

The study of hydroxyl complexation processes in Fe(III)–Fe(II)–Na(H)Cl–H₂O system

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Abstract. Hydroxyl complexation in the Fe(III)–Fe(II)–Na(H)Cl–H₂O system at different ionic strengths of the solution, temperature 308.16 K, was studied by the Clark–Nikolsky oxidation potential method. The formation of the following mono- and binuclear coordination compounds is shown $[\text{Fe}(\text{OH})(\text{H}_2\text{O})_5]^{2+}$; $[\text{Fe}_2(\text{OH})_2(\text{H}_2\text{O})_{10}]^{4+}$; $[\text{Fe}(\text{OH})_2(\text{H}_2\text{O})_4]^+$; $[\text{Fe}(\text{OH})_3(\text{H}_2\text{O})_3]^0$. It was found that Fe(II) does not form hydroxo complexes at the studied concentration parameters, and the pH range from 1.0 to 3.0. Above this pH value, Fe(III) hydrolyzes and forms a brown precipitate.

1 Introduction

Understanding the nature of electrolyte solutions, primarily the state of the solvent and dissolved substances, is important for explaining the reactions in solution. The main problem that arises in studying the interaction between the components of electrolyte solutions is the mutual influence of charged particles and water molecules as solvent and the structure of the basic particles. In this case, the hydrolysis of metal ions is of paramount importance due to its universal nature [1-4].

When studying the reaction of iron(III) hydrolysis in solutions, both the interaction of metal ions with the solvent and the influence of the background electrolyte ions used to maintain the constancy of the ionic strength on the structure of the iron complex hydrate shell should be taken into account. In the aquacomplex $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$, the coordinated water molecules are constantly exchanged for water molecules from the solution. Therefore, the nature and concentration of the background electrolyte significantly affect the structure of water as a solvent and the hydration processes in aqua complexes. The hydrolysis of iron(III) ions proceeds stepwise with the successive formation of hydrolytic products, and their dimerization at any stage of the reaction is possible [5-10].

To obtain quantitative information on the processes occurring in the Fe(III)–Fe(II)–NaHCl–H₂O system, to establish the exact composition of the most probable forms of Fe(III) hydroxo complexes, to calculate the hydrolysis constant, and to determine their dominance areas, we used the Clark–Nikolsky redox potential method [6, 201-203, 7, 94-96, 8, 74-77, 9, 27-29]. Sodium chloride was chosen as a background electrolyte. The studies were carried out at the following concentration conditions: $C_{\text{Fe(III)}} = C_{\text{Fe(II)}} = 0.001$ mol/l, temperature 308.16 K, and ionic strengths 0.1; 0.2; 0.25; 0.50; 1.00 [10- 11].

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2 Experimental procedure

The oxidation potential of the system is determined by potentiometry. Two galvanic elements are used:

Pt test solution || KCl (sat.), AgCl | Ag (I)

Ag | AgCl, (HCl 0.1 N) glass | test solution || KCl (sat.), AgCl | Ag (II)

The first galvanic cell is used to measure the system's EMF, and the second to determine the pH of the solution in the electrolytic cell. To prevent Fe(II) oxidation, inert gas (nitrogen or argon) is continuously passed through the working solution. EMF is measured using an ionometer [12].

The working solutions contain equimolecular Fe(II) and Fe(III) amounts. The ionic strength of the titrated solution is adjusted to a constant value with HCl and the titrant with a NaCl solution. The concentration of Fe(III) in the cell is minimum, and the titrant is maximum.

3 Results and Discussion

Hydroxyl complexation can be investigated by various methods [13-14]. In our works [15, 16]. The compositions of the formed iron hydroxo complexes were determined according to the theory of oxredmetry method based on the combined analysis of the slope tangents of the experimental dependences of the oxidation potential on the solution pH, pCO, and pCr. The curve of the dependence of the oxidative potential on pH (Fig. 1) indicates a decrease in the potential with increasing pH. This is associated with decreased Fe(III) in the system because part of the iron is consumed to form the hydroxo complex.

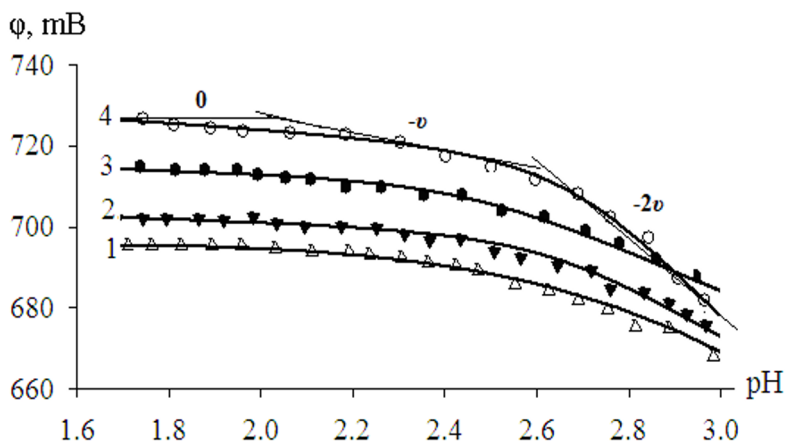
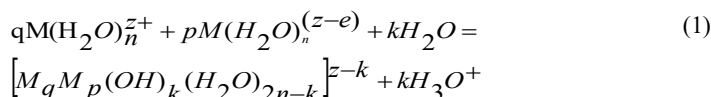


Fig. 1. Dependence of oxidative potential of Fe(III)-Fe(II)-Na(H) system Cl-H₂O from pH at temperature of 308.16 K, $C_{\text{Fe(III)}} = C_{\text{Fe(II)}} = 1 \cdot 10^{-3}$ mol/l. Curves refer to ionic forces of solution (mol/l). 1-0.10; 2-0.25; 30.50; 4-1.0. Points were obtained experimentally; curves were calculated using SigmaPlot 10.0 program.

The curves of the presented figure are obtained from experimental values of the oxidative potential of the system at different ionic forces of the solution. The calculations were carried out using the SigmaPlot 10.0 program [15]. If rectilinear sections are drawn on the curves of this figure, then the following values of the angular coefficients can be obtained: 0, -u and -2v, where: $= \frac{2.303RT}{F}$; R is the universal gas constant; T is the Kelvin temperature; F is the Faraday number. In addition, the following designations are introduced: q and p are

the numbers of atoms of the oxidized and reduced forms of the metal of the complexing agent, respectively, k is the total number of hydroxyl groups of the complex, s is the number of protons and l is the number of ligands in the formed complex, if there is a separate ligand in the solution.

The equation of the reaction of the formation of hydroxo complexes can be represented as:



where, q and p is the nuclearity of complex compounds; n - the coordination number of metals – oxidized and $M^{(z-e)+}$ - reduced forms; z^+ and $(z-e)^+$ charges of complex particles; $j = 2n - k$ the number of hydroxyl groups coordinated by a central atom;

For reaction (1), the equation of the oxidative potential of the system has the following form:

$$\varphi = \varphi^0 + \frac{\nu}{e} \cdot \lg \frac{[M^{z+}]}{[M^{(z-e)+}]} \quad (2)$$

To compile a material balance, it is necessary to find the equations of equilibrium concentrations of metal ions of oxidized and reduced forms; they will have the following form:

$$[M^{z+}] = \left(C_o \sum_1^q \sum_0^k q \beta_{qkj}^{1/q} G_{qkj}^{(q-1)/q} [M^{(z-e)+}]^q \right) \quad (3)$$

$$[M^{(z-e)+}] = \left(C_r \sum_1^p \sum_0^k p \beta_{pkj}^{1/p} G_{pkj}^{(p-1)/p} [M^{z+}]^p \right) \quad (4)$$

In these equalities G_{qkj} and G_{pkj} are the equilibrium concentrations of hydroxo complexes of oxidized and reduced forms of iron.

Taking into account equations (3) and (4), a new general equation of the oxidative potential for the systems is found: M_q - M_p -background electrolyte- H_2O :

$$\varphi = \varphi^0 + \frac{\nu}{e} \cdot \lg \frac{[M^{z+}]}{[M^{(z-e)+}]} = \varphi^0 + \frac{\nu}{e} \cdot \lg \frac{\left(C_o \sum_1^q \sum_0^k q \beta_{qkj}^{1/q} G_{qkj}^{(q-1)/q} [M^{(z-e)+}]^q \right)}{\left(C_r \sum_1^p \sum_0^k p \beta_{pkj}^{1/p} G_{pkj}^{(p-1)/p} [M^{z+}]^p \right)} \quad (5)$$

The partial derivative of equation (1) of pH has the following form:

$$\left(\frac{\partial \varphi}{\partial pH} \right)_{pC_o, pC_r} = \nu \left[\left(\frac{\nu}{p} \right) - \left(\frac{\nu}{q} \right) \right] \quad (6)$$

where v and y are the numbers of hydroxyl groups coordinated by the oxidized and reduced forms of the metal. Expression (6) gives the total number of hydroxyl ions coordinated around the complexing metal, calculated from the slope of the experimental curve φ from pH.

According to the theory of the method, to determine the number of Fe(III) atoms in a compound, it is necessary [17, 18] to have experimental dependences of the oxidative potential φ on pC_0 obtained at pH values corresponding to slopes $-v$ and $-2v$. This dependence is shown in Figure 2, where the experimental points within an error of $\pm 1-2$ mV deviate from the curve calculated according to the SigmaPlot 10.0 program. On the obtained dependence, linear sections with the tangent of the angle of inclination equal to $-u$ and $-u/2$ can be distinguished. The number of Fe(III) atoms in the hydroxo complex is determined by joint analysis of the slope of the curves $\varphi - pC_0$ and the first derivative of the general equation of the oxidative potential according to rSo:

$$\left(\frac{\partial \varphi}{\partial pC_0} \right)_{pH, pC_r} = -\frac{v}{q} \quad (7)$$

It follows from Equation 7 that q is equal to 1 and 2. Consequently, water-soluble mononuclear $[\text{Fe}(\text{OH})(\text{H}_2\text{O})_5]^{2+}$, $[\text{Fe}(\text{OH})_2(\text{H}_2\text{O})_4]^+$ and binuclear $[\text{Fe}_2(\text{OH})_2(\text{H}_2\text{O})_{10}]^{4+}$, $[\text{Fe}_2(\text{OH})(\text{H}_2\text{O})_{11}]^{5+}$ iron hydroxo compounds are formed in the solution.

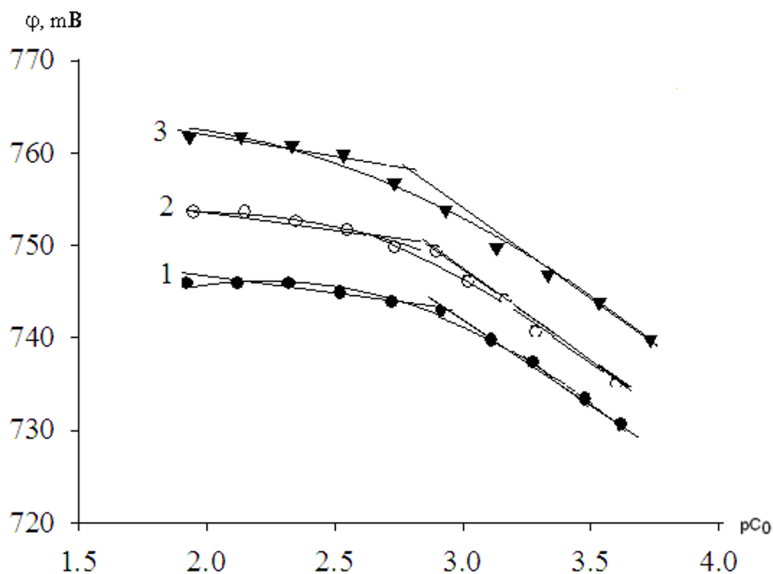


Fig. 2. Dependence of oxidative potential φ of Fe(III)-Fe(II)- Na(H)Cl-H₂O system from pC_0 at temperature of 308.16 K, $C_{\text{Fe(III)}} = C_{\text{Fe(II)}} = 1 \cdot 10^{-3}$ mol/l; pH = 2.20. Curves are calculated according to program SigmaPlot -10.0, and points are experimentally measured values at ionic forces of solution: 1 - 0.25; 2 - 0.50; 3 - 1.0.

The data from the first 2 figures allowed us to determine the number of iron(III) atoms and OH ions in the resulting compounds. The study of the experimental dependence of the oxidative potential φ on pC_r in the pH range of the solution 1.5 - 4.0 indicates that divalent iron does not form hydroxo complexes, because the slope of the experimental curve was zero.

Calculations using the oxidative function involve the calculation of equilibrium constants or the formation of hydroxo compounds of the general formula:

$$\left[M_q M_p (OH)_k \right]^{q+p-k}$$

To carry out the necessary calculations using computer programs, we used the principle of modeling equilibria in redox systems [18]. A stoichiometric matrix has been compiled (Table. 1), which includes the values of the slopes of the experimental dependence curves φ - pH; φ - pC_0 , the interval of their finding on the pH scale, the composition of the resulting complexes and their hydrolysis constants.

Based on the results obtained, for the studied system Fe(III)-Fe(II)-Na(H)Cl-H₂O, a stoichiometric matrix of slope values of all experimental curves was created (Table 1).

Table 1. Stoichiometric matrix of slope values of experimental dependences of oxidative potential on concentration parameters of Fe (III)-Fe (II)-Na(H)Cl-H₂O system at temperature of 298.16 K, ionic strength of solution is 1.0 mol/l

№	Interval slopes on the pH scale	Ratios of pH and pC_0 expressed in ν		Composition of the resulting complexes	(-lgK _{gpk}) Constants
		pH	pC_0		
				[Fe(OH)(H ₂ O) ₅] ²⁺	lgK ₁₀₁
1	1.6 – 2.8	- ν	- ν	[Fe ₂ (OH) ₂ (H ₂ O) ₁₀] ⁴⁺	lgK ₂₀₂
2	2.6 – 3.0	- ν	- $\nu/2$	[Fe(OH) ₂ (H ₂ O) ₄] ⁺	lgK ₁₀₂
3	2.3 – 3.1	- ν	- $\nu/2$, - ν	[Fe(OH) ₃ (H ₂ O) ₃] ⁰	lgK ₁₀₃
4	2.7 – 3.2	-2 ν	- $\nu/2$, - ν	[Fe(OH)(H ₂ O) ₅] ²⁺	lgK ₁₀₁

The chemical model of hydroxyl complexation is based on the data of the stoichiometric matrix (Table 2).

Table 2. Chemical model of hydroxyl complex compounds in Fe(III)-Fe(II)-Na(H)Cl – H₂O system.

№	Equilibrium equations of complexation
1	[Fe(H ₂ O) ₆] ³⁺ + HOH ↔ [Fe(OH)(H ₂ O) ₅] ²⁺ + H ₃ O ⁺
2	2[Fe(OH)(H ₂ O) ₅] ²⁺ ↔ [Fe ₂ (OH) ₂ (H ₂ O) ₁₀] ⁴⁺
3	[Fe(OH)(H ₂ O) ₅] ²⁺ + HOH ↔ [Fe(OH) ₂ (H ₂ O) ₄] ⁺ + H ₃ O ⁺
4	[Fe(OH) ₂ (H ₂ O) ₄] ⁺ + HOH ↔ [Fe(OH) ₃ (H ₂ O) ₃] ⁰ + H ₃ O ⁺

To compile models of chemical equilibria, it is necessary to consider all existing and interacting particles of the system, which are called basic. The basic thermodynamic parameters of the basic particles are called model ones. A mathematical matrix in the form of tables allows you to quickly calculate the necessary model parameters using computer programs.

The constants of the formation of iron(III) hydroxo complexes were calculated by iteration of the experimental and theoretical oxidative function [18]. According to the curves of the dependence of the oxidative potential on pH (Fig. 1), previously, under the condition $C_0 \neq C_r$, the numerical values of the experimental oxidative function were calculated according to the equation.

$$f_3^0 = \frac{C_r}{C_0} \exp \frac{(\varphi - \varphi^0)n}{\mathcal{G}} \quad (8)$$

Taking into account the composition of the resulting iron hydroxo complexes and the addition of fragments of the equations of the oxidative potential of the rectilinear sections of the dependence ϕ - pH, we have:

$$f_r^0 = \frac{h^3}{h^3 + K_{101} \cdot h^2 + 2K_{202}^{1/2} \cdot G_{202}^{1/2} \cdot h^2 + K_{102} \cdot h + K_{103}} \quad (9)$$

where: f_r^0 is theoretical oxidative function activity of hydrogen ions, $h = [H^+]$ is theoretical oxidative function activity of hydrogen ions.

Using the values of the oxidative potential at given pH values, the experimental oxidative function (f_e) values are determined, and its dependence on pH is constructed. The values of hydrolysis constants were determined by the iteration method of experimental (f_e) and theoretical (f_t) oxidative functions according to the Excel program. As an example (Fig. 3), the dependences of theoretical and experimental oxidative functions on pH at an ionic strength of 1.00 mol/l are presented. The theoretical curve of the oxidative function is approximated from the experimental one to the maximum coincidence

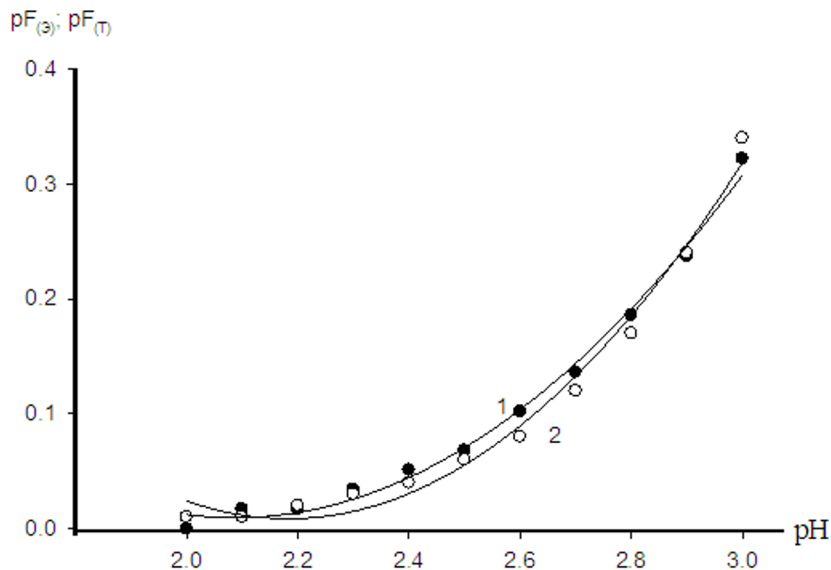


Fig. 3. Dependences of experimental $pf(e)$ and theoretical $pf(t)$ oxidative functions on pH of solution for Fe(III)-Fe(II)-Na(H)Cl-H₂O system at 308.16 K and ionic strength of solution is 0.50 mol/l. Curves: 1; 2 are experimental and theoretical, respectively.

As seen from the above figure, the theoretical curve coincides well with the experimental one, which indicates the results' reliability. The values of the true hydrolysis constants, fragments of the equation of the oxidative function, the interval of existence, and the maximum fraction of the formed coordination compound, are given in Tables 3 and 4. They are called the table of model parameters because it includes all the values determined using the compiled chemical model.

K_{101} , K_{202} , K_{102} and K_{103} – the values of the hydrolysis constants of the following hydroxo complexes: $[Fe(OH)(H_2O)_5]^{2+}$; $[Fe_2(OH)_2(H_2O)_{10}]^{4+}$; $[Fe(OH)_2(H_2O)_4]^+$ and $[Fe(OH)_3(H_2O)_3]^0$, respectively, G_{202} is the equilibrium concentration of the dimer

$[\text{Fe}_2(\text{OH})_2(\text{H}_2\text{O})_{10}]^{4+}$. For the intended composition (Table 1), the approximate values of the hydrolysis constants were calculated using fragments of the equation.

Table 3. Stability of Fe(III) hydroxyl complexes formed in Fe(III)-Fe(II)-Na(H)Cl - H₂O system at temperature of 308.16 K, $C_{\text{Fe(III)}} = C_{\text{Fe(II)}} = 1 \cdot 10^{-3}$, ionic strength of solution $I = 0.50$ mol / L.

№	Composition of the resulting complexes	The range of images. slope on the pH scale	The constant is stable - lgk_{apk}	Hydrolysis constant - lgk_{apk}
1	$[\text{Fe}(\text{OH})(\text{H}_2\text{O})_5]^{2+}$	1.6 – 2.8	11.18	2.82±0.03
2	$[\text{Fe}_2(\text{OH})_2(\text{H}_2\text{O})_{10}]^{4+}$	2.6 – 3.0	11.11	2.89±0.04
3	$[\text{Fe}(\text{OH})_2(\text{H}_2\text{O})_4]^+$	2.3 – 3.1	8.75	5.25±0.06
4	$[\text{Fe}(\text{OH})_3(\text{H}_2\text{O})_3]^0$	2.7 – 3.2	-	8.47±0.02

Table 4. Model parameters of Fe(III) hydroxyl complexes formed in Fe(III)-Fe(II)-Na(H)Cl - H₂O system at temperature of 308.16 K, $C_{\text{Fe(III)}} = C_{\text{Fe(II)}} = 1 \cdot 10^{-3}$, ionic strength of solution $I = 0.50$ mol/L.

№	Composition of the resulting complexes	α_{max} , %	pH	Partial equations of the oxidative function
1	$[\text{Fe}(\text{OH})(\text{H}_2\text{O})_5]^{2+}$	24	2.6	$1/K_{101}^{-1} \cdot h^{-1}$
2	$[\text{Fe}_2(\text{OH})_2(\text{H}_2\text{O})_{10}]^{4+}$	4.0	2.9	$1/2K_{202}^{1/2} \cdot G_{202}^{1/2} \cdot h^{-1}$
3	$[\text{Fe}(\text{OH})_2(\text{H}_2\text{O})_4]^+$	39	2.7	$1/K_{102} \cdot h^{-2}$
4	$[\text{Fe}(\text{OH})_3(\text{H}_2\text{O})_3]^0$	18	3.0	$1/K_{103} \cdot h^{-3}$

The molar fractions (degrees of accumulation) of complexes are calculated by the equations:

$$\alpha_{\text{Fe}^{3+}} = \frac{h^3}{h^3 + K_{101} \cdot h^2 + 2\sqrt{K_{202}} \cdot \sqrt{G_{\text{dimer}}} \cdot h^2 + K_{102} \cdot h + K_{103}} \cdot 100 \quad (10)$$

$$\alpha_{[\text{Fe}(\text{OH})(\text{H}_2\text{O})_5]^{2+}} = \frac{K_{101} \cdot h^2}{h^3 + K_{101} \cdot h^2 + 2\sqrt{K_{202}} \cdot \sqrt{G_{\text{dimer}}} \cdot h^2 + K_{102} \cdot h + K_{103}} \cdot 100 \quad (11)$$

$$\alpha_{[\text{Fe}_2(\text{OH})_2(\text{H}_2\text{O})_{10}]^{4+}} = \frac{2\sqrt{K_{202}} \cdot \sqrt{G_{\text{dimer}}} \cdot h^2}{h^3 + K_{101} \cdot h^2 + 2\sqrt{K_{202}} \cdot \sqrt{G_{\text{dimer}}} \cdot h^2 + K_{102} \cdot h + K_{103}} \cdot 100 \quad (12)$$

$$\alpha_{[\text{Fe}(\text{OH})_2(\text{H}_2\text{O})_4]^+} = \frac{K_{102} \cdot h}{h^3 + K_{101} \cdot h^2 + 2\sqrt{K_{202}} \cdot \sqrt{G_{\text{dimer}}} \cdot h^2 + K_{102} \cdot h + K_{103}} \cdot 100 \quad (13)$$

$$\alpha_{[\text{Fe}(\text{OH})_3(\text{H}_2\text{O})_3]^0} = \frac{K_{103}}{h^3 + K_{101} \cdot h^2 + 2\sqrt{K_{202}} \cdot \sqrt{G_{\text{dimer}}} \cdot h^2 + K_{102} \cdot h + K_{103}} \cdot 100 \quad (14)$$

The calculated values of the degrees of accumulation were used to construct a diagram of the distribution of Fe(III) hydroxo complexes (Fig. 4).

The maximum degree of accumulation falls on the complex of the composition $[\text{Fe}(\text{OH})_2(\text{H}_2\text{O})_4]^+$; it is 39 % at pH 2.7. In terms of stability, Fe(III) hydroxocomplexes form the following series: $[\text{Fe}(\text{OH})(\text{H}_2\text{O})_5]^{2+} < [\text{Fe}_2(\text{OH})_2(\text{H}_2\text{O})_{10}]^{4+} < [\text{Fe}(\text{OH})_2(\text{H}_2\text{O})_4]^+ < [\text{Fe}(\text{OH})_3(\text{H}_2\text{O})_3]^0$.

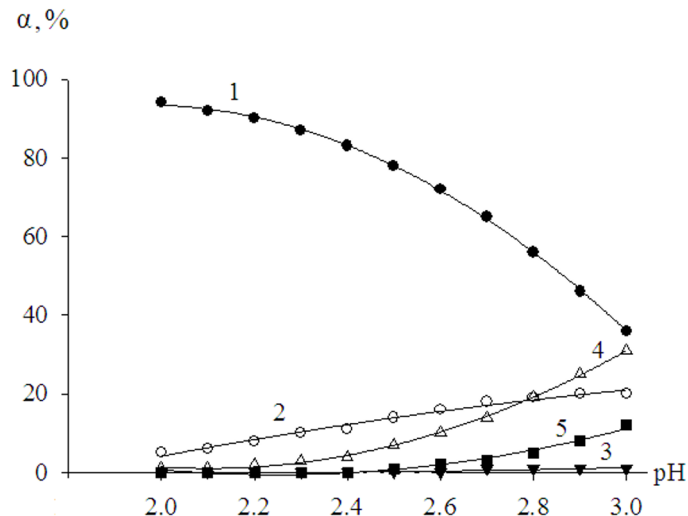


Fig. 4. Diagram of distribution of Fe (III) hydroxo complexes in Fe (III) – Fe (II) – Na(H)Cl – H₂O system at temperature of 308.16 K ionic strength of solution $I = 0.50$; $C_{\text{Fe(III)}} = C_{\text{Fe(II)}} = 1 \cdot 10^{-3}$ mol/L. 1 – $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$; 2 – $[\text{Fe}(\text{OH})(\text{H}_2\text{O})_5]^{2+}$; 3 – $[\text{Fe}_2(\text{OH})_2(\text{H}_2\text{O})_{10}]^{4+}$; 4 – $[\text{Fe}(\text{OH})_2(\text{H}_2\text{O})_4]^+$; 5 – $[\text{Fe}(\text{OH})_3(\text{H}_2\text{O})_3]^0$.

4 Conclusions

The greatest scientific interest is the study of the behavior and mechanisms of the formation of hydroxo complexes [19- 21]. In our work, the dependencies of the content of various hydroxo complexes in a certain medium are established. Thus, it was determined that the maximum content of hydroxo complexes is different in the studied pH range.

For example, for $[\text{Fe(III)}]$ content varies from 99 to 39 %, the maximum content for $[\text{Fe}(\text{OH})(\text{H}_2\text{O})_5]^{2+}$ is equal to 24 %, for $[\text{Fe}_2(\text{OH})_2(\text{H}_2\text{O})_{10}]^{4+}$ – 4%, for iron dihydroxide $[\text{Fe}(\text{OH})_2(\text{H}_2\text{O})_4]^+$ – 39 %, and for $[\text{Fe}(\text{OH})_3(\text{H}_2\text{O})_3]^0$ this parameter is 18 %.

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