and one can only make a rough estimation, as a rule. The relations of the colors of the plate to the percentage of sulphur is shown by the following table :

No color,	.	.	.	0.00 per cent.
Slightly yellowish,	.	.	.	0.01 "
Yellow,	.	.	.	0.02 "
Yellow-brown,	.	.	.	0.03 "
Brown,	.	.	.	0.04 "
Blue,	.	.	.	0.20 "

Between brown and blue there are numerous variations in color. A certain red tint signifies 0.10 per cent., but this can only be learnt by practice.

APPENDIX.

COLOR TEST FOR CARBON IN IRON AND STEEL.—By V. EGGERTZ.

(Translated from the Swedish by M. Troilius.)

The first description of this method appeared in the *Jernkontorets Annaler*, of 1862, page 54, and in the *Berg- und Hüttenmännische Zeitung*, 1863, page 373. Subsequently, several additions were made to this description in the *Jernkontorets Annaler*, 1874, page 176, and in the *Berg- und Hüttenmännische Zeitung*, 1875, page 440. At that time I considered that it would be practically sufficient to determine the carbon in tenths of a per cent., but I now find that even hundredths of a per cent. are required.

For commercial purposes, however, iron and steel is generally stamped with only whole or half tenths of a per cent. of carbon, it being chiefly for the softer irons and steels with carbon between 0.10 and 0.25 per cent., that the greater accuracy in determining the carbon is required.

Pure hydrate of oxide of iron, containing 0.1 gramme of iron and free from chlorine, will give a yellow-greenish solution when dissolved in 2.5 cc. of nitric acid, specific gravity 1.2. The solution will lighten somewhat on addition of 1.5 cc. of nitric acid, but not so much as when water is added instead of nitric acid. When hot, the solution has a much darker color than when cold. By adding 4 cc. of water in either of these cases, so as to make the bulk up to 8 cc., the iron color will be totally got rid of. Hence the rule for color tests for

carbon, that the solution of iron must be diluted with a volume of water at least equal to the volume of nitric acid used for dissolving the iron, and that the volume of the solution must not be smaller than 8 cc.* when the color is to be observed.

The nitric acid as well as the water must be entirely free from chlorine or hydrochloric acid, which otherwise will give a yellowish color† to the solution, even if present in only very small quantities. The quantity of nitric acid should correspond to a certain extent to the amount of carbon supposed to be present in the iron. For iron containing carbon 0.25 per cent., 2.5 cc. are thus used for each 0.1 gramme, for 0.3 per cent. 3 cc., for 0.5 per cent. 3.5 cc., and, finally, for 0.8 per cent. 4 cc. For steels with more carbon 5 cc. should be used. The last-named quantity is also to be used for white pig irons, of which, however, 0.05 gramme only are carefully weighed out for each test.

When the amount of carbon is quite unknown 2.5 cc. of nitric acid are added to start with, and then more until it is indicated by either the color or by the quantity of separated carbon-substance that no more acid is wanted. A little more acid than the quantity just mentioned does no harm, if only an equal volume of water is afterwards added. Thus for white pig iron 7 cc. may well be taken so as to prevent a too quick precipitation of organic matters after diluting. With too little acid a too dark solution will be obtained. Thus, if 0.1 gramme of steel containing 0.8 per cent. of carbon is dissolved in 2.5 cc. of nitric acid only, instead of 4 cc., the color of the solution will, after dilution, represent a percentage of carbon equal to about 0.9 per cent.

The iron for testing is to be finely divided either by filing,‡ by aid of a good clean file, or by means of a boring machine (which is preferable), by rubbing with some hard steel edges, or by crushing in a steel mortar, if it is very hard.

The test-tubes for dissolving the steel should have an internal diameter of 15 mm. and a length of 120 mm. The tubes are to be cleaned

* For each 0.1 gramme of iron.—M. T.

† Only 0.0001 gramme of chlorine produces a distinct yellow color in a solution of 0.1 gramme of iron (from hydrated oxide of iron) in 2.5 cc. of nitric acid. This color can be noticed even on addition of 1.5 cc. of nitric acid and 4 cc. of water. Upon further diluting, the coloration becomes less distinguishable.

‡ Worn-off particles from the file may, however, greatly increase the percentage of carbon in the iron.

out by means of coarse filter-paper rolled together and well cut at the edges. 0.1 gramme of the iron or steel for testing, or in the case of white pig, 0.05 gramme, carefully weighed, is then put into the tube and 2.5 cc. or more of nitric acid of specific gravity 1.2 added. The acid may be readily measured off in a little measuring glass of 10 mm. diameter and 75 mm. length, and graduated so as to show whole or half cubic centimetres. The tubes are covered with small watch-glasses (23 mm.) and put into a cylindrical vessel of copper-plate having a height of 100 mm. and a diameter at least of 120 mm. This vessel is covered with a copper plate provided with a thermometer and holes for the tubes. The holes are marked with engraved figures. The vessel is to contain water and a few grammes of paraffin to prevent the evaporation of the former, and is heated to 80°C. by means of a gas flame. This temperature should be kept up all the time. The tubes are shaken now and then, and the solution is completed when no further gas bubbles appear. One and a half to two hours, or sometimes longer, time is required to effect complete solution, the time depending upon the amount of carbon in the iron.

Up to the present time this has been the ordinary way of proceeding, but it is shown by experience that the keeping up of the temperature of 80°C. requires more care than has been generally taken. Of course, this has been of less influence as long as fresh weighings of the standard steel have been dissolved along with every set of samples for testing. But if it is desired to avoid the frequent dissolving of standard steel and to use permanent standard solutions (see further down) then the iron must always be dissolved at the same temperature. For this purpose it is the safest way to put the tubes into boiling water. By this means the time for dissolving may be shortened to about three-quarters of an hour, and the color of the fluid will then turn out a little darker than by dissolving at 80°C. If, occasionally, greater rapidity is required the solution may be effected by boiling over a lamp flame; between the flame and the tubes there should be put a brass gauze with the tubes resting on it. The color will in this case generally be somewhat darker than when dissolved at 100°C. The reason for heating to 80°C. instead of 100°C. has been, that in the latter case there is sometimes a reddish-yellowish deposit formed on the glass; when brought down into the fluid this matter has made the solution turbid. The deposit must

be dissolved by shaking if possible, otherwise it must be filtered off. It consists of nitric acid and oxide of iron. This is borne out by the fact that it also arises by heating a solution of pure hydrate of oxide of iron in nitric acid.

All gas bubbling having ceased, the tubes are taken out of the hot water and put into a beaker containing cold water. They should be covered in some way so as to be totally excluded from the daylight, which would soon give too light a color to the solutions. If protected in this way the solutions will retain their color for many days. The burette for determining the carbon should have 30 cc. capacity and be graduated into tenths of cubic centimetres and be provided with one large and one small mouthpiece at the upper end. The solution is brought into this burette through a filter, if necessary, as for instance if the fluid is turbid or graphite is present. Then distilled water is to be added; the quantity of water, including the water used for cleansing the test-tube, must be at least equal to the quantity of nitric acid used, and the total volume must not be less than 8 cc. when it is to be compared with the standard solution. The standard solution is prepared by dissolving standard steel. It should be diluted with water so as to make each cubic centimetre of the same to correspond to 0.1 per cent. of carbon. It may also be carefully diluted with more water so as to have each cubic centimetre = 0.05, 0.02, 0.01 or 0.005 per cent. of carbon, of 0.1 gramme of iron or steel used. For example, dissolve 0.1 gramme of standard steel, containing 0.8 per cent. of carbon, in 4 cc. of nitric acid and add water to 8 cc. The solution is to be carefully mixed after each addition of water; otherwise the lower part of the fluid will appear too dark. After mixing, at least one minute should be allowed for the fluid to run down along the sides of the burette before reading off, which is done at the upper border of the fluid.

The above-mentioned standard solutions, representing the total, half, fifth, tenth and twentieth of the standard, may be marked and used as follows:

	Percentage of carbon per cc. for 0.1 gramme of iron.	To be used for iron with a percentage of carbon =
N	0.10	0.8 and more
$\frac{1}{2}$ N	0.05	0.4 — 0.80
$\frac{1}{5}$ N	0.02	0.16 — 0.50
$\frac{1}{10}$ N	0.01	0.08 — 0.25
$\frac{1}{20}$ N	0.005	0.04 — 0.08

The smallest percentage of carbon* found in iron in this country is 0.04 per cent. A good daylight is required when using the last-named standard solution, and this remark applies to all colorimetric tests.

The dilution cannot be carried farther than twenty times the original solution in the case of using the ordinary burettes of about 12 mm. diameter. But one can sharply distinguish the difference in color between ordinary distilled water and a standard solution diluted to forty times its original volume and, therefore, if one uses burettes of 24 mm. diameter, it will be possible to approximately determine the carbon in iron containing only 0.02 per cent., supposing such soft iron to be produced. In this case measure off 1 cc. of the "N" solution in a delicate measuring tube and dilute to 40 cc. Thus 1 cc. will be equal to 0.0025 per cent. of carbon. Pour this standard solution into the 24 mm. tube. This tube should exactly correspond with another graduated 24 mm. tube, in which 0.4 gramme of the iron for testing is to be dissolved in 10 cc. of nitric acid, the solution diluted to 32 cc. and then more until the colors agree. For instance, if the colors agree at 35 cc. bulk, you find the percentage of carbon thus:

$$0.0025 \times 35 \div 4 = 0.022 \text{ per cent.}$$

Hitherto it has been the usual practice to compare colors by holding a piece of thin filter-paper behind the tubes. In this way, however, one is much dependent on the distribution of light in the room, which, therefore, ought to have only one window for this purpose. For most people's eyes the left-hand tube will appear somewhat darker than does the right-hand tube. For this reason it was originally prescribed that the tube containing the solution of iron or steel for testing should be kept to the right so as always to have it in the same position. In order to avoid being dependent on the room I work in, and to lessen the difference in color between the left- and right-hand tubes, I now most advantageously use a contrivance in the shape of a little camera, into which the tubes are put. The camera is made of wood 6 mm. thick, and is open at both ends. The inner sides are blackened. The internal height is 80 mm., width of forepart 26 mm. and width behind 120 mm. The tubes are put in the forepart through apertures in the upper part of the camera and steadied at the bottom by a gutter of copper plate and at the upper part by a brass wire. The

* By means of the iodine method 0.038 per cent. of carbon was once found in 8 grammes of a Swedish Lancashire iron.

box is closed at the wider end by means of thin filter-paper* nailed to it. Burettes and tubes should be closely of the same quality as to color, etc., and be wiped with clean linen before being put into the camera.

We have of late been using as standard steels two Bessemer steels, one of which contains .80 per cent. and the other one .16 per cent. of carbon. These steels are kept in pieces 12 mm. square. Samples from these pieces are always taken at right angles to the longitudinal axis. The carbon in these steels was determined by me by aid of the iodine method, as described in the *Jernkontorets Annaler*, 1862, page 47. 5 grammes were taken for each test. Three analyses of the harder steel showed .79, .80 and .82 per cent. of carbon, and two analyses of the steel containing .16 per cent. agreed very closely. These carbon determinations also agree very well with those of Dr. A. Tamm, whose results by combustion are described in the *Jernkontorets Annaler*, 1874. The iodine method is now modified, so that instead of using iodine direct for dissolving the iron or steel, a solution of iodine in iodide of iron is used. This is on account of the difficulty of obtaining pure iodine. The solution now used is prepared by dissolving 10 grammes of iron in 50 grammes of iodine and adding another 50 grammes of iodine, which is readily dissolved. The solution is filtered and water passed through the filter until a bulk of 100 cc. is reached. 10 cc. of this solution are required for 1 gramme of iron. With regard to the diminution in weight of filter-paper on being treated with acid, we have generally used platinum filters for collecting the carbonaceous matter. However it is somewhat difficult to obtain good platinum filters and, therefore, it seems preferable to use filter-paper which has been treated with hydrochloric and hydrofluoric acids,† whereby the inorganic ingredients are almost completely extracted. A filter of 60 mm. diameter thus treated does not give more than 0.0001 gramme ash. The carbonaceous mass should be dried on a water-bath at a temperature of 95°—98°C. For this purpose the mass is put into a crucible, and the crucible then put into a glass tube, closed at one end and having a length of 130 mm. and diameter of 35 mm. The upper end of the tube is closed by means

* This can also be effected by applying a screen holding a glass adapted to the eye as to convexity or concavity, and having about 40 mm. diameter.

† Fresenius' Zeitschrift, 1879, p. 582.

of a cork, through which hangs a thermometer. The crucible may be inserted or removed by means of a crooked brass wire.

Even for white pig iron better results than could be expected* are now obtained by the color test. The difficulty in this case lies in the use of only 0.05 gramme, and still diluting to a very large volume, which renders a slight error in observation very important. The solutions of white pig iron should be rapidly read off, for the reason that they soon become turbid with a precipitate of organic matter. This inconvenience may, to a great extent, be removed by using 7 cc. instead of 5 cc. of nitric acid.

The mode in which the carbon is present in the steel as "cement carbon," or "hardening carbon," does not influence the color test otherwise than to render the solution less dark in the latter case, when the carbon is more intimately combined with the iron. Thus a steel containing 0.8 per cent. of carbon showed only 0.55 per cent. after strong hardening and crushing. After reheating the steel to a low red heat, the original percentage of carbon was again obtained. By aid of the iodine method, 0.8 per cent. of carbon was found in 5 grammes of the hardened steel. A piece of iron containing 0.3 per cent. of carbon, upon being hammered down cold from 12 to 6 mm. square, showed the same carbon before and after hammering.

Different kinds of iron and steel behave very differently during dissolving, some solutions at once becoming colored, others only after heating. The final color, however, after diluting with water, generally tallies very well with the color of the standard. In case there should be any difference in tint—yellow or brown—it is best to notice the intensity of colors, which can be done by holding a thin filter-paper behind the tubes and observing through which tube one can most easily see certain dark points in the paper, etc. It is a matter of course that some persons are able to use the color test with greater advantage than others, according to their different eyesights; but eyesight may be considerably improved in this respect by practice, as shown by experience; in fact, only very few have hitherto been found incapable for this work.

The following experiments have been made with regard to the influence on color exercised by foreign substances in iron and steel:

Manganese.—0.05 gramme, contained in carbonate of manganese,

* Jernkontorets Annaler, 1874, and Berg- und Hüttenmännische Zeitung, 1875, p. 440.

was dissolved in 2·5 and 5 cc. of nitric acid. The color thus obtained was brown, owing to the presence of a little oxide of manganese. After heating to 100°C. a small precipitate was obtained (in all probability hydrated dioxide), and the solution assumed a slightly reddish color, which disappeared after diluting with water to 8 or 10 cc. These experiments show that the color test may be used for determining the carbon in ferromanganese. A ferromanganese containing 80 per cent. of manganese showed 4 per cent. of carbon by color test.

Phosphorus.—0·001 gramme, contained in phosphate of sodium, was added to a solution of 0·1 gramme of iron (as hydrated oxide) in 2·5 cc. of nitric acid and 2·5 cc. of water; no difference in color was noticed after this addition. Iron containing 5 per cent. of phosphorus is difficult to dissolve, and with 10 per cent. of phosphorus it is insoluble in nitric acid.

Sulphur.—0·001 gramme, contained in sulphate of magnesia, was added to a solution of iron in 2·5 cc. of nitric acid and 2·5 cc. of water. The color of the solution remained unaltered.

Neither did any alteration in color take place when 0·01 gramme of manganese, 0·01 gramme of phosphorus, and 0·001 gramme of sulphur (all contained in the above-mentioned salts) were added to a solution of 0·1 gramme of iron (as hydrated oxide) in 5 cc. of nitric acid and 5 cc. of water. The same quantities, when added to a solution of 0·1 gramme of standard steel with 0·8 per cent. of carbon, did not alter the color here either.

Copper.—0·001 gramme was dissolved in 2·5 cc. of nitric acid and 2·5 cc. of water. No coloring of the solution.

Silicon in iron and steel is, to a very great extent, dissolved in nitric acid on heating, even if lumps of silica be seen floating about at the beginning of the dissolving process. 0·4 per cent. of silicon in steel has no influence whatever on the color test. Silicious pig irons always contain graphite, which must be filtered off, together with the silica which may possibly have remained insoluble.

Tungsten in iron or steel is converted into tungstic acid (WO_3) when the iron or steel dissolves. It is insoluble, and must be filtered off.

Chromium.—0·002 gramme, contained in hydrated oxide, when dissolved in 2·5 cc. of nitric acid and 2·5 cc. of water, gave a grayish-bluish color to the solution. After addition of water to the double

volume this color grew less distinct. On addition of more water the color gradually grew fainter, and at 40 cc. it disappeared. It is difficult or even impossible to dissolve in nitric acid iron or steel containing much chromium.

Vanadium.—0.001 gramme, contained in vanadic acid (V_2O_5), when dissolved in 2.5 cc. of nitric acid, gives a faint yellow color to the solution. This color disappears after adding 2.5 cc. of water.

Nickel.—0.001 gramme, when dissolved in 2.5 cc. of nitric acid, gives a green color to the solution. This color remains visible after adding 2.5 cc. of water, but disappears at 8 or 10 cc. volume.

Cobalt.—0.001 gramme, when dissolved in 2.5 cc. of nitric acid, gives, as is well known, a red color to the solution. After adding water to 24 cc. this color is hardly visible, but may, however, not be considered removed before diluting to 40 cc.

In my paper of 1862 I pointed out the desirableness of having permanent standard solutions of *inorganic* matters, instead of those prepared by dissolving burnt sugar in spirits of wine, the latter solutions getting lighter after some time, particularly after prolonged exposure to the sunlight. Many propositions have been made of late, in various journals, with regard to this matter, and compounds of iron, cobalt and nickel, as well as potassium bichromate, have been recommended for the purpose. I have experimented upon several such mixtures, and lately, at the suggestion of Professor F. L. Ekman, I have tried the chlorides of iron, cobalt and copper, and I find that these yield the best results, as they allow of the production of any tint in yellow, brown or green. Such mixtures, diluted with water containing 0.5 per cent. of hydrochloric acid of 1.12 specific gravity, have been found to remain unaltered even after a long exposure to the sunlight. The mixture grows more and more yellow by adding hydrochloric acid drop by drop only. I prepare my standard solutions as follows: By adding to the neutral chlorides water containing 1.5 per cent. of hydrochloric acid for the chloride of iron, and 0.5 per cent. for the two other chlorides, I prepare solutions of a strength corresponding to 0.01 gramme of metal per cubic centimetre. Then 8 cc. of the nitric solution are mixed with 6 cc. of the cobalt solution and 3 cc. of the copper solution, and about 5 cc. of water containing 0.5 per cent. hydrochloric acid are added to the mixture. At a temperature of 18°C.* this solution shows exactly the same color as a

* This is an addition made since publishing the paper in the *Jernkontorets Annaler*.
—M. T.

solution of steel in dilute nitric acid corresponding to 0·1 per cent. of carbon per cubic centimetre. The solution may afterwards be diluted with water, containing 0·5 per cent. of hydrochloric acid, to any standard color required. The addition of water is almost directly proportional to the percentage of carbon. It need scarcely be mentioned that the quantities, 0·1 gramme for iron and steel, and 0·05 gramme for white pig iron, must be correctly weighed out when artificial standard solutions are used.

Now that permanent standard solutions can be obtained, it seems to be the right time to apply the old method for the determination of copper in ammoniacal solutions. This method was adopted by Mr. J. Blodget Britton, of Philadelphia (*Fresenius' Zeitschrift*, 1871, p. 245),† for colorimetric tests on iron and steel. He uses several tubes of the same size and quality, having equal volumes of standard solutions of different strengths in the different tubes, and dissolves the steels for testing in similar tubes, and finally dilutes these solutions with water to the same volume as the standard solutions. By comparing these he is able at once to determine to the hundredth of one per cent. by using 1 to 2 grammes for each test. He used burnt coffee for his standard solutions, which he considered to be better than burnt sugar.

Greater accuracy in color tests may also be obtained by looking at the solutions in the tubes from above, instead of from one side. In this case, however, the bottoms of the tubes must be perfectly alike. A suitable light must be provided, and the columns in the tubes must all have the same height.

I trust that a strict observation of the directions now given will essentially remove the difficulties which have been experienced in using the color test, as well in this country as abroad. It should also be borne in mind that the properties of iron and steel are not dependent on the amount of carbon only.

Before concluding this paper I have to thankfully acknowledge the service rendered by Mr. C. G. Dahlerus, mining engineer, in working out the results now published.

* From Mr. F. O. Söderberg, at the School of Mines, Stockholm, standard solutions, artificially prepared, as well as standard steels, are obtainable. The permanent solutions are kept in close glass tubes, to each of which belongs a corresponding burette. Those having burettes may send them to him in order to get suitable tubes for the standard solutions.