

## A modified design of a high temperature argon beta-ray detector for gas chromatography

In 1958, LOVELOCK<sup>1,2</sup> described an ultra sensitive detector for gas chromatographic work, whose operation is based on the unique ionisation properties of argon gas. Using this detector, which is relatively insensitive to changes in temperature, pressure and gas flow, as little as  $10^{-12}$  moles of most organic substances can be detected.

A high temperature is desirable for optimum sensitivity of the argon detector and is also essential to prevent condensation in gas chromatography of high boiling-point compounds. Although argon  $\beta$ -ray detectors are now used extensively, very little work has so far been done using this highly sensitive detector at temperatures above  $300^{\circ}$ .

There are two main difficulties in the use of these detectors at high temperatures: (i) the insulation of very high voltages at high temperatures, and (ii) the choice of suitable connecting tubes between the column and the detector body.

TERANISHI *et al.*<sup>3</sup> used silicone gaskets and glass tubing to connect the column to the detector and silicone rubber as an insulator. This limits the range to below  $240^{\circ}$ . UPHAM *et al.*<sup>4</sup> connected the column and detector directly with teflon sleeves. GUDZINOWICZ<sup>5</sup> and his coworkers used sapphire as an insulator at high temperatures and in a recent paper<sup>6</sup> they have replaced the usual silicone rubber or teflon connecting tubes between column and detector with glass to metal ball and socket connections. But even then the detector does not function properly at very high temperatures, *e.g.*

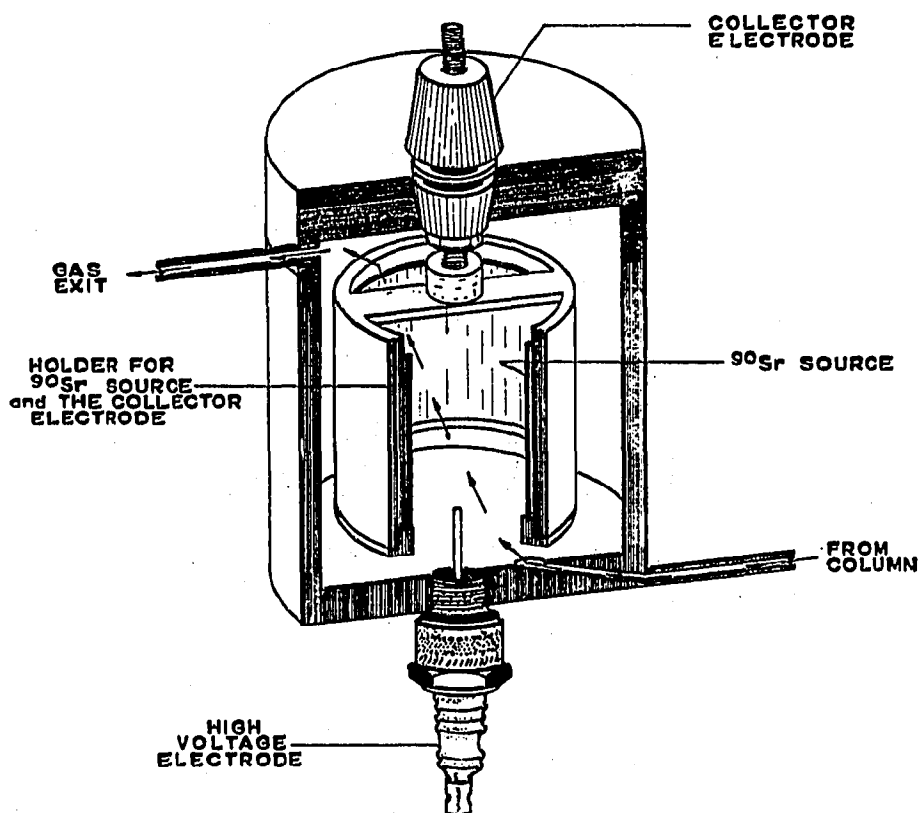


Fig. 1.  $\beta$ -Ray ionisation detector.

500°, when expansion of the metal to glass connection and electrical leakage through glass become serious problems.

To avoid these difficulties we have modified the design so that neither the high voltage nor the low voltage points are connected to the body of the detector. In the modified design the metal column is directly connected to the metal detector body using a swagelok coupler\* without any insulating material in between. The high voltage is applied through an extended spark plug probe and the collector electrode is isolated from the body of the detector by a porcelain insulated "Anticorona" plug. A gold coated <sup>90</sup>Sr source\*\* (10 mC) is used in order to prevent any diffusion of active <sup>90</sup>Sr at high temperatures. The details of the design are given in Fig. 1.

The detector has been working quite satisfactorily for about a year. It has been used at temperatures up to 500° in conjunction with our high temperature gas chromatographic unit<sup>7</sup> and it is felt that it can be worked at still higher temperatures.

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<sup>1</sup> J. E. LOVELOCK, *J. Chromatog.*, 1 (1958) 35.

<sup>2</sup> J. E. LOVELOCK, *Nature*, 182 (1958) 1663.

<sup>3</sup> R. TERANISHI, C. C. NIMMO AND J. CORSE, *Anal. Chem.*, 32 (1960) 896.

<sup>4</sup> F. T. UPHAM, F. T. LINDGREN AND A. V. NICHOLS, *Anal. Chem.*, 33 (1961) 845.

<sup>5</sup> B. J. GUDZINOWICZ AND W. R. SMITH, *Anal. Chem.*, 32 (1960) 1767.

<sup>6</sup> B. J. GUDZINOWICZ AND W. R. SMITH, *Anal. Chem.*, 33 (1961) 1135.

<sup>7</sup> R. M. IYER AND J. P. MITTAL, To be presented at the Golden Jubilee Session of the Indian Science Congress Association, 1963.

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\* A swagelok metal-metal union NO (400-6) supplied by Crawford Fitting Co., 884 East 140th Street, Cleveland, 10, Ohio, U.S.A.

\*\* Supplied by Radiochemical Centre, Amersham, U.K.

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## Chromatographie préparative sur plaques.

**Confection d'un mélange (gel de silice/plâtre) permettant un étalement homogène de couches épaisses et leur séchage sans craquelures.**

**Application au moyen d'un nouvel appareil**

La confection des couches épaisses de gel de silice pour la chromatographie préparative pose quelques problèmes. En effet, les mélanges de gel de silice et de plâtre commercialisés à l'heure actuelle sous le nom de Gel de silice G (selon STAHL) renferment en général 10 ou 13 % de sulfate de calcium (plâtre minute). Si l'on effectue la suspension de ces mélanges dans un volume d'eau double du poids d'adsorbant, la pâte ainsi obtenue durcit trop rapidement pour être étalée de façon homogène.

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