SOME EXPERIMENTS IN FILTRATION.*

BY WILBUR L. SCOVILLE.

It has long been known that in the filtration of solutions of solids through paper there is a slight absorption [1] of the solid by the paper and consequently a change in the strength of the solution. This absorption takes place from the first portion of the liquid which passes through the filter, and is so slight that it is disregarded except in close analytical work.

In pharmaceutical operations it is frequently necessary to employ a clarifying agent in addition to filter-paper, and it has seemed desirable to determine whether these agents have a material absorptive action on the solids, sufficient to make a real difference in the strength of the solution.

Since powders used in this way need a filter-paper also, the same size and grade of paper was used throughout the experiments, viz., a 5-inch white filter, holding, when filled, about two fluidounces.

Preliminary tests showed that 500 mils of water will pass through a paper of this size in 4 minutes, the same amount of alcohol in 8¾ minutes, and of diluted alcohol in 12 minutes.

Double filters required almost exactly twice as much time in each instance.

The clarifying agents chosen were paper pulp, purified talc, infusorial earth, and fullers' earth. Two hundred and fifty mils of solution was filtered through each of these, using 2 Gm. of paper pulp or 5 Gm. of powder for each experiment. The infusorial earth was free from carbonate, and yielded only a trace of soluble matter to water or diluted alcohol.

The conditions of filtration were the ordinary methods of pharmacy, the powder being mixed with about 50 mils of the solution and the mixture poured on the filter, to be followed as needed by the rest of the solution until all had passed through. Unless filtration was extremely slow, the funnel was not covered. Paper pulp was moistened with the solution, packed firmly in the apex of the funnel, and the solution poured upon it, no filter-paper being needed with this agent.

Three types of solutions were employed, namely, a neutral aqueous solution, an acidulated aqueous solution—usually containing one percent of citric acid—and a neutral solution in diluted alcohol.

The strengths of the various solutions corresponded to the usual strengths employed in pharmacy or medicine.

The following tables show the results, the strengths being indicated by grammes per 100 mils, and the changes in percentage.

---

* Read before the Section on Practical Pharmacy and Dispensing, A. Ph. A., San Francisco meeting.

[1] After considerable discussion of the paper a motion was passed asking that the Scientific Section take the matter of absorption in filtration under consideration and solicit papers on the subject.

Quinine Hydrochloride Solution, 2 Percent.

(Strength Stated as Anhydrous Quinine.)

(Acid Solution Containing 0.5 Percent Free Hydrochloric Acid.)

	Aqueous, Neutral			Aqueous, Acid			Alcoholic		
	Original solution	Filtrate	Change, percent	Original solution	Filtrate	Change, percent	Original solution	Filtrate	Change, percent
Paper pulp.....	1.706	1.706	00	1.700	1.696	− 0.2	1.672	1.624	− 2.8
Purified talc.....	1.706	1.676	− 1.1	1.700	1.690	− 0.6	1.672	1.670	− 0.01
Infusorial earth....	1.706	1.644	− 3.6	1.700	1.674	− 1.5	1.672	1.672	0.00
Fullers' earth.....	1.706	1.112	−34.8	1.700	1.326	−22.1	1.672	1.232	−26.2

Strychnine Sulphate, 1.28 Percent.

(Corresponding to 1.0 Percent Strychnine Alkaloid.)

(Acid Solution Containing 1 Percent Free Citric Acid.)

	Aqueous, Neutral			Aqueous, Acid			Alcoholic		
	Original solution	Filtrate	Change, percent	Original solution	Filtrate	Change, percent	Original solution	Filtrate	Change, percent
Paper pulp.....	1.000	0.989	− 1.1	1.000	0.995	− 0.5	1.000	0.995	− 0.5
Purified talc.....	1.000	0.996	− 0.7	1.000	0.996	− 0.4	1.000	1.009	+ 0.9
Infusorial earth....	1.000	0.973	− 2.7	1.000	0.960	− 3.5	1.000	1.046	+ 4.6
Fullers' earth.....	1.000	0.000	−100	1.000	0.000	−100	1.000	0.004	−99.6

Caffeine.

(Acid Solution Corresponding to Citrated Caffeine.)

	Aqueous, Neutral			Aqueous, Acid			Alcoholic		
	Original solution	Filtrate	Change, percent	Original solution	Filtrate	Change, percent	Original solution	Filtrate	Change, percent
Paper pulp.....	0.788	0.788	00	0.785	0.785	00	0.781	0.781	00
Purified talc.....	0.788	0.784	− 0.5	0.785	0.780	− 1.0	0.781	0.785	+ 0.56
Infusorial earth....	0.788	0.789	+	0.785	0.790	+ 0.6	0.781	0.783	+ 0.3
Fullers' earth.....	0.788	0.486	−35.3	0.785	0.466	−40.9	0.781	0.540	−31.5

Morphine Sulphate, 1.5 Percent.

(Corresponding to About 1.25 Gm. of Crystallized Morphine Per 100 Mils.)

	Aqueous, Neutral			Aqueous, Acid			Alcoholic		
	Original solution	Filtrate	Change, percent	Original solution	Filtrate	Change, percent	Original solution	Filtrate	Change, percent
Purified talc.....	1.242	1.2055	− 3.0	1.253	1.251	− 0.2	1.231	1.248	+ 1.3
Infusorial earth....	1.242	1.188	− 4.4	1.253	1.141	− 9.0	1.231	1.262	+ 2.5
Fullers' earth.....	1.242	0.700	−43.7	1.253	0.718	−42.7	1.231	0.757	−38.5

Atropine Sulphate, 0.7 Percent.

(Corresponding to 0.400 Gm. of Alkaloid Per 100 Mils.)

Agent	Aqueous, Neutral			Aqueous, Acid			Alcoholic		
	Original solution	Filtrate	Change, percent	Original solution	Filtrate	Change, percent	Original solution	Filtrate	Change, percent
Paper pulp	0.400	0.397	− 0.7	0.400	0.397	− 0.7	0.400	0.393	− 1.7
Purified talc	0.400	0.377	− 5.7	0.400	0.377	− 5.7	0.400	0.388	− 3.0
Infusorial earth	0.400	0.361	− 9.7	0.400	0.380	− 5.0	0.400	0.415	+ 4.0
Fullers' earth	0.400	0.00	−100	0.400	0.00	−100	0.400	0.00	−100

Acetanilid.

	35 percent alcohol solution			49 percent alcohol solution		
	Original solution	Filtrate	Change, percent	Original solution	Filtrate	Change, percent
Paper pulp	.....	.....	.....	3.400	3.420	+0.5
Purified talc	3.420	3.260	− 4.7	3.400	3.400	0.0
Infusorial earth	3.420	2.430	−29.0	3.400	3.505	+3.0
Fullers' earth	3.420	2.964	−13.4	3.400	3.350	−1.5

Two things are clear in these results. First, that excessive amounts of any filtering medium mean a loss in strength of the solution, and this loss is the greater the nearer the solution is to its saturation point. Weak solutions may show little or even no loss, while strong solutions show a considerable loss.

Second, fullers' earth is decidedly absorptive to alkaloidal solutions, but less so to neutral bodies. It will be noticed that the strychnine and atropine were entirely removed from solution by this agent, while other alkaloidal solutions lost a quarter to nearly half their strength.

These figures would doubtless vary with the proportion of earth employed, but even in small portions it will cause a loss.

The other three agents, paper pulp, purified talc, and infusorial earth, probably do not differ very much in their absorptive power, but they differ in their adaptability.

Paper pulp was found to filter quite readily, if not packed too firmly. It occasions little loss, but is not so efficient a clarifying agent as the powders.

In comparing talc with infusorial earth there is first to be noted that, while equal weights (5 Gm.) of these agents were used in the experiments, the infusorial earth is so much lighter than talc that more than twice as much, in bulk, of the earth was used and consequently the absorption appears greater.

Filtration was also much slower through the infusorial earth, and in the case of the alcoholic solutions the loss by evaporation from the filter more than counterbalances the loss by absorption, and the filtrates frequently show a gain in strength. Even when equal bulks of these two agents are used, infusorial earth is less than a quarter as rapid in action as talc, and, except for work in a filter press, is much less desirable as a clarifying medium.

Another factor to be observed in considering infusorial earth is that much of it contains carbonates, and even when these are not soluble in neutral liquids they may react with substances in solution.

In an experiment made in the early part of this investigation in which 1 percent solutions of strychnine (as sulphate) lost 49.1 percent, 42.6 percent, and 36.1 percent of their strength, respectively, the losses were found to be due to the presence of carbonate in the earth, though the carbonate was not sufficiently soluble in distilled water to produce an alkaline reaction.

For general use purified talc is the most satisfactory clarifying medium for pharmaceutical purposes, but care should be taken to avoid loss both of the solution and of strength, by excess. The smallest quantity that will do the work should be employed, both for economic reasons and for standard results.

Another point suggested by these experiments is that in making preparations which need to be clarified it is safer to adjust the final volume before clarifying than to make up to the full yield by passing enough menstruum through the filter to obtain it. Since the material absorbed in the filter is richer in dissolved or absorbed substances than the filtrate, washing with a relatively small amount of menstruum is not likely to displace it entirely, and the product is so much the weaker. The loss of a small portion of the product is better than the weakening of the whole.

LABORATORY OF PARKE, DAVIS & Co., Detroit, Mich.

PERFUMES.*

BASIC MATERIALS, PRODUCTION, PRESERVATION AND SELLING POINTS.

BY WILLIAM A. HALL.

In considering this subject, I am impelled chiefly to throw what light I can for an educative effect, and from that, by deduction and the experience such statements as may, if followed, result in a better understanding of this article of commerce, the limitations and means to be taken for our financial benefit.

From the earliest written records the item of *perfumes* has ever played an important part and always has it seemed to be not merely a luxury but a necessity. Writers have vied with each other in describing the art of perfume manufacturing, and many uses to which the products were put. From sage to savage, from ruler to the humblest citizen, has come the demand for this invisible scent or perfume, and to furnish it have collectors, chemists, steam and sail, perfumer and dealer been requisitioned.

The crude methods of the ancients have yielded to the refined processes of the present, but the facts are as true to-day as of yore, and emphasis should be laid here that the floral odors, as produced by Nature and conserved by man, cannot be successfully supplanted by the synthetical coal-tar products of the present, so freely exploited.

Rankness, as against fragrance, variation *vs.* stability, greater present profit *vs.* permanent future business, you may have, but who wishes to exchange the promise for the reality, when it comes to our commercial efforts?

If one rose is fair and sweet, as is universally acknowledged, think of nineteen hundred sixty tones of roses, the product of one season, in the Valley of Var in the south of France; think of one hundred tons of roses a day, often gathered at Grasse, the rose centre of that region.

At Grasse, they use three thousand pounds of rose leaves to make a pound of attar or *otto of roses,* the well-known oil distilled from the fragrant petals, and

* Read before the Section on Commercial Interests, A. Ph. A., San Francisco meeting.