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REVIEWS

PRINCIPLES OF INTRODUCING ORGANIC SUBSTANCES INTO A GAS CHROMATOGRAPHIC COLUMN BY THERMAL DESORPTION METHOD DEPENDING ON THE VOLATILITY OF THE MIXTURE COMPONENTS

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Abstract. A review of thermal desorption methods from concentrators in portable express gas chromatographs of the EKHO series for introducing samples of organic substances into a gas chromatographic column depending on the volatility range of substances in samples is presented. Methods for introducing samples without manual operations in EKHO devices are considered. The principles of introducing a sample with incomplete gas-dynamic isolation of the injection chamber and column when introducing samples of explosive vapors and arenes with different volatility of substances in the sample are described, as well as complete insulation of the injection chamber and column when introducing samples of C1–C5 saturated hydrocarbons with sharply different volatility.

Keywords: *gas chromatography, thermal desorption, volatility of mixture components, gas-dynamic isolation of the injection chamber*

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Adsorption concentration is widely used in the determination of trace amounts of organic substances. Depending on the analytical tasks, the determination of adsorbed substances can be carried out using thermal desorption (**TD**) of substances from a concentrator and without TD. In spectroscopic methods of analysis (X-ray fluorescence, neutron activation) The TD may not be used [1]. In spectrometric sampling gas analytical methods (gas chromatography, mass spectrometry) TD is widely used in the preparation of samples of trace amounts of organic substances [2, 3].

A series of portable, highly sensitive and high-speed EKHO gas chromatographs [4-7] uses concentration to determine trace amounts of a wide range of organic substances to increase the threshold sensitivity for detecting substances with a different range of volatility in analyzed samples. The input of a sample of substances is carried out by thermal desorption methods from concentrators in modes that provide short input times for effective separation of sample substances. The parameters and modes of TD were determined based on the theoretical results of the work [3].

Sorbents of concentrators are selected based on the condition of a high degree of concentration of the substances being determined, taking into account their volatility. Portable gas chromatographs of the EKHO series use three types of concentrators: flat, fine steel grids for detecting vapors of explosives and organochlorine pesticides, metal tubes 30 mm long with an inner diameter of 5.9 mm with a film of SE-30 sorbent on the inner surface for aromatic hydrocarbons C6–C8, and the same tubes filled with carbon fiber AHM, for alkanes C1–C5. Concentrators are characterized by the necessary sorption capacity and the ability to efficiently inject a sample using the TD method in a short time.

Steel grids are designed for explosive vapors, tubes with SE-30 sorbent for substances with slightly different volatility, and tubes with carbon fiber for mixtures of substances with sharply different volatility. The first concentrators are used to detect trace amounts of explosive vapors during anti-terrorist control [4, 5], the second - for the geochemical search for hydrocarbon deposits by registering aromatic hydrocarbons migrating from deposits on the Earth's surface [6, 7] by passive concentration by laying concentrators to a depth of 1 m; the third – for the actual geochemical search for hydrocarbon deposits by registration of C1–C5 alkanes on the Earth's surface, which also migrate from deposits [8, 9].

The sample is injected using the TD method either with incomplete insulation of the input chamber and column when the concentrator is heated, or with complete insulation.

The purpose of the review is to systematize methods for introducing samples into a gas chromatographic column by thermal desorption from concentrators, depending on the volatility of substances in the samples, without using manual operations in portable devices, which is necessary to reduce the duration of analysis in the field: from the detection of explosives to the search for hydrocarbon deposits. The volatility of the analytes was characterized by saturated vapor concentration, boiling point, and desorption energy.

TD methods with partial gas dynamic isolation of the input chamber (**IC**) from the column during heating of the concentrator are presented – for BB and for arenes C6–C8 with high and slightly different volatility, as well as with complete isolation of IC from the column for alkanes C1–C5 with sharply different volatility.

Table 1 shows the methods of TD sampling and the characteristics of the substances under consideration, which determine the volatility, such as saturated vapor concentration and boiling point.

The introduction of substances with high and slightly varying volatility (explosives and arenas). When entering, partial gas dynamic insulation of the IC and the column is used.

Injection of explosive vapors in an express GC EKHO-M device with a polycapillary column (**PCC**) with an explosive separation time of up to 20 s and an electronic capture detector [4]. For concentrating vapors of explosives, concentrators in the form of fine wire meshes with a wire diameter of d1 = 0.05 mm made of stainless steel are used, with the side length b of the square mesh opening being 0.08 mm (Fig. 1). The 7.5 mm diameter mesh is fixed in the shell. Such concentrators, proposed in the late 1980s [5, 15], are characterized by high efficiency in trapping explosive vapors for a short time of 5-10 seconds [16].

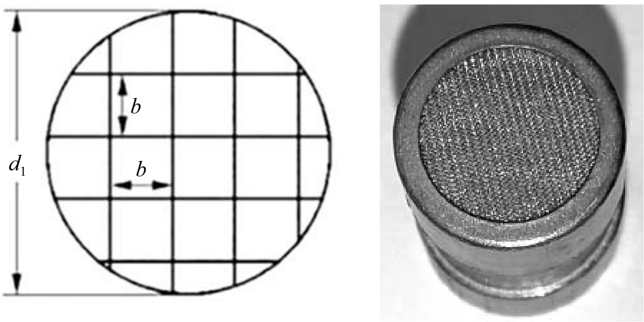

Fig. 1. Diagram of a metal mesh concentrator (on the left) and its appearance (on the right, enlarged).

The TD input scheme is shown in Fig. 2. IC *2* and PCC are connected by a thin capillary *4* with a diameter of 0.25 mm. During thermal desorption, the concentrator *1* is heated to 450-550 K for 2-3 s. Rapid sample injection for 0.5–1 s into the PCC is carried out by a carrier gas flow of 40 ml/min when the gas switch *6* is switched on. During the heating of the concentrator, the IC volume with the concentrator is partially gas-dynamically isolated from the PCC. For this purpose, during heating of the concentrator, a small return flow of 0.3–0.4 cm^3/

Table 1. Methods of sampling by thermal desorption and characteristics of volatility of substances

TD input method	Name of the substance	($c_{\text{vap. sat.}}$, ppt [10]) T_{boil}, °C [12]	Energy of desorption, kJ/mol	References
Gas engines- physical isolation of the IC and the column	2,4,6-TNT	(9400)	92	[11]
	Nitroglycerin	(580000)	86.7	
	p-Xylene	138.4	35.9	[13, 14]
	m-Xylene	139.1	36.3	
	o-Xylene	114.4	36.7	
	Toluene	110.8	33.5	
	Benzene	80.1	30.8	
Complete isolation of the IC and the column	Pentane	36.1	33.5*	[13, 14]
	Bhutan	-0.5	27.5*	
	Isobutane	-11.7		
	Propylene	-47.8		
	Ethan	-88.6	17.3*	
	Methane	-161.5	12*	

Note: *Desorption from graphitized carbon black.

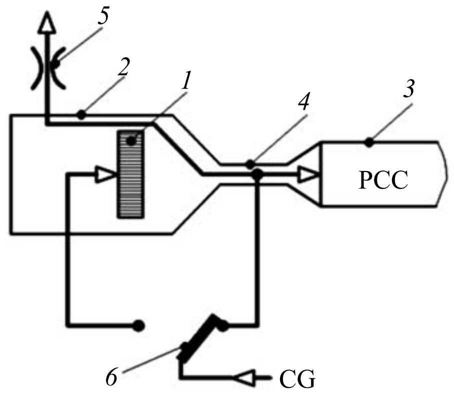

Fig. 2. Scheme for injecting substances with partial gas-dynamic isolation of the inlet chamber from the column. *1* — concentrator; *2* — thermostatically controlled inlet chamber; *3* — polycapillary column in the thermostat; *4* — thin capillary connecting the inlet chamber and the polycapillary column; *5* — choke, the gas flow through it protects against the ingress of interfering substances into the inlet zone; *6* — electromagnetic gas switch for sample insertion when switching the carrier gas line (CG).

min is supplied to IC through a thin capillary[4]. Valve *6* – thin capillary *4* – gap between the concentrator and the IC thermostat *2* – flow relief choke *5*. The reverse flow prevents substances from escaping from the IC. It has been experimentally established that a small amount of desorbable substances (no more than 10%) can be carried out of the IC by the reverse flow through the throttle *5*.

The satisfactory efficiency of injecting explosives with nitroglycerin (**NG**) and trinitrotoluene (**TNT**) in a short time is illustrated in Fig. 3 in the form of the relative magnitude of the signal at the output of the EKHO-M chromatograph depending on the injection time [16]. As can be seen, due to the higher volatility, NG is completely introduced in a shorter time of 0.4 s, and TNT is almost completely introduced in 1 s. In addition, despite the large difference in the volatility of substances measured by the concentration of saturated vapors, the rates of their thermal desorption from a flat grid concentrator do not differ much, and as a result, their input to the PCC in a short time, no more than 1 s.

The chromatogram of the mixture of substances in Fig. 4 with fairly symmetrical NG and TNT peaks demonstrates the satisfactory quality of the rapid injection.

The technical solution of the considered input from the TD is implemented in the EKHO-M device (Institute of Solid State Chemistry and Mechanochemistry of the Siberian Branch of the Russian Academy of Sciences (ISSCM SB RAS)) and is provided in publications [17, 18]. A more recent analogue is the portable gas chromatograph GHS–02P [19] with a similar TD-input of samples from a mesh concentrator (SIBEL LLC, Russia).

The introduction of C6–C8 *arenes* (benzene, toluene, xylenes) by the TD method during geochemical surveying is implemented in a polycapillary GC EKHO-FID-PC with a photoionization detector and a device for desorption input of samples from passive concentrators [7, 20]. A PCC with 1,000 capillaries with a diameter of 40 μm with a stationary liquid phase (SLP) liquid phase of SE-30 is used as a separation column. The separation time of the arenas is up to 100 seconds. The carrier gas is atmospheric air, purified by an integrated filter.

Concentrators in the form of tubes with a film of SE-

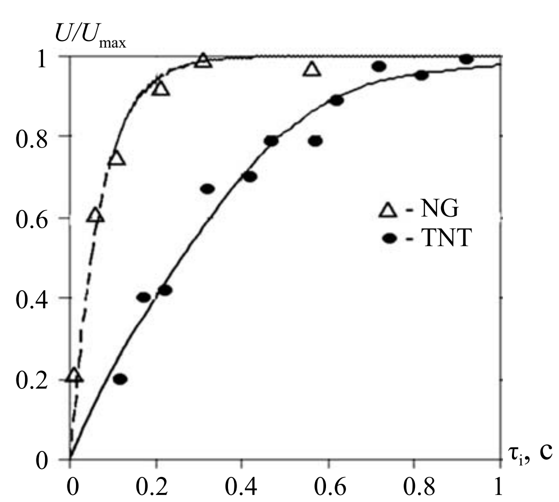

Fig. 3. Effect of the input time on the completeness of the sample input of nitroglycerin and trinitrotoluene [13]. The points are experimental data, the desorption temperature is 180°C.

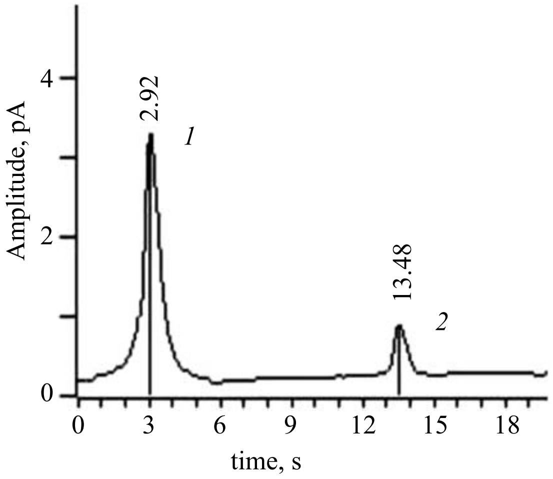

Fig. 4. Chromatogram of technical nitroglycerin with an admixture of trinitrotoluene. Polycapillary column manufactured by LLC Multichrome (Russia) (length 220 mm, 1100 capillaries with an inner diameter of 40 microns, SLP — stationary liquid phase SE-30), volume velocity of argon carrier gas 40 cm^3/min, sample insertion time 0.5 s.

30 sorbent [7, 20] on the inner surface for passive sampling are placed in the ground for 1-2 days. The measured average coefficients of concentration (enrichment) of arenes at room temperature are: benzene – 18.9, toluene – 72.4, *m*-xylene – 174.9, *o*-xylene – 190.5 (the enrichment coefficient was determined by the ratio of the magnitude of the GC signal received from a saturated concentrator to the magnitude of the signal from the calibration stream in which the concentrator was kept during calibration the hub). The introduction of samples from the concentrators into the PCC is carried out by the TD method.

It should be noted that TD in known sample injection devices is carried out in closed volumes, and a metering valve is used to inject the desorbed sample into the gas chromatographic (GCC) column [21, 22]. In other embodiments, the concentrator cartridge placed in the furnace is connected to a flow switch and equipped with a hollow needle inserted through a silicone membrane into the chromatograph evaporator [23-25]; the sample is injected into the GCC after the heating of the concentrator begins or the needle is inserted into the chromatograph evaporator.

The methods of sample input described above, as a rule, require energy–intensive (more than 100 watts only for the input device, for comparison, the consumption of GC EKHO is 40 W) and bulky equipment, which is unacceptable for an autonomous portable device, therefore, the authors of this review proposed an original device for sample input into the control panel without manual operations using the effect of gas-dynamic isolation of the chamber sample and column input during heating of the concentrator [6]. A schematic diagram of such an input is shown in Fig. 5. To enter the sample, the tubular concentrator *1* is placed in a thermostatically controlled input chamber *3*, heated to the required temperature, and maintained for a set time with the pneumatic electric valve (**PEV**) 5 closed and the PEV *4* open. At the end of heating, valve *4* closes and valve *5* opens at the same time. The desorbed sample is injected into the PCC *6* through a thin capillary *7* with a flow of 40 cm^3/min. Upon completion of the sample insertion, valve *5* closes, and valve *4* opens and the carrier gas (air) enters the GAC *6* and transports the sample through the PCC with a flow of 40 cm^3/min.

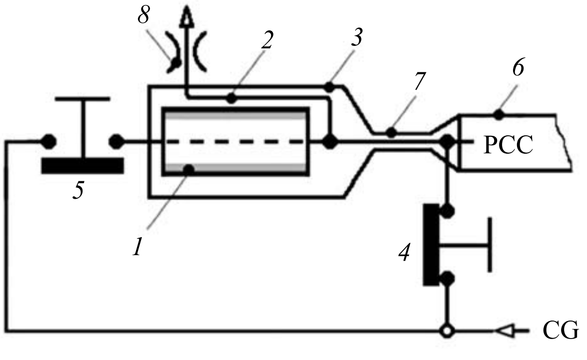

Fig. 5. Scheme of sample entry from the concentrator by thermal desorption with gas-dynamic isolation of the sample entry chamber during heating of the concentrator. The designations are given in the text when explaining the operation of the scheme.

The peculiarity of the sample input scheme is that during heating of the concentrator and analysis of the sample, the volume of the IC with the concentrator is gas-dynamically isolated from the PCC. For this purpose, during heating of the concentrator and analysis of the sample, a small return flow of 0.3 cm^3/min is supplied to IC through a thin capillary 7 through the valve *4* – thin capillary *7* – gap *2* between the concentrator and the thermostat – flow relief throttle *8*. The reverse flow prevents substances from escaping from the IC when valve *5* is closed. The amount of backflow is selected experimentally. The ratio of the locking and direct flows is approximately 1:100.

A small amount of desorbable substances can leave the IC volume mainly due to diffusion into the throttle channel *8* and be carried away by the reverse flow from the PCC. Input practice has shown that losses amount to no more than 10%. The low losses of arenes during this injection are due to the relatively insignificant difference in the volatility of arenes C6–C8 (benzene, toluene, xylenes) – the boiling points of these hydrocarbons are in the range of 80-144 °C [12]. Such an input system with gas dynamic insulation of IC and PCC for the duration of continuous heating of IC for 30-40 seconds at 200 °And the analysis of the sample for up to 150 seconds has been implemented in practice [7].

The efficiency of input with the TD-method of gas dynamic insulation of IC is illustrated in Figs. 6 and 7. A microgas system was used as a source of arenes [26], which provides concentrations in ppb of 68.6 benzene, 80.8 toluene, 25.5 *m*-xylene, and 39.2 *o*-xylene. Figure 6 shows the initial chromatogram of arenes, and Figure 7 shows the chromatogram with sample injection from the concentrator using the gas dynamic isolation method. The heating time of the concentrator is 33 s, the time of sample injection is 2.4 s. Taking into account the coefficients of arene enrichment with the concentrator, the ratios of peaks of substances recorded from the concentrator differ by no more than 10% for peaks recorded by direct injection of substances from Microgas, which indicates the acceptable effectiveness of the TD method with gas dynamic insulation of IC and PCC during heating of the concentrator and entering the sample into the column.

The EKHO-FID-PC GC with the TD principle described above-entering arene samples from a tubular concentrator [20] is used in field work on the geochemical search for hydrocarbon deposits [27]. A similar principle of TD-input of hydrocarbon samples from a tubular

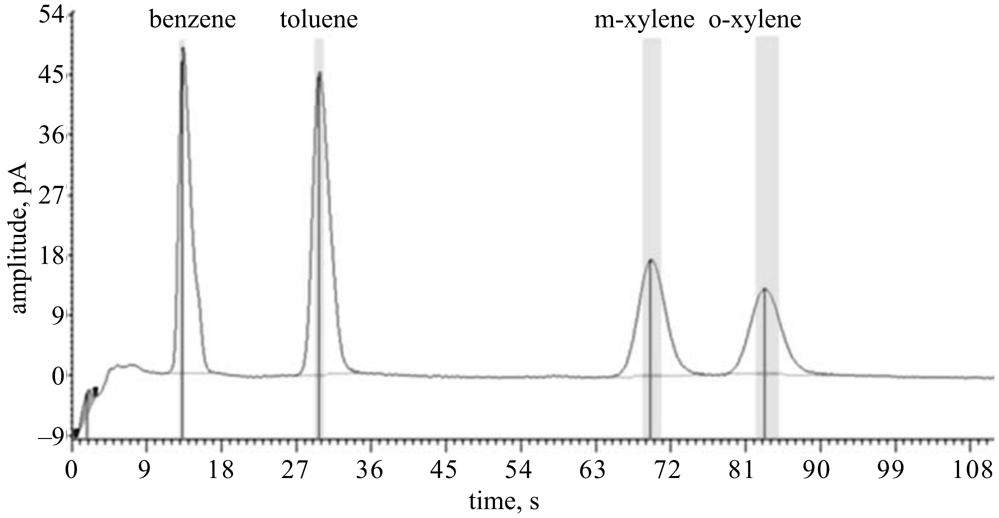

Fig. 6. Chromatogram of the Microgas sample. The type of the input device (injector) is a loop dispenser. The temperature of the injector is 200°C, the column is 40°C, and the detector is 120°C.

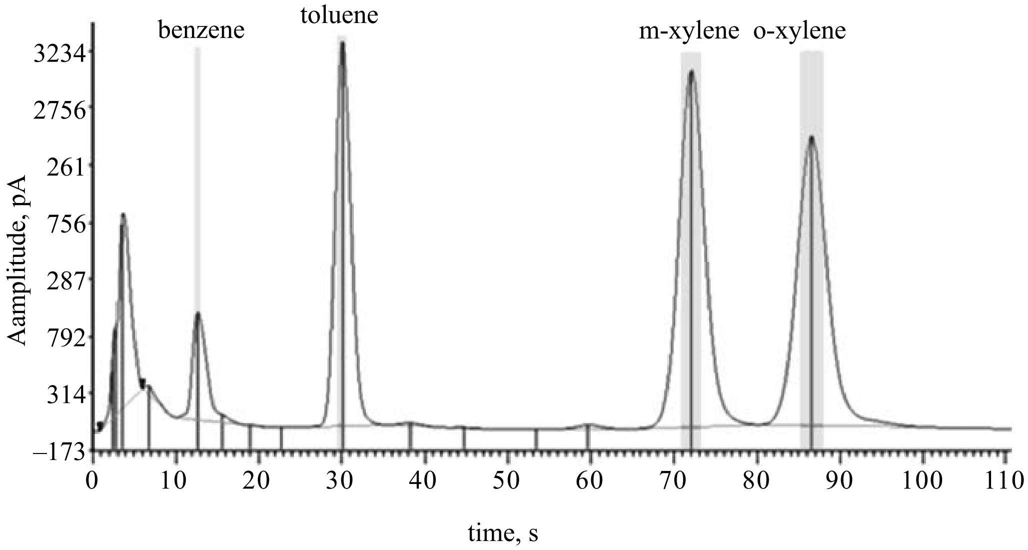

Fig. 7. Chromatogram of the sample from the concentrator. The temperature of the injector is 200°C, of the column is 40°C, and of the detector is 120°C. The heating time of the concentrator is 33 s, the sample input time is 2.4 s.

concentrator without manual operations is implemented in the analogue GC GHS-02PN (SIBEL LLC, Russia) [28]

Sample insertion with complete isolation of the input chamber and column during heating of the concentrator. The input is implemented in a portable EKHO-PID GC with a capillary column, a flame ionization detector and a helium carrier gas. The input scheme uses thermal desorption from a bulk sorbent (concentrator) of substances with sharply varying volatility, such as alkanes C1–C5, which are recorded in geochemical surveys when searching for hydrocarbon deposits [8, 9].

Alkanes C1–C5 vary greatly in volatility, their boiling points range from -161 °C to +36 °C, therefore, when the concentrator is heated, their evaporation rates vary greatly, which leads to a significant increase in evaporation time and unacceptable sample injection time if it is directly introduced into the column during desorption. In this case, thermal desorption is carried out in closed volumes, and a manually operated metering valve is used to insert the desorbed sample into the column [21, 22], which increases the bulkiness of the portable device and significantly reduces its speed, which is unacceptable in the field. To reduce the duration of the alkane sample input from the concentrator, two-stage thermal desorption (cryofocusing) is used [29] with a rather complex technical implementation of sorbent cooling. Cryofocusing is successfully used for the injection of explosives into the capillary column, which vary greatly in volatility (concentration of saturated vapor)

[30], and other organic substances [31]. Cryofocusing technologies effectively provide a significant reduction in the duration of sample insertion into the separation column of substances with significantly different volatility. However, such technologies are difficult to implement in portable devices.

For a portable device, an original sample input scheme was proposed in [32], in which desorption from the sorbent occurs in a completely sealed IC volume. IC sealing during desorption is carried out by electromagnetic valves for switching carrier gas flows. The software control of the solenoid valve switching ensures that samples are entered without manual operations. A schematic diagram of this method of sample injection is shown in Fig. 8. To enter the sample, the concentrator *1* with the sample is mounted on the working rod (not shown in Fig. 8) of the sample input device. The rod, together with the concentrator *1*, is inserted into the hot IC of the samples *2*, sealed with sealing elements (not shown in Fig. 8). After installing the concentrator in IC *2*, the pressure in IC increases, and at the signal of the pressure sensor (**PS**) *3*, the TD cycle of sample injection from the concentrator into the GCC is started. At the beginning of the sample injection cycle, the concentrator *1* is heated and the analyzed sample is desorbed into IC. During the heating of the concentrator, valve *4* is open and valves *5-7* are closed, thereby sealing the IC *2* of the sample with concentrator *1*, which ensures the safety and representativeness of the sample. At the same time, the carrier gas enters the analyzer through valve *4*, passes through the GCC and detector, ensuring their normal operation during sample heating, while there is no carrier gas flow through the sample's IC sealed by valves. After the desorption time has elapsed (the time is set by the control unit for all valves, not shown in Fig. 8), valve *4* closes, and valves *6* and *7* open, and the desorbed sample in the form of a short-term “plug” from the IC through valve *6* is transferred by the carrier gas flow to the GC analyzer. After the input is completed, valve *7* closes, valves *4* and *5* open, and the carrier gas passes through valve *4* and is divided into two streams. One larger stream of carrier gas enters the GCC and detector, ensuring the separation of sample substances into GCC and detection, and the other smaller stream (due to the presence of a flow limiter in the channel of valve *5*, not shown in Fig. 8) passes through valve *6*, IC with a concentrator, valve *5* with a flow limiter and is discharged into atmosphere, thus purifying the IC and concentrator. After the end of the analysis, the working rod with the concentrator is removed from the IC sample for the next analysis.

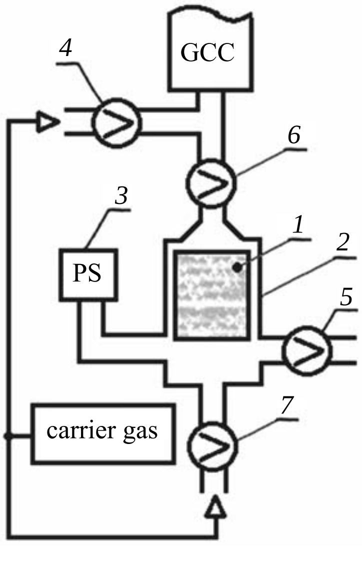

Fig. 8. Functional diagram of the sample input device from a volumetric concentrator. The designations are given in the text when explaining the operation of the scheme.

An example of the analysis of a mixture of light hydrocarbons with concentrations of 0.125 ppm (test gas mixture TU 6-16-2956-92) is shown in Fig. 9. Sampling for the concentrator was carried out by placing the concentrator in a 40 ml vial with a test gas mixture for 12 hours at room temperature. Analysis parameters: desorption temperature 270 °C, desorption time 1 min, injection time 4 s, flow rate of helium carrier gas through a chromatographic column 12 ml/min, column temperature 80 ° C.

The chromatogram in Fig. 9 with the separation of substances with greatly varying volatility from methane to butane demonstrates the satisfactory quality of thermal desorption injection without manual operations with complete sealing of the HF and software input control [32].

Consideration of the volatility of substances in the selection of a sample injection scheme into the column using thermal desorption. Let's consider the role of volatility using the example of desorption of explosive molecules from a flat mesh concentrator. With TD injection, two processes are implemented sequentially: desorption of substances at a given temperature of the concentrator and transfer (dosing) of the sample with a carrier gas into the column. The volatility of substances plays a role in both processes. Let's consider the process of desorption from the material of a flat mesh sorbent.

For the case of monomolecular TD from a flat sorbent material, we use the expression of the desorption rate of explosive molecules at a constant temperature T (K) [33]:

$$dN/dt = \gamma N \exp(-E/(RT)), \qquad (1)$$

where N is the number of molecules per sorbent surface unit, γ is the preexponential multiplier, E is the desorption energy, and $R = 8.314$ J/(mol·K) is the gas constant. Then the evaporation time of N molecules per unit surface of the sorbent can be estimated by the formula: $\tau_0 \exp(E_a/(RT))$, where τ_0 is the period of vibration of the molecules

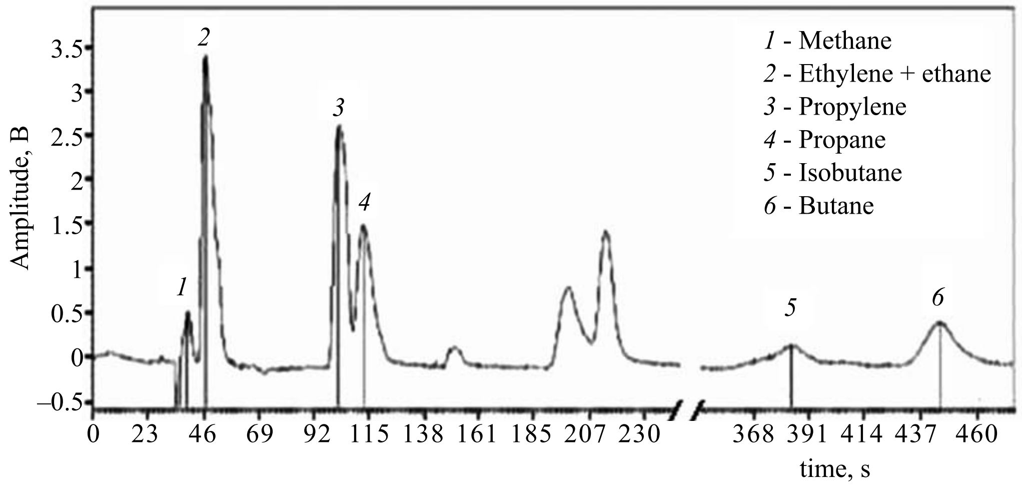

Fig. 9. Chromatogram of a test mixture with a hydrocarbon concentration of 0.125 ppm, selected by a concentrator from a 40 ml vial. Restek MXT-Q-PLOT column (30 m × 0.53 mm × 20 μm), column temperature 80 °C, flow rate through a gas chromatographic column 12 ml/min, flame ionization detector.

on the surface of the sorbent [16]. If we assume that during the desorption of NG and TNT, the values of t_0 for these substances are the same, then we obtain for the ratio θ the evaporation times of the molecules of these substances per unit surface of the concentrator:

$$\theta = \exp((E_{\mathrm{TNT}} - E_{\mathrm{NG}})/(RT)),$$

where E_{TNT} and E_{NG} are the desorption energies of TNT and NG molecules, respectively.

For E_{TNT} and E_{NG} from Table 1 and for $T = 453$ K we get $\theta = 3.8$. The small value of θ means that, despite the significant difference in the concentration of saturated vapor, the evaporation times from the material of the considered flat concentrator vary slightly. At the same time, we note that the experimental data in Fig. 3, illustrating the evaporation rate, confirm that the rates differ by no more than four times, which confirms the validity of the calculated estimate. This, in turn, also indicates the validity of our assumption about the small difference in the oscillation periods of NG and TNT molecules on the sorbent surface at the temperature under consideration.

To estimate the ratio of similar evaporation times of substances per unit area of sorbent materials in mixtures of arenes and alkanes under consideration, we use an approximate estimate θ for substances with extreme values of desorption energy: for arenes *o*-xylene–benzene (T = 473 K), for alkanes pentane–methane (T = 543 K). We obtain for arenes $\theta_{\mathrm{ar}} = 4.6$, for which gas-dynamic insulation of IC and columns is applied, as well as for explosive; and for alkanes $\theta_{\mathrm{al}} = 117$, where full insulation of HF and columns is applied with TD input. Thus, the value θ is quite sensitive to the types of TD inputs described. Therefore, it is possible to introduce a criterion (a kind of invariant) θ as the ratio of the evaporation times of substances per unit surface of the concentrator material, by the value of which it is possible to determine the expediency of using TD-input of samples from a concentrator with partial or complete isolation of the input chamber and column. For example, at $\theta \leqslant 10$, it should be considered advisable to use the simplest technical design of the TD input circuit with partial hydrodynamic insulation of the input chamber, which is important for portable devices, gas dynamic insulation of IC and the column.

* * *

Methods of thermal desorption in portable gas chromatographs of the EKHO series for introducing samples of organic substances from concentrators into a gas chromatographic column without manual operations, depending on the range of volatility of substances in the samples, are considered. The possibility of using partial gas-dynamic isolation of the water chamber and column during heating of the concentrator is shown by the example of introducing vapors of explosives and arenas. The insulation is carried out by a counterflow of carrier gas relative to the direction into the column. With sharply varying volatility of substances, for example, for the introduction of alkanes C1–C5, complete isolation of the inlet chamber and column is used during heating of the concentrator using a system of programmatically controlled electromagnetic switches of carrier gas flows, i.e. without manual operations, as in the case of traditional metering valves. To assess the possibility of using the simplest gas-dynamic insulation of the inlet chamber and column as a criterion, the ratio of evaporation times of substances in the sample per unit surface of the sorbent material is proposed. If the ratio of evaporation times is no more than 10, gas-dynamic insulation is acceptable.

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CONFLICT OF INTERESTS

The authors of this paper declare that they have no conflict of interest.

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