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Rhodium(I) dimethyl sulfoxide oxyquinolinato carbonyl complex, [Rh(Oxq)(CO)(DMSO)]. NMR and X-ray structure data



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ABSTRACT

A new rhodium(I) dimethyl sulfoxide (DMSO) 8-oxyquinolinato (Oxq) complex, [Rh(Oxq)(CO)(DMSO)] (I), has been prepared and characterized by IR, ¹H and ¹³C NMR, and X-ray data. DMSO is S-coordinated in the *trans*-N position. The values of v(CO), carbonyl δ^{13} C, as well as δ^{13} C and δ^{1} H of Oxq ligand for three L *trans*-N complexes, [RhOxq(CO)(L)] (L = NH₃, DMSO, and CO), define the intermediate position of DMSO within the ligand L series ranged in their net donating potency. Observed spin–spin couplings of Oxq ¹³C (C2, C3, C4a, C7, C8a) and ¹H (H2) nuclei to ¹⁰³Rh provide one more tool to study long-range interactions of Oxq rings with metallocenter and remote ligand L.

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Introduction

In our previous paper [1], we reported on the synthesis of rhodium(I) 8-oxyguinolinato carbonyl complex containing ammonia ligand, [Rh(Oxq)(CO)(NH₃)], and used spectral parameters of the carbonyl ligand to discuss ligand L effects in the series of [Rh(Oxq)(CO)(L)] complexes. Results presented in Ref. [1] showed that v(CO) stretching frequencies increased in the expected order of L: NH₃, NR₃ < Py, PBu₃ < PPh₃ < MeCN, C₈H₁₄ < P(OPh)₃ < CO, while carbonyl carbon chemical shift δ^{13} C decreased on passing from the left part of the series (ammonia and amines, tertiary phosphines) to its right part (olefin, phosphites, and carbonyl ligands). Following the traditional paradigm, we associated this series with the increase in π -acceptor potency of ligands L, decrease in their σ -donor potency, and hence with the decrease in their so called "net donating ability" [2]. In this work our aims were: (i) to examine the two-step procedure of $[Rh(Oxq)(CO)_2] \rightarrow [Rh(Oxq)(CO)(L)]$ conversion [1] with the example of a very popular ligand L = DMSO (dimethyl sulfoxide, see reviews [3-6], more recent publications, for instance [7] and

references therein); (ii) to determine the coordination mode of DMSO in the square planar rhodium(I) carbonyl complex; (iii) to establish the position of DMSO ligand within the above-cited series; and (iv) to trace the influence of ligand L on the NMR parameters of the oxyquinolinato ligand.

Results and discussion

Preparation of [Rh(Oxq)(CO)(DMSO)] (I)

The solution containing monocarbonyl intermediate (a mixture of [Rh(Oxq)(CO)(MeCN)] and $[Rh(Oxq)(CO)(Me_3N)]$ complexes) has been obtained by selective oxidation of the trans-N carbonyl group in $[Rh(Oxq)(CO)_2]$ with Me₃NO in acetonitrile solution as described in Ref. [1]. Addition of DMSO to that solution resulted directly in gradual precipitation of the desired complex, [Rh(Oxq)(-CO)(DMSO)] (I), in the form of goldish needle-like crystals. The crystals were isolated by filtration (1st fraction), and then, after evaporation of a part of solvent, the additional crystalline product was collected (2nd fraction). The two fractions were combined to give about 70% of total yield. Product I is stable on prolonged storage as solid. It is readily soluble in chloroform, methylene chloride, acetone, and tetrahydrofuran (THF).

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X-ray structure data

Crystal of I suitable for X-ray structure determination was taken directly from 1st fraction obtained as described above. The molecular structure of the complex is shown in Fig. 1. The selected bond lengths and angles are listed in Table 1. The molecule of I occupy a special position on the mirror plane and, consequently, possesses a strictly planar geometry excepting for the two methyl groups.

The major result obtained is the evidence of the expected DMSO coordination in I through sulfur atom (DMSO-S). All values of the bond lengths and angles lie within the intervals typical for squareplanar rhodium(I) complexes, in particular for (DMSO-S) complexes [8–11]. The value of the S–O bond length, 1.474(6) Å, is common with complexes containing (DMSO-S) ligand [6,8–11]. Central atom bonds to the oxyguinolinato donor atoms, N and O, in I differ markedly in their lengths from the corresponding values for ammonia analog (2.024(3) and 2.058(2), respectively [1]). Notice that the length of oxyquinolinato N to Rh bond increases in the order of *trans*-ligands $L = NH_3 < DMSO < P(OR)_3$: 2.024(3) [1]; 2.059(6) (this work); 2.097(2) for R = Ph [12] and 2.091(3) for R = 2,6-dimethyl Ph [13]). As to the C–O bond length in I, its ESD value is rather high which does not allow for including this bond length in the further discussion on the v(CO) - r(CO) interrelation, which we touched briefly in Ref. [1].

IR and NMR parameters of DMSO ligand

A strong, broad absorption band at 1108–1112 cm⁻¹ in the IR spectra of I in CHCl₃, CH₂Cl₂, and THF solutions should be assigned to the S–O stretching vibration. This frequency value is considerably higher than v(SO) of free DMSO (1055 cm⁻¹ [6,14]) and is indicative of the S-coordination of DMSO ligand [6,7,14,15]. In the ¹³C{¹H} spectrum of I (THF solution), the signal from two equivalent methyl carbons appears as a doublet at 46.69 ppm, ²J(CRh) = 2.1 Hz. The position of this signal is typical for DMSO-S in rhodium complexes [16,17]. In the methyl region, ¹H NMR spectrum of I (THF solution) exhibits one doublet at 3.29 ppm with intensity (6H) equal to the total intensity of six oxyquinolinato proton resonances. The position of this signal is typical for DMSO-S [6,16,17]. Its doublet splitting is apparently caused by coupling to ¹⁰³Rh nucleus, ³J(HRh) 0.7 Hz (confirmed by experiments at various operating frequencies).



Fig. 1. The structure of [Rh(Oxq)(CO)(DMSO)] (50% probability ellipsoids).

Table 1

Selected bond lengths (Å) and angles ($^{\circ}$) for the complex [Rh(Oxq)(CO)(DMSO)] (I) with estimated standard deviations in parentheses.

IR and NMR parameters of carbonyl ligand

The IR spectrum of **I** contains an intense carbonyl group stretching band, its position being solvent-dependent: 2000 cm⁻¹ in CHCl₃, 1988–1990 cm⁻¹ in CH₂Cl₂, THF, and acetone. Carbonyl carbon resonance in ¹³C{¹H} spectrum of **I** in THF solution appears as a doublet at 189.38 ppm with ¹J(CRh) 74.6 Hz. The carbonyl group spectral parameters pinpoint the place of S-coordinated DMSO within the series of ligands considered in Ref. [1] and cited above. In line with generally accepted notion articulated as early as in 1960s [18], DMSO-S belongs to the right part of that series, *i.e.* to the category of ligands with pronounced π -acceptor ability. In the series of [Rh(Oxq)(CO)(L)] complexes it is close to P(OPh)₃ (specifically with regard to v(CO) value) and C₈H₁₄ (specifically with regard to δ^{13} C value).

NMR parameters of oxyquinolinato ligand

¹³C{¹H} and ¹H NMR spectra of 8-oxyquinoline and 8oxyquinolinato complexes were studied widely within the last four decades. Our data presented below provide additional information concerning spin-spin coupling of ¹³C and ¹H nuclei of Oxq⁻ ligand to ¹⁰³Rh nucleus, and thus may stimulate further discussion on the bonding situation within the system of three condensed coplanar cycles (chelatoaromatic system [19]): two six-membered aromatic rings of Oxq⁻ ligand and a five-membered metal chelate ring, Rh-OCCN. The data obtained are presented in Tables 2-4 (the numbering of carbon atom positions is shown in Fig. 1; the hydrogen atoms are numbered in the same order). Assignments of the ¹H and ¹³C resonances suggested in Tables 2 and 3 are based on the published data [20-28] and supported by our heteronuclear 2D correlation experiment on complex I. Regarding the assignment of resonances H5 and H7, we presumed that in the spectrum of complex I the H7 resonance is shifted upfield in relation to H5, as this is commonly observed in the spectra of 8-hydroxyquinolines and their complexes [20–28].

Examination of Table 2 data shows that all the ¹³C chemical shifts of Oxq⁻ anion are more or less sensitive to the protonation effect (see the gray line for the molecule HOxq). One of these values changes dramatically (~15 ppm decrease), namely that for C8 carbon which is directly bound to the phenolato oxygen atom. By contrast, coordination to the rhodium metallocenter affects foremost C2, C4, and C7 carbons, chemical shifts of which increase more than by 4 ppm on passing from free Oxq⁻ anion to the dicarbonyl rhodium complex. The difference between protonation and coordination effects demonstrates that electron acceptor potency of H⁺ cation is directed predominantly toward oxygen atom of Oxq⁻ anion, whereas influence of rhodium metallocenter is rather delocalized. An important point is that all three, C2, C4, and C7, chemical shifts show the smallest increase for $L = NH_3$ and the greatest increase for L = CO, while the DMSO complex, I, occupies an intermediate position. Evidently these values of the chemical

Table	2
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Chemical shifts, δ^{13} C, ppm, of oxyquinolinato carbon resonances in the spectra of [Rh(Oxq)(CO)(L)] complexes (THF-d₈ solutions). Values of ¹³C to ¹⁰³Rh spin – spin coupling constants for doublet signals, ⁿJ(CRh), Hz, are given in parentheses. Data for neutral 8-hydroxyquinoline and its sodium salt are included.

	C2	C3	C4	C4a	C5	C6	C7	C8	C8a
NaOxq	145.45	120.39	136.47	131.98	114.85	129.74	107.87	169.58	146.38
HOxq	148.57	122,42	136.67	129.76	118.12	128.36	111.03	154.39	139.66
RhOxq(CO)(NH ₃)	148.12d (3.3)	122.01d (2.4)	136.93	131.64d (1.1)	110.86	130.64	113.93d (1.8)	170.28	145.90
RhOxq(CO)(DMSO)	150.02d (3.1)	122.16d (2.0)	139.43	131.53d (1.0)	112.44	130.82	115.24d (1.9)	168.99	145.02
$Rh(Oxq)(CO)_2$	150.86d (3.0)	122.17d (1.9)	140.95	131.46 (1.0)	112.94	131.32	115.89d (2.2)	169.35	143.99d (0.8)

shift are subjected to the net electron donor potency of ligand L: the strongest donor, NH₃, saturates the acceptor ability of metallocenter to the fullest extent and thus causes the minimum increase in the δ^{13} C value on coordination. Notice that Table 2 contains values of one more spectral parameter, spin—spin coupling constant, ⁿJ(CRh), for the ¹³C signals showing the doublet splitting. Noteworthy is the fact that in every case we observed this splitting, the ⁿJ(CRh) value for I lied between the values for [RhOxq(-CO)(NH₃)] and [Rh(Oxq)(CO)₂]. Apparently, these values also reflect the long-range effect of remote ligand L σ -donor/ π -acceptor properties.

Data presented in Table 3 demonstrate that protonation of $Oxq^$ anion results in the parallel increase of chemical shifts of all six aromatic ¹H nuclei (gray line). A similar effect is caused by $Oxq^$ coordination to rhodium metallocenter. The maximum increase of every δ^1 H value is observed for dicarbonyl complex, the minimum increase for ammonia complex, and DMSO complex, the minimum increase for ammonia complex, and DMSO complex, I, is again intermediate in all six cases. Our data are in reasonable agreement with published data on the thorium, uranyl, and rhenium complexes [21,28]. The δ^1 H values for oxyquinolinato ligand in these complexes are all higher than those for the free Oxq^- anion; especially high they are in case of the uranyl complex, $UO_2(Oxq)_2$ [21], that may reflect the superior electron acceptor potency of the uranyl group. The values for rhenium(V) and thorium(IV) complexes, [ReOCl₂(Oxq)(PPh₃)] and Th(Oxq)₄, are fairly close to the corresponding values for the [RhOxq(CO)(L)] complexes.

Table 4 represents data on mutual spin–spin couplings of oxyquinolinato aromatic protons. It should be noted that the H2 resonances in the spectra of rhodium complexes show an additional splitting because of spin–spin coupling to ¹⁰³Rh (see Table 3). As can be seen, the ³J(HRh) value for I falls once again in-between the values for [RhOxq(CO)(NH₃)] and [Rh(Oxq)(CO)₂].

Concluding remarks

Finally, it should be noted that the spectral parameters of carbonyl ligand, v(CO) and $\delta^{13}C$, for three [RhOxq(CO)(L)] complexes (L = NH₃, DMSO, and CO) define the expected in-between position of DMSO within the L ligand triad (and hence in the general ligand series ranged in their net donating potency). The intermediate position of DMSO in terms of ligand L effect on ¹³C and ¹H chemical shifts of oxyquinolinato ligand is remarkable as well.

Table 3

Chemical shifts, δ^{1} H, ppm, of oxyquinolinato proton resonances in the spectra of [Rh(Oxq)(CO)(L)] complexes (THF-d₈ solutions). Values of H2 proton to ¹⁰³Rh spin – spin coupling constants, ³J(HRh), Hz, are given in parentheses. Data for neutral 8-hydroxyquinoline and its sodium salt are included.

	H2	H3	H4	H5	H6	H7
NaOxq	8.34	7.09	7.92	6.70	7.13	6.59
HOxq	8.77	7.43	8.19	7.30	7.41	7.08
$RhOxq(CO)(NH_3)$	8.47 (2.1)	7.17	8.17	6.81	7.28	6.69
RhOxq(CO)(DMSO)	8.63 (1.8)	7.43	8.35	6.99	7.36	6.79
$Rh(Oxq)(CO)_2$	8.74 (1.5)	7.53	8.47	7.07	7.44	6.94

Observed spin–spin couplings of oxyquinolinato¹³C and ¹H nuclei to ¹⁰³Rh may provide one more tool to scrutinize long-range electronic interactions of oxyquinolinato rings with metallocenter and remote ligand L. Even though the variations in the values of NMR parameters may seem too small, we believe them worthy of consideration in view of their regular changes in the permanent sequence of ligands, NH₃–DMSO–CO. Perhaps the observed trends would motivate further efforts to quantify the ligand effects in the oxyquinolinato complexes with the computational chemistry techniques.

Experimental

All operations were carried out under dry argon using orthodox Schlenk techniques. All solvents were purified according to the standard procedures [29]. Deuterated solvents were stored over Al₂O₃ and distilled prior to use. Me₃NO·2H₂O was used as purchased from "Avocado Research Chemicals Ltd". [Rh(Acac)(CO)₂] was prepared by the published procedure [30]. $[Rh(Oxq)(CO)_2]$ and [Rh(Oxq)(CO)(NH₃)] were prepared as described in Ref. [1]. IR spectra were recorded on a Specord 75 IR instrument (CaF2 windows). NMR studies were performed at the Centre for Magnetic Resonance, St. Petersburg State University. The spectra were recorded on Bruker spectrometers DPX-300, AVANCE III 400, and AVANCE III 500 at operating frequencies 300.13, 400.13, and 500.03 MHz, respectively, (¹H spectra); 75.47, 100.61, and 125.73 MHz, respectively, (¹³C{¹H} spectra) in THF-d⁸ and CDCl₃ as solvents. The ¹H chemical shifts were referenced to TMS using solvent residual protons as internal standards, 7.26 ppm for CDCl₃: 1.72 ppm for THF. The ¹³C chemical shifts were measured using samples with natural ¹³C abundance. The ¹³C chemical shifts were referenced to TMS using solvent ¹³C as internal standards, 77.16 ppm for CDCl₃; 25.26 ppm for THF. Elemental analyses were performed with a Hewlett-Packard 185 microanalyzer.

Preparation of [Rh(Oxq)(CO)(DMSO)] (I)

To a suspension of $[Rh(Oxq)(CO)_2]$ (0.628 g, 2.07 mmol) in acetonitrile (MeCN, 10 ml) a freshly prepared solution of Me₃NO·2H₂O (0.230 g, 2.07 mmol) in MeCN (38 ml) was added dropwise on stirring at 0 °C. The starting dark suspension gradually turned into brownish-yellow homogeneous solution, which was then stirred for1 h at room temperature. IR spectrum of the reaction mixture thus obtained contained two carbonyl absorption bands

Table 4

Proton–proton coupling constants, Hz, in the spectra of oxyquinolinato anion, neutral oxyquinoline, and rhodium complexes, [Rh(Oxq)(CO)(L)], $L = NH_3$, DMSO, CO (THF-d₈ solutions).

	· ·					
	H2,H3	H2,H4	H3,H4	H5,H6	H5,H7	H6,H7
NaOxq	4.2	1.8	8.2	7.8	1.3	7.9
HOxq	4.2	1.6	8.3	8.3	1.3	7.6
RhOxq(CO)(L)	4.9	1.4	8.4	8.0	1.0	7.9

attributed [1] to two monocarbonyl moieties of general formula [Rh(Oxq)(CO)(L)]: 1972 cm⁻¹ (L = MeCN) and 1948 cm⁻¹ $(L = Me_3N)$, the last band being more intense. The reaction mixture was filtered, the solvent was removed under reduced pressure, and the black lustrous residue was re-dissolved in MeCN (15 ml). IR spectrum of the solution contained two carbonyl bands with the same maxima, but of reversed intensity ratio. The mixture of DMSO (4.2 ml) with MeCN (5 ml) was added to the solution. The goldish needle-like crystals of I were precipitated gradually. The product was collected by filtration, washed with diethyl ether and dried in vacuo (1st fraction). The volume of the mother liquid was reduced by 50%, and the additional portion of the product was collected in the same manner (2nd fraction). The two fractions were combined to give totally 0.504 g of I (69% yield). Anal. Calc. for C₁₂H₁₂NO₃SRh: C, 40.79; H, 3.40; N, 3.96. Found: C, 40.58; H, 3.47; N, 3.93%. The IR spectrum of its THF solution contains intense absorption bands with maxima at 2000 cm⁻¹in the carbonyl region and at 1108-1112 cm⁻¹ (sulfoxide SO group). NMR, δ , ppm, in THF-d⁸: ¹H 9.0–6.5 (6 signals from aromatic protons of Oxq⁻ ligand, 1H each, details see in Tables 3 and 4), 3.29d, ³J(HRh) 0.7 Hz (methyl protons of DMSO ligand, 6H); ¹³C 189.38d, ¹J(CRh) 74.6 Hz (1C, carbonyl ligand), 170–100 (9 signals from aromatic carbons of Oxq⁻ ligand, 1C each, details see in Table 2), 46.69 d, ²J(CRh) 2.1 Hz (2C, methyl carbons of DMSO ligand). In CDCl₃ (if not indicated, the integral intensities and splittings of the signals are the same as in THF-d⁸): ¹H 8.57 (H2), 8.26 (H4), 7.44 (H6), 7.34 (H3), 7.02 (H5), 6.93 (H7), 3.40 (methyl protons of DMSO ligand); ¹³C 188.00, ¹J(CRh) 74.2 Hz (carbonyl ligand), 167.53 (C8), 149.13 (C2), 144.26 (C8a), 138.93 (C4), 130.62 (C4a), 130.53 (C6), 121.26, (C3), 115.29 (C7), 112.62 (C5), 47.04 (methyl carbons of DMSO ligand).

Preparation of sodium 8-hydroxyquinolinate

To a solution of HOxq (0.303 g, 2.07 mmol) in methanol (MeOH, 20 ml) a solution of NaOH (0.083 g, 2.07 mmol) in MeOH (4 ml) was added on stirring. Removal of solvent under reduced pressure yielded the desired product as a yellow fine-crystalline solid. NMR (THF-d⁸, δ , ppm): ¹H 8.44, 7.22, 7.97, 6.44, 7.13, 6.42; ¹³C 169.58, 146.38, 145.45, 136.47, 131.98, 129.74, 120.39, 114.85, 107.87.

X-ray structure determination

Crystal of I ($C_{12}H_{12}NO_3SRh$, M = 353.20) monoclinic, space group $P2_1/m$, at T = 100 K: a = 8.276(4), b = 6.956(4), c = 11.267(6)Å, $\beta = 108.612(7)^{\circ}$, V = 614.7(5)Å³, Z = 2, F(000) = 352, $d_{calc} = 1.908$ g/cm³, $\mu = 1.557$ mm⁻¹. Intensities of total 5676 reflections (1562 independent reflections, $R_{int} = 0.059$) were collected on a Bruker three-circle diffractometer equipped with an SMART 1K CCD detector (λ (MoK_{α})-radiation, graphite monochromator, φ and ω scan mode, $\theta_{max} = 28^{\circ}$) and corrected for Lorentz and polarization effects and for absorption ($T_{min} = 0.736$, $T_{max} = 0.970$)[31]. The structure was determined by direct methods and refined by a full-matrix least squares technique on F^2 with anisotropic displacement parameters for all non-hydrogen atoms. The hydrogen atoms were placed in calculated positions and refined in riding mode with fixed isotropic displacement parameters. The final *R*-factors are $R_1 = 0.083$ for 1031 independent reflections with $I > 2\sigma(I)$ and $wR_2 = 0.223$ for all 1562 independent reflections. All calculations were carried out using the SHELXTL program [32]. Crystallographic data for I have been deposited with the Cambridge Crystallographic Data Center. CCDC 976848. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk or www.ccdc.cam. ac.uk).

Appendix A. Supplementary material

CCDC 976848 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/ data_request/cif.

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