



Synthesis, structure and photophysical properties of complexes [1-(9-anthracylmethyl)-3-alkylimidazol-2-ylidene]₂Pt(C≡CPh)₂

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Abstract

1-(9-Anthracenylmethyl)-3-octylimidazolium chloride (**1a**) or 1-(9-anthracenylmethyl)-3-ethylimidazolium iodide (**1b**) was treated with (cod)Pt(C≡CPh)₂ to afford the luminescent complexes [1-(9-anthracylmethyl)-3-alkylimidazol-2-ylidene]₂Pt(C≡CPh)₂ (**2a**: alkyl=C₈H₁₇, **2b**: alkyl=C₂H₅). Complex **2a** crystallizes in triclinic system, space group *P* $\bar{1}$, *a* = 8.615(3), *b* = 13.839(5), *c* = 13.870(5) Å, α = 85.275(6)°, β = 84.062(6)°, γ = 72.901(6)°, *Z* = 1. The photophysical properties of **2** were determined.

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1. Introduction

In recent years the discovery of stable *N*-heterocyclic carbenes has provided exciting new possibilities for the chemistry of carbene complexes [1–3]. While some of these carbenes are isolable, less stable relatives may be generated and used in situ [4,5]. *N*-heterocyclic carbenes metal complexes are usually stable for heat, air, and moisture [6], and the introduction of carbene ligands to metal complexes can now be performed under simple and mild conditions. This attractive feature stimulates extensive interest, and many complexes of transition metals with *N*-heterocyclic carbenes have been synthesized [7,8]. *N*-heterocyclic carbenes resemble phosphine ligands [9], and their complexes with transition metals can act as catalysts and catalyst precursors for some important transformations. As a new effort we have attempted to investigate the photophysical properties of some *N*-heterocyclic carbenes complexes. Herein we wish to report the synthesis, structure and photophysical properties of complex [1-(9-anthracylmethyl)-3-alkylimidazol-2-ylidene]₂Pt(C≡CPh)₂ (**2**).

2. Experimental section

2.1. General comments

All manipulations were performed using Schlenk techniques, and solvents were purified by standard procedures. All the reagents for syntheses and analyses were of analytical grade and used without further purification. The (cod)PtCl₂ and (cod)Pt(C≡CPh)₂ was prepared according to the literature methods [10,11]. Melting points were determined with a Boetius Block apparatus. FI-IR spectra were measured on a Bruker FI-IR Equinox-55 infrared spectrophotometer. ¹H and ¹³C{¹H} NMR spectra were recorded on a Varian Mercury Vx 300 spectrometer at 300 MHz and 75 MHz, respectively. Chemical shifts, δ , are reported in ppm relative to the internal standard TMS for both ¹H and ¹³C NMR. Elemental analyses were measured using a Perkin-Elmer 2400C Elemental Analyzer. The luminescent spectra and UV-absorption spectra were conducted on Cary Eclipse Fluorescence spectrophotometer and Cary 100 Bio U-Visible spectrophotometer.

2.2. 1-(9-Anthracenylmethyl)-3-octylimidazolium chloride (**1a**)

A 1, 4-dioxane solution of imidazole (7.829 g, 0.12 mol) was added to a suspension of oil-free sodium hydride

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(3.036 g, 0.13 mol) in 1, 4-dioxane (150 ml) and stirred for 1 h at 90 °C. Then 1, 4-dioxane (100 ml) solution of octane bromide (22.210 g, 0.12 mol) was dropwise added to the solution. The mixture was continued to stir for 48 h at 90 °C and a yellow solution was obtained. The solvent was removed with a rotary evaporator and H₂O (500 ml) was added to the residue. Then the solution was extracted with CH₂Cl₂ (3 × 100 ml), the extracting solution was dried with anhydrous MgSO₄. After removing CH₂Cl₂, a yellow liquid 9-octylimidazole was obtained by distillation. Yield: 6.413 g (80%). bp: 125–127 °C. ¹H NMR (300 MHz, CDCl₃): 0.88 (t, *J* = 6.6, 3H, CH₃), 1.27 (m, 10H, 5 × CH₂), 1.78 (m, 2H, CH₂), 3.94 (t, *J* = 7.2, 2H, CH₂), 6.92 (s, 1H, 4-imiH) (imi: imidazole), 7.06 (s, 1H, 5-imiH), 7.55 (s, 1H, 2-imiH).

A solution of 9-octylimidazole (3.965 g, 0.02 mol) and 9-chloromethylanthracene (4.983 g, 0.02 mol) in 1, 4-dioxane (150 ml) was stirred for 3 days under refluxing, and a yellow precipitate was formed. The yellow product was filtered and washed with 1, 4-dioxane. The yellow crystals of 1-(9-anthracenylmethyl)-3-octylimidazolium chloride are obtained by recrystallization from CH₂Cl₂/acetone. yield: 8.169 g (91%). mp: 166–168 °C. Anal. Calcd for C₂₆H₃₁ClN₂: C, 76.73; H, 7.68; N, 6.88. Found: C, 76.34; H, 7.68; N, 6.62. ¹H NMR (300 MHz, CDCl₃): 0.87 (t, *J* = 6.0, 3H, CH₃), 1.25 (m, *J* = 6.0, 10H, CH₂), 1.85 (m, *J* = 6.0, 2H, CH₂), 4.29 (t, *J* = 6.0, 2H, CH₂), 6.68 (s, 2H, CH₂An) (An: anthracene), 6.94 (s, 1H, AnH), 7.00 (s, 1H, 4 or 5-imiH) (imi: imidazole), 7.57 (t, *J* = 8.0, 2H, AnH), 7.65 (t, *J* = 8.0, 2H, AnH), 8.10 (d, *J* = 8.0, 2H, AnH), 8.38 (d, *J* = 8.0, 2H, AnH), 8.61 (s, 1H, 4 or 5-imiH), 11.45 (s, 1H, 2-imiH). ¹³C NMR (75 MHz, CDCl₃): 14.1 (CH₃), 22.5 (CH₂), 26.2 (CH₂), 28.9 (CH₂), 29.0 (CH₂), 30.2 (CH₂), 31.6 (CH₂), 45.5 (CCH₂N), 50.2 (AnCH₂N), 121.1, 121.5, 122.1 and 122.8, (AnC or 4, 5-imiC), 125.6, 128.3, 129.5, 130.5, 130.9 and 131.2 (AnC), 137.5 (2-imiC).

2.3. [1-(9-Anthracylmethyl)-3-octylimidazol-2-ylidene]₂Pt(C≡CPh)₂ (**2a**)

A stirred CH₃CN and THF (30 ml) suspension of KOBu^t (0.090 g, 0.80 mmol), 1, 3-(9-anthracylmethyl)octylimidazolium chloride (0.200 g, 0.49 mmol) and (cod)Pt(C≡CPh)₂ (0.124 g, 0.25 mmol) was refluxed for 8 h, during which time a brown solution was formed. The solvents were removed in vacuo, and H₂O (30 ml) was added to the residue, then the solution was extracted with CH₂Cl₂ (3 × 20 ml), and the extracting solution was dried with anhydrous MgSO₄, then concentrated to 15 and 3 ml hexane was added, a pale yellow powder **2a** was formed. Yield: 0.163 g (54%). mp: 207–209 °C. Anal. Calcd for C₆₈H₇₀N₄Pt: C, 71.75; H, 6.20; N, 4.92. Found: C, 71.48; H, 6.11; N, 4.76. ¹H NMR (300 MHz, CDCl₃): 0.61 (t, *J* = 7.2, 2 × 3H, CH₃), 1.48 (m, *J* = 7.2, 2 × 10H, CH₂), 2.17 (m, 2 × 2H, CH₂), 4.61 (t, *J* = 7.2, 2 × 2H, CH₂), 6.23 (s, 2 × 2H, CH₂An), 6.69 (s, 2 × 1H, PhH), 7.15–7.46

(m, 2 × 10H, imiH, PhH or AnH), 8.06 (d, *J* = 8.0, 2 × 2H, AnH), 8.56 (d, *J* = 8.0, 2 × 2H, AnH), 8.79 (s, 2 × 1H, AnH). ¹³C NMR (75 MHz, CDCl₃): 14.1 (CH₃), 22.5 (CH₂), 22.7 (CH₂), 27.0 (CH₂), 29.4 (CH₂), 31.0 (CH₂), 31.9 (CH₂), 47.3 (NCH₂CH₂), 51.2 (NCH₂An), 79.6 (C≡CPh), 107.0 (Pt≡C), 118.7 and 119.6, 120.5, 122.2, 122.8, 123.3, 124.3, 124.8, 125.3, 127.2, 128.3, 128.9, 131.3, 138.2 (AnC, PhC or 4, 5-imiC), 172.0 (C_{carbene}). IR (KBr; disk): ν (C≡C) 2096 (m) cm⁻¹.

2.4. [1-(9-Anthracylmethyl)-3-ethylimidazol-2-ylidene]₂Pt(C≡CPh)₂ (**2b**)

This complex was prepared in a manner analogous to that for **2a**, only with 1-(9-anthracenylmethyl)-3-ethylimidazolium iodide (**1b**) instead of 1-(9-anthracenylmethyl)-3-octylimidazolium chloride (**1a**). The pale yellow powder of [1-(9-anthracylmethyl)-3-ethylimidazol-2-ylidene]₂Pt(C≡CPh)₂ (**2b**) was obtained by recrystallization from CH₂Cl₂/CH₃OH. Yield: 1.218 g (52%) mp: 297–299 °C (dec). Anal. Calcd for C₅₆H₄₆N₄Pt: C, 69.34; H, 4.78; N, 5.78. Found: C, 69.21; H, 4.52; N, 5.44. ¹H NMR (300 MHz, CDCl₃): 1.67 (t, *J* = 7.2, 2 × 3H, CH₃), 4.73 (q, *J* = 7.2, 2 × 2H, CH₂), 6.63 (s, 2 × 2H, CH₂An), 6.71 (s, 2 × 1H, PhH), 7.21–7.62 (m, 2 × 10H, imiH, PhH or AnH), 8.06 (d, *J* = 8.0, 2 × 2H, AnH), 8.45 (s, 2 × 1H, AnH), 8.80 (d, *J* = 8.0, 2 × 2H, AnH), ¹³C NMR (75 MHz, CDCl₃): 16.5 (CH₃), 45.9 (NCH₂CH₃), 57.5 (NCH₂An), 79.9 (C≡CPh), 112.9 (Pt≡C), 119.5 and 123.5, 124.8, 125.6, 126.4, 126.6, 127.6, 128.1, 128.5, 129.2, 131.5, 131.8 (AnC, PhC or 4, 5-imiC), 171.1 (C_{carbene}). IR (KBr; disk): ν (C≡C) 2095 (m) cm⁻¹.

3. Structure determination

For complex **2a**, a selected single crystal was mounted on a Bruker SMART 1000 CCD diffractometer operating at 50 kV and 20 mA using Mo K α radiation (0.71073 Å). Data collection and reduction were performed using the SMART and SAINT software [12] with frames of 0.6° oscillation in the θ range 1.8 < θ < 25°. An empirical absorption correction was applied using the SADABS program [13]. The structures were solved by direct methods and all non-hydrogen atoms were subjected to anisotropic refinement by full-matrix least squares on F^2 using the SHELXTL package [14]. All hydrogen atoms were generated geometrically (C–H bond lengths fixed at 0.96 Å), assigned appropriated isotropic thermal parameters and included in structure factor calculations. Selected bond lengths (Å) and angles (°) and crystal data and structure refinement for **2a** are presented in Tables 1 and 2, respectively.

Table 1
Selected bond lengths (Å) and angles (°) for **2a**

Bond lengths		Bond angles		Bond angles	
Pt(1)–C(27)	2.011(4)	C(27)#1–Pt(1)–C(27)	180.0(4)	C(2)–N(2)–C(19)	126.1(4)
Pt(1)–C(1)	2.023(4)	C(27)#1–Pt(1)–C(1)	89.5(1)	N(1)–C(1)–N(2)	104.8(3)
N(1)–C(1)	1.353(5)	C(27)–Pt(1)–C(1)	90.4(1)	N(1)–C(1)–Pt(1)	127.8(3)
N(1)–C(3)	1.381(6)	C(1)–Pt(1)–C(1)#1	180.0(3)	N(2)–C(1)–Pt(1)	127.4(3)
N(1)–C(4)	1.457(6)	C(1)–N(1)–C(3)	110.3(4)	C(3)–C(2)–N(2)	107.2(4)
N(2)–C(1)	1.354(5)	C(1)–N(1)–C(4)	124.3(3)		
N(2)–C(2)	1.382(6)	C(3)–N(1)–C(4)	125.2(4)		
N(2)–C(19)	1.454(6)	C(1)–N(2)–C(2)	110.3(4)		
C(2)–C(3)	1.321(7)	C(1)–N(2)–C(19)	123.4(3)		

4. Results and discussion

1-(9-Anthracenylmethyl)-3-octylimidazolium chloride (**1a**) was prepared from imidazole by stepwise alkylation with 1-bromooctane followed by 9-chloromethylantracene in sequence, and was obtained as yellow powder. 1-(9-Anthracenylmethyl)-3-ethylimidazolium iodide (**1b**) was prepared according to the method of literature [15,16].

In CH₃CN and THF solution 1-(9-anthracenylmethyl)-3-octylimidazolium chloride (**1a**) or 1-(9-anthracenylmethyl)-3-ethylimidazolium iodide (**1b**) was deprotonated

by the use of strong base KOBu^t to yield 1-(9-anthracenylmethyl)-3-alkylimidazol-2-ylidene, then it reacts with (cod)Pt(C≡CPh)₂ to afford the luminescent complexes [1-(9-anthracenylmethyl)-3-alkylimidazol-2-ylidene]₂Pt(C≡CPh)₂ (**2a**: alkyl=C₈H₁₇, **2b**: alkyl=C₂H₅) (Scheme 1).

The yellow crystals of **2a**·CH₂Cl₂ suitable for X-ray diffraction were grown by evaporating slowly its CH₂Cl₂/CH₃OH solution at room temperature. Molecular structure and the crystal cell structure of the complex **2a** are depicted in Figs. 1 and 2, respectively. In the asymmetrically substituted carbene complex **2a** the metal (II) center and two imidazole rings lie in a same plane. The platinum atom and two PhC≡C[−] lie in another plane and perpendicular to above mentioned plane. Pairs of 1-(9-anthracenylmethyl)-3-octylimidazol-2-ylidene and PhC≡C[−] ligands are in a *trans* configuration, and two 9-anthracenyl groups and two octyl groups point to contrary directions due to the sterically demanding to form *trans-anti* geometry. The Pt–C_{carbene} and Pt–C_{acetyl} bond distances are similar (Pt–C_{carbene} 2.023(4) Å, Pt–C_{acetyl} 2.011(4) Å). The internal ring angle (N–C–N) at the carbene center is 110.3°, which is similar to that in some known platinum complexes [17–20]. The dihedral angle between anthracene and imidazole ring plane is 81.5°, and the dihedral angle between benzene and imidazole ring plane are 103.3°.

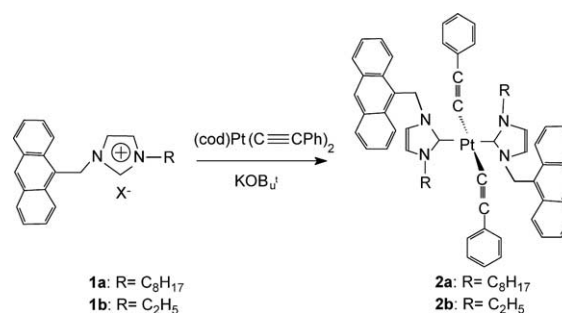
The ¹H NMR spectra of complexes **2** do not show the signal in 9–11.5 ppm, where the imidazolium C₂–H signals of precursors were found (9 ppm for **1a** and 11.45 ppm for **1b**). The C₄₍₅₎–H resonances of the imidazole ring show at

Table 2
Crystal data and structure refinement for **2a**

Empirical formula	C ₆₉ H ₇₂ Cl ₂ N ₄ Pt
Formula weight	1223.30
Crystal system	Triclinic
Space group	<i>P</i> $\bar{1}$
Unit cell dimension	
<i>A</i> (Å)	8.615(3)
<i>b</i> (Å)	13.839(5)
<i>c</i> (Å)	3.870(5)
α (°)	85.275(6)
β (°)	84.062(6)
γ (°)	72.901(6)
<i>V</i> (Å ³)	1569.7(10)
<i>Z</i>	1
<i>D</i> _{calc} (g cm ^{−3})	1.294
μ (mm ^{−1})	2.362
<i>F</i> (000)	626
<i>T</i> (K)	293(2)
λ (Å)	0.71073
Crystal size (mm)	0.40 × 0.22 × 0.20
2 θ Range (°)	2.52–26.41
Reflection collected	8732
Independent reflections	6205 [<i>R</i> _{int}] = 0.0281]
Refinement method	Full-matrix least-squares on <i>F</i> ²
Data/restraints/parameters	6205/24/385
Goodness-of-fit on <i>F</i> ^{2a}	1.013
Final <i>R</i> indices ^b [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> 1 = 0.0445, <i>wR</i> 2 = 0.0857
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0479, <i>wR</i> 2 = 0.0874
Largest difference, peak and hole (e Å ^{−3})	0.967 and −0.722

^a GOF = $[\sum \omega(F_o^2 - F_c^2)^2 / (n - p)]^{1/2}$, where *n* is the number of reflection and *p* is the number of parameters refined.

^b *R*1 = $\sum(|F_o| - |F_c|) / \sum|F_o|$; *wR*2 = $1/[\sigma^2(F_o^2) + (0.0691P) + 1.4100P]$ where *P* = $(F_o^2 + 2F_c^2)/3$.



Scheme 1.

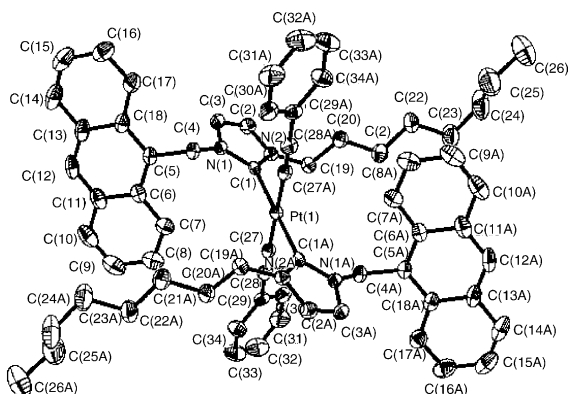


Fig. 1. Perspective view of **2a** and anisotropic displacement parameters depicting 30% probability. Hydrogen atoms have been omitted for clarity.

the low field, 7.1–7.5 ppm, indicating the substantial electron delocalization in the ring [21]. In the ^{13}C NMR spectra the signals for the carbene carbon of complexes **2** appear at 171–172 ppm, which is characteristic for a metal carbene signal [22,23]. In $\text{PhC}\equiv\text{C}_2$ unit the signals of carbons appear at ca. 80 ppm for C_1 and 110 ppm for C_2 , and the chemical shifts of C_2 are in lower field than those of C_1 , which may be ascribable to the withdrawing-electron effect of platinum atom. In the IR spectra the characteristic peaks of $\text{C}\equiv\text{C}$ are in ca. $2095\text{--}2096\text{ cm}^{-1}$. The complexes **2a** and **2b** are soluble in chloroform, and insoluble in diethyl ether and hydrocarbon solvents. The complexes are stable in air and moisture, and display high thermal stability.

The electronic absorption spectrum of **2a** (Fig. 3) shows two bands at ca. 315 and 340–400 nm. The former is the absorption spectrum of anthracene ring, and the latter is assigned to a metal-to-ligand charge transfer (MLCT) [$5d(\text{Pt}) \rightarrow \pi^*(\text{PhC}\equiv\text{C}^-)$] transition and the transition is likely to have some intraligand (IL) character [$\pi(\text{PhC}\equiv\text{C}^-) \rightarrow \pi^*(\text{PhC}\equiv\text{C}^-)$, ILCT] mixed into it [24]. The luminescent emission spectra of **1a** and **2a** are shown in Fig. 4 (the luminescent emission

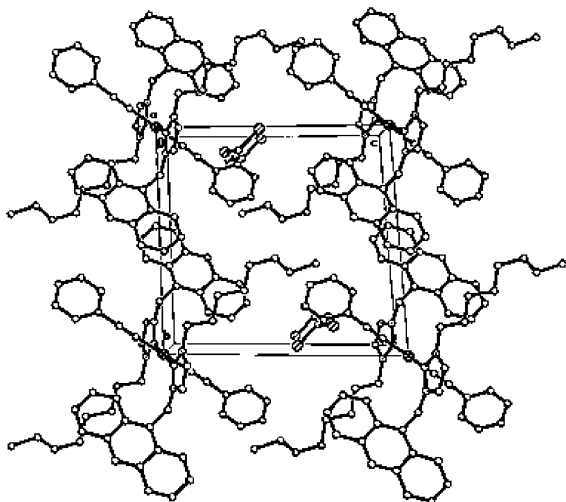


Fig. 2. Crystal cell structure of **a**.

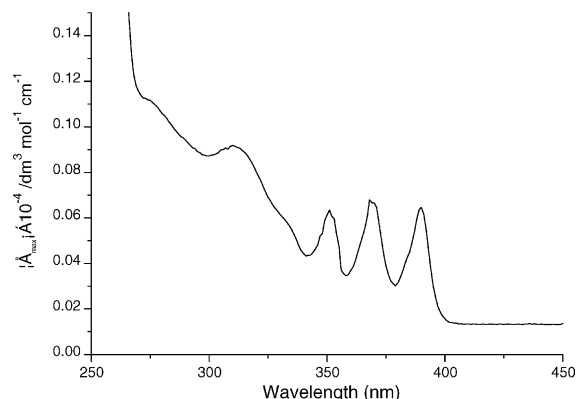


Fig. 3. UV-vis absorption spectrum of **2a** at 298 K in CH_2Cl_2 (5.0×10^{-6} M).

spectra of **1b** and **2b** are similar to those of **1a** and **2a**). In dichloromethane **2a** showed an anthracene-type fluorescence emission in 370–500 nm similar to but weaker than that of corresponding precursor **1a**. This can be attributed to transfer electrons of phenylacetylene through the platinum atom to the imidazole ring, which is helpful to the photoinduced electron transfer (PET) process of the lone-pair electron of the nitrogen atom to the anthracene ring [25].

Further investigation of new luminescent organometallic complexes from analogous precursors are being carried out. It is expected that the $\text{C}\equiv\text{C}$ units in the complexes might further coordinate with other metals, so that they can be become potential luminescent sensors for some metal ion.

5. Supplementary material

Crystallographic data for the structural analysis has been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 219731 for complex **2a**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ,

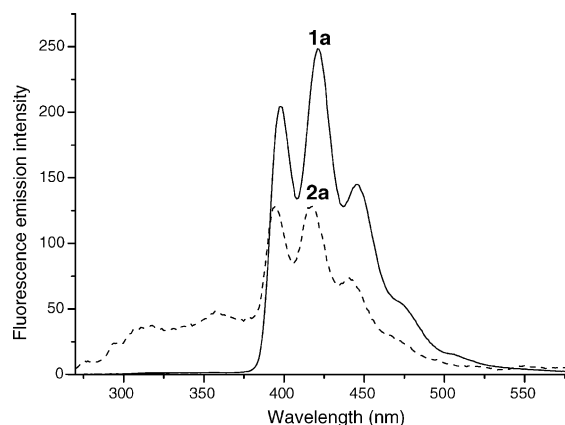


Fig. 4. Emission spectra of precursor **1a** (—) and complex **2a** (---) at 298 K upon excitation at 256 nm in CH_2Cl_2 (5.0×10^{-6} M).

UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>).

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