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Chromatographic determination of organohalogen compounds in swimming pool water

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The purpose of the work was to search for a sorbent with good selective properties for water purification. For this purpose, we determined the organohalogen compounds in the water of a swimming pool. A method of gas-liquid chromatography was used, using packed and capillary chromatographic columns. To concentrate the impurities of the organohalogen compounds, an automatic vapour-phase dispenser was used. This established that the swimming pool water, taken in the evening, had a complex composition of components, including 33 organohalogens, which have different toxicities for the human body. These are classed as belonging to the first and second groups of hazards. Identification of the individual component composition was carried out by determining the relative retention times of the test compounds and standard samples of the organohalogen compounds dissolved in methanol. Out of the entire range of the organohalogen compounds contained in the swimming pool water it was determined that the highest concentration of chloroform was 0.024 mg/dm^3 and the highest concentration of carbon tetrachloride was 0.018 mg/dm^3. The total content of organohalogen substances in the swimming pool water was 0.370 mg/dm^3, which increases the risk of human diseases while the swimming pool is in operation. It is proposed to clean the water in the swimming pools from toxic impurities by an adsorption method using rocks containing zeolite from the Tatar-Shatrashan deposit, which are characterized by a sufficiently high sorption capacity in relation to the organohalogen compounds.

Keywords: chromatography, sorbents, sorption capacity, organohalogen compounds, zeolite.

Introduction

In connection with the trend of improving the physical health of the population in the cities of Russia, intensive construction of swimming pools of various types and sizes has been observed. Swimming pools are complex technical facilities. One of the most important parts of these facilities is the water treatment system, which should ensure the purification of the water from impurities up to sanitary standards. The source water in the swimming pools is supplied through the water supply system of the cities or towns or using the underground sources. The sanitary and hygienic quality of the water entering the swimming pool has been steadily decreasing recently [1].

One of the main methods for purifying swimming pool water from priority pollutants is the chlorination process, which results in formation of organochlorine and other compounds accompanying them, which are characterized by their high toxicity [2]. They are rated as being second-class hazards and their composition is determined by chromatography or mass spectrometry analysis methods. These organochlorine compounds are included in the lists of priority pollutants in many foreign countries. They are formed during the disinfection of natural waters with chlorine compounds [3].

To determine the organohalogen compounds with low-boiling points in water, gas chromatography analysis methods with packed or capillary chromatographic columns are used. Polymethylsiloxanes with phenyl or cyanopropyl groups, polyethylene glycols with different molecular weights, as well as polar sorbents are usually used as stationary phases [4]. To determine the impurities of organohalogen compounds, an electron-capture, flame-photometric, or a mass-selective detector are mainly used. The dosage of the sample into the injector of a gas-liquid chromatograph is carried out using various techniques. The direct injection of a water sample allows us to eliminate loss of the analysed components prior to the sample preparation procedure. At the same time, the water, should it get inside the chromatographic apparatus, has a negative effect on the structural materials of the equipment, contributing to the failure of sorbents and stationary liquid phases as a result of hydrolysis processes. Therefore, a water trap is usually installed in front of the chromatographic column, the trap is cooled to a low temperature, which leads to a condensation of the water vapours and an increase in the sensitivity of determination of the organohalogen compounds [5].

For gas chromatography and determination of the volatile organohalogen compounds in a water medium, the most optimal solution is the vapour-phase analysis method, which allows obtaining information on the qualitative and quantitative composition of the analysed mixture. The most widely used method is a static version of vapour-phase analysis, which is a relatively simple and fast method [6]. In the case of a multi-stage variant of vapour-phase analysis, a dynamic method is used, which involves extracting the volatile components from the analysed water sample under the influence of an inert gas stream. Subsequently, the analysable substances are concentrated in the trap and desorbed into an injector of the gas chromatograph by rapid heating [7]. The aim of the work was to determine volatile organohalogen compounds in swimming pool water in real-time, using gas chromatography with a high-efficiency capillary chromatographic column and vapour-phase dosing of the sample into the chromatograph injector. The choice of sorbents is due to their high sorption capacity, which are used to purify the swimming pool water from organochlorine compounds.

Experimental

The experimental part of the work was carried out on a Perkin Elmer Calrus 580 chromatograph with a flame ionization detector having a lower detection limit for normal paraffin hydrocarbons of $5 \cdot 10^{-12}$ g/s. The work was carried out in the isothermal mode in the temperature range from 40 to 120°C, with the temperature of the chromatographic column being controlled programmatically. We used a 30 m long quartz capillary with an inner diameter of 0.32 mm. The division of the flow was 1:50, filled with 1,4-bis (dimethyl-siloxy) phenylene (dimethyl polysiloxane) with a film thickness of the stationary liquid phase of 0.25 μM.

The temperature of the injector was 110°C. The analysis was carried out at an initial temperature of the column of 35°C with an increase up to 200°C at a rate of 10 °C/min and exposure at this temperature for 6.5 minutes. Nitrogen with a flow rate of 1 ml/min through a capillary column was used as a carrier gas. The total analysis time of the volatile organohalogen components did not exceed 25 minutes.

A sampling of swimming pool water was carried out using vapour-phase analysis in a sealed container. The sample of water was sealed in a tightly sealed vial, placed in a thermostat, and heated to a specific temperature. Upon reaching the equilibrium between the liquid and a vapour phases, the sample was taken and dosed in the chromatographic column of a gas-liquid chromatograph.

A high-pressure sampling technique was used to dose the sample. With this technique, injection of the sample occurs when the internal pressure in the vial with the water sample is greater than the pressure in a head of the sampling module. When moving the sampling needle, the sample expands and enters the capillary of the transmission line. To carry out the process of sampling at high pressure, a necessary condition is that the pressure in the head of the column is higher than the internal pressure of the vapours in the vial containing the water sample. The gas-carrier was supplied through a module of the programmable pressure control for a vapour-phase dosemeter, and the module automatically increased the pressure from P_1 to P_2 at the start of the hydraulic pressure testing. After the vial was ventilated for a certain amount of time and the module returned the pressure to P_1.

Using vapour-phase analysis, only volatile compounds can enter the chromatographic column, while non-volatile components remain in the sample vial. Using the vapour-phase analysis method, the samples of the swimming pool water containing organohalogen compounds were analysed.

The vial containing the sample was shaken for 30 min. The time of pressure equalization was 3 min. The duration for needle insertion was 0.2 min. The thermostatically controlled temperature of the vial with the sample was 70°C. The temperature of the needle was 90°C. The temperature of the connecting line was 110°C. The duration of sample dosing was 0.15 min.

Fig.1. shows a detailed diagram of the water sample taken from the swimming pool. As can be seen from the figure, the analysed water sample fell into the vial of the vapour-phase dosimeter, which was thermostatically controlled to a temperature of 70°C. After that the system was pressed, which caused the sampling needle moves to the lower position, piercing the septum in the bottle cap. The vial was then pressurized with a gas-carrier at a pressure of P_2.

After the hydraulic pressure-testing phase, the valves of the solenoids V_1 and V_2 were closed, stopping the flow for the carrier-gas. The compressed gas from the vial flowed into the column, i.e. the taking of a water sample was carried out. After the preselected injection time, the valves of the solenoids V_1 and V_2 opened again, completing the sampling phase. The carrier-gas entered the column and was cut off from the vial containing the sample. This prevented additional sample vapours from entering the column. At the end of the injection time, the needle moved upward so that the analysed water stopped flowing from the vial. The system was once again subjected to a hydraulic test with the pressure P_2. After reaching equilibrium, the sample vial was hydraulic pressure tested, during which the vial was put under pressure equal to that of the gas-carrier. The excess gas pressure was vented through the needle cleaning port.

To obtain good reproducibility of the results, it is important that the pressure in the head of the column is greater than the pressure in the vial with the sample. At the beginning of the hydraulic pressure-testing phase, when the needle is lowered into the

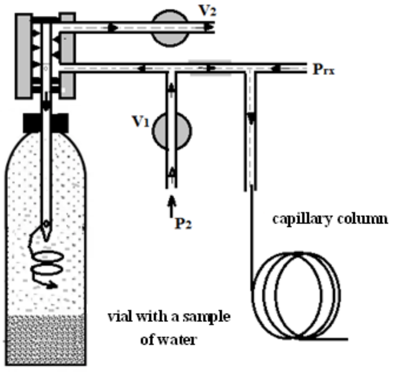

Fig. 1. Scheme of vapor-phase dosing of a water sample into an injector of the chromatograph. Hydraulic pressure testing. V_1 and V_2 are valves of the solenoids.

vial, the pressure should be reduced. A part of the pressure distributed between the injection port and the head of the column should be released into the atmosphere through an opening located at the bottom of the needle. Thus, if a difference in the pressure between the head of the column and the vial is very small, then part of the sample in the vial, existing as a vapour phase, will enter the stream of the gas-carrier before actual injection is performed, which would lead to an undesirable double injection. In order to prevent this, the pressure in the head of the column should be greater than the pressure in the vial at a pre-selected temperature of the thermostatic control, at least 1.2 times +20 kPa (100 kPa = 1 bar). When using capillary columns, the hydraulic pressure testing time was up to 3 minutes, which made it possible to achieve uniformity of the gas phase and good reproducibility of experimental data.

To reduce the degree of pollution, we purified the swimming pool water by adsorption methods. For this purpose, an adsorbent based on rocks containing zeolite from the Tatar-Shatrashan deposit were prepared. To obtain the adsorbent, the rocks were ground, the powder was mixed with a burnable additive and a water-based solution of plasticiser was used as a binder. The mixture was then subjected to heat treatment followed by hydrothermal crystallization in an alkaline solution. The formation of granules and their drying was carried out in accordance with the work presented in [6].

The adsorbent thus obtained was loaded into a chromatographic column, and its sorption properties were determined by ascending liquid column chromatography.

For the purification of swimming pool water from organochlorine compounds, synthetic or natural zeolites can serve as promising sorbents, which are characterized by their high sorption capacity in relation to organic compounds of various physicochemical natures. In the Republic of Tatarstan a large deposit of natural zeolites exists at the Tatar-Shatrashan deposit. It is interesting to study the sorption properties of these zeolites in relation to the organochlorine compounds that pollute the aquatic environment of swimming pools, since in these sports facilities there is direct human contact with these pollutants [8].

To assess the sorption properties of zeolite-containing rocks of the Tatar-Shatrashan deposit, we used the ascending liquid column chromatography method. For this purpose, we prepared chromatographic columns made of silicate glass with a length of 120 mm and an internal diameter of 4 mm. The column was filled with the studied sorbent, which was previously subjected to mechanical activation in accordance with [9]. The column was fixed on both sides with a glass fibre with a length of 10 mm. The determination of the sorption capacity in relation to organochlorine compounds was carried out by the gravimetric method. Organochlorine solvents were placed in a vial, into which the working end of the chromatographic column was lowered. In this case, due to the energy of the capillary forces of the sorbent under study, the organochlorine solvent began to rise along the sorption layer. After every 10 mm, the sorption time of the organochlorine solvent was recorded until the slip at 100 mm. The kinetic curves of the retention time dependence on the length of the sorption layer were then plotted.

The relative standard deviation when measuring the mass fraction of organochlorine compounds was no more than 5% with five parallel definitions. At the same time, the achievable detection limits in the case of vapour-phase analysis for organochlorine compounds were from 8 to 2 μg/l, which is also consistent with the literature data [10].

Results and discussion

Table 1 shows the results of gas chromatography analysis of the swimming pool water before entering the distribution system. The water comes from the Volga river basin and is pre-disinfected with sodium hypochlorite in the Vodokanal system of Kazan city of the Republic of Tatarstan. As

Table 1. Results of the gas chromatographic analysis of the swimming pool water before entering the distribution system

Item No.	Substance	Formula	T_{boil}, °C	SanPiN norms, mg/dm^3	C, mg/dm^3
1	Chloroform	$CHCl_3$	63.6	0.200	0.098
2	Carbon tetrachloride	CCl_4	76.5	0.006	0.008
3	1.2-Dichloroethane	$ClCH_2CH_2Cl$	83.6	-	0.003
4	Trichlorethylene	$ClC{=}CHCl$	86.9	0.005	0.005
5	Bromodichloromethane	$CHBrCl_2$	90.0	0.030	0.009
6	Dibromochloromethane	$CHBr_2Cl$	120.0	0.030	0.007
7	Tetrachlorethylene	$Cl_2C{=}CCl_2$	121.4	-	0.002
8	Chlorobenzene	C_6H_5Cl	132.0	0061	0.006
9	1.1.2.2 -Tetrachloroethane	$Cl_2CHCHCl_2$	147.0	-	0.006
10	Pentachloroethane	Cl_2CHCCl_3	162.0	-	0.002
Total concentration of organohalogen compounds					0.146

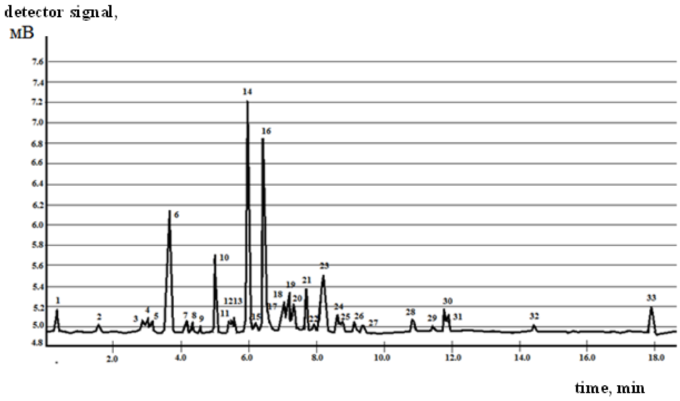

Fig. 2. Chromatogram of separation of the organohalogen compounds in the swimming pool water, obtained in the evening after the end of its operation. A chromatograph Perkin Elmer Calrus 680 with a flame ionization detector and a capillary column filled with 1,4-bis (dimethylsiloxane) phenylene dimethyl polysiloxane. Column length is 30m, inner diameter is 0.32 mm, film thickness of the stationary phase is 0.25mm. Mode: temperature programming. The order of the component exit corresponds to the data in table 2.

we can see in Table 1, the source water before entering the swimming pool is characterized by a high content of organohalogen compounds, especially chloroform (C=0.098 mg/dm^3). For many ingredients, the concentration of organohalogen compounds does not exceed SanPiN standards. However, the total content of organohalogen compounds is quite high (C=0.146 mg/dm^3).

During the operation of the swimming pool, it was of specific interest to determine a more complete composition of the organohalogen compounds, which are formed not only from anthropogenic sources, but also during water-healing procedures by humans. For this purpose, a water sample was taken in the swimming pool in the evening before it is closed at 21.00.

Identification of the analysed components was carried out based on the analysis of individual test substances, including state standard samples of organohalogen compounds dissolved in methanol.

The results of the analysis are given in Table 2. Figure 2 shows a chromatogram of the separation of the organohalogen compounds in the swimming pool water, which was obtained in the evening after the end of its operation.

Table 2. Results of the analysis of the swimming pool water after a completion of the water-healing process. Taking of the water samples at 21.00; T_{Tep}, °C is a temperature of the components exit during programming; t_{relat} is a relative retention time.

Item No	Substance	T_{boil}, °C	T_{Tep}, °C	t_{relat}, min	C, mg/dm^3	Hazard class
1	Difluorochloromethane	-40.8	35.0	0.05	0.001	4
2	Difluorodichloromethane	-29.8	35.0	0.21	0.01	4
3	Chloromethane	-24.2	43.7	0.41	0.01	2
4	Chlorethylene	-13.7	45.0	0.42	0.01	1
5	Chlorethane	12.3	48.9	0.49	0.01	4
6	Fluorodichloromethane	23.8	53.3	0.55	0.016	3
7	1.1-Dichloroethylene	31.8	57.5	0.61	0.003	2
8	2-Chloropropane	36.3	60.9	0.66	0.002	2
9	Dichloromethane	40.0	62.3	0.68	0.001	3
10	1-Chloropropane	46.9	65.0	0.72	0.012	2
11	Trans-1.2-Dichloroethylene	47.5	66.0	0.74	0.001	3
12	1.1-Dichloroethylene	57.3	72.1	0.81	0.001	2
13	Cis-1.2-Dichloroethylene	60.3	74.7	0.86	0.001	2
14	Chloroform	63.6	77.5	0.91	0.024	1
15	Bromochloromethane	70.0	81.9	0.96	0.001	2
16	Carbon tetrachloride	76.5	84.2	1.00	0.018	2
17	Benzene	80.0	86.1	1.03	0.001	2
18	1.2-Dichloroethane	83.5	88.0	1.05	0.002	1
19	Trichlorethylene	86.9	92.9	1.12	0.003	3
20	Bromodichloromethane	90.0	94.3	1.14	0.002	1
21	1.2-Dichloropropane	96.4	96.2	1.18	0.006	3
22	Bromtrichloromethane	104.7	97.3	1.21	0.001	2
23	Chlorpentane	107.9	99.0	1.25	0.008	2
24	2-Methyl-1.2-dichloropropane	108.0	101.8	1.29	0.002	3
25	1.1.2-Trichloroethane	108.0	104.3	1.31	0.001	2
26	Toluene	111.0	105.5	1.39	0.001	3
27	Dibromochloro-methane	120.0	113.1	1.41	0.001	2
28	Tetrachlorethylene	121.4	114.3	1.43	0.002	2
29	1.1.1.2-Tetrachloroethane	130.5	118.3	1.49	0.001	3
30	Chlorobenzene	132.0	123.6	1.56	0.003	2
31	Dibromodichloro-methane	135.0	124.2	1.58	0.001	2
32	1.1.2.2 -Tetrachloroethane	147.0	161.2	2.11	0.001	3
33	Pentachloroethane	162.0	194.4	2.59	0.003	2
Total concentration of organohalogen compounds					0.370	

As we can see in Table 2 and Fig. 2, the elution order of the components from the chromatographic column is determined by their boiling points. Moreover, the concentration of the individual components is small, but the total content of the organohalogen compounds is 0.370 mg/dm^3. In addition, the analysis revealed some organohalogen compounds belonging to the first and second hazard classes, which increase the risk of diseases in the human body.

Fig. 3 shows the dependence of the retention time (t_{10}, min) of organochlorine compounds on the length of the sorption layer of rocks containing zeolite of the Tatar-Shatrashan deposit. As we can see in the figure, the elution order of the individual components of the organochlorine compounds is not determined by their boiling points and it is associated with a nature of the intermolecular interaction in the system sorbate-sorbent. For example, trichlorethylene has a higher boiling point (86.2°C)

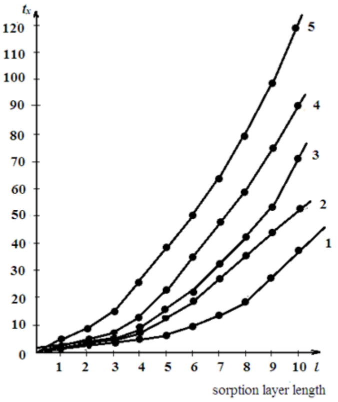

Fig. 3. Dependences of retention time of the organochlorine solvents on the length of the sorption layer of zeolite-containing rocks of the Tatar-Shatrashan deposit 1 – Trichlorethylene; 2 – Tetrachloromethane; 3 – Chloroform; 4 – 1,2-Dichloroethane; 5 - Benzyl chloride

Table 3. Physico-chemical and sorption properties of organochlorine compounds on various adsorbents (T_{boil}, °C is a boiling point; t_{10},min – retention time of the sorbat along the length of the sorption layer; A, %mass is a sorption capacity.

Item No.	Substance	T_{boil}, °C	μ	Synthetic zeolites NaX-13A		Natural zeolites	
				t_{10},min	A, %mass.	t_{10},min	A, %mass.
1	Chloroform	63.6	1.65	87	96	69	124
2	Carbon tetrachloride	76.5	0	67	84	54	155
3	1,2-Dichloroethane	83.6	1.75	61	97	88	66
4	Trichlorethylene	86.9	1.46	70	90	38	119
5	Benzyl chloride	179.0	1.15	112	86	117	142

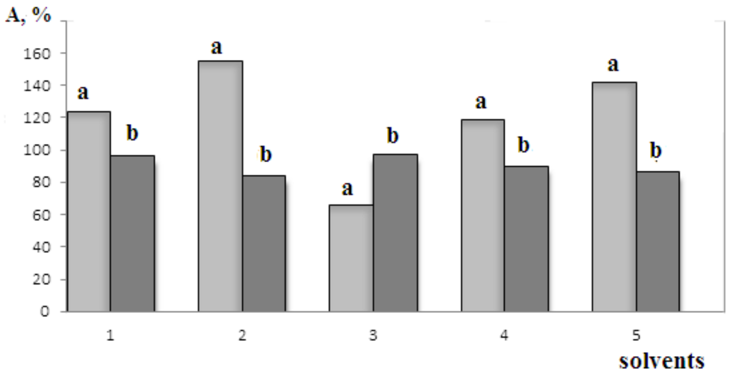

Fig. 4. Dependence of a dynamic sorption capacity of the organochlorine solvents on a nature of the adsorbent (a- NZ - natural zeolite; b - NaX - synthetic zeolite).
1 – chloroform, 2 – carbone tetrachloride, 3 – dichlorethane, 4 – trichloroethylene, 5 – benzyl chloride

than chloroform (63.5°C), dichloroethane (83.6°C) and carbon tetrachloride (76.5°C), and in the case of a non-polar adsorbent it should come out later than these components. However, it is eluted first from the chromatographic column, which is determined by its structural features.

Table 3 shows the physicochemical and sorption properties of organochlorine compounds on the various adsorbents.

As we can see in the table, unlike the NaX-13A synthetic zeolite, natural adsorbents, obtained from rocks containing zeolite from the Tatar-Shatrashan deposit, are characterized by a higher sorption capacity regarding almost all studied sorbents except 1,2-Dichloroethane.

Fig. 4 shows the dependence of the dynamic sorption capacity of the organochlorine compounds on the nature of the adsorbent.

As we can see in the figure, the adsorbents based on rocks containing zeolite from the Tatar-Shatrashan deposit have a higher sorption capacity than synthetic zeolites with respect to chloroform, carbon tetrachloride, trichlorethylene, and benzyl chloride. For 1,2-Dichloroethane, this dependence is not observed.

Conclusions

Based on the gas chromatography analysis of the swimming pool water in real time, it was found that it is polluted with organohalogen compounds, which have a negative impact on the human body and increase the risk of serious diseases. It is shown that the rocks containing zeolite from the Tatar-Shatrashan deposit are a promising sorbent for the purification of swimming pool water from organohalogen compounds. It was established that the sorption capacity of organochlorine compounds is higher for sorbents obtained from the rocks containing zeolite from the Tatarsko-Shatrashansky deposit in comparison to the NaX-13A synthetic zeolite, which is explained by the structural features of the materials under study. The dependences connecting the retention time of organochlorine solvents obtained under the conditions of the ascending version of column chromatography with the length of the sorption layer of the zeolite-containing rock of the Tatarsko-Shatrashansky deposit are found.

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Определение летучих галогенорганических соединений в воде плавательных бассейнов методом газо-жидкостной хроматографии

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Цель настоящей работы состояла в поиске оптимального по селективности сорбента для очистки воды. Для этого проводили определение летучих галогенорганических соединений в воде плавательного бассейна в период его эксплуатации методом газо-жидкостной хроматографии с использованием насадочных и капиллярных хроматографических колонок. Для концентрирования примесей галогенорганических соединений использовали автоматических парофазный дозатор, с помощью которого установлено, что вода плавательного бассейна, отобранная в вечернее время, имеет сложный компонентный состав, включающий 33 галогенорганические вещества, обладающие различной токсичностью для организма человека и относящиеся в основном к первой и второй группе опасности. Идентификацию индивидуального компонентного состава проводили на основе определения относительных времен удерживания тестовых соединений и государственных стандартных образцов галогенорганических соединений, растворенных в метаноле. Обнаружено, что из всего ассортимента галогенорганических соединений, содержащихся в воде плавательного бассейна, наиболее высокая концентрация отмечена для хлороформа 0.024 мг/дм^3 и тетрахлорметана 0.018 мг/дм^3. Общее содержание галогенорганических веществ в воде плавательного бассейна составляет 0.370 мг/дм^3, что повышает риск возникновения заболеваний человека в процессе его эксплуатации. Предложено очистку воды плавательных бассейнов от токсичных примесей проводить адсорбционным методом с использованием цеолитсодержащих пород Татарско-Шатрашанского месторождения, которые характеризуются достаточно высокой сорбционной ёмкостью по отношению к галогенорганическим соединениям.

Ключевые слова: хроматография, сорбенты, сорбционная способность, галогенорганические соединения, цеолиты.

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