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Vacuum Distillation of Zinc and Cadmium from Zinc Leach Purification Cake

Stiles R. Slosson

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VACUUM DISTILLATION OF ZINC AND CADMIUM
FROM ZINC LEACH PURIFICATION CAKE

By
Stiles R. Slosson

A Thesis
Submitted to the Department of Metallurgy
in Partial Fulfillment of the
Requirements for the Degree of
Bachelor of Science in Metallurgical Engineering

Montana School of Mines

Butte, Montana

May 15, 1951

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TABLE OF CONTENTS

	PAGE
INTRODUCTION	1
Principles of Vacuum Distillation	3
Notes on Zinc and Cadmium	5
PROBLEM	7
VACUUM DISTILLATION OF ZINC AND CADMIUM FROM ZINC LEACH PURIFICATION CAKE	7
Preparation of the Laboratory Sample	8
Development of a Suitable Apparatus	9
Preliminary Experiments	16
Experimental Procedure	18
Test No. I	20
Test No. II	20
DATA AND CALCULATIONS	21
Test No. I	23
Test No. II	23
Results of Test No. I	23
Results of Test No. II	26
DISCUSSION AND CONCLUSIONS	26
RECOMMENDATIONS	28
ACKNOWLEDGEMENT	30
BIBLIOGRAPHY	31

VACUUM DISTILLATION OF ZINC AND CADMIUM
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INTRODUCTION

The term "vacuum distillation" is used to describe the process employed when certain elements are separated either from their mother compounds or from one another by distilling them under reduced atmospheric pressure. The use of reduced pressure makes it possible to take advantage of the vapor pressures of the various elements and effect a separation between them at lower temperatures than can otherwise be used.

This method of producing metals is comparatively new and it had its greatest development in the years during and since World War II. In the early part of the war, it became apparent that existing facilities for the production of magnesium as well as those for purification in certain phases of uranium production were not adequate and this fact led to the development of mechanical pumps, diffusion pumps, and high vacuum gages which are suitable for large scale operations in the field of "vacuum metallurgy".

A "high vacuum" means low pressure which is far lower than can be obtained by simple reciprocating

pumps and water ejectors.

The most common unit of measurement of the pressure developed is the micron, which is one thousandth of a millimeter of mercury. Basing this on the fact that one atmosphere of pressure is 760 millimeters a micron is roughly one millionth of an atmosphere. However, in this paper all pressures will be measured in atmospheres which is more familiar to the writer. It has been found in experimental work on the subject that the best results in vacuum distillation have been obtained by using pressures in the range from 100 microns to one or less.

Although a compound mechanical pump in good working order can very easily produce the desired vacuum, it is an expensive piece of equipment and its pumping capacity is very small at low pressures. Therefore, the system which is used most often to produce low pressures is an arrangement whereby a mechanical pump and an oil diffusion pump are used in series. The mechanical pump takes the pressure down to about 200 microns and the diffusion pump does the rest, the results being a larger capacity and a lower ultimate pressure than can be produced ordinarily.

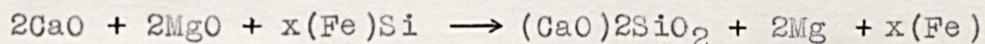
Accurate measurement of extremely low pressures is a difficult job. However, it can be accomplished

by means of the McLeod gauge in which the rarified atmosphere of an evacuated chamber is compressed into a much smaller space where it has an appreciable and measurable volume. There are various electrical gauges which give the pressure indirectly and have the advantage of giving a continuous reading.

Principles of Vacuum Distillation

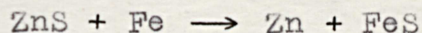
The successful separation of two or more metals by distillation depends on an appreciable difference in the vapor pressures of the metals. The vapor pressures of the common volatile metals have been fairly well established, but very little could be found concerning the vapor pressures of the compound being discussed in this paper.

The best known example of the group of metals which lend themselves nicely to vacuum distillation is furnished by magnesium in the ferrosilicon reduction of calcined dolomite according to the following reactions.



Many other elements such as calcium, sodium, lithium, and barium have been liberated by this method, and they are all produced in a manner similar to that of magnesium. However, other compounds and other

reducing agents could be used if the same conditions are obtained. Laboratory tests have been made showing that metallic zinc can be produced directly from sphalerite in a vacuum furnace according to the reaction:



The success of the process depends on the relatively high vapor pressure of zinc as compared to that of FeS or ZnS.

On the other hand, there is considerable proof that a pure metal can be gotten by applying vacuum distillation to volatilize the surrounding impurities away from the metal. Some difficulties are met in doing this such as the contamination of the product by carbon when too great an excess of it is used.

Probably the most recent advances that have been made in vacuum metallurgy have been accomplished at the Missouri School of Mines and Metallurgy where they have succeeded in removing zinc from Parkes' process crusts on a pilot plant scale. The space available in this paper does not permit a discussion of the process, but it is certain that it will be an important method of dezincing lead in the near future. **

** The bulk of the material contained in this introduction was taken from references 6,7, and 8.

Notes on Zinc and Cadmium 3&4

The atomic weight of zinc is 65.38. Dry air, or air free from CO_2 does not attack it at ordinary temperatures. The melting point is 419.40°C , and the boiling point is 920°C , but in a vacuum, zinc begins to sublime at 184°C .

The gas CO_2 begins to oxidize zinc at 700°C . What is commonly called "blue powder", is produced in the condensation of zinc vapor, and it is made up of finely divided metallic zinc, the particles being more or less coated with ZnO . It also contains such elements as Cd, Pb, As, Sb, and some Fe.

Zinc oxide (ZnO), is 80.26 % zinc. ($65.38\text{Zn} + 160 \rightarrow 81.38\text{ZnO} + 84,800 \text{ cal.}$) It occurs as zincite, a deep red to orange-yellow lustrous hexagonal mineral containing some Fe and Mn, and is formed by burning zinc. The oxide formed by burning zinc is amorphous white, changing to yellow upon being heated. The oxide is infusible, but volatile at elevated temperatures. It has been shown that ZnO is volatile above 1000°C and rapidly at 1300°C . The dissociation temperature to Zn and O_2 has been calculated as $3,817^\circ \text{C}$. ZnO is reduced to Zn by C, CO, and H_2 at elevated temperatures. The reduction by C is said to begin below the boiling point, and to

be complete at 1300° C. Reduction temperature varies with the character of the oxide and the reducing carbon.

The reaction $\text{ZnO} + \text{C} \rightarrow \text{Zn} + \text{CO}$ is nonreversible. Research by Lencauchez has shown that a gas with 99.50 % volatile CO and 0.5 % CO_2 exerts an oxidizing effect on zinc vapor at the temperature of its condensation. Sulphur has somewhat of a reducing effect on ZnO but the reaction is imperfect.

Cadmium

The atomic weight of cadmium is 112.41. Dry air does not attack the metal but moist air oxidizes it superficially. At red heat, cadmium burns to a yellowish-brown vapor.

Cadmium oxide (CdO), (87.50 % Cd, $112.41\text{Cd} + 16 \text{O} \leftrightarrow 128\text{CdO} + 66,300 \text{ cal.}$) Its volatilization is noticeable at 700° C and it attracts CO_2 from the air, is a strong base, and is readily reduced to metal by CO and C. Reduction by C begins at about 660° C. The only recognized cadmium mineral is greenockite with 77.70 % cadmium, which is honey-colored, citron-yellow to orange-yellow.

PROBLEM

The purpose of this investigation is to make preliminary tests using vacuum distillation in an attempt to remove cadmium and zinc from zinc leach purification cake, and at the same time, produce the two metals in pure enough form to allow direct marketing. It is also maybe possible to send the residue, which should be very high in Cu, to a copper smelter.

VACUUM DISTILLATION OF ZINC AND CADMIUM FROM ZINC LEACH PURIFICATION CAKE

The attempts made in this investigation to remove zinc and cadmium from purification cake could, if they had been proven successful, made the production of cadmium a much cheaper and safer process. Since most of the worlds' supply of cadmium is gotten from zinc plant residue, it is only fitting that some study be given to develop a more direct method for its liberation.

The cake which was tested in these experiments came from the purification department of the Anaconda Copper Mining Companies' zinc plant at Anaconda, Montana. According to the A.C.M. Co. assay, a typical analysis of the cake is given in Table I below.

Table I

% Zn	% Cu	% Cd	% Pb	Au oz/ton	Ag oz/ton
44.20	8.89	2.90	0.26	0.005	2.57

Preparation of the Laboratory Sample

The sample cut from the storage dump was equal to about 100 lbs and therefore the first step was to cut it down to about 25 lbs. This was done by coning and quartering. The amount of moisture contained in the cake was too high to allow easy handling so the sample was dried and then put through the rolls which were set at $\frac{1}{4}$ inches. A second pass was made with the rolls just touching. By riffing, the sample was further cut to about 10 lbs then mixed on a rolling cloth. From here, a $\frac{1}{2}$ -lb portion was taken, sent through the pulverizer, and sent to be assayed. The remainder of the sample was to serve as the heads for the actual experiments. The assay as received from the Bureau of Mines analyst, is given below in Table II.

Table II

% Cu	% Zn	% Fe	% Cd	% insol
10.56	41.80	0.50	1.60	0.40

The assay of the heads as gotten by polarographic analysis after a boiling water wash is given below in Table III.

Table III.

% Cu	% Zn	% Fe	% Cd	% insol
10.26	40.10	0.50	1.66	-----

Development of a Suitable Apparatus

Setting up a furnace with a workable condenser was one of the major problems of the investigation. After many trials with many different types of condensers it became obvious that, in this experiment which was carried out on such a small scale, it was impossible to make a satisfactory recovery of the volatilized materials.

The furnace used was an electric, tube-type furnace about 14 inches long and 6 inches in outside diameter. The electric heating coils were mounted in refractory holders about 12 inches long and 2 inches in diameter. These holders were two semi-circular shells which, when put together, formed a hollow cylinder through which a refractory tube could be placed. The furnace was connected to a 110-volt alternating current line. A rheostat with a number of different settings was placed in the circuit to enable the operator

to regulate the temperature of the furnace. With the 110-volt line as the power source, the maximum temperature obtainable after the furnace had been heating for 6 hours was about 1100° C. This temperature was high enough for the materials that were being reduced, but another 100° in the range would have been better.

The next step was to develop a chamber in which the reduction in a vacuum could be carried out. To accomplish this a sillimanite tube 24 inches long and three-fourths inches in diameter was fitted, in one end, with a rubber stopper, and the other end was connected to the condenser which in turn was connected to the vacuum pump. As previously stated, many types of condensers were tried and none of them would give the desired results. The first type of condenser was fashioned from a simple U-tube, and it did not work because it did not have enough surface area on which the vapors coming from the furnace could condense. The U-tube was connected to the reduction chamber by a piece of glass tubing in a rubber stopper which was fitted tightly in the end of the sillimanite tube.

For the purpose of denoting the pressure in the reduction chamber a manometer was put in the vacuum line in such a manner that a reading was given at all times. The manometer tube was graduated in inches and

the readings were thus given in inches of mercury.

Upon failure of the U-tube condenser, another type of condenser was made from a piece of glass tubing about 14 inches long and five-eighths inches in diameter. It was thought that by increasing the surface area a better condensation could be made, but this idea proved to be wrong. Not only did the fumes go straight on into the safety trap, but it was impossible to maintain an air-tight seal between the glass tube and the sillimanite tube so this condenser was discarded also.

The third type of condenser was made from a piece of glass tubing about 4 feet long and three-sixteenths inches in diameter. The tube was formed into a coil which could be put in a beaker of water and kept cool at all times. This condenser came closer to being satisfactory than any of the others tried because it was actually possible to condense zinc vapor. As in testing the other condensers, pure zinc in the form of zinc dust was mixed with powdered charcoal and volatilized under vacuum in the reduction chamber. The amount of zinc condensed was small in comparison with the amount volatilized, but this could be attributed to the fact that it was impossible to draw all of the vapor into the condenser due to the small opening.

Since zinc vapor condenses below 900° C, the mouth of the condenser, which was located in the cooler part of the furnace, was soon plugged up with zinc. This system, although it worked when pure zinc was volatilized, did not work when purification cake was substituted for the pure zinc. The main failure was not in the condenser itself, but the fact that there was too great an air leakage into the reduction chamber and reduction of the sample was not possible. Only after a thorough search was it discovered that the sillimanite tube had developed a series of tiny cracks which were allowing air to enter the chamber in such great quantities that the reduction medium could not combat the oxygen in it.

Since, with the type of furnace being used in the experiment, a tube had to be used for the reducing chamber, a quartz tube was next tried. The quartz tubes available in the laboratory were made from vitreous silica and their trade name is Vitreosil. A Vitreosil tube devitrifies when subjected to temperatures beyond 1100° C. This maximum temperature is lowered to 1000° C under strong reducing conditions such as were being used in these tests, therefore, the temperatures under which further experimentation could

be carried out were more or less limited to prolong the life of the tube.**

Along with the new tube, a new type condenser was developed. This last condenser was more or less a combination of the second and third types used. A piece of vitreosil 18 inches long and one-half inch in diameter was fitted with a rubber stopper in such a manner that about 6 inches of the tube could protrude into the reduction chamber to the place where the sample was to be located. On the opposite end of the small tube the coil condenser was placed, this in turn was connected to the vacuum pump. An almost air-tight seal was acquired with this set up and it was possible to maintain about .04 of an atmosphere of pressure in the chamber all the time.

The vacuum pump used was a Cenco Hy Vac oil pump manufactured by the Central Scientific Company. It is capable of reducing the pressure to a very low degree, but due to the hose connections in the hookup to the reduction chamber the lowest pressure reached was about .038 atmospheres, however this pressure could

** Taken from the instruction sheet which accompanies each new tube.

not be maintained all the time in actual practice. To make certain that no solid material could be drawn into the pump and cause damage, a trap was put in the vacuum line between the pump and the condenser. Although this was necessary to safeguard the pump, it was a source of a great deal of the air-leak in the system.

The method used to measure the temperature of the furnace was, at best, very crude. Before each run, a chromel-alumel thermocouple was placed in the tube for about ten minutes. The thermocouple was calibrated in degrees Farenheit, and since the tests were run using the Centegrade system it was necessary to convert from one scale to the other. The fact that a continuous reading could not be taken gave way to considerable error in the temperature at which a run was actually made. This was especially true when the temperature had to be raised or lowered just before a run was made.

Figure I, page 15, is a photograph of the assembled apparatus as it was finally developed.

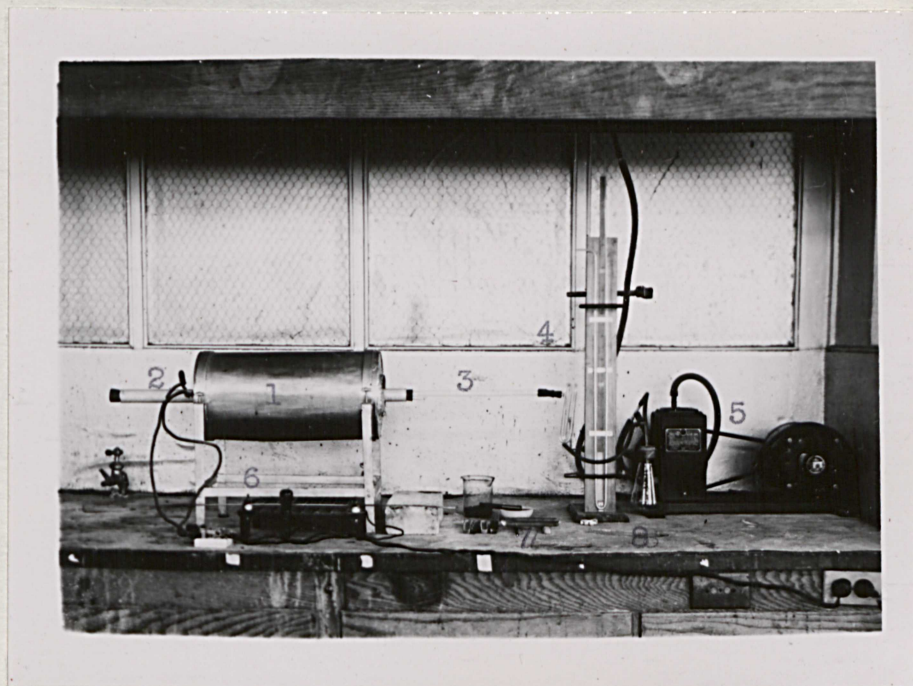


Figure I

Distillation Apparatus

- | | |
|---------------------|----------------|
| 1. Electric Furnace | 5. Vacuum Pump |
| 2. Reduction Tube | 6. Rheostat |
| 3. Condensing Tube | 7. Sample Boat |
| 4. Manometer | 8. Safety Trap |

Preliminary Experiments

The tests made on the cake were conducted on the assumption that the major constituents were present as oxides. To varify this assumption, a specimen was taken and given an X Ray analysis. The only conclusive evidence gotten was that there was a great deal of Zinc oxide present and it seemed to overshadow the other constituents.

The first tests made using the distillation apparatus were not successful. They were made on a qualitative basis to determine whether or not the process would work. A portion of the prepared sample was mixed with ground charcoal and placed in the reduction chamber in a small boat, the tube was sealed, and the vacuum pump was turned on. The temperature of the furnace was about 950° C. The greatest vacuum that could be gotten at this time was approximately 20 inches of mercury, and it did not seem high enough to aid in the reduction. It was noticed, however, that a considerable amount of moisture as well as SO₂ fumes were released. This test was performed using the first condenser and proved it was not practical.

The next test was made using a sample which had been washed in boiling water and filtered in an attempt

to remove the soluble sulphate which was thought to be causing the release of the sulphur. No apparent reduction was made during this test.

In order to ascertain whether the cake could or could not be reduced, a washed sample, after being pulverized in a mortar, was put in an induction furnace. The heat was gradually increased to about 900° C at which temperature fumes of CdO began to come off; the heat was further increased, and at about 1200° C ZnO fumes came off rapidly. The temperatures at which reduction took place meant little, but they did prove that cadmium and zinc would distill off.

With the proof that cadmium and zinc would distill from the cake, more tests were run in the vacuum furnace using both ground charcoal and ground coke. Coke did not work--probably because the temperatures used were too low.

Not until the last condenser was developed, and it was possible to obtain a vacuum of 0.04 atmospheres was much success in reducing the charge noted, but even this was not too convincing. Probably the step which was responsible for the best reduction was the one where the sample and the charcoal were pulverized to about 100 mesh and thoroughly mixed in equal portions before

being placed in the boat. This proved to be the key, for reduction could be made at temperatures as low as 700° C. (A finely pulverized charge works best in the tests made on Parkes' process crusts.⁸) The difficulty of getting a pure metal condensate still remained, however, because it seemed that with the small size sample used the constant air leak, though very small, would oxidize the zinc and cadmium vapors immediately, and only a small portion of them could be drawn into the condenser.

Upon finding it possible to reduce the cake, the next step was to run some quantitative tests to determine the effect of time and temperature. It was impossible to determine the true effect of pressure because it was necessary to maintain as high a vacuum as possible in these small-scale experiments.

Experimental Procedure

The procedure finally arrived at in making a test run is as follows:

1. A two-gram sample of the washed heads was weighed as accurately as possible on an analytical balance.
2. The weighed sample was placed in an alundum boat having a length of 5 inches, a width of

- one-half inch, and a depth of one-fourth inch.
3. The remaining space was then filled with powdered charcoal.
 4. This charge was poured into a beaker and mixed thoroughly.
 5. After reloading the boat with the mixed charge, the boat was carefully placed in the reduction chamber which was at the desired temperature.
 6. The tube was sealed and the vacuum pump was turned on. By means of a pinch cock on the hose, the air in the chamber was allowed to be slowly pumped out.
 7. When the run had been made for the desired length of time, the pump was shut off and the air was allowed to slowly return to the chamber before opening it and removing the boat.
 8. If the amount of condensate was large enough, the condenser was washed with acid to remove the condensate for analysis.
 9. The residue was prepared for analysis.

Test No. I

This set of runs was made using a two-gram sample. All charges were thoroughly mixed so that they would give consistent results.

Table IV

Run	Run Time Minutes	Pressure in. of Hg	Barometric Press., mm Hg	Temperature Degrees C
1	5:00	22.50	602.40	1000
2	10:00	22.50	602.40	1000
3	15:00	22.60	603.40	1000
4	20:00	22.60	603.40	1000
5	25:00	22.60	603.40	1000
6	30:00	22.60	603.40	1000

Test No. II

The charge for this set of runs was handled in the same manner as it was for the above test.

Table V

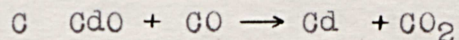
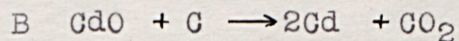
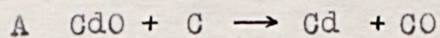
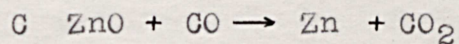
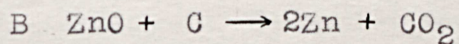
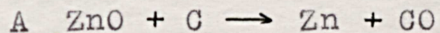
Run	Run Time Minutes	Pressure in. of Hg	Barometric Press., mm Hg	Temperature Degrees C
1	30:00	22.50	605.40	650
2	30:00	22.60	605.40	700
3	30:00	22.50	606.00	750
4	30:00	22.50	606.00	800

Table V (Cont.)

Run	Run Time Minutes	Pressure in. of Hg	Barometric Pres., mm Hg	Temperature Degrees C
5	30:00	22.50	606.50	850
6	30:00	22.50	606.50	900
7	30:00	22.50	607.60	950
8	30:00	22.50	608.00	1000

DATA AND CALCULATIONS

I-- Theoretically, there are three types of reactions that are possible in the reduction chamber and they are as follows:



Although all of these reactions undoubtedly take place in the chamber, reaction "A" probably is responsible for the bulk of the reduction. On this assumption, a charge can be calculated.

II-- By using the formula below, it is possible to calculate the actual pressure maintained in the reduction chamber during each run.

$$\frac{\text{Barometric Pressure} - (\text{Pressure in Tube})(25.40)}{760} = P$$

Test No. I

1. P = 0.0405 atmospheres
2. P = 0.0405 atmospheres
3. P = 0.0388 atmospheres
4. P = 0.0388 atmospheres
5. P = 0.0388 atmospheres
6. P = 0.0388 atmospheres

Test No. II

1. P = 0.0440 atmospheres
2. P = 0.0440 atmospheres
3. P = 0.0410 atmospheres
4. P = 0.0410 atmospheres
5. P = 0.0415 atmospheres
6. P = 0.0415 atmospheres
7. P = 0.0364 atmospheres
8. P = 0.0370 atmospheres

Results of Test No. I

The following data was gotten by polarographic analysis of the residue after each run.

Table VI

Run	Copper	Cadmium	Zinc
1	55 mg	below 0.05 mg	15.10 mg
2	99.50 mg	"	17.90 mg
3	85.00 mg	"	8.90 mg
4	67.20 mg	"	0.90 mg
5	88.50 mg	"	2.80 mg
6	90.00 mg	"	1.90 mg

A two-gram sample contains about 200 mg of copper, and by bringing all the zinc and cadmium values up to those values proportional to this figure, a smooth curve showing the trend of the zinc and cadmium left in the residue can be plotted.

Table VII

Run	Copper	Cadmium	Zinc
1	200 mg	below 0.05 mg	55.00 mg
2	200 mg	"	36.00 mg
3	200 mg	"	21.00 mg
4	200 mg	"	2.68 mg
5	200 mg	"	6.55 mg
6	200 mg	"	4.25 mg

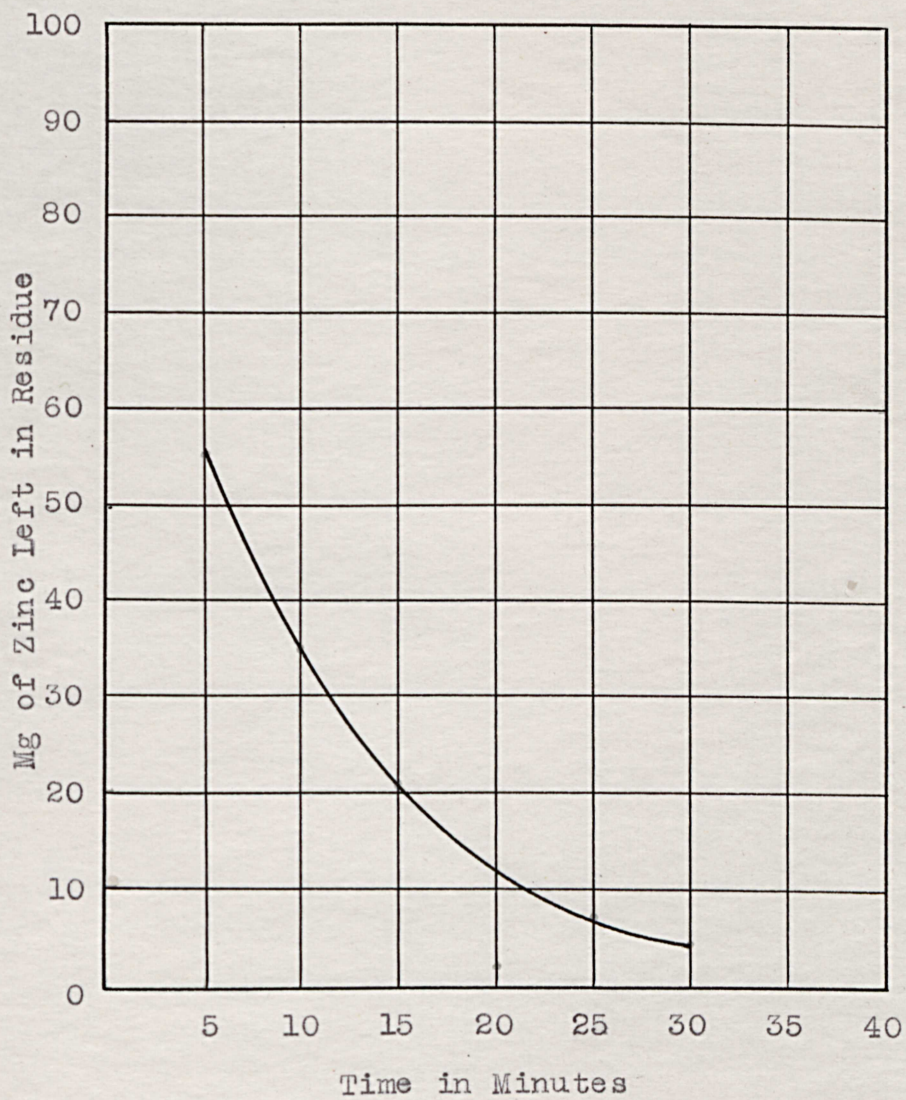


Figure II
Results of Test No. I

Results of Test No. II

The results for this test did not come out as it was hoped they would, but it is the writer's opinion that the difficulty lies in the preparation of the sample for analysis rather than the mechanics of the test.

By observing the residue after each run, it was noted that no reduction of ZnO was made below 750° C; from this temperature on, the reduction increased with the heat. It was not possible to determine whether or not CdO was reduced because of the small amount present in the original sample.

DISCUSSION AND CONCLUSIONS

A great deal of time was spent trying to develop a distillation system which would both reduce the cake and give a good condensate. However, it was never possible to get zinc and cadmium to condense in the metallic form. The exact cause for this failure is not known at this time, but it may be due to the small sample which did not contain enough zinc and cadmium to overcome the effects of the air leak. Whenever the tube was opened after a run there was always a deposit of zinc and cadmium oxide near the inlet to the condenser.

In the few instances when a large enough deposit of distillate was obtained, it was qualitatively analyzed and found to contain both zinc and cadmium. There always seemed to be a small amount of copper present but this was due to particles of the charge being carried into the condenser by the initial force of the vacuum pump taking air from the chamber.

In analyzing the residue the polarographic method was used. This did not prove too successful mainly because the samples were not prepared properly. The data of Test No. I, as has been stated, did not mean much until the results were recalculated. The graph in Figure II, Page 25, shows the desired results but the validity is questionable. It was expected that the cadmium had, for the most part, volatilized away and that the zinc would disappear as is shown.

On observing the residue after each run, it could be seen that below 750° C very little if any reduction took place; that is, it is possible and highly probable that cadmium was liberated but that zinc was not. The reduction increased as the temperature increased until at 900° C the zinc was almost all gone. The residue took on the black CuO color that was expected.

The conclusions that can be drawn from this investigation are as follows:

1. Purification cake can be reduced to liberate zinc and cadmium vapor at temperatures between 750° C and 1000° C under 0.04 atmospheres of pressure.
2. Cadmium will come off first and be deposited in the most distant portion of the condenser. Zinc vapor condenses behind the cadmium, thus the two can be separated.
3. The residue is high in Cu as was predicted. It ranged from 70% Cu to almost 98%, as gotten from Table VI, Page 24.
4. Although direct proof that the process will work is lacking, the investigator is satisfied it can be proven by further investigation.

RECOMMENDATIONS

With the limited amount of time available for this investigation, and much of that time being spent rigging up a furnace and condenser system which was not too successful, it was not possible to run the great number of tests needed to show the true effect of pressure, time, and temperature. Therefore, it is

recommended that further study be made, using a pot-type furnace with a water-cooled condenser. Also, a higher vacuum system should be tried--possibly by a combination set up as explained on Page 2. The whole experiment would give better results if it was carried out on a larger scale because the amount of vapor lost by precondenser condensation is large and by using a larger sample, more vapor would be available for collection in the correct place. A study should be made on the advisability of using natural gas continuously as a reducing agent which will also act as a suspension medium for the vapor released and carry it into the condenser.

22734

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BIBLIOGRAPHY

1. Foster, William, and Alyea, Hubert N., An Introduction to General Chemistry, Third Edition, D. Nostrand Company, Inc., New York, 1947.
2. Liddell, Donald M., Handbook of Nonferrous Metallurgy, (Recovery of the Metals), Second Edition, McGraw-Hill Book Company, Inc., New York, 1945.
3. Hofman, H. O., Metallurgy of Zinc and Cadmium, First Edition, McGraw-Hill Book Company, New York and London, 1922.
4. Ingalls, Walter Renton, The Metallurgy of Zinc and Cadmium, Second Edition, The Engineering and Mining Journal, New York, 1906.
5. Scott, G. Shaw, Editor, The Journal of the Institute of Metals, Volume VII, Institute of Metals, Caxton House, Westminster, S. W., 1922.
6. Schlechten, A. W., "Vacuum Distillation Tests Indicate A Better Way to Treat Parkes' Process Crusts", Engineering and Mining Journal, Volume 150, Number 12, December 1949, Page 80.

7. Schlechten, A. W., "Vacuum Metallurgy-- A New and Growing Industry", Engineering and Mining Journal, Volume 151, Number 7, July, 1950, Page 71.
8. Schlechten, A. W., and Doelling, R. F., "Vacuum Treatment of Parkes' Process Crusts on a Pilot Plant Scale", Journal of Metals, Volume 3, Number 4, April, 1951, Page 80.
9. Maier, C. G., "Zinc Smelting From a Chemical and Thermodynamic Viewpoint", U. S. Department of Commerce, Bureau of Mines Bulletin 324, Washington D. C., 1930.
10. Thompson, Maurice deKay, "The Total and Free Energies of Formation of the Oxides of Thirty-Two Metals", The Electrochemical Society, Inc., New York, 1942.