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Oxidative Dehydrogenation in Complexes of Transition Metals (Cu(II), Co(II), Ni(II)) with *N*,*N*'-Di(2-Hydroxybenzyl)diamines

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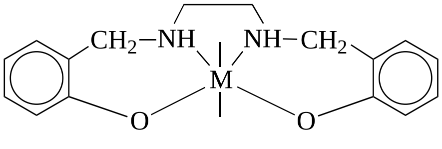
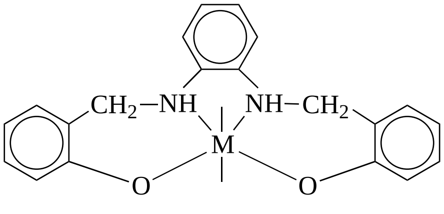
Abstract—Potentially tetradentate ligands *N*,*N*'-di(2-hydroxybenzyl)ethylenediamine (L^1) and *N*,*N*'-di(2-hydroxybenzyl)*o*-phenylenediamine (L^2) and complexes of Cu(II), Co(II), and Ni(II) with L^1 and L^2 were synthesized. The EPR and electronic spectroscopy methods were used to reveal the octahedral structure of the Cu(II) complex with L^1 in the solid state. In water–alcohol solutions, the Cu(II) and Ni(II) complexes with both ligands have distorted octahedral structures. The Co(II) complexes form dioxygen adduct with L^1. In the presence of oxygen, the ligands in the obtained complex compounds can undergo oxidative dehydrogenation with selective formation of the respective disalicylaldimines. In the case of L^2, the oxidative dehydrogenation is observed for the complexes of all studied metals in comparatively mild conditions ($T = 30°C$, methanol and other solvents), while in the case of L^1, it occurs only with the Co(II) complexes in the presence of pyridine.

The reactions of oxidative dehydrogenation of coordinated ligands are well known in the literature [1–4]. There are some publications on dehydrogenation of coordinated primary amines which convert them into nitriles [5–11], of diamines into diimines [12–19], as well as on dehydrogenation of the macrocyclic [20–29] and of other ligands [29–34].

While studying the properties of the previously unknown transition-metal complexes with *N*,*N*'-(2-hydroxybenzyl)diamines (ethylele- and *o*-phenylenediamine), we established that their ligands are also dehydrogenated to give the corresponding diimines.

This paper describes the synthesis, structure, and oxidative dehydrogenation of the Co(II), Ni(II), and Cu(II) complexes with *N*,*N*'-di(2-hydroxybenzyl)ethylenediamine (H_2DBED) and *N*,*N*'-di(2-hydroxybenzyl)*o*-phenylenediamine (H_2DBOPD).


M(DBED) · nH_2O


M(DBOPD) · nH_2O

EXPERIMENTAL

IR spectra (KBr pellets) were recorded on a Specord M80 (Carl Zeiss) or Mattson 1000 FTIR (Perkin–Elmer) spectrophotometer. The electronic absorption spectra were recorded on a Specord M40 and on a UV-240 (Shimadzu) spectrophotometer. The EPR spectra were registered on a RE 1306 and on a Radiopan (Poland) radiospectrometer. Derivatographic measurements were performed on a MOM (Hungary) derivatograph.

The oxygen absorption measurements were carried out with the aid of a thermostatically controlled gas buret connected to a thermostatically controlled reactor. Samples of solid complexes or a mixture of a solid acetate of the corresponding metal and a ligand were place in a reactor connected to a calibrated gas buret and a separatory funnel with a pressure leveling. The system was evacuated, while the reactor was filled with oxygen and then with a solvent.

The elemental analysis of the obtained compounds (see the table) was performed by the combustion (carbon and hydrogen) and Dumas (nitrogen) methods.

H_2DBED. Ethylene-*N*,*N*'-disalicylaldimine (26.8 g, 0.1 mol) was suspended in 40 ml of methanol, and

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Elemental analysis data for N,N'-di(2-hydroxybenzyl)diamines and their complexes

Compound	Empirical formula	Content (found/calcd.), %			mp, °C
		C	H	N	
H_2DBED	$C_{16}H_{20}N_2O_2$	70.33/70.56	7.35/7.35	10.63/10.29	123
Cu(DBED)(CH_3COO) · H_2O	$C_{18}H_{24}N_2O_5Cu$	52.83/52.48	6.03/5.87	6.65/6.80	179.5–181
Cu(DBED) · H_2O	$C_{16}H_{20}N_2O_3Cu$	54.50/54.61	5.68/5.72	7.82/7.96	201
Co(DBED) · O_2 · H_2O	$C_{16}H_{20}N_2O_5Co$	50.58/50.66	5.22/5.31	7.25/7.38	209.5–211
Ni(DBED) · $2H_2O$	$C_{16}H_{22}N_2O_4Ni$	52.25/52.35	6.52/6.58	7.59/7.63	222
H_2DBOPD	$C_{20}H_{20}N_2O_2$	74.82/74.98	6.18/6.29	8.64/8.74	110.5
Cu(DBOPD) · H_2O	$C_{20}H_{20}N_2O_3Cu$	60.02/60.07	5.01/5.04	6.95/7.00	192
Ni(DBOPD) · $2H_2O$	$C_{20}H_{22}N_2O_4Ni$	58.08/58.15	5.25/5.37	6.73/6.78	227

sodium borohydride (5.7 g, 0.15 mol) was added to it with intensive stirring in small portions. Once all the $NaBH_4$ had been added, stirring was continued for another 0.5 h, and then the colorless solution was diluted with a threefold amount of water and acidified with a 10% HCl solution to pH ~ 7.5. The precipitated compound was separated on a porous filter, washed with water, dried, recrystallized from a mixture of ethanol–water (1 : 1), and dried in vacuum.

Cu(DBED)(CH_3COO) · H_2O. *N*,*N*'-di(2-hydroxybenzyl)ethylenediamine (H_2DBED) (0.272 g, 1 mmol) was dissolved in 20 ml of methanol, and a solution of $Cu(CH_3COO)_2 \cdot H_2O$ (0.199 g, 1mmol) in 5 ml of water was added to it. After several hours, a finely crystalline precipitate of green color formed which was separated, washed with water on a filter, and dried in vacuum.

Cu(DBED) · H_2O. H_2DBED (0.544 g, 2 mmol) dissolved in 30 ml of ethanol was mixed with $Cu(OH)_2$ prepared by precipitation of aqueous $CuSO_4 \cdot 5H_2O$ (0.62 g, 2.5 mmol) with 5% NaOH. Some time later, dark green fine crystals precipitated which were separated and dried.

Co(DBED) · O_2 · H_2O was obtained similarly to the copper complex from $Co(CH_3COO)_2 \cdot 4H_2O$ and H_2DBED. A finely crystalline precipitate was formed from the obtained solution after it was allowed to stand in air, and the precipitate was separated and dried in vacuum.

Ni(DBED) · $2H_2O$ was obtained from $Ni(CH_3COO)_2 \cdot 2H_2O$ and H_2DBED by the procedure described above for the Cu(II) and Co(II) complexes. The obtained green solution was heated to boiling and allowed to stand and crystallize. In 15–20 min, fine lustrous crystals of blue color precipitated which were separated, washed with ethanol, and dried in vacuum.

Oxidative dehydrogenation of the Co(II) complex with *N*,*N*'-di(2-hydroxybenzyl)ethylenediamine. H_2DBED (0.272 g, 1 mmol) and $Co(CH_3COO)_2 \cdot 4H_2O$ (0.25 g, 1 mmol) were placed in a constant-temperature reactor (60°C) connected to a gas buret and filled with oxygen. Then, 40 ml of a dioxane–pyridine mixture (7 : 1) was supplied through a separating funnel, after which a homogeneous light violet solution of the cobalt complex was formed. When the reactor was filled with oxygen, the solution color changed to brown and oxygen absorption was observed to be almost complete in 15–20 min. Heating with simultaneous stirring was continued for another 1 h. The total absorption of O_2 was ~23 ml. The reaction mixture was evaporated to 1/3 of its volume, and the obtained brown precipitate was separated and dried in vacuum at 100°C. The IR spectrum of the compound formed is fully identical to that of Co(II) ethylene-*N*,*N*'-disalicylaldiminate (Fig. 1).

H_2DBOPD. *O*-phenylene-*N*,*N*'-di(salicylaldimine) (6.32 g, 2×10^{-2} mol) (mp = 147°C) was mixed with 50 ml of isopropyl alcohol, and sodium borohydride

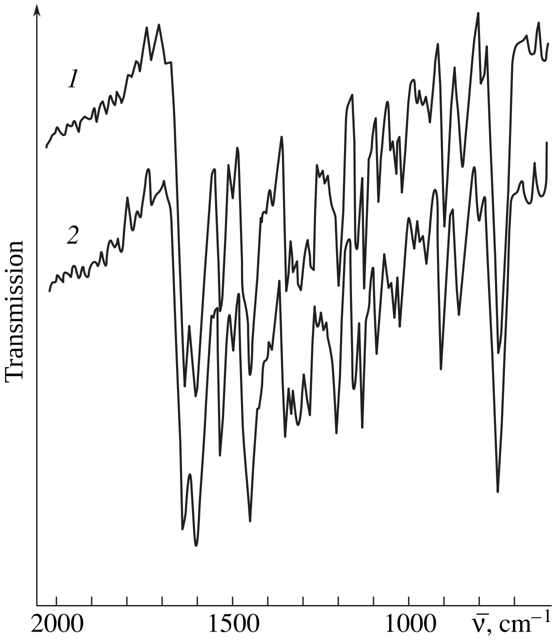


Fig. 1. IR spectra of the product of oxidative dehydrogenation of (*1*) Co(DBED) complex and (*2*) Co(II) ethylene-*N*,*N*'-di(salicylaldiminate).

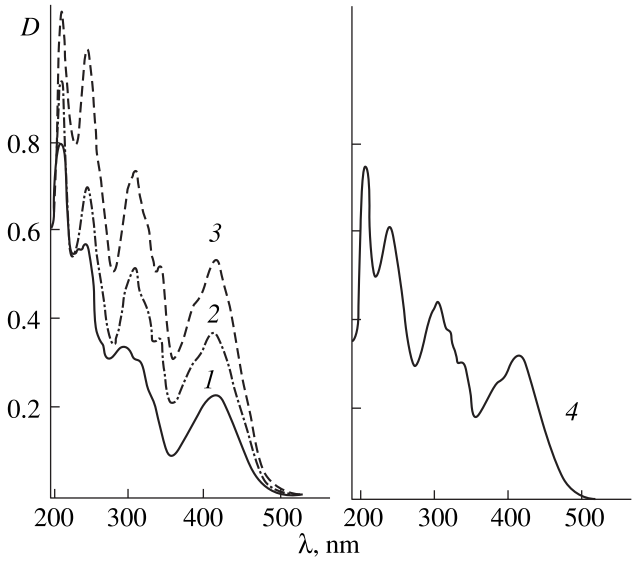


Fig. 2. Electronic absorption spectra of the methanol solutions of the products of Cu(DBOPD) · H_2O conversion in (*1*) dioxane, (*2*) tetrahydrofuran, (*3*) chloroform, and of (*4*) copper(II) *o*-phenylene-*N*,*N*'-di(salicylaldiminate) ($c = 10^{-3}$ mol/l).

was added to the mixture in small portions until the solution became colorless. The amount of $NaBH_4$ spent was 0.8–1.2 g. The reduced solution was diluted with a fivefold quantity of water and acidified to pH ~ 8.0. The obtained product (a nearly white precipitate) was separated, washed with water, dried, and recrystallized from benzene. Then, these white crystals were washed on a porous filter with ether and were dried.

Cu(DBOPD) · H_2O. H_2DBOPD (0.32 g, 1 mmol) was dissolved in 40 ml of methanol, and $Cu(CH_3COO)_2 \cdot H_2O$ in 5 ml of water was added to it. A finely crystalline green precipitate immediately formed which was then washed with water and dried in vacuum at 100°C. The yield was almost quantitative.

Ni(DBOPD) · $2H_2O$ was obtained by a procedure similar to that used to synthesize Cu(DBOPD) · H_2O. H_2DBOPD (0.32 g, 1 mmol) was dissolved in 30 ml of methanol and mixed with $Ni(CH_3COO)_2 \cdot 2H_2O$ (0.249 g, 1 mmol). The solution turned green. The compound precipitated as fine blue crystals.

Self-oxidative transformation of the Cu(DBOPD) · H_2O and Ni(DBOPD) · $2H_2O$ complexes. Cu(DBOPD) · H_2O (0.4 g) in 50 ml of tetrahydrofuran (THF) was boiled with a reflux condenser for 3 h. A dark brown solution was filtered off and allowed to stand for a long time, after which dark lustrous crystals precipitated from it. The yield was 0.30 g. mp > 250°C.

Similar results were obtained in the oxidation of a dioxane solution of Cu(DBOPD) · H_2O with pure oxygen in a temperature-constant reactor at 45°C.

The IR and electronic absorption spectra of the product of oxidative transformation of Cu(DBOPD) · H_2O fully coincide with the spectrum of copper(II) *o*-phenylene-*N*,*N*'-di(salicylaldiminate) (Fig. 2).

A similar procedure was used to carry out the oxidation of Ni(DBOPD) · $2H_2O$ in a temperature-controlled reactor. The obtained crystal precipitate had mp > 250°C, and its spectrum completely coincided with the spectrum of nickel (II) *o*-phenylene-*N*,*N*'-di(salicylaldiminate).

Oxidative dehydrogenation of the Co(II) complex with H_2DBOPD was run similar to that of the complex with H_2DBED. The dark brown compound obtained from the reaction mixture had mp > 250°C.

The IR spectrum of the product was identical with that of Co(II) *o*-phenylene-*N*,*N*'-di(salicylaldiminate).

RESULTS AND DISCUSSION

Complexes with H_2DBED

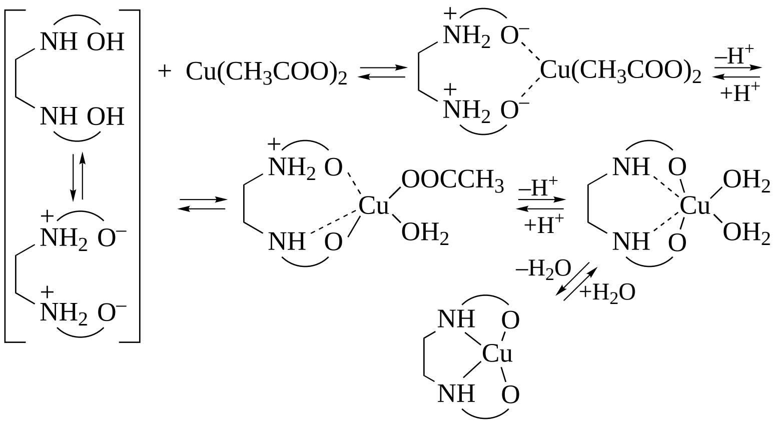
The IR spectra of H_2DBED contain a narrow intense band ν(NH) at 3285 cm^{-1} and a broad intense band in the range of 2500–2600 cm^{-1}, which should be assigned to the stretching vibrations of the amino group participating in the formation of a hydrogen bond with a phenol group. This is confirmed by the fact that the PMR spectrum lacks the H_2DBED signal at 5–6.5 ppm, which is typical of a secondary amine proton.

The electronic absorption spectrum of the H_2DBED methanol solution exhibits two bands due to absorption of a phenol group at 210 and 274 nm.

The H_2BDED behavior in complexation with the Cu(II) ion in water-methanol solutions is very much similar to that of tripod ligands [35].

In complexation of H_2DBED with copper(II) acetate, two characteristic bands appear in the electronic absorption spectrum: a band at 390 nm due to the ligand–metal charge transfer and a *d*–*d* band at ~600 nm. These bands allow one to establish the composition of the complex in solution depending on the pH of the medium. When the pH of the medium changes from 4.1 to 8.0, the band of the *d*–*d* transition shifts from 620 to 590 nm (Fig. 3). One can also observe a somewhat smaller (as compared with the *d*–*d* band) shift of the band due to the charge transfer from 388 to 379 nm. These shifts can be explained by a change in the coordination surrounding of the metal ion.

Complexation of Cu(II) with H_2DBED starts at pH > 4.5. At these values of pH, a band appears due to a charge transfer from the phenol oxygen to the metal ion. An increase in pH is accompanied by an increase in the intensity of this band, which attains its maximum at pH ~ 8. The processes occurring in the reaction mixture with increasing pH can be described by the following scheme:



Like in the case with tripod complexes [35], the shift of the *d–d* band upon a change in pH is evidently associated with the involvement of the nitrogen atoms into coordination. One can suggest that pH growth is accompanied by a gradual replacement of the oxygen atoms in the environment of the metal ion by the nitrogen atoms, thus increasing the strength of the ligand field and shifting the bands to the region of higher energies, which is experimentally observed.

The copper complex with H_2DBED separated in the crystalline state at pH ~ 7.5 has the composition Cu(DBED) · H_2O, and its IR and electronic spectra fully coincide with the spectra of the complex obtained from $Cu(OH)_2$. At the same time, the complex produced upon reacting copper acetate with H_2DBED in a water–methanol mixture (1 : 5) has a mixed-ligand composition $Cu(CH_3COO)$ · HDBED · H_2O.

The EPR spectrum of a polycrystalline sample of the Cu(DBED) · H_2O complex obtained at room temperature exhibits an intense, nearly symmetric, single band with ΔH = 150 Gs and g 2.107. This fact can suggest an octahedral environment of the Cu^{2+} ion in the solid state due to additional coordination by a central ion of two oxygen atoms (or nitrogen) from neighboring molecules.

The EPR spectrum of the Cu(DBED) solution in THF frozen at 84 K is typical of the distorted octahedral Cu(II) complexes and has the following parameters: $g_{\parallel} = 2.21$, $g_{\perp} = 2.04$, and $A_{\parallel} = 195$ Gs.

Cu(DBED) · H_2O poorly dissolved in common solvents, however, when a small amount of acetic acid was added, it dissolved in $CHCl_3$ ($CHCl_3 : CH_3COOH \approx 15 : 1$). The EPR isotropic signal of the complex solution in this mixture of solvents (A_{iso} = 75 Gs, g_{iso} = 2.113) fully corresponds to the isotropic EPR spectrum of the Cu(II) complex obtained from copper acetate and H_2DBED in methanol.

The nickel ions and H_2DBED form the Ni(DBED) · $2H_2O$ complex. The electronic absorption spectrum of this complex in water–alcohol (1 : 1) mixture (the bands at 340, 420 and 610 nm) and the value of the magnetic moment of this complex ($\mu_{eff} = 2.86\ \mu_B$ at 20°C) point to a distorted octahedral coordination of the central ion (evidently, an elongated octahedron). Freshly prepared lustrous blue crystals of the nickel complex become colorless with time, which is due to the loss of water molecules. According to data of thermogravimetric analysis, the first water molecule in the Ni(BDED)· $2H_2O$ complex is relatively weakly bound and removed at 120°C, while the second molecule is more strongly bound and is removed at 170°C.

Interaction of the Co(II) Complex with H_2DBED with Oxygen

Solutions of the Co(II) complex, which are prepared from the metal salt and H_2DBED in dioxane or in a mixture with pyridine, can absorb oxygen; this capability depends on the sequence of introduction of oxygen

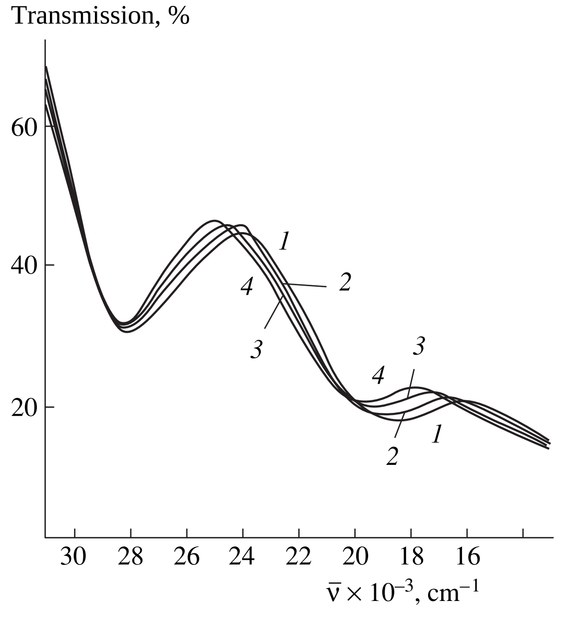
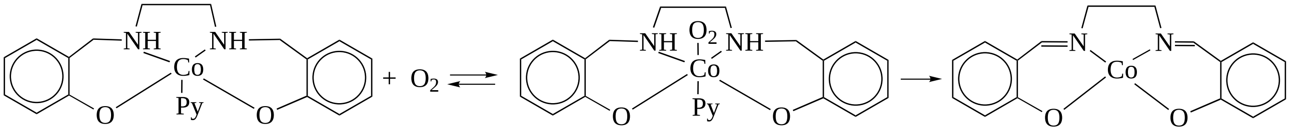


Fig. 3. Electronic absorption spectra of an aqueous solution of a mixture of $Cu(CH_3COO)_2 \cdot H_2O$ (10^{-3} mol/l) and H_2DBED (10^{-3} mol/l) at pH = (*1*) 4.8, (*2*) 5.8, (*3*) 6.7, and (*4*) 7.5.

and solvent into the reactor. When the system is first filled with oxygen and then the solvent is introduced after absorption of a small volume of oxygen, no further absorption is observed. The most probable explanation of this phenomenon is the formation of nonactive Co(III) complexes or stable binuclear peroxide complexes.

In typical experiments, 5×10^{-4} mol of metal acetate and of the ligand were placed in a reactor connected to a gas buret, the system was evacuated, and a dioxane–Py mixture (10 : 1) was introduced with stirring. Complexation was complete within several minutes. When oxygen was supplied, the solution color sharply changed from light violet-pink to dark brown and oxygen absorption occurred in time. The total amount of absorbed oxygen corresponded to a molar ratio of Co(DBED) : O_2 = 1 : 1. Subsequent heating of the solution at 60°C resulted in a complete conversion of the initial Co(DBED) to the respective Co(II) ethylene-*N*,*N*'-di(salicylaldiminate).

The reaction of H_2DBED and of cobalt acetate with oxygen in the methanol–water mixture at room temperature yields the oxygen adduct CoDBED · O_2 · H_2O. Taking this fact into account, one can suggest that the oxidative dehydrogenation in the presence of pyridine follows the scheme given below:



The Cu(II) and Ni(II) Complexes with H_2DBOPD

The electronic absorption spectrum of H_2DBOPD contains absorption bands (λ, nm (ε, mol^{-1} l cm^{-1})) due to the substituted phenol at 225 (1.8×10^4), 273 (9×10^2) and bands from the *o*-phenylenediamine fragment at 252 (1.2×10^3), 295 (4.7×10^2). In complexation with Cu(II) ion, all bands of the ligand undergo a slight bathochromic shift. In addition, bands at 418 nm (due to the charge transfer) and at 600 nm (due to the *d–d* transition) appear.

In the IR spectrum of Cu(DBOPD) · H_2O, the band due to the stretching vibration of the NH group is very broad and appears as a small peak in the region of 3208 cm^{-1}, whereas in the H_2DBOPD spectrum, it is very intense and is located in the region of 3220 cm^{-1}.

The Cu(DBOPD) · H_2O composition is confirmed by the data obtained from a thermogravimetric analysis. A 4.5% mass loss is observed in the temperature interval of 80–110°C and corresponds to the detachment of one water molecule (theoretical 4.5%).

The *d–d* band at 600 nm can point to a distorted octahedral structure of this complex. The pattern of the EPR spectrum of a polycrystalline Cu(DBOPD) · H_2O sample is typical of Cu(II) complexes with tetragonal symmetry.

The anisotropic constants $g_{\|} = 2.2095$, $g_{\perp} = 2.053$, and $A_{\|} = 191.7$ Gs were found from the EPR spectra of the frozen complex solution in THF.

When the Cu(DBOPD) · H_2O solution in $CHCl_3$ is heated in air, it gradually changes its color to dark brown and, then, dark lustrous needle crystals of a new complex precipitate. Analogous transformations of this complex were observed when it was dissolved in THF or dioxane and the obtained solutions were allowed to stand for several days. However, this transformation does not take place in the absence of oxygen.

One can suggest that the oxidative transformation occurs via the *o*-phenylenediamine fragment to yield a Cu(II) complex containing a quinone diimine fragment. Nevertheless, the analysis data and comparison of the electronic and IR spectra showed that in this case, a known complex, i.e., Cu(II) *o*-phenylele-*N*,*N*'-di(salicylaldiminate), is formed, the reaction being selective and having a yield close to the quantitative yield. It is noteworthy that the oxidation of the ligand itself under the same conditions gives a range of products and is likely to occur through the *o*-phenylenediamine fragment.

The selectivity discovered in the formation of Cu(II) *o*-phenylene-*N*,*N*'-di(salicylaldiminate) indicates that the oxidation reaction occurs in the coordinated ligands. It is safe to say that the initial stage of the oxidation reaction is the formation of an intermediate oxygen adduct M(DBOPD) · O_2. This can be indirectly confirmed by the fact that in the case of Co(II), which forms more stable oxygen adduct Co(DBED) · O_2, no Co(DBOPD) complex forms in the presence of oxygen.

In the IR spectrum of Ni(DBOPD) · $2H_2O$, the doublet band due to the vibration ν(NH) of a secondary amino group is at 3208 cm^{-1}. The UV region of the electronic absorption spectrum contains bands at 203, 240, and 280 nm and a low-intensity band at 450 nm; $\mu_{eff} = 2.92\ \mu_B$. These data point to a distorted octachedral structure of Ni(DBOPD) · $2H_2O$.

When this complex is heated in methanol in air, it gradually converts into Ni(II) *o*-phenylene-*N*,*N*'-di(salicylaldiminate), but at a considerably lower rate than in the case with the Cu(II) compound. The poor solubility of the nickel complex does not make it possible to quantitatively compare the rates of these processes for Cu(II) and Ni(II).

An attempt to obtain the cobalt complex with H_2DBOPD in air was a failure, and Co(II) *o*-phenylenedi(salicylaldiminate) always precipitated from the reaction mixture. In an inert atmosphere, Co(DBOPD) precipitated; however, it is vey difficult to obtain analytically pure samples because of its sensitivity to oxygen.

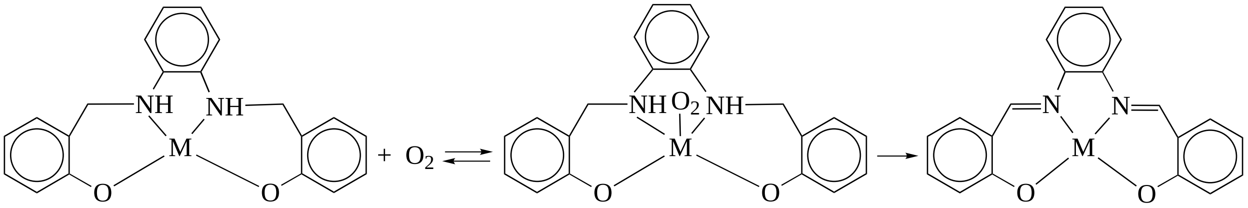
Interaction of the M(DBOPD) · 2H_2O Complexes with Oxygen

The Cu(DBOPD) · H_2O and Ni(DBOPD) · 2H_2O complexes in the solid state are stable in air; however, in solutions (methanol, dioxane, tetrahydrofuran), they undergo oxidative dehydrogenation. For Cu(DBOPD) · H_2O, the dehydrogenation with a selective formation of the corresponding Cu(II) *o*-phenylene-*N*,*N*'-di(salicylaldiminate) in an atmosphere of pure oxygen occurs at a noticeable rate already at 30°C. The oxidation of the nickel complex occurs at the same rate at a higher temperature (40–45°C).

Unlike analogous complexes with H_2DBED, the Cu(II) and Ni(II) complexes with H_2DBOPD more readily undergo oxidative dehydrogenation when exposed to oxygen. Thus, the oxidative dehydrogenation in the case of H_2DBED is only observed for the Co(II) complexes and under very rigid conditions (60°C, pyridine), whereas for the Cu(II) and Ni(II) complexes, dehydrogenation occurs under relatively mild conditions (30°C in a polar or weakly polar solvent, such as CH_3OH, $CHCl_3$, and others).

The main reason underlying this phenomenon is that the metal compounds with these ligands have different capabilities of forming active forms of complexes with oxygen. The presence of a redox-active *o*-phenylenediamine fragment, which is known to easily donate electrons, can favor the formation of active superoxide and peroxide complexes even in the case of metals that cannot give such forms with other ligands.

The mechanism of oxidative dehydrogenation in this case is evidently similar to that given above for the Co(DBED) complex and should include the formation of dioxygen adduct:



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