ART. XXVIII.—*On the Washing of Precipitates*; by
R. BUNSEN.*

A PRECIPITATE is washed either by filtration or by decantation: in the former case the portion of liquid not mechanically retained is allowed to drain from the precipitate; in the latter it is separated by simply pouring it away, the foreign substances contained in the precipitate being then removed by the repeated addition of some washing-fluid, in each successive portion of which the precipitate is, as far as possible, uniformly suspended, this process being continued until the amount of impurity becomes so minute that its presence may be entirely disregarded.

Supposing v to represent the volume of the moist precipitate remaining at the bottom of the vessel after decantation, or upon the filtrate after filtration, V the volume of wash-water employed at each successive decantation, n the number of decantations, and $\frac{1}{a}$ the fraction expressing the proportion of the original amount of impurity still remaining in the precipitate after n decantations, then

$$\left(\frac{v}{v+V}\right)^n = \frac{1}{a} \quad \dots \dots \dots (1)$$

Calling W the total volume of wash-water resulting from n decantations, then

$$nV = W; \quad \dots \dots \dots (2)$$

therefore

$$\left(1 + \frac{W}{nv}\right)^n = a,$$

or

$$W = nv(\sqrt[n]{a} - 1). \quad \dots \dots \dots (3)$$

If we differentiate W with respect to n and make the differ-

* From Lond., Edin. and Dublin Phil. Mag., Jan. 1869. Translated from the Ann. der Chem. und Pharm., vol. cxlviii, p. 269.

ential quotient equal to 0, then the minimum value of W becomes, when $n = \infty$,

$$W = v \text{ nat. log. } a. \quad . \quad . \quad . \quad . \quad . \quad (4)$$

Precipitates obtained in the course of chemical analysis may in all cases be assumed to be sufficiently washed when the impurity retained by them amounts to no more than the $\frac{1}{100000}$ part. Making therefore $a = 100000$ and $v = 1$, it results from equation (4) that the least quantity of fluid required in order to remove the impurity contained in a precipitate to the $\frac{1}{100000}$ part, amounts to eleven and a half times the volume occupied by the precipitate itself in the liquid in which it exists. It is evident, therefore, that the amount of water actually necessary to wash a precipitate the more nearly approaches this minimum the oftener we decant, and the smaller the quantity of washing-water we employ at each decantation.

Since some of the principal sources of error in analytical work consist in the incomplete or in the too protracted washing of precipitates, it becomes important to know how to ascertain the progress of the washing throughout the several stages of the process. By employing the same volume of water at each successive addition, and estimating its relation to that of the precipitate remaining at the bottom of the vessel or upon the filter, we can find from the following Table, calculated by

I 0 0 0 0 0			5 0 0 0 0			2 0 0 0 0			1 0 0 0 0		
I.	II.	III.	I.	II.	III.	I.	II.	III.	I.	II.	III.
$\frac{V}{v}$.	n .	W .	$\frac{V}{v}$.	n .	W .	$\frac{V}{v}$.	n .	W .	$\frac{V}{v}$.	n .	W .
0.5	28.4	14.2	0.5	26.7	13.3	0.5	24.4	12.2	0.5	22.7	11.4
1	16.6	16.6	1	15.6	15.6	1	14.3	14.3	1	13.3	13.3
2	10.5	21.0	2	9.8	19.7	2	9.0	18.0	2	8.4	16.8
3	8.3	24.9	3	7.8	23.4	3	7.1	21.4	3	6.6	19.9
4	7.1	28.6	4	6.7	26.9	4	6.1	24.6	4	5.7	22.9
5	6.4	32.1	5	6.0	30.2	5	5.5	27.6	5	5.1	25.7
6	5.9	35.5	6	5.6	33.4	6	5.1	30.5	6	4.7	28.4
7	5.5	38.8	7	5.2	36.4	7	4.8	33.3	7	4.4	31.0
8	5.2	42.0	8	4.9	39.4	8	4.5	36.1	8	4.2	33.5
9	5.0	45.0	9	4.7	42.3	9	4.3	38.7	9	4.0	36.0
10	4.8	48.0	10	4.5	45.1	10	4.1	41.3	10	3.8	38.4
11	4.6	51.0	11	4.4	47.9	11	4.0	43.8	11	3.7	40.8
12	4.5	53.9	12	4.2	50.6	12	3.9	46.3	12	3.6	43.1
13	4.4	56.4	13	4.1	53.3	13	3.8	48.8	13	3.5	45.4
14	4.2	59.4	14	4.0	55.8	14	3.7	51.1	14	3.4	47.5
15	4.2	62.3	15	3.9	58.5	15	3.6	53.6	15	3.3	49.8
16	4.1	65.0	16	3.8	61.1	16	3.5	56.0	16	3.3	53.0
17	4.0	67.8	17	3.7	63.6	17	3.4	58.3	17	3.2	54.2
18	3.9	70.4	18	3.7	66.1	18	3.4	60.5	18	3.1	56.3
19	3.8	74.3	19	3.6	68.6	19	3.3	62.8	19	3.1	58.4

means of the formula above given, the number of times it is necessary to decant in order to diminish the amount of impurity in the precipitate to the $\frac{1}{100000}$, $\frac{1}{50000}$, $\frac{1}{20000}$ or $\frac{1}{10000}$ part.

Column I. shows the relation between the volume of the precipitate and that of the washing-water employed for each successive decantation, column II. the number of decantations required to diminish the amount of impurity to the necessary extent, and column III. the total volume of water obtained from the several decantations.

When the washing-process is performed in a beaker, the relation between the volume of the precipitate and that of the liquid may be easily determined by holding a strip of paper along the side of the vessel and marking upon it the respective heights of the precipitate and supernatant liquid ; then on folding the portion of paper lying between the two marks in such a manner that each fold corresponds to the height occupied by the precipitate, the number of folds will give the argument in column I. to find in column II. the number of decantations needed to wash to the required extent. If the washing be conducted as in the ordinary method of filtration, funnels possessing an angle of 60° must be invariably employed, and the capacities of the various-sized filters once for all determined by means of a burette. After the precipitate has been brought upon the filter and allowed to drain, it is mixed as thoroughly as possible with water from a graduated washing-flask. Call the amount of water thus necessary to fill the filter $\mathfrak{u}$, and the capacity of the empty filter $\mathfrak{V}$, then $\frac{\mathfrak{u}}{\mathfrak{V}-\mathfrak{u}} = \frac{V}{v}$ in column I ; that is, the argument needed to find in column II the number of times it is necessary to refill the filter in order to wash the precipitate to the desired extent.

I by far prefer using this Table to employing the method generally followed of ascertaining the completion of the washing-process by evaporating a quantity of the filtrate on platinum-foil, since in the latter case it is only possible to obtain an infallible proof when we have to deal with a precipitate possessing an extremely high degree of insolubility ; if the precipitate be soluble to any marked extent, the result is completely illusory.

In the process of filtration as hitherto conducted, the time required is so long and the quantity of wash-water needed so great that some simplification of this continually recurring operation is in the highest degree desirable. The following method, which depends, not upon the removal of the impurity by simple attenuation, but upon its displacement by forcing the wash-water through the precipitate, appears to me to combine all the requisite conditions and therefore to satisfy the need.

The rapidity with which a liquid filters, depends, *cæteris paribus*, upon the difference which exists between the pressure upon its upper and lower surfaces. Supposing the filter to consist of a solid substance, the pores of which suffer no alteration by pressure or by any other influence, then the volume of liquid filtered in the unit of time is nearly proportional to the difference in pressure: this is clearly shown by the following experiments, made with pure water and a filter consisting of a thin plate of artificial pumice-stone. The thin plate of pumice was hermetically fastened into a funnel consisting of a graduated cylindrical glass vessel, the lower end of which was connected with a large thick flask by means of a tightly fitting caoutchouc cork. The pressure in the flask was then reduced by rarefying the air by means of a method to be described upon another occasion; and for each difference of pressure p , measured by a mercury column, the number of seconds t was observed which a given quantity of water occupied in passing through the filter. The following are the results:—

p . meter.	I.	
	t . "	pt .
0·179	91·7	16·4
0·190	81·0	15·4
0·282	52·9	14·9
0·472	33·0	15·6

In the ordinary process of filtration, p on the average amounts to no more than 0·004 to 0·008 meter. The advantage gained, therefore, is easily perceived when we can succeed by some simple, practicable and easily attainable method in multiplying this difference in pressure one or two hundred times, or, say, to an entire atmosphere, without running any risk of breaking the filter. The solution of this problem is very easy: an ordinary glass funnel has only to be so arranged that the filter can be completely adjusted to its sides even to the very apex of the cone. For this purpose a glass funnel is chosen possessing an angle of 60° , or as nearly 60° as possible, the walls of which must be completely free from inequalities of every description; and into it is placed a second funnel made of exceedingly thin platinum-foil, and the sides of which possess exactly the same inclination as those of the glass funnel. An ordinary paper filter is then introduced into this compound funnel in the usual manner; when carefully moistened and so adjusted that no air-bubbles are visible between it and the glass, this filter, when filled with a liquid, will support the pressure of an extra atmosphere without ever breaking.

The platinum funnel is easily made from thin platinum-foil in the following manner:—In the carefully chosen glass funnel

is placed a *perfectly accurately fitting* filter made of writing-paper; this is kept in position by dropping a little melted sealing-wax between its upper edge and the glass; the paper is next saturated with oil and filled with liquid plaster of Paris, and before the mixture solidifies a small wooden handle is placed in the center. After an hour or so the plaster cone with the adhering paper filter can be withdrawn by means of the handle from the funnel, to which it accurately corresponds. The paper on the outside of the cone is again covered with oil, and the whole carefully inserted into liquid plaster of Paris contained in a small crucible 4 or 5 centims. in height. After the mixture has solidified, the cone may be easily withdrawn; the adhering paper filter is then detached, and any small pieces of paper still remaining removed by gently rubbing with the finger. In this manner a solid cone is obtained accurately fitting into a hollow cone, and of which the angle of inclination perfectly corresponds with that of the glass funnel.

Fig. 1 represents the cones. By their help the small platinum funnel is made. A piece of platinum (fig. 2 shows the natural size)* is cut from foil of such a thickness that one square centimeter weighs about 0.154 grm., and from the center *a* a vertical incision is made by the scissors to the edge *cbd*. The small piece of foil is next rendered pliable by being heated to redness, and is placed upon the solid cone in such a manner that its center *a* touches the apex of the latter; the sides *a b d* are then closely pressed upon the plaster, and the remaining portion of the platinum wrapped as equally and as closely as possible around the cone. On again heating the foil to redness, pressing it once more upon the cone, and inserting the whole into the hollow cone and turning it round once or twice under a gentle pressure, the proper shape is completed. The platinum funnel, which should not allow of the transmission of light through its extreme point, even now possesses such stability that it may be immediately employed for any purpose. If desired, it may be made still stronger by soldering down the overlapping portion in one spot only to the upper edge of the foil by means of a grain or two of gold and borax; in general, however, this precaution is unnecessary. If the shape has in any degree altered during this latter process, it is simply necessary to drop the platinum funnel into the hollow cone and then to insert the solid cone, when by one or two turns of the latter the proper form may be immediately restored. The platinum funnel is placed in the bottom of the glass funnel,

* The size of this cut has been reduced about one third. The diameter of fig. 2, in the original drawing, is 2.5 millimeters.—Eds.

the dry paper filter then introduced in the ordinary manner, moistened, and freed from all adhering air-bubbles by pressure with the finger. A filter so arranged and in perfect contact with the glass, when filled with a liquid will support the pressure of an entire atmosphere without the least danger of breaking; and the interspace between the folds of the platinum-foil is perfectly sufficient to allow of the passage of a continuous stream of water.

In order to be able to produce the additional pressure of an atmosphere, the filtered liquid is received in a strong glass flask instead of in beakers.* This flask is closed by means

* These flasks must be somewhat thicker than those ordinarily used, in order to prevent the possibility of their giving way under the atmospheric pressure.

of a doubly perforated caoutchouc cork, through one of the holes of which the neck of the glass funnel is passed to a depth of from 5 to 8 centimeters (fig. 3); through the other is fitted a narrow tube open at both ends, the lower end of which is brought *exactly to the level of the lower surface of the cork*, to the other is adapted the caoutchouc tube connected with the apparatus (fig. 4) destined to produce the requisite difference in pressure: this apparatus will be described immediately. The flasks are placed in a metallic or porcelain vessel (fig. 3), in the conical contraction of which several strips of cloth are fastened. This method of supporting the flask has the advantage that, in one and the same vessel, flasks varying in size from 0.5 to 2.5 liters stand equally well, and that, by simply laying a cloth over the mouth of the vessel, the consequences of an explosion (which through inexperience or carelessness is possible) are rendered harmless.

It is impossible to employ any of the air-pumps at present in use to create the difference in pressure, since the filtrate not unfrequently contains chlorine, sulphurous acid, hydric sulphid, and other substances which would act injuriously upon the metallic portions of these instruments. I therefore employ a *water* air-pump constructed on the principle of Sprengel's mercury-pump, and which appears to me preferable to all other forms of air-pump for chemical purposes, since it effects a rarefaction to within 6 or 12 millimeters pressure of mercury.

Fig. 4 shows the arrangement of this pump. On opening the pinchcock *a*, water flows from the tube *l* into the enlarged glass vessel *b*, and thence down the leaden pipe *c*. This pipe has a diameter of about 8 millims., and extends downward to a depth of 30 or 40 feet, and ends in a sewer or other arrangement serving to convey the water away. The lower end of the tube *d* possesses a narrow opening; it is hermetically sealed into the wider tube *b*, and reaches nearly to the bottom of the latter. A manometer is attached to the upper continuation of this tube *d* by means of a side tube at *d'*; at *d''* is attached a strong thick caoutchouc tube possessing an internal diameter of 5 millims. and an external diameter of 12 millims.; this leads to the flask which is to be rendered vacuum, and is connected with it by means of the short narrowed tube *k*. Between the air-pump and the flask is placed the small thick glass vessel *f*, in which, when one washes with hot water, the steam which may be carried over is condensed. All the caoutchouc joinings are made with very thick tubing, the internal diameter of which amounts to about 5 millims., the external diameter to about 17 millims. The entire arrangement is screwed down upon a board fastened to the wall, in

such a manner that each separate piece of the apparatus is held by a single fastening only, in order to prevent the tubes being strained and broken by the possible warping of the board. On releasing the pinchcock *a*, water flows from the conduit *l* down the tube *c* to a depth of more than 30 feet, carrying with it the air which it sucks through the small opening of the tube *d* in the form of a continuous stream of bubbles. No advantage is gained by increasing the rapidity of the flow, since the friction exerted by the water upon the sides of the leaden pipe acts directly as a counter pressure, and a comparatively small increase in the rapidity of the flow is accompanied by a great increase in the amount of this friction. Accordingly at *g* is a second pinchcock, by which the stream can be once for all so regulated that, on completely opening the cock *a*, the friction, on account of the diminished rate of flow, is rendered sufficiently small to allow of the maximum degree of rarefaction. Such an apparatus, when properly regulated once for all by means of the cock *g*, exhausts in a comparatively short time the largest vessels to within a pressure of mercury equal to the tension of aqueous vapor at the temperature possessed by the stream.* The tension exerted by the water-stream in my laboratory, in which six of these pumps are used, amounts to about 7 millims. in winter and 10 millims. in summer. The filtration is made in the following manner: The flask standing in the metallic or porcelain vessel (fig. 3) is connected by means of the slightly drawn-out tube *k* with the caoutchouc tube *h* attached to the pump, the cock *a* having been previously opened and the properly fitted moistened filter filled with the liquid to be filtered. As usual, the clear supernatant fluid is first poured upon the filter; in a moment or two the filtrate runs through in a continuous stream, often so rapidly that one must hasten to keep up the supply of liquid, since it is advisable to maintain the filter as full as possible. After the precipitate has been entirely transferred, the filtrate passes through drop by drop, and the manometer not unfrequently now shows a pressure of an extra atmosphere. The filter may be filled (in fact this is to be recommended) with the precipitate to within a millimeter of its edge, since the precipitate, in consequence of the high pressure to which it is subjected, becomes squeezed into a thin layer broken up by innumerable fissures. As soon as the liquid has passed through and the first traces of this breaking up become evident, the precipitate will be found to have been so firmly pressed upon the

* The time required to obtain the above degree of exhaustion in a flask of from 1 to 3 liters capacity ranges from six to ten minutes; the quantity of water necessary amounts to about 40 or 50 liters.

paper, that on cautiously pouring water over it it remains completely undisturbed. The washing is effected by carefully pouring water down the side of the funnel to within a centimeter *above* the rim of the filter: the washing flask for this purpose is not applicable; the water must be poured from an open vessel. After the filter has in this manner been replenished four times with water and allowed to drain for a few minutes, it will be found to be already so far dried, in consequence of the high pressure to which it has been subjected, that without any further desiccation it may be withdrawn, together with the precipitate, from the funnel, and immediately ignited, with the precautions to be presently given, in the crucible.

If the porosity of a paper filter containing a precipitate were as unalterable as that of a pumice-stone filter, the experiments above described would show that the times required for filtration, according to the old method on the one hand, and the new one on the other, would be inversely proportional to the difference in pressure in each case; that is, by using the pump under the full pressure of about 740 millims., the time needed to wash a precipitate, occupying by the old process an hour, would at the utmost not amount to more than 30 seconds. In using these pumice filters (about which I will speak presently) to drain crystals from adhering mother liquors, or, say, to wash crystals of chromic acid by means of concentrated sulphuric acid and fuming nitric acid, the time occupied in the filtration is scarcely longer than that needed to pour a liquid slowly from one vessel to another. In filtering by means of paper, the precipitate gradually closes up the pores of the filter, and accordingly such an extraordinary acceleration as this can no longer be expected. But the following examples will show the saving of time and labor the method effects, even under all unfavorable conditions. For these experiments I have purposely chosen the hydrated chromium sesquioxide, since it is one of the most difficult of precipitates to wash thoroughly. A solution of chromium chlorid was prepared by acting with fuming hydrochloric acid upon potassium dichromate; and by means of a measuring-vessel, which allowed the amount of chromium to be estimated to within 0.0001 grm., successive portions of the liquid were withdrawn, and the chromium oxide contained in them precipitated with the usual precautions by ammonia. The volume of liquid, the quantity of ammonia employed, the time occupied in boiling and in permitting the precipitate to settle, the angle of inclination possessed by the funnel, and the size of the filter were the same in all the experiments. All the precipitates

were washed with hot water, and, after burning the filter, ignited over the blowpipe for a few minutes; in weighing, the platinum crucible was tared by one of about equal weight, and the position of equilibrium of the beam determined by vibrations.

I first attempted to filter one of the precipitates in the ordinary way. $\frac{V}{v}$ amounted to 2; and consequently, from the table, 8·4 fresh additions of water were required in order to wash the precipitate to the $\frac{1}{10000}$ part. The times required were as follows:—

In transferring the precipitate from the beaker	}	40'
and allowing it to drain - - -		
For the first addition of water to run through,		48
“ second “ “		70
“ third “ “		80
		<hr/>
Total length of time - - -		238

At this point the experiment was discontinued, as the filtrate became turbid. A second experiment failed from the same cause.

Accordingly I attempted to wash the precipitate by decantation. The volume of the precipitate amounted to about 30 cub. centims.; the quantity of water required to fill the beaker was seven times the volume of the precipitate; hence $\frac{V}{v}$ was 7, and the requisite number of decantations to reduce the amount of impurity to the $\frac{1}{50000}$ part was 5·2. The times observed were as follows:—

II.

For the first decantation to run through the filter...	15'
“ second “ “ “	12
“ third “ “ “	18
“ fourth “ “ “	15
“ fifth “ “ “	18
In transferring the precipitate to the filter	30

Time required in washing	108
Weight of the precipitate	0·2458 grm.
Volume of wash water nV	1050 cub. centims.

III.

Experiment repeated. Number of decantations 7. Other circumstances the same as in the foregoing determination.

Time required in washing	140'
Weight of the precipitate	0·2452 grm.
Volume of wash-water	1200 cub. centims.

IV.

After ten decantations.

Time required in washing	180'
Weight of the precipitate	0·2443 grm.
Volume of wash-water	1750 cub. centims.

By filtration with the platinum cone and the pump the following results were obtained:—

V.

In transferring the precipitate to the filter (17 cub. centims. water)	6'
For the first addition of water (25 cub. cent.) to run through,	2
“ second “ “ “	3
“ third “ “ “	2
“ fourth “ “ “	2
“ fifth “ “ “	2
In draining the precipitate	2
Time required	19
Weight of precipitate	0·2435 grm.
Volume of wash-water	142 cub. centims.
Pressure of manometer	0·576 meter.

VI.

In transferring the precipitate and allowing the water (18 cub. centims.) to run through	8'
For the first addition of water (25 cub. cent.) to run through,	4
“ second “ “ “	5
“ third “ “ “	5
“ fourth “ “ “	5
In draining the precipitate	1
Time required	28
Weight of precipitate	0·2434 grm.
Volume of wash-water	118 cub. centims.
Pressure	0·600 meter.

VII.

In transferring the precipitate and allowing the water (20 cub. centims.) to run through	4'
For the first addition of water (25 cub. cent.) to run through,	3
“ second “ “ “	3
“ third “ “ “	3
In draining the precipitate	3
Time required	16
Weight of precipitate	0·2432 grm.
Volume of wash-water	95 cub. centims.
Pressure	0·584 meter.

VIII.

In transferring with 25 cub. centims. of water	8'
For the first addition of 25 cub. centims. to run through ...	5
For the second addition of 25 cub. centims. to run through ..	5
In draining the precipitate	3
Time required	21
Weight of precipitate	0·2435 grm.
Volume of wash-water	72 cub. centims.
Pressure	0·593 meter.

IX.

In transferring with 15 cub. centims. of water and allow- ing it to run through	} 7'
For a single addition to run through	
In draining the precipitate	2
Time required	12
Weight of precipitate	0·2439 grm.
Volume of wash-water	41 cub. centims.
Pressure	0·572 meter.

X.

In transferring the precipitate with 13 cub. centims. of water, ..	5'
For a single addition of water (26 cub. cent.) to run through, ..	8
In draining the precipitate	1
Time required	14
Weight of precipitate	0·2439 grm.
Volume of wash-water	39 cub. centims.
Pressure	0·530 meter.

In washing, by means of decantation, in the ordinary manner, the amounts of chromium sesquioxyd found were as follows:—

II. 0·2458, after 5 decantations, washed to the	500000 part.
III. 0·2452 “ 7 “ “	200000 part.
IV. 0·2443 “ 10 “ “	100000000 part.
0·2451 mean.	

By the use of the pump:—

V. 0·2435, after 5 additions of water.	grm.
VI. 0·2434 “ 4 “ “	“
VII. 0·2432 “ 3 “ “	“
VIII. 0·2435 “ 2 “ “	“
IX. 0·2439 “ 1 addition of water.	“
X. 0·2439 “ 1 “ “	“
0·2436 mean.	

Hence the probable amount of chromium sesquioxyl contained in the solution, according to the experiments with the pump, was 0.2436 grm. : according to the old method of decantation it was somewhat higher, namely 0.2451 grm. This excess of 1.5 milligram shows that the adhesion of the soluble matters to the precipitate and to the filter is, in consequence of the greater pressure, more easily overcome in the new method than in the customary process; it follows, therefore, that we can obtain a more complete washing by the new method than by the old. The old process of decantation required 108 minutes and 1050 cub. centims. of water to effect a washing to the $\frac{1}{5000}$ part; the new, on the contrary, only 12 to 14 minutes, and not more than 39 to 41 cub. centims. of wash-water. If a precipitate be heated in a platinum crucible immediately after filtration by the older process, a portion will inevitably be projected out of the crucible. Hitherto, therefore, it has been necessary to dry the filter and precipitate before ignition. Now to dry a quantity of hydrated chromium sesquioxyl containing 0.2436 grm. Cr_2O_3 in a water-bath at 100°C . requires at least five hours; and, moreover, bringing the dried precipitate into the crucible, burning the filter, and gradually igniting the mass is in the highest degree tedious and troublesome. All this expenditure of time and labor may be saved by employing the new method. By its means a precipitate is as completely dried upon the filter in from 1 to 5 minutes as if it had been exposed from 5 to 8 hours in a drying-chamber; and it can immediately, filter and all, be thrown into a platinum or porcelain crucible and ignited without the slightest fear of its spurting. By operating in the following manner the filter burns quietly without flame or smoke; this phenomenon, although remarkable, easily admits of an explanation. The portion of filter-paper free from precipitate is tightly wrapped round the remainder of the filter in such a manner that the precipitate is enveloped in from four to six folds of clean paper. The whole is then dropped into the platinum or porcelain crucible lying obliquely upon a triangle over the lamp, and pushed down against its sides with the finger. The cover is then supported against the mouth of the crucible in the ordinary way, and the ignition commenced by heating the portion of the crucible in contact with the cover. When the flame has the proper size and position, the filter carbonizes quietly without any appearance of flame or considerable amount of smoke. When the carbonization proceeds too slowly, the flame is moved a little toward the bottom of the crucible. After some time the precipitate appears to be surrounded only by an extremely thin envelop of carbon, possessing exactly the form

(of course diminished in size) of the original filter ; the flame is then increased, and the crucible maintained at a bright-red heat until the carbon contained in this envelop is consumed. The combustion proceeds so quietly that the resulting ash surrounding the precipitate possesses, even to the smallest fold, the exact form of the original filter. If the ash shows here and there a dark color, it is simply necessary to heat the crucible over the blowpipe for a few minutes to effect the complete removal of the trace of carbon. This method of burning a filter is extremely convenient and accurate ; it is only necessary to give a little attention at first to the slow carbonization of the paper, after which the further progress of the operation may be left to itself.

Gelatinous, finely divided, granular, and crystalline precipitates, such as alumina, calcium oxalate, barium sulphate, silica, magnesium ammonium phosphate, &c., may with equal facility be treated in this manner ; so that even in this particular the work, in comparison with the method generally adopted, is considerably shortened and simplified.

From the above experiments it appears that the time necessary to filter and dry a quantity of chromium sesquioxyd, hitherto requiring about 7 hours, is reduced by the new method to 13 minutes. This saving of time is, moreover, proportionately greater in the case of precipitates more easily filtered than hydrated chromium sesquioxyd. Particularly is this so in separating a finely suspended precipitate from a large volume of water. Under these circumstances the clear fluid runs through the filter in a continuous stream, so rapidly that it is scarcely possible to maintain the supply ; the entire operation, in fact, requires scarcely more time than that necessary to pour a liquid from one vessel to another. Filtration, therefore, may be effected as quickly through the smallest as through the largest filter. Moreover the exceedingly small amount of water required to wash a precipitate completely renders unnecessary the tedious evaporations which by the older method are almost inevitable when the filtrate is needed for a further separation. Thus the introduction of impurities from the action of the liquid upon the dish in the course of evaporation is prevented ; and also the loss due to the slight solubility of the greater number of precipitates in the wash-water is reduced to a minimum. Supposing we had to analyze an alkaline chromate in which the quantity of chromic acid is equivalent to 0.2436 grm. chromic sesquioxyd, as in the above described experiments, then to determine the proportion of alkali we should, by using the older method, require the preliminary evaporation of about 1050 cub. centims. of liquid ; by the new method the evapora-

tion of 40 cub. centims. only is necessary. Now by employing the best form of water-bath, i. e., one possessing a constant water-level, such as is used in my laboratory, it is possible, under favorable circumstances, to evaporate in a porcelain dish 1 cub. centim. of water in 27 seconds. Consequently the evaporation of the filtrate obtained by the older method would occupy about eight hours, whilst by the new 18 minutes only are required. The total length of time needed to filter the chromium sesquioxyd, wash and dry the precipitate, and evaporate the filtrate is reduced, therefore, from 14 or 15 hours to about 32 minutes.

The experience I have subsequently gained in my laboratory, where the method has been in general use for the last nine months, fully confirms the above results. It has shown that, on the average, three or four analyses can now be made in the time formerly demanded by a single one.

Another and an inestimable advantage springs from the peculiar condition of a precipitate filtered by this method. It not unfrequently happens, even in the hands of experienced manipulators, in consequence of the agitation it is necessary to give to the contents of the filter to effect their complete washing, that the surface of the filter becomes injured and torn, so that the precipitate becomes mixed with filaments of paper; this is particularly the case in using hot water. Supposing the precipitate to consist of mixed hydrates of the sesquioxys (for example, iron and alumina), it will be found, on redissolving in an acid, that the filaments, like tartaric acid, prevent the complete separation of these substances by subsequent precipitation; thus the alumina will contain iron, and on precipitation by means of ammonium sulphid will be colored black. On the other hand, by employing the new method the precipitate coheres so firmly that the introduction of this source of error is impossible, even by using common gray filter-paper. The most gelatinous precipitates, as hydrated ferric oxyd, alumina, &c., adhere to the filter in a thin coherent layer, and may be removed, piece after piece, so completely that the paper remains perfectly clean and white. The advantage thus gained where it is necessary to transfer mixed precipitates to another vessel in order to effect their subsequent separation is evident.

The filter-pump, moreover, is exceedingly serviceable in separating precipitates or crystals from syrupy mother-liquors. Thus honey-sugar may be so completely separated from the thick viscid liquid in which it forms by a filter of coarse gray paper, that it remains only slightly colored, and by a single crystallization from alcohol may be obtained in small white shining needles. And since the bulk of the moist precipitates,

particularly that of the more gelatinous, is so much diminished under the high pressure, the precipitate only occupying one-third to one-sixth of its bulk under ordinary circumstances, a filter of one-third to one-sixth of the size usually employed may be taken, and thus the amount of ash proportionately essened.

As the water air-pump suffers no injury from the presence of corrosive vapors or gases, we can equally well employ it to filter liquids containing nitrous acid, sulphurous acid, fuming nitric acid, chlorine, bromine, volatile chlorids, &c. In such cases I use a peculiar filtering arrangement, consisting of a cylindrical glass vessel, the lower end of which is drawn out before the blowpipe to the form shown in fig. 5 ; in this drawn-out portion a thin plate, 1 or 2 millims. in thickness, of *artificial* pumice, such as is used by polishers, is packed water-tight by means of asbestos. This apparatus is arranged for the purpose required exactly as the funnel in the method of filtration by pressure above described. In order to have a number of these filters in readiness, a pumice-stone cylinder of the required diameter is turned in a lathe, and then the thin plates sawn off by means of a small hand-saw in the small wooden support shown in fig. 6. The upper surfaces of the plates may afterwards be rendered perfectly even by a coarse file.

By the aid of these pumice-stone filters many chemical products may be made, the preparation of which has hitherto been almost impossible. For the sake of example I take the preparation of pure dry chromic anhydrid ; in an hour it is easily possible to filter, wash, and dry crystals of this substance an inch in length. A solution of two parts of potassium dichromate in 20 parts of water mixed with ten parts concentrated sulphuric acid, deposits, after standing about 24 hours, numerous brilliant needles of chromic anhydrid. These may be drained from adhering mother-liquor upon the pumice filter by means of the pump, and in a few minutes completely washed by a small quantity of fuming nitric acid free from nitrous acid. A covering of tolerably strong sheet copper provided with two arms, as shown in fig. 5, is then placed around the tube ; by hanging lamps upon the arms the tube may be readily heated to about 60° or 80° C. ; and by connecting a chlorid-of-calcium tube with the upper end of the glass vessel, a current of dry air may be drawn through the apparatus by means of the pump, and thus in a comparatively short time large and brilliant crystals of chromic anhydrid, perfectly dry and free from all impurity, may be easily obtained.

A single pump of the above description costs, including the

lead piping, about 8 thalers (24 shillings); and experience has shown that five or six are amply sufficient for a laboratory of fifty or sixty students. The apparatus, as may readily be seen, can be applied in the operation of evaporating *in vacuo*; if, however, circumstances will not permit of its being adapted to this purpose, then a fall of 10 or 15 feet is sufficient to filter a precipitate according to the above described method, and so far to dry it that it can be immediately ignited in the crucible. It is therefore not absolutely necessary to employ an air-pump in this process of filtration; any apparatus producing a difference of pressure amounting to a quarter of an atmosphere is sufficient. The simple arrangement represented in fig. 7 is very useful, and is frequently employed in my laboratory. It consists of two equal-sized bottles, *a* and *a'*, of from 2 to 4 liters capacity, each of which is provided near the bottom with a small stopcock designed to regulate the flow of water. Suppose *a* filled with water and placed upon a shelf as high above the ground as possible and *a'* placed empty on the floor, and the two stopcocks connected by means of caoutchouc tubing *c*, then on allowing water to flow down the tube the air in the upper bottle becomes somewhat rarefied; and in order to employ the consequent difference in pressure (amounting to a column of mercury about 0.2 meter in height) for the purpose of filtration, it is only necessary to connect the mouth of the upper bottle with the tube of the filter-flask. When the water has ceased to flow, the position of the bottle is reversed, when the operation recommences. So small a pressure as 0.2 meter suffices to render the filter and its contents so far dry that they may be immediately withdrawn from the funnel and ignited without any other preliminary desiccation. The following experiment, made with a portion of the same solution of chromium used in the former determinations, will serve to show the saving of time effected by this simple arrangement:—

XI.

Transferring the precipitate with 14 cub. centims. of water.....	14'
For a single addition of 26 cub. centims. of wash-water to run through.....	7
To drain the precipitate.....	4
Time required in washing.....	25
Weight of the precipitate.....	0.2435 grm.
Volume of wash-water.....	40 cub. centims.
Pressure in manometer.....	0.184 meter.

This amount of chromium sesquioxide (0.2435 grm.) differs from the mean of the former experiments (0.2436 grm.) by one-

tenth of a milligram only, and shows that even by a pressure of 0.184 meter the washing is as complete by the single addition of 26 cub. centims. of water. The duration of the filtering process in the former experiments ranged from 12 to 14 minutes under a difference of pressure amounting to from 0.53 to 0.572 meter ; in the last experiment it required 25 minutes under a pressure of 0.184 meter, or about double the length of time. The time needed to analyze potassium chromate in the former case was reduced from 14 hours to 32 minutes ; by the latter method the reduction would be from 14 hours to 44 minutes.

The employment of the second method is particularly to be recommended to beginners in qualitative analysis. The experimenter needs only a single funnel, he is obliged to work carefully and cleanly, and the great saving of time and work amply compensates for the little trouble needed to reverse from time to time the position of the bottles.

I believe that the above-described water air-pump will soon become an indispensable piece of apparatus in chemical laboratories. It not only serves as the most convenient method of producing the differences in pressure required to accelerate the process of filtration, and of obtaining the necessary vacuum for evaporation ; it is equally adapted to purposes to which neither the mercury nor the ordinary pumps are in any way applicable. By its aid it is possible to calibrate a thermometer with the greatest accuracy, and to estimate the vapor-tension of such corrosive bodies as bromine, chromyl dichlorid, &c., by the simplest method possible, in which the necessary operations require scarcely more time than an ordinary determination of a boiling-point.

I purpose returning to these applications of the instrument in a future communication.