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A SIMPLIFIED DESIGN FOR AN ELECTRON CAPTURE DETECTOR

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SUMMARY

A simple, rugged electron capture detector based on a concentric tube design has been developed. The new detector functions as well as a current detector based on Kovar–glass insulations but has advantages in terms of ruggedness and ease and inexpensive repair.

INTRODUCTION

Electron capture detectors are noted for their extreme sensitivity, partial specificity and ease of contamination. Contamination can be a severe problem when the detector is used under less than optimal conditions[1]. Plating of the electron source by column effluent is a problem particularly when ^{3}H is used as a source due to the rather low operating temperature of the usual titanium tritide foils. Often this factor limits column operating temperature to less than optimum values.

Two means of partially overcoming plating problems have been used. The first is the use of high-temperature radioactive foils, such as ^{63}Ni or most recently $Sc^{3}H$, as the electron source. Detectors using these foils can be operated at much higher temperatures and column effluents are less likely to plate out and more easily revolatilized off of the foils. These detectors still have a problem with material plating out onto the foils which cannot be removed thermally. The second means of overcoming plating problems is through a detector design that enables easy foil maintenance to be carried out. The Varian concentric tube electron capture detector (U.S. patent 3,277,296) is an excellent example and coupled with the cleaning method of Holden and Wheatley[1], a foil may be removed, decontaminated and back in service in a matter of hours. The basic detector design is based on a Kovar–glass–Kovar–glass–Kovar function where the glass serves as an insulator. The relatively thin-walled glass insulator of the Kovar detector is quite fragile and, once broken, is generally beyond repair** and quite costly to replace. We wish to report on a modified concentric tube detector (modular cell) that, while having the advantages mentioned above, is rugged and easy to build or repair.

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** Two professional glass blowers, when requested to repair a broken Kovar detector, were not successful.

CONSTRUCTION OF THE DETECTOR CELL AND METHODOLOGY

The basic design features of the modular cell are shown in Fig. 1. The stainless-steel vent cap (I) the stainless-steel cathode (II) and the stainless-steel anode wafer (V) are easily machined out of stainless-steel stock. The anode tubing in this instance is made out of 17-gauge, stainless-steel syringe needle tubing (obtained from a 17-gauge hypodermic needle) and is friction-fitted into the hole in the stainless-steel anode wafer. The actual gauge size is relatively unimportant but must snugly fit the glass capillary tube section (IV and VI). With the large number of sizes of hypodermic needles available, the selection of glass capillary stock to give a snug fit is readily accomplished. The cathode–anode Pyrex insulator (IV) is made of stock Pyrex capillary tubing. A glass saw is used to cut the ends of the Pyrex tubing off square and the small depression in the insulator is made by grinding with a

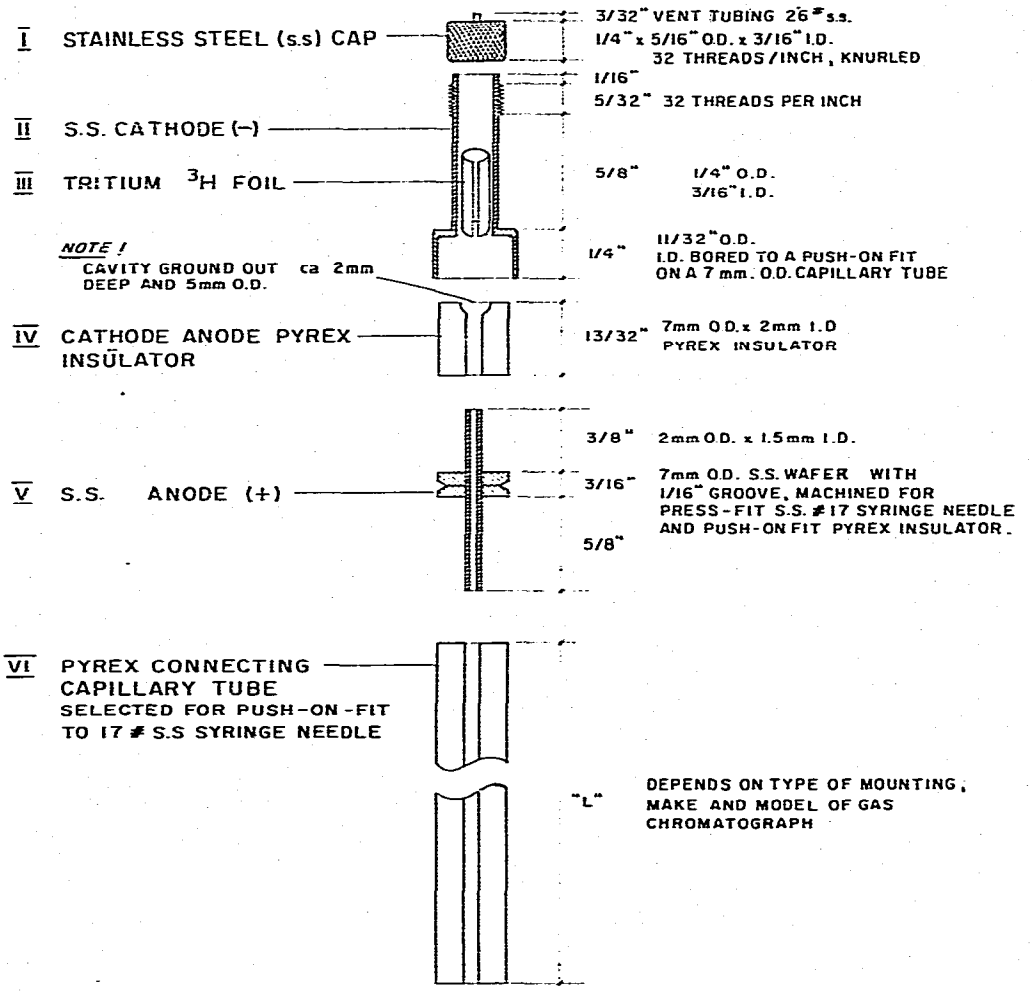

Fig. 1. Exploded view showing design features of individual components of the modular cell electron capture detector.

rounded Pyrex rod and carborundum powder. The detector uses a Varian 250-mCi Ti^3H_2 foil and, when assembled, is stacked onto a vertical glass capillary column effluent tube analogous to that described by Uthe *et al.*[2]. In this study the detector was mounted on a Microtek MT 220 Gas Chromatograph fitted with a 6 ft. × 0.25 in. I.D. glass column packed with 3% OV-1 on Chromosorb W (80–100 mesh) using nitrogen as a carrier gas at a flow-rate of 90 ml/min. The operating temperatures were 230°, 180° and 245° for the injection ports, column and detector block, respectively. All tests of detector operation were carried out using hexane solutions of lindane. In all cases comparison between the Kovar cell and the modular cell were carried out using the same column and same radioactive foil. Following the completion of a test, the foil was transferred back to the original detector and its response again determined to ensure stability of all operating parameters especially with regard to foil contamination.

The detector castle was a standard Varian design and Varian clip-on signal cables were used. The castle was mounted on a heating block turned out of aluminum stock. The heater block and the castle were insulated by asbestos paper overlaid with aluminum foil. The thermocouple and the heater cartridge were mounted on opposite sides of the heater block. The detector foil temperature was calibrated against the heater block temperature by removing the ^3H foil and placing a thermocouple inside the cell. For this particular design a heater block temperature of 245° gave a foil operating temperature of 214°.

RESULTS AND DISCUSSION

The linearity of response and the sensitivity of both the modular cell and the Kovar cell are shown in Fig. 2. The modular cell was slightly more sensitive than the Kovar cell, but both responses were within the same order of magnitude. No significant differences in the linearity of response were noticed between the two cells. There was no noticeable difference in noise when the detectors were operated at maximum sensitivity (Fig. 3). No evidence for oxygen contamination of the detector used in this study was found because the standing current equalled that of the sealed Kovar cell at operating voltages. This was rather surprising for it had been assumed that small leaks would be present in a friction-fit arrangement as used in the modular cell and would lead to increased noise over that present in a Swaged system such as the Kovar cell. The small upward baseline drift in the modular cell tracing is due to operation of the detector only a short time after placement onto the column.

The effect of varying the collection (d.c.) voltage on detector response is shown in Fig. 4. Again little difference was found between the two cells, both of them showing maximal response around 5 V and very similar response profiles. Standing current *vs.* voltage profiles are shown in Fig. 5. The Kovar cell is appreciably better at lower voltages, but at maximum current no great difference exists between the cells and at operational voltages (90 V) the standing currents of both cells are essentially the same. The small increase in current in the modular cell at 90 V d.c. as compared to the Kovar cell is probably due to the closer promixity of the anode to the cathode in the modular cell. This also indicates that no problem of current suppression due to oxygen level diffusion[3] through the friction-fit fittings occurred.

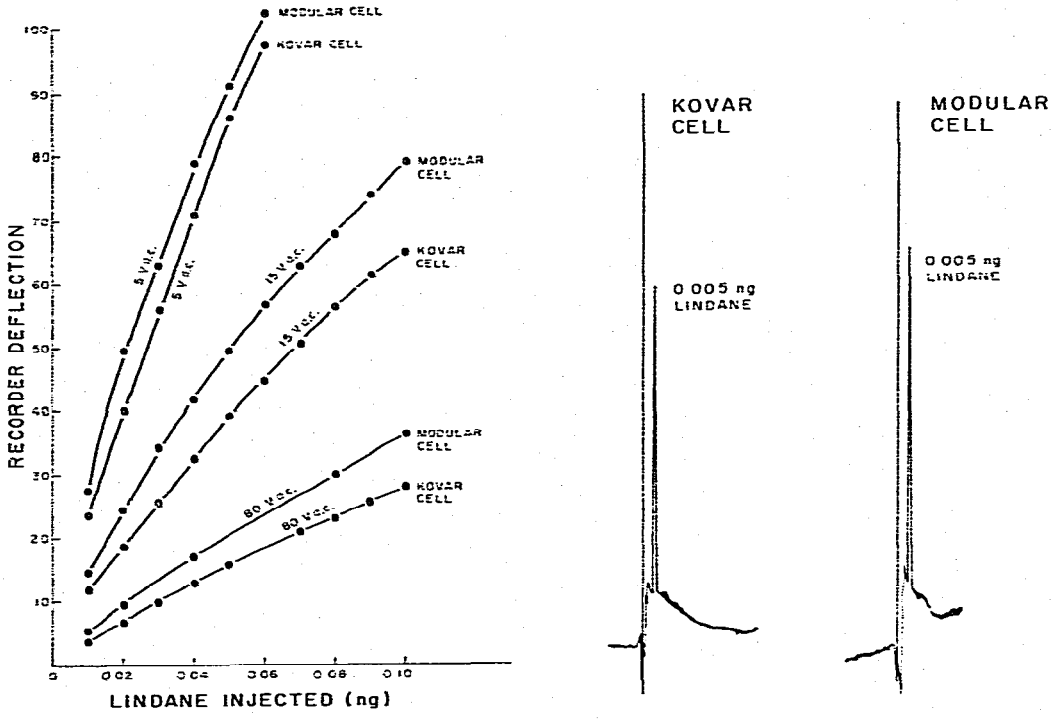

Fig. 2. Comparison of linearity of responses and sensitivity of response of the Kovar cell vs. modular cell electron capture detector.

Fig. 3. Comparison of recorder noise levels observed with Kovar cell and modular cell electron capture detectors operated in the maximum sensitivity ranges.

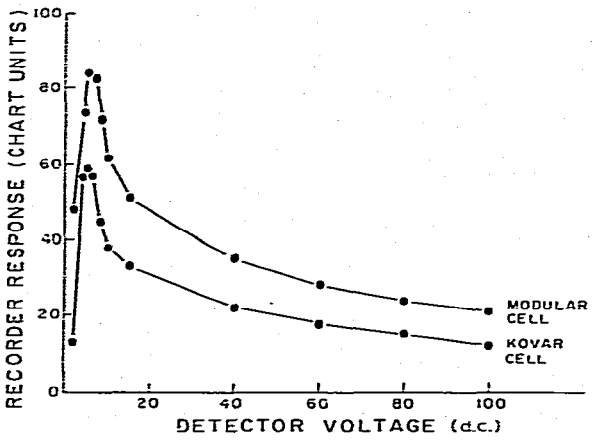

Fig. 4. Effects of varying collection voltage on Kovar and modular cell electron capture detectors.

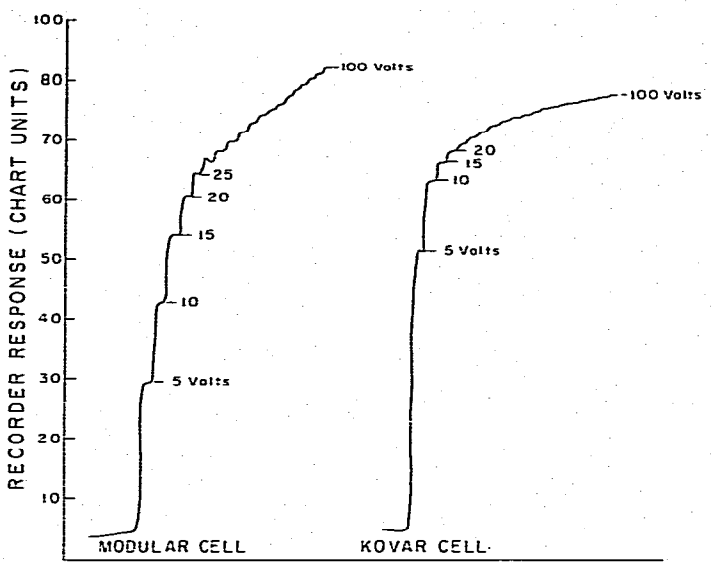

Fig. 5. Standing current (d.c. voltage) profiles of Kovar and modular cell electron capture detectors.

Use of both detectors in routine analysis of organochlorine residues has shown the modular cell to function as well as the Kovar cell. The frequency at which foils had to be removed and cleaned to restore detector performance was about the same. The ease with which the foil could be removed from the cathode (it is open at both ends) made maintenance relatively simple.

In summary, the modular cell functions as well as the Kovar cell. It has, however, certain advantages in terms of manufacture, ruggedness and serviceability. No evidence was found to suggest that joints tighter than friction-fit were necessary for good performance. The modular cell with a friction fit between the inner thin-walled stainless-steel tube and the outer thick Pyrex insulation tubes, when heated, would tend to form a tighter and tighter seal because the thermal expansion of stainless steel is greater than that of Pyrex. If a leak were a problem, a seal made with an electron-capture grade grease would probably suffice since pressures within the detector are only slightly above atmospheric pressure. The modular cell was not tested with any of the high-temperature foils, but there does not appear to be any major obstacle to their use, as is the case with the Kovar cell, at temperatures up to 300° (ref. 4) and use of a silica (quartz) cathode–anode insulator would extend the temperature range further[5]. No attempt was made to operate the modular cell in a pulsed mode as there seems to be little to be gained in operating concentric tube electron capture detectors in this mode.

Although no specific fragility tests were carried out, the use of a much thicker walled glass insulator in the modular cell as compared to that of the Kovar cell would indicate decreased fragility of the modular cell. The friction-fit functions of the modular cell would also not transmit imposed stress to the glass insulator from the non-glass portions as readily as would be the case with the Kovar cell.

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