Mn present. Per cent.	Mn found. Per cent.	Mn present. Per cent.	Mn found. Per cent.
0.415	0.423	...	....
0.415	0.414	...	....
0.415	0.414	1.00	0.990
0.415	0.420	1.00	0.989
0.442	0.440	1.00	0.982
0.442	0.460	1.00	0.980
0.442	0.460	1.00	1.010

LABORATORY OF THE E. AND G. BROOKE IRON CO.,
BIRDSBORO, PA.

THE RAPID DETERMINATION OF CARBON BY COMBUSTION.

BY GEORGE AUCHY.

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SOME experiences with the dry method of combustion having led the writer to the suspicion that the employment of a rapid rate of gas flow is occasionally attended with loss on account of incomplete oxidation by the copper oxide of the products of ignition and combustion, it was thought that it might be advantageous to use the Shimer apparatus with the modification of having the copper oxide in the crucible instead of in a tube.

In this plan the products of combustion are not forced through the copper oxide by the current of oxygen, but are carried in through the apparatus by the current of oxygen *after* oxidation by the copper oxide has been effected. Therefore, the time of contact with the copper oxide is entirely independent of the rate of gas flow employed, and the carbon compounds will be just as thoroughly oxidized by the copper oxide with a fast as with a slow rate. The carbon dioxide is formed and driven to the top of the layer of copper oxide by the heat of the blast-lamp. Is this period of contact with the copper oxide sufficient for complete oxidation? If so, the rapidity of the passage of oxygen is limited only by the capacity of the potash solution to absorb carbon dioxide rapidly and completely. With solution of 1.400 sp. gr. this capacity is very great.

This plan of having the copper oxide in the crucible had been tried by Dr. Shimer himself who in his original article speaks of an experiment in which the carbon sponge was covered with asbestos coated with copper oxide. The result, however, was a little low. It seemed to the writer to be worth while to do more experimenting in this direction. A new form of large size crucible was used. The one-half-hour-dried carbon residue and asbestos felt of good thickness, which had previously been transferred (without disturbing its shape) from a Hirsch funnel, smallest size, into a small watch-glass for drying, was transferred to the crucible, *carbon side up*[1]; the wad of asbestos used for cleaning out the funnel was placed to one side; both were pressed down evenly on the bottom by a thick glass rod with a spread-out end; the crucible was filled to nearly the point of reaching the inlet tube of the stopper when in position, with finely ground copper oxide freed completely from carbon, and thoroughly tested in this particular by preliminary blank tests; the copper oxide tube was omitted altogether; the blast-lamp was used for ignition, and purified oxygen (air should also serve) was used at a four- or five-bubble rate, allowing ten minutes for the combustion; the oxygen was used throughout, and the potash bulb was weighed filled with oxygen.[2] The following results were obtained:

No.	Regular combustion, 1-bubble rate. Carbon, per cent.	Shimer modified, 4- to 5-bubble rate. 10-minute combustion. Carbon, per cent.
3886	0.85	0.84
3642	0.96 0.95	0.95 0.96
3906 S	1.16	1.145
3819	1.015	1.00
3921	1.13	1.115
3818	1.11	1.12 1.115
3910	1.46 1.46	1.45

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[1] After the last washing of the carbon sponge, some of the copper oxide (3 spoonfuls—smallest size horn spoon) diffused in water was poured on and sucked down.

[2] This was proposed by Dr. Dudley who, however, deprecates the use of this plan in important work. In this the writer must disagree with him. But there is a precaution to be observed in this connection. When the bulbs filled with oxygen stand over night they lose weight, probably because of diffusion between the air on the outside and the oxygen within. We have found this loss to be about 0.0030 gram, but it would doubtless vary somewhat in different cases. A correction for this loss must be applied to the first combustion each morning, or, better, the bulbs should be filled with oxygen before use, whenever they stand for some time between successive combustions.

No.	Regular combustion, 1-bubble rate. Carbon, per cent.	Shimer modified, 4 to 5-bubble rate. 10-minute combustion. Carbon, per cent.
3911	0.79	0.795
3868	1.005 0.995	0.99
3954 L 2	1.12	1.105
3952	0.86	0.85
3979 S	0.925	0.19
1.03 S	1.03	1.034
3995	0.90	0.90
3996	0.925	0.92
3697	1.24	1.235
3697 S	1.245	1.245
3685	0.99	0.98
1.08 Std.	1.08	1.086
3929	0.765	0.76
3982	1.06	1.065 1.07
3989 S	1.04	1.025
3991	1.005	0.99
3988 L 2	1.13	1.14

These results, for practical purposes, are very fair indeed. The results are, on the average, 0.007 per cent. low. The doing away with the copper oxide tube is a gain in simplicity.

It was thought that possibly the copper oxide in the crucible, on account of its finely ground condition, might act as an absorbent for the chlorine and hydrochloric acid of the carbonaceous residue. The 1.03 carbon standard was determined with the glass bead tube (the hydrochloric acid absorbent) omitted, result, 1.03 per cent., indicating that all the hydrochloric acid and chlorine is absorbed by the finely ground copper oxide, and that the glass bead tube may therefore also be dispensed with. It would, however, require a considerable number of tests to prove conclusively that all the hydrochloric acid is retained in the crucible in every case. In the meantime, it is some gratification to know that the copper oxide at least helps to absorb it.

In the original Shimer method, the asbestos is placed in the crucible, carbon side down. It might be thought that in using this modification in order to insure a complete combustion, it would be well to make the asbestos layer as thin as possible.

The writer at first, in filtering, covered the asbestos with a layer of the copper oxide, and at the end of the filtration scraped off the carbon and copper oxide from the asbestos, and placed these in the crucible directly in the bottom and to one side of the asbestos. But in this plan the volatile portion of the carbonaceous residue seems to be driven off before the copper oxide has become sufficiently heated. Results 0.03 per cent. low were obtained in this way in cases of steel which by the process directed above came either right, or only 0.015 low. A good layer of asbestos seems therefore essential, and the writer also makes it a point not to dry thoroughly previous to combustion, but never longer than one-half hour at 90° C. The copper oxide, is, however, thoroughly dried by a short ignition in a platinum dish or crucible before each combustion.

Besides the results given above, there is other evidence which gives the writer confidence in this process. At these works it is the custom to take two samples from each heat of basic open-hearth steel, determining the carbon (and phosphorus) in each.[1] As these two samples may differ in carbon, a close agreement in the results in any one case, proves nothing, with regard to accuracy of method. But many such agreements—one test regular, the other by the Shimer modified—are entitled to be considered as evidence to the accuracy of the latter process. In cases of disagreement and the Shimer result coming low, the latter for a while were repeated by the regular process, and the results of the above table were for the most part obtained in this way.

This plan has now been abandoned on account of risk of getting into "hard" or "soft" spots twice in succession. We now mix together equal weights of drillings from six different samples (pieces of runner) of a heat, and make one combustion.

It is the writer's custom in his daily work to report carbon results in whole numbers or halves, as the case may be, and this custom has been adhered to in this table.

A very great improvement in connection with the carrying out of carbon determinations, and one which once tried will not be abandoned, is Dr. Sargent's plan of rechlorinating the used double

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[1] In cases of a greater variation than 0.06 per cent. a third sample (or fourth, if necessary) is taken. The average is taken, not of all the samples, but only of the two whose results agree within 0.06 per cent. The other, or others, are ignored, experiments having indicated that they are merely " hard spots " or " soft spots " as the case may be. Our experience is in agreement with Wahlberg's that low and medium carbon steels are more apt to vary than high carbon steels, and that phosphorus is more likely to vary than carbon.

chloride solution. There is, however, a source of error connected with its use that deserves to be known.

It would seem that solution of drillings should not be accomplished by allowing to stand at rest over night, except a brisk current of air, or brisk stirring by the machine be first used to drive off free chlorine. A current of air, or brisk stirring, will drive off the chlorine before it has time to do any harm to the separated carbon.

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NOTES.

Filtration in Determination of Crude Fiber.—All analysts of food products, or feeding-stuffs, are familiar with the difficulty which is often experienced in filtering off the acid and the alkaline extracts in the determination of crude fiber by the official method of the Association of Official Agricultural Chemists. Hence, I wish to suggest the following modification of the usual procedure, which I have found to give very satisfactory results.

Select a funnel of sufficient size to contain the entire bulk of the mixture to be filtered and fit into its point a small platinum filtering cone. Introduce enough ignited asbestos wool to fill the cone a little more than full. Upon moistening, the asbestos wool softens into a fluffy mass which may be drawn down by suction into a close firm filter. The mass to be filtered is now poured into the funnel with the usual care to avoid disturbing the asbestos layer, and suction applied. The filtrate obtained in this manner has always been found to be free of suspended particles of fiber. Filtration is very rapid except when working with finely ground flour, or spices, which tend to clog the filter and impede the flow of the filtrate. In such cases, place the funnel in an ordinary jacket of boiling water or steam, in order to secure hot filtration, transfer the entire mass to be filtered to the funnel and apply suction as usual. The filtration will then proceed at the proper temperature and without further attention from the operator, thus avoiding the two chief objections to the use of the Gooch filter as recommended by the official method. After filtration and washing are completed, transfer the contents of the funnel to a platinum dish,