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INFLUENCE OF CHROMATOGRAPHY CONDITIONS ON THE RETENTION OF SOME ISOQUINOLINE AND PYRIMIDINONE DERIVATIVES

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Abstract:

In this work, the chromatographic retention of pyrimidinone and isoquinoline derivatives under reversed-phase HPLC conditions was studied. Retention and separation factors were determined for various eluent compositions (CH3CN and H2O). The dependence of retention on the concentration of the organic modifier in the mobile phase was studied. In addition, studiedretention of the studied components depending on the nature of the substituents and their mutual arrangement.

Keywords: Is quinolines, pyrimidinones, RP HPLC, separation factor, sorbate, retention factor, eluent flow, acetonitrile, substituent nature.

INTRODUCTION

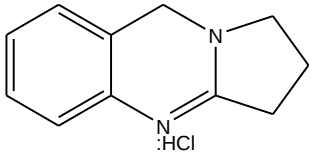
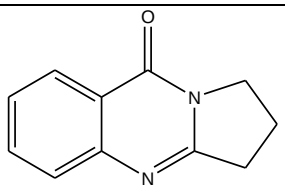
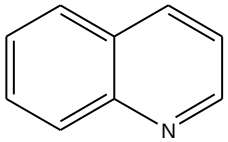
Currently, high performance liquid chromatography is one of the most promising methods used for the analysis of biologically active substances.in particular isoquinolines,as well as to identify the relationship "structure-property-activity". Many heterocyclic compounds in particularisoquinolines are widely used in biochemistry and medicine inas antioxidants, inhibitors, they are part of a number of natural substances and drugs that have antihypertensive, antibacterial, antiviral, antitumor and other types of pharmacological action [1]. Therefore, the development of new methods of synthesis, the study of their composition and properties is relevant. In the theory of liquid chromatography, a significant number of retention models are currently known that relate the parameters of a chromatographic system to various electronic and physicochemical characteristics of sorbates, such as polarizability, van der Waals volume or surface area of molecules, hydrophobicity factor, molecular bonding indices, molecular weight or boiling point, etc.

The results of HPLC analysis make it possible to control the purity of preparations and predict the properties of compounds based on the chromatographic behavior of sorbates.

The aim of our work was to studyinfluence of chromatographic conditions on the retention of some derivatives of pyrimidinone and isoquinoline series and eluent composition.

The objects of study were pyrimidinones and derivatives of the isoquinoline series, the formulas of which are presented in Table. 1.

Table 1.
Names and structural formulas of the studied sorbates[3]

No.	Structural formula	Name
1		Deoxypeganine hydrochloride
2		Deoxyvasicinone
3		Quinoline

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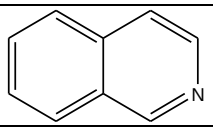
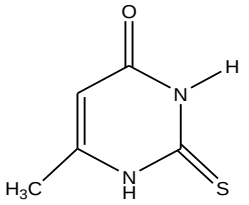
4		isoquinoline
5		2-thio-6-methylpyrimidinone

Patterns of retention of nitrogen-containing heterocyclic compounds, in particular, derivativespyrimidinone and isoquinoline derivativesinvestigated by HPLC.

It is known that in this variant of chromatography, specific intermolecular interactions with the mobile phase play a decisive role in the retention of sorbates, since in this case only weak dispersion interactions will be observed between the sorbent and sorbate [2].

The experiment was performed on an Agilent 1200 Series Rapid Resolution LC System liquid chromatograph with a UV detector. Detection was carried out at a wavelength of 254 nm. The sorbent was Exlipse XDB C-18, particle size 5 μm. The dimensions of the chromatographic column are 4,0x250 mm. A mixture of acetonitrile and water with an acetonitrile content of 15 to 40% (by volume) was used as eluents. Distilled water and acetonitrile were used to prepare the mobile phase. The eluent flow rate was 400 μl/min. Solutions of sorbates (concentration 10-4 mol/l) were prepared by dissolving individual samples in the appropriate mobile phase; the sample was injected in an amount of 20 ml.

Holdingderivativespyrimidinone and derivatives of the isoquinoline series were characterized by the value of the retention factor k

$$k = (t_R - t_M)/t_M ,$$

where t_R and t_M are the retention time of the sorbate and non-sorbable component, respectively;
The data obtained are presented in table. 2.

To study the influence of the nature of the mobile phase on chromatographic retention, a mixture of acetonitrile with water in different volume ratios (60:40, 70:30, 75:25, 80:20, 85:15) was used as an eluent [4].

The retention factors of the studied compounds are presented in Tables 2, respectively.

Table *2*

Retention times and factors of the studied sorbates using various mobile phases

No.	Mobile phases (H_2O/CH_3CN)									
	60/40		70/30		75/25		80/20		85/15	
	t_R, s	k	t_R, s	k	t_R, s	k	t_R, s	k	t_R, s	k
1	185,2	0,066	196,0	0,108	202,4	0,148	208,6	0,187	232,3	0,262
2	193,1	0,102	202,0	0,130	208,4	0,182	220,5	0,254	256,7	0,408
5	190,4	0,092	204,0	0,141	209,6	0,190	221,4	0,262	268,0	0,467
3	203,4	0,177	215,7	0,208	220,4	0,256	238,0	0,352	315,0	0,736
4	232,1	0,322	254.0	0,432	316,4	0,796	390,2	1,212	806,6	3,426

*The numbering of sorbates is given in accordance with Table. 1.

Table 3

Change in retention factors depending on the nature of substituents (mobile phase CH_3CN/H_2O – 15/85 vol.%)

p/n	Compound name	retention factor k
1	Isoquinoline	0,282
2	Deoxyvasicinone	0,546
3	2-thio-6-methylpyrimidinone	0,498

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From the analysis of the table. It can be seen from Table 2 that the system with a volume content of the organic modifier (CH_3CN) in the eluent equal to 15% has the best selectivity to the compounds under study.

From the table in Table. 3 it can be seen that pyrimidinone derivatives leave the column more slowly than derivatives of the isoquinoline series. This is obviously due to the fact that the molecule contains an additional 6п electron system, which increases the dispersion interactions with the octadecyl groups of the stationary phase.

It has been established that with an increase in the water content in the mobile phase, the retention time of the studied substances increases, which is explained by the solvophobic retention theory. It has been experimentally shown that when the content in the eluent is 40% acetonitrile sorbates elute faster from the column, which is accompanied by a reduced selectivity of such a chromatographic system for the compounds under consideration. As can be seen from table 2, an increase in water concentration from 60 to 85% leads to an increase in the retention characteristics and high selectivity of such a system to the compounds under study.

Analyzing table 2, it can be seen that at a constant concentration acetonitrile in the eluent, the retention strongly depends on the nature of the substituents and on their relative position.

Study of the Sorption of Nitrogen-Containing Certain Derivatives of Pyrimidinone-4 and Isoquinoline by HPLC on a Nonpolar Sorbent showed that this process proceeds most selectively a the ratio of the components of the binary mobile phaseacetonitrile/water (15/85% vol.) in isocratic mode.

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