

EPR study of mono-*o*-iminobenzosemiquinonato nickel(II) complexes with Ni–C σ -bond

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Abstract

New square-planar $(\text{Ph}_3\text{P})\text{Ni}^{\text{II}}(\textit{o}\text{-Tol})(\text{ISQ-Pr}^i)$ (**1**), $(\text{Ph}_3\text{P})\text{Ni}^{\text{II}}(\textit{o}\text{-Tol})(\text{ISQ-Me})$ (**2**), $(\text{Ph}_3\text{P})\text{Ni}^{\text{II}}(\textit{o}\text{-Tol})(\text{ISQ-Bu}^t)$ (**3**) nickel complexes (where $\text{ISQ-Pr}^i = 4,6\text{-di-}i\text{-tert-butyl-}N\text{-}(2,6\text{-di-}i\text{-propylphenyl})\text{-}o\text{-iminobenzosemiquinonate}$, $\text{ISQ-Me} = 4,6\text{-di-}i\text{-tert-butyl-}N\text{-}(2,6\text{-di-methylphenyl})\text{-}o\text{-iminobenzosemiquinonate}$, $\text{ISQ-Bu}^t = 4,6\text{-di-}i\text{-tert-butyl-}N\text{-}(2,5\text{-di-}i\text{-tert-butylphenyl})\text{-}o\text{-iminobenzosemiquinonate}$, $\textit{o}\text{-Tol} = \textit{o}$ -tolyl ligand) have been synthesized. Complexes contain σ -bound \textit{o} -tolyl and neutral donor ligand Ph_3P . The sterical hindrances of N -aryl in \textit{o} -iminobenzosemiquinonate ligands lead to the tetrahedral distortion of square-planar configurations of complexes as it was established using EPR spectroscopy.

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1. Introduction

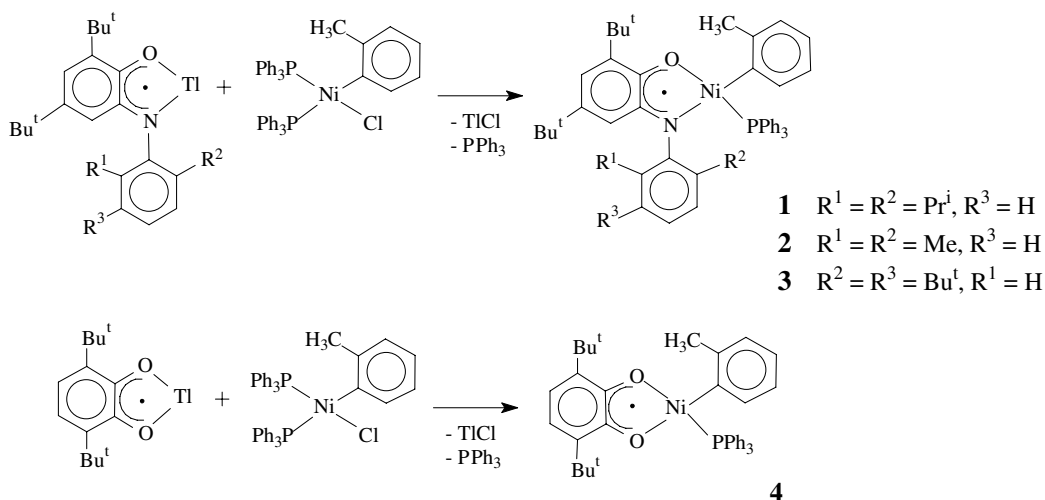
Organometallic compounds of nickel attracted a lot of attention because of their wide applications in organic synthesis and homogeneous catalysis. Introduction of \textit{o} -semiquinone (SQ) as a spin label in inner coordination sphere of organometallic compound in some cases allows to monitor complex composition, structure and dynamic processes occurring there by simple means of EPR spectroscopy [1–5]. \textit{o} -Semiquinone is an anion-radical of \textit{o} -quinone which is coordinated to the metal through two oxygen atoms. The most of spin density is localized in its π -system [6]. Parameters of EPR spectra of SQ-complexes (g -factor, number and intensity of components of hyperfine structure (HFS), hyperfine coupling constants) are very sensitive to the positions of magnetic nuclei relatively semiquinonic plane

[1,7,8]. A little number of papers mentions nickel complexes containing organic fragment and \textit{o} -semiquinone bonded to the metal [3c,9,10]. \textit{o} -Iminosemiquinones are analogues of \textit{o} -semiquinones which allow to introduce asymmetry in spin density distribution and evaluate influence of sterical hindrances of substituents at nitrogen atom bonded to the metal. There are no papers concerning organometallic complexes of nickel with \textit{o} -iminobenzosemiquinonato ligand and Ni–C bond in the scientific literature. In the present work we report the synthesis and the EPR study of series of \textit{o} -iminobenzosemiquinonato \textit{o} -tolyl nickel(II) complexes compared with their \textit{o} -semiquinonato analog.

2. Results and discussion

Complexes of nickel **1–4** were obtained through the exchange reaction of $(\text{PPh}_3)_2\text{Ni}(\textit{o}\text{-Tol})\text{Cl}$ with [4,6-di- \textit{tert} -butyl- N -aryl- \textit{o} -iminobenzosemiquinonato]thallium(I),

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Scheme 1.

where aryl = 2,6-di-*iso*-propylphenyl (**1**), 2,6-dimethylphenyl (**2**) or 2,5-di-*tert*-butylphenyl (**3**), or with [3,6-di-*tert*-butyl-*o*-benzosemiquinonato]thallium (Scheme 1).

All *o*-iminobenzosemiquinone complexes (**1–3**) were isolated and characterized by IR, EPR spectroscopy. Complexes **1–3** are the air stable microcrystalline solids colored from dark green to dark green-blue. Compounds are well soluble in different organic solvents (THF, toluene, benzene, diethyl ether, dichloromethane, methanol, acetone, and petroleum-ether). Unfortunately, we did not find the conditions to form X-ray suitable monocrystal materials. Complex **4** was generated and investigated in solution.

IR spectra of **1–3** contains bands related to all ligands of complexes. Triphenylphosphine is determined in complexes on the deformation vibrations $\delta(\text{C–P–C})$ at 525–530, 510 and 495 cm^{-1} , $\delta_{\perp}(\text{C–H, aromatic})$ at 735–695 cm^{-1} , stretching vibrations $\nu(\text{P–C})$ at 1100 cm^{-1} . Deformation symmetrical and asymmetrical vibrations $\delta_{\text{sim}}(\text{C–H})$ and $\delta_{\text{as}}(\text{C–H})$ of methyl groups in Me, Pr^i , Bu^t – 1365 and 1400 cm^{-1} . *o*-Iminobenzosemiquinone ligand in complexes exhibits absorption bands $\nu(\text{C–O})$ and $\nu(\text{C–N})$ between 1400 and 1500 cm^{-1} . Stretching vibrations $\nu(\text{C}_{\text{aryl}}\text{–N})$ are in range 1255–1260 cm^{-1} .

EPR spectrum of complex **4** in solution (Fig. 1) is doublet of triplets (1:2:1) with $g_i = 2.0100$ indicating the localization of odd electron mainly on organic ligand. The hyperfine coupling (HFC) constant $a(^1\text{H}) = 3.3$ G in triplet. This value is typical for SQ-ligands [11–14]. Doublet exhibits the hyperfine splitting due to ^{31}P nucleus ($I = 1/2, 100\%$) of the phosphine ligand in complex (Table 1).

As shown earlier, the HFC constant with phosphorus nucleus $a(^{31}\text{P})$ in *o*-semiquinone complexes is sensitive to the coordination geometry [1,3,7,8,13,14]. The HFC constant $a(^{31}\text{P})$ has value of 3–4 G for SQ-complexes with phosphines in a plane of *o*-semiquinone ligand,

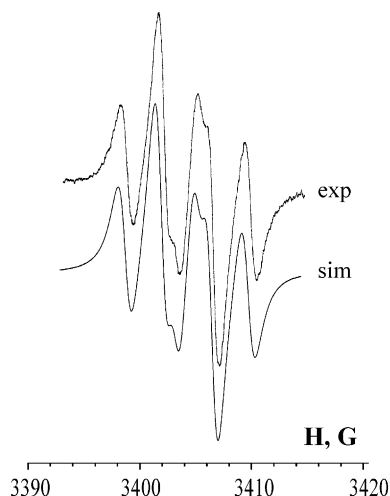


Fig. 1. Experimental X-band EPR spectrum of **4** in toluene at 290 K (top) and its simulation (bottom).

12–17 G for complexes with phosphine outside of the SQ-plane, and reaches values of 26–28 G for complexes with phosphorus in an apical position. For example, $a(^{31}\text{P}) = 3.2$ G for complex $(\text{Ph}_3\text{P})(\text{CO})\text{Rh}(3,6\text{-DBSQ})$ ¹ [3a] and 3.0 G for $(\text{Ph}_3\text{P})\text{PdCl}(3,5\text{-DBSQ})$ of the square-planar geometry [13], 3.2 G for $(\text{Ph}_3\text{P})_2\text{PtX}(3,6\text{-DBSQ})$ and 2.6 G for $(\text{Ph}_3\text{P})_2\text{PtX}(\text{O}^{\text{X}}\text{SQ}_{\text{Cl}})$ ² [14] of the tetrahedral pyramidal geometry with the ligand X being at the apex. In the other hand, for trigonal bipyramidal complex $(3,6\text{-DBSQ})\text{Rh}(\text{Ph}_3\text{P})_2(\text{CO})$ with phosphines in sites out of SQ-plane, HFC constants with two phosphorus nuclei $a(^{31}\text{P}) = 17.0$ G [3a]. For tetrahedral

¹ 3,6-DBSQ is 3,6-di-*tert*-butyl-*o*-benzosemiquinonate and 3,5-DBSQ is 3,5-di-*tert*-butyl-*o*-benzosemiquinonate.

² $\text{O}^{\text{X}}\text{SQ}_{\text{Cl}}$ is perchloroxantrenesemiquinonate.

Table 1
EPR spectral parameters of **1–4** in toluene at 293 K and in the solid state

Complex	g_{iso}	$a(^{31}\text{P})$ (G)	$a(\text{N})$ (G)	$a(\text{H5})$ (G)	$a(\text{H3})^{\text{a}}$ (G)	Anisotropic g -parameters	
1	2.0120	8.10	7.20	4.0	1.30	$g_{\parallel} = 2.0303$	$g_{\perp} = 2.0016$
2	2.0109	7.25	7.10	4.2	1.35	$g_{\parallel} = 2.0260$	$g_{\perp} = 2.0030$
3	2.0101	6.85	7.15	4.5	1.43	$g_{\parallel} = 2.0220$	$g_{\perp} = 2.0035$
4	2.0100	4.40	–	$a(\text{H4}) = a(\text{H5}) = 3.3$ G		$g_1 = 2.0255$	$g_2 = 2.0090$ $g_3 = 1.9956$

^a Although values $a(\text{H3})$ are less than the line widths on spectra these HFC constants are required for the satisfactory results of simulations.

$(\text{Ph}_3\text{P})_2\text{Cu}(3,6\text{-DBSQ})$ $a(^{31}\text{P}) = 17.6$ G, while there is the hyperfine splitting of 2.6 G on phosphorus nucleus for trigonal $(\text{Ph}_3\text{P})\text{Cu}(3,6\text{-DBSQ})$ [3b]. $a(^{31}\text{P}) = 26$ G for octahedral $(\text{Ph}_3\text{P})\text{Re}(\text{CO})_3(3,5\text{-DBSQ})$ where Ph_3P is in the apical site [15]. So, HFC constant $a(^{31}\text{P}) = 4.4$ G evidences the position of Ph_3P close to the plane of *o*-semiquinone ligand in **4**.

EPR spectra of **1–3** in solution are doublets of triplets (1:1:1) of doublets and reflect the HFC of unpaired electron with the ^{31}P , ^{14}N and proton nuclei. It was difficult to receive the values of HFC constants with nucleus of phosphorus, nitrogen and a proton from a spectra, because $a(^{31}\text{P}) \approx 2a(^1\text{H})$ and the imposing of components of a spectrum is observed. Therefore these spectra have been simulated. Fig. 2 displays an experimental (exp)

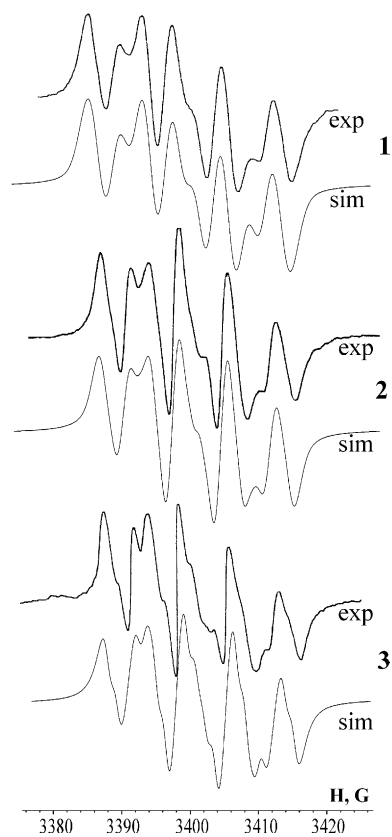


Fig. 2. Experimental X-band EPR spectra of **1–3** (toluene, 290 K) (exp) and their simulations (sim).

X-band EPR spectra of **1**, **2** and **3** and their simulations (sim).

Parameters of isotropic EPR spectra for complexes are summarized in Table 1. In solid state, complexes **1–3** exhibit anisotropic EPR spectra with axial g -tensor. EPR spectrum of **4** in the frozen toluene at 150 K has parameters of g -tensor: $g_1 = 2.0255$; $g_2 = 2.0090$; $g_3 = 1.9956$.

Value of HFC constant on phosphorus nucleus $a(^{31}\text{P}) = 8.1$ G for **1** allows to suggest the tetrahedral distorted square-planar structure for **1**. At transition from **1** to **2** and **3**, the volume of the substitutes in positions 2 and 6 of aryl fragment at nitrogen atom decreases. Reduction of the sterical repulsion of ligands in complex should result to that the configuration of complex will come nearer to clear square-planar owing to what HFC constant with a nucleus of phosphorus will go down. So, the reduction of HFC constant $a(^{31}\text{P})$ at transition from complex **1** through **2** to **3** confirms the assumed decreasing of the tetrahedral distortion of square-planar configurations of complexes.

The possibilities of spin-label *o*-semiquinonato ligands have been demonstrated for example on the exchange reactions of n - and π -donor ligands in *o*-semiquinonato copper(I) and rhodium(I) complexes [3]. By the example of **1**, we have investigated the behaviour of complexes in relation to various ligands using EPR spectroscopy. The following ligands were used: pyridine (Py), pyrazine (Pyz), 2,2'-dipyridyl (2,2'-dipy), NEt_3 , PMe_3 , PBu_3 , AsEt_3 , AsPh_3 , 2,6-dimethylphenylisocyanide (ArNC), NO. Exchange reactions are shown on Scheme 2. Under addition of Py, Pyz, NEt_3 , PMe_3 , AsEt_3 , ArNC, triphenylphosphine ligand is displaced in the nickel coordination sphere forming new four-coordinate adducts **1a–1f**. To receive values of HFC constants, EPR spectra have also been simulated. EPR spectral parameters of adducts are given in Table 2. EPR spectrum of PMe_3 adduct **1d** resembles spectrum of **1** with the only minor differences in HFC constants on magnetic nuclei of *o*-iminosemiquinone ligand. The HFC constant $a(^{31}\text{P})$ of this adduct is equal to that of **1**. It is explained by the larger basicity of PMe_3 relative to PPh_3 . EPR spectra in the cases of N-containing ligands (Fig. 3 for Py) and ArNC (Fig. 4) reflect only HFS on magnetic nuclei of *o*-iminosemiquinone. EPR spectrum (Fig. 5) of arsine-adduct **1f** contains in

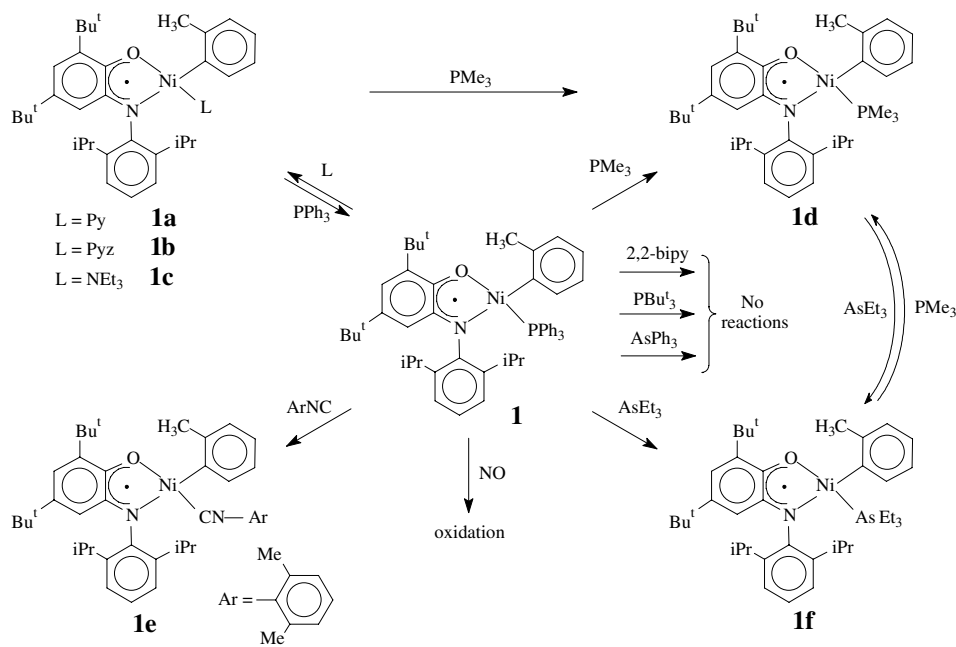


Table 2

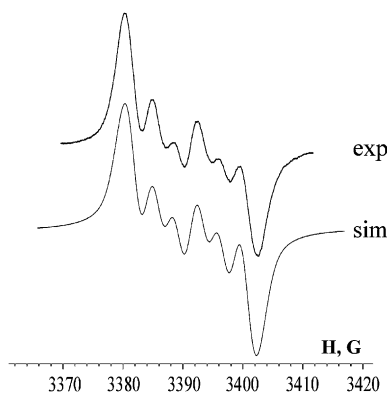
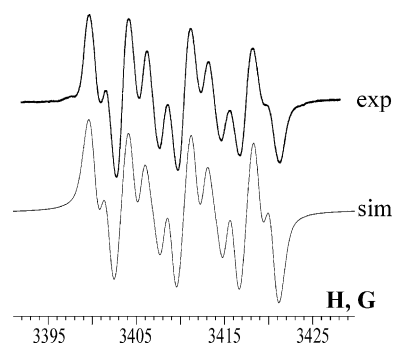
EPR spectral parameters of adducts of ligand exchange reactions of **1** (toluene, 293 K)

Adduct	Ligand	g_{iso}	$a(^{31}\text{P})$ (G)	$a(\text{N})$ (G)	$a(\text{H5})$ (G)	$a(\text{H3})$ (G)	$a(^{75}\text{As})$ (G)
1a	Py	2.0168	–	7.4	3.9	1.4	–
1b	Pyz	2.0135	–	7.3	4.1	1.4	–
1c	NEt ₃	2.0190	–	7.7	4.0	1.4	–
1d	PMe ₃	2.0117	8.1	7.3	4.4	1.5	–
1e	RNC	2.0058	–	7.1	4.4	1.6	–
1f	AsEt ₃	2.0152	–	7.4	4.1	1.5	9.3

addition the hyperfine splitting of 9.3 G on arsenic nucleus ^{75}As ($I = 3/2$, 100%). Noteworthy, complex **1** does not react with bulky ligands (PBU₃, AsPh₃, bidentate ligand 2,2'-dipy). This fact corroborates the claimed sterical hindered structure of complex. Reactions with nitrogen-containing substituting ligands are reversible

and require appreciable excess of these reagents to obtain pure spectra of substitution products. Addition of PMe₃ or AsEt₃ to **1a–1c** leads to observation of EPR spectra of **1d** or **1f** correspondingly.

All the adducts **1a–1f** are unstable and decompose in a solution during the short time (from several hours to

Fig. 3. X-band EPR spectrum (top) of pyridine adduct **1a** (toluene, 290 K) and its simulation (bottom).Fig. 4. X-band EPR spectrum (top) of isonitrile adduct **1e** (toluene, 290 K) and its simulation (bottom).

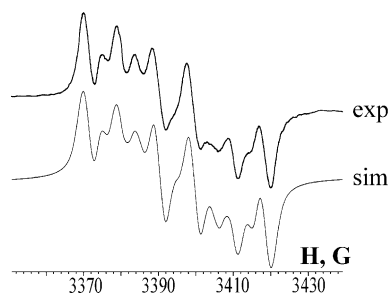


Fig. 5. X-band EPR spectrum (top) of triethylarsine adduct **1f** (toluene, 290 K) and its simulation (bottom).

one day). Complex reacts with nitric oxide with oxidation of *o*-iminobenzosemiquinonato ligand to *o*-iminobenzoquinone and decomposition of complex. During the reaction EPR spectrum of starting complex **1** disappears without appearance of any other EPR signals that indicates the formation of non-paramagnetic products of oxidation decomposition.

It should be noted that nickel complex keeps constant the coordination number in reactions, and in all cases the σ -bound *o*-tolyl ligand is kept. It suggests the sufficient stability of the given group in complex.

So, complexes **1–4** are complexes of nickel(II) containing *o*-iminobenzosemiquinonato or *o*-benzosemiquinonato anion-radical, σ -bound *o*-tolyl and neutral donor ligand PPh_3 . The geometry of complex **4** is close to the square-planar. The sterical hindrances of *N*-aryl in *o*-iminobenzosemiquinonato ligands lead to the tetrahedral distortion of square-planar configurations of complexes **1–3** as shown using EPR. Complexes are able to exchange of PPh_3 on some neutral donor ligands.

3. Experimental

3.1. General considerations

All reagents were grade. Solvents were purified by following standard methods [16]. *o*-Iminobenzoquinones were prepared according literature procedure [17]. The compound $(\text{Ph}_3\text{P})_2\text{Ni}(o\text{-Tol})\text{Cl}$ was prepared according to the known method [18]. Starting compounds [4,6-di-*tert*-butyl-*N*-aryl-*o*-iminobenzosemiquinonato]thallium(I) [19] and [3,6-di-*tert*-butyl-*o*-benzosemiquinonato]thallium(I) [20] were prepared according to the known procedures. All manipulations on complexes were performed in vacuum with exclusion of oxygen and moisture.

The infrared spectra of the complexes in the 4000–400 cm^{-1} range were recorded on a Specord M-80 spectrophotometer in nujol. EPR spectra were recorded on a Bruker ER 200 D-SRC spectrometer with ER041 MR microwave bridge, ER 4105 DR double resonator and

ER 4111 VT variable temperature unite. The spectra were simulated using WinEPR SimFonia v1.25 (Bruker) by iteration of the (an)isotropic *g* values, hyperfine coupling constants, and line widths. Measurements of paramagnetic centres concentrations in crystalline samples were performed using EPR by the comparison of the integral intensities of samples relative to that of standard (3,6-di-*tert*-butyl-*o*-benzosemiquinonato)(bis-(diphenylphosphino)ferrocene)copper(I) (3,6-DBSQ)Cu (dppfc) [21].

3.2. Syntheses

3.2.1. Preparation of (triphenylphosphine)-(*o*-tolyl)-(4,6-di-*tert*-butyl-*N*-(2,6-di-*iso*-propylphenyl)-*o*-iminobenzosemiquinonato)nickel(II) **1**

The solution of 4,6-di-*tert*-butyl-*N*-(2,6-di-*iso*-propylphenyl)-*o*-iminobenzosemiquinonato thallium(I) (prepared from a free *o*-iminobenzoquinone (0.379 g, 1 mmol) and an excess of thallium amalgam in 20 ml of THF [20]) was added to a solution of $(\text{Ph}_3\text{P})_2\text{Ni}(o\text{-Tol})\text{Cl}$ (0.709 g, 1 mmol) in ~50 ml of THF. After the solution was stirred at 20 °C for 1 h, the resulting deep green solution was filtered off to remove a precipitate of TiCl_4 , solvent was changed for toluene and solution was filtered again. The solution was allowed to stay for two days at –15 °C after slow evaporation of ~50% toluene. Resulting dark green microcrystalline material of **1** was collected by filtration, washed with cold toluene and dried in vacuum. In solid **1** is stable on air a long time, in solution – for a few hours. Complex is well-soluble in toluene, diethyl ether, dichloromethane, THF, benzene, and slightly soluble in hexane.

Yield is 0.46 g (58%). Anal. Calc. for $\text{C}_{51}\text{H}_{59}\text{ON-PNi}$ (%): C, 77.37; H, 7.51; P, 3.91; Ni, 7.41. Found: C, 77.05; H, 7.48; P, 3.71; Ni, 7.01. IR (nujol, cm^{-1}): 1575 w, 1535 w, 1440 s, 1400 w, 1365 m, 1330 m, 1260 m, 1175 m, 1100 m, 1030 w, 1000 w, 915 w, 860 w, 800 w, 770 w, 745 m, 735 s, 695 s, 530 s, 510 m, 495 m. Measured concentration of paramagnetic centers is 0.997 mol per mol of solid sample of **1** (99.7% of solid sample of **1**).

3.2.2. Preparation of (triphenylphosphine)-(*o*-tolyl)-(4,6-di-*tert*-butyl-*N*-(2,6-dimethylphenyl)-*o*-iminobenzosemiquinonato)nickel(II) **2**

Complex was prepared by the same way as **1**. Resulting product **2** was isolated as dark green microcrystalline powder. Complex is air stable in solid. Yield is 48%. IR (nujol, cm^{-1}): 1585 w, 1535 w, 1445 s, 1400 w, 1365 m, 1330 m, 1255 m, 1175 m, 1105 m, 1030 w, 1000 w, 915 w, 860 w, 770 w, 745 w, 735 m, 705 m, 695 m, 525 m, 510 m, 495 w. Measured concentration of paramagnetic centers is 0.993 mol per mol of solid sample of **1** (99.3% of solid sample of **2**).

3.2.3. Preparation of (triphenylphosphine)-(*o*-tolyl)-(4,6-di-*tert*-butyl-*N*-(2,5-di-*tert*-butylphenyl)-*o*-imino-benzosemiquinonato)nickel(II) **3**

Complex was synthesized in the same way as **1** but the isolation was performed from pentane. Product formed as dark blue-green microcrystalline material. Complex is air stable in solid. Yield is 42%. IR (nujol, cm^{-1}): 1580 w, 1535 w, 1440 s, 1400 w, 1365 m, 1330 m, 1255 m, 1185 m, 1100 m, 1030 w, 1000 w, 915 w, 860 w, 825 m, 770 w, 745 w, 735 m, 705 m, 695 s, 525 s, 510 m, 495 w. Measured concentration of paramagnetic centers is 0.994 mol per mol of solid sample of **3** (99.4% of solid sample of **3**).

3.2.4. Preparation of (triphenylphosphine)-(*o*-tolyl)-(3,6-di-*tert*-butyl-*o*-benzosemiquinonato)nickel(II) **4**

Complex was synthesized in the same way as **1** but using 3,6-di-*tert*-butyl-*o*-benzoquinone as starting reagent instead of *o*-iminobenzoquinone. During the reaction the green color appears. After the removing of thallium chloride from reaction mixture by filtration, the investigation of complex **4** was performed in solution without the isolation of **4**.

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