

Stirring Apparatus for the Investigation of Unstable Strongly Adsorbing Chemicals

By R. J. SAUCHUK, J. M. ANDERSON, and J. G. NAIRN

An apparatus is described which is useful for the evaluation of adsorption phenomena. The mixing apparatus provides the following features: an inert atmosphere to maintain maximum stability, a smooth, all-glass surface to eliminate spurious adsorption, a convenient method of analysis to permit maximum accuracy for rapid reactions, constant temperature, and controlled, measurable mixing speeds.

IN ORDER to provide an inert atmosphere for the adsorption studies of compounds subject to oxidation by air, a tightly-stoppered flask with a magnetic stirring device was employed. This method also provided a rapid, convenient method of analysis for kinetic studies. It was found that strong adsorption took place on all stirring bars covered with either Teflon or Tygon. Over short periods of time, a Pyrex sealed stirring bar was found to be satisfactory. However, prolonged use resulted in the formation of a ground-glass surface, both on the stirring bar and on the bottom of the reaction flask. This rough surface, which acted as an adsorption site, invalidated experimental results.

A floating, magnetic stirrer was designed to overcome this problem of spurious adsorption (Fig. 1). A Pyrex-covered magnetic bar $\frac{3}{8}$ in. \times $\frac{7}{8}$ in. was sealed to the bottom of a cylindrical float. The size of the float is dependent upon the following factors: the density of the solution, the height of the float above the magnetic stirrer, the speed of rotation, and the dimensions of the reaction flask. A mirror was enclosed within the float so that the speed of rotation could be determined by a photoelectric counting device. A hook was formed at the top of the float for ease of manipulation. The extent of agitation in the vessel can be controlled by varying the dimensions of the flat portion of the float.

To facilitate spectrophotometric analysis, a cell was sealed by means of a short extension onto the shoulder of a 250-ml. conical flask fitted with a standard taper $\frac{34}{45}$ stopper. The flask was placed in a constant-temperature bath, under which a magnetic stirrer was positioned. This closed system eliminated loss of volatile components and prevented exposure of the reaction mixture to atmospheric oxygen.

The speed of rotation was determined by allowing a beam of light from a 12-v. lamp to fall upon the mirror in the rotating float. The reflected light was allowed to sweep across a photoelectric cell, which by means of the electrical circuit (Fig. 2) activated an impulse counter. The magnetic

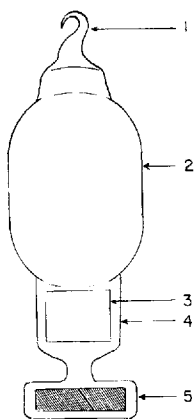


Fig. 1.—Floating magnetic stirrer. Key: 1, hook; 2, cylindrical body; 3, mirror; 4, flattened mirror casing; 5, Pyrex-covered magnetic bar.

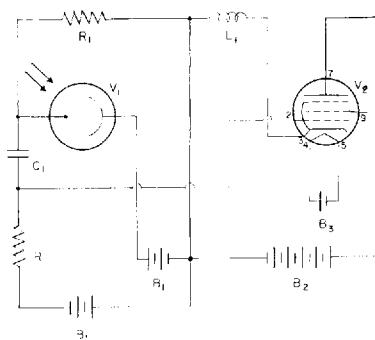


Fig. 2.—Electrical circuit for photoelectric counting device. Key: B₁, 45 v. (type B battery); B₂, 300 v. (power supply); B₃, 6 v. (filament supply); R₁, 22 megohms; C₁, 1 mf.; L₁, counter (Central Scientific Co.); V₁, photocell (Rogers 922 or equivalent); V₂, 6 CW5.

stirring motor was maintained at moderate temperatures by allowing cold water to pass through Tygon tubing encircling the housing. A more precise control of the mixing speed was obtained by supplying power to the magnetic stirrer through a variable transformer.

Satisfactory performance for counting and float stability up to 400 r.p.m. was obtained.

Received June 27, 1966, from the Faculty of Pharmacy and the Department of Physics, University of Toronto, Toronto, Ontario, Canada.

Accepted for publication August 15, 1966.

The authors are grateful to Mr. G. L. Bragg, who constructed the photoelectric counting device.

This investigation was supported in part by the Canadian Foundation for the Advancement of Pharmacy and the National Research Council of Canada.