As a third check a sample of pure dry nitroglycerine may be used.

At one time in my work the reduction tube was set by aid of a barometer, since shown to be faulty. A sample of potassium nitrate carefully purified showed in this case about 99 per cent. purity and a sample of sodium nitrate also carefully purified showed the same percentage of purity within the limits of error. The barometer setting of the reduction tube was therefore discarded and the correction factor introduced since it was manifest that, while either of the nitrates might be impure even after the most careful purification, it was in a high degree improbable that both should be impure and of exactly the same degree of impurity.

During the hot weather of summer there is a distinct advantage in having a large subtractive correction factor. In this way the gas may be measured nearly at the pressure of the atmosphere, thus avoiding the considerable strain on the stop-cock and consequent tendency to leakage.

The nitric oxide may be measured either dry or moist, more conveniently, however, in the latter condition. If so measured a few drops of water should be left in each tube. As the tension of the aqueous vapor is the same in each tube, it may be neglected in the calculations.

The sulphuric acid used in nitrometer work should be free from oxides of nitrogen and iron. It should be of 94 to 95 per cent. strength. In acid of 98 per cent. strength nitric oxide is quite freely soluble.

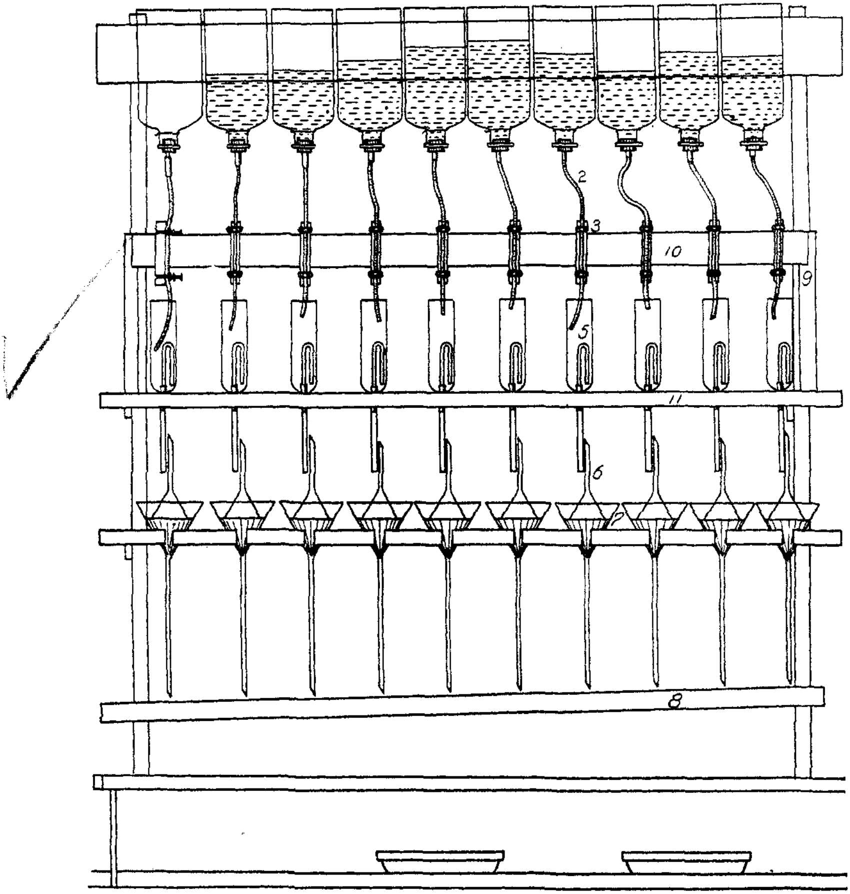
CALIFORNIA POWDER WORKS,
April, 1901.

AN AUTOMATIC FILTER-WASHER.

BY J. M. PICKEL.

Received June 21, 1901.

THE apparatus shown in the accompanying cut, consists of a battery of ten washers. The parts of each washer are: A reservoir, *1*, to contain the liquid with which the washing is done. A rubber tube, *2*, provided with two thumb-screw clamps, *3*, leading from the reservoir to the delivery vessel. A delivery vessel, *5*, provided interiorly with a small siphon, which delivers the washing liquid intermittently in small portions on the substance being washed. A larger funnel, *7*, containing the filter. A smaller inverted funnel, *6*, covering the substance. The object of this funnel is, while it prevents spattering, to throw the liquid around over the edge of the filter-paper, thus insuring that it be washed from the top downward at each delivery. It should be of such a diameter as almost, but not quite, to cover the filter-paper. A trough, *8*, which conveys the washings, in case they are not wanted, into the waste pipe. A supporting frame, *9*, *10*, *11*.



The apparatus is operated thus: The substance to be washed having been placed on the filters and covered with the inverted funnels, and the lower clamps having been closed, each reservoir is provided with a measured quantity of liquid adequate to the washing. To facilitate the measuring, the reservoirs should be graduated once for all. By means of the lower clamp discharge the liquid rapidly into the delivery vessel until the siphon overflows, then regulate (by the *lower* clamp) the flow so that the liquid shall fall, drop by drop, into the delivery vessel at such a rate that the siphon shall not overflow till after all the liquid has passed out of the filter. This regulation insures the intermittent washing of the filter with entirely fresh portions of liquid at each delivery, and, with a little practice and patience, can be accomplished in about ten to fifteen minutes for all ten of the washers. After the apparatus is once regulated, it requires no further attention—may be left going over night.

The upper clamp is not necessary, but is at times convenient; for example, if for any reason it is desired to stop the flow of liquid before the washing is completed, this is effected preferably by the upper clamp, thus leaving the "*set*" of the lower clamp undisturbed. The efficiency of the machine depends upon the ease and accuracy with which it may be regulated. The screw of the regulating clamp must, therefore, have a fine thread; the clamp must be firmly fixed in place, otherwise the merest touch will sometimes change the rate of flow. The washer at the extreme left of the cut shows a method of securing this fixedness. The clamps and attachment are there turned through an angle of 90°. A piece of wood about two inches long and of rectangular section passes through the clamp. Transversely across the back of the piece of wood are shallow grooves in which the clamps fit. These pieces are firmly attached by screws to the frame-piece marked *10*. Small wire brads, one just above and one just below the clamp, and each slightly pressing against it, are driven a short way into the wood completing thus the steadiness of the clamps. The arrangement here described is satisfactory; but doubtless a tube and glass cock, capable of delivering small drops (after the manner of a good burette), would be preferable.

The quantity of water delivered at each discharge of the siphon should, of course, be sufficient to cover the substance but not enough to flood the filter to overflowing. The capacity of the delivery vessel may be varied by adding to or taking from it, small glass beads, coarse sand, or fragments of glass. It is evident that, if the siphon is on the outside of the delivery vessel (after the manner of a Soxhlet extractor), the capacity of the vessel may easily be varied from its greatest volume down to an almost vanishing quantity by shoving down into it a tube closed at its lower end (a test-tube for example); a small plug of rubber wedged in between would hold the tube at any desired level. A Soxhlet can be utilized as a delivery vessel; one, in possession of the writer, whose normal maximum delivery is 43 cc. can, in the manner just described, be instantly made to deliver any lesser quantity down to 7 cc.

As is seen from the cut, the delivery vessels in the apparatus here described are what are known as carbon filters, for Gooch filters. Their dimensions are, for the large end, about 2 to 3 inches in length and $1\frac{1}{8}$-inch internal diameter, small or stem end, about 2 inches in length and $\frac{1}{4}$-inch internal diameter. The long arm of the siphon passes through the stem and is made water-tight by a short piece of rubber tubing. They were regulated once for all to deliver about 10 to 15 cc. The siphons should be well made, particular care being taken that they are not flattened, or contracted, at the bend. A siphon thus contracted requires, especially when it becomes somewhat soiled internally and does not readily wet, considerable pressure to force the water over; it will therefore vary considerably in the volume of liquid that it delivers at an overflow. For this reason also a narrow tall delivery vessel is to be preferred to one of larger diameter—there is less variation in the quantity of liquid delivered at each overflow of siphon in the former than in the latter. A diameter (internal) of not over an inch—of even three-quarters of an inch or less if practicable—is to be recommended. I especially call attention to this, because I found that some of my delivery vessels, which were set to deliver about 10 cc. would, owing to the causes here detailed, sometimes cause my filters to overflow (I was using quite small, 9 cm. filters). The reservoirs in this apparatus were made by cutting off, near the bottom, pint bottles. Small percolators would be neater.

The combination here described as an automatic filter-washer is, so far as the writer knows, new. At any rate, it was new to him and devised by him for washing water-soluble nitrogen (more especially nitric and ammoniacal nitrogen) out of mixed commercial fertilizers. It was used the past winter and spring for that purpose in the analysis of several hundred samples, and was found entirely satisfactory and a valuable labor-saver. Two nitrogen determinations were made in each case, one of total nitrogen, the other of residual nitrogen after washing with 300 cc. of distilled water.

There would seem to be no reason why this washer might not be used, with equal advantage, to take water-soluble phosphoric acid out of commercial fertilizers. A comparison of its work in this matter with the method of washing commonly practiced, was made with the following result on ten different fertilizers :

	Per cent.									
	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.
Water-soluble P_2O_5.	6.65	4.55	9.51	9.31	8.04	9.24	5.97	6.10	5.06	6.00
" "	6.42	3.78	8.92	8.75	7.88	8.65	6.10	5.68	4.72	5.42
Difference......	0.23	0.77	0.59	0.56	0.16	0.59		0.42	0.34	0.58

The percentages in the top line were obtained in the washings from the automatic washer; those in the second line were obtained in the usual way by directing a jet of water on the substance and thoroughly stirring up at each washing. Two grams of fertilizer were taken in each case, and the same quantity (or about the same quantity), a little less than 300 cc. of water, was used for washing in each case. In all except one case, the automatic washer took out more phosphoric acid than the usual method. This is all the more remarkable inasmuch as the washer does not *stir* the substance to any appreciable extent. The explanation of the difference is perhaps to be found in this circumstance ; the washer delivered the water in small portions of about 10 cc. at a time, whereas in the other case 30 to 40 cc. were used, the filter being larger; it thus came about that in the first case the samples got three to four times as many separate washings as in the second. There was this further circumstance: The machine, once set going, keeps up the work to the end, thus finishing the operation in a shorter time than did the hand washing, where the chemist was diverted by other operations which he was looking after at the same time. Such being the case, the phosphates were in contact with the water a shorter time in the first than the second instance, and the monocalcium phosphate had less opportunity (less time) to "*revert.*"

ANALYTICAL AND CONSULTING LABORATORY OF
DR. J. M. PICKEL, RALEIGH, N. C.,
June, 1901.