corresponding to a minimum detectable concentration of 0.3 p.p.m., but the poor loading characteristics of such a detector did not allow such large samples as before. In practice the minimum concentration which could be measured, with a standard deviation of about 10 %, ranges from 0.5 to 0.7 p.p.m.

Several difficulties must be taken into account when using the electron capture (EC) detector. As the EC detector is specific for certain types of compounds and does not have the same response to water and to hydrocarbons, it is necessary to prepare a plot of signal amplitude *vs.* water quantity, using known amounts of standards. The difficulty of obtaining samples with known low water contents can be overcome by analyzing a series of samples on the EC detector and on the thermal conductivity (TC) detector, and extrapolating the plot obtained from the TC detector sensitivity range to the range of the EC detector to zero concentration. This procedure is justified by the fact that in this range the detectors' response is linear.

Obviously the EC detector cannot be used when the sample contains excessively large amounts of electron-absorbing compounds (halogens, phosphorus and sulphur compounds, etc.) unless these compounds are removed with scrubber columns.

Furthermore, in order to avoid the temperature limit (225°) of the tritium detector, one could use a nickel EC detector which has a higher temperature limit. But the temperature range extension will not be large since the Porapak itself does not tolerate, at present, temperatures higher than 250°.

Other experiments are in progress in our laboratory to prepare stationary phases of new composition for other and better separations, and to improve the sensitivity of the method described.

Istituto di Chimica Industriale, Università di Genova, GIANRICO CASTELLO
Centro Nazionale di Chimica delle Macromolecole, STELIO MUNARI
Sezione V, Genova (Italy)

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Received May 22nd, 1967

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A simple microcell for photometric monitoring of flowing materials

A variety of cuvettes for the continuous photometric monitoring of flowing liquids are commercially available. But, none combines low cost with small sample size and a light path longer than 1 cm. A flow cuvette that combines these features is highly desirable. It could aid in such tasks as monitoring chromatographic effluents and measuring the optical densities of a series of small samples, especially if weakly absorbing solutions were involved.

In considering the desirable features such a cell should possess, we were led to

the same conclusions regarding design and choice of materials as those given by KIRK *et al.*[1] in their description of a cuvette for microanalysis. Their cuvette was not intended for flow-through service, but it differs in essentials from ours only as the intended functions of the sample access ports; consequently it lacks provision for automatic air-bubble elimination. Both cuvettes consist of a bored teflon* rod equipped with end windows (glass or optical quartz) and threaded teflon end pieces. Designed by KIRK *et al.*, this microcuvette is now commercially available**. It is 5 cm long, with a bore of either 2 mm or 4 mm.

We have converted a commercial Kirk cuvette to a flow-through cell (Figs. 1–3) by notching the inner wall at the exit and entrance holes to guide air bubbles

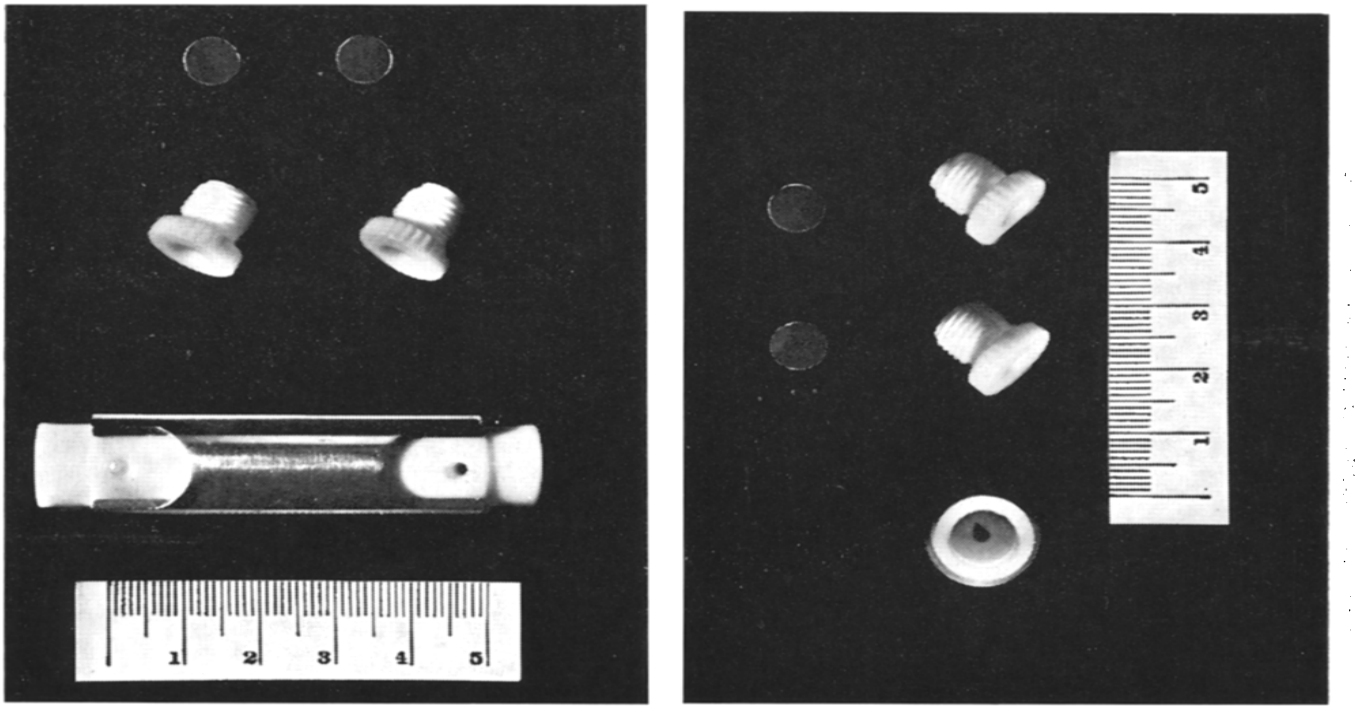

Fig. 1. Teflon-bodied flow cell. Modified commercial Kirk microcuvette with 5 cm long by 2 mm diameter cell volume. (a) Topview; (b) end view.

through the cell. Samples are introduced and removed through the teflon tubing. The AWG size No. 22 tubing may be held with tubing adapters***, which are tightly clamped to the inlet and outlet holes. In some analyses, the metal in the adapters interferes. Then the tubing can be press-fitted into the holes and held in place with black electrician's tape. AWG No. 16 tubing works well for this. Suitably supported this cell can be used in a recording spectrophotometer or, with a commercial cell holder, in a Beckman DU spectrophotometer equipped with an energy recording attachment.

Besides the obvious application of monitoring chromatographic columns, such a cell can be used to speed up microchemical photometric measurements (Fig. 4). We monitored the contents of the cell at 505 mμ with a Cary Model 11M spectrophoto-

* DuPont brand of polytetrafluoroethylene.
** Microchemical Specialities Co., Berkeley, Calif., U.S.A.
*** Beckman Instruments, Inc., Spinco Division, Palo Alto, Calif., U.S.A.

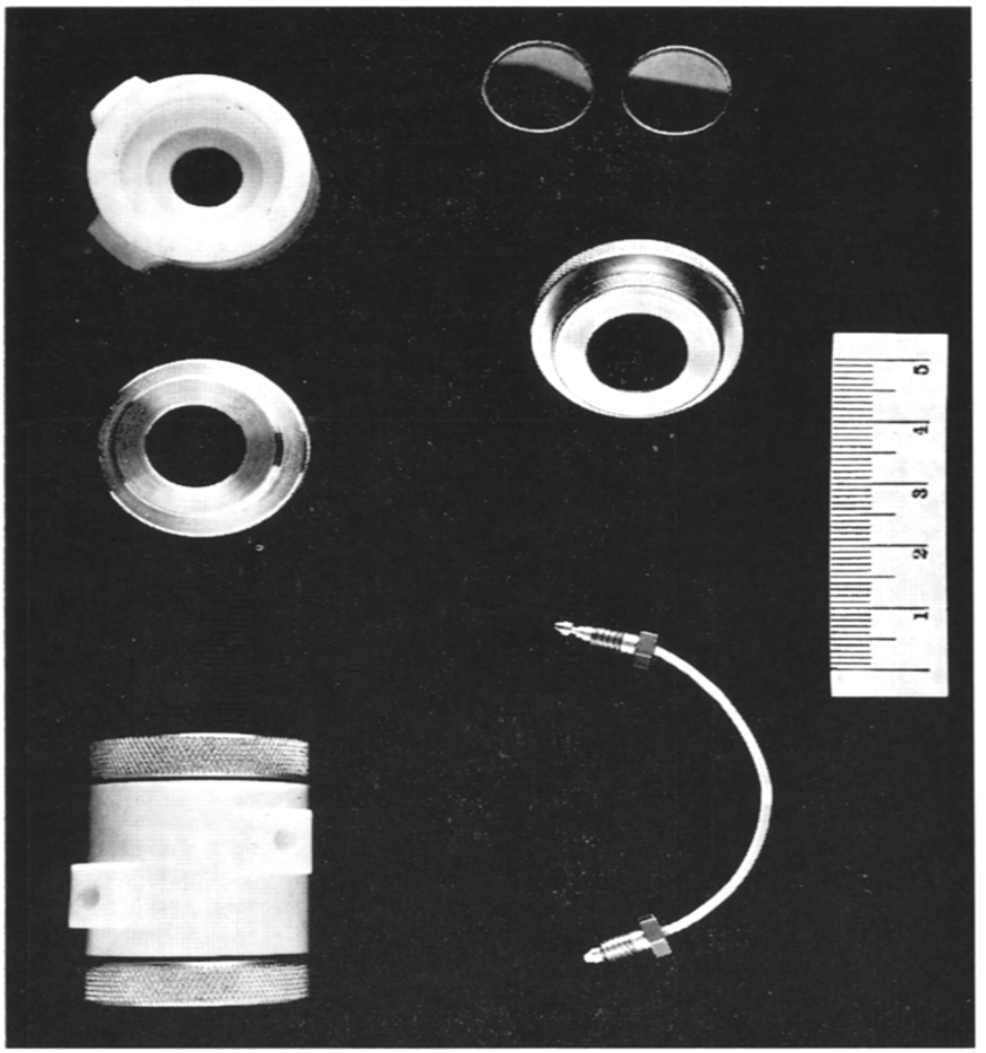

Fig. 2. Macrocuvette for polymer solutions.

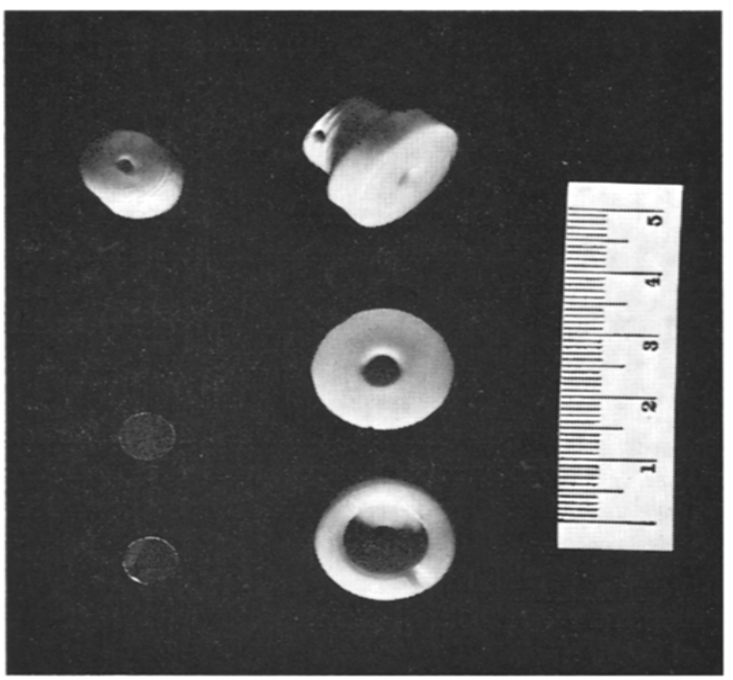

Fig. 3. Microcuvette with 1 cm long by 2 mm diameter cell volume.

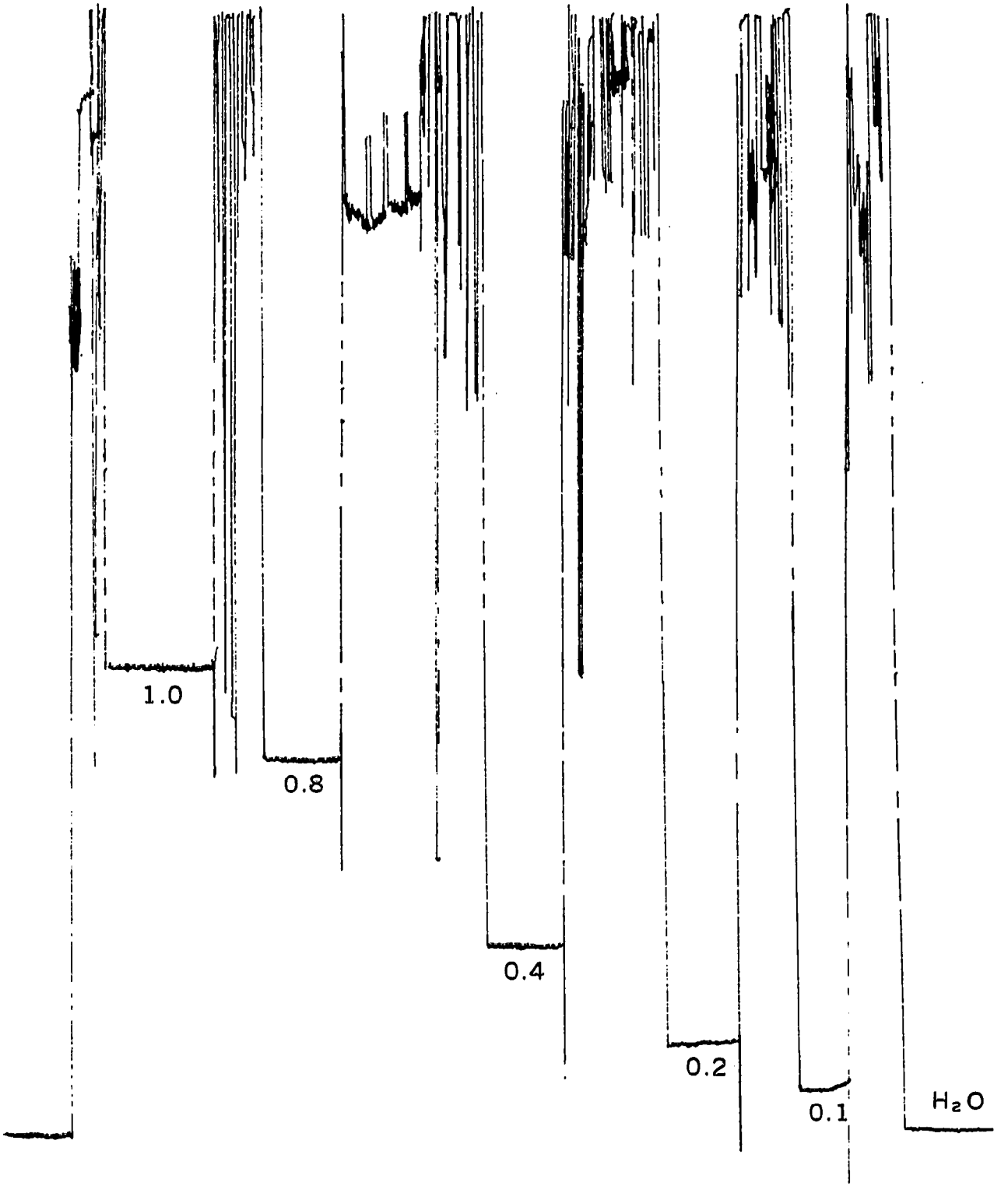

Fig. 4. Performance of 5 cm microflow cell with solutions of ferrous phenanthroline. Numbers refer to concentration of electrolytic iron in μg/ml. Ordinate is optical density at 508 mμ recorded by a Cary 11 M spectrophotometer.

meter while passing a series of aqueous solutions containing 0.1 μg/ml to 1 μg/ml of iron as the orange phenanthroline complex through the cell at a rate of about 1 ml per minute. To obtain uniform gentle suction, we coupled the outlet tube to a flask of water fitted with a 2-ft. vertical siphon outlet. Efficient rinsing of the cell between samples was insured by interjecting at least two air bubbles into the flow of each new sample. The presence of air bubbles moving through the cell interrupted the plotting of the samples' optical densities with regions of irregular traces. An occasional reluctant air bubble was removed by running a sample of methanol through the cell.

This flow cell system can satisfactorily record optical densities as small as 0.05 of samples as small as 1 ml. The actual minimum measurable quantity of material depends on its molar extinction coefficient. Using the ferrous phenanthroline complex, iron is measurable in concentrations in the range of 0.1 μg per ml.

Such a cell thus permits continuous measurement of optical densities of smaller volumes of more weakly absorbing solutions than is possible with other cells now commercially available or described in the literature[2-4].

We have constructed several flow cuvettes based on the same principles for other applications. The large cuvette shown in Fig. 2 was constructed to aid in monitoring solutions of cellulose carbanilate. Use of two cuvettes of different light path, in tandem, allows a wider range of concentrations to be monitored. The cuvettes can be completely dismantled for cleaning out viscous polymers.

The small cuvette in Fig. 3 has a 1 cm long by 2 mm diameter light path. Since it can also accept and eliminate air bubbles, the cuvette can be used to analyze a series of samples in microchemical analyses. In each cell, the windows are recessed. This feature protects them against breakage and scratches. They can be replaced easily and cheaply if damaged.

Mention of trade names and commercial enterprises or products is solely for necessary information. No endorsement is implied.

The authors wish to acknowledge the help of the Scientific Photographic Laboratory, University of California, Berkeley, Calif., in preparing the photographs, and of the Instrument Shops, U.S. Naval Radiological Defense Laboratory, San Francisco, Calif., in preparing the macro-flow cell.

Pacific Southwest Forest and Range Experiment Station, Forest Service, U.S. Department of Agriculture, Berkeley, Calif. 94701 (U.S.A.)

FRANK J. KILZER

United Research Service, Burlingame, Calif. 94011 (U.S.A.)

STANLEY B. MARTIN

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Received April 18th, 1967