Constant-Level Device for Batchwise Distillation

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Batches of about 50 liters of hydrochloric acid are distilled routinely in our laboratory for the following purposes: purification of analytical grade acid to obtain a grade suitable for application in rubidium–strontium age determinations of rocks and minerals; and recovery of used hydrochloric acid to obtain a grade suitable for cleaning glass and Teflon ware.

A simple regulator has been designed to control the flow from the bulk container to a 10-liter Pyrex distillation flask keeping the level in the latter constant. The regulator (Figure 1) consists essentially of an air inlet, which is controlled by the level of the liquid in the distillation flask, and two siphons. No mechanical valve is included in the design. The siphons have one leg in common, but to understand the operation, it may be helpful to think of two separate siphons, one connecting the liquids in the bulk container and regulator, and the other connecting the liquids in the regulator and distillation flask. The level in the distillation flask is adjusted by positioning the lower end of the air inlet (*a*) at the desired level.

Under equilibrium conditions, the level of the liquid in the outer tube of the regulator (*R*) and that in the bulk container (*C*) are equal, whereas the level in the distillation flask (*D*) corresponds to the level of the lower end of the air inlet.

The air pressure in the container and the closed volume of the regulator is lower than atmospheric by an amount determined by the specific mass of the liquid column above the lower end of the air inlet tube. If the liquid level in the distillation flask sinks below the adjusted level, liquid is siphoned from the regulator. Consequently, the equilibrium at the lower end of the air inlet is broken, and air bubbles rise through the liquid in the regulator. The equilibrium of the lefthand siphon is also broken and liquid starts to flow through the siphon till the air inlet is closed again.

Constructional details of the regulator are given in Figure 2. A safety overflow (*c, d,* and *i*) is included in the design: if an air leak occurs, the bulk container will empty through the overflow, rather than flood the distillation flask (so far, our only experience with the safety overflow is by simulated failures).

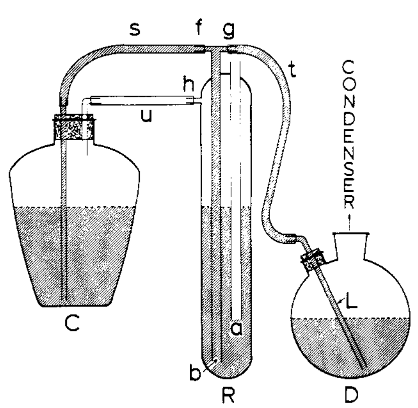

Figure 1. Principle of operation, showing connections between bulk storage container (*C*), regulator (*R*) and distillation flask (*D*)

(*a, b, f, g,* and *h*): refer to Figure 2; *L:* liquid inlet; (*s, t*): flexible PVC tubing, i.d. 6 mm, medical quality; (*u*): flexible PVC tubing, i.d. 8 mm

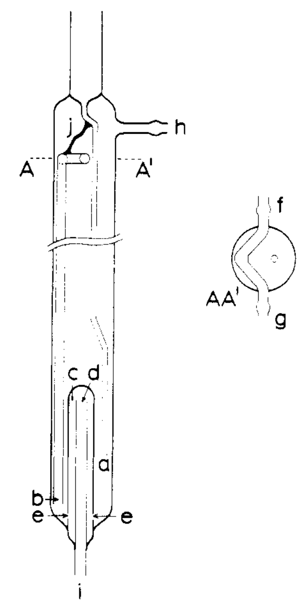

Figure 2. The regulator. Main tube i.d. 38 mm, overall length 80 cm

(*a*) Air inlet; o.d. 5 mm. (*b*) Common leg of twin siphon; o.d. 8 mm. (*c*) Cap of safety overflow; o.d. 18 mm. (*d*) Drainpipe of safety overflow; o.d. 10 mm. (*e*) 2–4 holes in cap; total cross section at least 50 mm^2. (*f*) Tubing connection to siphon leg in bulk storage container. (*g*) Tubing connection to liquid inlet in distillation flask.(*h*) Tubing connection to air volume of bulk storage container. (*i*) Tubing connection to safety drain-vessel. (*j*) Solid glass support. (*AA'*) Cross section showing branching of twin siphon

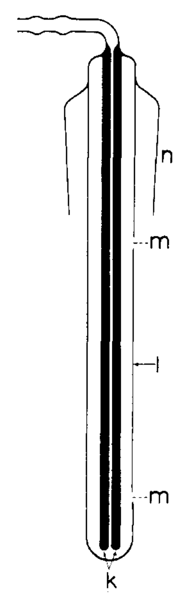

Figure 3. The liquid inlet

(*k*) Capillary tubing; i.d. 2.5 mm. (*l*) Protective sheath; o.d. 18 mm. (*m*) Holes in sheath. (*n*) Ground conical joint; cone 29/32

Non-professional glassblowers may prefer to construct the regulator and overflow as separate units. Another simplification would be to omit the common leg of the siphon and to attach the tubing connections (*f*) and (*g*) to the lower end of the outer tube (*e.g.*, at the height of *b*); the regulator will then function in the same way. The use of small "O" ring joints has been suggested to us, but such alternatives and their long-term testing are left to the interested reader.

We are aware of the fact that still simpler arrangements are possible; the air inlet tube could have been placed directly in the bulk container, and then a single syphon would have been sufficient. However, adjustment of the level would be less simple, and the risk of an air leak occurring, with subsequent flooding of the distillation flask, would be much greater.

The liquid inlet (Figure 3) in the distillation flask is protected by a glass sheath to prevent air and vapor bubbles from entering the siphon. Two holes in the sheath (*m*), one below and one above the liquid surface, allow free exchange of liquid and vapor with the distillation flask. The inlet tube (*k*) is a capillary to maintain a fairly rapid liquid stream during the distillation. Thus, air bubbles released during the warming up of the incoming liquid will be dragged along rather than accumulate in the siphon, while at the same time the warming up is diminished considerably.

The operation of the regulator is started as follows. Close off the drainpipe of the safety overflow with a stopper, disconnect the tubing at point (*h*) and blow to start the lefthand siphon. The siphon continues to flow even when the righthand siphon contains air: the PVC-to-glass transition at (*g*) prevents the air from entering the other siphon. Wait until the liquid levels in the regulator and container are equalized. Then close off the air inlet and the tubing connection (*h*) while blowing again in the tubing (*u*) to start the righthand siphon. Reconnect the tubing at (*h*). Wait until the liquid level inside the air inlet is well below the upper edge of the drainpipe (*d*) and remove the stopper from the drain.

The distillation may be left unattended overnight, provided that, in addition to the normal protection devices, provision is made to cut off the heater when the level in the distillation flask becomes too low (because of exhaustion or failure of the feed supply). We have a short platinum wire sealed into the outer wall of the regulator, at about 5 mm below the adjusted level, and a longer platinum wire sealed into the liquid inlet, which dips between the capillary tube and the protective sheath into the liquid. Thus, the liquid in regulator, siphon, and distillation flask is made part of an 8-V ac protection circuit.

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Simple, Vacuum-Operated Mixing Device

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Extensive use has been made in our laboratory of an ordinary vacuum-operated, windshield-wiper motor as a mixing device. By attaching a nickel-plated bronze flask holder with spring clamps to the oscillating arm of the motor as shown in Figure 1, the windshield-wiper motor has been used for gentle shaking or stirring of organic chemical reaction mixtures. Mixing of dilute solutions used for light scattering measurements has been achieved by using a lightweight cellholder, constructed of Duraluminum, with suitable slots to hold the cell tilted at a slight angle over the center of rotation attached to the oscillating arm. Thus, mixing was attained without wetting the stopper of the small (1 × 1 × 5 cm) cell used in the procedure, and without creating bubbles which can denature sensitive proteins. The device has also been useful for mixing solutions for absorption spectrophotometric measurements upon addition of small increments of reagents or after making dilutions in the cell.

The motor is mounted on a plate of Duraluminum which is attached to another plate as the supporting base. Less expensive supports would be as suitable. A piece of flexible tubing may be connected from the motor to either a vacuum line or water aspirator for operation of the mixing device. The device may be used in areas where electric outlets are not available or where the use of electric outlets is not suitable. Adjustment of the vacuum by the control knob allows a wide variation in mixing speed.

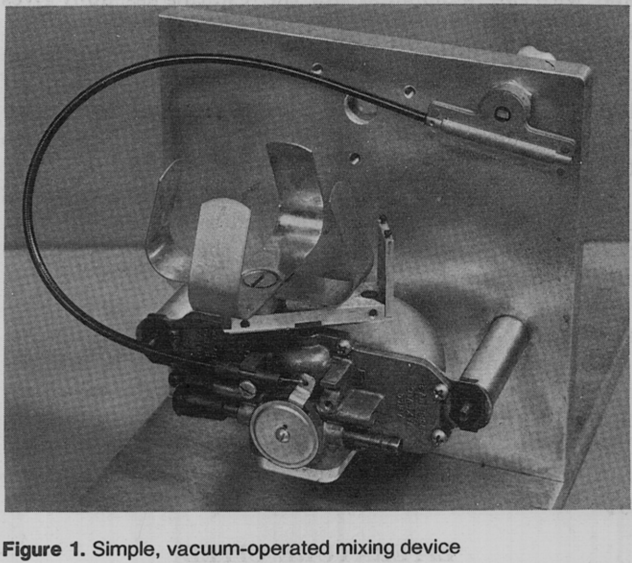

Figure 1. Simple, vacuum-operated mixing device

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