RESULTS.

| Chalcopyrite. Gram. | Iron. Gram. | Iron. Per cent. |
|---|---|---|
| 0.5800 | 0.1746 | 30.10 |
| 0.5800 | 0.1750 | 30.17 |

The question has been asked whether it is not possible, in a case like that of chalcopyrite, to render the original solution of the mineral ammoniacal and, not regarding the precipitated ferric hydrate, proceed with the electrolytic deposition of the copper. Many have felt that there was danger of iron entering into the deposit of copper, while others have declared that such a condition was not to be feared. During the past winter numerous tests of this question have been made in this laboratory, working with chalcopyrite. It has been found that if platinum dishes be employed as cathodes to receive the copper, the latter has invariably been contaminated with iron, but that if platinum cylinders, dipping into the ammoniacal solution contained in beaker-glasses, were used for cathodes the copper deposited upon them was free from iron.

The writer has had the assistance of Messrs. J. H. Graham and F. E. Walker in the experimental part of this study and would here acknowledge his indebtedness to them.

UNIVERSITY OF PENNSYLVANIA.

# THE USE OF POTASSIUM-FERRIC CHLORIDE FOR THE SOLUTION OF STEEL IN MAKING THE DETERMINATION OF CARBON.

BY GEO. WM. SARGENT.

Received July 27, 1900.

THE reaction considered to take place when steel or iron drillings dissolve in potassium-cupric chloride is expressed thus:

$$Fe + CuCl_2 = FeCl_2 + Cu \text{ and } Cu + CuCl_2 = Cu_2Cl_2.$$

This result expressed without indicating the intermediate reaction is written as follows:

$$Fe + 2CuCl_2 = FeCl_2 + Cu_2Cl_2.$$

The solution takes place more quickly and without any loss of carbon if hydrochloric acid (sp. gr. 1.2) is present in the ratio of 1 cc. to 13 cc. of the double chloride solution. The above equations do not contain the potassium chloride since the part enacted by it is not clearly understood; without it the drillings dissolve very much more slowly.

If instead of the copper salt, potassium-ferric chloride be used with just sufficient free hydrochloric acid present to prevent the separation of any basic iron salt, the drillings will be found to go into solution just as quickly and with no loss of carbon. The iron salt has the formula $FeCl_3,2KCl,H_2O$ and may be prepared by adding to a ferric chloride solution the requisite amount of potassium chloride and crystallizing out the double salt.

The chemically pure ferric chloride of commerce has the following composition:

| | Per cent. |
|---|---|
| Ferric chloride | 51.55 |
| Hydrochloric acid | 0.65 |
| Water | 48.80 |

If then to 267 grams of this commercial ferric chloride, 130.7 grams of potassium chloride are added and the whole, when dissolved in water, made up to 1 liter in volume, the resulting solution will be equivalent to the potassium-cupric chloride solution as usually prepared, except that it will contain free hydrochloric acid in the ratio of but 1 cc. to 225 cc. This amount is sufficient to prevent the formation of any basic salt and if this amount be much exceeded the acid will attack the drillings, giving off hydrogen and hydrocarbons. The reaction that takes place using the iron salt is analogous to that when the copper salt is used, leaving out of course the intermediate reaction, and is indicated by this equation:

$$Fe + 2FeCl_3 = 3FeCl_2.$$

If the solution is found to attack the drillings with evolution of hydrogen, the free hydrochloric acid must be neutralized until sufficient is left to just prevent the formation of basic salts of iron. The liquid should be a beautiful amber color when the conditions are perfect. As the drillings dissolve, the color becomes darker and of a greenish cast yet transparent so that when the solution of the drillings is complete, can more readily be ascertained than is the case when the copper salt is the solvent. The filtrate from the carbon is quite transparent, consequently whether the filtering medium has been a perfect one or not is quickly seen without the dilution of the filtrate as is required when potassium-cupric chloride is the decomposing agent.

This filtrate before it is contaminated with any wash-water, may be chlorinated and used again for the solution of a second lot of drillings. In this way, by chlorinating,[1] the filtrate from the carbon is made to decompose the next sample of drillings. When sufficient iron chloride has accumulated, add more potassium chloride and water to make the solution of the original strength. To illustrate, 200 cc. of the solution of $FeCl_3,2KCl$ containing about 50 grams of iron per liter are used in the decomposition of each factor weight, 2.7272 grams, of drillings. The liter will then contain besides the original amount 13.636 grams of iron. By chlorinating, four lots of 13.636 grams each or 54+ grams of iron may be dissolved in this liter. If 130.7 grams of potassium chloride be now added, the liter may be made two liters in volume. This operation of chlorinating, adding potassium chloride and diluting, may be carried on *ad infinitem*. It is, however, necessary at some point, determined by the amount of work done in the laboratory, to throw out a part of the accumulated solution.

In this way the waste product is utilized to assist in the next carbon determination and the expense materially reduced.

A table of results obtained by using both salts for the decomposition of the steels is appended.

| Sample. | Dissolved in $CuCl_2.2KCl$. Per cent. carbon. | Dissolved in $FeCl_3.2KCl$. Per cent. carbon. |
|---|---|---|
| Standard 0.97 per cent | 0.972 | 0.970 |
| " 0.97 " | 0.978 | 0.975 |
| " 0.97 " | 0.974 | 0.971 |
| " 0.97 " | 0.970 | 0.974 |
| " 0.97 " | 0.970 | .... |
| " 0.97 " | 0.968 | .... |
| No. 5616 | 0.677 | 0.676 |
| No. 5561 | 0.789 | 0.798 |
| No. 5566 | 0.806 | 0.803 |
| No. 5557 | 0.780 | 0.782 |
| No. 4367 (a tungsten steel) | 2.060 | 2.017 |
| Standard 1.15 per cent | 1.150 | 1.155 |
| " 1.15 " | 1.150 | 1.149 |
| " 1.15 " | 1.150 | 1.146 |
| " 1.15 " | 1.153 | 1.153 |
| " 1.15 " | 1.145 | 1.143 |
| " 1.15 " | .... | 1.146 |

---

[1] This Journal, **22**, 210.

It will be noticed that rather high carbon steels only have been dealt with and no irons. It is well known that mild steels and irons are more readily attacked by acid than high carbon steels. I have stated that the amount of free acid should not exceed 4.5 cc. per liter. This is a trifle more than sufficient to prevent any precipitation of basic iron salts, yet this solution, if allowed to stand upon a very mild steel or iron, will react with slight evolution of hydrogen. However if a very mild steel or iron be dissolved in this solution, the results vary from those obtained when the copper salt is the decomposing agent by amounts not exceeding the experimental error.

| Sample. | Dissolved in $CuCl_2.2KCl$. Per cent. carbon. | Dissolved in $FeCl_3.2KCl$. Per cent. carbon. |
|---|---|---|
| No. 23943 | 0.383 | 0.388 |
| No. 23943 | 0.375 | 0.383 |
| Cast iron {combined carbon = 0.126 per cent.} | 3.034 | 3.060 |

This would seem to indicate that the very dilute acid reacted upon the ferrite and not upon the cementite and that the carbon released from the $Fe_3C$ by the $FeCl_3$ was not readily taken up by the hydrogen produced from the acid acting upon the ferrite. That cementite resists the action of acid is well known and is shown by the difficulty experienced in dissolving high carbon steel, also by the action of very dilute acid upon a polished specimen of a low carbon steel.

The action of light upon chlorine water is to produce hydrochloric acid and oxygen.[1] Ferric chloride is also affected by the action of the light; hence it may be well to call attention to the fact that the chlorinated solution increases in acid content, particularly if it stands any length of time in the sunlight.

All the results obtaned in investigating the iron salt as a solvent have not been given, but sufficient to show that it can be used. The advantages derived from using it, especially where many determinations are made daily, will be apparent. The ease with which the point when all the drillings have gone into solution, is ascertained, the ready examination of the filtrate for any carbon that may have escaped the filter, and the economy recommend it.

LABORATORY OF THE CARPENTER STEEL CO.,
READING, PA.