

CHEMICAL KINETICS AND CATALYSIS

Oxidation of Co(II) by Ozone and Reactions of Co(III) in Solutions of Sulfuric Acid

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Abstract—Reactions of the oxidation of bivalent cobalt ions by ozone, of the spontaneous decomposition of trivalent cobalt, and of interactions between Co(III) and chloride ions in solutions of sulfuric acid are studied. The order and rate constant of the process of decomposition of Co(III) are determined. Information on the kinetics of the interaction between Co(III) and Cl^- is obtained. Kinetic patterns of the accumulation of Co(III) during the ozonation of solutions of CoSO_4 in sulfuric acid are explained. Molar absorption coefficients of Co(III) and Co^{2+} ions in the visible range of wavelengths are determined.

Keywords: ozone, trivalent cobalt ions, bivalent cobalt ions, rate constant of a chemical reaction, molar absorption coefficient

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INTRODUCTION

Ozonation is widely used to purify wastewaters and sewage waters of organic pollutants. However, certain organic compounds react somewhat with ozone, and their removal requires the use of catalysts. Of these, cobalt catalysts are some of the most efficient. They allow us to achieve complete mineralization of such substances inert with respect to ozone as oxalates and oxalic acid [1–3]. Adding Co^{2+} ions substantially increases the oxidation of chloride ions by ozone [4, 5]. The interaction between cobalt salts and ozone is a complex chemical process that includes stages of reduction–oxidation and complex formation. Under the action of ozone, Co^{2+} ions transform into Co(III) compounds that are very strong and active oxidizing agents. Information on the kinetics of the interaction between Co^{2+} and O_3 and concurrent reactions of Co(III) is required to develop new catalytic methods, and to intensify and control existing methods.

According to the available information, the reaction between ozone and cobalt ions in aqueous solutions was studied only in [6, 7] in solutions of perchloric acid within a narrow pH range; the rate constant of the reaction between Co^{2+} and O_3 was determined only within the temperature range of 0–2°C. The aims of this work were to study the kinetic patterns of the formation of Co(III) and its subsequent reactions during the ozonation of solutions of bivalent cobalt sulfate in sulfuric acid, to determine the order and rate

constant of the decomposition of Co(III), and to study the reaction between Co(III) and chloride ions.

EXPERIMENTAL

The following reagents were used in this work: distilled water, pure grade cobalt(II) sulfate heptahydrate $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$; sulfuric acid of special purity grade, hydrogen peroxide (30–40%) and potassium permanganate of pharmaceutical grade; titer standards of oxalic acid; chemically pure grade sodium chloride; and hydrochloric acid of special purity grade. Solutions of trivalent cobalt were obtained during the interaction between ozone and solutions of bivalent cobalt in sulfuric acid (0.001–0.1 M CoSO_4 in 0.1–8.2 M H_2SO_4) in a bubble-type reactor at room temperature (20–22°C). The experimental unit described in [8] was used. The reactor was a glass cylinder (internal diameter, 30 mm; height, 40 cm; operating volume, 200 mL) with a porous glass plate sealed in its bottom, through which ozonated oxygen was supplied. In the bottom part, the reactor had a side outlet for collecting samples of the reaction solution. Ozone was obtained by passing extra pure oxygen through a barrier discharge in an ozone generator. The concentration of ozone at the inlet to the reactor (10–70 g/m³) was monitored using a Medzon-254/5 photometer. The flow rate of the initial gas mixture was 21 L/h (std. cond.) in all of our experiments. The concentration of cobalt in the solutions was estimated by means of spectrophotometry. The spectra were registered and the

optical density measured on an Agilent 8453 UV–Vis spectrophotometer; the length of the liquid cell's optical path was 1 cm.

In finding molar absorption coefficients, the concentration of trivalent cobalt was determined by back titration with hydrogen peroxide. Up to 20 mL of a 0.1 M solution of H_2O_2 was added to 10 mL of a solution of sulfuric acid ($C(\text{H}_2\text{SO}_4) = 6\text{--}8.2\text{ M}$) with a Co(III) concentration of no more than 0.1 M. The reaction that occurred was $2\text{Co(III)} + \text{H}_2\text{O}_2 \rightarrow 2\text{Co}^{2+} + \text{O}_2 + 2\text{H}^+$. The amount of residual hydrogen peroxide was determined via titration with a 0.1 M solution of KMnO_4 . The equivalence point was determined from the emergence of a stable color of permanganate that was clearly visible against the pale reddish-pink color of bivalent cobalt. In our experiments, almost no catalytic decomposition of hydrogen peroxide occurred under the action of Co^{2+} ions. The concentration of Co(III) was calculated according to the formula

$$[\text{Co(III)}], \quad M = [V(\text{H}_2\text{O}_2)C(\text{H}_2\text{O}_2) - \frac{5}{2}V(\text{KMnO}_4)C(\text{KMnO}_4)] / V(\text{Co(III)}),$$

where $V(\text{H}_2\text{O}_2) = 20\text{ mL}$ is the volume of the added solution of peroxide; $C(\text{H}_2\text{O}_2)$ is the concentration of peroxide, M; $V(\text{KMnO}_4)$ is the volume of the solution of permanganate consumed for the titration, mL; $C(\text{KMnO}_4)$ is the concentration of permanganate, M; and $V(\text{Co(III)}) = 10\text{ mL}$ is the volume of the portion of the trivalent cobalt solution. The precise concentration of the initial solution of H_2O_2 was determined via permanganometric titration in the presence of sulfuric acid; the titer of KMnO_4 solution was determined using oxalic acid as the primary standard [9]. The molar absorption coefficient of Co(III), $\text{L mol}^{-1}\text{ cm}^{-1}$ at a particular wavelength λ , was calculated according to the formula

$$\varepsilon(\text{Co(III)})_\lambda = (A_\lambda - \varepsilon(\text{Co}^{2+})_\lambda([\text{Co}^{2+}]_0 - [\text{Co(III)}])) / [\text{Co(III)}],$$

where A is the optical density of the solution after ozonation; $[\text{Co(III)}]$ is the concentration of trivalent cobalt in this solution, M; $[\text{Co}^{2+}]_0$ is the concentration of bivalent cobalt in the initial solution, M; and $\varepsilon(\text{Co}^{2+})$ is the molar absorption coefficient of bivalent cobalt, $\text{L mol}^{-1}\text{ cm}^{-1}$.

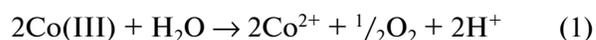
In studying the kinetics of the spontaneous decomposition of trivalent cobalt by means of spectrophotometry, the drop in its concentration in solutions of sulfuric acid was determined as a function of time. The order of the reaction (n) was determined using an integral method, so the integral or semi-integral value of n at which the kinetic curves in the $([\text{Co(III)}]^{(1-n)}, \text{time})$ coordinates $(\ln [\text{Co(III)}], \text{time})$ for $n = 1$) are most linear was sought.

In studying the interaction between trivalent cobalt and chloride ions, an equal volume of HCl or NaCl was rapidly added with a syringe to the solution of Co(III) in sulfuric acid in the cell of the spectrophotometer, and the absorption spectra were registered over time.

RESULTS AND DISCUSSION

Solutions of trivalent cobalt in sulfuric acid have two absorption maxima in the visible spectrum: 406 and 613 nm in 5 M H_2SO_4 and 409–410 and 617–618 nm in 8 M H_2SO_4 (see Fig. 1). The maxima shift slightly toward long wavelengths when the concentration of acid is increased, but the value of the molar absorption coefficient at the maximum remains almost unchanged. The following values were found for the molar absorption coefficients of Co(III) in aqueous solutions of sulfuric acid with concentrations of 1.8–8.2 M: $\varepsilon_{405\text{--}409\text{ nm}} = 52.2\text{ L mol}^{-1}\text{ cm}^{-1}$ and $\varepsilon_{611\text{--}617\text{ nm}} = 44.2\text{ L mol}^{-1}\text{ cm}^{-1}$. At 405–409 nm, the dependence of optical density A on the concentration of trivalent cobalt is linear at least up to $[\text{Co(III)}] = 0.021\text{ M}$ ($A = 1.05$), and up to $[\text{Co(III)}] = 0.071\text{ M}$ ($A = 3.15$) at 611–617 nm. The literature contains values of the absorption coefficients of Co(III) in solutions of perchloric acid: $\varepsilon_{402} = 39.6 \pm 0.2$ [10], $\varepsilon_{400} = 40.0 \pm 0.5$ [11], $\varepsilon_{602} = 34.5 \pm 0.1$ [12], $\varepsilon_{605} = 35.3$ [13], and $\varepsilon_{605} = 35.0 \pm 0.4\text{ L mol}^{-1}\text{ cm}^{-1}$ [11]. The spectrum of Co(III) in 9M H_2SO_4 was presented in [14] (the same spectrum was reproduced in [15]); from it, $\varepsilon_{409} \approx 56$ and $\varepsilon_{615} \approx 50\text{ L mol}^{-1}\text{ cm}^{-1}$ can be obtained. Our results on the absorption coefficients of Co(III) are therefore in agreement with the literature data.

In solutions of sulfuric acid, trivalent cobalt ions decompose according to the stoichiometric equation



and are thus always present along with Co^{2+} ions. In the visible region, the absorption maximum of $\text{Co}^{2+}(\text{aq.})$ is found at 511 nm (see Fig. 1). The molar absorption coefficients of bivalent cobalt determined in this work were $\varepsilon_{405} = 0.35$, $\varepsilon_{511} = 4.83$, and $\varepsilon_{613} = 0.34\text{ L mol}^{-1}\text{ cm}^{-1}$. According to literature data, $\varepsilon_{509} = 4.84\text{ L mol}^{-1}\text{ cm}^{-1}$ [13] for Co^{2+} in aqueous solutions of HClO_4 at $\lambda_{\text{max}} = 509\text{ nm}$; in 2 M H_2SO_4 , $\varepsilon_{511} \approx 6.6\text{ L mol}^{-1}\text{ cm}^{-1}$ [14, 15].

The kinetics of decomposition of trivalent cobalt was studied in aqueous solutions of 0.1–8.2 M sulfuric acid; here, the initial concentration of Co(III) was 3×10^{-3} to $7 \times 10^{-2}\text{ M}$. The reaction is of the second order; i.e., the drop in the concentration of trivalent cobalt is described by the differential equation $-d[\text{Co(III)}]/dt = k_1[\text{Co(III)}]^2$. The rate of decomposition falls dramatically as the concentration of sulfuric acid rises. The half-life period of Co(III) in 0.1 M H_2SO_4 is thus 6 min (at $[\text{Co(III)}]_0 = 3 \times 10^{-3}\text{ M}$); in 6 M H_2SO_4 , it

is 2×10^3 min (at $[\text{Co(III)}]_0 = 1 \times 10^{-2}$ M). We must therefore use high concentrations of H_2SO_4 in the reaction solution to obtain relatively stable preparations of Co(III). The experimental values of constant k_1 can be found using the approximation formula

$$k_1, \text{ L mol}^{-1} \text{ min}^{-1} = 3.26 \times 10^{-2} + 0.725/[\text{H}_2\text{SO}_4]^{1.9}, \quad (2)$$

where $[\text{H}_2\text{SO}_4]$ is the concentration of sulfuric acid, M. Here, the difference between the experimental and calculated values does not exceed 18%. Detailed studies of the kinetics and mechanism of reaction (1) were performed in [16–18]. The mechanism is quite complex and includes stages of the oligomerization and hydrolysis of Co(III) ions. The apparent order with respect to trivalent cobalt assumes values of 1.5–2; the reaction rate is inversely proportional to the concentration of H^+ ions, raised to the power of 1–2 [16–20].

The formation of Co(III) during the oxidation of bivalent cobalt ions by ozone is described by the stoichiometric equation



According to the data of [4, 5, 7], it is of the first kinetic order with respect to concentrations of Co^{2+} and O_3 ; i.e., the rate is determined by the equation $r_3 = k_3[\text{Co}^{2+}][\text{O}_3]$, where k_3 is the rate constant, $\text{L mol}^{-1} \text{ min}^{-1}$, and $[\text{Co}^{2+}]$ and $[\text{O}_3]$ are the current concentrations of bivalent cobalt and ozone in the reaction solution, M. The value of the rate constant of the reaction between Co^{2+} and O_3 , determined by the kinetics of the accumulation of CoAc^{2+} product in solutions of 0.2 M HClO_4 and $(1-40) \times 10^{-3}$ M CH_3COOH , is $37 \text{ L mol}^{-1} \text{ min}^{-1}$ ($0-2^\circ\text{C}$) [7]. Bivalent cobalt ions are efficient catalysts for the reaction between ozone and chloride ions. The values of the rate constant of reaction (3), found from the increase in the rate of chlorine formation upon adding Co^{2+} to the $\text{O}_3-\text{Cl}^--\text{H}^+$ reaction system, are $39.3 \text{ L mol}^{-1} \text{ min}^{-1}$ at 0°C [4] and $85 \text{ L mol}^{-1} \text{ min}^{-1}$ at 20°C [5]. Trivalent cobalt ions are unstable in aqueous solutions, so reaction (1) of their consumption with rate constant k_1 always occurs, along with reaction of formation (3).

Providing that the properties of the bubble-type reactor are close to those of an ideal mixing reactor, chemical transformations occur only in the liquid phase, and the rate of ozone dissolution is determined by the expression $k_L a (H_{\text{O}_3} C(\text{O}_3) - [\text{O}_3])$. The kinetics of the accumulation of trivalent cobalt during the ozonation of solutions of Co^{2+} is then described by a system of differential equations:

$$\begin{aligned} d[\text{Co(III)}]/dt &= k_3[\text{Co}^{2+}][\text{O}_3] - k_1[\text{Co(III)}]^2, \\ d[\text{O}_3]/dt &= k_L a (H_{\text{O}_3} C(\text{O}_3) - [\text{O}_3]) \\ &\quad - k_3[\text{Co}^{2+}][\text{O}_3], \end{aligned} \quad (4)$$

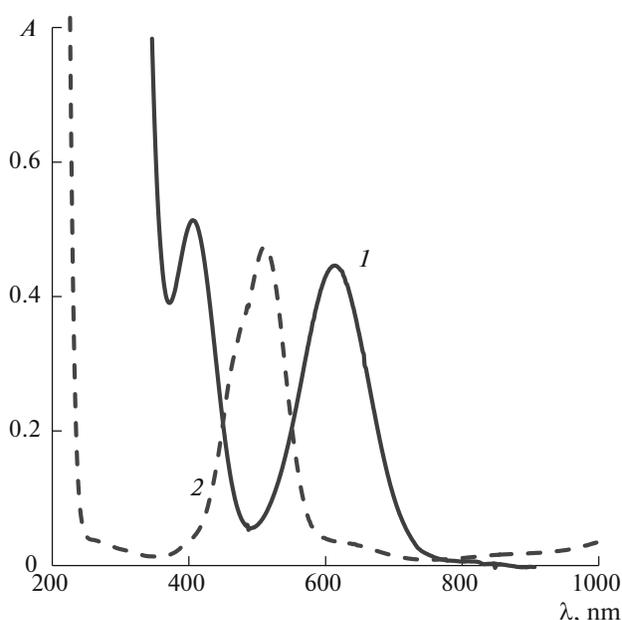


Fig. 1. Spectra of cobalt ions in solutions of sulfuric acid: (1) 0.01 M Co(III) in 5 M H_2SO_4 and (2) 0.1 M CoSO_4 in 0.5 M H_2SO_4 . The compensation solution was water; the optical path length was 1 cm.

$$\begin{aligned} \frac{V_{\text{gas}}}{V_{\text{liq}}} \frac{dC(\text{O}_3)}{dt} &= \frac{v}{V_{\text{liq}}} C(\text{O}_3)^\circ \\ &\quad - \frac{v}{V_{\text{liq}}} C(\text{O}_3) - k_L a (H_{\text{O}_3} C(\text{O}_3) - [\text{O}_3]) \end{aligned}$$

with the mass balance equation

$$[\text{Co}^{2+}] + [\text{Co(III)}] = [\text{Co}^{2+}]_0 \quad (5)$$

and initial conditions at $t = 0$

$$[\text{Co(III)}]_0 = 0, [\text{O}_3]_0 = 0, C(\text{O}_3)_0 = 0. \quad (6)$$

In expressions (4)–(6), $[\text{Co}^{2+}]_0$ is the initial concentration of CoSO_4 , M, $C(\text{O}_3)$ and $C(\text{O}_3)^\circ$ are the concentrations of ozone inside and at the inlet to the reactor, mol/L; $k_L a$ is the volumetric mass transfer coefficient, min^{-1} ; H_{O_3} is the dimensionless Henry constant of ozone equal to the ratio of molar volumetric concentrations in the solution and in the gas phase under equilibrium conditions, where both concentrations are expressed in the same units; $v = 21 \text{ L/h} = 0.35 \text{ L/min}$ is the flow rate of ozonated oxygen through the reactor; $V_{\text{liq}} = 0.2 \text{ L}$ is the volume of the liquid solution in the reactor; and V_{gas} is the volume of the gas phase in the reactor, L. The Henry constants of ozone H_{O_3} in solutions of sulfuric acid with different concentrations were determined in [21]; our understanding is that the values of H_{O_3} are almost the same in solutions of sulfuric acid with the addition of cobalt ions.

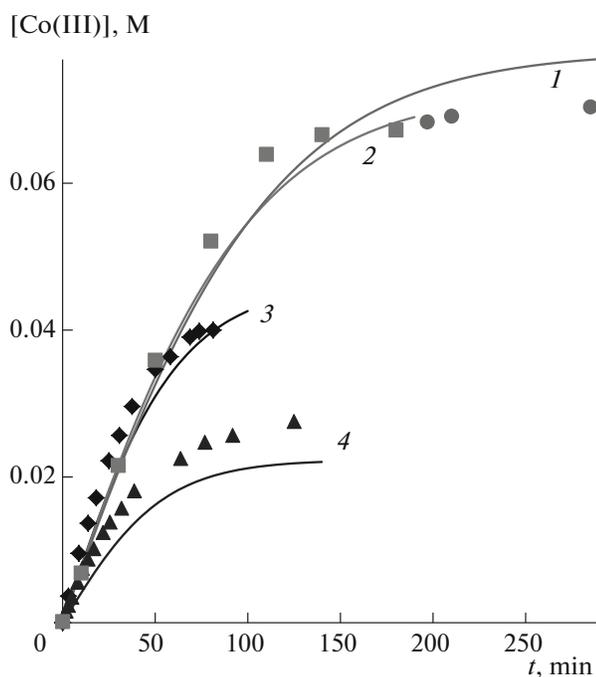


Fig. 2. Examples of dependences of the concentration of Co(III) on the duration of ozonation of solutions of 0.1 M CoSO₄ in sulfuric acid. Points denote experimental data; lines, the numerical solution to system (4)–(6). The values of constant k_1 were calculated according to formula (2): $k_3 = 85 \text{ L mol}^{-1} \text{ min}^{-1}$, $[\text{H}_2\text{SO}_4] = (1) 8.2, (2) 5, (3) 1.8,$ and $(4) 1 \text{ M}$; $C(\text{O}_3) = (1, 2) 70, (3) 60,$ and $(4) 30 \text{ g/m}^3$.

Problem (4)–(6) was solved numerically according to the Rosenbrock method using estimates of parameters $k_L a = 15 \text{ min}^{-1}$ (based on the data of [22]), $V_{\text{gas}} = 0.056 V_{\text{liq}}$, and $k_3 = 85 \text{ L mol}^{-1} \text{ min}^{-1}$ [5]. Constant k_1 was determined according to formula (2). As can be seen from Fig. 2, calculation dependences $[\text{Co(III)}](t)$ satisfactorily reproduce the experimental data. This testifies to the adequacy of kinetic model (4)–(6) of formation of Co(III) during the ozonation of solutions of CoSO₄ in sulfuric acid and confirms the correctness of the value of the rate constant of reaction (3) obtained in [5].

Trivalent cobalt ions have very strong oxidative properties and are one of the few substances that

actively oxidize chloride ions. The process is described by the stoichiometric equation



We performed a series of experiments devoted to investigating the kinetics of interaction between Co(III) and Cl⁻ (table). Typical spectra of the reaction system are shown in Fig. 3. The peak at 511 nm results from Co²⁺ ions, considerable concentrations of which were present in the initial solution. Other maxima are observed at 460 and 650 nm, and the absorption at these wavelengths falls rapidly over time. Comparing the spectra in Fig. 3 and the spectra of Co(III) and Co²⁺ in solutions of sulfuric acid with no chloride ions (Fig. 1), we may conclude that the two maxima correspond to the intermediate compound of Co(III) with Cl⁻.

It is known [23, 24] that reaction (7) proceeds in two stages. In the first stage, the intermediate compound, a chloride complex of trivalent cobalt CoCl²⁺ [23, 24], forms very fast (the formation of small quantities of CoOHCl⁺ is also possible [24]). At the second and limiting stage, there is relatively slow consumption of the complex. The mechanism of the second stage of reaction (7) is unknown. The available instruments allowed us to obtain spectra only at the second stage of the decomposition of CoCl²⁺. It can be shown that the difference between the optical densities at 650 nm at any time and upon completion of the reaction is directly proportional to the current concentration of the intermediate compound $[\text{CoCl}^{2+}] \sim A_{650}(t) - A_{650}(\infty)$, while the ratio of the current and initial concentrations is determined by the expression $[\text{CoCl}^{2+}]/[\text{CoCl}^{2+}]_0 = (A_{650}(t) - A_{650}(\infty))/(A_{650}(0) - A_{650}(\infty))$. The decomposition of CoCl²⁺ cannot be presented as a simple reaction with a certain order and rate constant because it is impossible to select any value of $n \geq 0$ at which the kinetic curves are linear within the $([\text{CoCl}^{2+}]^{(1-n)}, \text{time})$ coordinates $((\ln [\text{CoCl}^{2+}], \text{time})$ when $n = 1$). Kinetic curves in semilogarithmic coordinates are shown in Fig. 4. Let us determine the specific rate of the fall in the concentration of CoCl²⁺:

$$r_{\text{CoCl}^{2+}} = \frac{1}{[\text{CoCl}^{2+}]} \frac{d[\text{CoCl}^{2+}]}{dt} = \frac{d \ln [\text{CoCl}^{2+}]}{dt}.$$

Composition of initial solutions and specific rate of the reaction between Co(III) and chloride ions

No.	[Co(III)], M	[H ₂ SO ₄], M	[HCl], M	[NaCl], M	$r_{\text{CoCl}^{2+}}, \text{min}^{-1}$ ($t < 0.1 \text{ min}$)	$r_{\text{CoCl}^{2+}}, \text{min}^{-1}$ ($t > 0.4 \text{ min}$)
1	0.052	2	—	1	17	2.5
2	0.026	2	—	1	16	2.5
3	0.0089	4	0.33	—	26	1.9
4	0.01	1.8	0.25	—	6.4	1.4

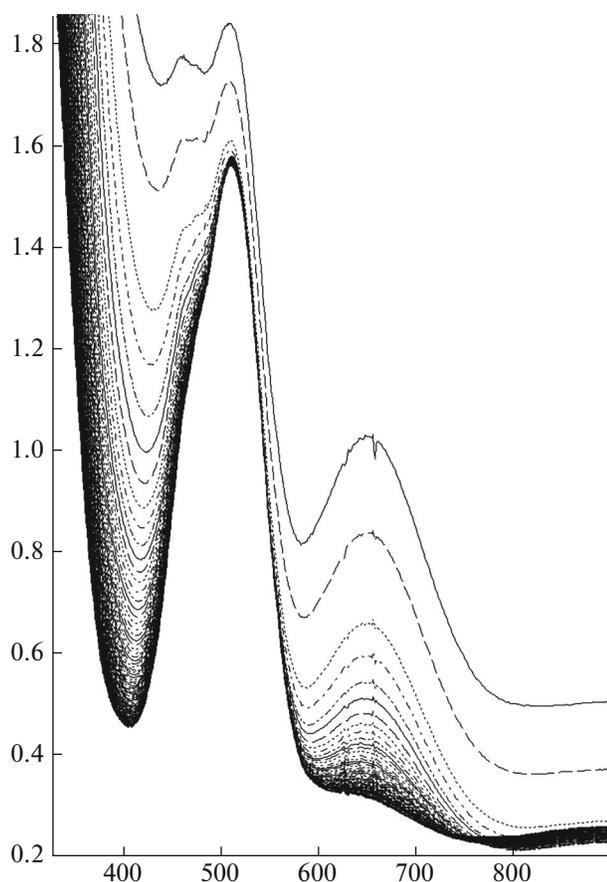


Fig. 3. Example of the change in the spectra of the Co(III) + Cl⁻ reaction system over time. The initial composition was [NaCl] = 1 M, [H₂SO₄] = 2 M, [Co(III)] = 0.078 M, and [Co²⁺] = 0.5 M. The spectra were registered at intervals of 1 s.

Its values, estimated from the linear sections of the kinetic curves in Fig. 4 using the equation

$$r_{\text{CoCl}^{2+}} = \frac{\Delta \ln[\text{CoCl}^{2+}]}{\Delta t} = \left(\frac{\ln \frac{A_{650}(t_2) - A_{650}(\infty)}{A_{650}(0) - A_{650}(\infty)}}{-\ln \frac{A_{650}(t_1) - A_{650}(\infty)}{A_{650}(0) - A_{650}(\infty)}} \right) / (t_2 - t_1)$$

are presented in the table. The specific rate is highest in the initial section at $t < 0.1$ min. It slows at $0.1 \text{ min} < t < 0.4$ min and reaches a constant value of $1.4\text{--}2.5 \text{ min}^{-1}$ when $t > 0.4$ min. We may tentatively conclude that the specific rate of decomposition of CoCl²⁺ does not depend on the initial concentration of trivalent cobalt and increases along with the concentrations of H⁺ and Cl⁻ in the reaction solution.

CONCLUSIONS

Our results allow detailed characterization of the kinetics of the accumulation and decomposition of Co(III) during the ozonation of solutions of CoSO₄ in

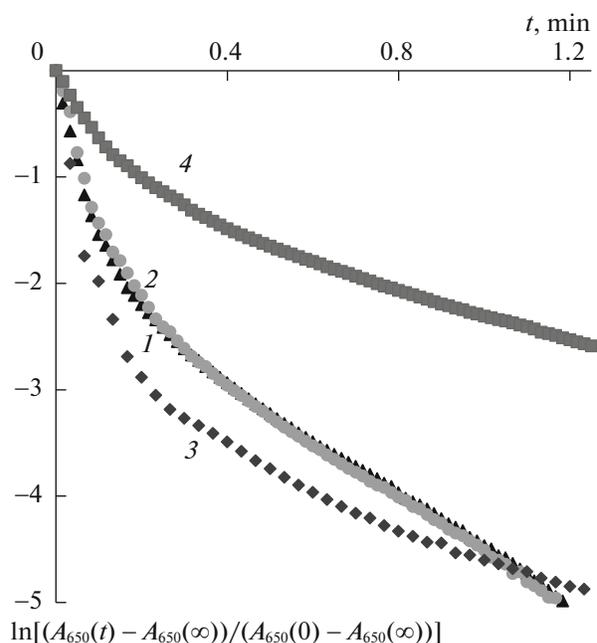


Fig. 4. Kinetic curves of the reduction of trivalent cobalt by chloride ions. The initial compositions of the reaction solutions are presented in the table.

sulfuric acid. The obtained data on the kinetics of interaction between Co(III) and Cl⁻ shows that catalytic oxidation of Cl⁻ according to the transfer mechanism $\text{Co}^{2+} \xrightarrow{+\text{O}_3} \text{Co(III)} \xrightarrow{+\text{Cl}^-} \text{Co}^{2+} + \text{Cl}_2$ is possible during the ozonation of acidic solutions of chlorides with additions of Co²⁺. The interaction between Co²⁺ and O₃ is the limiting stage.

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