# Synthesis, characterization, thermal and luminescent properties of thiophenol-functionalized platinum(II) bis(acetylide) complexes

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#### Abstract

Two thiophenol-functionalized *trans*-platinum(II) bis(acetylide) complexes, having one thiophenol moiety in each alkenyl backbone with general formula *trans*-[(PPh<sub>3</sub>)<sub>2</sub>Pt{C $\equiv$ C-Ar-CH=CH(SC<sub>6</sub>H<sub>5</sub>)}<sub>2</sub>], (**2a**-**2b**), (Ar = phenylene or 2,5-dimeth-ylphenylene), were synthesized in good yields and good regioselectivities. Compared to the absorption bands of the *trans*-platinum(II) bis(acetylide) complexes **1**, the positions of the lowest energy absorption bands were redshifted by 17–18 nm, after functionalization with thiophenol. For both of the platinum(II) complexes **2**, the lowest energy absorption bands in chloroform solution at room temperature were observed in the range 376–379 nm, and under excitation at the absorption maximum both complexes showed emission maxima in the range of 408–419 nm. All the new Pt complexes have been characterized by physico-chemical and spectroscopic methods.

## Introduction

In the past two decades, there has been growing interest in the design of conjugated systems that can give rise to materials with interesting properties such as rich luminescence spectra [1, 2], non-linear optical (NLO) properties [3], liquid crystallinity [4], molecular wires [5] and photovoltaic behavior [6, 7] based on their electronic, optical and magnetic properties [2]. Incorporation of heavy metals into an organic conjugated framework can elicit large effects on the electronic and optical properties of the resulting materials [2]. In particular, conjugated materials and their metallated

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derivatives show a wide range of intriguing properties useful for the development of optoelectronic devices such as organic light-emitting diodes (OLEDs) [8], photovoltaic cells [9], and field-effect transistors [10].

There are many M–C=C bond-forming reactions that are useful for the construction of a wide variety of transition metal alkynyl conjugated systems [11, 12]. In particular, the platinum(II) bis-phosphine bis-alkynyl system, with its simple square planar geometry, has been widely explored [13–15]. Recently, we have reported the palladium catalyzed synthesis of novel sulfur-containing *trans*-platinum(II) bis(acetylide) complexes having two phenylthio moieties in each alkenyl backbone [16] and the photochemical synthesis of selenium-containing *trans*-platinum(II) bis(acetylide) complexes having two phenylseleno moieties in each alkenyl backbone [17]. Although the radical-mediated thiolation of terminal alkyl/arylacetylenes is well precedented in synthetic organic chemistry [18, 19], it has not been well reported with regard to transition metal complexes [20, 21].

In this report, our attention is focused on the photochemical synthesis, characterization, thermal, and optical absorption and emission properties of two new *trans*-platinum(II) bis(acetylide) complexes, *trans*-[(Ph<sub>3</sub>P)<sub>2</sub>Pt{C $\equiv$ C-Ar-CH = CH(SC<sub>6</sub>H<sub>5</sub>)}<sub>2</sub>], (2) (Ar = phenylene or 2,5-dimethylphenylene), containing one thiophenol moiety in each alkenyl backbone, which are stabilized by



the presence of monodentate auxiliary triphenylphosphine ligands.

# Experimental

#### **Materials and methods**

All chemicals, except where stated, were purchased from commercial sources and used without further purification. The compounds  $HC \equiv C - C_6 H_4 - C \equiv CH [22], HC \equiv C - C_6 H_2(p CH_3$ )<sub>2</sub>-C=CH [22], trans-[(Ph<sub>3</sub>P)<sub>2</sub>Pt{C=C-C<sub>6</sub>H<sub>4</sub>-C=CH}<sub>2</sub>] [11], and *trans*-[(Ph<sub>3</sub>P)<sub>2</sub>Pt{C $\equiv$ C-C<sub>6</sub>H<sub>2</sub>(*p*-CH<sub>3</sub>)<sub>2</sub>-C $\equiv$ CH}<sub>2</sub>] [11] were prepared by literature methods. NMR spectra were recorded on a Bruker 400 MHz FT NMR spectrometer in CDCl<sub>3</sub>. <sup>31</sup>P NMR spectra were referenced to external trimethylphosphite. <sup>1</sup>H NMR spectra were referenced to internal TMS, and <sup>13</sup>C NMR spectra were referenced to solvent resonances. Infrared spectra were recorded in the solid state (KBr pellets) on a Shimadzu IR Prestige 21 Fourier transform infrared spectrometer. ESI-HR mass spectra were recorded on a JMS-T100LC spectrometer. Microanalyses were obtained from BCSIR, Dhaka. UV-Vis spectra were recorded with a Shimadzu UV-1800 spectrometer. The photoluminescence (PL) spectra were recorded with a Shimadzu RF-3501pc spectrofluorometer. Thermal analysis (Thermogravimetric analysis, TGA) of the platinum(II) complexes was performed with a Shimadzu TGA-50 thermogravimetric analyzer under flowing nitrogen. Samples were heated at 10 °C/min in a platinum crucible. Column chromatography was carried out with silica gel.

### Synthesis of complex 2a

A mixture of *trans*-[(Ph<sub>3</sub>P)<sub>2</sub>Pt{C $\equiv$ C–C<sub>6</sub>H<sub>4</sub>–C $\equiv$ CH}<sub>2</sub>] (1a) (0.097 g, 0.1 mmol) and thiophenol (0.027 g, 0.25 mmol) in chloroform (2 mL) was degassed under a nitrogen atmosphere and placed within a sealed tube. The mixture was photoirradiated with a 500 W tungsten lamp for 3 h (cool water was passed over the sealed tube to maintain room temperature). The completion of the reaction was determined by TLC. The reaction product was evaporated to dryness, and the crude product was purified by column chromatography, eluting with hexane and ethyl acetate with a ratio 5:1 ( $R_f$  value = 0.69), to give the title complex (2a) as a yellow solid in 70% yield (0.083 g), E/Z ratio: 66/34. IR (solid state, KBr): 2106 (C=C)  $cm^{-1}$ , <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): (*E*/*Z* ratio: 66/34): δ 7.83–7.76 (*m*, 12H, PPh), 7.42–7.19 (m, 28H, PPh, SPh), 7.09 (d, 1.37 × 2H, Ar–H), 6.9 (*d*, 1.63×2H, Ar–H), 6.67 (*d*, 1.31×1H,  $J_{H-H} = 15.6$  Hz), 6.57 (*d*, 1.31 × 1H,  $J_{H-H} = 15.6$  Hz), 6.39  $(d, 0.69 \times 1\text{H}, J_{H-H} = 11.2 \text{ Hz}), 6.32-6.29 [(d, 0.69 \times 1\text{H}, 1.2 \text{ Hz})]$  $J_{H-H} = 10.8$  Hz) and  $(d, 1.37 \times 2H, Ar-H)$ ] and 6.2 (d, d, d) = 10.8 Hz)

1.63 × 2H, Ar–H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 121.11, 121.15, 124.05, 124.88, 126.63, 127.0, 127.41, 127.64, 127.75, 127.81, 127.86, 129.03, 129.07, 129.37, 129.87, 130.20, 130.73, 130.98, 131.11, 131.28, 131.57, 132.64, 132.67, 134.99, 135.05, 135.11, 135.67 and 136.44; <sup>31</sup>P NMR (160.25 MHz, CDCl<sub>3</sub>): δ 19.28 ( $J_{Pt-P}$  = 2643 Hz); ESI-HRMS (M + Na<sup>+</sup>) = 1213.2559 (100%), Calc. mass: 1190.2998, Anal. Calc. for C<sub>68</sub>H<sub>52</sub>P<sub>2</sub>PtS<sub>2</sub>: C, 68.6; H, 4.4%. Found: C, 68.2; H, 4.0%; UV–Vis:  $\lambda_{max}$  = 379 nm ( $\varepsilon$  = 79,119 L mol<sup>-1</sup> cm<sup>-1</sup>).

#### Synthesis of complex 2b

A mixture of  $trans - [(Ph_3P)_2Pt\{C \equiv C - C_6H_2(p - C_6H_2)\}$  $CH_{3}_{2}-C\equiv CH_{2}$  (1b) (0.104 g, 0.1 mmol) and thiophenol (0.027 g, 0.25 mmol) in chloroform (2 mL) was degassed under a nitrogen atmosphere and placed within a sealed tube. The resulting reaction mixture was photoirradiated under a 500 W tungsten lamp for 3 h, using a flow of cool water to maintain room temperature. The completion of the reaction was determined by TLC. The reaction product was evaporated to dryness. The crude product was purified by column chromatography, eluting with hexane and ethyl acetate with a ratio 5:1 ( $R_f$  value = 0.80), to give the title complex (2b) as a yellow solid in 73% yield (0.091 g), E/Z ratio: 65/35. IR (solid state, KBr): 2099 (C≡C) cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): (*E*/Z ratio: 65/35):  $\delta$  7.81–7.78 (*m*, 12H, PPh), 7.40-7.18 (m, 28H, PPh, SPh), 7.04 (0.70×1H, Ar-H),  $6.91 (1.30 \times 1H, Ar-H), 6.84 (d, 1.30 \times 1H, J_{H-H} = 14.8 \text{ Hz}),$ 6.60–6.52 [{2H} = {6.58 (d,  $1.30 \times 1$ H,  $J_{H-H}$  = 15.2 Hz), 6.54 (d,  $0.70 \times 1$ H,  $J_{H-H} = 10.8$  Hz)}], 6.35 (d,  $0.70 \times 1$ H,  $J_{H-H} = 10.4$  Hz), 5.94–5.89 (m, 2H, Ar–H), 2.04–2.02 (m, 6H, Ar-*p*-CH<sub>3</sub>) and 1.53–1.39 (*m*, 6H, Ar-*p*-CH<sub>3</sub>);  $^{13}$ C NMR (100 MHz, CDCl<sub>3</sub>): δ18.92, 19.11, 19.60, 19.74, 121.21, 121.27, 124.31, 124.89, 126.20, 126.40, 126.74 127.72, 127.77, 127.82, 128.15, 128.98, 129.67, 130.14, 130.92, 131.12, 131.17, 131.22, 131.57, 131.71, 131.84, 133.19, 133.55, 135.13, 135.66, 136.19 and 136.71; <sup>31</sup>P NMR (161.83 MHz, CDCl<sub>3</sub>):  $\delta$  19.90 ( $J_{Pt-P} = 2670$  Hz); ESI-HRMS  $(M + Na^+) = 1269.3041$  (100%), Calc. mass: 1246.4058, Anal. Calc. for C<sub>72</sub>H<sub>60</sub>P<sub>2</sub>PtS<sub>2</sub>: C, 69.3; H, 4.8%. Found: C, 69.7; H, 5.0%; UV–Vis:  $\lambda_{max} = 376 \text{ nm} (\epsilon = 70,847)$  $L \text{ mol}^{-1} \text{ cm}^{-1}$ ).

## **Results and discussion**

## Syntheses

When a mixture of thiophenol and the platinum(II) bis(acetylide) complex, *trans*- $(Ph_3P)_2Pt\{C\equiv C-C_6H_4-C\equiv CH\}_2$  (1a), in chloroform was photoirradiated [20] with a tungsten lamp, a new



Scheme 1 Photochemical addition of thiophenol to platinum(II) bis(alkynylarylalkynyl) complexes. R = H (a) or  $CH_3$  (b)

 Table 1
 Optimization of addition reaction of thiophenol with various platinum(II) acetylides

Entry	Pt-acetylide, 1	Isolated Yield <sup>a</sup> (%), <b>2</b> , ( <i>E</i> / <i>Z</i> )
1	$\begin{array}{c} \textit{trans-}(Ph_3P)_2Pt\{C\equiv C-C_6H_4-\\C\equiv CH\}_2\ (\textbf{1a}) \end{array}$	70 ( <b>2a</b> ) (66/34)
2	trans- $(Ph_3P)_2Pt\{C\equiv C-C_6H_2(CH_3)_2-C\equiv CH\}_2$ (1b)	73 ( <b>2b</b> ) (65/35)

Reactions were carried out in chloroform solvent for 3 h under photoirradiation using *trans*-(Ph<sub>3</sub>P)<sub>2</sub>Pt{C $\equiv$ C–Ar–C $\equiv$ CH}<sub>2</sub> (0.1 mmol) 1 and benzenethiol (0.25 mmol)

<sup>a</sup>Inseparable mixtures of *E/Z* isomers were estimated by <sup>1</sup>H NMR

addition product (**2a**), a sulfur-containing platinum(II) bis(acetylide), was afforded in good yield (Scheme 1 and Table 1, entry 1). Its structural formula was deduced by physico-chemical and spectroscopic methods. Similarly, the reaction of thiophenol and platinum(II) complex, *trans*-(Ph<sub>3</sub>P)<sub>2</sub>Pt{C=C-C<sub>6</sub>H<sub>2</sub>(CH<sub>3</sub>)-C=CH}<sub>2</sub> (**1b**), in chloroform gave the addition product **2b** in 73% yield (Table 1; entry 2). *E/Z* isomers were identified according to the coupling constants of the vinylic protons [20]. The *E* isomer is formed predominantly over the *Z* isomer, probably owing to minimization of steric repulsion.

Both complexes **2** were isolated as yellow solids, after purification by silica column chromatography. They display good solubility in common organic solvents, but are insoluble in hexane. For comparison, the triphenyl phosphinebased platinum(II) starting materials **1** are poorly soluble in common organic solvents. These newly synthesized *trans*platinum(II) complexes were characterized by their IR, <sup>1</sup>H and  ${}^{31}P{H}$  NMR spectra and positive ESI-HR [M + Na]<sup>+</sup> mass spectra.

#### Characterization

The IR spectra of these platinum(II) bis(acetylide) complexes **2** display a single sharp  $\nu$ (C=C) absorption band in the range of 2099–2106 cm<sup>-1</sup>, confirming that the platinumacetylenic carbon bond (PtC=C) is conserved (Table 2) [16]. The IR spectra of complexes **2** showed no bands in the range of 3200–3300 cm<sup>-1</sup>, which is the characteristic region for the =C-H stretching vibration [20]. Thus, it is revealed that the terminal acetylenic groups have reacted with thiophenol. Hence, the IR spectra of complexes **2** confirm that the addition reaction has proceeded only at the terminal alkyne bond of the platinum(II) bis(alkynylarylalkynyl) complexes **1**.

In the <sup>1</sup>HNMR spectra, a new peak is observed for both complexes 2 in the range of 6.32–6.84 ppm, which is characteristic of vinyl protons (one vinyl proton is overlapped with the aromatic ring of the organic spacer). Meanwhile, complexes 2 did not show a signal from the terminal acetylenic proton (about 3 ppm) [18]. However, signals from the organic spacers, phenylthio moieties and phenyl phosphine protons were all observed in the expected regions. The <sup>31</sup>P{H}NMR spectra each showed a singlet along with two satellites due to coupling with <sup>195</sup>Pt, characteristic of a -Pt-(PR<sub>3</sub>)<sub>2</sub>-unit in trans geometry [21]. For complexes 2a and **2b**, the  $J_{Pt-P}$  values of 2643 and 2670 Hz, respectively, are in agreement with those previously reported for other square planar platinum(II) complexes with trans geometry [20, 23]; cis coupling constants are generally much larger (about 3500 Hz) [23]. The inseparable mixtures of E/Z isomers

 Table 2
 Selected spectroscopic data for the trans-platinum(II) complexes 2

Complex	IR (KBr) $v(C\equiv C)$ $(cm^{-1})$	<sup>1</sup> H-NMR (ppm) <sup>a</sup> [-CH=CH(SPh)], vinyl proton ( <i>E</i> / <i>Z</i> )	<sup>31</sup> P-NMR (ppm) <sup>b</sup>	ESI-HR mass $(M + Na^+)$ (Calc.) $m/z$
2a	2106	6.67 (d, 15.6 <sup>c</sup> ), 6.57 (d, 15.6 <sup>c</sup> ), 6.39 (d, 11.2 <sup>c</sup> ), 6.32 (d, 10.8 <sup>c</sup> ), (66/34)	19.28 (2643) <sup>d</sup>	1213.2559 (1190.2998)
2b	2099	6.84 (d, 14.8 <sup>c</sup> ), 6.58 (d, 15.2 <sup>c</sup> ), 6.53 (d, 10.8 <sup>c</sup> ), 6.35 (d, 10.4 <sup>c</sup> ), (65:35)	19.90 (2670) <sup>d</sup>	1269.3041 (1246.4058)

<sup>a</sup>Referenced to internal TMS

<sup>b</sup>Referenced to external trimethylphosphite

 $^{\rm c}J_{H\!-\!H}$ 

 ${}^{d}J_{Pt-P}$  value (in Hz) is given in parentheses

each displayed a sharp singlet in the <sup>31</sup>P{H}NMR, since the (*E/Z*), geometry is only associated with the terminal alkene, which is remote from the phosphine ligands. The complexes **2** showed intense molecular ion  $[M+Na]^+$  peaks in the positive ion ESI-HR mass spectra, observed at *m/z* 1213.2559 for **2a**, and at *m/z* 1269.3041 for **2b**. The stereochemistry of the *E/Z* isomers was assigned on the basis of the coupling constant values of the vinylic protons in the <sup>1</sup>H NMR spectra (Table 2); for instance, *trans*-platinum(II) complex **2a** showed coupling constants of the vinylic protons of 15.6 and 15.6 Hz for *E*, and 11.2 and 10.8 Hz for *Z* isomers [20].

#### **Optical absorption and emission properties**

The UV/Vis spectra of both complexes were recorded. In each case, the lowest energy absorption band in chloroform solution at room temperature was observed in the range of 376–379 nm. The values of  $\lambda_{\text{max}}$  are reported in Table 3. The absorption bands are slightly influenced by the presence of different organic spacers. The lowest energy band, in each case, was tentatively assigned to a predominantly  $\pi(C \equiv C) \rightarrow \pi^*(C \equiv C)$  transition by comparison with related systems [24–27], along with  $\pi(C=C) \rightarrow \pi^*(C=C)$  transitions on the alkenylaryl moiety [17, 28] but can be considered to have some LMCT character resulting from the possible admixture of platinum (n+1) p orbitals and a ligand  $\pi^*$ orbital [29]; their positions move to longer wavelengths due to coordination of acetylide ligands. Both complexes showed large extinction coefficients, suggestive of the presence of more extended  $\pi$ -conjugated systems through the sulfur bridge [30]. The UV/Vis absorption maxima of complexes 1a and 1b are observed at 361 and 359 nm, respectively, while those of their corresponding thiolated complexes 2a and 2b are observed at 379 and 376 nm, respectively (Table 3). In each case, a small redshift is observed, amounting to 18 and 17 nm for complexes 2a and 2b, respectively. This reveals that  $\pi$ -conjugation is extended through the metal site by mixing of the frontier orbitals of the metal and the ligand. A butyl phosphine *trans*-platinum(II) alkenylarylalkynyl complex, trans-[(PBu<sub>3</sub>)<sub>2</sub>Pt{C=C-C<sub>6</sub>H<sub>4</sub>-CH=CH(Ph)}<sub>2</sub>], having one phenyl ring in each alkenyl backbone, was reported by Schanze [28], and its UV/Vis absorption maximum was observed at 370 nm. Hence, compared to trans- $[(PBu_3)_2Pt{C \equiv C - C_6H_4 - CH = CH(Ph)}_2]$ , the sulfur-containing trans-platinum(II) complexes 2a and 2b are redshifted by 9 and 6 nm, respectively. Ethyl phosphine complexes, trans-[(Et<sub>3</sub>P)<sub>2</sub>Pt{C=C-C<sub>6</sub>H<sub>4</sub>-CH=CH(SC<sub>6</sub>H<sub>5</sub>)}<sub>2</sub>] and trans- $[(Et_3P)_2Pt\{C \equiv C - C_6H_2(CH_3)_2 - CH = CH(SC_6H_5)\}_2]$ , and trans-[(Et<sub>3</sub>P)<sub>2</sub>Pt{C=C-C<sub>6</sub>H<sub>4</sub>-CH=CH(SC<sub>6</sub>H<sub>4</sub>CH<sub>3</sub>-p)}<sub>2</sub>] and trans-[(Et<sub>3</sub>P)<sub>2</sub>Pt{C=C-C<sub>6</sub>H<sub>2</sub>(CH<sub>3</sub>)<sub>2</sub>-CH=CH(SC<sub>6</sub>H<sub>4</sub>CH<sub>3</sub>p) $_2$ ], were reported by Rahman [18, 19] and showed UV/ Vis absorption maxima at 366 and 362 [20], and 366 and 362 nm [21], respectively. Preliminary investigations of the luminescence properties of complexes 2a and 2b were also performed. The photoluminescence spectra recorded for the solutions of the complexes under excitation at the absorption maxima ( $\lambda_{max} = 376 - 379$  nm) showed emission maxima in the region 408–419 nm (Table 3; Figs. 1, 2). The room temperature photoluminescence spectra in chloroform of transplatinum(II) complexes 2a and 2b displayed emission bands in the blue region. This feature is attributed to emission from a singlet excited state (fluorescence) because of the small energy shift (Stokes shift) [16, 20, 31].

#### **Thermal stabilities**

The thermal properties of complexes 2 were examined by TGA under a nitrogen atmosphere. Analysis of the TG traces (heating rate 10 °C/min) for both complexes shows that they



Fig. 1 UV/Vis absorption and photoluminescence spectra of complex 2a at room temperature in chloroform solution

Table 3UV/Vis andphotoluminescence data forcomplexes, in CHCl3, at roomtemperature

Complexes <sup>a</sup>	$\lambda_{\max}$ (nm) Absorption	Complexes <sup>a</sup>	$\lambda_{\max}$ (nm) Absorption	$\varepsilon (L \text{ mol}^{-1} \text{ cm}^{-1})$	$\lambda_{\max}$ (nm) emission	Stokes shift (nm)
1a	361	2a	379	79,119	408	29
1b	359	2b	376	70,847	419	43

<sup>a</sup>The concentration is  $1 \times 10^{-5}$  M



Fig. 2 UV/Vis absorption and photoluminescence spectra of complex 2b at room temperature in chloroform solution

complexes <b>2a–2b</b> under TGA	complex	loss (%)	perature (°C)
	2a	78	356
	2b	84	427

PPh<sub>3</sub> groups plus two [{C=C-Ar-CH=CH(SC<sub>6</sub>H<sub>5</sub>)}<sub>2</sub>] groups

exhibit good thermal stabilities. Decomposition onsets at ca. 356 °C for **2a**, and 427 °C for **2b** were observed.

The peak decomposition temperature was defined as the first inflection point in the thermogravimetric curve, corresponding to a peak in the derivative of the TG data. The decomposition exotherms are sharp with single peaks. We have previously reported on ethyl phosphine containing platinum(II) bis(acetylides), whose decomposition exotherms are sharp with multiple peaks [21], but in the present case, we observed sharp single peaks. The decomposition step corresponds to the removal of two triphenylphosphine groups plus two thiolated acetylide, [{C=C-Ar-CH=CH(SC<sub>6</sub>H<sub>5</sub>)}<sub>2</sub>], groups from the platinum(II) bis(acetylides) complexes (Table 4) [32].

## Conclusions

We have synthesized two new platinum(II) bis(acetylide) complexes containing phenylthio moieties in their alkenyl backbones by the insertion of thiophenol into the terminal carbon–carbon triple bond of platinum(II) bis(acetylide) complexes under photoirradiation. Both of these platinum(II) complexes were isolated as yellow solids with good yields. Both complexes show emission bands in the blue region of the electromagnetic spectra and have good thermal stabilities. We are continuing to investigate various arene-containing extended alkynes of platinum(II) diacetylide complexes as reactants for the functionalization with substituted arylthiols, and the optical properties of the resulting products.

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