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CHROMATOGRAPHIC METHOD FOR HIGH-SPEED ANALYSIS OF GAS COMPONENTS

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SUMMARY

A new method is proposed for the definition of gas components with quickly changing compositions, based on the use of several parallel chromatographic channels, efficient columns and selective detection of the analysed substances.

INTRODUCTION

High-speed gas analysis is of interest both in scientific research and industrial processes. As an example of such a process we may take the production of steel, where the melting process lasts only 12–15 min and timely analytical control determines the quality of the metal produced and the conversion efficiency. The high rate of the physico-chemical processes occurring in the metal pool requires continuous (or quasi-continuous) information on the hydrogen, oxygen, carbon monoxide and dioxide contents of the waste gases.

In research^{1,2} aimed at solving the task of fast chromatographic definition of gaseous components, new methods such as frontal adsorption and high-speed stepping chromatography⁵ were suggested and studied experimentally. The first of these is nearly independent of the changes in the chromatographic parameters. The resulting chromatograms can easily and accurately be interpreted and the method is simple to perform. The analysis of model mixtures required only 35 s. The second method involving stepping chromatography required only 15–20 s for analysis of an analogous mixture. It is difficult to perform and does not provide accurate measurements. It should be mentioned that these methods have few advantages in comparison with the traditional elution variant of chromatographic analysis. A reduction in the duration of analysis for complete separation of the components and the use of one or a few connected columns causes serious technical difficulties.

In order to reduce the analysis duration efficiently, we now propose a new variant which allows in the use of several chromatographic channels, synchronous dosing of the analysed samples and selective definition of the components³.

EXPERIMENTAL

The system shown in Fig. 1 was employed. It is important to stress that in the upper channel the selective definition of the oxygen component by a thermochemical detector without a separating column was performed. The separation of hydrogen and carbon monoxide and the selective definition of components by a thermochemical detector took place in the middle channel. The definition of carbon dioxide was achieved by a thermal conductivity detector after the chromatographic separation of carbon dioxide from lighter components.

Packed capillary columns with inner diameters from 0.6 to 2 mm ensured high efficiency and high linear velocity of the carrier gas.

Rapidity of detection was ensured by the use of microdetectors with a chamber volume of 30–50 μl .

The use of a rod injector did not cause interruption of the carrier gas flow and excluded the possibility of false signals in the dosing process.

RESULTS AND DISCUSSION

Table I shows the sorbents which gave the optimum results. The aim was to achieve a complete separation of the components within the minimum time.

The results of this study confirm the previous conclusions⁴ that the quality of packing and the particle diameter of the sorbent have a great effect on the column efficiency and penetrability. The optimum separation was achieved when the ratio of the column diameter to that of the sorbent particles was 0.25–0.2. A decrease in the column diameter, particle diameter and narrowing of the fractional composition of the sorbent increased the column efficiency and penetrability, but the increase in column resistance necessitated the employment of higher pressures of the carrier gas.

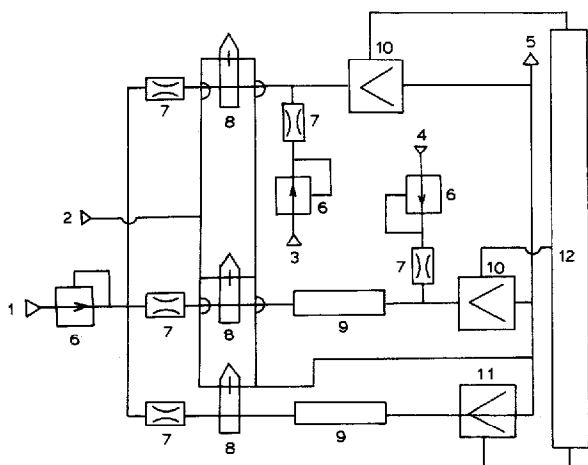


Fig. 1. Scheme of the chromatographic system. 1 = Carrier gas (helium) inlet; 2 = analysed gas inlet; 3 = hydrogen inlet; 4 = oxygen or air inlet; 5 = gas outlet; 6 = pressure regulator; 7 = fluidic resistor; 8 = injector; 9 = packed capillary column; 10 = thermochemical detector; 11 = thermal conductivity detector; 12 = recorder.

TABLE I
SORBENTS USED

<i>Sorbent</i>	<i>Particle diameter (mm)</i>
Molecular sieves 5 A	0.1-0.15
Molecular sieves 4 A	0.15-0.2
Activated carbon SKT-2	0.1-0.15
Activated carbon Saran	0.15-0.2
Polysorb-1	0.1-0.25
Porapak	0.15-0.18
Silica gel ASK	0.15-0.2
Silica gel KSM	0.15-0.2
Polysorb-4	0.1-0.25

A complete separation of hydrogen and carbon monoxide was obtained on the columns (20 cm × 1 mm I.D.) packed with SKT-2 and Saran. The most rapid definition of the carbon dioxide component was obtained on the column (50 cm × 1 mm I.D.) packed with silica gel ASK. In these cases the analysis time did not exceed 10 s.

The suggested method allows the analysis time to be reduced with incomplete separation of the components and the use of computers.

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