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THE DETERMINATION OF OXYGEN IN COPPER.

By S. DICKSON.

(*Read at the Meeting, March* 1, 1905.)

The processes for the determination of oxygen in copper in use at the present time, and on which reliance can be placed, are two in number:

1. That devised by Mr. Bertram Blount.
2. That devised by Mr. Leonard Archbutt.

The first process is based on the complete fusion of the metal in a current of dry hydrogen, and the water formed collected in a U-tube containing pumice saturated with sulphuric acid.

The second process consists in heating the metal (in a fine state of division) to

redness in hydrogen, and the loss in weight is taken as representing the amount of oxygen present. In considering the above processes, and assuming both methods of equal accuracy, the analyst would most probably select that which would be more easily conducted, and for which the apparatus necessary is to be found in everyday use in the laboratory. There is, however, one objection which limits the usefulness of the latter process, and that is, the pieces of metal must be of very small dimensions. The first process, on the other hand, has no such drawback; the size of the pieces is immaterial, and a lump may be and is used. The only objections are the high temperature required for the fusion of the copper, to attain which special apparatus is necessary, and, when using a blast furnace, the heating of the absorption apparatus by radiation, it being by no means easy to insulate it thoroughly. The somewhat lower results obtained by this method, when compared with those given by the process to be described, are, I think, due to such heating. The use of an electrically heated furnace, as described in the February number of this journal by the same author, would no doubt obviate the defect.

In considering the objections to the foregoing methods, and in an attempt to overcome the same, it occurred to me that the property of combination (or solution) of a high melting-point metal with one of low melting-point, at or about the fusing-point of the latter, would probably give the desired result—viz., complete fusion at a comparatively low temperature.

The following experiments were conducted to ascertain if oxygen could be determined accurately in this manner. The method adopted was as follows:

Ten grams of copper in one piece, and 20 grams of tin (previously fused in hydrogen), were placed in a boat and heated in a porcelain tube until complete fusion had taken place, dry hydrogen passing through the apparatus as in the fusion process. The water formed caught in the ordinary sulphuric acid and pumice U-tube.

Comparative determinations on the same samples were also made by the fusion method, and two by a *modified* Archbutt method, the gain in weight of the U-tube, and not the loss in weight of the metal, being taken as representing the oxygen present.

	Blast Furnace Method: Fusion Lump.	Heating Small Piece only.	Copper and Tin Lumps.
Sample I.	0·15	0·140	0·166
,, Ia.	prolonged heating.	0·166	—
,, II.	0·107	—	0·115
,, III.	0·163	0·174	0·188
,, IV.	0·132	—	0·146
,, V.	0·161	—	0·194
,, VI.	0·021	—	0·022

A blank experiment was made, using 10·9 grams of copper previously fused by the blast in hydrogen, and 24·1 grams of tin, also fused in hydrogen: the gain in weight of the U-tube amounted to 0·0001 gram.

To ascertain the limit of size permitted by the Archbutt method, an experiment was conducted on strips of copper, the dimensions of which were $\frac{1}{32}$ inch by $\frac{1}{16}$ inch by 1 inch. The metal was heated in a glass combustion-tube at as high a temperature as possible without undue softening of the glass.

The amount of oxygen found was 0·061 per cent. The heating was then continued for thirty minutes, but there was no increase in the weight of the U-tube.

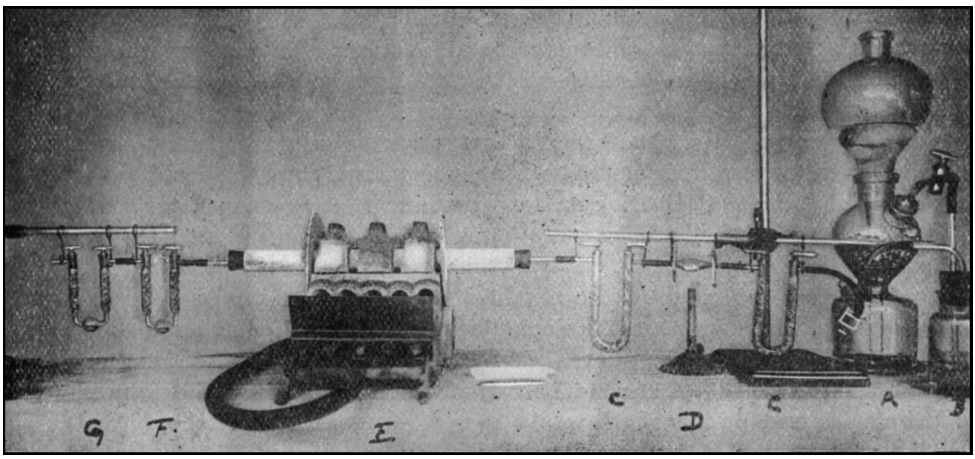
The original amount found was 0·115 per cent. (No. 11); therefore a deficit of 0·054 remains unaccounted for.

Twenty grammes of tin were then added to the metal used in the above experiment and the whole fused; the amount of oxygen remaining was:

Oxygen ... ... 0·050 per cent.

—or a total of 0·111 per cent., instead of 0·115 per cent. obtained by the tin solution method. A reasonably close agreement.

A further experiment was made on copper wire of 0·035 inch diameter. Ten grams were heated in the furnace to a red heat for two and a half hours, at as high a temperature as the glass tube would stand. The gain in weight amounted to 0·0075 gram. It was reheated for another period of one hour, and when weighed



A, Hydrogen generator; B, wash-bottle; C, C, sulphuric acid and pumice drying-tubes; D, platinized asbestos heated with Bunsen burner; E, furnace with porcelain tube; F, weighed absorption-tube; G, guard-tube.

the increase amounted to 0·0008 gram. Again, after a further heating for one hour no increase occurred. The amount found by the copper-tin solution method being 0·0100 gram, or a deficit by the heating method of 0·0017 gram.

The apparatus consists of a hydrogen generator, drying-tube, platinized asbestos bulb tube heated by Bunsen burner for the elimination of oxygen, drying-tube, connected with the porcelain tube, $1\frac{1}{8}$ inch external diameter, in which is placed the porcelain boat containing the copper and tin. The furnace consists of a section of Fletcher, Russell and Co.'s tube furnace, which is heated by a battery of five Bunsen burners. The absorption apparatus is a U-tube containing pumice saturated with sulphuric acid, and a similar one as a guard-tube. In conclusion, I have to thank Mr. R. H. Stanger, in whose laboratories the work has been carried out, and Mr. R. Doresa for the care he has taken in making the determinations.

Discussion.

The President (Mr. Bevan) having invited discussion,

Mr. Archbutt said that it was not altogether correct to speak of the "Archbutt method" for the determination of oxygen in copper. The method referred to was originally devised by Professor Hampe. He (Mr. Archbutt) had tested it, and, having found it to be a good method, had adopted it for his own use. He might claim also to have shown that it gave the whole of the oxygen without the trouble of fusing the copper, which was necessary in Mr. Blount's method. As he had mentioned in describing the method before the Society, comparisons of the two methods had been made, with the kind assistance of Mr. Blount; and with one exception, for which an explanation was given, the results had agreed very well. He was therefore not prepared to find that an increased quantity of oxygen was obtained as the result of adding tin to the copper. He did not understand why, in the experiment in which 0·061 per cent. of oxygen was obtained after one hour's simple heating of the copper in hydrogen, more was not obtained after heating for a further thirty minutes, if the copper contained more. The action certainly commenced on the outside, but he thought it gradually penetrated to the interior; and although he could quite understand that the oxygen might be given up slowly if the piece of copper were thick, he thought it would have been all given up eventually. He had not, however, experimented on the subject, and therefore could only express surprise at the result of Mr. Dickson's experiment. Since his paper was read he had used some specially-made silica glass bulbs, which could be heated to a very high temperature. He had found, however, that this was not really necessary, for practically the same results were obtained by heating to a good red heat in an ordinary Bohemian glass bulb. Sometimes a trifling amount more oxygen was obtained at the high temperature. Mr. Dickson seemed only to have noted the increase in the weight of the U-tube. He (Mr. Archbutt) took the loss of weight of the copper, and he thought it would have been more satisfactory if Mr. Dickson had done this as well as weighing the U-tube, because the one would have checked the other. The method which he used was, of course, more suitable for copper in very small turnings, which he could obtain without difficulty. If he had to deal with copper in large lumps, he should probably prefer such a method as Mr. Dickson's. His remarks had been called forth mainly by the discrepancies, which he should not have expected, in the analytical figures.

Mr. Blount said that the author's process was certainly a very ingenious one. The only objection he could urge against it was that he did not like adding unnecessary materials in large quantity in any process of analysis.

Mr. J. H. B. Jenkins said that, in connection with the method used by Mr. Archbutt, the concave surface of the fine copper turnings was full of fissures and minute cracks, so that the surface actually exposed to the reducing action of the hydrogen was very great; and this fact should be borne in mind in comparing the behaviour of these small pieces of turnings with that of a solid block.

Mr. Dickson, in reply, said that the whole question of the suitability of the different processes turned upon the state of division in which the metal was obtainable. Drillings or turnings were composed of very fine scrapings, agglomerated,

perhaps, by the action of the tool, but still in a very fine state of division; and he quite agreed that, where it was possible to get the copper so finely divided, the whole of the oxygen could be estimated by a simple heating process. With a wire, however, it was difficult, if not impossible, to get the copper into such a fine state of division, and in that case a process of mere heating would fail. His reason for not weighing the copper was simply one of convenience, the experiments having been conducted in tubes. He had started with the idea that the heating method was quite a good one when the particles were fine, and had not considered it necessary to go any farther under those conditions. As to the effect of heating when the pieces of copper were larger, he showed a tube in which some pieces of copper wire had been heated to the highest temperature available, and, although the severity of the treatment was apparent, the attempt to obtain the whole of the oxygen in that way had distinctly failed.

Mr. ARCHBUTT said that it would probably have been better had the heating not been so severe.

Mr. DICKSON, however, thought that the higher the temperature the better the results would be.

The PRESIDENT said that the fact that Mr. Dickson had obtained a larger proportion of oxygen than either of the other processes yielded seemed to be in his favour, as he had shown that practically nothing was obtained in the blank experiment.