tank by slit rubber stoppers, which fit in tsockets in he sheet-copper, partially covering the condenser, and at the bottom are bound by rubber tubing, beneath and above, to the brass tubes soldered to the floor of the condenser. The provision for independent worms relieves the necessity for putting the whole system out of service for repairs. The condenser is of $\frac{1}{8}$-inch copper, 24 ounces per square foot, partially covered by the sheet-copper, as mentioned, which is stiffened by braces soldered to the overflow pipe, also extending beyond its walls, acting as a support for the flasks, which are held in position by the bolts *d*.

The water is supplied by pipes *a* and *a*, and removed by the overflow pipe *e*, insuring satisfactory circulation.

The apparatus is supported on a circular shelf 36 inches in diameter (Fig. 3), which rests upon an octagonal table 52 inches wide, having two funnels on either side connected with the overflow pipe for convenience when washing the Nessler tubes. These are held in position by sockets beneath each worm, and, when not in service, are stored on shelves beneath the table.

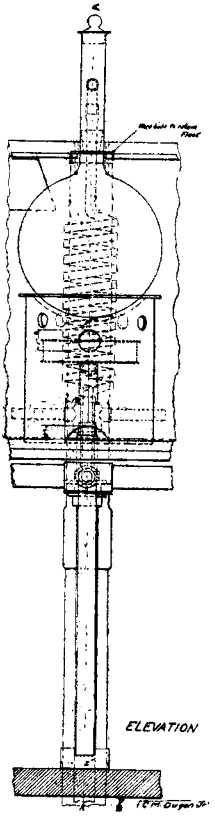

Fig. 3.

CITY HALL, PHILADELPHIA.

NOTES ON THE DETERMINATION OF NITROGEN AS NITRITES, IN WATERS.[1]

BY ROBERT SPURR WESTON.

Received January 12, 1905.

THE determination of small amounts of nitrogen as nitrite is usually made by one of three methods, namely, the iodometric method,[2] the metaphenylene-diamine-method,[3] or the naphthylamine method.[4]

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[1] Read at the Philadelphia Meeting of the American Chemical Society.

[2] *Pharm. Trans.*, 1881, 286.

[3] Sutton's "Volumetric Analysis," 8th Ed., p. 485.

[4] Warrington: *J. Chem. Soc.*, 1881, 231.

These methods are all colorimetric, the first developing the iodo-starch blue, the second "Bismark-brown," and the third the red color of the azo dye (azo-α-amino-naphthalene-parazo-benzene-sulphonic acid).

The first (Iodide) and second (Griess) of these methods are rarely used in America, while the third (Griess-Warrington), more or less modified, is employed quite generally.

The modifications of the Griess-Warrington method concern the strength, amount and kind of acid used; otherwise the analytical processes are similar. Either hydrochloric or acetic acid is used to produce the necessary acidity.

The original Griess-Warrington method was carried out with hydrochloric acid. Ilosvay[1] claimed, however, that acetic acid should be used, as it induces a more rapid development of color and more uniform gradation of tint. He also claimed that the reagents keep better when made with acetic acid.

In 1894 J. W. Ellms,[2] studied the Griess-Warrington method and arrived at the conclusion that while the presence of too much or too strong hydrochloric acid prevents the reaction of the naphthylmine-hydrochloride with the phenyl compound (para-diazo-benzene-sulphonic acid) to form the azo dye, as much as 2 cc. of 1 :4 hydrochloric acid in 100 cc. of the sample could be used and at the same time the color would develop rapidly and the tint would be of satisfactory depth. His conclusions, after comparison with Ilosvay's method were that the latter method resulted in a rather slower development of the color and for small quantities of nitrites had no special advantage over the original Griess-Warrington method, when the proper quantity of hydrochloric acid was used.

At the Lawrence Experiment Station[3] the use of acid, with the exception of a small amount used in the preparation of the reagents, is omitted.

The writer, holding views entirely in accord with those of Ellms, determined to make an attempt to harmonize these entirely opposite opinions.

To do this, it was necessary to compare the tints produced with some permanent standard. Such a standard was made by

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[1] *Bull. Soc. Chim.* (3), **2**, 347 (1889).

[2] This study was made in the Boston Laboratory of the Massachusetts State Board of Health, but the results were never published.

[3] Report Mass. Board of Health, 1890. "Purification of Water and Sewage."

diluting Ziehl's phenol-fuchsine solution to such a degree that it exceeded the maximum color produced by 0.1 mg. of nitrogen as nitrite in a liter. This standard did not fade appreciably during the course of the experiment, and matched the diazo color almost exactly in all dilutions.

REAGENTS.

The solutions used were as follows:

GRIESS-WARRINGTON.

		Reaction in terms of normal acid.
A. Sulphanilic acid	8 grams	
Hydrochloric acid	10 cc.	
Water to make to	1000 cc.	+ 1.6 N.
B. α-Naphthylamine	8 grams	
Hydrochloric acid (conc.)	8 cc.	
Water to make to	1000 cc.	+ 0.9 N.
C. Hydrochloric acid	——	+ 5 N.

ILOSVAY.

D. Sulphanilic acid	3.3 cc.	+ 4.8 N.
Acetic acid (sp. gr. 1.044)	1000 cc.	
E. α-Naphthylamine	0.5 g.	+ 3.9 N.
Acetic acid	1000 cc.	
F. Acetic acid	——	+ 5 N.

The standard nitrite solution contained 0.0001 gram of nitrogen per liter.

EXPERIMENTS.

Using the above reagents, a number of experiments were made to determine the speed of reaction, of which those given below are typical. The procedure was that recommended by the Committee of the American Public Health Association,[1] as follows:

Measure 100 cc. of the decolorized sample (decolorized by adding aluminum hydroxide free from nitrite), or a smaller portion diluted to 100 cc. into a Nessler comparison tube. At the same time make a set of standards by diluting various volumes of standard nitrite solution in comparison tubes to 100 cc. with nitrite-free water. Add the reagents; mix; allow to stand until the color develops; compare the samples with the standards. Make a blank determination in all cases to correct for the presence of nitrite in the air, the water and the reagents.

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[1] Jour. Am. Public Health Asso., 1904.

EXPERIMENT I.—COMPARISON OF GRIESS-WARRINGTON + HYDROCHLORIC ACID, WITH ILOSVAY METHOD.

a. Speed of reaction with 2 cc. of each A and B and 1 cc. of C to each 100 cc. of sample.

b. Ditto with 8 cc. of each D and E.

Readings of color developed are in terms of permanent standard.

The figures represent the number of cubic centimeters of standard fuchsine solution in 100 cubic centimeters of the sample compared.

Time	Cc. of standard nitrite solution.					
	a. Griess method.			*b.* Ilosvay's method.		
minutes.	3.	10.	20.	3.	10.	20.
3	0	0	0	0	0	4
5	0	0	0	0	1	5
10	0	0	0	0	2	8
15	0	0	0	2	4	10
30	3	4	6	4	8	18
60	4	8	11	6	13	26

EXPERIMENT II.—COMPARISON OF SPEEDS OF REACTION BY GRIESS METHOD, WITH AND WITHOUT THE ADDITION OF 1 CC. HYDROCHLORIC ACID, WITH ILOSVAY'S METHOD.

Readings of color developed are in terms of permanent standard.

Temperature = 22° C.

a. 2 cc. A : 2 cc. B — No hydrochloric acid.

Time	Cc. of standard nitrite solution.					
minutes.	1.	5.	10.	25.	50.	100.
3	4	10	20	35	75	100
5	4	10	23	37	--	140
10	5	10	24	38	75	150
15	5	10	24	38	80	150
30	5	11	24	38	80	150
60	5	11	24	38	80	150
150	5	12	24	38	80	160

b. 2 cc. A : 2 cc. B + 1 cc. hydrochloric acid C.[1]

Time	Cc. of standard nitrite solution.					
minutes.	1.	5.	10.	25.	50.	100.
3	1	4	6	10	18	30
5	3	6	8	19	25	45
10	4	7	10	25	42	60
15	5	8	11	28	50	75
30	5	8	14	37	70	130
60	5	9	16	39	75	130
120	6	9	20	40	75	130

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[1] As described by Richards and Woodman, "Air, Water and Food."

c. 2 cc. D and 2 cc. E (Ilosvay).[1]

Time minutes.	Cc. of standard nitrite solution.					
	1.	5.	10.	25.	50.	100.
1	4	7	10	25	30	15
5	6	11	22	35	80	120
10	6	11	22	35	78	130
15	6	12	22	36	80	150
30	6	12	22	38	78	160
60	6	12	22	38	78	160

Experiment III.—Comparison of the Relative Effects of Organic and Inorganic Acids on the Speed of Reaction with :

A. Griess-Warrington method with no acid.
B. Ditto with 5 cc. hydrochloric acid, 5 N.
C. Ilosvay's method.
D. Ditto with 5 cc. acetic acid, 5 N.

Readings are in terms of permanent standard.

Time minutes.	A.			B.			C.			D.		
	Cubic centimeters of standard nitrate solution.											
	1.	25.	50.	1.	25.	50.	1.	25.	50.	1.	25.	50.
3	3	14	26	0	4	6	5	28	34	5	28	29
5	4	25	40	0	6	8	5	32	55	5	33	50
10	5	28	65	1	8	14	5	34	80	5	35	80
15	5	30	70	1	10	21	5	35	80	5	35	80
30	5	34	80	3	15	32	5	35	80	5	35	80
24 hrs	6	37	83	6	37	78	6	37	85	6	38	83

Experiment IV.—Comparison of the Speeds of Reaction with Different Volumes of Ilosvay's Reagents, D and E, with :

A. 1 cc. of each reagent to 100 cc. of sample.
B. 2 cc. of each reagent to 100 cc. of sample.
C. 8 cc. of each reagent to 100 cc. of sample.

Readings are in terms of permanent standard.

Time minutes.	A.				B.				C.			
	1.	10.	25.	50.	1.	10.	25.	50.	1.	10.	25.	50.
3	0	0	1	2	0	2	7	10	6	13	30	60
5	1	2	3	5	2	5	13	23	7	15	33	75
10	2	4	6	12	4	9	26	35	7	16	35	80
15	3	5	11	23	6	13	30	50	7	20	35	80
30	7	12	27	36	8	20	34	70	8	22	36	80
16 hrs	13	31	47	84	15	31	47	87	–	22	48	90

All of the above experiments were made with a standard solution of sodium nitrite. To ascertain if similar data would be obtained with calcium salts, the following experiment, using a standard solution of calcium nitrite (1 cc. = 0.0000001 gram of nitrogen), was made.

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[1] As described by Leffmann, "Examination of Water."

EXPERIMENT V.—COMPARISON OF THE GRIESS-WARRINGTON (WITHOUT ADDITIONAL ACID) AND ILOSVAY METHODS, USING STANDARD CALCIUM NITRITE SOLUTION.

Readings are in terms of permanent standard.

Time minutes.	Griess method.				Ilosvay method.			
	Cubic centimeters of standard calcium nitrate.							
	1.	5.	10.	20.	1.	5.	10.	20.
3	2	7	12	22	4	11	16	30
5	3	11	13	24	4	12	17	32
10	3	12	16	30	4	12	17	34
25	3	12	16	33	4	12	17	34

Blank tubes with distilled water and the reagents showed varying amounts of nitrite, equivalent to from 0 to 6 cc. of the standard solution, dependent, to a large degree upon the period of exposure to the air of the laboratory.

CONCLUSIONS.

The following conclusions may be drawn from these experiments:

(1) An excess of hydrochloric acid interferes with the delicacy of the reaction.

(2) An excess of acetic acid does not interfere with the delicacy of the reaction.

(3) The speed of reaction varies with the amount of hydrochloric acid added.

(4) These phenomena hold true with both calcium and sodium nitrite, probably with all nitrites of the alkalies and alkaline earths.

(5) The speed of reaction is affected in some degree by the excess of reagents.

(6) The method as modified by Ilosvay is more rapid than the original Griess-Warrington method.

(7) There are no apparent differences in the quality of the color (the tint) produced by the various modifications of the method.

In view of the above it may be asked why many workers have believed that the Ilosvay modification was inadvisable,—in fact, the writer apparently duplicated many of the above experiments with contrary results. The reason for this discrepancy is that mixing was practiced in one case, not in the other. If the samples are mixed, the heavy acetic acid (Ilosvay) solutions are diffused through the sample; if not, they sink to the bottom and the reaction is delayed. This difficulty is not so noticeable with the aqueous solutions, often used without mixing, the hydrochloric acid being added subsequently.

In water analysis the Ilosvay modification would not be preferred above the Griess-Warrington without addition of extra acid, were it not for the variation in the alkalinities of natural waters, making the use of small additions of hydrochloric acid inaccurate. Enough acid should be added to nullify the error due to the varying alkalinity; otherwise, waters of high alkalinity would react quicker than those of low, due to differences in acidity at the time of the development of the color. If enough hydrochloric acid were added to accomplish this end, the reaction would be retarded. Acetic acid, however, may be added with impunity.

The addition to a decolorized sample of water of 5 cc. of each of Ilosvay's reagents, followed by mixing the samples, produces a color which, after ten minutes' standing, may be compared with standard solutions similarly treated.

The only improvement in the method which the writer can suggest is that the reagents be made more concentrated than Ilosvay suggested, because the use of 5 cc. of each reagent increases the volume of the samples to an inconvenient degree. The writer would recommend, therefore, that the sulphanilic acid solution be made by dissolving 8 grams of the salt in a liter of dilute acetic acid, and that the a-naphthylamine solution also contain 8 grams of the salt in 1 liter. To effect the solution of the naphthylamine dissolve it as completely as possible in the acetic acid, warming, if necessary. After a few hours filter the solution through absorbent cotton. Two cc. of each of these reagents will be found to be sufficient.

The writer is indebted to his assistant, Mr. R. K. Hale, for making many of the readings.

'4 BEACON STREET, BOSTON.

A NEW FILTER.

BY PORTER W. SHIMER.

Received December 24, 1904.

THERE are a number of sources of inaccuracy in the methods of filtration commonly used in quantitative chemical analysis that we seem to have become accustomed to, for, if we think of them at all, we are inclined to accept them as unavoidable. For example, we are rarely able to get filter-paper that can be relied on, under all circumstances, to retain every trace of finely divided