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Possible Optimization of Separation Conditions for Au III, Se IV and Te IV Ions by Thin-layer Chromatography with the Use of Different Supports

Możliwości optymalizacji warunków rozdziału jonów Au III, Se IV i Te IV metodą chromatografii cienkowarstwowej z zastosowaniem różnych nośników

Возможности оптимализации условий разделения ионов Au III, Se IV и Te IV методом тонкослойной хроматографии при использовании разных носителей

Sensitive, quick and simple TLC methods for the investigation of inorganic ion mixtures are frequently used for analytical studies. In process dealing with industrial methods of Au recovery from deposit it often happens that the separation of Au, Se and Te ions, that appear together in a mixture, is needed.

According to the theory, the behaviour of Au, Se and Te ions in inorganic acids solutions as well as the previously investigated [1] possibilities for separation of the ions under discussion on Al_2O_3 thin-layers lead to the following

conclusion: differences in R_F values and Au, Se and Te ion diffusion in solutions of HCl, HBr and NH_4SCN are explained by the process of complex formation of the ions under investigation in given solutions and the ability of Al_2O_3 for sorption of the complex ions that have been formed.

Thus, the mechanism of ion separation on Al_2O_3 is associated with processes of sorption and complex formation, and the separation of ions under investigations depends upon sorption of the complex ions that have been formed and the difference in stability constants of the complexes formed in moderately and strongly acid solutions.

In this paper there have been presented the results obtained from the investigations of the establishment of optimal separation conditions, identification and determination of microgram amounts of Au (III), Se(IV) and Te(IV) ions by TLC method with the use of silica gel, glass powder and solutions of inorganic and organic acids or other organic solvents and base solutions as mobile phases.

Certain kinds of glasses possess cationexchange properties with respect to many inorganic acids. Therefore, it would be interesting to investigate possibilities for the application of glass powder as support for separation and identification of Au(III), Se(IV) and Te(IV) ions. It seems to be even more interesting when we point to the fact that glass has never been used previously as support in TLC method for inorganic ions analysis.

EXPERIMENTAL

The experiments have been conducted with the use of TLC adsorption method. As adsorbents were used KSK N^o 2,5 n silica gel; Al₂O₃ AB-17 and glass powder with the same parameters as in the case of remaining supports, together with their mixtures of different compositions. Solutions of the following organic acids have been used as mobile phases: acetic acid, chloracetic acid, trichloroacetic acid, oxalic acid, tartaric acid, citric acid and their potassium or sodium salts. There have been also used solutions of inorganic acids: HBr, HCl, HNO₃ and NaOH base as well as solutions of chosen organic solvents, that is, aliphatic alcohols: methanol, ethanol and buthanol. As mobile phases there have been also used different mixtures of the above mentioned solvents with different compositions.

In this paper there has been investigated the influence of thickness of adsorbent layer on chromatographic process as well. For this Al₂O₃ has been applied with different thickness of layer, i.e. 1.00; 0.50; 0.25 and 0.10 mm. Solutions containing Au(III), Se(IV) and Te(IV) ions have been put on plates covered with thin layer of adsorbent.

Chromatograms have been developed through ascending technique in Stahl's chambers on the distance of 10 cm. Chromatograms were visualized by spraying the plates first with the solution of tin dichloride in HCl and then with 50% solution of potassium thiocyanate.

RESULTS AND DISCUSSION

As it follows from the experiments that have been conducted fine glass powder weakly sorbs Au(III), Se(IV) and Te(IV) ions in acid environment. The absence of "tails" and high R_F values (0.95) of the ions investigated on supports of this kind confirms the above statement. In the first experimental series there have been investigated possibilities for application of water solutions of H_3PO_4 and HBr acids as well as mixtures solutions of HBr and n-butanol with different volume ratios. The basic conclusions that is drawn from the analysis of the data obtained (tab. 1) is the following: when glass powder is used as support in moderate and strong acid solutions, R_F values of the ions under investigations not

Tab. 1. Dependences between R_F values of Au(III), Se(IV) and Te(IV) ions and concentration of acid mobile phase on glass powder

Acids	Concentration mol/dm ³	R_F values of		
		Se IV	Te IV	Au III
HBr	0.05	0.40	0.00	0.00-0.14
	0.10	0.30-0.79	0.00	0.00-0.27
	0.20	0.35-0.80	0.00-0.18	0.00-0.31
	0.60	0.53-0.89	0.00-0.25	0.15-0.81
	1.00	0.95	0.95	0.95
	6.00	0.95	0.95	0.95
HNO ₃	0.10	0.47	0.47	0.00-0.32
	0.25	0.64	0.64	0.00-0.43
	0.50	0.80	0.80	0.00-0.57
	0.60	0.85	0.85	0.00-0.65
	1.00	0.96	0.97	0.98
	8.00	0.99	0.99	0.99
H ₃ PO ₄	0.05	0.11	0.00	0.00-0.16
	0.10	0.79	0.70	0.00-0.20
	0.25	0.85	0.80	0.00-0.29
	0.60	0.90	0.87	0.00-0.59
	1.00	0.95	0.88	0.96

depend upon the properties of the acids applied because of the absence of sorption of the obtained ion forms [2].

The data from tab. 2 show that the application of pure n-buthanol as mobile phase does not cause the migration:

Tab. 2. Dependences between R_F values of Se(IV), Te(IV) and Au(III) ions and mobile phase composition on glass powder

Volume ratios $\frac{\text{n-butanol}}{\text{HBr 6 n}}$	R_F values of		
	Se(IV)	Te(IV)	Au(III)
n-butanol	0.00	0.00	0.00-0.01
1:99	0.34	0.34	0.92
1:39	0.51	0.51	0.92
1:19	0.70	0.70	0.92
1:9	0.96	0.96	0.96
9:1	0.95	0.92	0.96
HBr 6 n	0.98	0.98	0.98

of Se(IV), Te(IV) and Au(III) ions. However, the addition of small quantities of HBr leads to the increase of R_F values, which is particularly visible in the case of Au(III) ions. In proper conditions Au(III) ions completely separate from Se(IV) and Te(IV) ions, and spots of the latter ones covered one another.

In order to increase the mobility differences of the ions there have been used mixtures of glass powder and Al_2O_3 in different compositions as supports. HCl and HBr solutions with different concentrations have been used as mobile phases. As one can see in tab. 3, on the thin layer of mixed sorbent in HCl environment, the spots of the ions under experiment take elongated shape due to great sorption properties of Al_2O_3 in relation to the ions, particularly to Se(IV). The same image is obtained in HBr solutions, but it is less clear.

Tab. 3. Dependences between R_F values of Se(IV), Te(IV) and Au(III) ions and sorbent compositions and concentration of HCl and HBr acids

Volume ratios glass powder	mobile phases	concentration (mol/dm ³)	R_F values for		
			Se(IV)	Te(IV)	Au(III)
Al ₂ O ₃					
9:1	HCl	0.60	0.00-0.11	0.00-0.09	0.00-0.22
		1.50	0.00-0.12	0.00-0.10	0.00-0.25
		3.00	0.00-0.15	0.00-0.21	0.00-0.46
		4.50	0.00-0.20	0.00-0.86	0.00-0.63
		5.25	0.00-0.21	0.00-0.85	0.00-0.81
		6.00	0.00-0.22	0.00-0.99	0.00-0.92
3:1	HCl	0.60	0.00-0.01	0.00-0.01	0.00-0.01
		1.50	0.00-0.02	0.00-0.02	0.00-0.02
		3.00	0.00-0.06	0.00-0.12	0.00-0.05
		4.50	0.00-0.07	0.00-0.57	0.00-0.27
		5.25	0.00-0.10	0.00-0.85	0.00-0.85
		6.00	0.00-0.08	0.00-0.91	0.00-0.91
3:1	HBr	0.60	0.00-0.04	0.00-0.03	0.00
		1.50	0.00-0.05	0.00-0.09	0.00-0.02
		3.00	0.00-0.05	0.00-0.75	0.00-0.88
		4.50	0.00-0.08	0.00-0.93	0.00-0.91
		5.25	0.00-0.09	0.00-0.95	0.00-0.94
		6.00	0.00-0.09	0.00-0.96	0.00-0.96

In each mixed supports composition and at different concentrations of mobile phase, the spots of all the ions cover one another. As it can be seen from the data presented in tab. 4 the addition of anionit to glass powder rapidly decreases R_F values of the ions due to the increase of sorption properties of supports. The greater the quantity of ion exchanger the stronger acid concentration is needed to displace Se(IV) and Te(IV) ions. Au(III) ions, in almost all mixed support compositions of glass powder and AB-17 anionit, remain at start line. This means that, due to sorption properties, the ions under investigations may be arranged as follows: Au(III) > Te(IV) > Se(IV). In the range of 0.1-0.5 n concentra-

Tab. 4. Dependences between R_F values of Se(IV), Te(IV) and Au(III) ions and sorbent composition and concentration of HBr acid

Volume ratios glass powder Al ₂ O ₃	HBr concentration	R_F values of		
		Se(IV)	Te(IV)	Au(III)
199:1	0.01	0.00-0.14	0.00	0.00
	0.05	0.00-0.57	0.00	0.00
	0.10	0.79	0.00-0.07	0.00
	0.25	0.81	0.47	0.00-0.09
	0.50	0.89	0.57	0.00-0.12
	1.00	0.89	0.89	0.00-0.12
99:1	3.00	0.00-0.06	0.00	0.00
	4.50	0.00-0.14	0.00-0.02	0.00
	5.25	0.00-0.20	0.00-0.07	0.00
	6.00	0.00-0.26	0.00-0.09	0.00
39:1	3.00	0.00	0.00	0.00
	4.50	0.00-0.20	0.00	0.00
	5.25	0.00-0.22	0.00-0.05	0.00
	6.00	0.00-0.23	0.00-0.10	0.00

tions of HBr, at volume ratio of AB-17 anionit in chloride form to glass powder being equal to 1:199, Au(III), Se(IV) and Te(IV) ions separate quantitatively. Au(III) ions are at a smallest distance from the start line, Se(IV) ions are nearest to the finish line and Te(IV) ions are between them. It is to be supposed that the application of anionit mixtures with glass powder or any other non-active supports will give new possibilities for separation of the ions under experiment, not only through TLC method but also by means of Liquid Column Chromatography.

The application of solutions of acetic acid ($K = 1.8 \times 10^{-5}$), chloroacetic acid ($K = 1.4 \times 10^{-3}$) and trichloroacetic acid ($K = 1.3 \times 10^{-1}$) as well as solutions of their salts is better in comparison with inorganic acids, because we do not

need a complementary organic solvent to replace the front of mobile phase and the spots we obtain during the investigation are not always broaded.

The relationships between R_F values of Au(III), Se(IV) and Te(IV) ions on thin layer of silica gel and the properties and concentration of mobile phase in systems of acetic and chloracetic acids are presented in tab. 5. As it can be seen Te(IV) ions remain at the start line in solutions of acetic acid and in its sodium salt. The phenomenon can be explained on the basis of sorption of TeO^{2+} and $TeOOH^+$ cations by silica gel which results from cationexchange properties of the support.

Te(IV) ions take greater R_F values with the increase of mobile phase acidity and the application of stronger chloracetic and trichloracetic acids. The R_F values are as greater as 0.86 (tab. 6) in 1 M solution of the latter.

The same phenomenon can be observed in the case of salt solutions of appropriate acids. It may be explained by the fact that the support changes into hydrogen form with the increase of acidity and as a result Te(IV) ions move together with the solvent front of mobile phase. Se(IV) ions behaviour is the same as Te(IV) ions behaviour, but the difference is that Se(IV) ions form a broaded spot that does not migrate from the start line in the environment of acetic acid and its sodium salt. Se(IV) ions spots take a compact shape when solutions of chloracetic and trichloracetic acids are used as mobile phases. The spot position on chromatographic plate does not depend on Se amount spread up to 0.008 mg. In the environment of 0.05-1 M trichloracetic acid the spots of

Tab. 5. Dependences between R_F values of Se(IV), Te(IV) and Au(III) ions and nature and composition of mobile phase on silica gel KSK N° 2,5 n

Mobile phases	concentration mol/dm ³	R_F values of		
		Se(IV)	Te(IV)	Au(III)
CH ₃ CO ₂ H	0.0001	0.00-0.47	0.00	0.99
	0.10	0.00-0.49	0.00	0.99
	1.00	0.00-0.48	0.00	0.98
ClCH ₂ CO ₂ H	0.0001	0.00-0.37	0.00	0.66
	0.01	0.12-0.44	0.00	0.99
	0.10	0.10-0.45	0.00-0.13	0.99
	0.25	0.26-0.54	0.00-0.27	0.89
	0.50	0.72	0.15-0.43	0.88
	1.00	0.43-0.70	0.08-0.50	0.88
CH ₃ CO ₂ Na	0.0001	0.00-0.34	0.00	0.99
	0.10	0.00-0.45	0.00	0.87
	1.00	0.00-0.72	0.00	0.89
ClCH ₂ CO ₂ Na	0.0001	0.00-0.25	0.00	0.98
	0.01	0.00-0.29	0.00	0.98
	0.10	0.00-0.40	0.00	0.98
	0.25	0.28-0.74	0.00	0.93
	0.50	0.29-0.65	0.00	0.99
	1.00	0.29-0.65	0.00	0.99

Tab. 6. Dependences between R_F values of Se(IV), Te(IV) and Au(III) ions and Cl₃CCO₂H acid concentration on silica gel KSK N° 2,5 n

Cl ₃ CCO ₂ H concentration mol/dm ³	R_F values of		
	Se IV	Te IV	Au III
0.0001	0.00-0.56	0.00	0.99
0.001	0.00-0.46	0.00	0.99
0.01	0.00-0.30	0.00-0.07	0.99
0.05	0.37	0.00-0.21	0.99
0.10	0.40	0.00-0.47	0.99
0.25	0.85	0.00-0.70	0.81
0.50	0.86	0.86	0.81
1.00	0.86	0.86	0.89

Se(IV) ions are compact and their R_F values are in a range of 0.37-0.86 accordingly (tab. 6).

The behaviour of Au(III) ions is completely different from that of Se(IV) and Te(IV) ions. In mobile phases that have been applied, depending upon the kind of acid and its concentration, Au(III) ions may be in different anions forms and thus they migrate along silica gel plate almost together with the front of mobile phase (tab. 5 and 6):

The mechanism of ion separation on thin layer of silica gel is of ionexchange character, particularly in the solutions in which dissociate function groups have not been blocked yet.

Ion separation begins to proceed according to sorption mechanism as a result of the increase of mobile phase acidity. According to its sorption properties in relation to the ions under investigation, silica gel takes the middle position between Al_2O_3 and glass powder.

Due to the strong sorption of all ions on Al_2O_3 in acetic acid environment, it has been impossible to make a complete separation of these ions. Se(IV) and Te(IV) ions remain at the start line in all phases that are used, whereas Au(III) ions while slowly reduce to metal form broaded spots with their beginning at the start line. The same behaviour is observed in the case of the remaining three ions when we apply solutions of chloracetic and trichloracetic acids and solutions of their sodium salts as mobile phases [3,4].

When Al_2O_3 is applied as support and citric acid as mobile phase, Se(IV) and Te(IV) ions migrate only slightly, they practically remain at the start line. All the ions that are investigated remain motionless at the start line in tartaric and oxalic acids.

Au(III) ions reduce to metallic form in concentrated solutions of oxalic acid, tartaric and citric acids on Al_2O_3 layer, Te(IV) ions always remain on start, Se(IV) ions migrate slightly. On the other hand, R_F values for Au(III) ions become smaller together with the decrease of salt concentration in mobile phase.

Au(III) ions spots are broadened on chromatogram for all concentrations of the salting component of mobile phase. Au ions migration is weakest one in citrate potassium solutions. In concentration ranging from 0.001 M to 0.0001 M of acid Au(III) ions remain on start, and at concentrations of 0.05 and 0.25 M R_F values for Au(III) ions are 0.008 and 0.003, respectively. It follows from the experiments that if Al_2O_3 is used as support and solutions of oxalic, tartaric and citric acids and their potassium salts solutions are applied as mobile phases, then the separation of Au(III), Se(IV) and Te(IV) ions is not achieved. When we use NH_4OH solutions as mobile phases and Al_2O_3 as support, we get Se, Te and Au ions that are motionless at the start line. At a greater concentration of NH_4OH (17,5 M) only Se(IV) and Te(IV) ions move, but the R_F values corresponding to the ions are 0.06 and 0.02, respectively. When we use water solutions of NH_4OH as mobile phases, Au(III) ions remain on start. On the other hand, R_F values increase for Se and Te ions together with the growth of base concentration.

When 0.6 M solution of NaOH is used, R_F values for Se(IV) and Te(IV) ions are 0.58 and 0.42 respectively. At a concentration of 4 M NaOH, R_F values for the ions are the greatest ones, i.e., they are equal to 0.86 and 0.71 respectively. The further increase of base concentration does not change R_F values of

the ions under investigation and it makes Al_2O_3 layer fluffly. Thus, the greatest differences in R_F values for Se and Te ions are obtained at concentration range of 0.3-0.6 M NaOH.

To make the separation conditions of Au(III), Se(IV) and Te(IV) ions optimal there have been used as mobile phases monohydroxy aliphatic alcohols (methanol, ethanol and buthanol) in mixtures with 3 M solution of NaOH in different volume ratios. The addition of methanol, ethanol and buthanol to NaOH solution decreases R_F values of Se(IV) and Te(IV) ions (tab. 7).

Tab. 7. Dependences between R_F values of Se(IV), Te(IV) and Au(III) ions and mobile phase composition

Alcohol	Volume ratio alcohol	R_F values of	
	3 M NaOH	Se(IV)	Te(IV)
methanol	1:0	0.00	0.00
	1:1	0.00	0.19
	1:3	0.54	0.52
	1:9	0.76	0.73
ethanol	1:0	0.00	0.00
	3:1	0.05	0.05
	1:1	0.35	0.35
	1:3	0.72	0.67
buthanol	1:0	0.00	0.00
	3:1	0.00	0.00
	1:1	0.36	0.34
	1:3	0.66	0.62
	1:9	0.69	0.65

At volume ratios of alcohol up to 3 M NaOH from 9:1 to 1:1 Se and Te ions practically do not migrate from the start line, and the further increase of base concentration leads to the same increase of R_F values of the ions.

There have been also investigated the mixture of ethanol and NaOH solution at the same volume ratio (1:1) but at

different concentrations of NaOH solution. The results obtained prove that there is no possibility for the quantitative separation of Se(IV) and Te(IV) ions because their spots cover one another in part. As supports there have been used mixtures of Al_2O_3 with glass powder as well as Al_2O_3 and silica gel with different weight ratios.

The data contained in tab.8 show that chemically pure silica gel possesses adsorption properties in relation to Se(IV) and Te(IV) ions. The decrease of R_F values for the ions under

Tab. 8. Dependences between R_F values of Se(IV) and Te(IV) ions and sorbent composition

sorbent composition	weigh ratio	mobile phase	R_F values of	
			Se IV	Te IV
Al_2O_3 : SiO_2	9:1	NaOH (6N)	0.56	0.45
	3:1		0.50	0.42
	1:1		0.36	0.29
	1:3		0.29	0.21
	1:9		0.21	0.21
Al_2O_3 : silica gel	3:1	NaOH (6N)	0.31	0.27
	4:1		0.37	0.38
	9:1		0.47	0.39
	19:1		0.53	0.46
Al_2O_3 : glass powder	9:1	NaOH 3N : NH_4OH : H_2O (10:25:65)	0.50	0.34
	3:1		0.54	0.39
	1:1		0.60	0.40
	1:3		0.78	0.58
	1:9		0.94	0.76
	1:19		0.96	0.82

Au (III) ions remain at the start line

investigation accompanied by the increase of silica gel amount in mixture with Al_2O_3 proves the above statements. Glass powder and KSK N^o 6N silica gel do not show any adsorption properties. Irrespective of the relatively great difference of R_F values for Se(IV) and Te(IV) ions, separation

and identification of the ions disturbed by the "tails" formed when the mixture of Al_2O_3 and glass powder (1:1) is used as support.

In order to do away with the unprofitable phenomenon the influence of the thickness of support layer upon chromatographic process has been studied [5].

The R_F values obtained for the ions with the use of Al_2O_3 with layer thickness of 1.0; 0.5; 0.25 and 0.10 mm as support and 3 M NaOH : 17.5 M NaOH : H_2O mixtures with different volume ratios as mobile phases are shown in tab. 9.

Tab. 9. Dependences between R_F values of Se(IV) and Te(IV) ions and sorbent composition and layer thickness

Mobile phase composition NaOH (3 n) : NH_4OH (17.5 n) : H_2O	layer thickness mm	R_F values of	
		Se(IV)	Te(IV)
10:25:65	1.00	0.47	0.32
	0.50	0.49	0.34
	0.25	0.50	0.34
	0.10	0.52	0.34
5:25:70	0.10	0.48	0.30
10:25:65	0.10	0.52	0.34
15:25:60	0.10	0.58	0.47
25:25:50	0.10	0.68	0.52

Au(III) ions remain at the start line

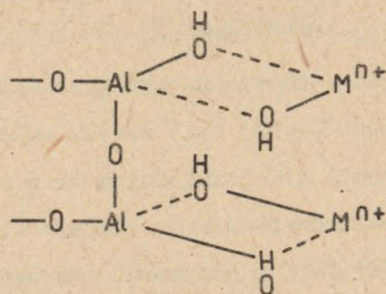
The data obtained show that the decrease in thickness of Al_2O_3 support layer leads to the slight increase of R_F values for Se(IV) and Te(IV) ions. There are "tails" on every chromatogram.

The investigation of relationship between spot area and Te(IV) and Se(IV) ion concentration shows that TLC method is useful in base systems for separation and identification of Au(III), Se(IV) and Te(IV) ions, and in some cases it is

helpful in semiquantitative determination of Se and Te ions when they are together present in the mixtures under investigation. The lower limit for determination of the ions investigated is 0.5 μg amount for Te and Se [6].

The experiments that have been made show that both acidic and basic solutions are useful as mobile phases for the separation of Au(III), Se(IV) and Te(IV) ions through TLC method with the application of Al_2O_3 adsorbent with the second degree of activity.

Taking into consideration the present view upon structure of surface groups of Al_2O_3 sorbent as well as the structure of Au(III), Se(IV) and Te(IV) ions in weak-basic, neutral and weak-acidic solutions, the probable scheme for sorption of the above ions is as follows: Al_2O_3 possesses in its structure limiting groups of OH, $\left[(\text{OH})_2 (\text{Al}_2\text{O} (\text{Al}_2\text{O}_3)_{z+1} \text{O}) \right]$, that are functional groups [7]. From this point of view, ion sorption on such a sorbent probably assumes the character of chemisorption that is associated with the formation of connections with OH groups. This may be presented in the following graphic form:



When we compare the results obtained through TLC method and those achieved by paper chromatography, it may be said that, due to sensitivity and quickness of analysis and possibility for quantitative determination of ions under investigation, TLC method is more effective than paper chromatography.

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STRESZCZENIE

Prowadzone badania wskazują na szerokie możliwości chromatografii cienkowarstwowej jako metody rozdzielania i identyfikacji mieszanin jonów nieorganicznych oraz w badaniu mechanizmu procesu chromatograficznego. Okazuje się, że mechanizm rozdzielania związany jest z procesami tworzenia różnych form kompleksowych badanych jonów i ich sorpcją przez zastosowane adsorbenty. Trwałość utworzonych jonów kompleksowych oraz ich powinowactwo do fazy stacjonarnej warunkują rozdział badanych jonów nieorganicznych.

Śledzono zmiany wyników procesu chromatograficznego przy zmianach rodzaju i składu fazy ruchomej i stacjonarnej. W prezentowanym artykule przedstawione są wyniki prac nad ustalaniem optymalnych warunków prowadzenia analizy metoda chromatografii cienkowsarstwowej.

РЕЗЮМЕ

Проведенные исследования указывают на широкие возможности тонкослойной хроматографии как метода разделения и идентификации смесей неорганических ионов, а также в исследовании механизма хроматографического процесса. Оказывается, что механизм разделения связан с процессами образования разных комплексных форм исследованных ионов и их сорбированием через использованные адсорбенты. Прочность образованных комплексных ионов, а также их сродство со стационарной фазой обуславливают разделение неорганических ионов.

Наблюдались смены результатов хроматографического процесса при изменениях вида и состава фаз - подвижной и стационарной. В показанной работе представлены результаты исследований над установлением оптимальных условий проведения анализа методом тонкослойной хроматографии.

