THE TREATMENT OF COPPER CONCENTRATES BY WET METHOD

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I.

INTRODUCTION

Many metallurgists have tried to recover copper from flotation concentrates by hydrometallurgical methods. No one has developed an economical process to compete with the smelting process which is applied in the present copper industries. The treatments used in the hydrometallurgical process are roasting, leaching, and recovery of copper from solutions. Methods are reported from A.I.M.E. and Eng. & Min. Jnl. such as: (1) leaching the calcine by acid and chloridizing roasting of the residues(1); (2) leaching the calcine by water or acid and smelting the residues.(2) The first method shows a good extraction of copper, but not a good extraction

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(1) L. Addicks. Possibilities in the Wet Treatment of Copper Concentrates. Trans. A.I.M.E. (1916), 55, 856.

(2) W.E. Greenawalt. Electrolytic Copper from Copper Concentrates. Eng. & Min. Jnl. (1930), 129, 241-244.

of silver, and the ore contains practically no gold. The second method has possibilities of recovering all the gold and silver by smelting. However, it is not economical to build a small smelting plant for this purpose only, or to ship the large amount of residues to the reduction plants.

In recent years metallurgists have applied the sulphuric acid baking method in the production of soluble sulphates from mixed oxide ore, followed by leaching the sulphates by water.(1),(2) Later on this method is applied to the treatment of roasted copper concentrates.(3) It is successfully compared to some other methods, but the recovery of silver is not successful. The extraction of gold from residue by the cyanidation method still cannot compete with the smelting method, and the high cost of the roasting and leaching equipment must be considered in the plant installation.

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(1) C.F. Floe and C.R. Hayward. Differential Production of Soluble Sulphates from Mixtures of Metallic Oxides, A.I.M.E. Tech. Pub. 735 (1936).

(2) B.H. Strom. Roasting with Sulphuric Acid Increases Zinc Recovery. Eng. & Min. Jnl. (1930) <u>129</u>, 79.

(3) C.F. Floe. Extraction of Copper from Roasted Concentrates by Sulphuric Acid Baking. A.I.M.E. Tech. Pub. 768 (1937).

The author believes that the hydrometallurgical method will be able to compete with the smelting method, if the following problems can be solved: (1) copper and precious metals can be completely extracted from the ores; (2) the leaching solvent (sulphuric acid) can be produced from the roaster gases; (3) precious metals can be recovered by an economical method; (4) simple equipment can be used; and (5) the whole operation is near the mine.

The purpose of this thesis is an attempt to extract all the copper and precious metals from the sulphide copper concentrates - chalcopyrite. The experiments are under four main headings:

(1) - Sulphatizing roast.

(2) - Leaching by water and dilute sulphuric acid.

(3) - Chloridizing residue.

(4) - Recovery of copper and precious metals from solutions.

The problems to be worked out are:

(1) - To determine the influence of the temperature and time of sulphatizing roast in the formation of sulphates from sulphide copper concentrates in order that a high extraction of copper can be made.

(2) - To determine the amount of iron in the water leaching solution of roasted ores at various temperatures.

(3) - To determine the percentage of copper leach from roasted ores following the five per cent dilute H_2SO_4 leach on the residue.

(4) - To determine by the chloridization method the possibility of extracting all copper and precious metals (gold and silver) which remain in the leached residues.

(5) - To determine by the chloridization method the optimum condition necessary for the formation of chlorides from leached residues which contain a few per cent of copper, gold and silver.

II.

THEORY OF PROCESS AND REVIEW OF PREVIOUS WORK

In preparation for the laboratory work, the author reviewed: (1) the literature on oxidizing, sulphatizing, and chlorizing; (2) previous theses written in the Department of Metallurgy; (3) reference books, "General Metallurgy" by Hofman, "Metallurgy of Copper" by Hofman and Hayward, "An Outline of Metallurgical Practice" by Hayward, and so forth.

1. Oxidizing and Sulphatizing Roast

Metallic sulphide is heated gradually with an access of air to a temperature at which the metallic oxide and sulphur dioxide are formed. The operation is called an oxidizing roast.

The most important reaction taking place in the oxidizing roast is as follows:

$$MS + 3O = MO + SO_2$$

In the absence of air and in contact with metallic oxides, a part of the sulphur dioxide is converted into sulphur trioxide. The sulphur trioxide is formed in such a way that it may combine with metallic oxide to become sulphate. This operation is called a sulphatizing roast.

The reactions probably take place as follows:

$$MO + SO_3 \rightleftharpoons MSO_4$$

$$3SO_2 \rightleftharpoons 2SO_3 + S$$

$$SO_2 + 2MO \rightleftharpoons M_2O$$

$$SO_2 + 3Fe_2O_3 \rightleftharpoons SO_3 + 2Fe_3O_4$$

The essential factors for roasting are: (1) size and composition of the ore; (2) temperature; (3) frequent rabbling; and (4) kind and amount of catalyzer.

Application of the common methods used for sulphide ores, such as an oxidizing roast and sulphuric acid leach, leaching with ferric sulphate, or leaching with ferric chloride, is not very successful with chalcopyrite ores. The reasons are: (a) in an oxidizing roast the copper forms copper ferrite ($CuO.Fe_2O_3$, $Cu_2O.Fe_2O_3$) and copper silicates, all of which are quite insoluble in dilute sulphuric acid; (b) in the commercial application such a long treatment for a complete conversion by means of ferric sulphate or ferric chloride and a large amount of iron in the solution for electrolysis is impossible if profits are expected.

Hofman(1) stated that FeS_2 or $FeSO_4$ helps the ores convert from sulphide to sulphate in the sulphatizing roast. The other sulphatizing agents are $Al_2(SO_4)_3$ to $NaHSO_4$.

Goolbaugh(2) treated the sulphide ores or mattes in three steps: (1) sulphide ores or mattes roast to oxides; (2) sulphatizing roast the oxides; and (3) sulphates are leached out. The precious metals, silver and gold, are recovered from smelting the residues.

Aarts and Aarts(3) indicated that iron oxide is a catalytic agent for sulphatizing roast and

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(1) H.O. Hofman. General Metallurgy, 406, New York, McGraw Hill Book Co.

(2) M.T. Goolbaugh. U.S. Patent 1,315,761. Sept. 9, 1919.

(3) Aarts and Aarts. British patent 119,867.

three conditions are determined before the ore is sulphatized: (1) amount of sulphur in the ores; (2) amount of the catalyst; and (3) composition of the ores.

Westly(1) worked with the treatment of metalliferous fines containing copper.

Dudley, Jr.(2) pointed out that the formation and decomposition of sulphates during roasting is dependent on the following conditions: temperature; time; the partial pressures of the reacting gases (sulphur dioxide and trioxide); and the amount of catalytic agent present.

The two important equilibrium reactions are:

$$MO + SO_3 \rightleftharpoons MSO_4$$

$$SO_2 + 1/2\ O \rightleftharpoons SO_3$$

Bugbee and Turner(3) indicated that 600°C. was the best temperature for the sulphatizing of

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(1) G.C. Westly. U.S. Patent 1,266,731 and 1,266,732, May 21, 1918.

(2) B. Dudley, Jr. Met. Chem. Engr., <u>13</u>, pp. 221-6 and 303-8, April 1915.

(3) Bugbee and Turner. Study in the Sulphatizing of Some Metallic Oxides. Thesis: Mass. Institute of Technology (1920).

cupric oxide. The catalyzer was shown to aid in the formation of SO_3 in the work. Time for roasting did not have much effect on the extraction of copper.

Nash's results(1) showed that 400°C. was the temperature to use for the best extraction of copper and the catalyzer was quite suitable to the formation of sulfates. No test was made with CuO at a temperature lower than 400°C.

Brown(2) agreed with Nash on the effect of the catalytic agent on the extraction. CuO to SO_3 are increased if the amount of Fe_2O_3 is increased. For ferric oxide catalysts, the formation of SO_3 is converted from SO_2 and air.

Erickson and Johnson's thesis(3) indicated that the highest extraction of copper is obtained between 500 to 600°C. High extractions are manifestly due to favorable conditions of SO_2 concentration and time.

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(1) H.L. Nash. The Catalytic Effect of Ferric Oxide on the Sulphatizing of Metallic Oxides. Thesis: Mass. Inst. of Tech. (1920).

(2) A.A. Brown. Investigation of the Catalytic Effect of Ferric Oxide on the Sulphatizing of Metallic Oxides. Thesis: Mass. Inst. of Tech. (1920).

(3) Erickson and Johnson. Sulphatizing of Metallic Oxides. Thesis: Mass. Inst. of Tech. (1921).

Eighty per cent copper was extracted by Heymans(1) from the oxidizing roast of chalcopyrite ore. The ore was roasted 90 minutes at 450°C. and then leached by five per cent (by weight) of sulphuric acid. There was an extraction of 44.5 per cent of copper from the chalcopyrite ore if the muffle was kept closed for three hours at 300°C. Ferric sulphate or ferric chloride solution was used for leaching.

Greenawalt(2) suggested a treatment of a variety of simpler copper, lead, gold, and silver ores by the following steps: (a) oxidizing roast the ores; (b) leaching the copper by water from the roasted ores; (c) sintering and smelting the leached residues in lead blast furnace, silver and gold may be obtained from leach bullion.

The work done by Floe(3) was to determine the optimum condition necessary for the formation of

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(1) W. J. Heymans. The Extraction of Copper from Chalcopyrite Concentrates by Wet Methods. Thesis: Mass. Inst. of Tech. (1927).

(2) W.E. Greenawalt. Electrolytic Copper from Copper Concentrates. Eng. & Min. Jnl., <u>129</u>, 241-244, 1930.

(3) C.F. Floe and C.R. Hayward. Differential Production of Soluble Sulphates from Mixtures of Metallic Oxides. A.I.M.E. Tech. Pub. 735, (1936).

sulfates from pure metallic oxides by baking with sulfuric acid, and to study the nature of the reactions involved in sulfating various metallic oxides. In general, the results of treating roasted chalcopyrite show that the method of acid baking is not superior to ordinary acid leaching unless ferrites are present.

One hundred per cent of copper was extracted with ten per cent H_2SO_4 acid leach.

2. Chloridizing Roast

The following statement is introduced from "Metallurgy of Copper" by Hofman and Hayward, to explain the chloridizing reactions:

"The chloridation of copper is due largely to the presence in burned pyrite of $CuSO_4$, which, acting upon NaCl, formed $CuCl_2$ and Na_2SO_4, and to the decomposing effect of $FeSO_4$, either present as such or formed by the oxidation of FeS. The $FeSO_4$ from both sources could act upon NaCl and form $FeCl_2$, $FeCl_3$, and Na_2SO_4; or, after it had been decomposed by heat, the SO_3 set free would convert Cu_2S into $CuSO_4$; or, acting upon NaCl, it would give Cl (which would chloridize Cu_2S) and HCl in the presence of H_2O (and chloridize CuO).

The roast products from the chloridizing roasting of ore are a mixture of cupric chloride, cupric oxide, cupric sulphate, cupreous chloride, sodium sulphate, ferric chloride, ferric oxide; also sulphate and chlorine combinations of the other metals contained in the ores. If silver and gold are contained in the ores, the silver and gold chlorides are formed."

Longmaid-Henderson process.(1) This process was used to treat the residues or cinders of the sulphide ores from the manufacture of sulphuric acid. The ores contained three to six per cent sulphur and three to six per cent copper.

If any small amount of silver and gold is present in the ores, the chloride compounds of silver and gold are formed, and are soluble in the leaching solution.

The following chemical reactions(2) of the Longmaid-Henderson process which probably take place during the roasting operation are:

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(1) C.R. Hayward. Outline of Metallurgical Practice, 145. New York. D. Van Nostrand Co.

(2) C.R. Hayward. Outline of Metallurgical Practice, 146. New York. D. Van Nostrand Co.

1) $2FeS_2 + 7O_2 = Fe_2(SO_4)_3 + SO_2$

$Fe_2(SO_4)_3 + 6NaCl = 3Na_2SO_4 + 2FeCl_3$

2) $2Cu_2S + 5O_2 = 2CuSO_4 + 2CuO$

$2CuO + 2SO_2 + O_2 = 2CuSO_4$

$3CuO + Fe_2(SO_4)_3 = Fe_2O_3 + 3CuSO_4$

3) $CuSO_4 + 2NaCl = CuCl_2 + Na_2SO_4$

$3CuO + 2FeCl_3 = Fe_2O_3 + 3CuCl_2$

4) $Cu_2S + 4Cl + 3O = 2CuCl_2 + SO_3$

$3Cu_2S + 4FeCl_3 + 15O = 6CuCl_2 + 2Fe_2O_3 + 3SO_3$

5) $2NaCl + SO_3 + H_2O = Na_2SO_4 + 2HCl$

6) $Ag_2SO_4 + 2NaCl = 2AgCl + Na_2SO_4$

7) $Au + 3Cl = AuCl_3$

Hofman and Hayward(1) stated that burned pyrite to be treated by the chloridizing process must contain less than 20 per cent gangue. The

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(1) H.O. Hofman and C.R. Hayward. Metallurgy of Copper, 325. McGraw-Hill Book Co.,Inc., New York.

copper is usually less than six per cent in the ores. A satisfactory percentage of $CuCl_2$ is obtained by using 1 to 1.5 parts of sulphur for every part of copper.

Kothny(1) has shown that the temperature of the chloridizing is between 500 to 600°C.

Croasdale(2) extracted 80 to 90 per cent copper from the raw oxide mixed with 10 per cent NaCl by a chloridization roast at 550 to 700°C. for six hours. It included the 69 per cent of the volatilized copper.

Raw chalcopyrite ore mixed with 12.5 per cent NaCl was roasted for 1.5 to 2 hours. The total per cent extraction of the copper was 92.0.

Volatilized copper is 44.1 per cent and soluble copper is 47.9 per cent.

Addicks(3) applied the chloridizing roast to the acid leaching residue of oxidizing roasted ores.

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(1) - Kothny: see H.O. Hofman and C.R. Hayward. Metallurgy of Copper, 325. McGraw Hill Book Co., New York.

(2) - Croasdale, Stuart. Leaching Experiments on the Ajo Ores. Trans. A.I.M.E., XLIX, 1914, New York.

(3) - L. Addicks. Possibilities in the Wet Treatment of Copper Concentrates. Trans. Eng. & Min. Jnl., 129, 241-244, (1930).

The residues mixed with 7.5 NaCl were roasted in an electric muffle furnace for one and one-half hours at 975°F. and leached in a liquid containing five per cent Na_2SO_4, five per cent NaCl, five per cent $FeCl_2$, and 0.5 per cent HCl + H_2SO_4. The results show a 99 per cent copper and a 79 per cent silver extraction.

From Sanderson's experiments,(1) a chloridizing roast can be applied to a chalcopyrite flotation concentrate with a maximum solubility in water of 78 per cent copper and in acid of 20 per cent copper. The total extraction of copper is 98 per cent when roasting at 400°C. with 50 per cent salt.

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(1) A.F. Sanderson. Chloridizing Roasting of a Chalcopyrite Concentrate. Thesis: Mass. Inst. of Tech., 1935.

III.

MATERIALS, EQUIPMENT AND ANALYTICAL METHODS

1. MATERIALS

(a) - Ore for Sulphatizing Roast

The ordinary chalcopyrite flotation concentrate which we decided to use for research work was found to contain not enough silver and gold after the assay test. Therefore, the special ore had to be made up for the purpose as follows:

1. Weighed 10 kg. of ordinary ore and 3 kg. chalcopyrite ore of Laboratory Box 11 which contains 4.25 gold and 24.52 silver.
2. Mixed both ores thoroughly and passed through 20 mesh.
3. Ground large particles by disc grinder and passed through 20 mesh.
4. Mixed the new ore thoroughly by split shovel.
5. 400 grams ore were taken from split shovel and ground to pass through 100 mesh. The ore was kept as a standard sample.

The chief mineral constituent of the ore was chalcopyrite. The small amount of gangue material was mainly siliceous. The chemical analysis of the mixture was:

Copper	24.6 percent
Iron	28.2 percent
Sulphur	22.1 percent
Gold	1.07 ounces per ton
Silver	6.08 ounces per ton

(b) - Residue for Chlorizing Roast

The residue for the chlorizing roast was obtained after leaching with water and five per cent (by volume) H_2SO_4 solution of sulphatizing roasted ore "C_5". "C_5" was sulphatizing roasted for five hours at 500°C. The residue contained a large amount of iron oxides, a small amount of sulphur, and a small amount of gangue material (siliceous).

Its chemical analysis was:

Copper	3.84 percent
Gold	2.32 ounces per ton
Silver	14.82 ounces per ton

Water

The water used in the leaching was distilled water.

Sulphuric Acid

The sulphuric acid used in the leaching of the sulphatizing and chloridizing ore was: mol. weight = 98.08; specific gravity = 1.84; and Min. 95.5 percent.

Hydrochloric Acid

The hydrochloric acid used in the leaching of chloridizing was mol. weight = 36.468; specific gravity = 1.1878; and Min. 37 percent.

Sodium Chloride

The sodium chloride used in the chloridizing roasting and leaching was ordinary commercial salt.

2. EQUIPMENT

Roasting Furnace

The furnace used for sulphatizing and chloridizing roasting was a muffle type, electric resistance furnace, connected in series with two rheostats and an ammeter. The temperature of the furnace could be maintained constant at any point up to a certain limited degree.

The furnace was built in such a way as to have a low roof which kept the gases generating from roasting in contact with the ore. Its construction, dimensions and electrical connection are given in Figure 1.

Roasting Dish

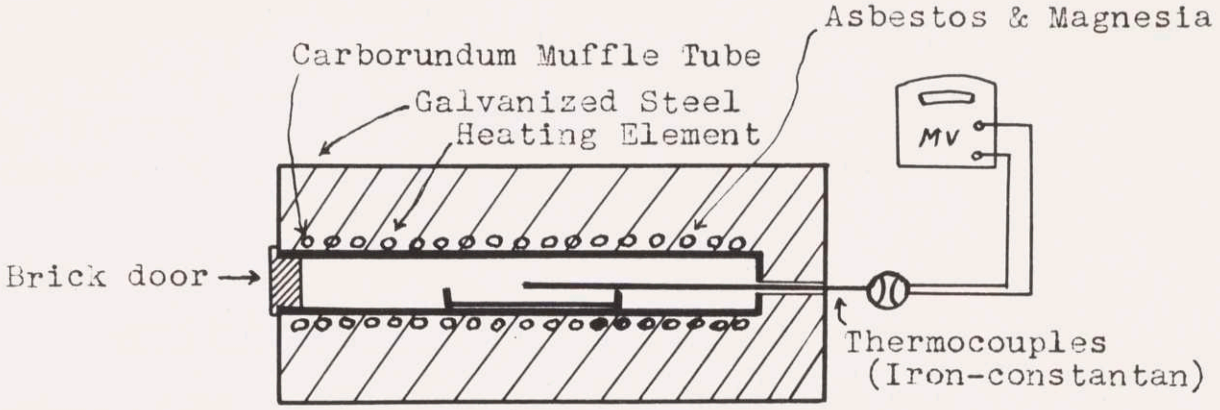
The roasting dish was a fire clay dish which was 9 inches by 6 inches by one-half inch deep.

Rabbling Rod

The rabbling rod was a three-eighths inch steel rod which was bent at one end. The rod was used for rabbling the ore and for drawing the roasting dish out of the furnace.

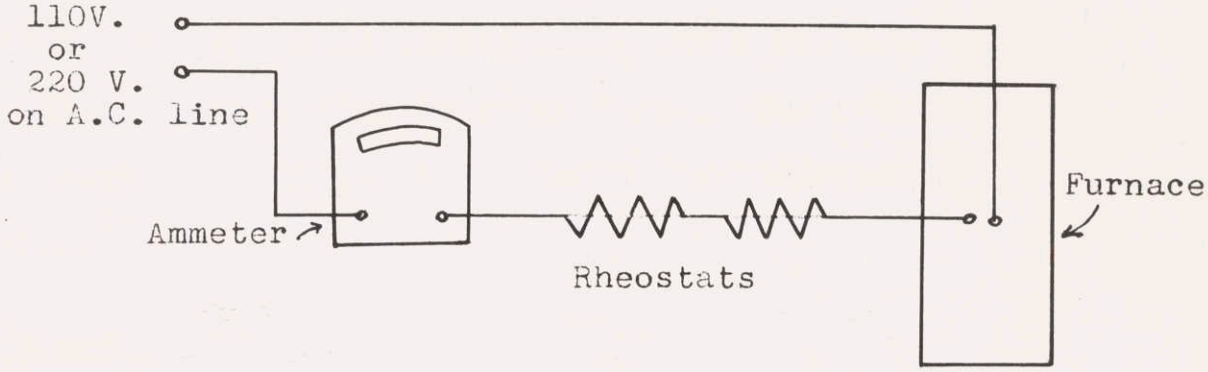
Thermocouples

An iron-constantan thermocouple, protected by a silica tube, was connected to the millivolt meter for the temperature reading.



(A)-Sketch of Muffle Furnace

Furnace size: 26"x15-1/2"x9-1/2"
Muffle size: 21"x9x2-3/4"



(B)-Electrical connection of the Operation

FIGURE 1

(A) - Sketch of Furnace used in Roasting.
(B) - Electrical Connection of the Operation.

220 V. was connected when the temperature of furnace was up to 600°C.

3. ANALYTICAL METHODS

Copper

Copper was determined by the standard iodide method which was outlined by Low.(1)

Iron

Iron was determined by the standard permanganate method outlined by Low.(1) This test was only applied to the iron contained in water leach solutions.

Total Sulphur and Sulphate Sulphur

Total sulphur and sulphate sulphur were determined by adding an excess $BaCl_2$ and a few c.c. of HCl to the solution to be analyzed. The precipitate was weighed as $BaSO_4$. The methods are described by Keffer.(2)

Gold and Silver

Gold and silver were determined by assay methods described by Bugbee.(3)

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(1) A.H. Low. Technical Methods of Ore Analysis. McGraw Hill Book Co., New York.

(2) R. Keffer. Methods in Non-Ferrous Metallurgical Analysis. McGraw Hill Book Co., New York.

(3) E.E. Bugbee. A Textbook of Fire Assaying. John Wiley & Sons, Inc., New York.

IV.

EXTRACTION OF COPPER BY SULPHATIZING METHOD

1. EXPERIMENTAL METHODS

(a) - Sulphatizing Roasting

A resistance electric muffle was used for roasting, connected in series with two rheostats which controlled the current. Through a small opening at the rear end of the muffle, a thermocouple was introduced to measure the temperature. The front of the muffle was closed by a brick with no admission of air. Two hundred and fifty gram samples were roasted each time. The ore, after being weighed, was placed in the clay roasting dish which was pushed into the muffle. The current was turned on and the furnace heated up slowly to the roasting temperature. During the roasting period, the ore was rabbled every fifteen minutes with a rabbling rod. The ore was sampled by drawing out the roasting dish from the furnace. Fifty gram samples were taken out each time.

NOTE:

The variation in roasting temperature must never exceed ten degrees. During the roasting period lumps were formed. The best results were obtained by breaking these lumps. The roasting temperature between 450°C. to 500°C. was found difficult to control, the reaction of the ore having a tendency to raise the temperature.

The appearance of the products was different corresponding to the roasting time and temperature. Dark blue and some brown products were obtained from low roasting temperatures such as 300°C. and 400°C. and shorter times such as 30 and 60 minutes. The appearance of the products was brown or reddish brick color with increasing roasting time and temperature.

There are five regular roasting runs, divided into Series A, B, C, D and E, and two additional runs: Series CD and C_5-C_{55}. The charges for each run under various time and temperature are given in Table I.

For complete details of experimental work see the Appendix.

(b) - Leaching

Water Leaching

There were 28 products from the sulphatizing tests. A 10-gram sample of each roasting product was leached by agitating with 100 c.c. distilled hot water at about 90°C.-100°C. for an hour. After leaching, the solutions were filtered off and were measured in volume. The residues were dried and weighed. The amount of copper was determined by an analysis of the residues, and iron and total sulphate sulphur by the solutions.

Acid Leaching

The residues from tests showing the best extractions were leached again, using a 2.5 gram sample, by agitating with dilute sulphuric acid solution containing 5 c.c. H_2SO_4 and 95 c.c. H_2O at about 90°C.-100°C. for an hour. After leaching, the solutions were filtered off and were measured in volume; the residues were dried and weighed. The amount of copper was determined by an analysis of the residue, and iron by the solution.

The data and results for the leaching tests are given in Table I and Figures 2 to 7. More details of the experimental work can be obtained in the Appendix.

TABLE I

Data and Results

Sulphatizing Chalcopyrite with Various Temperatures and Times

TABLE I

DATA AND RESULTS

Sulphatizing Chalcopyrite with Various Temperatures and Times

Series No.	A at 300°C.				B at 400°C.				C at 500°C.				D at 600°C.				E at 700°C.				CD* at 550°C.				* at 500°C.	
Samples No.	A_1	A_2	A_3	A_4	B_1	B_2	B_3	B_4	C_1	C_2	C_3	C_4	D_1	D_2	D_3	D_4	E_1	E_2	E_3	E_4	CD_1	CD_2	CD_3	CD_4	C_5	C_{55}
ROASTING																										
Charge = 250 gms. for each series																										
Time, Minutes	30	60	120	240	30	60	120	240	30	60	120	240	30	60	120	240	30	60	120	240	30	60	120	240	300	330
Calcine draw from furnace, gms...	50	50	50	96	50	50	50	109	50	50	50	104	50	50	50	83	50	50	50	65	50	50	50	83	100°	110°
Analysis of Calcine, percent Cu .	22.8	22.8	22.4	23.2	22.4	22.8	21.3	21.3	23.7	21.8	21.5	21.4	25.4	25.0	23.2	23.2	25.5	25.7	24.3	25.4	25.2	24.6	22.8	21.6	20.95	21.08
percent S ..	23.84	24.4	24.6	23.8	23.9	21.1	19.1	19.1	14.0	10.6	10.5	10.7	16.1	8.3	6.5	7.0	19.5	12.5	3.5	1.3	16.5	8.5	6.8	8.5	9.7	9.6
percent SO_4.	6.8	5.4	6.0	6.5	6.3	5.6	5.8	7.1	5.7	6.4	8.0	8.5	4.2	7.4	9.2	1.1	1.3	2.0	3.4	1.8	3.0	6.8	16.5	18.4	25.6	25.6
percent S as SO_4	2.3	1.8	2.7	3.0	2.9	2.6	2.7	3.3	2.6	2.9	3.7	4.0	1.9	3.4	4.2	0.5	0.6	0.9	1.5	0.6	1.0	2.3	5.5	6.1	8.6	8.6
LEACHING																										
Temperature = 90°C.-100°C. Leach solution H_2O = 100 c.c. Sample = 10 gms. Time = 1 hour																										
Weight of leach residues, gms.....	8.86	8.85	8.84	8.90	8.56	7.64	6.69	6.45	8.23	6.23	5.93	5.80	9.29	8.79	8.16	7.92	9.62	9.50	9.65	9.78	7.01	7.32	8.85	9.37	5.87	5.77
Leach residues, percent Cu	23.9	23.6	23.0	23.4	23.7	24.5	23.0	22.1	22.7	15.9	14.1	12.8	22.7	24.0	18.3	18.0	26.1	25.9	25.2	25.5	24.1	23.5				
insoluble Cu. gms.	2.39	2.36	2.30	2.34	2.37	2.45	2.30	2.21	2.27	1.59	1.41	1.28	2.27	2.40	1.83	1.80	2.61	2.59	2.52	2.55	2.41	2.35	1.72	1.50	1.17	1.20
Fe. dissolved in leaching, gms....	0.25	0.20	0.24	0.23	0.32	0.46	0.61	0.60	0.17	0.19	0.47	0.19	.038	.035	.027	.037	.019	.025	.041	.016	.035	.046	.066	.080	.150	.153
Percent Cu. extracted in leaching..	7.03	9.63	9.06	9.38	9.40	18.0	24.5	31.5	21.5	54.8	56.2	65.5	17.3	16.0	3.58	37.9	1.96	6.62	0.41	1.97	33.1	30.0	33.2	34.7	67.4	67.2
Percent Fe. extracted in leaching..	2.5	2.0	2.4	2.3	3.2	4.6	6.1	6.0	1.7	1.9	4.7	1.9	0.38	0.35	0.27	0.37	0.19	0.25	0.41	0.16	0.35	0.46	0.66	0.80	1.50	1.53
Ratio of Fe. and Cu. in leaching solutions	1.55	0.91	1.1	1.08	1.45	1.1	1.18	0.88	0.32	0.16	.386	.138	.086	.087	.032	.042	.047	0.14	.414	0.32	.0417	.062	.087	.107	.107	.109

* Additional sulphatizing test on samples of chalcopyrite.

° C_5 and C_{55} total charge = 200 grams.

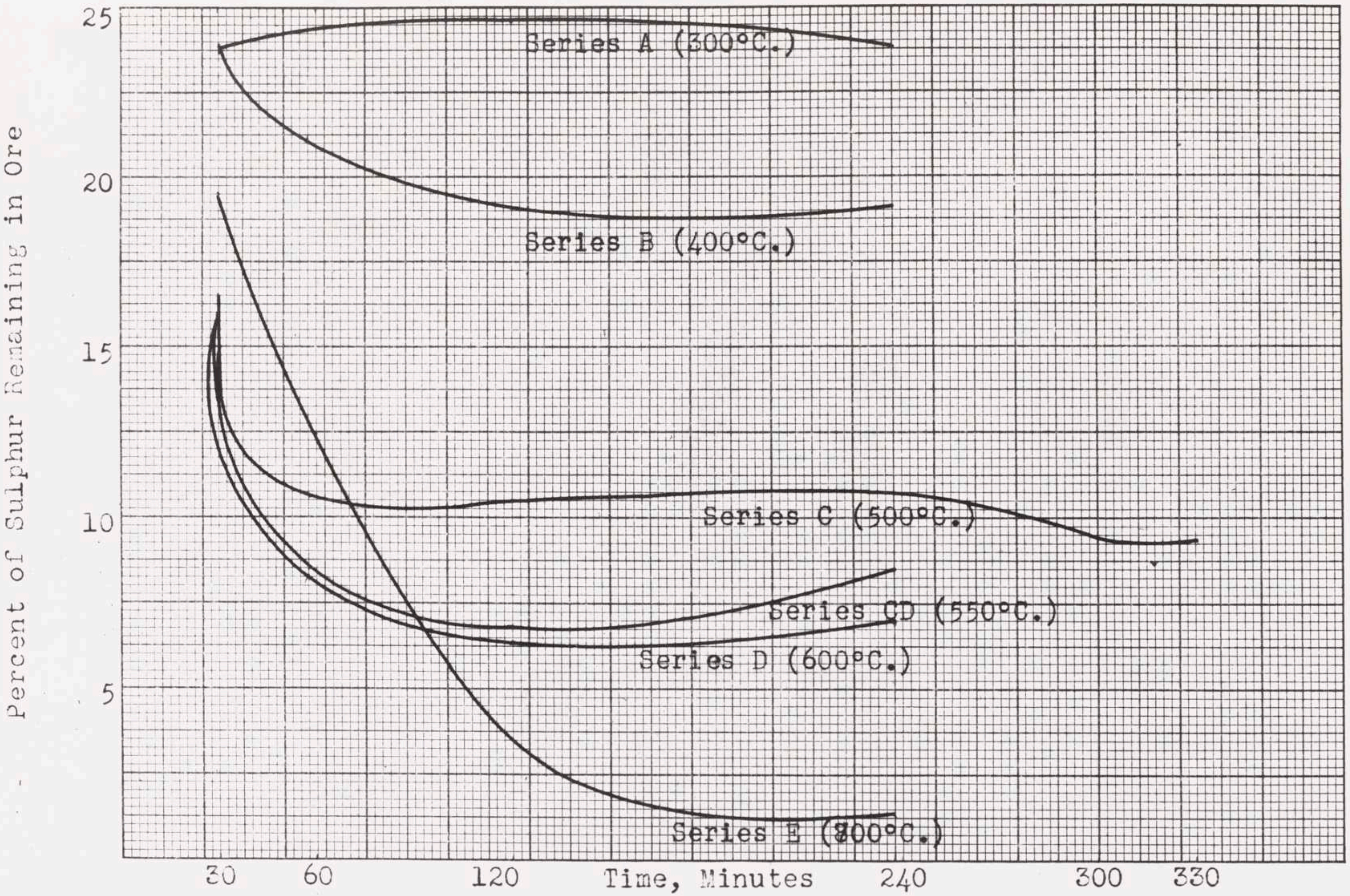

FIGURE 2

Effect of Time on Sulphur Removal During Roasting
at Various Temperatures

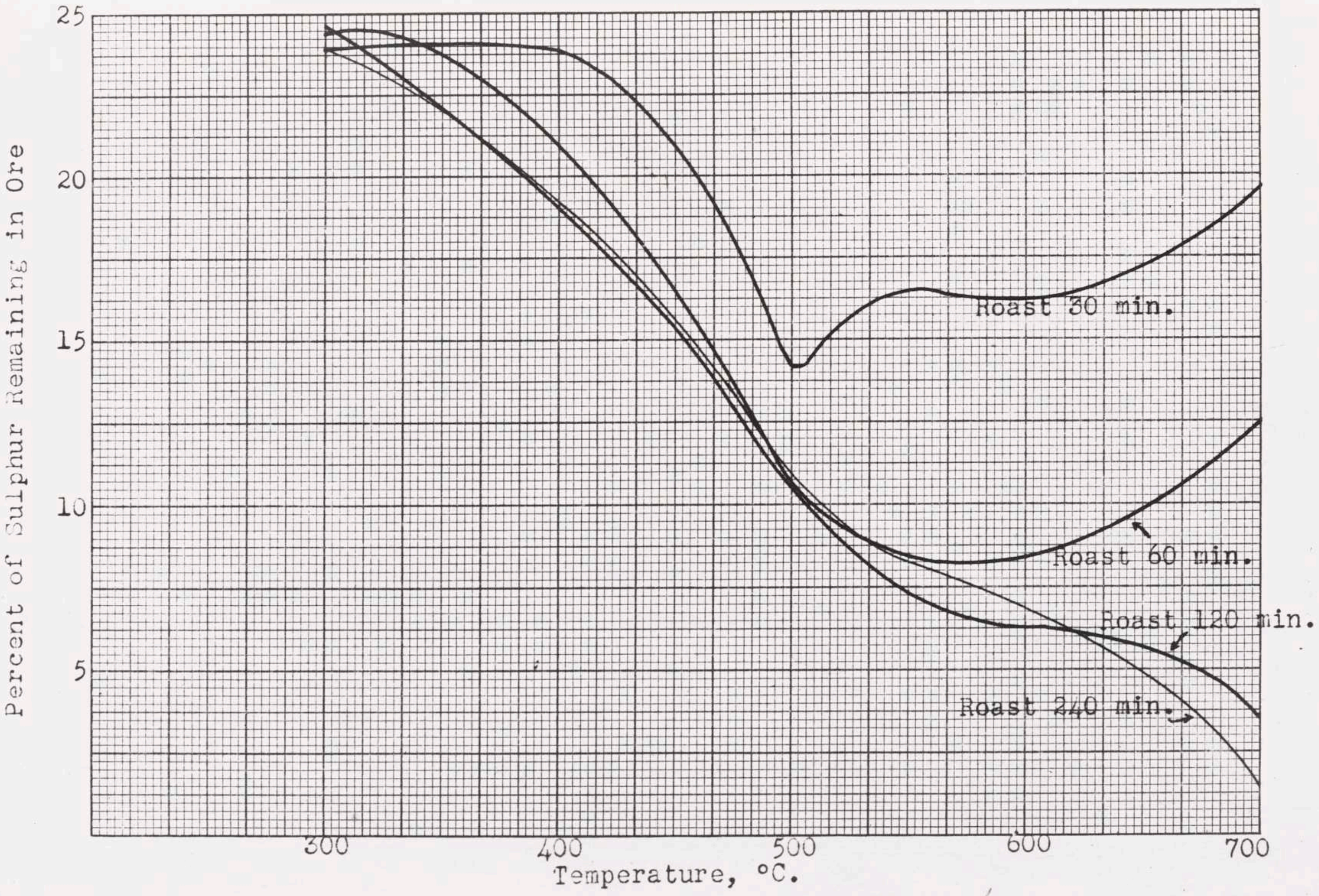

FIGURE 3

Effect of Temperature on Sulphur Removal During Roasting for Different Times

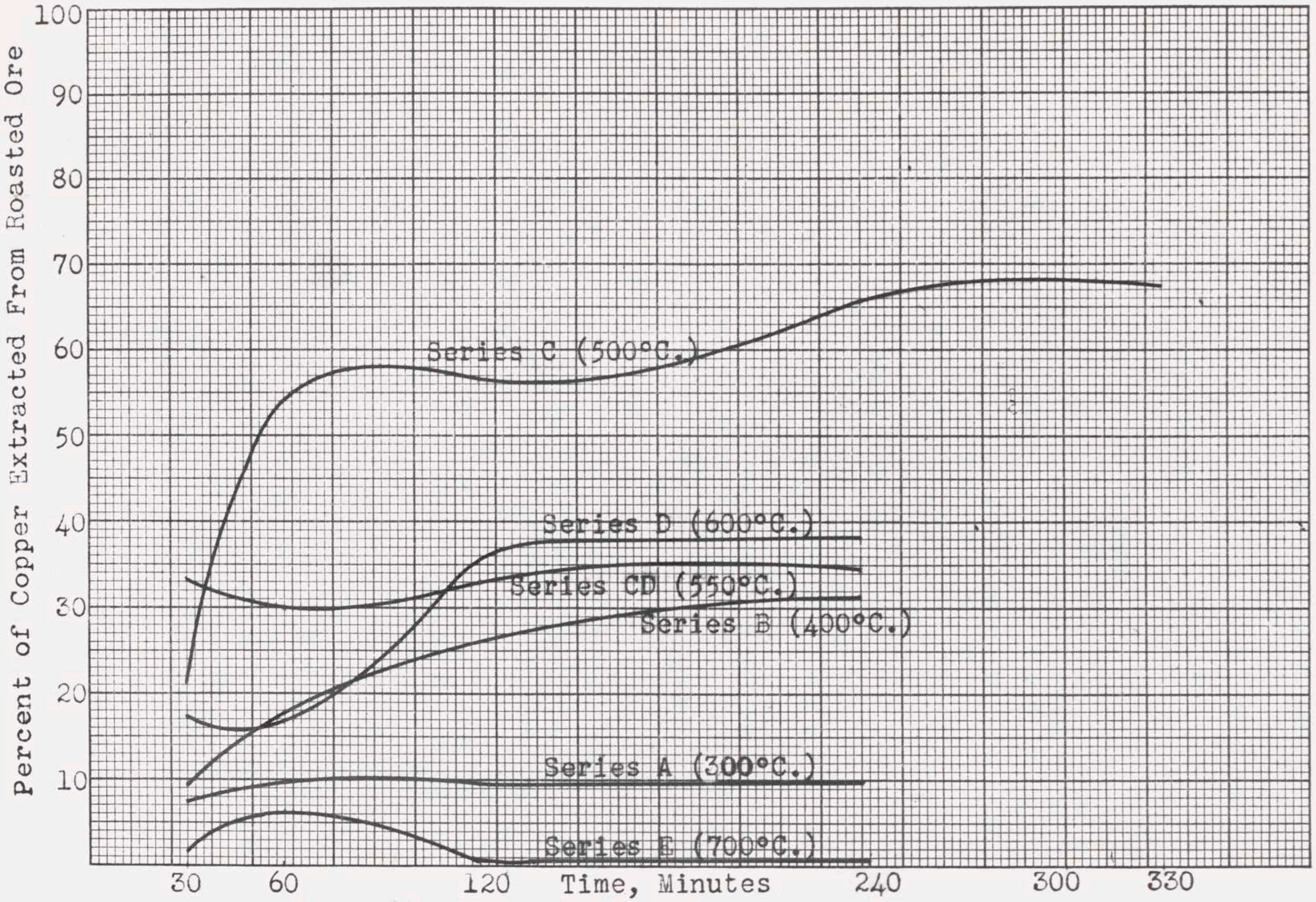

FIGURE 4

Effect of Time on Copper Extraction At Various Temperatures

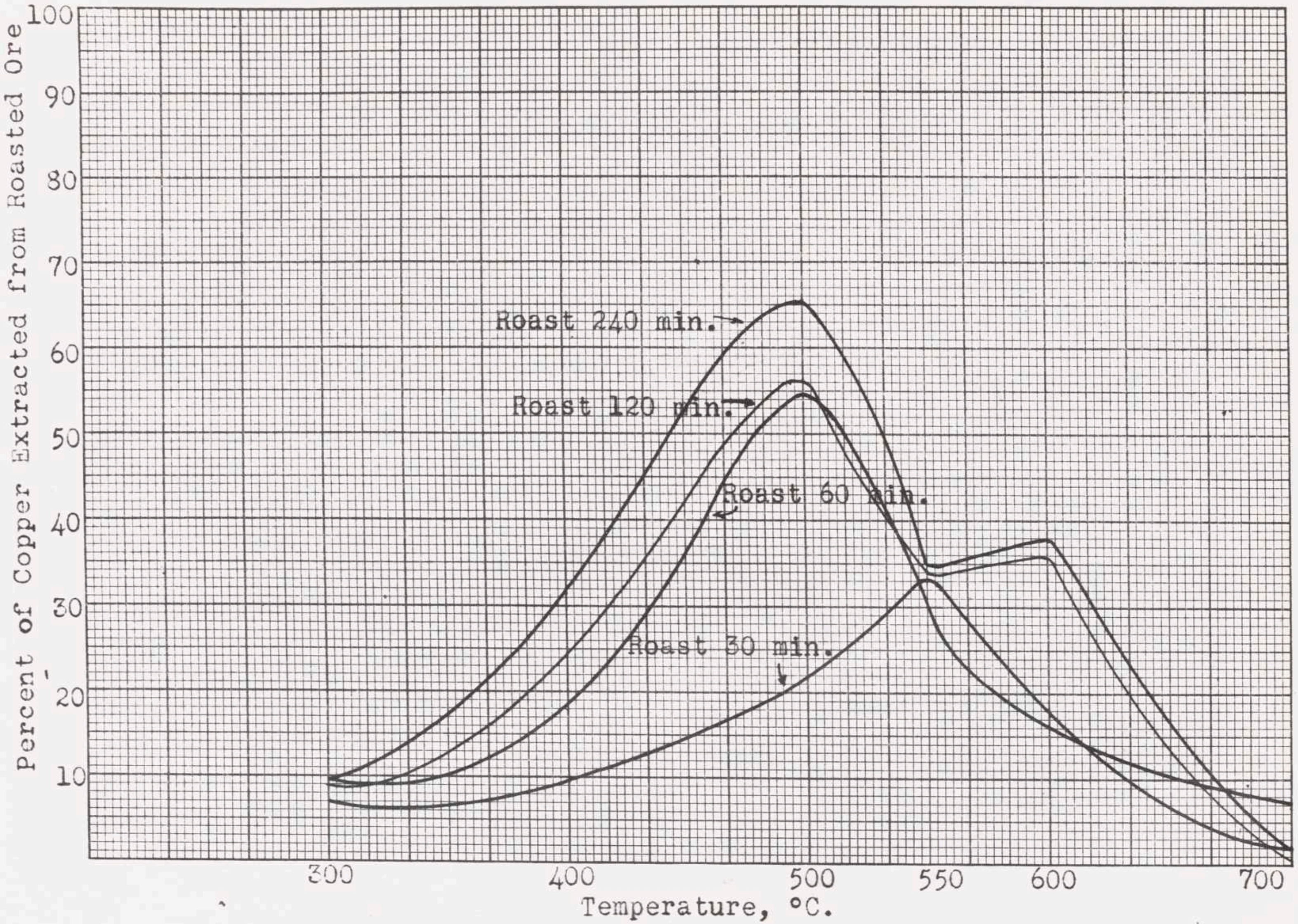

FIGURE 5

Effect of Temperature on Copper Extraction For Various Times

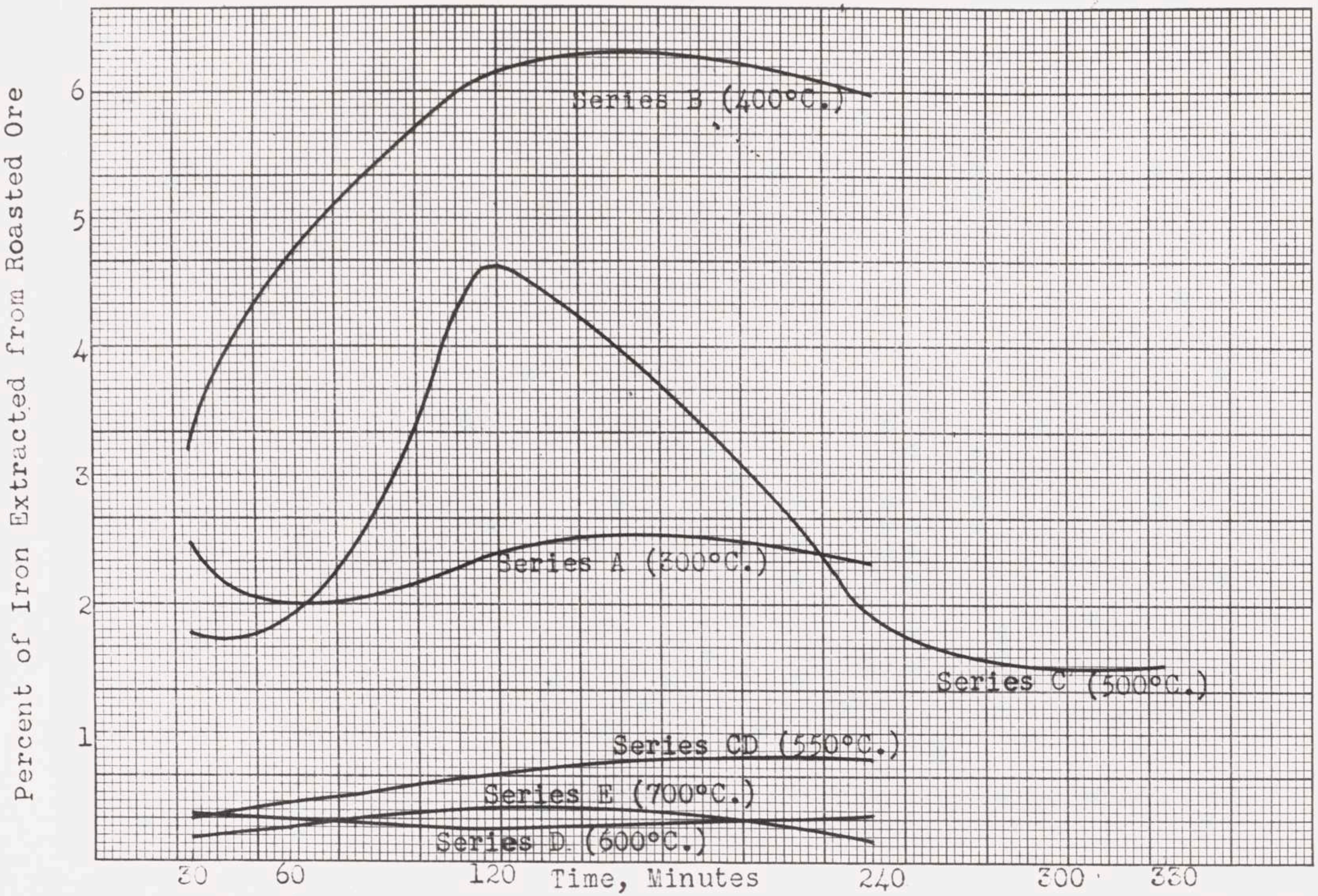

FIGURE 6

Effect of Time on Iron Extraction at Various Temperatures

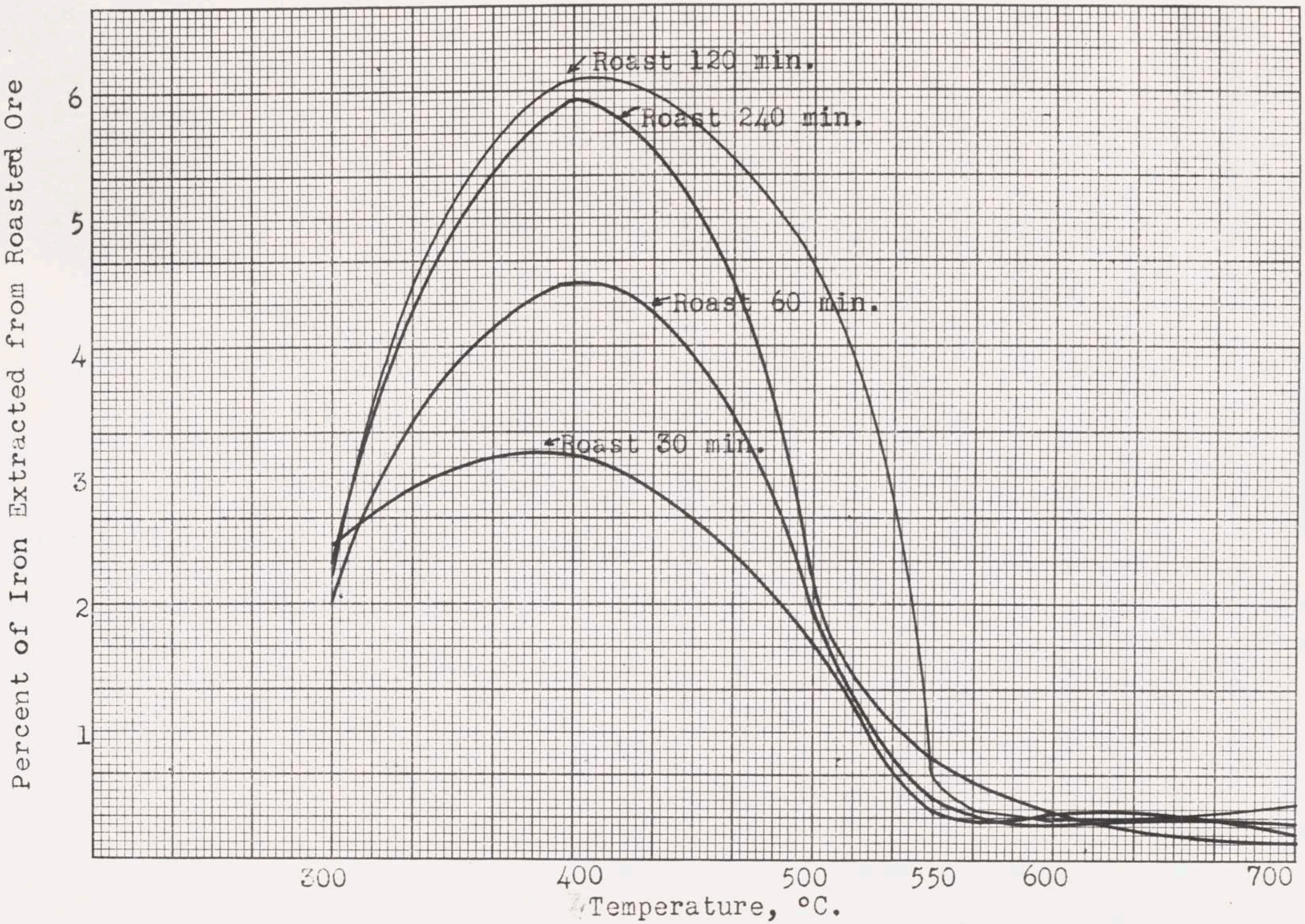

FIGURE 7

Effect of Temperature on Iron Extraction for Various Times

From the data and results of water leaching Sample C_4 was shown to contain the highest extraction of copper among all roasted ores under different conditions.

The water leached residue of Sample C_4 was leached by five per cent (by volume) sulphuric acid solution. The data and results are given in Table II.

Additional runs - Series CD and C_5-C_{55}- were made. Series CD gave no better results than Sample C_4 on the water leaching tests. The better extraction of copper on water leaching tests of C_5 and C_{55} were obtained. The data and results are shown in Table II.

The summary of the water and acid leaching results of Samples C_4, C_5 and C_{55} are given in Table III. No curves are necessary to plot for an explanation of the results of the acid leaching tests.

TABLE II

DATA AND RESULTS

Acid Leaching Tests on the Water Leached Residues of the Roasted Ore Samples C_4, C_5 and C_{55}

Samples No.	C_4	C_5	C_{55}
Weight of the water leached residue used, gms.	2.5	5	5
Percent of Cu. in the residue	12.75	11.70	12.00
Weight of Cu. in the residues, gms..	0.319	0.585	0.600
Leach solution (5 c.c. H_2SO_4 plus 95 c.c. H_2O), c.c.	100	100	100
Leach temperature, °C.	90-100	90-100	90-100
Leach time, hr.	1	1	1
Weight of the leach residues, gms...	1.51	3.466	3.470
Acid leach residue, percent Cu	3.94	3.55	3.50
insoluble Cu., gms	.0596	.1230	.1214
Percent Cu. extracted in leaching (Reference to the water leach residue)	81.0	79.0	80.0
Percent Cu. extracted in leaching (Reference to the roasted ore)	28.2	29.66	30.4
Fe. dissolved in leaching, gms.	.0713	.167	.168
Percent Fe. extracted in leaching ...	2.85	3.34	3.37
Ratio of Fe. and Cu. in leach solution	.274	0.36	0.35

TABLE III

Summary of the Water and Acid Leaching Results of Samples C_4, C_5 and C_{55}

Samples No.	Roast time, min.	Roast T, °C.	Leached	Cu. extracted percent	Fe. extracted percent	Fe. / Cu.
C_4	240	500	H_2O	65.5	1.94	.138
			5% H_2SO_4	28.2	2.85	.274
TOTAL				93.7		
C_5	300	500	H_2O	67.4	1.50	.107
			5% H_2SO_4	29.66	3.34	.36
TOTAL				97.06		
C_{55}	330	500	H_2O	66.6	1.53	.109
			5% H_2SO_4	30.4	3.37	.35
TOTAL				97.00		

III. DISCUSSION OF RESULTS

The data and results that have been recorded in the previous pages are discussed as follows:

(a) - Effect of time on copper extraction, on iron extraction and on sulphur removal during roasting at various temperatures.

Figure 4 showed the Copper extraction was not affected by the different times at 300°C.. Extraction was gradually increased by increasing the temperature. The maximum extraction obtained by roasting was for 240 min. at 500°C. and gradually decreased until 700°C.

Figure 6 showed the iron extraction was not affected by the different times at 300°C. Extraction gradually increased to a maximum as the temperature was raised to 400-430°C.

The iron extraction was quite low when the temperature was 550°C. or over.

Figure 2 showed the sulphur removal during roasting was not affected by the different times at 300°C. Amount of sulphur was decreased by increasing the temperature.

(b) - Effect of temperature on copper extraction, on iron extraction, and on sulphur removal during roasting at different times.

Figure 5 showed the copper extraction was affected by the various roasting temperatures for different times. The extraction was not increased by increasing the roasting time at 300°C. and 700°C. The extraction was gradually increased by increasing the roasting time up to 120 minutes at 400°C., 550°C. and 600°C. The extraction was nearly constant when the roasting time was increased at the above three temperatures. The extraction was suddenly increased when roasting time was increased from 30 to 60 minutes at 500°C. and still further increased up to 300 minutes, but no increase occurred beyond this time.

Figure 7 showed the iron extraction was affected by the various roasting temperatures for different times. The remarkable increase in extraction was at 400°C. and 500°C. The maximum extraction was obtained when the ore was roasted for 120 minutes.

Figure 3 showed the sulphur removal during roasting was affected by various temperature for different times, from 30 to 120 minutes except 300°C. and below. The sulphur in the ore was practically constant after 120 minutes roasting.

(c) - The results principally show that water and acid leaching of the sulphatizing ore will give a good extraction of copper and low percent soluble iron. The extraction is nearly as good as that obtained by smelting and by acid baking method.(1)

(d) - Duplication of results may be obtained under similar conditions. For example, the sample C_5 was successfully duplicated on a large-scale test to obtain material for further experimental work.

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(1) - C.F. Floe: Extraction of Copper from Roasted Concentrates by Sulphuric Acid Baking. A.I.M.E. Tech. Pub. 768 (1937).

V.

EXTRACTION OF COPPER AND PRECIOUS METALS (AU. AND AG.) BY CHLORIDIZING METHOD

1. EXPERIMENTAL METHOD

(a) - Chlorizing Roast

Two thousand gram samples "C_5" water and acid leached residue were needed for the chloridizing tests. The roasting furnace only contained 350 grams ore for each run. One hundred and fifty grams residue was obtained from 350 grams roasted ore after water and acid leaching. Therefore, fourteen 5-hour runs were made for this purpose.

The equipment and procedure for chlorizing roast were about the same as in sulphatizing roast. The difference was only mixing the roasting sample thoroughly with salt and rabbling the roasting sample every twenty minutes instead of fifteen minutes. About two hundred grams of "C_5" residue were used in each roast. Three samples were taken at various periods during each run.

NOTES:

Fumes of sulphur dioxide and chlorine were evolved shortly after the roast began in all roasts. The temperature of the furnace was suddenly increased by the evolution of gas. Therefore, it was necessary to control the heat for the roasting period.

The charge on roasting changed from light brownish red to the dirty brownish red with spots of green copper chloride.

Leaching

The procedure was the same as the leaching test described in sulphatizing extraction except the chloridizing leaching solution had the composition as follows:

H_2O = 100 c.c.

HCl = 5 c.c.

H_2SO_4 = 5 c.c.

$NaCl$ = 5 gms.

The leaching samples placed on the hot plate bump badly so the samples were leached on the steam plate for two hours at 70 to 80°C.

Assaying

After leaching, the residues were assayed for gold and silver as described in Bugbee's "A Textbook of Fire Assaying". The detailed procedures of assaying for sulphide and oxide ore are given in the Appendix.

The data and results of chlorizing roast, leaching and assaying are given in Table IV and Figures 8-9. More details of the experimental work may be obtained in the Appendix.

TABLE IV

Data and Results

Chloridizing the Sulphatizing Leached Residue at Various Temperatures and Various Times.

Series No.
Samples No.

ROASTING

Weight of Charg
leached residu
Percent re
Percent Na
Roasting Time,
Roasting sample
furnace, gms.
Analysis of cal
Percent Cu ..
Weight of gms

LEACHING

Weight of Calci
Leach solution
H_2O, 5 c.c. Hc
Leach temperatu
Leach time = 2

Weight of the l
Percent Cu. ext
(Reference to
residue), sol
per 1 gm. Cal
Percent Cu. ext
tilization (R
Cu. in residu
Weight of Cu.
per 1 gm. Ca
Total percent C
dizing test .

ASSAYING

Weight of the c
used = 1/2 A.T

Weight of Au. i
Weight of Ag. i
Percent of Au.
Percent of Ag.

TABLE IV

Chloridizing the Sulphatizing Leached Residue at Various Temperatures and Various Times.

DATA AND RESULTS

Series No.	M at 400°C.											
Samples No.	M_{12}	M_{13}	M_{14}	M_{22}	M_{23}	M_{24}	M_{32}	M_{33}	M_{34}	M_{42}	M_{43}	M_{44}
ROASTING												
Weight of Charge (sulphatizing leached residue* + NaCl), gms.	200	200	200	211	211	211	223.5	223.5	223.5	237	237	237
Percent residue in charge	95	95	95	90	90	90	85	85	85	80	80	80
Percent NaCl in charge	5	5	5	10	10	10	15	15	15	20	20	20
Roasting Time, hr.	2	3	4	2	3	4	2	3	4	2	3	4
Roasting sample draw from furnace, gms.	65	65	67.5	67	67	74	70	70	85	80	80	80
Analysis of calcine: Percent Cu	3.84	3.84	3.85	3.83	3.83	3.85	3.86	3.87	3.84	3.84	3.84	3.86
Weight of gms. Cu./1 gm. Calcine	.0384	.0384	.0385	.0383	.0383	.0385	.0386	.0387	.0384	.0384	.0384	.0386
LEACHING Weight of Calcine used = 40 gms. Leach solution (5 gms. NaCl, 100 c.c. H_2O, 5 c.c. Hcl, 5 c.c. H_2SO_4)= 160 cc. Leach temperature = 70-80°C. Leach time = 2 hrs.												
Weight of the leach residue, gms.	29.33	30.15	29.95	27.2	28.35	28.0	26.15	26.05	26.55	24.8	23.85	23.80
Percent Cu. extracted in leaching	100	100	100	100	100	100	100	100	100	100	100	100
(Reference to the sulphatizing leach residue), soluble Cu. in leach, gms. Cu. per 1 gm. Calcine	.0384	.0384	.0385	.0383	.0383	.0385	.0386	.0387	.0384	.0384	.0384	.0386
Percent Cu. extracted, or loss by volatilization (Reference to the percent of Cu. in residue used for chlorization)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Weight of Cu. by volatization, gms. Cu. per 1 gm. Calcine	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Total percent Cu. extracted from chloridizing test	100	100	100	100	100	100	100	100	100	100	100	100
ASSAYING Weight of the chloridizing leached residue used = 1/2 A.T. or 14.5083 gms.												
Weight of Au. in the residue, oz./ton	2.2	2.58	2.56	2.04	2.12	2.12	1.80	1.70	2.52	0.00	2.06	1.80
Weight of Ag. in the residue, oz./ton	10.48	9.86	11.56	6.68	6.44	10.88	5.84	5.60	3.26	0.00	4.62	3.32
Percent of Au. extracted, oz./ton	25.6	12.1	13.4	34.0	28.2	28.8	40.4	44.3	15.10	100	33.4	42.2
Percent of Ag. extracted, oz./ton	44.0	47.0	38.4	66.0	66.0	41.6	69.9	71.0	82.8	100	76.7	83.3

Series No.	N at 500°C.											
Samples No.	N_{12}	N_{13}	N_{14}	N_{22}	N_{23}	N_{24}	N_{32}	N_{33}	N_{34}	N_{42}	N_{43}	N_{44}
ROASTING												
Weight of Charge (sulphatizing leached residue* + NaCl), gms.	200	200	200	187.7	187.7	187.7	188.2	188.2	188.2	200	200	200
Percent residue in charge	95	95	95	90	90	90	85	85	85	80	80	80
Percent NaCl in charge	5	5	5	10	10	10	15	15	15	20	20	20
Roasting Time, hr.	2	3	4	2	3	4	2	3	4	2	3	4
Roasting sample draw from furnace, gms.	65	65	65	55	60	58	55	55	75	65	65	65
Analysis of calcine: Percent Cu	3.61	3.50	3.46	3.54	3.47	3.44	3.47	3.47	3.40	3.50	3.46	3.37
Weight of gms. Cu./1 gm. Calcine	.0361	.0350	.0346	.0354	.0347	.0344	.0347	.0347	.0340	.0350	.0346	.0337
LEACHING												
Weight of the leach residue, gms.	32.25	31.05	32.0	31.9	29.75	30.0	27.43	27.6	27.3	26.65	24.70	25.85
Percent Cu. extracted in leaching	94.0	91.4	90.1	92.2	90.4	89.6	90.9	90.9	88.6	90.9	90.1	87.7
(Reference to the sulphatizing leach residue), soluble Cu. in leach, gms. Cu. per 1 gm. Calcine	.0361	.0350	.0346	.0354	.0347	.0344	.0349	.0349	.0340	.0350	.0346	.0337
Percent Cu. extracted, or loss by volatilization (Reference to the percent of Cu. in residue used for chlorization)	6.0	8.6	9.9	7.8	9.6	10.4	9.1	9.1	11.4	9.1	9.9	12.3
Weight of Cu. by volatization, gms. Cu. per 1 gm. Calcine	.0023	.0034	.0038	.0030	.0037	.0040	.0035	.0035	.0044	.0035	.0038	.0047
Total percent Cu. extracted from chloridizing test	100	100	100	100	100	100	100	100	100	100	100	100
ASSAYING												
Weight of Au. in the residue, oz./ton	1.12	0.20	0.68	2.12	1.86	2.08	2.18	1.60	2.50	3.64	1.80	2.16
Weight of Ag. in the residue, oz./ton	10.02	10.40	7.52	3.00	7.68	5.96	224.34	4.76	7.40	2.84	2.70	0.26
Percent of Au. extracted, oz./ton	5.77	93.0	75.6	19.2	33.8	25.4	24.3	81.3	13.6	0.00	39.8	24.4
Percent of Ag. extracted, oz./ton	42.5	44.0	57.4	52.0	57.2	66.4	0.00	73.8	60.0	80.0	86.0	86.0

Series No.	O at 600°C.											
Samples No.	O_{12}	O_{13}	O_{14}	O_{22}	O_{23}	O_{24}	O_{32}	O_{33}	O_{34}	O_{42}	O_{43}	O_{44}
ROASTING												
Weight of Charge (sulphatizing leached residue* + NaCl), gms.	158	158	158	187.7	187.7	187.7	188.2	188.2	188.2	200	200	200
Percent residue in charge	95	95	95	90	90	90	85	85	85	80	80	80
Percent NaCl in charge	5	5	5	10	10	10	15	15	15	20	20	20
Roasting Time, hr.	2	3	4	2	3	4	2	3	4	2	3	4
Roasting sample draw from furnace, gms.	50	50	50	60	60	45	60	60	67	60	60	73
Analysis of calcine: Percent Cu	3.50	1.90	1.23	3.40	3.70	2.72	3.34	3.64	3.00	3.02	3.00	1.80
Weight of gms. Cu./1 gm. Calcine	.0350	.0190	.0123	.0340	.037	.0272	.0334	.0364	.0300	.0302	.0300	.0186
LEACHING												
Weight of the leach residue, gms.	31.4	31.85	32.6	29.2	29.6	30.15	26.45	27.2	27.1	24.84	25.0	24.8
Percent Cu. extracted in leaching	91.1	49.9	32.0	88.8	91.1	70.8	87.0	92.2	78.2	78.7	78.2	48.4
(Reference to the sulphatizing leach residue), soluble Cu. in leach, gms. Cu. per 1 gm. Calcine	.0350	.019	.0123	.0340	.035	.0303	.0334	.0354	.0300	.0302	.0300	.0186
Percent Cu. extracted, or loss by volatilization (Reference to the percent of Cu. in residue used for chlorization)	8.9	50.1	68.0	11.2	8.9	29.2	13.0	7.8	21.8	21.3	21.8	51.6
Weight of Cu. by volatization, gms. Cu. per 1 gm. Calcine	.0034	.0194	.0261	.0044	.0034	.0081	.0050	.0030	.0084	.0082	.0084	.0198
Total percent Cu. extracted from chloridizing test	100	100	100	100	100	100	100	100	100	100	100	100
ASSAYING												
Weight of Au. in the residue, oz./ton	0.04	0.12	0.20	0.92	1.60	1.00	2.08	1.86	1.48	2.80	3.32	3.90
Weight of Ag. in the residue, oz./ton	8.36	4.20	4.52	10.0	10.32	8.32	4.20	7.26	4.72	2.40	0.18	0.06
Percent of Au. extracted, oz./ton	98.58	96.04	93.38	68.0	43.2	64.2	30.0	35.8	12.2	5.9	0.00	0.00
Percent of Ag. extracted, oz./ton	53.2	78.6	73.7	45.3	42.6	52.8	78.0	60.7	74.6	87.5	99.2	99.8

* Analysis of the Residue:

Cu. = 3.84%
Au. = 2.32 oz./ton
Ag. = 14.82 oz./ton

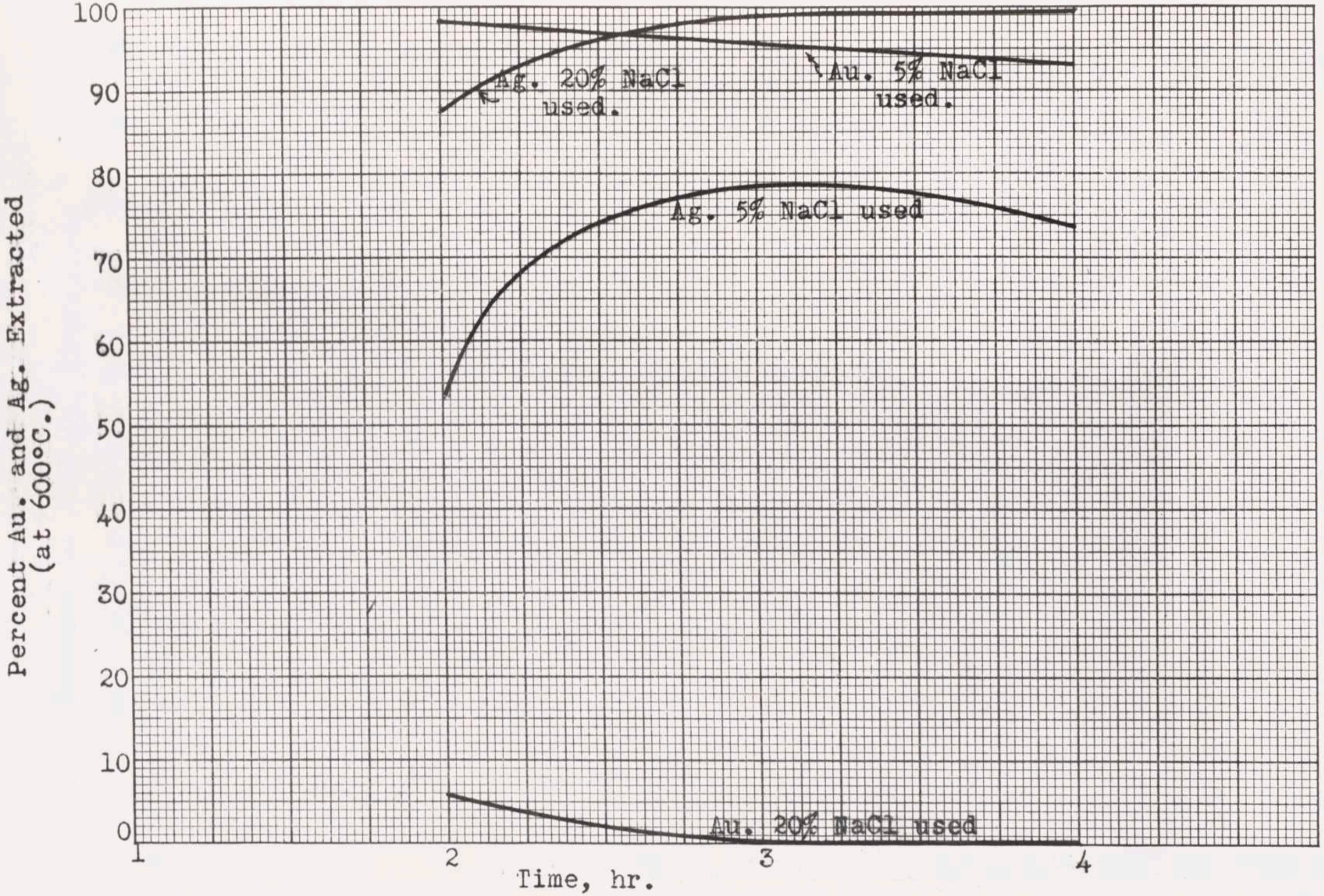

FIGURE 8

Effect of Percent NaCl used on Au. and Ag. at a Definite Temperature for Different Times.

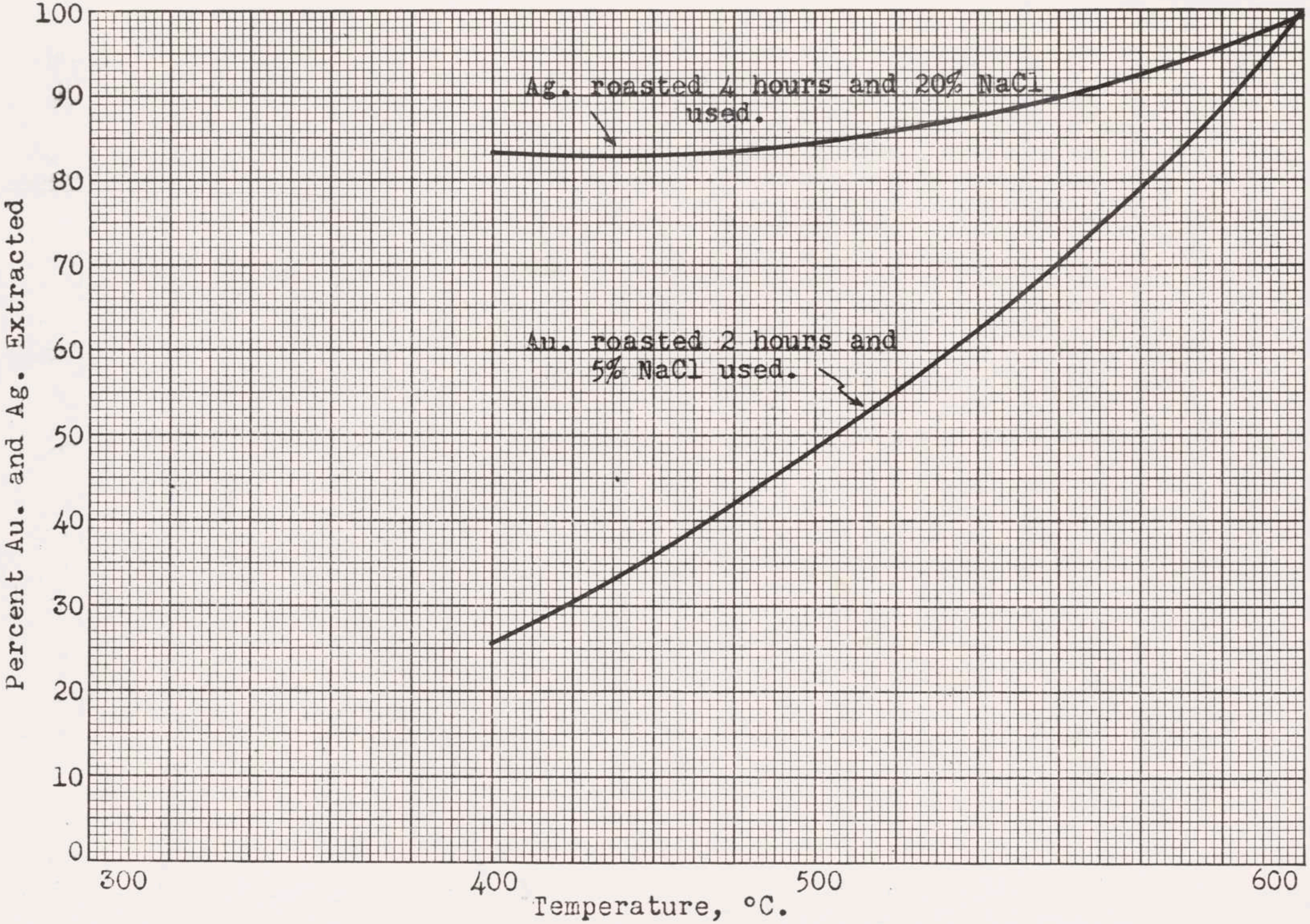

FIGURE 9

Effect of Percent NaCl used on Au. and Ag. Extraction at a Definite Times at Various Temperatures.

III. DISUCSSION OF RESULTS

The cyanidation test is not conveniently applied to the residue containing a few percent copper with gold and silver. The most logical method of treating them is by the chloridizing roast. The results show that a very good extraction of copper, gold and silver is readily obtained. The discussions of results are as follows:

(a) - By analysis of calcine for copper, it was found that there was no copper volatilization after roasting at 400°C. for different times and for different percent NaCl used. But the copper contained in the calcines was completely extracted by leaching.

The percent of volatilization of copper was obtained at 500°C. and 600°C. gradually increasing with the larger percent NaCl used. The copper remaining in the calcines was practically leached out.

(b) - In the assaying test of the residue for gold and silver, the large amount of silver obtained from Sample N_{32} was unbelievable. Possibly it came from the ore itself or the materials used for assaying. One hundred percent extraction of gold and silver from Sample M_{42} was quite doubtful until a duplication test was made to prove it. A very good extraction of gold and a reasonably good extraction of silver from Samples O_{12}, O_{13} and O_{14} of Series O were obtained. A very good extraction of silver and practically no extraction of gold were found in Samples O_{42}, O_{43} and O_{44} of Series O. Figure 8 is self-explanatory. Figure 9 shows that the percent extraction of gold was increased by 5% NaCl used and for two hour roasting at various temperatures. The maximum extraction of gold was obtained at 600°C. or a little higher. The percent extraction of silver was increased by increasing the temperature for four hours roasting and by using 20% NaCl. The maximum percent extraction was obtained at 600°C.

VI.

SUMMARY AND GENERAL CONCLUSIONS

(1) - Ninety-seven percent extraction of copper from sulphatizing roasted concentrates can be obtained by first water leaching followed by 5% H_2SO_4 leaching. The ore has to be roasted at 500°C. for five hours.

(2) - Under the above conditions, only 1.5 percent iron per one gram calcine is soluble by water leaching. The ratio of iron and copper in the water leached solution is 0.107. 3.34 percent iron per one gram calcine is soluble by acid leaching. The ratio of iron and copper in the acid leached solution is 0.36. This amount of soluble iron is not harmful to the electrolysis of copper from the leached solutions.

(3) - From three to four percent of copper remaining in the water and acid leached residue treated by chloridizing roast can be extracted completely by leaching, or by leaching and volatilizing. The roasting conditions are given in Table IV.

(4) - Gold and silver can be recovered from the leached residues by chloridizing. 98.58 percent gold can be extracted by chloridizing at 600°C. for two hours and by using 5% NaCl. At the same time 53.2 percent silver can be extracted. 99.8 percent silver can be extracted by chloridizing at 600°C. for four hours, and by using 20% NaCl. Practically no gold is extracted under the same conditions.

This experiment shows that the total extraction of copper is 100 percent, the maximum extraction of gold is 98.58 percent, and the maximum extraction of silver is 99.8 percent.

The sample can be duplicated at any time under the same conditions by careful control.

If enough interest will be created in this process to compete with the smelting, the next step necessary is the construction of a small laboratory plant in order to duplicate the results similar to those obtained in practice. Then a pilot-plant should be built to check carefully the process before erecting a big plant for commercial-scale production.

VII.

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VIII.

APPENDIX

I.-

II. -

Appendix I

TABLE V

Detailed Results of Sulphatizing Roasting and H_2O Leaching of Concentrates

Series No.	A at 300°C.				B at 400°C.				C at 500°C.			
Samples No.	A_1	A_2	A_3	A_4	B_1	B_2	B_3	B_4	C_1	C_2	C_3	C_4
ROASTING												
Charge = 250 gms.												
Rabble every 15 min.												
Time of roasting, min.	30	60	120	240	30	60	120	240	30	60	120	240
Wt. Calc. draw from furnace, gms.	50	50	50	96	50	50	50	109	50	50	50	104
Calc. analysis (.5 gm. sample)												
Wt. Cu. in 1 gm. Calc., gms.	.228	.228	.224	.232	.224	.228	.213	.213	.237	.218	.215	.214
%Cu. in Calcine	22.8	22.8	22.4	23.2	22.4	22.8	21.3	21.3	23.7	21.8	21.5	21.4
Wt. S. in 1 gm. Calc., gms.	.238	.244	.246	.238	.239	.211	.191	.191	.140	.106	.105	.107
%S. in Calcine	23.8	24.4	24.6	23.8	23.9	21.1	19.1	19.1	14.0	10.6	10.5	10.7
LEACHING												
Sample = 10 gms.												
Temp. = 90-100°C.												
Time = 1 hr.												
H_2O = 100 c.c.												
Wt. residue after leaching, gms........	8.86	8.85	8.84	8.90	8.56	7.64	6.69	6.45	8.23	6.23	5.93	5.80
Analysis of Residue: (.5 gm. sample)												
Wt. Cu. in 1 gm. Res., gms.	.239	.236	.23	.234	.237	.245	.23	.22	.227	.159	.141	.128
%Cu. in 1 gm. residue...	23.9	23.6	23.0	23.4	23.7	24.5	23.0	22.1	22.7	15.9	14.1	12.8
Tot. Cu. in tot. res., gms.	2.12	2.06	2.03	2.08	2.02	1.87	1.61	1.45	1.86	0.99	0.84	0.74
%Cu. extracted in leach soln	7.0	9.7	9.1	9.4	9.4	18.0	24.5	31.9	21.5	54.8	56.2	65.5
cc. soln. after leaching.	100	100	100	125	110	110	110	100	125	135	110	135
Analysis of the Leaching Soln:												
Wt. Fe. in 1 gm. Calc. in leach solution, gms.......	.025	.020	.024	.023	.032	.046	.061	.060	.017	.019	.047	.019
Percent Fe................	2.5	2.0	2.4	2.3	3.2	4.6	6.1	5.0	1.7	1.9	4.7	1.9
$\%SO_4$ in the solution	6.78	5.36	5.98	6.49	6.29	5.62	5.83	7.09	5.71	6.40	8.01	8.46
%S. as SO_4 in the soln....	2.26	1.78	2.74	2.98	2.89	2.57	2.67	3.25	2.62	2.93	3.67	3.98
Ratio Fe. and Cu. in leach solution	1.55	0.91	1.10	1.08	1.45	1.1	1.18	0.88	0.32	0.16	0.39	0.14

TABLE V (con'd)

Series No.	D at 600°C.				E at 700°C.				CD* at 550°C.				at 500°C.	
Samples No.	D_1	D_2	D_3	D_4	E_1	E_2	E_3	E_4	CD_1	CD_2	CD_3	CD_4	C_5	C_{55}
ROASTING														
Charge = 250 gms.														
Rabble every 15 min.														
Time of roasting, min.	30	60	120	240	30	60	120	240	30	60	120	240	300	330
Wt. Calc. draw from furnace, gms.	50	50	50	83	50	50	50	65	50	50	50	83	100	110
Calc. analysis(.5gm.sample)														
Wt.Cu. in 1 gm.Calc.,gms.	.254	.250	.232	.232	.255	.257	.243	.254	.252	.246	.228	.216	.210	.211
%Cu. in Calcine	25.4	25.0	23.2	23.2	25.5	25.7	24.3	25.4	25.2	24.6	22.8	21.6	21.0	21.1
Wt.S. in 1 gm.Calc.,gms.	.161	.083	.065	.070	.195	.125	.035	.013	.165	.085	.068	.085	.097	.096
%S. in Calcine	16.1	8.3	6.5	7.0	19.5	12.5	3.5	1.3	16.5	8.5	6.8	8.5	9.7	9.6
LEACHING														
Sample = 10 gms.														
Temp. = 90-100°C.														
Time = 1 hr.														
H_2O = 100 c.c.														
Wt. residue after leaching, gms.	9.29	8.79	8.16	7.92	9.62	9.50	9.65	9.78	7.01	7.32	8.85	9.37	5.87	5.77
A nalysis of Residue (.5 sample)														
Wt.Cu. in 1 gm.Res.,gms.	.227	.239	.183	.18	.261	.259	.252	.255	.241	.235	.172	.15	.117	.12
%Cu. in 1 gm. residue...	22.7	23.9	18.3	18.0	26.1	25.9	25.2	25.5	24.1	23.5	17.2	15.0	11.7	12.0
Tot.Cu. in tot.res., gms.	2.10	2.10	1.49	1.45	2.50	2.40	2.42	2.49	1.68	1.72	1.52	1.41	1.37	1.39
%Cu.extracted in leach sol.	7.3	16.0	35.8	37.9	1.96	6.6	.41	1.97	33.1	30.0	33.2	34.7	67.4	67.2
cc. soln. after leaching..	125	110	135	110	125	110	135	110	100	100	100	100	150	150
Analysis of the Leaching Soln.														
Wt.Fe. in 1 gm.Calc. in leach solution, gms.	.038	.035	.027	.037	.019	.025	.041	.016	.035	.046	.066	.080	.015	.015
Percent Fe	.38	.35	.27	.37	.19	.25	.41	.16	.35	.46	.66	.80	1.5	1.5
%SO_4 in the solution	4.18	7.36	9.15	1.14	1.26	1.96	3.37	1.81	2.99	6.80	16.5	18.4	12.8	12.78
%S. as SO_4 in the soln....	1.93	3.41	4.19	0.52	0.57	0.89	1.54	0.60	.997	2.27	5.5	6.14	4.28	4.28
Ratio Fe. and Cu. in leach solution	.09	.09	.03	.04	0.47	.14	.41	.32	.04	.06	.09	.11	.11	.11

* Additional sulphatizing test on

° C_5 to C_{55} total charge = 200 gms.

Appendix I

TABLE VI

Acid Leaching of Water Leached Residue
Sample C_4, C_5, C_{55}

Sample No.	C_4	C_5	C_{55}
Total weight of the residue after H_2O leaching, gms.	5.8	5.77	5.77
Weight of sample, gms.	2.5	5.0	5.0
Leach temperature, °C.	90-100	90-100	90-100
Leach time, hr.	1	1	1
Leach solution ($5\%H_2SO_4+95\%H_2O$), c.c.	100	100	100
Weight of residue after acid leaching, gms	1.51	3.47	3.47
Volume of solution after acid leach, c.c.	100	100	100
Analysis of residue (1 gm. sample)			
Weight of Cu. in 1 gm. residue, gm....	.0394	.0355	.0350
Percent Cu. in 1 gm. residue, gm.	3.94	3.55	3.50
Percent Cu. extracted in leaching.....	28.2	29.66	30.4
Analysis of Leach Solution (sample= 10 cc)			
Weight of Fe. extracted in leaching per 1 water leach residue, gms.	.0285	.0334	.0334
Percent Fe. extracted in leaching per 1 water leach residue, gms.	2.85	3.34	3.34
Ratio of Fe. and Cu. in leach solution ..	.274	.360	.350

Appendix I

TABLE VII

Roasted Ore Dissolved in H_2O and Acid Leaching
Sample C_5

Charge, gms.	20	340	340
Weight of H_2O leach residue, gm.	11.74	195	182
Weight of 5% H_2SO_4 leach residue, gms	8.1	140	141
Percent ore dissolved in H_2O leaching	41.3	42.6	46.5
Percent ore dissolved in acid leaching	18.4	16.3	12.1
Total ore dissolved in H_2O and acid leaching	60	59	58.5

Appendix I

TABLE VIII

Data and Results of Copper* Extraction from Chloridizing Test

	Chloridizing Calcine Wt.Cu. per gm.	Chloridizing Volatization Wt.Cu./gm.	Chloridizing Volatization %Cu.	Wt.Cu. extracted in leach soln.	% Cu. extracted in leach soln.	Total % Cu. extracted volatization & leaching
M_{12}	.0384	.000	0.00	.0384	100	100
M_{13}	.0384	.000	0.00	.0384	100	100
M_{14}	.0384	.000	0.00	.0384	100	100
M_{22}	.0383	.000	0.00	.0384	100	100
M_{23}	.0383	.000	0.00	.0384	100	100
M_{24}	.0384	.000	0.00	.0384	100	100
M_{32}	.0384	.000	0.00	.0384	100	100
M_{33}	.0384	.000	0.00	.0384	100	100
M_{34}	.0384	.000	0.00	.0384	100	100
M_{42}	.0384	.000	0.00	.0384	100	100
M_{43}	.0384	.000	0.00	.0384	100	100
M_{44}	.0384	.000	0.00	.0384	100	100

continued

* Sample used:
= .0384 gm. Cu. per 1 gm. sample

TABLE VIII (con'd)

Data and Results of Copper* Extraction from Chloridizing Test

	Chloridizing Calcine Wt. Cu. per gm.	Chloridizing Volatization Wt.Cu./gm.	Chloridizing Volatization %Cu.	Wt.Cu. extracted in leach soln.	%Cu. extracted in leach soln.	Total % Cu. extracted volatization & leaching
N_{12}	.0361	.0023	6.0	.0361	94.0	100
N_{13}	.0350	.0034	8.6	.0350	91.4	100
N_{14}	.0346	.0038	9.9	.0346	90.1	100
N_{22}	.0354	.0030	7.8	.0354	92.2	100
N_{23}	.0347	.0037	9.6	.0347	90.4	100
N_{24}	.0344	.0040	10.4	.0344	89.6	100
N_{32}	.0349	.0035	9.1	.0344	90.9	100
N_{33}	.0349	.0035	9.1	.0347	90.9	100
N_{34}	.0340	.0044	11.4	.0340	88.6	100
N_{42}	.0350	.0035	9.1	.0350	90.9	100
N_{43}	.0346	.0038	9.9	.0346	90.1	100
N_{44}	.0337	.0047	12.3	.0337	87.7	100

continued

* Sample used:
= .0384 gm. Cu. per 1 gm. sample

Appendix I

TABLE VIII (con'd)

Data and Results of Copper* Extraction from Chloridizing Test

	Chloridizing Calcine Wt. Cu. per gm.	Chloridizing Volatization Wt.Cu./gm.	Chloridizing Volatization %Cu.	Wt.Cu. extracted in leach soln.	% Cu. extracted in leach soln.	Total % Cu. extracted volatization & leaching
O_{12}	.0350	.0034	8.9	.0350	91.1	100
O_{13}	.0190	.0194	50.1	.0190	49.9	100
O_{14}	.0123	.0261	68.0	.0123	32.0	100
O_{22}	.0340	.0044	11.2	.0340	88.8	100
O_{23}	.0350	.0034	8.9	.0350	91.1	100
O_{24}	.0303	.0081	29.2	.0303	70.8	100
O_{32}	.0354	.0050	13.0	.0334	87.0	100
O_{33}	.0364	.0030	7.8	.0354	92.2	100
O_{34}	.0300	.0084	21.8	.0300	78.2	100
O_{42}	.0302	.0082	21.3	.0302	78.7	100
O_{43}	.0300	.0084	21.8	.0300	78.2	100
O_{44}	.0186	.0198	51.6	.0386	48.4	100

* Sample used:
= .0384 gm. Cu. per 1 gm. sample

Appendix I

TABLE IX

Data and Results of Au. and Ag.* Extraction from Chloridizing Test

Series No.	M											
Samples No.	M_{12}	M_{13}	M_{14}	M_{22}	M_{23}	M_{24}	M_{32}	M_{33}	M_{34}	M_{42}	M_{43}	M_{44}
Sample used for leaching (contained salt)	= 40 gms.											
Ex.Wt. Calcine used for leaching ...	38	38	38	36	36	36	34	34	34	32	32	32
Wt.Au. in exact wt. calc.,oz./ton...	3.027	3.027	3.027	2.87	2.87	2.87	2.71	2.71	2.71	2.54	2.54	2.54
Wt.Ag. in exact wt. calc.,oz./ton...	19.3	19.3	19.3	18.3	18.3	18.3	17.3	17.3	17.3	16.3	16.3	16.3
Wt. leached residue, gms.	29.83	30.15	29.95	27.2	28.35	28.0	26.15	26.05	26.55	24.8	23.85	23.80
Wt.Au. in leached res.,oz./ton....	2.2	2.58	2.56	2.04	2.12	2.12	1.80	1.7	2.52	0.0	2.06	1.80
Tot.Wt.Au. in tot. leach.res.,oz/ton	2.26	2.66	2.62	1.9	2.06	2.04	1.62	1.52	2.3	0.00	1.69	1.47
Wt.Au. extracted from 40 gm. sample oz./ton	.772	.367	.407	.97	.81	.83	1.095	1.20	0.41	2.54	0.85	1.07
Percent Au. extracted	25.6	12.1	13.4	34.0	28.2	28.8	40.4	44.3	15.1	100	33.4	42.2
Wt.Ag. in leached residue,oz/ton.	10.46	9.86	11.56	6.68	6.44	10.88	5.84	5.60	3.26	0.00	4.62	3.32
Tot.wt.Ag. in tot. leach res,oz/ton	10.79	10.24	11.9	6.25	6.26	10.47	5.24	5.01	29.7	0.00	3.80	2.72
Wt.Ag. extracted from 40 gm.sample oz./ton	8.51	9.06	7.4	12.05	12.04	7.63	12.06	12.29	14.33	16.3	12.5	13.58
Percent Ag. extracted	44	47	38.4	66	66	41.6	69.9	71	82.8	100	76.7	83.3

* Sample used: Au. ...... 2.32 oz./ton
Ag. ...... 14.82 oz/ton

TABLE IX (con'd)

Data and Results of Au. and Ag.* Extraction from Chloridizing Test

Series No.						N						
Samples No.	N12	N13	N14	N22	N23	N24	N32	N33	N34	N42	N43	N44
Sample used for leaching = 40 gms. (contained salt)												
Ex.Wt.Calcine used for leaching...	38	38	38	36	36	36	34	34	34	32	32	32
Wt.Au. in exact wt. calc.,oz./ton..	3.027	3.027	0.027	2.87	2.87	2.87	2.71	2.71	2.71	2.54	2.54	2.54
Wt.Ag. in exact wt. calc.,oz./ton..	19.3	19.3	19.3	18.3	18.3	18.3	17.3	17.3	17.3	16.3	16.3	16.3
Wt. leached residue, gms.......	32.25	31.05	32.00	31.90	29.75	30.0	27.43	27.60	27.30	26.65	24.70	25.85
Wt.Au. in leached res.,oz./ton...	1.12	0.20	0.68	2.12	1.86	2.08	2.18	1.60	2.5	3.64	1.80	2.16
Tot.Wt.Au. in tot. leach.res.,oz/ton	1.28	0.214	0.746	2.32	1.90	2.14	2.05	1.51	2.34	3.44	1.53	1.92
Wt.Au. extracted from 40 gm. sample oz./ton	1.747	2.813	2.281	0.55	0.97	0.73	0.66	2.20	0.37	0.00	1.01	0.62
Percent Au. extracted.........	57.7	93.0	75.6	19.2	33.8	25.4	24.3	81.3	13.6	0.00	39.8	24.4
Wt.Ag. in leached residue,oz/ton.	10.0	10.4	7.52	8.0	7.68	5.96	2.24	4.76	7.4	2.84	2.7	0.26
Tot.wt.Ag. in tot. leach res,oz/ton	11.1	10.8	8.25	8.8	7.84	6.15	100	4.52	6.95	2.6	2.3	2.31
Wt.Ag. extracted from 40 gm. sample oz./ton	8.2	8.5	11.05	9.5	10.5	12.15	0.0	12.78	10.35	13.0	14.0	13.99
Percent Ag. extracted	42.5	44.0	57.4	52	57.2	66.4	0.0	73.8	60	80	86	86

TABLE IX (con'd)

Data and Results of Au. and Ag.* Extraction from Chloridizing Test

Series No.	0											
Samples No.	012	013	014	022	023	024	032	033	034	042	043	044
Sample used for leaching = 40 gms. (contained salt)												
Ex.Wt.Calcine used for leaching..	38	38	38	36	36	36	34	34	34	32	32	32
Wt.Au. in exact wt. calc.,oz./ton..	3.027	3.027	3.027	2.87	2.87	2.87	2.71	2.71	2.71	2.54	2.54	2.54
Wt.Ag. in exact wt. calc.,oz./ton..	19.3	19.3	19.3	18.3	18.3	18.3	17.3	17.3	17.3	16.3	16.3	16.3
Wt. leached residue, gms.......	31.4	31.85	32.6	29.2	29.6	30.15	26.45	27.2	27.1	24.84	25.0	24.8
Wt.Au. in leached res., oz./ton..	0.04	0.12	0.20	0.92	1.60	1.00	2.08	1.86	1.48	2.80	3.32	3.90
Tot.Wt.Au. in tot. leach.res,oz/ton	.043	.131	.224	.922	1.63	1.03	1.89	1.74	1.38	2.39	2.84	3.28
Wt.Au. extracted from 40 gm. sample oz./ton	2.987	2.896	2.803	1.948	1.24	1.84	0.82	0.97	0.33	0.15	0.00	0.00
Percent Au. extracted	98.58	96.04	93.38	68.0	43.2	64.2	30.0	35.8	12.2	5.9	0.00	0.00
Wt.Ag. in leached residue,oz/ton.	8.36	4.2	4.52	10.0	10.3	8.32	4.2	7.26	4.72	2.4	0.18	0.06
Tot.Wt.Ag. in tot. leach res,oz/ton	9.06	4.06	5.06	10.0	10.5	8.62	3.82	6.80	4.4	2.05	0.16	0.05
Wt.Ag. extracted from 40 gm. sample oz./ton.........	10.24	15.24	14.24	8.3	7.8	9.68	13.5	10.5	12.9	14.3	16.1	16.3
Percent Ag. extracted	53.2	78.6	73.9	45.3	42.6	52.8	78	60.7	74.6	87.5	99.2	99.8

II. CHEMICAL ANALYSIS

Short Iodide Method for Determining Copper in Concentrates and Other Products

(1) - Treat 0.5 gram sample in an 8-ounce Erlenmeyer flask with 10 c.c. Hcl and 5 c.c. HNO_3. Heat gently until the decomposition of the sample is complete, then add 4 c.c. H_2SO_4 and boil to almost complete dryness. Cool.

(2) - Add 50 c.c. of H_2O and heat to dissolve the assays. Add 5 c.c. bromine water and boil to expel.

Remove the boiling solution from the heat and add cautiously strong NH_4OH until ammoniacal, though no excess. Add 7 c.c. glacial acetic acid and boil for one-half minute. Cool.

(3) - Add 2 grams of sodium fluoride, 20 c.c. KI solution (50 gms. per liter). Titrate with standard sodium thiosulphate until the brown iodine color is faint; then add starch liquor (more) and continue the titrate until no further decolorization. Now add a little more KI to see whether the operation is actually finished.

Calculation

$$\text{c.c. } Na_2S_2O_3 \text{ x N of } Na_2S_2O_3 \text{ x } \frac{63.57}{1000} \text{ x } \frac{100\%}{\text{wt. of sample}} = \%\text{Cu.}$$

$$\text{c.c. } Na_2S_2O_3 \text{ x N of } Na_2S_2O_3 \text{ x } \frac{63.57}{1000} \text{ x } \frac{100\%}{\text{c.c. sample}} = \text{gm. Fe per liter.}$$

Standardization of $Na_2S_2O_3$ solution.

Make up a solution containing about 19.5 grams of $Na_2S_2O_3 \cdot 5\ H_2O$ per liter. Standardize this as follows: weigh 0.2 gram of pure copper-foil and place in an 8-ounce flask. Add 5 c.c. glacial acetic acid, which dissolves the copper. Dilute the solution and boil to expel the red fumes. Add ammonia in slight excess. Add about 5 c.c. of glacial acetic acid and boil the solution for a few minutes. Cool. Add 6 c.c. of a 50 per cent solution of KI and titrate with $Na_2S_2O_3$. solution.

Calculation

$$\frac{\text{No. of gms. of copper used}}{\text{No. of c.c. of } Na_2S_2O_3 \text{ used}} = \text{copper standard.}$$

Method for Determining Iron on Acid Solutions

(1)- 2 c.c. sample into 250 c.c. Erlenmeyer; add very little 1-1 HCl (none if solution has been acidified with HCl during leaching process.

(2) - Boil for few minutes -- very few, as the solution will boil to dryness in a very short time!! -- take off hot plate and immediately $SnCl_2$ dropwise, avoiding more than a slight excess. A color change will take place on the addition of not too much $SnCl_2$.

Cool. Add water until volume is about 100 c.c. Then add an excess of $HgCl_2$ -- a white silvery precipitate of Hg_2Cl_2 will form. Let stand three minutes and titrate with $KMnO_4$ to permanent pink color.

NOTE: If too much HCl has been added, the end point will disappear and it is best to do the experiment over if accurate results are to be obtained.

Calculations

$$\text{c.c. } KMnO_4 \text{ x N of } KMnO_4 \text{ x } \frac{55.8}{1000} \text{ x } \frac{1000}{\text{c.c. sample}} = \text{gm. Fe/liter}$$

$$\text{c.c. } KMnO_4 \text{ x N of } KMnO_4 \text{ x } \frac{55.8}{1000} \text{ x } \frac{100\%}{\text{wt. of sample}} = \%\text{Fe.}$$

Standard of $KMnO_4$ solution (3.160 gms. $KMnO_4$ per 1000 c.c. H_2O)

Weigh 0.20 gram of pure iron wire and dissolve in an 8-ounce flask in a mixture of 5 c.c. each of HNO_3 and HCl. When the wire has dissolved, add 5 c.c. of H_2SO_4 and boil to expel the HCl and HNO_3 acid and most of the sulphuric also. Add H_2O and heat to dissolve all compounds into the solution. Cool and titrate with $KMnO_4$ solution.

Calculation

$$\frac{\text{No. of gms. of Cu. used}}{\text{No. of c.c. of } KMnO_4 \text{ used}} = \text{Fe. standard}$$

The detailed description is given in Low's TECHNICAL METHODS OF ORE ANALYSIS.

Method for Determining Sulphur (over 15%) in Concentrates and Other Products

(1) - Weigh 0.5 gram sample into a 250 c.c. beaker; add 10 c.c. of water, 1 gram of $KClO_3$ and 20 c.c. of HNO_3.

Place on the hot plate to dry. Cool; add 10 c.c. of HCl and dry it again.

(2) - Add 10 c.c. of HCl and 75 c.c. of H_2O to boil the dry assays into solution. Filter. The filtrate is received in a 250 c.c. beaker.

(3) - Heat the filtrate to boiling point. Add 10 c.c. of hot $BaCl_2$ solution (100 gms./1000 c.c. H_2O) and boil for a few minutes.

(4) - Settle; filter and wash. Place the precipitate in a clay cup, ignite, cool, and weigh as $Ba\ SO_4$.

Calculation

Wt. of $BaSO_4$ x 0.1373 = wt. of S.

Method for Determining Sulphur as Sulphate in Copper Solutions

(1) - Pipette 10 c.c. of copper solution into a 250 c.c. beaker. Add a small amount of powdered Aluminum metal, and let stand until the solution is colorless and completely reduced.

(2) - Filter. Add a few c.c. of HCl and heat to boil the solution. Add 10 c.c. of $BaCl_2$ solution (100 gms./1000 c.c. H_2O) and boil a few minutes.

(3) - Settle, filter, wash, and place the precipitate in a crucible; ignite, cool and weigh as $BaSO_4$.

Calculation

Wt. of $BaSO_4$ x 0.4115 = Wt. of SO_4

Method of Assaying for Determining Gold and Silver

I. Flux used for 1/2 A.T. Ore

(1) -- Sulphide Ore

Ore	1/2 A.T. (=14.083 gms.)
Na_2CO_3	15 gms.
Borax glass	5 gms.
Litharge	120 gms.
Nitre	21 gms.
Silica	12 gms.

(2) -- Oxide Ore

Ore	1/2 A.T.
Na_2CO_3	15 gms.
Borax glass	6 gms.
Litharge	60 gms.
Flour	2.5 gms.
Silica	6.5 gms.

II. Procedure

(1) - Weigh and mix thoroughly the charge in the assay clay crucible (30-gram sizes).
Place the charge in the muffle furnace for about 45 minutes at about 900°C.

(2) - Pour the molten products into the crucible mold. Cool and remove the lead button (about 30 gms.) from slag. Pound the button to a square form.

(3) - Heat the cupel in a good hot furnace (about 800°C.). Drop the button quickly into the cupel. Lead is formed as oxide which goes into the air. Gold and silver are left in the cupel. Weigh the bead (Au. + Ag.).

(4) - Pound the bead and dissolve by weak parting acid (1 HNO_3: % H_2O) in a porcelain crucible. Heat it on a hot plate but do not boil. When the reaction ceases, all silver is dissolved into the acid solution. Decant the solution and add strong parting acid (1 HNO_3: 1 H_2O) to dissolve the silver again. Heat the solution for a few minutes but do not boil. Decant the solution, wash, dry and weigh the residue as gold.

<u>Calculation</u>(1)

$$\text{Wt. of Au.(mg.)} \times 2 = \text{Au. } \frac{\text{oz.}}{\text{ton}}$$

$$\text{Wt. of Ag.(mg.)} \times 2 = \text{Ag. } \frac{\text{oz.}}{\text{ton}}$$

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(1) - Bugbee, E.E.: Textbook of Fire Assaying, 86. New York, John Wiley & Sons, Inc.