Devices Useful for Oxygen-Filled Flask Combustions

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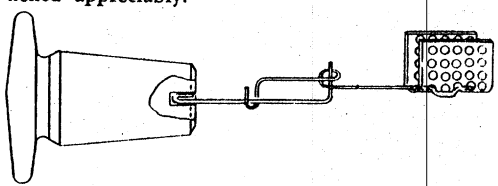
Technics involving the oxygen-filled flask combustion of organic samples have been found useful for many determinations. Several devices which expedite and simplify the combustion procedure have been developed in the course of a study of the determination of phosphorus in leather (*1*). These devices include a self-demountable sample carrier, a tray for multiple samples, and a pipet drainer.

The sample carrier with its supporting wire (*2*) is so arranged that the former can be readily disengaged. This allows the sample carrier to be immersed in any absorbent solution for effective solubilization of combustion residues without opening the flask. The tray for multiple samples permits the convenient transport of a series of either solid or liquid samples.

The pipet drainer (*3*) allows easy positioning of pipets with control of the drain angle. It can be used with a wide range of pipet sizes, 2- to 100-ml. capacity, and also permits the draining of one or more pipets while others are being filled. Large groups of samples or survey procedures requiring dilutions above or below the suspected value can thus be handled expeditiously.

It may be noted that a conventional 500-ml. flat-bottomed flask having a 24/40 std. taper joint was found suitable as a combustion chamber. Safety precautions were observed as in all closed-flask combustions.

Self-demountable Sample Carrier. The illustrated sample carrier is fashioned from a 0.01-in. thick platinum sheet (1¼ by ⅝ in.) having 144 holes (0.062-in. diam.) per sq. in. This sheet is bent to give a ⅛-in. clearance between the open sides. A carrier of platinum-rhodium is more rigid and withstands heat better than platinum, but breaks at the creases if the sides are flexed appreciably.

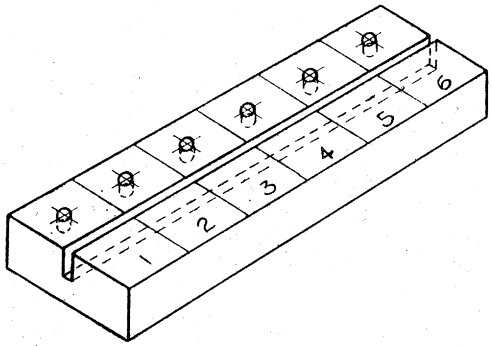


A hole is drilled into the end of a No. 24 flat-head stopper and provides an anchor for a length of platinum-rhodium (20%) wire (platinum wire is too soft and flexible). The wire is bent back on itself at one end, in order to anchor it when inserted to the stopper hole, thus obtaining a firm fitting without the need of an extraneous binding substance. The other end of the wire is bent at a right angle so that the exposed length is ¾ in.

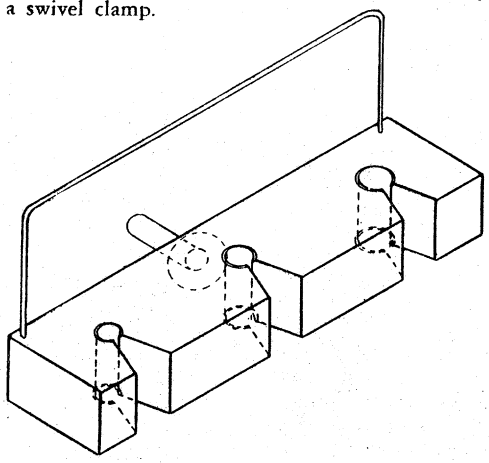
A piece of either 20-gauge platinum or platinum-rhodium wire is bent and connected to the sample carrier, as illustrated. This wire is attached to the carrier either by winding through the holes in the perforated sheet or by spot welding. In use, the sample carrier is disengaged by longitudinal rotation of the inclined flask with the glass stopper in place.

When not in use, the glass stopper, with or without the attached sample carrier, can be conveniently supported by two nails driven into a wood block at a slight angle about ¾ in. apart. Pieces of rubber tubing over the nails provide a non-slip cushion for holding the assembly at the groove in the glass stopper. Three such holders can be placed on a piece of wood 8 by 1.5 in.

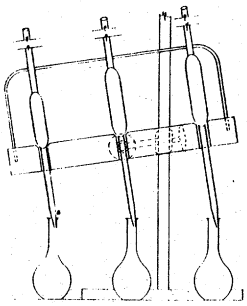
Sample Tray. The illustrated tray fabricated from a block of hard wood (6 by 2 by ¾ in.) can be used to transport a series of solid samples contained in filter paper or liquid samples in gelatin capsules. A strip of aluminum foil, cemented in the central trough (⅜-in. depth, ⅛-in. width) and extending about ¼ in. on either side, permits easy insertion and removal of the solid samples. Samples in gelatin capsules are placed in the holes (¼-in. diam., ¼-in. depth).



Pipet Drainer. The illustrated pipet drainer is composed of a pipet rack attached to a stand by a swivel clamp.



For the rack, three holes, 5/16-in., ⅜-in., and 7/16-in. diam., resp., are drilled and countersunk in a block of wood (2½ by 10 by 1½ in.) and a ¾-in. wedge opening is cut into each hole. A support shaft is positioned on the back of the block and a length of stiff wire is inserted into two holes in the top, as illustrated.



Inclination of the block permits pipets of different sizes (i.e., 2-5 ml., 5-25 ml., and 15-100 ml. pipets in the 3 respective holes) to drain into flasks that are equal in size. Rotation of the block makes it possible to drain pipets identical in capacity into receiving flasks of equal size.

References. (1) Weaver, E. A., *Analyst,* in press. (2) Weaver, E. A., Pollack, R. L., "Self-demountable Support," U. S. patent pending. (3) Weaver, E. A., "Pipet Drainer," U. S. patent pending.

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