

blue color with starch solution. The figures are grams of thio-sulphate, equivalent to 1 cc. of iodine solution.

| Weight of sample. | Volume of iodine solution. cc. | Standard of solution. |
|-------------------|-----------------------------------|-----------------------|
| 0.8833 | 27.45 | 0.03228 |
| 0.7288 | 22.57 | 0.03227 |
| 0.9319 | 28.90 | 0.03224 |
| 0.6807 | 21.07 | 0.03229 |

The standard of the same iodine solution, as determined by the direct method with a weighed quantity of iodine, was 0.03228.

Three days later the following determinations were made with the same substance in order to see if there was any deterioration of the anhydrous salt within that time, due, perhaps, to absorption of moisture or other cause.

| Weight of sample. | Volume of iodine solution. cc. | Standard of solution. |
|-------------------|-----------------------------------|-----------------------|
| 0.9404 | 29.30 | 0.03210 |
| 0.8338 | 25.92 | 0.03217 |
| 0.6100 | 18.92 | 0.03224 |
| 0.8518 | 26.47 | 0.03218 |

Two determinations by the direct method, made at the same time, gave 0.03217 and 0.03221.

Except for the first determination, the agreement is all that could be desired. There is more than likely a small experimental error in the first determination.

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Note on the Effect of Old Filter-paper on Iodate-free Potassium Iodide.—In the course of some experiments with potassium iodide it developed that a dilute solution (1 in 10) of iodate-free salt, after filtering through the ordinary filter-paper of the laboratory and acidification with a few drops of dilute sulphuric acid after the manner of the United States Pharmacopœia test for iodate,¹ oftentimes showed a distinct brownish coloration after

¹ The Pharmacopœia of 1890 recommends the use of starch paste to render the liberation of iodine evident by the formation of the characteristic starch-iodine blue. We find that the yellow coloration of the free iodine is as delicate and as easily seen in a colorless clear solution as the starch reaction. Hence we omitted the starch paste. We worked with 1 gram of potassium iodide in 10 cc. of water instead of 1 gram in 20 cc. as directed in the Pharmacopœia. In all these tests it is necessary to run a blank on the iodide solution along with the test, since, as is well known, the acidified potassium iodide solution becomes brownish from the oxidation of the hydriodic acid by the oxygen of the air. If the time of standing be short, say three minutes, the blank solutions will be practically water-white, whereas the solutions treated with old filter-paper will be distinctly yellowish. The coloration corresponds, of course, to a very small weight of iodine: For 20 cc. of the solution, it required only two or three drops of N/100 thiosulphate solution to cause a complete bleaching.

several minutes standing, due, of course, to the liberation of traces of iodine. With some filters and with washed glass wool the effect was absent and the acidified, clear solution remained water-white for a few minutes. The iodate-free potassium iodide was prepared by acting upon a slightly alkaline, dilute solution of the salt in nitrite-free water with magnesium powder and shaking. This solution, after being filtered to remove the magnesium, gave the above slight test for iodate, indicating the paper as the cause of the trouble.

Samples of paper from other rooms in the same laboratory were then tested in the above manner, and also with indigo solution, with the result that out of sixteen samples of various papers all but two colored the potassium iodide solution and these two did not bleach the indigo. The indigo reaction was tried on four of the samples which colored the iodide and each time the blue solution bleached slightly, but distinctly. The papers tested included the ordinary sheet filter-paper, factory filters (gray and white), Schleicher & Schüll's quantitative (washed), and several grades of Dreverhoff's best. All these papers had been kept in different laboratory rooms and in different drawers for times varying from several months to a year.

It is quite well-known that filter-paper absorbs or occludes gases somewhat in the manner of charcoal when exposed, and recently H. R. Procter¹ has pointed out that filters, originally free from acidity or alkalinity, easily absorb acids or ammonia from the laboratory air in sufficient quantity to disturb titrations for hardness in water analysis and, accordingly, he recommends thorough washing and rejection of part of the first filtrates. Mansier,² working with solutions, found that filter-paper, like bone-coal, has the power of fixing certain substances in the fibers so that washing will not remove them. For example, he found that a 15 cm. filter, previously thoroughly extracted with dilute hydrochloric acid followed by distilled water, lowered the strength of a normal solution of sodium hydroxide by about a twentieth of its original value. The paper was immersed in the solution, which, presumably, was then decanted and titrated.

L. W. Andrews³ has detected nitrites in filter-paper which has stood for some time exposed to laboratory air. With these facts

¹ *J. Soc. Chem. Ind.*, January 15, 1904.

² Mansier : *J. Pharm. Chim.* [6], 16, 6-116 (1902) ; abstract in *Centrbl.*, 2, 768 (1902).

³ Private communication: Dr. Andrews further states that some of the papers containing no nitrites still liberated iodine from potassium iodide. The cause of this is not known to us.

in mind we tested old papers again for nitrous acid by the characteristic Griess reaction along with the various grades of *fresh papers*, obtained directly from Eimer & Amend. The procedure was as follows: Six sheets (7 cm.) of the fresh paper from different parts of the pack were torn to pieces and treated for fifteen minutes with 60 cc. of nitrite-free water, and this solution was poured into Nessler tubes, about 2 cc. of the sulphanilic acid solution added, followed by about 2 cc. of the α -naphthylamine solution. The amounts of nitrogen present as nitrites were estimated colorimetrically by comparing the colors obtained from the filter-paper solutions with a scale of colors produced by adding definite amounts of a determinate sodium nitrite solution, as is usual in water analysis.¹ Quantities of peroxide of hydrogen and of sodium hypobromite, greatly in excess of the equivalent of the maximum amount of nitrite, measured in the filter-paper, gave no characteristic coloration when treated with the Griess reagents. Measured in this way, the six sheets (7 cm.), representing 3 to 4 grams of the various fresh papers, including Eimer & Amend's best white, The Baker & Adamson Chemical Co.'s hydrofluoric and hydrochloric washed, Munktell's Swedish paper and Schleicher & Schüll's, gave only *very slight tests* for nitrites, corresponding roughly to from 0.0001 mg. to 0.0005 mg. of nitrogen, according to the different makes used. On the other hand, the old paper which had been kept in the laboratory for some time invariably gave a *strong reaction*. Thus two sheets (7 cm.) of Eimer & Amend's best white from a drawer in the laboratory showed over 0.0020 mg. of nitrogen, corresponding to more than 0.0060 mg. of nitrogen for six papers, and two sheets of Schleicher & Schüll's (7 cm., No. 589) from the same drawer showed over 0.0008 mg., corresponding to more than 0.0024 mg. for six papers. We found, furthermore, that 2 grams of a large Prat-Dumas filter, which had been kept in an office room off from the laboratory, showed 0.0001 mg. of nitrogen, but the same amount of a similar filter, which had remained in the laboratory for some time, showed 0.0020 mg. nitrogen. As would be expected, the fresh papers showed no coloration with potassium iodide solutions and of the Prat-Dumas paper above mentioned, the sheets stored in the laboratory gave a strong reaction, whereas that stored in the office desk showed none.

¹ Solutions according to Richards and Woodman: "Air, Water, and Food." α -naphthylamine 8 grams, concentrated hydrochloric acid 8 cc., water 992 cc. Sulphanilic acid 8 grams in 1 liter of water.

The above experiments confirm the results of Andrews and show that just as the absorption of acid vapors by filter-paper exposed to laboratory air may render the paper unfit for delicate titrations, as, for example, the determination of the hardness of water, so also the absorption of nitrous vapors may take place to such an extent as to oxidize solutions of iodate-free potassium iodide when filtered through such paper. With the paper used in these experiments the oxidation, although representing very small amounts of free iodine, was readily detectable by the appearance of a yellowish coloration when such a solution is acidified.

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