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BACKFLUSHING FILTERS FOR FIELD PROCESSING OF WATER SAMPLES PRIOR TO TRACE-ELEMENT ANALYSES

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Menlo Park, California
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ILLUSTRATIONS

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ABSTRACT

A portable unit is described for filtering water samples at field sites in such a manner that the filtrate is suitable for analysis not only of major constituents but also of trace elements at the microgram-per-liter level. A battery-operated peristaltic pump forces the water sample through medical-grade silicone tubing into and through an all-plastic in-line filter which can be backflushed when sediment clogs the filter membrane. Initial filtration rate exceeds 500 mℓ/minute and, because of the backflushing feature, a total time for filtering high-sediment-bearing water samples is greatly reduced.

Field filtration of sediment-bearing water samples has been a slow process because filter membranes or filter papers (referred to simply as membranes or papers hereafter) may clog rapidly, thereby greatly lengthening the time for processing samples. As field filtration becomes a more common procedure, especially in the processing of natural waters prior to trace-element analysis, the need for suitable equipment has become increasingly evident. In response to this need, equipment has been devised for field filtration which is comprised in part of commercially available materials and in part of custom-made apparatus. The equipment has been used successfully for more than 2 years by the authors and their colleagues.

The filtration equipment is basically very simple. It is composed of a reversible battery-operated peristaltic pump which forces the sediment-laden water through flexible tubing into a plate-type "filter" (the term "filter" includes membrane amd support structures). There the sediment is retained by a membrane while the filtrate passes through to a collection vessel. The membrane is supported both above and below by a plastic screen, which permits water flow in either direction without disruption of the membrane. In operation, the practice is to place the intake tube in the water sample to be filtered and the peristaltic pump is run in the forward direction until filter clogging reduces output to an unacceptably slow rate. At that point the pump is stopped, the output tube is held under the surface of the filtrate, the inlet tube is switched to a separate container and the pump is then operated rapidly and briefly in reverse. This action creates a vacuum on the top of the filter membrane causing backflow of filtrate and lifting of the sediment off the membrane. As soon as most of the sediment removal has occurred, the pump is stopped, the intake tubing is replaced in the water sample, and the pump again run forward. If the sample volume is large compared to the required volume of filtrate, there is no need to switch the intake line to a separate container on backflushing because the sediment concentration in the orginal sample may not increase enough to be a problem. Such a situation exists when the inlet tubing is placed directly in a stream.

It is frequently worthwhile to repeat the backflushing step after a short forward run before continuing with prolonged filtration. Our experience has been that there is some loss of filtration capacity despite the backflushing. However, 50 to 70 percent of the initial capacity is generally retained after the first backflushing of suspended river sediment, and thereafter the capacity declines slowly with each filtration cycle.

The backflushing filters have been built in two sizes to meet two different requirements. For the average muddy water sample where 1-2 ℓ of filtrate are adequate, a 142-mm-diameter filter is acceptable; however, if more than a few liters of sample are needed and sediment concentration is high, a 293-mm-diameter filter greatly speeds the filtration. Alternately, the 293-mm filter can be used where a large sample of suspended sediment must be collected from a stream containing only small concentrations of sediment. In this case, higher capacity pumps can be used to rapidy filter the larger volumes of water needed.

A picture of the 142-mm filter is shown in figure 1. The top Lucite plate (shown bottom-side up on the right in the photograph) has a set of distributary grooves on the bottom side which connect to a central entry tube. Below the top plate is a polyester screen rimmed with a seal of resilient silicone. Next is the membrane which sets on a second rimmed polyester screen (membrane and screen not shown). On the bottom, with its upper surface grooved for collection of filtered water and holding a silicone "O" ring in an outside circular groove, is the lower Lucite plate (stacking sequence for the 142-mm filter is as shown for screens and membrane of the 293-mm filter in figure 4). When the flip-off tightening bolts on the bottom Lucite plate are secured, the membrane is held in a porous sandwich of polyester screen sealed at the outer edge by the "O" ring. A vent on the upper plate allows air overlying the membrane to escape prior to the beginning of filtration. This vent is closed after the air is exhausted. Figure 2 shows the dimensions for the 142-mm filter and figure 3 is a photograph of the 142-mm filter and battery-operated peristaltic pump set up as normally used under field conditions.

All parts for the 142-mm filter can be machined or readily purchased except the rimmed polyester screens. These are made by cutting circular 100-mesh polyester screens to size and then impregnating the edges with liquid silicone to which a catalyst has been added (Dow Corning Corp. silastic 392 elastomer 1/). The silicone is then smoothed and allowed to set. At the time this report was written attempts were being made to obtain a commercial source for these rimmed polyester screens.

The 293-mm filter has the same basic design (figures 4 and 5) as the 142-mm filter, but a center bolt is provided for added strength and an additional "O" ring and central rims on the screens are necessary. (Suitable support screens for the 293-mm filter can be obtained from Nuclepore Corporation, Pleasanton, CA, or from various laboratory supply companies). The inlet ports are positioned to aid distribution of flow over the filter, and two collection ports are also provided. Plans for the 293-mm filter are shown as figure 4. In the most recent models radial distributary grooves in the upper plate and collection grooves in the lower plate are not at right angles in order to avoid interference between flip-off bolts and connections to inlet or outlet ports.

1/ The use of brand names in this report is for identification purposes only and does not imply endorsement by the U. S. Geological Survey.

The filtering operation can be described as follows. After placing the membrane between two screens which are resting on the bottom plate, the top plate is installed and bolts are tightened firmly by hand. The filter is then placed on edge in the position indicated for the "top plate" which is shown in the upper right corner of figure 4. Clamp A is closed and clamp B is open when pumping of the sample begins. This causes water to flow into the filter through the bottom port and fill the space between the top filter plate and the upstream side of the membrane. Air is exhausted through the upper port and the tubing held in open clamp B. When water begins to issue from the upper port, clamp A is opened to allow air in the tubing to escape and clamp B is closed. The filter can then be placed in a stable horizontal position with little or no air present to reduce filtration rate. If a little air remains in the system, it will propably be removed with the first backflush. After filtering is completed, care should be taken on dismantling the filter to assure that the lower screen is not contaminated by sediment caught on the membrane or upper screen. A clean membrane should be kept between screens in the filter to prevent contact of the soiled upper and clean lower screens and one should avoid accidental interchange of the filter screens, i.e., the sediment screen should never be used on the filtrate side of the membrane. Thorough cleaning of the whole unit is recommended before extended storage.

The equipment described is believed to provide a minimum of contamination or absorptive losses while, at the same time, allowing effective removal of suspended solids. Medical-grade silicone tubing is used throughout the system. It has relatively long life in the peristaltic pump and does not detectably alter the trace-element content of the stream waters at the fractional microgram-per-liter level. Clear silicone "O" rings are used for the same reason. Both rubber and neoprene "O" rings contain several percent Zn, which rapidly contaminates water samples.

The reversible battery-operated peristaltic pump is a modified version of a commercial unit (available from several laboratory supply houses as a "Masterflex" portable pump). As supplied, the pump can be operated in one direction only from 115V AC, 12V DC or from an internal rechargeable battery. The motor itself operates from 12V DC and is reversible by installing a double-pole, double-throw, center-off switch in the lines to the motor. If desired, a speed control can also be added by placing a rheostat in series with the motor. We have used a 10-ohm, 25-watt rheostat successfully. Care must be taken to assure that the motor is not reversed without an intermediate stop, because the gears may be stripped by very rapid reversal.

For backflushing, the silicone tubing used should have a thick enough wall that it will not collapse when vacuum is applied. This can be achieved if silicone tubing of 3/16 inch I.D. and 3/8 inch O.D. is used with an appropriate pump head. Such an arrangement has a pumping capacity of about 650 mℓ/minute.

If valid trace-element data are to be obtained, considerable care must be taken to reduce addition or removal of trace elements by the membrane itself. Various investigators (Jenkins, 1968; Marvin and others, 1970, 1972; Zirino and Healy, 1971; Kennedy, V. C. and Zellweger, G. W., unpublished data, 1975) have shown that the membranes can release Na, Mg, Ca, Cu, Zn, NO_3 and PO_4. Less well recognized is the problem of absorption of trace elements by the filters. Sandell (1944) called attention to the fact that filter paper may adsorb such material as Pb and Cu, but little work has been done to attack the problem since that time.

In the normal filtration procedure, as sediment builds up on the membrane the flow-through rate decreases greatly and there is enough contact time for significant leaching of contaminants from the membrane or for sorptive losses of some metals to the membrane. When trace-metal concentrations are near background, i.e., in the low or fractional microgram-per-liter range, such membrane effects should not be ignored. Where trace-metal levels are several tens of micrograms-per-liter, as is true for some streams in industrialized areas, these effects may not be important. When the backflushing procedure is used, the original buildup of sediment will occur during the period when initial leaching and equilibration of the water with the membrane takes place. After backflushing the filter to increase the flow rate, the actual sample to be analyzed can be taken when the flow rate is relatively high so as to reduce contact time with the membrane. If a sample were taken early in the initial filtration when flow rate was high, contaminants easily removed from the membrane would also be included inthe sample and sorption effects would be emphasized also.

Although a completely satisfactory method of separating suspended particulates from natural waters is not available now, the equipment described appears to be as good as can be made using existing materials. For the 142-mm filter a minimum of 1-2 ℓ of water sample should pass through the system at a moderate flow before any sample is retained. Should there be significant clogging of the filter, a backflushing step following the wasting of at least 1 ℓ would be appropriate. When using the 293-mm filter and the peristaltic pump described to concentrate a suspended-sediment sample, there is merit in using a 0.1 or 0.22 μm pore size instead of a 0.4 or 0.45 μm pore size because the backflushing appears to be somewhat more effective with the finer pore sizes. When 0.4 or 0.45 μm membranes are clogged and a backflushing rate of 500-600 mℓ/minute is used, a part of the filter cake lifts off and the resultant open area(s) permits easy passage of all the backflushing water the pump can draw. The remainder of the filter cake can remain relatively undisturbed. When smaller pore sizes are used, the partial removal of filter cake seems to be less common. If a larger capacity pump were used with the 293-mm filter, this propably would not be a problem. When fine clay or algae are the dominant components of suspended sediment, the backflushing system is least efficient, even though improvement on filtering rate can still be obtained compared to filtration without backflushing.

In summary, the equipment described should enable one to obtain easily and rapidly in the field a filtered water sample whose composition represents as closely as practicable the dissolved constituents in sediment-bearing natural waters.

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ILLUSTRATIONS

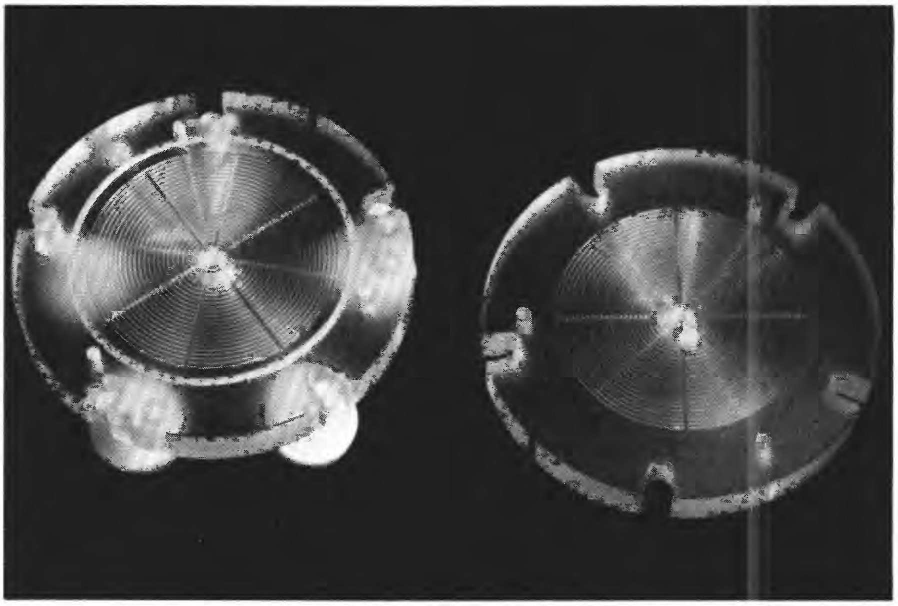


Figure 1. Photograph of a 142-millimeter filter

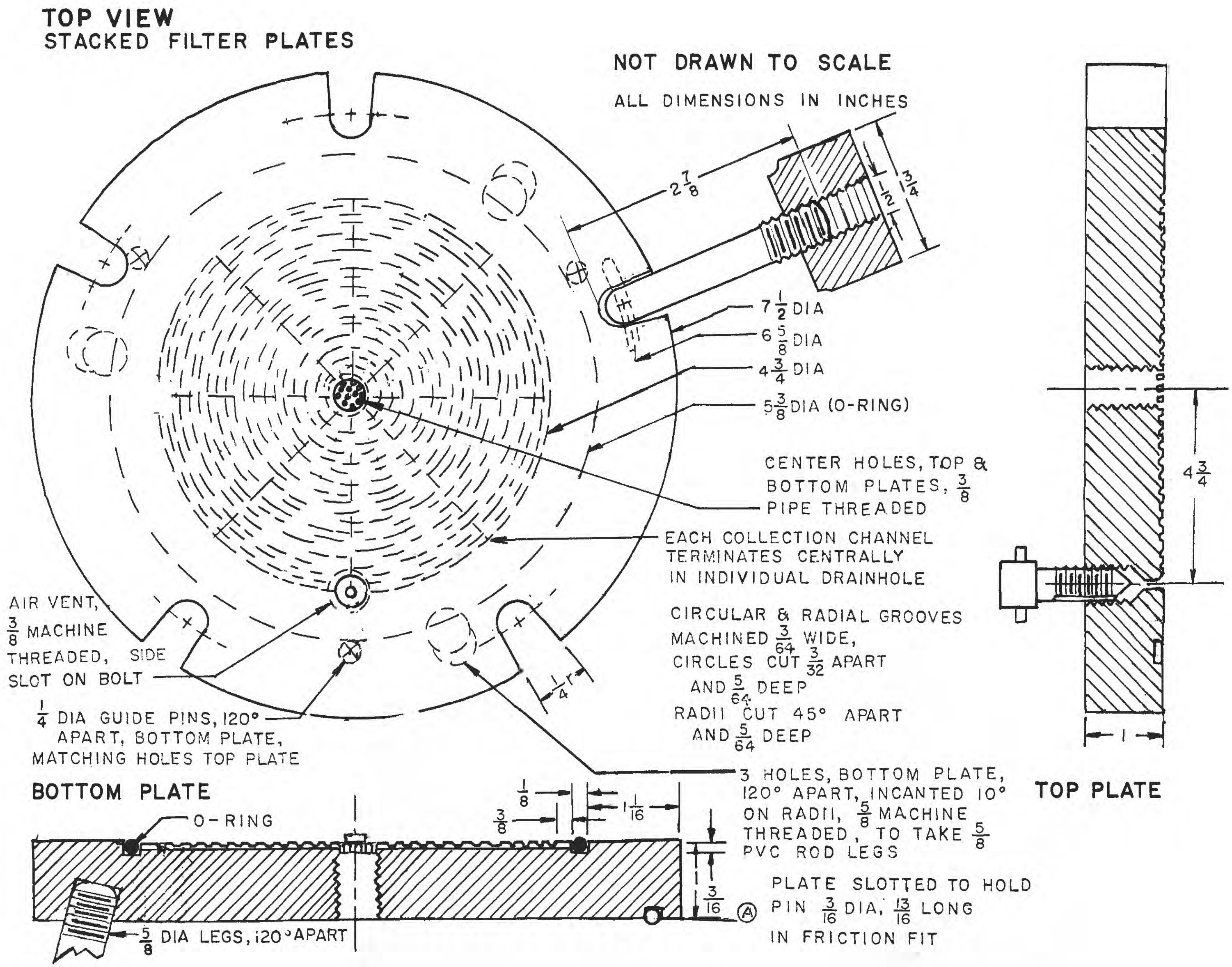


Figure 2. 142-millimeter backflushing filter

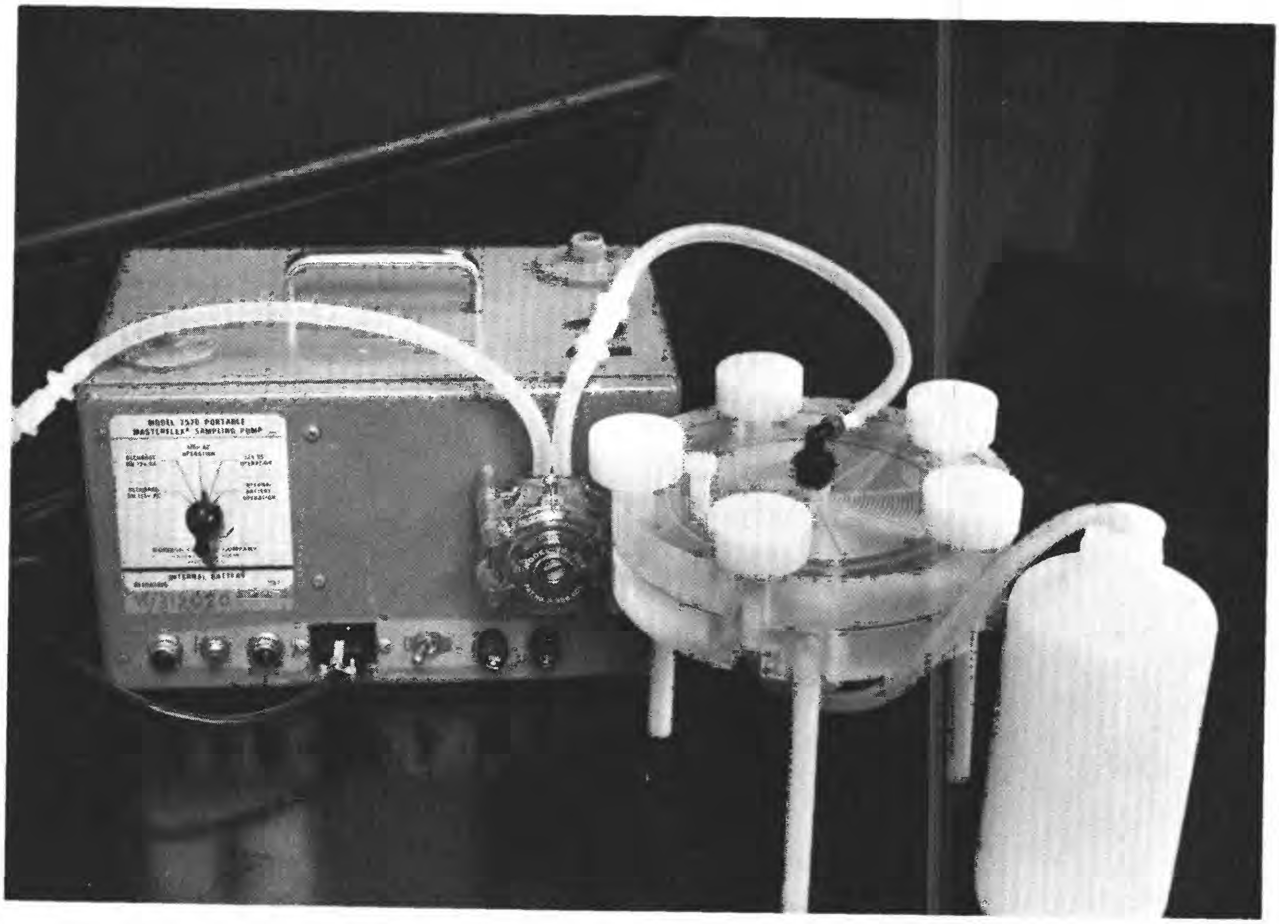


Figure 3. Photograph of 142-millimeter filter connected to battery-operated peristaltic pump

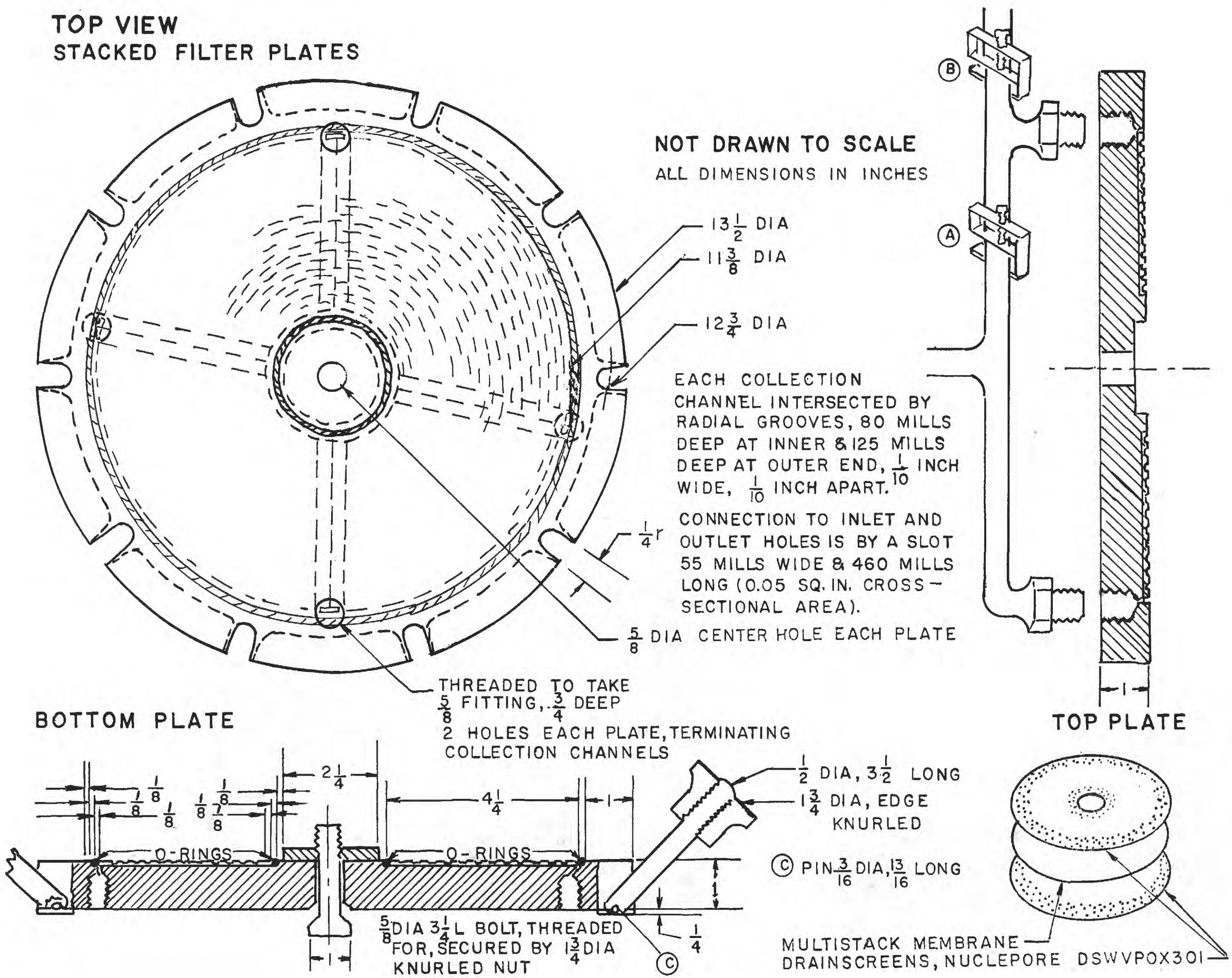
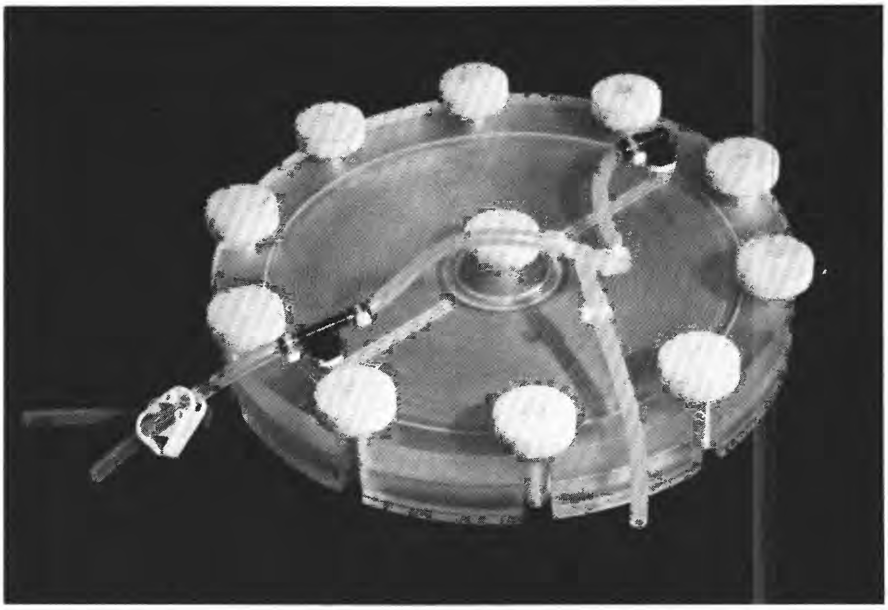


Figure 4. 293-millimeter backflushing filter



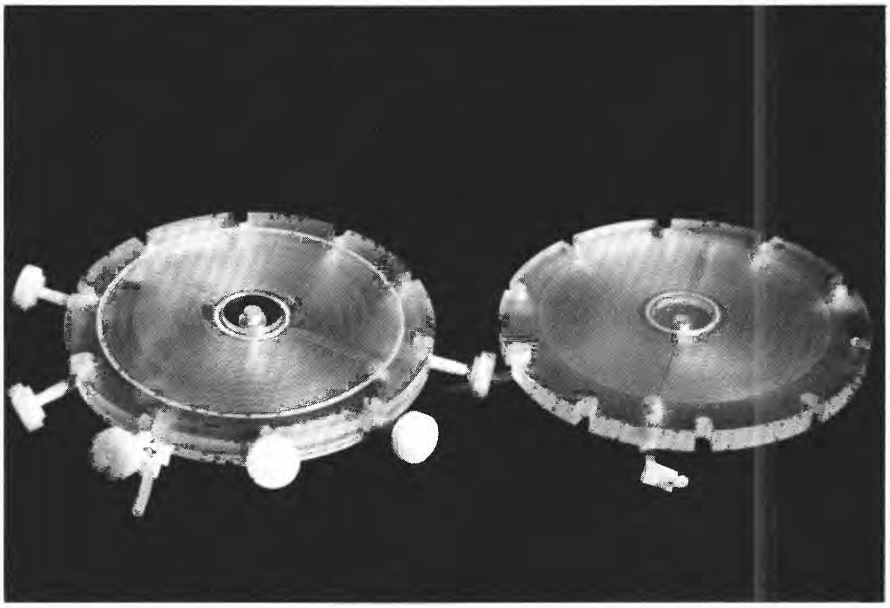


Figure 5. Photographs of 293-millimeter filter