Notes

CHROM. 6205

A simplified electron capture detector

In gas chromatographic work the radioactive foil electron capture detector is widely used for the detection of chlorinated hydrocarbon residues, such as those found in pesticides. It responds only to those molecules which readily capture electrons, such as many oxygen- and halogen-containing materials as well as unsaturated compounds.

Although the electron capture detector is relatively simple in design and maintenance, some general features can be improved. These modifications to the detector result in: (1) the elimination of pyrolysis in the gas chromatograph by means of a complete all-glass construction; (2) simpler cleaning procedures; and (3) low manufacturing costs of the detector.

The theory of operation of the proposed detector does not differ from the conventional concentric tube detector, which has been described in detail by several authors[1-3].

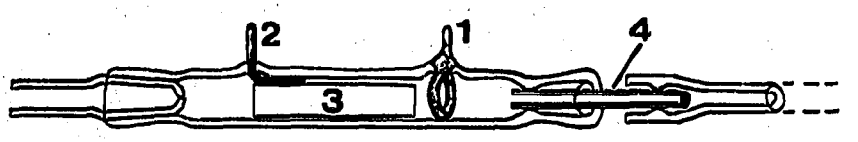

Fig. 1. Principle of design of the simplified electron capture detector. 1 = Collector; 2 = cathode; 3 = titanium tritide foil; 4 = PTFE tubing.

Construction

The basic design of the detector is shown in Fig. 1. The body of the detector is made of Pyrex glass with collector (1) and cathode (2) in platinum. The radioactive source is a 250-mCi, cylindrically wrapped titanium tritide foil (3). The detector is attached to the column by means of a short piece of PTFE tubing (4). To ensure a firm and gas-tight connection between the glass and PTFE tubes, both the detector cell and the exit of the column are provided with an "inner neck". When testing with this type of connection, it was found to be gas-tight up to 10 kg/m^2. No interference from the connection was observed when incorporated in a gas chromatograph–mass spectrometer system.

The whole construction is kept in the detector oven, and signal and ionization voltage cables are connected to the detector cell by means of small spring-holders, directly attached to the protruding platinum electrodes.

Discussion

When comparing the performance of the present detector with that of a conventional concentric tube detector (Varian 1700), sensitivity and stability were found to be similar. Due to the construction, however, the all-glass detector is easier to dismantle for cleaning the radioactive foil, and not as liable to damage as the conventional Kovar cell. It can be cleaned and re-used in less than 45 min.

When detectors are operated at suboptimal temperatures, decreased sensitivity and reduced working life of the radioactive foil sometimes result from condensation of impurities. In constructions where the detector cell is protruding from the detector oven, the actual cell temperature has been found to be significantly lower than that recorded for the oven. With the present modification, the detector cell can be completely immersed in the oven, thereby excluding possible temperature disparities.

Certain pesticides are decomposed when they are exposed to hot metal surfaces[4]. Therefore, glass columns are now frequently used instead of metal columns. In most instruments, however, the detection system is connected to the column by means of metal tubings. These tubings, placed in the detector oven, usually have a higher temperature than the column, and thereby increase the risk of pyrolysis. The use of a glass–PTFE system from the injection septa to the detector cell would seem to minimize the degradation within the gas chromatograph.

The proposed detector is also very cheap: approximately five modified all-glass detectors can be made for the cost of one conventional Kovar cell.

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