tube is immersed in water, during the passage of a gas containing hydrogen and oxygen, and by so doing cause a partial combustion of any hydrocarbons that may be present. It is important, therefore, that the palladium should be distributed as uniformly as possible throughout the asbestos. As a result of the ignition, after impregnation with palladium chloride solution, the asbestos becomes somewhat more rigid and the bundle of fibers may be readily pushed, by help of a copper wire, far into the narrow glass tube.

[CONTRIBUTION FROM THE SHEFFIELD LABORATORY OF YALE UNIVERSITY.]

ON THE SEPARATION OF TUNGSTIC AND SILICIC ACIDS.

BY H. L. WELLS AND F. J. METZGER.

Received April 15, 1901.

IN a recent number of a German periodical[1] appears an article by Otto Herting, of Philadelphia, in which the assertion is made that the method given in the text-books for expelling silica from tungstic acid by means of hydrofluoric acid is incorrect. This statement is made on the ground of alleged numerous quantitative experiments with mixtures of pure tungstic acid and pure ignited silicic acid, but no details in regard to the results are given. Herting believes that upon ignition, silicic and tungstic acids form a silicotungstic acid which is volatile when treated with hydrofluoric acid, and finally says that he should be pleased if, by means of his article, he should bring about the more careful study of the " action of hydrofluoric acid upon tungstic acid in the presence of silicic acid."

Since Herting's statement throws doubt upon a method that is generally used, we have undertaken an examination of the matter. For this purpose, we dissolved some of Kahlbaum's tungstic acid in ammonia, precipitated with nitric acid, washed with water by decantation, digested repeatedly with sulphuric acid of sp. gr. 1.378 to separate any molybdic acid that might possibly be present,[2] washed the residue and ignited it. The tungstic acid thus prepared was used for the experiments that follow.

A weighed quantity of tungstic acid in a platinum crucible

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[1] *Ztschr. angew Chem.*, 1901, 165.

[2] See Ruegenberger and Smith : This Journal, **22**, 772.

was mixed with about an equal quantity of pure silica. The mixture was covered with dilute sulphuric acid, a liberal amount of pure hydrofluoric acid added, the liquid carefully evaporated, and the residue ignited over a Bunsen burner. Then another portion of silica was added and the operation repeated. The results are shown in the following table:

	Taken.		Tungsten trioxide found after	
	Tungsten trioxide. Gram.	Silica. Gram.	First operation. Gram.	Second operation. Gram.
I.	0.1928	0.2	0.1927	0.1928
II.	0.2097	0.2	0.2097	0.2096
III.	0.2100	0.2	0.2099	0.2100
IV.	0.1999	0.2	0.2000	0.1998

The greatest error found in these experiments is 0.0001 gram and they show that the process is perfectly exact under these conditions.

Other experiments showed that long ignition of the mixed tungstic and silicic acids over the Bunsen burner before expelling the silicic acid had absolutely no effect upon the results. It was thought that in the absence of sulphuric acid a loss might occur by the treatment of tungstic acid with hydrofluoric acid, and the following experiments were made to test this point, no sulphuric acid being used:

Taken.		
Tungsten trioxide. Gram.	Silica. Gram.	Tungsten trioxide found. Gram.
0.1983	0.2	0.1983
0.2102	0.2	0.2100
0.2106	0.2	0.2105
0.1996	0.2	0.1994

In these experiments, the greatest error, 0.0002 gram, is well within reasonable limits; hence, it is evident that the absence of sulphuric acid has no effect. It is to be noticed that in these cases also, the tungstic acid was ignited by the Bunsen flame only.

Attention should be called to the fact that tungstic acid must not be ignited by means of the blast-lamp, since at the temperature thus produced, it volatilizes to a considerable extent. The books of reference do not give proper warning in regard to this matter. The following table gives the results of a series of experiments made by heating some of the substance (which showed no loss of weight over the Bunsen burner) over the blast-lamp in a platinum crucible:

	Weight of tungsten trioxide. Gram.	Loss. Gram.
Taken	0.3007	......
Ignited for two minutes	0.2978	0.0029
“ “ “ “ again	0.2962	0.0016
“ “ “ “ “	0.2946	0.0016
“ “ “ “ “	0.2932	0.0014
“ “ “ “ “	0.2924	0.0008
“ “ “ “ “	0.2916	0.0008
“ “ “ “ “	0.2906	0.0010
“ “ five “ “	0.2872	0.0034
Total loss		0.0135

All the ignitions except the last were made with a lamp provided with a water-blast, which gave a flame of only moderate power. The last ignition was made with a lamp connected with a foot bellows, which gave a considerably higher temperature. It is noticeable that the losses show a tendency to diminish after the first ignition, but this is probably due to a change in the physical condition of the oxide rather than to the removal of some more volatile substance. It is hardly possible that our carefully purified tungstic acid, which showed no loss when heated with a good Bunsen burner, could contain an amount of molybdic acid or other volatile substance sufficient to give the results that have been obtained. The loss shown in the table above amounts to nearly 5 per cent., while inan other experiment 0.1955 gram of tungstic acid lost over 7 per cent. after heating with the blast-lamp for twenty minutes. It should be stated that the platinum crucible in which these ignitions were made showed no loss in weight after it had been cleaned.

We have shown that Herting's criticism of the usual method for separating silicic and tungstic acids is without foundation, and it appears probable that his difficulties were due to igniting tungstic acid at a too elevated temperature.

SHEFFIELD SCIENTIFIC SCHOOL,
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POTASSIUM PERSELENATE[1]—PRELIMINARY NOTE.

BY L. M. DENNIS AND O. W. BROWN.

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POTASSIUM perselenate was prepared by the electrolysis of a saturated solution of potassium selenate containing a little free selenic acid. This solution was placed in a 150 cc.

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[1] The experimental work herein described was performed by Mr. Brown in the spring of 1898. It has been impossible to resume the work until recently, but it is hoped that the investigation may soon be carried to completion.