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A NEW UNIVERSAL RADIOIONIZING DETECTOR FOR GAS CHROMATOGRAPHY: MODEL DNW

J. LASA AND T. OWSIAK
Institute of Nuclear Research, VI Department, Cracow (Poland)
AND
D. KOSTEWICZ
Institute of Nuclear Techniques, Academy of Mining and Metallurgy, Cracow (Poland)

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SUMMARY

The basic parameters characterizing the new construction of a detector—model DNW—are presented in this paper. This detector can be used as a cross-section, argon or electron-capture type detector. Nickel-63 was used as the source of ionizing radiation in the detector. The detector can work at temperatures up to 400°.

INTRODUCTION

In addition to such classical detectors for gas chromatography as the flame-ionizing detector and the catharometer, three types of radioionizing detector are currently being used. They are designated according to the type of physical phenomenon which plays the most important role in the detectors and are: the cross-section detector, the argon detector and the electron-capture detector.

In recent years—as a result of the investigations of several research laboratories dealing with the construction of such devices—a characteristic form for the active space has been worked out for all of these detectors.

The cross-section type detector is generally characterized by the cylindrical configuration of the electrodes. The mutual distance between them is chosen in such a way that homogeneity of the electrical field inside the active space of the detector is assured[1,2].

The argon detector can be constructed in a similar manner to the cross-section type which was developed by BOTHE[3], or it can have the electrode configuration which was proposed by LOVELOCK[4]. In the latter type of the geometrical configuration, the electrical field extending between the detector electrodes is not homogeneous and causes the increased production of metastable atoms around the electrodes. In consequence, the sensitivity and dynamic range of the detector signal increase.

The electron-capture type detector, especially if it operates under d.c. supply

conditions, should be such that the configuration and form of the electrodes and active space ensure the non-homogeneity of the electrical field which is necessary for ionic recombination.

All the above-mentioned configurations of the active space can be obtained easily in the detector described in this paper. The required nature of the detector response is achieved by changing the end of the appropriate electrode, and the use of a suitable carrier gas and voltage supply.

CONSTRUCTION OF THE DETECTOR

The detector is shown in Fig. 1. It consists of a head (Fig. 1a) in which there are electrodes, 2 and 3, and insulators, 5, 6 and 7. The casing, 8, is screwed into the head. The casing is fitted with a supporting collar, 9.

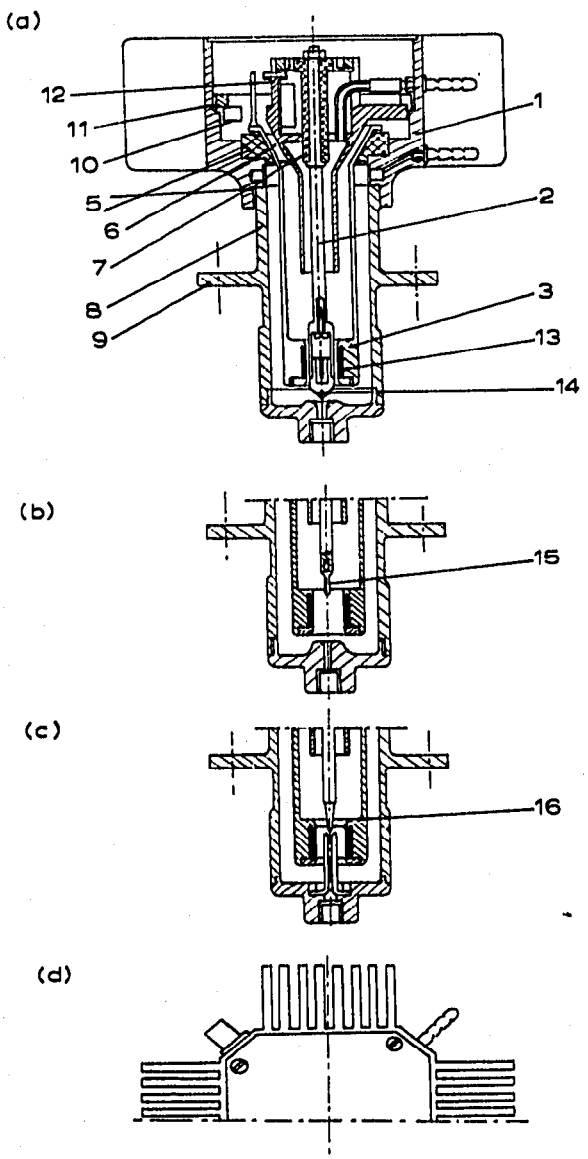


Fig. 1. Cross-section of the detector–Model DNW.

The detector is fastened on the thermostat in such a way that its active space is within the thermostat while the detector head is outside. The electrode insulators which are in the detector head are made of teflon. The detector head has cooling fins (Fig. 1d) which ensure a practically constant temperature for the insulators 5 and 6. Insulator 7 is inside the electrostatic screen, 10, which is fixed in the detector head by means of a lock-nut, 11. Insulator 7 is cooled by water which flows through a brass cylinder, 12, placed in the screen, 10. Such a cooling system for the insulators makes it possible to keep their temperature constant to that of the thermostat, inside which the active space of the detector is situated.

The geometrical configuration of the active space corresponding to a particular detector type is attained by changing the end of electrode 2. The active space of the detector is inside the radiation source, 13, which is placed on the end of electrode 3. ^{63}Ni is used as the source of beta particles in the detector model DNW.

Fig. 1a shows the geometrical configuration of the active space corresponding to a cross section-type detector. The end of electrode 2 is a cylinder, 14. The distance between its surface and the inner surface of radiation source is 1 mm.

Fig. 1b shows the geometrical configuration of the active space corresponding to an electron-capture type detector. The electrode 2 ends with the terminal 15, the size of which ensures such an non-homogeneity of the electrical field inside the detector that its sensitivity for electronegative components reaches its optimum.

The geometrical configuration of the active space of the argon detector is shown in the Fig. 1c. In this case the electrode 2 is terminated by the terminal 16 the shape of which permits the ionization of gas to commence inside the detector at a fairly low voltage supply.

A view of the detector head is shown in Fig. 1d. On the side of the detector head are the leads for the voltage supply and the electrometer, as well as the inlet and outlet for the cooling water and the outlet for the carrier gas.

THE WORKING CONDITIONS FOR THE DETECTOR

The use of an appropriate carrier gas and an adequate voltage supply are necessary factors for the instigation of any definite physical phenomena in the active space of the detector.

The cross-section type detector shown in Fig. 1a can work with hydrogen or helium as the carrier gas with a voltage supply of 100 V.

The electron-capture type detector (Fig. 1b) has been examined with nitrogen as carrier gas and with a d.c. voltage supply. With the sizes of terminal 15 (Fig. 1b) of electrode 2 used, the optimum voltage supply was 9 V.

The argon-type detector (Fig. 1c) works at voltage of 1000 to 1500 V with argon as the carrier gas.

THE DEPENDENCE OF THE IONIZATION CURRENT OF THE DETECTOR ON THE TEMPERATURE

The idea behind the construction of the detector in question was, apart from its universality, to increase detector stability at variable temperatures. This property of the detector has been achieved by placing the insulators of the electrodes 2 and 3

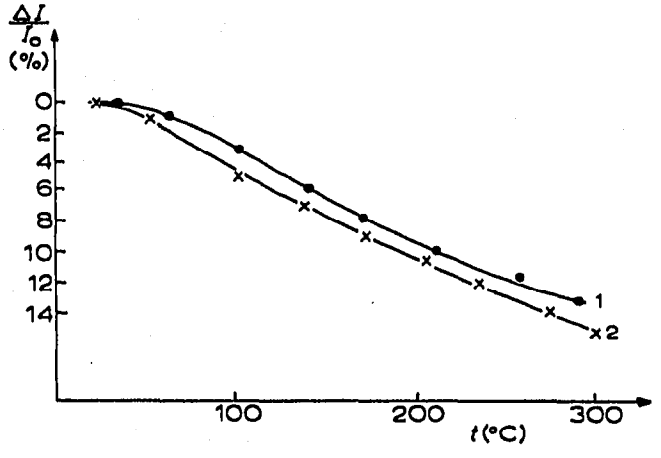
outside the active space of the detector, which is situated within the variable temperature zone of the thermostat. In the various designs of radioionizing detectors met so far, the insulators of the electrodes are situated near the active space of the detector, so they are subject to variable temperatures. A change of temperature of the insulators causes them to have different electrical resistances which in turn means a change in leakage currents that influence the "drift" of the zero line. Hence it would appear that by assuring a steady temperature for the insulators, the stability of the detector increases. The "drift" of the zero line then only results from changes in the physical property of the gases with respect to their different temperatures.

Figs. 2, 3 and 4 show the percentage changes of the ionization current of the detector when working as a cross-section, electron-capture and argon-type detector with varying temperatures. When water flows through the cooling cylinder 12 (Fig. 1a), the response of the detector is designated "with cooling". When the detector works without water flowing through the cylinder and is cooled only by natural air circulation we designated it "without cooling".

Fig. 2 shows the change of ionization current of the detector with configuration of the electrodes according to Fig. 1a, with helium as carrier gas. The voltage supply is 100 V. It is sufficient to secure the response of the detector within the saturation current.

In Fig. 2, curve 1 shows the response of the detector with cooling, and curve 2—without cooling. In this case, the limited efficiency of the cooling system on insulator 7 is surprising; the stability of the zero line of the detector with cooling is only about 20% better than without cooling.

In Fig. 3 the results of analogous investigations of the electron-capture type detector with the geometrical configuration of the electrode as shown in Fig. 1b are presented. The detector was examined with nitrogen as carrier gas with a d.c. voltage supply. In this case, the increase of the temperature of the active space of the detector gives a rise in the ionization current. The above dependence does not agree with the



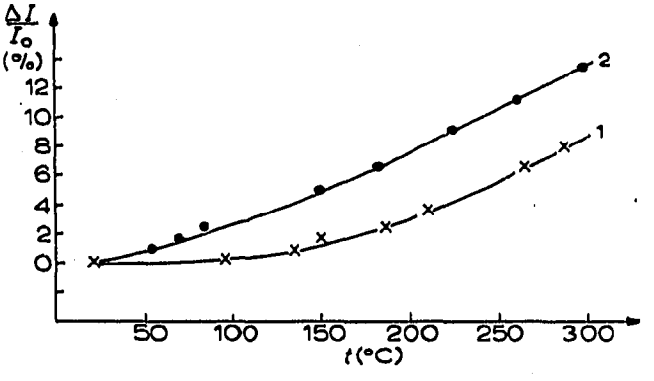


Fig. 2. Dependence of the cross-section type detector current on its temperature, carrier gas—He: V_0 = 26 ml/min; voltage supply = 100 V; I = $1.6 \cdot 10^{-10}$ A. Curve 1 = with cooling; curve 2 = without cooling.

Fig. 3. Dependence of the electron-capture type detector current on its temperature; carrier gas—N_2: V_0 = 50 ml/min; voltage supply = 9 V; I = $2 \cdot 10^{-9}$ A. Curve 1 = with cooling; curve 2 = without cooling.

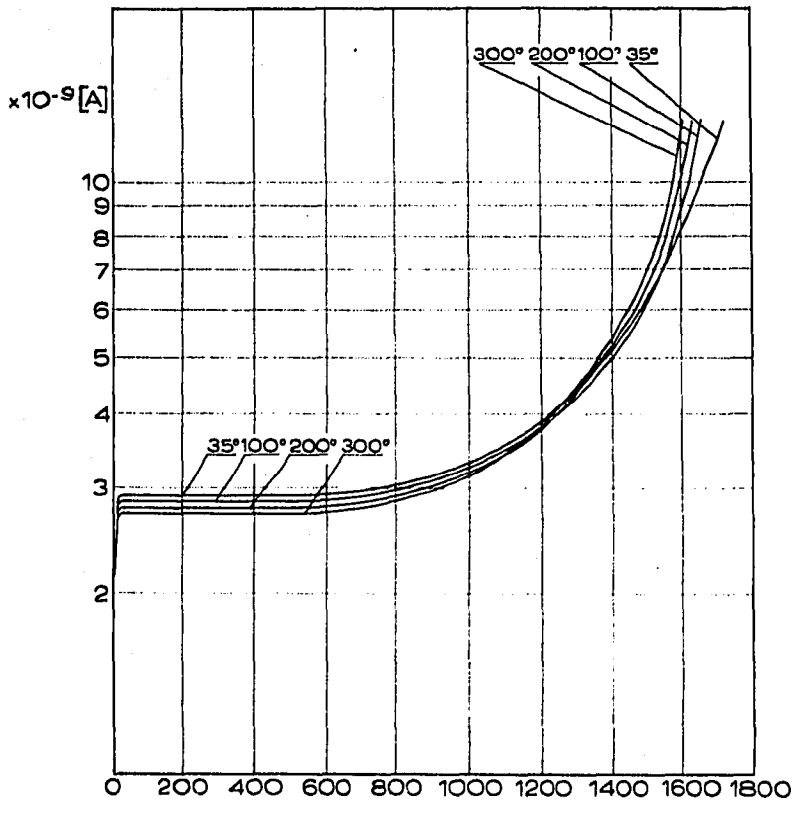


Fig. 4. Current–voltage characteristics of the argon-type detector at different voltages supplies; carrier gas = argon; V_g = 50 ml/min.

expected one and is rather difficult to explain. In Fig. 3, curve 1 shows the response of the detector with cooling, and curve 2—without cooling. As is seen the effectiveness of the cooling system is better than with the cross-section type detector. When the detector is operated with cooling, its drift zero line is smaller by about 50% than without cooling.

Different results have been obtained for the argon-type detector (as in Fig. 1c). Fig. 4 shows the current–voltage characteristics of this detector which works with argon as carrier gas, different temperatures of the active space being used.

For a voltage supply up to 600 V, an increase of the temperature in the detector results in the expected decrease of the saturation of the ionization current.

With higher voltage supply, gas amplification occurs in the detector. The value of the gas amplification coefficient depends exponentially upon the temperature. Thus, enhancement of the detector temperature causes an increase in its ionization current. It can be seen in Fig. 4 that these characteristics overlap one another in the voltage supply range of 1000 to 1400 V.

The comparison of the relative changes in the ionizing current for the detector with and without cooling is shown in Fig. 5. When the detector is supplied with 100 V and argon is used as carrier gas, the effectiveness of the additional cooling is better than when the detector works under the same conditions with helium as carrier gas. In this case, the stability of zero line is better by about 55% when the detector is cooled (curve 2, Fig. 5).

For a voltage supply of 1100 V the drift of the zero line of the detector with

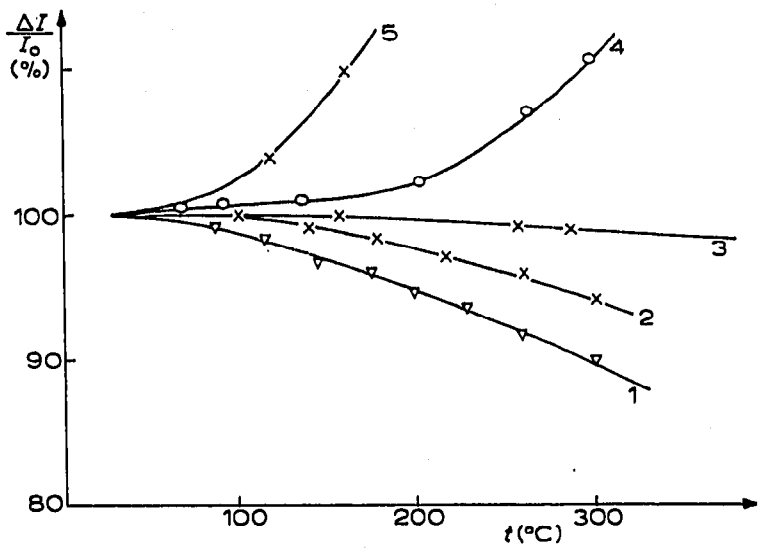


Fig. 5. Dependence of the argon-type detector current on its temperature; carrier gas–argon; V_g = 50 ml/min. Curve 1 = voltage supply 100 V with cooling; curve 2 = 100 V without cooling; curve 3 = 1100 V with cooling; curve 4 = 1500 V with cooling; curve 5 = 1500 V without cooling.

cooling—according to Fig. 4—is smaller; and at 300° amounts to 2% (curve 3, Fig. 5).

When the argon detector operates at a higher voltage, the effectiveness of the cooling system is the highest. Curve 4 (Fig. 5) is with respect to the detector with a voltage up to 1500 V with cooling, and curve 5 is without cooling.

CONCLUSIONS

After analysing the results presented in Figs. 2–5 with regard to the operation of the DNW Model detector, one can say that the drift of the zero line of the detector is connected with the flow of current through the insulators and depends upon the different properties of the gases at variable temperatures. The value of the leakage current depends upon the voltage at which the detector works and on the temperature of its insulators. It has been observed that the value of this current also depends upon the kind of gas flowing through the detector.

On the basis of the results presented it can be stated that the detector—Model DNW has very good thermal stability, particularly when it is operated with additional cooling.

Another practical advantage of the present detector is the possibility of working at up to 400° and of using three types of detectors in one *viz.* the cross-section type, electron-capture type and argon-type detector.

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