Anion Exchange Recovery of Gold (I)
from Thiocyanate Solutions in the Presence of Iron (III),
Silver (I) and Some Non Ferrous Metal Ions

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We investigate ion exchange equilibria of thiocyanate complexes of gold (I), iron (III), copper (II) and zinc (II) during sorption from individual solutions on anion exchangers AV-17-8, AM-2B and AN-251 (commercial samples made in Russia). It was found out that there is a dependency between the basicity of anion exchangers and the selectivity of gold (I) recovery from thiocyanate multicomponent solutions (at simultaneous presence of Fe (III), Cu (II), Zn (II) and Ag (I) ions). As a result, the sorption sequences for thiocyanate metal complexes were obtained.

Keywords: gold, thiocyanate, sorption, anion exchange, resin.

Introduction

The stability of thiocyanate ions in acidic medium, as well as their greater selectivity compared to cyanide ions, is the main reason why thiocyanate ions are prospective for recovery of gold from gold-containing raw materials. Another reason for that is that thiocyanate ions are less toxic than cyanides.

It is known that gold (I) thiocyanate complexes are less stable ($\lg \beta_i = 25$ [1,2,3]) than cyanide complexes ($\lg \beta_i = 38$ [1,2,]). However, the thermodynamic analysis of Au – Fe (III) – SCN^- – H_2O [4,5] showed that in approximation to real conditions (i.e. at concentrations of gold and iron (III) less than 0.01 mol/L) it is possible to obtain $\left[Au(SCN)_4\right]^-$ and $\left[Au(SCN)_2\right]^-$ complexes during the dissolution of gold. The research [6,7]

resulted in successful sorption recovery of gold from arsenopyrite concentrates after breakdown with acidic thiocyanate solutions.

The authors [8,9] have pointed out the problems arising when the dissolution of gold is carried out with thiocarbamide, thiosulfate or iodide, as alternative to cyanide reagents. These problems are the choice of oxidizer, which must be effective and "soft" at the same time, and the choice of effective recovery method of gold from the obtained solutions. Noteworthy, a number of investigators [4,6] has proposed to use iron (III) ions, hydrogen peroxide etc. as the oxidizers for effective gold dissolution by thiocyanate ions. The most appealing for that are iron (III) ions because of their presence in gold-containing raw materials. However, at present time there is a lack

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of studies of sorption recovery of gold on various sorbents. Therefore, our investigation is focused on sorption of gold from thiocyanate solutions on a number of anion exchangers.

Experimental

The sorption of thiocyanate complexes of gold (I), iron (III), copper (II) and zinc (II) was studied using anion exchangers AV-17-8, AM-2B and AN-251 with different basicity. AV-17-8 is the strong basic sorbent ($pK_a > 1.5$) with polystyrene matrix and gel structure. It contains quaternary ammonium base as the functional groups. AM-2B is the macroporous anion exchanger of neutral basicity ($pK_a \sim 1.5$–4) with also polystyrene matrix. Its functional groups are quaternary ammonium base as well as tertiary amine groups. AN-251 is the weak basic anion exchanger ($pK_a \sim 5$) with vinylpyridine matrix and macroporous structure. Its functional groups are pyridine rings.

Before use, the anion exchangers were treated according to the standard methods [10].

The solutions of thiocyanate gold (I) complexes were obtained from stock solution of $HAuCl_4$ by means of gold (III) reduction by thiocyanate in the presence of KOH [11]:

$$3[AuCl_4]^- + 7SCN^- + 4H_2O \rightarrow$$
$$\rightarrow 3[Au(SCN)_2]^- + SO_4^{2-} + CN^- + \quad (1)$$
$$+ 8H^+ + 12Cl^-$$
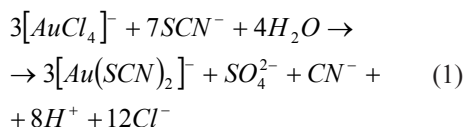
For this purpose, 50.0 mL of $HAuCl_4$ solution with the concentration of 2.0 g/L were diluted by distilled water in a 1 L glass up to the volume of 900 mL. The pH value in this solution was adjusted up to ~ 2 by hydrochloric acid. Then 50.0 mL of 2M KSCN solution was slowly added under agitation by magnetic mixer, and the pH value of the obtained solution was adjusted up to

~ 8 by 10% KOH solution. Over a period of 1 h, pH value of this solution was reduced up to ~ 2 (or ~ 4). Then the solution was placed into a 1 L glass volumetric flask and adjusted up to the mark.

The solutions of gold thiocyanate complexes containing iron (III) and other metal ions, were prepared from the above-mentioned solution. Before the adjustment of solution in a 1 L volumetric flask, 10.0 mL of 0.1 M $FeSO_4$, $CuSO_4$, $ZnSO_4$ and $AgNO_3$ solutions were added to the thiocyanate solution of gold (I). Table 1 contains the data on composition and concentrations of multicomponent solution.

The absorption spectra of investigated solutions were recorded in the wavelength interval of 190–720 nm (spectrophotometer "SF–46", Russia) using a quartz cuvet with 1 cm pathlength.

The individual solutions of iron (III), copper (II) and zinc (II) were prepared from accurately weighed salts samples $FeSO_4 \cdot H_2O$, $CuSO_4 \cdot 5 H_2O$, $ZnSO_4 \cdot 2 H_2O$. These samples were dissolved in 0.1 M KSCN solution with pH ~ 2 (adjusted with HCl).

The concentration of gold in individual and multicomponent solutions as well as concentrations of copper (II), iron (III), zinc (II) and silver (I) at their simultaneous presence were determined by flame atomic absorption method using spectrophotometer "Saturn 2M" (Russia). The concentrations of metal ions in the individual solutions were determined by a photometrical method: iron (III) with sulfosalicylic acid ($\lambda = 420$ nm) [12], copper (II) with thiocyanate at 360 nm [13], zinc (II) with xylenol orange at 420 nm [14].

Table 1. Composition of initial multicomponent solution. pH=2, $C_{KSCN} = 0.1$ mol/L

Compound	Au	Ag	Fe	Cu	Zn	SCN⁻
Concentration, mmol/L	0,23	0,10	2,9	0,10	0,23	0,1
Concentration, mg/L	45	10	162	6,5	7,5	$5,8 \cdot 10^3$

The sorption isotherms were obtained by variation of molar ratio [10,15,16]. For this purpose, the air-dried samples (0.1 g) of anion exchangers in the appropriate form were stirred at 20ºC with 25.0–1000.0 mL of metal ion solution (with concentration $1 \cdot 10^{-3}$ mol/L). Thus, the ratio of resin functional groups quantity in the sample (determined as exchange capacity to Cl⁻ ions) to metal quantity in the contacting solution varied from 3:1 to 1:3. After 24 – 48 h, the equilibrium concentration of metal ions in solution was measured, and the exchange capacity (EC) of the anion exchangers was calculated from the formula:

$$EC = \frac{\left(C_{in} - C_{eq}\right)}{m} \cdot V_S, \qquad (2)$$

where C_{in} and C_{eq} are molar concentrations of the initial and equilibrium solutions, respectively; m is resin mass, g; V_S is volume of the contacting solution, mL.

The sorption of gold from multicomponent solutions was studied through the following scheme. 0.1 L of solution, prepared in accordance with the above-mentioned method, was stirred at 20ºC with the resin sample (0.1 g). After 48 h the solution and anion exchanger were separated and 1.0 mL aliquot was sampled to determine the concentration of metal ions. Then the separated solution was stirred again with the fresh resin sample over a period of 48 h. After that, the resin and solution were separated and 1.0 mL aliquot was sampled again. These operations were repeated 3 times. Then exchange capacity, recovery degree (R), distribution ratio (D) and separation coefficient K_S were calculated for each stage according to the following expressions:

$$R = \frac{\left(C_{in} - C_{eq}\right)}{C_{in}} \cdot 100\%; \qquad (3)$$

$$D = \frac{EC}{C_{eq}} \cdot 1000; \qquad (4)$$

$$K_{S_{Me_1/Me_2}} = \frac{D_{Me_1}}{D_{Me_2}}, \qquad (5)$$

where D_{Me_1} and D_{Me_2} are distribution ratios of the metals separated.

Results and Discussion

It is known that gold is usually contained in the sources of complex composition, where the principal component is scattered among many other metals. The examples could be pyrite or arsenopyrite ores of Russia and S.A.R., containing large amounts of iron, ores of Australia and gold-containing slime, with large amounts of copper [17]. Besides that, primary and secondary raw materials may contain silver, zinc, cobalt, nickel as well as platinum group metals. The problem is that these metals might form anionic complexes with thiocyanate ions and though compete with gold when concentrating from solutions. Therefore, the recovery of gold can be done effectively by means of selective sorption methods, which provide good selectivity through high exchange capacity and good kinetic characteristics of ion exchangers [18].

The main affinity factors of anion exchangers with complex anions of transition metals are the basicity of ion exchanger's functional groups and the charge of recovered species [19]. The propensity of metal ions to form single-charged or multi-charged complexes can also influence the selectivity of sorption for these metals [20].

Using the stage stability constants of metal thiocyanate complexes [1] and method described in [21], we plotted the mole fraction distribution diagrams for various complex ions, depending on concentration of thiocyanate ions in solution. These distribution diagrams allowed us to reveal the ionic state of gold (I), iron (III), copper (II) and zinc (II) in the investigated systems. As we found out, gold (I) exists in a wide range of pH in the form of $\left[Au(SCN)_2\right]^-$ complex ion, whereas iron (III), copper (II) and zinc (II) may

exist in various complex forms, with the charge depending on SCN^- concentration in solution. If SCN^- concentration is about 0.1 mol/L, iron (III) and copper (II) are in the form of $[Fe(SCN)_6]^{3-}$ and $[Cu(SCN)_4]^{2-}$, respectively. Zinc exists there as $[Zn(SCN)_4]^{2-}$ and $[Zn(SCN)_3]^-$ complexes. With the decrease in SCN^- concentration in solution, the relative increase of (-1)-charged copper (II) and zinc (II) complexes takes place. Simultaneously, iron $[Fe(SCN)_2]^+$ complexes appear in solution, and their relative share increases substantially.

We investigated the ion exchange equilibria by means of sorption isotherms of thiocyanate gold (I), iron (III), copper (II) and zinc (II) complexes during their recovery from individual solutions on anion exchangers with different basicity. The results are presented in Fig. 1, 2, 5 and 6. All the curves can be considered Langmuir's isotherms, i.e. the curves are abrupt in the range of low equilibrium concentrations of recovered species, and come to equilibrium in the range of high concentrations. Therefore, the sorption probably proceeds according to the anion exchange mechanism:

$$m\overline{RAn} + [Me(SCN)_n]^{m-} \rightarrow$$
$$\rightarrow \overline{R_m[Me(SCN)_n]} + mAn^-, \qquad (6)$$
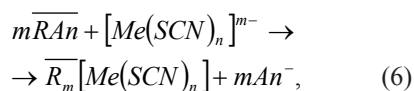
where An^- is the counter-ion of initial anion exchanger form.

Taking into account that iron (III) was used as an oxidizer for thiocyanate leaching process, it is a matter of scientific and practical interest to study ion exchange equilibrium in the system Fe(III) – SCN^- - H_2O – anion exchanger in more detail. It is known [6,7] that the iron (III) concentration is about 1-2 mmol/L after the sorption leaching of gold from arsenopyrite concentrates after breakdown with acidic method. That is why we investigated the sorption equilibrium of thiocyanate iron (III) complexes in the equilibrium concentrations range up to 2 mmol/L.

Sorption isotherms (Fig. 2) show that the investigated anion exchangers recover thiocyanate iron (III) complexes from solutions at pH=2 and thiocyanate concentration 0.1 mol/L. The saturation of anion exchangers is achieved at more than 0.6 mmol/L iron (III) equilibrium concentration in solution. However, the exchange capacity of sorbents depends on their basicity, and is considerably reduced when it decreases. For instance, the maximum exchange capacity is about 0.6 mmol/g for AV-17-8, 0.4 mmol/g for AM-2B and 0.1 mmol/g or less for AN-251. This can be explained by the fact that the strong basic anion exchangers show the greatest affinity with metal ions, which are likely to form multi-charged complexes. Since in our experiments (pH=2; C_{SCN^-} =0.1 mol/L) iron (III) existed in solution mostly in the form of triple-charged complex ions (ignoring hydrolysis), AV-17-8 tends to these ions.

We have also investigated the sorption equilibria at the so-called "aging" of thiocyanate iron (III) complexes solutions. Fig. 2 contains isotherms of iron (III) sorption both from fresh solutions and solutions kept over 48 h. The maximum exchange capacity of AV-17-8 to iron (III) after its sorption from aged solution diminishes approximately to 20 %, but the shape of sorption isotherm remains convex as before (Fig. 2, curves 1 and 2). The sorption equilibrium on weak basic anion exchanger AN-251 is almost the same (Fig. 2, curves 5 and 6). In contrast, the sorption of iron (III) from aged solutions on neutral basic anion exchanger AM-2B proceeds with sharp decrease in affinity, which can be easily noticed by change in isotherm's shape (Fig. 2, curves 3 and 4). The convex curve becomes a straight line (determination coefficient is ~ 0.98). Probably this can be explained by hydrolysis of thiocyanate iron (III) complexes [22]: at pH=2 it proceeds slowly, causing the formation of asymmetric mixed complexes, poorly held on anion exchangers with weak basic functional
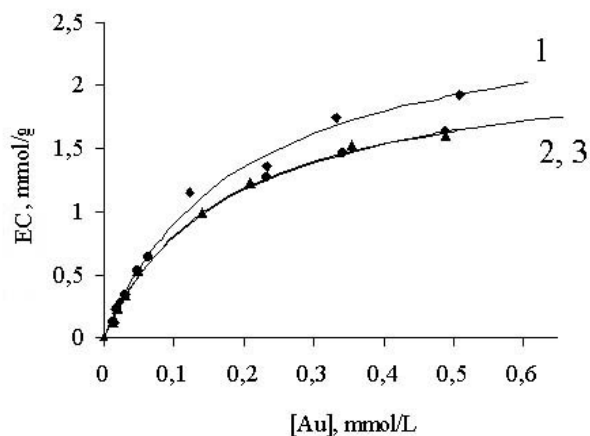
Fig. 1. Sorption isotherms of thiocyanate complexes of gold (I) on anion exchangers in initial chloride form. pH=2, C_{KSCN} = 0.1 mol/L. 1 – AM-2B; 2 – AV-17-8; 3 – AN-251
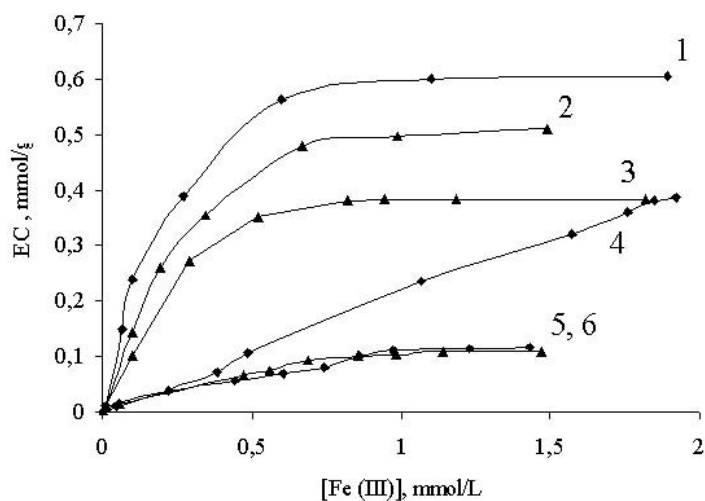

Fig. 2. Sorption isotherms of iron (III) from thiocyanate solutions, pH=2, C_{KSCN} = 0.1 mol/L. 1,2 – AV-17-8; 3,4 – AM-2B; 5,6 – AN-251. 1,3,5 – sorption from fresh solutions; 2,4,6 – sorption from solutions kept over 48 h
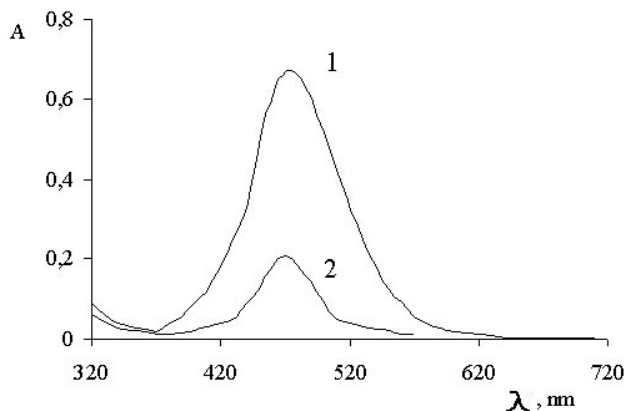

Fig. 3. Electron absorption spectra of thiocyanate solutions of iron (III); 1 - fresh solution; 2 – kept during 24 h

groups. It is known [6] that the reduction of iron (III) by SCN^- ions can take place in thiocyanate solutions. However, the qualitative reactions [23] in investigated solutions indicated the presence of slight amounts of Fe (II). It should be noted, that the observed reduction in absorption maximum intensity at ~ 470 nm after 48 h of "aging" the thiocyanate iron (III) solution (Fig. 3) also points out to hydrolysis of Fe (III).

The concentration of thiocyanate ions in contacting solution has a substantial effect on recovery of iron (III) (Fig. 4). At SCN^- concentration of 0.01 mol/L, all the investigated anion exchangers practically do not recover iron (III). This decrease in exchange capacity of anion exchangers is a consequence of change in ionic state of iron (III): cationic complexes $\left[Fe(SCN)_2\right]^+$ exist mainly in solution at C_{SCN^-} =0.01 mol/L. These complexes do not permeate into the resin phase because of Donnan's potential.

Thus, it should be noted that the investigated anion exchangers do not possess high affinity with thiocyanate iron (III) complexes, and iron (III) would not compete with gold during its recovery. At the same time, the obtained data indicate the relatively high probability of "poisoning effect" of anion exchangers by $\left[Fe(SCN)_6\right]^{3-}$. This effect, discussed in the works [17, 18], reveals itself by decrease in exchange capacity of anion exchangers for gold due to the "poisoning" by complex ions $\left[Fe(CN)_6\right]^{3-}$ and $\left[Fe(CN)_6\right]^{4-}$.

As we mentioned before, the usual components of gold-containing raw materials are iron, copper and zinc. Their percentage share can be dozens of times higher than of gold. That is why we have investigated the ion exchange equilibria in the systems Cu (II) – SCN^- – H_2O – anion exchanger and Zn (II) – SCN^- – H_2O – anion exchanger. The results are shown in Fig. 5. When equilibrium concentrations of copper and zinc ions in solutions are about 0.2 mmol/L, approximately 80% of maximum exchange capacity of anion

exchangers for Cu (II) and Zn (II) are achieved. With the increase in their concentrations, the exchange capacity for copper (II) and zinc (II) ions is slowly rising. Such a trend can be probably explained by formation of monovalent complexes of copper and zinc, as the ratios Cu (II)/SCN^- or Zn (II)/SCN^- are decreasing. They are changed in the resin phase during its saturation with these ions.

We have also investigated the effect of SCN^- ions in sorbent phase on the sorption equilibrium of copper and zinc. It was determined that initial SCN^- form of anion exchangers does not alter the shape of sorption isotherms, i.e. has no effect on their sorption.

In general, it can be stated that anion exchangers AV-17-8, AM-2B and AN-251 recover thiocyanate complexes of iron (III), copper (II) and zinc (II) only to some extent. The saturation of sorbents with these complexes, even if their concentration are of 0.1 mol/L, is not higher than 20% of their exchange capacity for Cl⁻ ions. At the same time, the anion exchangers investigated show great affinity with thiocyanate complexes of gold. The high exchange capacity is achieved at low equilibrium concentrations of gold in solution. This behavior is presented at Fig. 6. It should be noted that in our previous work [24] we have indicated the great affinity between investigated anion exchangers and thiocyanate complexes of silver (I). That is why in the present investigation we paid attention to the opportunities of selective recovery of gold in presence of silver and other associated metal ions.

It can be seen from the Tables 2 and 3 that all the anion exchangers investigated possess the high sorption abilities to gold during its sorption from multicomponent thiocyanate solutions. On the first stage of sorption (i.e. without excess of sorbent), the anion exchangers recover up to 73–83% of gold, and on the second stage (twofold excess of sorbent) the recovery degree goes up
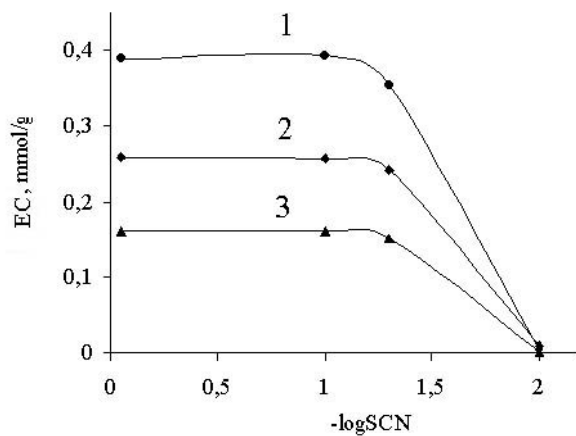
Fig. 4. Dependence of anion exchanger capacity for iron (III) on concentration of thiocyanate ions in contacting solution. 1 – AV-17-8; 2 – AM-2B; 3 – AN-251
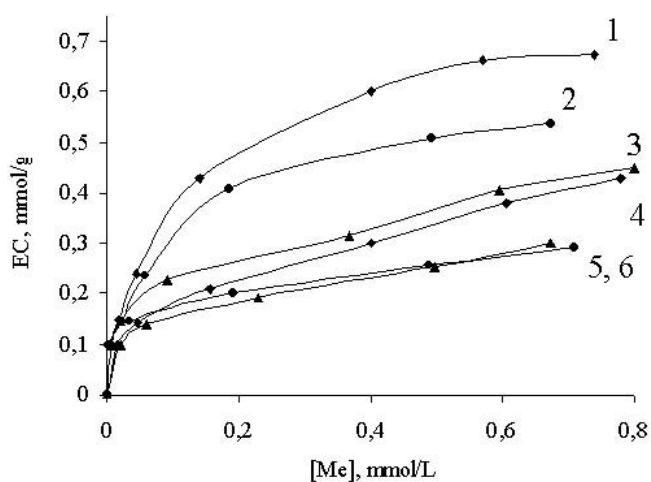

Fig. 5. Sorption isotherms of thiocyanate complexes on anion exchangers.
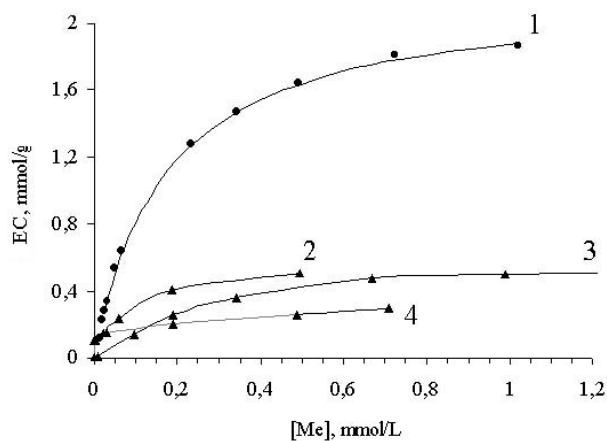AM-2B: 1 – Cu(II), 4 – Zn (II); AV-17-8: 2 - Cu(II), 6 - Zn (II); AN-251: 3 - Cu(II), 5 - Zn (II)

Fig. 6. Sorption isotherms of metal thiocyanate complexes on anion exchanger AV-17-8 in initial chloride form.
1 – Au (I); 2 - Cu(II); 3 – Fe (III); 4 - Zn (II)

Table 2. Metal ion recovery degrees during stage sorption from multicomponent solution

| Anion exchanger	Stage	Au	Ag	Fe	Cu	Zn
AV-17-8	1	77,8	90	10,5	98,2	46,7
	2	95,6	98,9	22,8	98,2	73,3
	3	99,8	98,9	40,1	98,2	88
AM-2B	1	73,3	72,2	7,4	98,2	46, 7
	2	91,1	98, 9	22, 9	98,2	74
	3	94,4	98, 9	30,9	98,2	86
AN-251	1	83,3	55, 6	7,4	61	56,8
	2	97,8	88, 9	7,4	64	79,3
	3	99,8	98, 9	22, 9	98	86,7

Table 3. Distribution ratios of metal thiocyanate complexes during stage sorption from multicomponent solution

| Anion exchanger	Stage	Au	Ag	Fe	Cu	Zn
AV-17-8	1	3500	9000	117	54000	875
	2	3800	7600	152	0	950
	3	17100	0	260	0	1100
AM-2B	1	2750	2600	80	54000	875
	2	1900	22800	190	0	999
	3	540	0	105	0	771
AN-251	1	5000	1250	80	1750	1308
	2	6175	2850	0	0	1042
	3	8100	8100	180	17100	495

~ 98%. It is visible through distribution ratios (Table 3), that weak basic resin AN-251 shows the highest affinity with gold (I) thiocyanate complexes, whereas the strong basic sorbent AV-17-8 sorbs the thiocyanate complexes of silver to a greater extent. The anion exchangers AV-17-8 and AM-2B possess the high sorption ability to the thiocyanate complexes of copper (II).

During the first stage of sorption on AN-251, the distribution ratios for gold are higher than for silver and copper on the first stage of recovery. As a result, the recovery degrees account to 83.3%, 55.6% and 63.6% for gold, silver and copper, respectively (Table 2). On the second stage of sorption, when gold is mostly recovered from solution, the anion exchanger AN-251 sorbs silver. Copper is recovered on AN-251 only on the third stage of sorption, i.e. after isolation of gold and silver from solutions to 99% and 96%, respectively. Therefore, the metal thiocyanate complexes are recovered on weak basic anion

exchanger AN-251 in accordance with the following sequence:

Au (I) >> Ag (I) > Cu (II) > Zn (II) >> Fe (III).
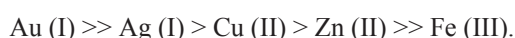
The neutral basic anion exchanger AM-2B shows less selectivity to gold than to silver, but the metal complexes are recovered practically in the same sequence like on the anion exchanger AN-251:

Au (I) > Ag (I) > Cu (II) > Zn (II) >> Fe (III).
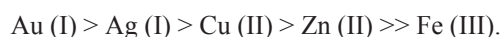
The strong basic anion exchanger AV-17-8 exhibits the great affinity with thiocyanate complexes of silver and recovers the metal complexes in the following sequence:

Ag (I) > Cu (II) ~ Au (I) > Zn (II) >> Fe (III).
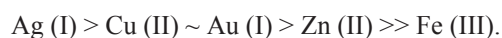
The anion exchangers investigated show weak sorption abilities to thiocyanate complexes of iron (III). However, it should be noted that AN-251 is saturated with metal complexes to a lesser extent (Table 4). The mentioned sequences can also be illustrated by separation coefficients

Table 4. Sorption of gold (I) from multicomponent solution on anion exchangers AV-17-8, AM-2B and AN-251

Metal	Au	Ag	Fe	Cu	Zn
Anion exchanger	AV-17-8				
Stage	EC, mg/g				
1	35	8,1	17	5,4	3,5
2	7,6	0,8	19	< 0,1	1,9
3	1,71	< 0,1	25,2	< 0,1	1,0
Separation ratio	$K_{f(Au/сумма)}$	$K_{f(Au/Ag)}$	$K_{f(Au/Fe)}$	$K_{f(Au/Cu)}$	$K_{f(Au/Zn)}$
	1,7	0,4	30,0	0,1	4
Anion exchanger	AM-2B				
Stage	EC, mg/g				
1	33	6,5	12	5,4	3,5
2	7,6	2,3	23,8	< 0,1	2,0
3	1,4	< 0,1	11,7	< 0,1	0,81
Separation ratio	$K_{f(Au/сумм)}$	$K_{f(Au/Ag)}$	$K_{f(Au/Fe)}$	$K_{f(Au/Cu)}$	$K_{f(Au/Zn)}$
	4,5	1,1	34,4	0,1	3,1
Anionexchanger	AN-251				
Stage	EC, mg/g				
1	37,5	5	12	3,5	4,3
2	6,2	2,9	< 0,1	< 0,1	1,6
3	0,8	0,8	22,5	1,7	0,5
Separation ratio	$K_{f(Au/сумма)}$	$K_{f(Au/Ag)}$	$K_{f(Au/Fe)}$	$K_{f(Au/Cu)}$	$K_{f(Au/Zn)}$
	8,9	4	62,5	2,9	3,8

calculated for the first sorption stage (Table 4). These values are 8.9; 4.5 and 1.7 for AN-251, AM-2B and AV-17-8, respectively. Therefore, the selectivity of these sorbents to gold grows in the following sequence:

AV-17-8 < AM-2B < AN-251.

Noteworthy, this sequence is in agreement with the basicity of these sorbents.

The observed patterns are in good correspondence with the conception of affinity of anion exchangers. As a rule, the strong basic resins show greater selectivity to multicharged anions. That is why AV-17-8 is more selective to thiocyanate complexes of silver, which form stable triple-charged anionic complexes with thiocyanate ions. At the same time, the weak basic anion exchangers show greater affinity with monovalent anionic metal complexes, the most stable of which are thiocyanate complexes of gold (I).

Our results allow to conclude, that weak basic anion exchangers are the most selective to thiocyanate complexes of gold (I). That is why they can be recommended for recovery of gold from multicomponent solutions contained copper (II), silver (I), zinc (II) and iron (III) ions. The effective simultaneous recovery of gold and silver can be carried out on neutral or strong basic anion exchangers.

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