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Note

An electron capture detector cell

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The construction of an electron capture detector (ECD) cell designed for use in a Varian 1860 gas chromatograph instead of the original ECD normally used with this equipment is described. It is simple and rapid to replace the original with the new cell. A high-temperature liquid thermostat was also designed in order to improve the thermal stability of the new detector system.

The need for a new cell arose because we are concerned with problems that demand accurate measurements of physico-chemical quantities; we are especially interested in measurements of the temperature dependence of electron capture processes^{1,2}. For such purposes, the new cell was designed so as to obtain the following desirable properties: the pulse sampling method of the ECD should be the normal mode of operation and the new cell must allow this mode of operation³; the cell must be constructed in such a way that it can be easily and efficiently cleaned; the temperature of the detector system should be constant in the range $\pm 0.02^\circ$; the cell should be as rigid and compact as possible so as to be suitable for use in long, heavy-duty work; as we had a Varian 1860 instrument at our disposal, we wished to construct a cell suitable for use with this apparatus, making the whole measurement easy and rapid; the cell should be easily removed and exchanged for other commercial detectors if necessary.

In order to meet these requirements, we constructed the cell of PTFE because of its strength, thermal stability, good electrical insulation and high heat capacity. The electrodes are made of stainless steel; one is connected to the pulse generator and the other to the electrometer amplifier of the chromatograph. The connections are made with coaxial cables. The structure of the cell is shown schematically in Figs. 1 and 2. It consists of five parts that are screwed together with stainless-steel screws. The upper part of the detector cell reaches into the thermostat vessel and the lower part is connected to the column outlet*.

The thermostat vessel consists of two parts: the inner part with a screw-on cover is made of stainless steel and the outer part is made of aluminium sheet. The space between the two parts is filled with glass-wool for thermal insulation. The thermostating liquid, circulating through the vessel and controlled by a conventional Höppler thermostat, is glycerol.

* Detailed plans of the detector cell can be obtained from the authors.

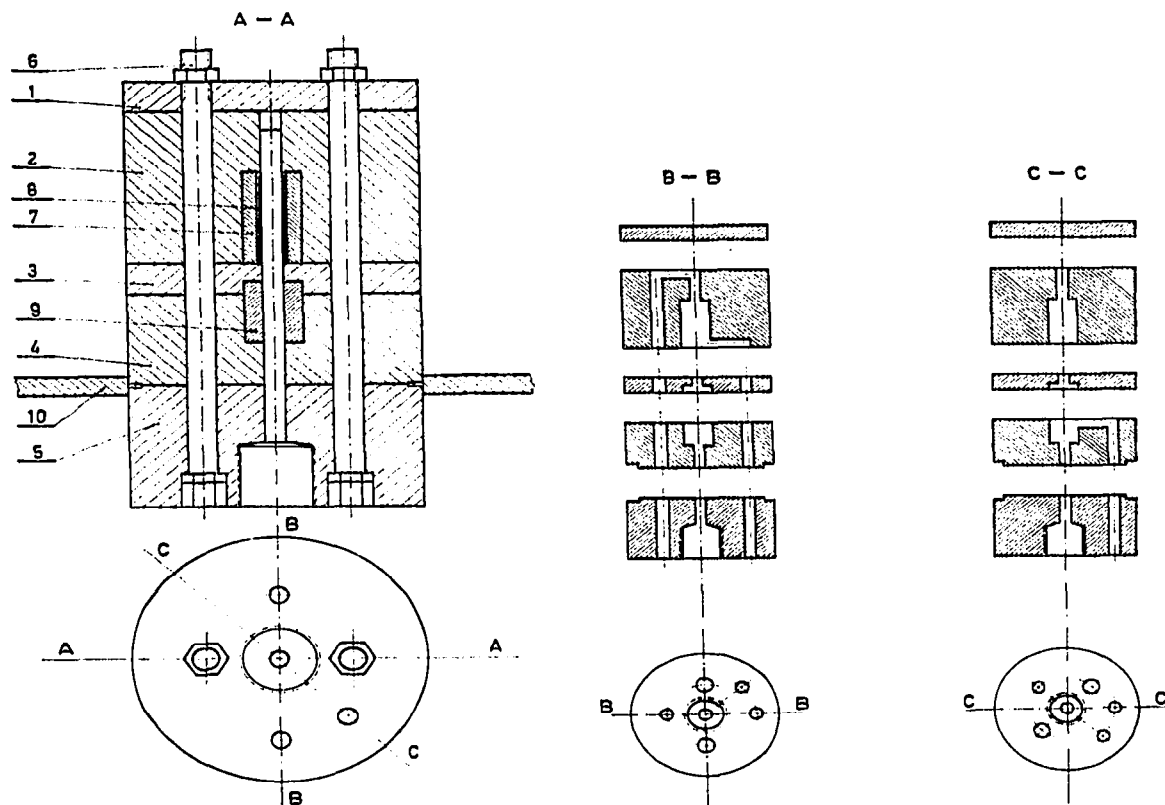


Fig. 1. Cross-section of the cell (A-A), and bottom view. 1, Cover; 2, cathode housing; 3, insulation between the electrodes; 4, anode housing; 5, connector between the detector and column outlet; 6, stainless-steel screw; 7, cathode cylinder; 8, radioactive foil; 9, anode cylinder; 10, bottom of thermostat vessel.

Fig. 2. Enlarged cross-sections of the cell (B-B and C-C), and bottom views.

The pulse generator, specially constructed in this institute, generates a square waveform with widely variable characteristics. The duration of the pulses varies between 7 and 500 μsec , the time between pulses varies between 20 μsec and 20 msec, and the amplitude is continuously variable between 0 and 100 V.

We compared the performances of the original detector cell with those of the new cell. If one uses the same tritium foil, the standing current in the two cells is identical in both the direct current and pulsed methods. This is not surprising, as the geometries of the electrodes and of the inter-electrode space are virtually identical in both instances. We tested the magnitude of the signal for both cells with a dilute solution (1 ppm) of carbon tetrachloride in hexane under the same conditions and found the same results in both instances. The advantage of the new cell lies in its robustness and thermal stability. It can serve reliably for long periods even if exposed to rough handling.

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