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CRYSTAL STRUCTURE OF PALLADIUM(II) COMPLEX WITH 2,2′-DIPYRIDYLAMINE AND 4-TOLUENESULFONYL-L-SERINE

E. Mehdipour[1], H. Bahrami[1], S. Shamaei[2], V. Amani[3]

[1]*Department of Chemistry, Faculty of Science, Lorestan University, Khoramabad, Iran*
E-mail: e_mehdipour@yahoo.com
[2]*Department of Chemistry, Faculty of Science, Khoramabad Branch, Islamic Azad University, Tehran, Iran*
[3]*Young Researchers and Elite Club, Yadegar-e-Imam Khomeini (RAH) Branch, Islamic Azad University, Tehran, Iran*

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The [Pd(dpa)(tsser)] complex (**1**) is prepared from the reaction of $PdCl_2$ and 2,2′-dipyridylamine (dpa) with 4-toluenesulfonyl-L-serine ($tsserH_2$). This complex is characterized by spectral methods (IR, UV-Vis, ^{1}H NMR, and luminescence), elemental analysis, thermal analysis (TG, DTA), and single crystal X-ray diffraction. X-ray structure determinations show that in this complex, Pd^{II} atoms are four-coordinated in a distorted square-planar configuration by two N atoms from a bidentate 2,2′-dipyridylamine ligand and one N atom and one O atom from a bidentate $tsser^{2-}$ ligand.

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Keywords: crystal structure, palladium(II) complex, 4-toluenesulfonyl-L-serine, 2,2′-dipyridylamine, thermal gravimetric, differential thermal analyses, luminescent properties.

INTRODUCTION

There is a significant similarity between the coordination chemistry of palladium(II) and platinum(II) compounds; therefore, palladium(II) complexes were investigated for developing new antitumor agents [1—7]. Palladium(II) complexes also have applications in catalysis [8—11], electronics, optics [12—14] and antibacterials and antifungals [15—17]. Only eight palladium(II) mixed-ligand complexes with a formula of [Pd(L)(amino acid)] [18—23] have been synthesized and fully characterized by spectroscopic methods and their structures were studied by the single crystal X-ray diffraction method. Furthermore, all of these palladium(II) mixed-ligand complexes have shown significant cytotoxic activity against several human tumor cell lines. The effect of the chemical structure on the cytotoxic properties has also been reported [24—28]. It should be noted that the investigation of the relation between the chemical structure and cytotoxic properties is needed to synthesize similar complexes with small differences in their compositions. Thus, in the present paper, we report the synthesis, characterization, and crystal structure of a new palladium(II) mixed-ligand complex containing 2,2′-dipyridylamine and 4-toluenesulfonyl-L-serine.

Materials and instrumentation. All chemicals were purchased from Merck and Aldrich. Infrared spectra (4000—250 cm^{-1}) of solid samples were taken as 1 % dispersion in CsI pellets using a Shimadzu-470 spectrometer. NMR was recorded on a Bruker AC-300 spectrometer for protons at 300.13 MHz in DMSO-d_6. Melting points were obtained on a Kofler Heizbank Rechart type 7841 melting point apparatus. Elemental analysis was performed using a Heraeus CHN—O Rapid analyzer.

Table 1

Infrared frequencies of dpa, tsserH_2, *and complex* **1** (cm^{-1})

Compound	ν(N—H)	$\nu(O—H)_{COOH}$	ν(C—H)	ν(CO)	ν_{as}(COO)	ν_s(COO)	ν(C=N), ν(N=N)	ν(ring deformation)	ν(Pd—X)[a]
dpa	3423m	—	3250m	—	—	—	1605s	1147m	—
			3099m				1529s	997m	
			2938m				1463s	769s	
							1437	741m	
							1343m	735w	
							1314m	624w	
								599w	
								523w	
tsserH_2	3277s	3410br	3022w	1732s	—	—	1593m	1162s	—
			2959m				1315s	1098m	
			2942m				1185m	1023s	
								971m	
								842s	
								824s	
								685s	
								575m	
								559m	
1	3444m	—	3136w	—	1630s	1361s	1531m	1138s	440m
			3077w				1480s	1087m	383m
			2987w				1419w	1040m	330w
							1278m	974m	261m
								806m	
								672m	
								615m	
								586m	
								568m	
								533m	

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[a] X is N and O.

Thermal behavior was measured with an STA 503 Bähr apparatus. UV-Vis spectra were recorded on a Shimadzu 2100 spectrometer using a 1 cm path length cell DMSO at room temperature. Luminescence spectra were recorded on a Perkin Elmer LS 45 using a 1 cm path length cell.

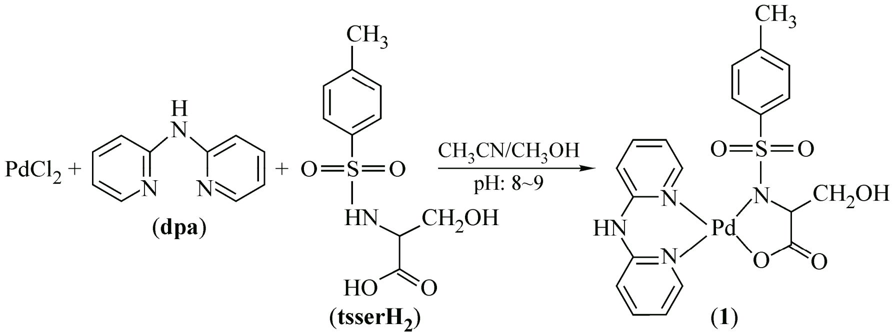
Synthesis of 4-toluenesulfonyl-L-serine (tsserH_2). 4-Toluenesulfonyl-L-amino acid was prepared according to the procedure previously described in [19]. Yield 73.3 %, m.p. 228 °C. Infrared frequencies (CsI, cm^{-1}) are reported in Table 1. UV-Vis (DMSO, 4.2×10^{-5} M): $\lambda_{max} = 258$, 264 and 274 nm. ^{1}H NMR (DMSO-d_6, ppm): 12.45 (s, br, 1H, COOH), 7.91 (d, $J = 8.7$ Hz, 1H, NH), 7.67 (d, $J = 7.8$ Hz, 2H, ArH), 7.34 (d, $J = 7.8$ Hz, 2H, ArH), 4.97 (s, 1H, OH), 3.74—3.68 (m, 1H, CH), 3.62—3.46 (m, 2H, CH_2), 2.36 (s, 3H, CH_3). Elemental analysis: $C_{10}H_{13}NO_5S$ (%) (259.28); anal. calcd (%): C 46.32, H 5.01, N 5.40. Found: C 46.01, H 4.97, N 5.35.

Synthesis of [Pd(4,4′-dmbipy)(tsser)] (1). A suspension of $PdCl_2$ (0.09 g, 0.5 mmol) in CH_3CN (50 ml) was stirred and warmed to 70 °C, giving an orange solution. Then, 2,2′-dipyridylamine (0.09 g, 0.5 mmol) in CH_3CN (10 ml) was added and the mixture was stirred at 70 °C. After 10 min, a mixture of 4-toluenesulfonyl-L-serine (0.13 g, 0.5 mmol) in 20 ml CH_3OH and 2 ml NaOH (0.50 M in an aqueous solution, 1.0 mmol) was added and the contents refluxed for 3 h. The solution was filtered and allowed to crystallize at room temperature, whereupon yellow block crystals formed after two weeks (yield 0.20 g, 74.8 %, m.p. 250 °C). Infrared frequencies (CsI, cm^{-1}) are reported in Table 1. UV-Vis (DMSO, 4.4×10^{-5} M): λ_{max} = 262, 305 and 326 nm. ^{1}H NMR (DMSO-d_6, ppm): 11.01 (s, 1H, NH, dpa). 8.88 (d, J = 5.7 Hz, 1H, dpa), 8.04 (d, J = 5.7 Hz, 1H, dpa), 8.00—7.92 (m, 2H, dpa), 7.79 (d, J = 7.8 Hz, 2H, ArH, tssr), 7.23—7.20 (m, 4H, ArH, tsser and dpa), 7.12 (t, 2H, dpa), 5.07 (s, 1 H, OH, tsser), 3.94—3.90 (m, 1H, CH, tsser), 3.65—3.46 (m, 2H, CH_2, tsser), 2.30 (s, 3H, CH_3, tsser). Elemental analysis: $C_{20}H_{20}N_4O_5PdS$ (%) (534.87); anal. calcd. (%): C 44.91, H 3.74, N 10.47. Found: C 44.52, H 3.71, N 10.36.

Single crystal X-ray diffraction analysis. The X-ray diffraction measurements were made on a Bruker APEX II CCD area detector diffractometer at 298 K (MoK_α radiation, graphite monochromator, λ = 0.71073 Å). The structure of **1** was solved by SHELX-97 and absorption corrections were done using the SADABS programs [29, 30]. Softwares including Bruker APEX II (data collection and cell refinement) [31], Bruker SHELXTL (data reduction) [32], and WinGX (publication material) [33] were properly used. The molecular graphics programs used were ORTEP-3 for WINDOWS [34], PLATON, and MERCURY [35]. Crystal chemical information on the structure investigated was deposited with the Cambridge Structural Database under number CCDC 1015082, from where it can be obtained free of charge on request at the following website: www.ccdc.cam.ac.uk/products/csd/faqs.

RESULTS AND DISCUSSION

Synthesis of [Pd(dpa)(tsser)] (1). 4-Toluenesulfonyl-L-serine was synthesized according to the reported procedure [19]. Compound **1** was also obtained from the reaction of one equivalent of $PdCl_2$ and one equivalent of 2,2′-dipyridylamine ligand with one equivalent of the 4-toluenesulfonyl-L-serine ligand in a mixture of CH_3CN/CH_3OH (3:1) and in the presence of a 0.5 M sodium hydroxide solution. Suitable crystals of **1** for X-ray diffraction studies were grown by slow evaporation of its solution at room temperature over two weeks. The synthetic route of **1** is shown in Scheme 1.



Scheme 1. The preparation method of compound **1**

Description of the molecular structure of [Pd(dpa)(tsser)] (1). Crystallographic data for **1** are given in Table 2. The ORTEP view with the numbering scheme for **1** is shown in Fig. 1. This compound crystallized in the monoclinic $P2_1$ space group. As shown in Fig. 1, each palladium(II) cation is four-coordinated in a distorted square-planar configuration by two N atoms from a bidentate 2,2′-dipyridylamine ligand and one N atom and one O atom from a bidentate $tsser^{2-}$ anion. The Pd—N_{dpa} bond lengths are 2.002(7) Å and 2.029(7) Å, the Pd—N_{tsser} bond length is 2.020(7) Å, and the Pd—O bond length is 2.018(6) Å. The Pd—N and Pd—O bond distances are within the ranges of those for other analogical palladium(II) complexes [19—23]. The N1—Pd—N3, N1—Pd—N4, N3—Pd—O3, and N4—Pd—O3 bond angles are 89.7(3), 94.9(3), 93.6(3), and 82.3(3)° respectively. The angle between planar N1—Pd1—N3 and planar O1—Pd1—N4 is 8.04°, which indicates that the Pd1—O3—N3—N1—N4 plane is slightly distorted. The pyridine rings are also strongly distorted from planarity.

Table 2

Crystallographic and structure refinement data for complex **1**

Parameter	Value
Formula	$C_{20}H_{20}N_4O_5PdS$
Temperature, K	298(2)
Molecular weight	534.87
Wavelength, Å	MoK_α (0.71073)
Crystal system	Monoclinic
Space group	$P2_1$
a, b, c, Å; β, deg.	7.3714(8), 16.5799(12), 8.7178(8); 101.018(8)
V, Å^3	1045.82(17)
Z	2
ρ_{calc}, g/cm^3	1.699
μ, mm^{-1}	1.028
F(000)	540
Crystal size, mm	0.30×0.27×0.26
Range of data collection over θ, deg.	2.38—26.99
Intervals of reflection indices	$-8 \le h \le 9$, $-21 \le k \le 21$, $-11 \le l \le 11$
Number of meas. / indep. reflections	7229 / 4563 (R_{int} = 0.0333)
Completeness over θ = 26.99, %	99.9
R factors over $F^2 > 2\sigma(F^2)$	R_1 = 0.0296, wR_2 = 0.0664
R factors over all reflections	R_1 = 0.0368, wR_2 = 0.0676
S	0.961
Residual electron density (min / max), e/Å^{-3}	0.533 / –0.445

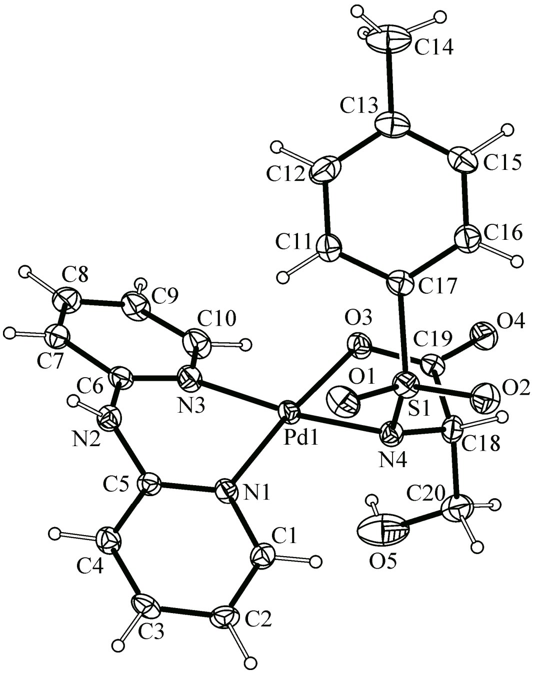


Fig. 1. The labeled diagram of [Pd(dpa)(tsser)] (**1**). Thermal ellipsoids are at a 30 % probability level

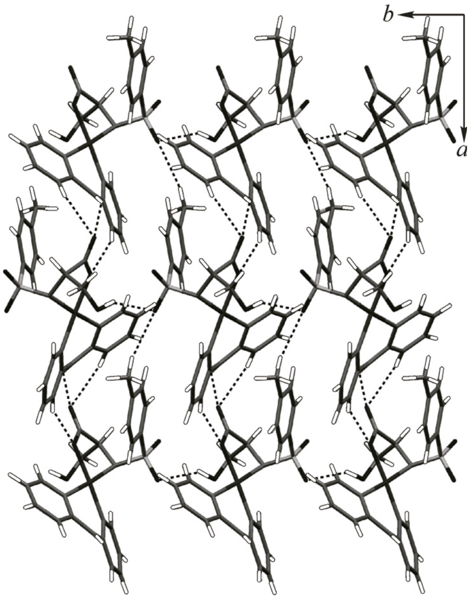


Fig. 2. Intermolecular N—H···O and C–H···O hydrogen bonds in [Pd(dpa)(tsser)] (**1**) viewed along the *c* axis

Table 3

Hydrogen bond geometry for complex **1** (Å *and* deg.)

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O4$^{\#1}$	0.86	2.15	2.811(11)	134
O5—H5···O1$^{\#2}$	0.93(16)	2.16(14)	2.946(13)	142
C1—H1···N4	0.93	2.55	3.054(12)	115
C3—H3···O2$^{\#3}$	0.93	2.57	3.266(13)	132
C4—H4···O3$^{\#1}$	0.93	2.42	3.273(12)	153
C7—H7···O4$^{\#1}$	0.93	2.57	3.274(13)	133
C10—H10···O3	0.93	2.45	2.958(15)	114
C16—H16···O2	0.93	2.51	2.890(13)	104
C18—H18···O2	0.98	2.44	2.927(13)	110

Symmetry operation: $^{\#1}$–*x*, 1/2+*y*, 1–*z*; $^{\#2}$1+*x*, *y*, *z*; $^{\#3}$ 1–*x*, 1/2+*y*, 2–*z*.

The mean planes of A (N1/C1—C5) and B (N3/C6—C10) rings make the following dihedral angle with each other: A/B = 34.25°.

In the crystal structure of **1** (Fig. 2) there are no π···π interactions between the pyridine and phenyl rings, and only intermolecular N—H···O, O—H···O and C—H···O hydrogen bonds (Table 3) are effective in the stabilization of the crystal structure and the formation of the 3-D supramolecular assembly.

Spectroscopic characterization of tsserH$_2$ and 1 compounds. The infrared spectra in Table 1 show the vibrational frequencies for free and coordinated dpa and tsserH$_2$ ligands in compound **1**. The medium absorption band at 3444 cm^{-1} in the IR spectrum of **1** has been assigned to ν(N—H)$_{dpa}$ [36, 37]. The vibrational bands at 3410 cm^{-1} and 3277 cm^{-1} are assigned to ν(O—H)$_{COOH}$ and ν(N—H), respectively, for tsserH$_2$. These bands disappear for complex **1**, showing that the tsserH$_2$ ligand has been deprotonated and that Pd—O and Pd—N bonds have formed [19—22]. The weak absorption bands present in the region of 3136 cm^{-1} to 2938 cm^{-1} are due to the C—H stretching of phenyl and pyridine rings and methyl groups [38]. The strong band at 1732 cm^{-1} in the IR spectrum of tsserH$_2$ has been assigned to ν(CO). For the free ligand this band is shifted to lower frequencies upon coordination in **1**. The strong bands at 1630 cm^{-1} and 1361 cm^{-1} for complex **1** are assigned to ν$_{as}$(COO) and ν$_s$(COO), respectively. The frequency difference Δν [Δν = ν$_{as}$(COO) – ν$_s$(COO)] is correlated with the coordination mode of a carboxylate ligand. The Δν difference is 269 cm^{-1} for compound **1**, which is greater than that of sodium acetate (164 cm^{-1}), as anticipated for a monodentate mode of the carboxylate ligand [39, 40]. Moreover, several bands in the range 500—1605 cm^{-1} are related to the heteroaromatic ring mode vibrations for C=C, C=N, C—C, and C—N [36, 38]. New bands in the range 261—440 cm^{-1} are assigned to Pd—N$_{dpa}$, Pd—N$_{tsser}$, and Pd—O$_{tsser}$ stretching vibrations [37, 41—44].

The UV-Vis spectra of a dimethyl sulfoxide solution of tsserH$_2$ and **1** have a broad band in the range 262—274 nm, assigned to the ligand π—π* transition, and two broad bands at 305 and 326 nm for **1** are assigned to the metal-to-ligand charge transfer (MLCT) [19—22].

The ^{1}H NMR spectra of the title compounds in the DMSO-*d*$_6$ solution are given in Fig. 3. As shown in Fig. 3, the ^{1}H NMR spectrum of 4-toluenesulfonyl-L-serine exhibited a broad singlet at 12.45 ppm for the —COOH group and a doublet at 7.91 ppm for the —NH group. These signals are absent in the spectrum of compound **1**, indicating the removal of —COOH and —NH protons and the formation of Pd—O and Pd—N bonds. As shown in Fig. 1, the X-ray structure of compound **1** shows that half of the 2,2′-dipyridylamine ligands lie in the diamagnetic protection zone of the phenyl aromatic ring. Thus, this phenomenon corroborates with the chemical shifts observed for H9, H10, and H12 in NMR.

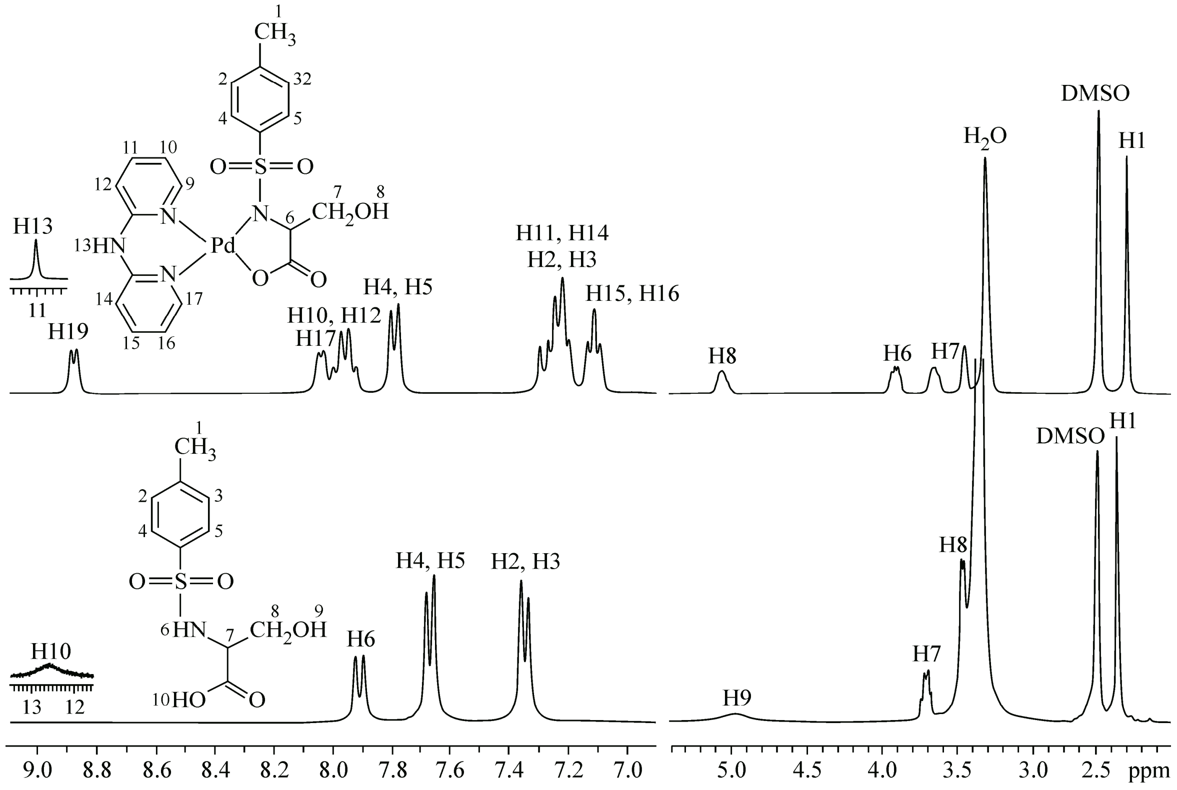


Fig. 3. ^{1}H NMR spectra of the tsserH$_2$ ligand and [Pd(dpa)(tsser)] (**1**) complex in DMSO-d_6

The luminescence emission spectra of dpa, tsserH$_2$, and **1** were obtained in the DMSO solution at room temperature and the results are displayed in Fig. 4. As shown in Fig. 4 (λ_{ex} = 360 nm), dpa, tsserH$_2$, and **1** exhibit broad luminescent emission centered at 408, 419, and 429 nm, respectively. There are red shifts of the emission energies of dpa and tsserH$_2$ after coordination to the Pd(II) cation in complex **1**. The shapes and intensities of the luminescence emission spectra of dpa, tsserH$_2$, and complex **1** are similar, therefore, the emission properties of complex **1** are believed to originate from $\pi^* \to \pi$ or $\pi^* \to n$ transitions in dpa and tsserH$_2$ ligands. The observed red shift for **1** can be explained by a decrease in the energy gap between the ground and excited states in the dpa and tsserH$_2$ ligands upon complexation with Pd(II). The same trend is found for other complexes with different metal centers [45—49].

Thermal studies of [Pd(dpa)(tsser)] (1). The thermal stability of **1** have been determined on single crystal samples between 30—780 °C in the air atmosphere with a heating rate of 10 °C·min^{-1} by thermogravimetric (TG) and differential thermal analyses (DTA). The thermogravimetric analysis of **1** shows that chemical decomposition starts at around 240 °C and ends at around 590 °C with a weight loss of 75.8 % corresponding to the removal of one 2,2′-dipyridylamine ligand and one 4-toluenesulfonyl-L-serine ligand (calcd.: 77.1 %). The final residual weight is 24.2 % corresponding to PdO (calcd. 22.9 %) [50, 51]. The differential thermal analysis of complex **1** displays three distinct endothermic peaks at 242, 385, and 587 °C and three distinct exothermic peaks at 252, 446, and 524 °C.

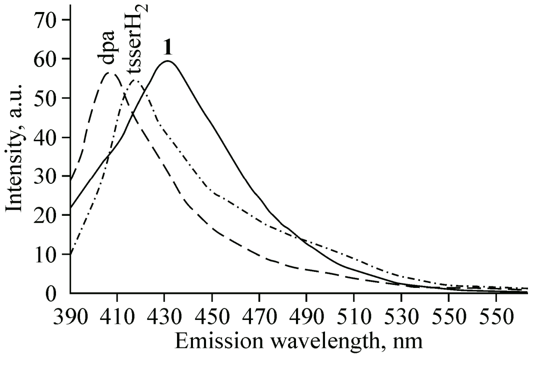


Fig. 4. Luminescence spectra of dpa (5.26×10^{-4} M), tsserH$_2$ (5.24×10^{-4} M), and **1** (5.25×10^{-4} M) in DMSO at room temperature; excitation wavelength = 360 nm

CONCLUSIONS

In this work, a new palladium(II) mixed-ligand complex [Pd(dpa)(tsser)] (**1**), containing the 4-toluenesulfonyl-L-serine and 2,2′-dipyridylamine ligands, has been synthesized and its structure was determined by the single crystal X-ray diffraction method. The structural analysis of this complex shows that the Pd atom has a distorted square planar geometry through the coordination of two N atoms from the bidentate dpa ligand and one N atom and one O atom from the bidentate $tsser^{2-}$ ligand. Also from the thermal analysis it is found that this compound decomposed between 240—590 °C by losing 75.8 % of its weight corresponding to the removal of one 2,2′-dipyridylamine ligand and one 4-toluenesulfonyl-L-serine ligand.

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