Experimental Treatment of Side Products Formed in Smelting the Silver Lead Ore of Newburyport Mass.

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A sample of about four and a quarter tons weight of ore from the Merrimac Mine, consisting largely of third grade, with less than ten per cent. of first class ore, was sent to the Institute by the superintendant, Mr. Edgar Shaw, for experimental treatment. It was worked upon by the following gentlemen as a subject for a graduating thesis.

Messrs Flint and Stimpson took charge of the separation of ore from the gangue, with a view of ascertaining the efficiency of our apparatus, as applied to ore of this grade.

Messrs Jenney and Wood took the middlegrade ore to seek an economical method of working it.

Mr Hibbard and myself investigated the rich products, or smelting ore in order to work it thoroughly, and separate everything of value.

Mr Hibbard paid particular attention to the cupelling, while I studied the treatment of the various residues, while both worked together in roasting, smelting, and refining.

On a rough calculation the ore as sent us consisted of

Galena	6 %
Mispickel	3 "
Blende	8 "
Siderite	10 "
Chalcopyrite	1 "
Pyrite	5 "
Tetrahedrite	.5 "
Total ore	33.5 %
Quartz	40. %
Serpentine	15 "
Chlorite	10 "
Total gangue	65. %

I afterwards found in the treatment of the ore, that my estimate of the total ore should not have been over 25 % of the amount sent us.

The ore was crushed to 1/2 in. and passed over a cone, thence the heavier parts over the jigs, and the lighter through a series of Spitz-kastens, and thence over the side bump table; the richer tailings were then treated on the end bump, and the richest of these products repassed over the side bump.

The separation was made into three parts, 1° Gangue, consisting of 65 % of the total ore and assaying ($4) four dollars per ton in silver.

2° Middlegrade, containing the Blend, Siderite, Mispickel etc. and assaying ($9) nine dollars per ton in gold, and ($12) twelve dollars per ton in silver.

3° Rich, containing Galena, Tetrahedrite, larger part of Pyrite etc. and assaying ($14.42) fourteen dollars and fortytwo cents per ton in gold, and ($21.67) twenty one dollars and sixty seven cents in silver.

Full accounts of the Gangue, its percentages of lead, silver, etc. will be found in the essays by Messrs Flint and Stimpson, while the Middlegrade is described by Messrs Jenney and Wood, so that I shall now confine myself to the treatment of the third product, the rich grade ore called smelting ore.

There was run to the cone a total of 8485 lbs of the crushed ore, from which Mr Hibbard and myself received,

Smelting ore 572 lbs.
Very rich " " 56 ".
Slimes from overflow tank 154 lbs.

The ordinary smelting ore was roasted, as will be described hereafter, but the 56 lbs of very rich were retained intact for use in cutting away the sows which might form in the furnace, and to furnish sulphur for the treatment of the various residues.

Analyses of the above three products will be found on page 13

It will be noticed that the slimes were very poor, containing only 7.60 o/o of Lead to 45.79 o/o of Silica, so that only a few pounds were used and that only for the silica contained.

The smelting ore was considered a very good product, the small amount of silica showing an excellent separation. It contained

Pb	39.5 o/o
S	18.9 o/o
SiO_2	6.2 o/o.

We thus had a good ore to experiment on, ores being roasted and smelted in the Upper Hartz containing only 4 - 10 o/o of lead.

For roasting we used the hollow bed reverberatory furnace in the Institute laboratory, and the results, although not new, may serve to check some of the former runs, hence I give below the tabulated results.

No. of Charge	Time charged	Time drawn	Interval	Wt ore	Wt coal
1	10.15 AM	1.55 PM	3.40	46 lbs	187 lbs
2	2.05 PM	5.58 "	3.53	46	45 5/8
3	6.03 "	— "	—	46	51 2/8
4	10.03 "	2.00 AM	3.57	46	82 7/8
5	2.05 AM	6.00 "	3.55	46	57 3/8
6	6.05 "	10.00 "	3.55	46	53 7/8
7	10.07 "	2.00 PM	3.53	46	48 6/8
8	2.03 PM	5.55 "	3.52	46	52 3/8
9	6.05 "	9.55 "	3.50	46	72 2/8
10	10.05 "	2.00 AM	3.55	46	49 3/8
11	2.07 AM	5.57 "	3.50	46	45 3/8
12	6.00 "	9.50 "	3.50	46	59 5/8
Total 12			46.30	572	835 6/8
Average			3.52 1/2	46 (47 2/3 ?)	69 31/48

Fire lit at 7.20 AM.

The total ore charged to the furnace was 572 lbs, and the ore drawn amounted to 465½ lbs. The analyses of the two compared take the following form - according to Mr. Hibbards analyses.

Ore charged		Ore drawn.
Pb	39.5	43.94
Fe	14.6	17.80
S	18.9	3.28
Al_2O_3	.7	—
Cu	.6	.83
SiO_2	6.2	6.59
MgO	3.3	3.98
ZnO } MnO }	.7	.92
As	trace	

The percentage lost, amounts to 18.6 % of the ore as charged, of which 16.2 is due to the loss of S, 1.7 to Pb, and the remainder due to Zn As etc. with the exception of 1.62 lbs not cleared from the furnace until agglomerating.

After thus seeing where the losses occurred, it would be well to criticise the working of the furnace itself.

Our results show that it requires about 117 lbs of coal to start a fire in the furnace, and afterwards, an average of $69\frac{3}{48}$ lbs every four hours to roast at the heat required.

The average amount of coal could undoubtedly have been greatly reduced, if the furnace had been in charge of an experienced fireman, or even of the same man throughout, but the men being shifted once every four hours, each man was compelled to learn to regulate it himself, thus wasting coal.

This waste is shown by charges No 4 & 12, at both of which an amount greatly above the average was used, on account of the previous man allowing his fire to die down too much; thus requiring more coal to build it up, than would have sufficed to have kept a good heat all the time.

From the above we see that for each pound of ore roasted 1.45 lbs of coal were used.

The average time required for the absolute roasting of the charges was 3 hrs. 52.5 Min., the remaining 7½ minutes of each shift of four hours, being devoted to drawing and charging, and the shortness of the time, shows that the furnaces were as well managed as was possible under the circumstances.

The working of each roast was intended to begin with a rather low heat, and after running along with a medium heat, stirring, as we found it necessary to do, every five or ten minutes for about three hours, the heat was raised as high as was possible without agglomerating the ore, for the remaining hour being stirred every four or five minutes.

The small amount of fume at the end of three and a half hours, showed a pretty thorough roast, with the exception of the last charge of sixty six pounds, which was not as well roasted as the others, on account of its bulk.

In agglomerating the ore, which was of course necessary, its fineness unfitting it for the blast furnace without preparation, we obtained the following results, as given on the next page

Time	Remarks.
10.05 AM	Fire started
10.56	$116\frac{1}{2}$ lbs ore charged
11.55	Charge drawn
12.05	125 lbs ore charged
1.10	Charge drawn.
1.20	120 lbs ore charged
2.30	Charge drawn
2.44	$104\frac{3}{8}$ lbs charged
3.55	Charge drawn.
4.30	Finished cleaning out furnace.

Summation

hrs min		
4.25	Total time actually employed in agglomerating	
6 25	Total time labor employed	
	Total coal used	252 lbs.
	Weight of ore charged	$465\frac{7}{8}$ "
	Weight of ore drawn	$467\frac{4}{8}$ "

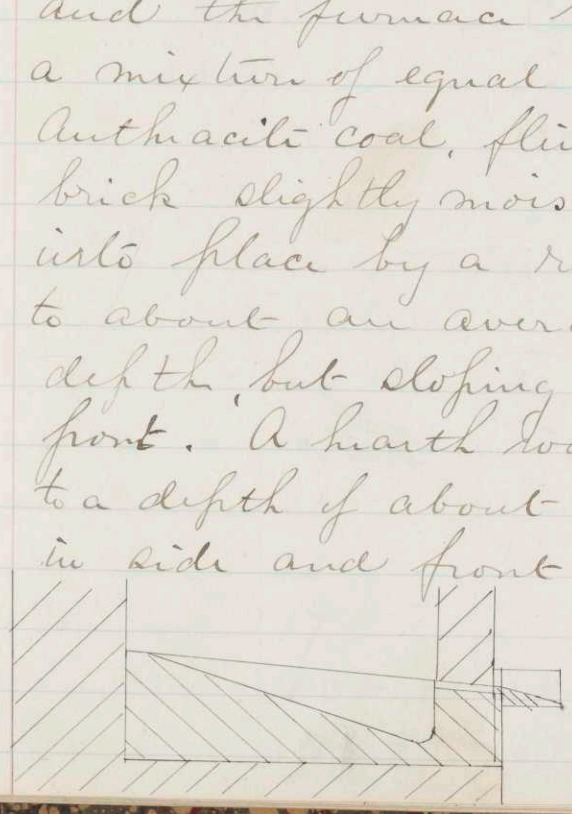
Supposing no loss in agglomerating, as we are entitled to, the actual loss being very small, the $1\frac{5}{8}$ lbs drawn, over the amount charged is accounted for, as above stated, by the fact that after roasting, ~~and~~ that amount was left in to be scraped out after the agglomeration.

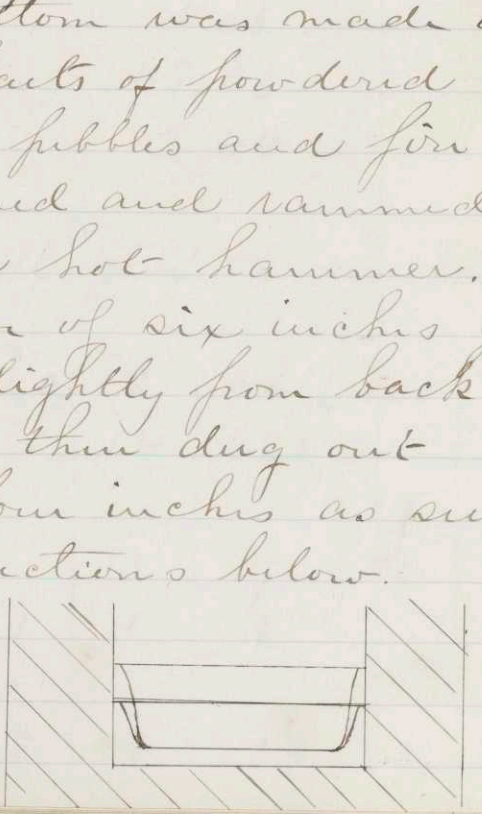
The working of the blast furnace was on the whole rather unsatisfactory, being made so from three causes,

1° An undue weight of slag planned.
2° A poor slag planned.
3° A cold furnace at the end of the run leaving an undue amount of furnace ends to smelt

The furnace itself is an ordinary cupola furnace, carrying a column of ore and flux from three to three and a half feet high, and one and a half feet square in plan section.

There was no forehearth used, and the furnace bottom was made of a mixture of equal parts of powdered Anthracite coal, flint pebbles and fire brick slightly moistened and rammed into place by a red hot hammer, to about an average of six inches in depth, but sloping slightly from back to front. A hearth was then dug out to a depth of about four inches as seen in side and front sections below.





There are three tuyeres, 3/4 inches in diameter, thus giving .84 sq. in. of section of blast per sq. ft. of furnace section, the blast being run at a pressure of 15/16 in. of mercury or 7 1/2 oz per sq. ft.

The slag which seemed best, and which was planned for was one obtained by Mr. Shockley in a former run of a similar ore.

A table of his work shows

Planned		Obtained
29.56	SiO_2	30.54
9.23	Al_2O_3	8.71
4.41	CaO	4.66
56.77	FeO	48.86
	PbO	2.44

The following table will give the result of our calculations.

Table I

	Wt.	SiO_2	Fe	Al_2O_3	S	Pb	CaO	Vol.
Ore	467 1/2	32.	83.	5.	14	221		112
Limestone	36						19	17
Galena	20				3	17		
Slimes	17	8		1	1	1		6
Cinder	150	21	84	12			3	
Total	690 1/2	51	167	18	18	239	22	135

The column marked Vol. gives the weights of the elements not considered in planning, including the volatile matters.

The charges planned are given in Table II

Charge	I	II	III	IV	V	VI	VII
Cinder	9 5/8	13	16	19 2/8			
Limestone	2 3/8	3	3 7/8	4 5/8			
Slime	1 1/8	1 4/8	1 7/8	2 2/8			
Galena	1 3/8	1 6/8	2 1/8	2 5/8			
Ore	30	40	50	60			
Total weight	44 4/8	59 2/8	72 7/8	88 6/8			
Coke	10	10	10	10	10	10	10
Buggy 6							—
Buggy 7						—	
Basic Roaster				40			

The analysis of the substances charged as follows, only partial analysis being made in the majority of cases, as more were not needed.

By Mr Hibbard — Cinder		By myself — Limestone	
SiO_2	14.55	SiO_2	.72
FeO	70.89	CaO	55.45
Al_2O_3	8.08	CO_2	43.60
CaO	2.38		99.77
	95.90		

Ore by Mr Hibbard	
SiO_2	6.2
Pb	39.5
S	18.9
Cu	.6
Al_2O_3	.7
Fe	14.6
MgO	3.3
ZnO } MnO }	.7
	84.5

Slimes by myself.	
SiO_2	45.79
Pb	7.61
S	5.47
Cu	.42
Al_2O_3	8.95
Fe	.21
MgO	12.87
CaO	5.59
As	1.41
Sb	trace
Zn	.37
~~Mg~~	
CO_2	10.43
	99.12

Galena by myself.	
Pb	52.20
S	19.27
Fe	17.31
SiO_2	2.04
	90.82

A fire was lit the afternoon before the run was to be made, so that the furnace was thoroughly heated and at 9.55 the next morning the blast was turned on, and the run really began; the particulars of which will be found on the succeeding pages in Table III, so that only a short explanation will be necessary to complete the record.

Tap Record Time	Interval	No. of Buggy	Feed Record Time	Interval	Depth before Charging	Charge	Notes.
4.30 PM							Fire lit
							2 hods charcoal
							3 .. coke.
9.55 AM				Blast	on.		5 hods coke.
			10.33 AM				15 lbs coke.
			10.40		$1\frac{1}{4}$ ft	V	
			10.45	.05		V	
10.50			10.50	.05		V	Slag runs
			10.57	.07	$2\frac{3}{4}$	V	
			11.13	.06	2	I	
			11.20	.07	$2\frac{1}{8}$	I	
			11.26	.06	$2\frac{1}{8}$	I	
11.30		6					
11.33	.03	7	11.33	.07	2	I	Plugged
11.42	.09	8					Tapped } Some lead
11.43	.01						Plugged }
			11.44	.11		III	
11.51	.08	9	11.51	.07		III	Tapped
11.52	.01						Plugged
12 00	.08	10					T
12 01	.01						P
12 10	.09	11					T
12.11	.01		12.11	.20	$2\frac{1}{4}$	IV	P { Globules of
12.21	.10	12					T Pb. on top.
12.22	.01		12.22	.11	$2\frac{1}{2}$	IV	P

Tap Record — Feed Record — Notes

Time	Int.	Buggy	Time	Int	Depth	Charge	Notes
12.32 AM	.10	13					T Fewer globules
12.33	.01						P
			12.36 AM	.14	2½	IV	
12.43	.10	14					T Foul on top
12.44	.01						P
			12.50	.14	2	I	
12.54	.10	15					T Foul on top
12.55	.01						P
							Furnace cold. Showing too ~~much~~ little coke.
1.05 PM	.10	16					T Foul
1.05½	.00½						P
			1.07 PM	.17	1¾	VI	
			1.13	.06	2	VII	
1.15	.09½	17					T Foul.
1.16	.01						P
1.26	.10	18					T Foul
1.27	.01						P
1.37	.10	19					T Foul
1.38	.01						P
			1.40	.27	1¼		
1.47	.09	20					T very Foul
1.55	.08		1.55	.15			Blast off
2.00	.05		2.00	.05			Front down

As soon as the furnace was judged hot enough, the first charge of 40 lbs. of Basic Reverb Slag was put in for the purpose of starting the regular working of the furnace; it was followed by two other charges of the same character before the slag began to run, when a fourth was added after which ore and flux were fed.

The tap hole was left open until the first signs of the coming of the lead, when it was closed, and tapped every ten minutes during the run, the slag and metal accumulating sufficiently in that time to fill a buggy.

As will be seen from the table the charges were added in the ratio of

4 lbs charge ~~to~~	1 lb coke
4	do.
4	do.
4	do.
4.45	do.
4.45	do.
4.45	do.
4.45	do.
7.275	do.
7.275	do.
8.875	do.
8.875	do.
8.875	do.
4.	do.

The last two or three heavy charges were too much and cooled the furnace off so much, that salamanders were formed in the back and on the sides of the furnace.

It has been found by repeated experiments that our furnace burns ten pounds of coke every fifteen minutes, hence in feeding, that amount must be added with a larger or smaller proportion of material to be smelted, as the furnace is too hot or too cold.

The record shows that the amount of coke used in starting our fire was eight hods plus fifteen pounds of coke or about (250 lbs) two hundred and fifty lbs. The amount used in running it was (160 lbs) one hundred and sixty pounds a total of (410 lbs) four hundred and ten pounds, or a little less than one pound of coke to one pound of ore.

It will be noticed in looking over the Notes attached to the Records, that as soon as the lead began to flow, small spots of it remained on the surface of the slag in the buggies, instead of settling to the bottom.

They were very small

not over an eighth of an inch in diameter, but there must have been at least four or five of them on every square inch of slag surface. We were entirely at a loss to account for the presence of these globules as the slag was very liquid indeed.

There did not appear to be any regularity about them, as they were just as numerous when the furnace chilled, as they were when it was hottest.

One buggy would be covered with them, the next would have very few, while the next would be as bad as the first.

The slag run, weighed four hundred pounds, and was mostly a very foul product, however some specimens of pure slag were picked out for analysis, the results of which are

Slag -

Planned		Obtained	Intended to be planned.
19.43	SiO_2	24.28	30.54
68.15	FeO	52.20	48.86
5.73	Al_2O_3	10 48	8.71
7.00	CaO	5.30	4 66
	MgO	1.37	
	S	.79	Cu .25
	Pb	4.44	

The discrepancy observed in the analysis on the foregoing page are due principally to the fact that while Mr. Shockley's Slag called for 167 lbs of FeO, we by mistake planned for 167 lbs of Fe, the result of which was that the walls of the furnace were cut away not only by their naturally tending to flux with the slag but also by the extremely basic slag sucking SiO_2.

After cooling, the slag was crushed and passed through a sieve of twelve meshes to the inch, thus giving a mixture of a large per.cent. of lead with a small amount of uncrushed slag on the sieve; the larger pieces of lead were then picked out, and the remainder jigged by hand extracting altogether

By ordinary sifting	5 lbs.
By jigging	1 lbs
Total	6 lbs

I now come to my especial portion of the run viz:- the treatment of the furnace ends residues etc.

The furnace ends were picked over, and the unsmelted ore, lead etc. separated, amounting to 113 lbs.

Without making an analysis, I estimated that about the right proportions of ore and flux would be found in the ends, and proceeded immediately to smelt them in black lead crucibles, holding about 17 lbs each, adding the six lbs of impure lead from the slag of the blast furnace.

Owing to defective flues in the furnaces used, I was unable to obtain as good a melt as I could wish, but succeeded in getting 19 7/8 lbs of lead which together with the 130 lbs from the blast furnace, amounted to 149 7/8 lbs of lead thus far.

The loss of lead in the slag was very heavy, a result due partly to the uncertainty of having the right flux, partly to a large admixture of coke, and partly to the want of a perfect fusion, the three causes together taking, about 25 lbs of lead as nearly as could be determined in the mixed slag.

The total lead now on hand to be refined amounted to 149 7/8 lbs.

A fire was lit in the large crucible furnace, and 43 lbs of lead charged into a No. 25 black lead crucible, half an hour later 21 lbs were added, the whole charge was then kept a little below a boiling heat for six hours, being stirred and skimmed from time to time as the impurities, mixed with litharge, collected.

Toward the end of the operation, a small quantity of lead was dipped out at short intervals in order to change the level, as the litharge cut away the crucible very badly, and it was found to save it very much. In all 15 lbs were removed and charged with 64 1/2 lbs of new lead into the crucible for the next refining.

It was kept at the same heat, as the first charge, for four hours, the greater rapidity of refining being due to the addition of a few crystals of nitre every few minutes toward the end of the operation.

The action of the nitre is twofold, first it purifies by causing the lead to boil, and second by oxydizing it, thus allowing the litharge to carry off the impurities.

The third charge of 31 lbs with five or ten pounds from the second, was refined for only two and a quarter hours, nitre being added every few minutes during the whole operation, beginning as soon as the first slag had been skimmed off.

The results showed the advantage of using the nitre very forcibly, both in the diminished time required to refine, and in the softer lead produced, that from the last charge being the best, and that from the first being the hardest.

I have not ascertained the cost of nitre, but as it is not very expensive, I should reccommend its use in at least all small refining operations such as we have in the laboratory at the Institute.

Our refining gave us 111 lbs of a very good soft lead, and 38⅞ lbs of skimmings, i.e. slag enclosed in the lead, impure litharge etc.

Mr. Hibbard now took the

lead and sweated it to still further refine it, thus giving $89\frac{3}{4}$ lbs of lead, containing almost no impurities, and 21 lbs of residues, the loss of $\frac{1}{4}$ lb probably being due to the inaccuracy of the scales used, as there was no chance for loss during the operation

I took the

Skimmings	$38\frac{7}{8}$ lbs
Residues	21 lbs.
Galena	12 lbs
Total	$71\frac{7}{8}$ lbs.

and smelted them for the production of a copper matt, as it was ascertained that the major part of the copper in the ore, which contained only .6 % had gone into the residues of various sorts.

The Galena was added in sufficient quantity to furnish all the sulphur necessary.

The mixture was smelted in three charges, taking, on an average, only two hours to melt, as the heat required was not very intense. The products were

Lead	$43\frac{5}{8}$ lbs
Matt + Speiss	$12\frac{3}{8}$ "
Slag	$11\frac{7}{8}$ "
Total	$67\frac{7}{8}$ lbs.

The total weight shows a loss of four pounds, from volatilization and partial boiling over of the slag.

I succeeded in getting by this smelt, four very distinct layers. at the bottom the lead, then 1/4 inches of an arsenical speiss containing 8.6 o/o of sulphur, and 26.4 o/o of arsenic, then 3/4 inches of a pure concentrated matt, which I afterward regretted neglecting to analyze by itself, while the whole was covered by about 1/3 inches of a very good looking basic slag.

The 43 5/8 lbs of lead obtained were refined with nitre and sweated giving

Skimmings	6 lbs
Sweatings	6 "
Lead	25 5/8 "
Total	37 5/8

This discrepancy between the total unrefined lead and the total weight of the products, was caused by the loss of a six pound ~~pig~~ ~~sa~~ by one of the students, and which we were unable to recover.

I now had twelve pounds of residues and twenty six and a quarter pounds of what was called galena, although it only contained as was afterward ascertained by analysis about 58 o/o of Galena.

My total charge in smelting the above was,

Galena for copper matt	6.25 lbs
Galena reduced by Fe_3O_4	20. "
Magnetite	6.5 "
Coal	1.5
Skimmings + Sweatings	12.
Total	46.25 lbs

Galena for copper matt was to furnish the sulphur.

For analysis of the galena see page 13.

There resulted from the above,

13.25 lbs	Matt + Speiss
12. "	Lead
20. "	Slag
45.25 lbs Total.	

Matt and speiss were not well defined, the former having a large per. cent. of iron and small per. cent. of copper.

The poor matt was due to the fact, that the galena carried 17.3 o/o of iron which was ignored, as I had no analysis at the time.

I next melted the two matts together in order to get a sample for analysis, the result of which was,

First Matt. $25\frac{5}{8}$ lbs.		First Matt Slag.	
S	24.10 %		
Pb	14.17 "		
Cu	8.56 "	Cu	3.8 %
Fe_2O_3	49.72 "	Pb	8.4 %
Al_2O_3	.98 "		
SiO_2	1.16 "		
	98.69.		

The apparent want of completeness in the above is due to the fact that some of the iron exists as sulphide which would make the total nearer 100.

The same is the case in the second matt, as will be seen.

In roasting the first matt I used, as a kiln an old black lead crucible with the bottom knocked out, and a grate inserted.

The matt was crushed to about one and a half inches and a charge of eight pounds placed in the kiln, with three quarters of a pound of charcoal, when the fire was lit, and on an average burnt five and a half hours.

The ore was roasted twice

in that way, the operation consuming three days and leaving still 20.3 % of sulphur in the matt.

I then decided to use a double crucible, the top one inverted over the lower and with its bottom out as an exit for the fumes, and crush the matt to 1/2 inches and use only about one pound of charcoal per charge, and putting in the entire matt at one time.

When after two roastings lasting about twenty hours each, as nearly as I could judge, I found that the percentage of sulphur had been reduced to 14.8 %, thus showing a decided advantage, both in saving of fuel, about 44 %, and time, due partly to the finer condition of the ore, and partly to the larger kiln retaining the heat better.

However on the whole, I think that in an experimental laboratory, such small amounts may best be roasted in a reverberatory, roasting a calculated per cent dead and mixing with the unroasted, giving the amount of sulphur desired.

I now smelted for a second matt, using glass containing 74.43 % of

SiO_2 as a flux and mixing.

Ore	25 5/8 lbs	
Glass	5 "	
Charcoal	4/8	
Total		31 1/8 lbs.

I succeded in getting thirteen pounds of matt of a composition given below, and a slag of eighteen pounds-

Second Matt.		Second Matt Slag	
S	20.42	Cu	2.79
Pb	20.27	Pb	1.13
Cu	12.12		
Fe_2O_3	44.69		
SiO_2	1.20		
	98.70		

This second matt was now crushed to 1/12, and roasted in iron pans in open crucible furnaces.

As the heat was all below the pans, the result was a very imperfect roast, leaving 10.1 o/o of sulphur still in the matt, and a small loss through holes eaten by sulphur in the bottom of the pan. The matt was roasted in two charges of six and a half pounds each, and for one hour and three quarters.

I then recrushed, and roasted in the small reverberatory used for cupelling, using as a bed a plate of fire brick luted into an iron cupel ring sixteen inches square, by a mixture of three parts firebrick and one part fire clay moistened and rammed in.

The matt was put in in two charges, the average length of roast being one and a quarter hours and when the ore appeared roasted dead, no smell of SO_2 being noticeable, it was drawn.

I then smelted for black copper charging,

Matt	13 lbs
Glass	9 lbs
Coal	1 lbs.
Total	23 lbs

and instead of copper as I expected, having no time to make a determination of sulphur in the last roast, I obtained a third matt of five pounds weight.

Third Matt		Third matt slag, 18 lbs.	
Cu	20.41 %	Cu	.54
Pb	19.27 "	Pb	3.41

This was immediately roasted dead, in an iron pan in the large muffle furnace, taking one hour and three quarters, and smelted with what I judged sufficient glass and coal, having no analysis to go by on account of insufficient time, and gave me.

Copper	.5 lbs.	Slag	.5 lbs.
Cu	60.44	Cu	14.42
Pb	26.72	Pb	2.02

The copper was of a reddish gray color and looked like a very good matt, the gray being due to the amount of lead which was alloyed with it.

In refining, the copper was boiled five hours in a black lead crucible, when it had been reduced to 5.4 oz in weight. I should have refined it still further, if I had been accustomed to the operation, but thought at that time that it was complete, owing to the peculiar copper green of the melted copper, so that I poured it into an ingot, and weighed it as stated above.

I now wished to purify the copper still more, by a wett process.

and part from it, the gold and silver, it containing of the latter 6.57 o/o and of the former .12 o/o, to this end I cut the ingot into small pieces and dissolved in dilute nitric acid, the metal all going up in about three hours. I then filtered of the undissolved residue and precipitated the silver by hydrochloric acid as AgCl, and decanted the supernatant liquor, placing the AgCl in a warm place to dry.

I now digested the first undissolved residue with Aqua Regia and succeeded in dissolving it almost completely, there not being but a gram or two of it, after solution the fluid was diluted with water and crystals of oxalic acid added in sufficient quantity to precipitate the gold, which was then filtered off, dried, ignited, and weighed giving a button of pure gold weighing .1846 of a gram, being almost exactly .12 o/o as given by the assay.

The AgCl was now ignited in a Hessian crucible, in the forge, with a very little lead to prevent volatilization of silver and assist in reducing it, some charcoal, and a little borax giving a button of silver weighing 9.75 grms. a little less than the assay, which called for ten grams.

The copper being now dissolved as nitrate, the solution was evaporated to dryness and the $Cu\,2(NO_3)$ reduced in a Hessian crucible, with a little charcoal, producing an ingot of copper weighing 3.2 oz as its analysis demanded.

I rescued from the furnace ends and residues, beside some silver and gold which Mr. Hibbard has parted, the following as the result of my work.

Lead about	60 lbs @ .06 per lb	$3.60
Copper	3.2 oz @ .40 per lb	.08
Silver	19.5 grms @ $1.30 per oz	.44
Gold	.1846 grms @ $20.60 per oz	.12
	Total value	$4.24

Appendix

Losses -

Lead. As only 132 lbs or 55.65 % of lead were obtained from 239 lbs in the ore, I give the following table showing the approximate losses at different stages of the work.

Loss from volatilization in roasting	9.7 lbs
Loss in blast furnace slag	17.7 "
Loss from volatilization in blast furnace and crucibles	38.
Loss in slag of crucible melts	25.
Pig lost	6.
Unaccounted for; in fumes from smelting residues, refining and crucible slags	10.6
Total loss	107. lbs

Total lead obtained	132 lbs	or 55.65 %
" " lost	107 lbs	" 44.35 %
" " in ore	239	100

Copper.

Loss in blast furnace slag	1.0 lb.
" " 1st Matt slag	.5 "
" " 2d " "	.5 "
" " 3d " "	.1 "
" " Copper slag	.7 "
Unaccounted for, due in great measure to loss in Speiss	.8 "
Total copper lost	3.6 "
" " recovered	.2 "
Total copper	3.8 lbs

The large losses are due to the fact that the percentage of copper was so small in each case, as compared with the material handled.

Summary of process used.

In order to give a fuller understanding of the process used, so that it can be seen at a glance, I give on the next page a tabular view of the whole.

- Ore. Roasted
  - Ore Agglomerated
    - Ore Smelted
      - Lead
      - Slag
      - Fume
      - Furnace ends Smelted
        - Lead
        - Slag
    - Furnace ends
    - Loss from fume
  - Loss from fume

- Refined
  - Pig lead Sweated
    - Soft lead – Mr. Hibba
    - Residues *
  - Skimmings *

- * Smelt with Galena
  - Lead
  - Matt — Half roasted + Smelted with Galena
    - Lead
    - Matt — Half roasted + smelted with Galena
      - Matt — Roasted dead
        - Copper † Refined
          - Copper
          - Residue
        - Slag
      - Slag
    - Slag
  - Slag

- † Dissolved in HNO_3
  - Gold
  - Solution plus HCl
    - $AgCl$ Smelted to Ag.
    - Solution evaporated to dryness + Smelted to Copper.

The Smelting of a Silver-lead ore from Newburyport, and the Separation of the products,

By
Henry D. Hibbard.
M. I. T. '77.

Index.

Introduction.

The ore worked was an argentiferous galena from the Merrimac mine Newburyport. It ranked at the mine as a third class ore too poor to work. It contained only about 6 o/o galena.

A mineralogical examination of the ore revealed the following minerals. Galena, Quartz, Pyrite, chalcopyrite, Siderite, Blende, Chlorite, Arsenopyrite, Tetrahedrite, Dolomite, Felspar and Serpentine. This latter made up a large part of the mass.

The ore was weighed out of the canvas sacks in which it was received and was sledged to a size (about 2" cube), which could be fed to a small Blake's crusher. The total weight was about 4 1/2 tons of 2000 lbs each.

It was next crushed, then passed

between rolls and sifted through a ½" sieve. The fine ore was then concentrated by means of the machines in the mining laboratory, and from it was obtained 628 lbs. smelting ore. Of this, 56 lbs, the Table No 1 product, was not roasted but was kept to use in the blast furnace to prevent the formation of "sows".

The plan actually followed in the concentration is shown by the table on the next page.

The amalgamating ore, or second product, was worked separately, and its treatment does not come within the province of this article.

The composition of the 572 lbs. which were roasted was as follows:-

SiO_2	6.2
Fe	14.6
Al_2O_3	.7
Pb	39.5
Cu	.6
$ZnO + MnO$	.7
MgO	3.3
S	18.9
$CO_2 + O + As + Sb + Ag$ not determ	15.5
	100.0

The Table number one product contained 52 o/o of lead.

- Ore to $\frac{1''}{12}$ on cone
  - Concentration. Smelting
  - Overflow on Spitzkasten
    - 1 $\frac{1''}{20}$ On jig
      - Jig I rejigged on $\frac{1''}{20}$
        - Jig I Smelting ore
        - Jig II and overflow Amalg. ore
        - Siftings
      - Jig II Amalgamating ore;
      - Siftings
        - Concent. on cone
          - Concent. Smelting ore.
          - Overflow to side bump table
            - Smelting
            - Amalg. ore.
        - Tailings Amalg. ore
        - siftings Smelting
      - Overflow Tailings
    - 2, 3, 4 Each on End Bump Table by itself
      - Concentration on E. B. Table
        - Concent on E. B. T.
          - Smelting ore
          - Amalg. Ore.
        - Waste
      - Waste.
    - Slimes

The plan of work followed in the treatment of the smelting ore was as follows:-

I Roasting.
II Agglomerating.
III Smelting for lead
IV Refining the lead.
V Sweating the lead.
VI Zincing and sweating 3 times.
VII Expelling zinc from alloy.
VIII Cupelling.
IX Parting of button.
X Recovery of lead from cupel.

I Roasting.

The roasting was done in a hollow bed reverberatory furnace. Twelve (12) charges were made of which 11 were of 46 lbs. and the twelfth was of 66 lbs. The time allowed each charge was four (4) hours which seemed somewhat long for the smaller charges, but the charge of 66 lbs fumed considerably of sulphurous acid on being drawn. The final result of the roasting of the lot was the reducing the percentage of sulphur from 18.9 to 3.28 which was very fair. The loss of lead from fuming was very considerable being 9.1 o/o of the whole lead in the roast. The weight of the roasted ore was 467½ lbs.

II Agglomerating.

This consisted in heating the roasted ore to a bright redness in the same furnace used in roasting, in charges of about 120 lbs each. The heat was sufficiently great to partially fuse the ore which then, on being stirred, agglomerated, and it was then drawn out of the furnace.

There was no loss of weight during the agglomeration which showed that the roasting had been thorough. The ore at the end of the operation was left in the state of lumps varying in size from that of a pea, or less, to masses weighing several pounds.

III Smelting for lead.

The slag calculated for as a model was a uni-silicate of the following composition:-

SiO_2	30.54
FeO	48.86
Al_2O_3	8.71
CaO	4.66

The ingredients used in the run were ore, tap cinder, limestone, slimes, and galena. To obtain the above slag the analyses of all the ingredients was of course necessary, at least the percentages of the above named oxides. These were determined to be:-

Puddle, or tap cinder.

SiO_2	14.55
FeO	70.89
Al_2O_3	8.08
CaO	2.38
	95.90

The limestone was considered pure, that is as having 56 o/o CaO.

Slimes

SiO_2	45.79	S	5.47
Pb	7.61	Cu	.42
Al_2O_3	8.95	As	1.41
CaO	4.01	Zn }	.37
Fe	21.	Mn }	
CO_2	10.42	Mg.	1.72

The Table No 1, or so-called galena, had 53 o/o lead and also probably a considerable percentage of pyrite.

The charges used in the run were:-

	I	II	III	IV	V	VI
Ore.	30	40	50	60		
Cinder.	$9\frac{5}{8}$	13	16	$19\frac{1}{4}$		
Limestone.	$2\frac{3}{8}$	3	$3\frac{7}{8}$	$4\frac{5}{8}$		
Galena.	$1\frac{3}{8}$	$1\frac{3}{4}$	2	$2\frac{5}{8}$		
Slimes.	$1\frac{1}{8}$	$1\frac{1}{2}$	$1\frac{7}{8}$	$2\frac{1}{4}$		
Basic Revere.					40	
Buggies 5.6+7.						65
Charge.	$44\frac{1}{2}$	$59\frac{1}{4}$	$73\frac{3}{4}$	$88\frac{3}{4}$	40	65
Coke.	10	10	10	10	10	10

The slag actually obtained by the use of these charges was of the following composition.

SiO_2	24.28
FeO	52.20
Al_2O_3	10.48
Pb	4.44
CaO	5.30
MgO	1.87
S	.79
Cu	.25
	99.11

This slag was too basic and cut the furnace rather badly but a fusible slag is necessary in such short runs.

The percentage of lead was also very considerable. The weight of the slag obtained was about 400lbs and the weight of crude lead or bullion 130lbs

The smelting was done in a blast furnace having a section 12"×18", and 4 ft. high to the charging door. The furnace was supplied with three $\frac{3}{4}$" tuyeres, one on each side and one at the back, and the blast was furnished by a No 0 Sturtevant blower driven about 4000 revolutions per minute, which gave a pressure of $\frac{1}{2}$ lb. to the square inch.

The furnace was prepared for the run by ramming the bottom with steep (a mixture of equal parts of fireclay, ground firebrick, and anthracite coal, powdered.). A red hot hammer was used, the heat of which partially baked the clay in the steep giving a hard and impervious bottom. This was raised up sufficiently to allow the bottom to be hollowed out into a basin to form a receptacle for the lead, the taphole was carefully cut out, the front of the furnace built up and all was ready to fire up.

The fire was started without

the blast the evening before the day of the run in order to have the furnace hot. The next morning the blast was put on and coke fed rapidly to get up the heat. When this was well up basic Revere slag was charged to get everything going smoothly, and then ore was charged.

The following tabular record will furnish the details of the run.

Tap record			Feed Record.				Remarks
Time	Interval	No Buggy	Time	Interval	Depth before charging	Charge.	
4.30							March 14 Fire lit. shavings 2 hods charcoal + 2 hods coke.
9.55							Mch. 15 blast on 3 hods.
			10.20				3 hods coke
			10.33				1 hod coke.
			10.40	5'	$1\frac{1}{4}'$	V	
			10.45	5'		V	
		1	10.50	7'		V	slag begins to run.
		2	10.57	7'	$2\frac{3}{4}'$	V	
		3	11.13	16'	2'	I	
		4	11.20	7'	$2\frac{1}{8}'$	I	
		5	11.26	6'	$2\frac{1}{8}$	I	
11.30		6					tapped
11.33		7	11.33	7'	2'	I	plugged

Time	Interval	Buggy	Time	Interval	Depth	Charge	
11.42	5'	8					Tapped. first lead
11.43							plugged.
			11.44	11'		III	
			11.50	6'		III	
1151	8'	9					tapped
1152	8'						plugged.
12.00		10					tapped
12.01							plugged
12.10	9'	11					tapped
12.11			12.11	21'	$2\frac{1}{4}$"	IV	plugged
12.21	10'	12					tapped
12.22			12.22	11'	$2\frac{1}{2}$	IV	plugged
12.32	10'	13					tapped
12.33			1236	14'	$2\frac{1}{2}$	IV	plugged
12.43	10'	14					tapped
1244							plugged
12.54	10'	15	1250	14'	2	I	tapped
12.55							plugged
1.05	10'	16					tapped
1.06			1.07	17'	$1\frac{3}{4}$	VI	plugged
1.15	9'	17	1.13	6'	2	10lb coke	tapped
1.16							plugged
126	10'	18					tapped
127							plugged
137	10'	19					tapped
138			1.40	27	$1\frac{1}{4}$		plugged
1.47		20					tapped very foul slag
1.55							blast off.

The front of the furnace was knocked down at the end of the run.

The slag was all foul as in every buggy there were shots and drops of lead on top & through the interior of the pigs and also sprays on the outside. Under these conditions it was deemed advisable to crush and roll it which was accordingly done. The sprays and larger shot were flattened out and would not pass through the sieve though many small ones undoubtedly did. A wet assay of this impure slag gave 5.31 o/o lead or nearly a percent more than the pure slag contained. The pure slag sample was taken from buggies 10-16 inclusive and the impure slag from buggy 8 to the end.

There was recovered by the crushing and jigging of some residues 6 lbs lead.

The furnace was much too cool at the end of the run and there was a large mass of ore and slag left in it. These furnace endings weighed 112 lbs and were fused with coal in black lead pots though with indifferent success, as none but an intense white heat gave a good separation of lead and slag.

The crude bullion obtained assayed 64 oz. silver and $54. gold to the ton.

It seems now as if the use of tap cinder was unwise as it unnecessarily increased the bulk of the slag, which increased the loss of lead. The ore having more iron in it than was needed to flux the silica and almost the sulphur as well, a better run would probably have been made with the ore, galena magnetite and limestone, with perhaps a little of the slimes.

In adding up the ore charges we find that only 440 lbs were charged showing a loss of 27½" which we are unable to account for.

The loss of lead by the incomplete fusions of the furnace endings was also considerable but its amount cannot be determined. About 19 lbs. lead was saved in these fusions.

IV Refining the lead.

This operation consisted in heating the lead to a driving heat in black lead crucibles and skimming the slag which floated on the surface.

The first crucible full was refined by the oxidation of the air alone, but on the second one nitre was thrown at intervals which assisted greatly in shortening the operation. The slag, a very basic silicate was very fusible and somewhat difficult to skim at first, but on trial it was found that a little fine bituminous coal thrown in would stick it together on coking, so as to be readily skimmed, thus materially diminishing the amount of labor required. The surface required to be kept as clear as possible to allow oxidation to go on.

Poling the lead was impracticable, as the violent commotion caused by the evolution and expansion of the gases led to loss of lead by spattering.

The lead cut the crucibles so badly at its surface, that it was found to be expedient to ladle a little out occasionally, thus bringing a fresh portion of the crucible to the wear.

The skimmings from the refining were all carefully saved and smelted with the residues from sweating, and galena, for lead and matte (For treatment of the matte see Mr. Baldwin's thesis). The lead obtained was refined and sweated giving new skimmings which were smelted and so on. The fourth skimmings + residues were not smelted.

The total amount of lead refined was

Lead from smelt and crucible fusions	149 lbs
" " 1st skimmings and galena	$43\frac{5}{8}$ "
" " 2d " " "	12 "
" " 3d " " "	9 "
	$213\frac{5}{8}$ "

The 149 lbs gave:-

Refined lead,	111 lbs
Skimmings,	$38\frac{7}{8}$ "
	$149\frac{7}{8}$ "

The 45 lbs gave:-

Refined lead,	$37\frac{5}{8}$ "
Skimmings,	6 "
	$43\frac{5}{8}$ "

The 12 lbs gave:-

Refined lead,	$9\frac{1}{2}$ "
Skimmings,	$2\frac{1}{2}$ "
	12 "

The 9 lbs gave:-

Refined lead,	$7\frac{1}{2}$ "
Skimmings,	$1\frac{1}{2}$ "
	9

Of the $213\frac{5}{8}$ lbs. lead refined considerable was handled several times so that the real quantity of lead smelted out it was impossible to determine.

The total wt. of refined lead obtained was $165\frac{5}{8}$ lbs. and total skimmings $48\frac{7}{8}$ lbs.

The fourth skimmings being only $1\frac{1}{2}$ lbs were not smelted.

V Sweating or Liquation

In this operation the lead was heated on a sloping sheet iron bed heated by gas. The lead when liquefied ran off and was collected in ladles while the copper and other solid impurities remained behind. The lead of course carried the silver and gold with it. The principal object of sweating was to eliminate the copper. The residues were smelted with the refining skimmings.

The first lot of 111 lbs refined lead gave:

Pure lead,	$89\frac{3}{4}$ lbs	80.82 o/o
Residues,	21 "	18.94 "
Total product,	$110\frac{3}{4}$ "	
Loss,	$\frac{1}{4}$	.24 "
Total,	111	100.00 "

2^d lot of $37\frac{5}{8}$ lbs. gave:-

Lead,	$31\frac{3}{8}$ lbs	83.50 o/o
Residue,	6	15.95 "
Product,	$37\frac{3}{8}$	
Loss,	$\frac{1}{4}$	.65 "
	$37\frac{5}{8}$	100.00

3^d lot of $9\frac{1}{2}$ lbs gave:-

Lead,	$8\frac{3}{4}$ lbs	92.10
Residue,	$\frac{3}{4}$ "	7.90
Product,	$9\frac{1}{2}$ "	
Loss,	0 "	
Total,	$9\frac{1}{2}$ "	100.00

4th lot of $7\frac{1}{2}$ lbs

Lead,	$6\frac{3}{4}$ lbs	90.00%
Residue,	$\frac{3}{4}$ "	10.00 "
	$7\frac{1}{2}$ "	100.00 "
Total pure lead.	$136\frac{1}{4}$ "	

VI Zincing and Sweating.
Parke's process.

When a small proportion of melted zinc is stirred into a quantity of melted argentiferous lead, the silver is seized upon by the zinc which forms a scum floating on the surface. This scum is separated either by skimming when large quantities are worked or by sweating when the quantity is small. The latter method was used in this case.

The lead was heated to near driving in an iron pot, and the zinc, almost at a burning heat stirred rapidly in. The whole was then ladled into moulds. The pigs thus moulded were subsequently sweated in the same manner as the first sweating to get rid of the copper.

For the first zincing only $8\frac{3}{4}$ lbs lead were put in as the rest of the lead ($6\frac{3}{4}$ lbs) was not sweated from the copper. One pound of zinc was melted in a crucible and was burning when poured in.

The sweating after zincing gave:—

Lead,	$78\frac{3}{4}$ lbs.	or	93.75
Zinc silver lead alloy,	$4\frac{1}{4}$	"	5.06
Total product.	83	"	98.81
Loss,	1		1.19
Total wt. of Zn + Pb,	84		100.00

For assays of the leads obtained see end of this section.

For the second zincing were put in the pot the $78\frac{3}{4}$ lbs from the last and also the $6\frac{3}{4}$ lbs of original lead mentioned above, making in all $85\frac{1}{2}$ lbs. 1 lb. zinc was added.

The result of the sweating was:—

Lead,	85 lbs.	or	98.27%
Alloy,	$1\frac{1}{8}$ "		1.30
Products,	$86\frac{1}{8}$		99.57
Loss,	$\frac{3}{8}$		.43
Total,	$86\frac{1}{2}$		100.00

Before proceeding to the third zincing of this lead, all the lead obtained from the skimmings and residues, which we shall speak of as residual lead was brought to this stage of the process. The results follow:—

1st zincing and sweating of residual lead.

Total lead zinced $46\frac{7}{8}$ lbs. $\frac{3}{8}$ lb. zn. added

Lead obtained,	$44\frac{3}{8}$ lbs.	or	93.92 %
Alloy,	2 "		4.23 "
Product,	$46\frac{3}{8}$ "		98.15 "
Loss,	$\frac{7}{8}$ "		1.85 "
Total lead + zinc,	$47\frac{1}{4}$ "		100.00 "

2d zincing & sweating.

Lead,	43.3/4 lbs.	or	98.04
Alloy,	5/8 "		1.40
Product,	44 3/8 "		99.44
Loss,	1/4 "		.56
Total,	44 5/8 "		100.00

For the final zincing all the lead was put in making a total weight of $128\frac{3}{4}$ lbs. and $1\frac{1}{4}$ lbs. zinc were added. The lead obtained from the sweating was resweated to get out as much zinc as possible.

The result was:-

Lead,	$126\frac{7}{8}$ lbs.	or	97.60 %
Alloy,	$2\frac{5}{8}$ "	"	2.02 "
Product,	$129\frac{1}{2}$ "		99.62 "
Loss,	$\frac{1}{2}$ "		.38
Total,	130 "		100.00

The assays of all the leads down to the end of zincing were:-

Sample.		oz. Ag to a ton.
1. Bullion,	$54. gold	64.
2. Sweated lead,		62.
3. Lead from 1st zincing,		16.
4. " " 2d " ,		2.48
5. Residual lead,		48.
6. Sweated residual lead,		49.
7. Lead from 1st zincing residual lead,		18.72
8. " " 2d " " " ,		.99
9. 1st Sweating 3d " of all the lead,		.22
10. 2d " " " " " "		.27

As there was some rich lead added before the second zincing of the original lead the value of the resultant lead, 2.48 oz. does not give a fair showing of the value of the process. The second zincing of the residual lead however yielding a lead with only 1 oz. to the ton was good and probably in practice two zincings would be found sufficient to desilverize much richer leads. The finished lead from the third zincing yielding, taking the average of the two assays only ¼ oz. silver to the ton, was good. The amount of zinc added to the leads were approximately 1 percent of the whole, the repetition of the process doing the work much more effectively than a large amount of zinc at once.

VII Expelling Zinc from alloy.

The amount of zinc silver lead alloy obtained from the zincings and sweatings was eleven (11) pounds in all and this contained about $3\frac{1}{2}$ lbs of zinc which was next to be got rid of.

The apparatus used consisted of two crucibles, one larger than the other

arranged as shown in the figure. The lower one held the alloy and the upper one which had a hole in the bottom was filled with ignited charcoal. The joint between the two was securely sealed with mortar and the lower crucible heated to bright redness. The zinc was volatilized in the metallic state, passed up through the charcoal and was burned on coming in contact with air, or passed up the chimney as zinc. The object of the upper crucible filled with burning charcoal was to prevent access of air and so the formation of infusible zinc oxide over the surface of the alloy.

In about two hours the zinc ceased to come off and the lead was poured.

Only 6 lbs. lead was got owing to the formation of considerable dross which was fused down in two large fires yielding $1\frac{1}{2}$ lbs more lead.

The final result was.

Rich silver-lead	6 lbs or	54.54 o/o
" " " (dross)	$1\frac{1}{2}$ "	13.63 "
Zinc	$3\frac{1}{2}$ "	31.83 "
Total.	11 "	100.00 "

The rich silver-lead was somewhat impure and was refined a little in a large fire and from that cast into long slim ingots, in which form it was ready for the cupel.

VIII Cupellation.

This was done in a small reverberatory furnace the bed of which was occupied by the cupel

The cupel itself required the utmost care in making, the first one made being hammered unevenly and so it split into layers on being dried. The second one was very good and gave entire satisfaction

The ring in which it was made was elliptical in form about 13 inches long, 9 inches broad and 2 inches deep.

The boneash was moistened until it made a compact mass when squeezed in the hand. It was thoroughly mixed and then sifted through a $\frac{1}{20}''$ sieve. The layers put in were each a fourth of the whole amount in the cupel. Each layer was rammed first with a pestle and then by light blows of a large faced hammer until it seemed perfectly hard to the finger end but was easily cut with the nail. After each layer was rammed its surface was thoroughly scratched with an iron point and the next layer was put in. It is better to have one man do the whole work of ramming as he can give equal hardness to his blows thus insuring homogeneity.

the
tie
wrinkle

After drying a few days the cupel was hollowed out on its upper

surface in the manner shown in the figure and a hole cut through it which is also represented in the figure. The hollow was to recive the lead, the hole to allow the litharge to run off, and the three little grooves were channels for the litharge to reach the hole.

The cupel was next placed in

the furnace, the tube A for the introduction of the lead and the tuyere t were placed in position projecting over the hollow and the fire was started. The heat was very slowly raised to avoid the flying of the cupel, and on arriving at a bright red heat the first lead was introduced by means of the tube A before-mentioned.

The fire required to be forced to the utmost to start the lead driving. The lead was a little impure containing probably some zinc which formed a ring around the edge, and gave signs of covering the lead but was soon absorbed by the cupel. As driving commenced a blast of air was blown in through the tuyere which was of ⅛" gas pipe. The blast was blown by a water injector. Considerable air entered also by the tube for the introduction of the lead and the driving when once commenced was well kept up. The rest of the lead was added from time to time until all in. A half a pound of litharge ran over but all the rest was absorbed by the cupel. The amount of lead cupelled was 8¾ lbs. and the time of blicking after driving commenced was 3 hours and 10 minutes.

The button contained a little copper in spite of the refinings and sweatings. Its weight was 14. grammes or $3\frac{2}{5}$ ounces. A little spattering occurred during cupellation an a few little pieces of lead in cracks were scorified and cupelled giving .4 gramme. The cracks in the cupel were few and unimportant and the whole operation was highly successful.

IX Separating the button.

The silver and gold button was treated with nitric acid which dissolved the silver and copper, leaving the gold as an insoluble black residue. This was filtered off, ignited in a porcelain crucible and weighed. The solution of the silver was diluted to about a liter and the silver was precipitated as chloride by the addition of chlorhydric acid. The precipitate was washed by decantation and covered with chlorhydric acid diluted with an equal bulk of water. Several pieces of spelter (crude zinc) put in and the whole left two or three days without stirring. At the end of that time the silver chloride had all been reduced to metallic silver which was washed by decantation and dried.

It was subsequently run down in a crucible with a little lead, blicked and poured into an ingot mould giving fine silver.

The gold obtained was 4.22 grm. = $2.80

" silver	"	107.89 "	= $3.80
total value			$6.60

X Recovering Lead from cupel.

The portions of the cupel impregnated with litharge were separated from the rest, ground fine, by being rolled and smelted in crucibles to recover the lead. The ingredients to flux the bone ash were magnetite, and glass, coal being used to effect the reduction.

The proportions were.

Cupel	16 $\frac{1}{2}$ lbs.
Magnetite	8 "
Glass	8 "
Coal (anthracite)	2 $\frac{3}{8}$ "

A very strong heat being required, a blast was put on a crucible furnace. The fusions were quite satisfactory. The lead obtained was refined a little and cast into pigs. Its wt. was 5 $\frac{3}{8}$ lbs.

On the pigs of lead at the bottoms of the buggies were cakes of phosphide of iron, a hard brittle substance having an appearance somewhat like cast iron. The phosphorus was reduced from the phosphate of lime in the bone ash and united with some iron reduced from the magnetite.

The addition of this lead to that previously obtained raised the total pure lead to 132¼ lbs.

Conclusion.

In summing up in order to find the percentages of loss in the different operations we get the table below.

The whole amount of lead introduced into the operations was:-

In Smelting ore,	225.94 lbs
" Galena,	29.12 "
" Slimes,	.80 "
Total.	255.86
The amount of lead obtained	132.25
Loss	123.61
Percentage of lead	51.69
" " loss	48.31
	100.00

The percentages of the losses at each operation and also the weights follow:-

Table of Losses.

Loss of lead in agglomeration (27½ lbs),	11.08 lbs	4.33%
" " " " roasting,	20.52 "	8.02 "
" " " " slag,	21.24 "	8.30 "
" " " " skimmings,	1.00 "	.39 "
" " " " matte,	4.00 "	1.57 "
" " " " zincing & sweating,	3.50 "	1.36 "
" " " " cupel smelting,	3.25 "	1.27 "
" " " " samples for assays,	1.00 "	.39 "
" " " " crucible fusions fumes, blast furnace bricks, steep &c.	58.02 "	22.68
Total loss,	123.61 "	48.31 "

The assay value of the smelting ore was:-

Silver,	$23.47
Gold,	14.42

Assuming the Table No I products to be of the same value, the whole amount of each in the ore was

Silver			$7.32
Gold			4.50
The silver obtained was	$3.80	or	52.05 %
" " lost,	" 3.52		47.95 "
Total	7.32		100.00
The Gold obtained was	$2.80	or	62.22 %
" " lost,	" 1.70		37.78 "
	4.50		100.00

the percentages of silver and lead obtained are about the same, that of the gold being 10 a/o higher though why it should be so does not appear.

Taken as a whole the run was the most complete and perhaps as satisfactory as any ever made at the Institute.

Henry D. Hibbard.

Appendix.

I

The original ore from the mine was of the following composition.

Pb	5.23
S	5.45
Fe	4.24
FeO	9.24
Cu	.32
As	.27
Al_2O_3	8.30
MnO	1.01
Zn	.15
CaO	1.16
MgO	1.39
Alkalies	1.95
CO_2	7.05
SiO_2	52.75
H_2O	.96
	99.44

The minerals were calculated from this analysis and found to consist of the following percentages.

Galena	6.04
Pyrite	7.26
Siderite (containing Mn &c)	18.51
Chalcopyrite	.93

Arsenopyrite	.90
Siliceous gangue	65.24
	99.44

II

The formula of the slag calculated for was $\frac{1}{2}(Al_2O_3)6(FeO.CaO.PbO)4SiO_2$. The slag obtained is represented by the following formula $(Al_2O_3)8(FeO.PbO.CaO)5SiO_2$

III

A precaution taken in ramming the cupel was to slightly moisten the upper surface of each layer after being scratched, before the introduction of the next layer. It was thought to be of great service in rendering the cupel more as one solid piece, preventing the layers from separating.

IV

The following tables are designed to show more clearly than heretofore the places of loss and the amount at each place.

Lead.

Substance	wt. in lb.	% of lead	lbs. lead	loss since last operation
Concentration	572	39.5	226	
Roasted ore	476	44.	205.5	20 1/2
Agglomeration	440	44	223.52	11
Galena	56	52		
Slimes	10 1/2	7.6		
Crude Bullion	149 approx	?	?	
Refined "	137 1/8 "	100	137 1/8	81 7/8
Sweated "	136 1/4	100	136 1/4	7/8
Desilverized lead	126 7/8	100	137 7/8	
Zn-Ag-Pb alloy	11	68		
Zinc	3 1/2 subtracted		3 1/2	
			134 3/8	1 7/8
Distilled lead	7 1/2		8 1/2	
Samples	1			
Cupel bottom	16	52	8 1/4	1/4
Lead from cupel	5	100	5	3 1/4
Total Loss.				123 5/8

Gold.

Substance	lbs. wt.	% of Au	value Au	loss since last operation
Concentration	572	.00239	4.50	
Galena	56			
Bullion crude	149	.00913	4.08	.42
Button	3.67 oz.	3.686	2.80	1.28
Gold obtained	.1357 oz.	100.	2.80	
				1.70

Silver.

Substance	wt.	% Ag	wt Ag	Loss since last operation
Concentration	572 lbs			
Galena	56 lbs	.067	6.15 oz.	
Roasted ore	476 lbs	—	—	—
Agglomeration	440 lbs			
Galena	56 "	—	—	—
Slimes	$10\frac{1}{2}$ "			
Crude bullion	149 "	.1935	4.77 "	1.38 oz
Sweated lead	$89\frac{3}{4}$ "	.211		
" "	$31\frac{3}{8}$ "	.1683	3.76	1.01 oz.
" "	$15\frac{1}{2}$ "	.1683 approx		
Zinced lead	$126\frac{7}{8}$ "	.0007	.02	
Ag-Zn-Pb alloy	11 "	2.294	3.68	.08
Distilled lead	$7\frac{1}{2}$ "	—	—	—
Silver button	3.67 oz.	94.25	3.47	.21
Cupel bottoms	16 lbs	—	—	
Lead from cupel bottom	5 "			
				2.68