A feed adapter for continuous automatic transfer of solutions into vacuum evaporation systems

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SUMMARY The design of a simply constructed feed adapter for continuous transportation of solutions into a vacuum evaporation system is described.

KEY WORDS feed adapter · continuous solution transfer · vacuum evaporation

SEVERAL CONTINUOUS FEED devices have been described for use with vacuum evaporators (1–8). Such devices[1] permit the automatic transfer of liquid into the evaporation system and continuous rather than batch type operation. Some of these have one or more of the following disadvantages: high cost, presence of grease on connections, or the necessity for frequent supervision. This report describes a feed adapter which has been designed and found useful for continuous transfer of solutions into a vacuum evaporation system and which eliminates or minimizes these disadvantages.

The adapter consists of two glass cylinders, one inside the other, glass intake tubing equipped with a stopcock attached to the outer cylinder, and fittings to hold the assembly together. The apparatus is illustrated in Fig. 1.

[1] Available from, for example, Matheson Scientific, Inc., P.O. Box 85R, East Rutherford, N.J.; Kopp Scientific, Inc., 401 Broadway, New York, N.Y.; and Buchler Instruments, Inc., 1327 16th St., Fort Lee, N.J.

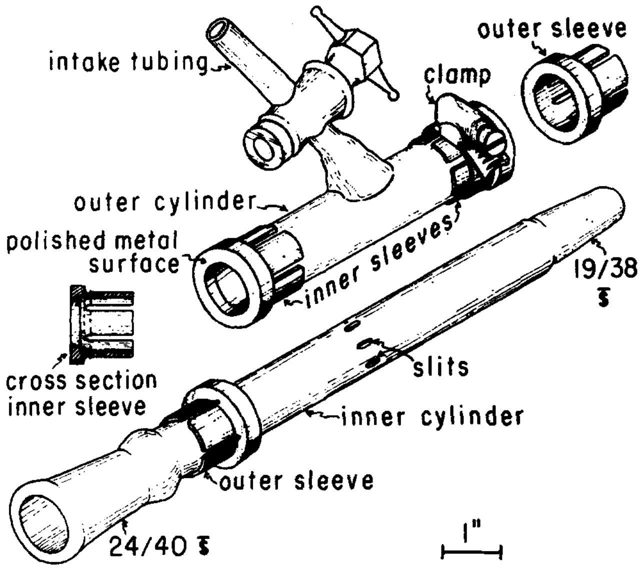

Fig. 1. Continuous feed adapter.

The cylinders are manufactured from precision bore pyrex tubing, and the inner cylinder is ground and polished to fit inside the outer cylinder with the narrowest possible tolerance that still permits rotation of the inner cylinder. The wall thickness of each tube is approximately $^{3}/_{32}$ inch. The inner cylinder is longer than the outer cylinder and contains a standard ground-glass joint at each end for attachment to a trap and a round bottom flask. These cylinders can be constructed with any convenient dimensions, but in the apparatus illustrated the i.d. of the outer cylinder is 0.7877 inch; the inner cylinder has a female 24/40 glass joint at the upper end and a male 19/38 joint at the lower end. The glass intake tubing which bears the stopcock is fastened to the upper surface of the outer cylinder at approximately a 45° angle. The intake port may be fitted with a convenient ball-and-socket joint for attachment of glass tubing from the solution reservoir. Several slits about 1 mm wide and 4 mm long are cut around the circumference of the inner cylinder below the attachment of the glass intake tubing (see below).

The cylinders are held in position by a pair of brass sleeves at each end. One brass sleeve of each pair is secured to the shoulder of the outer cylinder and the other sleeve is fastened to the inner cylinder so that one sleeve can revolve against the other. Metal clamps with a screw for adjustment are employed to secure the brass sleeves to the glass cylinders.

Solutions to be evaporated enter through the glass intake port and flow into the inner cylinder through the slits and then into the round bottom flask attached to the lower end of the adapter (Fig. 2). Solvent is evaporated by means of the vacuum system which in this case is employed in conjunction with a Rinco rotary evaporator. The Rinco evaporator turns the trap, the inner cylinder of the feed adapter, and the round bottom flask; only the outer cylinder remains stationary. The outer cylinder can be held stationary by means of a clamp or other similar arrangement which overcomes the torque induced by rotation of the inner cylinder. Capillary attraction of fluid between the polished glass surfaces of the two cylinders provides self-lubrication of the adapter, and no grease need be used. The solution flow rate is adjusted by means of the stopcock. We prefer the stopcock to be of Teflon, and such a stopcock containing a needle valve appears to be a satisfactory arrangement for making fine adjustments in flow rate. The solution reservoir may be located on the bench top below the adapter since the vacuum system can easily siphon liquid in any desired direction. Preliminary experience with the vacuum system and the solvent employed will establish a suitable flow rate so that solution intake and solvent evaporation will proceed at equal rates, and the intake stopcock need not be altered again. Evaporation may be hastened by the application of heat to the round bottom flask if desirable.

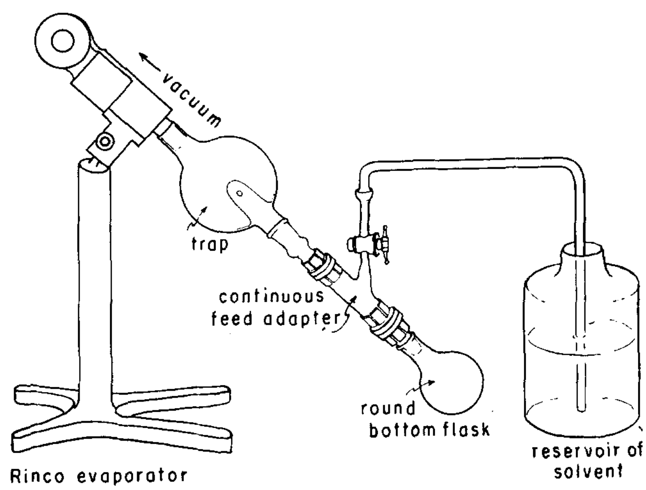

Fig. 2. Feed adapter and assembly for continuous solvent transfer and evaporation.

Considerable difficulty was experienced in designing sleeves suitable for prolonged use, but the present arrangement appears satisfactory. Each sleeve is circular and revolves against its pair on a $^{1}/_{4}$ inch polished surface which is coated with about 0.001 inch chromium. To induce flexibility where the clamp is employed, the sleeve wall is $^{1}/_{16}$ inch thick, is partly slotted and contains a groove. The glass shoulder of the outer cylinder rests against a metal ridge on the inside of the appropriate sleeve, and care should be taken that the inside diameter of the hole at the end of this sleeve is equal to or greater than the i.d. of the outer cylinder, or contact may occur between metal and the inner cylinder which rotates within this hole. The inner diameter of the two outer sleeves is less than the diameter of the inner sleeves because of the difference in o.d. of the cylinders. The sleeves are machined to fit over the cylinders with as close a tolerance as possible while remaining easily removable. The apparatus can be disassembled easily for cleaning.

Leakage of solvent from the opening between the glass surfaces of the two cylinders was negligible. The red dye Sudan III dissolved in chloroform has been used to test the system, and a faint red ring (0.5–1 mm) was observed on the polished surface of the outer sleeves when the adapter was run continuously for many hours. When certain highly volatile solvents are used, ice may accumulate on the outer surface of the adapter below the glass intake tubing. This may be prevented by permitting some intake of air into the vacuum system leading to the Rinco evaporator.

The feed adapter has been found to be capable of transferring 1300–1500 ml of chloroform per hour, with the vacuum system evaporating that amount when employed in conjunction with a vacuum pressure of 80–100 mm Hg and a water bath temperature of $<40°$ under the round bottom flask. If more rapid inflow and evaporation is required, it is recommended that larger diameter tubing be used for construction of the adapter since at high flow rates the upward passage of the vapor stream tends to interfere with the downward descent of liquid at the narrowest part of the adapter.

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