A sensitive probe for the Photovolt densitometer

Polycyclic hydrocarbons, separated into distinct spots on paper chromatograms, can be measured quantitatively by the visible fluorescence emitted under ultra-violet light. For this purpose we were using a Photovolt Densitometer type 501A, with ultra-violet source, and ultra-violet sensitive phototube. We considered that the sensitivity obtained with this arrangement was insufficient. Sensitivity could presumably have been improved by the purchase of a rather expensive photomultiplier photometer. Some preliminary experiments led us to believe that the requisite sensitivity could be obtained rather simply, and very inexpensively, using cadmium sulphide single crystal cells.

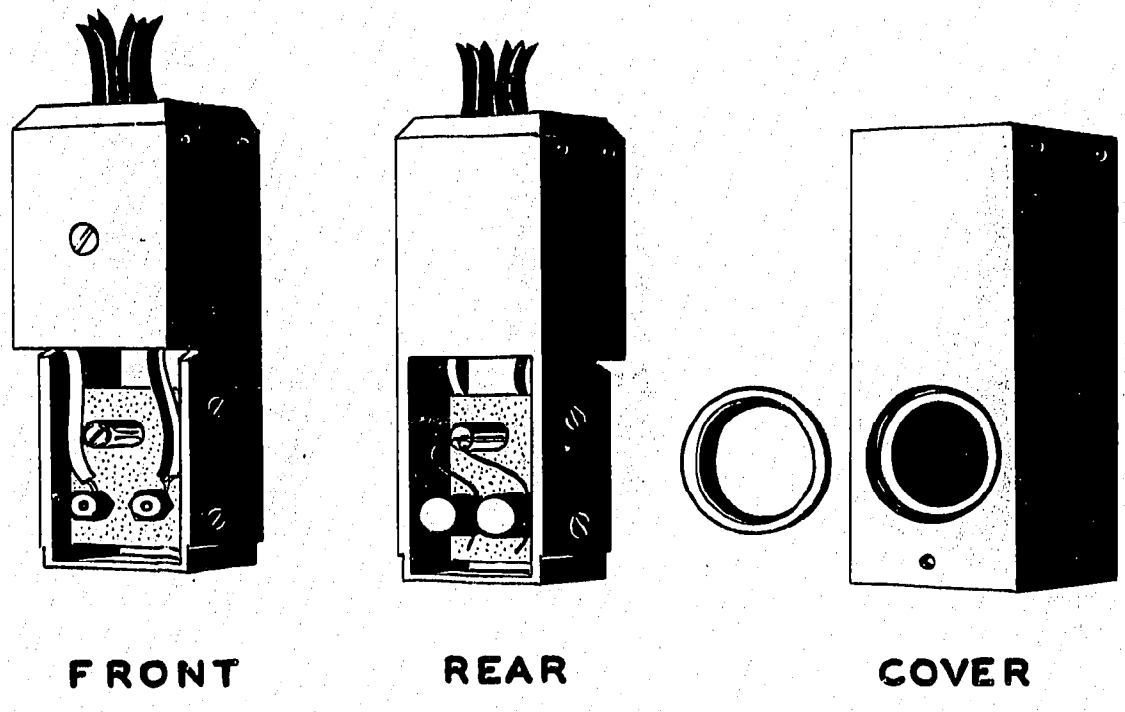

Fig. 1.

These preliminary experiments indicated that such a photocell (CL-2 of Clairex Computer N.Y.C., $ 3.50) showed an increase of about one hundred times for visible light over the original phototube. Accordingly, an adapter to hold this photocell was built for use with the Photovolt densitometer. The original probe was disassembled, and the phototube removed. By brazing aluminum angle stock together, an insert box was constructed fitted with covers on both ends, and cut out so that it would slip into the original cover without interfering with the filter holder ring. Inside the insert box the crystal cell was supported in a tight fitting hole drilled in a piece of plexiglass which also carried two binding posts. The photocell and the original cable were connected to the binding posts, the insert box slipped into the original rectangular housing, and the probe was ready to use (see Fig. 1).

Since the dark resistance of the crystal cell is much lower (1000 MΩ for a CL-2) it was necessary to adjust the coarse zero control, accessible on removing the top of the Photovolt photometer 501A.

In order to test the new probe a number of chromatograms of polycyclic hydrocarbons were run on a Photovolt type 520 densitometer, equipped with an ultra-violet source. The readings were recorded on a 10 mV Brown recorder. The same

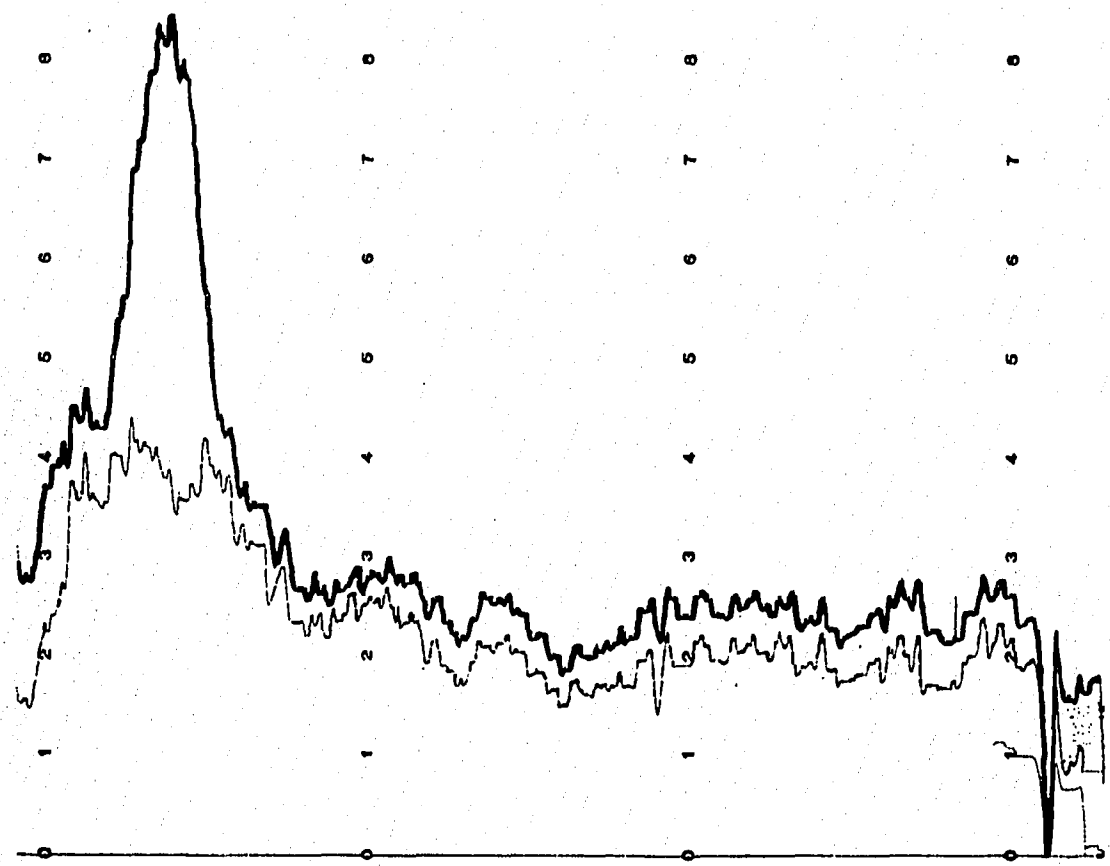

Fig. 2.

chromatogram was run using the Photovolt ultra-violet probe type B. Comparative tracings are shown in Fig. 2, for a chromatogram of 5 μg of pyrene. Microgram, and fractional microgram amounts also gave adequate response.

The Clairex cell gave sufficient sensitivity in range 1, whereas the phototube required the photometer range switch to be set at 2 or 3. By using the recommended ultra-violet filter No. 445 on the probe, the Clairex cell gave twice the sensitivity of the original phototube, although the CL-2 has a spectral peak at 520 mμ. Furthermore, we experienced less interference from line voltage fluctuations, when using the crystal photocell, due to the slower response time.

A further application was to use the new crystal probe to measure the light intensity on the screen of the Philips EM100 Electron Microscope. Here again the CL-2 showed its superiority over the ordinary phototube by a factor of approximately

200 on the meter reading. The phototube probe was practically useless at low intensities, whereas the crystal cell gave adequate readings well below the minimum intensity needed for focussing.

In our opinion, the sensitivity of a large number of photometric devices can be improved by substituting crystal photocells in place of phototubes provided:

(a) the lower internal resistance of the crystal cell can be accommodated;

(b) the longer response and decay time is not objectionable;

(c) the comparatively high temperature coefficient can be compensated. The manufacturer's value of temperature drift for the CL-2 is 0.1% per 1°. This is negligible for the average laboratory in temperate climates.

Another significant advantage is the maximum operating voltage of 300 V DC as compared to 90 V for most phototubes. The extremely small size of the crystal photocell is a distinct advantage in probe design. Since these cells are so inexpensive we believe that they will find increasing application in photometry.

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Improved resolution on paper chromatograms

In paper chromatography the original solution is commonly allowed to spread over a small circular area. This results, after development, in the spots corresponding to the different substances being irregular circles. Two substances with close R_F values cannot easily be distinguished since overlapping spots appear only as a rather more irregular circle. On one-dimensional chromatograms this is sometimes overcome by applying the original solution as a long streak, but this introduces difficulties and cannot be used for two-dimensional chromatograms. The method described here is applicable to one- or two-dimensional chromatograms and enables substances with very close R_F values to be distinguished.

The solution for analysis (10 μl) is applied to the paper and spreads as a small circular spot. This is dried and the operation repeated as many times as is needed to concentrate the material. The solvent (*e.g.* water if the original solution is aqueous) is applied in three 10 μl portions to the centre of the dried spot, drying between applications. As the solvent spreads out it carries the solutes to the periphery of the original spot, forming a ring with an empty centre. On running the chromatogram as usual, each substance runs at its characteristic rate forming a discrete spot which maintains its ring shape, though there is some spread and the ring changes to a solid circle at R_F values greater than about 0.4. It is very much easier to detect overlapping rings than overlapping circles, particularly if the centres nearly coincide, and one can be