Copper-Containing Copolymers Based on the Norbornene Monomers. Synthesis, Photo-, and Electroluminescent Properties

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Abstract—New copper(I) complexes containing the norbornene-substituted phenanthroline ligand were synthesized. Based on these compounds, new carbon-chain copper-containing copolymers possessing the photo- and electroluminescent properties were obtained by the metathesis polymerization.

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To date, there is a significant progress on the development and practical application of organic lightemitting diodes (OLEDs) [1, 2]. However, studies aimed at improving the performance of OLED-devices permanently develop. One of the challenges in this area is creating new emitting materials.

Organic and metal-containing polymers are promising electroluminophores. The significant advantages of polymeric emitters compared with the lowmolecular analogs is that the nanolayers of polymer emitter can be obtained by the jet printing or spincoating, whereas the emission layers of the lowmolecular luminophores are produced by the expensive high vacuum evaporation. Furthermore, the use of the polymeric emitters makes possible to produce the OLED-devices of large area, which is almost unattainable with the low-molecular luminophores.

The polymers containing the luminescent metal complexes are of particular interest, since such light emitters can generate a variety of colors in almost the entire range of the visible spectrum.

Recently, the platinum and iridium-containing polymers have been synthesized and successfully used as emissive materials in organic light-emitting diodes [3–6].

It is known that a number of the low-molecular copper(I) complexes has effective electroluminescent properties [7–9]. Data on the electroluminescent copper-containing polymers are absent in the literature.

In this work we report on the synthesis of new copper(I) complexes with norbornene-substituted phenanthroline ligand and of the first electro-luminescent copper copolymers obtained from them by the ring-opening metathesis polymerization (ROMP).

The ROMP reactions are widely used for the preparation of polymers containing a variety of functional groups in the side chains, including the metal complex fragments [10, 11]. The same method we used for the synthesis of copper-containing polymer materials.

Recently, the synthesis and efficient photoluminescence properties of the copper(I) complex containing 1-ethyl-2-phenyl-1*H*-imidazo[4,5-*f*]-1,10-phenanthroline ligand has been reported [12]. To obtain the same norbornene-containing copper derivative, capable to act as a copper-containing monomer, we used two alternative ways, shown in Fig. 1.

1-(Bicyclo[2.2.1]hept-5-ene-2-pentyl)-2-phenyl-1Himidazo[4,5-f]-1,10-phenanthroline I was used as the starting reagent in both cases. In the first method, the initially obtained norbornene-substituted phenanthroline ligand II reacts with the acetonitrile copper(I) complex in the presence of triphenylphosphine or bis (2-diphenylphosphino)phenyl ether (DPEphos) to the form the desired copper monomers III and IV. In the second method in the initial stage the copper complexes containing ligand I were formed. Then the hydrogen atom in the imidazole ring of diimine ligand





 $L = 2 PPh_3$ (III), DPEphos (IV).

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 $L = 2 PPh_3$ (P1), DPEphos (P2).



bound to the copper is replaced by a norbornene moiety. As a result, complexes **III** and **IV** form.

Compounds **III** and **IV** were isolated as the airstable yellow solids. NMR spectroscopy revealed that the complexes are a mixture of *endo-* and *exo-*isomers (80:20).

9-{5-(Bicyclo[2.2.1]hept-5-en-2-yl-pentyl)}-9*H*-carbazole was used as a comonomer to obtain the coppercontaining copolymer, since the carbazole groups in the polymer emitters improve their charge-transporting properties and electroluminescent characteristics [2]. Copolymerization proceeds in the presence of Grubbs' third generation catalyst at room temperature and results in the target copper copolymers in high yields.

The ratio of carbazole- and copper-containing monomers n:m equals 18:1. The amount of catalyst was 1.0 mol% relative to the total amount of the monomers. According to the TLC monitoring, the copolymerizetion completed within 6 h. The isolated copolymers (Table 1) are air-stable yellow solids, which are well soluble in THF, CH₂Cl₂, and CHCl₃.

The study of the photophysical properties of the copolymers obtained showed that the electron

absorption spectra of the copper-containing monomers III, IV and polymer products P1, P2 (Fig. 1, Table 2) are similar and contain the absorption bands in the range of 250–325 nm attrubuted to the $\pi \rightarrow \pi^*$ -transitions in the aromatic systems of phosphine and substituted phenanthroline ligands. Similar to the spectra of known copper complexes with phenanthroline ligands [8, 12], the broad bands of lower intensity in the range of 325–400 nm may be assigned to the metal-to-ligand charge transfer (MLCT).

The photoluminescence spectra of monomers **III**, **IV** and copolymers P1, P2 (Fig. 2, Table 2) in thin films contain the broad bands at 520–560 nm related to the MLCT transitions. In the spectra of copolymers there are also the high energy excimers of carbazole groups (375–380 nm) [13], indicating the incomplete transfer of excitation energy from the polymer matrix to the luminophore copper complexes.

To study the electroluminescent properties of the synthesized copolymers, we fabricated the coppercontaining three-layer model OLED-devices of ITO/ Cu-copolymer/BATH/Alq₃/Yb configuration, in which a layer of the indium oxide doped tin oxide (ITO) served as the anode. The emissive layer consists of the

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Fig. 1. Absorption spectra of the complexes (a) **III**, **IV** and (b) copolymers P1, P2 in CH_2Cl_2 . (*1*) **III**, P1; (*2*) **IV**, P2.

copolymer P1 or P2. 4,7-Diphenyl-1,10-phenanthroline (BATH) and aluminum tris-(8-hydroxyquinolate) (Alq₃) were used as a hole-blocking and electron-transporting layers, respectively. The ytterbium metal layer served as the cathode. The electro-luminescence spectra of the copolymers P1 and P2, as well as the performance characteristics of the OLED-based devices are shown in Figs 3 and 4.

In the electroluminescence spectra there are the broad bands with maxima at 525 (P1) and 560 nm

Table 1. Molecular-mass characteristics and degradationtemperature of copolymers P1, P2

Copolymer	$ar{M}_{ m w}$	$ar{M}_n$	$ar{M}_{ m w}/ar{M}_n$	$T_{\rm d}$, °C ^a
P1	33460	20950	1.60	293
P2	23200	12170	1.91	338
P2	23200	12170	1.91	33

^a Temperature at 5% mass loss.



(P2), corresponding to the MLCT transitions in polymer copper complexes. The absence of emission of the polymer matrix evidences the effective transfer of the excitation energy from the carbazole fragments to the copper-containing luminescent centers by the Förster mechanism [14]. The emission band in the electroluminescence spectra of the copolymer P2 containing the DPEphos ligand at the copper atom is shifted to the long-wave region by 35 nm compared to the emission of the copolymer P1, containing the luminophore copper complexes with the PPh₃ ligands. A similar shift of the emission band was observed in the electroluminescence spectra of the low molecular weight copper complexes with PPh₃ and DPEphos ligands [7]. OLEDs based on the copolymers P1 and P2 showed a low brightness [8 cd m^{-2} at 19 V (P1) and 9 cd m^{-2} at 20 V (P2)]. This is probably due to the fact that the relaxation of the excited states of copper complexes in the electroluminescence occurs largely

Table 2. Photophysical characteristics of compounds III,IV, P1, and P2

Compound	λ_{max}^{abs} nm ($\epsilon \times 10^{-5}$ l mol ⁻¹ cm ⁻¹), CH ₂ Cl ₂	λ_{\max}^{em} , nm (film)
III	275 (0.80), 300 (sh, 0.33), 318 (sh, 0.10), 338 (0.21), 420 (sh, 0.008)	530
IV	280 (0.39), 302 (sh, 0.19), 318 (sh, 0.10), 337(0.12), 420 (sh, 0.02)	560
P1	267 (4.70), 284 (3.00), 296 (3.26), 319 (sh, 1.03), 335 (1.43), 347 (1.53)	375, 520
P2	265 (4.28), 285 (1.97), 296 (2.88), 320 (sh, 0.55), 333 (0.79), 347 (0.89)	375, 525

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Fig. 3. Electroluminescence spectra of ITO/Cu-copolymer/ BATH/Alq₃/Yb OLEDs at the maximal brightness.

by non-radiative way. The current-voltage and and brightness-voltage relations (Fig. 4) are typical for OLEDs.

with copper(I) complexes Thus. new the norbornene-containing phenanthroline ligands were synthesized. Based on these complexes, we obtained via the ROMP method previously unknown carbonchain copolymers containing in the side chains luminophore copper complexes and hole-transporting carbazole fragments. All the compounds are luminescent. In the photoluminescence spectra of the coppercontaining copolymers, there are the emission bands of carbazole groups excimers (375 nm) and the band at 520-525 nm corresponding to the MLCT transitions in the copper complexes. The electroluminescence spectra of copolymers contain only the bands (525 and 560 nm) of the MLCT transitions in the copper complexes. The maximal brightness of the nonoptimized OLED-devices based on the copper copolymers is 8-9 cd m⁻² at 19.5–20 V. Obviously, the polymer emitters can be improved by changing the ligand environment in the luminophore copper complexes, as well as by optimizing the ratio of the charge-transporting and luminescent units.

EXPERIMENTAL

All operations with the easily oxidizable and hydrolysable substances were carried out in a vacuum or under argon using standard Schlenk techniques. Ligand I [15, 16], [Cu(MeCN)₄]BF₄ [17], bicyclo [2.2.1]hept-5-en-2-ylpentylbromide [NBE(CH₂)₅Br] [18], 9-{5-(bicyclo[2.2.1]hept-5-en-2-ylpentyl)}-9*H*carbazole [NBE(CH₂)₅carb] [19], (H₂IMes)(3-Br-py)₂. (Cl)₂Ru=CHPh (Grubbs' catalyst of III generation)



Fig. 4. (a) The current-voltage and (b) the currentluminance characteristics of the OLEDs based on the (1) P1 and (2) P2 copolymers.

[20, 21] were synthesized as previously described. Triphenylphosphine (PPh₃), bis(2-diphenylphosphino)-phenyl ether (DPEphos), aluminum tris(8-hydroxy-quinolate) (Alq₃) and 4,7-diphenyl-1,10-phenan-throline (BATH), (Aldrich) were used without further purification.

The ¹H and ¹³C–{¹H} NMR spectra were obtained on a Bruker DPX-200 [200 MHz (¹H), 50 MHz (¹³C)] and Bruker Avance III-400 [400 MHz (¹H), 100 MHz (¹³C)] spectrometers, internal reference tetramethylsilane. The IR spectra were taken on a FSM 1201 FTIR spectrometer from KBr pellets (**II**, **III**, **IV**) or thin films between the KBr plates (copolymer samples). The electron absorption spectra were recorded on a Perkin Elmer Lambda 25 UV/Vis spectrometer. The photoluminescence spectra were obtained on a Perkin Elmer LS 55 photoluminescence spectrometer.

The molecular mass distribution of polymers was determined by the gel permeation chromatography (GPC) on a Knauer chromatograph equipped with a Smartline RID 2300 differential refractometer as a detector with a set of two Phenomenex columns filled with Phenogel sorbent (the pore size 10^4 and 10^5 Å, eluent THF, 2 ml min⁻¹, 40°C). The columns were calibrated using 13 polystyrene standards.

The uncorrected melting points of the monomers were determined in evacuated sealed capillaries. Thermal gravimetric analysis (TGA) of the copolymers was made by a Perkin Elmer PYRIS 6 TGA thermogravimeter in a stream of dry nitrogen (the flow rate $80 \text{ cm}^3 \text{ min}^{-1}$, the heating rate 5°C min⁻¹).

The electroluminescence spectra, current-voltage and voltage-brightness characteristics were obtained for the model OLED-devices without encapsulation using an automated PC-coupled complex, including a GW INSTEK PPE-3323 power supply, GW INSTEK GDM-8246 digital multimeter and Ocean Optics USB 2000 spectrofluorimeter.

1-{5-(Bicyclo[2.2.1]hept-5-en-2-ylpentyl)}-2-phenyl-1H-imidazo[4,5-f]-1,10-phenanthrolinobis(triphenylphosphine)copper tetrafluoroborate (III). a. To a solution of compound I (0.82 g, 2.8 mmol) in 30 ml of DMF was added in small portions 0.10 g of NaH (4.0 mmol). When the hydrogen evolution ceased, the reaction mixture was stirred for 2 h at 80°C. To the resulting solution was added NBE(CH₂)₅Br (0.81 g, 3.3 mmol). The mixture was stirred for 12 h at 80°C. After cooling to room temperature, the reaction mixture was poured into 100 ml of distilled water. The products were extracted with chloroform $(3 \times 50 \text{ ml})$. The combined extracts were washed with distilled water (3×50 ml) and dried with anhydrous magnesium sulfate. The solvent was removed by evaporation in a vacuum; the residue was recrystallized from ethanol. 1-{5-(Bicyclo[2.2.1]hept-5-en-2-ylpentyl)}-2-phenyl-1*H*-imidazo[4,5-*f*]-1,10-phenanthroline (II) was obtained in 44% (0.55 g) yield as the microcrystalline colorless substance, mp 176-178°C. IR spectrum, v, cm^{-1} : 3053 w (=C-H, C-H_{Ar}), 1562 w, 1550 w (C-N), 1513 w, 1400 w (C_{Ar}-C_{Ar}), 1260 w, 1180 w, 1158 w, 810 m, 777 w, 741 m, 702 m. ¹H NMR spectrum (CDCl₃), δ , ppm: 9.47 m (2H), 9.06 d.d (1H), 8.55 d.d (1H), 7.73-7.66 m (4H), 7.59-7.56 m (3H), 6.05 d.d (0.8 Hendo), 6.02 m (0.4 Hexo), 5.80 d.d (0.8 Hendo), 4.58

t (2H), 2.70 m (2H), 1.90 m (11H), 1.30–1.15 m (1H), 0.42–0.36 m (1H). ¹³C NMR spectrum (CDCl₃), δ_C , ppm: 153.96, 148.96, 147.70, 144.83, 144.25, 137.07, 136.78, 132.18, 130.43, 130.38, 129.92, 129.89, 128.93, 128.12, 124.80, 124.19, 123.53, 122.57, 120.13, 49.55, 46.84, 45.33, 42.45, 38.57, 34.39, 32.30, 30.13, 27.90, 26.49. Found, %: C 81.34; H 6.52. C₃₁H₃₀N₄. Calculated,%: C 81.22; H 6.55.

A mixture of [Cu(MeCN)₄]BF₄ (0.2 g, 0.6 mmol) and PPh₃ (0.34 g, 1.2 mmol) in 10 ml of CH₂Cl₂ was stirred for 1 h at room temperature. To the reaction mixture was added compound II (0.18 g, 0.6 mmol) in 10 ml of CH_2Cl_2 . The mixture was stirred for 2 h at room temperature. After the removal of solvent and volatile products the residue was washed with hexane and dried in a vacuum. The complex III was obtained (0.62 g, 86%) as a microcrystalline yellow solid, mp $202-205^{\circ}$ C. IR spectrum. v. cm⁻¹: 3050 m (=C-H. C-H_{Ar}), 1585 w, 1572 w (C-N), 1547 w, 1435 s, 1403 w (C_{Ar}-C_{Ar}), 1261 w, 1180 w, 1158 w, 810 w, 747 s, 694 s. ¹H NMR spectrum (CDCl₃), δ, ppm: 9.26 br. s (2H), 8.46 br. s (4H), 7.69–7.54 m (5H), 7.31 t (9H), 7.15 m (11H), 7.08 m (10H), 6.10 d.d (0.8 H_{endo}), 6.02 m (0.4 Hexo) 5.91 d.d (0.8 Hendo), 3.40 m (2H), 2.74 m (2H), 1.84 m (3H), 1.43–1.03 m (10H). ¹³C NMR spectrum (CDCl₃), δ_{C} , ppm: 145.07, 140.60, 137.00, 133.21, 132.32, 132.02, 131.58, 129.90, 129.02, 128.71, 128.07, 127.56, 123.51, 49.60, 45.54, 38.68, 36.37, 34.57, 34.00, 32.85, 32.43, 28.42, 27.28. Found, %: C 71.09; H 5.32. C₆₇H₆₀BCuF₄N₄P₂. Calculated, %: C 70.96; H 5.29.

b. A mixture of [Cu(MeCN)₄]BF₄ (0.14 g, 0.44 mmol) and PPh₃ (0.23 g, 0.88 mmol) in 10 ml of CH₂Cl₂ was stirred for 1 h at room temperature. To this solution was added compound I (0.13 g, 0.44 mmol) in 10 ml of CH₂Cl₂ and the mixture was stirred for 2 h at room temperature. The solvent and volatiles were removed by evaporation in a vacuum. To a solution of the resulting complex [(PPh₃)₂Cu(I)]BF₄ [22] in 10 ml of THF was added in small portions NaH (0.011 g, 0.44 mmol). The reaction mixture was stirred for 2 h at room temperature. Then to the resulting mixture was added NBE(CH₂)₅Br (0.11 g, 0.45 mmol) in 5 ml of THF. The mixture was stirred for 18 h at room temperature. The precipitated NaBr was separated by centrifugation. After the solvent removal, the residue was washed with hexane and dried in a vacuum. Yield 0.42 g (83%). The IR and NMR spectral data are identical to those of compound III obtained by the method mentioned above.

1-(5-Bicyclo[2.2.1]hept-5-en-2-ylpentyl)-2-phenyl-1H-imidazo[4,5-f]-1,10-phenanthrolino[bis(2-diphenylphosphino)phenyl ether|copper tetrafluoroborate (IV). a. Synthesis of compound IV was similar to that of the complex III. Yield 0.64 g (87%), microcrystalline yellow solid, mp 260-265°C (decomp.). IR spectrum, v, cm⁻¹: 3050 w (=C-H, C-H_{Ar}), 1586 w, 1572 w (C–N), 1547 w, 1477 w, 1402 w (C_{Ar}–C_{Ar}), 1308 w, 1260 m, 1183 m (C-H), 1158 w, 810 m, 744 m, 697 m. ¹H NMR spectrum (CDCl₃), δ , ppm: 9.17 d (2H), 8.57 d (2H), 8.34 d (2H), 7.45–7.39 m (5H), 7.07 t (14H), 7.00–6.94 m (14H), 6.10 d.d (0.8 H_{endo}), 6.02 m (0.4 Hexo) 5.91 d.d (0.8 Hendo), 3.74 (1H), 3.40 t (1H), 2.74 m (2H), 1.85 m (3H), 1.43-1.05 m (10H). 13 C NMR spectrum (CDCl₃), δ_{C} , ppm: 146.67, 137.00, 134.39, 133.23, 133.06, 132.90, 131.67, 131.50, 131.17, 130.83, 129.90, 128.64, 128.55, 128.45, 128.37, 126.95, 124.96, 123.29, 120.31, 49.59, 45.42, 42.54, 36.67, 34.57, 33.99, 32.42, 30.11, 28.42, 27.78, 25.63. Found, %: C 70.16; H 5.11. C₆₇H₅₈B· CuF₄N₄OP₂. Calculated, %: C 70.10; H 5.06.

b. A mixture of $[Cu(MeCN)_4]BF_4$ (0.14 g, 0.44 mmol) and DPEphos (0.24 g, 0.44 mmol) in 10 ml of CH₂Cl₂ was stirred for 1 h at room temperature. To this solution was added compound I (0.13 g, 0.44 mmol) in 10 ml of CH₂Cl₂, and the mixture was stirred for 2 h at room temperature. The solvent and volatiles were removed by evaporation in a vacuum. The resulting complex [(DPEphos)Cu(I)]BF₄ [23] was dissolved in 10 ml of THF. To this solution was added in small portions NaH (0.011 g, 0.44 mmol). The reaction mixture was stirred for 2 h at room temperature. Then, to the resulting solution was added NBE(CH₂)₅Br (0.11 g, 0.45 mmol) in 5 ml of THF, and the mixture was stirred for 18 h at room temperature. The precipitated NaBr was separated by centrifugation. After the solvent removal, the residue was washed with hexane and dried in a vacuum. Yield 0.43 g (85%). The IR and NMR spectral data are identical to those of compound IV obtained by the method mentioned above.

Synthesis of copolymers P1, P2. To a solution of the monomer III (0.04 g, 0.034 mmol) and NBE· $(CH_2)_5$ carb (0.2 g, 0.62 mmol) in 5 ml of CH_2Cl_2 was added a solution of the Grubbs' catalyst of III generation (0.006 g, 0.0067 mmol) in 1 ml of CH_2Cl_2 . The mixture was stirred at room temperature. The polymerization process was monitored by TLC. After the reaction completed (6 h), to the reaction mixture was added a few drops of ethyl vinyl ether to decompose the catalyst. The reaction mixture was stirred for an additional 30 min. The resulting polymer was precipitated with hexane followed by reprecipitation with hexane from CH₂Cl₂ and drying in a vacuum at room temperature to a constant weight. The copolymer P1 was obtained as a pale yellow solid in 92% yield (0.22 g). IR spectrum, v, cm⁻¹: 2930 s, 2855 m (C_{Ar}–H), 1627 w, 1598 m, 1482 s, 1452 s (C_{Ar}–C_{Ar}), 1346 m, 1325 s (C–N), 1263 m, 1231 m, 1154 m, 971 w, 748 s, 725 s, 618 w. ¹H NMR spectrum (CDCl₃), δ , ppm: 8.05 d (22H), 7.36–7.17 m (192H), 5.20–5.14 m (25H), 4.19–4.06 m (20H), 2.68 m (10H), 2.17 m (18H), 1.76 m (101H), 1.26–1.00 m (158H). Found, %: C 84.80; H 7.70. C₄₉₉H₅₄₆BCuF₄N₂₂P₂. Calculated, %: C 84.87; H 7.74.

Copolymerization of the monomer **IV** (0.04 g, 0.035 mmol) and NBE(CH₂)₅carb (0.2 g, 0.62 mmol) was carried out similarly at room temperature for 6 h. The copolymer P2 was obtained as a pale yellow solid in 95% yield (0.23 g). IR spectrum, v, cm⁻¹: 1482 s, 1461 s, 1452 s (C_{Ar}-C_{Ar}), 1346 m, 1325 s (C-N), 263 m, 1231 m, 1154 m, 971 w, 748 s, 725 s, 618 w. ¹H NMR spectrum (CDCl₃), δ , ppm: 8.05 d (24H), 7.35–7.17 m (171H), 5.23–5.17 m (29H), 4.18 m (24 H), 2.75 m (13H), 2.39 m (9H), 1.80–1.59 m (122H), 1.26–1.00 m (152H). Found, %: C 84.76; H 7.56. C₄₉₉H₅₄₄BCuF₄N₂₂OP₂. Calculated, %: C 84.71; H 7.69.

OLED-Devices fabrication. A glass ITO-coated (120 nm, 15 Ω cm⁻², Lum Tec) plate, which acts as the anode, was used as a support for the OLED-devices of ITO/Cu-copolymer (40 nm)/BATH (30 nm)/Alq₃ (30 nm)/Yb (150 nm) configuration. The emission layer of the copolymer was deposited from its CH₂Cl₂ solution (5 mg ml⁻¹) using a Spincoat G3-8 centrifuge $(3000 \text{ rev min}^{-1}, 30 \text{ s})$ and dried in a vacuum at 70°C for 3 h. The layer thickness was determined by a META-900 ellipsometer. A hole-blocking layer of BATH, the electron-transporting layer Alq₃ and a Yb layer (Aldrich), which plays a cathode role, was deposited by evaporation in a vacuum (10^{-6} mm Hg) from the heat-resisting evaporators. The layer thickness was monitored by a calibrated quartz resonator. The active area of the device is a circle with a diameter of 5 mm.

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