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## Hydro-electric treatment of Joplin-Miami zinc concentrate

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Hydro-Electric Treatment of Joplin-Miami Zinc Concentrates

By

David Francis Walsh

A

Thesis

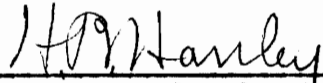
submitted to the faculty of the  
School of Mines and Metallurgy of the University of Missouri  
in partial fulfillment of the work required for the

Degree of  
Master of Science  
in  
Metallurgy

Rolla, Mo.

1924

approved:

  
\_\_\_\_\_  
Assoc. Prof. of Metallurgy.

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## Part 1.

### General

#### 1. Present Practice in the Zinc Industry:

At the present day there are three principal methods by which zinc may be extracted from its ores: the Belgian retort process, the electric smelting process, and hydro-metallurgical treatment. The recovery of zinc by the Belgian retort process is admittedly awkward and expensive, and, yet, the bulk of the zinc used throughout the world is produced by that method. It seems odd that zinc ores should still be smelted on the scale that they are in the retort process: small unit charge, low thermal efficiency (12.%), short life equipment, and only eighty-five per cent (average) zinc recovery. Furnaces for reducing and distilling zinc from its roasted ores using electricity as the source of heat (slagging and dry distillation types<sup>1</sup>) have been designed, tested, and in a

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<sup>1</sup> De Laval Furnace (slagging); Fulton's Electrothermic Dry Distillation Furnace.

number of places put into practice; but, only in Norway and Sweden, where electric power is unusually cheap, have they met with much success, and, then, there still remains the difficulty of zinc vapor condensation. Considerable experimental work is being done on this problem and it seems evident from the results obtained so far that in the near future condensation will fade away as a "ghostly" question. Hydro-metallurgic treatment as is known to-day consists in the process of lixiviation, dissolution of zinc from its ores by chemical reagents, followed by the precipitation of metallic zinc, either by chemical or electrolytic reaction. Chemical precipitation is only of slight importance, being used chiefly for the production of zinc compounds and not metallic zinc; it is the latter method, electrolytic deposition, that offers the better means of solution for building up a commercially economical process for the recovery of zinc -- a process to take the place of the Belgian retort process.

While the hydro-electric metallurgy of zinc has

been practiced for over thirty years\*, not enough was

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\* at Winnington, England, e.g.; Bruner, Mond & Co.

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generally known about working details until the plants of the Anaconda Copper Company<sup>1</sup> and the Consolidated Mining and Smelting Company<sup>2</sup> installed their present electrolytic processes.

## 2. The Future of Hydro-Electric Treatment:

\*\*\*New conditions are arising, which will bring

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\*\* W.E.Greenawalt, Trans. A.I.M. & M.E., January 1924,  
'Greenawalt Electrolytic Copper Extraction Process.'

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the wet method into prominence; while freight rates and increasing cost of fuel will tend to limit smelting to highly favored localities. The possibilities offered by the installation of hydro-electric plants and the greatly enlarged range of power transmission will greatly widen the field of electrolytic methods.

\*Electrolytic methods are ideally adapted to hydro-electric development, for the load is constant thru-

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1 Anaconda Copper Co., Great Falls, Montana, - 1915

2 Consolidated Mining & Smelting Co., Trail, B. C.

out the full twenty-four hours, and the cost of both installation and operation of hydro-electric plants is not appreciably greater when operating for twenty-four hours than when operating for eight or ten hours; whereas, in fuel-power generation, as also in smelting, the cost is more or less in proportion to the fuel and power used. Nevertheless, fuel-power generation presents advantages in electrolytic methods, as the power plant may be located in a favorable place while the leaching and electrolytic plants may be located at the mine."

What Greenawalt has said when speaking on the future of copper hydro-metallurgic treatment may also be said as regards the outlook for the development of the hydro-electric treatment of zinc ores. Electricity stands out to-day as the most important and cheapest source of energy. Its utilization as such is still in its youth. We can only dream of the possibilities which might develop from its greater use.

There is no doubt but that electric- and electro- methods will shortly prevail in all branches of metallurgy.

One of the great faults found with the retort process is its low thermal efficiency -- a high consumption of coal or coke, both as a fuel and as a reducer. Electric smelting, as in the case of the retort practice, requires a reducing agent, carbon in the form of coke, which weighs heavily in the cost column at this day of high-costing coke. While cheap electric power, in conjunction with relatively high fuel cost, is a favorable condition for economic electric smelting (e.g., Fulton's Electrothermic Dry Distillation Furnace), power consumption is a function of the amount of furnace charge, whereas the power consumption of electrolysis is a function of the amount of zinc deposited. With this in mind we see a probable reason for the recent popularity as regards the advantages of hydro-electric treatment.



Looking over the list of hydro-electric operators in the zinc industry do-day, amongst those outstanding -- Anaconda Copper Company, output: 150 tons per day; Consolidated Mining and Smelting Company, output: 75 tons per day; and the Electrolytic Zinc Company of Australasia, output: 100 tons per day -- we see that the extraction of zinc from ores by the hydro-electric process, acid leaching and electrolytic deposition, is now a well established process. Of course, this process like all others that completely revolutionize an old established industry, being still in its infancy, cannot as yet be economically operated save on a large scale.

World Electrolytic Zinc Production for the year 1923.

United States	75,000
Consolidated Mining & Smelting Co. TRAIL, B.C.	46,000
Electrolytic Zinc Company of Australasia	50,000
England	10,000
Miscellaneous	10,000
	<hr/>
Total -- (short tons) -	191,000

Average Prices for Zinc (1917 - 1922)

	1917	1918	1919	1920	1921	1922
Grade A	14.0	11.1	8.3	8.4	5.6	6.0
Prime						
Western	8.9	8.0	7.0	7.8	4.7	5.7

Note: above statistics as per Siebenthal and Stall.

Production of Zinc in the United States (1917-1922)

(short tons)

	1917	1918	1919	1920	1921	1922
Electrolytic	25,209	38,916	27,056	51,626	11,559	55,369
Distilled, Primary	644,364	479,011	438,687	411,751	188,941	298,908
Distilled, Secondary	16,835	9,918	19,748	21,371	17,573	32,988
Total -	<u>686,408</u>	<u>527,845</u>	<u>485,491</u>	<u>484,748</u>	<u>218,073</u>	<u>387,265</u>

World Production of Zinc: Apportioned according to grade:

	1917	1918	1919	1920	1921	1922
Grade A	97,708	129,344	45,377	80,713	24,084	67,459
B (inter- mediate)	69,189	68,987	39,173	33,893	4,996	36,402
Brass Special	148,749	9,854	140,917	59,811	47,814	49,980
Prime Western	370,763	230,093	260,024	310,331	141,179	233,424
Total -	<u>686,409</u>	<u>438,278</u>	<u>485,491</u>	<u>484,748</u>	<u>218,073</u>	<u>387,265</u>

Note: Above statistics as per Siebenthal and Stall.

### 3. Statement of Problem:

#### A. Zinc Ferrate:

In the pioneer days of the electrolytic zinc process it was observed that a fairly large amount of zinc would be readily leached from the calcines by dilute sulphuric acid but that the remainder (sometimes a very appreciable amount), was dissolved only by boiling in dilute sulphuric for long periods. At first, this retarded solution was attributed to the protective action of a coating of lead sulphate which often formed around the zinc particles, but this idea was soon proven false, for upon removing this lead sulphate "protective coating" (by treatment with sodium acetate solution), and re-treating with dilute sulphuric no increase in solubility of zinc was obtained.

Upon investigation<sup>1</sup> this insoluble-in-cold-dilute-sulphuric-acid residue was found to be

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<sup>1</sup> Hamilton, Murray, and McIntosh, Canadian Mining Inst. Bulletin, July 1917.

soluble in boiling acid in the ratio of one atomic weight of zinc to two of iron, approximately. The iron and zinc was found to exist as an oxide compound of the formula<sup>1</sup>  $ZnO \cdot Fe_2O_3$ .

Very little information is available as regards the formation of insoluble compounds in the roasting of zinc ores other than those of the complex type, such as marmetite (where the iron sulphide occurs in solid solution with the zinc sulphide as an isomorphous form), -- and that is only general as regards influencing conditions -- hence, it was thot advisable to investigate the formation of this insoluble compound, zinc ferrate, especially in the roasting of the ores with which we were directly concerned -- zinc concentrates from the Miami-Joplin District.

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1 Prost, Bulletin de l'Assoc. Belge des Chimistes, thru Ingalls: Met. of Zinc and Cadmium.

B. Flotation Concentrates from the

Miami-Joplin District:

In the Missouri-Oklahoma district there are tons of flotation concentrates produced running 59% (average) zinc, 3. - 4% lead, and 0.34% cadmium which is now being shipped to the smelters to be treated in the retort process plants. At the smelter this concentrate is subject to a "fines penalty charge" of \$1.06 per ton. Further, altho containing other metals than zinc, no attempt is made to recover these as by-products. In addition, it might be said that the entire production of jig concentrates and table concentrates, which at present are treated in the retort plants (where a zinc recovery of only 85% is made), are open for improvement as regards their process of treatment.

Are these concentrates amenable to commercially economical treatment by the hydro-electric process? Can such a process be placed on a competitive basis with present practice, due allowance being accredited to the former by virtue of increased recovery of zinc and increased returns thru the recovery of by-products? Such are the problems in brief confronting investigation and for which answers and solutions were sought.

## Thirty Leading Shippers of Zinc Concentrates

### Joplin-Miami District

Name	(For the year 1923)	Tonnage
Golden Rod M & S Co (Okla)		31,762
Commerce M & R - Webber		31,324
St. L. S & R Co (Okla)		31,212
Eagle-Picher Lead Co (Okla)		27,480
Underwriters Land Co (Okla)		23,225
Skelton L & Zn Co (Okla)		23,113
Vinegar Hill Zn Co - Barr (Kans)		21,185
Barnsdall Zn Co (Kans)		15,915
Butte-Kansas M Co (Mo)		15,787
Chanute Spelter Co (Kans)		15,496
Admiralty Zn Co (Okla)		13,107
Huttig L & Zn Co (Okla)		13,106
Federal M & S Co - Brewster (Okla)		12,410
Dorothy Bell M Co (Okla)		12,049
Commerce West Side (Kans)		10,609
Bilharz M Co (Okla)		10,533
Vinegar Hill - Texas (Okla)		10,147
Rialto M Co (Okla)		10,295
Quapaw M Co - Chubb (Okla)		9,748
Okla. Woodchuck		9,534
Black Eagle M Co (Okla)		8,767
Anna Beaver M Co (Okla)		8,160
Lion M Co (Okla)		7,594
Domado M Co		7,402
Vantage M Co (Okla)		7,193
Amer. Zn L & S Co (Mo)		7,191
Loweyer M Co		7,091
Queen Esther M Co		6,383
Premier Zn Co		6,293
United Zinc - Manhattan		6,203

For the year 1923 there was produced 649,000 tons from the tri-State district, an average of 1,800 tons per day. This was the largest yearly production ever made from this district — 125,000 tons greater. A purchase of 688,545 tons was reported. This includes the above production figure and also storage piles, etc., from the district.

For the period January 1st to April 29th, 1924,

Thirty of the Leading Producers report the following:

Name	tonnage
St. L. S & R Co (Okla)	20,529
Federal M & S Co (Okla)	12,129
Commerce M & R Co (Okla)	34,266
Eagle-Picher Lead Co (Okla)	11,270
Golden Rod M & S Co (Okla)	10,336
Vinegar Hill (Okla)	7,836
Skelton L & Zn Co (Okla)	7,803
Underwriters Land Co (Okla)	6,838
Butte-Kansas (Mo)	6,582
Castey Kring Brandt - N.Y.	5,362
Quapaw M Co (Okla)	4,742
Anna Beaver (Okla)	4,633
Barnsdall Zn Co (Mo)	4,361
Admiralty Zn Co (Okla)	4,538
Chanute Spelter Co (Kans)	4,484
Dorothy Bell M Co (Okla)	4,236
Huttig L & Zn Co (Okla)	3,020
Domado M Co	2,867
White M Co	2,834
Bilharz M Co (Okla)	2,761
United Zn Co	2,455
Vantage M Co (Okla)	2,376
Cherokee L & Zn Co (Kans)	2,261
Wade M Co (Kans)	2,152
Commonwealth M Co (Okla)	2,132
Black Eagle M Co (Okla)	2,126
Lowyer M Co	2,103
Lion M Co (Okla)	2,065
Amer. L Zn & S Co (Mo)	2,073
Amalgamated Zn Co (Okla)	2,070

The market on zinc concentrates (60.0% Zn) listed  
\$38. as of May 1st, 1924.

(The figures on Tri-State production as listed above and on  
preceding page were taken from the Joplin Globe paper.)



#### 4. Method of Attack:

A. In order to determine the influence of time, temperature, percentage of iron, and degree of mixture on the formation of ferrate, a series of tests were made with these factors varying. Chemical analyses were made on the resulting calcines to determine the relative percentages formed of oxide zinc, sulphate zinc, and insoluble-in-dilute-sulphuric-acid zinc, i.e., zinc as ferrate.

B. Experimental work, consisting of roasting the concentrates, leaching the calcines, and purification of the electrolyte, was performed on concentrates from seven different mills in the Miami-Joplin district; and, from data obtained, a process and equipment was outlined for a proposed plant to treat this type of zinc ore.

As Greenawalt says, there is a prevailing idea that the hydro-electric process is only applicable to the treatment of complex ores -- and not without reason, for to-day the only commercial application of

hydro-electric metallurgy is in the treatment of ores which have been found not amenable to retort practice. But why limit it to complex ores? With the idea in mind that there was no apparent reason why the hydro-electric process could not be adapted to the treatment of Miami-Joplin zinc ores and a recovery made of the metallic values (other than zinc) as by-products, a series of tests were made experimenting with the main features of such a process on these ores.

The experimental work embodied in this thesis was performed under the supervision of Professor H. R. Hanley in the metallurgy laboratories of the School of Mines and Metallurgy.

Altho this is not the proper place for acknowledgements, the writer wishes to take this opportunity to thank Professor Hanley for his aid and kindly suggestions. To his practical and experienced advice is to be accredited the organization under which "Commercial Applications" was set forth.

## Part 11.

### Experimental Investigation

The problem being of a two-fold nature, it is best that the discussion of the procedure be handled under two distinct headings. Under "Roasting" the experimental work, together with results and conclusions, concerning the formation of zinc ferrate will be outlined. Under "Joplin-Miami Flotation Concentrates" the experimental work performed on the concentrates from the seven different mills in the S.W. Missouri-Oklahoma district will be covered.

#### 1. Roasting:

Sulphide ores must be roasted to convert the zinc into zinc oxide and sulphate so that it can be dissolved in dilute sulphuric acid solution. There are three general schemes possible for roasting this material: (1), the oxidizing roast wherein there is complete elimination of sulphur, the zinc remaining as zinc oxide; (2), the

sulphatizing roast, the object of which is to oxidize all of the zinc sulphide to zinc sulphate; and (3), a roast, the degree of oxidation varying between the above two. Zinc oxide is readily soluble in dilute sulphuric acid solution and zinc sulphate is readily soluble in water. While the second scheme gives a product which, in the leach, would greatly reduce the acid consumption, still, on account of the difficulty of temperature control and thoro removal of sulphide zinc in roasting, together with the fact that there would be an excess of regenerated acid developed in the cells that could not be utilized, there is a tendency in commercial practice to effect this third scheme (obtaining both the sulphate and oxide). In so doing, the risk of leaving unoxidized sulphide in the calcines is minimized. This is greatly desired, for one pound of sulphur left as zinc sulphide holds up two pounds of zinc in the leach.

Against this there is the fact that for every pound of zinc roasted to zinc oxide one and one-half pounds of sulphuric acid must be used, whereas with zinc sulphate no acid is required. The loss of zinc which might be incurred thru inefficient oxidation of zinc sulphide is the factor which has caused the change in methods of roast -- from the sulphate roast to an almost complete oxide roast (to four per cent Zinc as  $ZnSO_4$  content average). As regards the acid necessary to leach this high oxide thus formed, there is no increased cost inasmuch as the anode-regeneration of sulphuric acid produces sufficient acid to leach effectively. Poor recovery in hydro-electric practice is attributed mainly to the difficulty of roasting the ore without the formation of ferrate of zinc; hence the advisability of determining the effect of iron (in different forms) on the formation of this insoluble compound when roasting for acid leach.

Procedure:

Seven series of roasting tests were performed using a constant weight of iron and varying the amount of zinc in increasing increments. Zinc and iron were introduced into the admixtures in the forms as shown below in the six combinations:

ZnO	plus	Fe <sub>2</sub> O <sub>3</sub>
ZnO	plus	FeS <sub>2</sub>
ZnS	plus	Fe <sub>2</sub> O <sub>3</sub>
ZnS	plus	FeS <sub>2</sub>
Zn (dust)	plus	Fe <sub>2</sub> O <sub>3</sub>
Zn (dust)	plus	FeS <sub>2</sub>

The weighed quantities of each combination were carefully triturated in an agate mortar, placed each in a four-inch roasting dish and roasted in an oil-burner muffle. Fairly accurate temperature control was maintained thruout all roasts, iron-constantin thermocouples previously standardized being used, one in each furnace placed in a most representative uniform-heat distribution position on the floor of the muffle.

Eight hour roasts were made on most of the series. Rabbling was performed as stated on separate series logs.

Upon completion of the roasts dishes were removed, calcines weighed then ground in agate mortar, and placed in marked paper bags for future reference.

Soluble zinc was determined by ammonia chloride (solution: 200 gms.  $\text{NH}_4\text{Cl}$ , 500 cc.  $\text{NH}_4\text{OH}$ , 750 cc.  $\text{H}_2\text{O}$ ), 30 minutes at 80 degrees Centigrade, leach.

Sulphate zinc was determined by hot water leach.

Zinc remaining in the residue left from ammonia chloride leach was figured by subtracting "Soluble zinc" from total zinc in calcines, this result being termed "zinc as ferrate".

With the data thus obtained curves were plotted to graphically show the effect of time of roast, temperature of roast, excess of zinc present, and degree of prior mechanical mixture on the per cent of ferrate formed.



Series No. 1. a.

ZnO and Fe<sub>2</sub>O<sub>3</sub> ; 8 hours at 600 deg. C.; no rabbling.

Sample No.	Gms. ZnO	Gms. Fe <sub>2</sub> O <sub>3</sub>	%excess zinc	Gms.Zn. in mix.	Weight calcines	NH <sub>4</sub> Cl sol. zinc, %	Gms. sol. Zn.	Gms. insol. Zn.	Per cent Ferrate formed.
48	4.86	8.0	20	3.90	12.80	26.40	3.38	0.52	16.0 (?)
49	5.67	8.0	40	4.56	13.62	22.50	3.06	1.50	46.3
51	7.29	8.0	80	5.83	15.22	26.40	4.02	1.83	56.4
52	8.10	8.0	100	6.50	16.07	33.80	5.42	1.08	33.2

Series 1. b.

ZnO and Fe<sub>2</sub>O<sub>3</sub> 6 hours at 600 deg. C. No rabbling.

Sample No.	Gms. ZnO	Gms. Fe <sub>2</sub> O <sub>3</sub>	%excess zinc	Gms. Zn. in mix.	Weight calcines	NH <sub>4</sub> Cl sol. zinc, %	Gms. sol. Zn.	Gms. insol. Zn.	Per cent Ferrate formed
7	4.05	8.0	00	3.25	12.05	19.05	2.3	0.95	29.2
8	4.46	8.0	10	3.58	12.25	20.50	2.51	1.07	32.9
9	4.86	8.0	20	3.90	12.75	22.05	2.81	1.09	33.0
10	5.26	8.0	30	4.23	13.25	19.40	2.57	1.66	50.9
11	5.67	8.0	40	4.56	13.75	20.10	2.77	1.79	55.1
12	6.08	8.0	50	4.88	14.00	23.90	3.35	1.53	47.0
13	6.48	8.0	60	5.20	14.25	25.60	3.65	1.55	47.7
14	6.88	8.0	70	5.52	14.75	25.10	3.70	1.82	56.0
15	7.29	8.0	80	5.85	15.25	29.50	4.50	1.35	41.5
16	7.69	8.0	90	6.17	15.75	31.50	4.96	1.21	37.2
17	8.10	8.0	100	6.50	16.25	33.70	5.48	1.02	31.4
18 <del>x</del>	4.05	8.0	00	3.25	12.05	24.00	2.89	.36	11.0
19 <del>x</del>	4.05	8.0	00	3.25	12.05	26.40	3.18	.07	2.1
20 <del>x</del>	4.05	8.0	00	3.25	12.05	26.80	3.22	.03	0.9
21 <del>x</del>	4.05	8.0	00	3.25	12.05	26.80	3.22	.03	0.9

~~x~~ numbers 18, 19, 20, and 21 were roasted for 6 hours at 500 deg.C, 400 deg.C, 300 deg.C, and 200 deg.C., respectively.



Series No. 11. a

Zn (dust) and Fe<sub>2</sub>O<sub>3</sub> 8 hours at 600 deg. C. fair rabbling.

Sample No.	Gms. Zn. dust	Gms. Fe <sub>2</sub> O <sub>3</sub>	%excess zinc	Gms. Zn. in mix.	Weight calcines	NH <sub>4</sub> Cl sol. zinc, %	Gms. sol. Zn.	Gms. Insol. Zn.	Per cent Ferrate formed
59	3.27	8.0	00	3.25	11.70	2.18	.25	3.00	92.3
60	3.61	8.0	10	3.58	12.00	3.12	.37	3.21	98.8
61	3.93	8.0	20	3.90	12.40	4.21	.52	3.38	104.0
62	4.26	8.0	30	4.23	12.84	6.21	.80	3.43	105.5
63	4.59	8.0	40	4.56	13.12	8.75	1.15	3.41	104.8
64	4.92	8.0	50	4.88	13.60	10.75	1.46	3.42	105.1

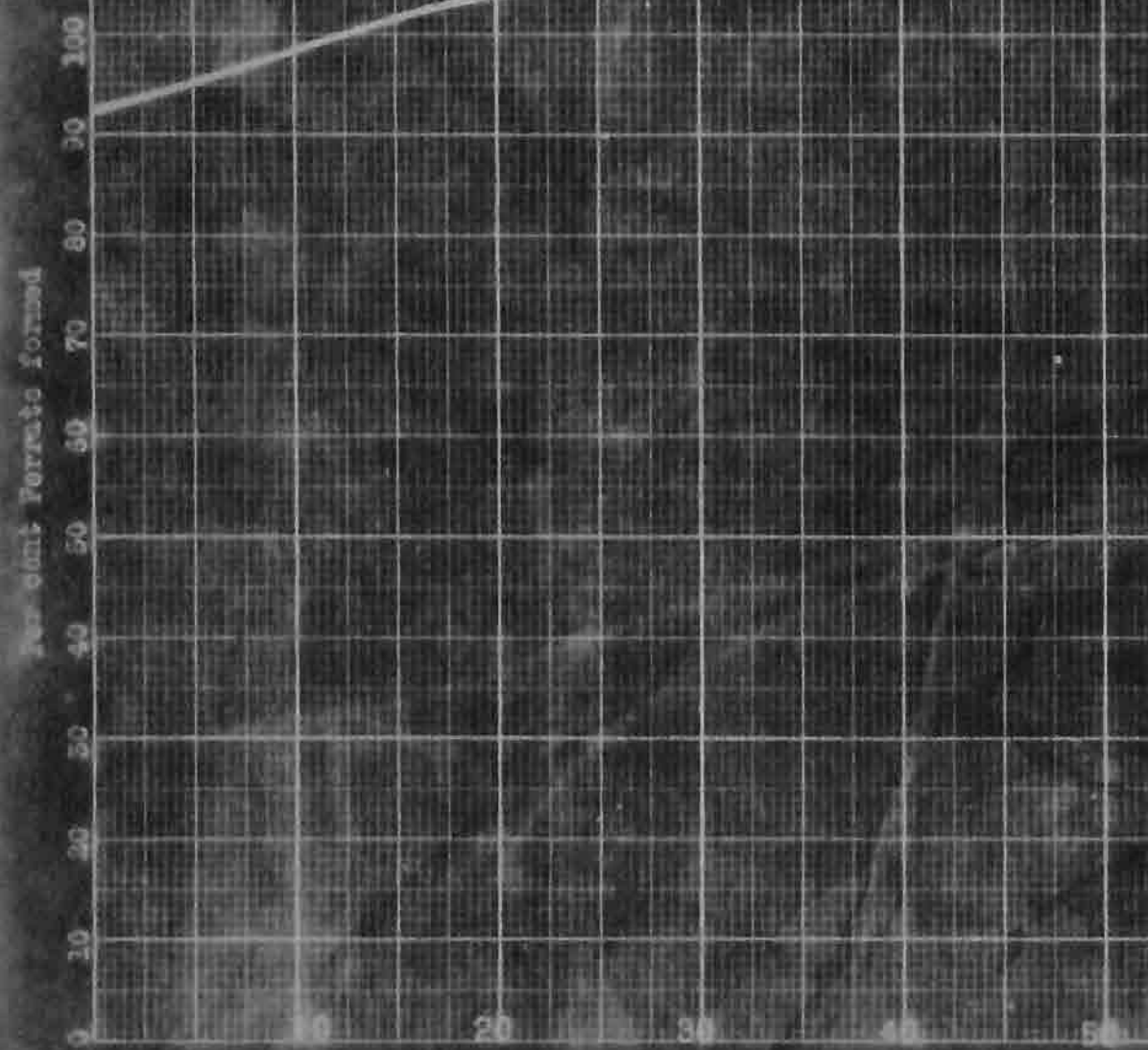
Note: Zinc dust used analyzed 99.3% Zn.

A duplicate experiment on the above series gave practically the same results:

Sample No.	Per cent Ferrate formed
59 b	90.0
60 b	100.0
61 b	102.0
62 b	106.0
63 b	108.0
64 b	107.0

SERIES NO. 11.

ZN (DUST) AND IRON OXIDE  
9 HOURS AT 600 DEG. C.



PER CENT EXCESS ZINC

SERIES NO. 11.

Series No. 111. a

Zn (dust) and  $2\text{FeS}_2$ ; 8 hours at 600 deg. C.; fair rabbling.

Sample No.	Gms. Zn. dust	Gms. $\text{FeS}_2$	%excess zinc	Gms. Zn. in mix.	Weight calcines	$\text{NH}_4\text{Cl}$ sol. zinc, %	Gms. sol. Zn.	Gms. Insol. Zn.	Per cent Ferrate formed
65	3.27	12.	00	3.25	13.00	12.95	1.68	1.57	48.3
66	3.61	12.	10	3.58	13.35	13.75	1.83	1.75	53.8
67	3.93	12.	20	3.90	14.02	15.02	2.10	1.80	55.3
68	4.26	12.	30	4.23	14.42	16.55	2.39	1.85	56.7
69	4.59	12.	40	4.56	15.07	16.95	2.55	2.01	61.8
70	4.92	12.	50	4.88	15.65	17.75	2.78	2.10	64.6

Note: Zinc dust used analyzed 99.3% Zn.

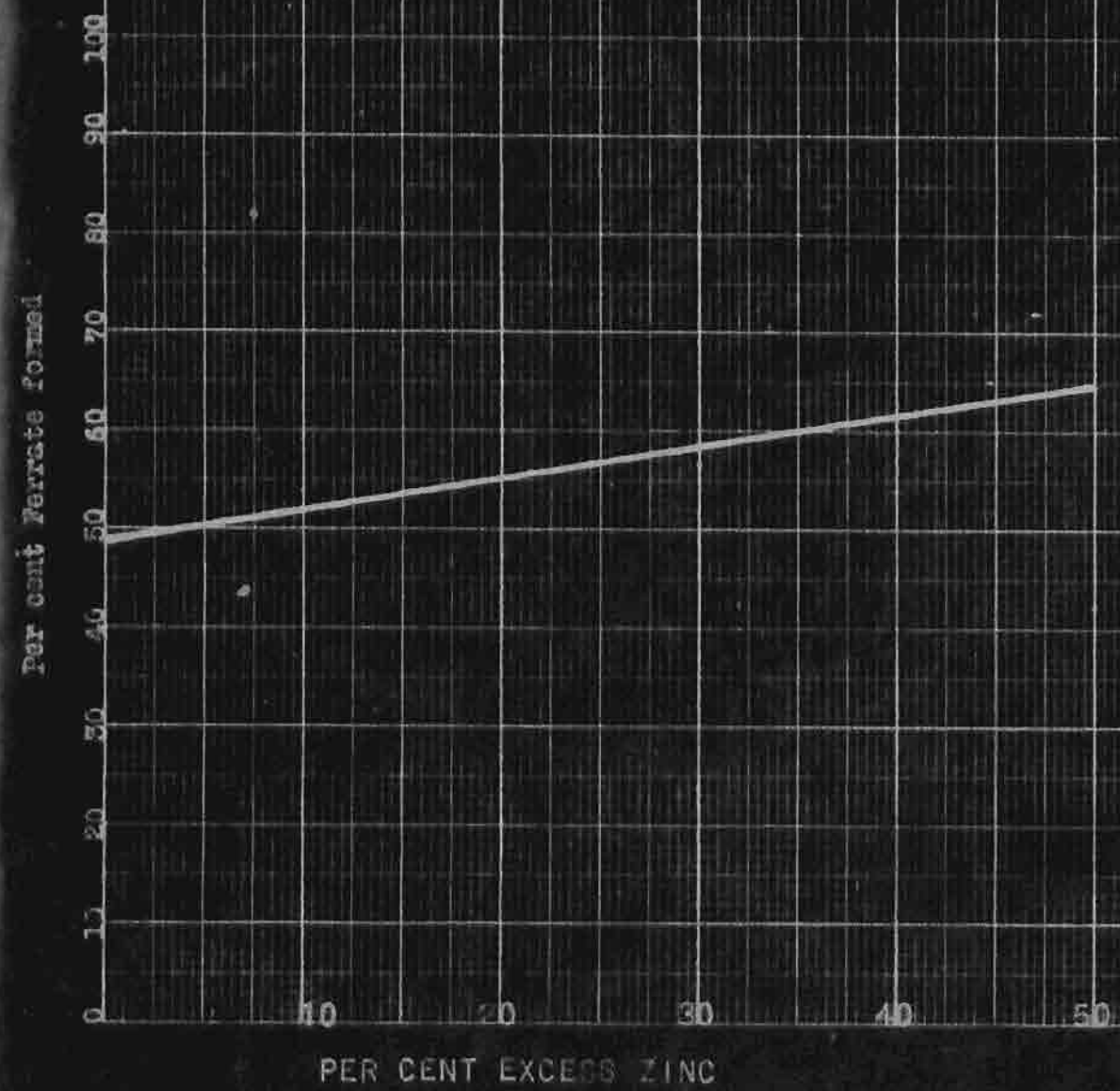
A duplicate experiment on the above series gave practically the same results:

Sample No.	Per cent Ferrate formed
65 b	42.3
66 b	50.5
67 b	54.0
68 b	56.2
69 b	59.5
70 b	63.0

SERIES 111.

AN (DUST) AND PYRITE

8 HOURS AT 600 DEG. C.



Series No. 1V.

ZnO and  $2\text{FeS}_2$  ; 8 hours at 600 deg. C.; good rabbling.

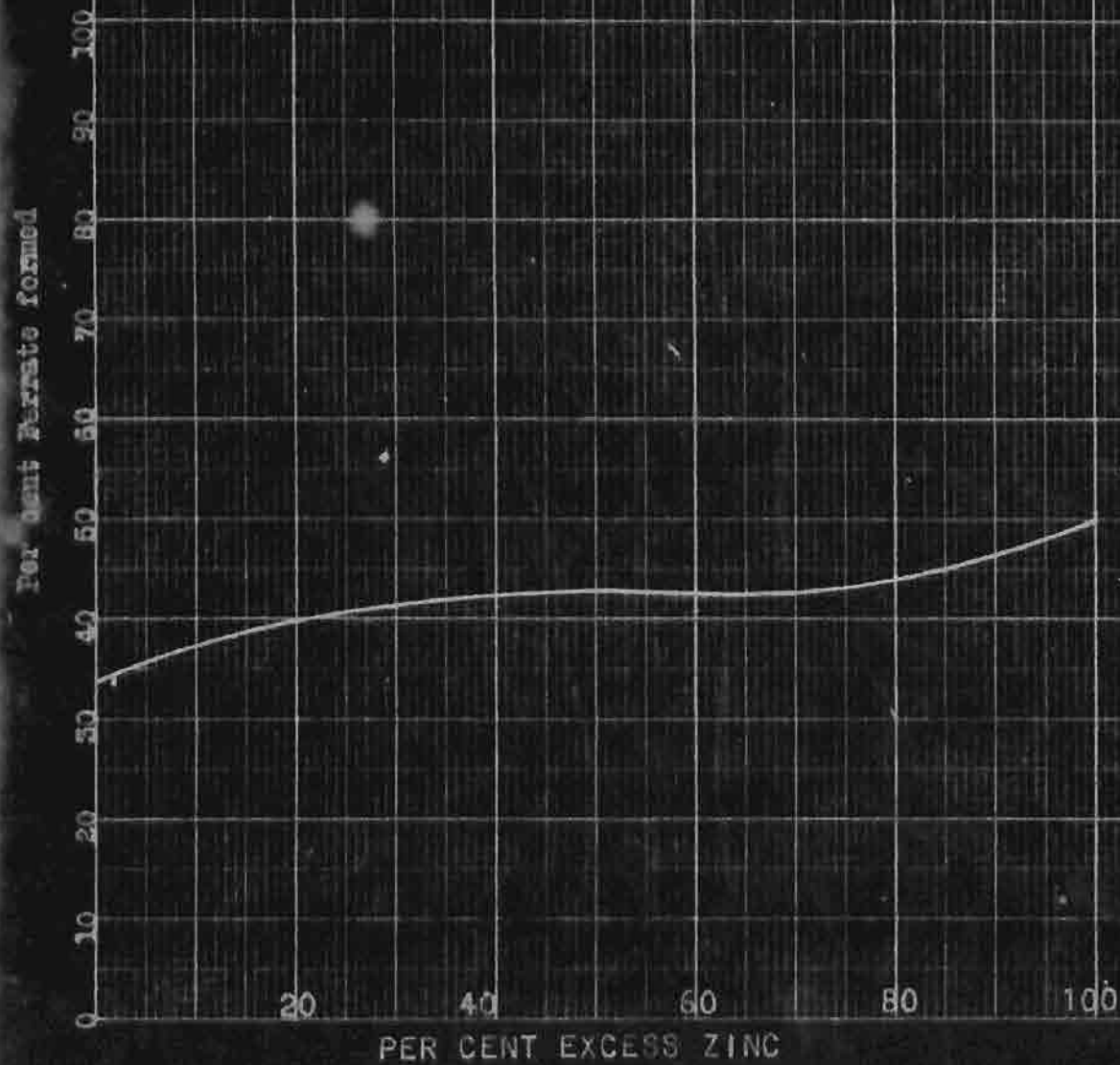
Sample No.	Gms. ZnO	Gms. $\text{FeS}_2$	%excess zinc	Gms.Zn. in mix.	Weight calcines	$\text{NH}_4\text{Cl}$ sol. zinc, %	Gms. sol. Zn.	Gms. Insol. Zn.	Per cent Ferrate formed
22	2.43	7.2	00	1.95	8.3	15.55	1.29	.66	33.85
23	2.92	7.2	20	2.34	8.9	17.60	1.56	.78	40.00
24	3.40	7.2	40	2.73	9.5	20.21	1.92	.81	41.51
25	3.89	7.2	60	3.12	10.19	22.85	2.33	.79	40.50
26	4.37	7.2	80	3.51	10.62	24.90	2.65	.86	44.10
27	4.86	7.2	100	3.90	11.08	26.42	2.91	.99	50.80



SERIES NO. 4

ZINC AND PYRITE

8 HOURS AT 600 DEG. C.



SERIES NO. 1V.

Series No. V. a

ZnS and 2FeS<sub>2</sub> ; 8 hours at 600 deg. C. ; good rabbling.

Sample No.	Gms. ZnS	Gms. FeS <sub>2</sub>	%excess zinc	Gms. Zn. in mix.	Weight calcines	NH <sub>4</sub> Cl sol. zinc, %	Gms. sol. Zn.	Gms. Insol. Zn.	Per cent Ferrate formed
53	3.40	7.2	2.07	1.98	8.5	20.7	1.76	0.22	11.35
54	4.08	7.2	22.7	2.38	9.2	23.6	2.17	0.21	10.84
55	4.76	7.2	42.8	2.77	9.92	25.6	2.54	0.25	12.90
56	5.44	7.2	63.4	3.17	10.65	27.2	2.90	0.27	13.90
57	6.12	7.2	84.0	3.57	11.22	28.0	3.14	0.43	21.90
58	6.80	7.2	104.0	3.96	11.83	29.4	3.47	0.49	25.25

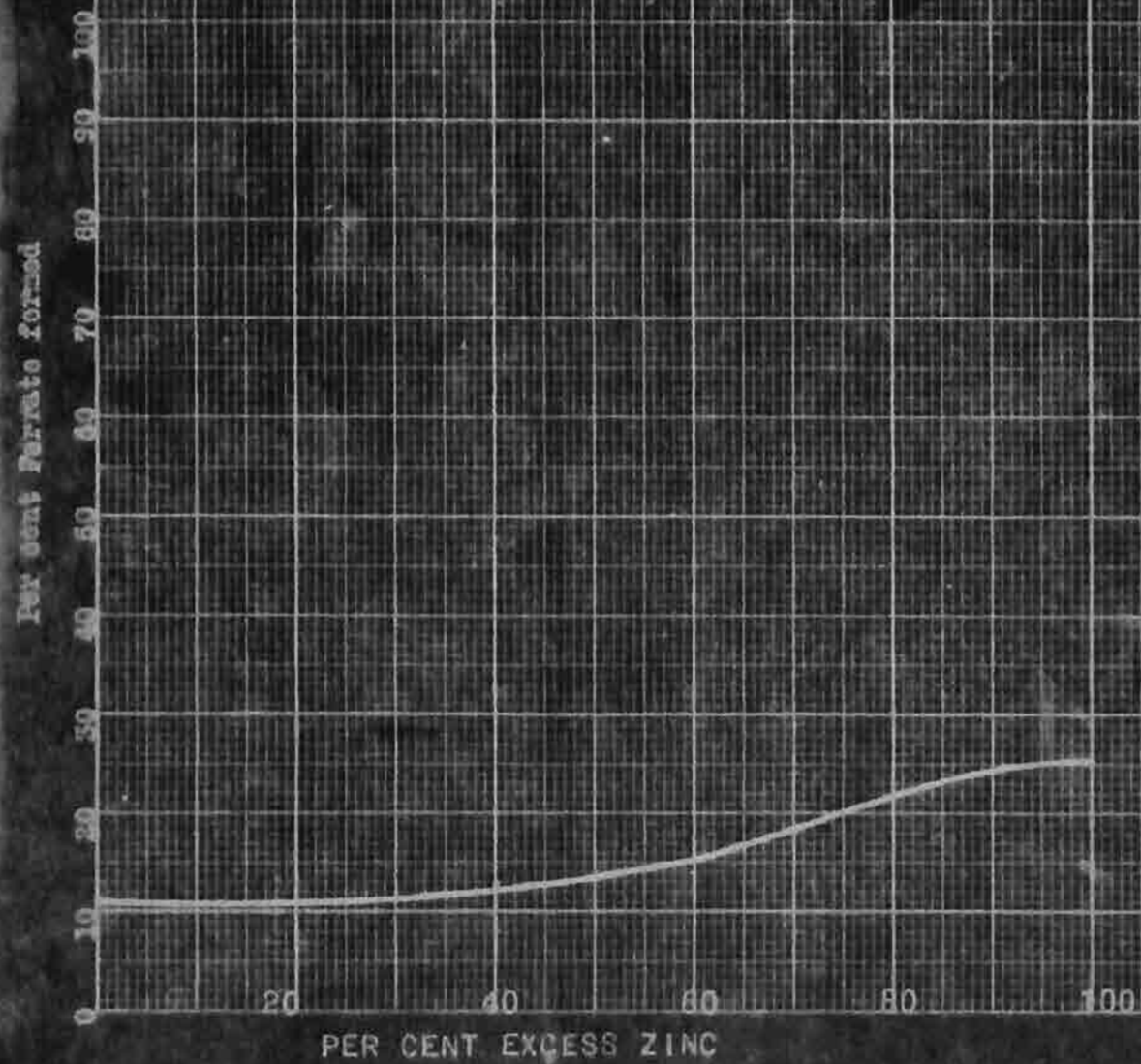
Note: ZnS used was in the form of Blende (flotation concentrates from Vinegar Hill Mill) — assay: 58.23% Zn.

FeS<sub>2</sub> used analyzed 99.0% pure FeS<sub>2</sub> .

SERIES V-A

ZNS AND PYRITE

8 HOURS AT 500 DEG. C.



EUGENE I. SERIES NO. V-A YORK, NO. W. BX

Series No. V. b

Zns and 2FeS<sub>2</sub> ; 8 hours at 700 deg. C. ; good rabbling.

Sample No.	Gms. ZnS	Gms. FeS <sub>2</sub>	%excess zinc	Weight calcines	Gms.Zn in mix.	NH <sub>4</sub> Cl sol. zinc, %	Gms. sol. Zn.	Gms. Insol. Zn.	Per cent Ferrate formed.
35	3.40	7.2	2.07	7.80	1.98	18.90	1.47	.51	26.3
36	4.08	7.2	22.7	8.37	2.38	19.25	1.61	.77	39.7
37	4.76	7.2	42.8	8.90	2.77	20.00	1.78	.99	51.0
38	5.44	7.2	63.4	9.51	3.17	24.90	2.37	.80	41.2
39	6.12	7.2	84.0	10.15	3.57	27.0 0	2.74	.83	42.8
40	6.80	7.2	104.0	10.65	3.96	27.10	2.88	1.08	55.6

Note: ZnS used was in the form of Blende (flotation concentrates from the Vinegar Hill Mill) -- assay: 58.23% Zn.

Pyrite used analyzed 99.0% FeS<sub>2</sub>.

SERIES V-B

ZNS AND PYRITE

8 HOURS AT 700 DEG. C.

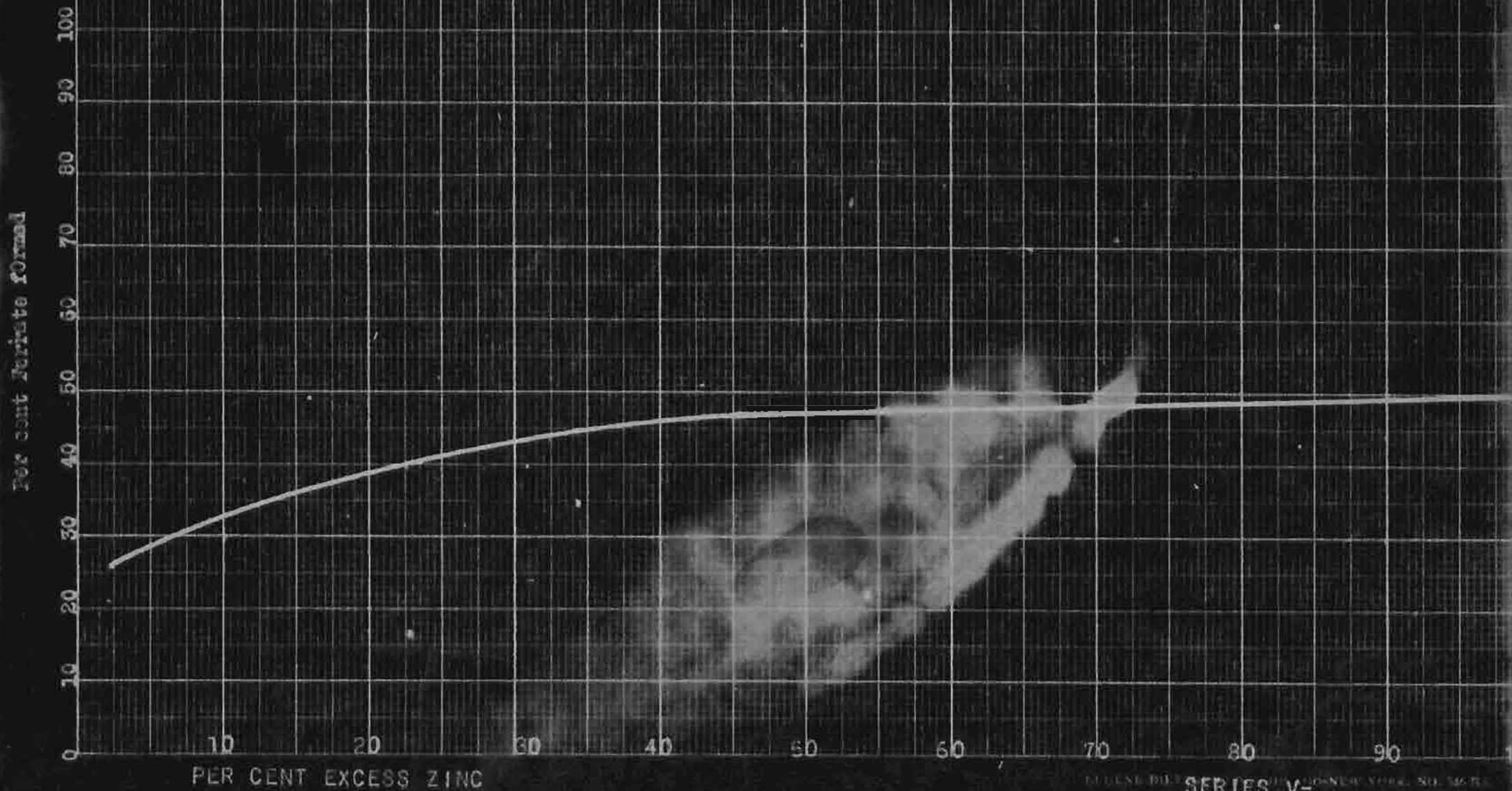


ILLUSTRATION BY THE BUREAU OF MINES, U.S. DEPARTMENT OF THE INTERIOR  
SERIES V-B  
B.

Series No. VI.

ZnS and  $\text{Fe}_2\text{O}_3$  ; 8 hours at 600 deg. C. ; good rabbling.

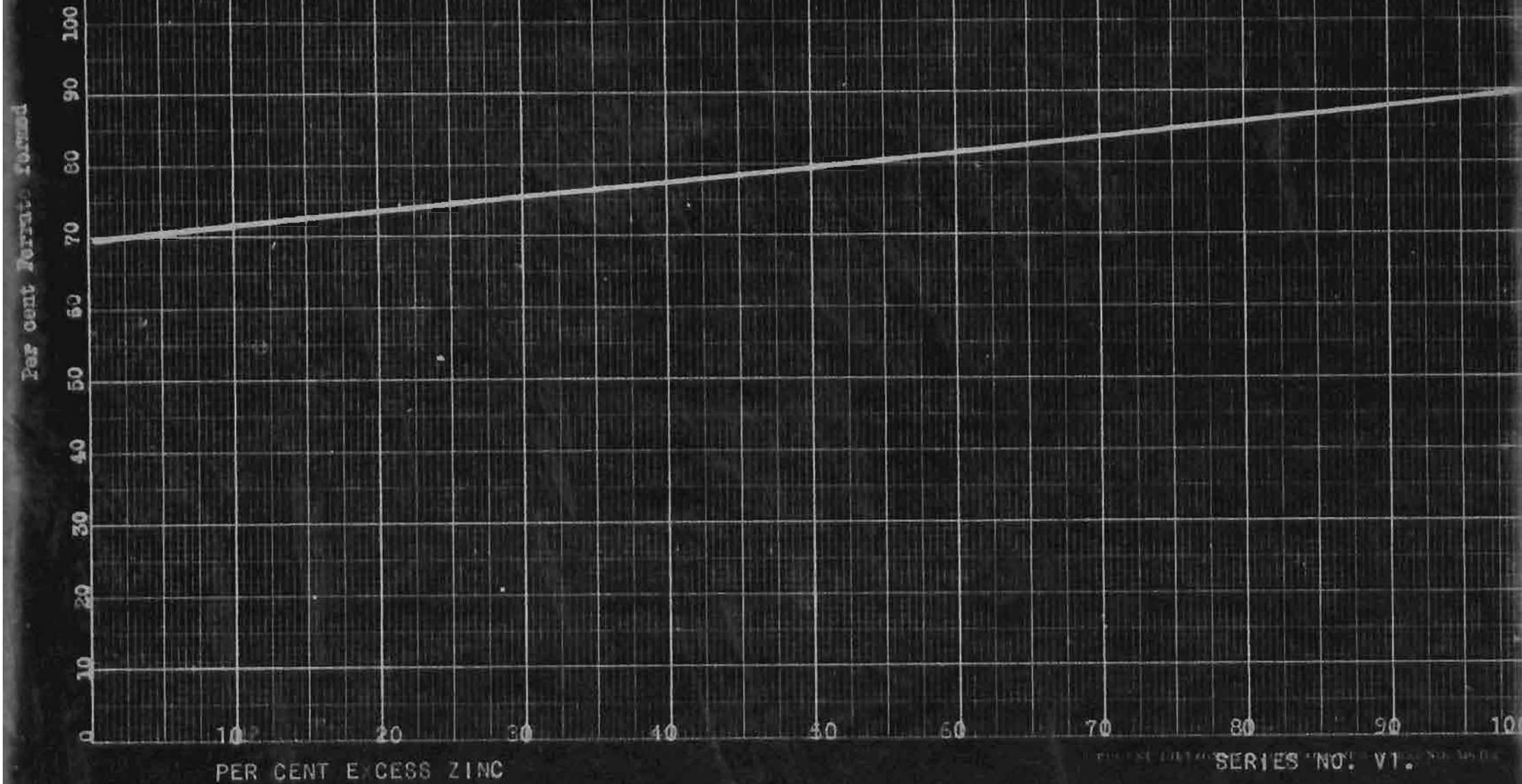
Sample No.	Gms. ZnS	Gms. $\text{Fe}_2\text{O}_3$	%excess zinc	Gms. Zn. in mix.	Weight calcines	$\text{NH}_4\text{Cl}$ sol. zinc, %	Gms. sol. Zn.	Gms. Insol. Zn.	Per cent Ferrate formed.
29	3.40	4.8	1.02	1.98	8.03	7.78	.624	1.356	69.8
30	4.08	4.8	21.4	2.38	8.70	10.80	.940	1.44	74.3
31	4.76	4.8	41.3	2.77	9.40	14.20	1.335	1.435	74.1
32	5.44	4.8	62.4	3.17	10.15	16.00	1.63	1.54	79.6
33	6.12	4.8	82.2	3.57	11.05	17.10	1.89	1.68	86.6
34	6.80	4.8	102.0	3.96	11.75	20.70	2.43	1.53	78.9

Note: ZnS used was in the form of Blende (flotation concentrates from Vinegar Hill Mill) -- assay: 58.23% Zn.

SERIES NO. VI.

ZNS AND IRON OXIDE

8 HOURS AT 600 DEG. C.



SERIES NO. VI.

Series No. VII.

Zn (dust) and Fe<sub>2</sub>O<sub>3</sub> ; 8 hours at 600 deg. C. ; no rabbling.  
in SO<sub>2</sub> atmosphere.

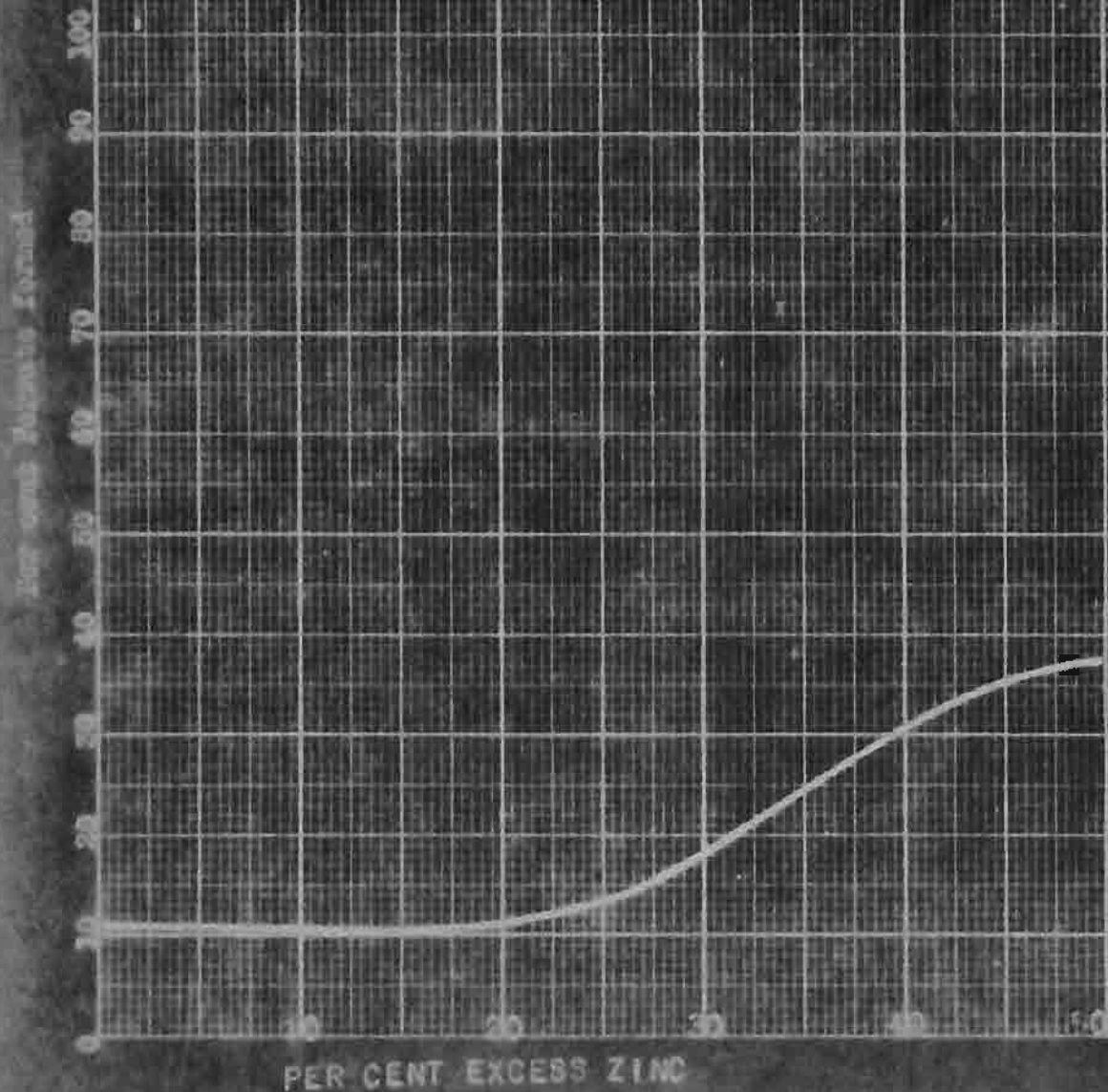
Sample No.	Gms. Zn. dust	Gms. Fe <sub>2</sub> O <sub>3</sub>	%excess zinc	Gms.Zn. in mix.	Weight calcines	NH <sub>4</sub> Cl sol. zinc, %	Gms. sol. Zn.	Gms. Insol. Zn.	Per cent Ferrate formed
71	3.27	8.0	00	3.25	15.2	18.7	2.84	.41	12.6
72	3.61	8.0	10	3.58	16.2	20.2	3.27	.31	9.54
73	3.93	8.0	20	3.90	17.05	20.8	3.54	.36	11.08
74	4.26	8.0	30	4.23	17.8	20.2	3.60	.63	19.4
75	4.59	8.0	40	4.56	17.45	19.2	3.35	1.21	37.2
76	4.92	8.0	50	4.88	18.3	20.5	3.75	1.13	34.8

Note: SO<sub>2</sub> concentration in muffle maintained at 2%, fairly constant.



SERIES NO. VII

ZINC (DUST) IRON OXIDE  
8 HOURS AT 500 DEG. C.  
IN SULPHUR DIOXIDE ATM.



Series 1. ZnO and Fe<sub>2</sub>O<sub>3</sub>

The zinc and the iron are both in a highly oxidized state to start with. The heating is a mere "baking" which causes approximately the highest formation of ferrate when sixty per cent excess zinc is used.

Series 11. Zn (dust) and Fe<sub>2</sub>O<sub>3</sub>

In this case the zinc is oxidized in the presence of iron oxide. In this series the highest formation of ferrate of the entire group (seven series) was obtained: 100 per cent ferrate with only 10 per cent excess zinc. This may be attributed to the fact that intimacy of contact of zinc and iron (or iron oxide) is greatest and that volume change due to the zinc taking on one oxygen atom results in even greater intimacy.

Series 111. Zn (dust) and 2FeS<sub>2</sub>

Here we have oxidation of both zinc and iron, the maximum amount of ferrate forming being sixty-five per cent.

The presence of SO<sub>2</sub> (from oxidation of the pyrite) decomposes some of the temporarily formed ferrate --- resulting in a lesser amount of the insoluble compound formed.

Series IV. ZnO and 2FeS<sub>2</sub>

There is no oxidation of the zinc, but there is practically complete oxidation of the pyrite.

The curve shows a maximum amount of ferrate with 100 per cent excess of zinc. This is not fully understood. It is probably due to the dilution of the SO<sub>2</sub> with an increase in ZnO. Nos. 22 to 27 inc., all contain the same amount (gms weight) of pyrite and, consequently, give off the same volume of SO<sub>2</sub> upon roasting; but, inasmuch as there is an increased amount of mass (due to increased amount of ZnO) in the numerically ascending lots, there results a greater dilution of the pyrite and hence a greater dilution of the SO<sub>2</sub>. The practically straight line seems to indicate that the increased amount of ZnO directly reduces the "retardation" effect of the SO<sub>2</sub>.

Series V. ZnS and 2FeS<sub>2</sub>

Both the zinc and the iron are oxidized together. In this series there is only a small amount of ferrate formed -- 10 to 25 per cent, the latter at 100 per cent excess zinc. This shows the influence of SO<sub>2</sub>

in decomposing the ferrate which has already formed. The effect of the  $\text{SO}_2$  is truly a decomposing action and not merely a retardation one --- as has been proven by heating 100 per cent ferrate in an  $\text{SO}_2$  atmosphere, concentration and time being sufficient all ferrate is decomposed to  $\text{ZnSO}_4$ ,  $\text{FeSO}_4$ , and  $\text{Fe}_2\text{O}_3$ .

Series VI.  $\text{ZnS}$  and  $\text{Fe}_2\text{O}_3$

Oxidation of zinc; no oxidation of iron.

A high formation of ferrate was obtained which is in conformity with the general results of the entire group. This high formation is quite similar to that obtained by roasting Zn (dust) and  $\text{Fe}_2\text{O}_3$ .

Series VII. Zn (dust) and  $\text{Fe}_2\text{O}_3$  in  $\text{SO}_2$  Atmosphere.

The zinc is oxidized in the presence of iron oxide. This is identical with series 11. save that  $\text{SO}_2$  was introduced into the muffle in this series' test. The conditions which, as in series 11., were conducive to high formation of ferrate were offset as far as final results were concerned by the decomposing effect of the  $\text{SO}_2$  atmosphere.

In order to determine the effect of increasing amounts of iron in the concentrates upon the extraction of zinc from calcines resulting from roasting such concentrates, a roast-leach test was made as follows:

8 hour roast at 600 deg. C.; rabbling every 20 minutes

Lot No.	Weight of concentrates	Weight of FeS <sub>2</sub> added	Per cent iron
1	50. gms.	0.0 gms.	.6
2	50.	1.075	1.57
3	50.	3.225	3.38
4	50.	5.325	5.23
5	50.	7.525	6.61
6	50.	9.675	8.05
7	50.	15.050	11.20
8	50.	20.425	13.90

A 10 gm. sample of each of the above lots (calcines) was leached in ammonia chloride leaching solution (200 gms. NH<sub>4</sub>Cl, 500 cc. NH<sub>4</sub>OH, 750 cc. H<sub>2</sub>O) for fifteen minutes at 75 deg. C., iron removed, and filtrate assayed for zinc.

Results of Roast-Leach Test:

Lot No.	Weight of calcines	Total Zn in calcines	Zn leached	Per Cent of total Zn extrd.
1	45.6 gms.	29.1 gms.	28.4 gms.	97.8
2	47.4	29.1	28.4	97.8
3	50.2	29.1	28.3	97.5
4	51.5	29.1	27.2	93.4
5	52.2	29.1	27.24	93.7
6	56.4	29.1	28.2	97.0
7	60.2	29.1	27.4	94.2
8	63.5	29.1	27.5	94.7

CURVE SHOWING THE EFFECT OF INCREASING  
AMOUNTS OF IRON OF THE PER CENT OF  
EXTRACTION OF ZINC FROM COASTED COPPER  
CONCENTRATES OF GRAVITY 6.5, TO 10.3 PER CENT  
IRON.

Conclusions: Sec. 1, Part 11.

Ferrate Investigation

1. The combinations of iron and zinc may be listed in the order of increasing percentage of ferrate formed as follows:
  1. ZnS plus  $2\text{FeS}_2$
  2. ZnO plus  $2\text{FeS}_2$
  3. ZnS plus  $\text{Fe}_2\text{O}_3$
  4. Zn plus  $2\text{FeS}_2$
  5. ZnO plus  $\text{Fe}_2\text{O}_3$
  6. Zn plus  $\text{Fe}_2\text{O}_3$
2. Sulphur dioxide gas has a decomposing effect on ferrate.
3. Oxidation of the zinc and iron need not take place simultaneously to produce a high formation of ferrate.
4. The greater the intimacy of contact of the zinc and iron particles, the greater the percentage of ferrate formed.
5. At 700 deg. C. there is a marked increase in the formation of ferrate over 600 deg. C. practice.
6. Prolonged heating gives higher formation.
7. The percentage of Zn as  $\text{ZnSO}_4$  is inversely proportional to the percentage of ferrate formed.
8. In general, the greater the amount of rabbling, the lower is the formation of ferrate in all series.

9. The formation of ferrate in the roasting of flotation concentrates from the Joplin-Miami district is practically negligible.

10. Even when these concentrates are mixed with iron as high as 14% (see page 41) there is no appreciable decrease in the solubility of the zinc. The rise and drop in the extraction figures is due no doubt to insufficient roasting in the case of those lots which have corresponding low results.

11. The existence of the iron and zinc particles merely as a mechanical mixture is not conducive to the formation of ferrate as is the case in roasting marmetite where there exists isomorphous crystallization of zinc and iron sulphides.



2. Joplin-Miami Flotation Concentrates:

A series of tests were run on the flotation concentrates from the following mills:

sample no. as tested.	mill	analysis				
		Zn.	Fe.	Cu.	Cd.	Pb.
H <sub>1</sub>	Vinegar Hill Zn Co., Baxter Spgs, Kans.	58.23	.60	.1	.39	—
H <sub>2</sub>	C. M. & Ref. Co., Carden, Okla.	59.67	.62	.1	.255	—
H <sub>3</sub>	St. L. S & Ref. Co., Picher, Okla.	60.81	.51	.1	.355	4.16
H <sub>4</sub>	Federal M & S Co., Kockerville, Okla.	60.50	.62	.1	.346	4.59
H <sub>5</sub>	Okla. Woodchuck Zn & Pb Co., Okla.	57.19	.72	.1	.322	6.30
H <sub>6</sub>	Golden Rod M & S Co., Carden, Okla.	59.67	.51	.1	.305	1.98
H <sub>7</sub>	Eagle-Picher Lead Co., Picher, Okla.	58.22	.51	.1	.391	4.20

A. Reast:

Acid Consumption Test:

2 gms. of roasted concentrates,

100 cc. of 4.05% H<sub>2</sub>SO<sub>4</sub>,

agitated 10 minutes at 60 deg. C.,

filtered, residue washed completely, and free

acidity determined on filtrate by titrating with

a std. base (methyl orange as indicator).

Test showed an average acid consumption of 87. %.

Solubility Test:

Concentrates: H<sub>(1-7)</sub> roasted at 600 deg. C. in 200 gm. lots. Calcines were weighed, assayed for oxidized zinc, water-soluble zinc, the necessary acid needed for solution calculated, and a dilute sulphuric acid (13.5%) leach made. As a check, in several cases, both filtrate and residue were analyzed for zinc in order to determine "solubility".

Result of roast:

Sample No.	gms. conc.	gms. calcines	% reduction in weight	%oxidized zinc	%SO <sub>4</sub> zinc
H <sub>1</sub>	200	186	7.0	62.4	4.0
H <sub>2</sub>	200	184	8.0	64.2	4.0
H <sub>3</sub>	200	185	7.5	61.6	2.0
H <sub>4</sub>	200	184	8.0	62.0	1.3
H <sub>5</sub>	200	192	4.0	58.9	6.2
H <sub>6</sub>	200	181	9.5	57.0	.8
H <sub>7</sub>	200	185	7.5	60.2	1.0

Result of Leaching:

Solubility Test:

Sample; No.	;	Calcines			Residues			; Insol.Zn.;	Sol. Zn.	
		Wt.	% Zn.	Gms. Zn;	Wt.	% Zn.	Gms. Zn;		%	A
H <sub>1</sub>	a	50.	62.7	31.36	7.75	11.02	.854	2.72	97.3	98.0
H <sub>2</sub>	a	50.	64.85	32.47	6.05	25.44	1.54	4.73	95.3	94.5
	b	50.	64.85	32.47	3.75	7.52	.28	.87	99.1	—
H <sub>3</sub>	b	50.	65.70	32.85	3.83	3.30	.126	.38	99.6	—
H <sub>4</sub>	b	50.	65.60	32.80	4.29	3.38	.145	.44	99.5	—
H <sub>5</sub>	a	50.	59.50	29.80	8.35	16.07	1.34	4.50	95.5	95.7
	b	50.	59.50	29.80	6.25	7.13	.45	1.50	98.5	—
H <sub>6</sub>	b	50.	65.7	32.90	3.27	4.1	.134	.41	99.5	—
H <sub>7</sub>	b	50.	62.50	31.25	5.52	4.23	.233	.745	99.2	—

Note: Under "Sol. Zn." column A the analyses of the residues is used to compute the "solubility"; column B lists the "solubility" as computed from the zinc determination of the filtrates.

### B. Leaching Tests:

The idea was to determine the maximum percentage of zinc that could be leached from the concentrates under the most favorable conditions.

The apparatus used for leaching was as follows:

An ordinary pulp balance was used to weigh the charges and residues. The 50-gm. leaches were made in a three-liter glass bottle (inverted with the bottom removed). Air agitation was maintained by inserting a glass tube down into the charge (touching the rubber stopper which was inserted in the necked crown of the bottle). A Buchner funnel and suction pump were used for filtering.

Dilute sulphuric acid was used for leaching. A 13.5% acid solution was used as it gave an electrolyte of the desired zinc concentration.

Five leaches were made on 50-gm. lots of a mixture containing equal parts (by weight) of the seven calcines, H(1-7), strength of acid being the same in all, but the time of leach varying as follows: no. 1 - 15 min., no. 2 - 30 min., no. 3 - 1 hr., no. 4 - 2 hrs., no. 5 - 3.5 hrs.

The pulp from the leaches was filtered, acidity determined on undiluted filtrate, residues washed, weighed, and analyzed for zinc. Relative solubilities were then calculated.

Results of leaches, time of leach varying.

No.	filtrate	free acid	weight residue	% Zn. in residue	% Zn. extracted solubility
1	425. cc.	9.57 gm.	4.82	19.59	97.0
2	450.	9.97	5.13	8.68	98.5
3	440.	10.17	4.80	4.33	99.4
4	400.	9.75	4.73	3.98	99.5
5	405.	9.90	4.69	3.75	99.5

Assuming No. 3 (time of agitation: 1 hr) to give apparent satisfactory extraction, but, for safety, allowing an extension of a half hour, a leach was made on 150. gms. of the above type calcines, just enough acid being used as to give a slightly acid filtrate (this amount having been determined from the above tabulated results), temperature of leach being maintained around 65 deg. C., and air agitation maintained for 1.5 hours. Results as tabulated below:

Weight calcines	%Zn. in calcines	Gms. Zn. in calcines	Weight residue	%Zn. in residue	Gms. Zn. in residue	%Zn. extr'd. solubility.
150.00	63.80	95.70	15.18	5.67	.86	99.1

Note: The undiluted filtrate from this last leach had a sp.gr. of 1.265.

In order to determine the "solubility" or per cent extraction of zinc from Joplin-Miami flotation concentrates roasted in a manner more representative of commercial practice, a leach was made on calcines obtained as follows:

30 lbs. flo. conc. from ST. L. Sm. & Ref. Co. Mill,  
Picher, Okla.

12 hour slow roast in a mechanicalized electric roaster.

Roaster: 1 inch cast iron circular hearth, 24 inch in diam., under which was installed a removable Ni-Chrome heating element (not imbedded).

Average temp. of hearth: 600. deg. C.; average voltage maintained: 112.; average amperage: 94.; rabble arm rate: 1 r.p.m. Zn as  $ZnSO_4$  obtained: 4.0%

The leach was made on 150. gms. calcines, acid and time being the same as in the last-mentioned leach, but temperature being lower — no external heat was added. The heat of reaction ( $ZnO + H_2SO_4$ ) was sufficient to raise the temperature of the pulp to 45 deg. C. (room temp. being 25 deg. C.).

An analysis of the residue from this test showed an extraction of 98.55 per cent, zinc solubility.

The undiluted filtrate from this leach analyzed as follows:

Zinc	Iron	Cadmium	Free acid
74.6gm./L	.301gm./L	.34gm./L	1.055gm./L

#### Purification Test:

A 500 cc. sample of the acid filtrate, containing chiefly  $ZnSO_4$  but also some  $CdSO_4$  and  $CuSO_4$ , was subjected to two treatments with zinc dust, agitation being maintained while adding the zinc, and a careful filtration effected immediately following each zinc dust treatment.

The solution remaining (filtrate from above) was tested for nickel and copper. Result was negative for both.

A 2-hour leach was made on 150 gms. of calcines, the acid pulp neutralized with fresh calcines and 5. gms. of  $MnO_2$  added. After one hour further agitation with air, the pulp was filtered and the filtrate tested for ferrous and ferric iron. The ferrous test was negative, but the ferric test showed a slight trace of  $Fe^{+++}$  present — some colloidal ferric hydrate undoubtedly passed thru the filter.

### Purification of the Zinc-Sulphate Solution:

One of the most important steps in the electrolytic zinc process is the purification of the solution prior to its discharge into the cells. All attempts to deposit zinc electrolytically from impure solutions have proven failures; for efficient deposition an absolutely pure solution is essential.

#### Iron as an impurity:

Ferrous iron is harmful. It will be oxidized at the anodes and current is used for this action. Besides this wasteful consumption of current, there is a liability of the iron being entrapped or deposited in the cathode.

Practically all of the soluble iron contained in the solution is in the ferrous condition. When this solution is neutralized (by fresh calcines) or treated direct with a base, the iron compound formed is not completely insoluble, and during subsequent filtrations, part of this compound passes thru the filter. The effective removal can be obtained by thoro oxidation of the iron in neutral solution by the addition of a small quantity of manganese dioxide, or by air agitation, the latter, however,



not being as effective or active as the former owing to the fact that the air which passes up thru the solution is not in a very finely divided state (the bubbles being large do not have intimate contact with the entire solution). Limerock or fresh calcines serve sufficiently well to precipitate the bulk of the iron prior to filtration, but for a more thoro removal manganese dioxide is needed.

The following equations represent the reactions occurring during the removal of iron:

Oxidation:

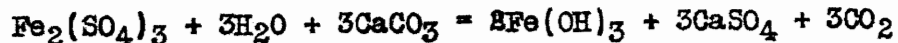


Upon neutralization of the free acid, precipitation of the iron as hydrate occurs carrying down with it arsenic and antimony as ferric-arsenate and antimonate, both of which are insoluble in neutral or basic solution.

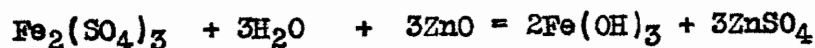
Neutralization:      Precipitation resulting:



Upon the addition of limerock:



Upon the addition of ZnO (fresh calcines):



#### Arsenic and Antimony as Impurities:

Both arsenic and antimony, even in very small quantities, especially the latter, are highly detrimental to good deposition. Removal occurs upon the precipitation of the hydrate of iron. The ferric hydrate interacts with the arsenic and antimony to give ferric-arsenate and ferric-antimonate. If insufficient iron is present in the leaching solution, it is necessary to add a solution of ferric sulphate in order to insure complete removal of the arsenic and antimony.

#### Nickel and Cobalt as Impurities:

Both of these elements have proven to be most deleterious in the cell room; being similar to iron in that they have an -ic and an -ous form, the presence of the -ous form uses up current at the anode, and in the -ic form deposits at the cathode thereby contaminating the zinc. It is the action at the anode that causes the trouble — both cobalt and nickel require a high oxidation potential to convert them to the -ic state.

¶Considerable experimental work has been performed on solutions varying in cobalt and nickel content: The treatment of the solution with strong oxidizing agents (PbO<sub>2</sub>, permanganates, and ozonized air), with blue powder, nitroso-  

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¶ O.C. Balston, Electrolytic Dep. and Hydro-Met. of Zinc.

beta-naphthol, and the addition of glue and use of sack diaphragms in the cell have all proven well worth considering.

#### Copper as an Impurity:

Copper is detrimental to good deposits. The amount in the solution determines its action. Large amounts were found to deposit upon the cathode and then drop off in spongy form. Small amounts cause black patches on the cathodes and hinder zinc deposition.

The removal of copper by the addition of zinc dust has proven very successful, commercially. About 80.% of the copper comes out with the first treatment with zinc dust. Decantation and filtration followed by a second addition of zinc results in practically a copper-free solution.

#### Cadmium as an Impurity:

Cadmium will deposit out of solution along with the zinc. Thoro removal of cadmium is necessary in order to produce grade A zinc. Upon the addition of the first charge of zinc dust considerable cadmium will precipitate along with the copper, but as the solution stands a re-solution of the cadmium occurs. By subjecting the solution to immediate filtration after the first treatment with

zinc dust, this re-solution is prevented, and by a second treatment of zinc dust, practically all cadmium is precipitated. The solution resulting, containing a small amount of cadmium in suspension is immediately pumped thru a clarifying filter-press, the effluent flowing to the cell room feed tank.

D. Deposition:

Spangy zinc results from two chief causes: 1, very low acidity or basic solution; 2, presence of impurities like copper and arsenic. Poor circulation of the electrolyte tends to cause formation of sprouts, trees, etc. Pinholes are due to the adherence of small bubbles of  $H_2$ ; larger holes are due to the re-solution of zinc, due to presence of impurities, especially cobalt, nickel, antimony and arsenic. The effect of impurities has been mentioned under section C. and only the important electrolytic effects are mentioned below:

ferrous iron ---- not harmful.  
ferric iron --- is one of the main causes of anode corrosion -- maximum amount permissible = 25 mg./L.  
antimony and arsenic --- causes re-solution of the zinc and, hence, produces poor deposit.  
cobalt --- is the worst impurity -- causes re-solution of zinc.  
cadmium -- no effect save that it reduces the purity of the zinc deposit.  
chlorides -- causes serious corrosion of the anodes.  
maximum amount permissible = 50 mg./L.

## Part 111.

### Commercial Applications

#### 1. Proposed Process:

In general the process proposed for the extraction of zinc from the concentrates from the Joplin-Miami District and the recovery of zinc in metallic form may be described as a sulphate-leaching electrolytic deposition process.

The concentrates are first roasted (to about 2 - 5 per cent zinc as zinc sulphate content) and then subjected to a dilute sulphuric acid leach. The sulphate solution resulting is next purified of all elements which have been found detrimental in the efficient operation of further treatment. The now practically pure zinc sulphate solution is subjected to electrolytic action, zinc in metallic form being deposited on the cathodes and free sulphuric acid, which is used again for leaching, regenerated at the anodes.

Detailed explanation may best be obtained by reference to the diagrammatic outline of the process attached on last page.

## 2. General Description of the Proposed Plant:

The basis for the proposed commercial plant is the daily treatment of 200 tons of 59.% zinc concentrates, producing approximately 113. tons of slab zinc per day.

The first step in the treatment will be roasting. This preliminary treatment of oxidation of the sulphide will be performed in Wedge zinc roasters (similar to those in use at Anaconda and at Trail).

The second step will consist in the leaching of the calcines in dilute sulphuric acid solution in Pachucas tanks. Filtration will follow as step three. Purification of the electrolyte by the addition of zinc dust will constitute a fourth step, and electrolytic deposition of the zinc from the sulphate solution, the fifth. The last step, which is necessary in both the retort and the electrolytic process, will be the melting down of the zinc and casting into slab form.

## Outline of Departments.

### 200 tons of 59. per cent Zinc Concentrates:

1. Receiving Bins     )  
  )     one building.  
    Regrinding Plant )
2. Roasting Plant:  
    six Wedge roasters, mechanically rabbled, water cooled rabble arms and column, 7-hearth, 25 ft. outside diam. 21 ft. inside diam.  
  
    2250 sq.ft. effective roasting area per furnace.  
  
    Flue dust collector stack.
3. Leaching Plant:  
    Pachuca Agitators,  
    Dorr Classifiers,  
    Dorr Thickeners,  
    Oliver Filter and Shriver Filter Press.  
    Air Compressor, 2-1500 cu.ft./min. at 30#.
4. Power Sub-Station:
5. Zinc Melting Plant:
6. Offices, laboratory, and Plant Yard Equipment.

Operations:

Roasting:

Delivered to roasters 200 tons of 59. % zinc

Concentrates:-----118.0 tons zinc

assuming a roasting loss of 1.% ----- 1.18

In calcines -----116.82 tons zinc

assuming a 10.% shrinkage in weight (in roasting):

$200 - .10 \times 200 = 180$  tons calcines (65.% Zn)

one roaster will handle about 40 tons per day --

about five roasters needed --

install six roasters for safety.

Calcines from roasters: 180 tons:

65.% Zn.	{	3.% Zn as Sulphate -----	5.4 tons
		62.% Zn as oxide -----	111.42 tons
			<u>116.82 tons Zinc.</u>

---

By conveyor to Leaching Plant.



Leaching:

Leaching results in the laboratory show 99.4% solubility. Assuming 98.% solubility there will be  $.98 \times 116.82 = 114.5$  tons of soluble zinc in the 180 tons of calcines received at the leaching plant.

114.5 tons soluble zinc  
5.4 tons zinc as zinc sulphate (3.%)

---

109.1 tons zinc requiring acid for leaching.

Acid consumption will be as follows:

$109.1 \times 3/2 = 163.6$  tons  $H_2SO_4$

$163.6/180 \times 100 = 90.7\%$  acid consumption

(Laboratory tests show 87.% acid consumption)

Referring to page 66 "Cell Room", there will be seen the following relative quantities:

Neutral Feed to cells ---- 8.5 % Zinc

Acid Effluent from cells -- 2.5 % Zinc

$H_2SO_4$  in Effluent --- 9.0 %

In all there will be 1975 tons of acid effluent (9.%).

(Refer to page 67 for computations).

This will carry 2.5% Zinc or 49.35 tons.

The residue from leaching 180 tons of calcines will amount to less than one-fifth this weight. (Laboratory tests showed a 10 to 1 reduction in weight.)

Assuming a reduction in weight of 5 to 1, then from 180 tons of calcines there will be 36 tons of residue.

This residue goes to a classifier to take out sands. These sands will largely be unroasted ore, which will be re-ground and returned to the roaster --(circulating load). With proper roast this only becomes a safety valve to insure proper oxidation of the zinc sulphide.

Pachucas needed:

A 15 x 30 tank has a volume capacity of 3882. cu.ft. and will hold 121.5 tons of solution.

$121.5/1975 \times 36 = 2.21$  tons of residue per tank.

$2.21/2.7$  (sp.gr.) = .82 or approximately 1 ton (vol) residue.

$121.5 - 1. = 120.5$  tons solution (less residue).

Assuming a two-hour leach:

$8$  hr. shift /  $2 = 4$  batches per shift, or 482 tons soln.

Hence there will be needed  $1975/482 = 4$  tanks.

Inasmuch as leaching is done in one 8-hour shift, there is no reason to provide many extra tanks as there will be two 8-hour shifts to take care of any emergency leach necessary. However, the installation of five is recommended.

The leach is considered to be performed in a double leaching system: neutral and acid circuit.

Considering the neutral circuit:

The nearly neutral acid overflow from the acid circuit to-

gether with about 30% of the cell effluent is delivered to the head pachuca of the neutral side. In this and the subsequent pachuca the iron is oxidized by means of  $MnO_2$ . The iron is fully oxidized by the time it reaches the third tank, at which place the fresh calcines are added. The other two pachucas are simply digestors and coagulators. There is not sufficient manganese present in the concentrates to oxidize the iron, but an addition of  $MnO_2$  to the system will, to a great extent, be regenerated as  $MnSO_4$  and subsequently to  $MnO_2$  for following batches.

Following the fifth neutral pachuca there is a classifier, then two neutral Dorr, and three acid pachucas, these latter to be followed by two acid Dorr.

**Necessary Dorr:**

There will be 36 tons of solids to be separated from 1975 tons of solution, but for safety consider 40 tons to be handled.

**Assuming 21 sq.ft./ton/day settling area of one Dorr:**

40 tons will then require a settling area of 840 sq.ft., which is provided for by two 40 x 10 Dorr tanks with equipment. Assuming the spigot discharge from the Dorr to have a ratio of 2 liquid to 1 solid, the 40 tons of solids will therefore contain 80 tons of liquid.

Filtering:

The residue contains approximately 20.% Pb as  $PbSO_4$ ,  $SiO_2$ ,  $Fe_2O_3$ ,  $Al_2O_3$ ,  $CaO$ , and some Zn as  $ZnSO_4$  and  $ZnS$  (see zinc figures at bottom of this page), and offers very little difficulty in filtering. Following a filter cycle of: 2 minutes for cake formation, 2 minutes for washing, and 1 minute for drying, and practice as listed: cake thickness -  $5/16$  inch, vacuum - 23 inches, temperature - 30 deg. C., and sp.gr. of filtrate - 1.26, a tonnage of 6.52 dry solids can be obtained for one foot horizontal of a 12 ft. Oliver Filter. For 8 ft. horizontal,  $8 \times 6.52 = 52$  tons can be removed.

Provision is made in the plant plans for two such filters, the second filter to receive the pulp after repulping from the first filter. This repulping is to be done in a weak solution which gradually becomes fortified in zinc.

The filter discharge residue amounting to 36 tons (as previously assumed) will contain about 8.% zinc:

6.45% insoluble zinc ( $ZnS$ )	-----	2.32 tons
1.55% soluble zinc (entrained)	---	.56 tons
<u>8.00% total zinc</u>		<u>2.88 tons,</u>

thereby creating a zinc loss of  $2.88/118 \times 100 = 2.44\%$

Purification:

Zinc Dust Agitators:

Five 10 ft. diam. x 15 ft. depth Devereux agitators are suggested. The stream is deflected from tank 16 (see Diagramatic Outline of Proposed Process at end of thesis) to classifier 17 from which the coarse zinc is sent back to a ball mill 18, thence to tank 15, and the over-flow sent to Derr thickener 20 - 19. This over-flow from 20 - 19 flows to tank 21 to which an excess of zinc dust is added to completely precipitate the cadmium.

Classifier 24 classifies coarse and fine zinc: the coarse flows back to the head zinc tank 15; the over-flow goes to Derr 25 - 26. Spiget from 25 - 26 also returns to the head zinc tank 15; the over-flow from these Derrs continues on to the clarifier press 27, the filtrate resulting, being pure neutral zinc sulphate solution, goes into Feed Tank 28.

Cadmium Removal:

200 tons of concentrates at .338% cadmium =	0.676 tons
allowing for a 10.% roasting loss	<u>.068</u>
Cadmium in calcines =	.608 tons

Theoretically it would take 707 lbs. of zinc, if all the cadmium was soluble, to effect the precipitation of these 1216. lbs. of cadmium. For the efficient as well as complete removal of all the cadmium and any copper present, about 1.5 tons of zinc dust is used.

Cadmium solubility determination:

Undiluted filtrate from a leach made on 150 gms. of calcines assayed 0.34 gms./L. In the 150 gms. of calcines there was .507 gms. of cadmium (.338% of 150.). The total undiluted filtrate, which amounted to 1265. cc, should have assayed .401 gms. Cd./L (.507/1.265) — assuming 100% solubility. The actual assay showed a cadmium content of .34 gms./L; hence, there was obtained  $.34/.401 \times 100 = 85\%$  solubility.

With 85.% of the total contained-in-calcines-cadmium soluble, the precipitate resulting upon the addition of the 1.5 tons of zinc dust will contain about 1034. lbs. cadmium and 2399. lbs. zinc. Subsequent dissolving of cadmium and zinc and reprecipitating the cadmium will return 90.% of the zinc to the solution system.

$.9 \times 2399 = 2160 \text{ lbs.} = 1.08 \text{ tons soluble zinc (redissolved)}$

$2399 - 2160 = 239. \text{ lbs.} = .1195 \text{ tons zinc (lost)}$

This loss amounts to  $.1195/118. \times 100 = .104\%$

The .517 tons of cadmium upon precipitation will cause the dissolution of  $.517 \times (65.37/112.4) = .30 \text{ tons of zinc dust.}$

Cell Room:

Assuming the cell plant feed to contain 85. gms./L (zinc) and an average deposition (solution depletion) of 60. gms. per liter, the effluent will run 2.5% zinc. The zinc deposition of 60.gms./L will result in an acid regeneration of  $60. \times 3/2 = 90. \text{ gms. H}_2\text{SO}_4 \text{ per liter (9.\% sulphuric).}$

Adding up the zinc tonnage thus far calculated that will be in solution:

New zinc (page 60) -----	114.50	tons
Zinc in solution feed (1975 tons at 2.5% Zn.) ----	49.35	
Zinc dissolved by Cd. ppt. (page 66) -----	.30	
Excess zinc dust dissolved (page 66) -----	1.08	
	<hr/>	
	165.23	
less soluble zinc entrained in residue (page 63) -	.56	
	<hr/>	
	164.67	
less zinc in cell effluent (page 60) -----	49.35	
	<hr/>	
	115.32	
plus zinc dissolved from dross (page 68) -----	3.05	
	<hr/>	
Net tonnage to cathodes -----	118.37	tons

This cathodic deposition will regenerate:  $118.37 \times \frac{3}{2} = 177.5$  tons of sulphuric acid. This acid will constitute 9% of the cell effluent:  $177.5 / .09 = 1975$  tons. This will carry 2.5% zinc or 49.35 tons zinc.

In melting this 118.37 tons cathode zinc in a coal or gas fired reverberatory there will result in the production of 96.1% Bar Zinc — 113.76 tons.

This leaves 4.61 tons of zinc as dross. The dross is baked to remove chlorine coming from the sal-ammoniac used in melting. Then classified and two products obtained: fines and coarse. The fines are largely particles of ZnO and metallic zinc, while the coarse is all metallic zinc.



The coarse is ground in a ball mill to proper fineness. About 1.5 tons of the ground over-size is sent to the purification department and the fines from the classifier together with the balance of the ground oversize and also the ground undersize are dissolved in acid, the solution resulting being sent to general solution system.

$4.61 \text{ tons dross} - 1.5 \text{ tons zinc dust} = 3.11 \text{ tons.}$

assuming 98.% recovery of this by solution:

$.98 \times 3.11 = 3.05 \text{ tons zinc in solution sent to system.}$

General Quantity Flow Sheet

To Roaster: 200 tons concentrates -----	118.00	tons zinc
less roasting loss -----	1.18	
	116.82	
plus cell effluent (1975 tons at 2.5% zinc) -----	49.35	
	166.17	
To Leaching Plant -----	166.17	
less soluble and insoluble zinc in residue -----	2.88	
	163.29	" "
To Purification Tanks -----	163.29	

To the solution containing 163.29 tons zinc there are added 1.5 tons zinc dust for precipitating purpose, .30 tons of which goes into solution as the cadmium precipitates out. Upon redissolving of excess zinc dust in the zinc-cadmium precipitate, 1.08 tons of this 1.5 tons are also taken back into the solution. Then, there will be:

$$163.29 + 1.08 + .30 = 164.67 \text{ tons zinc in solution.}$$

To this amount there is also added 3.05 tons from dissolution of the excess dross (see page 68).

$$164.67 + 3.05 = 167.72 \text{ tons zinc which goes to the Cell Plant for electrolytic treatment.}$$

All but 49.35 tons of this (see page 67) are deposited, hence there is a cathodic deposit of:

$$167.72 - 49.35 = \underline{118.37 \text{ tons zinc.}}$$

Metal Balance Sheet

Solution System

Zinc Put In		Zinc Taken Out	
From Concentrates	118.00	Roaster loss	1.18
From Cell Effluent	49.35	Residue loss	2.88
From Zinc Dust	1.50	Cd-ppt. loss	.12
From Zinc Dross	3.05	As Cathodes	118.37
		Spent Electrolyte	49.35
	171.90		171.90

Melting Department

Zinc Put In		Zinc Taken Out	
Cathodes	118.37	Bar Zinc	113.76
		Dross	4.61
	118.37		118.37

Acid Balance Sheet

H <sub>2</sub> SO <sub>4</sub> Put In		H <sub>2</sub> SO <sub>4</sub> Taken Up	
Cell Room Regeneration 3/2 x 118.37 (page 69)	177.5	Zinc in calcines requiring acid 109.1 x 3/2 (page 60)	163.6
		Dissolving dross 3.05 x 3/2 (page 60)	4.57
		Dissolving excess zinc dust 1.08 x 3/2 (page 66)	1.62
		Dissolving zinc in Zn-Cd ppt. .30 x 3/2 (page 66)	.45
		Excess Acid (marginal safety)	7.26
	177.5		177.5

Recoveries:

Recovery of cathode zinc in terms of zinc in concentrates:

$$\frac{(163.29 - 49.35)}{118} \times 100 = 96.5 \%$$

Recovery of bar zinc in terms of zinc in concentrates:

$$\frac{(163.29 - 49.35) \times .961}{118} \times 100 = 92.7 \% \text{ (for one time thru)}$$

The dross, 96.1% of which is redissolved, is taken back into the system and is ultimately recovered as bar zinc. Therefore, the continuous daily bar zinc recovery in terms of zinc in concentrates is:

$$\frac{(163.29 - 49.35) - 3.9\% \text{ of } 4.61}{118} \times 100 = 96.35 \%$$

For Plant Recovery, the figure 92.7% has been taken for "Costs Comparison" somputations — ignoring the re-entry of the dross just as a matter of safety.

Losses:

Roaster Loss	----- 1.00 %	---	1.16 tons	(page 59)
Leaching loss	----- 2.44	---	2.88	(page 63)
Purification loss	- .10	---	.12	(page 66)
Dross loss	----- .05	---	.06	(page 68)

total loss 3.59 % --- 4.24 tons

Remarks:

The Roaster loss taken at 1.‰ is much too high. In practice this would amount to but a trace. The dust is returnable thru the recovery of same by using flue collectors, Cottrells, etc.

Residue losses are smaller than herein computed. Actual leaches showed approximately a ten to one reduction in weight from calcines to residue instead of a five to one ratio as was figured. This would make a correspondingly smaller soluble zinc ( $ZnSO_4$ ) loss entrained in the filter cake of lesser weight.

The addition of an insufficient amount of zinc dust to the first two zinc agitators gives a rich cadmium precipitate. A large excess of zinc dust in the other agitator insures complete removal of cadmium. A regrinding of the zinc-cadmium from the second tank is probably unnecessary.

### 3. Costs -- Comparison:

The Anaconda Electrolytic Zinc Plant which was built during war times cost about \$30,000. per ton slab zinc per day. This figure was a bit high due to two reasons: 1st, the general high prices of material on account of war-time demands; and, 2nd, the newness of the process -- everything having been untried on a commercial scale resulted in considerable re-vamping of design. The same plant could probably be built for \$24,000. per ton at the present date. On account of the small weight of residue, the leach department could be reduced somewhat. Normally, the percentage department cost referred to the total cost is about as follows:

Receiving & Regrinding Plant	1.94
Roasting Plant	13.10
Leaching Plant	11.35
Electrolytic Cell Plant	32.60
Sub-Station (power)	26.40
Zinc melting Plant	4.62
Office, Labs., and Yard	9.75

Inasmuch as the Leaching Plant is only about 11.35% of the total plant, a large saving in this department would not greatly affect the total cost. Taking the total cost at \$2,600,000., 11.35% for the leaching plant

would amount to \$294,000. If this was cut in half, the total cost would amount to

$\$2,600,000. - 294,000./2 = \$2,453,000.$  or  
\$22,200. per ton daily capacity.

(These are general figures only, and serve merely to show a probably trend.)

The operating costs of electrolytic plants compared with retort plants are composed of radically different quantitative amounts. The electrolytic plant requires small labor charges but high fixed charges; whereas, the retort plant carries low fixed charges and very high operating charges.

The power charges in an electrolytic plant may be apportioned as follows:

Regrinding plant	0.28 per cent
Roasting plant	.22
Leaching plant	1.43
General	.28
Electrolytic power	97.79
	<hr/>
	100.

Using a current efficiency of 87.5%, voltage at 3.5, there can be obtained a deposition of

$2.69 \times .875 = 2.35 \#$  zinc per 1000 amp. hr., and a power consumption of

$3.5 \text{ KW} / 2.35\# = 1.49 \text{ KWH per lb. of zinc (2980KWH/ton)}$

With Rotary Converter installation, the conversion losses would amount to less than 9%. Assuming this figure, however, to be the case and assuming other losses at 1%, there would be an efficiency of 90%.

$2980. / .9 = 3310.$  KWH per ton A.C. cathode zinc.

With a melting efficiency of 96%, the power per ton of bar zinc would be:  $3310. / .96 = 3450.$  KWH. (electrolytic power). This would amount to 2070. KWH. per ton of 60% concentrates.

The total power for one ton of Bar Zinc produced would be:  $3450. / .9779 = 3530.$  KWH., since the electrolytic power equals 97.79% of the total power to the plant.



Power Cost

Power Plant Cost

In order to arrive at a fair figure for the cost of power to charge to the cost of producing electrolytic zinc, a brief outline together with calculations and a summary of charges for the cost of a modern coal-burning power plant (KWH capacity sufficient to handle the treatment of 200 tons of concentrates per day), is herewith set forth — cost per KWH figures for 100% and 75% load factor are shown at coal costs of \$2.00, \$3.00, and \$4.00, respectively.

Under the following conditions for power generation a modern coal-burning plant will develop power (electrical) in terms of coal weight as listed at the end of this paragraph:

steam economy: 250# pressure, 200 deg. F. superheat.

	power factor	# steam / KWH	
	1.00	11.5	
	0.80	11.7	
at average load:	# steam / KWH	B.T.U.s above 70. deg. F./KWH.	
Turbine	11.50	14,700	
Auxiliary	.92	1,175	
Misc.	.46	598	
	<u>12.98</u>	<u>16,473</u>	
credit for return to feed water:		<u>1,023</u>	
Net	-----	15,450	

Assuming the steam generator equipment efficiency to be 73%, then the B.T.U. requirement will be:  
 $15,450 / .73 = 21,160$  B.T.U.s, which, in terms of coal (of 12,700 B.T.U. value), equals:  
 $21,160 / 12,700 = 1.665$  #. Therefore, 1.665 # of coal is equivalent to 1.0 KWH.

On page 75 it is shown that the power consumption was approximately 3600. KWH per ton of Bar Zinc produced. With an output of 113.8 tons of Bar Zinc the KW load would be:  $\frac{3600 \times 113.8}{24} = 17,070$  KW.

For safety a plant of 20,000 KW capacity was taken as the basis for the following outline.

Pre-war cost = \$75. per installed KW capacity.

1921 cost = \$135. per installed KW capacity.

1924 cost = \$175. per installed KW capacity.

At the 1924 cost rate (which was advised by the Illinois Power & Light Company) the total cost of the power plant was figured:

$$20,000 \times 175. = \underline{\$3,500,000.}$$

Coal Consumption:

at 100.% load factor:

$$113.8 \times 3600 \times 365 = 150,000,000 \text{ KWH/Year}$$

$$150,000,000 \times 1.665 = 250,000 \text{ tons coal per year}$$

at 75.% load factor:

$$.75 \times 150,000,000 = 112,500,000 \text{ KWH/Year}$$

$$.75 \times 250,000 = 187,500 \text{ tons coal per year}$$

Coal Cost:

	100.% load factor	75.% load factor
at \$2. per ton	\$250,000.	\$187,500.
at \$3. per ton	375,000.	281,250.
at \$4. per ton	500,000.	375,000.

Basis upon which the following Power Cost was calculated:

cost of plant -----	\$3,500,000.
daily bar zinc production -----	113.8 tons
power consumption rate -----	3,600. KWH/ ton bar Zn.
coal-power equivalence -----	1.665#/KWH.

Power Cost Sheet

	100.% load factor		75.% load factor	
K W H per year	150,000,000		112,500,000	
	Yearly	cost per KWH	Yearly	cost per KWH
Insurance - 1.5% of plant cost	% 52,500.		\$ 52,500.	
Amortization - 10 yrs @ 5% cpd.	277,778.		277,778.	
Interest on total investment, 8.%	280,000.		280,000.	
Taxes, 2% of plant cost	70,000.		70,000.	
Maintenance @ 3% of plant cost	105,000.		105,000.	
Operating labor, salaries @ \$125. per day	45,625.		45,625.	
Misc. Operating supplies	10,000.		10,000.	
Total Cost ex- clusive of coal	\$ 840,903.	.00561	\$ 840,903.	.00748
Coal Cost: @ \$2./ton:	\$ 250,000.		\$ 187,500.	
@ \$3./ton:	375,000.		281,250.	
@ \$4./ton:	500,000.		375,000.	
Total Power Cost with -				
Coal @ \$2./ton:	\$1,090,903.	.00727	\$1,028,403.	.00914
@ \$3./ton:	1,215,903.	.00811	1,122,153.	.00997
@ \$4./ton:	1,340,903.	.00894	1,215,903.	.01081

On the basis of treating 73,000 tons of concentrates per year the following power costs per ton of concentrates would appear for \$2., \$3., and \$4. coal:

\$2. per ton coal:

100% load factor:  $1,090,903/73,000 = \$14.94$

75% " " :  $1,028,403/73,000 \times .75 = \$18.78$

\$3. per ton coal:

100% load factor:  $1,215,903/73,000 = \$16.66$

75% " " :  $1,122,153/73,000 \times .75 = \$20.495$

\$4. per ton coal:

100% load factor:  $1,340,903/73,000 = \$18.36$

75% " " :  $1,215,903/73,000 \times .75 = \$22.208$

### Electrolytic Plant Cost

Considering \$2,600,000. as the cost of the electrolytic plant (exclusive of power generation) for treating 200 tons of concentrates per day, the following Over-head Cost distribution would be a fair representation of the yearly "capital charges".

Amortization - 10 yrs @ 5% cpd	\$ 206,200.
Insurance - 1.5% of plant cost	39,000.
Interest for 1/2 period @ 5%	65,000.
Taxes, 2% of plant cost	52,000.
Medical Liabilities	8,000.

Total ----- \$ 370,200. per year, or  
 $370,200 / 365 = \$1014.20$  per day, or  
 $1014.20 / 200 = \underline{\$5.07}$  per ton of concentrates.

With labor charges and materials totaling an average of \$8.50 per ton, and allowing a "miscellaneous" charge of \$1.00 per ton, the cost of treating one ton of concentrates (exclusive of power) can be listed as:

Labor & Materials -----	\$8.50
Capital Charges -----	5.07
Miscellaneous -----	1.00
	<u>\$14.57</u> per ton.conc.

With coal at \$2., \$3., and \$4. per ton, and load factors of 100% and 75%, the total cost of electrolytic treatment of zinc concentrates would be:

\$2. Coal — 100% L.F. )	14.57 + 14.94	=	<u>\$29.51 / ton.</u>
power: \$.00727/KWH )			
— 75% L.F. )	14.57 + 18.78	=	<u>33.35 / ton.</u>
power: \$.00914/KWH )			
\$3. Coal — 100% L.F. )	14.57 + 16.66	=	<u>31.23 / ton.</u>
power: \$.00811/KWH )			
— 75% L.F. )	14.57 + 20.495	=	<u>35.065 / ton.</u>
power: \$.00997/KWH )			
\$4. Coal — 100% L.F. )	14.57 + 18.36	=	<u>32.93 / ton.</u>
power: \$.00894/KWH )			
— 75% L.F. )	14.57 + 22.21	=	<u>36.78 / ton.</u>
power: \$.01081/KWH )			

Assuming a power cost of \$.008 / KWH, and using a power consumption of 2070 KWH per ton of 60% concentrates (see page ), a fair figure for the total electrolytic cost per ton of concentrates would be:

$$(2070 \times \$.008) + \$14.57 = \underline{\underline{\$31.13}}$$

For the purpose of arriving at a comparison between the cost of treatment of the Retort Plant and the cost of treatment of the Electrolytic Plant it is correct to deduct from the latter cost the differential recovery value as derived below:

Considering one ton of concentrates (2000 lbs.):

Grade of Conc:	40.%	50.%	60.%
Recovery:			
Electrolytic	85.%	89.%	92.7%
Retort	80.	83.	85.
Zinc recovered: #/ton conc.			
Electrolytic	680.	890.	1112.4
Retort	<u>640.</u>	<u>830.</u>	<u>1020.</u>
Difference --	40. #	60. #	92.4 #

Value of Excess Zinc by the Electrolytic Process:

Zinc @ 5¢	\$2.00	\$3.00	\$4.62
" @ 6¢	2.40	3.60	5.54
" @ 7¢	2.80	4.20	6.47
" @ 8¢	3.20	4.80	7.39

Allowing for a one-quarter cent premium on the Electrolytic product, this would amount to:

\$1.70	\$2.225	\$2.78
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Adding the premium increase to the respective values of the excess zinc by the Electrolytic Process, a total differential recovery value per ton conc. is listed below:

Zinc @ 5¢	\$3.70	\$5.225	\$7.40
" @ 6¢	4.10	5.885	8.32
" @ 7¢	4.50	6.425	9.25
" @ 8¢	4.90	7.025	10.17



Electrolytic Plant treatment cost = \$31.13 per ton 60% concentrates. (page 82).

Deducting differential recovery values for the same grade concentrates:

Zinc At 5¢	\$31.13	- 7.40	= \$23.73
" " 6¢	31.13	- 8.32	= 22.81
" " 7¢	31.13	- 9.25	= 21.88
" " 8¢	31.13	-10.17	= 20.96

It might be brought out at this point that the power consumption for the Electrolytic Plant is practically independent of the grade of ore treated, and is 3600 (in round numbers) KWH per ton of zinc output regardless as to whether that zinc is made from 60.% concentrates or from 25.% ores. On the other hand, with the retort process as well as with the electric smelting process, the power consumption, the coal consumption, and in fact the costs as a whole, are practically the same for a ton of material treated, without regard to the zinc available from that ton of material.

Lead as a By-Product:

A further credit to the Electrolytic Zinc Plant is the residue containing lead, about 90% of which can readily be recovered by brine leaching, as per O.C.Ralston. From treating 200 tons of concentrates per day a daily residue of 36 tons (averaging a little better than 20.% Pb) was obtained (see page 61). Disposal of this residue to a lead smelter would make a marked saving which would decrease the per ton cost of the electrolytic zinc.

Using several different smelter schedules to figure the value of this residue, one of which is shown below, an average figure of \$16.90 per ton was obtained:

Smelter Schedule Calculations:

$$(90.\% \text{ of } \$4.00) + 1/2 (4.50 - 4.00) + 6.80 - 4.50 = \$6.15$$

$$\$0.47 + 1/5 (6.15 - 4.00) = \$0.90 \text{ (per unit)}$$

Residue assay: 20.% Pb, 3.% Fe, and 15.% SiO<sub>2</sub>

$$\begin{array}{r} \text{Credit for lead} \text{ --- } 20.0 - 1.5 = 18.5 \text{ units} \\ 18.5 @ \$0.90 = \quad \quad \quad \$16.65 \end{array}$$

$$\text{Credit for iron} \text{ --- } 3 \text{ units } @ 10\text{¢} = \quad \quad \quad .30$$

$$\text{total credit} \text{ ----} \quad \quad \quad \underline{\$16.95}$$

$$\text{Debit for SiO}_2 \text{ --- } 15 \text{ units } @ 10\text{¢} = \quad \quad \quad \$ 1.50$$

$$\text{Debit for smelter treatment} \quad \quad \quad = \quad \quad \quad 5.00$$

$$\text{Debit for freight} \quad \quad \quad \quad \quad \quad = \quad \quad \quad \underline{3.00}$$

$$\text{total debit} \text{ ----} \quad \quad \quad \underline{\quad \quad \quad 9.50}$$

$$\text{net credit} \text{ ----} \quad \quad \quad \underline{\quad \quad \quad \$ 7.45/\text{ton.}}$$

For 36 tons this credit would amount to \$268.20 per day, or \$1.34 per ton concentrates — which can be credited to the general returns from the Electrolytic Zinc Plant.

In addition to the lead, the flotation concentrates (and also the jig concentrates) carry about .34% cadmium. A plant treating 200 tons of concentrates per day at .34% cadmium would yield about .52 tons Cd./day — as only about 76.5% of the total cadmium in the concentrates could be recovered; hence, the cadmium output would amount to about 1040. lbs. per day. At the present day the cadmium market could not absorb this; hence, it becomes necessary to seek a new demand for its use. Such an outlet would be feasible thru the manufacture and sale of cadmium solder, a product which has been developed somewhat and which would upon further exploitation undoubtedly furnish an avenue of cadmium disposal at a profit.

### Uses of Metallic Cadmium:

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An extract taken from an article: "Electrolytic Cadmium", by H. R. Hanley; Chem. and Met. Engr., Dec. 29, 1920.

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"Cadmium statistics collected by Siebenthal in 1918 show that the use of this metal in the manufacture of solders has received some attention. This was of special interest during the late war, when in the first three-quarters of the year 1918 there was a scarcity of tin. It was known early in the war that the Germans, without resources in tin but with a fair supply of cadmium, were substituting that metal for tin. It is now known that solders with 10 per cent Cd and 10 per cent Sn were used and the cadmium content in other solders ranged as high as 30 per cent Cd with 2 per cent Sn. This authority quotes the following solder composition:

parts Pb.	parts Cd.	parts Sn.	Melting range deg. C.
84.4	7.6	5.	212 - 253
90.6	7.9	1.5	237 - 267
87.5	7.5	5.	235 - 368

"The cost of materials for tin and cadmium solders of various formulas, Oct. 1, 1918, is shown in table 11. At these prices solder composed of 80 parts of lead, 10 parts of tin and 10 parts of cadmium can be made as cheaply as half-and-half solder with cadmium at \$3. per lb. AT the close of

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Table 11. Cost of Material in 100 lb. Solder,  
as of Oct. 1, 1918.  
lead 8.05¢, tin 80.¢, cadmium \$1.50 per lb.

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	50 Pb-50 Sn;	60 Pb-40 Sn;	80 Pb-10 Sn-10 Cd;	92 Pb-8 Cd
Pb	\$ 4.03	\$ 4.83	\$ 6.44	\$ 7.41
Sn	40.00	32.00	8.00	-----
Cd	-----	-----	15.00	12.00
	-----	-----	-----	-----
	\$44.03	\$36.83	\$29.44	\$19.41

---

1918, with a lower price for tin and lead, the price of the foregoing combination is as shown in table 111. With the average prices before the war of about 4.5¢ per lb. for lead, 35.¢ per lb. for tin and 80.¢ per lb. for cadmium, the foregoing solders would cost respectively, 19.7¢, 16.7¢, 15.1¢, and 10.5¢ a lb. for the raw materials."

Table 111. Cost at Close of 1918.  
lead 5.75¢, tin 69.¢, cadmium \$1.50 per lb.

50 Pb-50 Sn; 60 Pb-40 Sn; 80 Pb-10 Sn-10 Cd; 92 Pb-8 Cd.				
Pb	\$ 2.88	\$ 3.45	\$ 4.60	\$ 5.29
Sn	34.50	27.60	6.90	—
Cd	—	—	15.00	12.00
	—————	—————	—————	—————
	\$37.38	\$31.05	\$26.50	\$17.29

The following table is given in order to show the relative costs of the above-mentioned solders at the present-day prices, viz., lead 7.25¢, tin 41.¢, and cadmium 60.¢ per lb., as per E. & M. J-P., May 1924.

Costs of Material in 100 lbs. Solder.  
As of May 24, 1924.

50 Pb-50 Sn; 60 Pb-40 Sn; 80 Pb-10 Sn-10 Cd; 92 Pb-8 Cd.				
Pb	\$ 3.63	\$ 4.35	\$ 5.80	\$ 6.67
Sn	20.50	16.40	4.10	—
Cd	—	—	6.00	4.80
	—————	—————	—————	—————
	\$24.13	\$20.75	\$ 15.90	\$11.47

### Cadmium as a By-Product:

Process for the recovery of cadmium from the Zn-Cu-Cd mud:

In order to derive as high a profit as possible by the recovery and sale of cadmium it is advisable to include a department for the treatment of the cadmium mud and the manufacture of cadmium solder in the Electrolytic Plant.

For the recovery of cadmium from the above-mentioned mud, such a process as <sup>was</sup> ~~is~~ in practice at Kennett, California and which was just recently installed in Tasmania (where a daily output of 500 lbs. of cadmium is made) would be quite suitable. The process consists in six steps:

1. Leaching the zinc-copper-cadmium mud with dilute sulphuric acid at about 60 deg. C. This dissolves the cadmium and zinc but not the un-oxidized copper.
2. Separation of solution from the residual copper mud by filtration. (Copper must not be permitted to oxidize.)
3. Precipitation of cadmium from solution as sponge metal by the addition of an insufficient amount of zinc dust. The zinc sulphate solution remaining is sent to the solution system of the Electrolytic Zinc department.
4. Solution of the cadmium in acid solution (cadmium electrolyte), the acid being regenerated in the electrolytic cells.

5. Removal of iron (and thallium when necessary) from the solution; addition of lime for iron precipitation and the addition of sodium dichromate (see below) for thallium removal.

6. Electrolytic deposition: 80. gm./L cadmium solution depleted to 25. gm./L.

If thallium is present in the Zn-Cu-Cd mud, special means for its removal must be used prior to electrolytic deposition, for it has a decidedly detrimental effect on the character of the cadmium deposit, and further causes an undue amount of oxidation of the cadmium in the melting of the cathodes.

#### Removal of Thallium.

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An extract taken from an article: "Electrolytic Cadmium", by H. R. Hanley; Chem. and Met. Engr., Dec. 29, 1920.

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"Thallium resembles lead and manganese in some of its reactions and has two valences. The different characteristics of the lower and higher valence of the metal in solution have an important bearing on its separation from cadmium. Thallous compounds are not precipitated by hydrated lime, limerock, or other bases, whereas cadmium is precipitated by almost all bases except limerock. Thallic compounds are very easily reduced and the higher valence can be obtained only by strong oxidizing agents, such as potassium permanganate, bromine, iodine, etc., or by the anodic oxygen produced in the electrolytic cell. The higher form is transitory only and is slowly reduced on standing, after the effect of the oxidizing agent is gone.

"Precipitation of cadmium by a base in presence of the soluble thallous salts is possible, but involves the

handling of large quantities of cadmium precipitate. The removal of thallium in the thallic state by a base after electrolysis is not economical, because of the loss of acid which would be sustained, nor is it feasible, because of the indefinite valence of the element. In neutral solution thallic salts can be precipitated completely by sodium bichromate without producing any chemical effect on the cadmium, but cannot be precipitated from the solutions that are slightly acid. This gives a simple method of removing thallium from the solution before electrolysis as a chromate, and this step is performed when the metal has reached a concentration of 0.3 gm./L. One pound of commercial sodium bichromate will remove approximately 1.36 lbs. thallium."

When arsenic is present in the Zn-Cu-Cd mud the process as previously outlined is changed slightly, the steps being substantially the same as shown but their order of sequence is changed --- the removal of the arsenic with the iron is brought about before the precipitation of the cadmium as sponge metal. Thruout, great care is exercised to have the arsine that is formed removed so as not to poison the plant atmosphere.

Electrolytic deposition is performed using vertically rotating disc cathodes (aluminum) set 9 inches apart. Double anodes, that is, two anodes per each cathode, has proven most successful. Operation with the double anodes implies the use of a cell having a volume approximately double that for regular anode installation. Because of the high ratio between the cell volume and the immersed cathode area, it is feasible



to operate each unit as an accumulating acid cell. Such cells require no feed and the electrolyte gradually increases in acidity as the cadmium decreases in concentration. Practically no attention is required except to fill the cells, strip the deposits (which is easily done on account of the large clearance space between cathodes), and discharge the acid electrolyte.

Cost of cadmium recovery:

Power:

Theoretically, there is 4.61 lbs. cathode cadmium deposited per 1000. amp.-hr. With a current of 1200 amp. cell voltage of 4. volts, line loss of .4 volt, and generator efficiency of 84.%, the power required for one cell, is:

$$24(1200 \times 4.4) / .84 = 151. \text{ KWH for 24 hr. operation.}$$

The cadmium produced in twenty-four hours will be at 85% current efficiency:  $24 \times 4.61 \times 1.2 \times .85 = 113 \#$

The KWH required per lb. of cathode cadmium will be:

$$151 / 113 = 1.335 \text{ KWH} / \# \text{ Cd.}$$

The power required for the recovery of 1030 lbs. of cadmium would be:  $1030 \times 1.335 = 1375. \text{ KWH}$ , which, at  $\$.008 / \text{KWH}$  would amount to:  $\$11.00$ .

Labor Cost:

The time element involved in the production of 1030# of cadmium may be stated in the number of man-hours, each department being listed separately.

Leaching, purification, solution, etc. -----	66.7
Cell room operation, including stripping ---	12.8
Melting to bars, drying, etc. -----	20.6
	-----
Total -----	100.1

This total of hours at an average rate of 50¢/hr. would amount to \$50.05

Estimating a general expense of \$41., the total cost of producing 1030 lbs. of cadmium would be:

Power -----	\$ 11.00
Labor -----	50.05
Gen. Expense -----	41.00
	-----

total ----- \$102.05 , or,

when allowing for 97% recovery of the cadmium from the cadmium mud, =  $102.05 / .97 \times 1030 =$   
10.21 ¢ per lb. For safety assume the cost to be 12¢ per lb.

Manufacture of cadmium solder:

The profit to be derived from the manufacture and sale of a solder of the following composition, lead 60% - tin 10% - cadmium 30%, is shown below in the computations in order to arrive at an approximate figure with which the Electrolytic Plant may be credited for the recovery of cadmium as a by-product.

Basis of computations: 100 lbs. solder.

Pb @ 7.¢ / lb., Sn @ 40.¢ / lb., and Cd @ 12.¢ / lb.

	%	Cost of material
Pb	60	\$ 4.20
Sn	10	4.00
Cd	30	3.60

\$11.80 material  
4.00 labor

Total/100# \$15.80 = 15.8¢ per # Cd solder.

Regular "Half-and-half" solder would cost as follows:

Pb @ 7.¢ / lb., Sn @ 40.¢ / lb.

	%	Cost of material
Pb	50	\$ 3.50
Sn	50	20.00

Cost of material \$23.50  
labor 4.00

Total/100# \$27.50 = 27.5¢ per # Regular solder.

27.5 - 15.8 = 11.7 ¢ difference

Figuring the market price of regular solder at 30.¢ per lb., and allowing for a drop in price for the cadmium solder of

2.5¢ per lb., the apparent market value for this cadmium solder would be 27.5¢ / lb.

Then  $27.5 - 15.8 = 11.7$  ¢ profit per lb.

With a daily net production of 1000 lbs. of cadmium from 200 tons of concentrates treated in the Electrolytic Plant there would be a daily production of  $1000 / .3 = 3333$  lbs. of cadmium solder. This would give a total profit of  $3333 \times 11.7 / 100 = \$390$  / day.

$\$390 / 200 = \underline{\$1.95}$  per ton concentrates -- which can be credited to the general returns from the Electrolytic Zinc Plant.

Final Cost sheet for Electrolytic Zinc: 60 % concentrates, basis.

Total Cost of Electrolytic Zinc	-----	\$31.13 / ton 60% conc.
Deduct credit due to increased recovery	-----	\$ 4.62
Deduct credit due to premium price of product	-----	2.78
Deduct credit for lead product recovery	-----	1.34
Deduct credit for cadmium recovery (solder sale)	-----	1.95
	-----	
Total credits	-----	10.69
Net Cost of Electrolytic Zinc	-----	\$20.44 / ton 60% conc.

Comparison:

The average cost of producing metal from calcine in a highly perfected regenerative furnace plant (Retort Process) operating at full capacity is ----- \$25.00/ton conc.

Net Electrolytic Process Cost ----- 20.44/ton conc.

a difference in favor of Electrolytic Process \$ 4.36/ton con.

Granting that with the installation and growth of the Electrolytic Practice in the Tri-State field there would soon disappear the one-quarter cent premium as above accredited, there still would be an appreciable balance (difference) in the cost of zinc production in favor of the Electrolytic Process, viz., \$1.58 per ton concentrates.

## Part IV.

### Summary and Conclusions:

It is well known that the production of zinc from ores is almost exclusively accomplished by the Retort Process. This process requires for its success:

1. High grade concentrates,
2. Cheap fuel, and
3. Cheap labor.

The high grade concentrate is fairly easy to produce with ore from the Joplin-Miami field. In other fields, however, this is by no means assured — especially in the case of ores containing fair quantities of lead and iron. As the grade of concentrate is lowered the cost of treatment per ton of concentrate increases.

The subject of fuel and its combustion is a matter of greatest importance. There is no other major metal of commerce which is produced at the expense of so much coal per pound of metal produced as is the case with zinc. In most cases the ore must be carried to the fuel supply rather than the fuel to the smelting plant. Even

under this premise the cost of fuel and labor constitute over 80.% of the total cost.

Furthermore, it appears that it is difficult to mechanicalize the Retort Process. Much thot and effort have been given to this feature and, altho material saving has resulted as a consequence of this, there still remains many essential steps that must be carried out by hand labor.

Notwithstanding the simplicity of the distillation process in theory, its practical application is attended by so many inherent difficulties that development on the lines that have been followed in the metallurgy of lead and copper, especially the introduction of furnaces of large capacity, and labor-saving devices, has been impossible, and, consequently, the metallurgy of zinc remains to-day essentially the same as at the time of its early practice. Chief among these difficulties is the ease with which zinc vapor can be oxidized and the necessity of producing a gas of high percentage of zinc. This limits the size of the vessels in which the reduction ( $ZnO$  to  $Zn$ ) can be effected to retorts of small capacity. The handling of the calcines in and out of

the retorts and the general maintenance of the retorts and various devices are features difficult of mechanicalization and, therefore, must be done by hand. This entails a large amount of labor.

The recovery of zinc in the Retort Process is not more than 80.% for 40.% concentrates and 87.% for 60.% concentrates, altho in some cases it has been reported to reach 89. to 90.% on the highest grade of concentrates.

The Electrolytic Process is a process capable of almost unlimited mechanicalization. For obvious reasons it is more economic to treat high grade concentrates; yet, the extremely high grade concentrates are not essential. The Electrolytic Process can treat economically concentrates as low as 30.% zinc under certain conditions. It is significant that as the grade of concentrates offered for treatment is lowered, the treatment cost per ton of concentrates decreases for the Electrolytic Process; whereas, it will increase, as previously stated, for the Retort Process. It is seen then that the position of the Electrolytic Process is strengthened where lower grade concentrates are to be treated.



The cost of Electrolytic and Retort Plants is a matter of major importance. The Electrolytic Plant without the primary and secondary power station will be comparable, as regards the cost of construction, with the Retort Plant, which is about \$8500. to \$9500. per ton of concentrates per day. The Electrolytic Plant will carry the secondary power station investment and, in the end, the total investment for the Electrolytic Plant will be much greater than for the Retort Plant. Considering the primary power station as a separate self-sustained investment -- which it would undoubtedly result in, as the adoption and installation of the Electrolytic Process in the Tri-State field would present apparent promising opportunities to power plant promotion -- is the most reasonable manner in which to compare the two zinc processes and their respective plants as regards costs.

The Electrolytic Process must have cheap power. A modern coal-burning power plant, however, will develop sufficient KWH's from one ton

of coal to electrolytically deposit .34 ton of zinc in marketable form. In contrast to this, one ton of coal used in the Retort Plant will produce only .255 tons of zinc. The following calculations are added for explanation:

**Electrolytic Plant:**

1 KWH is equivalent to 1.665 lbs. of coal  
 1 ton coal equals 2000/1.665 or 1200 KWH  
 1 ton of bar zinc requires 3530 KWH, or  
 3530/1200 equals 2.94 tons of coal  
 1 ton of coal, therefore, will equal  
 1/2.94 or .34 tons zinc.

**Retort Plant:**

Coal for heating -----	3000 lbs.
Coal for reduction -----	750 lbs.
Coal for blue powder ----	130 lbs.
Coal for luting -----	120 lbs.

Total coal required ----- 4000 lbs. or 2 tons

That is, 2 tons of coal per 85.% (recovery)  
 per one ton of 60.% concentrates.  
 .85 x .6) x 2000 equals 1020 lbs. zinc  
 Therefore, one ton of coal equals  
 1020/2 or 510 lbs. or .255 tons of zinc.

It will be seen then that one of the chief and expensive items of the Electrolytic Process is less costly than the similar item (heating and reducing fuel) in the Retort.

On account of the large power plant KWH capacity that is necessary to supply the Electrolytic Plant the incorporation of a separate company and the building of a plant to supply not only the demand of the Electrolytic Plant but also other industry, <sup>would result</sup> -- in which case special rates could undoubtedly be secured by the Electrolytic Plant for power on account of its steady 24-hour (high load factor) demand, and consequently the result would be a decrease cost of zinc production.

For the purpose of making the power plant promotion more attractive to investors, a 10 % interest on investment could be paid to stockholders instead of the 8 % as herein calculated under "Power Cost". This 2 % increase in capital cost would result in a unit power cost rise of .00047/KWH, which would result in an increase of  $(3530 \times .00047 \times 113.8) / 200$  or \$0.925 per ton of concentrates. Permitting <sup>increase in power cost</sup> this, there still would be a difference in <sup>^</sup> production costs in favor of the Electrolytic Process.

This thesis was only intended to roughly outline the statement of the problem in preparing to treat Joplin-Miami concentrates by the Hydro-Electric Process, and hence, throughout, the term "for safety" has appeared — as a result of which, the equipment needed and the recoveries of zinc, lead, and cadmium, made, were over and under calculated, respectively. As a consequence, the final figure on the Cost of Producing Electrolytic Zinc per ton of 60% concentrates is probably higher than actual practice would make it.

Summing up the Credits:

1. Increased recovery of zinc: from 85% (Retort) to 92.7 % (Electrolytic) — laboratory test showed 96.4 % — and probably as high as 95 % (per F. Laist),
2. Probably lower cost per pound, taking increased recovery into consideration,
3. Probably a premium price for a part of the production — this, in time, would disappear,
4. Sale of lead as a by-product,
5. Sale of cadmium as a metal or as a solder,
6. If power was developed by water falls, great conservation of coal and gas, and
7. Treatment of lower grade concentrates would be a feature in which the Retort Plant could not compete,

it is seen that from a metallurgical and economic viewpoint the Treatment of the Joplin-Miami Zinc Concentrates by the Hydro-Electric Practice is but a matter of time — how long? — it is difficult to say, for it will mean the complete revolution of one of the oldest processes of metallurgical industry.

Part V.

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