

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.

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A NEW FILTER.

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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

precipitates, such as barium sulphate. With the common filter and funnel this precipitate is liable either to pass, in appreciable amount, through the body of the paper or to mount over its top and pass down the channel made by the fold.

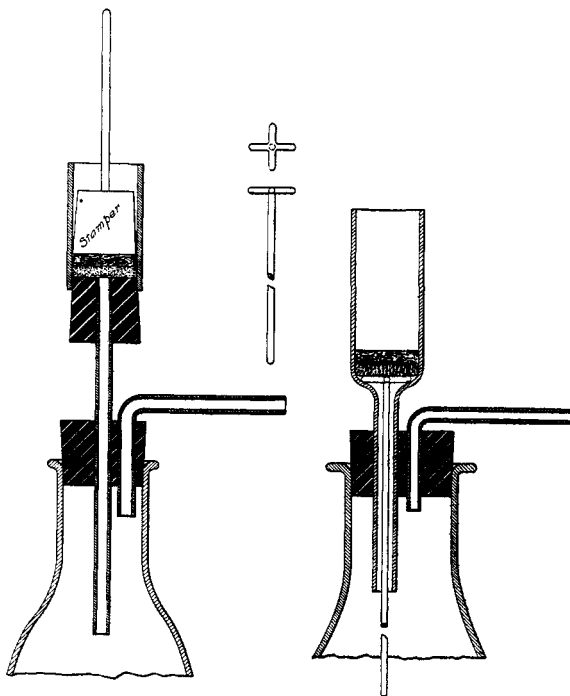
The use of double filters lessens this trouble, but does not altogether remove it. The double filter, moreover, is more difficult to wash and, when iron must be removed from the paper by the use of hydrochloric acid, as in the determination of sulphur in iron, the loss of barium sulphate by solution may become quite appreciable. The extra reducing action of double filters in the ignition of certain precipitates should also not be overlooked.

As another example, let us consider the matter of washing a precipitate on a 3- or 4-inch filter in a glass funnel. If the precipitate is small, it will take longer to wash the solution out of the paper than out of the precipitate, which is not altogether as it should be. As a concrete example let us take a small phosphomolybdate precipitate from a pig iron guaranteed not to go over 0.03 per cent. phosphorus. When this phosphorus is to be determined volumetrically, the washing out of every trace of the molybdate solution is a matter that requires skilful and conscientious manipulation, for, as Dr. Dudley has shown, an inexpert analyst may easily leave enough molybdate solution in the paper to cause an error of 0.005 per cent.

Again, in order that washing may be thorough, the paper must fit perfectly into the funnel, that is, it must be in contact with the sides of the funnel at every point, without bubble or wrinkle anywhere. For this purpose the funnel must have an angle of exactly 60° , and we all know how hard it is to get funnels that are quite satisfactory in this respect.

With a view to getting a filter free from these defects, I have devised one which has been in successful use in my laboratory for nearly a year. One form of the apparatus consists of a glass tube, cut off square at both ends, two inches long and one inch in internal diameter. The edges should be left sharp and not rounded in the flame. In the bottom of the tube is a rubber stopper, fitted with a glass tube for attachment to the suction flask. On the stopper, when inserted into the tube, is a disk of piano-felt, $\frac{3}{16}$ -inch thick, fitting closely into the tube. This piano-felt may be variously replaced, as will be mentioned further on. The filter-tube is now ready for the filter. Take unwashed

Swedish filter-paper in any convenient amount, crush it into a ball in the hand and place it in a large ceresine hydrofluoric acid bottle from which the upper part has been cut. Add hydrochloric acid (sp. gr. 1.12 to 1.18) and a little hydrofluoric acid and stir vigorously with a paraffin-coated wooden stirrer until the paper



has become a mass of fine, soft pulp. Let it stand for a few minutes and then add distilled water. In preparing a filter, pour some of this pulp into a beaker, dilute further with distilled water and pour enough on the felt, under suction, to make a filter of the desired thickness. Wash two or three times with water and the filter is ready for filtration of gelatinous precipitates as in silicon determination, and for the smaller precipitates of iron and alumina. For large precipitates of iron and alumina a large filter surface is desirable and the old method of filtration is preferred.

In forming the filter for barium sulphate and crystalline precipitates generally, after depositing the pulp to the depth of about $\frac{1}{4}$ inch, it must be well compacted by hard stamping. For

this purpose it is convenient to use a solid rubber stopper, the larger end of which is only a little smaller than the inner diameter of the tube. A hole is made in the smaller end, but not deep enough to pass quite through, for the insertion of a short piece of glass rod as a handle.

Barium sulphate may be filtered and washed rapidly, under suction, on this filter without fear of a trace getting into the filtrate. When filtration and washing are completed, turn off the suction and remove the filter-tube from the stopper. Take the stamper and push the felt and filter up until the filter projects beyond the tube, when it can be readily removed from the felt by the use of curved, brass forceps; or, if preferred, the upper end of the tube may be inserted into the weighed crucible and the felt and filter may be pushed at once into it, when the felt can be readily removed and the precipitate and filter ignited. Any precipitate adhering to the sides of the tube is taken along by the outgoing filter. Occasionally a tube is found which is not quite uniform in diameter and a little of the precipitate remains in places on the sides. Such a tube is, of course, thrown aside. When precipitates are not to be ignited immediately they may be conveniently preserved, each on a small glass square, ground on one side for convenience in labeling, or they may be laid aside on small pieces of filter-paper, properly labeled.

The weight of these filters varies, of course, with their thickness, but it is in all cases less than the weight of an ordinary filter. The ash is quite negligible, for it takes the ash from six filters to make 0.1 mg., which is only one-third of the ash of the best double-washed 11 cm. paper. Filtration is rapid, as are also the operations of charring and incineration, with, consequently, minimum risk of reducing action in the crucible.

One of the greatest advantages is in the washing of precipitates. By reason of the direct downward movement of the wash-water it is possible to wash thoroughly with much less water than in the usual method of filtration, thus reducing both loss by solubility of precipitates and the dangers of incomplete washing. In dissolving the yellow phosphomolybdate precipitate, this may be done either by dissolving under suction into a small Erlenmeyer flask, or, if greatest speed is not necessary, the filter may be removed and placed, with the precipitate upwards, in an ordinary funnel. By pressing down the edges of the filter into

contact with the sides of the funnel by means of a glass rod the solution in ammonia may be made and the filtrate allowed to run into a beaker.

A few results, comparing old and new filters, follow:

	Old.	New.
Silicon in pig iron.....	2.720	2.717
Sulphur in coal—Sample A.....	1.740	1.736
“ “ “ “ B.....	1.811	1.810

Duplicate sulphurs in pig iron, both new filters..... 0.066 0.066

It is essential to keep the filters and flasks which are used in sulphur determinations for this purpose only, otherwise there is risk of contaminating them with sulphuric acid or soluble sulphates. The filtering apparatus may be conveniently arranged in any desired number along a piece of gas-pipe, coated inside with an acid-proof coating, and attached to the suction. The filters are united with this pipe at short intervals and each tube leading to a filter is provided with a valve so that any number may be put on or off at pleasure. The receptacle for the filtrate may be a gas-bottle or an Erlenmeyer flask and the transference to a beaker is easily made.

For filtering the carbon from steel or pig iron this form of filter is very satisfactory. The tube is $\frac{7}{8}$ inch in diameter, a plug of glass wool is used in place of the felt and finely divided ignited asbestos is deposited upon it. A properly prepared filter need not be thicker than $\frac{1}{8}$ inch. After filtration and washing, the tube is disconnected and the filter is pushed out of the upper end of the tube. The carbon, with asbestos filter, is readily lifted off the glass wool by use of curved forceps and placed, for drying, in a properly labeled scorifier previously heated on the edge of a hot plate, not above 100° C. The scorifier is jarred smartly against the table so as to bring the wet filter in close contact with its absorbent surface. The drying, in an air-bath, or on the cooler part of the hot plate, is rapid. The removal of the carbon from the tube is complete, but the end of the tube must be cut off square and must be neither flared nor drawn in. The carbon, thus filtered, is in an exceptionally convenient form for transference to the crucible for combustion.

When, in ordinary analytical work, it is desired to use stronger acids or other solvents which might act upon the piano-felt or rubber stopper, a filter-tube 1 inch in internal diameter, drawn out

at the lower end, is used. Through the stem of the tube and projecting beyond it far enough to let the filter be pushed out of the tube, is a glass rod to the upper end of which is fastened a perforated disk, or filter-plate, either of glass, porcelain or platinum.

Another useful form of the apparatus consists of the filter-tube, with the glass rod, to the upper end of which are fused two short pieces of glass rod previously fused together at right angles. For the rapid filtration of such gelatinous precipitates as those from silicon in pig iron an extra open material on which to deposit the felt is desirable. For this purpose upholsterer's horse hair may be sewed to the upper end of the rod, preferably with horse hair; a horse-hair felt manufactured into insoles for shoes makes a very good base for the deposition of the filter. For precipitates which cause slow filtration, the felt is never stamped down. For filtering carbons the same arrangement is very useful, the horse hair being, however, replaced by a mass of glass wool, which need not be fastened to the rod.

Absorbent cotton may be used as a base on which to deposit the felt, but piano-felt is more open and is more resistant to the action of acids. In the determination of silicon in pig iron by the Drown method absorbent cotton may, in certain cases, be used in place of the paper felt.

In filtering barium sulphate it is necessary to compact the felt upon a firm support, as in the arrangement first described, in order to make the filter absolutely tight. The horse-hair base is too springy for this purpose.

Since, in the system of filtration described, a filter-pump is necessary, it may be of interest to mention that, in the writer's laboratory, where the hydrant pressure is not satisfactory for use in creating vacuum, a Gould air-pressure or vacuum-pump, with hand-power, is used. The pump is attached to two large cylinders, one for compressed air, the other for vacuum. For blast the apparatus is very much better than foot-blowers, for only occasional attention is required and the blast is steady and of any strength desired. For suction also, only occasional attention is necessary, and the degree of vacuum may be regulated at will.