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Transformations of $[Au(CN)_2]^-$ at the Contact with Activated Carbon Surface

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Abstract—We have studied the processes occurring when alkaline aqueous solution of $Na[Au(CN)_2]$ is brought in contact with activated carbon surface. It has been shown that the adsorption of the gold cyanide complex occurs via several independent routes, one of them being accompanied with the partial breakdown of the inner sphere of the compound and free cyanide ions release into the solution. The latter process decreases the gold recovery by aqueous NaOH during the high-temperature (150°C) desorption. A prolonged storage of the adsorbent saturated with $[Au(CN)_2]^-$ in air produces a similar result. The reasons of the observed phenomena have been discussed.

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Dicyanoaurate(I) complex ion, $[Au(CN)_2]^-$, is known for extreme stability; its stability constant value $\beta_{1,2}$ ranges from 10^{37} to 2.8×10^{39} as reported in [1] and references therein. Therefore, the inner sphere substitution reactions of this anion are hindered. On the other hand, the inner sphere of the complex is fairly labile [2]. The cyanide ligands lability is increased due to their *trans*-effect, which is particularly strong in the linear complexes of gold(I) [3]. Thus, the ligand substitution in $^-NC–Au^I–CN^-$ is possible, when a large excess of the ligand with the weaker *trans*-influence than that of cyanide ion is present in the solution. Besides the substituting group nature, the possibility of binding the eliminating cyanide ion is an important factor of $[Au(CN)_2]^-$ reactivity. The HCN acid, conjugate with the cyanide ion, is very weak, in contrast to the cases of many other pseudo-halogenide ligands. Therefore, in the acidic solutions the probability of cyanide ligand exchange is strongly increased, as compared to the alkaline media. Indeed, in the acidic solution, $[Au(CN)_2]^-$ interacts with the thiol and thione ligands [4–6] as well as with iodide anion [7]; at least one of the cyanide ligands is thus substituted, and HCN is released into the solution.

The mechanism of $[Au(CN)_2]^-$ adsorption onto activated carbon has remained unclear so far. Various mechanisms of the gold(I) complex ions binding to activated carbon are discussed in [8]. According to some suggestions, the adsorption of $[Au(CN)_2]^-$ from alkaline solutions might be accompanied with elimination of CN^- from the inner sphere of the complex due to the formation of the AuCN fragments or $[Au_n(CN)_{n+1}]^-$ ($n \geq 4$) oligomers in the carbon phase. In such adsorbates, gold atom is bound to donor centers of the adsorbent via the nucleophilic substitution reaction in the inner sphere of the $[Au(CN)_2]^-$ ion. In particular, it has been shown that the activated carbon containing about 7% of sulfur (likely, in the form of surface-bound thiophene-like groups) exhibits a higher capacity towards $[Au(CN)_2]^-$ adsorption from alkaline aqueous solutions (pH 10.5) as compared to the sulfur-free carbon [9]. It has been suggested that the enhanced adsorption in the above-mentioned case is due to the substitution of one of the cyanide ligands in the Au(I) linear complex ion with the thiophene fragment containing a donor sulfur atom.

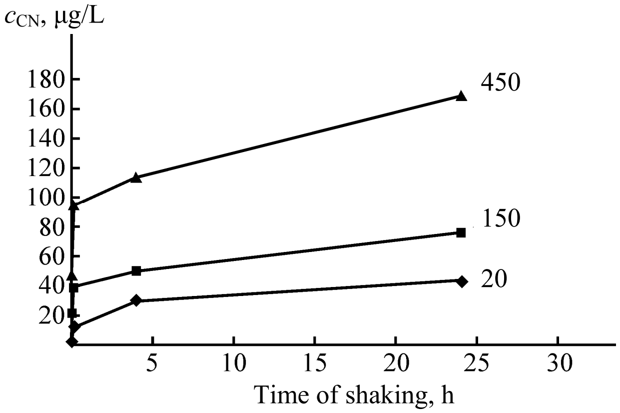
On the other hand, the photochemically induced substitution of both cyanide ions with chloride ones has been observed in acetonitrile solution of the stable anion, *trans*-$[Au(CN)_2Cl_2]^-$ [10]. It cannot be excluded that the process occurs via the intramolecular redox reaction of the REOA type, through intermediate Au(I) or Au(II) complexes with lower coordination number as compared to the initial Au(III) complex.

In this work, we have studied the behavior of Na $[Au(CN)_2]$ in the course of adsorption from aqueous solutions with the activated carbon; high-temperature (145°C) desorption of gold cyanide complex by aqueous NaOH was evaluated as well.

In the adsorption experiment, a weighed sample of crystalline $Na[Au(CN)_2]$ was dissolved in the aqueous solution of NaOH (at pH of 10.47). The concentration of cyanide anion was determined in the so prepared solution; it was then considered a reference (back-ground) concentration. The activated carbon (Goldcarb WSC 208C-GR, washed from the ashes and dried) was introduced to the solution. The mixture was then isolated from air and stirred in the laboratory shaker, the free cyanide concentration was determined after certain shaking times (10 min, 4 h, and 24 h). Experimental results are collected in the figure, the CN^- concentrations plotted in the figure are given after subtraction of the back-ground value.

As seen in the Figure, after addition of activated carbon to the alkaline solution of $Na[Au(CN)_2]$, free cyanide ions were detected in the solution. Their concentration increased with increasing concentration of dicyanoaurate(I) ions. The release of CN^- started immediately after the adsorbent addition; however, it continued steadily during the next 24 h. Therefore the adsorption of $Na[Au(CN)_2]$ onto the activated carbon was accompanied by the partial removal of cyanide ion from the inner sphere of the complex. It should be noted that according to [11] the release of cyanide in the form of HCN occurred when activated carbon, treated with 0.1 mol/L KCl at pH 10 and saturated with $[Au(CN)_2]^-$, was heated at about 300°C. Crystalline $Na[Au(CN)_2]$ decomposed into metal gold, cyanogen, and sodium cyanide at 462°C [12].

The true concentration of free cyanide ions released from the inner sphere of the $[Au(CN)_2]^-$ anion upon interaction with activated carbon could hardly be determined. According to [13], KCN was almost irreversibly adsorbed from its aqueous solution in contact with charcoal. The adsorbed cyanide ions could not be then detected neither after treatment the charcoal with sulfuric acid in a vacuum nor after interaction with aqueous picric acid. The desorption of CN^- from the charcoal was not facilitated by treatment with aqueous Na_2S or by boiling with aqueous NaOH [13]. Later it was demonstrated in [14] that a part of the cyanide anions adsorbed by the activated carbon from aqueous KCN solution was in fact oxidized by air oxygen to form CNO^-, the process was catalyzed by Cu(II) compounds.



Concentration of CN^- as a function of time of $Na[Au(CN)_2]$ solution shaking with activated carbon at pH 10.5. Concentration of Au(I) in the solution (mg/L) is shown near the curves.

Further reason for irreversible adsorption of cyanide ions onto the activated carbons could be their nucleophilic addition to the aldehyde or ketone groups present on the activated carbon surface. In this case, depending on the nature of the fragments bound to the carbonyl groups, either α-oxynitriles (further hydrolyzed to α-oxyacids in the aqueous solutions), α-oxyketones, or cyanohydrins could be formed at the surface [15]. The study of the carbon fibers in contact with aqueous NaCN by the X-ray photoelectron spectroscopy (XPS) revealed the appearance of symmetric signal with the N 1*s* bond energy of 399.5 eV, nitrogen concentration at the surface being 1.8 mol % [15]. In the literature, the bond energy of the N 1*s* line in the range of 399.6–399.7 eV was assigned to nitrile group [16].

In order to evaluate the participation of cyanide ion oxidation in the adsorption of NaCN by the activated carbon we studied this process in air and under anaerobic conditions. In the latter case, the sample of activated carbon was immersed into NaOH solution (starting pH of 10.50) and was refluxed during 3 h at bubbling with purified nitrogen of extra pure grade. After that, the solution was cooled to room temperature under the nitrogen stream, and a sample of solid NaCN was added. Further operations were performed under bubbling with nitrogen through the suspension in the anaerobic atmosphere, sampling the solution with the Schott's adapter. A similar experiment was performed

NaCN adsorption with activated carbon under aerobic and anaerobic conditions

Time of activated carbon contact with NaCN solution, h	Concentration of CN^- in the solution in contact with activated carbon, mg/L	
	aerobic conditions	anaerobic conditions
0	114.76	92.70
4	23.20	75.71
8	2.10	59.51
12	0.88	44.98
16	0.12	28.29
20	0.08	12.77
24	0.06	6.16

in air (the activated carbon was not refluxed with NaOH in that case). The results are collected in the table.

From the tabulated data it followed that air oxygen significantly affected the residual concentration of CN^- in contact with the activated carbon. We cannot be sure that even under anaerobic conditions oxygen was completely removed from the carbon surface. Thus, the results shown in the Figure could only be used for qualitative interpretation.

Previously it was shown that the treatment with NaOH at 145°C led to almost quantitative (>98%) desorption of gold(I) from the carbon surface [17]. Even in the absence of NaCN in the desorbing solution, gold(I) was detected in the eluate in the form of $[Au(CN)_2]^-$ anion. In order to evaluate the amount of CN^- eliminated from the inner sphere of $Na[Au(CN)_2]$ in the course of its adsorption onto the activated carbon, we used the high-temperature autoclave desorption of Au(I) cyanide complex with aqueous solution of NaOH. In the case of elution of the adsorbent washed of the free NaCN, only gold(I) present on the surface in the form of $[Au(CN)_2]^-$ could be desorbed. On the contrary, the desorption of gold(I) present in the form of AC–AuCN (AC denoting the activated carbon donor centers) by NaCN-free alkaline solution was unlikely. Gold(I) in the form of $[Au_n(CN)_{n+1}]^-$ ($n \geq 4$) clusters should undergo a minor desorption, its extent decreasing with increasing n.

Under the conditions of the common industrial process, the adsorption of gold(I) cyanide complex occurred in the presence of excess of free NaCN (usually 50 to 450 mg/L). Beside the so-called “irreversible” adsorption of cyanide anions leading in fact to its conversion into other compounds, the reversible adsorption of free cyanide ions was possible. The latter process could occur via the ion exchange of CN^- with the OH^- ions from the double electrical layer of the carbon surface, or via physical adsorption of NaCN in the sorbent pores. Thus, high-temperature desorption of Au(I) cyanide complexes with alkali under industrial conditions occurred in the presence of excess cyanide, if the carbon was not washed prior with acid. The presence of free cyanide was evidenced by significant release of ammonia in the course of high-temperature (135–155°C) desorption of $[Au(CN)_2]^-$. The ammonia release was due to the decomposition of CN^- according to the equation below [18].

$$CN^- + 3H_2O \rightarrow NH_3 + CO_2 + H_2 + OH^-.$$

The presence of free NaCN in the extracting solution facilitated gold(I) removal from the carbon surface [17]. In order to eliminate the influence of free cyanide, the following experiment was performed. The solution of $Na[Au(CN)_2]$ [200.0 mL, 14.80 mg/L of gold (I)] was divided in two equal portions. Crystalline NaCN was added to one of the portions, to the final concentration of 300 mg/L, the second portion was used as a reference. Activated carbon (2.00 g) was added to both portions, and the mixtures were stirred with the laboratory shaker during 18 h. Then, the carbon was separated, the solutions were analyzed for residual Au(I), whereas the carbon samples were thoroughly washed with distilled water and subjected to desorption in the laboratory autoclave. In the absence of the added NaCN, the adsorption of gold(I) cyanide complex was somewhat enhanced. The final Au(I) concentrations in the solutions were 4.70 mg/L and 5.07 mg/L, respectively. In both cases, the desorption stage was performed at 145.0±0.5°C by NaOH solutions (20 g/L). The amount of the eluate was the same in both cases, 27.5 bed volumes. Gold(I) recovery from the carbon sample treated with NaCN was 99.3%, being only 92.4% in the case of the reference sample. Thus, about 7% of the $[Au(CN)_2]^-$ complex anions released cyanide ion from the inner sphere under the experimental conditions.

The conversion of the $[Au(CN)_2]^-$ anion adsorbed onto the activated carbon occurred in the course of its prolonged storage in air. In particular, the carbon samples were saturated by cyanide solutions to contain

3.67 mg/g of gold and 4.06 mg/g of silver. In the course of desorption with NaOH solution (10 g/L, 150.0±0.5°C) performed immediately after the saturation and washing the sorbent with distilled water, 98.6% of gold and 90.5% of silver was recovered. The same carbon samples, after 3 months storage in the dry state (without special oven drying) released only 62.7% of gold and 54.1% of silver under the same conditions. Gold recovery could be up to 96.8% when a mixture of NaOH (10 g/L) and NaCN (0.5 g/L) was used as the eluent; however, the recovery of silver was still reduced to 81.0%. Thus, it was proved that the gold cyanide complex adsorbed onto the activated carbon was gradually converted into the other form(s).

It should be noted that the study of the $[Au(CN)_2]^-$ anion adsorbed onto the activated carbon granules by means of ^{197}Au Mössbauer spectroscopy [19] showed the presence of two gold compounds at the surface of the wet sorbent in the case of uptake from alkaline solutions (pH 10.2). One of the compounds was the $[Au(CN)_2]^-$ ion somehow bound to the matrix (95% of the total gold amount). Its quadrupole splitting and isomeric shift as compared with those of crystalline $K[Au(CN)_2]$ revealed that due to binding with gold(I) the central atom nucleus was less shielded, which indirectly evidenced the additional interaction with the π-acceptor ligands. On the other hand, the only complex compounds of Au(I) with coordination number higher than 2 (3 or 4) described so far were of the non-electrolyte ($[AuXL_n]^0$; $n = 2, 3$, L was neutral monodentate ligand) or of the cationic type [20].

The second gold compound (5% of the total gold amount) could be assigned to the Au(III) complex with the cyanide ligands according to the isomeric shift and the quadrupole splitting values. This unusual carbon-adsorbed gold compound was preserved in the course of vacuum drying at room temperature, but was converted into $[Au(CN)_2]^-$ after 22 hours of drying at 60°C in air or after prolonged storage (several months).

The XPS study [21, 22] showed that after $[Au(CN)_2]^-$ adsorption onto the activated carbon surface part of gold was present in the form of Au(III) compound, however, the major part was assigned to Au(I) according to the $4f_{7/2}$ bond energy. In most of other XPS studies of activated carbons, only Au(I) compounds were detected after adsorption of $[Au(CN)_2]^-$ from aqueous solutions. The analysis of these results should take into account that the activated carbons were dried prior to XPS experiment. As was mentioned above, the drying conditions could significantly change the composition and structure of adsorbate.

The nature of the cation (H^+, K^+, Ca^{2+}, Gd^{3+}) adsorbed together with $[Au(CN)_2]^-$ did not influence the Lamb–Mössbauer factor, proving that they were not localized in the nearest surrounding of the gold atom [19]. Thus, the cyanide complex was not adsorbed in the form of ion associate with the studied cations.

The cyanide ligand elimination from the inner sphere of $[Au(CN)_2]^-$ could be regarded as nucleophilic substitution of CN^-; it should be noted that the latter process required the presence of donor centers on the adsorbent surface. The adsorption of $[Au(CN)_2]^-$ onto the activated carbon surface under standard conditions occurred in the solutions saturated with air oxygen and was accompanied with molecular oxygen absorption. When the oxygen-free adsorbent was used, and the process was performed under anaerobic conditions, the adsorbent capacity towards $[Au(CN)_2]^-$ was significantly decreased as compared with aerobic conditions [23], and the adsorption was noticeably slowed down [24]. When immersed into the oxygen-saturated distilled water, redox processes occurred at the activated carbon surface leading to the appearance of the reactive species ($O_2^{-\cdot}$, $OH^{\cdot}$, and hydrogen peroxide) [15, 25]. In turn, they induced the activated carbon surface oxidation resulting in the formation of nitrogen- and oxygen-containing groups, release of CO and CO_2, and the appearance of carbene-like unsaturated carbon atoms on the surface. Like other carbenes, such ligands revealed strong *trans*-effect [26] and could be regarded as peripheral carbon atoms of graphene [27]. They could participate in the nucleophilic substitution in the inner sphere of $[Au(CN)_2]^-$ leading to strong binding of the gold complex to the adsorbent surface due to the formation of AC–AuCN compounds.

On the other hand, the HOMO of $[Au(CN)_2]^-$ belonged mainly to the Au(I) atom. As a result of the formation of superoxide anion-radical near the activated carbon surface, the $[Au(CN)_2]^-$ anion could be subjected to single-electron or two-electron oxidation. In the case of two-electron oxidation, the process occurred via oxidative addition accompanied by the formation of square planar Au(III) complex with the $^-NC{-}Au^{III}{-}CN^-$ fragment remaining unchanged and the new ligands (for instance, water molecules) added. The substitution of one of the water molecules with the carbene-like carbon atom led to strong anchoring of the Au(III) complex on the activated carbon surface.

Being strong single-electron oxidant, the OH radicals in alkaline medium could oxidize $[Au(CN)_2]^-$ anions into gold(II) compound, $[(OH)Au(CN)_2]^-$ [28]. These reactive intermediate could be further oxidized into stable ions, $[O{=}Au^{III}(CN)_2]^-$ or $[(HO)_2Au(CN)_2]^-$, or disproportionate to form either $[Au(CN)_2]^-$ and $[O{=}Au^{III}(CN)_2]^-$ or $[(HO)_2Au(CN)_2]^-$ [28].

We attempted to evaluate the possibility of intramolecular redox reaction between the central atom and the coordinated cyanide ligand in the Au(II) complex in the frame of the Fridman intramolecular potentials theory [29], using the correlation $E^0_{X2/2X-} = -0.763+0.990E^0_{1e}$ (V) between the standard single-electron redox potentials of the ligand E^0_{1e} and its standard oxidative dimerization potential $E^0_{X2/2X-}$ [30]. To do so, we used the standard redox potentials in the Au^{2+}/Au^+ (1.8 V [31]) and CN^0/CN^- (2.59 V [32]) pairs and assumed that the stability constant of the Au(II) cyanide complex was between the corresponding values of $[Au(CN)_2]^-$ and $[Au(CN)_4]^-$ ($\log K_{stab}$ = 39 and 82, respectively [1]). According to Fridman theory, the difference between the intramolecular potentials of Au(II) and the coordinated CN^- ($\Delta E'$) could be estimated, and thus the stability of the complex towards the intramolecular redox reaction could be predicted. The calculated $\Delta E'$ value was negative (less than –0.39 V); therefore, in the course of Au(II) cyanide complex formation in the solution via the single-electron oxidation of $[Au(CN)_2]^-$, its reduction to Au(I) complex could not occur via the intramolecular redox reaction involving the cyanide ligand oxidation. Furthermore, such process should be accompanied with the decrease in the Au(II) coordination number and the elimination of the second cyanide ion from the inner sphere of the complex; that was not observed in the Mosseri experiment [28].

Note that in the alkaline aqueous solutions (pH 10.0–10.5), the primary products of Au(II) compound oxidation or disproportionation should be the corresponding aqua-cyanide complexes of Au(III). Thus, the adsorption of Au(III) cyanide compounds could occur due to the substitution of the water molecule with the donor carbene-like carbon atom. The analysis of experimental results on the adsorption of thermodynamically stable cyanide complexes of the transition metals onto the activated carbons revealed that the ions with planar or linear coordination of the central atom were mainly adsorbed [33].

EXPERIMENTAL

The activated carbon Goldcarb WSC 208C-GR was washed from the ashes as previously described [12] prior to the adsorption experiments.

The concentration of cyanide anions in the aqueous solution was determined by spectrophotometry (pyridine-barbiturate reaction, in quartz cell, optical path of 1.01 cm, KFK-3) [34]. Concentrations of gold and silver in the solution were determined by atom absorption spectroscopy (SpektrAA 220 Varian, oxygen-acetylene flame).

Desorption of gold and silver from the saturated activated carbon was performed in the specially constructed laboratory autoclave.

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