

SOME EXPERIENCES IN THE CONSTRUCTION OF GAS CHROMATOGRAPHS

By I. KÖVÁRI and SZ. FÉNYI

Institute of Organic Chemistry, József Attila University, Szeged

(Received May 5, 1964)

On basis of certain consideration our gas chromatograph has been fitted with thermal conductivity cell. The detector body is a wrought brass block, and it is a flow-detector type. As a gauge resistance-wire a tungsten spiral was used. Thermostating was accomplished with air thermostat. This thermostat might be used up till 250 °C with ± 1 °C accuracy.

The rapid spreading of gas chromatography is due to the cheapness of the devices used, on the one, and naturally due to the wide field of application on the other. The employment of relatively simple appliances makes possible the successful approach to "domestic" construction of the instrument at universities and research institutes. A number of home and foreign textbooks [1, 2, 3, 4, 5, 6, 7] deal with the development achieved so far, which highly facilitates the possibility for domestic construction.

According to literature the construction of a gas chromatograph of any type (gas-solid, gas-liquid) is determined by the quality of the following three units:

- detector
- thermostating
- recording of the detector signal.

Among detectors the thermal conductivity cells are the most wide-spread ones; though these are not the most sensitive devices, they accomplish certain tasks satisfactorily. On basis of this consideration our gas chromatograph has been fitted with such a detector. The detector and its dimensions are shown in Figure 1.

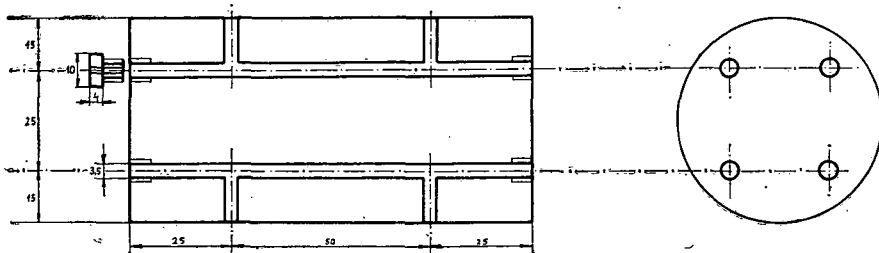


Fig. 1. Detector body and its dimensions

The detector body is a wrought brass block provided with four borings, of which two opposite ones are the comparing and measuring cells, respectively. The other two opposite borings contain the two wires of constant resistance of the Wheatstonebridge. Thus our device is of a flow-detector type. As gauge resistance-wire a tungsten spiral was used. The spirals were connected to brass wire by spelter and provided with bilateral terminals. The space of gas was prevented by drawing home the screws in Figure 1, applying klingerit and asbestos packing. Using this packing experiments were carried out at 200 °C with absolute certainty.

The electric circuit of the detector is shown in Figure 2. The rough and fine compensation of the bridge is done with potentiometers R_5 and R_6 , respectively. The total bridge-current is regulable with potentiometer R_7 within the range of 50 and 240 mA. The detector is fed with a 6V battery. The R_3 and R_4 constant resistances are placed in the detector body for the sake of thermostating, the output signal of the detector being thus insensitive against any change in temperature. $R_1 = R_2 = R_3 = R_4 = 20$ ohm, $R_5 = 5$ ohm, and $R_6 = R_7 = 100$ ohm.

The relationship between the integral detector signal and the amount of sample introduced was checked. This proved to be linear (see Figure 3) in accordance with literary data (8).

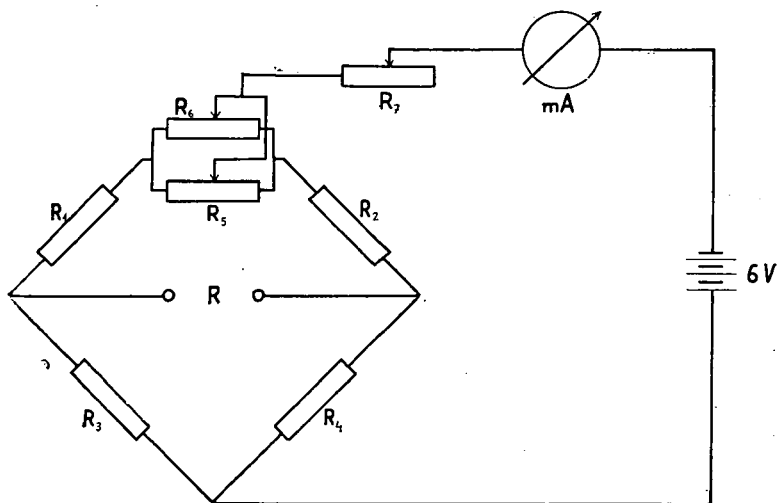


Fig. 2. Electric circuit of the detector

The sensitivity of the detector (change in signal over change in sample amount) is the slope of the corresponding straight lines in Figure 4. Figure 4 was obtained by plotting these slope values (sensitivity) against bridge-current. Similar results were obtained by M. Spalma, too (8).

Thermostating was accomplished with air-thermostat. The volume of the thermostat was 30 liter, since the apparatus is intended to be used for preparative purposes as well, in the future. The heatregulation circuit of the thermostat is shown in Figure 5. This thermostat might be used up till 250 °C with ± 1 °C. accuracy.

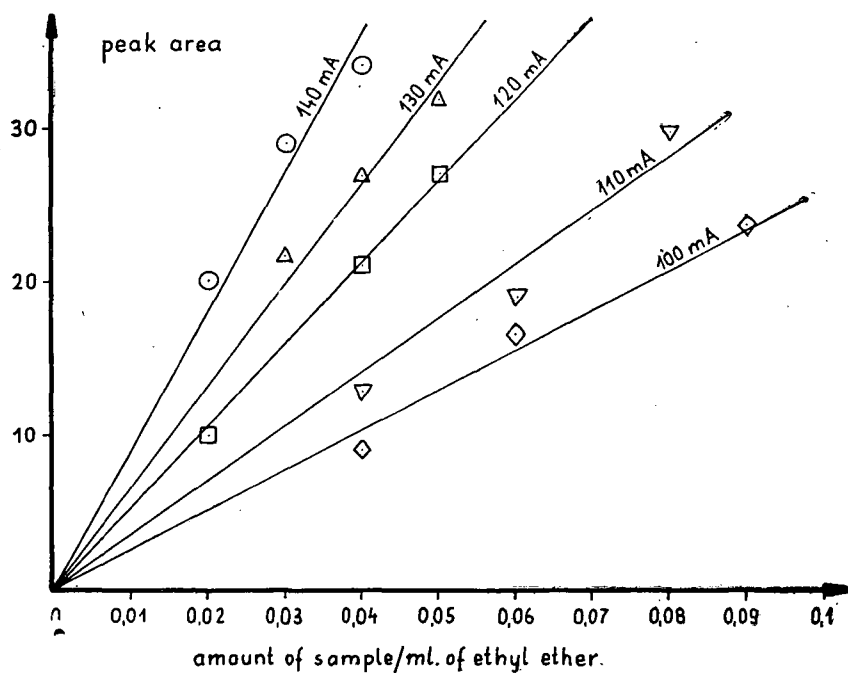


Fig. 3. Relationship between integral detector signal and sample amount

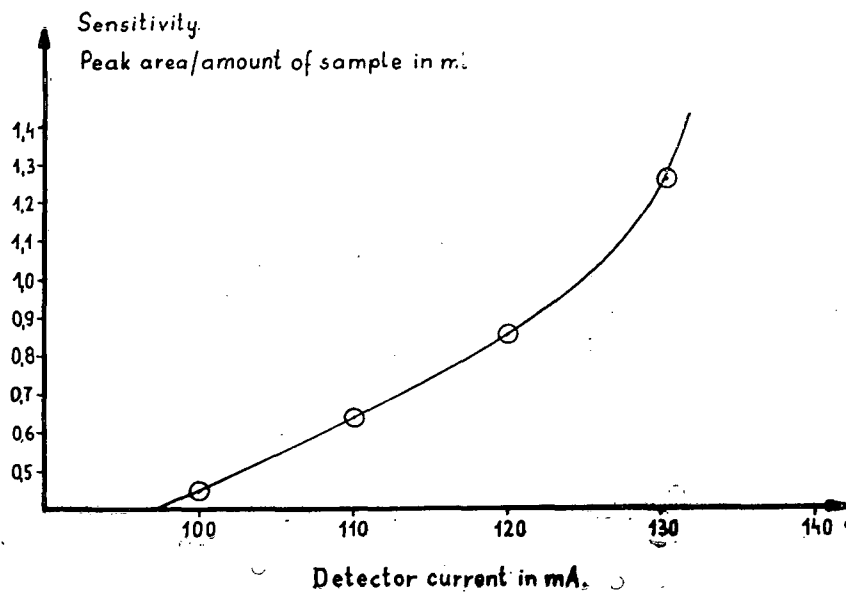


Fig. 4. Sensitivity of the detector

The principal arrangement of the thermostat was made on basis of scheme of the Organic Chemical Research Institute, Budapest. The detector signal was recorded by a J. C. Eckardt single trace compensograph of 0–2 mV measuring range. This

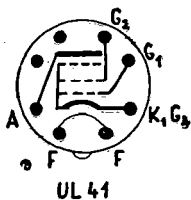
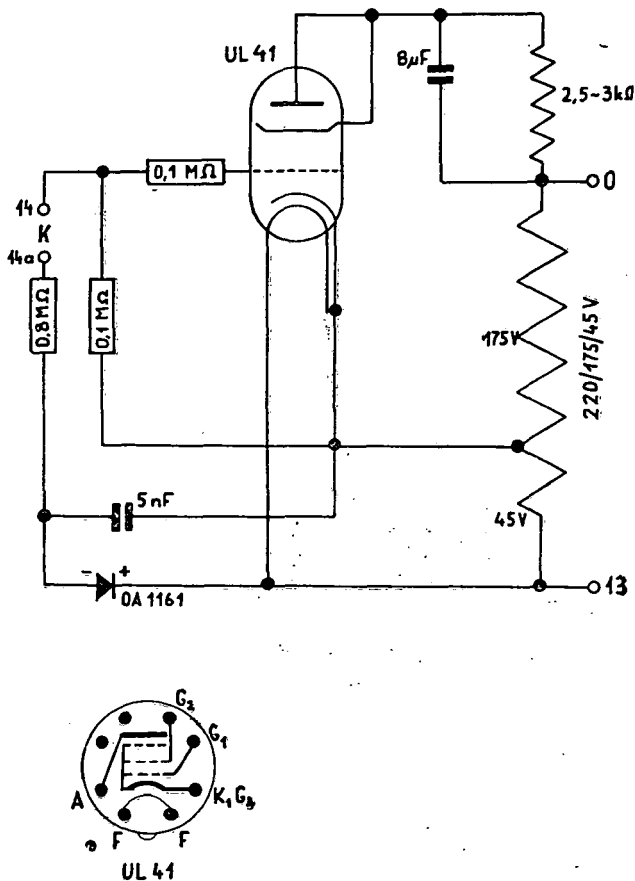


Fig. 5. Heat-regulation circuit of the thermostat

compensograph could directly be connected to the Wheatstone-bridge, without any fitting parts.

* * *

Thanks are due to Mr. S. KÓNYA technician of the department for the execution of the apparatus.

References

- [1] Bayer, E.: Gaschromatographie (Springer-Verlag, Berlin—Göttingen—Heidelberg, 1959).
- [2] Kaiser, R.: Gas—Chromatographie (Akad. Verlagsges, Leipzig, 1962).
- [3] Kenlemans, A. J. M.: Gas—Cromatographie (Verlag Chemie, Weinheim Bergstrasse 1959).

- [4] *Phillips, C.*: Gas-chromatography (Butterworths, London, 1956).
[5] *Schay, G.*: A gázkromatográfia alapjai (Akadémiai kiadó, Budapest, 1961).
[6] *Szepesy, L.*: Gázkromatográfia (Műszaki Könyvkiadó, Budapest, 1963).
[7] *Vámos, E.*: Kromatográfia (Műszaki Könyvkiadó, Budapest, 1959).
[8] *Spála, M.*: Fortschrittsberichte zur Gas—Chromatographie. (Akademie-Verlag, Berlin) p. 199..

ОПЫТЫ ПОСТРОЕНИЯ ГАЗХРОМАТОГРАФА

И. Кевари и С. Фени

На основе некоторых размышлений наш газхроматограф снабжен хорошей теплопроводящей камерой. Тело самого детектора приготавливается из кованной меди, переносного типа. В роде сопротивления употребилась волфрамовая спираль. Воздушный термостат служил термостатом, регулирующий до 250 С точностью $\pm 1^\circ$.

