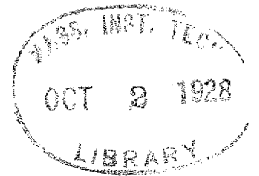


Chem. Eng. g
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thesis
1928



A STUDY OF THE ROASTING OF PYRITES ORE
IN A HERRESHOFF FURNACE

A THESIS
SUBMITTED TO THE FACULTY
OF THE
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE
DEGREE OF BACHELOR OF SCIENCE

Submitted by:

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Professor A.L.Merrill
Secretary of the Faculty
Massachusetts Institute of Technology

Dear Sir:

We present herewith a thesis, "A Study of the Roasting of Pyrites Ore in a Herreshoff Furnace", in partial fulfillment of the requirements for the degree of Bachelor of Science.

Very respectfully yours,

Joseph A. Jamison

Benjamin F. Miller

Edmund L. Welcyng

In appreciation to Charles M. Cooper
of the Institute, under whose direc-
tion this thesis was done; and to Messrs.
C. Dean and J. Clark of the Merrimac Chem-
ical Company for their kind assistance.

THESIS

Chas. B. ...

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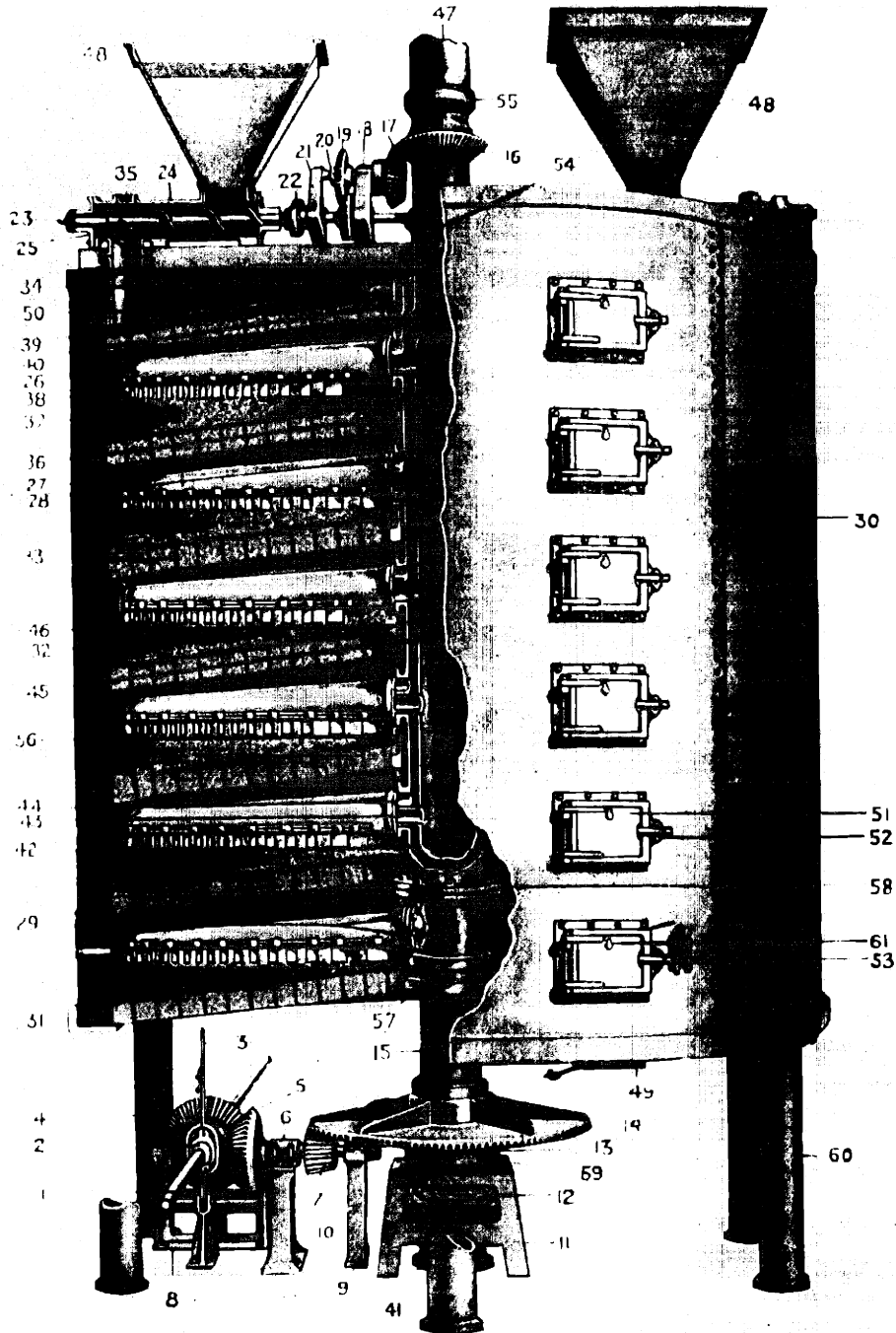
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Introduction.

The contact sulphuric acid plant of the Merrimac Chemical Company at South Wilmington, Massachusetts manufactures sulfuric acid and oleum of market strengths. The sulfur dioxide used in making the acid is produced by roasting pyrites in a Herreshoff furnace. This method has only recently been adopted; rotary sulphur burners having been previously employed. The pyrites are by-product fines from the Canadian copper flotation industry.

Because this equipment is new to both the company and the operators, it has been the object of this thesis to gather all possible information and data concerning the roasting of the particular ore used. It is hoped that these data and results will help solve the present and future difficulties in the operation of Herreshoff furnaces.



Herrshoff, mechanical roasting furnace.

The Herreshoff Roasting Furnace.

1. Main driving shaft.
2. Starting clutch.
3. Starting lever.
4. Main driving gear.
5. Transmission gear.
6. Pillow block.
7. Pinion wheel.
8. Floor stand.
9. Pedestal.
10. Pillow block or pedestal.
11. Main shaft floor stand.
12. Stuffing box and gland.
13. Ball bearing collar.
14. Driving rack or spur wheel.
15. Main revolving shaft.
- 16 21. Feed drive.
22. Flange coupling.
23. Worm gear bearing.
24. Worm gear housing.
25. Worm feed.
26. Rabble arm or rake arm.
27. Arm flange shaft.
28. Shaft flange for arm.
29. Flange bolts.
30. Boiler plate casing.
31. Base flange or base plate ring.
32. Red brick.
33. Fire brick.
34. Outer drop hole or feed spout.
35. Poke hole.
36. Inner drop hole shelves, 2-4 and 6.
37. Cinder bed.
38. Removable blades or plows.
39. 40. Seal lutes.
41. Cool air intake.
42. Central hollow shaft.

(continued)

The Herreshoff Roasting Furnace (continued)

43. Cool air tube-out.
44. Warm air tube-return.
45. Annular air chamber.
46. Outer shell of central shaft.
47. Hot air stack.
48. Feed hopper.
49. Cinder outlet with damper.
50. Sulphur-dioxide chamber.
51. Inspection door, brick lined.
52. Door latch.
53. Combustion air inlet with adjustable slide.
54. Top bearing.
55. Stack seal (lute).
56. Shelf seal (lute) (3) and (5).
57. Bottom seal (lute) (1).
58. Flange connecting outer and inner shaft.
59. Ball bearing (bearing plate).
60. Columns.
61. Peep hole in inspection door.

N.B. The above explanatory notes and the accompanying photograph of the Herreshoff Roasting Furnace taken from "Pyrites in Canada" (1912) Mines Branch, No. 167. (Canadian Government Publication).

Measurements:

Outside diameter:16 feet.
Height:29 feet.
Hearth area:1710 sq.feet
Number of hearths:12.

Operation of the Herreshoff Furnace.

The accompanying diagram shows a six hearth Herreshoff Furnace of an earlier type than the one in use at the Merrimac Chemical Company. The work of this thesis was conducted on a twelve hearth furnace which differs from the one shown in the diagram only in the method of feed. Instead of a screw conveyor, a preheating hearth is used. The ore is rabbled around the roof of the furnace exactly as on the hearths below in order to dry and preheat the feed. This ore drops thru a luted feed hole onto the outside of number one (top) hearth; and is rabbled toward the central shaft where it drops onto number two hearth inside; and so on ~~it~~ travels over the twelve hearths. The air for combustion is admitted on the lowest hearth, so that the path of the gases is always upward thru the furnace. The burner gases are taken from number one hearth by suction, and are sent thru the centrifugal dust collector; thence to the cooling and cleaning equipment of the contact plant.

The combustion within the furnace is entirely self-supporting. The cold ore is brought up to the temperature of combustion by heat interchange between it and the hot gases rising from the hearths below. This temperature is reached on the first hearth. Lower

down in the furnace & heat interchange of a reverse nature proceeds. Here the hot ore gives up heat to the cold gases rising from the lowest hearths.

The rabble arms are cooled by air which is blown up the central shaft and circulated thru the arms as shown in the illustration.

Pyrometers, placed in 1-1/2 inch iron wells, extending 18 inches into the hearth and 19 inches above the bed, are installed on each hearth. By noting the recorded temperature the operator can observe whether the furnace is properly functioning. However, this does not give him any information as to the quality and amount of gas being produced. The amount of gas can be controlled by means of a Connersville blower and a by-pass. The quality of the gas is determined by gas analysis.

Summarized Method of Procedure.

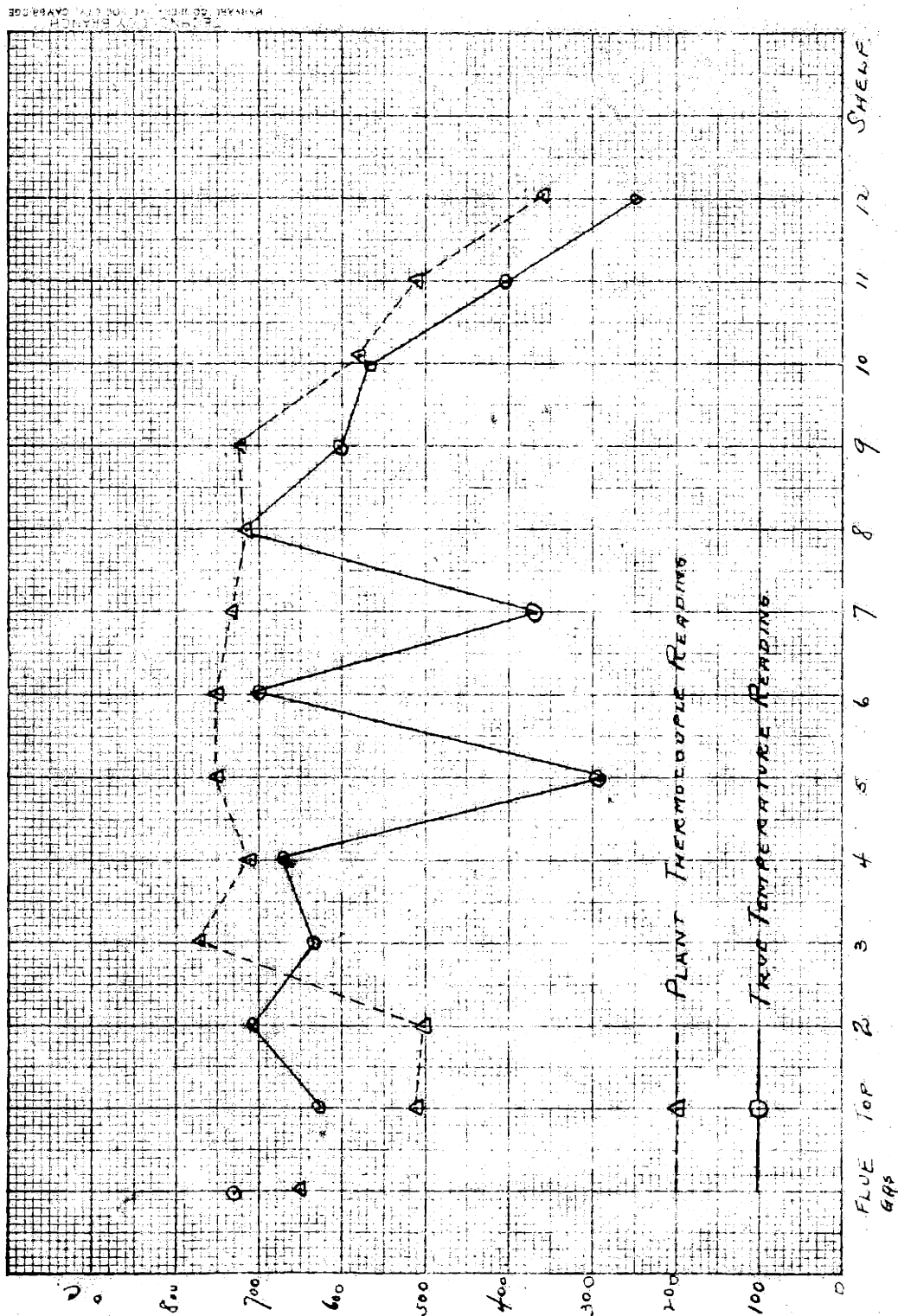
The work of this thesis can be divided into three parts: (I) Calibration of the Furnace Pyrometers; (II) Heat and Material Balance on the Herreshoff Furnace; and (III) A Hearth Study of the Furnace.

n For the calibration of the furnace pyrometers gas temperatures were measured by means of a true temperature thermocouple, and also by the use of a bare thermocouple. The bed temperatures were obtained by a bare thermocouple, and also by an optical pyrometer.

The Heat and Material balance data were obtained over a test run of 6.3 hours. The feed was weighed; and analyzed for sulfur, iron and moisture. The burner gases were analyzed for SO_2 , and O_2 ; its volume was computed by a sulfurbalance. The cinder and dust were weighed; the former analyzed for sulfur and iron. The amount of cooling air was measured with a Pitot tube. Temperatures were taken from the Brown Recording Pyrometers or with mercury thermometers.

The Hearth Study consisted of sampling and analyzing the incoming and outgoing ore on each of the hearths.

RESULTS.



COMPARISON OF TRUE AND PYROMETER TEMPERATURES

FIGURE 1.

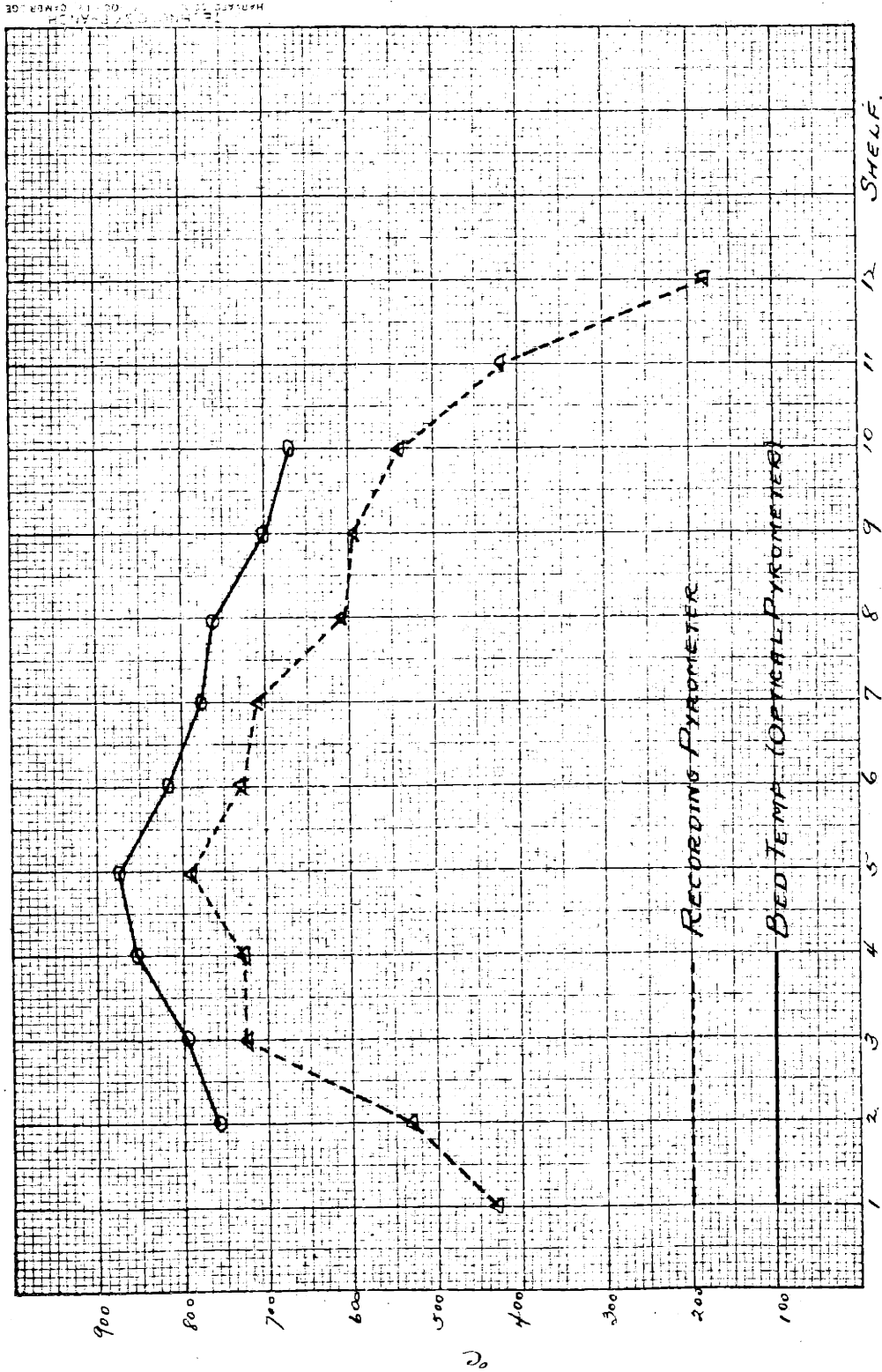


FIGURE 2.
 COMPARISON OF OPTICAL PYROMETER READINGS WITH RECORDING PYROMETERS.

Results

Flow of Materials, Basis: 1 hour

Input:

- a. 2424 lbs bone dry ore
 - 1070 lbs sulfur
 - 960 lbs iron
 - 390 lbs gangue
 - 12.3 lbs moisture
- b. 104,000 cu.ft. (8,400 lbs) of air at standard conditions for combustion
- c. 106,000 cu.ft. (8,550 lbs) of cooling air at standard conditions

Output:

- a. 1527 lbs cinder
 - 30.5 lbs sulfur
 - 805 lbs iron
 - 348 lbs gangue
- b. 62.5 lbs dust
 - 1.3 lbs sulfur
 - 33 lbs iron
 - 14 lbs gangue
- c. 100,400 cu.ft. burner gases (standard conditions)
 - 11,700 cu.ft. SO₂
 - 6,900 do O₂
 - 81,800 do N₂
 - 247 do water vapor
- d. 106,000 cu.ft. of cooling air at standard conditions.

Results: Material Balance

Sulfur Balance,

Input

Feed 1070 lbs

Output

Cinders 30.5
Gas (diff.) 1039.5

1070 lbs

Iron Balance,

Input

Feed 960 lbs

Output

Cinders 805
Dust 33
Unaccounted for 122

960 lbs

% unaccounted for: 12.7

Gangue Balance,

Input

Feed 390 lbs

Output

Cinders 348
Dust 14
Unaccounted for 28

390 lbs

% unaccounted for: 7.2

Results, Heat Balance

Heat of Combustion of Ore:

2430 B.t.u. per lb.

Heat Balance: Basis: 1 hour
Datum line 68° F.

Input:

Feed	5,880,000 B.t.u.	100%
------	------------------	------

Output:

a. Sensible Heat

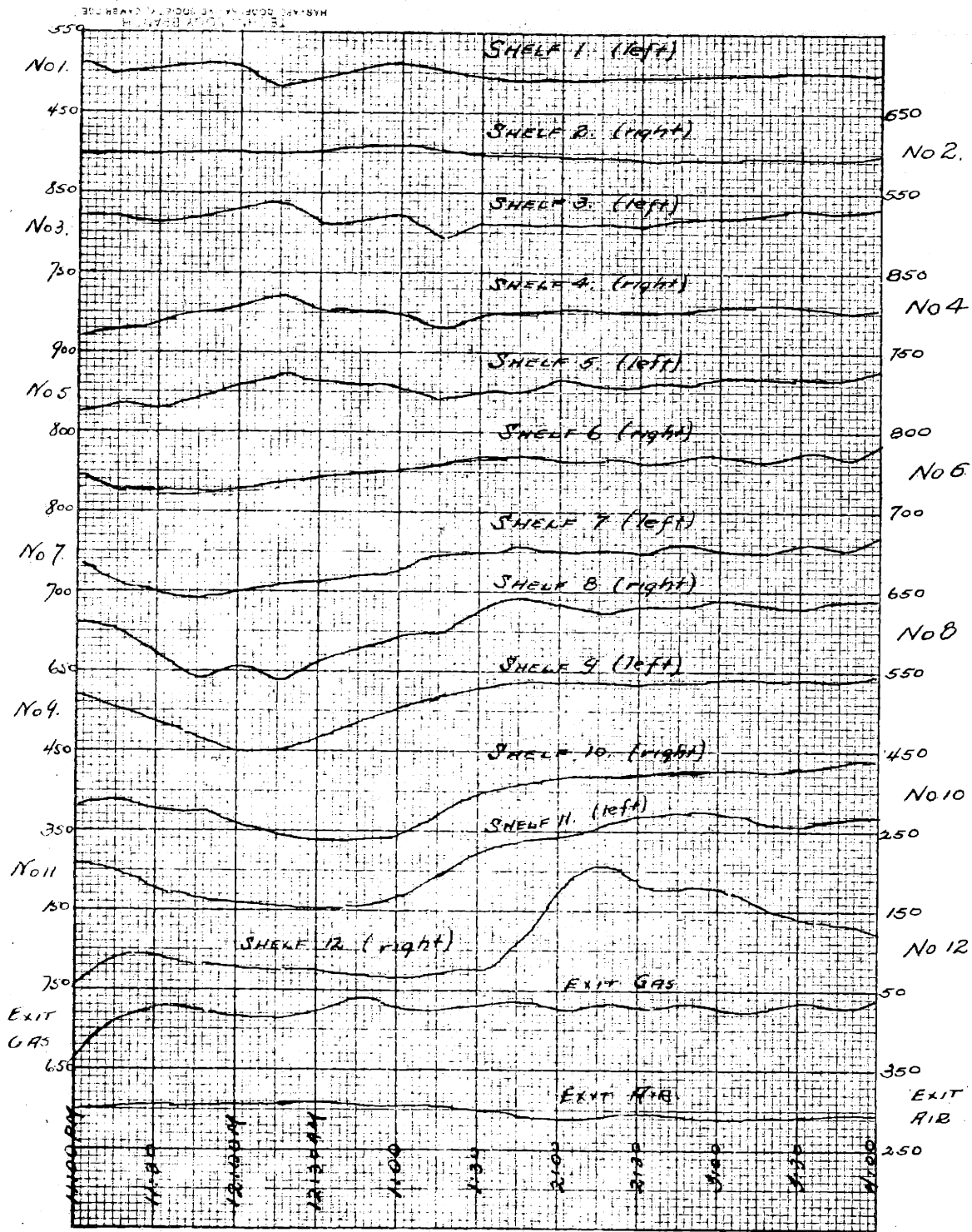
Burner gases	3,059,000 B.t.u.	52.1% of input
Cooling air	1,070,000	18.2
In cinders	115,000	2.0
In dust	13,400	0.23
In ore	10,500	0.18

b. Unburnt ore in cinder	167,000	2.8
--------------------------	---------	-----

c. Heat to vaporize water on preheating hearth	53,700	0.92
--	--------	------

d. Radiation and unaccounted for	<u>1,392,000</u>	<u>23.6</u>
----------------------------------	------------------	-------------

Totals	5,880,000 B.t.u.	100 %
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TEMPERATURES ON RECORDING PYROMETERS (°C)
FIGURE 3.

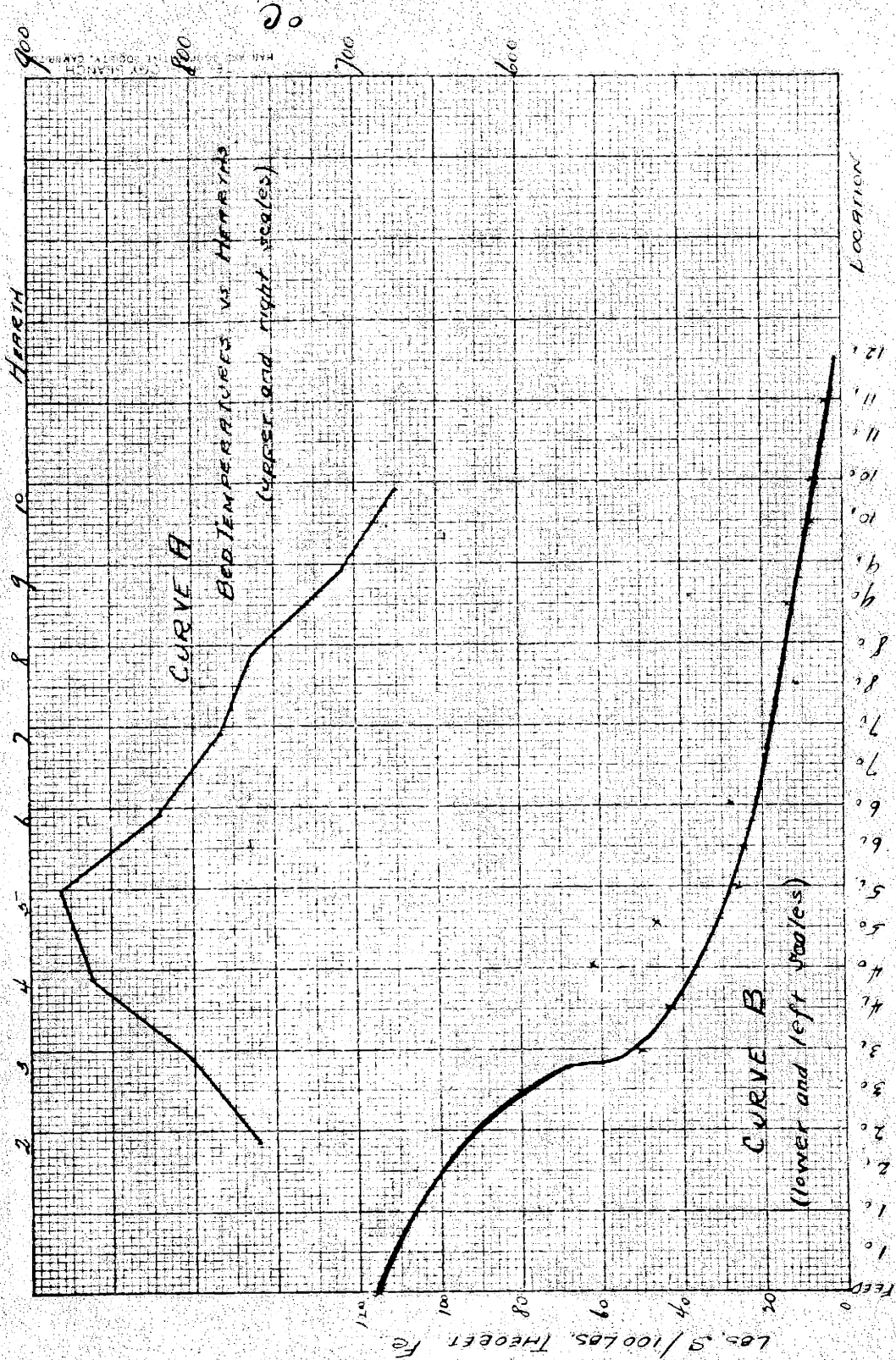


FIGURE 4 HEARTH STUDY

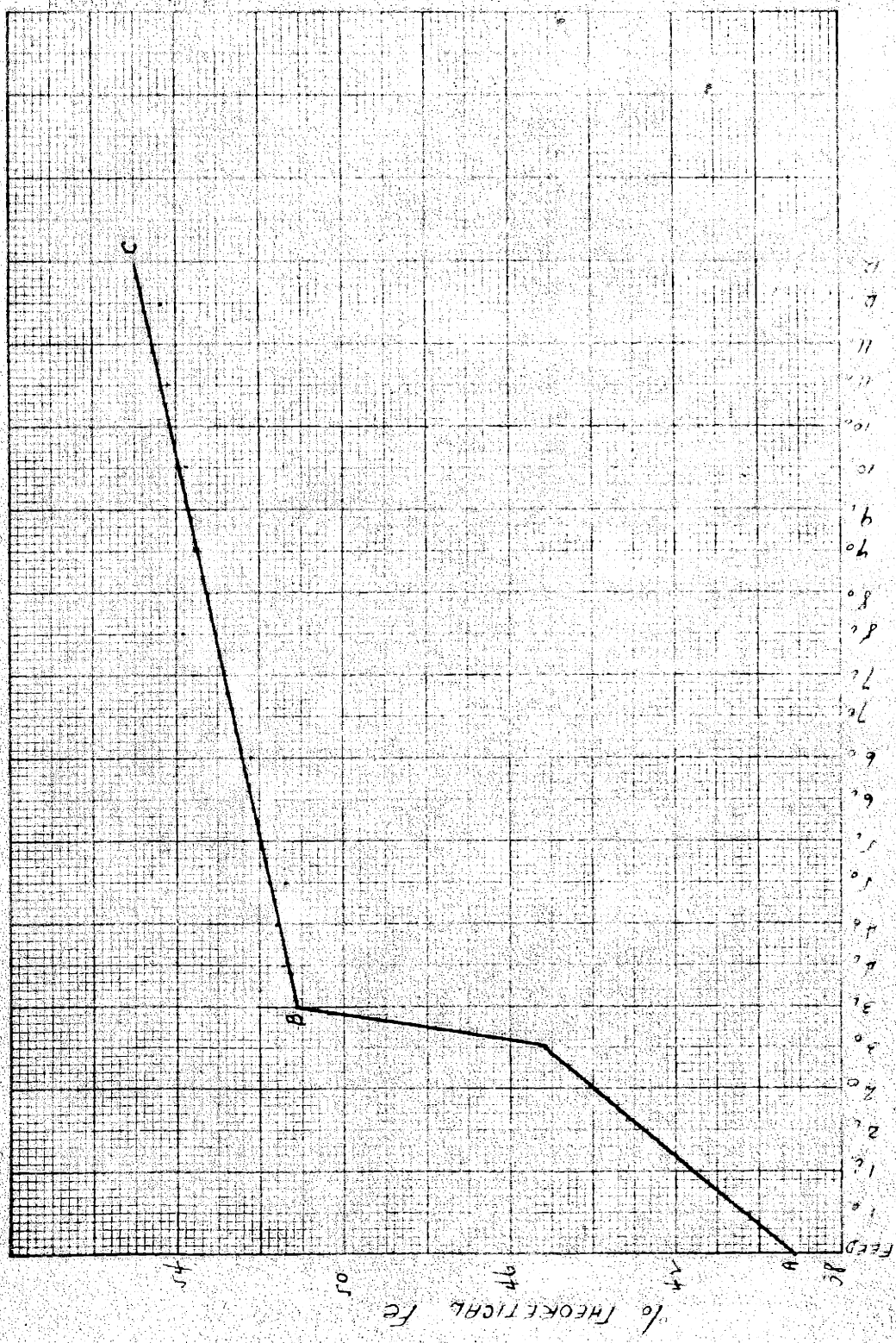


FIGURE 5. RATE OF FE CONTENT INCREASE

Discussion of Results

I. Calibration of Furnace Pyrometers.

It can be seen from an inspection of Figures 1 and 2 that the furnace pyrometers, in general, record a temperature between the bed temperature and the true gas temperature. The pyrometer temperatures are lower than the bed temperatures on all hearths, as shown in Figure 2. Figure 1 shows that the plant thermocouples read higher than the true gas temperatures except in the flue and on hearths one and two. This is due to the fact that, in the flue, the walls are colder than the main body of the gas since the flue is not insulated and is exposed to the outside air. This means that the thermocouple-well radiates heat to the cooler surrounding walls, and, hence, reads below the true temperature. A similar case exists on the first two hearths where the cold feed is at a lower temperature than the hot gas rising from the hearths below, so that the thermocouple-well in contact with the hot gas radiates heat to the cold ore; and thus reads a lower temperature than the true gas temperature, on these hearths. However, when the ore reaches hearth three, it is in active combustion and attains a temperature higher than the main body of gas above it. Hence, it begins to radiate heat to the thermocouple-well,

which consequently reads higher than the true temperature. This condition exists from hearth three to hearth 12.

On hearths five and seven (Figure I), the true gas temperature was surprisingly low in comparison with the temperatures shown by the recording pyrometers. On number five hearth this was due to excessive leakage of air thru the doors. Hearth seven was well luted at the time so that air leakage from the outside was at a minimum. Hence the low reading was probably due to a large air leak in the central shaft or in the rabble arms on number seven hearth.

Discussion of Results.

II. Material Balance.

The results show discrepancies in the iron and gangue balances. These, it is believed, can hardly be due to errors in procedure since all instruments were calibrated and weights and analyses were checked. There are two possibilities which might account for these discordant results. The first, that the furnace was not at equilibrium; the second, that ore was piling up on the hearths due to the breakage of rabble teeth. Since the test was started after the furnace had been observed to have been operating at constant conditions for six hours, and since the hearth temperatures, as shown in Figure 3, were reasonably constant, it is believed that the furnace was at equilibrium; and that the discrepancies in the balance are due to the fact that ore was building up on the hearths. This conclusion is well in accord with the fact that arms on hearths three and four had to be renewed on the following day. The conditions under which this test was run were the most constant obtained in two weeks of observation. During the time that the work on this thesis was being conducted the furnace was almost in daily repair.

Discussion of Results

III. Heat of Combustion of Ore.

The value for the heat of combustion of the ore was obtained by thermochemical computations based on its analysis. The value of 2430 B.t.u. per pound of ore checks well with that of 2340 B.t.u. per pound of ore obtained from Lunge's value for theoretical pyrites.

Discussion of Results

IV. Heat Balance.

The heat balance shows a considerable discrepancy which is much greater than is reasonable to expect from radiation losses. The two largest factors in the heat output are the sensible heat in the gases and the cooling air. The cooling air volume was measured directly; but the volume of burner gas was computed from a sulfur balance. An inspection of the gas analyses will show that there is too much oxygen present for a burner gas resulting from pyrites roasting. Because all precautions were taken in the method of analysis and because the accuracy of the determination of oxygen in pyrogallol acid can hardly be questioned, it is believed that the method of determining sulfur dioxide by chromic acid absorption gives high results. The furnace operator's analyses during the test showed even higher sulfur dioxide than our data. Thus, the accuracy of this method seems doubtful. It can be seen that a lower value for sulfur dioxide would compensate the heat balance on the output side since the largest item, the sensible heat in the gases, was obtained by a sulfur balance.

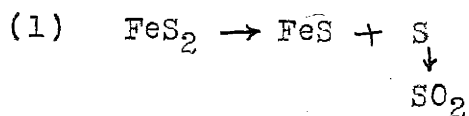
It is well to note that the percentages given for the heat in the cooling air, cinders, dust, etc. are based on the accurately determined heat input,

that is, they are stated as percentages of the total heat input rather than the total heat output; and, hence, stand correct since they are the result of direct measurements.

Discussion of Results

V. Hearth Study.

From Figures 4 and 5 it can be seen that the "flash" of the first atom of sulfur in the ore takes place on the first three hearths. Below that, the reaction $2 \text{FeS} + 7 \text{O} \rightarrow \text{Fe}_2\text{O}_3 + 2 \text{SO}_2$ proceeds. Figure 4 shows a rapid burning off of sulfur until the fourth hearth is reached, from then on the sulfur is burned off gradually. Figure 5 shows a rapid change in the iron content on the first three hearths. If we consider the reactions to be



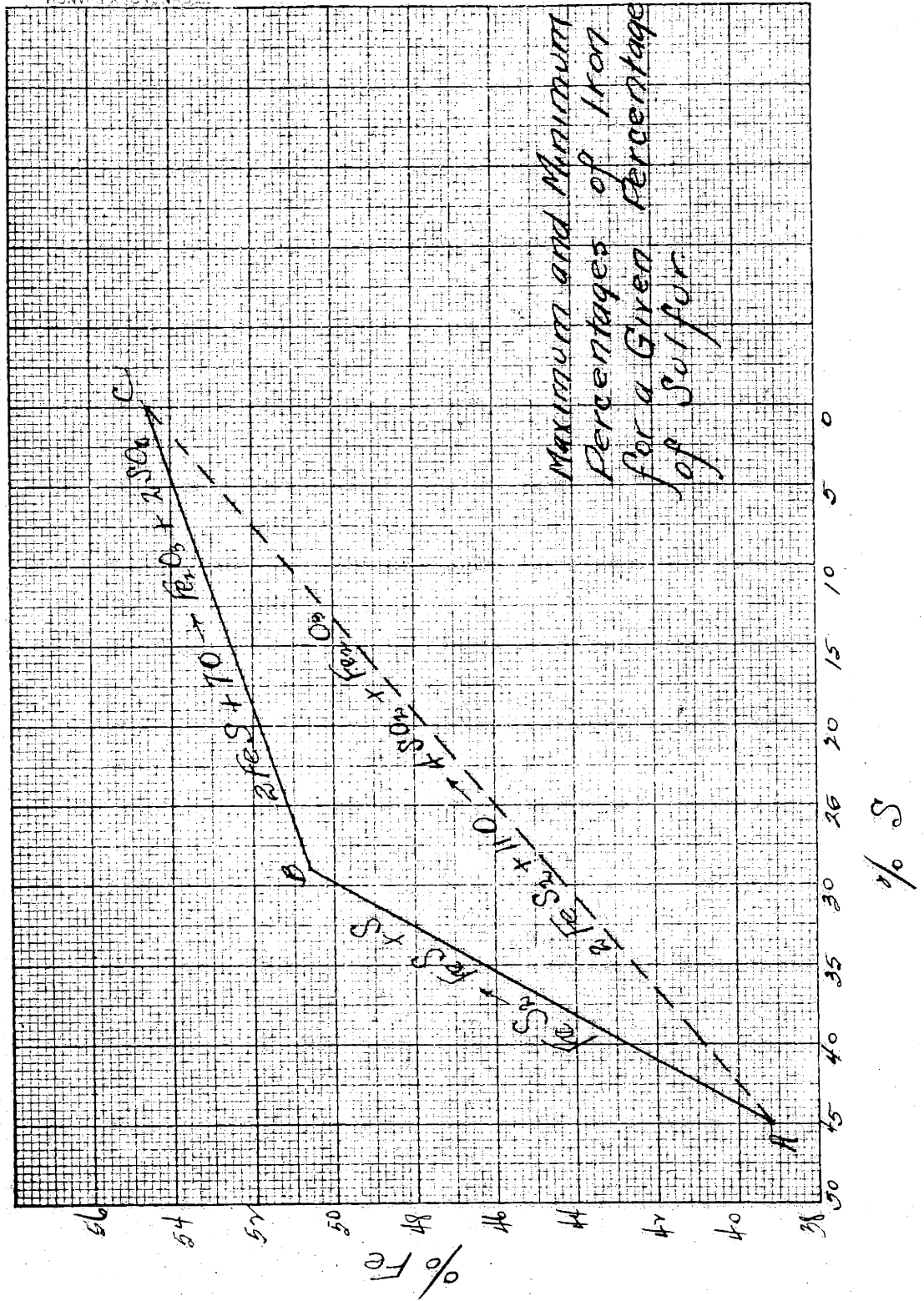
then it can be seen that while reaction (1) is proceeding alone the increase of iron content is rapid because sulfur is being burnt off, and no oxygen is added to the iron. This corresponds to the line AB of Figure 5. However, when this reaction is completed reaction (2) begins. No doubt reaction (2) proceeds simultaneously with reaction (1), but at a much slower rate so that its effect above hearth 4 is small in comparison with that of reaction (1).

The bed temperatures (plot A) as shown in Figure 4 are in accordance with the amount of sulfur combustion (curve B) on each hearth. That is, the greatest

rate of change in temperature comes between hearths three and four (curve A), whereas curve B shows that the largest amount of sulfur has been ~~burned~~ off on hearth three. Then, since the slope of curve B is gradual thereafter, this shows again that the "flash" must have occurred on the first three hearths.

It is interesting to note that curve B indicates that 71% of the sulfur has been burned off on the first five shelves. The curve also shows that all the other hearths are effective to a certain extent in burning off ~~of~~ sulfur.

Curve B of Figure 4 shows actual sulfur analyses on the basis of the iron that theoretically would have to be in the sample if the reaction proceeded according to the line ABC of Figure 6 (Appendix). That is, the values for iron are the maximum that could be obtained for any given sulfur analysis. This had to be done because the iron analyses given by the Zimmerman-Rheinhardt method were in many cases far above the maximum that calculation shows could have been obtained. The sulfur analyses were taken as correct, and the curves based on them.



Maximum and Minimum
 Percentages of Iron
 for a Given Percentage
 of Sulfur

FIGURE 6.

Conclusions

- I. That the recording pyrometers give a temperature between that of the actual bed and gas temperatures.

- II. That under present conditions of operation the "flash" of the first atom of sulfur in the ore occurs on the first three hearths.

Recommendation

That in further study of this furnace, observations be made of the effect of the variables given under "Expansion of Introduction".

APPENDIX.

Expansion of Introduction

I. Present operation of the furnace:

The furnace is roasting approximately thirty tons of ore per twenty-four hour day, which yields a gas containing 9 to 12 percent sulfur dioxide. The central shaft rotates at a speed of 1.6 revolution per minute. The cinder contains on the average two percent of unburnt sulfur.

II. Difficulties in operation:

The chief difficulty in operation is the control of hearth temperatures in order to prevent slagging of the ore. This is especially important because the slag builds up on the rabble arms and causes them to break off. Slagging on hearths three four and five has been especially troublesome in the operation of this furnace. The operators attempt to prevent slagging by keeping the temperature on these hearths as low as possible. If the temperature begins to rise above 800 °C. the shelves are cooled by opening ^{doors} ~~doors~~ and allowing cold air to enter. This cools the hearth, but is bad from the standpoint of operation because it interferes with the normal equilibrium which the furnace is designed to operate. The operation of the Herreshoff has been improved by speeding up the central shaft feed

to the present figure, and keeping the amount of feed constant. This means that the ore travels thru the furnace at a faster rate so that the amount of combustion on the upper hearths, where the roasting proceeds most rapidly, is reduced.

III. Possible Changes in Operation:

Operation may be changed by any of the following methods:

- (a) Change speed of central shaft.
- (b) Change amount of feed.
- (c) Use of preheated air for combustion.
- (d) Recirculation of burner gases within the furnace.

- (e) Change of the angle of throw of the rabble teeth.

- (a) The effect of changing the speed of the central shaft has been explained above under II.
- (b) A change in the amount of feed does not change the time of travel of the ore thru the furnace, but changes the amount of ore available for roasting on each hearth. If the feed be increased without other changes being made, it would most likely result in excessively high temperatures on the upper hearths and possibly a very high sulfur content in the cinder as a result of the low roasting capacity of the

and cannot be removed. The preheated air would still be useful in increasing the capacity of the furnace since more ore would be roasted if the temperature of the lower hearths could be raised to make them more effective than at present.

The Manufacture of Contact Sulfuric Acid.

Strong sulfuric acid and oleum are today produced almost entirely by the contact process. Weaker acids of strength 50 to 60 °Be' can still be made profitably by the older chamber process for use in the fertilizer industry. Chamber acid is seldom concentrated to obtain oil of vitriol since it cannot compete with contact acid in the making of stronger acids. The great advantage of the contact plant is in its ability to produce desired strength of acid without expensive evaporation.

Contact acid is obtained by absorbing sulfur trioxide in 98% sulfuric acid, and later diluting to the desired strength. The trioxide is formed by the oxidation of sulfur dioxide in converters containing a contact mass of platinized asbestos. The sulfur dioxide is produced by the combustion of sulfur in rotary burners, or by the roasting of pyrites in a Herreshoff or similar type furnace. The gas must be cleaned of all dust, mist, moisture and arsenic before being sent to the catalyzer so that the expensive platinum will not be poisoned.

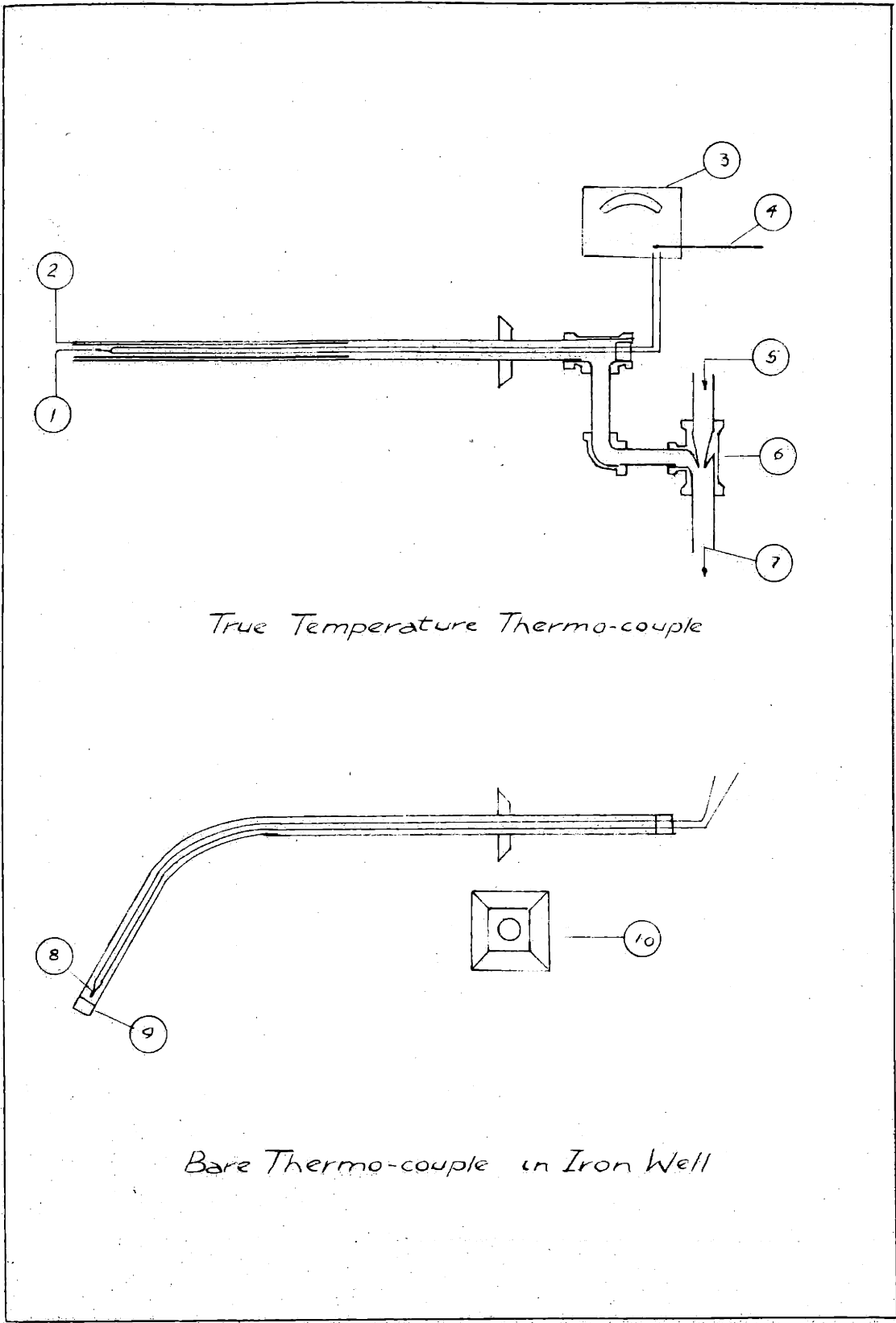
Method of Procedure.

I. Calibration of Furnace Pyrometers.

A. True-Temperature Measurements.

The true-temperature thermocouple was constructed as shown in the diagram. The couple was made of No. 14 (B & S) chromel-alumel wire; and was calibrated in an electric furnace against a standard couple within the temperature range expected in the furnace. A Weston millivoltmeter was used to measure the electromotive force set up in the couple. The true-temperature was obtained by drawing the hot gases, thru a 1/2 inch iron pipe which held the couple, at a velocity of 40 feet per second by means of a compressed air ejector. To eliminate the effect of radiation from the hot bed or walls, the couple was protected by a 3/8 inch pyrex tube which fitted tightly into the half-inch iron pipe. This thermocouple was used to measure the true temperature of the gases above the bed.

In making the measurements the true-temperature couple was placed 18 inches into the furnace thru the peep-hole in the door nearest the recording pyrometer. In order to prevent cold air from leaking in at the peep-hole, an air-tight



True Temperature Thermo-couple

Bare Thermo-couple in Iron Well

FIGURE 7.

Construction of Thermocouples.

- (1) No.14 (B & S) chromel-alumel couple.
- (2) 3/8 inch pyrex tube inside of 1/2 inch iron pipe.
- (3) Weston millivoltmeter.
- (4) Mercury thermometer at cold junction.
- (5) Compressed air.
- (6) Compressed air ejector.
- (7) Exhaust gases.
- (8) No. 14 (B&S) chromel-alumel couple.
- (9) 1/2 inch iron well.
- (10) Air-tight guard.

fitting was made. The hot gases were drawn past the couple until the millivoltmeter recorded a constant temperature. Readings were taken and compared with those of the Brown Recording Pyrometer. Readings were taken on each of the twelve hearths, and of the burner gases in the flue. Later, the compressed air was shut off and four readings were taken without the high gas velocity past the thermocouple.

B. Bare Couple Measurement of Gas Temperature.

These measurements were made by placing the same calibrated couple of No. 14 (B.&S.) chromel-alumel wire into a 1/2 inch iron well. The electromotive force set up was measured with a Weston millivoltmeter. The well was inserted 18 inches into the furnace thru the peep-hole in the door nearest the recording pyrometer. When placed in the furnace under these conditions, the bare couple approximated the position of the recording pyrometer. In this manner it was possible to obtain gas temperatures at number seven hearth.

C. Bed Temperature Measurements with Thermocouple.

These measurements were made by placing the same bare couple, described above, into the bed on the hearth thru the peep-hole of the door nearest the recording pyrometer. The reading on the Weston millivoltmeter and that of the corresponding recording pyrometer were then taken. Readings were thus secured on hearths seven and twelve.

II. Heat and Material Balance.

A. Feed.

The feed was measured by obtaining the time required for one complete revolution of the rake arm, and weighing the amount of ore that was raked off in that period. The ore fed during the revolution was collected upon a wooden shield placed on the arms and weighed on a platform balance. These readings were made every half hour.

B. Cinder.

The cinder was wheeled out by hand and weighed on a platform scale.

C. Gas Analysis.

Samples of the burner gas were taken from the flue at half-hour intervals. The gas was analyzed in an Orsat apparatus by first absorbing the SO_2 in chromic acid solution, and then passing the remainder of the gas into pyrogallie acid which absorbed the oxygen. The unabsorbed gas was assumed to be nitrogen.

The chromic acid solution was made by dissolving 165 grams of sodium dichromate in 125 ml. of water and adding 40 ml. of 66° Baume sulfuric acid.

The pyro solution was made by mixing a solution of 35 grams of sodium hydroxide in 105 grams of water with a solution of 35 grams of pyrogallie acid in 105 grams of water.

Mercury was used to draw the gases from the flae. In order to prevent the dry gases from taking up any moisture while being passed thru the absorption solutions, the mercury was covered with a very thin layer of water. Thus the gases were saturated before entering the absorption tubes. The gas burettee was water-jacketed and kept at a constant temperature.

D. Dust.

At the beginning of the run the cyclone dust collector located in the burner gas flue was completely emptied. During the test the dust was collected at several hour intervals and weighed upon a platform scale.

E. Temperature Measurements.

The Brown recording instruments were read at half-hour intervals during the test. These gave the gas temperatures on each hearth, of the exit gas, and also the temperature of the exit cooling air. The temperatures of the incoming cooling air, and of the ore feed, and of the cinders were taken with a mercury thermometer.

F. Measurement of Amount of Cooling Air.

The air used for cooling the rabble arms was measured by placing a 17 foot sheet metal pipe (11.6 inches diameter) at the intake of the blower. At a point eight feet from the blower a Pitot tube was inserted into the pipe. A pipe factor was

determined by the eleven point system. Readings were made upon a ten-to-one inclined water manometer at distances from the center equal to: $0.316r$; $0.548r$; $0.707r$; $0.837r$; and $0.949r$; where r is the radius of the pipe in inches.

Six readings, which for all practical purposes were constant, were made upon the water manometer at half-hour intervals during the test. It was then necessary to remove the pipe, because it interfered with the workmen. Since the speed of the blower was not changed during the run, it was assumed that the amount of cooling air per hour was constant during the run.

G. Methods of Sampling and Analysis.

During the test composite samples were taken of the hopper ore, feed ore, and of the cinder.

Several methods of analysis of iron pyrites were investigated and tried on the samples. The following methods were found to be not

applicable: Lunge method of using reversed aqua regia; Allen and Bishop method of using a mixture of bromine and carbon tetrachloride; and the ordinary method of using a fusion mixture of sodium peroxide-sodium carbonate. These left large quantities of the sample undissolved and consequently gave low results. The method of analysis finally adopted, and which gave the best results, was this: 0.5000 gram of dry sample was mixed with a fusion mixture of 10 grams sodium peroxide and 0.4 gram sugar carbon. Part of the fusion mixture was used to line the nickel crucibles in which the reactions were carried out. The ore mixed with the fusion mixture was then placed into the properly-lined crucible and the mass set off by a glowing string inserted thru a hole in the nickel cover. The crucible during fusion rested in a bath of cold water which absorbed the intense heat given off during the reaction. The above fusion mixture has been suggested by the U.S. Bureau of Standards.

The contents of the crucible upon cooling were dissolved in 150 ml. of hot water. When the reaction ceased, 5 ml. of HCl (s.gr. 1.12) saturated with bromine were added. The mixture was then heated to boiling and the ferric hydroxide filtered thru a Swedish filter paper and washed free from sulfate. The filtrate was acidified with HCl (s.gr. 1.12) and evaporated to dryness to dehydrate the silica. The mass was taken up in 2 ml. of HCl (s.gr. 1.2), 100 ml. of hot water added and the solution filtered. The silica was washed until free from sulfate. Sulfur was determined in the filtrate by the Lunge method.

Iron was determined by dissolving the ferric hydroxide precipitate from the above determination with 15 ml. of HCl. This solution was combined with the filtrate from the BaSO_4 precipitation. The iron was determined volumetrically by the method of Zimmerman-Renhardt. Both ore and cinder samples were analyzed by the above methods.

III. Hearth Study: Combustion of Sulfur.

This phase of the work was done by taking two samples from each hearth. One sample was taken as the ore fell on the ~~hearth~~ and the other as the ore fell off the shelf. Twenty four samples were thus taken; a sample of the feed was also taken. One samples were taken with an iron spade. The hot ore was placed in a practically air-tight can, which was chilled in a bath of cold water. Thus it was hoped to prevent further combustion from taking place.

The samples were dried, ground and analysed for sulfur and iron by the methods outlined above.

Just before sampling, bed temperatures were taken with an optical pyrometer, also the corresponding recording pyrometer temperature.

DATA.

I. CALIBRATION OF PYROMETERS.

Temperatures of Burner taken with True Temperature Thermocouple

Shelf	mv.	temp.	cold junction	True temp.	Well temp.
Top	27.2	605	21.5	626	508
2	29.8	680	22.5	702	495
3	27.2	605	23.5	628	765
4	28.4	640	28.5	669	710
5	15.2	260	24.0	284	750
6	29.6	670	27.5	698	750
7	15.2-16.8	310-365	19.0	329-384	730
8	30.2	690	20.5	711	710
9	26.8	580	20.0	600	720
10	25.2	550	15.5	565	575
11	19.8	380	17.2	400	505
12	13.8	220	17.5	237	352

Temperatures of Burner taken with True Temperature Thermocouple functioning as a Bare Thermocouple.

10 *	20.0	400	20.5	420	500
10 **	20.4	410	20.5	430	500
11 **	15.4	270	21.0.	291	425
11 *	12.4	180	21.0	201	425

*with compressed air
 ** without compressed air

Temperatures of Beds and Gases as taken with a Protected Thermocouple

Shelf	Place	mv	temp.	cold junction	couple temp.	plant's pyrometer
7	bed	36.5	870	30	900	825
7	gas	29.0	655	30	685	790
12	bed	18.7	360	20	380	335
Flue gas		29.8	695	30	725*	650

*Flue gas temperature taken with a true temperature thermocouple.

DATA.

II. HEAT AND MATERIAL BALANCE.

DATA:

Temperature Readings (°C.) on Recording Pyrometers

Shelf	10:30	11:15	11:30	11:45	Midnite
Top	530	500	505	510	510
2	600	600	595	600	600
3	830	820	815	820	835
4	870	780	795	805	815
5	835	825	840	835	860
6	760	730	725	725	730
7	740	710	700	690	700
8	610	600	570	540	510
9	590	560	525	480	440
10	320	330	300	270	250
11	270	250	210	190	170
12	70	95	90	85	80
Exit gas	675	720	730	720	715
Exit air	300	305	305	310	305

Shelf	12:15	12:30	1:00	1:15	1:30	1:45
Top	485	495	515	505	495	495
2	600	605	610	600	600	600
3	845	815	825	795	815	815
4	825	805	800	785	800	800
5	875	865	860	840	850	850
6	740	745	750	760	760	765
7	710	715	730	745	745	760
8	540	560	600	600	625	625
9	450	485	560	580	610	610
10	230	220	230	305	350	375
11	160	145	145	200	295	340
12	75	70	65	60	75	110
Exit gas	715	725	745	725	730	730
Exit air	310	305	300	305	300	300

Weight of dust from centrifugal blower:

150, 125, and 123 lbs.

DATA: (continued)

Shelf	2:00	2:15	2:30	2:45	3:00	3:15
Top	490	490	490	495	495	500
2	595	595	590	590	590	595
3	815	810	810	820	820	825
4	805	800	800	800	810	805
5	865	855	855	855	865	865
6	760	765	760	765	765	760
7	750	750	745	760	750	745
8	630	620	630	630	635	630
9	615	610	615	620	630	625
10	390	400	405	415	415	410
11	360	370	375	375	375	360
12	190	210	210	180	170	170
Exit gas	735	725	730	725	730	720
Exit air	290	290	290	285	285	285

Shelf	3:30	3:45	4:00	4:15	5:00
Top	500	500	505	500	500
2	595	595	595	595	595
3	825	825	830	830	815
4	805	800	805	805	800
5	865	865	870	860	865
6	770	765	775	765	765
7	760	750	760	750	750
8	630	635	635	640	640
9	625	630	625	625	630
10	415	420	425	425	415
11	365	370	370	370	340
12	150	135	130	215	115
Exit gas	725	730	725	730	735
Exit air	285	290	285	290	290

DATA:(continued)

Weight of cinders

Time	Lbs. cinders	Time	Lbs. cinders
10:37 pm	262	2:12 am	192
10:40	168	2:13	183
10:55	196	2:40	183
10:57	223	2:40	169
11:05	205	2:41	215
11:09	211	2:42	198
11:20	216	2:43	188
11:30	228	2:45	171
11:40	280	2:50	205
11:45	173	2:55	138
11:55	182	3:05	174
12:30 am	180	3:10	208
12:33	131	3:25	180
1:05	167	3:25	206
1:10	197	3:45	205
1:20	220	3:50	163
1:30	220	3:55	196
1:45	207	4:26	194
1:50	193	4:30	140
1:52	200	4:30	190
1:53	228	4:30	203
1:54	175	4:35	212
1:55	192	4:35	188
1:56	210	5:00	164
2:10	207	5:00	203

Temperature of cinders leaving shelf no. 12

Time	°C.
2:30	350
3:10	260
3:55	190

DATA: (continued)

Calibration of Ore Feed:

Time	Seconds/revolution	Lbs./revolution
10:30 pm	37	26.0
11:00	37	26.0
11:30	37	26.0
12:00 m	37	26.0
12:30 am	37	24.0
1:00	37	25.0
1:30	37	25.0
2:00	37	27.0
2:30	37	27.0
3:00	37	25.5
3:30	37	26.5
4:00	37	25.0
4:30	37	24.5
5:00	<u>37</u>	<u>25.0</u>
Average	37	25.6

Temperatures (taken with a 360 °C. mercury thermometer)

Time	cold ore	feed ore	outlet air
10:30 pm	20	70	290
11:00	20	69	297
11:30	22	78	307
12:00 m	24	85	305
12:30 am	23	84	305
1:00	22	82	307
1:30	20	104	295
2:00	21	95	295
2:30	18	70	(for further readings see pyrometer re- cording temps.)
3:00	18	104	
3:30	21	74	
4:00	18	76	
4:30	18	70	
5:00	<u>20</u>	<u>80</u>	
Average	20 °C.	82 °C.	

DATA: (continued)

Pitot Tube Readings to Determine Pipe Factor:

Horizontal diameter of pipe: 11.6 in.

Point Readings (in. water at 6.5 °C)

1	0.58	0.38
2	0.60	0.48
3	0.60	0.48
4	0.62	0.48
5	0.64	0.48
6	0.68	0.50
7	0.76	0.50
8	0.76	0.50
9	0.80	0.50
10	0.69	0.48
11	<u>0.60</u>	<u>0.46</u>
Average	0.67	0.48

Pitot Tube readings during test:

Time Reading (in. water at 6.6 °C.)

10:45 pm	0.44	
11:05	0.42	Pitot Tube readings after 1:00 A.M. were discontinued due to the necessity of removing pipe.
11:40	0.38	
12:00 m	0.42	
12:30	0.40	
1:00	<u>0.38</u>	
Average	0.41	

Temperature of inlet cooling air:

Time	°C.
10:50 pm	6.5
11:42	6.0
1:10 am	4.0
2:05	3.1
3:00	3.0
4:05	<u>3.0</u>
Average	4.3

DATA: (continued)

Analyses of EXit Gases:

Time	% CO ₂	% O ₂
10:30 pm	9.9	7.6
11:30	11.7	-
12:00 m	10.2	8.8
12:30 am	10.2	9.0
1:00	10.2	7.0
1:30	14.0	4.0
1:45	11.9	5.9
2:30	12.6	5.8
3:10	12.0	7.0
3:30	11.4	-
4:30	14.4	-
5:00	<u>11.4</u>	<u>*</u>
Average	11.7	6.9

Analytical Data

I. Heat and Material Balance:

A. Moisture determination:

Sample: wet ore from hopper:

% moisture*-- 2.7 (dry basis)

Sample: ore to feed hole:

% moisture--- 0.506 (dry basis)

B. Analysis of feed: (dry basis)

No. 1

No. 2

40.5% Fe

39.2% Fe

45.0% S

43.5% S

C. Analysis of cinder:

52.5% Fe

52.7% Fe

2.07% S

1.92% S

DATA.

III. HEARTH STUDY.

Hearth Study: Summarized Data

<u>Place taken</u>	<u>Sample % S</u>	<u>Analysis % Fe</u>	<u>Theoret. % Fe</u>	<u>theoret. lbs. S/100 lbs. Fe.</u>
Feed	44.9	39.8	39.2	114.0
2 in.*	(47.2)	40.3		
3 out**	36.5	52.0	45.3	80.8
3 in.	25.7	55.3	51.1	50.3
4 in.	22.2	54.9	51.5	43.2
4 out.	30.4	55.0	49.6	61.3
5 out.	23.5	56.1	51.4	45.7
5 in.	14.1	56.7	52.6	26.8
6 in.	13.6	56.7	53.0	25.6
6 out.	14.2	53.5	52.7	27.0
8 in.	5.68	54.5	53.8	10.5
9 out.	7.61	61.1	53.6	14.2
10 in.	6.45	53.7	53.7	8.3
11 out.	2.88	58.7	54.2	5.3
12 in.	1.53	54.8	54.4	2.8

* 2 in. means Hearth number two at a point near the central shaft

** 3 out. means Hearth number three at a point near the shell of the furnace.

Temperatures of Beds as taken with an Optical Pyrometer
and Temperatures of Hearths as recorded on plant Pyrometer.

Shelf No.	Plant Pyrometer	Optical Pyr. of Bed
1	430 °C	
2	530	760 °C.
3	725	795
4	725	855
5	790	875
6	730	815
7	710	775
8	605	760
9	595	700
10	440	670
11	415	
12	180	

SUMMARIZED DATA FOR HEAT AND MATERIAL
BALANCES.

Basis: 1 hour

2490 lbs. wet ore from hopper
2.7% moisture (dry basis)

Analysis on dry basis: 44.3% S, 39.8% Fe, 15.9% gangue
Moisture in ore to feed hole: 0.506% (dry basis)

Cinder: 1527 lbs. 2% S, 52.6% Fe.

Dust from centrifugal collector: 62.5 lbs.

Exit burner gasses: 11.7% SO₂, 6.9% O₂, 81.4% N₂.

Temperature of ore from hopper: 20 °C.

Temperature of ore to feed hole: 82 °C.

Temperature of inlet cooling air: 4.3 °C.

Temperature of outlet cooling air: 295 °C.

Temperature of cinders leaving No. 12 shelf: 300°C.

Temperature of exit burner gasses: (recording pyrometer)
725 °C.

CALCULATIONS.

CALCULATIONS:

Pipe factor for cooling-air pipe:

First exploration with Pitot tube,

average reading: 0.67 in water
 center reading: 0.68 in water
 temperature: 43.7 °F. (6.6 °C)

density of air at 43.7°F.,

$$\frac{29 \times 492}{359 \times 503.7} = 0.0790 \text{ lb./cu. ft.}$$

$$h_{\text{average}} , \frac{0.67 \times 62.3}{12 \times .0790} = 44.2 \text{ ft. air}$$

$$h_{\text{center}} , \frac{0.68 \times 62.3}{12 \times .0790} = 44.8 \text{ ft. air}$$

$$u_{\text{average}} , \sqrt{2 g h} = \sqrt{64.4 \times 44.2} = 53.3 \text{ ft./sec.}$$

$$u_{\text{center}} , \sqrt{64.4 \times 44.8} = 53.8 \text{ ft./sec.}$$

$$\text{Pipe factor (1): } \frac{u_{\text{ave.}}}{u_{\text{center}}} = \frac{53.3}{53.8} = 0.991$$

Second exploration with Pitot tube:

average reading: 0.48 in water
 center reading: 0.50 in water

$$h_{\text{ave.}} \frac{0.48 \times 62.3}{12 \times 0.0790} = 31.5 \text{ ft. of air}$$

$$h_{\text{center}} \frac{0.50 \times 62.3}{12 \times 0.0790} = 33.0 \text{ ft of air}$$

$$u_{\text{ave.}} \sqrt{64.4 \times 31.5} = 45.0 \text{ ft.air/sec.}$$

$$u_{\text{center}} \sqrt{64.4 \times 33.0} = 46.0 \text{ ft.air/sec.}$$

CALCULATIONS: (continued)

$$\text{Pipe factor (2): } \frac{u_{\text{ave.}}}{u_{\text{center}}} = \frac{45.0}{46.0} = 0.978$$

$$\text{Average pipe factor: } \frac{0.991 + 0.978}{2} = 0.985$$

Calculation of amount of cooling air:

average reading: 0.41 in. water
average temperature: 39.8°F. (4.3°C.)
diameter of pipe: 11.6 inches

Density of air:

$$\frac{29 \times 492}{359 \times 498.8} = 0.0795 \text{ lbs. cu.ft.}$$

$$h_{\text{ave.}} = \frac{0.41 \times 62.3}{12 \times 0.0795} = 26.8 \text{ ft. air}$$

$$u_{\text{ave.}} = 0.985 \sqrt{64.4 \times 26.8} = 40.9 \text{ ft./sec.}$$

Pounds of air / second:

$$\frac{(11.6)^2 \times 0.785 \times 40.9 \times 0.0795}{144} = 238 \text{ #air/sec.}$$

$$\text{or } \frac{2.38}{0.0795} = 29.5 \text{ cu.ft.air/sec.}$$

HEAT AND MATERIAL BALANCES.

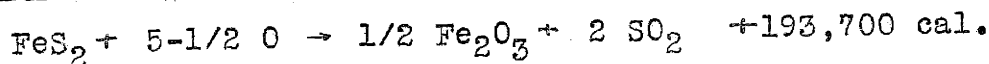
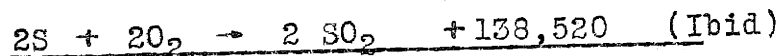
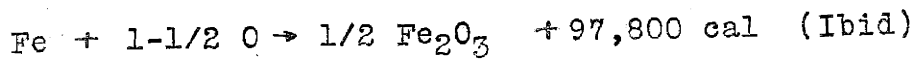
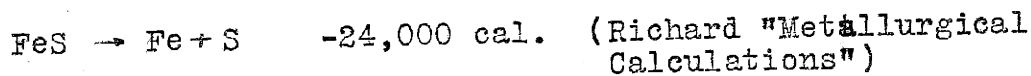
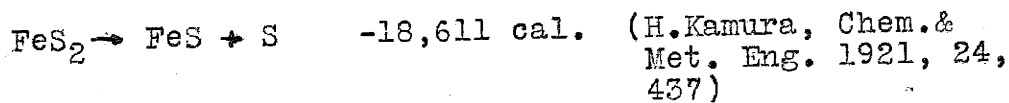
Heat of Combustion of Ore:

Basis: 100lbs ore,

44.3 lbs S
39.8 lbs Fe
15.9 lbs inert

55.34 x 44.3 / 2 x 32.06 = 39.2 lbs iron for theoretical pyrites

Theoretical pyrites assumed.



193,700 C.h.u. / lb mol 1.8 x 193,700 =

348,000 B.t.u. / lb mol

348,000 / (55.84 + 64.12) = 2910 B.t.u. / lb mol of theoretical pyrites

44.3 + 39.2 = 83.5% of ore is theoretical pyrites

2910 x 0.835 = 2430 B.t.u. / lb of ore

1557 x 1.8 x 0.835 = 2340 B.t.u. / lb of ore

Lunge (Vol. I part II page 60) gives 1557 gm. cal per gm. mol as the heat of combustion of theoretical pyrites.

CALCULATIONS FOR MATERIAL BALANCE.

Basis: 1 hour

Feed: $2490 \times 100 / 102.7 = 2424$ lb b.d. ore
 $2424 \times 0.027 = 66$ lb moisture in inert feed
 $2424 \times 0.00506 = 12.3$ lb moisture to feed hole
 $66 - 12.3 = 53.7$ lb moisture evaporated on
preheating hearth
 $2424 \times 0.443 = 1070$ lb S
 $2424 \times 0.398 = 960$ lb Fe
 $2424 - (1070 + 960) = 390$ lb gangue

Cinder:

$1527 \times 0.02 = 30.5$ lb S
 $1597 \times 52.6 / 111.7 = 75.6\%$ Fe_2O_3
 $100 - (75.2 + 2) = 22.8\%$ gangue
 $1527 \times 0.228 = 348$ lb gangue in cinder
 $1527 \times 0.528 = 805$ lb Fe in cinder

Dust:

(Assume same analysis as cinder) 62.5 lbs
 $62.5 \times 0.526 = 33.0$ lb Fe
 $62.5 \times 0.02 = 1.3$ lb S (negligible)
 $62.5 \times 0.228 = 14.0$ lb gangue

Gas:

Analysis: SO_2 11.7%; O_2 6.9%; N_2 81.4%.

Material Balance (continued)

$$(1070 - 30.5) / 32 = 32.5 \text{ mols S burned}$$

also 32.5 mols SO_2 formed

$$32.5 \times 6.9 / 11.7 = 19.2 \text{ mols } \text{O}_2$$

$$32.5 \times 81.4 / 11.7 = 228 \text{ mols } \text{N}_2$$

Cubic feet of gas at S.C.

32.5 x 359	11,700	cu ft SO_2
19.2 x 359	6,900	O_2
228 x 359	<u>81,800</u>	N_2

100,400 cu ft of burner gas

Air for combustion:

$$228 \times 100 \times 359 / 79 = 104,000 \text{ cu. ft.}$$

Calculations for Heat Balance.

Basis: 1 hour datum line 68°F. (20°C)

Input

Feed: 2424 x 2430 = 5,880,000 B.t.u.

Output

Sensible heat

Burner gases: 725 °C. corrected to 810 °C.
(1490 °F.) by correction ration 725/650
previously obtained by true temperature
thermocouple.

SO₂ : 32.5 x (16000- 300) = 510,000 B.t.u.

N₂ & O₂ : (228 19.2)(10,500 - 250) =
2,540,000

Moisture in gas :

12.3(13,000-280) / 18 = 8,700

Cinder: 1527x0.15(572-68) = 115,000

Dust: 62.5x0.15(1490-68) = 13,400

Ore: 2424x0.15x16x1.8 = 10,500

Cooling air:

2.38x3600x0.238(295-4)x1.8 =
1,070,000

Unburnt ore in cinder:

1527x0.02x111.9 / 64.1 =

57.2 lb of theo. FeS₂

57.2 x 2910 = 167,000

Latent heat of water vapor on
preheating hearth

53.7 x 1000 = 53,700

SUMMARIZED MATERIAL BALANCE

By Sulphur Balance,

Input

Feed 1070 lbs

Output

Cinders 30.5
Gas (by diff.) 1039.5

1070 lbs

By Iron Balance

Input

Feed 960 lbs

Output

Cinders 805
Dust 33
Unaccounted for 122

960 lbs

% unaccounted for: $122 \times 100 / 960$

12.7%

By Gangue Balance

Input

Feed 390 lbs

Output

Cinders 348
Dust 14
Unaccounted 28

390 lbs

% unaccounted for: 7.2%

SUMMARIZED HEAT BALANCE

Input

Feed 5,880,000 B.t.u.

Output

Sensible heat of burner gases

SO₂ 510,000

N₂ & O₂ 2,540,000

Moisture in gas 8,700

Sensible heat

Cinder 115,000

Dust 13,400

Cooling air 1,070,000

Unburnt ore in cinder 167,000

Latent heat of moisture in vapor on preheating hearth 53,700

Sensible heat in ore 10,500

4,488,300 B.t.u.

Specific Heats, and Heats of Reaction.

Specific Heats (Landolt-Bornstein)

Fe_2O_3	(24 °C.)	0.1600
FeS	(17-98°C.)	0.1357
Fe_7S_8	(0.-100°C.)	0.1459

Heats of Reaction.

FeS_2 FeS S -18,611 cal. (H.Kamura, Chem.& Met.
Eng. 1921,24,437)

FeS_2 $5-1/2 \text{ O}$ $1/2 \text{ Fe}_2\text{O}_3$ 2 SO_2 1557 cal.
(from Lunge's calculations,
p.60, vol. I part I, "Manu-
facture of Sulfuric Acid")

FeS Fe S -24,000 cal. (Richards, "Metallurgical
Calculations)

Fe $1-1/2 \text{ O}$ $1/2 \text{ Fe}_2\text{O}_3$ 97,800 cal (Ibid)

S O_2 SO_2 69,260 cal. (Ibid)

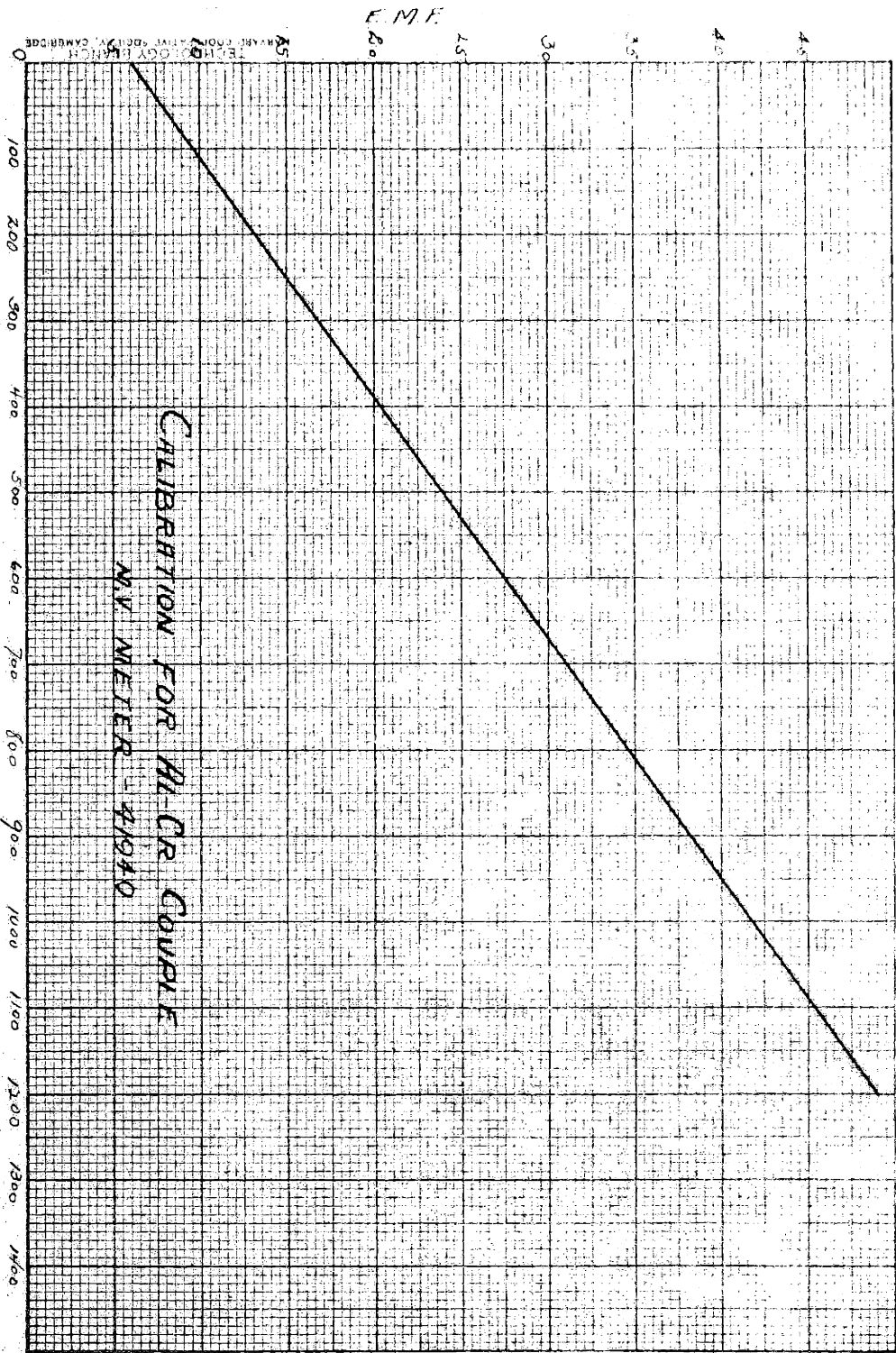


FIGURE 8.