A THESIS.

Submitted by J.H. Gilbreth in partial fulfillment of requirements for B.S. degree in chemical engineering.

THE SEPARATION OF ZINC AND COPPER IN BRASS

In the analysis of brass, the seperation of copper from zinc by two precipitations is very tedious and requires a great deal of time. If one precipitation will successfully effect the seperation, it is a waste of time to precipitate twice. The two leading authories differ on this point . Fresenius, an old and established authority, declares that the only method for seperating copper from zinc is by two precipitations; while Treadwell, a latter and well known author, states that a complete seperation of copper from zinc may be effected with only one precipitation. The following is the method.

Fresenius:-a. To effect the seperation of the oxides of the fifth group from those of the first three groups by means of sulphuretted hydrogen, it is necassary simply that the reaction of the solution be acid , the nature of the acid being of no consequence. But , to effect the seperation of the oxides of the fifthe group from those of the fourth group , the presence of a free mineral acid is indespensable; otherwise, zinc, and under some circumstances, also cobalt and nickel may be coprecipitated.

b. But even the addition of hydrochloric acid to the fluid will not always prevent the coprecipitation of the zinc.

the zinc. Rivot and bBouquet declare a complete seperation of copper from zinc by means of sulphuretted hydrogen, altogether impracitical. Calvert states that he has arrived at the same conclusion. On the other hand, Spirgatis concurs with H. Rose in maintaining that complete seperation of copper from zinc maybe effect by means of sulphuretted hydrogen, in presence of a sufficient quantity of free acid.

In this conflict of opinions I thought it necessary to subject this method ince more to a searching investigation. I therefore instructed one of the students of the labaratory, Mr. Grundmann, to make a series of experiments in the matter, in view of settling the question.

The results proved incontestably that copper maybe seperated from zinc by sulphuretted hydrogen, if the f following instructions are strictly complied with:-

Add to the copper and zinc a copius amount of hydrochloric acid(e.g., to 0.2 grms. of oxide of copper in 25c.c. of solution, 10c.c. of hydrochloric acid of 1.1sp.gr.), conduct into the fluid sulphuretted hydrogen largely in excess, filter before the excess of sulphuretted hydrogen has had time to escape or become decomposed, wash with sulphuretted hydrogen water, dry, roast redisolve in nitro hydrochloric acid evaporate nearly to dryness, add water and hydrochloric acid, as above, and precipitate again in sulphuretted hydrogen. This second precipitation is free from zinc; it is treated as sulphide of copper.

On the other hand the more recent book has come out under the authority of Treadwell, showing that it is not necessary

not necessary to precipitate copper more than one time to seperate it from zinc .

Treadwell:- The filtrate from lead is evaporated until the alchol is completly removed,100c.c. of water are added, the solution is heated to boiling, and hydrogen sulphide is conducted into it until it becomes cold, when the copper sulphide is filtered off, washed first with hydrogen sulphide water containing in every 100c.c.c., 20c.c. of double-normal sulphuric acid and at the end with 5per cent ascetic acid which is saturated with hydrogen sulphide,until the filtrate gives no precipitate on being treated with barium chloride. The copper is determined as Cu2s.

In as much as these two authorities differ I have done this work with a view of determining whether or not the shorter of the two methods (Treadwell's) may be used in seperating copper from zinc.

Method used by me in obtaining results here given; To 25c.c. of the solution containing 0.2 grms. each of copper and zinc water and hydrochloric acid are added in varying proportions. Hydrogen sulphide is then passed into the solution. As soon as all the copper has been precipitated it is filtered and thoroughly washed by decantation with water containing hydrogen sulphide , care being taken that the copper does not oxidise. The wash is boiled to expel the H2S and tested with nitric acid and silver nitrate. After the precipitation has been washed clean it is dried and ignited over a Busen flame. It is then transfered to a beaker and

dissolved in nitric acid. 10c.c. of sulphuric acid are then added and the solution is evaporated until the nitric acid has been expelled, indicated by the white fumes of the sulphuric. After cooling, 200c.c. of water are added and hydrogen sulphide is again passed into the solution, which has first been brought to boiling . When the precipitation is complete the copper is filtered and washed a very little. The filtrate now contains the zinc which was precipitated with the copper on the first precipitation. This solution containing the zinc is made ammoniacily with ammonium hydroxid and then the zinc is precipitated with ammonium sulphide. The zinc sulphide settles slowly, requiring two or three or four hours. After it has subsided the ZNS is filtered, washed, dried and weighed. The Zn is then computed. There was so small a quantity of Zns that it was all undoubtly oxidized and consequently the precipitate was weighed as Zno.

A solution was prepared for the analyses, of copper sulphate and zinc sulphate according to the following.

Preparation of a solution which shall contain in 25c.c. 02. grm. copper and 02. grm. zinc, from copper sulphate and zinc sulphate. Ccopper sulphate, CuSO4. 5H2O. Zinc zulphate, ZnSO4, 7H2O.

Atomic weights.

Cu	63.57	Zn	65.37
S	32.07	S	32.07
O4	64.00	O4	64.00
5H2	10.08	7H2	14.11
50	80.00	70	112.00
	249.72		287.55

249. 72÷ 63.57= 3.9282÷ 5= 0.7856 grms. copper sulphate will contain 0.2 grm. copper.

278.55÷ 65.37=4.3973÷ 5=0.8797 grms. zinc zulphate contains 0.2 grm zinc.

One litter contains forty 25c.c. portions.

40×0.7858=31.4240 grms copper sulphate.

40×0.6537=35.1760 grms. zinc sulphate.

Therefore , 31.4240 grms. copper sulphate and 35.1760 grms. zinc sulphate dissolved in 1000c.c. water will yield 0.2. grm. copper and 0.2 grm zinc for 25c.c. of solution at room tempture.

Thirty seperate analysis were made and the results obtained from each analysis, with the varying conditions is given in tabulated form.

Number	Amount of Cu in 25cc of solution.	Amount of Zn in 25cc of solution.	HCl, number of cc.	Water, ee number cc.	Temptura, degrees C.	Amount Zn precipitated.	per cent of Zn precipitated.	Average per cent of Zn precipitated
1	0.2	0.2	5	200	100	0.0045	2.25	
6	0.2	0.2	5	400	100	0.0024	1.20	
11	0.2	0.2	5	200	20	0.0033	1.65	
16	0.2	0.2	5	400	20	0.0043	2.15	
21	0.2	0.2	5	200	-	0.0159	7.85	
26	0.2	0.2	5	400	-	0.0085	4.25	
								3.23
2	0.2	0.2	10	200	100	0.0043	2.15	
7	0.2	0.2	10	400	100	0.0021	1.05	
12	0.2	0.2	10	200	20	0.0032	1.60	
17	0.2	0.2	10	400	20	0.0019	0.95	
22	0.2	0.2	10	200	-	0.0026	1.30	
27	0.2	0.2	10	400	-	0.0025	1.25	
								1.38
3	0.2	0.2	15	200	100	0.0022	1.12	
8	0.2	0.2	15	400	100	0.0013	0.65	
13	0.2	0.2	15	200	20	0.0024	1.20	
18	0.2	0.2	15	400	20	0.0016	0.80	
23	0.2	0.2	15	200	-	0.0012	0.60	
28	0.2	0.2	15	400	-	0.0011	0.55	
								0.82
4	0.2	0.2	20	200	100	0.0014	0.72	
9	0.2	0.2	20	400	100	0.0006	0.30	
14	0.2	0.2	20	200	20	0.0005	0.19	
19	0.2	0.2	20	400	20	0.0013	0.65	
24	0.2	0.2	20	200	-	0.0003	0.15	

Number	Amount of Cu in 25cc of solution.	Amount of Zn in 25cc of solution.	HCl, number of cc.	Water, number of cc.	Tempture, degrees C.	Amount of Zn precipitated.	Per cent. of Zn precipitated.	Average per cent. of Zn precipitated.
29	0.2	0.2	20	400	-	0.0006	0.30	
								0.30
5	0.2	0.2	25	200	100	0.0005	0.24	
10	0.2	0.2	25	400	100	0.0004	0.20	
15	0.2	0.2	25	200	20	0.0006	0.30	
20	0.2	0.2	25	400	20	0.0005	0.25	
25	0.2	0.2	25	200	-	0.0001	0.05	
30	0.2	0.2	25	400	-	0.0004	0.20	
								0.20

** Brought to boiling and precipitated while boiling.

From a study of results given on page it will be seen that the amount of zinc precipitated with the copper varies directly with the amount of acid used. Experment No. 25 shows the least amount of zinc precipitated, only 0.05 per cent going down . This was effected by adding 25c.c. hydrochloric acid and 200c.c. water to the solution which was then brought to boiling and hydrogen sulphide allowed to pass into it while cooling.

In view of the fact that so small amount of zinc is precipitated with the copper in experiment No. 25, it can very well be considered neglible in ordinary analysis. Hence it is concluded that one precipitation, with hydrogen sulphide is sufficient to seperate copper from zinc in a copper and zinc solution.