

LOSS THROUGH VOLATILIZATION.

Loss of metals from volatilization during chloridizing roasting was known already, when Plattner undertook a study of the conditions and extent of this loss in an extensive series of experiments on both oxidizing and chloridizing roasting. The results of his experiments were published in his "Metallurgische Rostprozesse" (1856). He mentioned considerable loss of silver chloride when it came in contact with other easily volatilized chlorides, and discussed at length the volatile products of the chloridizing roasting, but he failed to record any loss of gold by volatilization.

Percy[2] gives an account of experiments made by Thompson and Aiken in 1838, on a method of assaying and purifying gold, which they said was based on the well-known fact that "not only has gold no affinity for chlorine at a red heat, but actually parts with it at that temperature. This is not true of other metals with which gold is alloyed, so it affords an easy and certain means of separation"; also that "the presence of alkali chlorides seems to have the effect of preventing the volatilization of the chloride of silver."

Prof. S. B. Christy[3] quotes an anonymous writer in the Chemical News, March 12, 1869, who says that if rock containing gold is heated to a bright cherry red in a current of chlorine gas the gold will combine at high temperature with the chlorine and becomes volatile therewith, whereas at points where the temperature is less high the chloride of gold is again decomposed and the gold deposited. Debray[3a] says that "while gold chloride is dissociated by heat at 200° C. in an atmosphere of chlorine," he also says that Boyle was the first writer to mention that gold chloride is volatile to some degree without dissociation.

In 1880 Charles H. Crosby,[4] in charge of the Pioneer Reduction Works at Nevada City, Calif., found great difficulty in treating pyritic ore from the Murchie mine, where about 75 per cent of the value was in gold and the rest in silver. To extract the silver it was necessary to roast with salt; when the ore was roasted without salt no loss by volatilization was sustained, but when roasted with 3 per cent salt 30 per cent of the gold and 50 per cent of the silver were lost by volatilization. Crosby attributed these losses to the tellurides, which he supposed to be present in the ore, and not to the salt used in roasting.

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[2] Percy, John, Work cited, p. 402.

[3] Christy, S. B., The losses in roasting gold ores and the volatility of gold: Trans. Am. Inst. Min. Eng., vol. 17, 1888–89, p. 8.

[3a] Debray, H., Note sur le chlorure d'or: Compt. rend., t. 69, 1869, p. 984.

[4] Christy, S. B., work cited, p. 3.

G. Küstel[5] said, "If salt is present during the roasting the chloride of tellurium volatilizes voluminously, and it is possible that under this condition the tellurium causes the gold to volatilize likewise." He mentioned his own experience of the loss of 8 per cent of the gold before the ore had reached a red heat when roasted with 4 per cent salt, and he expressed the opinion that the loss might reach 20 per cent by increasing the time and temperature of roasting, but he was not certain that this loss was due to the salt. He also said that "many manipulators complain of considerable loss of gold with some kinds of sulphurets, which they could not avoid by any regulation of heat or general treatment."

The first person to recognize the volatility of gold in chloridizing roasting, and to bring this subject prominently before the metallurgical world, was C. H. Aaron.[6] On roasting some gold-bearing pyrites with less than 3 per cent salt, in a reverberatory furnace, at his plant at Melrose, Calif., he suffered a loss of some $3,000 worth of gold by volatilization. This loss led to considerable investigation on his part and aroused the interest of metallurgists engaged in this work throughout the country, so that during the next few years of the early eighties many experiments were made at western metallurgical plants to determine the actual as well as the possible losses of both gold and silver by volatilization, and to devise means of overcoming them.

Aaron, after learning the cause of his monetary loss, confined his efforts to the prevention of future losses by reducing the amount of salt used and the time of its application during roasting. He came to the conclusion "that gold is volatilized in some form not easily condensible," but neither he nor Küstel gives details of their experiments. In his earlier treatise[7] Aaron said:

> Roasting does not "destroy" the base metals, as commonly stated. Roasting with salt to put silver in the form of chloride puts copper, lead, etc., in similar condition, and they are not driven off to any extent. True, that by modified roasting, base metals can be changed in a measure not to be amalgamated, but this causes loss and expense and is not wholly successful, besides interfering with the amalgamation of the silver. Chloridizing roasting of silver ore is not so difficult as some suppose or pretend, but it requires practice.

Küstel[8] states that "base metals as sulphates take up their share of salt and consume a large portion, but as their chlorides are volatile, the salt is a means of getting rid of a great deal of the metals during roasting which are not desirable in the ore for subsequent treatment of silver."

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[5] Küstel, Guido, Roasting of gold and silver ores and the extraction of their respective metals without quicksilver. San Francisco. 1880, p. 57.

[6] Aaron, C. H., Leaching gold and silver ores. The Plattner and Kiss process. A practical treatise. San Francisco. 1881, p. 121.

[7] Aaron, C. H., A practical treatise on testing and working silver ores. San Francisco. 1876, pp. 35–36.

[8] Küstel, Guido, work cited, p. 29.

Professor Christy,[9] of the University of California, with the cooperation of his students investigated this subject in the best chlorination mills in California, as well as in the laboratory, during the early eighties. Their experiments dealt wholly in establishing the fact that losses occur during chloridizing roasting and in the determination of the conditions that produce these losses. Their conclusions are that losses of both gold and silver increase (1) with increased percentage of salt added during the roast; (2) with increase of time and temperature during roasting; and (3) when salt is added after a long oxidizing roast instead of at the start. Christy qualifies the last statement by saying that "while there is a rapid volatilization of gold and silver when salt is added at the end of the roast, the gold chloride is quickly decomposed by the SO_2 gases and by the raw ore itself in the cooler end of the furnace, and the gold is redeposited in metallic form so that the actual loss is not so great as when salt is added at the beginning of the roast." These experiments were confined largely to the treatment of the gold-bearing pyritic concentrates from the California stamp mills.

Stetefeldt,[10] when engaged in examining the gold ores of Las Minas, Vera Cruz, Mexico, in 1885, found to his great astonishment "enormous losses of gold by volatilization," when these ores were roasted in a muffle or reverberatory furnace. His opinion was that the gold escaped as a double salt, and if this was true, the loss of gold depended on the volatility of the chloride with which the gold chloride was combined. He concluded his comments as follows:

> I have repeatedly expressed the opinion that the volatilization of metals in roasting is principally a function of time. My experiments in the chloridizing roasting of silver ores have established this fact in the case of silver. Why should it not hold good with regard to gold? The advantages of instantaneous roasting, as done in the Stetefeldt furnace, are as yet very little understood and appreciated by metallurgists.

TREATMENT OF COPPER ORES.

Chlorine was first used for the treatment of copper ores about 1860,[11] when William Henderson, of Scotland, patented a method for extracting copper from pyritic residues. Pyrites from Spain and Norway, which were used throughout Europe for making sulphuric acid, contained from 3 to 8 per cent of copper. After the pyrites were roasted for the manufacture of sulphuric acid, the residues were again roasted with salt, by the Henderson process, to convert the copper into chloride. The chloridized ore was then leached with water or dilute acid, and the copper was precipitated from the solution by iron.

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[9] Christy, S. B., Work cited, pp. 3–44.

[10] Stetefeldt, C. A., The amalgamation of gold ores, and the loss of gold in chloridizing-roasting, with especial reference to roasting in a Stetefeldt furnace: Am. Inst. Min. Eng., vol. 14, 1886, pp. 336–351.

[11] Eissler, Manuel, The hydrometallurgy of copper, being an account of processes adopted in the hydrometallurgical treatment of cupriferous ores, including the manufacture of copper, vitriol, with chapters on the sources of supply of copper and the roasting of copper ores. London and New York. 1902. pp. 87–104.

The gases from chloridizing roasting contained sulphurous acid, hydrochloric acid, and chlorine. To prevent these gases from becoming noxious to the neighborhood, and to utilize their acids, they were passed through coke towers sprayed with water, which collected not only the acids but any volatilized metallic chlorides as well. The water from the condensing towers was used for leaching the chloridized ore.

In European practice, the limit of copper content in ore amenable to this process was 6 to 8 per cent; the sulphur content should not exceed one and one-half the equivalent of copper, otherwise the consumption of salt would be too great. If the sulphur content in the residue was too low, raw pyrites were added. To prevent excessive loss by volatilization the temperature of roasting was kept at a dark-red heat and was never allowed to exceed a cherry red. About 15 per cent salt was used in roasting. This process was patented in the United States in 1866 and is further described on this page.

When the residues or ores contained silver, that metal was precipitated from solution as an iodide by the Claudet process before the precipitation of the copper.

About the same time, 1862, ferrous chloride was suggested by Schaffner and Unger, of Germany, as a solvent for copper in ores. This method was developed later in the United States into the Hunt and Douglas process. It was designed to treat oxidized copper ores, whereas the Doetsch process, using ferric chloride, was employed at Rio Tinto to treat sulphide ores.

The use of chlorine, or the chloridizing roast, in the metallurgy of lead, zinc, and other base metals, was never developed into a commercial process, but was restricted chiefly to eliminating these metals from complex ores preparatory to some other treatment. There was no thought of extraction by volatilization, and the recovery of the volatilized chlorides, if attempted, was only a phase of the general treatment of the ores. For further discussion of this subject, see the following pages.

UNITED STATES PATENTS ISSUED BEFORE 1900.

Previous to 1900 I find but two United States patents relating to the volatilization and recovery of metals by means of chloridizing roasting. These patents were granted to William Henderson,[12] of Glasgow, Scotland, and to Arthur Chanute,[13] of Denver, Colo.

The Henderson patent is divided into three parts. The second part only covers the treatment of sulphide ores of copper and silver by roasting with salt, as follows:

> If the proportion of sulphur existing in the ore is more than one and a half times as much as the metal or metals to be extracted, it should be reduced by calcination;

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[12] Henderson, W., U. S. patent 60514, Dec. 18, 1866.

[13] Chanute, A., U. S. patent 501559, July 18, 1893.

or if the ore contains much silica the proportion of sulphur may be even lower than equal proportions. The ores are reduced to a fine powder generally—the finer the better—although for copper ores very rich in iron a sieve with eight holes to the inch will be as fine as can be conveniently worked in the tanks. The ores are then mixed with from 5 to 50 per cent of common salt. The mixture is placed in retorts or close calcining furnaces connected with a condensing apparatus and heated at once to a bright red heat; the volatilized chlorides, passing into the condensing apparatus, are condensed with water. The ore is withdrawn from the furnace when it ceases to smoke strongly, and the metals left in the ore are leached out by the hot acid solution coming from the condensing apparatus.

From the practical application of this process in Europe, as well as at the works of the Pennsylvania Manufacturing Co. in Philadelphia and at Natrona, Pa., it is clearly evident that the condensing chambers were installed not primarily to collect the volatilized metallic chlorides but to prevent the noxious acid gases from escaping into the atmosphere, and at the same time to recover the acids for leaching the chloridized ore. Every effort was made to prevent the volatilization of metallic chlorides by roasting at low temperatures, and the recovery of any metal in this manner was purely incidental to the process. The third part of this patent describes a process for making sponge iron to be used in precipitating the copper and silver from the solution.

The Chanute patent relates particularly to the treatment of complex lead-zinc ores containing gold and silver. Such ores are mixed with chloride or sodium or other haloid salts and subjected to "a considerable degree of heat, whereby the chloride of silver, gold, lead, and zinc are readily formed and vaporized." These metallic fumes were caught and saved by means of bags of textile material. "The ore remaining in the furnace after the metallic haloids have been driven off may be smelted in the usual manner, or if it contains too high a percentage of zinc it may again be roasted with salt, and more metallic value may be extracted in the form of fumes." The ore is finely crushed and mixed with varying percentages of salt, depending upon the composition of the ore, "but usually 25 per cent of salt is sufficient."

Under the specifications of those patents the residual ore, after chloridizing roasting, was either leached or smelted. The patentees thereby preclude any claim for the complete volatilizating of the metals, attempting to provide only for the recovery of losses through volatilization.

Both patentees recommend excessive quantities of salt in roasting, which, with most ores, would produce an easily fusible slag and prevent the volatilization of the metals. The Chanute patent is vague and provides neither for the complete nor the preferential volatilization of metals from complex ores.

Some years later a similar patent was granted to Guy de Bechil, of Paris.[14] He proposed mixing complex lead-zinc ores with salt and treating them in a cupola furnace. A highly oxidizing atmosphere was maintained in the upper part of the furnace and a reducing atmosphere in the lower part. By this means the inventor claimed to volatilize, in one operation, practically all of the zinc as chloride, and to leave most of the lead in the ore to be reduced to a metallic state or matte in the lower part of the furnace. The zinc chloride was collected in condensing towers or other suitable apparatus. The operation of this process as described would be an impossible metallurgical feat.

One other United States patent should be noted. It was granted to H. J. M. Puistienne,[15] of Paris. He produced a matte by smelting copper ores with sulphur, calcium chloride, and the necessary fluxes, "but if the copper ore contains arsenic, antimony, zinc, nickel, cobalt, or other injurious or deleterious matters in quantities of sufficient importance, the pulverized ore should be first mixed with chloride of calcium or sodium, and the mixture then roasted in order to volatilize the deleterious metals in the state of chloride." No provision was made for the recovery of these metals.

After the foregoing patents the Brazelle,[16] Larsen,[17] and Swinburne-Ashcroft [18] patents relating to the volatilization of certain metals by means of chlorine gas at a red heat were issued, but these do not relate to the subject of chloridizing roasting.

RECOVERY OF FUME.

The metals volatilized as chlorides were recognized as metallurgical losses, and such losses were kept at a minimum by roasting at low temperatures, hence no special apparatus except the ordinary dust chambers was devised for their recovery. Dust chambers were considered essential by all metallurgists when roasting fine ores in White-Howell or Stetefeldt furnaces.

The injection of steam into the dust chambers was sometimes recommended, as the steam on condensing in the cooler parts of the flue carried down with it the solid matter from the furnace gases. Little or no distinction was made between flue dust and fume.

It is true that water sprays and other forms of water condensation had been recommended, but the only place I know where it reached commercial application was at the Holden Lixiviation Works, Aspen, Colo. Here occurred a volatilization loss of about 10 per cent of the silver, as chloride, from the Stetefeldt furnace. A large chamber, built beyond the usual dust chambers, was filled with a checkerwork

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[14] de Bechil, Guy, U. S. patent 681609, Aug. 27, 1901.
[15] Puistienne, H. J. M., U. S. patent 34591, Mar. 4, 1862.
[16] Brazelle, B., U. S. patent 529476, Nov. 20, 1894.
[17] Larsen, A. S., U. S. patent 607287, July 12, 1898.
[18] Swinburne, J., and Ashcroft, E. A., U. S. patent 695126, Mar. 11, 1902.

of wooden fence pickets, which were sprayed with water. Much silver was thus saved, but the recovery was not complete.

Bags of textile material had already been used for the recovery of zinc oxide. They had also been recently adapted to the recovery of fumes from lead blast-furnaces, and Chanute had recommended their use for the recovery of metallic chloride fumes. It was generally recognized, however, that bags were impracticable for collecting metalliferous particles from the corrosive acid gases produced in either oxidizing or chloridizing roasting.

A forerunner of the Cottrell precipitator, which was patented by A. O. Walker, of England, is described as follows:[19]

> He collects the fine particles of metals suspended in the fumes by means of a discharge of high potential electricity from metal points or edges, or other projections, situated in the flue passage, chamber, or other receptacle, and so placed that the current of air or gas containing the finely divided material or metallic vapor is carried or passes in close proximity to the discharging points. The discharge from the points electrifies the air or gas and the charged air or gas then acts on the finely divided matter, causing it to cohere, condense, and deposit.

This process is said to have worked very efficiently, but evidently it did not advance beyond the experimental stage.

SUMMARY OF INVESTIGATIONS BEFORE 1900.

Other well-known metallurgists who had investigated the losses of metals by volatilization up to the time of my own work in 1896–1898 might be mentioned, but no one had laid a foundation or established definite laws from which a commercially complete volatilization of metals could be accomplished; nor had anyone proposed utilization as a metallurgical process whereby metals might be volatilized completely from their ores by the chloridizing roast and then recovered as fume, the gangue being discharged from the furnace as of too low value to warrant further treatment. All effort had been expended in one direction—to prevent the loss of gold and silver by volatilization while preparing ores for treatment by amalgamation or lixiviation. Much that was published concerning volatilization losses was contradictory, and each investigator usually confined his experiments to the particular ore in which he was interested at the time without arriving at results applicable to all ores.

Sulphur was considered essential to chloridization, and, if not already in the ore, it was added as elemental sulphur, as ferrous sulphate, or as raw pyrite. Mattes and pyritic ores were roasted to reduce their sulphur content before the addition of salt, in order to prevent excessive consumption of salt. In practice the percentage of sulphur used varied from 3 to 15 per cent. It was recognized that silica would replace sulphur to some extent in the decomposition of salt. Schnabel[20] states that "ores containing manganese peroxide

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[19] Eissler, Manuel, Metallurgy of gold. 4th ed. 1896, p. 219.

[20] Schnabel, Carl, Handbook of metallurgy, vol. 1, 1905, p. 682.

do not need the presence of pyrites for chloridizing roasting." Küstel[21] also asserts that "some cupriferous ores, especially if other base metals are present and no sulphur (or very little), will give sometimes a good chloridizing roasting without any addition of green vitriol or other sulphur combinations." The salt used in commercial practice varied from 3 to 20 per cent, "depending chiefly upon the character of the ore, arrangement for roasting, and the manner in which the process is worked."

No definite relations whereby the most efficient results could be obtained in either chloridization or volatilization were ever established between the percentages of salt and sulphur used and the percentages of the base metals in the ore. Henderson placed the sulphur limit at "one and one-half times as much as the metal or metals to be extracted," a percentage that is too high for the greatest efficiency; at the same time he allowed the salt used a range of 5 to 50 per cent.

The only record I have been able to find approaching a definite relation between the percentages of salt and sulphur used in chloridizing roasting is the statement[22] that Mr. Falkenau, of San Francisco, in experimenting with ores from Virginia City, Nev., found the highest chloridization of both gold and silver was obtained with 10 per cent salt and 3 per cent sulphur. No mention is made of any volatilization of these metals, but Falkenau found that approximately theoretical proportions of salt and sulphur gave the most efficient chloridization.

Loss of gold by volatilization was not known for a long time after the discovery of the loss by that means of silver and the base metals. When the volatilization of gold was discovered, it was not thought serious in commercial practice provided precautions were taken to prevent it. No attempt was ever made to volatilize gold completely on a commercial scale, although Christy, Stetefeldt, and others obtained in the laboratory results ranging from 20 to 88 per cent volatilization.

Plattner, Patera, Roessner, Kiss, and Hofmann devised methods for extracting gold from gold-silver ores by converting the gold into its chloride by means of chlorine gas after the ores had been subjected to a chloridizing roast and the silver had been removed by a solvent. This procedure indicates that little or no gold was lost by volatilization in commercial practice. On the other hand, in Australia chloridizing roasting of gold-silver ores was abandoned entirely on account of the high losses of gold by volatilization.

Silver losses by volatilization were generally recognized, especially when base metals were present in the ore, but every effort was made

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[21] Küstel, Guido, Roasting of gold and silver ores and the extraction of their respective metals without quicksilver, 1880, p. 27.

[22] Eissler, Manuel, Metallurgy of gold, 1895, p. 224.

to reduce these losses to a minimum so that in commercial practice they seldom exceeded 10 per cent. Laboratory tests are recorded in which volatilization losses up to 80 per cent of the silver were obtained, but no attempt was ever made to complete the volatilization on a larger scale.

The volatilization of base metals during chloridizing roasting was utilized as a means to eliminate deleterious elements from gold and silver ores rather than to recover these metals for profit.

Increases in the time and temperature of roasting increased the losses of gold and silver, according to the experience of all metallurgists, so the roasting was always done at a dull-red heat to prevent loss by volatilization. In other words, only one end of a temperature range was determined, and that was the temperature at which the metallic chlorides began to volatilize. This delimited the field of all former commercial practices and fixed the danger line for losses. No temperature or time was ever determined that would effect complete loss of all the metals.

THE POHLÉ-CROASDALE PROCESS AND ITS DEVELOPMENT.

EARLY EXPERIMENTS.

My work on the volatilization of metals as chlorides began in the early nineties. From 1891 to 1893 I was employed as chemist at the Holden Lixiviation Works in Aspen, Colo., where Aspen silver ores were treated by the Russell process. The base metals contained in these ores, although in small amounts, gave a great deal of trouble, and much of my time was spent in research to increase the efficiency of the company's practice.

During this work in 1892 and 1893 Cripple Creek began to develop into a prominent gold district and the prospect arose of extending metallurgical operations into that section; hence I conducted an exhaustive series of experiments on these ores by the barrel chlorination process after first submitting the ores to an oxidizing roast. During the period of my investigation of the Cripple Creek ores my experimental furnace was used for a short time for the chloridizing roasting of silver ores. On resuming the oxidizing roasting of Cripple Creek ores, I sustained a loss of over 90 per cent of the gold on account of the small amount of salt retained in the lining of the furnace, and to get satisfactory results again with the Cripple Creek ores I had to rebuild the furnace.

This was an interesting clue for subsequent investigations and all the phenomena were carefully noted, but further experimentation was prohibited by the panic of 1893, which closed all silver-leaching plants in the United States. I was not able to renew research in this direction until 1896. when I accepted the position of chief chemist for the Globe Smelting Co., of Denver, Colo. While there I met Edwin C. Pohlé, then chief assayer for the same company. I learned that he had been experimenting in a similar manner with the silver-bearing ores from Aspen, Colo., particularly those containing large amounts of baritè. Much to his astonishment, on roasting these ores with salt he sustained almost a complete loss of the silver.

After comparing our notes we reached the conclusion that a process might be developed whereby all of the valuable metals could be volatilized from an ore as chlorides and collected in a condensed form, the gangue passing from the roasting furnace to the tailing dump devoid of commercial value. At that time the older processes for leaching gold and silver ores were being replaced by the cyanide process, but there was, and still remains, a big field for a process that will treat refractory low-grade siliceous ores, more or less remote from smelting centers, particularly gold and silver ores containing small percentages of copper or lead.

As mentioned above, we were employed at the Globe smelting plant in Denver, where the bag house was in successful use for collecing lead blast-furnace fumes; hence we anticipated no difficulty in the recovery of the metals after we had driven them from the ore by chloridizing roasting. We therefore planned systematic experimentation to determine what conditions would yield the desired results.

Variations were made in the time and temperature of roasting and in the percentages of salt and sulphur used, until the points were determined at which a commercially complete volatilization of the metals could be accomplished most efficiently. For this purpose a Cripple Creek gold ore was used, and the roasting was always done in a highly oxidizing atmosphere. Volatilization was completed within 30 minutes after the cold ore was charged into the highest temperature of the furnace, or within 20 minutes if the ore was warmed by the heated gases before entering the furnace. As to range of temperature, volatilization began at a dull-red heat and was completed at a bright-red heat, the volatilizing temperature ranging between 750° and 1,050° C.

PROPORTION OF SALT.

Ten per cent of salt produced complete volatilization of the gold, the percentage of salt bearing a direct relation to the sulphur used; for this particular ore 5 to 7½ per cent of the salt would have accomplished the same result. The highest volatilization was obtained when the salt and the sulphur used were in proportions to form a definite chemical combination according to the following equation:

$$2\ NaCl+S+2O_2=Na_2SO_4+Cl_2$$

This series of tests gave us a basis for experimenting on all ores, including complex lead-zinc ores carrying gold and silver.

From the equation given above, it is obvious that an excess of sulphur in an ore is an unnecessary consumer of salt. Therefore, in an ore containing a base metal that is volatile as a chloride, if sulphur be added, or reduced by roasting, to an amount that will form the normal sulphide of the base metal or metals to be volatilized, and the theoretical amount of salt added, then roasting at the proper temperature should volatilize that base metal, along with any gold or silver present, and leave no metallic value, or only the nonvolatile metals, in the gangue. This assumption proved correct by actual experiment; from a complex lead-zinc ore the lead was completely volatilized, carrying with it the gold and silver, whereas practically all the zinc was left in the gangue.

The summation of the chemical reactions relating to the chloridization of the base metals may be represented as follows:

$$CuS+2NaCl+2O_2=CuCl_2+Na_2SO_4$$
$$PbS+2NaCl+2O_2=PbCl_2+Na_2SO_4$$

From these equations may be determined the relative proportions of salt and sulphur that insure the most efficient volatilization. These relations apply to all metals if the ore and salt are ground infinitely fine, so that there is an intimate contact and direct chemical action between each particle of salt and each particle of mineral—in practice it is not necessary to crush either salt or ore finer than 20 mesh, or 0.75 mm., which is about the economical limit for fine grinding.

Any attempt to obtain a chemical reaction in a dry mixture like ore and salt is very different from obtaining a chemical reaction with a solution of salt and ore, such as takes place in any leaching operation. The percentage of salt necessary to form a chemical combination with less than 1 ounce of gold or a few ounces of silver in a ton of ore would be so small that much of the salt would be volatilized before it could be brought into actual contact with the gold or silver mineral by any method of dry mixing; hence calculations based on these reactions can not be applied to gold and silver ores that contain no base metals. In order to insure actual contact of the chemical reagents involved, salt must be added in excess of the theoretical amount required for the gold and silver present, and seldom can less than 5 per cent be used. The percentage of sulphur required would then be based on the amount of salt used.

ROASTING WITHOUT SULPHUR.

Metals can sometimes be volatilized by roasting with salt alone, no sulphur being necessary. An example of this kind was an ore from the Black Warrior mine at Miami, Ariz., where the copper occurred as a silicate or a hydrosilicate. In this ore the "silicic acid" combined readily with the sodium in the salt and liberated enough chlorine to combine with nearly all of the copper.

Lead, being volatile in nearly all its combinations, might likewise occur in ores which would not require the addition of sulphur to bring about its volatilization through chloridizing roasting; sulphur is a common constituent of nearly all ores, and as lead requires only one-seventh of its weight in sulphur to form the normal sulphide, advantage would seldom be gained by roasting without sulphur.[23]

PLANT EXPERIMENTS AT CAMDEN, N. J.

Our experiments were completed in 1898 and arrangements were made with the Globe Smelting Co. to install the process on a commercial scale. About this time (1899) the American Smelting & Refining Co. was formed, and into it all Colorado smelting companies were merged. When the Globe Smelting Co. was absorbed by the larger company, our contracts were canceled and, much to our disadvantage, we had to seek other capital to demonstrate our process.

Finally, the Metal Volatilization Co. was formed for this purpose, with W. W. Gibbs, of Philadelphia, as president, and Edwin N. Hawkins, of Denver, as vice president and general manager. A small experimental plant was constructed at Camden, N. J., during the latter part of 1900. The next two years were taken up chiefly with patent litigation; then a larger experimental plant was constructed at Denver to treat ore on a tonnage basis. This plant was operated from 1903 to 1906. Much time was spent in working out problems pertaining to the furnace and to fume condensation.

All our preliminary work concerning the relative proportions of salt, sulphur, and base metals held true and constant in the larger tests.

After trying several types of roasting furnaces, we found the White-Howell or cement-kiln type the most suitable for our purpose.

For the collection of fumes we naturally installed bags at first. Both cotton and woolen fabrics soon disintegrated from the corrosive action of the acids in the fumes, and this system had to be temporarily abandoned. We then tried water condensation in every conceivable form. Towers filled with gravel and sprayed heavily with water were useless, even when the gravel was reduced almost to the fineness of sand; we reached the capacity of our fans in forcing the furnace gases through the towers. The same held true for filters of textile fabrics sprayed with water. The copper chloride fumes were particularly difficult to collect.

Finally, we made a tower in which were placed, about 1 foot apart, a number of superimposed horizontal burlap filters, and water was

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[23] Some of the experiments reported in this bulletin show that sulphur is not necessary in the chloridizing of lead compounds and that it is actually a hindrance where the object is to chloridize and volatilize simultaneously. Mr. Croasdale, however, makes a faithful report of conditions as he knew them

sprayed on top of each filter. A positive blower of the Root type, lined with lead to preserve it from the corrosive action of the fumes, was placed in the flue system ahead of the condensing tower. The gases were forced through the lower filter in the condensing tower, a certain amount of water being held on top of the filter, and the gases forming a bubble at each mesh in the burlap. Each bubble collided with another, so that the gases not only passed through the wet filter but through 2 or 3 inches of water foam on top of the filter. Water gages were placed on the side of the tower so that the depth of water held on top of each filter could be observed and regulated; 8 to 10 inches of water could be held on top of each filter, but a depth of 2 inches was more efficient, as more agitation was produced.

After passing through the first filter and the water on top of it, the gases reunited to pass through the second filter and the water on top of that, in the manner just described, and so on through the tower. It took seven or eight of these filters to collect all of the copper chloride fumes; the fumes were stopped eventually, but the apparatus became too difficult to manage and was impracticable.

By this time our funds were exhausted, but before stopping entirely we decided to try more experiments with textile fabrics for collecting fumes. We built a small gas-fired furnace and connected it with a woolen bag. The fumes, before entering the bag, were so thoroughly cooled in a long flue that practically all the corrosive sulphuric acid was condensed and the bag then recovered the fumes successfully without evident injury. We were never able to continue this demonstration on a large scale and over a long period of time, so it is yet undetermined how long the bags would give service.

PLANT EXPERIMENTS AT MAYER, ARIZ.

In the meantime, the Globe Smelting Co., licensed to use the process, had built a plant at Mayer, Ariz.[24] (See p. 28.) In this plant, asbestos bags were installed to collect the fumes, but for some reason they, too, failed. All the bags disintegrated at the top. Probably the heat and the corrosive action of the gases loosened the fibers; then the vibration produced by the fan, combined with the great weight of the bag, which was not properly supported, pulled the fabric apart.

Metallic chlorides are, of course, volatilized in the form of gas or vapor. In an atmosphere of furnace gases from the chloridizing roast, these chlorides seem to condense into colloidal particles that have no power or tendency to agglomerate into particles large enough to be influenced by gravity, water, or low temperature. This fact was more noticeable with the chlorides of copper and silver than with the chlorides of the other metals—although I think it is true of all the

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[24] Fairchild, O. H., The application of the Pohlé-Croasdale process: Min. and Sci. Press, vol. 93, 1906, pp. 293–294.

volatile metallic chlorides—and undoubtedly Aaron was right when he said that "gold is volatilized in some form not easily condensible." Unfortunately, physical chemistry was not then a recognized science, otherwise it might have helped us understand this problem. The difficulty of wetting fume is now well known. For instance, in the laboratory it is possible to form ammonium chloride fumes by union of ammonia vapor and hydrochloric acid gas of such physical quality that the gas in which they are suspended can be passed through several wash bottles in series without being scrubbed clean of ammonium chloride.

The impinging of the chloride particles against a dry textile fabric seemed to be more effective than any practicable form of water condensation, especially after the fabric became coated with the fume itself, but in our experimental plant we could never separate the bag filters long enough to make a clean-up on account of the corrosive action of the acids. No form of bag filtration, as is well known, has ever been used successfully for the collection of solid matter from oxidizing gases from a roasting furnace unless the acids were first neutralized. Gases from chloridizing roasting contain not only hydrochloric acid but must contain an abnormal proportion of sulphuric acid from the oxidizing action of the chlorine. Both acids would be very corrosive on either cotton or woolen fabrics, unless they were neutralized or removed with the moisture by cooling in a long flue system.

I have often wondered what the results would have been had the Globe Smelting Co. gone ahead with the proposed demonstration of our process. The fumes from our furnaces would probably have been turned into the bag house with the fumes from the lead blast furnace and might have been successfully recovered without our ever learning their true character.

At this time a curious division of opinion arose concerning the possibilities of our process. Lead and zinc metallurgists accustomed to the collection of fumes in bags were positive about the complete recovery of values, but were skeptical about our ability to volatilize all of the metals from the ore. On the other hand, metallurgists familiar with chloridizing roasting were positive about the volatilization of the metals, but were skeptical about their recovery as fume. It is certain that all the metallurgists from Mr. Henderson's time to 1900 who recommended water condensation, dust chambers, or bags for the complete recovery of metallic chloride fumes assumed more than they knew, and not until the Cottrell process of electrical precipitation was developed commercially was a really satisfactory method of recovery attained. The acidity of the fumes is especially favorable for this method of precipitation, and the electrical discharge agglomerates the fume into particles large enough to be recovered by gravity and electrical attraction.

The results of our work were not published, for obvious reasons, although they were known to some extent among western metallurgists and by editors of the technical press. As long as the Globe Smelting Co. was interested and we were working in its laboratories, we expected to finish the large-scale experiments before publishing anything.

PATENT LITIGATION.

When we made other arrangements for financing our experimental plant, we filed application for a United States patent on January 23, 1900. Before the patent was issued we availed ourselves of the time allowed by the Patent Office to confirm our original experiments in a complete operating plant and to determine any changes that might be necessary. During this time Robert McKnight, who may have had some idea of treating gold ores in this manner, obtained the exact phraseology of three of our five claims and filed an interference to our application. This led to litigation through the highest United States courts and resulted in a complete adjudication of our rights and the granting to us of a basic patent for the "commercially complete volatilization of metals as chlorides or oxychlorides from their ores, and collecting the same by suitable condensing chambers." This patent was the first that had been granted for a process in which metals were completely volatilized from their ores as chlorides and recovered as fumes without an auxiliary treatment of the gangue by leaching or smelting.

Owing to the interference with our original application for patent and the subsequent litigation, our application for patent was renewed December 3, 1900, and the patent was finally issued October 20, 1903. In the meantime patents were granted to us by 33 or more foreign countries, covering every section on earth where mining and metallurgy were recognized arts. The results of our completed work were published in the Engineering and Mining Journal September 23, 1903. As the Cottrell precipitator has been developed to commercial application and its value has been demonstrated during the last few years as an efficient apparatus for the recovery of metallic chloride fumes from furnace gases, the original Pohlé-Croasdale process for the complete volatilization of metals from their ores now stands ready to take its place in the metallurgical world.

INVESTIGATIONS FROM 1906 TO 1921.

For several years after the closing of our experimental plant in Denver in 1906 little was done to further exploit this method of ore treatment.

In 1912 Ben Howe (see p. 26, (1), (2), and (3)) developed a volatilization process for the treatment of gold ores at the Gwalia Consolidated Mines, in Australia. This process was described in the press as something new in metallurgy, although Howe duplicated in almost every particular the work I had done 10 to 15 years before. The process was again fully discussed in the technical press of America and in the Mining Magazine, of London.

About this time the Anaconda Copper Co. began leaching its mill tailings for copper. At first a little salt was added during the roast to chloridize the silver in the tailing. This method, of course, caused some loss of both copper and silver by volatilization, and the Cottrell precipitator was first applied for the recovery of these fumes.

In 1917 the technical staff of the Intermountain station of the Bureau of Mines published[25] the results of their work on the volatilization process during the years of 1914–1917. In collaboration with mining companies they have since conducted a great deal of research on the application of this process to the low-grade oxidized lead ores of Utah. H. R. Layng[26] has done similar work in San Francisco.

During the past few years several mining companies in the United States and Mexico have operated experimental volatilization plants. The Western Metallurgical Co., of Los Angeles, Calif., has also completed investigations that have satisfied it as to the commercial practicability of the process.

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[25] Ralston, O. C., Williams, C. E., Udy, M. J., and Holt, G. J., Salt in the metallurgy of lead: Trans. Am. Inst. Min. Eng., vol. 57, 1917, pp. 634–645.

[26] Layng, H. R., Chloridizing processes: Min. and Sci. Press, vol. 120, 1920, pp. 77–83.

ABSTRACT OF BEN HOWE'S WORK.

The experiments of Ben Howe mentioned in the preceding discussion of the history of the volatilization process were conducted on ores from the Gwalia Consolidated Mining Co.'s properties in western Australia. The ore presented serious difficulties in opposition to the metallurgical methods common in general practice.

In 1910 A. T. Fray, a chemist and assayer at the mine, discovered that gold was lost when the ore was heated with salt. As research was being done to find a metallurgical method for treating the ore, a thorough investigation was made, a summary of which follows.

The gangue in the Gwalia Consolidated ore is composed mainly of a silicate of alumina, with quartz and calcite.

Assay of the ore of Gwalia Consolidated Mining Co.

Gold, (Au) ounces per ton	0.5
Arsenic (As), per cent	1.5
Antimony (Sb), per cent	0.5
Sulphur (S), per cent	2–3

The mineral is finely disseminated throughout the ore, and ordinary concentrating methods were ineffective, whereas cyaniding after roasting yielded only about 75 per cent of the gold.

Uninformed of Pohlé and Croasdale's work (see p. 26, (1)), Mr. Howe believed he had discovered a new metallurgical process, and it was not until 1913, at the time of his large kiln tests, that he was informed of earlier work having been done at Denver, Colo., and elsewhere. The fact that two separate investigations were conducted with practically duplicate results and that both investigators concluded their work facing the same difficulty, namely, the recovery of fume from the furnace gases, is of primary interest.

LABORATORY TESTS.

According to Mr. Howe (see p. 26, (3)), preliminary volatilization tests were made in the assay muffle furnace. The charge, consisting of two assay ton weights of finely crushed ore, was thoroughly mixed with 5 per cent sodium chloride and spread over a roasting dish 4 to 5 inches in diameter and placed in the rear of an assay muffle, at a temperature about 1,000° C., for about 30 minutes.

The rate of volatilization proved to be directly dependent on the temperature and the mesh of the ore treated, the temperature needing to be maintained as high as possible (1,000° C. for best results) without sintering the charge. The test on 20-mesh material produced 92 to 94 per cent extraction. "Equivalent extraction was obtained on 100-mesh material in 10 minutes, 40-mesh in 30 minutes, 30-mesh in 50 minutes."

SMALL KILN TESTS.

This small plant (see p. 26, (3)) consisted of a rotary brick-lined furnace 13 inches diameter inside and 72 inches long, fired by a kerosene and hot-air burner. The furnace made one revolution a minute and had a slope of 1 inch for 6 feet. The ore previously mixed with 5 per cent salt was fed regularly by a screw conveyer into the cool end of the furnace. This end was connected to a small dust chamber, which in turn joined on to a 200-gallon iron vat. To avoid the trouble of fitting an air-tight top to this vat, it was placed upside down in a water seal, and made air-tight thereby. The vat was fitted with three compartments similar to a "zinc box," but with an air-tight lid. This box had nine compartments, 8 by 8 by 12 inches each, filled with broken quartz, the lower lips of the partitions dipping half an inch under water. In this way the gases had to bubble nine times through water before reaching the cylindrical receiver. This receiver served merely to catch any drops of water mechanically carried in the draft, the finer drops being caught in a square box filled with twigs and oakum. The draft was created in the furnace, and the fume drawn through this rather elaborate system of washers by a vacuum pump connected to the back of the oakum box. A vacuum of 1½ to 4 inches of mercury, according to the height of water in the "zinc box," was required for the suction of the gases through the plant.

A careful test was made on a parcel of ore put through the small furnace, which ran continuously for 83 hours. The test was watched by Messrs. W. A. Macleod and W. B. Blyth, representatives of Bewick Moreing, and Mr. Harley Wright, representing Hooper & Speak. All parties reported this small plant to be quite successful. The volatilization of the gold over this period averaged 92 per cent, according to the assays of the headings and residues; and the gold actually recovered was within a few per cent of this figure. More could not be expected from an experimental furnace working on such a small lot of ore.

The favorable results that were obtained in this small installation led to the construction of a large plant, which, according to Mr. Howe, was as follows:

We found one or two accessories in the plant that worked well, and noted others to be avoided. Our furnace was 5 feet in diameter by 27 feet long. It should have been 45 or 50 feet for this diameter. Our furnace was red hot even at the cool end, so that the heat losses were excessive. The greater length would not only have conserved the heat better, but by giving a greater length in which to roast the ore would have increased the capacity of the furnace. In the 27-foot furnace the first 12 feet were taken up by the ore roasting, leaving only 15 feet for the volatilization. This meant that we had to run with too thin a layer of ore. For the number of times that a given amount of ore is turned and exposed in 15 feet twice the amount of ore would be exposed equally often in 30 feet. The brick lining to this furnace was only 4 inches thick (all we could obtain at the time) when it should have been 9 inches thick. A large loss of heat by radiation resulted from the thin lining. We found with this big furnace that a slope of 1 inch per 6 feet and a speed of one revolution per minute were about right.

Our gas burner was simply a 10-inch pipe passing from the producer through the fire box and cut off flush with the mouth of the furnace. A 2-inch pipe delivering preheated air was screwed into a blank flange at the back of this pipe, so that hot air and gas were mixed in the last 6 feet of the pipe before burning at the nozzle. Both air and gas were at a temperature of 400° to 500° C., and under these conditions we had no trouble in keeping the furnace temperature between 1,000° to 1,200° C. The amount of air per minute passing through this furnace when treating 20 tons per day was 1,200 cubic feet.

At the cool end of the furnace the opening was contracted to 2 feet in diameter, and this opened direct to a dust box made of thin iron plates. The dimensions of this dust box were 8 feet long, 8 feet high, and 2 feet wide. A small dust chamber with baffles is more effective than a large chamber without baffles. The baffles are made of hanging rods and not fixed plates.

To clear the rods, all a workman has to do, once every hour or so, is to give the rods a shake with a piece of wood. Being free to move, they commence to swing about and knock against each other, which clears them immediately.

A push conveyer working in the bottom of the dust chamber will feed ore into the furnace and at the same time automatically return any dust that settles in the chamber. Screw conveyers should not be used. They are not to be compared with push conveyers for dry ore, particularly if there is any heat about.

In passing through the dust chamber the furnace gases are cooled considerably, and 80 per cent of their dust should be retained here. It is highly important to settle as much of the dust as possible without condensing too much sublimate.

The fume leaving the dust chamber is made to enter a medium-sized scrubber, which should be built of wood. For a furnace treating 30 tons per day this scrubber should be about 10 feet in diameter by 12 or 15 feet high.

Water circulating from top to bottom through the scrubber will separate out the dust and about 25 per cent of the gold. The residue thus retained should assay about 100 ounces gold per ton.

On leaving the scrubber the gases are quite cool and practically free from dust. It now only remains to break them up very finely in water before discharging them into the atmosphere. There are two methods I should recommend for doing this: (1) Pull them by means of a Root's blower, through water, causing them to bubble several times before they finally reach the blower, or (2) pass them direct into an apparatus that beats and churns the gases thoroughly with water. In either case it must not be forgotten that wood, earthenware, or lead (regulus metal, 17 per cent antimony and 83 per cent lead, is good) are the only materials that can be used.

The secret of successful absorption seems to be to break the gases up into fine particles in water and to use the same water over again. Owing to the prominence given to the failures of Mr. Croasdale in America, and of the Gwalia plant in Australia, many engineers have doubted whether the fume absorption will ever be successful. I can assure them that it already has been successfully accomplished in my first plant, but on this point I can give testimony other than my own.

Before I left the Gwalia Consolidated I offered to exhibit this apparatus under test, but I insisted on a third man being present to watch the test. Unfortunately, the directors could not agree to this, and so I have no test figures to give of its working.

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(1) Croasdale, Stuart, Volatilization of metals as chlorides: Eng. and Min. Jour., vol. 76, 1903, p. 312; Mining Mag. (London), vol. 10, 1914, p. 200.

(2) Howe, Ben, Description of Howe's volatilization process: Monthly Jour., Western Australia Chamber of Mines, December, 1912.

(3) ———, Gold recovery by volatilization: Min. and Sci. Press, vol. 106. 1913 p. 484.

(4) HOWE, BEN, The volatilization process—A record of experimental work at Gwalia Consolidated Gold Mine: Mining Mag. (London), vol. 9, 1913, p. 437.
(5) METALLURGICAL AND CHEMICAL ENGINEERING, Recovery of gold by volatilization: Vol. 11, 1913, p. 220.
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(7) ———, The Howe volatilization process: Vol. 107, Oct. 4, 1913, p. 535.
(8) MINING MAGAZINE (London), Extracting gold by volatilization: Vol. 8, 1913, p. 219.
(9) PARMELEE, H. C., Volatilization processes in the metallurgy of gold: Met. and Chem. Eng., vol. 11, 1913, p. 230.

PAST ATTEMPTS TO COMMERCIALIZE THE POHLÉ-CROASDALE PROCESS.

RIGBY MINING-REDUCTION CO. MAYER, ARIZ.

The plant of the Rigby Mining-Reduction Co. at Mayer, Ariz., was one of the first attempts to commercialize the Pohlé-Croasdale process. Owing to many difficulties, it proved a failure, according to O. H. Fairchild,[27] who has described it in some detail.

From Fairchild's description of the plant it was noted that the crushing and preparation of the ore was similar to that in most dry-crushing plants. The ore was reduced to a 20-mesh size, mixed with salt, and fed automatically to the furnaces. From this point the plant consisted of units, each one being independent and consisting of a furnace—the rotary type 36 feet long, 5 feet 6 inches in diameter at one end and 6 feet 6 inches in diameter at the other end—a set of collecting chambers, and cooling flues, a blower, with its connecting pipes, a condensing chamber, and a bag house. There was no stack to the furnaces, the draft being caused by the vacuum created by the blowers. All gases were drawn into the condensing chambers and bag house. The precipitate or flue dust was taken to the filter house and treated in leaching tanks for separation of the copper content, the residue being subsequently refined. The plant was supposed to have a capacity of 125 tons a day.

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[27] Fairchild, O. H., The application of the Pohlé-Croasdale process: Min. and Sci. Press, Sept. 1, 1906, vol. 93, pp. 263-264.

PLANT AT FAIRBANK, ARIZ.

By Walter A. Schmidt.[27a]

The chloridizing-volatilization plant built at Fairbank, Ariz., was the first commercial attempt to recover metals by volatilization and electrical precipitation, and it is especially interesting, not only as regards the development of electrical precipitation, but also as regards the development of metallurgy. The plant was put in operation in March, 1918. It was built and first operated by J. H. Hirt & Co. Before the work was completed, Mr. Hirt withdrew his interest in the original company, and a new organization, the Contention Co., was effected, of which Mr. Charles Biesel, of El Paso, Tex., was general manager.

The plant was located near an old tailings dump of a mill built to treat the ores from the mines near Tombstone, Ariz. This dump contains approximately 165,000 tons of tailings, carrying about 5 per cent lead, 5 ounces silver, and $1.40 worth of gold. Large sums of money had been spent in trying various cyanide and leaching processes on these tailings.

The plant at Fairbank consisted of a kiln, 60 feet long by 5 feet in diameter, which had been used by a cement plant as a drier. It was oil-fired, and only one burner was used in the preliminary tests. The fume passed out of the kiln into a small settling chamber, and then into the treater.

The treater comprised one unit of 64 pipes, 14 feet long by 12 inches in diameter, inclosed in a shell made of two old cyanide tanks put together to form a cylinder. Discharge electrodes were of No. 16 iron wire. One electrical unit furnished the power.

The power house contained two oil-fired boilers, one about 45 horsepower, the other about 30 horsepower. There were also two steam engines, one about 35 horsepower and the other about 30 horsepower. The latter engine was used exclusively for the motor generator which supplied the power for the electrical precipitators.

The transformer was manufactured by the American Transformer Co., having a capacity of 20 kv.a. It has a voltage of 220/440 to 100,000 volts. The motor generator was built by the Westinghouse Electric & Manufacturing Co. and was of 15-kv.a., 220-volt, 60-cycle capacity. The switchboard also was of Westinghouse manufacture.

[27a] President, Western Precipitation Co. ,Los Angeles, Calif.

Many difficulties were encountered in running the plant. Insulators cracked on account of deposits of moisture and carbon; the boiler capacity was inadequate, and numerous electrical troubles occurred, owing perhaps not so much to the equipment as to lack of properly trained operators. The electrical difficulties were remedied, for after a few hours spent in adjusting and cleaning various parts of the equipment complete precipitation of the fume was obtained. One of the main difficulties encountered in operation was the feeding of the tailings into the kiln, as part of the tailings were dry, part wet and sticky. The indications were that preliminary drying would be necessary to insure steady and uniform operation.

In view of the fact that so many changes seemed necessary, such as increased boiler capacity and adequate conveying apparatus, requiring the expenditure of a large additional sum of money, and as the profits to be realized from the treatment of available tailings did not seem to justify such expenditure, operations were discontinued July 1, 1918, and the plant was closed and dismantled.

SCOPE OF EXPERIMENTAL WORK DONE AT THE INTERMOUNTAIN EXPERIMENT STATION.

As stated in the introduction, the investigation of the volatilization process by the Bureau of Mines Intermountain experiment station, in cooperation with the department of metallurgical research, University of Utah, began about 1916.

Lyon and Ralston[28] have pointed out the necessity of investigating this process, and have discussed the results of experimental work on different types of ores.

The first experimental work on the process at the station was conducted in muffle tests; improved methods of research have been since evolved, especially in the development of the electric-tube apparatus and the small-scale laboratory experimental plant. (See pp. 33 to 36.)

As a result of the small-scale experiments, which showed that valuable metal could be extracted by this method from various types of ores, a semicommercial installation was made which provided for experiments on a larger scale.

These experiments have been classified and the types of ores have been segregated, so that information relative to the amenability of the volatilization process to any type of ore can be obtained readily by reference to the tables in this bulletin. Most of the experiments were conducted on oxidized and semioxidized ores, as the need of such a process for these ores was more pressing than for the sulphide ores.

COOPERATION OF MINING COMPANIES.

After the encouraging results on a semicommercial scale at the experiment station, work was done in cooperation with several mining companies to demonstrate what the process could accomplish on a commercial scale. These results are described later.

More than 150 mining companies from every part of the United States and many foreign countries have made application to the experiment station for cooperative work on this process. Many of these companies have sent representatives to be present when the experiments were made and visitors numbering into the thousands, interested in the process, have come to the Intermountain station to discuss the process, to see the equipment of the laboratories, and to observe demonstrations of the process.

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[28] Lyon, D. A., and Ralston, O. C., Innovations in the metallurgy of lead: Bull. 157, Bureau of Mines, 1918, 176 pp.

METHODS OF EXPERIMENTATION.

ANALYSIS OF ORE SAMPLES.

In order to obtain data for further experiments on a larger scale, it is of course essential to conduct many preliminary experiments on any ore.

After the analysis has been made and physical characteristics of the minerals in the ore studied, the usual procedure is to conduct experiments either in a muffle or in an electric-tube furnace in order to obtain preliminary operating data. These methods of experiment are described in some detail on page 33.

The ores were invariably analyzed both for their chemical and their mineralogical constituents, and were examined by the microscope to determine the physical condition of the minerals. The chemical analysis showed the various amounts of metals and other constituents present, and the microscopic analysis revealed how the minerals occurred. By the microscopic examination it was possible to determine accurately the size of crushing necessary to liberate the greatest amount of mineral from the gangue. The compactness or porosity of the ores in which the minerals occurred was an important factor; usually the compact ores required finer crushing.

When the crushing size of the ore had been determined, it was necessary to calculate from assays and analyses the amount of chloridizing salts to add to the charge in order to insure enough chlorine to unite with the metals and to insure complete chloridization and volatilization of the minerals.

MUFFLE TESTS.

The assay muffle was generally used and was heated to a temperature necessary to effect volatilization. This temperature varied somewhat, but for ores carrying silver it commonly was about 1,050° C. As a rule, 100 grams of crushed ore was used, being thoroughly mixed with a certain percentage of salt or calcium chloride. These mixed charges were put in a 5-inch or 6-inch roasting dish, which was placed in the heated furnace. Several series of tests were made in which the varying factors were the following: Time of roast, temperature of furnace, fineness of crushing of ores, and varying amounts of salt and calcium chloride.

The residues after roasting were weighed and assayed, and the percentages of metals volatilized were calculated. In these experiments none of the fumes evolved were caught.

ELECTRIC-TUBE TESTS.

The electric tube shown in Figure 1 consisted of a horizontal electric-tube furnace containing a silica or porcelain tube. In the tube was placed a small boat containing the mixed charges. It was possible to pass varying amounts of air through the tube and to collect the fumes and measure the gases; for this reason and because of the better control of temperature, the apparatus was more desirable for preliminary experiments.

SMALL-SCALE TESTS.

The equipment used for these experiments is described on page 34, and is shown in Plate I, *A* and *B*, page 34.

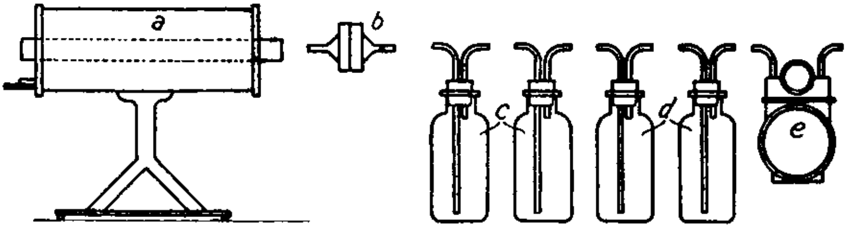


FIGURE 1.—Diagram showing electric-tube apparatus set up for tests: *a*, electric-tube furnace; *b*, Mohr filter; *c*, distilled water; *d*, standard $AgNO_3$ solution; *e*, gas meter.

The furnace was fired with a blowtorch, using low-pressure artificial gas or oil and air at about 20-pound pressure. The control of the temperature was satisfactory, any desired temperature up to 1,250° C. being maintained without difficulty.

Tests were made in the small laboratory plant both as research and as preliminaries to the large kiln tests. From 10 to 25 pound samples proved desirable for these experiments. The ore, with part of the chloridizing reagents, was fed into the upper end of the furnace by means of a mechanical ore feeder, the remaining part of the chloridizing reagents being injected directly upon the charge in the front or heated end of the furnace.

METHOD OF CONDUCTING ELECTRIC-TUBE TESTS.

The material to be treated was put in a porcelain boat 16 by 100 millimeters and submitted to the heated furnace, being placed in a porcelain tube 1 by 12 inches, which protected the heating elements from the action of corrosive gases. The gases from the furnace were first passed through a Mohr filter, where the solid matter was collected, then through four wash bottles—the first two containing distilled water and the last two standard solutions of silver nitrate.

In all bottles the gases bubbled through 4 inches of liquid, and, as shown by the chlorine balance obtained in most tests, the gases leaving the last bottle were usually free from chlorine. It was thought that, after the solids had been removed by the filter, the wash solutions would remove the free chlorine and hydrochloric acid from the gases, and that the silver nitrate solutions would be used as guard solutions to catch any fume going through the filter, for it was known that only water washing would not remove this fume from the gases. Water seemingly did not wet the fume. Usually the greater part of the chlorine was removed before the gases reached the silver nitrate solutions; sometimes these solutions collected a considerable part of the chlorine, and in a few tests some chlorine escaped from the last wash bottle.

As the volume of gases drawn over the charge in the furnace influenced the rate of the evaporation of the chlorides, the gases from the wash bottles were drawn through a wet type of gas meter, graduated in liters. The temperature and vacuum on the meter and the meter readings were recorded at regular intervals. The volume was regulated by means of a filter pump.

A platinum and platinum with 10 per cent rhodium thermocouple, suitably protected in a porcelain tube, was used to measure the temperature which was read in degrees centigrade registered by a millivoltmeter.

PROCEDURE.

The furnace was first heated to the desired temperature, the wash bottles were properly filled with their respective solutions, all connections were properly fitted, and the desired quantity of gas was passed through the system. The charge was then put to the furnace and the data were recorded at regular intervals. Upon completion of the test all products were collected, analyzed, and assayed. The computations made showed the metal extracted, the total gas volume, the fume collected, and other results.

LABORATORY VOLATILIZATION PLANT.

The small laboratory plant described herein was designed by C. C. Stevenson, research fellow, and built by C. E. Orhn, mechanic, University of Utah. It was patterned to include the basic features of a commercial installation, and so arranged that any one condition could be changed, leaving all others constant. The flexibility of such a plant offered a splendid opportunity to make direct observations of the effects produced under different conditions. The equipment consisted of a rotary kiln, dust chamber, and a double-unit Cottrell electrostatic precipitator.

FURNACE.

The furnace was 36 inches long and 6 inches inside diameter for the first 24 inches from the feed end. The remaining 12 inches had a diameter of 8 inches. The furnace was lined with fire brick with silocel filling between them.

A 2-inch transite board nose ring in the feed end of the furnace kept the charge from working back into the dust chamber. A 1½-inch beveled fire-brick nose ring in the enlarged end or fire box caused the charge to remain in the heated zone of the furnace for as long a period of time as was desired.

The shell, made of ¼-inch sheet iron, was 14 inches in diameter and 36 inches long, with two steel tires 1¼ inches wide and ½ inch thick, welded to the outside 9 inches from each end. The furnace was mounted on four rollers with bearings suitably fastened to an adjustable 2¼-inch timber frame. The rollers were partly submerged in a small sheet-iron vessel with circulating water to prevent overheating of the bearings and to protect them from the heat from the furnace, as shown in Plate I, *A*.

The upper, or feed end of the kiln, projected into a $\frac{1}{16}$-inch sheet-iron dust chamber 24 inches high, 20 inches wide, and 5 inches long. A slide door at the bottom provided a means for recovering the dust. A 4-inch asbestos-jacketed stovepipe conducted the gases laden with fume from the furnace. Provision was made whereby the gases might or might not be passed through the treaters.

COTTRELL ELECTROSTATIC PRECIPITATOR.

The fume precipitator was a double-unit Cottrell treater (see Pl. I, *B*), one unit having two vertical pipes, and the other unit four vertical pipes 4 inches in diameter and 5 feet long. The units could be used separately or in parallel, which was equivalent to using either a two, four, or six tube treater. An insulated No. 16 gage nichrome wire suspended through the center of each pipe was charged with pulsating direct current at 30,000 to 50,000 volts potential.

Suitable inclosures for the ends of the pipes were made of No. 14 gage sheet iron. The gases entered the treaters at the top and were drawn off at the bottom, as shown in Plate II, *B*.

GAS CIRCULATION.

A ¼-horsepower induction motor operating a No. 00 Buffalo Forge blower caused the gases to circulate through the system. The gas velocity through the system was regulated by slide dampers suitably located in the flue system.

ELECTRICAL TREATER EQUIPMENT.

One General Electric transformer, type K, Form E. V., 60-cycle, capacity 5 kv.a., 50,000 volts to 100, 220, and 400 volts, was used to furnish the necessary potential. One overload circuit breaker, General Electric type C, Form G, two poles, 25 amperes, and 250 volts, placed in the primary circuit, afforded the necessary overload protection.

VOLTAGE CONTROL.

The high-tension voltage was controlled by means of a General Electric potential regulator, type M I R S 2–2–60, Form L K, 60 cycles, 2 kv.a., primary voltage 110 to 220, secondary amperage 18.2 to 9.1, placed in the primary circuit, together with a resistance; capacity 0–3.2 ohms, 16 amperes.

RECTIFIER.

The alternating-current high-tension voltage was converted into a direct current by means of a rotary-disk rectifier, similar to the Western Precipitation Co.'s No. 20.

ORE FEEDER.

The charge was mechanically fed into the rotary kiln by a screw feeder, which was essentially a 1-inch auger bit, revolved inside of a 1-inch water-cooled pipe. (See Plate II, *A*.)

The 1-inch pipe was water-jacketed from the discharge end to the hopper with a 2-inch pipe covering. A hopper with a capacity of about 10 kg. of ore was connected to the 1-inch pipe. A slot cut in the pipe exposed the auger bit at the bottom of the hopper.

VARIABLE SPEED TRANSMISSION.

The power from an alternating-current motor, type S. A., Form S. I., 110 volts, 2.3 amperes, speed 1,140 r. p. m., 1/12 hp., 1 phase, 60 cycles, was transmitted to the kiln and ore feeder through a system of bicycle sprockets and chain, as shown in Plate II, *A*.

KILN SPEEDS.

Accommodations were made for three kiln speeds—1⅓, 2⅛, and 2¾ revolutions per minute, respectively. The same chain could be shifted to any of the three combinations.

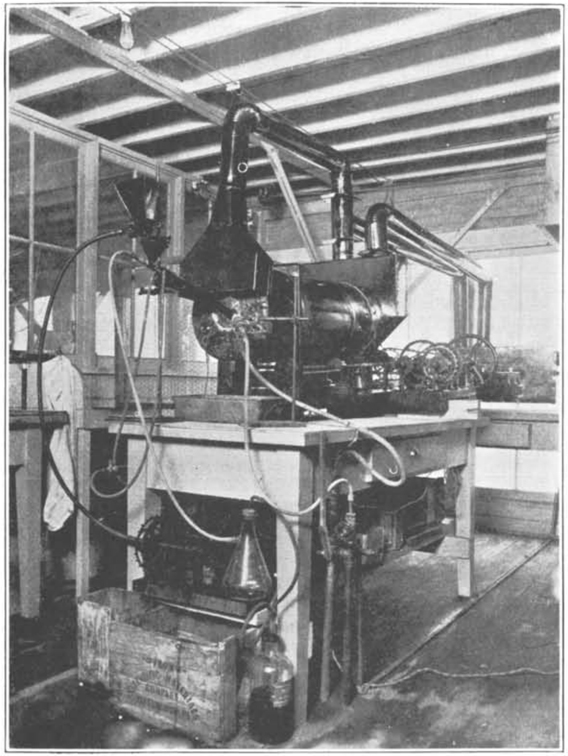
VARIABLE ORE FEED.

A similar combination of sprockets transmitted power to the ore feeder. Eight speeds revolving the auger bit 3 to 10 revolutions per minute were available. This arrangement was equivalent to feeding about 1,000 to 3,000 grams of ore per hour.

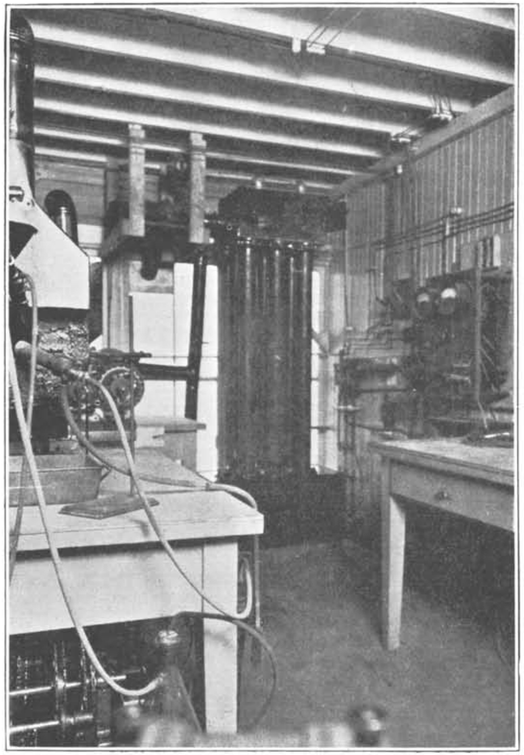
SUMMARY OF FEATURES OF PLANT.

A temperature range up to 1,250° C. could be maintained throughout a test with considerable accuracy.

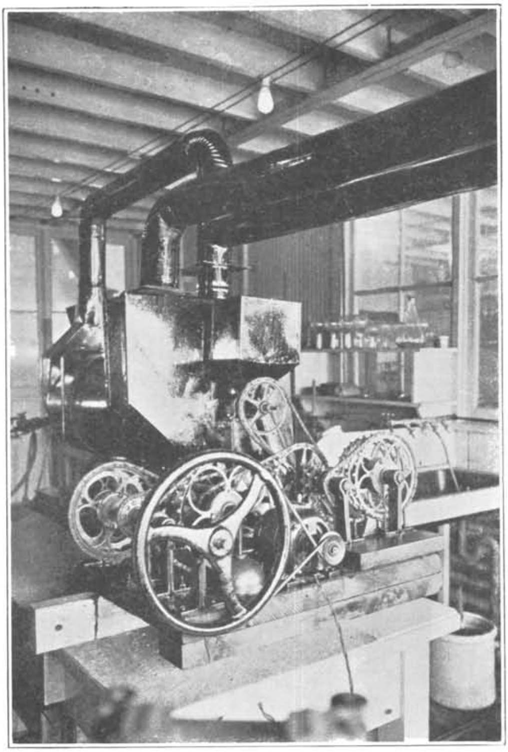
Three different kiln speeds, approximately one, two, or three revolutions a minute, were possible. Eight speeds for the ore feeder having a capacity of 1,000 to 3,000 grams an hour, because the rate of feed may be different for each ore treated.



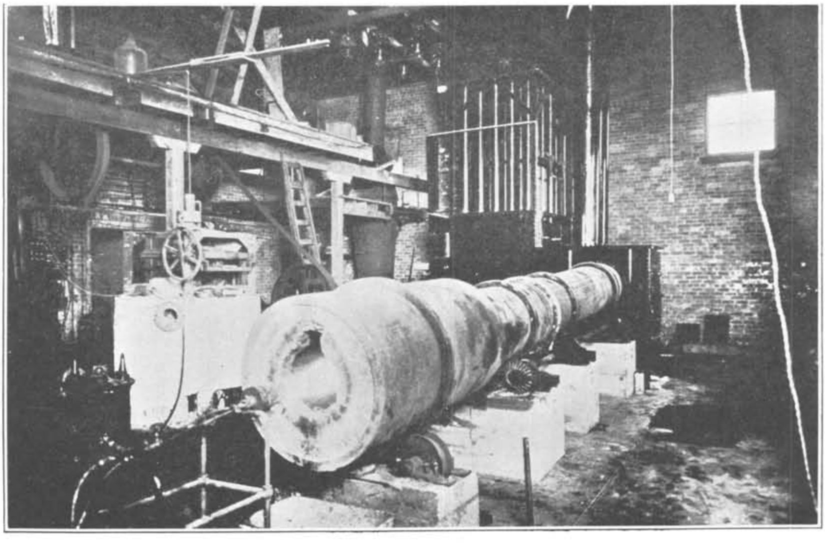
A. SMALL-SCALE LABORATORY KILN AND CHLORIDE INJECTOR.



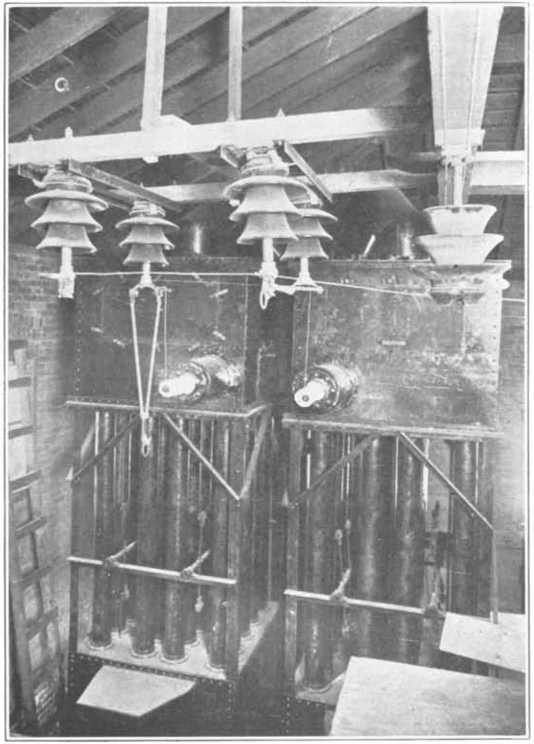
B. COTTRELL TREATERS, 2 AND 4, OR 6 UNITS, IN SMALL-SCALE LABORATORY INSTALLATION.



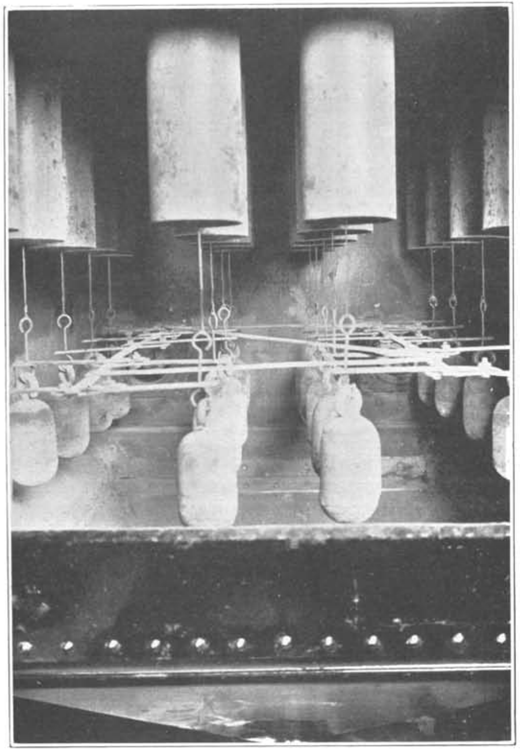
A. VARIABLE-SPEED MECHANISM FOR REGULATING KILN SPEED AND ORE FEED; SMALL-SCALE LABORATORY INSTALLATION.



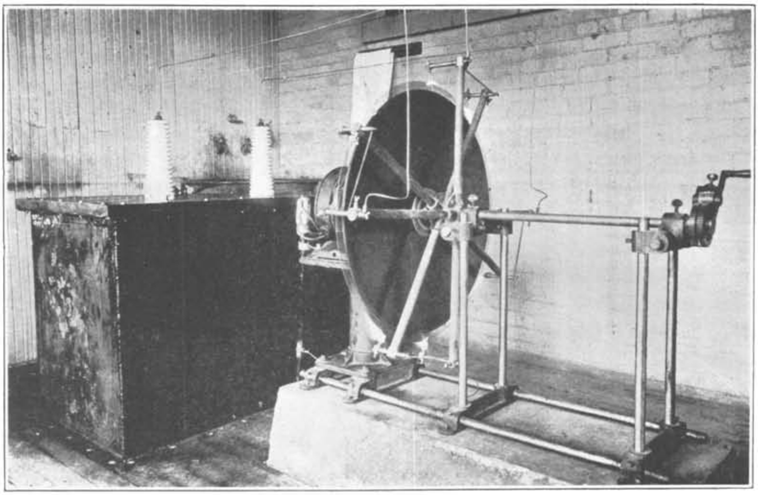
B. ROTARY 20-FOOT KILN, WEDGE ROASTING FURNACE (LEFT), AND COTTRELL TREATERS (REAR); HAUCK BURNER IN OPERATION SHOWS HEATING OF KILN BY DIRECT FLAME; SEMICOMMERCIAL INSTALLATION.



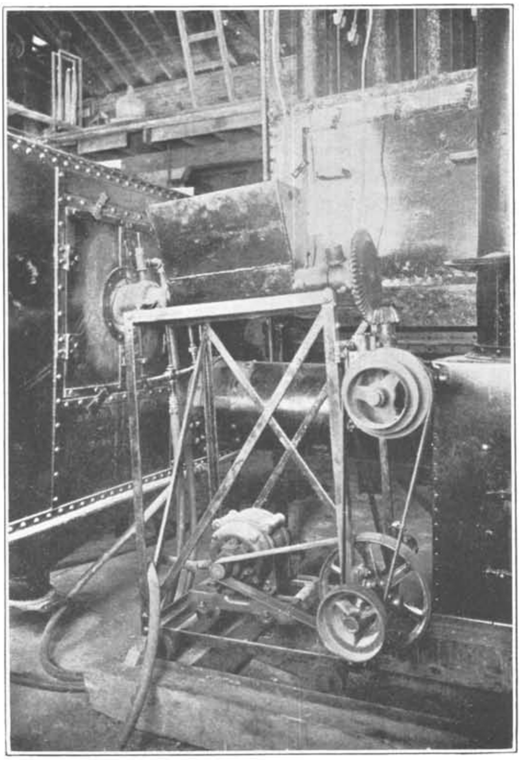
A. TWO 20-TUBE UNITS OF COTTRELL TREATERS AND INSULATORS.



B. DISCHARGE END OF COTTRELL TREATERS SHOWING METHODS OF KEEPING HIGH-VOLTAGE WIRES INSULATED AND IN TENSION.



A. RECTIFIER AND TRANSFORMER USED TO TRANSFORM VOLTAGE UP TO 100,000 VOLTS.



B. WATER-COOLED ORE FEEDER, VARIABLE-SPEED, USED TO FEED ORE INTO THE 20-FOOT ROTARY KILN.

Electrostatic fume arrestor, which could include either two, four, or six pipes.

The gas velocity was under immediate control by means of slide dampers, suitably located in the flue system.

A wide variation of potential could be supplied to the treaters.

BEHAVIOR IN TESTS.

The mechanical operation of this plant was very satisfactory and tests were made on a large variety of ores. A new feature was added to the installation wherein part of the chloridizing reagents—namely, calcium chloride—could be admitted to the charge in the front end or heated zone of the furnace at regular intervals during the test.

Experiments on many ores were conducted and it was possible to accumulate all necessary data for large-scale experiments. It was possible to collect practically all the fume and usually in experiments on lots of 3 to 5 kilograms of ore a metallurgical balance could be obtained. The semicommercial plant described later was used for large lots of ore and to make confirmatory tests based on data accumulated in the plant just described.

CHLORIDE INJECTOR.

A mechanical chloride injector as designed by C. C. Stevenson was built for the purpose of supplying the halogen salt at a constant rate to the heated charge in the furnace. (See Pl. I, *A*.) The essential feature of the mechanism was a screw feeder, revolving in the bottom of a hopper, which discharged the salt into a cone-shaped hopper at a constant rate. The apex of the cone hopper was connected to an orifice leading to the inside of a $\frac{1}{2}$-inch water-jacketed pipe.

A jet of air under any desired pressure from a $\frac{3}{16}$-inch nozzle, as shown in the drawing, caused a suction through the orifice, which conducted the chlorides by means of a draft of air into the furnace.

The screw feeder was driven by means of a $\frac{1}{12}$-hp. alternating-current motor, speed 1,140 r. p. m., through a variable speed transmission composed of a Jones reduction gear and a system of bicycle sprockets and chains. From the sprocket transmission a flexible automobile speedometer cable was directly connected to the screw feeder.

CAPACITY OF SCREW FEEDER.

There were 48 speed adjustments of the screw feeder ranging from about $\frac{1}{5}$ to $1\frac{1}{3}$ r. p. m., which could feed from about 75 to 485 grams of -16-mesh calcium chloride ($CaC1_2.2H_2O$) an hour. As good results were obtained by using the mechanical chloride injector as by feeding the chloridizing reagent by hand. The results obtained in the small kiln were later duplicated in the semicommercial plant.

PRESENT SEMICOMMERCIAL INSTALLATION.

The plant installed at the station, excepting the treaters, was designed by Thomas Varley, F. G. Moses, and J. C. Morgan, Bureau of Mines metallurgists. The Western Precipitation Co., of Los Angeles, Calif., designed the treaters and all electrical equipment.

The plant consists of a cement-type rotary kiln, dust chambers, Cottrell electrostatic precipitator, and auxiliary equipment.

ROTARY KILN.

The furnace is 20 feet long, with an internal diameter of 13 inches for the first 15 feet from the feed end and 21 inches for the last 5 feet, or fire end. (See Pl. II, *B*.) A sheet-metal nose ring at the feed end prevents the charge from traveling back into the dust chamber, and a fire-brick nose ring in the opposite end of the furnace retains the ore charge in the enlarged section of the fire box for a longer roasting period.

The shell is constructed in two sections, each 10 feet long, of ¼-inch boiler plate, with two cast-iron tires on each section. The tires are bolted to the shell 2 feet from the nearest end. The joining ends of the sections are provided with a vertical 1½-inch angle-iron flange riveted on the circumference of the shell 1 inch from the end. Twelve equally spaced slots $\frac{9}{16}$ inch wide and 1¼ inches deep provide bolt holes for fastening the sections together. The inside diameter of the shell is 23 inches in the 15-foot length and 28 inches in the enlarged end. The shell is lined with a single layer of No. 6 arch fire brick in the 15-foot length and No. 7 arch brick in the enlarged end. The kiln is mounted on trunnions suitably fastened on wooden blocks to concrete foundations, giving it an adjustable slope. For most purposes a slope of half an inch a foot is suitable. The kiln is provided with five speeds, ranging from about one to three revolutions a minute.

DUST CHAMBER AND GAS FLUES.

The feed end of the kiln projects into a ⅛-inch sheet-iron dust chamber, 28 inches wide, 44 inches high, and 62 inches long, with a hoppered bottom provided with a sliding door, from which the dust is removed and returned to the feeder. From this first dust chamber a 13-inch sheet-iron cylindrical flue connects to a second dust chamber, 24 inches wide, 45 inches long, and 45 inches high. Two vertical metal flues 13 inches in diameter connect the top of the second dust chamber with the tops of the Cottrell treater units and have suitable damper arrangements, so that the treaters may be used separately or in parallel.

COTTRELL ELECTROSTATIC PRECIPITATOR.

The fume-arresting device as designed by the Western Precipitation Co., of Los Angeles, Calif., comprises two separate Cottrell precipitator units, shown in Plate III, *A*. Each unit consists of 20 vertical, 6-inch pipes, 10 feet long. Through the center of each pipe is suspended an insulated No. 16-gage, nichrome wire. A 20-pound weight suspended on the end of each wire maintains the proper tension and an insulated metal-web fixture holds the wire in place.

Sheet-iron metal chambers, 4½ by 4½ by 4½ feet, provided with the necessary doors, form inclosures for the ends of the pipes. The furnace gases, laden with fume, enter the treaters at the top and leave at the bottom. The lower chamber is provided with a hopper-shaped bottom, from which the fume is recovered, also an outlet for the spent gases. (See Pl. III, *B*.)

The suspended wires are negatively charged with a high-tension, direct-current voltage, ranging from zero to 100,000 volts. The electrical pressure on these insulated wires is raised to a point where it causes a corona brush discharge between the wire and the grounded pipe, which sweeps the solid matter from the gases and deposits it on the inside of the pipes.

Electrical equipment (5 kv.a.) used in connection with the treaters (see Pl. IV, A).

One transformer, Thordason type, A. S. C., No. 5340-2, 50-cycle, 220-440-100,000 volts.

One rectifier, Western Precipitation Co.'s No. 20.

One autotransformer with voltage taps at intervals of 55 volts from 0 to 550. This transformer regulates the primary voltage supplied to the main transformer.

One carbon circuit-breaker, Westinghouse Electric Co., type C. D., style No. 168301 B, 10 amperes, 600 volts, placed between secondary of the autotransformer and the primary of the main transformer, affording the necessary protection against overload.

One Westinghouse electric direct-current ammeter, type S. L., style No. S. L. 20166, which measures the amperage supplied to the treaters.

One alternating-current ammeter, Westinghouse Electric Co.'s type S. M., style No. 194355, 45 to 65 cycles, 5 amperes in meter circuit.

One alternating-current voltmeter, Westinghouse Electric Co.'s type S. M., style No. 156390, 45 to 65 cycles, 600 volts on meter and resistance.

One alternating-current wattmeter, Westinghouse Electric Co.'s type S. M., style No. 5 L. A. 17189, 60 cycles, 5 amperes, 400 volts, 2,000 watts in circuit, measures the electrical quantities supplied to the primary circuit of the main transformer. (See Pl. IV, *A*.)

HEATING EQUIPMENT.

The furnace is heated by means of a direct flame playing on the inside lining of the enlarged end of the kiln. A high-pressure Hauck burner, using gas oil and air under 40 pounds pressure, has given satisfactory results. A compressor, size 6 by 6, N. S. B., is used to furnish the necessary air pressure.

GAS CIRCULATION.

The gases are circulated through the system by means of a turbine fan driven by a 3-horsepower, 200-volt, 60-cycle, 3-phase motor, located between the treater and the stack. The gas velocities are regulated by means of slide dampers conveniently situated in both the inlet and outlet flues to the treaters.

ORE-FEEDING DEVICE.

The ore-feeding device used was designed by J. C. Morgan, assistant chemist, Bureau of Mines, and C. E. Ohrn, mechanician, University of Utah. The ore charge is mechanically fed into the upper and cool end of the furnace by means of a 3-inch screw feeder, operating inside of a 3½-inch O. D. steel boiler tube four extra wire gages thick. This tube is surrounded from the discharge end to the ore hopper by a 6-inch pipe, which serves as a water jacket.

The screw is rotated by means of a 2-horsepower, 60-cycle, 3-phase, 220-volt motor. The transmission arrangement from the motor to the feeder is essentially a pair of step-cone pulleys. Seven combinations are available with the cone pulleys, which vary the screw feeder speed from 3.16 to 7.75 r. p. m. This combination affords a charge feed of about 100 to 300 pounds an hour with intermediate adjustments. The feeder equipment is separately constructed on a portable frame, mounted on four wheels so that it may be withdrawn from the furnace when not in use, as illustrated in Plate IV, *B*.

OPERATION OF SEMICOMMERCIAL UNIT.

In the semicommercial installation the experiments were conducted on the basis of results and data obtained in the muffle, electric tube, and small-scale laboratory installation. Tests in this plant were made on lots of 200 pounds to several tons, and the results obtained indicated what might be expected in commercial operation.

The ores to be treated were crushed to the desired size and mixed with the necessary amount of salt or other haloid, put into the hoppered screw feeder, and fed into the 20-foot kiln in any desired quantity. When the kiln was heated to the desired temperature by the Hauck burner, the voltage was regulated in the treater, and the feeder started. The time required for the charge to pass through the kiln depended on the revolutions per minute as controlled by a series of step pulleys. (See Pl. II, *B*, p. 35.)

TEMPERATURE CONTROL.

Temperatures were taken at the fire end of the kiln by means of an optical pyrometer and in the flues with thermometers. The temperature was controlled entirely by the burner flame.

GASES.

The velocity of the gases through the kiln was controlled by dates in the pipe between the treaters and the exhaust fan, which were regulated as desired, according to the amount of fumes given off, or by means of Pitot tube readings taken in the gas conduits.

SOLIDS.

When so desired, total solid determinations were made on the gases entering and leaving the treater. The difference in amounts of solids represented the effectiveness of the treater in collecting the fume. The apparatus and method of determination have been described by the Western Precipitation Co.[29]

PRODUCTS.

The calcine discharged from the kiln was collected, weighed, and after cooling was sampled for assay. An appreciable amount of the charge often overflowed at the feeder, was caught in the dust chamber, then returned to the kiln through the feeder. All products were weighed and sampled for assay so that a metal balance might be obtained.

OPERATION OF THE COTTRELL PRECIPITATOR.

The operation of the Cottrell precipitator is illustrated schematically in Figure 2. The pipe *a*, or collecting electrode, terminates in headers *b* and *c*. The discharge electrode *d* is suspended axially in the pipe, and is insulated from it at *e*, and held taut by weight *f*. Gases enter from flue *g* and pass through pipe *a*, and when the current is on and the precipitator in operation, the charged particles are deposited on the electrode *a*, and the gases pass through the pipe to the outlet *h*. The deposited particles are dislodged from the electrodes by means of rapping devices which deliver a sharp blow on the pipe, the collected material falling into the hopper *i*. If liquid in nature, the material may run down the surface of the electrodes into a sump or other suitable receiver.

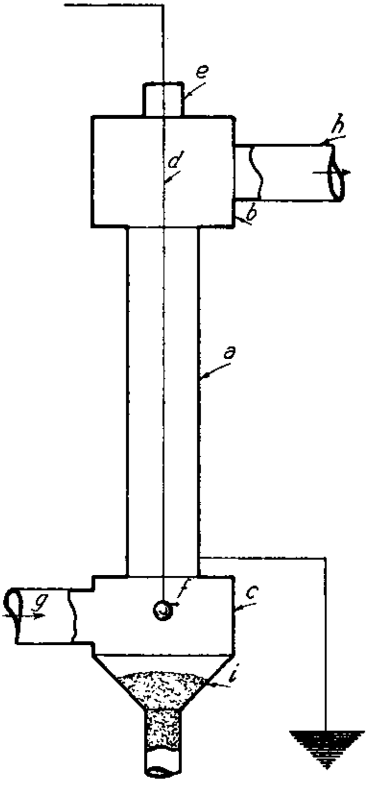


FIGURE 2.—Diagram illustrating the operation of the Cottrell precipitator: *a*, collecting electrode terminating in headers *b* and *c*; *d*, discharge electrode; *e*, insulator; *f*, weight; *g*, point where gases enter; *h*, outlet; *i*, hopper.

OPERATION OF BRÜCKNER FURNACE.

The Brückner furnace is heated to the desired temperature with the same burner as that used for the 20-foot kiln, and the prepared charge is shoveled into the furnace through the opening in the fire end. This furnace is used for charges of 20 to 200 pounds and has the advantage that a charge may be held in the furnace for any

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[29] Western Precipitation Co., Methods of determination of velocity, volume, and dust concentration in gases. Los Angeles, Calif.

desired length of time with perfect temperature control. The thickness of the bed of ore can also be controlled by the amount of material or charge put in the furnace. Grab samples taken at regular intervals show the rate of the volatilization of the metals.

This furnace is a revolving iron drum, 5 feet 6 inches long by 2 feet 5 inches in diameter, lined with fire brick and having openings 13 inches in diameter in each end. One end is connected to the flue leading to the treaters, and the furnace is fired through the other with the same burner as that used in heating the 20-foot kiln. By mechanical devices the furnace can be revolved at any desired speed. The gases are conducted through the treater, and the velocity measurements and total solid determinations may be made as in operating the 20-foot kiln.

The residue is cleaned out of the furnace and after cooling is weighed and sampled for assaying. All products are weighed and sampled in order to obtain a metal balance.

PROCESS APPLIED TO VARIOUS COMPLEX ORES.

VOLATILIZATION OF GOLD.

The volatility of gold and the formation of various chlorides is discussed briefly on page 8. Some research is being conducted on the physical chemistry of gold chloridization and volatilization, but at present little more can be said than what has already been quoted on preceding pages. The behavior of gold ores during volatilization and the amount of gold volatilized and subsequently precipitated in the treaters indicate that whatever may be the chemical reaction or the conditions best suited for volatilization, the gold is readily volatilized under very simple experimental conditions.

GOLDEN REWARD ORES.

Some experiments were conducted with ores from the Golden Reward Mining Co., Deadwood, S. Dak., which assayed 0.42 ounce of gold a ton. A series of experiments was conducted in the muffle at 900° C. temperature for one hour. The varying amounts of salt used and the percentages of gold volatilized are shown in Table 1. The amount of the gold volatilized ranges from 28½ to 90.8 per cent, which was obtained by the addition of 8 per cent salt and 4 per cent lime. The addition of the lime was made in order to prevent the charge from fusing.

TABLE 1.—*Results of tests with Golden Reward ore from Deadwood, S. Dak.*

Weight of ore.	Reagents.			Weight of calcine.	Gold in calcine.	Gold volatilized.
	NaCl.	CaO.	S.			
Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Grams.*	*Ounces.*	*Per cent.*
100	2			87.0	0.30	37.8
100	4			87.3	.12	75.0
100	6			88.6	.08	83.1
100	8			89.3	.05	89.4
100	8	4		96.4	.04	90.8
100	8		2	89.8	.05	89.3
100	8	4	2	96.4	.05	88.5
100	4	(1)		85.8	.35	28.5

(1) Moistened.

TROJAN GOLD ORE.

A series of experiments were carried on with ores from the Trojan Mining Co., Deadwood, S. Dak. A 100-pound sample of low-grade ore, "blue rock," was received from the Trojan Mining Co. for tests on the extraction of the gold by volatilization. The ore was first crushed through a ¼-inch screen, then split down to get a 10-pound sample, which was ground until all had passed a 100-mesh screen. From the minus 100-mesh material a head sample was taken, which analyzed as follows:

Analysis of minus 100-mesh sample of "blue rock" from Deadwood, S. Dak.

Au....ounces..	0.82	Fe....per cent..	5.74
Ag....do....	1.32	Al_2O_3....do....	1.60
Pb....per cent..	.66	Insoluble....do....	87.20
Zn....do....	1.59		

Volatilization tests were made to ascertain the effect of various amounts of salt as a chloridizing agent and the effect of different additional reagents. Experiments, as shown in Table 2, were conducted on the ore crushed to different degrees of fineness, that through a 100-mesh screen giving the best results. Ore was crushed through 100 mesh for all tests and the temperature was 900° C. The tests were conducted by M. H. Smith.

TABLE 2.—*Results of tests with Trojan mine gold ore from Deadwood, S. Dak.*

Test No.	Weight, ore.	Reagents.	Weight.	Time.	Weight, calcine.	Au in calcine.	Gold volatilized.
	Grams.		*Grams.*	*Hours.*	*Grams.*	*Ounce.*	*Per cent.*
11	100	NaCl	6	1	100.6	0.13	84.1
12	100	NaCl	8	1	100.8	.13	84.0
13	100	NaCl FeS_2	4 1	1	98.3	.14	83.2
14	100	NaCl FeS_2	4 2	1	100.5	.23	71.4
15	100	NaCl	6	1½	98.5	.09	89.2
16	100	NaCl	8	1½	97.5	.07	91.7
17	100	NaCl FeS_2	6 2	1	100.5	.12	85.3
18	100	NaCl FeS_2	8 2	1	101.5	.09	88.9
19	100	NaCl CaO	8 4	1	107.5	.10	86.9
20	100	NaCl CaO	8 8	1	111.0	.10	86.5

Consistent increase in extraction due to finer grinding was the outstanding feature of these tests on 100-mesh ore. Tests 11, 12, 13, and 14 gave direct comparison with the 30-mesh materials (not tabulated), as they were duplicates of previous tests. The increase in extraction ranged from 15 to 20 per cent. Tests 15 and 16 were made to determine the influence of a longer time in the furnace and showed a much better extraction than tests using a shorter time. (Compare tests 15 and 16 directly with tests 11 and 12.) An increase in the amount of salt while using pyrite resulted in steadily increasing volatilization, as shown in tests 17 and 18. In the last two tests, 19 and 20, large amounts of lime were tried, but with no apparent advantage, as was also the result with the 30-mesh material.

In conclusion, it should be said that though the tests on 30-mesh ore were not encouraging, the tests on finer ore were much better. Volatilization of gold was greatly helped by high temperature, being at 1,000° or 1,100 ° C., much above that at 900° C. The point of sintering determines the limits to which the temperature can be carried with any particular ore.

VOLATILITY OF GOLD IN MIXED ORES.

In nearly all complex ores gold is one of the mineral constituents, and in most experiments the volatilization of this metal was usually rather high, as noted in experiments on Ruby Hill ore, Table 13 (p.58). The assay of this sample was 0.238 ounce gold a ton, 2.82 ounces silver, 3.75 per cent lead. The experiments showed that 92.9 per cent of the gold was volatilized.

In another set of experiments (p. 65), on the Pacific mine ore, which assayed gold, 0.25 ounce; silver, 1.15 ounces; copper, 1.08 per cent, the results indicated that 80 per cent of the gold was volatilized. In many of the tabulations the percentage of gold volatilized was comparatively high, probably averaging higher than for most other metals.

VOLATILIZATION OF SILVER.

Numerous experiments were conducted with the volatilization of silver chloride from argentiferous ores. In addition to experimentation on ores, research was done along physicochemical lines, both at the Intermountain station, at Salt Lake, Utah, and the Pacific station, at Berkeley, Calif. The investigations included the determination of the vapor pressure of the silver chloride at various temperatures; also an examination of the hydrolysis of silver chloride by water vapor, resulting in the formation of metallic silver. Approximate determinations were made of the equilibrium, at various temperatures, between silver, silver chloride, water vapor, and hydrochloric acid. The temperatures at which several common minerals of silver were oxidized or disassociated were noted.

From the results of the last-mentioned experiments it seems that the roasting of silver minerals tends to produce metallic silver. Chloridizing roasting of silver minerals, therefore, is essentially a problem of chloridizing metallic silver and subsequent volatilization of silver chloride. In the furnace, conditions must be maintained that prevent the silver from being hydrolyzed and from reverting to the metallic state before passing from the ore as silver chloride during volatilization. After further study a more detailed report covering these experiments will be published.

Silver is not easily chloridized and volatilized, and has so far offered greater difficulty than any of the common metals. It seems extremely sensitive to atmospheric conditions in the furnace and probably is easily affected by the gangue constituents of the ore.

Silver minerals occur in many low-grade and complex ores of lead, zinc, and copper, silver sometimes being of major importance, especially in economic value. A high percentage extraction is required to make any process for such ores commercially feasible.

In many ores the silver has minor value. When silver is present with rather high percentages of lead and copper, it may not seem economical to provide the conditions for making high extractions. The tabulations shown and discussions of the ore treated in this report deal with ores in which silver is the principal content. Under complex ores silver is also discussed. Typical ores described are as follows: Deer Trail ore from Marysvale district, Utah; Ruby Hill ore, Eureka, Nev.; Tintic Standard Mining Co.'s ore, Eureka, Utah; typical silver ore representing the Creede district, Creede, Colo.; Ontario Mining Co.'s oxidized ore, Park City, Utah; Darwin silver ore, Darwin, Calif.; Ramshorn Mining Co.'s ores, Bayhorse, Idaho; and the Silver Reef Consolidated Mining Co.'s ores, Silver Reef, Washington County, Utah. Each of these ores is discussed separately and tabulations are shown covering the experiments in detail.

ONTARIO MINE DUMP ORE.

Ontario dump ore is essentially a silver ore representing a large tonnage on the dumps at the Ontario mine, Park City, Utah. The analysis was as follows:

Analysis of silver ore from Ontario mine, Park City, Utah.

Au	ounces	0.01	Fe	per cent	2.33
Ag	do	8.98	CaO	do	3.10
Pb	per cent	.65	Insoluble	do	85.20
Zn		Trace.	Al_2O_3	do	3.88

After encouraging preliminary tests, the sample was crushed to −16-mesh size and treated in the semicommercial plant. A temperature of 1,000° to 1,050° C. was maintained in the hottest part of the kiln during the test.

Results of volatilization tests on silver ore from Ontario mine, Park City, Utah.

SUMMARY OF DATA.

Charge.	Weight of charge.	Size or ore.	Percentage of total charge.
	Pounds.		
Ore	1,566	Through 16-mesh	
Calcium chloride	47		Less than 3.
Salt	94		Less than 6.
Lime	94		Do.
Total mix	1,801		100.

PRODUCTS AND ASSAYS.

Item.	Weight.	Assay.		
		Ag.	Insoluble.	Cl.
	Pounds.	*Ounces.*	*Per cent.*	*Per cent.*
Heads	1,556	8.98		
Calcine discharged	1,500	2.50		
Calcine in kiln	91	2.30		
Dust-chamber dust	38	7.36		
Flue dust	6	26.40		
Grab sample No. 1		8.10		
Grab sample No. 2		1.88		
Fume	72½	113.80	23.50	30.32
Total	1,707½			

The loss in weight of the charge was 93½ pounds, or 5.18 per cent.

A more complete analysis of the fume than is shown in the preceding table follows:

Analysis of the fume of volatilization tests with silver ore from Ontario mine.

Au	ounces	0.35	Fe	per cent.	1.75
Ag	do	113.80	Insoluble	do	23.50
Pb	per cent.	10.9	As		None.
Zn	do	5.9	Cl	per cent.	30.32
Cu	do	1.18			

Distribution of silver in test of ore from Ontario mine.

	Ag.	
	Units.	Per cent.
Heads	14,062.7	100.0
Calcine discharged	3,750	26.5
Calcine in kiln	209.3	1.5
Dusts	438.1	3.0
Fume	9,665.2	69.0
	14,062.7	100.0

VOLUME OF GAS, TOTAL SOLIDS, AND TREATER EFFICIENCY.

The gas was sampled as it entered and as it left the treater, the fume content being caught in heated filters and the moisture content being condensed in weighed flasks. The following data were obtained over a period of 50 minutes.

Data of gas sampled on entering and on leaving the treater.

	Entering treater.	Leaving treater.
Average temperature	117° C.	48° C.
Average velocity	7.33 feet per second.	6.11 feet per second.
Average volume	541 cubic feet per minute	495 cubic feet per minute.
Average draft	0.009 inch water	0.008 inch water.
Solids in gas, grains	0.661 per cubic foot	0.00101 per cubic foot.
Solids in gas, pounds	3.15 per hour	0.00428 per hour.
Cl in gas, grains	0.00	0.00235 per cubic foot.
HCl in gas, grains	0.34 per cubic foot	0.056 per cubic foot.

Approximately 50 cubic feet of air a minute leaked in between the two points of measurement. The treater efficiency as determined from the fume content of the gases was 99.87 per cent.

ONTARIO SILVER MINING CO. OXIDIZED ORE.

The ore used for this test from Park City, Utah, assayed as follows:

Assay of silver ore from Ontario Mining Co.

Ag	ounces	7.78	Fe	per cent	3.17
Pb	per cent	0.75	CaO	do	0.33
Zn		Trace.	S	do	1.42
Au		Trace.	Insoluble	do	83.80

Results of several experiments indicated that, in order to attain satisfactory recoveries, crushing to –16-mesh size was necessary. The reagents for volatilization had also been determined as well as the temperature of the furnace and the time of treatment required. Data were taken on gases for volume, velocity, and total solids. These results are not tabulated as they are similar in all experiments; a typical case is described in some detail on page 46.

Proportions of charge used in testing silver ore from Ontario Silver Mining Co.

Charge.	Pounds
Crude ore crushed to –16 mesh	200
Calcium chloride	6
Salt	12
Limestone	12
Sulphur	2
Total mixed charge	232

The test was made in the semicommercial plant, and all products were caught, weighed, and analyzed as shown in the tabulated form. The temperature of kiln was kept as near 1,000° C. as possible, and the speed of the kiln was one revolution a minute. Limestone was added to prevent fusion of the calcine in the furnace, and sulphur was added to increase the percentage of silver volatilization. Comparative tests indicated equally high extractions without sulphur. Elemental sulphur was added and was probably burned off before reaching the hottest part of the furnace.

TABLE 3.—*Summary of volatilization test of silver ore from Ontario Silver Mining Co., Park City, Utah.*

Material.	Weight.		Assay.		Percentage of original crude ore content.	
	Pounds.	Per cent.	Ag.	Pb.	Ag.	Pb.
			Ounces.	*Per cent.*		
Original crude ore	200.0	100.0	7.78	0.75	100.0	100.0
Calcines	188.0	94.0	1.34	.12	16.2	14.8
Flue dusts	14.5	7.2	10.00	.25	9.3	6.1
Fume	10.5	5.2	108.7	11.14	76.0	78.0
Total	[a]213.0	106.2			101.5	98.9

[a] Increase in weight accounted for by the addition of limestone on the charge.

This test was one of 12 or more made on the ore from the Ontario Silver Mining Co.'s mine and was as near an average as it is possible to present. As shown in Table 3, the silver and lead actually recovered in the fume was 76 and 78 per cent, respectively. The flue dust contained 9.3 per cent of the silver and 6.1 per cent of the lead. This dust from several tests was accumulated, dampened, and re-treated in the furnace, with the result that 75 per cent of the silver and 80 per cent of the lead was recovered. If these extractions were applied to the test tabulated above, the actual recoveries would be 83 per cent of the silver and 82.8 per cent of the lead.

SILVER REEF ORE.

Several lots of Silver Reef ore were received, including a number of small samples for small-scale tests and about a ton of ore for the large-scale tests representing a large deposit in Washington County, southern Utah. The assays of the small samples varied from 4 to 22.5 ounces silver a ton and 0.10 to 2.5 per cent copper; the assay of the large sample showed approximately 10 ounces silver and 1.4 per cent copper.

Silver Reef ore is an imperfectly consolidated sandstone composed of rounded and angular grains of sand with a small amount of mica and garnet. Crushing for treatment was not difficult.

Part of the ore was crushed to minus 48 mesh and carefully sized; the 100, 150, and 200 mesh products were concentrated by panning with a horn spoon. Examination of these concentrates under the microscope by R. E. Head established the following facts:

The minerals identified were native silver, argentite, malachite, chalcocite, and pyrite.

SILVER IN SILVER REEF ORE.

Silver occurs in this ore in the form of native silver, but mainly as the sulphide, argentite, which carries practically all of the silver present in the ore. The native element was found and identified by microchemical tests, but was negligible. When examined under the microscope, iron-stained nodules selected from sample 4 gave indications of being high in silver and copper. A picked sample of this material assayed 100.61 ounces of silver.

COPPER IN SILVER REEF ORE.

The copper in the sample examined was in the form of the carbonate, malachite, and the sulphide, chalcocite. The relative amounts of carbonate and sulphide were estimated, the form of occurrence being governed to some extent, no doubt, by the degree of exposure to oxidizing influences. In sample 22 practically all of the copper was in the carbonate form, whereas in sample 4 approximately 40 per cent of the copper was in the sulphide form. Hence any combination of carbonate and sulphide may possibly occur, ranging from all carbonate to all sulphide.

IRON IN SILVER REEF ORE.

In sample 4, pyrite occurred in sufficient quantity to warrant its presence being noted with the other minerals. Its percentage ran very low.

TABLE 4.—*Preliminary muffle tests of Silver Reef ore.*

Test No.	Weight of ore.	FeS_2.	NaCl.	Time.	Temperature.	Loss in weight.	Heads, Ag.	Cu.	Calcines.		Metal volatilized.	
									Ag.	Cu.	Ag.	Cu.
	Grams.	*Per ct.*	*Per ct.*	*Minutes.*	°*C.*	*Per ct.*	*Ounces.*	*Per ct.*	*Oz.*	*Per ct.*	*Per ct.*	*Per ct.*
2	100	2	5	60	933	5.0	8.1	0.80	1.9	0.07	77.7	91.7
3	100	4	5	60	933	5.0	8.1	.80	2.0	.10	76.8	88.3
4	100	....	5	75	919	4.5	8.1	.80	2.0	.15	76.4	82.5
5	100	....	5	75	919	3.0	8.1	.80	2.8	.20	66.4	75.7
8	100	4	10	60	916	5.0	7.6	.77	1.9	.05	76.3	93.7
9	100	....	10	90	900	3.0	7.0	.82	1.6	.10	78.0	88.2
10	100	....	10	90	900	2.0	7.0	.82	1.3	.07	82.0	91.7
11	100	....	10	120	945	1.5	7.0	.82	1.5	.15	79.7	83.4
			$CaCl_2$.									
12	100	....	10	90	965	5.0	7.0	.82	3.6	.12	51.2	86.1
13	100	....	10	90	965	5.0	7.0	.82	2.0	.10	72.8	88.4
14	100	....	10	120	945	2.0	7.0	.82	.9	.10	87.4	88.0
15	100	....	10	120	945	1.5	7.0	.82	.9	.05	87.3	95.5

Test 13 was similar to test 12 except that half of the calcium chloride was added at the beginning of the roast, and the remaining 5 per cent after roasting 30 minutes. This double roasting gave better silver extraction, although the amount of calcium chloride used was the same as in test 12, in which all the reagent was added at one time. The only feasible way of doing this double roast in commercial practice would seem to be by using two kilns or furnaces in series.

Test 14 was roasted for the same time and at the same temperatures as test 11, but the pyrite and half the salt was added at the beginning, and the remaining 5 per cent salt after 30 minutes roasting. Comparison with tests 10 and 11 shows that double roasting raised the extraction of the silver.

Test 15 was roasted for the same time and at the same temperature as test 14, and with the same quantity of salt and pyrite, but half of both the salt and pyrite was added at the beginning of the roast, and the remainder after 30 minutes roasting.

TABLE 5.—*Data of tests of Silver Reef ore.*

[Experiments conducted in 20-foot kiln.]

Test No.	Weight of ore.	Reagents.			Mesh ore.	Time.	Temperature.	Assay.				Metal volatilized.	
								Heads.		Calcines.			
		NaCl.	CaO.	S.				Ag.	Cu.	Ag.	Cu.	Ag.	Cu.
	Pounds.	*Per ct.*	*Per ct.*	*Per ct.*		*Minutes.*	*° C.*	*Ozs.*	*Per ct.*	*Ozs.*	*Per ct.*	*Per ct.*	*Per ct.*
1......	200	4	2	....	28	80	1,102	10.63	2.15	2.42	0.52	77.7	75.7
2......	200	5	2.5	....	20	80	1,107	9.68	1.97	2.96	.36	69.5	81.7
3......	200	4	2	....	28	90	1,005	9.54	2.20	1.90	.51	80.2	76.5
4......	250	8	0	2	28	90	1,050	9.88	1.40	2.28	.26	77.0	81.7
5......	240	8	2.4	2	28	90	1,050	10.34	1.30	1.64	.25	84.2	80.6

When the above experiments were conducted, the treaters were not working properly and no record was made of the precipitation of the fumes. Hence a series of tests was also made to determine the efficiency of the treaters in collecting silver from furnace gases and to check previous results. The general character of ore has been described (p. 49), assaying as follows: Silver, 7.48 ounces; copper, 0.73 per cent; and mercury, 0.045 per cent. The charges consisted of 250 pounds of ore mixed with 8 per cent salt and 2.4 per cent lime. To one charge 6.5 per cent pyrite was added, and to the other 2 per cent sulphur. The temperature was maintained at about 1,050° C. In the last 5 feet, or the enlarged part of the kiln, the ore remained in this heated zone for about one hour. The speed of the kiln was one revolution a minute, requiring about two hours for the passage of ore through the kiln. The volume of air passing through the kiln was reduced to exactly enough to carry away the fumes as fast as they formed.

Tables 6 and 7 show the weights of reagents and products, the assays of products, and the distribution of values.

TABLE 6.—*Results of first treater test of Silver Reef ore.*

	Weight.	Reagents.				Assays.			Crude metal content		
		NaCl.	S.	Pyrite.	Lime.	Ag.	Cu.	Hg.	Ag.	Cu.	Hg.
Heads................	*Lbs.* 250	*P.ct.* 8	*P.ct.* 0	*P.ct.* 6.5	*P.ct.* 2.4	*Oz.* 7.48	*P.ct.* 0.73	*P.ct.* 0.045	*P.ct.* 100.0	*P.ct.* 100.0	*P.ct.* 100.0
Calcine discharge.....	17					5.38	.22	.015	3.1	2.0	2.
Do................	108.5					1.80	.10	.020	10.4	6.0	19.
Calcine in kiln.........	73					2.24	.22	.010	8.7	8.8	6.5
Dust chamber.........	36					5.20	.43	.020	10.0	8.5	6.4
Flue dust.............	9.5					6.50	.53	.025	3.7	2.7	2.1
Fume.................	17.5					62.3	5.60	.190	58.3	53.7	29.5
Loss.................	....					......	......	......	5.8	18.3	34.0
Metals volatilized (by difference)...........	....					......	......	......	64.1	72.0	63.5

TABLE 7.—*Results of second treater test of Silver Reef ore.*

	Weight.	Reagents.				Assays.			Crude metal content.		
		NaCl.	S.	Pyrite.	Lime.	Ag.	Cu.	Hg.	Ag.	Cu.	Hg.
	Lbs.	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*	*Oz.*	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*	*P.ct.*
Heads	250	8	2	0	2.4	7.48	0.73	0.045	100.0	100.0	100.0
Calcine discharge	19					1.50	.22	.015	1.7	2.3	2.6
Do	98.5					1.20	.10	.012	6.3	5.4	10.5
Calcine in kiln	72					2.04	.32	.010	7.8	12.6	9.5
Dust chamber	30.5					5.46	.53	.017	8.9	8.8	4.6
Flue dust	9.5					6.38	.73	.022	3.2	3.8	31.9
Fume	......					58.15	5.60	.220	62.2	61.3	39.1
Loss	......					......	......	......	9.9	5.8	31.8
Metals volatilized (by difference)	......					......	......	......	72.1	67.1	70.9

In these experiments the amount of silver and copper shown to be volatilized was greater than the amount shown as recovered in the fume. This difference was due to difficulty in thoroughly cleaning the treaters. Most experiments balance closely in this respect, whereas others in the calculation of a metallurgical balance show a slight increase in metals. Comparison of tests 1 and 2 shows that the addition of pyrite in test 1 had no effect on the percentage of metals volatilized; in fact, test 2 showed a higher percentage of the silver volatilized. The effect of elemental sulphur was not noted, as the authors held the opinion that it burned off before reaching the hottest part of the kiln, where it would influence reactions affecting volatilization.

VOLATILIZATION OF LEAD.

Lead is usually the most easily volatilized of all the metals thus far investigated. It forms a volatile chloride more easily than any other metal by the chloride volatilization process.

Tests were tabulated of two typical ores containing practically no valuable metals except lead. Practically all the lead was volatilized from the ore, as shown in Tables 8 and 9.

Lead has been present in nearly every kind of ore treated by volatilization at the Intermountain station and in nearly every test its recovery or extraction has been higher than that of any other mineral constituent. These tests are included under complex ores and silver-lead ores (pp. 57–63).

ELECTRIC POINT OXIDIZED LEAD ORE.

The sample of this ore was furnished by the Electric Point Mining Co., Northport, Wash., and assayed as follows:

Assay of lead ore from Electric Point Mining Co.

Ag	Trace.	CaO .... per cent..	1.96
Pb .... per cent..	15.15	S .... do....	.12
Zn .... do....	1.84	Insoluble .... do....	7.89
Cu .... do....	0.0	MgO	Trace.
Fe .... do....	37.60		

This ore, which represented a large deposit, was not high grade enough to ship directly to smelting plants. As lead was present as a carbonate, gravity concentration would recover only part of the value—approximately 60 to 70 per cent under the best conditions.

The data tabulated resulted from muffle tests. As previous work on the more complex ores indicated that muffle tests and small-scale experiments showed what could be done in the larger plants, no large-scale test was made.

The material treated was crushed to −30 mesh and heated in the muffle at a temperature of approximately 950° C. Table 8 gives the amount of reagents used, the time of heating, the temperature, the weight of calcines, and the percentage of extraction in eight experiments.

TABLE 8.—*Data resulting from eight experiments with Washington lead ore.*

Test No.	Weight of ore.	Reagents.		Time.	Temperature.	Weight calcines.	Pb in calcine.	Pb extracted.
		$CaCl_2$.	NaCl.					
	Grams.	*Grams.*	*Grams.*	*Hours.*	°. *C.*	*Grams.*	*Per cent.*	*Per cent.*
1	100	8.1	....	1½	950	71.5	1.92	90.3
2	100	12.2	....	1½	950	72.9	.22	98.4
3	100	....	8.6	1½	950	76.5	8.54	57.2
4	100	....	12.9	1½	950	73.7	6.34	69.2
5	100	6.6	1.7	1½	950	72.9	2.70	87.0
6	100	6.6	6.0	1½	950	71.1	.85	96.0
7	100	12.0	3.0	1½	950	73.9	Trace.	99.9
8	100	12.0	5.0	1½	950	72.7	Trace.	99.9

These eight tests showed that from 57 to 99.9 per cent of the lead was volatilized from the ore, and indicated also that calcium chloride was a better agent than salt for the volatilization of lead.

GLADSTONE MOUNTAIN ORE.

Gladstone Mountain ore is a typical oxidized lead ore assaying as follows:

Assay of Gladstone Mountain lead ore.

Pb....................per cent..	17.6	Insoluble...............per cent..	64.8
Fe.......................do....	7.0	Cu, Zn, Au, Ag, Mn, and CaO......	Nil.

Tests with Gladstone Mountain ore were conducted in the muffle furnace for one hour, with furnace temperature from 850° to 930° C., on 100-gram samples of the ore crushed to −16-mesh size. Table 9 gives the results of three tests.

TABLE 9.—*Data resulting from three muffle tests with Gladstone Mountain lead ore.*

Test No.	Reagent.		Weight of calcine.	Pb in calcine.	Pb extracted.
	NaCl.	$CaCo_3$			
	Per cent.	*Per cent.*	*Grams.*	*Per cent.*	*Per cent.*
1	12.5	....	83	0.52	97.5
2	12.5	10.0	89	Trace.	99.9
3	15.0	....	84	0.33	98.4

In the tests where salt alone was used the charge slagged, which was prevented in test 2 by the addition of 10 per cent of limestone. A slightly higher recovery resulted. Other tests duplicated the results tabulated.

VOLATILIZATION OF COPPER.

Copper volatilizes readily in the chloride volatilization process. Copper forms with chlorine two salts—cuprous chloride (Cu_2Cl_2) and cupric chloride ($CuCl_2$). Cuprous chloride is the more stable at elevated temperatures. The chlorides, even at temperatures below redness, are volatile. When heated the cupric salt changes to cuprous, and volatilizes as cuprous salt. In an atmosphere of hydrochloric acid, however, the fumes of cuprous chloride may be partly raised to the cupric form, hence collected fumes are usually mixtures of the two chlorides. Oxychlorides of copper may take some part in these changes but, according to tests on collected copper chloride fumes, not an important one.

The reactions between sodium or calcium chloride and the oxidized ores of copper are rapid at moderate temperatures; as extremes of neither time nor temperature are required, the volatilization process for copper from ores is desirable commercially. The percentage of copper in the fume runs high, and the reduction of the chloride fume to metal by fusing it with lime and coke affords a simple method of treatment.

The presence of some sulphur in copper ores probably helps rather than hinders the chloridizing reactions. On the other hand, if the proportion of sulphide is large, copper sulphate occurs in the fume; when reduced, such fume gives considerable copper matte along with metallic copper. Little iron is volatilized, and the matte occurring with the metal runs high in copper.

POPE SHENON COPPER ORES.

The ores in the Pope Shenon mine, Salmon, Idaho, especially those in the upper level, comprise a mixture of oxides, carbonates, and sulphides, the first two predominating. No valuable metals except copper exist in these ores, and methods of concentration and flotation have not proved amenable for its recovery.

The Salt Lake station received for experimental purposes several lots of the Pope Shenon ore, with which preliminary experiments were conducted in muffle and electric-tube furnaces. Almost a complete extraction of the copper was indicated, especially when calcium chloride served as the volatilization reagent.

After the preliminary experiments many tests were made in the semicommercial plant, which was equipped with a 20-foot rotary kiln and Cottrell treaters to recover the fumes. Table 10 shows the results of two representative experiments. In test 2 grab samples taken at different intervals showed that the calcines being discharged assayed 0.30, 0.40, 0.45, and 0.66 per cent copper, though the average calcine discharged assayed 0.84 per cent copper, as indicated in Table 9. Material remaining in the kiln raised materially the grade of the discharged calcine.

In fact, the total recovery of copper in the form of fume would have been much higher had not some calcines that were higher in copper content than the discharged calcine remained in the kiln.

The following data (Tables 10 and 11) show the weights and assays of the products, together with the percentage of recovery or loss of the copper content:

TABLE 10.—*Data of volatilization test 1 of Idaho copper ore.*

CHARGE FOR TEST 1.

Charge of ore, –16 mesh .... pounds..	75
Calcium chloride .... do....	14
Time in furnace .... hour..	1
Temperature .... °C..	900

RESULTS OF TEST 1.

Material.	Weight.		Assay.	Recovery or loss.
	Pounds.	Per cent.	Cu.	Total Cu.
			Per cent.	*Per cent.*
Crude ore	75.0	100.0	7.65	100.0
Calcine	76.5	102.0	.675	9.0
Fume	12.0	....	41.10	86.0
Total	[a] 88.5	....	....	95.0

[a] Increase in weight was due to addition of reagent.

TABLE 11.—*Data of volatilization test 2 of Idaho copper ore.*

CHARGE FOR TEST 2.

	Pounds
Charge of ore, –16 mesh	330.0
Calcium chloride	33.0
Salt	16.5
Total charge	379.5

RESULTS OF TEST 2.

Material.	Weight.		Assay.		Recovery or loss.
	Pounds.	Per cent.	Cu.	Cl.	Total Cu.
			Per cent.	*Per cent.*	*Per cent.*
Crude ore	330.0	100.0	4.87	....	100.0
Calcine	322.5	97.7	.84	0.12	16.8
Fume	39.1	11.7	34.85	20.55	84.0
Total	[a] 361.6	109.4	....	....	100.8

[a] Increase in weight was due to addition of salt and calcium chloride.

The following table gives data on the solid matter in the ore:

Data on solid matter in Pope Shenon copper ore.

Average velocity in flue, feet per second		8.36
Average volume in flue, cubic feet per minute		617
Solid matter per cubic foot	grains	3.34
Solid matter per hour	pounds	17.6
Free chlorine per cubic foot	grains	.003
Free chlorine per hour	pounds	.064
Hydrochloric acid per cubic foot	grains	.73
Hydrochloric acid per hour	pounds	3.86

VOLATILIZATION OF ZINC ORES.

Behavior of zinc in ores treated by the volatilization process depends largely on the arrangement of the mineral in the ore, also on the atmosphere that is maintained in the furnace during treatment. Experiments have indicated that in a strictly oxidizing atmosphere little zinc is volatilized, especially in the zinc carbonate ores of the Goodsprings, Nev., district.

The tests of the Yellow Pine Mining Co.'s ores and of other ores from the Goodsprings district were made on zinc concentrates in which the zinc content ranged from 30 to 40 per cent, the silver and lead content varying from 4 to 8 ounces and from 4 to 8 per cent, respectively. The quantity of silver and lead volatilized ranged from about 50 to 90 per cent, whereas the total amount of zinc volatilized generally averaged less than 1 per cent.

Analysis of the fume precipitated in volatilization tests on these ores indicated high assays in silver and lead and 3 to 6 per cent in the zinc assay.

BEHAVIOR OF ZINC IN COMPLEX ORES.

The behavior of zinc is very erratic, the conditions under which it is volatilized probably depending largely on the sulphur content in the ore. The presence of sulphur causes a complicated series of chemical reactions during the usual treatment, consequently a variable quantity of the zinc is driven off with other volatile materials. When crude ores high in sulphur content have been treated, most experiments indicated nearly complete volatilization of the zinc, but when the ore was given a preliminary roast to reduce the sulphur content, the amount of zinc volatilized varied considerably.

EFFECT OF VOLATILIZATION OF ZINC IN COMPLEX ORES.

The presence of zinc in carbonate ores containing silver, lead, and zinc indicates that by volatilization a clean-cut separation can be made between the lead and zinc. On the other hand, in treating complex sulphide ores much of the zinc was volatilized in nearly every experiment and was subsequently precipitated in the Cottrell treaters as chloride with the other metal chlorides.

FUME PRODUCTS.

The precipitated fume from complex sulphide ores requires special hydrometallurgical methods to separate the silver and lead from the zinc. The fume is nearly as complex as the original ore, and if hydrometallurgical methods were required to effect separation probably the zinc should be leached from the roasted ores and the leached residues treated by volatilization for the recovery of the silver and lead.

VOLATILIZATION OF THE MORE COMPLEX ORES.

LEAD-SILVER OXIDIZED ORES.

Most of the ores tested by the volatilization process contained more than one valuable mineral, usually a combination of silver, lead, copper, and zinc. For a description of the silver-lead-zinc ores, see pages 75 to 81. The more typical ores—representing silver, lead, and some copper—were those of the Tintic Standard Mining Co., Eureka, Utah, the Ruby Hill Development Co., Eureka, Nev., and the Nevada United Mines Leasing Co., East Ely, Nev.

Many experiments were conducted on somewhat similar ores, but as these were representative of large tonnages in the districts named and were carbonates, hence not amenable to concentration methods, they received special attention. The tabulated results show the assays of the material treated, the calcines produced, and the recoveries of each mineral constituent of the ores.

SILVER-LEAD ORE FROM EUREKA, UTAH.

The sample tested was an oxidized ore from the Tintic Standard Mining Co., Eureka, Utah, and assayed as follows:

Assay of Tintic Standard ore.

Ag	ounces	15.6
Pb	per cent	7.33
CaO	do	3.7

The test was made in the semicommercial plant, and all products were caught, weighed, sampled, and assayed as shown in Table 12. The temperature was kept as near 1,000° C. as possible, and the rate of feeding the ore into the furnace was 100 pounds per hour.

TABLE 12.—*Data of volatilization test of complex ores from Eureka, Utah.*

CHARGE PER TEST, IN POUNDS.

Ore crushed to −16 mesh	500
Calcium chloride	25
Limestone	50
Salt	25
Total charge	600

RESULTS OF TESTS.

Material.	Weight.		Assay.		Percentage crude ore content.	
	Pounds.	Per cent.	Ag.	Pb.	Ag.	Pb.
			Ounces.	*Per cent.*		
Crude ore	500	100.0	15.60	7.33	100.0	100.0
Calcine	425	85.0	2.87	.20	15.6	2.3
Fume	104	20.8	62.40	34.42	83.0	97.4
Total	529	105.8			98.6	99.7

Actual recoveries in fume show 83 per cent of the silver and 97.4 per cent of the lead. The addition of limestone was necessary to prevent the ore from fusing in the furnace, and accounts for the excess in weight in the totals over the original crude ore treated.

GOLD-SILVER-LEAD ORE FROM EUREKA, NEV.

A series of tests was made by E. P. Barrett to determine the possibility of using chloride volatilization to recover the gold, silver, and lead values in ore of the Ruby Hill Development Co., Eureka, Nev.; 100-gram charges of ore crushed to −20 mesh were mixed with the chloridizing reagents and treated in a muffle furnace.

The assay of the original ore was as follows:

Assay of complex ores of Ruby Hill Development Co.

Ag	ounces	7.66	Fe	per cent	30.00
Au	do	.34	S	do	.45
Pb	per cent	5.30	CaO	do	10.40
Cu	do	.29			

Table 13 gives the charges, time of heating, temperature, assays, and extractions of the tests.

TABLE 13.—*Results of volatilization tests of complex ores of Ruby Hill Development Co.*

Test No.	Weight of ore.	Reagents.		Time.	Temperature.	Calcine.	Calcine assays.					Percentage extraction.		
		NaCl.	CaCl$_2$.				Au.	Ag.	Pb.	Fe.	Cl.	Au.	Ag.	Pb.
	Grams.	*Grams.*	*Grams.*	*Hours.*	°*C.*	*Grams.*								
1	100	6		1	950	75		0.70	1.36	37.7	0.61		94.1	80.7
2	100	3	3	1	950	76		.16		40.0	.81		98.4	100.0
3	100		6	1	950	77		.36		38.0	1.55		96.5	100.0
4	100	6		2	950	74		Trace.	.67	38.7	.19		100.0	90.3
5	100		6	2	950	72		None.		37.3	.39		100.0	100.0
6	100	3	3	2	950	72	Trace.	Trace.		36.2	.42	100.0	100.0	100.0
7	100	6		½	1,000	74		Trace.	1.10	37.3	.32		100.0	84.6
8[a]	100	3	3	½	1,000	72	Trace.	Trace.	.60	37.3	.19	100.0	100.0	91.8
9[a]	100		6	½	1,000	75		.16		37.3	.51		98.6	100.0

[a] Tests 8 and 9 sintered somewhat at 1,000° C. temperature.

These tests indicated that a charge mixed with 3 per cent salt and 3 per cent calcium chloride give almost complete extractions of the lead, gold, and silver values, if heated to 950° C. for about one and one-half hours; if the temperature were increased, the same extractions would be obtained in less than an hour.

SILVER-LEAD ORE FROM EAST ELY, NEV.

A 100-pound sample of oxidized lead ore from the Nevada-United Mines Leasing Co., East Ely, Nev., was analyzed, a proportionate part being crushed through 30 mesh for volatilization in the muffle furnace. The analysis of the ore was as follows:

Analysis of lead ore from East Ely, Nev.

Au		Trace.	Fe	per cent	43.25
Ag	ounces	2.72	CaO		Trace.
Pb	per cent	10.12	MgO		Trace.
Cu	do	.11	S	per cent	1.08
Zn	do	.79	Insoluble	do	10.64

The quantity of salt necessary to combine with all of the lead in the ore as lead chloride was 5.7 per cent, and the amount of calcium chloride required to accomplish the same purpose was 5.4 per cent of the weight of the ore. The charges used were determined on the basis of simple fractions of the amounts of chloridizing agent theoretically necessary. For instance, charge 1, using one and one-fourth times the amount of chloridizing agent required for the lead, was a mixture of three-fourths the theoretical part of $CaCl_2$, and one-half the theoretical part of NaCl. The fractions of the theoretical quantities of reagents used are determined easily by inspection of the weights of the reagents used.

The first two charges were made with a 25 per cent excess of reagent, the next two with a 50 per cent, and the last two with a 75 per cent excess of reagent. In each pair of tests—1 and 2, 3 and 4, 5 and 6—the volatilization proved to be best for both the lead and silver in the test having the highest ratio of salt to calcium chloride. In other words, the use of salt in place of calcium chloride seems advantageous. It is doubtful, however, whether a charge of salt alone would be as good as mixtures, because salt by itself does not chloridize lead as readily or as thoroughly as does calcium chloride. The ore had not enough silver to make that metal as important as the lead. Mechanical action of the lead chloride vapor in sweeping out with it the vapor of silver chloride probably accounts for the silver extraction following the extraction of lead so closely. Hence a complete extraction of the lead, as in test 5, would also represent the best extraction of the silver obtainable.

TABLE 14.—*Data on volatilization tests of silver-lead ore from East Ely, Nev.*

Test No.	Weight of ore.	Reagents.		Weight of calcine.	Calcine.		Extraction.	
		NaCl.	$CaCl_2$.		Ag.	Pb.	Ag.	Pb.
	Grams.	*Grams.*	*Grams.*	*Grams.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	100	2.9	4.1	79.7	3.34	1.78	2.0	86.0
2	100	1.4	5.4	79.8	2.78	2.60	18.4	79.5
3	100	2.9	5.4	79.4	1.28	.59	62.6	95.4
4	100	1.4	6.8	80.2	1.54	1.78	54.5	85.9
5	100	2.9	6.8	79.3	.90	Trace.	73.8	99.9
6	100	1.5	8.1	79.1	1.22	.84	64.5	93.4

EXPERIMENTS USING ARTIFICIAL SILVER-LEAD ORES.

By L. G. Gerhardt.[30]

A series of experiments was made on a mixture of Gladstone Mountain and O. K. silver ores to study the factors controlling the extraction of lead and silver from ores. The O. K. silver ore used contained 26 ounces of silver per ton with only a trace of lead. The Gladstone Mountain ore contained 8 per cent lead with a trace of silver. By using different amounts of each ore a mixture containing varying amounts of silver and lead could be obtained. Forty-one 100-gram charges were mixed containing the amounts of silver and lead recorded in Table 15. As 100 grams of the ore were used, these figures can be read as percentages of lead and ounces of silver per ton.

The roasts were made in an assay muffle. All charges were roasted for one and one-half hours at a temperature of 850° to 950° C. Varying amounts of pyrite were added to the charges to study the effect upon the extractions of the metals. The pyrite used contained 44.5 per cent Fe and 50.9 per cent sulphur. One and one-quarter equivalents of salt was added to each roast with a minimum of 2.5 per cent salt.

One of the chief reasons for conducting these tests with varying amounts of lead and silver was to observe the extraction of the silver. In the old chlorination roasting process the loss of silver by volatilization was said to be roughly proportional to the volume of the fume of base metal, the silver being carried away mechanically by the fume. Data of tests 1 to 5 show that just the reverse is true.

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[30] Gerhardt, L. G., graduate thesis, 1918, University of Utah, Salt Lake City, Utah.

TABLE 15.—*Data of tests of artificial ores with varying quantities of lead and silver.*

Test No.	NaCl.	Pyrite (44.5 per cent Fe).	Assay of mixture.		Metal volatilized.	
			Pb.	Ag.	Pb.	Ag.
	Per cent.		*Per cent.*	*Ounces.*	*Per cent.*	*Per cent.*
1	6.7	....	8.0	Trace.	85.0	....
2	5.9	....	6.0	6.5	94.0	45.7
3	4.4	....	4.0	13.0	96.2	44.2
4	2.5	....	2.0	19.5	92.0	62.0
5	2.5	....	0.0	26.1	....	76.3
6	2.5	2.5	Trace.	26.1	....	25.3
7	2.5	5.0	Trace.	26.1	....	21.0
8	2.5	10.0	Trace.	26.1	....	15.3
9	2.5	20.0	Trace.	26.1	....	18.8
10	5.9	2.5	2.0	19.5	99.9	12.8
11	5.9	5.0	2.0	19.5	99.9	88.2
12	5.9	10.0	2.0	19.5	91.0	20.0
13	5.9	20.0	2.0	19.5	25.0	35.9
14	4.4	2.5	4.0	13.0	90.4	16.9
15	4.4	5.0	4.0	13.0	51.3	....
16	4.4	10.0	4.0	13.1	55.5	4.5
17	4.4	20.0	4.0	13.1	34.4	19.2
18	5.9	2.5	6.0	6.5	56.6	15.4
19	5.9	5.0	6.0	6.5	50.9	26.1
20	5.9	10.0	6.0	6.5	25.0	13.2
21	5.9	20.0	6.0	6.5	25.0	13.2
22	6.7	2.5	8.0	Trace.	61.8	....
23	6.7	5.0	8.0	Trace.	43.7	....
24	6.7	10.0	8.0	Trace.	42.5	....
25	6.7	20.0	8.0	Trace.	20.0	....
26	2.5	2.5	2.0	19.5	49.0	86.3
27	2.5	5.0	2.0	19.5	51.0	29.2
28	2.5	10.0	2.0	19.5	23.0	16.9
29	2.5	20.0	2.0	19.5	15.0	17.9
30	2.5	2.5	2.0	21.9	40.0	24.2
31	5.0	2.5	2.0	21.9	84.5	15.1
32	10.0	2.5	2.0	21.9	73.0	4.0
33	15.0	2.5	2.0	21.9	72.5	0.0
34	5.0	2.5	2.0	21.9	99.9	77.6
35	5.0	5.0	2.0	21.9	99.9	51.2
36	5.0	10.0	2.0	21.9	79.0	32.4
37	5.0	15.0	2.0	21.9	61.5	13.7
38	2.5	2.5	2.0	21.9	99.9	73.5
39	5.0	5.0	2.0	21.9	99.0	59.9
40	10.0	10.0	2.0	21.9	99.0	49.8
41	15.0	15.0	2.0	21.9	99.0	43.4

In comparing these figures it should be remembered that the tests were run in batches and. therefore, compare much better within batches than between batches. An assay muffle is so difficult to bring to a desired temperature, and to hold it there constantly, that discrepancies in the results are unavoidable. Had the tests been carried out in an electrically heated muffle the difference would not have been as great. Keeping this in mind, two sets of curves have been drawn to show the effect of increasing additions of pyrite on the chloridizing volatilization of lead and silver, respectively.

The curves showing the effect of pyrite on the silver chloridizing are given in Figure 3. Curve *a*, taken from tests 5 to 9, is for a mixture containing only silver, the ore carrying 26.1 ounces of silver a ton, and 2.5 per cent salt. It can be seen that the addition of only 2.5 per cent of pyrite caused a marked drop in the chloridizing efficiency of the salt for silver. Possibly this has some connection with the fact that the salt can be converted to sodium sulphate by the sulphur dioxide resulting from the pyrite; 1 part of pyrite will convert 1.6 parts of salt, assuming the reaction to be 100 per cent complete.

Enough pyrite was added to convert completely all the salt to sodium sulphate during the roasting. Quantities of pyrite over 2.5 per cent were greatly in excess of the amount necessary for this purpose. Though the old ideas on chloridizing roasting were based on the formation of a chloridizing atmosphere through the medium of the oxides

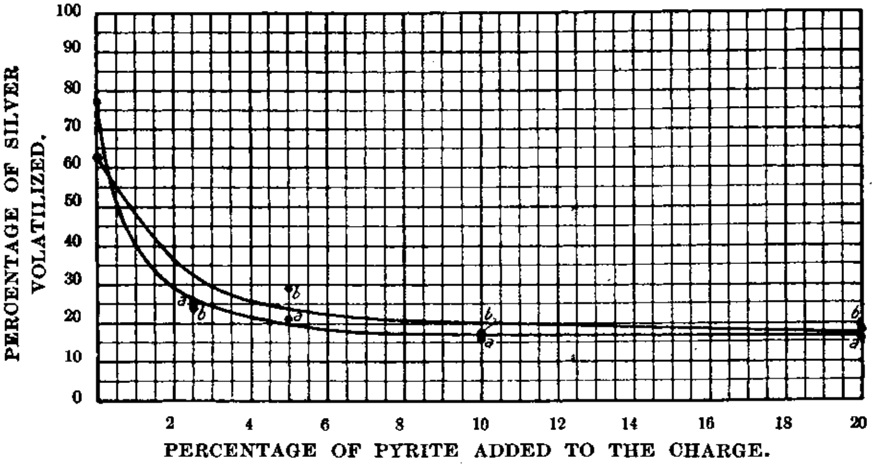


FIGURE 3.—Influence of pyrite on the chloride volatilization of silver: *a*, 2 per cent lead, 19.5 ounces silver to the ton; *b*, no lead present, 26.1 ounces silver.

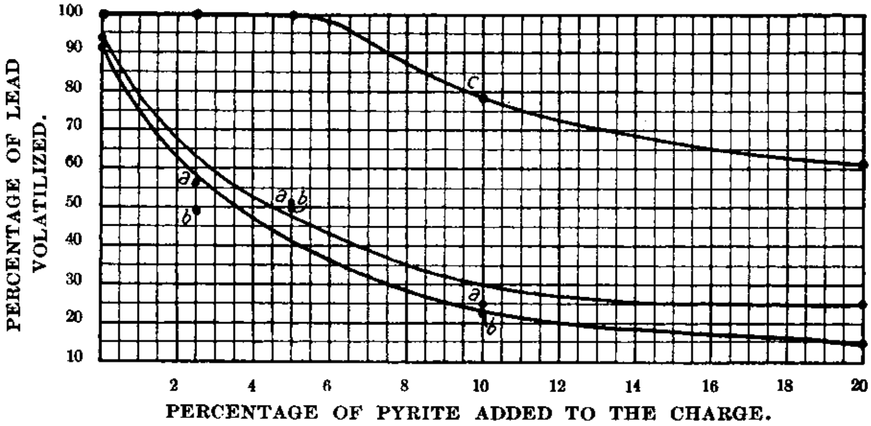


FIGURE 4.—Influence of pyrite on chloride volatilization of lead: *a*, 6 per cent lead, 5.9 per cent salt; *b*, 2 per cent lead, 2.5 per cent salt; *c*, 2 per cent lead, 5 per cent salt.

a is from tests 2, 18, 19, 20, and 21, Table 15.
b is from tests 4, 26, 27, 28, and 29, Table 15.
c is from tests 34, 35, 36, and 37, Table 15.

of sulphur resulting from the roasting of metal sulphides, the efficiency of chloride volatilization fell off with the addition of pyrite in the dry atmosphere of the muffle at the temperature range of 850° to 950° C. The reason for this decrease in efficiency is not known.

Curve *b* of Figure 3 is for tests run as nearly as possible under the same conditions as those in curve *a*, except that 2 per cent of lead was present in the ore mixture, the value of silver being reduced to

19.5 ounces a ton. Nearly always in the presence of the lead slightly less silver was volatilized than when no lead was present. The differences between curves *a* and *b*, however, are doubtless within the limits of error of the work. In both curves the chloridizing volatilization of the silver is visibly hindered by the presence of the pyrite in the charge. If the roasting had been carried on at lower temperatures with the object of forming soluble chlorides (but not allowing the temperature to rise to a point where they would volatilize) the claims of the old chloridizing roasting metallurgists would probably have been justified. Under our conditions, however, they are not.

Similar curves made for the lead contents of some of these roasting tests are shown in Figure 4. Curve *a*, prepared from tests 2, 18, 19, 20, and 21, was for an ore mixture containing 6 per cent lead and 5.9 per cent salt. Curve *b*, from tests 4, 26, 27, 28, and 29, was for an ore mixture containing 2 per cent lead and 2.5 per cent salt; and curve *c*, from points 34, 35, 36, and 37, was for an ore mixture with 2 per cent lead and 5 per cent salt.

The conclusions that can be drawn from these curves are: (1) The addition of pyrite to a charge lowers greatly the efficiency of chloridizing and volatilizing lead in the dry atmosphere of a muffle at temperatures between 850° and 950° C.; (2) this bad effect is much more pronounced when equal weights of salts and pyrite are proportionately greater than the lead; when the weight of salt is more than double the weight of the lead, much more pyrite must be added to cause a deleterious effect.

These tests illustrate one fundamental difference between conditions in the muffle tests and in the larger-scale tests in rotating kilns. In the muffle, the ore sample is put directly into a hot zone in dry air and requires only a short time to reach the temperature of the muffle. In the kiln the ore gradually rises in temperature as it progresses down the kiln, and it is also subjected to the action of products of the combustion of the fuel used for firing the kiln. Conditions in the older chloridizing roasting practice were more like those in the rotating kiln, and perhaps pyrite or other metallic sulphides can be satisfactorily chloridized at the lower roasting temperatures, to be followed by volatilization of these chlorides when the charge reaches the hotter parts of the kiln. These varying effects are to be investigated later.

OTHER OXIDIZED ORES.

LEAD-ZINC-SILVER ORE FROM KLONDYKE, ARIZ.

A 75-pound sample of ore from the Aravaipa Leasing Co., Klondyke, Ariz., was used for volatilization tests, which were made in the muffle furnace. This ore is a lead-zinc-silver combination with a small amount of copper in a quartz gangue.

After the ore was crushed through a ¼-inch mesh it was mixed thoroughly and quartered, one-quarter being put through a 30-mesh screen. Analysis of the ore is given below:

Analysis of lead-zinc-silver ore from Klondyke, Ariz.

Au	ounces	0.01	Fe	per cent	4.40
Ag	do	4.02	CaO	do	10.60
Pb	per cent	5.26	S	do	.99
Zn	do	2.24	Al_2O_3	do	9.30
Cu	do	.36	Insoluble	do	62.40

In making tests, a mixture of calcium chloride and salt was used for the chloridization. Three per cent salt was used in all tests, this being considered a large enough quantity to chloridize the silver. In the first four tests the amount of calcium chloride used was 150, 175, 200, and 250 per cent of that theoretically necessary for the chloridization of the lead. No account was taken of the reagent necessary for the zinc. In the subsequent tests, using various reagents, the charge of 200 per cent of the theoretical calcium chloride, together with salt equal to 3 per cent of the weight of the ore, was taken as a common factor, and the different reagents were added to this mixture. The tests were run for an hour and a half at a temperature of 900° C.

The results indicated that the process might be favorably applied to an ore such as the Aravaipa. A simple mixture of salt and calcium

TABLE 16.—*Data covering volatilization tests of lead-zinc-silver ore from Klondyke, Ariz.*

Weight of ore.	Reagents.		Other reagents.	Weight of calcine.	Analysis of calcines.			Metal volatilized.		
	$CaCl_2$.	NaCl.			Ag.	Pb.	Zn.	Ag.	Pb.	Zn.
Grams.	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
100	4.2	3		90.6	0.60	0.42	0.00	86.5	96.8	100
100	4.9	3		92.4	.40	.33	0.00	90.8	94.2	100
100	5.6	3		90.0	.30	.29	0.00	93.3	95.0	100
100	7.0	3		91.1	.35	.33	0.00	92.1	94.3	100
100	5.6	3	2 CaO	95.7	.35	.42	0.00	91.7	92.3	100
100	5.6	3	4 CaO	97.3	.35	42	0.00	91.5	92.2	100
100	5.6	3	6 CaO	98.5	.33	.42	0.00	91.9	93.8	100
100	5.6	3	8 CaO	98.3	.35	.42	0.00	91.4	92.1	100
100	5.6	3	1 S	94.0	.43	.42	0.00	89.9	92.5	100
100	5.6	3	2 S	95.0	.45	.42	0.00	89.4	92.4	100
100	5.6	3	3 S	94.5	.66	.29	0.00	84.5	94.8	100
100	5.6	3	1 S; 2 CaO	96.5	.37	.42	0.00	91.1	93.2	100
100	5.6	3	2 S; 4 CaO	99.0	.45	.38	0.00	88.9	92.8	100
100	5.6	3	3 S; 6 CaO	100.5	.35	.62	0.00	91.7	88.2	100
100	5.6	3	6 Niter	90.5	.35	.54	0.00	92.1	90.7	100
100	5.6	3	8 Niter	90.0	.51	.43	0.00	88.6	92.6	100
100	5.6	3	10 Niter	95.0	.45	.33	0.00	89.4	94.0	100
100	5.6	3	12 Niter	95.5	.45	.42	0.00	89.3	92.4	100

chloride allows extraction of more than 90 per cent of the lead and silver and also volatilizes all of the zinc.

A noteworthy feature of these tests was that almost every calcine was sintered. That sintering did not affect volatilization, as it usually does, was probably due to the fact that the ore sintered rather slowly, giving the metals plenty of time to chloridize and volatilize. Larger-scale tests would show whether good results could be obtained in spite of the tendency to sinter.

GOLD-SILVER-COPPER ORE FROM LUDLOW, CALIF.

The gold-silver-copper ore from Pacific mine, Ludlow, Calif., was a semioxidized material containing approximately 0.37 ounce gold, 1.4 ounces silver, and 1.3 per cent copper, in which the angular fragments of siliceous material were cemented together partly with iron oxide and partly by copper minerals deposited in the remaining interstices. The iron oxide varied in color from black to red. The copper was present in the form of the carbonates, malachite and azurite.

Experiments were made on about 2 tons of this ore in lots of 200 to 1,000 pounds. The final test showed that 84.3 per cent of the gold, 66.3 per cent of the silver, and 76.6 per cent of the copper was volatilized. These recoveries on the sample treated correspond to a tailing assaying 0.05 ounce gold, 0.50 ounce silver, and 0.31 per cent copper.

The charge was 8 per cent chloridizing reagent, consisting of 3 parts ($NaCl$) and 1 part ($CaCl_2$), and 3.4 per cent sulphur. The ore was ground to pass a 30-mesh screen, and the maximum temperature at which it was treated was about 1,000° C.

LEAD-VANADIUM ORE FROM GOODSPRINGS, NEV.

A test on lead-vanadium ore from Goodsprings, Nev., was made by E. P. Barrett to determine the possibility of using the chloride volatilization process as a means of separating the lead from the vanadium in this ore.

A charge of 100 grams of ore, crushed to −60 mesh and mixed with 10 per cent salt, was placed in a cold muffle, gradually heated to 1,000° C, and held at that temperature for about an hour. The charge showed a slight tendency to stick to the roasting dishes.

The results indicated that this process might be a means of separation of the lead and vanadium, as about 85 per cent of the lead and only 14 per cent of the vanadium volatilized. The grade of the vanadium was increased from 2.9 per cent in the heads to 4.9 per cent in the calcine.

Assay of products in percentages.

Product.	Pb.	V_2O_5.	CaO.	Fe.	Insoluble.
Heads	9.40	2.90	22.60	2.50	1.60
Calcine	2.80	4.90	39.50	4.68	3.20

Distribution of lead and vanadium.

Product.	Weight.	Assay, Pb.	Total Pb.	Assay, V_2O_5.	Total V_2O_5.
	Grams.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Heads	100	9.40	100.0	2.90	100.0
Calcine	52	2.80	15.5	4.90	86.2
Volatilized			84.5		13.8

SULPHIDE ORES.

The complex sulphide ores, especially of the zinc, were very erratic in behavior during volatilization treatment. The presence of sulphur increased the quantity of salt or other chloridizing reagent needed to volatilize the metals, and as sulphide ores slag or fuse badly they lower the efficiency of the furnace.

In sulphide ores lead seems to be the most easily volatilized metal, silver, copper, and gold following very closely. The quantity of zinc recovered ranged from a few to 100 per cent.

PREROASTING.

The general experience has been that ores containing sulphur in excess of 5 per cent required a preliminary roast in order to reduce the sulphur content, preferably to 1 or 2 per cent. Roasting of complex ores demanded extreme care and control of temperature. If zinc was present, part of the zinc sulphide was frequently converted to zinc, and sulphates of other metals were also formed.

Under conditions that probably could be maintained if thoroughly understood, the amount of zinc volatilized after preroasting of some ores, especially the Sunnyside ores, from Eureka, Colo., was as great as from crude ores. The results of experiments on several complex sulphide ores, both raw and roasted, are given below; the percentage of silver, lead, and copper volatilized was unusually high, the zinc being erratic.

SILVER-LEAD ORE FROM PARK CITY, UTAH.

The low-grade sulphur ore used in the following series of tests came from the lower workings of the Ontario Silver Mining Co.'s property, Park City, Utah. The analysis was as follows:

Analysis of low-grade sulphide ore from Park City, Utah.

Au	ounces	0.01	Fe	per cent	6.53
Ag	do	13.80	CaO	do	3.55
Pb	per cent	4.36	S	do	11.48
Zn	do	10.00	Insoluble	do	60.60
Cu	do	.38			

The ore was treated without a preliminary roast to eliminate the sulphur. The tests were conducted in a semicommercial laboratory plant, using the 20-foot kiln and the large Cottrell treaters. The speed of the kiln was one revolution per minute, requiring about two hours for the passage of the ore through the furnace. The temperature was maintained close to 1,050° C. in the hottest part of the furnace.

TABLE 17.—*Volatization of silver and lead from low-grade sulphur ore from Park City, Utah.*

CHARGE FOR TESTS, IN POUNDS.

Ore crushed, −16 mesh	854	
Salt	102.5	(12 per cent).
Calcium chloride	34.2	(4 per cent).
Lime	68.5	(8 per cent).
Total mix	1,059.2	

PRODUCTS AND ASSAYS.

	Weight.	Ag.	Pb.	Zn.	Insoluble.	Cl.
	Pounds.	*Ounces.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Crude ore treated	854	13.80	4.36	10.00	60.00	....
Discharged calcine	530	5.08	Trace.	3.98	....	0.40
Calcine in kiln	171	7.72	None.	7.15	....	....
Dust-chamber dust	26	12.12	4.58	7.65	....	....
Flue dust	5	12.95	5.84	7.25	....	....
Fume [a]	108	65.00	30.30	33.40	4.50	27.54
Total product [b]	840	....	....	....	....	....

[a] The fume carried also 1.22 per cent copper and 1.40 per cent iron.

[b] The loss in weight of the mix was 219.2 pounds, or 20.7 per cent. The loss in weight of ore was 14 pounds, or 1.6 per cent.

DISTRIBUTION OF METALS.

	Ag.		Pb.		Zn.	
	Units.	Per cent.	Units.	Per cent.	Units.	Per cent.
Crude ore treated	11,785.2	100.0	3,723	100.0	8,540	100.0
Calcine discharge	2,692.4	22.9	....	0.0	2,109.4	24.7
Calcine in kiln	1,320.1	11.2	....	0.0	1,222.7	14.3
Dusts	379.9	3.2	148.3	4.0	235.2	2.8
Fume	7,010.0	58.6	3,272.6	88.0	3,610.8	32.2
Loss	382.8	4.1	302.1	8.0	1,361.9	16.0
Total	11,785.2	100.0	3,723	100.0	8,540	100.0

LEAD-ZINC-SILVER ORE FROM EUREKA, COLO.

A sample of complex lead-zinc sulphide ore carrying silver, with a gangue of quartz and rhodonite, was obtained from the Sunnyside Mining & Milling Co., of Eureka, Colo., for use in volatilization tests for the recovery of the lead and silver. All tests were made in the muffle furnace. The sample was crushed until it all passed a ¼-inch screen, and was then well mixed and quartered. One quarter was ground through a 30-mesh screen for volatilization tests, and the remainder was ground to sizes necessary for table concentration and flotation tests. The sample of the ore assayed as follows:

Assays of lead-zinc sulphide ore from Eureka, Colo.

Au	ounces	0.10	Fe	per cent	4.27
Ag	do	2.64	CaO	do	3.05
Pb	per cent	6.05	S	do	15.83
Zn	do	12.30	Insoluble	do	61.08
Cu	do	.52			

The first tests, on the −30-mesh material, tried the chloride volatilization treatment for recovering the lead and silver from the raw ore.

The lead proved relatively easy to volatilize, but the silver presented a more difficult problem. The best silver recovery was about 55 per cent, and only 25 per cent was volatilized from one sample. Large percentages of the zinc also volatilized. A great difficulty lay in the tendency of the ore to sinter because of the easily fusible metal sulphides. Sintering not only makes the ore hard to handle in the kiln, owing to its stickiness, but also prevents the gases from coming in contact with all of the metal to chloridize The results of the volatilization tests are given below:

TABLE 18.—*Data on volatilization tests of lead-zinc ore from Eureka, Colo.*

Weight of ore.	Reagents.		Time.	Temperature.	Weight of calcine.	Analysis of calcine.			Metal volatilized.		
	NaCl.	$CaCl_2$.				Ag.	Pb.	Zn.	Ag.	Pb.	Zn.
Grams.	*Grams.*	*Grams.*	*Hours.*	° *C.*	*Grams.*				*Per ct.*	*Per ct.*	*Per ct.*
100	2.5	7.5	1½	900	91.9	1.30	Trace.	7.64	54.7	99.9	44.5
100	5	5	1½	900	87.0	1.54	Trace.	6.34	49.3	99.9	55.0
100	5	10	1½	900	96.3	2.06	Trace.	6.31	24.9	99.9	50.6
100	7.5	7.5	1½	900	95.0	2.00	Trace.	3.53	28.0	99.9	72.8

Following the volatilization tests—which, although not entirely conclusive, indicated the inapplicability of the process to the raw sulphide ore—concentrating tests were made to produce a sulphide concentrate containing most of the economic metallic minerals, which could be used for subsequent volatilization. One table concentration test was made on material passing a 30-mesh and retained on a 60-mesh screen; a second table test was run upon material crushed through a 60-mesh and retained on a 100-mesh screen. The concentrate from each test was used for volatilization tests; the middling from the first test which was crushed through 80 mesh was mixed together with the middling and slime from the second test with raw ore and used in a flotation test. This test produced a third concentrate for volatilization, and the three tailings which were discarded.

On account of the probability of their sintering when heated, the concentrates were first given an oxidizing roast to reduce their sulphur content. The three products were put in separate shallow pans and were roasted at the same time in a gas-oven furnace. The roast was completed in seven hours, and the temperatures were approximately as follows:

Temperatures of concentrates in oxidizing roast.

	C.		C.
First hour	350	Fifth hour	650
Second hour	475	Sixth hour	730
Third hour	550	Seventh hour	800
Fourth hour	600		

Table 19 gives the results of the oxidizing roast of concentrates.

TABLE 19.—*Data on oxidizing roast of concentrates of lead-zinc ore from Eureka, Colo.*

	Table test 1.	Table test 2.	Flotation test.
Before roasting:			
Weight of concentrate.....grams..	165	210	363
Analysis of concentrate—			
Ag.....ounces..	5.04	7.74	13.02
Pb.....per cent..	10.64	17.80	19.20
Zn.....do....	16.80	24.1	22.6
S.....do....	11.90	17.50	18.42
Time of roast.....hours..	7	7	7
After roasting:			
Weight of calcine.....grams..	148	191	326
Analysis of calcine—			
Ag.....ounces..	4.82	8.34	12.56
Pb.....per cent..	10.23	20.63	21.18
Zn.....do....	15.40	22.2	20.42
S.....do....	1.84	3.14	4.65

The chloride volatilization of calcine from table test 1 follows:

Analysis of roasted concentrate in test 1.

Ag.....ounces..	4.82
Pb.....per cent..	10.23
Zn.....do....	15.40

In test 1, 50 per cent excess $CaCl_2$ was used over that necessary for the lead, and 5 per cent NaCl for the silver. The charge was 60 grams of roasted concentrate, 5.4 grams $CaCl_2$, 3 grams NaCl. The weight of the calcines was 54 grams.

Analysis of calcine and extractions.

		Metal volatilized.
		Per cent.
Ag.....ounces..	0.60	88.8
Pb.....per cent..	.41	96.4
Zn.....do....	17.9	None.

In test 2, 100 per cent excess $CaCl_2$ was used for the lead, and 5 per cent NaCl for the silver. The charge was 60 grams roasted concentrate, 7.2 grams $CaCl_2$, 3 grams NaCl. The weight of the calcine was 53 grams.

Analysis of calcine and extractions of metal.

		Metal volatilized.
		Per cent.
Ag.....ounces..	0.75	86.4
Pb.....per cent..	.33	97.2
Zn.....do....	14.0	19.7

Chloride volatilization of calcine from table test 2 follows:

Analysis of roasted concentrate in test 2.

Ag.....ounces..	8.34
Pb.....per cent..	20.62
Zn.....do....	22.2

In test 3, 50 per cent excess $CaCl_2$ over that necessary for the lead was used, and 6 per cent NaCl for the silver. The charge was 50 grams roasted concentrate, 8.3 grams $CaCl_2$, 3 grams NaCl. The weight of the calcine was 40 grams. Analysis of calcine and percentage of extraction follows:

Analysis of calcine and extractions of test 3.

		Metal volatilized.
		Per cent.
Ag ....ounces..	0.90	92.3
Pb	Trace.	99.9
Zn ....per cent..	24.2	12.8

In test 4, 100 per cent $CaCl_2$ excess was used for lead, and 6 per cent NaCl for the silver. The charge was 50 grams roasted concentrate, 11.1 grams $CaCl_2$, 3 grams NaCl. The weight of calcine was 39.1 grams. Analysis of calcine and recovery of metal follows:

Analysis of calcine and extractions in test 4.

		Metal volatilized.
		Per cent.
Ag ....ounces..	0.65	93.9
Pb ....per cent..	.41	98.4
Zn ....do....	22.2	21.8

Chloride volatilization of the calcine from flotation test follows:

Analysis of roasted concentrate.

Ag ....ounces..	12.56
Pb ....per cent..	21.18
Zn ....do..	20.42

In test 5, 50 per cent $CaCl_2$ over amount required for the lead was used, and 8 per cent NaCl for the silver. The charge was 50 grams roasted concentrate, 8.6 grams $CaCl_2$, 4 grams NaCl. The weight of the calcine was 40.2 grams. Analysis of the calcine and extractions of the metal follows:

Analysis of calcine and recovery in test 5.

		Metal volatilized.
		Per cent.
Ag ....ounces..	1.10	93.0
Pb ....per cent..	.37	98.6
Zn ....do....	18.3	28.0

In test 6, 100 per cent excess $CaCl_2$ for lead was used, and 8 per cent $NaCl_2$ for silver. The charge was 50 grams roasted concentrate, 11.4 grams $CaCl_2$, 4 grams NaCl. The weight of the calcine was 39.4 grams. Analysis of calcine and extractions of metal follows:

Analysis of calcine, and recovery in test 6.

		Metal volatilized.
		Per cent.
Ag .... ounces..	1.20	92.4
Pb .... per cent..	.28	98.9
Zn .... do....	17.6	32.2

In test 7, 50 per cent excess $CaCl_2$ for lead was used, 8 per cent NaCl, and 4 per cent CaO for the silver. The charge was 50 grams roasted concentrate, 8.6 grams $CaCl_2$, 4 grams NaCl, 2 grams CaO: The weight of calcine was 41.8 grams. Analysis of calcine and percentage of metal recovered follows:

Analysis of calcine, and extractions in test 7.

		Metal volatilized.
		Per cent.
Ag .... ounces..	1.00	93.4
Pb .... per cent..	.37	98.5
Zn .... do....	16.5	32.4

In test 8, 100 per cent excess $CaCl_2$ for lead was used, 8 per cent NaCl, and 8 per cent CaO for silver. The charge was 50 grams roasted concentrate, 11.4 grams $CaCl_2$, 4 grams NaCl, 4 grams CaO. The weight of calcine was 44.4 grams. Analysis of calcine and percentage of recovery of metal follows:

Analysis of calcine, and extractions in test 8.

		Metal volatilized.
		Per cent.
Ag .... ounces..	0.52	96.4
Pb .... per cent..	.33	98.6
Zn .... do....	17.2	25.3

The results obtained in the foregoing volatilization tests with Sunnyside ore are regarded as very good. Such high extractions of the silver are unusual. No trouble was encountered from sintering. The extractions made on the roasted concentrate indicate that the raw ore should be roasted and the products volatilized. No tests were made in the concentration of the zinc in volatilization of tailings.

SILVER AND LEAD FROM ZINC CONCENTRATE FROM GOODSPRINGS, NEV.

By E. P. Barrett.

The Yellow Pine mine at Goodsprings, in southwestern Nevada, yields an ore nearly free from gangue constituents. The ore as mined contains 8 to 12 per cent lead, 8 to 12 ounces silver, and 30 to 35 per cent zinc, and is milled in a gravity concentration plant where separation of the lead and zinc is made. The ore is nearly all carbonate, except a small amount of galena, and the lead concentrates contain considerable zinc; the zinc concentrates contain lead and silver in quantities ranging from 3 to 7 per cent and 3 to 7 ounces, respectively. The lead and silver in the zinc concentrate is a total loss, as well as an expense; on a basis of 35 to 50 tons per day the loss becomes serious. The following tests were made with the object of recovering this lead and silver:

MUFFLE EXPERIMENTS.

The first preliminary tests on the chloride volatilization of the silver and lead from Yellow Pine ore, on 100-gram charges, were made to determine the conditions that would give the highest volatilization of these metals, the varying factors being temperature, time of heating, fineness of crushing, and the kind and amount of chloridizing reagent. The results obtained with calcium chloride were slightly better than those with salt, but the higher cost of calcium chloride prevented its use on a commercial scale. About 90 per cent of the silver and lead were volatilized when salt was the chloridizing reagent. The charge was heated to 1,000° C. for 30 minutes.

LABORATORY EXPERIMENTS.

Following the muffle tests, C. C. Stevenson carried out five experiments in the 3-foot kiln, equipped with a screw feeder and a calcium chloride injector, as already described (p. 37). During some of the tests calcium chloride was injected into the heated part of the kiln. Assays of products made from this series of tests indicated that high percentages of the silver and lead can be volatilized in a rotary kiln using salt or both salt and calcium chloride.

SEMICOMMERCIAL EXPERIMENTS.

Two series of tests were made on 200-pound samples in the 20-foot kiln. Salt alone was used in some tests as the chloridizing agent;

in other tests both salt and calcium chloride were used. Here, again, is evidence that an increase of temperature increases the percentages of the silver and lead volatilized, and that calcium chloride causes somewhat higher percentages of metals to be volatilized than does salt alone. Tables 20 and 21 were compiled by Mr. Barrett from data on these tests.

TABLE 20.—*Data of tests on Nevada zinc concentrates in 20-foot kiln.*

Test No.	Date.	Weight.			Time.		Temperature.	Volatilized.		Collected.		Loss.	
		Ore.	NaCl.	CaCl.₂	Hours.	Minutes.		Ag.	Pb.	Ag.	Pb.	Ag.	Pb.
	1919.	*Lbs.*	*Lbs.*	*Lbs.*			°*C.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
1	July 13	210	....	12.6	1	15	800	21.9	50.8	23.8	31.6	—2.0	18.2
2	16	300	13.5	....	1	15	850	32.9	25.8	20.5	16.8	12.4	9.0
3	17	208	....	....	1	15	850	8.2	13.6	10.1	3.2	—1.9	10.4
4	18	210	4.2	8.4	1	15	850	35.3	49.1	38.3	36.6	—3.0	12.5
5	22	200	6.0	4.0	1	15	950	64.1	39.0	28.3	41.2	35.8	—2.2
6	28	200	9.0	....	1	15	950	49.2	47.2	41.1	29.6	8.1	17.6
7	Aug. 1	200	9.0	....	2	00	950	47.6	48.6	34.8	30.8	12.8	17.8
8	Nov. 5	475	28.5	28.5	....	....	1,000	48.6	53.3	29.1	22.7	19.5	30.6
Average (omitting test 3)								42.8	44.8	31.9	29.9	11.9	14.8
Average, NaCl only, 4½ per cent								43.2	40.5	....	....	....	....

TABLE 21.—*Data of tests on Nevada zinc concentrates, based on assays of first discharged calcine and on average discharged calcine.*

Test No.	Date.	Weight of concentrates.	Decrease in weight	Heading assays.		First discharged calcine.				Average discharged calcine.			
						Assays.		Volatilized.		Assays.		Volatilized.	
				Ag.	Pb.	Ag.	Pb.	Ag.	Pb.	Ag.	Pb.	Ag.	Pb.
	1919.	*Lbs.*	*P. ct.*	*Ozs.*	*P. ct.*	*Ozs.*	*P. ct.*	*P. ct.*	*P. ct.*	*Ozs.*	*P. ct.*	*P. ct.*	*P. ct.*
1	July 15	210	5.7	7.34	6.58	5.64	3.10	27.5	55.6	6.22	3.71	20.2	46.8
2	16	300	14.8	7.14	6.30	6.20	4.92	25.1	33.3	5.76	5.21	31.2	29.5
4	18	210	17.5	6.40	6.19	4.14	3.68	46.8	51.1	4.87	3.87	37.4	48.5
5	22	200	12.3	6.58	6.39	2.40	3.46	68.2	52.5	2.36	4.22	68.5	42.2
6	28	200	21.0	6.58	6.39	5.16	4.34	38.0	46.4	4.98	4.57	40.2	43.6
7	Aug. 1	200	16.2	6.58	6.39	3.58	2.86	54.5	62.5	3.87	3.37	50.7	55.7
	Average	....	....	....	....	....	....	43.4	50.2	....	....	41.3	44.4

J. C. Morgan and C. C. Stevenson made in the 20-foot kiln tests in which calcium chloride was injected at the firing end of the kiln. During these tests some calcines were discharged which carried small amounts of silver and lead, indicating that to inject calcium chloride into the the hot end of the furnace was more efficient than to add it with the feed. Salt was mixed with the ore before feeding to the kiln.

EXPERIMENTS IN THE 20-FOOT KILN.

A careful test was made to check test 5 (p. 70), and to obtain data from which extractions could be calculated. The nose ring in the end of the enlarged part of the kiln was removed to prevent the ore from forming there a thick bed. The speed of the kiln was one revolution per minute, and the ore was fed at the rate of about 150 pounds per hour.

Forty minutes was spent in getting to the junction of the furnace, and 20 minutes in the enlarged part of the furnace where it is assumed that most of the volatilization takes place. The ore was mixed with 10 per cent salt, and was fed into the cold end of the furnace. The following schedule shows the time of the different operations:

a. m.
11. 30. Fire started.
p. m.
12. 30. Feeder started.
1.10. Charge at junction 980° C.
1.30. Discharge began 933° C.
2.00. Feed all in 1,037° C.; 40 pounds overflow refed to kiln.
2.15. Temperature, 1,021° C.
2.40. Grab sample taken just as charge reaches junction.
3.20. Temperature, 963° C.
3.45. Shutdown; all discharged.

The average of the series of 12 tests later made in the 50-foot kiln at Goodsprings was approximately the same as the average of 7 tests in the 20-foot kiln at Salt Lake City.

Comparison of tests made at Goodsprings and at Salt Lake.

Item.	Amount volatilized.	
	Silver.	Lead.
	Per cent.	*Per cent.*
At Salt Lake City, 20-foot kiln	42. 8	44. 8
At Goodsprings, 50-foot kiln	41. 3	45. 1
The average of three tests in the Brückner furnace on 140, 187, and 195 pounds ore	63. 9	85. 4
The average of the above three tests in the Brückner, computing the values in the grab samples as being the same as the material left in the furnace	77. 6	97. 9
In the thin-bedded test in the 20-foot kiln	40. 6	60. 6

Here, again, the amount of metals extracted was close to that volatilized in thick-bedded tests, and in the 50-foot kiln at Goodsprings. Experiments were made to determine the difference in metals volatilized with thick and thin beds of ore in the kiln. In the thin-bedded test the time of heating was much shorter—only 20 minutes in the enlarged section of the kiln—and the grab sample showed that the silver content, as the charge entered that section, was 4.3 ounces; this amount was reduced to about 2 ounces by the time the ore was discharged. In the last run the metals in the fume collected accounted for all the metals volatilized.

In a rotating kiln, therefore, the greatest amount of metals is volatilized when the charge of ore and salt is heated in a thin bed in the furnace for about 45 minutes at 1,000° C.

PLANT TESTS.

YELLOW PINE MINING CO., GOODSPRINGS, NEV.

The small-scale and semicommercial volatilization experiments by E. P. Barrett on the silver-lead-zinc ores from the Yellow Pine mine, Goodsprings, Nev., gave such favorable results that the Yellow Pine Mining Co. offered to the Bureau of Mines the use of their plant at Goodsprings, Nev., in which to make volatilization tests on a commercial scale. (See Pl. V, *A*.) This offer was accepted, and representatives of the Bureau of Mines carried on a series of tests in cooperation with the Yellow Pine Mining Co.

The Yellow Pine Mining Co. operates a 75-ton mill and calcination plant at Goodsprings, Nev.; the plant is equipped with a complete concentrator. The ore is all high grade, containing little or no gangue material, so that the mill produces zinc concentrate, lead concentrate, and a slime product that can not be separated into zinc and lead concentrates.

MICROSCOPIC EXAMINATION.

Microscopic examination of a representative sample of Yellow Pine ore showed it to be a typical oxidized ore, the sulphide content being practically negligible. The zinc and lead are present chiefly in the carbonate form, although minor amounts of zinc and lead sulphides, together with a small amount of pyrite, can be identified in concentrates obtained by careful panning with a horn spoon. The gangue is largely siliceous in nature, but contains some lime carbonate and is stained with iron oxide.

Although the usual microscopic methods were used in examining the Yellow Pine ore and products, positive determination of any silver mineral in the low-grade ore has not been reached. Careful scrutiny of panned concentrates of screen products failed to get visible evidence of the presence of silver; hence several fire assays were made upon samples of the products examined in order to determine approximately the location of the silver. The results of these tests indicated that much of the silver was carried by the residual sulphides, probably as finely disseminated argentite, the remainder lying with the balance of the ore. The puzzling feature was the relatively high silver content of the gangue, careful examination of which in the crushed condition failed to give any clue to the presence of silver.

CALCINING PLANT.

The zinc concentrate made in the mill, which was originally shipped to zinc smelters in Kansas and Oklahoma, assayed about 33 per cent zinc, 4 to 6 per cent lead, and 4 to 6 ounces silver. The silver and lead in the zinc concentrate was a total loss, as neither of these metals was paid for by the purchaser of the concentrate.

Mr. Kirk installed a calcining kiln, 50 feet long and 6 feet in diameter, to dry the ore, which contained approximately 20 per cent moisture as it left the mill, and to heat it simultaneously to about 800° C., in order to drive off the carbon dioxide from the ore. By this procedure the grade of the zinc concentrate was raised to approximately 38 per cent zinc. All material calcined and treated in the kiln was ungraded, the maximum size being about one-fourth inch.

VOLATILIZATION EXPERIMENTS.

The procedure in the Goodsprings tests was to mix the zinc concentrate and the requisite quantity of salt before feeding the material into the 50-foot kiln. The temperature was raised to between 800° and 1,000° C. in order to volatilize the silver and lead.

In most of the experiments conducted at Salt Lake both salt and calcium chloride were used for chloridizing. In the first experiments at the Goodsprings plant both calcium chloride and salt were used, but the high cost of calcium chloride and the extreme difficulty in preparing it and in mixing it with the ores made it prohibitive for commercial use, hence the other tests were performed with salt alone as a chloridizing agent. The tests were begun at the plant in February, 1920. Mechanical difficulties were encountered with the blower and the oil burner, and consumed much time before they were finally overcome.

OPERATIONS OF ROTARY KILN.

It soon became obvious that the ore did not remain in the kiln long enough at the proper temperature to effect complete chloridizing volatilization of the silver and lead. The speed of the kiln was decreased somewhat, with better results. Several times, when the kiln was shut down, the ore was sampled at intervals of 3 feet along the length of the kiln to determine the rate at which the metals volatilized. After the kiln was cool enough, an examination was made which showed that the first 20 feet of the kiln served only as a drier, and there the ore hardened somewhat and adhered to the sides of the furnace. In the next 20 feet the ore and salt were brought up to the required temperature, only the last 10 feet in the kiln being available for volatilization. Many experiments made to determine the time necessary for the ore to pass through the kiln showed it to be usually between 40 and 50 minutes, so that the ore remained in the heated zone only 10 or 12 minutes.

SCREEN TEST OF ORE FROM CONVEYER

A screen test of a sample taken from the conveyer feeding into the kiln showed the following percentages:

Screen test of Yellow Pine ore being fed into rotary kiln.

	Weight.	Cumulative weight.
	Per cent.	*Per cent.*
On 4-mesh screen	42. 0	....
On 8-mesh screen	27. 6	69. 6
On 10-mesh screen	6. 4	76. 0
On 20-mesh screen	6. 0	82. 0
On 48-mesh screen	6. 8	88. 8
On 65-mesh screen	1. 4	90. 2
On 100-mesh screen	1. 8	92. 0
Through 100-mesh screen	7. 8	99. 8

DEMONSTRATION TREATER.

The Western Precipitation Co., Los Angeles, Calif., in order to demonstrate the efficiency of the Cottrell electrostatic precipitator in collecting chloride fumes, installed at the plant, under the direction of Mr. H. V. Welch, a six-pipe treater complete with a 15-kv.a., 220-volt, alternating-current generator, 220 to 100,000 volt transformer, rectifier, and switchboard. The Yellow Pine Mining Co. furnished motors and fans to complete the precipitation plant. The treater was erected beside the concrete base of the stack and was so connected with a fan that 300 cubic feet of gases a minute could be drawn from the flue and passed through it. (See Plate V, *B*.) The average volume of gas passing through the main stack was about 5,000 cubic feet a minute. This small treater worked very satisfactorily, only a slight haze being seen above the outlet at the top of the treater. Plate VI shows comparison of densities of fume passing through the main stack and through the treater.

FIRST PLANT TEST.

Test 1 was made over a period of three hours during which the feed was 6,000 pounds of wet ore, 16.6 per cent moisture, plus 300 pounds of salt an hour. The salt was added to the ore on the pan conveyer feeding the kiln. The temperature in the hottest part of the kiln was about 1,000° C., at which point there was a slight tendency for the ore to fuse and "ball up," and the calcine was discharged at a temperature just below the fusion point. The consumption of oil was about 25 gallons an hour, or 8 gallons a ton of feed. The gases entered the treater at about 185° C. at a velocity of 6 feet a second through the pipes. There was visual clearance from the fume at all times. The fume, which was almost white, was analyzed by Mr. Nestell, analyst for the Western Precipitation Co., who reported as follows:

Analysis of fume from Cottrell treater, test 1.

Au	ounce	0.04	K	per cent	0.55
Ag	ounces	28.80	Na	do	8.62
Pb	per cent	42.40	SO_4	do	6.34
Cu	do	.44	Cl	do	29.12
Zn	do	6.30	H_2O	do	3.24
Fe	do	1.79			

The amounts of silver and lead volatilized in this test were 42 per cent and 41.8 per cent, respectively.

SECOND PLANT TEST.

The speed of revolution of the kiln was reduced in test 2 to one revolution in about two and one-half minutes in order to increase the length of time necessary for the ore to travel through the kiln. The charge was heated for a longer time, and thus a greater amount of the silver and lead was volatilized. The charge was 4,600 pounds of wet ore, 18 per cent moisture, plus 225 pounds of salt an hour. The salt was added on the pan conveyer and was mixed with the ore in the upper part of the kiln. Assays of the products of this test follow:

Assays of products of second plant test.

Item.	Ag.	Pb.	Zn.	Cl.
	Ounces.	*Per cent.*	*Per cent.*	*Per cent.*
Feed	3.40	3.90	36.4	....
Calcine	1.56	2.00	44.3	1.86
Fume	34.72	37.90	6.2	30.27

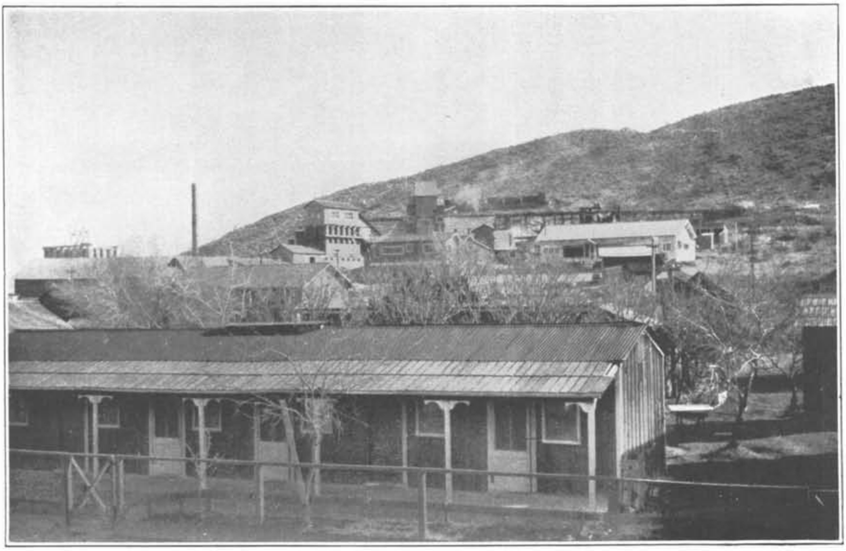
The loss in weight was 18 per cent, and the amounts of silver and lead volatilized were 62.3 per cent and 58 per cent, respectively.

THIRD PLANT TEST.

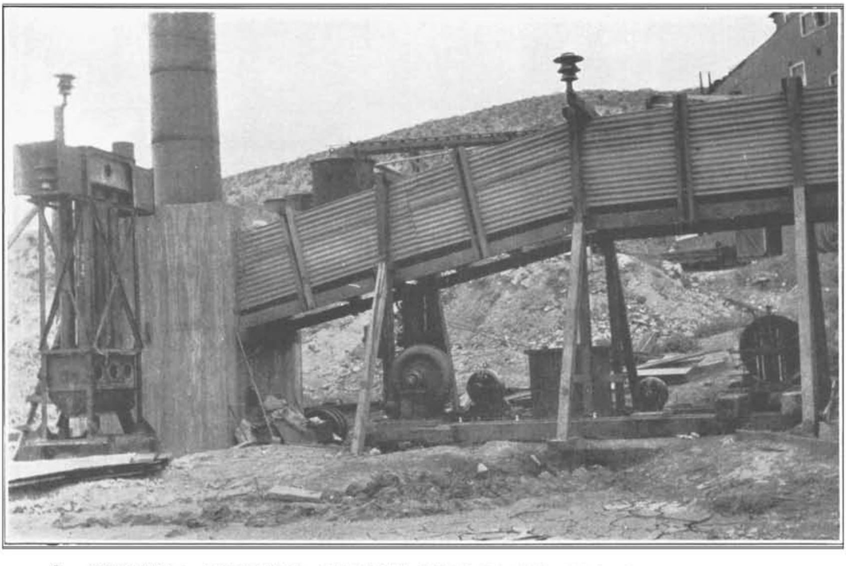
The speed of the kiln in test 3 was one revolution in about two and one-half minutes. The mill had been shut down, thus allowing the ore in the bins to dry out and reducing the moisture content in the feed. The charge was 6,000 pounds of wet ore, 5 per cent moisture, plus 360 pounds of salt an hour. A temperature of 1,000° C. was maintained in the lower part of the kiln throughout this test. Assays of the products follow:

Assays of products of third plant test.

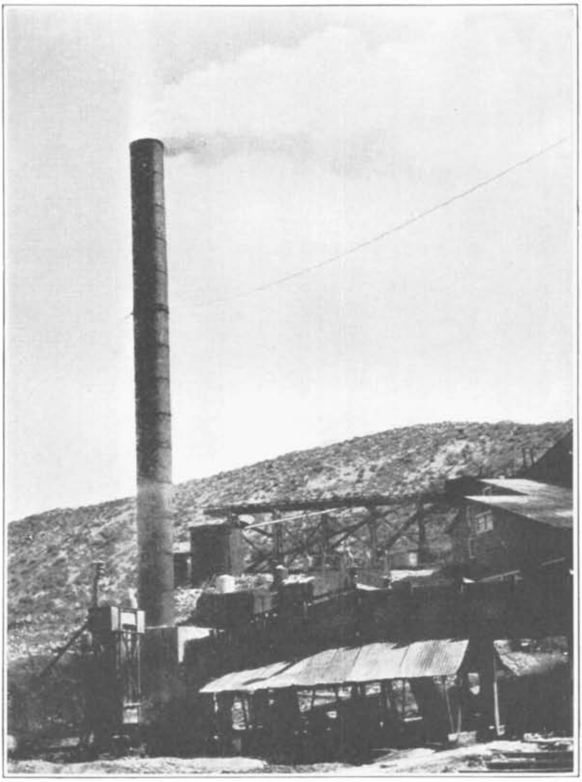
Item.	Ag.	Au.	Pb.	Zn.	Cl.
	Ounces.	*Ounce.*	*Per cent.*	*Per cent.*	*Per cent.*
Feed	4.00	....	4.00	37.5	....
Calcine	2.58	....	1.80	45.0	....
Fume	35.20	0.08	30.80	7.2	33.5



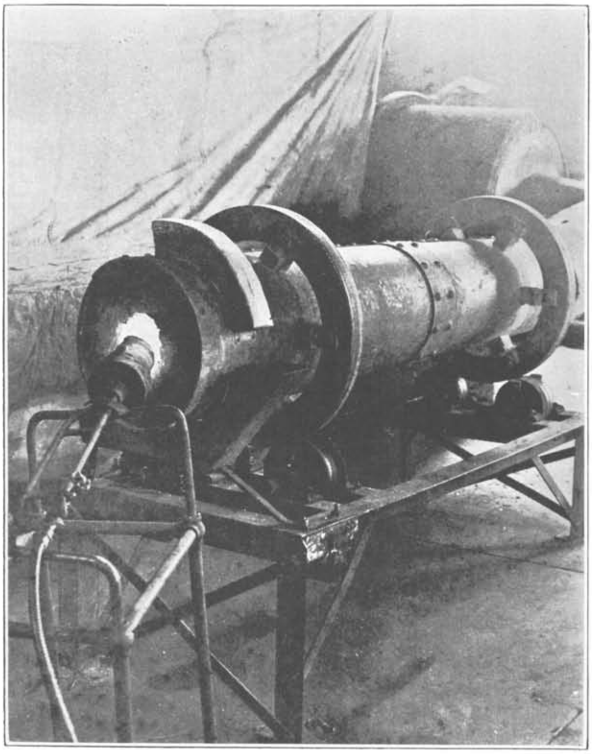
A. GENERAL VIEW OF YELLOW PINE MILL, GOODSPRINGS, NEV.



B. COTTRELL TREATERS, YELLOW PINE PLANT, GOODSPRINGS, NEV.



CHLORIDE FUMES ESCAPING THROUGH STACK, AT BASE OF WHICH IS A SMALL UNIT OF A COTTRELL PRECIPITATOR, YELLOW PINE PLANT. GOODSPRINGS, NEV.



ROTARY KILN, SHOWING SCOOP FEEDER AT FIRE END OF FURNACE; BUILT AFTER KIRK'S DESIGN.

The loss in weight was 16.7 per cent, and the amounts of silver and lead volatilized were 48.2 per cent and 62.5 per cent, respectively.

FOURTH PLANT TEST.

Test 4 was made to determine the possibilities of recovering by volatilization the silver and lead in the mill slime. The charge was about 4,000 pounds of slime, 20 per cent moisture, plus 240 pounds of salt. The regular feed to the kiln was shut off for 30 minutes, allowing the ore burden in the upper part to pass down the kiln, so that only a little mixing of the slime with left-over concentrate was necessary. The charge dusted much more than the regular charge of concentrate, but not as much as might be expected with material practically all finer than 200-mesh size. Assays of the products of this test were as follows:

Assays of products of test 4.

Item.	Ag.	Pb.	Zn.	Cl.	Insoluble.
	Ounces.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Slime	5.43	6.00	32.00		
Calcine	4.20	3.60	39.70		
Fume	28.00	31.30	14.30	19.6	6.20

The reduction in weight was 16.8 per cent, and the amounts of silver and lead volatilized were 35.7 and 50 per cent, respectively. Test 4 gave better results than were expected. The percentage of fume from insoluble matter would as a matter of course be higher than from concentrate. The higher zinc content was undoubtedly due to mechanical losses through dusting of the charge, rather than to higher volatilization of the zinc. This test shows that the slime could be dewatered and mixed with the coarse concentrate and the mixture fed without difficulty into the kiln. The daily tonnage of slime represents about 15 per cent of the concentrate tonnage from 9 to 10 tons.

LOSSES OF METAL AS DUST.

In order to determine the losses from dust being carried up the stack in the usual operation of the calcination plant, a test was made passing part of the flue gas through the treater. The gases in the stack were at 200° C., with a velocity of 12 feet a second, their volume being 5,040 cubic feet a minute; 297 cubic feet, or 5.89 per cent, of the flue gas was deflected to the treater, entering it at a temperature of 187° C., with a velocity of 39.23 feet a second. The dust collected at a point 10 feet from the base of the stack amounted to 0.0686 grams a cubic foot of gas. The loss from the top of the stack may be somewhat smaller than this figure. The treater was operated at 45,450 volts and gave almost perfect visual clearance at all times. The feed to the kiln was 5,400 pounds of ore per hour, and the duration of the test was four hours. An assay of the dust follows:

Assay of dust taken 10 feet from base of stack.

Material.	Ag.	Pb.	Zn.	Insoluble.
	Ounces.	*Per cent.*	*Per cent.*	*Per cent.*
Dust from stack	11.9	8.4	39.6	13.4

The loss per eight-hour shift was approximately 2.38 ounces of silver, 33.6 pounds of lead, and 158 pounds of zinc. The fume from the volatilization tests assayed about 1 per cent of insoluble matter, a quantity much less than the insoluble matter in the dust. Hence dusting was probably decreased by the addition of salt to the charge.

CONCLUSIONS FROM EXPERIMENTS.

The experiments tabulated and described above are but a few of those made at the plant of the Yellow Pine Mining Co., at Goodsprings, Nev. Tests tabulated as laboratory experiments are but a few of many made at the Intermountain experiment station of the Bureau of Mines. The results recorded here prove that the chloridizing volatilization process can be applied to this type of ore. With proper machinery and with right plant conditions a higher extraction of the silver and lead can no doubt be obtained.

The equipment used at Goodsprings was not perfect; moreover, the tests were made during the regular operations of the calcining plant. The greatest difficulty in effecting high extraction seemed to be that the ore was not held long enough in the part of the furnace maintained at the desired temperature to effect complete volatilization. In addition to the present kiln or the substitution for it of a Brückner kiln was suggested to the management of the Yellow Pine plant.

The silver and lead in the calcine shipped to the zinc smelters is a total loss to the mining company. The recovery of only 50 per cent of each of these metals would more than pay the cost of operating a volatilization plant.

The fume collected in the treater was assayed many times, a fair average being as follows:

Average assay of fume in treater.

Gold	ounces	0.08	Zinc	per cent	6.1
Silver	do	34.8	Chlorine	do	28.2
Lead	per cent	35.6	Insoluble	do	1.0

Laboratory experiments in reducing similar fume have easily yielded silver-lead bullion and calcium chloride slag. The latter could be used again as a chloridizing agent. Unfortunately, not enough fume was made to conduct semicommercial experiments. Similar fume is sold in Utah to the lead smelters, and is smelted to base bullion after admixture with lead and silver ores.

Salt alone should be used as a chloridizing agent; it can be procured very cheaply at the plant and can be handled without difficulty.

The general depression of the metal market and the high freight rates forced the Yellow Pine Mining Co. to suspend operations in the autumn of 1920, about the time the final report was made covering the foregoing experimental work. On the resumption of activities this company might well install a plant designed especially to recover metals now being lost.

POPE SHENON MINING CO.

The Pope Shenon Mining Co. owns several patented mining claims in the vicinity of Salmon City, Idaho. The principal value in the ore extracted is copper, which exists in several different mineral forms, the upper workings being chiefly malachite, azurite, cuprite, chalcocite, and chalcopyrite. After several years of development, the company decided to build a reduction plant, as the grade of ore mined was not high enough in copper for direct shipment to smelters. Experimental work in concentration and flotation failed to recover the oxidized minerals in the ore and a material loss of copper content.

EXPERIMENTAL WORK.

A series of experiments was conducted at the Intermountain station, consisting of muffle tests, electric-tube tests, small laboratory tests, and, finally, tests on lots ranging from 200 up to 2,000 pounds in the semicommercial plant.

The results of these preliminary experiments indicated clearly that high extraction of the copper could be obtained by chloride volatilization and that practically all of the volatilized copper chloride could be recovered in the Cottrell treaters.

DESIGN OF PLANT.

Under the direction of Dr. R. H. Bradford, of the department of metallurgy, University of Utah, the company constructed a plant, based on the laboratory results, to treat approximately 35 to 50 tons of ore in 24 hours. The crushing plant was designed with capacity to crush 150 tons in 24 hours, and the general plant was so arranged that additional furnaces could be added at any time in order to increase the tonnage of ore treated. As it is not necessary to crush the ore finer than −16 mesh, crushers and rolls, screens, and elevators constitute the total equipment in the crushing plant.

FURNACE.

The furnace installed, a rotary kiln 42 inches in diameter and 25 feet 3 inches long, is heated by the flame from an oil burner in a short fire box at the fire end of the furnace. At the flue end are a short flue and dust chamber to conduct the fumes to the Cottrell precipitator and to settle the coarse dust carried over by the rather strong draft resulting from a fan placed at the end of the dust chamber near the Cottrell treaters.

COTTRELL PRECIPITATOR.

The Cottrell precipitator consists of 36 iron pipes, 15 feet long and 8 inches in diameter, open at both ends. An iron wire in the center of each pipe carries a 65,000-volt electric current, and the pipe serves as the other electrode. A brush or corona discharge of electricity passes from the wire to the pipe, and the solid matter in the fumes is precipitated completely on the inside of the pipe and on the wire. At intervals the pipes and wires are shaken, and the precipitated copper chloride is collected below in the hopper-bottomed chamber. A screw conveyer carries the fume from the hopper to a small bucket elevator leading to a 25-ton storage bin.

FUSION FURNACE.

From the storage bin the fume and a calculated quantity of coke and limestone is fed by a screw conveyer to a reverberatory fusion furnace. This furnace, which it is necessary to run only about half the time, is built large enough to fuse down all the fume from the volatilization furnace. In the melting furnace the lime takes the chlorine from the copper chloride, and the carbon of the coke reduces the copper to the metallic condition. A temperature high enough to melt copper is maintained in the fusion furnace, calcium chloride slag being skimmed off at intervals. As the copper accumulates, it is also tapped off into molds ready for the refinery.

The two experimental runs summarized in Table 22 are probably representative of the more successful tests conducted at intervals during the summer of 1920.

The tests were made with oxidized ore from the dump crushed to −10 mesh and assaying 3.5 per cent Cu, 4 per cent Fe, 73 per cent insoluble, and 0.40 per cent S. The plant operated continuously for 12 hours, treating 18 tons of ore mixed with 1,080 pounds of common salt crushed to −10 mesh. The temperature was approximately 900° C.

TABLE 22.—*Summary of test on oxidized Idaho ore from Pope Shenon plant.*

Material.	Weight.		Assay.		Total Cu in head sample.
	Pounds.	Per cent.	Cu.	Cu.	
			Per cent.	*Pounds.*	*Per cent.*
Crude ore treated	36,000	100.0	3.50	1,260	100.0
Calcine discharged	34,200	95.0	.56	192	15.2
Precipitated fume	2,551	7.1	41.00	1,046	83.0
Total	[a]36,751	102.1	....	1,238	98.2

[a] Increase in weight was due to the addition of salt.

In a test at the Pope Shenon plant on August 18, 1920, 17 tons of crude ore was treated. The assay of the ore showed 3 per cent Cu, 2 per cent as sulphides, and 1 per cent as oxides and carbonates. The rate of feed of the ore was 35 tons in 24 hours. One thousand pounds of salt was used, or 3 per cent of the ore charge. The temperature was a red heat, approximately 850° to 900° C. The Cottrell treaters gave almost perfect clearance. An average assay discharged in the calcine 0.75 per cent Cu, the lowest sample discharging 0.45 Cu. The recovery of copper in fume was 70 per cent. Table 23 gives a summary of a day's run at the Pope Shenon plant on above date.

TABLE 23.—*Day's experimental run at Pope Shenon plant.*

Material.	Weight.		Assay.		Total Cu in head sample.
	Pounds.	Per cent.	Cu.	Cu.	
			Per cent.	*Pounds.*	*Per cent.*
Original crude ore	3,400	100.0	3.00	1,020	100.0
Calcine discharged	32,300	95.0	.75	242	23.7
Fume collected	1,786	5.2	40.00	714	70.0
Flue dusts	275	.8	1.50	41	4.0
Total	34,361	101.0	....	997	97.7

BLAISDELL COSCOTITLAN SYNDICATE.

The Blaisdell Coscotitlan Syndicate, Pachuca, Hidalgo, Mexico, first investigated the chloride volitalization process for the treatment of "patio tailings" at their metallurgical plant at Pachuca, Hidalgo, Mexico. The preliminary experiments were carried on in cooperation with the Western Precipitation Co., Los Angeles, Calif., and with the Salt Lake station of the Bureau of Mines. As a result of the experiments, which indicated that the process might be successful in recovering metals from the ore, a pilot plant was designed and built.

After success had been attained in the small plant, a large one was built and was operated successfully for some time. In a communication to the Bureau of Mines, P. A. Babb, general manager of the

Blaisdell Coscotitlan Syndicate, described the operation of the plant and indicated that this plant, too, was successful. He wrote in part as follows:

> We have no fault to find with our extractions—Au, Ag, Cu, and Hg—for they were well over 90 per cent except on silver, which was 75 per cent, and good on the material in question (patio tailings). Incidentally, we had perfect extractions on small quantities of lead and zinc, which were of no commercial interest.
>
> The Cottrell worked splendidly. Very little chlorine reached the exhaust fan; we sprayed the charged gases before they passed to the Cottrell treaters. On solids in the gases (exclusive of water-soluble salts) the recovery in the treater was practically 100 per cent. We determined the treater efficiency on the water soluble salts (although not quite fair to the treater) and obtained around 97 per cent clearance.
>
> The prices of metals is the reason we are not employing the process just now. We require 75-cent silver. We believe we had worked out all the fundamental mechanical difficulties. The oil consumption will be 12 gallons of 18,500 B. t. u. fuel oil per dry metric ton, and this without preheating air or any insulation. Our kiln is 125 feet by 8 feet diameter, shell lined with 9-inch concrete-clinker brick. On our material we find it is essential that charge (in the kiln) rolls or cascades as against sliding, that is, presents new surfaces continually as it travels down the kiln; and under these conditions we expect very little not to be reclaimed. No advantage was found with calcium chloride, but 5 per cent of CaO is necessary to reach the proper finishing temperature.
>
> ### FUME TREATMENT.
>
> Our scheme was to wet the gases pretty thoroughly and so condense the water vapor that flushing would be automatic in the treater. We had not reached that stage when we stopped the runs, but felt very hopeful it would be developed. There was always considerable HCl in the gases and circuit in which the fumes were handled; hence, we had practically all our copper and mercury in the soluble or "ic" form. The sludge from the treater was filtered and water washed. The filtrate went to scrap-iron precipitation, removing copper, mercury, considerable gold and silver (lead also dropped out here); the liquor left (a strong saline solution of sodium and potassium chlorides and some sulphates, also iron and zinc chloride) was employed to wet down the entering charge into the kiln.
>
> The solids we proposed to hyposulphite for recovery of silver, which responded excellently in a very short contact, and gold. The gold extraction was not so good, and we had not quite worked out this factor before discontinuing operations. However, the sludge would go again to the kiln for treatment.

BRÜCKNER FURNACE THIN-BEDDED EXPERIMENTS.

The Brückner furnace at the Intermountain experiment station was used for a series of 100-pound tests, in which a thin bed of ore was maintained at the desired temperature for any convenient length of time. Grab samples were taken every 15 minutes, and from the assays of these samples curves were drawn showing the amounts of silver and lead left in the calcine in the furnace. Four tests were made, the curves in Figure 4 showing the averages of the series. The ore treated was the zinc concentrates from the Yellow Pine mill, Goodsprings, Nev.

No attempt was made to determine the actual quantities of silver and lead volatilized. The only factor considered in these tests was the length of time necessary to heat the charge of ore and salt in order to reduce the amounts of silver and lead in the calcine to a minimum.

The data show that by heating to 1,000° C. for 165 minutes the amount of lead is reduced to 0.1 per cent and in 195 minutes the silver to 1.1 ounces. These results lead one to believe that length of time in heating was the important factor to consider in the volatilization of the silver and lead from this material. But comparison of the above results with those obtained in the muffle when over 80 per cent of the silver and lead was volatilized in 15 minutes by heating to 1,000° C. shows that time is not the most important factor.

Barrett believed that conditions approximating those in the muffle could be obtained by heating in a Brückner furnace a thin bed of ore mixed with chloridizing reagent, and that high percentages of the metals could be volatilized in the same length of time as in the muffle tests. Three experiments were made with ores, 100-pound tests of which had previously been made in the Brückner furnace, 150 minutes heating being required to volatilize quantities of metals that could be volatilized in the muffle in 40 minutes. Each test showed high percentages of metal volatilized and compared well with muffle tests.

In order to determine whether or not consistent results could be obtained by this process, a series of four tests was made, as follows:

TABLE 24.—*Test 1 with thin bed of ore in Brückner furnace.*

November 22, 1920.

Time.	Operation.	Temperature.	Assay.		
			Ag.	Pb.	Cl.
a. m.		*° C.*	*Ounces.*	*Per cent.*	*Per cent.*
9.45	Fire started.				
10.45	Ore charged plus 10 per cent NaCl	1,021	4.02	2.15	....
10.55	Fuming	940	....	....	....
11.00	Dense fumes, grab 1	1,000	3.44	1.25	4.85
11.05	Dense fumes, grab 2	1,000	2.64	.70	....
11.10	Dense fumes, grab 3	1,000	2.34	.50	2.35
11.20	Shut down, grab 4	975	1.88	.35	1.35

TABLE 25.—*Test 2 with thin bed of ore in Brückner furnace.*

Time.	Operation.	Temperature.	Assay.		
			Ag.	Pb.	Cl.
p. m.		*° C.*	*Ounces.*	*Per cent.*	*Per cent.*
1.25	Ore charged plus 10 per cent NaCl	990	4.02	2.15	....
1.35	Fuming	920	....	....	....
1.40	Dense fumes	950	....	....	....
1.45	Dense fumes, grab 1	1,021	3.56	1.20	4.45
1.50	....	950	....	....	....
1.55	....	975	....	....	....
2.00	Dense fumes, grab 2	975	2.24	.55	....
2.10	Dense fumes, grab 3	975	1.82	.30	1.75
2.25	Shut down, grab 4	....	1.80	.45	1.60

TABLE 26.—*Test 3 with thin bed of ore in Brückner furnace.*

November 23, 1920.

Time.	Operation.	Temperature.	Assay.		
			Ag.	Pb.	Cl.
p. m.		*° C.*	*Ounces.*	*Per cent.*	*Per cent.*
1.15	Fire started.				
1.45	Ore charged plus 10 per cent NaCl	....	4.02	2.15	6.00
1.55	Dense fumes	965	....	....	....
2.00	Dense fumes, grab 1	980	2.84	1.05	....
2.05	Dense fumes	1,005	....	....	....
2.15	Dense fumes, grab 2	1,000	2.70	.45	....
2.30	Dense fumes, grab 3	965	2.18	.45	3.06
2.40	Shut down, grab 4	981	1.92	.45	2.50

TABLE 27.—*Test 4 with thin bed of ore in Brückner furnace.*

Time.	Operation.	Temperature.	Assay.		
			Ag.	Pb.	Cl.
a. m.		*° C.*	*Ounces.*	*Per cent.*	*Per cent.*
3.00	Ore charged plus 10 per cent NaCl	....	4.02	2.15	6.00
3.10	Dense fumes	933	....	....	....
3.15	Dense fumes, grab 1	993	3.72	1.45	4.55
3.30	Dense fumes, grab 2	1,000	2.50	.75	2.15
3.45	Fuming, grab 3	1,000	1.30	.55	1.40
3.55	Shut down, grab 4	1,010	1.04	.50	1.00

In order to determine the relative effects of slow and rapid heating, a test was made in the 20-foot kiln, using about 150 pounds feed an hour and one revolution of the kiln in 18 seconds.

TABLE 28.—*Test 5 made in 20-foot kiln with rapid heating.*

Time.	Operation.	Temperature.	Assay.		
			Ag.	Pb.	Cl.
a. m.		*° C.*	*Ounces.*	*Per cent.*	*Per cent.*
9.20	Fire started.				
10.15	Ore feed started, 10 per cent NaCl, fire box	900	2.75	3.48	....
10.40	Ore at junction	980	....	....	....
10.45	Feed all in.				
10.50	Very dense fumes, grab 1	1,010	.84	.55	....
11.00	Very dense fumes, grab 2	965	1.08	.70	1.05
11.10	Very dense fumes, grab 3	980	1.12	.45	.95
11.20	Shut down, grab 4	....	.70	.30	.40

The purpose of these runs was to determine whether or not the amounts of silver and lead in the calcine could be consistently reduced to small percentages in duplicate tests in which the charges were heated for approximately the same time—30 to 50 minutes—as in the muffle.

At the time of making thin-bedded tests, it was thought that this method had not been heretofore understood. Later, however, attention has been called to the following statement by Hofman:[31]

> The logical consequence of the above facts (a high heat indirectly causes a larger loss of silver by the expulsion of larger quantities of volatile chlorides, etc.) is that the operator, while he endeavors to obtain a high silver chlorination, should at the same time be careful to expel as little as possible of the volatile chlorides. He will be greatly assisted in this endeavor by keeping the ore in a thick layer and by using low heat and plenty of air.

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[31] Hofman, O., Hydrometallurgy of silver, 1907, p. 20.

And to the following from the same source:

> If a small charge is thinly spread over a large hearth, more volatile chlorides will be expelled, and the ore will lose more in weight and in silver than when a larger charge is roasted in the same furnace. This is the reason why, as a rule, the loss in weight and in silver in a large Brückner furnace, in which the ore lies 2 feet thick, is less than in a reverberatory, and why small samples roasted on a roasting dish in a muffle show so much greater loss of silver than the same ore does when roasting on a large scale in the furnace.

Blamey Stevens[32] writes in the Mining and Scientific Press, July 12, 1919:

> Apparently good results, usually obtainable in the laboratory, are due to the facility of excess of air to the thin layer of ore in the muffle.

Stuart Croasdale[33] states, August 9, 1919:

> The volatilization of the metals can be completed in 20 to 30 minutes and better results can be obtained by passing a thin stream of ore through the furnace at this rate of speed than by carrying a heavier bed of ore and roasting a longer period of time.

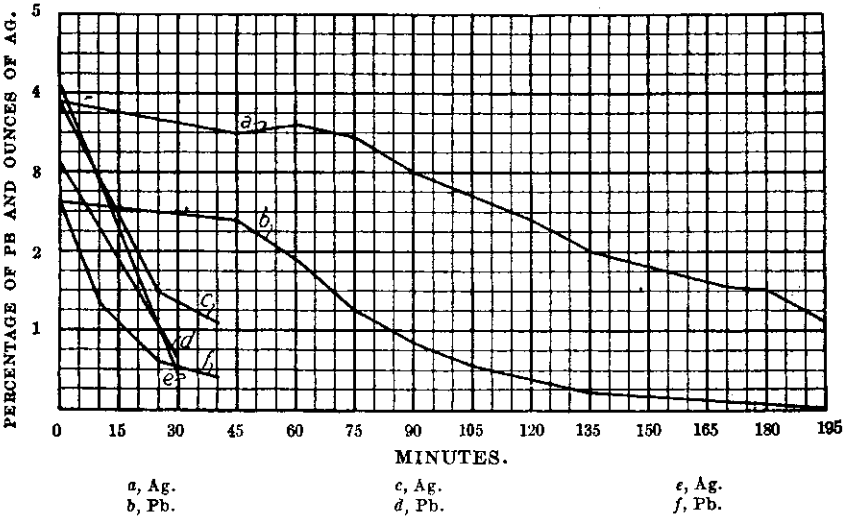

FIGURE 5.—Average assays of grab samples taken in four runs on Brückner furnace: *a*, *c*, and *e*, silver; *b*, *d*, and *f*, lead.

February 21, 1920,[34] Mr. Croasdale further states:

> I desire to call Mr. Layng's attention to my paper on the "Volatilization of metals as chlorides" (Eng. and Min. Jour., Aug. 29, 1903), where my original experiments on time variation showed that the maximum commercial volatilization was reached in 30 to 40 minutes after the cold ore was charged into the furnace. Our 25-foot furnace was not a failure so far as actual volatilization was concerned, but when it was charged to capacity with cold ore it was not long enough to give the ore more than 10 or 15 minutes roast at the volatilization temperature. When fed with a small stream of ore, so that the ore was heated to the proper temperature soon after entering the furnace and had nearly the full length of the furnace, or, in other words, 20 to 30 minutes exposure to the volatilization temperature, the results were all that could be desired.

Curves are given (Fig. 5) showing comparison of assays of grab samples taken during volatilization tests in the Brückner furnace with assays obtained in a muffle test on an oxidized silver-lead-zinc ore.

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[32] Stevens, Blamey, The chloride-volatilization process: Min. and Sci. Press, vol. 119, 1919, p. 45.

[33] Croasdale, Stuart, The chloride-volatilization process: Min. and Sci. Press, vol. 119, 1919, p. 183.

[34] Croasdale, Stuart, Chloridizing process: Min. and Sci. Press, vol. 120, 1920, p. 259.

Curves *a* and *b* represent the average assays of grab samples taken during four runs in the Brückner furnace on 195, 187, 140, and 110 pounds of ore, respectively. Ten per cent salt was used in each test, and the temperature was about 1,000° C. The thickness of ore bed was about 4½ inches. Curves *c* and *f*, terminating on the 40-minute line, represent assays of grab samples taken during a run in the Brückner furnace on 20 pounds of ore; 10 per cent salt was used in this test and the temperature was about 1,000° C. The thickness of the ore bed was about 1¾ inches. Curves *d* and *e*, terminating on the 30-minute line, represent the assays obtained from one of the best muffle tests made on this ore. Six per cent salt was used in this test and the temperature was about 1,000° C. The thickness of ore bed was about one-half an inch.

PARALLEL-FLOW VOLATILIZATION FURNACE.

DESCRIPTION.

The above experiments on silver and lead ores in the Brückner furnace indicated that when the charge in the furnace formed a thin bed only, and was quickly brought up to a temperature high enough for volatilization, higher extractions were made of the silver and lead than with a heavier feed in the furnace, or with a thick bed that required a longer time to bring the ore up to the desired temperature. In other words, the thin-bedded charge, quickly heated, showed decided advantage over a slowly heated thick-bedded charge.

The Brückner furnace could not be operated continuously, and ideal conditions could not be obtained. A parallel-flow furnace was designed by M. P. Kirk, and was installed on a Brückner furnace at Harbor City, Calif. This furnace had a scoop feeder to admit the crude ore, previously mixed with salt, into the heated end of the furnace. (See Pl. VII.) The ore, when heated, passed through the furnace, flowing in the same direction as the flue gas, and discharged continuously at the opposite end of the kiln. The furnace was 7 feet long, 10 inches internal diameter, and had a slope of about one-half inch to the foot. It was provided with a variable speed drive and could be revolved from one revolution in three minutes to three revolutions in one minute. The maximum capacity was about 75 pounds an hour.

The kiln was fired with the same burner used in the 20-foot kiln, and the gases evolved in this kiln were passed through the other kiln and into the Cottrell treaters for precipitation of the fumes. The feed was introduced through an opening in the side of the furnace, just inside a ring of brick, which prevented the charge from overflowing at the end of the kiln.

To the kiln was riveted a scoop which, revolving through a sheet-iron hopper, picked up the charge and emptied it into the kiln. The scoop practically cleaned the hopper during every revolution, so that the charge in the furnace was controlled by the quantity of material placed in the hopper.

TESTS WITH THE 7-FOOT KILN.

TEST 1.

Test 1 was made May 27, 1921, by E. P. Barrett to try out the new kiln in which the ore entered at the fire end and was heated rapidly to maximum temperature. The charge was 50 pounds of ore plus 5 pounds of salt and was fed at the rate of 50 pounds per hour. The speed of the kiln was two revolutions a minute, and the discharge began 13 minutes after the feed was started.

As the run was made to test the mechanical features of the furnace, no data were collected except assays of the heads and the discharged residue. The feeder gave satisfaction in every respect. It was expected that the charge would remain in the kiln for a longer period of time. Owing to the small diameter of the kiln and the high pressure of the gases from the burner, the finer material was frequently picked up and carried through the entire length of the kiln and caught in the dust box.

Assay of feed and discharged residue of test 1.

Material.	Assay.	
	Ag.	Pb.
	Ounces.	*Per cent.*
Feed	3.82	2.19
Residue	.98	.64

The above assays indicate that the 7-foot parallel-flow furnace should be satisfactory for this ore.

TEST 2.

This test was made on May 28, 1921, to check results obtained on the previous run and to collect data on the volatilization of the silver and lead. The charge was 60 pounds of Yellow Pine ore mixed with 3 pounds of salt.

The speed of the kiln was one revolution in 42 seconds. The feed was at the rate of 60 pounds per hour, and the temperature was about 1,000° C. The depth of the charge was about 1¼ inches. Material began discharging in 20 minutes after the feed started. The metal in the product was distributed as follows:

Distribution of metals from test 2.

Material.	Weight.	Ag.		Pb.	
		Ounces.	Per cent total.	Per cent.	Per cent total.
	Pounds.				
Heading	60	3.82	100.0	2.19	100.0
Residue	45	1.00	19.7	.64	21.9
Volatilized			80.3		78.1

About 2 pounds of dust collected in the pipe connection between the kiln and the flue. This dust was not sampled, hence the datum on the metals volatilized includes the silver and lead in the dust. These results are very satisfactory, and further work will be done to perfect the mechanical operations of the 7-foot parallel-flow furnace.

TESTS WITH RING IN KILN.

This run was made May 30, 1921, by E. P. Barrett, after a ring had been placed in the discharge end of the kiln to cut down the opening to about 3½ inches, and thereby decrease the velocity of the gases in the kiln. It was thought this feature would eliminate part of the dust losses. An opening was also cut in the side of the kiln just back of the ring, so that some residue would be discharged at every revolution of the kiln. The ring prevented discharging over the end of the kiln.

Difficulty was experienced in heating after the ring was installed, as the products of combustion frequently remained inside the furnace and prevented the gases from traveling away from the burner. The supply of oxygen to support combustion of all the oil vapors was thus cut down.

In the run made on ore from the Sultan mine at Goodsprings, Nev., the charge was 50 pounds of ore plus 3 per cent salt. The time required in the furnace for the feed to discharge was 13 minutes. The temperature in the kiln was about 1,000° C. The rate of feed was 50 pounds an hour. The distribution of metal in the product was as follows:

Distribution of values in product from test with Sultan ore.

Material.	Weight.	Ag.		Pb.		Zn.	
		Ounces.	Per cent total.	Per cent.	Per cent total.	Per cent.	Per cent total.
	Pounds.						
Heads	50	8.10	100.0	3.68	100.0	20.30	100.0
Calcine	31	4.38	33.4	1.71	28.7	25.80	76.8
Dust	4	5.66	5.6	1.62	3.5	20.30	8.0
Volatilized			61.0		67.8		15.2

The percentage of zinc volatilized in this test is thought to be due to reducing conditions inside the furnace caused by the prevention of free circulation of gases through the furnace by the ring. The extraction of silver and lead was also reduced.

SUMMARY OF REPORT.

Beginning with the discovery of losses of chloride vapors in the old chloridizing roasting methods, this bulletin outlines the history of the processes of chloride volatilization and the final recognition of the possibility of causing all the valuable metal content of an ore to chloridize and volatilize, providing an efficient device could be applied to recover the fume. The development of the Cottrell precipitator provided this device. It remained for the Bureau of Mines to put together all these facts and to revivify the older work. A complete discussion of the chemistry of the chloridizing volatilization treatment of ores is not attempted, but attention is called to the fact that most previous investigators in chloridizing felt that the addition of sulphides to the roasting charge was necessary in order to provide sulphurous gases that could act on the salt used for chloridizing, thus producing a chloridizing atmosphere. The work of the bureau on oxidized ores has shown that the presence of sulphur compounds is not only unnecessary but often is actually deleterious at the temperatures used. A more thorough study of the chemistry involved in chloridizing and volatilizing various metals is in progress.

The testing of numerous ores and the various chloridizing furnaces developed in the study of this method of ore treatment occupies the main part of this report. Numerous tests are recorded and each furnace is described. The first tests were made with mixtures of oxidized ores and salt in shallow roasting dishes placed in ordinary assayers' muffles heated to various temperatures, then in the electric-tube furnaces previously described. The horizontal rotating kiln was next tested in various modifications. It was difficult to treat the ore long enough in an ordinary rotating kiln in which the feed entered the cold end where the furnace gases discharged, and the hot ore discharged at the fire end of the kiln. Finally, abandoning for the moment principles of heat conservation, it was found that a horizontal rotating kiln with the ore fed at the hot end of the furnace and flowing in a parallel direction to the furnace gases through the kiln gave much better conditions, especially when the ore was maintained in a thin bed rather than in a thick one, allowing better aeration and providing more surface from which the volatile chlorides could vaporize.

Semicommercial tests by both parallel and countercurrent flow kilns are described, and the present condition of this method is shown to be in need of tests on a larger scale. The excellent mechanical and chemical results obtained on the "patio tailings" by a commercial plant operated by the Blaisdell Coscotitlan Co. are most encouraging.

The disposal or treatment of the fume is receiving most careful attention. It has been found necessary to install dust settlers between the furnace and the treaters to remove foreign materials from the chloride fumes. Several devices are now on the market, one that was built and set up in the laboratories at the Salt Lake station proving very effective.

On a clean fume little or no difficulty is experienced in reducing the fumes, either lead or copper, to the metallic form in the presence of proper amounts of lime or limestone and powdered coke, coal, or charcoal. Several different furnaces have been tried, and with carefully controlled temperatures a very small percentage of the metal chlorides have been volatilized in the reduction process.

Various hydrometallurgical methods are being tested, several, such as hot and cold water and acid leaches. also acidified brine, having proved feasible. The presence of zinc in the fume has a marked influence, both on pyrometallurgical and hydrometallurgical methods. Of these subjects special investigations and studies are being made.

PARTIAL LIST OF PATENTS RELATING TO THE CHLORIDE VOLATILIZATION PROCESS.

The following partial list of patents relating to the chloride volatilization process deals with the chloridization of the valuable metals and their subsequent volatilization and recovery and with the apparatus used in the process of treatment, including apparatus for electrostatic precipitation and other mechanical devices for the recovery of fumes.

Methods pertaining to volatilization in treatment of ores. 693,982, Feb. 25, 1902, Gaz. vol. 98, p. 1556. McKnight, Robert, Philadelphia, Pa., assignor to Metallic Condenser Co., a corporation of Delaware.

741,712, Oct. 20, 1903, Gaz. vol. 106, p. 1826. Pohlé, Edwin C., and Croasdale, S., Denver, Colo., assignors to Metal Volatilization Co., Philadelphia, Pa., a corporation of New Jersey.

811,085, Jan. 30, 1906, Gaz. vol. 120, p. 1251. Pohlé, Edwin C., Reno, Nev.

Process of recovering values from sulphide ores. 927,046, July 6, 1909, Gaz. vol. 144, p. 115. Hogel, Hascal A., New York, N. Y.

Method of treating ores. 1,111,976, Sept. 29, 1914, Gaz. vol. 206, p. 1256. Titus, Court C., Helena, Mont., assignor to Montana Metallurgical Co., Helena, Mont., a corporation of Montana.

Process of treating complex refractory ores of silver and gold. 1,169,530, Jan. 25, 1916, Gaz. vol. 222, p. 1211. Clawson, Seldon I., Salt Lake City, Utah.

Process of volatilizing ores. 1,192,037, July 25, 1916, Gaz. vol. 228, p. 1136. Clawson, Seldon I., Salt Lake City, Utah.

Process of volatilizing metals. 1,264,586, Apr. 30, 1918, Gaz. vol. 249, p. 1119. Wigton, George H., Eureka, Utah.

Method of treating siliceous ores. 1,305,327, June 3, 1919, Gaz. vol. 263, p. 19. Wigton, George H., Eureka, Utah.

Method of treating siliceous ores. 1,338,271, Apr. 27, 1920, Gaz. vol. 273, p. 691. Welch, Harry V., Los Angeles, Calif., assignor to International Precipitation Co., Los Angeles, Calif., a corporation of California.

Process for recovery of metals from their ores. Fume-arresting devices. 737,003, Aug. 25, 1903, vol. 105, p. 1821. McKnight, Robert, Philadelphia, Pa.

Apparatus for condensing fumes formed in volatilization processes. 796,956, Aug. 8, 1905, Gaz. vol. 117, p. 1754. Clawson, S. I., Salt Lake City, Utah.

Fume arrester. 880,506, Mar. 3, 1908, Sp. p. 29, Dr. p. 7, Gaz. vol. 133, p. 8. Clawson, S. I., Salt Lake City, Utah.

Fume arrester. 882,073, Mar. 17, 1908, Sp. p. 3197, Dr. pp. 679–680, Gaz. vol. 133, p. 582. Moffitt, John R., Denver, Colo.

Fume-condensing apparatus. 895,729, Aug. 11, 1908, Gaz. vol. 135, p. 1233. Cottrell, Frederick G., Berkeley, Calif., assignor to International Precipitation Co., San Francisco, Calif., a corporation of California.

Art of separating suspended particles from gaseous bodies. 907,379, Dec. 22, 1908, Gaz. vol. 137, p. 1808. Laurent, Louis C., Denver, Colo.

Fume-condensing apparatus. 922,260, May 18, 1909, Sp. p. 4464, Gaz. vol. 142, p. 774. Clawson, S. I., Salt Lake City, Utah.

Apparatus for condensing and purifying smoke, etc. 922,516, May 25, 1909, Sp. p. 4975, Gaz. vol. 142, p. 885. Rockliff, W. A., and Booth, J. W., Salt Lake City, Utah.

Fume arrester. 13,090, Mar. 8, 1910, Sp. p. 2700, Gaz. vol. 152, p. 466. Howard, Henry, Boston, Mass.

Fume arrester (reissue). 952,970, Mar. 22, 1910, Sp. p. 5127, Gaz. vol. 152, p. 921. Whitmore, Claude C., Butte, Mont.

Fume arrester. 972,637, Oct. 11, 1910, Sp. p. 2469, Gaz. vol. 159, p. 420. Parsons, William F., Irvine, Ky.

Fume arrester and smoke purifier. 980,257, Jan. 3, 1911, Sp. p. 148, Gaz. vol. 162, p. 29. Heslewood, William R., Berkeley, Calif., assignor, by direct and mesne assignments, to South Fork Smelting Co., Oakland, Calif.

Fume-condensing apparatus. 991,823, May 9, 1911, Sp. p. 214, Gaz. vol. 166, p. 381. de Benedictis, T., San Francisco, Calif., assignor of one-half to F. C. Amorose, San Francisco, Calif.

Fume condenser. 990,009, Apr. 18, 1911, Sp. p. 3685, Gaz. vol. 165, p. 638. Richards, George C., Berkeley, Calif.

Fume-condensing compound apparatus. 1,031,089, July 2, 1912, Gaz. vol. 180, p. 86. Royce, Lorenzo D., Oakland, and Wallerstein, L., San Francisco, Calif.

Fume arrester. 1,035,422, Aug. 13, 1912, Gaz. vol. 181, p. 376. Cottrell, Frederick G., Berkeley, Calif., and Burns, H. A., Oakland, Calif., assignors to International Precipitation Co., San Francisco, Calif., a corporation of California.

Apparatus for separating suspended particles from gaseous bodies. 1,057,783, Apr. 1, 1913, Gaz. vol. 189, p. 132. Tomkins, Alfred, Pittsburgh, Pa.

Apparatus for arresting fumes. 1,083,057, Dec. 30, 1913, Gaz. vol. 197, p. 1202 Clawson, Selden I., Salt Lake City, Utah.

Fume arrester. 1,132,124, Mar. 16, 1915, Gaz. vol. 212, p. 867. Schmidt, Walter A., and Roberts, G. C., Los Angeles, Calif., assignors to International Precipitation Co., Los Angeles, Calif., a corporation of California.

Apparatus for separating suspended particles from gases. 1,204,906, Nov. 14, 1916, Gaz. vol. 232, p. 504. Schmidt, Walter A., Los Angeles, Calif., and Bradley, L., East Orange, N. J., assignors to Research Corporation, New York, N. Y., a corporation of New York.

Method and means for recovering certain constituents from gaseous bodies. 1,218,354, Mar. 6, 1917, Gaz. vol. 236, p. 162. Baldwin, William J., Brooklyn, N. Y.

Process for the separation of dust particles and smoke from air and gases. 1,250,088, Dec. 11, 1917, Gaz. vol. 245, p. 510. Herbert, A. Burns, Los Angeles, Calif., assignor to International Precipitation Co., Los Angeles, Calif., a corporation of California.

Apparatus for electrical separation of suspended particles from gases. 1,292,561, Jan. 28, 1919, Gaz. vol. 258, p. 687. Baldwin, William J., Brooklyn, N. Y.

Apparatus for separating foreign materials from gases. 1,298,409, Mar. 25, 1919, Gaz. vol. 260, p. 658. Schmidt, Walter A., Los Angeles, Calif., assignor to International Precipitation Co., Los Angeles, Calif., a corporation of California.

Process and apparatus for separating finely divided materials. 1,302,281, Apr. 29, 1919, Gaz. vol. 261, p. 897. Bates, William H., jr., Boston, Mass.

Metallurgical smoke filter. 1,319,706, Oct. 28, 1919, Gaz. vol. 267, p. 499. Carl, Wm. J., Hedberg, New Britain, Conn., assignor to Research Corporation, New York, N. Y., a corporation of New York.

Apparatus for the electrical treatment of gases. 1,325,136, Dec. 16, 1919, Gaz. vol. 269, p. 467. Bradley, Linn, East Orange, N. J., assignor to Research Corporation, New York, N. Y., a corporation of New York.

Apparatus for electrical treatment of gas. 1,329,237, Jan. 27, 1920, Gaz. vol. 270, p. 569. Frisbie, Howard I., of Anaconda, Mont.

Electrical precipitator. 1,329,817, Feb. 3, 1920, Gaz. vol. 271, p. 88. Wolcott, Edson R., Los Angeles, Calif., assignor to International Precipitation Co., Los Angeles, Calif., a corporation of California.

Art of precipitating suspended material from gases. 1,329,818, Feb. 3, 1920, Gaz. vol. 271, p. 88. Wolcott, Edson R., Los Angeles, Calif., assignor to International Precipitation Co., Los Angeles, Calif., a corporation of California.

Method of collecting suspended material from furnace gases. 1,329,859, Feb. 3, 1920, Gaz. vol. 271, p. 96. Schmidt, Walter A., Los Angeles, Calif., and Bradley, Linn, East Orange, N. J., assignors to Research Corporation, New York, N. Y., a corporation of New York.

Apparatus for electrical treatment of gases. 1,331,225, Feb. 17, 1920, Gaz. vol. 271, p. 451. Wolcott, Edson Ray, Los Angeles, Calif., assignor to International Precipitation Co., Los Angeles, Calif., a corporation of California.

Art of precipitating suspended material from gases. 1,342,651, June 8, 1920, Gaz. vol. 275, p. 255. Squires, Harold W., Amityville, N. Y., assignor to Research Corporation, New York, N. Y., a corporation of New York.

Miscellaneous equipment. 1,067,974, July 22, 1913, Gaz. vol. 192, p. 810. Cottrell, Frederick G., Berkeley, Calif., assignor to International Precipitation Co., Los Angeles, Calif., a corporation of California.

Method of discharge of electricity into gases. 1,143,175, June 15, 1915, Gaz. vol. 215, p. 808. Cottrell, Frederick G., Berkeley, and Burns, H. A., Oakland, Calif., assignors to International Precipitation Co., San Francisco, Calif., a corporation of California.

Synchronous electrical contact maker. 1,356,462, Oct. 19, 1920, Gaz. vol. 279, p. 488. Nesbit, Arthur P., Wilkinsburg, Pa., assignor to International Precipitation Co., Los Angeles, Calif., a corporation of California.

Apparatus for the electrical precipitation of suspended matter in gaseous and fluid bodies. 1,204,907, Nov. 14, 1916, Gaz. vol. 232, p. 504. Schmidt, Walter A., Los Angeles, Calif., assignor to International Precipitation Co., Los Angeles, Calif., a corporation of California.

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