lution and the heat of fusion of its solvent are among the most brilliant results of exact science.

This energy method, with its fruitful principle of virtual changes, is rearing the framework of a coherent theory of chemical processes, and it is through this development of a science of exact relations that we must hope to be relieved from the thralldom of unproven, frequently unprovable and always unnecessary mechanical hypotheses. Through this appears for the first time in the history of chemistry a prospect of dealing directly with facts as such, *and only with facts.*

WORK OF THE COMMITTEES ON THE "INTERNATIONAL STANDARDS FOR THE ANALYSIS OF IRON AND STEEL."[1]

By John W. Langley, Cleveland, Ohio.

THE history of the organization and work of these committees up to the present year is briefly as follows:

In the summer of 1888 it was the fortune of the present writer to present the subject of the desirability of establishing a set of samples of steel which should be analyzed with extreme care, in order that they might become standards to which scientific and commercial analyses of iron and steel could be subsequently referred; also, that greater uniformity in the results of analyses might be brought about, since these standards would bear towards analytical methods somewhat the position which the original units of weight and length, the gram and meter, or the pound and yard, do to the mechanical arts.

The plan met with hearty co-operation, with the result that committees were appointed in Sweden, Germany, France, England, and America to receive the material and see that the necessary analyses were executed.

These committees were constituted as follows:

In Sweden, Prof. Richard Åkerman; in Germany, by the authority of the minister of public works, the execution of the analyses has been entrusted to the Konigliche Chemisch Technischen Versuchungsanstalt, in Berlin; in France, Mons. Ferd.

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[1] Read before the World's Congress of Chemists, August 22, 1893.

Gautier; in England, the British Association for the Advancement of Science; in America, a joint committee from members of the University of Michigan and of the American Society of Civil Engineers.

This latter body appointed a committee of seven analysts as follows: Andrew A. Blair, Philadelphia, Pa.; Regis Chauvenet, Golden, Colorado; Thomas M. Drown, Boston, Mass.; Charles B. Dudley, Altoona, Pa.; John W. Langley, Cleveland, Ohio; Albert B. Prescott, Ann Arbor, Mich.; P. W. Shimer, Easton, Pa.

The material for analysis at first consisted of four samples of steel prepared in this country. To this was subsequently added a fifth standard selected in England by the committee for that country.

Reports of progress have been made by the English committee in the Reports of the British Association for the Advancement of Science, volumes for 1888, 1889, 1890, 1891, 1892, the last one particularly containing a full account of the work on the four original standards.

In the United States the attention of the committee was early drawn to discrepancies caused by different methods of analysis. They therefore prepared a set of supplementary samples known as the experimental standards and to be used only in reviewing methods of analysis. A full report of their work in this line will be found in the Transactions of the American Institute of Mining Engineers, volume **19**, 1890, and a brief one in volume **20.**

The Swedish committee through Professor Akerman has made a report which will be given shortly.

The outcome of the American committee's work on the modes of determining carbon resulted in calling attention to important errors likely to occur when using double chloride of copper and ammonium as a solvent for the steel owing to the great difficulty of obtaining this salt free from pyridin and other tarry products. They recommend substituting chloride of potassium for chloride of ammonium, which is found to be a complete remedy. They also found that contrary to the usual practice this solvent must not be neutral, but must be rendered strongly acid with from five to ten per cent. of its volume of strong hydrochloric acid.

They also call attention to the great tendency which chlorine compounds have to pass out of the combustion apparatus and to lodge in the absorption train, thus increasing the apparent weight of carbonic acid, and they give methods for arresting it.

During the past year this committee has continued its work. The communication from Professor Drown given below enumerates very fully the precautions necessary and some inevitable causes of variation in carbon determinations.

MASSACHUSETTS INSTITUTE OF TECHNOLOGY,
BOSTON, JULY 29, 1893.

PROF. JOHN W. LANGLEY, *Chairman, Committee on*
International Standards for the Analysis of Iron and Steel;

DEAR SIR: I give you herewith the results of my determinations of the carbon in the experimental steel standard. The sample examined was marked "hammered."

The amount of carbon found in six consecutive analyses was:

Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
1.050	1.057	1.054	1.030	1.054	1.053

These results were all obtained by precisely the same process and method of manipulation. The agreement is satisfactory with the exception of the fourth. I know of no reason why this result should have been lower than the others. The average of the six determinations is 1.0497. If the fourth determination is omitted, which I think it reasonable to do, the average of the remaining five determinations is 1.0536 per cent.

The process used was as follows: Three grams of the steel were treated with 200 cc. of a solution of copper-potassium chloride (300 grams to the liter) and fifteen cc. of hydrochloric acid (sp. gr. 1.2). After complete solution of the iron the carbon was filtered off on an asbestos-lined platinum boat, thoroughly washed with hydrochloric acid, and then with water until the washings gave no reaction with silver nitrate. After drying, the boat was put into a porcelain tube and the carbon burned in a current of oxygen.

The oxygen used was passed over red hot copper oxide, then through caustic potash, then over calcium chloride before it entered the porcelain tube. The tube contained at its farther end several inches of copper oxide and a coil of metallic silver, which were heated to redness before the carbon was ignited.

The gases on leaving the porcelain tube were passed, first, through a small bulb tube containing an acid solution of ferrous sulphate, then over anhydrous copper sulphate on pumice, and finally over calcium chloride before entering the potash bulb. The latter was provided with a small extension tube containing calcium chloride.

Before beginning each combustion two or three blank combustions were made to see that there was no source of error in the apparatus itself. The oxygen was swept out with air (free from moisture and carbonic acid) and the potash bulbs were closed with rubber tubing plugged with capillary glass tubes.

The silver coil, ferrous sulphate, and anhydrous copper sulphate were all used for the purpose of absorbing hydrochloric acid or chlorine. In Mr. P. W. Shimer's communication on this subject he calls attention to the fact that absorbents of hydrochloric acid are unnecessary if the acid is entirely washed out in the original filtration, an operation which would seem to be not at all difficult. I have long held the same view as Mr. Shimer, and yet I am compelled to admit that better agreeing results can be regularly and consecutively obtained if all possible precautions are taken at the time of the combustion against the entrance of chlorine or hydrochloric acid into the potash bulbs.

I am indebted to Dr. C. B. Dudley for the suggestion of the use of ferrous sulphate, and I think it a valuable addition to the process. The solution is made by adding concentrated sulphuric acid to a saturated solution of ferrous sulphate until the latter begins to crystallize out.

A series of determinations made in all respects like those given above, except that this small bulb of ferrous sulphate was not inserted, gave the following results:

Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
1.073	1.080	1.051	1.051	1.037	1.040	1.049	1.069

There are three abnormally high results in this series, which do not appear in the series in which the ferrous sulphate was used.

In the course of this study I have made several hundred blank combustions to determine what errors might be introduced in the apparatus and manipulation. On some days there would be a constant gain in weight in the potash bulbs, on other days a constant loss, and again, at other times, the bulbs would not change in weight, in spite of the fact that the conditions of the experiments as to time, quantities of oxygen and air passed, and arrangement of apparatus were the same. On moist days, as every chemist of experience has observed, it is almost impossible to get a satisfactory weight of the potash bulbs, and it is also well known that differences of temperature between laboratory and weighing room must always be taken into consideration. The very least error, plus or minus, which one must count upon in the final weight of carbonic acid is, I think, 0.0003 gram. This can not be thought large when one considers the great weight of the bulbs, the large amount of oxygen and air (one or two liters) passing through them, the large surface of glass exposed, and the fluctuating conditions of humidity and temperature in the laboratory and weighing room. And yet this three-tenths of a milligram amounts to 0.003 per cent. of the carbon when three grams of steel are used for the analysis. It seems to me that a variation plus or minus of 0.005 per cent. on carbon in steel is the very least that should be allowed in view of the many sources of error inherent in the combustion process as now generally used. And I am inclined to think that it would be very seldom that the variation on ten consecutive determinations would fall within this limit, even with chemists of experience in this line of work.

I have satisfied myself by many experiments that a small extension tube filled with very small fragments of calcium chloride attached to the potash bulbs is entirely adequate to collect any moisture that may be carried over from the potash solution, and that the latter does not allow any carbonic acid to pass through. The use of solid caustic potash in the extension tube is therefore unnecessary. Neither have I found any advantage in the addition of a drop or two of water in the bulb of the calcium chloride tube to moisten the air after it has passed the anhydrous copper sulphate.

In addition to the work above given I have made a large number of determinations of the carbon in the experimental standard under different conditions. Some of the results thus obtained it may be worth while to record. A determination made in the same series as those first given, and in precisely the same manner, except that copper-ammonium chloride was used instead of the potassium salt, gave the per cent. of carbon as 1.053. This would indicate that the ammonium salt can be safely used when free from carbon.[1] That cupric chloride acidified with hydrochloric acid can be used for the solution of the iron has already been shown by other members of the committee. I have made several experiments in this direction with the view of using this method exclusively, and thus avoid the necessity of using a crystallized double salt of copper. Cupric chloride acidified with twenty per cent. of strong hydrochloric acid dissolves iron with reasonable promptness and gives good results. The addition of forty per cent. of hydrochloric acid is apt to cause loss of carbon. But hydrochloric acid is not as good a solvent of cuprous chloride as the alkaline chlorides.

It seemed obvious that the addition of potassium or sodium chloride to the acidified copper solution would give as good results as a solution of copper-potassium chloride. Several determinations made in this way gave fairly good results. Copper oxide, containing some metallic copper, was dissolved in hydrochloric acid, chlorine gas was passed into the solution to oxidize the cuprous chloride, and the solution was then evaporated to small bulk to expel the free chlorine; it contained some free acid. The amount of copper in this solution was determined by the battery in order that the proper amount should be used in dissolving the steel. Three grams of steel were treated with an amount of this solu-

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[1] This copper-ammonium chloride was obtained from Messrs. Baker and Adamson, Easton, Pa.

tion which contained twelve grams of copper, fifteen cc. of hydrochloric acid (sp. gr. 1.2) and sixty cc. of a solution of common salt containing 320 grams to the liter. The steel dissolved readily. The filtration of the carbon and the combustion were made precisely as above described with all the precaution for the interception of chlorine and hydrochloric acid. The results obtained for the carbon in three experiments were:

Per cent.	Per cent.	Per cent.	Average per cent.
1.055	1.043	1.059	1.052

A similar experiment, using potassium chloride instead of sodium chloride, gave 1.051 per cent. carbon.

I have also made a large number of experiments with silver sulphate as a solvent for the steel, but I did not obtain any satisfactory results in this way.

I take pleasure in acknowledging my great indebtedness to Mr. Wm. S. Davenport, instructor in analytical chemistry in the Massachusetts Institute of Technology, for the analytical work of this investigation, and for many valuable suggestions regarding the processes employed.

THOMAS M. DROWN.

The original four standards have now been analyzed in three countries with the following result.

The English committee give as the mean of their determinations:

ENGLISH COMMITTEE.

	Standard No. 1.	Standard No. 2.	Standard No. 3.	Standard No. 4.
Carbon	1.414	0.816	0.476	0.151
Silicon	0.263	0.191	0.141	0.008
Sulphur	0.006	0.007	0.008	0.039
Phosphorus	0.018	0.014	0.021	0.078
Manganese	0.259	0.141	0.145	0.130

The Swedish committee through Professor Åkerman give their means as:

SWEDISH COMMITTEE.

	Standard No. 1.	Standard No. 2.	Standard No. 3.	Standard No. 4.
Carbon	1.45	0.84	0.50	0.17
Silicon	0.257	0.185	0.15	0.015
Sulphur	0.008	0.004	0.006	0.048
Phosphorus	0.022	0.015	0.021	0.102
Manganese	0.282	0.145	0.17	0.13

The American members have not held a joint meeting this year and there are one or two analysts who have not completed their work, so that the averages given below may suffer some small modification in the future.

AMERICAN COMMITTEE.

	Standard No. 1.	Standard No. 2.	Standard No. 3.	Standard No. 4.
Carbon	1.44	0.807	0.452	0.16
Silicon	0.270	0.202	0.152	0.015
Sulphur	0.004	0.004	0.004	0.038
Phosphorus	0.016	0.010	0.015	0.088
Manganese	0.254	0.124	0.140	0.098

The German committee has devoted much attention to the study of methods of analysis in connection with the international standards, but has not yet announced its results. The French committee has made no report.

STANDARDIZING THE TORSION VISCOSIMETER.

BY O. S. DOOLITTLE, CHEMIST, P. & R. R. CO.

Received October 24, 1893.

IN the article published in the June number of *Drugs, Oils, and Paints*,[1] on the Torsion Viscosimeter, the method recommended for expressing the viscosity was in the number of degrees of retardation between the first and second complete arcs. Further experience has shown that although this is the best method for any one instrument, yet when it comes to comparing different instruments it is too delicate. It has been found that two instruments cannot be made to show the same number of degrees of retardation throughout a wide range of viscosity without requiring an amount of care and exactness in the manufacture which greatly adds to their cost.

In order to overcome this difficulty each instrument is now standardized against pure cane sugar solutions, after the manner proposed by Mr. S. M. Babcock (*J. Anal. Chem.*, **1,** 151,) in a similar case, the viscosity being expressed in the number of grams of sugar contained in 100 cc. of the syrup at 60° F., which will give the retardation designated at 80° F. These readings are obtained by taking a number of solutions contain-

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[1] See this JOURNAL, March, 1893.