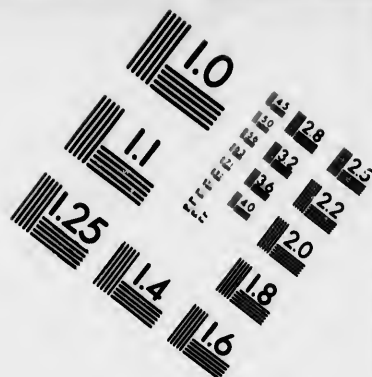
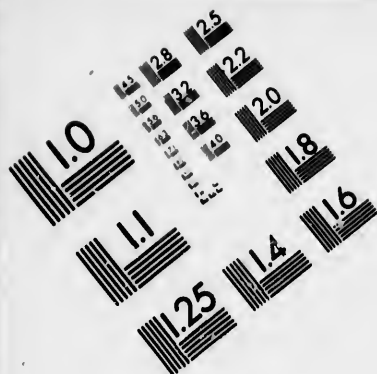
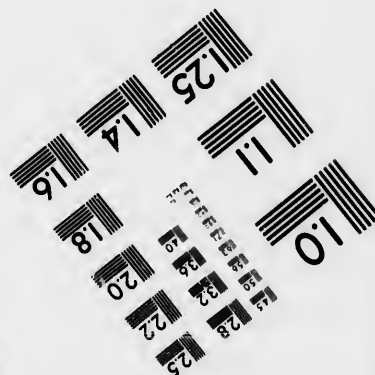
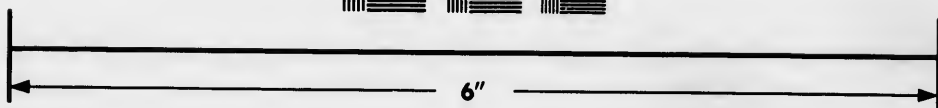
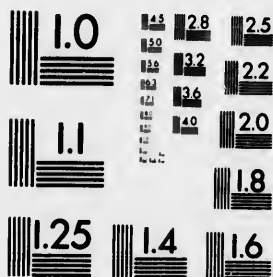




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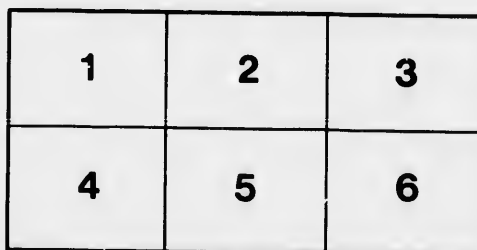
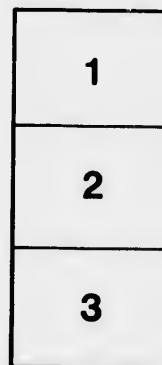
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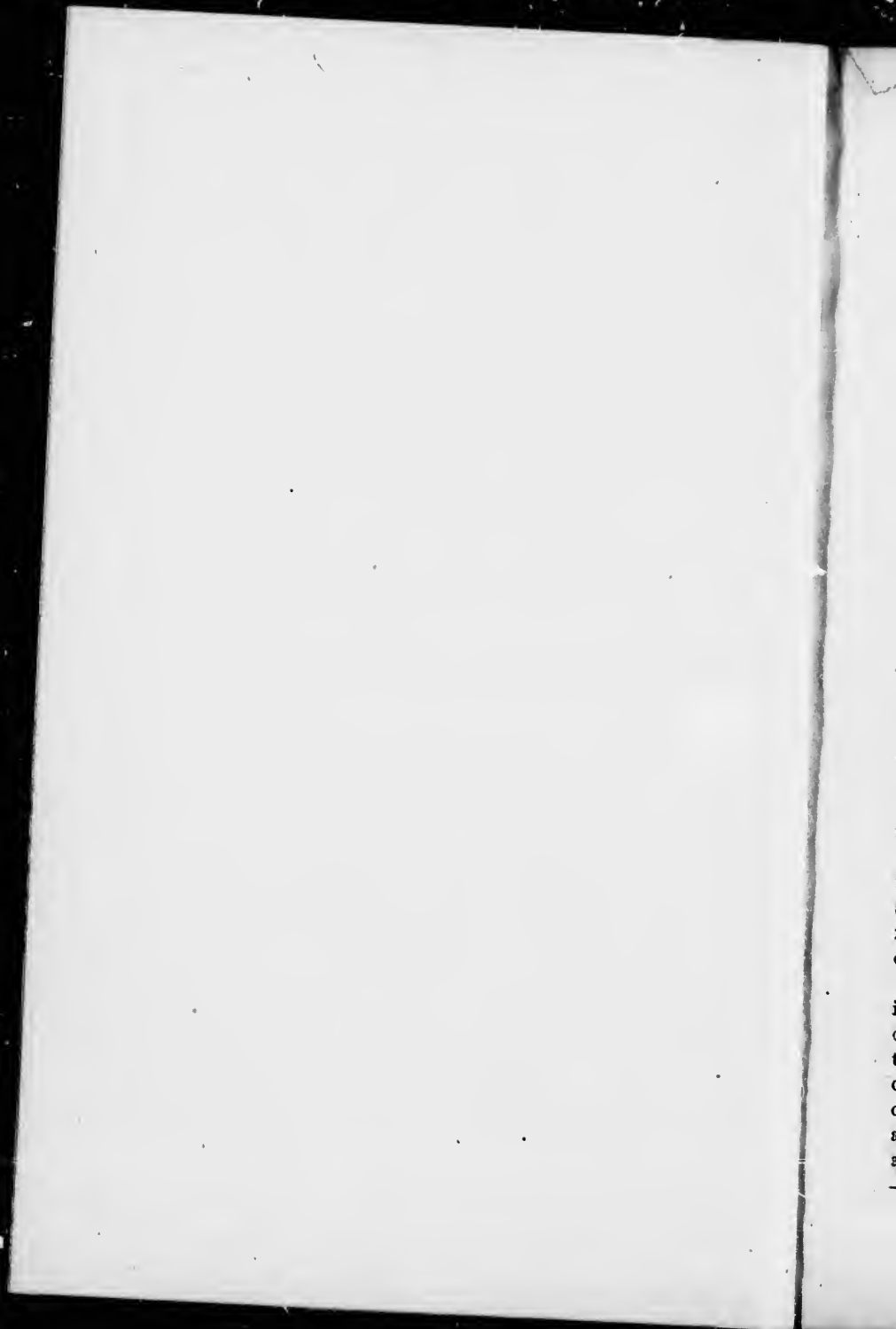
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ON THE EXTRACTION OF COPPER FROM ITS ORES IN THE HUMID WAY.

By THOMAS MACFARLANE.

From the Canadian Naturalist for June 1865.

In the last Report of the Geological Survey of Canada reference was made to the poor pyritous copper ores of the Eastern Townships; and with regard to the best method of utilizing them, it was remarked as follows: "It is much to be desired that some of the various methods which have been proposed for removing the copper in a soluble form, could be applied to these ores."* The importance of this question has by no means diminished, since the publication of the report alluded to. The recent discovery, in the neighborhood of Lennoxville, of several very promising beds of pyritiferous copper ore, the difficulty of concentrating these by any of the usual mechanical processes of ore-dressing, and the obstacles to the establishment of smelting-houses near to the mines for the production of ingot copper, all combine to render this subject one of somewhat more than ordinary interest.

The processes which have been from time to time proposed, and put into practical operation, for the humid treatment of copper ores are so numerous and diverse, that I shall not attempt to describe them minutely. I shall merely refer to some of the more important among them, and especially to those which bear some resemblance in principle to the method pursued in certain experiments which I have performed on a small scale, with various Canadian ores, in order to the extraction of the copper contained in them. The results of these experiments having been very satisfactory, I shall proceed to describe them, and, in conclusion, refer to the manner in which the method of extraction founded on them might be most advantageously carried out on a large scale.

1. One of the oldest modes of producing copper in the wet way is the precipitating it, by means of metallic iron, from the water of mines, or that resulting from lixiviating old waste-heaps in their neighborhood. These waters contain the copper in the form of sulphates, derived doubtless from the oxidation of sulphureted ores. This process is or was not long ago carried on at Schmöllnitz and Neusohl in Hungary, at Moldava in Croatia, in Anglesea, and at Rammelsberg in the Lower Hartz.

* Geology of Canada, page 736.

2. Closely allied to this process is the one still followed at Faldal in Norway, where an artificial oxidation takes the place of the natural oxidation of similar ores. They are roasted in heaps, and then lixiviated.

3. At Stadtbergen in Westphalia, and at Linz on the Rhine, the vapors evolved in roasting various sulphurets are brought into contact with poor ores containing malachite, and with oxidized ores containing cupric oxide. After these have been acted on for several weeks, they are lixiviated in the usual manner.

4. Dilute muriatic and sulphuric acids, hyposulphite of soda, and even ammonia, have been proposed and occasionally used for dissolving out the copper of oxidized ores.

5. Much resembling the process mentioned under 2, is the method in which the oxidation is performed by calcining in reverberatory furnaces. At various Russian smelting works and in Mansfeldt this process is applied, but in no case does the extraction appear to be at all complete. A large quantity of the copper is removed in the soluble form, but fully as much remains in the residue, which is subject to further metallurgical treatment.

6. The first stage of Bankart's process is the same as the foregoing ones. Rich Cuban sulphurets are first calcined by themselves in reverberatory furnaces, and then lixiviated; the residues are mixed with a fresh portion of raw ore and again calcined. The peroxide of iron contained in the calcined ore causes the conversion into sulphuric acid of a portion of sulphur which would otherwise escape as sulphurous acid. The additional amount of sulphuric acid thus formed contributes of course to rendering the copper soluble. This principle is doubtless correct, but there appear to have been other circumstances which interfered with the practical application of the process.

7. Longmaid calcines pyritous ores with common salt, and then lixiviates. In his process there is doubtless a larger amount of copper rendered soluble than when the sulphurets are calcined alone; but the residues, even when abundance of sulphur is present, are far from being free from copper. The process which I have adopted in the experiments about to be described may be said to be a combination of the two last mentioned methods,—Bankart's and Longmaid's.

8. Henderson's process differs from Longmaid's merely in this particular, that the calcination is performed at such a temperature as to cause the volatilization of the copper in the form of a sub-

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chloride. The latter is condensed and treated further. This process labors under the same disadvantage as Longmaid's with regard to copper in the residues.

Among the various processes just enumerated, those which are most advantageous in economical respects, and which are best suited to the local circumstances of Canada, are evidently those in which few or no extraneous substances are required to render the copper soluble, and in which the acid necessary to form the soluble salt of copper is derived from the oxidation of elements present in the ore itself. At first sight the simplest and possibly most efficient means of rendering the copper soluble would appear to be that of merely calcining the sulphuret ores and lixiviating the product. Almost all such ores, whether containing copper glance, purple copper, or copper pyrites, certainly yield by such treatment more or less of their copper in a soluble state; but the amount of the metal so yielded in proportion to the quantity contained in the ore, is invariably very small indeed. Even with ores which contain a very considerable excess of sulphur, the calcination must be very carefully conducted if the extraction is at all to approach completeness. In the first stage of calcination sulphurous acid is given off; in the second sulphate of iron is formed; during the third stage it is necessary to heat the charge rather strongly in order to decompose the sulphate of iron, and transfer its sulphuric acid to the oxide of copper. In so doing there is a danger of too much heat being applied, and even of the sulphate of copper being decomposed. It is therefore extremely difficult to regulate this process, and the disadvantages of its being performed at too low a temperature consist not only in the extraction being imperfect, but also in the deposition of basic salts of iron while the copper is being precipitated. In this way the latter becomes so contaminated that many specimens of copper produced by cementation do not yield by assay over 60 per cent of the pure metal.

These disadvantages are to a very great extent removed by the use of common salt. By its action the sulphate of iron first formed is decomposed at a low temperature, and sulphate of soda and chloride of iron produced. The latter seems to be easily decomposed, and its chlorine transferred, in part at least, to the copper. On treating the calcined product with water, a solution is obtained which is very pure, often containing merely a trace of iron, in which case the deposition of basic iron salts becomes impossible. But although common salt may remedy the defects here indicated,

it cannot make up for a deficiency of sulphur in the original ore; and with or without common salt that ore will be treated with the greatest advantage which contains the largest proportion of sulphur. In proof of this the two following experiments may be cited:

1000 grains of slimes from the Bruce Mines, containing 5.20 per cent. of copper in the state of copper pyrites, but no admixture of iron pyrites, were calcined with 100 grains of common salt. The result yielded to water 13.7 grains of copper. Hence only 37.9 per cent. of the copper was extracted.

500 grains of ore from Escott, containing 10.4 per cent. of copper and an admixture of iron pyrites, were calcined with 100 grains of salt. The product yielded to water 40 grains of copper. Consequently 76.9 per cent. of the metal was removed.

In reflecting on the necessity of a sufficient supply of sulphur, it occurred to me that any means which would facilitate the conversion of the sulphur into sulphuric acid, instead of allowing it to escape as sulphurous acid, would be of advantage if applied in such calcining operations as those here referred to. Having in former experiments* demonstrated that the addition of peroxide of iron increased the amount of sulphuric acid formed in such calcinations, it occurred to me that it might be possible even with an ore containing no more sulphur, in proportion to the copper, than copper pyrites, to convert so much of it into sulphuric acid, as would in the presence of common salt develop sufficient chloride to render the whole of the copper soluble. I accordingly calcined the following materials together:

200 grains copper pyrites.
400 grains ferric oxide.
200 grains salt.

800 grains in all.

The copper pyrites, which was from Escott, contained 23.5 per cent; = 47 grains of copper. The residue after calcination weighed 657 grains, and on analysis gave—

Insoluble iron oxide.....	67.9
Insoluble cupric oxide.....	2.2 = 1.8 copper.
Sulphate of soda.....	10.1
Chloride of copper.....	3.4 = 1.6 copper.
Chloride of sodium (by difference)... ..	16.4
	100.0

* Canadian Naturalist, vol. vii, p. 196.

From this it appeared that in the residue there were present of copper—

In an insoluble state.... 11.82 grs. = 25.15 per cent.
In a soluble state..... 10.51 " = 22.36 "

22.33 grs.

Amount of copper lost.. 24.67 " = 52.49 "

47.00 grs.

The only way in which this extraordinary loss of 52.49 per cent. can be accounted for, is by supposing it to have been volatilized; and other circumstances confirm this view. The calcined substances contained 66.35 grains of sulphate of soda. Consequently 53.11 grains of common salt must have been decomposed, and 32.08 grains of chlorine liberated from it. Of this, 11.82 grains are found in combination with the copper in a soluble state. The remaining 20.26 grains were more than sufficient to form sub-chloride with the 24.67 grains of copper volatilized. The whole of the 32.08 grains of chlorine were not however sufficient to convert the forty-seven grains of copper contained in the ore into soluble protochloride.

From this experiment it became evident, 1st, that even with the use of a large quantity of peroxide of iron, it is difficult to treat copper pyrites so as to produce enough of sulphuric acid to render all the copper soluble; and 2nd, the calcination must be performed at as low a temperature as possible, in order to prevent the volatilization of the copper. The experiment was therefore performed a second time, care being taken to keep the temperature low. The same materials, in the same proportions, were used, and the product this time from the 800 grains weighed 792 grains, and contained—

Iron oxide and other insoluble matter.. 66.00
Sulphate of soda. 17.60
Chloride of copper..... 9.64 = 4.55 copper.
Chloride of sodium (by difference).... 6.76

100.00

Thirty-six grains of copper out of the original forty-seven were this time removed in a soluble form; equalling seventy-six per cent, instead of the twenty-two per cent, of the former experiment. Further, it appears that 139 grains of sulphate of soda were formed; and consequently 111 of chloride of sodium decomposed, and sixty

seven of chlorine liberated. The latter quantity was sufficient to convert fifty-nine grains of copper into proto-chloride. It becomes therefore difficult to say why the whole copper contents, 47 grains, were not extracted. Since the temperature was kept very moderate, the only other cause which may possibly have affected the result unfavorably, was the continuing of the calcination beyond the proper point, and the consequent decomposition of proto-chloride into insoluble sub-chloride of copper.

In order to ascertain whether the copper unaccounted for in the last experiment remained in the residue or was volatilized, the following experiment was made.

200 grains copper pyrites.

500 grains iron oxide.

200 grains salt.

900 grains were mixed together.

The pyrites contained 23.6 per cent, equal to 47.2 grains of copper. The mixture was calcined at a low temperature until the evolution of sulphurous acid and then of chlorine ceased. The result weighed 911 grains, and contained—

Iron oxide and other insoluble matter....	67.33	
Cupric oxide.....	1.87	= 1.5 copper.
Sulphate of soda.....	12.6	
Chloride of copper.....	8.14	= 3.8 copper.
Chloride of sodium (by difference).....	10.06	

100.00

It is evident from this that seventy-three per cent of the copper contained in the original ore was rendered soluble, and that twenty-seven per cent was left in the residue insoluble, but whether in the state of sub-chloride or oxide I did not ascertain. If we calculate from the amount of sulphate of soda formed in this experiment, it would appear that but a very little more chlorine was developed than was necessary to take up the whole of the copper as proto-chloride, so that it is most reasonable to suppose that the copper which remained insoluble was in the state of oxide, and had never had an opportunity of uniting with chlorine. The result of the experiment as to the volatilization of the copper was decisive, inasmuch as the whole of the copper was found in the product; therefore none could have been sublimed. The experiment also indicated that in order to a perfect extraction, an excess of chlorine must be presented to the copper.

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It will be recollected that the experiments just described were made with an ore poor in sulphur; containing no more of that element in proportion to the copper than is contained in copper pyrites. The results not proving satisfactory, I proceeded to experiment upon ores containing more sulphur, taking care always to keep the temperature very low, and to interrupt the calcination so soon as the development of sulphurous acid had ceased, and while that of chlorine and other gases was going on. The following are some of these experiments, with their results:

500 grains ore from Escott, containing 10.4 per cent copper (and both iron pyrites and copper pyrites), were calcined with 500 grains of iron oxide and 200 grains of salt. The product weighed 1184 grains, and gave with water a solution from which iron precipitated 49.6 grains of copper. Since fifty-two grains were present in the ore operated on, it is evident that 94.2 per cent of the copper was removed in a soluble form.

500 grains ore from Escott, containing 4.5 per cent of copper, and a considerable amount of iron pyrites, were calcined with 100 grains of iron oxide and 100 grains of salt. The result weighed 618 grains, and when heated with water yielded a solution containing 22.2 grains copper. 22.5 grains were present in the original ore; hence 97.7 per cent of the copper was dissolved.

300 grains of the same Escott ore, with 4.5 per cent of copper, 300 grains of ore from Bruce Mines containing 8.7 per cent of copper (and no iron pyrites), 300 grains of iron oxide, and 300 grains of salt, were calcined until chlorine was strongly developed. The result treated with water gave a green solution containing thirty-six grains of copper. Since the ore used in this experiment contained 39.6 grains copper, 90.9 per cent of the metal was removed in a soluble state.

300 grains of Bruce Mine ore of 8.7 per cent, 200 grains of iron pyrites from Brockville, 300 grains of iron oxide, and 200 grains of salt were calcined in the manner above mentioned. The product gave with water a solution containing 27.1 grains copper. The ore contained only 26.1 grains, and the excess may have been derived from the iron pyrites. In this case, therefore, the whole of the copper of the ore was dissolved.

The same mixture as the preceding, calcined for a longer time, yielded only 14.5 grains of copper in a soluble state.

400 grains of Bruce Mine slimes of 5.1 per cent, 400 grains of iron oxide, 100 grains of pyrites from Brockville, and 100 grains

of salt yielded, on calcination, a product from which water dissolved 18.6 grains of copper. The ore contained 20.4 grains; hence 91.1 per cent of the copper were dissolved.

200 grains of Escott copper pyrites of 23.6 per cent, 100 grains of iron pyrites, 800 grains of iron oxide, and 200 grains of salt were calcined together. The product weighed 1349 grains, from which water dissolved 46.6 grains of copper. Consequently ninety-eight per cent of the copper contents of the ore were rendered soluble.

In order to prove that the iron oxide is essential to producing this favorable result, the last experiment was repeated, the iron oxide being excluded from the mixture. On calcining and lixiviating, only 33.4 grains of copper were dissolved; that is, seventy per cent only instead of ninety-eight per cent.

The foregoing experiments were performed on plates of fire-clay in the muffle of a cupelling furnace. The iron oxide used was prepared by calcining iron pyrites with salt, and then lixiviating and drying. The following trials were made in a manner approximating more to the method that would necessarily be adopted in carrying out the process on a large scale. The mixtures were merely stirred on an iron plate lying upon two ignited billets of wood in a common stove. And further it will be seen that the oxide of iron necessary in the process was obtained from the ore experimented on. This ore was from the Capel Mine near Lennoxville, and had the following chemical composition:

Copper ..	8.60
Iron	30.31
Sulphur (by difference).....	34.34
Silica and argillaceous matter.....	26.75
	100.00

These figures correspond to the following mineralogical composition:

Copper pyrites	24.72
Iron pyrites.....	40.79
Quartz, &c.....	26.75
	101.26

It may be said that in round numbers this ore consisted of one half iron pyrites, one fourth copper pyrites, and one fourth rock. The first calcinations made with it were principally for the purpose of obtaining the necessary iron oxide. Nevertheless, the quantity of copper extracted was at the same time determined.

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10,000 grains of the ore were first calcined with 2,000 grains of salt, and without oxide of iron. The result weighed 10,860 grains. Water extracted from it 0.8 per cent of copper, and diluted sulphuric acid dissolved further a quantity of flocculent matter containing 1.8 per cent. Of the 860 grains copper contained in the original ore there were therefore,

Dissolved by water.....	86.88 grains = 10 per cent.
Dissolved by dilute acid.....	195.48 " " 22 "
Left in the residue.....	577.64 " " 68 "
	860.00

The product of the foregoing experiment after lixiviation and drying assayed 6.4 per cent. copper. A second calcination was performed with it and other substances having copper contents, as follows:

400 grains lixiviated product of above experi- ment containing 6.4 per cent.....	= 256 grains copper.
2000 grains fresh ore of 8.6 per cent.....	= 172 "
1200 grains salt.....	—
7200 grains mixture with.....	428 grains copper.

After being calcined in the usual manner the whole weighed 7850 grains, and contained four per cent of copper soluble in water slightly acidulated with sulphuric acid, and 1.11 per cent of copper insoluble therein. Of the 428 grains there were therefore,

Dissolved.....	314 grains.
Left insoluble.....	87 "
Unaccounted for.....	27 "
	428 grains.

Thus 73.3 per cent of the original copper contents were obtained in a soluble form.

The product of the foregoing experiment was, as in the first case, lixiviated and dried. It then contained $1\frac{1}{2}$ per cent of copper, and was calcined a third time, with fresh ore and salt in the following proportions:

4800 grains residue with $1\frac{1}{2}$ per cent....	= 64.0 grains copper.
2400 grains fresh ore of 8.6 per cent.....	= 204.5 "
1440 grains common salt.....	—
8640 grains, containing.....	270.4 grains copper.

This mixture was treated in the usual manner. The product weighed 8900 grains, and contained

Soluble.....	2.77 per cent.	= 246 grains copper.
Insoluble.....	.25 "	22.2 " "
Unaccounted for.....	1.3 "	" "
		270.4 " "

Thus 91.4 per cent of the copper contained in the ore originally used was recovered in a soluble form.

The residue from the experiment last described, after thorough lixiviation and drying, contained 0.44 per cent copper. Now very few of the slags from copper furnaces contain less than this percentage, and the refuse products of ordinary ore in dressing-operations seldom assay less than 1.5 per cent. In having produced therefore a residue containing as low a percentage as the above, it may be assumed that a point was raised where its copper contents might be disregarded. This residue consists, however, almost exclusively of iron oxide and the earthy matrix of the ore, and by virtue of the former is of value for mixing with fresh ore to produce the oxidation of the sulphur, and the consequent extraction of the copper contained therein. In order to test its usefulness in this respect a fourth calcination was made, with the following mixture:

2000 grains residue of 0.44 per cent	= 8.8 grains copper.
1000 " fresh ore of 8.6 "	= 86 "
600 " salt.....	

3600

94.8 grains copper.

The product weighed 3710 grains, and contained as follows:

Iron oxide and insoluble matter.....	72.13
Cupric oxide insoluble in water and dilute sulphuric acid.....	.27 = 0.22 copper.
Cupric oxide soluble in dilute sulphuric acid.....	.13 = 0.11 "
Sulphate of soda.....	19.22 = 4.33 sulphur.
Chloride of copper.....	4.7 = 2.22 copper.
Perchloride of iron.....	1.42
Chloride of sodium, by difference.....	2.13

100.00

Of the 94.8 grains copper contained in the original ore, there were consequently

Dissolved by water.....	82.36 grains = 86.88 per cent.
Dissolved by dilute acid..	4.08 " = 4.3 "
Left in residue.....	8.16 " = 8.6 "
Unaccounted for	.20 " = .22 "

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ins copper.

"

ns copper.

as follows :

.22 copper.

11 "

33 sulphur.

22 copper.

ore, there

cent.

If however we neglect the 8.3 grains copper contained in the residue used, which it is perfectly reasonable to do, then 95.7 per cent. of the copper in the ore is dissolved in water alone, and the whole of it on the additional use of a slight quantity of dilute sulphuric acid. One of the most striking facts deducible from the results of this experiment is that 46.64 per cent of the sulphur contained in the original ore was converted into sulphuric acid, and then into sulphate of soda. The amount of chlorine liberated by the formation of such a large quantity of this salt, must have been greatly in excess of what was necessary to render the copper soluble.

On account of this latter circumstance I attempted to reduce the quantity of salt used, and made four calcinations in the proportions of two parts of residue, one part of fresh ore, and one fourth of a part of salt. The ingredients, used with their copper contents, were as follows.

	1.	2.	3 & 4.
	GRS.	GRS.	GRS.
Residue.....	2000 with 8.8 Cu	1500 with 6.6 Cu	2000 with 20 Cu
Fresh ore.....	1000 " 86.0 "	750 " 64.5 "	1000 " 86 "
Salt.....	250 "	175 "	250 "
	3250 with 94.8 Cu	2425 with 71.1 Cu	3250 with 106 Cu

The products of these four experiments contained of copper respectively :

	1.	2.	3.	4.
	GRS.	GRS.	GRS.	GRS.
Soluble in water.....	59.11	40.5	62.22	66.00
Soluble in dilute SO ₃	14.69	7.3	3.60	10.00
Insoluble	} 21.00 {	7.3	18.18	17.21
Unaccounted for.....		16.0	22.00	12.79
	94.8	71.1	106.00	106.00

These results were by no means encouraging, yet the quantity of salt was quite sufficient for chloridizing the copper. One fact must however be mentioned with regard to these trials; they were very diligently stirred during the calcination.

No other resource was left but to return to experimenting with a larger quantity of salt. Accordingly the following ingredients were mixed and calcined :

2950 grains residue of 1.0 per cent. =	29.5 grains copper.
1500 " fresh ore of 8.6 " =	129.0 "
750 " salt.....	
5200 grains containing.....	158.5 grains copper.

They gave a product weighing 5270 grains, and containing

2.33 per cent Cu soluble in water	= 122.79
0.22 " " in dilute acid	= 11.59
0.33 " insoluble	= 17.16
Unaccounted for	= 6.96

158.50

Neither was this result extremely satisfactory. This mixture also was stirred diligently during the operation.

In order to ascertain whether the stirring exercised any injurious effect upon the completeness of the extraction, the following mixture was calcined:

2000 grains residue of 1.1 per cent	= 22 grains copper.
1000 " fresh ore of 8.6 "	= 86 "
500 " salt.....	

3500 grains containing..... 108 grains copper.

The operation was performed with as little stirring as possible, and the result weighed 3800 grains, containing

2.55 per cent soluble in water	= 96.90 grains copper.
.22 " " in dilute acid	= 8.36 "
.22 " left insoluble	= 4.18 "

109.44

It will be seen that this calcination was eminently successful, and that all the copper contained in the fresh ore was extracted. It was therefore very evident that diligent stirring was injurious.

Encouraged by this result I repeated the calcination with the smaller quantity of salt which I had previously used; the mixture being left almost wholly undisturbed during the operation. The ingredients were

2000 grains residue of 1.1 per cent	= 22
1000 " fresh ore of 8.6 "	= 86
250 " salt....	

3250 grains with..... 108 grains copper.

They gave on calcination a product of 3405 grains, containing 2.77 per cent, equal to 94.31 grains copper soluble in water; a result conclusively establishing that the more undisturbed the materials are during calcination, the more complete is the extraction.

The foregoing experiment was performed with one fourth part of salt to one of fresh ore. The result having been satisfactory,

the following mixture containing only one fifth part of salt was calcined :

2000 grains residue of 1·1 per cent	= 22 grains copper.
1000 " fresh ore of 8·6 " "	= 86 " "
200 " salt.....	

3200 grains containing.....108 grains copper.

This experiment was made with the same care as the others, and with as little stirring as possible. The result weighed 3250 grains, and contained

2·33 per cent. soluble in water	= 76·7 grains copper.
·44 " " in dilute acid	= 14·3 " "
Unaccounted for	= 17·0 " "

108·0

Since therefore only 71 per cent of the copper was extracted, it follows that one fourth of salt is the minimum quantity which can be used, and at the same time a complete extraction accomplished.

I here closed this series of experiments, having obtained all the definite results sought for. It appears certain that 95 per cent of the copper contained in an ore such as that produced by the Capel mine can be extracted in the humid way by calcining it with twice its weight of impure iron oxide (perhaps less would be sufficient), and one fourth of its weight of salt ; provided always that the operation is performed at a very low temperature, that it is not continued beyond a certain point, and that while it is going on the materials are stirred as little as possible. Whether equally good results can be obtained in practically applying this process, is a matter which can only be decided by experience ; but there appears to be no reason for doubting that it would be completely successful on the large scale.



