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THE CHLORIDE VOLATILIZATION OF SILVER AND LEAD  
FROM ZINC CONCENTRATE.

BY

EDWARD PHILLIP BARRETT.

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A

THESIS

submitted to the faculty of the  
SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI  
in partial fulfillment of the work required for the  
Degree Of  
METALLURGICAL ENGINEER

Rolla, Mo.

1924.

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Approved by

*Chas. J. Clayton*

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## PREFACE.

This thesis is presented to the faculty of the School of Mines and Metallurgy of the University of Missouri in partial fulfillment of the work required for the Degree of Metallurgical Engineer, and consists of excerpts from Bull. 211 of the Bureau of Mines, Department of the Interior, entitled, "The Chloride Volatilization Process of Ore Treatment," by Messrs. Varley, Barrett, Stevenson, and Bradford, copy of which is attached hereto, for reference as to history, purpose and scope of experimental work carried on by the Bureau of Mines, and the application of the chloride volatilization process to various ores. The extracts from the bulletin cover a portion of the work conducted by the author on the chloride volatilization process while he was attached to the Intermountain Station of the Bureau of Mines at Salt Lake, and include laboratory experiments and plant operations carried on by him personally or under his direction during 1918, 1919, and 1920. He is indebted for their assistance and cooperation to the members of the Bureau of Mines and cooperating companies interested in this investigation.



EXCERPTS FROM  
"THE CHLORIDE VOLATILIZATION PROCESS OF ORE TREATMENT"  
BY  
Thomas Varley, Edward P. Barrett, C. C. Stevenson,  
and R. H. Bradford.<sup>1</sup>

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<sup>1</sup> Bull. 211, U. S. Bureau of Mines, 1923, copy attached hereto.

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THE CHLORIDE VOLATILIZATION OF SILVER AND LEAD  
FROM ZINC CONCENTRATE.

BY

EDWARD PHILLIP BARRETT.

---

SILVER AND LEAD FROM ZINC CONCENTRATE FROM GOODSPRINGS, NEVADA.

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 described on Page 37 of Bull. 211. 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 1 and 2 were compiled by Mr. Barrett from data on these tests.

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 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.

TABLE 1.

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

Test No.	Date	Weight			Time		Temperature	Volatilized		Collected		Loss	
		Ore	NaCl	CaCl <sub>2</sub>	Hrs.	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	...	..	1000	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 2.

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.		
	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

### Experiments in the 20-foot kiln.

A careful test was made to check test 5 (Page 70 of Bull. 211) 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  $980^{\circ}$  C.

1.30. Discharge began  $933^{\circ}$  C.

2.00. Feed all in  $1,037^{\circ}$  C.; 40 pounds overflow refeed to kiln.

2.15. Temperature,  $1,021^{\circ}$  C.

2.40. Grab sample taken just as charge reaches junction.

3.20. Temperature, 963° C.

3.45. Shut down; 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 of 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.

The small-scale and semicommercial volatilization experiments by E. P. Barrett on the silver-lead-zinc ores from the Yellow Pine mine, Goodsprings, Nevada, gave such favorable results that the Yellow Pine Mining Co. offered to the Bureau of Mines the use of their plant at Goodsprings, Nevada, in which to make volatilization tests on a commercial scale. (See Plate I.) 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.

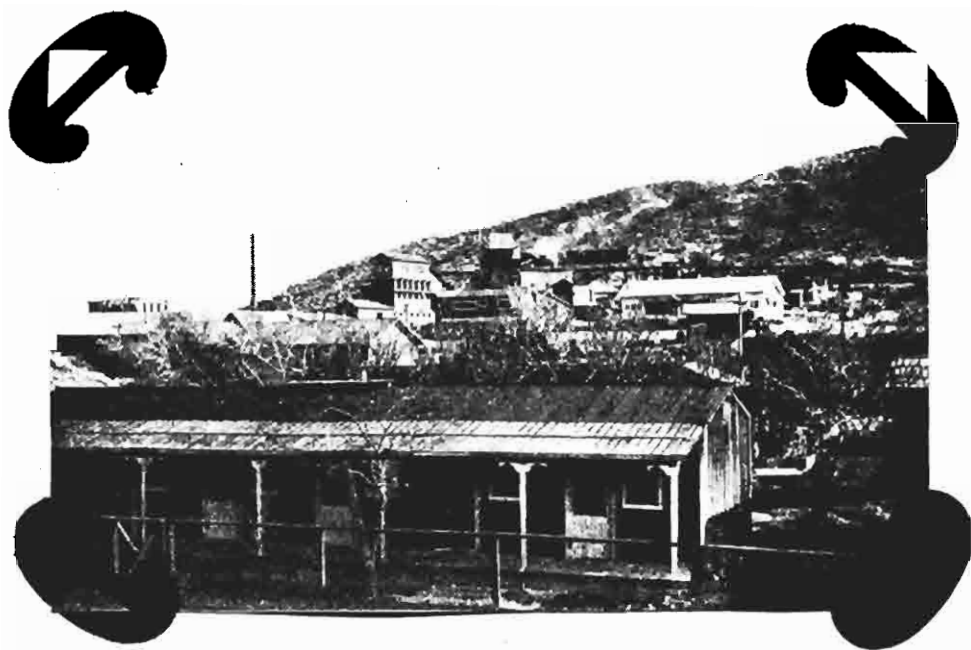


PLATE I.

General view of Yellow Pine Mill, Goodsprings, Nev.

The Yellow Pine Mining Co. operates a 75-ton mill and calcination plant at Goodsprings, Nevada; 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 II. 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 III 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 mois-



PLATE II.

Cottrell treaters, Yellow Pine Plant, Goodsprings, Nev.



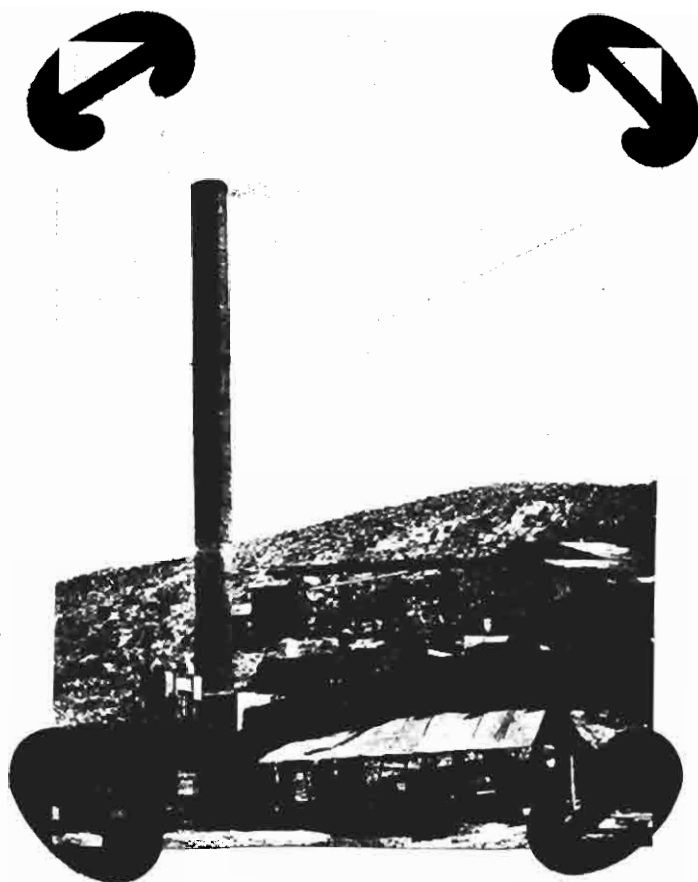


PLATE III.

Chloride fumes escaping through stack, at base of which is a small unit of a Cottrell precipitator, Yellow Pine Plant, Goodsprings, Nev.

ture, 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 <sub>4</sub> .....do..... 6.34
Cu.....do..... .44	Cl.....do.....29.12
Zn.....do..... 6.30	H <sub>2</sub> O.....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 of 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

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
	Cunces	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..	Insoluble....do..... 1.0
35.6	



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.

### 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 1 showing the averages of the series. The ore treated was the zinc concentrates from the Yellow Pine mill, Goodsprings, Nevada.

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<sup>o</sup> 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 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<sup>o</sup> C.

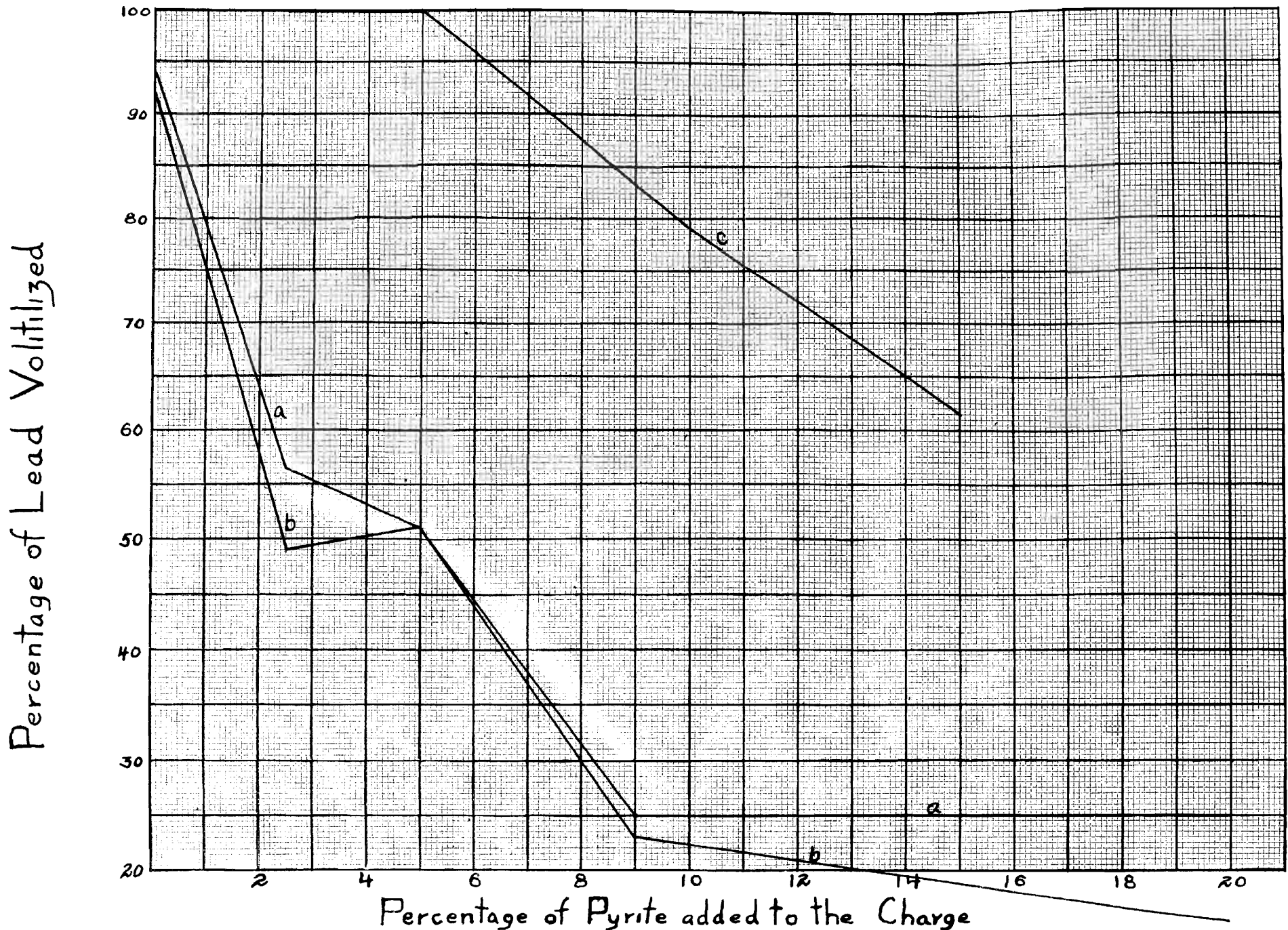


Fig 1.-Influence of chloride Volatilization of lead: a, 6% lead, 5.9% salt; b, 2% lead, 2.5% salt; c, 2% lead, 5% salt.

shows that time is not the most important factor.

The author 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.

Test 1 with thin bed of ore in Brückner furnace.

November 22, 1920.

Time a.m.	Operation	Tem- pera- ture ° C.	Assay		
			Ag. oz.	Pb. %	Cl. %
9.45	Fire started				
10.45	Ore charged plus 10% 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

Test 2 with thin bed of ore in Brückner furnace.

Time p.m.	Operation	Tem- pera- ture ° C.	Assay		
			Ag. oz.	Pb. %	Cl. %
1.25	Ore charged plus 10% 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

Test 3 with thin bed of ore in Brückner furnace.

November 23, 1920.

Time p.m.	Operation	Tem- pera- ture °C.	Assay		
			Ag. oz.	Pb. %	Cl. %
1.15	Fire started				
1.45	Ore charged plus 10% 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	3.05
2.40	Shut down, grab 4	981	1.92	.45	2.50

Test 4 with thin bed of ore in Brückner furnace.

Time a.m.	Operation	Tem- pera- ture °C.	Assay		
			Ag. oz.	Pb. %	Cl. %
3.00	Ore charged plus 10% 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.

Test 5 made in 20-foot kiln with rapid heating.

Time a.m.	Operation	Tem- pera- ture °C.	Assay		
			Ag. oz.	Pb. %	Cl. %
9.20	Fire started				
10.15	Ore feed started, 10% 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:<sup>1</sup>

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<sup>1</sup>Hofman, O., Hydrometallurgy of silver, 1907, p. 20.  
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"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."

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 does when roasting on a large scale in the furnace."



Blamey Stevens<sup>2</sup> writes in the Mining and Scientific

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<sup>2</sup>Stevens, Blamey, The chloride-volatilization Process: Min. and Sci. Press, vol. 119, 1919, p. 45.  
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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<sup>3</sup> states, August 9, 1919:

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<sup>3</sup>Croasdale, Stuart, The chloride-volatilization process: Min. and Sci. Press, vol. 119, 1919, p. 183.  
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"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."

February 21, 1920,<sup>4</sup> 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."

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<sup>4</sup>Croasdale, Stuart, Chloridizing process: Min. and Sci. Press, vol. 120, 1920, p. 259.  
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Curves are given (Fig. 2) 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.

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 1000° C. The thickness of ore bed was about  $4\frac{1}{2}$  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 1000° C. The thickness of the ore bed was about  $1\frac{3}{4}$  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 1000° C. The thickness of ore bed was about one-half an inch.

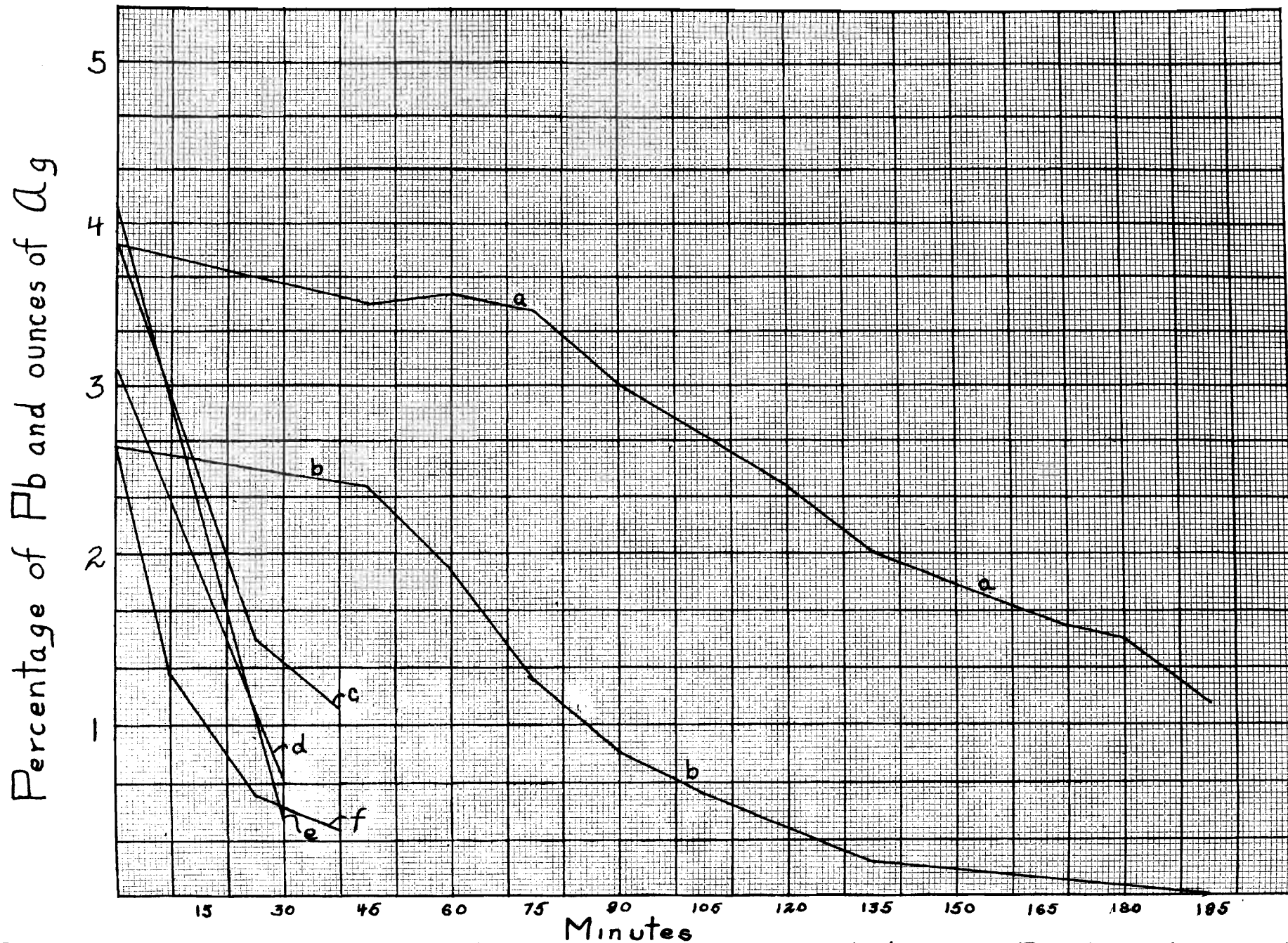


Fig 2.- Comparison of average assays of grab samples taken during tests: a & b, Brückner furnace, thick bed; d & e, muffle tests; c & f, Brückner furnace, thin bed.

### 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, California. This furnace had a scoop feeder to admit the crude ore, previously mixed with salt, into the heated end of the furnace. (See Plate IV) 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

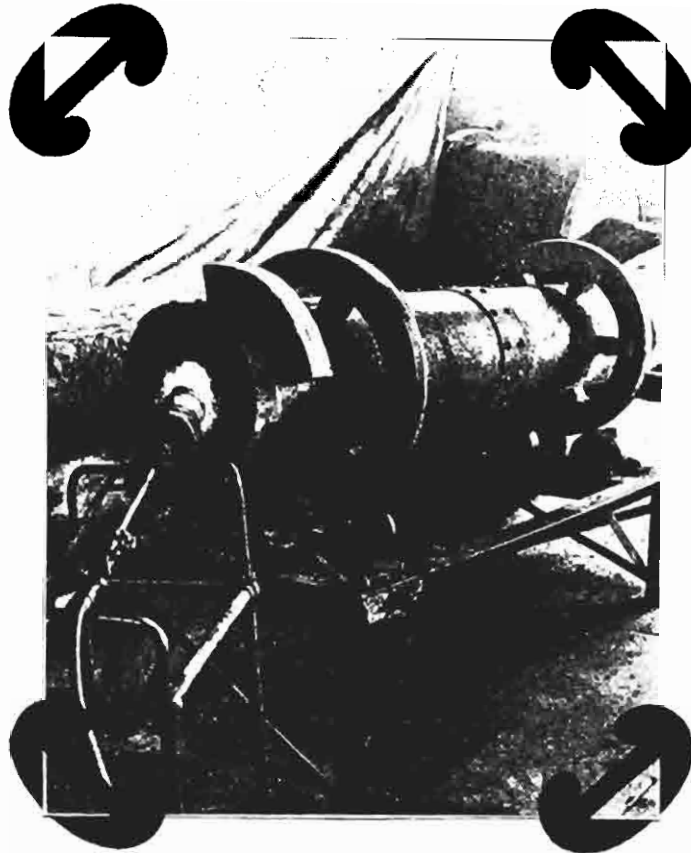


PLATE IV.

Rotary kiln, showing scoop feeder at fire end of furnace, built after Kirk's design.

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 . oz.	Pb. %
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 1000° C. The depth of the charge was about 1-1/4 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 lb.	Ag.		Pb.	
		Oz.	% total	%	% total
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  $3\frac{1}{2}$  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, Nevada, 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  $1000^{\circ}$  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 lb.	Ag.		Pb.		Zn.	
		oz.	% total	% total	% total	% total	
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.

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