per cent boron. The effectiveness of No. 1 Grainal is greater than would be expected from the boron alone. We have also used in the past Grainal No. 10, which was a zirconium-titanium alloy.

These additions are added in the ladle after the aluminum, Alsifer, or other strong deoxidizers. They can also be added in the molds, preferably if the material is preheated.

Our additions range from 1.8 to 3.6 lb. per net ton of ingots, depending upon the application involved. For the 0.5 per cent boron alloy, this amounts to an addition of 0.0005 to 0.001 per cent boron added. With our present analytical methods, I do not believe we are justified in figuring the percentage recovery. After all, an analytical error of 0.0005 per cent boron would be 50 to 100 per cent of the amount added. In the last analysis, the real test of effectiveness of the intensifier treatment is the hardenability of the steel as measured by the Jominy test or some other test, and not the analytical figure for recovery of the element added. With Grainal No. 1, our vanadium recovery is apparently close to 100 per cent.

We find that the effectiveness of the intensifiers is not strictly in proportion to the amount added according to the multiplicative factors of Grossmann and others, which seem to hold true for the additions of the usual alloying elements in larger amounts. The additions seem to be somewhat more effective on some steel compositions than on others. In other words, we believe the effect is to some extent a specific property of the steel being treated as well as of the intensifier, and is not calculable by a constant multiplying factor. This is a tentative conclusion based on indications of current evidence.

For boron determinations, we use the colorimetric method with quinalizarin. Our results are reproducible to 0.0002 to 0.0003 per cent boron by the same or different analysts. We have been able to check the new Bureau of Standards standard sample No. 151, which has a certificate value of 0.0027 per cent boron, within that range; that is, we get from 0.0025 to 0.0030 per cent boron in this sample.

We used to report boron to the nearest 0.0001 per cent. We are now attempting to read in between standards and report to the nearest 0.00001, but we do not put too much faith in accuracy better than 0.0005 per cent.

The Chairman.—The last discussion will be by Mr. L. C. Flickinger, of the Youngstown Sheet and Tube Company.

**Methods and Accuracy of Boron Determinations by the Quinalizarin Method**

*By L. C. Flickinger*

Boron as determined by the quinalizarin method is dependent upon the change in color of the organic reagent (tetrahydroxy-anthraquinone) in the presence of extremely small amounts of boron. That the reagent is sensitive to small amounts of boron is indicated by the fact that a very decided color change occurs on the addition of one micromilligram of boron, and that 0.2-micromilligram additions are distinguishable.

The method is especially suited to the determination of boron in steel of the order of 0.0005 to 0.0030 per cent, for the reason that fairly large samples may be used and the colors developed therefrom happen to fall within the sensitive range of the indicator. We have found that with colors produced by the presence of boron exceeding the equivalent of 0.0030 per cent differentiation is difficult. It should be recognized, therefore, that in order to obtain the greatest accuracy, modifications must be made in the method in order to produce colors that fall within this narrow band. This may be accomplished by
different initial weights of samples, aliquot portions and various types of standards.

In order to use this procedure successfully it is quite essential to have a good grade of quinalizarin and sulphuric acid of proper quality and strength. Standard colors should show no evidence of fading over a long period of time. Colors developed from samples of steel should be equally permanent. Unless such conditions hold, accurate determinations cannot be made. The procedure in detail was published in *Steel* of April 5, 1943.

In order that the producer and the consumer of boron-treated steel may be enabled to correlate test data and performance, it is essential that the amount of boron be known to the extent of its effectiveness. Since by the use of the quinalizarin method we have been able to detect the equivalent of 0.0001 per cent addition and have consistently checked 0.0002 per cent on many samples, we feel that the method conveniently affords this information. The method is quite adaptable to checking the efficiency of boron additions, the extent of fading, and relationship to hardenability at a minimum expenditure of time.

We have been able to add various amounts of boron as boric acid to steel samples and have had excellent recoveries; for example, to steel analyzing 0.0024 per cent boron we have added the equivalent of 0.0010 per cent boron and obtained 100 per cent recovery. Also, we have regularly obtained good recoveries upon adding small amounts of boric acid to boron-free steel. Combinations of previously analyzed boron steels have also checked quite satisfactorily.

While the procedure was established for carbon and molybdenum steels, we have found that with some modifications the method can be used for rather highly alloyed steels. The preparation of the standards in such cases is important. By the addition of calculated amounts of the alloying elements to the standards, colors or shades are obtained that closely approximate the colors obtained from alloy-steel samples.

As mentioned, a good grade of quinalizarin is most essential for accurate work. The prewar product was recrystallized by the Eastman Kodak Co. from an imported crude. With this crude unavailable, much work was required before an equivalent product could be marketed. The finished product varies from batch to batch, some showing excellent properties and others being almost worthless. In our efforts to make the method more available, we have checked in our laboratory the various batches of quinalizarin made by the Eastman Kodak Co., and have advised whether or not the product was sufficiently sensitive for the colorimetric boron determination. However, the chemist must satisfy himself that his quinalizarin at hand meets the requirements mentioned, in order that the method may yield the best results.

On that analytical question, I want to add that vanadium appears to interfere with the insoluble fraction but the addition of a small amount of ferrous sulphate removes that interference.

P. A. MacIsaac.—Only recently we have conducted tests on additions of Silcag to 40 C steel. We have no data on recoveries of boron additions or accuracy of determinations.

T. S. Washburn presiding

The Chairman.—I would like to point out that anyone who has not had an opportunity to offer discussion as we have had to drive through with the program is asked to send it in, as it still can be presented in the Proceedings.

I want to thank you who have presented discussions and the others for their indulgence.

The session adjourned at 5.10 p.m.