

PYRITIC SMELTING OF A NICKEL-COPPER SULPHIDE ORE.

Hiram Niel Crichton

and

Bert Samuel Wohlgemuth.

Massachusetts Institute of Technology.

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INTRODUCTION.

The subject of this thesis is pyritic smelting in the Institute blast furnace of a mixture of copper-nickel matte. cupriferous pyrite. With the ordinary reducing smelting in and the Institute blast furnace, 18% of coke is used which furnishes a reducing atmosphere. In pyritic smelting, no coke or only a small amount is used, and this causes the smelting to take place under oxidizing conditions. The exidation of the iron and sulphur of the sulphide of iron and the formation of the slag, furnish the whole or the greater part of the heat necessary. For the success of the process, it is necessary to have an abundant supply of air at the right pressure and a sufficient quantity of silica to slag off the iron which is not required for the desired grade of matte. The matte concentration is affected by the amount of air furnished, by the quantity of silicia present and by the coke added. The slag composition under given conditions is always constant. An increase of silica will not change this composition, but causes the formation of a larger quantity of slag and a rise in the grade of the mette.ackslash

MATERIALS.

The ore smelted consisted of equal parts of coppernickel matte and cupriferous pyrite.

<u>Matte</u>. The matte used was a mixture of four lots of copper matter and one lot of nickel matte produced at the Institute in laboratory runs and in a thesis run of 1908. The five lots of matter

were separately broken by hand to 30 mm. and screwed on 8 mm. sieve to remove the fines, as only the oversize was to be used. This was sampled by fractional selection, one tenth part by weight of each lot being reserved as sample. The samples were combined and reduced to 1/4 inch in the Sturtevant roll-jaw crusher. The combined sample was then halved in the Jones sampler and the reserved portion put through the Hendrick-Bolthoff sample grinder to pass a 30 mesh sieve, then split-shoveled down to 485 grams and this ground on the bucking plate to pass a 100 mesh screen. The matte was found to be magnetic, hence the metallic iron particles from the machines could not be removed with a magnet.

The matte was analyzed for Cu, Ni, Fe, Pb, S, CaO, Alg 0_3 and SiO₂

The sample for analysis was treated with a solution of bromide in HNO_3 (Sp.Gr.1.42). After action ceased, concentrated HO/was added, and the solution boiled until no more substance was dissolved. The residue was then filtered, fused with $Na_2 CO_3$ and added to the main solution. The solution was then evapcrated to dryness, dehydrated, HO/and water were added, and the silica filtered off, washed, ignited, and weighed.

x Foot-note from 1st. page: -- Thesis No. 341 by A. H. Bradford and C. A. Gibbons, Jr.

H2 S was passed into the slightly acid filtrate, the sulphides of copper and lead were precipitated and filtered. The precipitate was dissolved in H N Og and the solution electrolyzed. The copper was weighed as Cu and the lead as PbO.2

The filtrate was evaporated nearly to dryness with $H_2S O_4$, water was added, and the solution passed through a Jones reductor. The iron was determined by titrating with K MnO₄, After titrating the solution was evaporated to about 250 C.C., and the nickel precipitated by the dimethylglyoxime method and weighed as $C_8 H_{14}^{-N} O_4^{-N}$.

A fresh sample was taken for the determination of CaO and Al $_2$ O₃. It was put into solution as above, SiO₂ , Cu and Pb were separated as shown, and the combined hydroxides of aluminum and iron thrown down by means of a double ammonia precipitation, filtered, ignited and weighed. As some nickel is carried down its amount has to be determined. This was done by fusing the ignited precipitate with KHSO₄, taking up with water, precipitating with dimethylglyoxime, and weighing. Deducting the weight of the previously determined oxides of iron and nickel gave the Al₂ O₃.

The calcium was precipitated in the filtrate as calcium oxalate. The precipitate was dissolved in H $_2$ SO₄ and titrated hot with KMnO₄.

Sulphur was determined in one sample by the Fresenius method of fusing with Na₂ CO₃ and K N O₃ and in another by the Glaser method of fusing with Na₂O₂.

The matte was found to contain some magnetic oxide of iron. The analysis gave the following results:

	Cu	Ni	Fe	Pb	S	CaO	A1_0_	Si0	o#	Total
70	9.54	2.35	55.32	• 42	25.94	•46	.45	.63	4.89	100

0 as FeO and Fe_30_4 .

<u>Pyrite</u>. The pyrite used came from the Eustis mine at Capleton, Province of Quebec, Canada. The ore was a massive copperbearing pyrite with some quartz as gangue. It was broken by hand to pass a 30 mm. screen, the smalls, finer than 8 mm. were removed by screening. The coarse ore was halved by fractional selection and one half sampled by taking pieces from it when spread on the floor. The sample was crushed in the Sturtevant roll-jaw crusher, cut in half by means of the Jones sampler and ground in the Hendrick, Bolthoff sample grinder. The pulp was then split-shoveled to 210 grams and this amount bucked through a 100-mesh sieve. The metallic iron from the wear of the machines was removed with a magnet.

The pyrite was analyzed for SiO_2 , Fe, Al_2O_3 . S, and Cu. The SiO_2 , combined exides of iron and aluminum and S were determined in one sample, as followd:

A sample of about 5/10 gram. of pyrite was dissolved in 20 c.c. of a frehly-made mixture of three volumes HNO3 (Sp.gr.1.42) and one volume of HC1 (Sp. gr. 1.20). When disintegration was complete 5c.c. of sodium carbonate solution were added. The whole was evaporated to dryness, treated with 5c.c. HC1 and dehydrated. The residue was moistened with

lc.c. HCl, 100 c.c. of hot water were added, the whole heated to beiling and the silice filtered off, washed, and ignited and weighed. A test with HF showed that the insoluble residue was silica.

The filtrate was treated with bromine water to oxidize the iron. The combined oxides of iron and aluminum were precipitated by pouring the hot filtrate into an excess of ammonia, filtering off the hydroxides. They were re-dissolved in HCl and re-precipitated with ammonia. This second filtrate and the washing were added to the first filtrate. The precipitate was ignited and weighed as Fe_203 and Al_203 Sulphur was determined as Ba SO4 by precipitating with Ba Cl2 in the hot filtrate.

Iron was determined from the second sample of pyrite by dissolving in the same mixture of nitric acid and hydrochloric acid as above, filtering, fusing the residue with sodium carbonate, adding water and suphuric acid (conc.), evaporated until fumes are given off, passing through a Jones reductor and titrating with KMnO₄. Copper was determined in the third sample of pyrite. It was dissolved as above, neutralized with ammonia, made slightly acid with hydrochToric acid, heated to boiling and hydrogen sulphide passed in. The precipitated copper sulphide was filtered off, dissolved in nitric acid and electrolyzed. The following are the results of the analysis.

	Fe	S	Cu	Si0 ₂	A1203	Total
%	40.62	45.11	1.25	10.12	2.07	99.17

The fluxes used were limestone and quartz.

Limestone. The limestone used was a very pure white marble. It was sampled, crushed, and ground to about 30-mesh in the same way as the pyrite. The sample was split-shoveled to 300 grams and then bucked through a 100-mesh screen. All metallic iron present was removed with a magnet. The analysis was carried out in about the same way as in the Spathic Iron Ore Proceedure, common in the Institute Laboratories.

The analysis was as follows:

	CaO	MgO	FeO	Si02	C02#	Total
%	54,16	1.13	• 35	.21	43.89	99.74

Calculated.

Quartz. The quartz was the same used in the thesis of Messre. Bradford and Gibbons, Jr., in 1908. Their analysis gave:

	Si02	FeO	A1203	Total
8/0	96.61	1.50	0.49	98.60

For use in the smelt the quartz was broken up by hand to pass through a 30 mm. screen. The fines were not removed. <u>Fuel</u>. The fuel used was Everett coke taken from the Institute bins. A sufficient quantity was put aside for the run and sampled by reserving a piece from every fourth shovelful. The sample was broken by hand, then crushed and ground in the same way as the pyrite. It was split-shoveled down to 100 grams and this amount bucked through a 100-mesh screen. The following is "approximate" analysis of the coke.

Fixed Car	bon	· • • • • • • • • •	. 77.02	Z
Volatile	Matter		. 6.76	%
Ash		• • • • • • • • • • •	.16.22	%
Moisture	(Undried	Sample).	00.92	ħ

The ash gave, upon analysis,

• :	A1203 Mg0	Si02	Fe ₂ 03	Al203	1.80	Alk's	Total
6/0	6.39 253	32.10	35,10	15.75	3.75	4.38	100.00

In the subjoined Table I are assembled the results of

the above analyses.

Materials	Silz	Fe	FeO	FezQ	CaO	MgO	AlzO3	COz	0	S	Cy	Ni	Pb.
Matte	0,63	55.32			046		0.45		4.89	25.94	9,54	2.35	042
Pyrite	/0.12	. 40.62					2.07	A - 4		45.11	1.25		
Limeste	0.21		D.35		54.16	1.13	•	43.89					
Quartz	96.61		1.50				0.49					\$ O3	AlK's
CoKe Ash	32.10		x 2303	35,10	6.39	2.53	15.75					3.75	<i>4.3</i> 8

For convenience in calculating the slag the percentage of magnesia and alumina were changed into equivalent percentages of lime and added to the CaO column. This was done as follows.

 $\frac{\text{Mol. Wt. of Ca0}}{\text{Mol. Wt. of Al}_2 0_3} \times \text{ x Al}_2 0_3 = \text{Equivalent % of Ca0}$

 $\frac{\text{Mol. Wt. of Ca0}}{\text{Mol. Wt. of Mg0}} \times \% \text{ Mg0} = \text{Equivalent } \% \text{ of Ca0.}$

Table No. I is thus changed as given below in Table No. II.

Materials	Si02	FeO	CaO	S	Cu	Ni	Pb
Matte	0.63	71.12	0.71	25.94	9.54	2.35	0.42
Pyrite	10.12	52.22	1.13	45.11	1.25		
Quartz	96.61	1.50	0.27				
Limestone	0.21	0.35	55.73				
Coke Ash	32.10.	31.59	18.53	1.50			

DESCRIPTION OF THE BLAST FURNACE.

The blast furnace of the laboratory is similar to the Arizona copper blast furnace. It consists of a water-jacketed beiler shell resting on four cast iron columns. The working height is 46" and the distance from the bottom to the level of the tuyeres is 14". The diameter at the top of the furnace is 23-1/4" and at the bottom 17-1/2",

There are seven tuyeres -- four 1-3/8" and three 2" in diameter. The bustle pipe is 6" in diameter. This is shown in the drawing.

The crucible was put in the furnace as follows: the bottom plate was placed in position and clamped. Then a mixture of 3 parts coke, 1-1/2 part ashes and 1 part fire clay, was introduced through the charging door and tamped firmly into place. The furnace was filled up to the level of the tuyeres with this material firmly tamped down. Then the plate which closed the breast of the furnace was removed and the central part of the compact brasque dug out leaving the crucible with the cross section shown in the drawing.

THE BLOWER.

The blower which supplied the air to the furnace is a Root No. 1/2. It has a rated capacity of 487 cubic feet of free air per minute, running at a rate of 325 R. P.M. The blast main is 6" in diameter.



The volume of air furnished to the furnace was measured with a Pitot tube and two U-tube manemeters, giving respectively the static and velocity pressure heads.





Static Pressure

CALCULATION OF CHARGE.

It was decided to smelt a mixture of equal parts of matte and pyrite and to pruduce a matte with 30% copper and nickel.

Starting with 50 Kgs. matte and 50 Kgs. pyrite. The mixture contains 5.40 Kgs. Cu, 1.17 Kgs. Ni and .21 Kgs. Pb. Total = 6.78 Kgs. Cu, Ni, Pb. These 6.78 Kgs. = 30% of Wt. of matte, Hence 6.78 kgs. = 30% of Wt. of matte, Hence 6.78 x 100 = 22.6 Kgs. is the Wt. of matte to be produced per 100 Kgs. ore. New 5.40 Kgs. copper = 6.76 Kgs. Cu₂S 1.17 " Ni = 2.13 " Ni₃S₂ 0.21 " Pb = 0.24 " Pb S Total = 9.13 " Matte.

The FeS in the mette will be

22.60 - 9.13 = 13.47 Kgs.

To supply the FeO necessary for this $13.47 \times \frac{\text{FeO}}{\text{FeS}}$ 12 Kgs. coke per 100 kgs. mette and pyrite were used.

A former run, similar in cherecter to the present one showed that an addition of 12 Kgs. coke to 100 Kgs. of ore would supplement the heat generated by the combustion of iron and sulphur -- hence the same amount of fuel this time.

The slag chosen had the following composition: Si 02' 36.8 %; FeO, 51.2 %; CaO, 12.0%. It lies within the range of pyritic slag.

The table No. III shows that the proposed charge

contains 62.29 Kgs. FeO.

From this total will have to be deducted the number of Kgs. FeO going into the matte.

62.29 - 11.03 = 51.26 Kgs. FeO to be fluxed.

The Silica necessary for this FeO equals $\frac{36.8}{51.2}$ x 51.26

= 36.84 Kgs.

Table Nc. III shows that there is 6.00 Kgs. silica present. Hence, Si 0_2 to be supplied equals 36.84 - 6.00 = 30.84 Kgs. The quartz used contains 95.53 % available Si 0_2 ? Therefore quartz that has to be added $\frac{30.84}{95.53} \times 100 = 32.29^{\text{Kgs.}}$

To find the CaO necessary the silica was used as the basis of calculation. As shown by table No. III there are now present 37.20 Kgs. of SiO₂. CaO necessary for this is

 $\frac{12}{36.8} \times 37.20 = 12.14 \text{ Kgs}.$ There are already present 1.37 Kgs. CaO. Hence the CaO to be supplied is

12.14 - 1.37 = 10.77 Kgs. The limestone contains .35% FeO. As this is equivalent to 12 \times .35 % FeO = .08% CaO, there is 55.73 - .08 = 55.65% CaO available in the limestone. Hence, the limestone necessary to furnish the CaO is

 $\frac{10.77}{55.65}$ x 100 = 19.36 Kgs.

			1.			1.			1	<u> </u>	
* SiO2 in + Includ \$ 8.0 Kgs	3/ag	CoKe.	Total	Limeste	Quartz	Total	Coke Ash.	Pyrite.	Matte		Naterials
tor est		12:00	153,60	19.36	32.29	101.95	1.95	50	50		WŁ Kgs.
for is	36			0.21*	96.61		32.10	10,12	0.63	0%	Si
negle matte	5.8		37,24	0.04	31.20	6,00	0.63	5.06	0.31	wt.	O_2
eted. and + .92	5,			0.35	1.50		31.59	57.22	71.12	0/0	وع
ylag 7 m	2		+ 62.84	0.07	0.48	62.29	0.62	26,11	35.56	Wt	0
L/1	12			55.73	0.27		18.53	1.13	0.71	0/0	C_{d}
103 Kg	0		12.15	10.78	0.99	1.28	3.5.0	0.56	036	WZ.	0
teo	_						1.50	<u>45</u> /I	25.94	0/0	S
for n						35.56	.03	22.56	12.97	Wt	
ratte								1.25	9.5H	0/0	C
						5.40		0.63	4.77	WŻ.	×
									2.35	0/0	\$
						1.17			1.17	wt	×.
									0.42	0/0	7
					- -	0.21			0.21	WZ.	6
		7.91\$	100.00	12.77	21.29			32.97	32.97		Charge.

TABLE NO. III.

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DESCRIPTION OF RUN AND DATA SHEETS.

The furnace was blown in at 8.45 AM . The blowing in charge consisted of paper, kindling wood, charcoal, coke, copper slag and some fine copper matte. The individual charges and the order in which they were fed are shown in the weigher and feeder's data sheets. The blowing in charges were the same as those used in the laboratory for blowing in the blast furnace for a normal reducing fusion. The furnace, filled with the blowing in charge. was lighted, later, a tuyere was inserted in the tap-hole and a gentle blast turned on. This was kept going until the charcoal and coke were burning freely. Then the four 1-3/8" tuyeres were inserted and the furnace run a while until it got hot enough for the pyritic charge. Now the three 2" tuteres were put in end the regular pyritic charges of 100 Kgs. of ore and flux with 8 Kgs. of coke fed.

During the run the following reads were taken: temperature of jacket water at inlet and outlet, etatic and velocity heads of the blast, meisture (Grains per cubic feet) in the air, beremetric pressure, atmospheric temperature, and the temperature of the slag. The jacket water was measured at intervals by weighing the overflow during a half minute. Chilled slag samples were obtained from every slag part. Two flue gas analyses were also made during the run.

The furnace worked very well, the slag showed great fluidity and the same was the case with the matte of which a very large amount was produced. An artificial bosh was formed in the furnace as shown by the drawing, the tuyeres were black most of the time, but the blast penetrated the wall accretion without difficulty.

The run lasted till 2.15 P. M. The total emount of materials fed was 230 Kgs., slag; 236.8 Kgs., matte; 216.9 Kgs., pyrite; 139.9 Kgs., quartz; 72.1 Kgs., limestone; and 84.1 Kgs., coke.

WEIGHER.

÷	Time	Charge	Caka	0	re.	C/	FI	ύχ.	C· ,	
	weighed	No.	Core.	Matte.	Pyrite.	Slag.	Quartz	Limest		
	8.45	,		3 Å 3 _	lods hods	Kind Cha	king peoal		_	
		2	36							
		3	18			50				
	9.10	4	18			100				
\rightarrow	9.20	5	18.	20	- S	80	0	0	N. R. Perry	
	9.45	6	8.	33.0	33.1	}	21.3	12.8	u the r	
١	10.00	7	8	33.0	33.0		213	12.8	(i (j	
· /ړ	10.37	8	8	35.0	3300		21.2	12-8	· · · · ·	
′a √ 	40 47	9	8	33.0	330		213	12.8	~ ~	:
V	1,0,55	70	Σ_{1}	33.0	33.0		213	12.8	L 2	
	11.15		8	33.0	330		213	12.8		
- 1	11,45	12	4.6	18:18	18.8		12.1	7.3	In reducing change to	18.5 gruat
>	12,43	13	R.,		. -		-		End of Reputri Smith	62 A gree
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600 FEEDER Chirg Feed door Top Flue Gas No. Top Chirg Hot-Cold Time Temp Signature. Time Coke Charge 3 hodes Kin 3 hods Charcoal 1 2 3 4 5 3 mile Colo 9.25 N. R. Perry The cher 10.31 6 2.6" Hor -10.37 7 1 Cold -10.45 8 1. Warn gasanaly -1055 9 ,6" male 11 ar 1.3" Cold 11.15 11.32 10 gas analypi. made at 11 2'.0" Warm 12:04 11.55 Terry /2 12°F at 1'6" 212% 1223 12 12,43 13 2'0° bot 0120 18. 1213 19 2'0" hot 10

ASSISTANT TAPPER

	Time	Blast	Press	Overf	low Water	F'd Water	Athmo	Signature.
		Static	Vel.	Temp.	wt	Temp.	Temp.	
	9:55	5.5	• 36	510	per 1 min	12.5°	600	RaBickune
	10:05	6.45	.36	780		12:5	63.7	16
	10:10	6.45	.36	96.2		12.1	64.2	7/
	#10:16	6.25	-37	78.2		12.1	64.0	"
	10:26	6.35	37	65.0		12°./	66.0	11
	10:35	4.9	, 59	78.0		120.1	65.9	."
	10:40	4.9	60	82 ? . 1		12.01	66.3	11
	10:44 10:49 -	5.2 5.45	.58	92°.0. 96: 3	L	12.01	66.0	"
. (10:55 14)11:03	5.98	.58	99.0	6.8 K	12.01 12.1	67.3 67.7	11 11
	1:14-	6.40	.60	939.0	7.0 K.	12.1	67.2	
	11:22	6.80	.60	92.0		12.1	68.2	ļ
	11:36	6.70	161	92.0	m rlr	12.2	68.0	4 11
	11:47	6.9	.62	931.0	1.0 1	12.1	69.0	4
	12:00-	6.71	.62	91.2	6.9K	12.3	69.1	11
	12116	7.35	. 6/	89.0	MAL	12.4	68.8	"
	12:32	7.20	.61	84.50	7.2.1	12.1	67.0	" 1
	R:44	7.15	.61	83.5	4.11	1.2.1	70:0	1/
	12:55	7.05	. 62	68.7	/· / (12-0	70.3	Y I
	1:26	6.75	. 6 /	90.50		12.1	40.3	"
	1:37	7.05	.67	67. "		12.1	Y0.0	4
(X	1:53	7.00	.622	64.0		12.1	69.0	11 4
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	х.							
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X Pyritic fuelt.

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TAPPER.

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										Jugerer	
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										le a a va	
										1 - 2 7,15	
9.50	9.53										Lind he
7,00	1.53										Aj N. Crichton
10.07	10.04	12									
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10 22	10.20	ļ		.						Thursday	
	10.35									1 miles	
10,405	10 16 4									lary Tuneres	
10.40	10.79									fall closed	
10.55	10.57										
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11.14.	11.15							1.		Turpeter geli	i i i i i
11.24	11.26	1				••		17.	, n	large ant	¢.
11 30	11.39	2	Not	th	bh	th	bk	1h	bh	••	11
11 36	11.37	1						br	"	Slay acid	• 1
11.46	11.47	1				••		. 17	1.	<i>'</i>	
12.00	12.01	1	•••	•		۰.					e.
12.16	12.17	1	"			••	br	dr	br	<i>r</i> ,	• `
12.22	12.23	ľ	dh	dh	dh	dh	dk	dh	dk	1-	••
12.32	12.33	1	ic	te.	"	14	"	0	••	11	
12.44	12.43		· 10		r	t ⁱ	e	, .	"	۰, ۲	
2.55	12.56		•	11	"	"	41	"	"		t t
1,10	1.11	//		"	· ''		••	"			• •
1.25	1.20	' ,									
0	1721		(C	*	"	ч	'n	••	••	·• .	• •
1.33	1.55	2	n	"	"	••		••	••		••
1											

br = bright bk = black dr = dark

Time.	Slag No.	Hygrom. Reading.	Barom. Pressure.	Remarks.	Signature.
9.50	1	41.3	29.88	Thick - Guite warm	R.L. BEAles
10.02	2	39.50	29.88	Hot - More fluid.	R.L. BEAlES
10.10	3	40.50	••	p. e. 11	91. 99
10.13	4	. F1	۹.	" fluid	"
10.20	5	4	"		10 In 10 In 1
10.26	7	45 50		" first matte	u 7
10.33	9	"	14	" Very liquid -	11 10
10.40	10		14		ti v
10 55	11	50%	29.90		B Wohlgemuth
11.00	12	n. Relev	1.		
/1.03	13		4	Pyritic Smelt	B Wohlgemuth
11.24	15	45	v	Liquid fot	۲
11.36	17	4 4 1	••	Jenny slag 1120°C	(,
12 00	19	3.6 ct. 00	15	sauge and prince	ο του του του β ηλατού του
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12.22	21	J.2 J2	11	1 11 30	, (
.1232	22	v	PC	<i>III40</i>	//
12.44	23	n	" "	11 /140	e1
/10	25			At lo'clack power cutok	,•
1.25	26	it i		" 1:3mm power un	
137	27	//	ļ		()
153	28			Blowing Out 2.15	"

SLAGMAN.

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CLEAN UP.

The products of the smelt were divided into pyritic sleg, pyritic matte, preliminary matte, preliminary and final slag and odds and ends. Towards the end of the proliminary run, the pots were kept in the order in which they were filled, so as to keep separate the preliminary slag and matt from the pyritic and to distinguish them when cold by the difference in appearance of the slag. The preliminary and also the final slag were black in color, vitreous in lustre while the pyritic slag was black but was dull and non-vitrecus. All the matte produced after that which had been fed with the blowing in charges was considered to be pyritic matte. The odds and ends included the crust forming the bosh or wall accretion, the matte-scaked portion of the crucible, and the material drawn from the furnace after the last tap excepting the unburned coke which was picked out by hand.

It was decided not to sample and analyze the preliminary matte, as, being equal in weight to the matte added during the preliminary run, its composition was assumed to have remained unchanged, excepting the amount of copper that it probably took up from the blowing in slag. All other products were broken by hand to about egg-size and then sampled by fractional selection. A hod of each was preserved as a sample, and a hodful crushed, ground, and bucked to pass a 100-mesh screen in the same way as was done with the samples of ore for the smelt.

Analysis of Products. The products of the run were analyzed for their copper, nickel, and sulphur contents. In addition, there ware determined, in the pyritic matte, the iron, in the chilled sample of the pyritic slag; the FeO, SiO CaO, MgO, and Al 0. The methods of analysis were similar to those used with the raw materials of the smelt. Some difficulty was experienced in determining the copper electrolytically in the pyritic slag and in the edds and ends. The results were checked by the method of tiration with potassium iodate (KIO3) devised by G. S. Jamieson, L. H. Levy, and H. L. Wells and published in the journal of the American Chemical Society, 1908, Vol. I, Page 760.

TABLE NO. IV. ANALYSIS OF PRODUCTS. Matérials Cu Ni S Fe Matte 8.73 1.95 26.25 58.65 Pyritic Slag 0.76 0.06 2.75 Prel. & Final 0.28 0.00 1.48 Slag. Odds & Ends 1.59 0.29 2.51

Materials	S	FeO	Si02	CaO	MgO	A1203
Pyritic Slag Chilled Sample	2.57	28.80	37.35	18.34	0.86	4.61

GAS ANALYSIS.

	C0 ₂ &್ S0 ₂	0	CO	N
I	19.6%	0.6%	1.8%	78.0%
II	20.6%	0.4%	3.4%	75.6%

		No. I	No. II
-	so ₂	0.35%	0.15%

ACCOUNT OF STOCK.

The blowing in charges contained 20 Kgs. of matte, the analysis of which was not known. For this reason, a complete account of the copper is not possible.

The first 20 Kgs. of matte produced resulted from the matte mixture and foul slag used in the blowing in and wes assumed to contain all copper not accounted for in other products. The belance sheet was made, using this assumption.

The balance sheet of sulphur was calculated only for the pyritic run.

BALANCE SHEET OF MATERIALS.

		T	ABLE NO	VI			······································
Materials Wt.in		19 C	opper		Nickel		
Fed	Kgs.	5/0	Kgs	Total	ę,	Wt.kg.	% Of
Matte	216.8	9.54	20.68	80.79	2.35	5.09	100
Pyrite	216.8	1.25	2.70	10.54			
Matte Mix	20 [#] 0		` .			• •	
Foul Slag	330.0	0.67	2.22	8.67			
Quartz	139.9						
Limestone	84.1						
Coke	160.6			·	1		
Total	1168.2		25.60			5.09	

This analysis unknown as it was decided just before the

run to add the matte mixture.

TABLE NO. VII.

Sand in the shift of the	•	TAB	TE NO.	VII.			
Materials	Wt. in		Copper			Nickel	
Produced	Kgs.	1/2	Kgs	% of Totai	%	Kgs	% of Total
Matte	216.4	8,73	18.89	73:80	1.95	4.22	88.6 5
Prelim.Matte	20.0	7	1.90#	7.41			
Pyritic Slag	256.0	0.76	1.95	7.62	0.06	0.15	3.15
Prelim & Fin Slag	255.6	0.28	0.72	2.81			
Odds & Ends	134.4	1.59	2.14	8.36	0.29	0.39	8.20
Total	88.24	· · · · · · · · · · · · · · · · · · ·	25.60			4.76	

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Since the Cu in the matte used when blowing in is notknown a complete account of the Cu is not possible.

BALANCE SHEET OF SULPHUR.

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Materials Charged.	Weight Kgs.	70	Sulphur. Wt.Kgs.	% of total.
Matte	216.8	25,94	56.24	36.50
Pyrite	216.8	45.11	97.80	63.46
Coke	52.6	0.14	0.07	0.04
Total	486.2		154.11	100.00

TABLE NO. VIII.

TABLE NO. IX.

Materials	Weight	Sulphur.					
Produced.	Kgs.	%	Wt. Kgs.	% of total			
Matte	216.4	26.25	56.80	84.49			
Pyritic Slag	256.0	2.75	7.04	10.50			
Odds & E nds	134.4	2.51	3.37	5.01			
Total	606.8		67.21	100.00			

CALCULATION OF THEORETICAL BALANCE SHEET OF MATERIALS

FOR ONE PYRITIC CHARGE.

There are three methods of calculating a Theoretical Balance Sheet of Material. They are based on, (1) total materials; (2) definite weight of matte produced; (3) materials of a single charge.

The irregularities in the running of the furnace during blowing in and blowing out make it difficult to ohtain reliable data for these two periods and also to define the limits of the run proper. The same holds good for making a definite amount of matte produced, the basis of calculation. In the third method, these errors are avoided, as the calculations are based upon data taken while the furnace is running normally. It was therefore chosen for making a theoretical balance sheet of material.

Since the heat required to smelt the charge depends largely upon the form in the elements are chemically combined, it is necessary to calculate a rational analysis of the materials charged. This is done as follows: <u>Matte</u>. From the analysis of the materit is known that the iron is present as FeS, FeO, Fe₃O₄ and the remainder is assumed to exist as metallic iron. The amount of oxygen present is assumed to be the per cent not acounted for in the chemical analysis. With these assumptions, the amounts of FeS, FeO, Fe₃O₄ and Fe were calculated as follows:

Iron present is FeS.

This is found by calculating the sulphur necessary to unite with the Cu, Ni, and Pb present to form $Cu_2 S$, $Ni_3 S_2$ and Pbs and assuming the remaining S to exist as FeS.

Total sulphur present = 25.94%S as Cu₂S Ni₃S₂and Pbs = 3.34%S to form FeS is 25.94 - 3.34 = 22.60%Fe equivelent to this Sulphur as FeS $\frac{56}{32} \times 22.60 = 39.46\%$

FeS present equals 62.06%

The amount of FeO present is determined by assuming it is in the cratic 40 to the silica present, that is, that it is in the form of slag pellets.

Silica present equals .63%

Feo present equals $40 \times .63 = .84\%$

This is equivalent to .65% iron and .19% Oxygen. The remainder of the iron exists as Fe_3O_4 and metallic Fe. The amount of oxygen in the Fe_3O_4 is obtained by subtracting from 100% the total percentage as found by analysis (95.11%) and the percent oxygen in the FeO(.19%). This leaves : .70% as the amount of oxygen combined with

iron in the form of Fe_30_4 .

Fe in Fe₃0₄ that is equivalent to 4.70% 0 = 12.34% . This gives 17.04% of Fe₃0₄.

The remaining iron is the metallic iron and its per cent is found by subtracting from the total Fe, the emount present as FeS, FeO, and Fe₃O₄. Total Fe equals 55.32%

Metallic Fe equals 55.32 - (39.46 .65 12.34) = 2.87%The Alg O3,Ca O and Si O₂ are assumed to exist in

that form.

<u>Pyrite.The pyrite contains 1.25%</u> Cu which is assumed to exist as Cu₂S. The remaining S is considered to be in

the form of FeS and FeS_2 , the amount of each being calculated as follows:

S as FeS and Fe $S_2 = total S - S$ as Cu₂S.

Total S equals 45.11%.

S as Cy S equals.32%.

S as FeS and $\text{FeS}_2 = 45.11 - .32 = 44.79\%$. Fe as Fes and $\text{FeS}_2 = \text{total}$ Fe = 40.62\%.

Now Let X = % Fes, then <u>32</u>. X = % S as FeS.

" $Y = \% FeS_2$, " $\frac{64}{120}$ Y = % S as Fe S₂.

Also the % FeS + % FeS₂ = % Fe + % S as FeS and FeS₂.

(1) S as FeS and FeS₂ = $44.79 = \frac{32}{88} \times \frac{64}{120} \times \frac{64}{120$

From equations (1) and (2)

% FeS = X = 4.51% $\% FeS_2 = Y = 80.90\%$

The Alg03 and Si 02 are assumed to exist in that form. The remaining .83% unaccounted for by analysis is assumed to be water of crystallization in the aluminum silicate. QuartzThe SiQ, FeO and AlgOz are assumed to exist in that form. The remaining 1.40% unaccounted for by the analysis is designated in the table No. XII as X. It is assumed to be slag-forming material.

<u>limestone</u>. The CaO, MgO, FeO, and SiO₂ are recorded as obtained by analysis. The 44.15% unaccounted for is assumed to be CO.

<u>Coke</u>. The proximate analysis based on dry coke gave 77.02% fixed carbon and 16.22% ash. The sulphur in the latter probably exists as SO_3 (.61%). The SO_3 is volatile at the furnace temperature leaving 15.61% of slag-forming material. The 6.76% unaccounted for is the volatile matter in the coke. The moisture accompanying the 7.91 Kgs. of coke, as obtained by analysis, is .73 Kgs.

Calculation of Blast.

There are two methods of calculation of the blast, one using the flue gas analysis and the amount of carbon per charge, the other by means of Pitot tube measurements of velocity head and static pressure head.

Method Using Gas Analysis.

The volume of flue gas is first calculated by dividing the total weight of carbon in one charge by the weight of carbon (as CO and CO₂) in $1-\frac{3}{m}$ of gas. Average Gas Analysis.

CO2, 19.85%; CO, 2.60%; SO2, .25%; 0,.50%; N,76.80%.

C in 1 m of flue gas = (% of CO2 in flue gas % CO in flue gas) x (Wt. of C in 1 m³ of CO or CO2 under stendard conditions). Wt. of C in 1 m ³ of CO or CO gunder standard conditions = .54 Kg. " " " flue gas = (.1985 .026) x .54 = .12 Kg. Carbon entering gas from coke = 6.09 Kgs. (See Table XII.) limestone = $\frac{12}{44}$ x 5.64 = 1.54Kgs. Total carbon in gas = C from coke + C from limestone. " = 6.09 + 1.54 = 7.63 Kgs. Volume of flue gas per charge at 0°C and 760 mm. $=\frac{7.63}{.12}$ = 63.58 m³ It was assumed that all the nitrogen in the flue gases was that introduced by the blast. Volume of N = %N in flue gas x volume of flue gas. Volume of N = .768 x 63.58 = 48.83 m^3 . Volume of air = $\frac{\text{volume of N}}{\frac{7}{5} \text{ of N} \text{ in air } x 100.}$ % of N in air = 79.2 % by volume. Volume of air to furnish 48.83 m³ N equals m³ dry air at 0 and 760 mm. $\frac{48.83}{79.2} \times 100 = 61.65$ Wt. of 1 m³ dry air at 0° and 760 mm. = 1.293 Kgs. "61.65"" " = 79.71 Conditions at time of run. Atmospheric temp. = 69° F = $20^{\circ}_{.5}$ С Barometric pressure = 29.90 "= approx. standard. 61.65 m³ dry air at 0° and 760 mm = 293.5 x 61.65 = 66.28 m^3 dry air at 20.5 C and 760 mm_{\bullet}^{270}

The moisture accompanying this air is calculated as follows:

Hygrometer readings gave 3.6 grains water to cubic foot air.

3.6 grains per cubic foot equals 8.27 grams per m³. Wt.H₂O in blast per charge = Vol. of blast x Kgs. H₂O per m³ = $\frac{66.28 \times 8.27}{1000}$ = .55 Kgs. H₂O.

Method using Pitot Tube measurements.

The volume of the blast was calculated from the Pitot tube measurements as follows:

Vol. of air percharge = (Vel. per Sec.) x (A.of cross section of pipe) x (of one charge in passing through furnace in seconds).

The velocity is calculated by the formula $V = \sqrt{2}$ gh where <u>h</u> is the velocity head in terms of air.

Velocity head = .61" water.

Water is 772 x as heavy as air at 0° and 760 mm.

Therefore velocity head $= .61 \times 772 = 470.9$ " air = 39.24' air.

Substituting in the formula

 $V = \sqrt{64 \times (42.55)} = 52.08'$ per sec.

Area of cross section of blast main = .195 sq. ft.

Rate of charging equals one charge in 21 minutes or 1,260 sec. Static pressure head = 6.97" water = 1/4 lb. per sq. inch.

Therefore Vol. of air per charge $= 52.08 \times .196 \times 1260$

= 12865 cu.ft. air at 20.5 end 1/4 lb.

12865 cu. ft. air at 20.5 and 1/4 lb. = 364 m^3 at 20.5 & 1/4# 364 m^3 " " " " " = 344 " air at 0° and 760 mm.

NOTE. There is a great difference between the volumes of air obtained by the two methods. This is due either to faulty gas samples or to the lack of calibration of the Pitot tubes or to both. The volume calculated from the gas analysis was used in the calculations, and not that obtained from the Pitot tube measurements, as the gas analysis had to enter the calculation of the heat balance.

The materials are distributed under the heads of Matte, Slag and Gas. The copper, nickel, and lead are all figured as entering the matte, the small amounts in the slag are assumed to exist there as matte particles. The weight of matte produced from one charge was calculated from the weight of copper in the charge and the per cent of copper in the matte produced. The weights of iron and sulphur entering the matte were calculated from the analysis of the matte formed. All solid substances not entering the matte were assumed to form slag and the remaining volatile and combustible matter to enter the gas.

Wt. matte produced per charge = wt. Cu per charge x 100.
% Cu in matte
Wt. Cu per charge = 3.55 Kgs.
% Cu in mette = 8.73%.

Matte produced per charge $= \frac{3.55}{8.73}$ x 100 = 40.67 Kgs.

The sulphur is assigned as follows:

The Cu S, Ni S , Pbs, and FeS (amounting to 2 3 2 27.62 Kgs), were assigned to the matte. These contained 9.20 Kgs. sulphur. The sulphur in the matte produced is 25.25% (by analysis). Therefore, the 40.67 Kgs. of matte contain 10.68 Kgs. sulphur. The amount of S still to be furnished = 10.68 - 9.20 = 1.48 Kgs. This is equivalent to 3.67 Kgs. FeS which must be furnished by the FeS₂. The remaining sulphur is assigned to the gas. The iron is assigned as follows:

The total iron entering the mate as indicated above equals 16.30 Kgs.

The matte produced contains both metallic iron and magnetic oxide of iron. The amount of the latter was not determined but is estimated to correspond to 3% of oxygen. Total wt. of sulphides = 31.29 Kgs. The weight Fe and Fe₃ 0_4 =40.67- 31.29 = 9.38 Kgs. Wt. 0 in matte = .03 x 40.67 = 1.22 Kgs. This is equivalent to 4.42 Kgs. Fe₃ 0_4 . Wt. metallic Fe = 9.38 - 4.42 = 4.96 Kgs. The Fe₃ 0_4 is assumed to be supplied by the Fe₃ 0_4 in the matte charged. The remainder of the latter Fe₃ 0_4 is assumed to be reduced to Fe0 and 0 and to enter the slag and gas respectively. The metallic iron (4.96Kgs.) is assumed to c come from the FeS_2 . Of the FeS_2 , 8.63 Kgs. (4.96 Kgs. as Fe and 3.67 Kgs. asFeS) enter the matte,. The Fe (5.30 Kgs) is assigned to the slag and remaining S (12.74 Kgs.) to the gas.

The assignment of the remaining materials requires no comment except that of the I.76 Kgs. of O from the blast which is assigned to the slag. It is the O required to oxidize the metallic iron from the matte charged and the iron which enters theslag from the FeS₂.

THEORETICAL BALANCE SHEET OF MATERIALS.

TABLE No.XII.

DEBIT			CRED	IT	
	20	Wt. kg.	Matte	Slag	Gas
Matte (32.97 kg.)				
Cu ₂ S	11.95	3.94	3.94		
Ni3Sg	3.21	1.05	1.05		
PbS	0.49	0.16	0.16		
FeS	62.06	20.46	20.46		
FeO	0.84	0.28		0.28	
Fe ₃ 04	17.04	5.62	4.42	1.11	0.09
Fe	2.87	0.95	•	0.95	
A1203	0.45	0.15		0.15	
SiO2	0.63	0.21		0.21	÷
CaO	0.46	0.15		0.15	
Pyrite (32.97 kg	.)				
Cu ₂ S	1.57	0.52	0.52		
FeS	4.51	1.49	1.49		•
FeS2	80.90	26.67	8.63	5.30	12.74
A1203	2.07	0.68		0.68	
Sio2	10.12	3.34		3.34	
H20	0.83	0.27			0.27
Total Weight.		65.94	40.67	12.17	13.10

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DEB		CRI	EDIT		
Quartz (21.29kg.)	%	Wt.kg.	Matte	Slag	Gas
Si02	96.61	20.57		20.57	
FeO	1.50	0.32		0.32	
A1203	0.49	0.10		0.10	
X	1.40	0.30		0.30	
Limestone (12.77)					
CaO	54.16	6.92		6.92	•
MgO	1.13	0.14		0.14	·
Fe 0	0.35	0.04		0.04	
Si02	0.21	0.03		0.03	
CO2	44.15	5.64			5.64
Coke (dry) (7.91)	an di Santa A				. 1
Fixed C	77.02	6.09	· ·	۰ ۱	6.09
Ash	15.61	1.24	· · · ·	1.24	
SO3	0.61	0.05			0.05
Volatile Matter	6.76	0.53	•		0.53
Moisture (0.07)	 	0.07			0.07
31ast (dry) (79.7]	L)			•	
N	76.90	61.30			61.30
0	23.10	18.41	•	1.76	16.65
H ₂ O (0.55 kg.)		0.55	•		0.55
Total	с. т. 2	122.30	00.00	31.42	90.88
Wts. from page 36 TOTAL WEIGHTS	•	65.94 188.24	<u>40.67</u> 40.67	<u>12.17</u> 43.59	<u>13.10</u> 103.98

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THERMAL BALANCE.

The figures used for heats of formation, specific heats, etc. are those given by J.W.Richards in his "Metallurgical Calculations". Those marked "approx". do not apply directly to the materials in this case. But since determinations of specific heats, heats of fusion, etc. on the different materials were not practicable, figures were selected which seemed to apply most nearly to each case.

Heat Generated.

- (I) Oxidation C to CO₂ (8100 Cal. per Kgs. of C)
 - C burned to $CO_2 = (Vol. of gas X \% CO_2 X Wt. C in 1$ m³ CO₂) - (Wt. C in CO₂ from lmste)

 $63.58 \times .1985 \times .54 = 6.83 \text{ Kgs}.$

6.83 - 1.54 = 5.29 Kgs.

 $5.29 \times 8100 = 44,793$ Cal.

(2) Oxidation C to CO. (2430 Cal. per Kg. C.)

C burned to CO = Total C in coke - C burned to CO_2 . 6.09 -5.29 = .80 Kg.

 $.80 \times 2430 = 1944$ Cal.

- (3) Oxidation S to SO₂ (2164 Cal. per Kg. S)
 - % SO₂ in gas x m³ of gas = m³ of SO₂. (Gas analysis) Wt. of l m³ H x Density of SO₂ x $\frac{S}{SO_2}$ = Wt. S in l m³

of S02.

.0025 x 63.58 =.159 m³ of SO₂

.159 (.0896 x 32 x $\underline{32}$) = .227 Kgs. S burned. This does not agree with the of S assigned to gas in the theoretical balance sheet. The difference is assumed

- to have been distilled off as free sulphur. .227 x 2164 = 491 Cal.
 - (4) Oxidation of Fe to FeO. (1173 Cal. per Kg. Fe.)
 Wt. Fe oxidized = .95 5.30 = 6.25 Kgs. (See Table No. XII)

 $6.25 \times 1173 = 7331$ Cal.

- (5) Heat of Formation of Matte.All copper and iron exist as sulphides in the ore.The heat of union of FeS andCu₂S is unknown.
- (6) Heat of Formation of Slag. (150 Cal. per Kg. approx.)
 Wt. slag made = 43,59 Kgs.

 $43.59 \times 150 = 6538$ Cal.

(7) Sensible Heat in Blast at room temp. (20.5) (Sp. Heat .25 approx.)

Vol. x Sp. Ht. x Temp. = 66.28 x .3036 x 20.5. = 413 Cal.

(8) SensibleHeat in Charge at room temp. (20.5)

(Sp. Heat = .25 approx.)

Wt. xSp. Ht. x Temp. = 107.91 x .25 x 20.5 = 553 Cal.

HEAT ABSORBED.

- (a) Reduction of Fe₃O₄ to FeO. (1023 Cal. per Kg. FeO.)
 Wt. FeO from Fe₃O₄ = 1.11Kgs.
 1.11 x1023 =1135 Cal.
 - (b) Reduction FeS₂ to Fe (428 Cal. per Kg. Fe Approx.) The heat of reduction of FeS₂ to Fe is not known. Since it probably is not much larger than FeS to Fe,

the latter figure is used.

Wt. Fe from FeS₂ = 10.26 Kgs. (See distribution of matte constituents, page 34.)

 $10.26 \times 428 = 4391$ Cal.

- #
- (c) Decomposition of Limestone. (1026 Cal. per Kg. CO₂)
 # Figure for CaCO₃ to CO₂ used since the amount of MgCO₃ is small.

Wt. CO_2 from $CaCO_3 = 5.64$ Kgs.

 $5.64 \times 1026 = 5787$ Cal.

(d) Heat in Slag (Temp. = 1130 C.) Heat in melted slag at 1100 = 300 Cal. per Kg. (approx.) 43.59 x 300 = 13077 Cal. To heat from 1100 to 1130 C. (Sp. Ht. = .27 approx.) 43.59 x .27 x 30 = 353 Cal.

Total Heat = 13077 + 353 = 13430 Call.

(e) Heat in Matte. (Temp. = 1130 C)
Heat in melted matte at 1000 = 200 Cal.(approx.)
40.67 x 200 = 8134 Cal.
To heat from 1000 to 1130 (Sp. Ht. = .285 approx.)
40.67 x .285 x 130 = 1507 Cal.

Total Heat = 8134 + 1507 = 9641 Cal. (f) Heat in Gases. (212 F = 100 C.)

(1) CO + N + O = 79.90%.

(These gases have same specific heat.)

 $.7990 \times 63.58 = 50.80 \text{ m}^3$

Sp. Ht. per $m^3 = (.303 + .000027t)$

Heat = $50.80 \times 100 (.303 + .000027 \times 100) =$

1553 CAL.

Debit, Kg. Calories.	. Credit, Kg. Calories.
Burning C to CO ₂ - 44,793	Reduction Fe ₃ 0 ₄ to FeO - 1,135
Burning C to CO 1,944	Reduction FeS ₂ to Fe 4,391
Burning S to SO ₂ - 491	Decomposition of $CaCO_3 - 5,787$
Burning Fe to FeO - 7,331	Heat in Slag 13,430
Formation of Slag - 6,538	Heat in Matte 9,641
Sensible Heat in Blast 413	Heat in Gases 3,162
Sensible Heat in Charge553	Heat in Cooling Water <u>11,613</u>
Formation of Matte(?)	49,159 Radiation(by Difference) <u>12,904</u>
62,063	62,063

XIII. THERMAL BALANCE FOR ONE CHARGE.

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SUMMARY.

The sulphur elimination obtained in this run was 56.38 per cent of the sulphur present, instead of 79.64% as expected. If the analysis of the flue gas is correct about 98% of the sulphur eliminated was driven off in the form of sulphur vapor and not as sulphur dioxide. The sample of gas must have been faulty, as the concentration obtained, --1.6 into 1 -- gives a greater pyritic effect although it falls below the calculated concentration of 4.4 into 1.

The thermal balance shows that the larger part of the heat in the furnace was furnished by the coke and not by the oxidation of the iron and sulphur as should have been the case.

The slag formed contained 32.37 % FeO instead of the calculated 51.2%, the silica contents was 41.98% instead of the calculated 36.08%. The percentage of lime ran much higher than was intended. These facts show that sufficient silica was present but that the iron was not oxidized and therefore could not enter the slag but went into the matte. Assuming the air supply to be sufficient, the coke must have prevented the desired pytitic effect.

A decrease in the amount of coke used would therefore correct the smelting conditions.