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PHYSICAL CHEMISTRY OF SOLUTIONS

Coordination of 5,10,15,20-Tetraphenyl-21*H*,23*H*-porphin by Rhenium in Various Oxidation States

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Abstract—A method was developed for the synthesis of three rhenium complexes, (5,10,15,20-tetraphenyl-21*H*,23*H*-porphinato)(phenoxo)rhenium(III) (PhO)ReTPP, (5,10,15,20-tetraphenyl-21*H*,23*H*-porphinato)(chloro)rhenium(III) (Cl)ReTPP, and μ-oxo-bis[(oxo)-(5,10,15,20-tetraphenyl-21*H*,23*H*-porphinato)rhenium(V)] $[O{=}ReTPP]_2O$, by one reaction between porphyrin H_2TPP and H_2ReCl_6 in boiling phenol. In the complex formation reaction accompanied by the redox process, only the metal cation is involved in the transformation. Rhenium(IV) as chlororhenic acid disproportionates without participation of solvent or porphyrin to give Re(III) and Re(V) complexes. The chemical structures of the products were established by spectral and elemental analysis. Characteristics of the UV, Vis, IR, and ^{1}H NMR spectra, the chromatographic mobility, and stability of the complexes were determined.

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Group 7 and 8 elements of the Periodic System have high coordinating ability. The diversity of oxidation states varying from 0 to +6 and coordination numbers in the coordination compounds gives rise to numerous combinations: 23 for Re, 17 for Ru, and 21 for Os [1]. Many complexes exhibit high catalytic and biological activities. Owing to the ability of macrocyclic ligands, especially aromatic, to stabilize unstable oxidation states of metals, the porphyrin complexes of these metals (metal porphyrins, MP) can be used as components of inorganic and organometallic materials [2].

A large number of publications consider the synthesis and properties of MP [3] and the practical use of rhenium macrocyclic coordination compounds, in particular, in radiopharmacology for photodynamic therapy [4, 5]. However, rhenium porphyrin complexes remain little studied. The published data are limited to the works dealing with the synthesis and study of monomers and dimers of Re(I), Re(II), Re(III), and Re(V) compounds of various structure [6–11].

This communication reports the new method of simultaneous synthesis of three rhenium complexes: (5,10,15,20-tetraphenyl-21*H*,23*H*-porphinato)(phenoxo)rhenium(III) (PhO)ReTPP, (5,10,15,20-tetraphenyl-21*H*,23*H*-porphinato)(chloro)rhenium(III) (Cl)ReTPP, and μ-oxo-bis[(oxo)(5,10,15,20-tetraphenyl-21*H*,23*H*-porphinato)rhenium(V)] $[O{=}ReTPP]_2O$, in one reaction. The quantitative spectral characteristics and stability data for the complexes are presented. These data were used, together with elemental analysis, to determine the structures of the complexes.

EXPERIMENTAL

The starting rhenium compound H_2ReCl_6 was prepared by the reaction of ReO_2 with HCl on heating.

(5,10,15,20-Tetraphenyl-21*H*,23*H*-porphinato)(phenoxo)rhenium(III) (PhO)ReTPP, (5,10,15,20-tetraphenyl-21*H*,23*H*-porphinato)(chloro)rhenium(III) (Cl)ReTPP, and μ-oxo-bis[(oxo)(5,10,15,20- tetraphenyl-21*H*,23*H*-porphinato)]rhenium(V) $[O{=}ReTPP]_2O$. H_2TPP andH_2ReCl_6 in 1 : 2 molar ratio were refluxed in phenol at 454 K for 2 h. After reaction completion detected by the disappearance of band for H_2TPP and termination of changes in the UV/Vis spectrum of the reaction mixture in chloroform, the contents of the reaction vessel were cooled and, after dilution with water, the products were extracted into chloroform. The $CHCl_3$ solution was repeatedly washed with warm water to remove phenol, concentrated by partly evaporating the solvent, and chromatographed on a column with Al_2O_3 (Brockman activity II) using chloroform. This gave two zones, a diffuse green zone (Fig. 1, line *1*) and concentrated green-brown zone (Fig. 1, *2*). The substances isolated from the first and second zones were chromatographed once again.

The green-zone compound was chromatographed on a silica gel (40/100 Chemapol) column, elution being performed first by benzene and then by a $CHCl_3$ c C_2H_5OH mixture (1 : 1). This gave three zones (orange, lilac, and green): (1) (Cl)ReTPP with the UV/Vis spectrum of a typical MP (Fig. 2, *1*); (2) H_2TPP; (3) $[O{=}ReTPP]_2O$ (Fig. 2, *2*).

Chromatography of the substance of the green-brown zone on a silica gel column using first benzene and then a $CHCl_3$ c C_2H_5OH mixture (1 : 1) gave pink

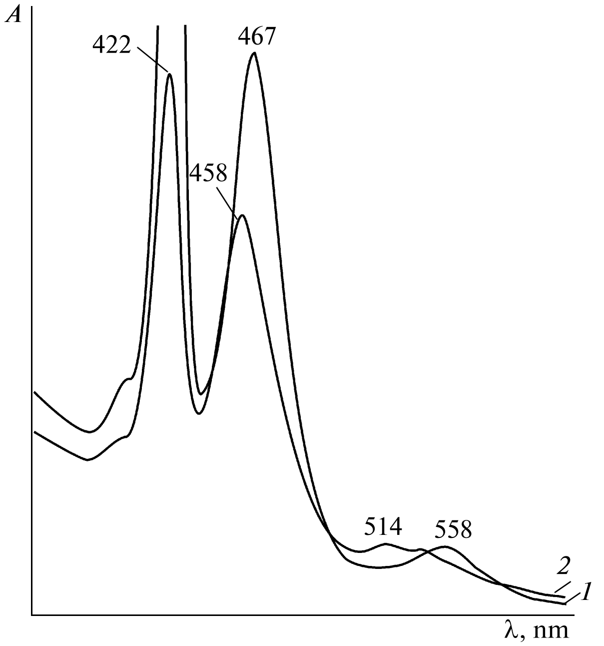


Fig. 1. UV/Vis spectra of samples isolated upon the first chromatography (Al_2O_3, elution with $CHCl_3$) of the products of reaction of H_2ReCl_6 with H_2TPP: (*1*) green zone; (*2*) green-brown zone.

(PhO)ReTPP (Fig. 2, *3*) and green $[O{=}ReTPP]_2O$ zones.

Solid amorphous samples were isolated from solutions by solvent evaporation at room temperature. The individual character and chromatographic purity of the products isolated in this way were confirmed by TLC on Silufol plates using the $CHCl_3$–2% C_2H_5OH solvent for $[O{=}ReTPP]_2O$ ($R_f = 0.42$) and benzene for (Cl)ReTPP ($R_f = 0.80$) and (PhO)ReTPP ($R_f = 0.83$).

(Cl)ReTPP. Yield 0.5%. UV/Vis ($CHCl_3$, λ_{max}, nm): 555 (sh), 525, 495 (sh), 416. UV/Vis (AcOH, λ_{max}, nm): 540 (sh), 520, 480, 437 (sh), 417.

IR (300–2000 cm^{-1}) (KBr pellets, ν, cm^{-1}): phenyl groups, 705, 759 (γ(C–H)); 1075, 1172 (δ(C–H)); 1466, 1595, 1614 (ν(C=C)); pyrrole groups, 799 (γ(C–H)); 1029 ((C_3–C_4), ν(C–N), δ(C–H)); 1350 (ν(C–N)); 1377 (ν(C=N)); coordination center, 408 (Re–N); 374, 392 (Re–Cl).

The ^1H NMR spectrum was not studied due to low product yield. The product was identified based on the hypso-type UV/Vis spectrum typical of metal porphyrins [12] (table).

(PhO)ReTPP. Yield 7%. UV/Vis ($CHCl_3$, λ_{max}, nm, (logε): 660 (sh), 551 (2.3), 436 (sh), 425 (4.3).

IR (solid random layer, ν, cm^{-1}): phenyl groups, 702, 758 (γ(C–H)); 1068, 1178 (δ(C–H)); 1487, 1578, 1600 (ν(C=C)); 2956, 3060 (ν(C–H)); pyrrole groups, 806 (γ(C–H)); 997 ((C_3–C_4), ν(C–N), δ(C–H)); 1340 (ν(C–N)); 1377 (ν(C=N)); coordination center, 418 (Re–N); 663 (Re–O); axial ligand, 1265, 1462, 1542 (–OPh).

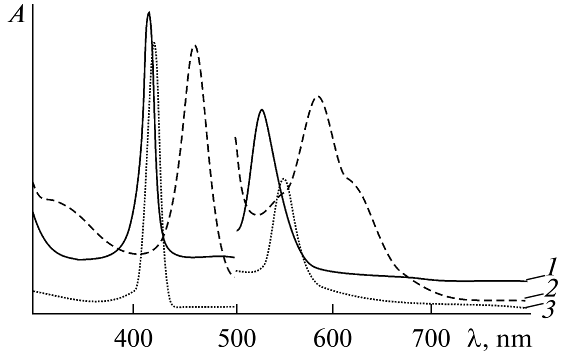


Fig. 2. UV/Vis spectra in $CHCl_3$ of (*1*) (Cl)ReTPP, (*2*) $[O{=}ReTPP]_2O$, and (*3*) (PhO)ReTPP.

^1H NMR ($CDCl_3$, δ, ppm): 8.95 (d, $8H_\beta$), 8.25, 8.15 (d, m, $8H_o$); 7.80 $8H_m$), 7.55 (m, $4H_p$), 3.63 (s, $2H_o$ (OPh)), 4,71 (s, $2H_m$ (OPh)), 5.37 (s, $1H_p$, (OPh)).

$[O{=}ReTPP]_2O$. Yield 75%. UV/Vis ($CHCl_3$, λ_{max}, nm (logε): 620 (sh), 582 (4.51), 461 (5.34), 333 (4.88).

IR (solid random layer, ν, cm^{-1}): phenyl groups, 702, 754 (γ(C–H)); 1072, 1179 (δ(C–H)); 1485, 1575, 1597 (ν(C=C)); 2954, 3056 (ν(C–H)); pyrrole groups, 801 (γ(C–H)); 1018 ((C_3–C_4), ν(C–N), δ(C–H)); 1341 (ν(C–N)); 1440 (ν(C=N)); coordination centre, 418 (Re–N); 568, 630, 854 (Re–O–Re); 961, 946 (Re–O).

^1H NMR ($CDCl_3$, δ, ppm): 9.07 (d, $8H_o$, J = 7.6 Hz), 8.79 (s, $16H_\beta$), 8.01 (t, $8H_m$, J = 7.6 Hz), 7.60 (t, $8H_p$, J = 7.6 Hz), 7.47 (d, $8H_{o'}$, J = 7.6 Hz), 7.04, 6.87 (t, t, $8H_{m'}$, J = 7.6 Hz).

^1H NMR (D_2SO_4, δ, ppm): 9.13 (m, $8H_\beta$), 8.52, 8.23 (t, t, $8H_o$), 8.06 (t, $8H_m$, 4 H_p).

For $C_{88}H_{56}N_8O_3Re_2$ anal. calcd. (%): C, 64.22; H, 3.41; N, 6.81.

Found (%): C, 63.14; H, 3.52; N, 5.59.

The UV/Vis spectra were measured on Agilent 8453 UV-Vis and Specord M-400 spectrophotometers; IR spectra were recorded on a VERTEX 80v spectrometer; the ^1H NMR spectrum was recorded on a Bruker AVANCE-500 spectrometer, using TMS as the internal standard; elemental analysis was performed on a CHNS-O Analyzer Flash EA 1112 Series.

The solid layers of complexes for IR spectroscopy was formed by evaporating the $CHCl_3$ solvent from a solution of the complex on a silicon plate.

RESULTS AND DISCUSSION

Analysis of published data [13] indicates that metal carbonyls are used most often as complexing ions for the synthesis of Group 7 and 8 metal porphyrin complexes. In this case, CO ligands are always present at

Metal porphyrins with hypso-type UV/Vis spectrum and parameters of the spectrum in $CHCl_3$

Metal porphyrin MP	M configuration	λ_{max}, nm (logε)	Refs.
$(Cl)_2HfTPP$	d^0	*414*(*4.64*)[a], 461(3.91), 497(3.67), **538(4.16)**[b], 563(sh)	[14]
$(Cl)_2TiTPP$	d^2	373(5.11), *416*(*4.39*), **494(4.14)**, 521(sh)	[15]
(Cl)ReTPP	d^4	*416*, 495(sh), **525**, 555(sh)	
(Cl)ReTPP[c]	d^4	*417*, 437(sh), 480, **520**, 540(sh)	
(Cl)RhTPP	d^6	*421*(*5.30*), **533(4.33)**, 569(3.72), 592(3.28)	[16]
(Cl)IrTPP	d^6	395(sh), *418*(*4.78*), 476(sh), 508(sh), **549(3.42)**, 588(2.81), 640(2.63)	[17]
$(Cl)_2PtTPP$	d^6	*436*(*4.57*), 517(4.26), **548(4.45)**, 587(4.19)	[18]
PdTPP	d^8	*416*(*5.50*), 480(3.68), **521(4.45)**, 552(sh), 602(3.14)	[19]
PdTPP(*i*-OCH_3)$_4$	d^8	*422*(*5.12*), **525(4.37)**, 638(3.30)	[20]
PtTPP	d^8	*402*(*4.84*), **511(4.11)**, 451(3.92)	[18]

[a] Soret band; [b] Q(0.1) band [13]; [c] in AcOH.

the coordination center, which considerably affects the properties of MP and, therefore, it is not always desirable. The use of oxides of metals in higher oxidation states solves this problem only partially as their inertness increases the time of synthesis up to 7 h in the case of Re_2O_7 and 2,3,7,8,12,13,17,18-octaethyl-21*H*,23*H*-porphin (H_2OEP) [20]. In this study, stable chlororhenic acid H_2ReCl_6 was used as the complex cation donor instead of the volatile and extremely toxic $Re_2(CO)_{10}$. This acid was prepared from rhenium(IV) oxide by the reaction

$$ReO_2 + 6HCl = H_2ReCl_6 + 2H_2O. \qquad (1)$$

The coordination of H_2TPP resulting in the formation of several individual rhenium(III) and rhenium(V) compounds is a complex redox process. According to [1], these oxidation states of rhenium give rise to the largest number of coordination compounds. The coordination of the porphyrin ligand by highly charged metal cations accompanied by redox processes is known for indium, lead, molybdenum, tungsten, and iridium complexes [21, 22]. In the first-mentioned case, the stable metal oxidation state (+3) did not change; however, the redox reaction between the solvent, boiling phenol, and porphyrin afforded indium(III) complexes with porphyrin and with its reduced form, chlorin. In all other cases, the metal cation and phenol or porphyrin underwent a redox reaction. In the reaction of H_2ReCl_6 with H_2TPP, the redox transformation involves only the rhenium(IV) cation as follows from the absence of traces of reduced H_2TPP in the reaction mixture.

Using spectral methods and elemental analysis, it is possible to determine the chemical structure of the coordination center in the synthesized rhenium complexes. The metal porphyrin nature of the compounds is confirmed by the IR spectra, which exhibit absorption bands for the bonds and fragments of pyrrole and phenyl groups. The absence of strong absorption at 964 cm^{-1} corresponding to in-plane bending vibrations, δ(N–H), in H_2TPP implies that the porphyrin ligand is not coordinated in the molecular form (so-called sitting atop complex) but is coordinated as the TPP^{2-} anion. The IR spectra of (PhO)ReTPP and $[O{=}ReTPP]_2O$ shows absorption with a maximum at 418 cm^{-1} corresponding to Re–N vibrations; in the case of (Cl)ReTPP, the maximum is shifted to 408 cm^{-1}. This may be due to the reduced force constant of the Re–N bonds caused by conjugation of Cl electron pairs with the d_π orbitals of Re. In the spectra of complexes with oxygen-containing axial ligands, the frequency of the absorption maximum does not depend on the metal oxidation state. A similar situation is observed for the platinum porphyrin complexes with 2+, 3+, and 4+ oxidation states of the metal for which

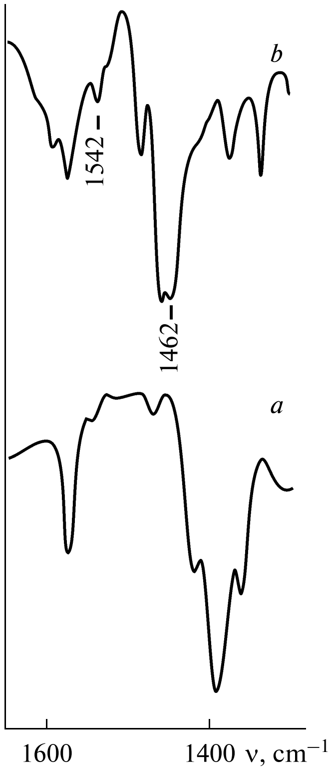


Fig. 3. IR spectra of (*a*) $[O{=}ReTPP]_2O$ and (*b*) (PhO)ReTPP in the region of phenyl C=C vibrations. Solid random layer.

the Pt–N vibration frequency in the spectra remains constant [18].

The spectrum of $[O{=}ReTPP]_2O$ contains absorption bands for the Re–O–Re vibrations in the μ-oxo bridge; in particular, the band at 854 cm^{-1} close to that for μ-O$(FeOEP)_2$ [23]. The presence of medium-intensity absorption at 650–550 cm^{-1} (630 and 568 cm^{-1}) is also indicative of the μ-oxo dimer [24]. The presence of the oxo ligand in the first coordination sphere is indicated by 961 and 946 cm^{-1} bands typical of Re=O bonds. For example, the ν(Re=O) frequency is 976 cm^{-1} in the spectrum of (oxo)(ethoxo)(N_2O_2-calix[4]arene)rhenium(V), O=Re(N_2O_2-calix)OEt [25].

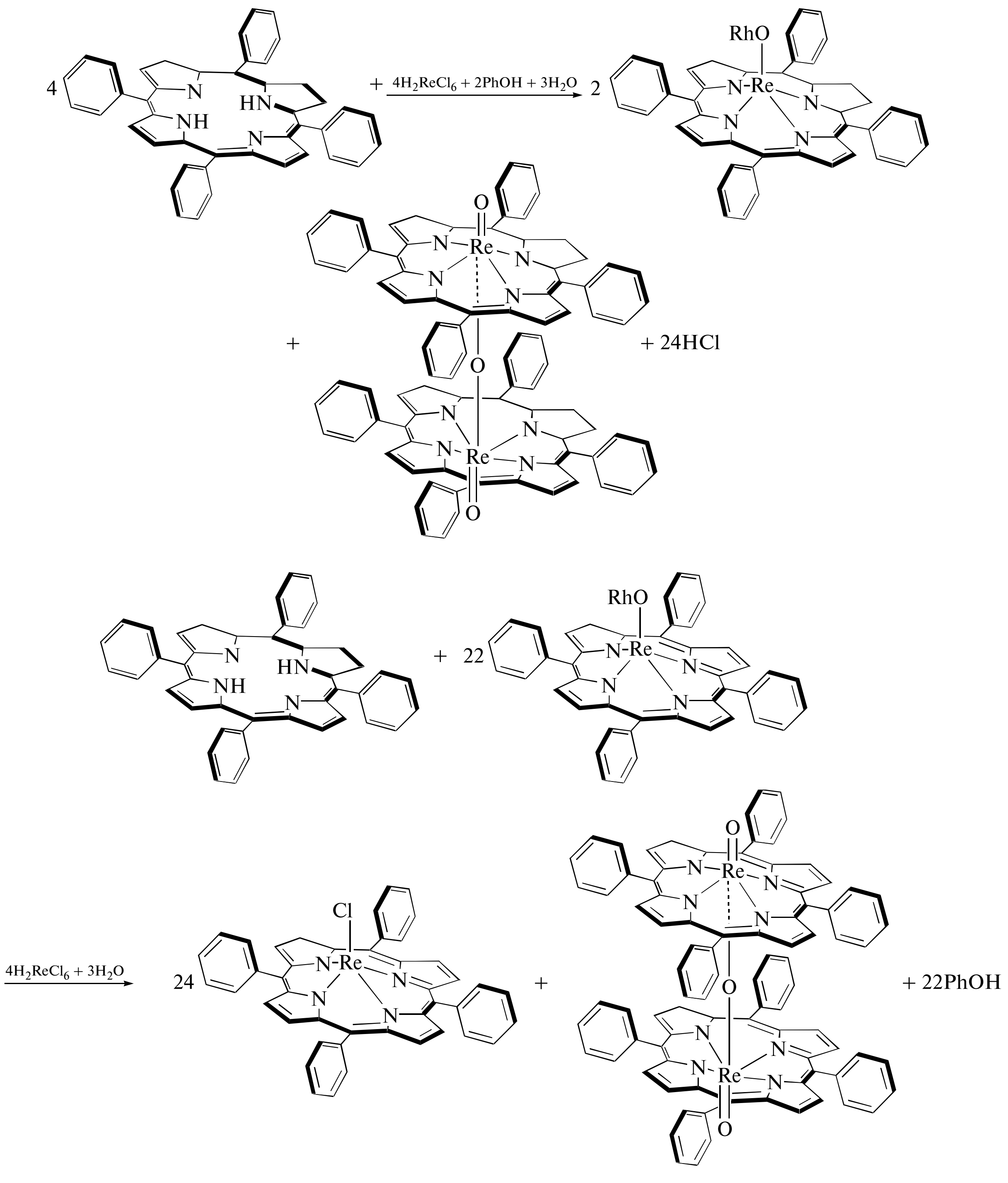
No Re–O–Re bands are recorded in the IR spectrum of (PhO)ReTPP. There is absorption with the maximum at ν = 663 cm^{-1} due to the Re–O bond vibrations [26]. The presence of the phenoxy ligand in (PhO)ReTPP is confirmed by a new intense absorption in the IR spectrum of the compound (medium-intensity bands in the whole spectrum) with a maximum at 1285 cm^{-1} (ν(C–O)). In addition, in the region of benzene C=C bonds (1487, 1578, 1600 cm^{-1} and 1485, 1575, 1597 cm^{-1} for (PhO)ReTPP and $[O{=}ReTPP]_2O$, respectively), additional absorption bands of the same nature are present; these bands at 1462 and 1542 cm^{-1} are due to phenyl in the coordinated phenoxy ligand (Fig. 3). The 374 and 392 cm^{-1} bands for (Cl)ReTPP (or 342 cm^{-1} in the spectrum of $[ReCl_6]^{2-}$ [27]) may be due to the axial ligand.

The –OPh ligand can be detected also by the presence of signals of *o*-, *m*-, and *p*-protons in the ^{1}H NMR spectrum of (PhO)ReTPP; these signals are shifted upfield with respect to the proton signals of free phenol [28]. No additional signals that could be assigned to the protons of axial ligands were found in the ^{1}H NMR spectrum of $[O{=}ReTPP]_2O$. The ^{1}H NMR spectrum of $[O{=}ReTPP]_2O$ in $CDCl_3$ differs from the spectra for free H_2TPP [29] or 1 : 1 MP, but bears considerable resemblance to the spectrum of μ-oxo dimer $[HORuTPP]_2O$ [30]. The calculation of the number of protons from the integrated curves gave more protons of each sort than in the case of 1 : 1 complexes. The signal splitting in the spectra of metal porphyrin dimers is caused by change in the symmetry dictated by the porphyrin ring. In the spectrum of $[O{=}ReTPP]_2O$, the pyrrole protons $H_β$ are responsible for a sharp singlet at δ = 8.79 ppm; the protons of the *meso*-phenyl groups H_p account for a triplet at δ = 7.60 ppm, while the H_o and H_m signals are split into two doublets and two triplets, respectively. (The high-field signal is additionally split into two triplets). The signal splitting for these sorts of protons is due to the arrangement of the phenyl groups at an angle to the macrocycle plane. The bands for $H_β$ and H_p and the H_o and H_m protons located inside the cavity formed by two macrocycles were shifted upfield relative to the signals in the spectrum of $[O{=}ReTPP]_2O$ in D_2SO_4 where the complex is monomeric. The ^{1}H NMR spectra of $[O{=}ReTPP]_2O$ in $CDCl_3$ and D_2SO_4 differ appreciably, first of all, by the number of signals in the spectrum. The number of protons calculated from the integrated curves in the spectrum in D_2SO_4 corresponds to monomeric rhenium(V) complex with H_2TPP.

The formation of μ-oxo dimer cannot be treated as incidental. The possibility of formation of this type of compound is supported by the existence of $Re_2O_3Cl_4Py_4$ and $[ReO(salpd)]_2O$ (salpd is propane-1,3-diyl-bis-salicylideneiminate), which were isolated upon the reaction of $ReOCl_3(PPh_3)_2$ with wet pyridine [4] and AlkOH [25], respectively. The porphyrin complexes containing μ-oxo bridge are also known for iron(III) [31, 32], manganese [33, 34], and ruthenium(IV) [30].

The UV/Vis spectra of (Cl)ReTPP, (PhO)ReTPP, and $[O{=}ReTPP]_2O$ (Fig. 2) are different. The spectrum of Re(V) μ-oxo dimer exhibits a band with a maximum at 350 nm, which is missing in the spectra of the H_2TPP complexes with Re(III). A similar absorption in the UV region was noted for W(V) and Mo(V) porphyrin complexes [26].

Considering the conditions of reaction between H_2TPP and H_2ReCl_6 and the composition of the resulting product mixture, the following overall reaction scheme of the formation of complexes can be suggested:



It is obvious that the formation of three Re complexes depends on the ratio of the rates of reactions involving phenol or the phenoxy complex and is largely due to different stoichiometric coefficients at (PhO)ReTPP. However, it should not depend on the H_2ReCl_6 to H_2TPP molar ratio, as follows from performing the reaction at H_2ReCl_6 to H_2TPP ratios of 1 : 2 and 1 : 5. The low yield of the chloride complex (Cl)ReTPP becomes understandable from the proposed scheme. Indeed, this complex is formed with participation of the phenoxy complex (PhO)ReTPP from H_2ReCl_6 and H_2TPP, which are consumed for the formation of (PhO)ReTPP in the first reaction. In the case of low formation rate of (PhO)ReTPP, the relative yields of complexes may be expected to shift toward (Cl)ReTPP with time. However, in reality, after 10 h of the synthesis, the yield of (PhO)ReTPP markedly decreases, and (Cl)ReTPP is not detected at

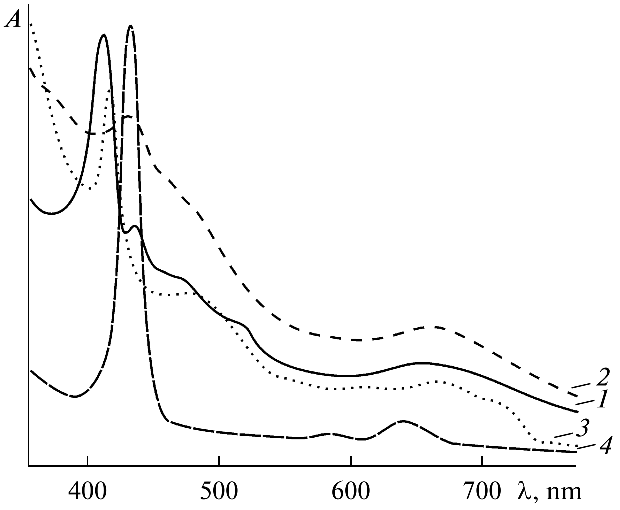


Fig. 4. UV/Vis spectra of: (Cl)ReTPP in AcOH–3 M H_2SO_4: (*1*) immediately after dissolution and (*2*) after keeping for 3 h at 398 K, and (*3*) in $CHCl_3$ after precipitation on ice; (*4*) (PhO)ReTPP in AcOH–3 M H_2SO_4 (the spectrum of H_4TPP^{2+}).

all in a mixture of H_2TPP and μ-oxo dimer. This may be due to the air oxygen oxidation (in the presence of an acid) of Re(III) in both complexes, (Cl)ReTPP and (PhO)ReTPP, which proceeds with time, as in the case of OsTPP [35]. The subsequent formation of the μ-oxo dimer of the complex oxidized in this way may be a result of gradual evaporation of HCl from the reaction medium at high temperature. Simple destruction of Re(III) complexes at the coordination center under the action of HCl is unlikely, as follows from the stability data obtained for the synthesized complexes.

A comparison of the UV/Vis spectra of the obtained complexes in $CHCl_3$ and in protic media (Fig. 4) shows that (Cl)ReTPP and $[O{=}ReTPP]_2O$ remain in the coordinated state even in AcOH mixtures with 15 M H_2SO_4, which attests to high stability of the complexs. By examining the UV/Vis spectra in an AcOH–H_2SO_4 mixture, a new form for these two complexes, i.e., the product of single-electron oxidation of the aromatic macrocycle, was detected [35]. The least stable coordination center is that in (PhO)ReTPP, which forms the protonated dication of H_2TPP in a AcOH–H_2SO_4 mixture (Fig. 4); this species was detected even in 100% AcOH. Most probably, this is caused by the $-J$ effect of the phenoxy ligand, which appears if the p_π–d_π bond of Re with the PhO oxygen is perpendicular to the benzene ring plane [29]. The state of the complexes in acids is supported by the isolation of complexes after reprecipitation from solutions (Fig. 4).

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