crucible, covered, and annealed at a bright red heat in a muffle for fifteen minutes.

An interesting report was presented in 1908 by H. Kinder,[8] of the work of a chemical commission appointed by the German Society of Ironworkers, to inquire into the estimation of sulphur in iron and steel. The figures obtained show that the highest results were got by the use of strong hydrochloric acid, 1.19 specific gravity, and quick evolution, and that under these conditions the use of a red-hot tube is unnecessary.

In a laboratory where the ordinary routine consists in the analysis of samples varying from the best hæmatite iron to high phosphorus foundry iron, and from the purest transformer iron through the many varieties of steel used for steel castings to complex alloy steels, the applicability of a method for general use is severely tested.

I have found that many samples of iron and steel do not evolve their full sulphur contents after merely annealing in a non-oxidizing atmosphere, when treated with concentrated hydrochloric acid. MacFarlane and Gregory's method of annealing with cream of tartar, however, gave me excellent results with some brands of iron with which I had previously not succeeded; but with other brands, and particularly with nickel-chromium steels and other self-hardening material for which I was very anxious to find a quick method of determining the sulphur, the full amount was not obtained. I am not overlooking the fact that the method was only designed for irons.

My experience agrees with the finding of the German Commission as to the importance of using concentrated acid and rapid evolution. Moreover, in many experiments, I passed the gases from the absorption flask through a red-hot tube and into a second flask, but the amount of sulphide obtained in the second solution was negligible.

In order to find a more active reagent than cream of tartar, I tried various substances, and finally chose potassium ferrocyanide. The effect of heat upon this substance is to decompose it into carbide of iron, potassium cyanide, and nitrogen. The result is, that the drillings are more or less carbonized, and a strongly reducing atmosphere obtained.

Experiments made to determine the length of time required for annealing, indicated that with less than twenty minutes, low results are obtained. On the other hand, no advantage is usually to be gained by exceeding this period, whilst in the case of steel drillings, oxidation is then liable to occur.

Annealing experiments with different amounts of ferro-cyanide indicated that 0.25 gramme mixed with 5 grammes of the sample, was the most suitable amount. If more is used, cyanogen compounds may be driven into the absorption flask, especially if the condensing tube gets hot. The result is, that when the titration is made by adding iodine to the contents of the absorption flask without filtering, acidified only with acetic acid, an excess of iodine may be added beyond that required to dissolve the sulphide, without any colour being imparted to the solution. (A drop of starch solution added here gives the characteristic blue colour.) On adding dilute hydrochloric acid, the brown colour due to the excess of iodine appears, and the titration is proceeded with as usual, the result being unaffected, notwithstanding.

Experience has shown that in order to obtain correct results it is absolutely necessary to control the temperature of annealing. As will be seen from the table of results the correct temperature lies between 750 deg. C. and 850 deg. C.

If there are no means of measuring the temperature available, it is necessary to work a standard of known sulphur content with each batch of samples annealed.

An indication as to whether the sulphur is being evolved correctly is furnished by the colour of the precipitated cadmium sulphide. This should be distinctly yellow. A pale precipitate is usually associated with a low result, and suggests that the annealing has not been conducted properly.

In order to promote uniformity in the rate of solution, which is important, it is advisable that the drillings of the sample should be of the same degree of fineness as the standard, and of similar composition. Reference is made below to the use of a standard.

The method I have adopted is as follows: 5 grammes of the sample are mixed, as far as is practicable, with 0.25 gramme of pure finely powdered anhydrous potassium ferro-cyanide, and wrapped in one 9-centimetre filter paper if the sample is a graphitic iron, or two papers if it is a steel or white iron, placed in a small porcelain crucible (Royal Berlin O.A.), covered with the lid, and annealed at 750 deg. C. to 850 deg. C for twenty minutes in a closed muffle. After the annealing and subsequent slow cooling outside the muffle, the drillings should still be covered practically completely by the charred filter paper, if the temperature has not been above 850 deg. C. or they have not been in the muffle too long. I make a point of observing the appearance of the annealed drillings very closely, because low results are obtained if the paper is completely burnt away from the top of them. After cooling, the crucible is emptied into a glass mortar, and the slightly caked drillings loosened with a pestle. They are then brushed on to a small piece of stiff paper, which is rolled into cylindrical shape so that it will fit right into the neck of the evolution flask, and allow of the drillings being transferred to the flask without touching its neck or sides. The evolution flask is connected with a condensing tube 6 inches by 1 inch, containing about 2 inches of water, and standing in a conical beaker filled with cold water. A tube from this dips into a flask containing 60 cubic centimetres of the cadmium chloride solution, which again is connected with another flask containing more cadmium solution. Fifty cubic centimetres of concentrated hydrochloric acid are now added, and heat is immediately applied to the flask until the frothing of the contents indicates that the solution is well in progress. The flame of the burner used to heat the flask is lowered. As soon as the speed of the bubbles of gas passing through the cadmium solution begins to slacken, the flame of the burner is raised so that the liquid in the flask just boils. The boiling is continued so long as any appreciable amount of gas is given off from the solution. The apparatus is then disconnected, and iodine solution added in excess to the contents of the absorption flask — usually nothing is found in the safety flask; then 10 cubic centimetres or so of diluted hydrochloric acid (one part acid and two parts water) are added, and the liquid shaken to complete the solution of the sulphide. The excess of iodine is then titrated with sodium thiosulphate, starch solution being added when the iodine colour has nearly disappeared. (The air in the apparatus may be washed out by carbonic acid before the addition of the acid to commence with, and again at the end of solution of the drillings, if desired, though