The Experimental Working, by Wet and Dry Methods, of a Low-Grade Silver and Gold Ore, from Newburyport, Mass.

Thesis
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The Experimental Working, by Wet and Dry Methods, of a Low Grade Silver and Gold ore from Newbury.

When the above was chosen as a thesis-subject, it was intended to make certain experiments with the aim of finding a process for an ore from the Four Rock mine at Newbury.

A lot of this ore sent last year to the Institute yielded, by washing, a product consisting mainly of argentiferous zinc-blende.

On account of the high percentage of zinc contained in this product, smelting with a lead ore in the blast furnace would have occasioned a great loss of silver carried away in the furnace smoke.

To obviate this difficulty. it was decided to try certain wet processes which have come into use within a few years.

This year. however, it was found impossible to obtain ore from the Four Rock mine, and a product separated in the washing of ore from the Merrimack mine was substituted.

As the work of conducting the experiments was assigned to Mr. Jenney and myself, the following division of the principal wet methods was made.

The processes were arranged in two groups, and lots drawn for the choice of groups.

By this arrangement the following processes fell to the writer:—

Washoe amalgamation, Augustin, Patera, Ziervogel.

For the sake of comparison a smelting process was afterwards added to this group.

To more clearly present the principles upon which the experiments were conducted, brief descriptions of the above named processes are given.

Chapter I.

A description of certain processes for silver extraction.

Washoe Process.

This process is based upon the following chemical facts:—

I. That when common salt is added to a solution of sulphate of copper, sulphate of soda and di-chloride of copper are formed.

II. That by action of di-chloride of copper upon sulphide of silver, chloride of silver is formed.

III. That a solution of common salt dissolves chloride of silver, especially if the solution be heated.

IV. That when a solution of chloride of silver is brought in contact with metallic mercury, mercurous chloride is formed and metallic silver is liberated. If an excess of mercury is present the silver is immediately amalgamated.

V. That when mercurous chloride is brought in contact with metallic iron, protochloride of iron is formed and metallic mercury liberated.

The finely crushed ore is placed in an iron amalgamating pan and ground with water until in the state of an impalpable mud. Sulphate of copper, common salt and mercury are then added and the grinding continued. As the reactions take place much more readily if the mass is heated, steam is blown in. After grinding with the mercury for a time, the mass is run out into settling tanks where the mercury falls to the bottom and is drawn off. By heating the amalgam in a retort, the mercury is driven off and the silver remains as a porous mass.

When the character of the ore, upon which the experiments were to be made, was known, it was decided not to try this process as amalgamation in the presence of so large an amount of lead would at the best be very imperfect.

For a full description of this process and the apparatus employed the reader is referred to:- Mining and Metallurgy of Gold and Silver, by J. A. Phillips.

Augustin's Process.

This process was formerly employed at the Gottesbelohnung works, near Eisleben in Prussian Saxony, for the extraction of silver from argentiferous copper mattes. It has never been applied to the working of ores, probably from the fact that nearly all ores contain more or less lead, antimony, arsenic or other impurities which cause a loss of silver through volatilization or prevent a complete conversion of the silver into chloride.

The principles upon which this process depends are as follows:—

I. That when sulphuretted argentiferous ores or products are roasted with free access of air and at proper temperatures, sulphate of silver is formed.

II. That when ores thus roasted are mixed with common salt and roasted again, the sulphuric anhydride of the ore reacts upon the common salt forming sulphate of soda, while the silver is converted into chloride.

III. That the chloride of silver is soluble in a hot concentrated solution of common salt.

IV. That the silver may be precipitated from this solution by means of metallic copper.

As conducted at the Gottesbelohnung works, Augustin's process is as follows:—

The copper matte, finely ground and sifted, is roasted at a low red heat on the upper hearth of a double reverberatory furnace for about five hours, in charges of about four hundred-weights; it is afterwards roasted for two hours longer on the lower hearth, and then for about three hours at an increased temperature.

By this operation the silver is mostly transformed into sulphate, while the iron and copper are mostly changed into oxides with some little basic sulphates.

The roasted mass is now taken from the furnace and ground with 1.–6. per cent. of common salt. It is then roasted again for about three quarters of an hour at a heat below redness.

This second roasting produces chloride of silver together with some chlorides of iron and copper.

The mass, still hot from the roasting, is carried to the lixiviating tubs where a hot, strong solution of common salt is passed through it until the liquid which runs out at the bottom of the tub ceases to whiten a clean piece of copper.

The liquid is conducted from the outlets of the lixiviating tubs to the precipitating tubs, where, by means of metallic copper the silver is precipitated.

The copper which passes into solution is precipitated by metallic iron.

The cement silver is washed with water and acids to free it from impurities and is then dried and refined.

Augustin's process has been employed at Freiberg for the extraction of silver from copper mattes, and at the Boston and Colorado smelting works at, Black Hawk Colorado, it is now employed for the desilverizing of residues from Ziervogel's process.

At both these places the details of the process are more or less modified to suit the nature of the product worked upon, but the general principles remain the same.

For a more complete account see:–
Crookes and Röhrig's translation of Kerl's Metallurgy, and Vol IV of the Transactions of the American Institute of Mining Engineers, p. 295.

Ziervogel's Process.

This process is based upon the following facts:—

I, That when a finely pulverized matte, consisting of the sulphides of iron and copper with a proportion of silver, is roasted with free access of air, sulphate of iron is first formed.

As the roasting proceeds, the sulphate of iron is decomposed liberating sulphuric anhydride and leaving peroxide of iron.

The sulphuric anhydride acting upon the sulphide of copper forms suboxide of copper and sulphate of copper, the former being converted into peroxide by the oxygen of the air while the latter breaks up into sulphuric anhydride and cupric oxide

The sulphuric anhydride resulting from this latter decomposition acts upon the sulphide of silver forming sulphate of silver and sulphurous anhydride.

II. That sulphate of silver is readily soluble in warm water.

III That the silver may be precipitated from a solution of sulphate of silver, by means of metallic copper.

The main difficulty in carrying out this process is with the roasting, which must be arrested just before the last portions of sulphate of copper are decomposed otherwise some sulphate of silver is decomposed with the formation of metallic silver and sulphuric anhydride; if, on the other hand, the roasting be arrested too soon, some silver will remain as sulphide.

On experiment it was found impracticable to apply this process to the ore under consideration, therefore for a complete description, the reader is referred to:— Vol. I Crookes and Röhrigs translation of Kerls metallurgy; Mining and Metallurgy of gold and silver, by J. A. Phillips; p. 285. Vol. IV Transactions of American Institute of Mining Engineers.

Von Patera's Process.

This process consists in:—

I Roasting the ore in a reverberatory furnace, and at the same time blowing steam upon the charge.

II Roasting the ore a second time with the addition of common salt and sulphate of iron if the ore does not already contain enough sulphates, steam being blown in as before.

III Leaching the chlorinated ore with hot water for the removal of the chlorides of lead, copper and zinc.

IV Leaching the ore with a cold solution of hyposulphite of soda.

V Precipitating the silver from the hyposulphite solution by means of poly-sulphide of sodium.

VI Filtering off the precipitated sulphides and reducing them to metallic silver by ignition in a muffle.

This process is carried on at Joachimsthal as follows:—

The ores treated are very rich and composed of a great number of mineral species.

These ores, coarsely powdered, are roasted in a reverberatory furnace of peculiar construction.

The grate is about six inches wide and seven feet long. The bed of the furnace is about ten feet wide and six feet from the fire-bridge to the flues. The fireplace is separated from the bed by a sort of firebridge, consisting of an iron tube covered with fire-clay and pierced with a number of small holes on the side next the bed. Through this bridge, steam is admitted to the charge.

As soon as the charge is heated to redness, steam is turned on.

At the end of four hours the charge is drawn and ground between millstones, with 4.–9. per cent. of common salt, according to the richness of the ore.

The ore is then recharged and roasted with steam until a sample, lixiviated with hyposulphite of soda solution, does not retain more than .1–.2 per cent. of silver in its residues.

The charge is then drawn, sifted and moistened with water. It is next transfered to the lixiviating tubs where it is leached, first with cold, then with hot and again with cold water.

The washed mass is next treated with a cold, aqueous solution of hyposulphite of soda, until the liquor dropping from the tub no longer gives any precipitate with polysulphide of sodium.

The liquor resulting from the lixiviation, and holding in solution the double hyposulphite of silver and sodium, is next run into the precipitating tanks.

Here polysulphide of sodium is added in just sufficient quantity to precipitate the silver.

The resulting sulphide, after being allowed to settle, and drained of most of the liquor by decantation, is filtered in a canvass bag.

After washing and drying, it is placed in a muffle and heated to redness.

The residue is melted with iron in crucibles, affording silver of 960 – 980 fineness.

As neither of the processes yet described is adapted to the extraction of both gold and silver, and as the ore to be experimented on contained both these met als, with about 10 per cent. of lead, it was decided to try a modification of the following smelting process employed at Freiberg for the treatment of poor auriferous silver ores.

Poor silver ores and pyritous ores are so mixed that the amount of matte produced by smelting them may be from 40.-50. percent. of the total weight of ores operated upon. This mixture is then smelted in a blast-furnace. The resulting raw matte is then roasted in heaps. A mixture is then made with roasted lead ores, silver ores and the roasted raw matte, in such proportions as to contain 18.-20. per cent. of lead. This mixture is smelted and yields lead which is treated by cupellation.

Chapter II.

The Ore.

As previously stated, the ore, upon which the experiments have been performed, was obtained as a middle product in the washing of ore from the Merrimack mine. Four and one third tons of low-grade ore were crushed to one twelfth inch, and treated in the washing machines of the Mining Laboratory, so as to yield three products, a rich smelting ore, a middle grade product, and waste tailings.

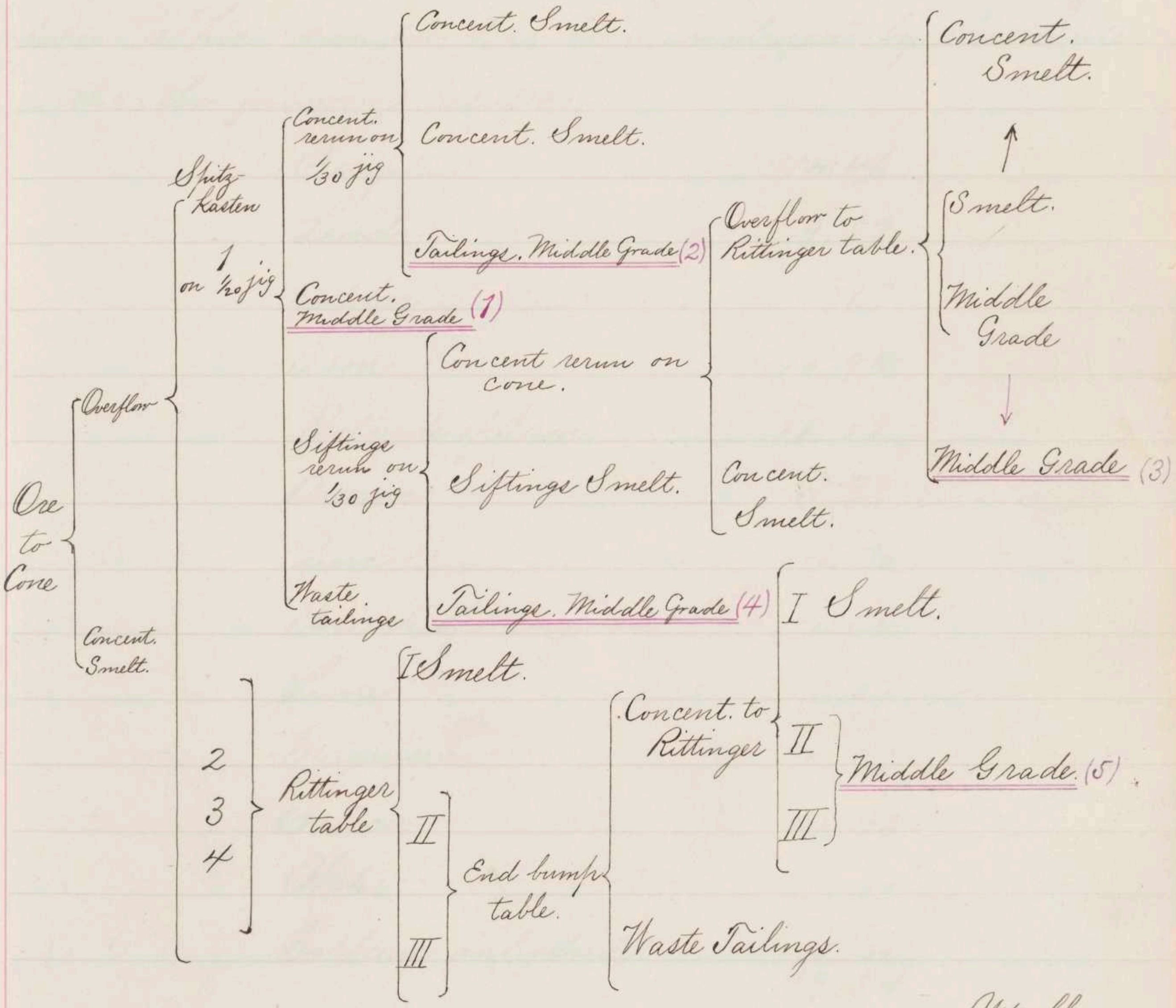
(On the following page will be found a scheme of the washing, as given by Messrs. Flint and Stimpson.)

While breaking up the ore, as it came from the mine, the following minerals exclusive of the vein rock, were determined:—

Galena.	Chalcopyrite.	Blende.
Siderite	Arsenopyrite.	Quartz
Pyrite	Tetrahedrite,	Serpentine.

Limonite was found on two or three specimens, but in such small quantities as not to be taken into account.

Scheme of washing as given by Messrs. Flint and Stimpson.



The products underscored made up the product experimented upon in the following proportions:—

These products were dried, thoroughly mixed and sampled.

	Wt. lbs.
(1)	377½
(2)	417
(3)	193⅞
(4)	121¾
(5)	174¾
Total	= 1284⅞

The Analysis of the Ore.

When thoroughly dried, the products making up the middle grade ore were well mixed and a sample taken. This sample has been analyzed by the writer with the following results:—

Sulphur	14.46
Lead	9.87
Copper	.67
Iron	10.94
Protoxide of Iron	18.18
Arsenic	.99
Zinc	1.70
Protoxide of Manganese	1.56
Lime	1.07
Magnesia	3.07
Silica	21.42
Water	.40
Carbonic anhydride	15.01
	99.34

The following are some of the methods used in the analysis of the ore:—

Sulphur:— The ore was decomposed with an excess of aqua-regia. The resulting acid solution and residue from the decomposition were transferred to an evaporating dish and evaporated to dryness with an excess of carbonate of soda. The residue from the evaporation was treated with strong hydrochloric acid and again evaporated to dryness. The residue from the second evaporation was then digested with water and the solution filtered. After making the filtrate slightly acid with hydrochloric acid, chloride of barium was added and the sulphur weighed as sulphate of barium.

Lead:— determined as sulphide.

Copper:- precipitated by the battery.

Iron:— determined by titration with bichromate of potash.

Arsenic:— weighed as arseniate of ammonia and magnesia.

Zinc and manganese:- precipitated as carbonates and weighed as oxides.

Magnesia:— weighed as pyrophosphate.

Water:— determined by mixing about three grammes of ore with a large excess of chromate of lead and heating the mixture in a hard glass tube connected with a chloride of calcium tube. After having been heated to redness for about five minutes, a current of dry air was passed through the apparatus to displace any carbonic anhydride which might have resulted from decomposition of carbonates. The increase in weight of the calcium tubes was the weight of the water expelled from the ore.

The object in view in mixing the chromate of lead with the ore was the oxidation of the sulphides to form sulphate of lead and thus prevent the sublimation of sulphur.

Carbonic acid:— determined by treating the ore in a small flask with a mixture of dilute sulphuric acid and bichromate of potash. The flask was heated gently at first and finally to boiling. The gas evolved was conducted through an acid solution of sulphate of copper and into potash bulbs. The object of the addition of the bichromate of potash was the oxidation of sulphides to prevent the formation of sulphuretted hydrogen, while the copper solution was used to take up any sulphuretted hydrogen that might have escaped decomposition

From the analysis of the ore the table of mineral percentages on the following page has been calculated.

						Siderite							
	Galena	Chalcopyrite	Arsenopyrite	Blende	Pyrite	$FeOCO_2$	$MnOCO_2$	$CaOCO_2$	$MgOCO_2$	Serpentine	Quartz	Water	Percentages of Elements
Sulphur	1.53	.68	.42	.84	10.99								14.46
Lead	9.87												9.87
Copper		.67											.67
Iron		.59	.74		9.62	14.13							25.08
Arsenic			.99										.99
Zinc				1.70									1.70
Oxygen						4.04							4.04
CO_2						11.09	.97	.84	2.11				15.01
MnO							1.56						1.56
CaO								1.07					1.07
MgO									1.92	1.15			3.07
H_2O										.34		.06	.40
SiO_2										1.18	20.24		21.42
Mineral Percent's.	11.40	1.94	2.15	2.54	20.61	29.26	2.53	1.91	4.03	2.67	20.24	.06	99.34
						37.73							

Assays of the ore, made by the writer give;—

	percentage	dollars per ton
Silver	.0384	12.32
Gold	.0007	4.22
Total		16.54

Chapter III

The Experiments.

The following experiments were conducted in the metallurgical laboratory:-

I Qualitative experiment upon fifty pounds of ore to determine if it were possible to extract any silver by a wet process.

II The working of one hundred pounds of ore by Augustin's process.

III Qualitative experiment upon five hundred gramme of ore, to determine the possibility of using Ziervogel's process.

IV The working of one hundred pounds of ore by Von Patera's process.

V The smelting of one hundred pounds of ore for matte and the subsequent treatment of one fourth of the matte for gold and silver.

I Qualitative Experiment.

On account of the presence of so high a percentage of lead, it was doubtful whether it would be possible to extract any of the silver by a wet process; accordingly, fifty pounds of ore were subjected to a chlorination roasting and subsequent lixiviation after Augustin's method. The following are the details of the experiment:—

The furnace chosen for this, as well as for the subsequent roastings, was the hollow bed reverberatory, as easier of access than the flat bed furnace. The fuel used was Cumberland coal.

When the furnace was heated to dull redness, fifty pounds of ore crushed to one fortieth of an inch were charged. The working door was closed for about half an hour, to allow the charge to become heated. On reopening the door the charge was found to be fuming and the sulphur burning. At the end of the first hour the mass was glowing brightly from the burning sulphur. At about this time a singular phenomenon was noticed; The charge seemed to have become liquid, flowing down to the tap hole, so that all attempts to distribute it evenly over the hearth were futile. This state of things which lasted about an half hour was due to the tendency of the escaping carbonic anhydride to lift the ore particles. The heat was gradually increased, the fire being kept open to allow free access of air, and the charge stirred every ten or fifteen minutes.

At the end of three hours the smell of sulphurous acid was no longer perceptible; the roasting, however, was continued at a cherry red heat for half an hour longer, when the charge was drawn. Samples, taken at the end of three and three and one half hours respectively, showed the following percentages of SO_3:—

End of three hours, 7.55 per cent

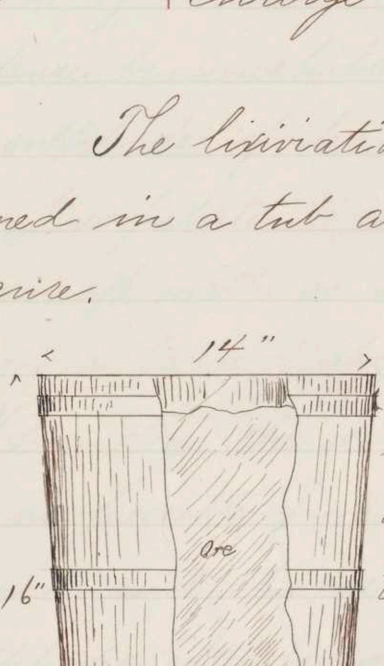
End of three and one half hours 7.68 per cent.

As soon as cool, the roasted ore was divided into two equal charges and each charge ground in the Chilian mill with one pound or about five per cent of common salt. The grinding of each charge occupied about an hour, at the end of which time the ore was in the state of a very fine dark red powder.

The furnace being at a dull red heat, the mixture of ore and salt was charged for chlorination After half an hour's warming, the charge was stirred; dense white fumes were evolved, rendering it impossible to see the parts of the charge farthest from the door. In about an hour the charge had assumed the light, almost flocculent appearance, known as "woolly." The fire was urged until the furnace was at a red heat. The charge was stirred every ten minutes, and, at the end of two hours from the time of charging, while still fuming, was drawn. By the roasting, the charge had increased considerably in bulk, and was in the state of an exceedingly fine, light powder. The following is a summary of the roasting:—

Time	Remarks	Wt. Coal, lbs.
5.10 A.M.	Fire lighted	146 3/8
9.30	Fifty pounds charged for oxidation	
1.00 P.M.	Charge drawn. Weight = 38 3/4 lbs.	54 3/8
5.00	Charged for chlorination 39 3/4 lbs.	
	One pound lost in grinding.	
7.00	Charge drawn. Weight = 36 7/8	43 1/2
		244 1/4

The lixiviation of the roasted ore was performed in a tub arranged as represented in the figure.



On the bottom of the tub was first spread a layer of coarse pebbles. Over this a layer of pebbles which had passed through a half inch and over a quarter inch sieve, then a layer of gravel which had passed through an eighth and over a sixteenth inch sieve. On this gravel bottom the ore was placed. Upon the ore a hot, strong solution of common salt was poured and after percolating through the mass was collected in large glass bottles placed under the spout. In order to expedite the leaching, which at first proceeded very slowly, a stopper provided with two tubes was inserted in the neck of a bottle; one tube was connected with an aspirator while the other was connected with the spout at the bottom of the tub. By this means the liquid was caused to flow in a stream from the tub.

To the liquid contained in the bottles, poly-sulphide of sodium was added in excess. Immediately a dense brownish-black precipitate, consisting mainly of sulphide of lead, was thrown down. After allowing the precipitate to settle the supernatant liquid was drawn off with a siphon and the sulphides thrown upon a large paper filter and dried.

When dry, the sulphides were separated as completely as possible from the paper; the paper was then burned and the ashes added to the sulphides. The sulphides, after being mixed with an equal weight of granulated lead, were fused in a Hessian crucible. The resulting lead button was cupelled leaving a silver button weighing .68 grms. An assay of the ore after the oxidizing roasting shows .0415 per cent. of silver. An assay of the residues from the lixiviation gives .026 percent. of silver. As it was omitted to take a sample of the chlorinated ore, the loss during chlorination cannot be determined.

The following figures show the loss of silver at the different stages of the operation:—

	Wt. lbs.	Percentage of original silver
Loss by oxidizing roasting	= .0031	16.2
" between O. roasting and cupil'n	= .0052	27.1
" in residues	= .0094	48.9
Total loss	= .0177	92.2
Obtained by cupellation	= .0015	7.8
Original silver	.0192	100.0

The heavy loss during the oxidizing roasting is attributed mainly to the using of too high temperatures and continuing the roasting for a too long time. In the lixiviation, special pains were not taken to have the solution saturated or boiling; also, as the experiment was intended to be merely qualitative, the leaching was not continued for a sufficiently long time to extract all the chloride of silver. This may in some measure account for the rich residues.

Experiment II:- Augustine Process.

One hundred pounds of ore, crushed to one fortieth of an inch, were subjected, first to an oxidizing and then to a chlorinating roasting. As the details of the roasting were very similar to those in the first experiment, the following summary only, is given:-

Time	Remarks	Wt charge lbs.	Wt coal lbs.
5.20 A.M.	Fire lighted		112 3/4
9.00	Charge I in for oxidizing. Furnace dull red	50	
11.30	" " out. Furnace cherry red.	41 1/8	40 1/2
11.45	" II in for oxidizing. Clinkers in fire	50	
	" I is ground with 2.5 lbs. salt.		
P.M. 2.15	" II drawn.	41 3/8	62 1/8
2.20	" I Charged for chlorination	38 3/4	
	" II ground with 2.5 lbs salt.		
4.30	" I out. Furnace dull red	34 1/2	27 1/8
4.45	" II charged for chlorination	48 1/2	
6.55	" II drawn	45 1/2	46 3/8
	Obtained by sweeping furnace when cold	2 1/2	
	Total coal		288 7/8

It will be seen from the above that the oxidizing roasting was one hour shorter than in the case of the experimental roast. The heat used in the chlorination was also much lower than in the experimental roast.

The charge when cool was divided into two portions and each placed in the lixiviating tub and leached, first with cold water, then with hot water and finally with a boiling, saturated solution of common salt. The liquid obtained by leaching twice with cold water gave tests for sulphuric acid and chlorine but no precipitate with sulphide of sodium. The leaching with hot water was continued until five of the two gallon bottles had been filled. From the liquid in the first bottle of the five, crystals of sulphate of soda separated, on cooling. The contents of all the bottles gave strong tests for chlorine and sulphuric acid but no precipitate with sulphide of sodium. Leaching with brine was then commenced and continued until six bottles had been filled. The brine was heated, first by blowing in steam, then to boiling over a large Bunsen lamp. From the liquid in the first four bottles a white crystalline precipitate separated on cooling. This, on examination proved to be principally chloride of lead. The precipitate in the first bottle gave a strong test for silver; that in the second, a trace. After the first two bottles not the slightest trace of silver could be detected. The separation of the chloride of lead was at one time so rapid as to choke the spout leading from the leaching tub to the bottles.

On account of the small amount of silver, it was decided to use poly-sulphide of sodium, instead of metallic copper, for the precipitation. The precipitated sulphides in the first two, but in none of the subsequent bottles gave tests for silver. The precipitated sulphides were collected on filters and dried, the filters being burned as in experiment I. The weight of the sulphides thus obtained, was 1044 grammes. The sulphides were then mixed with 500 grammes of soda, 20 grammes of borax, 20 grammes of coal and 300 grammes of iron scraps. This mixture was fused in a No 7. Hessian crucible. Owing to the cracking of the crucible, the charge ran into the ash pit of the furnace. The ashes were collected, crushed between rolls and panned. The lead resulting from the panning, together with what portion of the charge had not run through, was fused in a large five Hessian crucible. The weight of the soft lead thus obtained was 357. grammes.

This lead, cupelled in the large muffle, yielded a button of silver weighing 4.11 grammes. This button has been parted, yielding .0444 gramme of gold. As sulphide of gold is very soluble in alkaline sulphides, the presence of gold in the silver button may be due to some sulphide of gold having been dragged down by the great mass of lead sulphide. The presence of chloride of lead in the liquid obtained by leaching with brine, while not a trace of lead was detected in the liquid from the hot water leaching, may have been due to the formation, during roasting, of an oxy-chloride of lead insoluble in water. Assays of the ore at the end of the chlorination roasting, and of the residues from the lixiviation give;—

	Silver per cent.	Gold per cent.
At end of Chlorination:	.0393	—
Residues	.0207	.0004.

The following figures show the losses at the various steps:—

	Wt. silver lbs.	Percentage of original silver
By Roasting	= .0060	15.6
Between Chlorination and Cupellation	= .0078	20.3
In residues	= .0155	40.4
Total loss	.0293	76.3
Obtained by cupellation	.0091	23.7
Original silver	.0384	100.0

	Wt. Gold lbs.	Percentage of original gold
Roasting, precipitation &c. &c.	.0003	42.9
Residues.	.0003	42.9
Total loss	.0006	85.8
Obtained	.0001	14.3
Original Gold	.0007	100.1

Experiment III: Ziervogel's Process.

To remove the doubts regarding the possibility of extracting any of the silver by this method, 500 grammes of ore were placed in an iron pan and roasted in the large muffle. The heat, which was kept low during the first part of the operation, was gradually increased, until at the end of one and one half hours, when the roasting was completed, the muffle was bright red. The ore thus roasted was placed in a glass flask, covered with water and allowed to remain at a temperature of 100°C. for several hours. The contents of the flask was then thrown upon a large paper filter and washed with boiling water. The resulting filtrate failed to give the slightest precipitate with chloride of barium or sulphydrate of ammonium.

It is possible that the sulphate of lead fusing so coated over the sulphide of silver as to prevent oxidation. It is also probable that the arsenic acid formed by roasting, united with the silver to a certain extent, forming insoluble arseniate of silver.

In view of these results, the idea of working a larger quantity of ore by this method was abandoned.

Experiment IV:— Von Patera's Process.

Before trying this method it was necessary to introduce steam into the furnace. This was effected by entering a quarter inch pipe at the tap hole, and by means of three elbows, connecting it inside the furnace with another quarter inch pipe fastened against the side of the firebridge next the bed. This latter pipe was perforated with eleven holes at equal distances from each other, each hole being about one twentieth of an inch in diameter. By means of a cock outside the furnace the supply of steam could be regulated. One hundred pounds of ore were operated upon in two charges of fify pounds each. The following is the record kept during the roasting:—

			Wt. lbs	
Time.	Remarks	Charge.	Coal.	Coke.
5.10 A.M.	Fire lighted		115 1/8	
9.10	Furnace dull red. Charge I in	50		
10.00	Charge red hot. Steam turned on.			
11.40	charge has ceased fuming. Five percent of salt stirred in. Fumes not so dense as in previous chlorinations			
P.M. 12.40	Two percent of salt stirred in.			
1.30	Charge ~~I~~ out. Furnace at dull red heat		59 3/8	52 1/8
1.35	Charge II in	50		

Time	Remarks	Charge	Coal	Coke
2.25	Charge at red heat, steam on			
4.05	Charge ceased fuming. Five percent of salt in			
5.05	Two percent. of salt added			
6.00	Furnace dull red. Charge II drawn		46⅛	58
	Total weight after roasting	76¾		
	Total fuel		220⅝	110⅛

As the supply of Cumberland coal fell short it was found necessary to use a mixture of coal and coke. The large amount of fuel required to keep the furnace sufficiently hot was due to the heat being carried off by the water vapor. The lixiviating apparatus was the same used in former experiments. The ore was divided into two charges and each lixiviated as follows:—

First a leaching with cold water for the purpose of removing any excess of common salt which would have caused a loss of silver in the following leaching. Secondly, a leaching with hot water to remove chlorides of lead, zinc &c. Thirdly a leaching with cold water to cool the mass preparatory to leaching with the hyposulphite solution. Finally a leaching with a solution of hyposulphite of soda.

The first leaching was continued until the resulting liquor gave ~~only~~ a faint test for chlorine. Contrary to expectation the liquors from the second leaching gave not the slightest precipitate with sulphydrate of ammonium

The hyposulphite solution was made by dissolving one half pound of cystallized hyposulphite of soda in one gallon of water. This solution after having been passed several times through the charge, was poured upon the charge and the spout at the bottom of the tub closed. After remaining over night, the liquid was drawn off and the ore leached with a fresh quantity of the hyposulphite solution, the last portions of which were removed by leaching with water. Poly-sulphide of sodium was then added to the liquor from the hyposulphite leaching. The precipitated sulphides after being dried and the filters burned, as in preceding experiments, weighed about 90 grammes. These sulphides were mixed with 150 grammes of proof lead and fused in a Hessian crucible, yielding about 200 grammes of lead. This lead gave, on cupellation a silver button weighing 4.3 grammes. This button on parting yielded .0605 grammes of gold.

Assays give the following results:—

	Percentage of Silver	Percentage of Gold
At end of chlorination	.0325	——
Residues	.0165	.0005

The following figures give the losses:—

	Silver wt. lbs.	Percent. of Orig. Silver.
By chlorination	.0135	35.2
Between Chlorination and Cupellation	.0039	10.2
Residues	.0115	29.9
Total loss	.0289	75.3
Obtained	.0095	24.7
Original silver	.0384	100.0

	Gold wt. lbs	Percent. of Orig Gold.
By chlorination, lixiviation &c.	.00022	31.4
Residues	.00035	50.0
Total loss	.00057	81.4
Obtained	.00013	18.6
Original Gold	.00070	100.0

By action of the steam, hydrochloric acid gas is formed which converts the arsenic into a volatile chloride. If as has been suggested the greater part of the silver is contained in an arsenical tetrahedrite, the loss of so large a percentage during roasting may in some measure be accounted for. At Joachimsthal the loss of over ten percent of the silver in an arsenical ore is prevented by connecting the furnace with dust chambers.

Experiment V. Smelting Process.

Previous to the smelting of the ore, two matte assays were made. The first was made by fusing 10 grammes of ore in a Hessian crucible with 25 grammes of borax, 15 grammes of glass and 20 grammes of salt. The ore was put at the bottom of the crucible and the fluxes placed over it in the order of their naming. A piece of coal was laid on top of the charge and the crucible covered. The crucible was kept at a bright yellow heat for an half hour. The result of this assay was 37½ per cent. of matte.

The second assay was made by fusing 50 grammes of ore in a Hessian crucible with the simple addition of a piece of coal, to keep a reducing atmosphere. The heat was the same as in the first assay. On cooling, the contents of the crucible was examined and found to have been fused and apparently quite liquid, a clean button of matte separating from the slag. The slag was glassy and siliceous. The results of this assay was 45 percent of matte.

Having succeeded so well in fusing a small quantity of the ore without the addition of any flux, and the percentage of matte being about that desired, it was decided to undertake the fusion of a larger quantity of the ore in black lead crucibles, without adding a flux, relying upon the siderite to furnish bases necessary to the formation of a slag.

Accordingly, each of two Nº 25 black lead crucibles was charged with a mixture of 16 lbs of ore and ½ lb. of anthracite coal dust. It was soon found that the charges were too large, for as soon as the crucibles became heated to redness, the escaping carbonic acid gas caused a sort of ebullition and threw part of the charges out of the crucibles. To prevent this, the crucibles were covered with sheet iron. After spending five hours in a vain attempt to fuse the charges, the blast was attached. In about half an hour after connecting the blast, one of the charges was poured. The slag was thick and ropy. After pouring the other charge which was in much the same condition as the first, operations were suspended for the day. When cool, the slag and matte were examined. The slag was so siliceous as to appear quite stony. Through out the matte, fine globules of lead were scattered, standing out in drops on the surface of a piece broken while hot. Owing to the difficult fusibility of the slag the matte didnot separate well, therefore all thought of repeating this experiment was abandoned.

It was then decided to undertake the smelting of another portion of ore with the addition of magnetite to furnish iron for the slag. A mixture was accordingly made of 12 lbs of ore, 3 lbs of magnetite and ½ lb. of coal. Each of two black lead crucibles was charged with a mixture of this kind and placed in a large crucible furnace. With the aid of a blast this mixture was easily fused, six pours being made in an afternoon. The slag was very liquid and on cooling separated cleanly from the matte. The crucibles were examined after the run and found to be nearly intact. 104 lbs. of ore gave 46½ lbs. of matte. This matte resembled in all respects that from the fusion without flux. The slag showed a slight tendency to crystallize. In making the mixture, a slag slightly more ~~fusible~~ acid than a uni-silicate was calculated for. The matte was crushed to one twelfth of an inch and sampled. A partial analysis shows the following composition:—

Sulphur	26.50	per cent.
Lead	14.82	" "
Copper	1.36	" "
Iron	46.39	" "

The crushed matte was then roasted for six hours in the hollow bed reverberatory furnace, at the end of which time the smell of sulphurous acid had ceased. The fuel used was coke and Cumberland coal, of which 173¼ lbs of coke and 51⅛ lbs. of coal were consumed in heating the furnace and in roasting. The weight of the charge when drawn was 47¾ lbs. showing an increase in weight by the roasting. This might at first seem rather strange, but a determination of the sulphur in the roasted matte shows 3.10 percent. By calulation this is found to be slightly in excess of the amount of sulphur required to convert all the lead to sulphate. Calculating the iron to have become changed to peroxide, the copper, partly to sulphate and partly to oxide and all the lead to sulphate, the other elements remaining unchanged, there should be an increase in weight.

The roasted matte was now mixed with coal and glass in such proportions as to give a slag containing about 30 per cent. of silica provided that the 3.10 per cent. of sulphur remained in the matte. A mixture of 12 parts matte, 4 parts glass and ½ part anthracite dust was charged into a black lead crucible. With the aid of the blast the charge was melted. On pouring the not very liquid mass, the crucible was found much eaten. In the next charge the amount of glass was increased to 4½ lbs. This charge acted much like the first. The third charge ran through the crucible putting a stop to farther operations.

On examination it was found that no matte had been formed, but instead a curious alloy of lead, copper and iron.

As globules of this alloy were scattered throughout the slag, the whole slag was passed between the rolls and sifted. The flattened globules which remained on the sieve, together with the cake from the bottom of the buggy were fused with a mixture of soda, coal, pyrite and borax. The object of the addition of the pyrite was the removal of iron and copper in a matte. The lead resulting from the fusion weighed 282 grammes. This lead gave on cupellation, and parting of the resulting silver button, 1.44 grammes silver and .0496 gramme gold. Assays give the following:—

Raw matte	.0705 per cent silver	.0011 per cent gold
Roasted "	.0694 " "	"

The losses were:—	Silver wt lbs	Percentage of orig. silver
First smelting	.0011	10.5
Roast	.0002	2.1
Second smelt. + cupellation	.0051	53.1
Total loss	.0064	65.7
Obtained	.0032	34.0
Started with	.0096	99.7

	Wt. gold lbs	Percentage of original gold.
Smelting + roasting	.000043	24.6
Second smelt. & Cupell'n.	.000013	7.4
Total loss	.000056	32.0
Obtained	.000119	68.0
Started with	.000175	100.0

It is probable that had there been sufficient sulphur to form a matte, soft lead would have been obtained in the second fusion and the heavy loss of silver would not have occurred. The remedy would naturally be, to give the matte a shorter roasting.

In spite of the poor result of the last experiment, it seems as if there could be no doubt that the smelting process is the only one adapted to the working of an ore like the one under consideration. The amount of zinc and arsenic is too small to be of much consequence in the blast furnace. The presence of so high a percentage of lead while it unfits the ore for amalgamation or solution methods is highly favorable to a smelting operation. Hence it is concluded that instead of attempting to work this product by itself it should be charged into the blast furnace with the product separated as smelting ore.

Note:— The values used in calculating the assays at the bottom of page 18 are, Silver $1.10 per oz. Gold $20.67 per oz.