A Dry Method of Determining Zinc contained in Brass, Bronze and other similar Alloys.

By

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The ordinary methods, whether volumetric or gravimetric, used for determining the proportion of zinc contained in brass, bronze and other similar alloys, necessitate in the first place the separation of the copper and then the estimation of the zinc, in consequence of which they are not considered very convenient when it is necessary to analyse numerous samples at one time. The electrolytic method also has its disadvantages. In using this method, it is difficult to know the best analytical conditions, so that it is not very reliable except for those who have much practice in it.

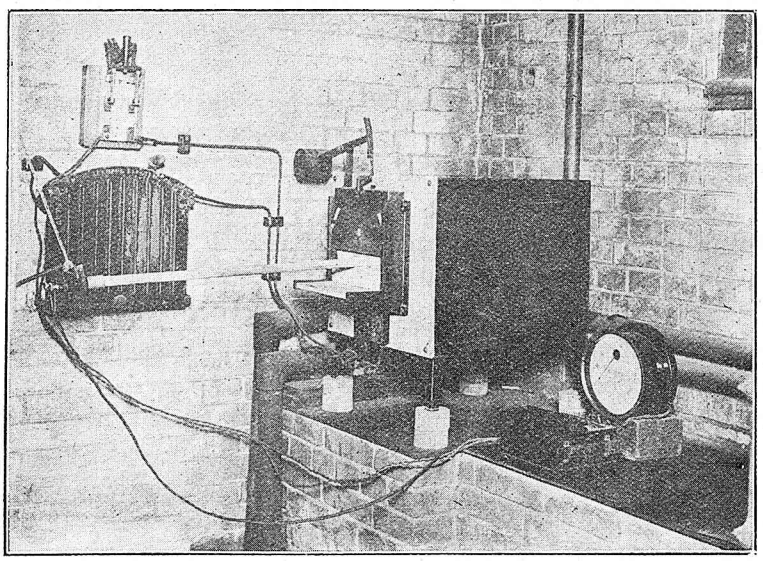
Our method consists in taking advantage of the low volatility of tin, which is however very liable to form alloys with copper, thereby replacing zinc in the original alloys, on being heated a little higher than the volatilization-point of the zinc. Sir T. K. Rose[1] determined the percentage of zinc in brass by heating 1 grm. of the alloy in a carbon crucible for two hours at a temperature of about 1375°C. Such a high temperature must however be liable to raise the oxidation of the copper; moreover, the means of obtaining so high a temperature will usually be lacking in ordinary laboratories or workshops. Our method is on the contrary much simpler and more accurate, and in so far as it is a dry process, does not require much practice, so that there is little danger of making mistakes by which the results would be affected in many ways.

1 Jour. Soc. Chem. Ind., **33**, 170.

The method of procedure is as follows: Weigh out accurately 500 mgms. of a sample, and mix it with the same weight of pure tin. Place these in a small graphite crucible, 2·7 cm. in height, 3 cm. in the external and 2·1 cm. in the internal diameter, and cover them well with wood-charcoal with which the crucible has previously been filled, then put a graphite lid on it. This crucible, after being preheated in an air-bath at about 230°C. for 30 to 40 minutes, is transferred into Hoskin's electric resistance furnace kept at 980°C., and is there subjected to reactions for $1\frac{1}{2}$ hours. After the reaction are over, take the crucible out of the furnace and allow it to cool. The metallic button is picked out, well brushed, and weighed. The difference in weight, which is due to the zinc having been volatilized, multiplied by 2/10, gives the percentage of the zinc.

The most convenient form of Hoskin's furnace is of the F. D. type, the size of which is $7\frac{1}{2}'' \times 5\frac{1}{4}'' \times 14''$. It can be used without any more modification than that, except, perhaps, the enlarging of the draught-hole. Forty crucibles can be placed at one time in the furnace, and therefore, if it is necessary to carry out two analyses for each sample, there is still room for 20 different ones.

In the present investigation, we heated 15 crucibles each charged with the same alloy in pieces at one operation, and repeated this operation twice, so that we were able to have 30 analyses for each sample. This results obtained were always checked with those found with the wet process. The following figure shows the arrangement in use.



Experiment No. I:- 5% Zn-brass
(94·18% Cu, 5·71% Zn, 0·01% Pb)

No.	First charge		No.	Second charge	
	Wt-loss in mgs.	Zinc in %		Wt-loss in mgs.	Zinc in %
1	29·0	5·80	1	28·6	5·72
2	28·8	5·76	2	28·7	5·74
3	28·8	5·76	3	28·9	5·78
4	28·3	5·66	4	28·3	5·66
5	28·7	5·74	5	28·7	5·74
6	28·9	5·78	6	28·3	5·66
7	28·9	5·78	7	28·6	5·72
8	28·8	5·76	8	28·5	5·70
9	28·2	5·64	9	29·0	5·80
10	28·9	5·78	10	28·6	5·72
11	29·0	5·80	11	28·6	5·72
12	28·9	5·78	12	28·6	5·72
13	28·8	5·76	13	28·9	5·78
14	28·2	5·64	14	29·0	5·80
15	28·7	5·74	15	28·7	5·74
Mean	28·7	5·74	Mean	28·7	5·74
Max.	29·0	5·80	Max.	29·0	5·80
Min.	28·2	5·64	Min.	28·3	5·66
Dif.	0·8	0·16	Dif.	0·7	0·14

Experiment No. II:- 10% Zn-brass
(89·20% Cu, 10·54% Zn, 0·15% Pb).

No.	First charge		No.	Second charge	
	Wt-loss in mgs.	Zinc in %		Wt-loss in mgs.	Zinc in %
1	52·8	10·56	1	52·0	10·60
2	53·3	10·66	2	52·7	10·54
3	53·4	10·68	3	53·1	10·62
4	52·8	10·56	4	52·7	10·54
5	53·0	10·60	5	53·3	10·66
6	53·3	10·66	6	52·6	10·52
7	52·8	10·56	7	53·0	10·60

8	53·2	10.64	8	53·2	10.64
9	53·2	10.64	9	52.6	10.52
10	53·1	10.62	10	52·7	10.54
11	52.8	10.56	11	53·0	10.60
12	53·4	10.68	12	53·0	10.60
13	52.8	10.56	13	53·1	10.62
14	53·2	10.64	14	52.6	10.52
15	53·0	10.60	15	53·3	10.66
Mean	53·1	10.62	Mean	52·9	10.58
Max.	53·4	10.68	Max.	53·3	10.66
Min.	52.8	10.56	Min.	52.6	10.52
Dif.	0.6	0.12	Dif.	0·7	0.14

Experiment No. III:– 5% Zn-brass
(84·04% Cu, 15·66% Zn, 0 13% Pb).

No.	First charge		No.	Second charge	
	Wt-loss in mgs.	Zinc in %		Wt-loss in mgs.	Zinc in %
1	78·9	15.78	1	78·9	15.78
2	78·7	15.74	2	78.6	15.72
3	79·1	15.82	3	78·4	15.68
4	78.8	15.76	4	79·3	15.86
5	78.6	15.72	5	79·1	15.82
6	79·2	15.84	6	78·9	15.78
7	78.8	15.76	7	79·5	15.90
8	78.6	15.72	8	79·4	15.88
9	78·4	15.68	9	78·7	15.74
10	78·7	15.74	10	79·3	15.86
11	78.6	15.72	11	78·5	15.70
12	79·0	15.80	12	79·1	15.82
13	79·3	15.86	13	78·7	15.74
14	78.6	15.72	14	78.6	15.72
15	78·3	15.66	15	78·9	15.78
Mean	78.8	15.76	Mean	78·9	15.78
Max.	79·3	15.86	Max.	79·5	15.90
Min.	78·3	15.66	Min.	78·4	15.68
Dif.	1.0	0.20	Dif.	1·1	0.22

Experiment No. IV:– 20% Zn-brass
(78·60% Cu, 20·97% Zn, 0·26% Pb, 0·15% Fe).

No.	First charge		No.	Second charge	
	Wt-loss in mgs.	Zinc in %		Wt-loss in mgs.	Zinc in %
1	105·3	21·06	1	105·7	21·14
2	105·8	21·16	2	105·0	21·00
3	105·0	21·00	3	105·0	21·00
4	105·1	21·02	4	105·8	21·16
5	105·8	21·16	5	105·3	21·06
6	105·5	21·10	6	105·3	21·06
7	105·6	21·12	7	105·6	21·12
8	105·2	21·04	8	105·7	21·14
9	105·4	21·08	9	105·5	21·10
10	105·6	21·12	10	105·2	21·04
11	105·3	21·06	11	105·3	21·06
12	105·6	21·12	12	105·0	21·00
13	105·0	21·00	13	105·7	21·14
14	105·4	21·08	14	105·8	21·16
15	105·8	21·16	15	105·2	21·04
Mean	105·4	21·08	Mean	105·5	21·10
Max.	105·8	21·16	Max.	105·8	21·16
Min.	105·0	21·00	Min.	105·0	21·00
Dif.	0·8	0·16	Dif.	0·8	0·16

Experiment No. V:– 30% Zn-brass
(70·17% Cu, 29·42% Zn, 0·26% Pb).

No.	First charge		No.	Second charge	
	Wt-loss in mgs.	Zinc in %		Wt-loss in mgs.	Zinc in %
1	147·8	29·56	1	147·5	29·50
2	147·9	29·58	2	147·2	29·44
3	147·7	29·54	3	147·6	29·52
4	147·8	29·56	4	147·8	29·56
5	147·1	29·42	5	147·5	29·50
6	148·1	29·62	6	148·1	29·62
7	147·2	29 44	7	148·0	29·60

8	148·1	29·62	8	147·1	29·42
9	147·6	29·52	9	147·1	29·42
10	147·3	29·46	10	147·2	29·44
11	147·8	29·56	11	147·5	29·50
12	147·2	29·44	12	148·0	29·60
13	147·9	29·58	13	147·6	29·52
14	147·6	29·52	14	147·1	29·42
15	147·1	29·42	15	148·1	29·62
Mean	147·6	29·52	Mean	147·6	29·52
Max.	148·1	29·62	Max.	148·1	29·62
Min.	147·1	29·42	Min.	147·1	29·42
Dif.	1·0	0·20	Dif.	1·0	0·20

Experiment No. VI:– 40% Zn-brass
(60·16% Cu, 39·32% Zn, 0·24% Pb).

No.	First charge		No.	Second charge	
	Wt-loss in mgs.	Zinc in %		Wt-loss in mgs.	Zinc in %
1	39·40	197·0	1	197·5	39·50
2	39·42	197·1	2	197·0	39·40
3	39·38	196·9	3	197·0	39·40
4	39·42	197·1	4	197·2	39·44
5	39·48	197·4	5	197·5	39·50
6	39·40	197·0	6	197·4	39·48
7	39·46	197·3	7	197·0	39·40
8	39·46	197·3	8	197·5	39·50
9	39·44	197·2	9	197·5	39·50
10	39·48	197·4	10	197·0	39·40
11	39·40	197·0	11	197·4	39·48
12	39·46	197·3	12	197·0	39·40
13	39·38	196·9	13	197·0	39·40
14	39·44	197·2	14	197·5	39·50
15	39·48	197·4	15	197·4	39·48
Mean	39·44	197·2	Mean	197·3	39·46
Max.	39·48	197·4	Max.	197·5	39·50
Min.	39·38	196·9	Min.	197·0	39·40
Dif.	0·10	0·5	Dif.	0·5	0·10

Experiment No. VII:– Bronze No. 1
(88·72% Cu, 8·66% Sn, 2·55% Zn, 0·02% Fe).

No.	First charge		No.	Second charge	
	Wt-loss in mgs.	Zinc in %		Wt-loss in mgs.	Zinc in %
1	12·7	2·54	1	13·1	2·62
2	12·6	2·52	2	12·6	2·52
3	13·1	2·62	3	13·1	2·62
4	12·6	2·52	4	12·9	2·58
5	13·1	2·62	5	12·6	2·52
6	12·8	2·52	6	12·9	2·58
7	12·8	2·56	7	12·4	2·48
8	12·5	2·50	8	12·6	2·52
9	12·4	2·48	9	12·7	2·54
10	12·4	2·48	10	12·9	2·58
11	13·1	2·62	11	12·5	2·50
12	12·5	2·50	12	12·6	2·52
13	12·5	2·50	13	12·4	2·48
14	12·6	2·52	14	12·8	2·56
15	12·6	2·52	15	12·9	2·58
Mean	12·7	2·54	Mean	12·7	2·54
Max.	13·1	2·62	Max.	13·1	2·62
Min.	12·4	2·48	Min.	12·4	2·48
Dif.	0·7	0·14	Dif.	0·7	0·14

Experiment No. VIII:– Bronze No. 2
(78·32% Cu, 4·37% Sn, 16·91% Zn, 0·21% Pb).

No.	First charge		No.	Second charge	
	Wt-loss in mgs.	Zinc in %		Wt-loss in mgs.	Zinc in %
1	84·0	16·80	1	83·9	16·78
2	84·1	16·82	2	83·8	16·76
3	84·0	16·80	3	83·9	16·78
4	84·3	16·86	4	84·4	16·88
5	84·8	16·96	5	84·3	16·86
6	84·5	16·90	6	84·4	16·88
7	84·2	16·84	7	84·0	16·80

8	84.0	16.80	8	84.2	16.84
9	84.2	16.84	9	84.5	16.90
10	84.4	16.88	10	84.5	16.90
11	84.5	16.90	11	84.1	16.82
12	84.0	16.80	12	83.9	16.78
13	84.5	16.90	13	84.1	16.82
14	84.5	16.90	14	84.3	16.86
15	84.3	16.86	15	84.2	16.84
Mean	84.3	16.86	Mean	84.2	16.84
Max.	84.8	16.96	Max.	84.5	16.90
Min.	84.0	16.80	Min.	83.8	16.76
Dif.	0.8	0.16	Dif.	0.7	0.14

Experiment No. IX:- Brass containing 0.5% lead (79.64% Cu, 19.51% Zn, 0.47% Pb, 0.28% Fe).

No.	First charge		No.	Second charge	
	Wt-loss in mgs.	Zinc in %		Wt-loss in mgs.	Zinc in %
1	99.4	19.88	1	99.3	19.86
2	99.0	19.80	2	98.7	19.74
3	98.6	19.72	3	98.5	19.70
4	99.1	19.82	4	99.1	19.82
5	99.1	19.82	5	99.0	19.80
6	99.1	19.82	6	99.3	19.86
7	98.7	19.74	7	98.4	19.68
8	98.8	19.76	8	98.5	19.70
9	99.0	19.80	9	99.1	19.82
10	99.3	19.86	10	99.1	19.82
11	99.4	19.88	11	99.3	19.86
12	98.7	19.74	12	98.4	19.68
13	98.6	19.72	13	98.5	19.70
14	99.0	19.80	14	99.1	19.82
15	99.1	19 82	15	99.0	19.80
Mean	99.0	19.80	Mean	98.9	19.78
Max.	99.4	19.88	Max.	99.3	19.86
Min.	98.6	19.72	Min.	98.4	19.68
Dif.	0.8	0.16	Dif.	0.9	0.18

Experiment No. X:- Brass containing 1·0% lead (68·21% Cu, 30·43% Zn, 1·16% Pb, 0·17% Fe).

No.	First charge		No.	Second charge	
	Wt-loss in mgs.	Zinc in %		Wt-loss in mgs.	Zinc in %
1	154·5	30·90	1	153·9	30·78
2	153·6	30·72	2	153·6	30·72
3	153·7	30·74	3	154·4	30·88
4	154·1	30·82	4	153·7	30·74
5	153·9	30·78	5	154·0	30·80
6	154·5	30·90	6	154·1	30·82
7	154·0	30·80	7	153·8	30·76
8	153·9	30·78	8	153·9	30·78
9	154·1	30·82	9	153·8	30·76
10	153·7	30·74	10	153·5	30·70
11	154·5	30·90	11	153·9	30·78
12	154·0	30·80	12	153·8	30·76
13	153·7	30·74	13	154·4	30·88
14	154·1	30·82	14	153·8	30·76
15	153·9	30·78	15	154·0	30·80
Mean	154·0	30·80	Mean	153·9	30·78
Max.	154·5	30·90	Max.	154·4	30·88
Min.	153·6	30·72	Min.	153·5	30·70
Dif.	0·9	0·18	Dif.	0·9	0·18

Experiment No. XI:- Brass containing 1% iron (58·62% Cu, 39·85% Zn, 1·16% Fe, 0·29% Pb).

No.	First charge		No.	Second charge	
	Wt-loss in mgs.	Zinc in %		Wt-loss in mgs.	Zinc in %
1	200·4	40·08	1	200·7	40·14
2	200·6	40·12	2	200·3	40·06
3	200·2	40·04	3	200·5	40·10
4	200·2	40·04	4	200·7	40·14
5	200·7	40·14	5	200·8	40·16
6	200·8	40·16	6	200·6	40·12
7	200·3	40·06	7	200·4	40·08

8	200·2	40·16	8	200·8	40·16
9	200·7	40·14	9	200·7	40·14
10	200·1	40·02	10	200·9	40·18
11	200·9	40·18	11	200·9	40·18
12	200·9	40·18	12	200·9	40·18
13	200·3	40·06	13	200·8	40·16
14	200·8	40·16	14	200·5	40·10
15	200·7	40·14	15	201·0	40·20
Mean	200·5	40·10	Mean	200·7	40·14
Max.	200·9	40·18	Max.	201·0	40·20
Min.	200·1	40·02	Min.	200·3	40·06
Dif.	0·8	0·16	Dif.	0·7	0·14

Experiment No. XII:– Brass containing 2% iron (73·38% Cu, 24·34% Zn, 2·10% Fe, 0·16% Pb).

No.	First charge		No.	Second charge	
	Wt-loss in mgs.	Zinc in %		Wt-loss in mgs.	Zinc in %
1	123·3	24·66	1	123·4	24·68
2	123·4	24·68	2	123·7	24·74
3	123·2	24·64	3	123·3	24·66
4	123·3	24·66	4	123·2	24·64
5	123·7	24·74	5	123·5	24·70
6	123·9	24·78	6	123·1	24·62
7	123·2	24·64	7	123·2	24·64
8	123·2	24·64	8	123·1	24·62
9	123·2	24·64	9	123·1	24·62
10	123·8	24·76	10	123·2	24·64
11	123·9	24·78	11	123·6	24·72
12	123·5	24·70	12	123·3	24·66
13	123·3	24·66	13	123·6	24·72
14	123·6	24·72	14	123·5	24·70
15	123·9	24·78	15	123·9	24·78
Mean	123·5	24·70	Mean	123·4	24·68
Max.	123·9	24·78	Max.	123·9	24·78
Min.	123·2	24·64	Min.	123·1	24·62
Dif.	0·7	0·14	Dif.	0·8	0·16

Résumé.

The results obtained with our method are always within the limits of allowable error when compared with those with the wet process, as will be seen from the following table:

Alloys	Zn-% with wet process	Zn-% with dry process	Difference
5% Zinc-Brass	5·71	5·74	0·03
10% 〃	10·54	10·60	0·06
15% 〃	15·66	15·77	0·11
20% 〃	20·97	21·09	0·12
30% 〃	29·42	29·52	0·10
40% 〃	39·32	39·45	0·13
Bronze No. 1	2·55	2·54	0·01
Bronze No. 2	16·91	16·85	0·06

That an alloy contains lead or iron besides zinc does not affect the results, if the quantities of these metals are not much over certain limits. The following table explains this fact clearly:

Alloys	Zn-% with wet process	Zn-% with dry process	Difference
Brass containing 0·5% Pb	19·51	19·79	0·28
Brass 〃 1·0% Pb	30·43	30·79	0·36
Brass 〃 1·0% Fe	39·85	40·12	0·27
Brass 〃 2·0% Fe	24·34	24·69	0·35

This experiment was conducted at the Assay Laboratory of the Imperial Mint, Osaka.