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Cyclometallated Iridium(III) Complex with the Norbornene-Substituted Pyrazolonate Ligand and Related Copolymers: Syntheses, Structures, and Photophysical Properties

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Abstract—New cyclometallated iridium(III) complex $(NBEpz)Ir(Ppy)_2 \cdot 2CH_2Cl_2$ (**I**) (NBEpzH is 1-phenyl-3-methyl-4-(5-bicyclo[2.2.1]hept-5-en-2-yl)-5-pyrazolone, PpyH is 2-phenylpyridine) is synthesized and structurally characterized. Compound **I** undergoes metathesis polymerization and forms new iridium-containing copolymers. The photophysical properties of complex **I** and related copolymers are studied.

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INTRODUCTION

Cyclometallated iridium(III) complexes have efficient photo- and electroluminescence properties and are used as emission materials for organic light-emitting diodes (**OLED**) [1, 2], chemisensors [3], and biomarkers [4]. The luminescence efficiency of the iridium compounds and the color of the radiation depend considerably on the nature of the ligands bound to the metal [5, 6]. The variation of the ligand environment makes it possible to control the luminescence characteristics and, hence, investigations in the field of the synthesis of new cyclometallated iridium complexes and studies of their photo- and electroluminescence properties are being currently developed. The electroluminescent iridium-containing polymers have recently been synthesized, and OLED devices generating the radiation of diverse colors were constructed on the basis of these polymers [7–11].

In this work, we report the synthesis of the new cyclometallated iridium(III) complex with the norbornene-substituted pyrazolonate ligand. The complex obtained undergoes metathesis copolymerization with carbazole-containing monomers and forms copolymers with intense photoluminescence in the green spectral range.

EXPERIMENTAL

All procedures with easily oxidizable and hydrolyzable substances were carried out in vacuo or under argon using the standard Schlenk technique. The solvents used were thoroughly purified and degassed. 1-Phenyl-3-methyl-4-(5-bicyclo[2.2.1]hept-5-en-2-yl)-5-pyrazolone [12], $[(Ppy)_2IrCl]_2$ [13], $(H_2IMes)(3\text{-}Br\text{-}Py)_2(Cl)_2Ru = CHPh$ (Grubbs catalyst of the third generation) [14, 15], and bicyclo[2.2.1]hept-5-en-2-yl(9*H*-carbazol-9-yl)methanone (*endo* isomer (**2-*endo***) and *exo* isomer (**2-*exo***)) [16] were synthesized using known procedures.

^1H and $^{13}C\{1H\}$ NMR spectra were recorded on Bruker DPX-200 (^1H NMR: 200MHz, ^{13}C NMR: 50 MHz) and Bruker Avance III-400 (^1H NMR: 400 MHz, ^{13}C NMR: 100 MHz) spectrometers. Chemical shifts are given in ppm relative to tetramethylsilane. IR spectra were recorded on an FSM 1201 FT-IR spectrometer. A sample of complex **I** was prepared by pressing a pellet with the substance to KBr ratio equal to 1 : 200. Samples of polymers **P1** and **P2** were prepared as thin films between KBr plates. The molecular weight distribution of the polymers was determined by gel permeation chromatography on a Smartline RID 2300 differential refractometer as a detector and a set of two Phenomenex columns packed with the Phenogel sorbent (pore size 10^4 and 10^5 Å, respectively). Tetrahydrofuran (THF) served as an eluent (2 mL/min, 40°C). The columns were calibrated by 13 polystyrene standards.

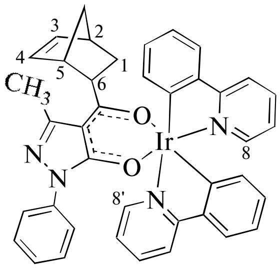
Differential scanning calorimetry was carried out on a DSC 204 F1 Phoenix instrument (Netzsch) in a dry argon flow (flow rate 20 cm^3/min, heating rate 5°C/min).

The absorption spectra of the iridium complex and copolymers in a CH_2Cl_2 solution were recorded on a PerkinElmer Lambda 25 spectrometer. Photoluminescence (**PL**) spectra were detected on a PerkinElmer LS 55 fluorescence spectrometer. The relative quantum yields of compound **I** and copolymers *P*1 and *P*2 were determined at ambient temperature in degassed CH_2Cl_2 solutions. The values of quantum yields were calculated relatively to Rhodamine 6G in ethanol (Φ = 0.95) [17] using a described procedure [18].

Synthesis of (NBEpz)Ir(Ppy)$_2$ · 2CH$_2$Cl$_2$ (I). 1-Phenyl-3-methyl-4-(5-bicyclo[2.2.1]hept-5-en-2-yl)-5-pyrazolone (0.1086 g, 0.369 mmol) in DME (5 mL) was gradually added to a suspension of NaH (0.01 g, 0.417 mmol) in DME (5 mL) under argon. After dihydrogen stopped evolving, the mixture was stirred at ambient temperature for 2 h and filtered off. The obtained solution of sodium pyrazolonate was added to a solution of [(Ppy)$_2$IrCl]$_2$ (0.20 g, 0.187 mmol) in DME (15 mL). The reaction mixture was refluxed with stirring for 16 h. After the solvent was replaced by CH$_2$Cl$_2$, a yellow solution was separated from a colorless precipitate by centrifuging. The slow evaporation of the solvent at ambient temperature gave 0.33 g (92%) of complex **I** as stable in air yellow crystals.

IR (KBr) ν, cm^{-1}: 3054 ν, 3042 ν, 1095 ν, 1063 ν, 1030 ν, 1004 ν, 755 $\gamma(C_{Ar}{-}H)$; 2926 ν, 1397 β_s, 1313 β, 1158 $\beta(C_{Alk}{-}H)$; 1603 $\nu_{as}(C\cdots O)$; 1592 ν, 1498 ν_{as}, 1475 ν, 1265 $\nu(C{=}C_{Ar})$; 1527 ν, 1452 ν, 1397 ν(pyrazole ring); 906 ν, 876 ν(C–C); 693 β, 631 β, 613 ν(chelate ring); 512 ν, 485 ν, 475 ν(Ir–O).

According to the NMR spectroscopic data, complex **I** is a mixture of *endo* and *exo* isomers.



***Endo* isomer** (66%). ^{1}H NMR (CDCl$_3$), δ, ppm: 8.58 d (1H, *J* = 5.6 Hz, H^8), 8.18 d (1H, *J* = 5.7 Hz, H$^{8'}$), 7.77 t (2H, Ar), 7.65 m (4H, Ar), 7.49 m (2H, Ar), 7.08 m (2H, Ar), 6.96 m (3H, Ar), 6.77 m (2H, Ar), 6.63 m (2H, Ar), 6.26 m (2H, Ar), 5.37 dd (1H, *J* = 5.4 Hz, 3.0 Hz, H^3), 5.30 s (4H, CH$_2$), 4.35 dd (1H, *J* = 5.5 Hz, 2.8 Hz, H^4), 3.43 m (1H, H^6), 2.80 br.s and 2.62 br.s (1H each, H^2 and H^5), 2.44 s (3H, Me), 1.54 m (1H, H^1), 1.18 m (2H, H^7 and H$^{7'}$), 0.66 m (1H, H$^{1'}$).

***Exo* isomer** (34%). ^{1}H NMR (CDCl$_3$), δ, ppm: 8.46 d (1H, *J* = 5.6 Hz, H^8), 8.41 d (1H, *J* = 5.9 Hz, H$^{8'}$), 7.80–6.20 m (19H, Ar), 6.00 m (2H, H^3 and H^4), 5.30 s (4H, CH$_2$), 2.57 br.s and 2.54 br.s (1H each, H^2 and H^5), 2.41 s (3H, Me), 2.24 m (1H, H^6), 1.80–0.80 m (4H, H^1, H$^{1'}$, H^7 and H$^{'}$).

All isomers. ^{13}C NMR (CDCl$_3$), δ, ppm: 193.9, 191.8, 169.0, 168.6, 161.8, 161.7, 149.4, 148.6, 148.5, 148.2, 147.6, 146.6, 145.5, 145.1, 144.6, 139.4, 138.8, 138.6, 137.1, 137.0, 136.7, 133.5, 133.3, 133.2, 130.5, 128.9, 128.5, 123.9, 123.5, 123.4, 121.4, 121.3, 119.3, 118.34, 118.27, 117.93, 117.87, 105.5, 53.4, 49.8, 49.5, 48.2, 47.9, 46.8, 46.5, 46.0, 44.9, 43.2, 42.4, 41.9, 32.5, 29.7, 28.7, 28.4, 18.2, 17.8.

For C$_{42}$H$_{37}$N$_4$O$_2$Cl$_4$Ir

anal. calcd., %:	C, 52.34;	H, 3.87.
Found, %:	C, 52.25;	H, 3.80.

Synthesis of copolymer *P*1. The Grubbs catalyst of the third generation (0.0038 g, 0.046 mmol) in CH$_2$Cl$_2$ (3 mL) was added to a mixture of monomers **I** (0.05 g, 0.052 mmol) and 2-*exo* (0.12 g, 0.042 mmol) in CH$_2$Cl$_2$ (5 mL). The mixture was stirred at ambient temperature. The course of the reaction was monitored by thin layer chromatography. After the end of copolymerization (2 h), several droplets of vinyl ethyl ether was added to the reaction mixture to decompose the catalyst, and the mixture was stirred for 20 min more. The copolymer formed was precipitated with methanol, additionally purified by reprecipitation with methanol from CH$_2$Cl$_2$, and dried in vacuo at ambient temperature to a constant weight. The yield of copolymer *P*1 as a yellow powder was 0.15 g (84%).

IR, ν, cm^{-1}: 3122 ν(C=C–H), 3057 ν, 1098 ν, 1062 ν, 1035 ν, 1007 ν, 754 $\gamma(C_{Ar}{-}H)$; 2959 ν(C–H), 1375 β, 1158 $\beta(C_{Alk}{-}H)$; 1687 ν(N–C=O); 1604 $\nu_{as}(C\cdots O)$; 1595 ν, 1490 ν, 1477 ν, 1238 $\nu(C{=}C_{Ar})$; 1533 ν(pyrazole ring); 972 ν(C–C); 693 β, 631 β, 620 ν(chelate ring); 512 ν, 482 ν(Ir–O).

^{1}H NMR (CDCl$_3$), 200 MHz, δ, ppm: 0.6–4.00 m (75H), 4.64–5.45 m (20H), 6.81–7.42 m (55H, Ar), 7.47–8.17 m (55H, Ar).

For C$_{200}$H$_{169}$N$_{12}$O$_{10}$Ir

anal. calcd., %:	C, 77.67;	H, 5.51.
Found, %:	C, 77.64;	H, 5.56.

Copolymer *P*2 was synthesized from compound **I** (0.05 g, 0.052 mmol) and 2-*endo* (0.12 g, 0.042 mmol) similarly to the above procedure. The copolymerization time was 3 h. The yield was 0.13 g (76%).

IR, ν, cm^{-1}: 3122 ν(C=C–H), 3060 ν, 1098 ν, 1062 ν, 1034 ν, 1006 ν, 754 $\gamma(C_{Ar}{-}H)$; 2944 ν(C–H), 1313 β, 1158 $\beta(C_{Alk}{-}H)$; 1687 ν(N–C=O); 1604 $\nu_{as}(C\cdots O)$; 1595 ν, 1490 ν, 1477 ν, 1238 $\nu(C{=}C_{Ar})$; 1533 ν(pyrazole ring); 972 ν, 910 ν(C–C); 693 β, 631 β, 619 ν(chelate ring); 512 ν, 480 ν(Ir–O).

^{1}H NMR (CDCl$_3$, 200 MHz), δ, ppm: 0.6–4.19 (64H), 4.60–5.71 (20H), 6.81–7.49 (60H), 7.49–8.26 (60H).

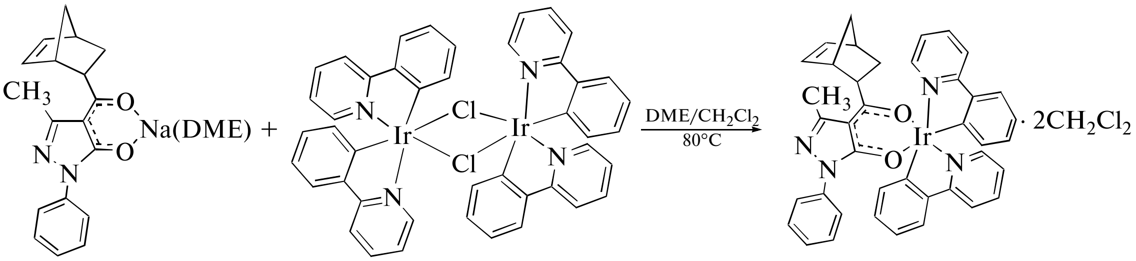
For C$_{200}$H$_{169}$N$_{12}$O$_{10}$Ir

anal. calcd., %:	C, 77.67;	H, 5.51.
Found, %:	C, 77.62;	H, 5.58.

X-ray diffraction analysis of compound I was carried out on a Smart Apex diffractometer (MoK_α, graphite monochromator). The structure was solved by a direct method followed by the full-matrix least-squares refinement for F^2 using a described program [19]. An absorption correction was applied using the SADABS program [20]. All non-hydrogen atoms were refined in the anisotropic approximation. Hydrogen atoms were placed in the geometrically calculated positions and refined in the riding model. The crystallographic data and the main refinement parameters are given in Table 1. Crystallographic information for compound **I** was deposited with the Cambridge Crystallographic Data Centre (no. 921082; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

Norbornene-substituted iridium complex **I** was synthesized by the reaction of substituted sodium pyrazolonate with iridium(III) chloride containing the phenylenepyridine ligands



(I)

Compound **I** was synthesized in a high yield as a stable in air yellow crystalline substance soluble in chloroform, CH_2Cl_2, and DME. The NMR studies showed that the product was a mixture of *endo* and *exo* isomers in a ratio of 66 : 34.

The structure of complex **I** was determined by X-ray diffraction analysis (Fig. 1, Table 2). The iridium atom has a distorted octahedral coordination mode. The base of the octahedron is formed by the O(1), O(2), C(11), and C(22) atoms, and the N(1) and N(2) atoms occupy the axial positions. The Ir(1) atom lies in the plane of the base, and the shift from the plane is only 0.007 Å. The dihedral angles between the 2-phenylenepyridine and pyrazolonate ligands lie in a narrow range of 85.13°–87.26°.

The Ir–C (1.986(3), 1.995(3) Å), Ir–N (2.028(2), 2.029(3) Å), and Ir–O (2.155(2), 2.168(2) Å) bond lengths in compound **I** are comparable with similar distances in complexes $Ir(Ppy)_2(L^1)$ and $Ir(Ppy)_2(L^2)$ (L^1 is 1-phenyl-3-methyl-4-benzoyl-5-pyrazolone, L^2 is 1-phenyl-3-methyl-4-phenylacetyl-5-pyrazolone): Ir–C 1.962(7)–1.988(6), Ir–N 2.023(5)–2.042(5), and Ir–O 2.167(4)–2.174(4) Å [4]. The O(1)–C(23) distance (1.273(4) Å) in compound **I** somewhat exceeds that in pyrazolone NBEpzH (1.253(2) Å) [12]. The dihedral angle between the plane of pyrazole ring **A** and phenyl substituent **B** (49.96°) noticeably exceeds a similar parameter in complexes $Ir(Ppy)_2(L^1)$ (39.92°), $Ir(Ppy)_2(L^2)$ (12.83°) [4] and in pyrazolone NBEpzH (11.28°) [12]. Probably, such a wide range in changing this parameter is due to distinctions in crystal packings of molecules of the iridium complexes. The 2-phenylenepyridine substituents in compound **I** are almost planar. The dihedral angles between the plane of the pyridine and phenylene fragments are 0.68° and 2.55°. According to [12, 21–23], this parameter is 0.98°–11.15° in the related iridium complexes.

It is known that the introduction of carbazole groups into the polymer emitters improves their charge-transport properties and electroluminescence characteristics [1, 24]. Therefore, the carbazole-containing comonomers (2-*endo* and 2-*exo*) were used for the synthesis of the iridium-containing copolymers.

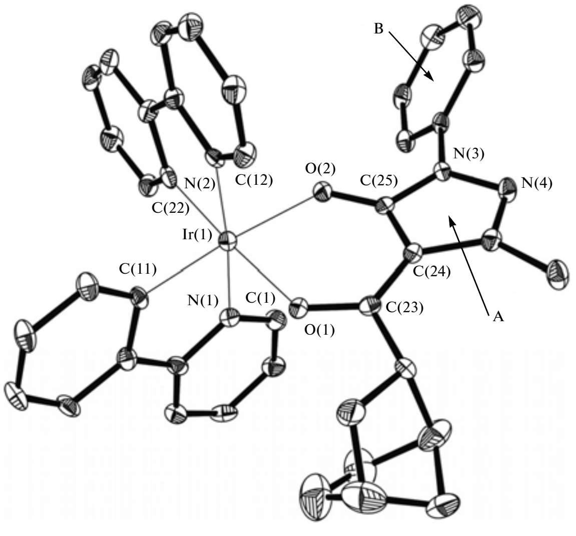
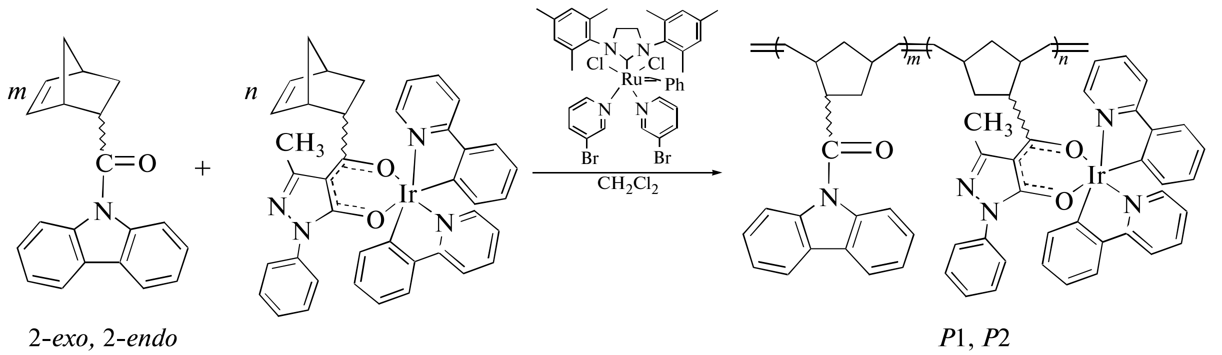


Fig. 1. Structure of complex **I**. Thermal ellipsoids are presented with 30% probability. Molecules of crystallization CH_2Cl_2 are omitted.

The copolymerization reactions occur in the presence of the Grubbs catalyst of the third generation at ambient temperature and afford iridium-containing copolymers *P*1 and *P*2



The initial ratio of the iridium- and carbazole-containing comonomers *m* : *n* was 8 : 1, and 1 mol % Grubbs catalyst with respect to the total amount of the comonomers introduced into the reaction was used. The reaction time was monitored by thin layer chromatography. The copolymerization of compound **I** and comonomer 2-*endo* occurs within 3 h, and the reaction involving more reactive comonomer 2-*exo* completes within 2 h. Copolymers *P*1 and *P*2 were isolated as stable in air yellow powders highly soluble in THF, CH_2Cl_2, and $CHCl_3$. Their molecular weight characteristics and the temperatures of glassy transition and decomposition are given in Table 3.

The study of the photophysical properties of the synthesized compounds showed that the absorption spectrum of complex **I** (Fig. 2, Table 4) was similar to the spectra of the known pyrazolonate iridium(III) complexes $Ir(Ppy)_2(L^1)$ and $Ir(Ppy)_2(L^2)$ [4].

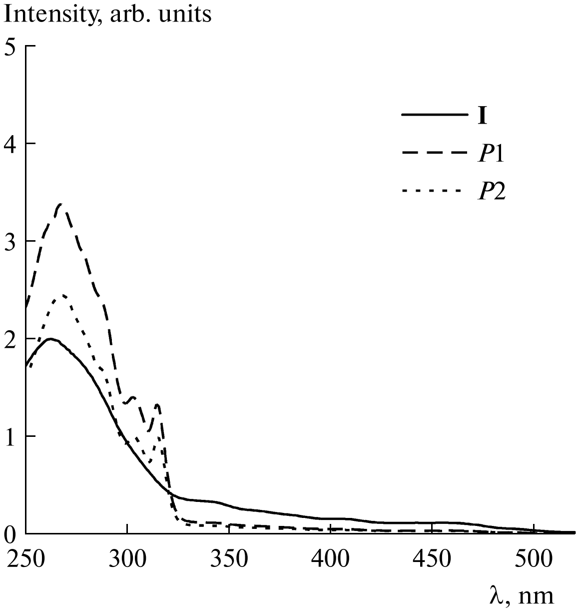


Fig. 2. Absorption spectra of complex **I** and copolymers *P*1 and *P*2 in CH_2Cl_2.

The intense broad band with a maximum at 262 nm is related to the π → π* transitions in the phenylenepyridine and pyrazolonate ligands. By analogy to [4, 25], the weak bands at 340–490 nm can be attributed to metal-to-ligand charge-transfer (MLCT) transitions mixed with ligand-to-ligand charge-transfer transitions. The absorption spectra of copolymers *P*1 and *P*2 contain the bands characteristic of complex **I** and additional intense bands at 300–340 nm caused by the π → π* transitions in the carbazole fragments.

The PL spectra of complex **I** and copolymers *P*1 and *P*2 are similar in both solutions and in thin film (Fig. 3, Table 4) and exhibit broad bands with maxima at 516–522 nm assigned to the MLCT transitions in the cyclometallated iridium complexes.

The absence of emission bands of the polymer matrix in the spectra of copolymers *P*1 and *P*2 indicates the efficient transfer of the excitation energy from the carbazole groups to the iridium-containing fragments following the Förster mechanism [26]. The quantum yield of complex **I** (4.1%) is noticeably lower than the quantum yields of copolymers *P*1 (6.5%) and *P*2 (8.4%). The higher yields of the copolymers are probably due to the fact that the iridium-containing fragments bound to the polymer chain are noticeably screened by the carbazole groups and, hence, their interaction with the solvent resulting in nonradiative losses is hindered.

The electroluminescence properties of copolymers *P*1 and *P*2 are presently under study. The preliminary results showed that the synthesized iridium-containing copolymers were efficient electroluminophores emitting the yellow-green light with the luminance exceeding 500 cd/m^2.

Thus, the new cyclometallated iridium(III) complex with the pyrazolonate ligand was synthesized, and the related copolymers with carbazole-containing units were obtained. All compounds were found to possess luminescence activity. The PL spectra of the iridium complex and copolymers contain intense

Table 1. Crystallographic data and the parameters of X-ray diffraction experiments and refinement for complex **I**

Parameter	Value
M	963.76
Temperature, K	100(2)
Crystal system	Monoclinic
Space group	$P2_1/c$
a, Å	13.1005(14)
b, Å	10.7279(11)
c, Å	28.138(3)
β, deg	90.771(3)
V, Å^3	3954.1(7)
Z	4
F(000)	1912
ρ_{calcd}, g/m^3	1.619
μ, mm^{-1}	3.688
Crystal size, mm	0.26 × 0.20 × 0.11
θ Range, deg	2.03–26.00
Ranges of reflection indices	$-16 \le h \le 16, -13 \le k \le 13, -34 \le l \le 34$
Number of reflections	33043
Number of independent reflections	7744
R_{int}	0.0814
Goodness-of-fit (F^2)	1.005
R_1, wR_2 ($I > 2\sigma(I)$)	0.0412, 0.0776
R_1, wR_2 (for all parameters)	0.0752, 0.0845
$\Delta\rho_{\max}/\Delta\rho_{\min}$, e Å^{-3}	1.474/–0.998

Table 2. Selected bond lengths (Å) and bond angles (deg) in complex **I**

Bond	d, Å	Angle	ω, deg
Ir(1)–O(1)	2.155(2)	C(22)Ir(1)C(11)	91.2(1)
Ir(1)–O(2)	2.168(2)	C(22)Ir(1)N(2)	81.2(1)
Ir(1)–N(1)	2.029(3)	C(11)Ir(1)N(2)	95.0(1)
Ir(1)–N(2)	2.028(2)	C(22)Ir(1)N(1)	95.2(1)
Ir(1)–C(11)	1.995(3)	C(11)Ir(1)N(1)	81.1(1)
Ir(1)–C(22)	1.986(3)	N(2)Ir(1)N(1)	174.7(1)
O(1)–C(23)	1.273(4)	C(22)Ir(1)O(1)	175.1(1)
O(2)–C(25)	1.273(4)	C(11)Ir(1)O(1)	90.7(1)
N(1)–C(1)	1.350(4)	N(2)Ir(1)O(1)	94.1(1)
N(1)–C(5)	1.374(4)	N(1)Ir(1)O(1)	89.6(1)
N(2)–C(12)	1.346(4)	C(22)Ir(1)O(2)	90.9(1)
N(2)–C(16)	1.381(4)	C(11)Ir(1)O(2)	174.3(1)
N(3)–C(25)	1.367(4)	N(2)Ir(1)O(2)	90.5(1)
N(3)–N(4)	1.388(4)	N(1)Ir(1)O(2)	93.4(1)
N(3)–C(34)	1.447(4)	O(1)Ir(1)O(2)	87.6(1)
N(4)–C(26)	1.327(4)	O(1)C(23)C(27)	114.9(3)
C(24)–C(26)	1.436(4)	O(2)C(25)N(3)	121.0(3)
C(32)–C(33)	1.314(4)	C(25)N(3)C(34)	126.7(3)
C(23)–C(24)	1.414(5)		
C(24)–C(25)	1.429(4)		

Table 3. Characteristics of copolymers $P1$ and $P2$

Copolymer	$\bar{M}_w \times 10^{-3}$	$\bar{M}_n \times 10^{-3}$	$\bar{M}_w/\bar{M}_n$	T_g, °C	T_d, °C
$P1$	21.5	12.2	1.8	183	336
$P2$	40.4	29.6	1.4	109	347

Table 4. Photophysical characteristics of complex **I** and copolymers *P*1 and *P*2

Compound	λ_{abs}, nm (logε)	λ_{em}, nm		Quantum yield in CH_2Cl_2, %
		film	in CH_2Cl_2	
I	262 (5.10), 340 (4.32), 365 (4.17), 380 (4.08), 405 (3.98), 455 (3.86), 490 (3.51)	529	516	4.1
*P*1	267 (5.35), 286 sh (5.20), 302 (4.96), 314 (4.95), 341 sh (3.87), 365 sh (3.72), 383 sh (3.60), 405 sh (3.49), 455 sh (3.33), 490 sh (2.86)	521	520	6.5
*P*2	267 (5.33), 287 sh (5.17), 303 (4.94), 315 (4.93), 343 sh (3.86), 367 sh (3.72), 384 sh (3.62), 407 sh (3.53), 457 sh (3.39), 490 sh (3.07)	522	520	8.4

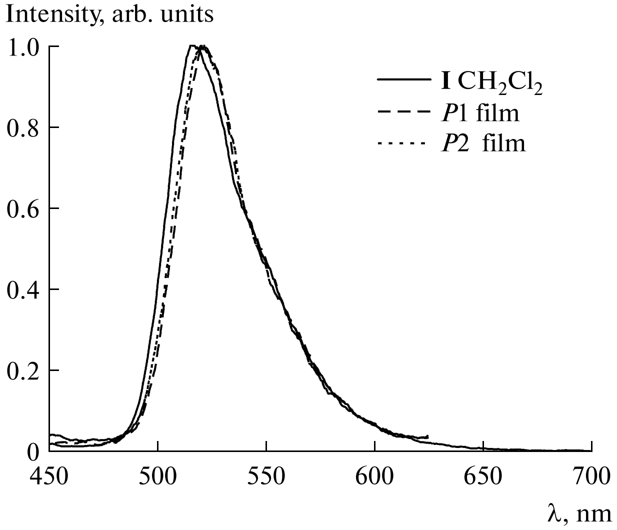


Fig. 3. PL spectra of complex **I** in a CH_2Cl_2 solution at ambient temperature (λ_{exc} = 360 nm) and copolymers *P*1 and *P*2 in thin films (λ_{exc} = 340 nm).

bands with maxima at 516–522 nm caused by the MLCT transitions in the iridium-containing fragments.

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