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# A systematic identification of efficiency enrichment between thiazole and benzothiazole based yellow iridium(III) complexes

Cite this: DOI: 10.1039/x0xx00000x

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Received 00th January 2012,  
Accepted 00th January 2012

DOI: 10.1039/x0xx00000x

www.rsc.org/

Four cyclometalated heteroleptic Ir(III) complexes [(Et-Cz-BTz)<sub>2</sub>Ir(pic)], [(Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O)], [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic)], and [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O)] containing benzothiazole linked ethylcarbazole (Et-Cz-BTz) as a main ligand has been successfully synthesized for solution-processed phosphorescent organic light-emitting diodes (PhOLEDs). All the Ir(III) complexes emit bright yellow (541–582 nm) phosphorescence at room temperature. Among the four Ir(III) complexes, the [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic)] showed the best luminous efficiency of 60 cd/A and external quantum efficiency (EQE) of 19% with CIE coordinates (0.467, 0.524), which is one of the best efficiencies for solution-processed yellow PhOLEDs using heteroleptic Ir(III) complexes as an emitting layer. Interestingly, upon replacing the thiazole linking group in the main ligand by benzothiazole, the EQE was increased from 6.08 to 19%.

## 1. Introduction

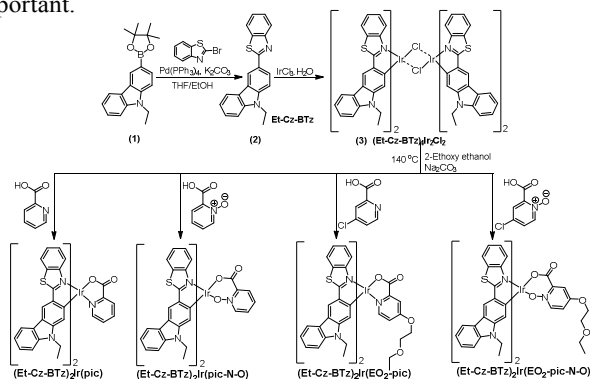
Phosphorescence organic light-emitting diodes (PhOLEDs) based on heavy metal centred phosphorescent emitters have currently paid more attention due to their potential applications in full color flat panel displays and solid state lighting.<sup>1–4</sup> Particularly, these kind of emitters in the emitting layer (EML) can harvest both singlet and triplet electro-generated excitons, which leads to increase of the internal quantum efficiency as high as the theoretical value of 100%.<sup>5–8</sup> Particularly, the emitters based on Ir(III) complexes have become the most successful phosphorescent candidates in recent days owing to their favourable electrochemical and photophysical properties, flexible color tunability, short triplet state lifetimes, and the ability to participate in/outer sphere electron transfer reactions.<sup>9–11</sup> Even though various colors of phosphorescent Ir(III) complexes can be prepared by varying the structure of the ligands, the yellow emitting Ir(III) complexes have attracted more research interest because of their utility in the fabrication of two emitting component white OLEDs with blue emitters.<sup>12</sup>

Upon considering the efficiency point of view, the achieved maximum efficiencies of solution-processed yellow PhOLEDs are not high as like other primary color materials (Blue, Green, and Red ~20%). Recently, Bryce et al. have been reported the systematic study of substituent effects of Ir(III) complexes containing carbazole (Cz)-pyridine based main ligands for the solution-processed OLEDs, which were fabricated using poly(N-vinylcarbazole) as a high triplet energy host material and as a hole transport material, and achieved the highest external quantum efficiency (EQE) of 12%.<sup>10</sup> Further, we introduced ethylcarbazole-thiazole (Et-Cz-Tz) based main

ligands in the Ir(III) complexes and studied their efficiency in solution-processed PhOLEDs using m-MTDATA:TPBI as host materials; among the Ir(III) complexes, the captain Ir(III) complex shows the maximum EQE of 6.08%.<sup>13</sup> Additionally, the introduction of a benzothiazole (BTz) based framework into the ligands of Ir(III) complexes leads efficient yellow emission.<sup>11</sup> Even though various electron donating/withdrawing groups (OMe, Me, CF<sub>3</sub>, F, CN, etc.) or a more extended chromophore are introduced either in the phenyl ring or to the BTz moiety, the phosphorescence of the resultant Ir(III) complexes is mainly localized within a narrow color range around yellow with few exceptions.<sup>14–16</sup> Very recently, Li et al. have reported Ir(III) complexes with Cz-BTz based ligand systems for vacuum-deposited PhOLEDs and observed the EQE of 23%.<sup>12</sup>

Even though the PhOLEDs fabricated using thermal high vacuum deposition techniques show better device performance, the solution process techniques, such as spin coating and inkjet printing, have more advantages than the former because of their complicated fabrication process and limited utilization of the expensive materials.<sup>17–19</sup> Thus, much attention has been given in the area of solution-processed PhOLEDs to design and preparation of suitable small molecules and optimization of device architectures. In order to fabricate the solution-processed PhOLEDs, the Ir(III) complexes are prepared using oligomeric or polymeric ligand systems,<sup>20,21</sup> or dendrimers.<sup>22,23</sup> Upon introduction of these macromolecular systems into the Ir(III) complexes, the purity of resultant material was decreased. Additionally, multi-step reaction sequences are required to attain the macromolecular ligands. Thus, the solution-processed counterparts, which combine the advantageous features of both

polymeric and small molecule materials, have been developed. Under this circumstance, the developments of solution process heavy metal centred phosphorescent emitters are of great important.



**Scheme 1.** Synthetic route for  $(\text{Et-Cz-BTz})_2\text{Ir}(\text{pic})$ ,  $(\text{Et-Cz-BTz})_2\text{Ir}(\text{pic-N-O})$ ,  $(\text{Et-Cz-BTz})_2\text{Ir}(\text{EO}_2\text{-pic})$ , and  $(\text{Et-Cz-BTz})_2\text{Ir}(\text{EO}_2\text{-pic-N-O})$ .

In this report, in order to give further validation of the above discussions, we glued the BTz to N-ethylcarbazole (Et-Cz-BTz) as a main ligand in Ir(III) complexes for solution-processed PhOLEDs, where the solubilizing ethoxyethanol group was introduced into the ancillary ligands by a tandem reaction approach.<sup>13</sup> Here, we synthesized four cyclometalated heteroleptic Ir(III) complexes using Et-Cz-BTz as a main ligand and picolinic acid [ $(\text{Et-Cz-BTz})_2\text{Ir}(\text{pic})$ ], picolinic acid N-oxide [ $(\text{Et-Cz-BTz})_2\text{Ir}(\text{pic-N-O})$ ], 4-(2-ethoxyethoxy)picolinic acid [ $(\text{Et-Cz-BTz})_2\text{Ir}(\text{EO}_2\text{-pic})$ ], and 4-(2-ethoxyethoxy)picolinic acid N-oxide [ $(\text{Et-Cz-BTz})_2\text{Ir}(\text{EO}_2\text{-pic-N-O})$ ] as an ancillary ligands. All the Ir(III) complexes show bright yellow emission (541–582 nm) at room temperature. The photophysical, electrochemical properties, and the performance of solution-processed PhOLEDs of these Ir(III) complexes were investigated systematically.

## 2. Experimental

### 2.1. General Information

All chemicals and reagents were purchased from Aldrich Chemical Co., and it was used without further purification. The THF was purified by appropriate procedures and the other solvents were used as received. 3-Bromo-9-ethyl-9H-carbazole, 9-ethyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-9H-carbazole (1) and 4-chloropicolinic acid N-oxide were synthesized using our previously reported procedures. All reactions were systematically monitored by thin layer chromatography with Merck pre-coated aluminum plates. <sup>1</sup>H- and <sup>13</sup>C- NMR spectra were recorded on a Varian Mercury Plus 300 MHz spectrometer in CDCl<sub>3</sub> using tetramethylsilane as an internal standard. High resolution mass spectra were obtained from the Korea Basic Science and Institute, Daegu Centre (HR-ESI Mass). TGA and DSC thermograms were obtained with Mettler Toledo TGA/SDTA 851e and DSC 822e analyser under an N<sub>2</sub> atmosphere at a heating rate of 10 °C/min, respectively. The UV-visible absorption and the fluorescence spectra were recorded with a JASCO V-570 and Hitachi F-4500 fluorescence spectrophotometers at room temperature, respectively. CV measurements were performed with a CHI 600C potentiostat (CH Instruments), which is equipped with a platinum disc as

the working electrode, a platinum wire as the counter electrode, and a Ag/AgCl as the reference electrode, at a scan rate of 100 mVs<sup>-1</sup> using anhydrous chloroform and 0.1 M tetrabutylammonium tetrafluoroborate (TBABF<sub>4</sub>) as the solvent and electrolyte, respectively. The potentials were referenced to the ferrocene/ferrocenium redox couple (Fc/Fc<sup>+</sup>). It was assumed that the redox potential of Fc/Fc<sup>+</sup> had an absolute energy level of -4.8 eV under vacuum. All electrochemical experiments were carried out at room temperature.

### 2.2. Synthesis of 2-(9-ethyl-9H-carbazol-3-yl)benzothiazole (Et-Cz-BTz) (2)

2-Bromobenzothiazole (6.66 g, 31.13 mmol), K<sub>2</sub>CO<sub>3</sub> (12.89 g, 93.39 mmol), and tetrakis(triphenylphosphine)palladium (1.79 g, 1.56 mmol) were added to a solution of 9-ethyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-9H-carbazole (1) (10 g, 31.13 mmol) in 100 mL of THF under N<sub>2</sub> atmosphere. The reaction mixture was heated to 65 °C for 15 h. After completion of the reaction, the solution was cooled to room temperature, poured into water and extracted with EA. The organic layer was washed with water, brine solution, and dried over anhydrous MgSO<sub>4</sub>. The solvent was removed under reduced pressure and the crude product was purified by column chromatography on silica gel using EA/hexane (2:8 v/v) as an eluent to give a 2-(9-ethyl-9H-carbazol-3-yl)benzothiazole (Et-Cz-BTz) (6.2 g, 62%) as a white solid. m.p. 142 °C. <sup>1</sup>H- NMR (300 MHz, CDCl<sub>3</sub>): δ (ppm): 8.86 (s, 1H), 8.22–8.19 (dd, 2H), 8.09 (d, 1H), 7.92 (d, 1H), 7.55–7.28 (m, 6H), 4.44–4.37 (q, 2H), 1.48 (t, 3H). <sup>13</sup>C- NMR (CDCl<sub>3</sub>): δ 169.6, 154.6, 141.8, 140.8, 135.2, 126.6, 126.4, 125.8, 124.9, 124.8, 123.7, 123.3, 122.9, 121.7, 121.1, 120.3, 119.9, 109.1, 108.9, 38.0, 14.1.

### 2.3. General procedure for the synthesis of Ir(III) complexes

All the Ir(III) complexes were synthesized by applying the similar procedure reported by our group.<sup>13,24–26</sup> The general procedure for the synthesis of Ir(III) complexes is as follows: the compound 2 (6 g, 18.26 mmol) and IrCl<sub>3</sub>·3H<sub>2</sub>O (2.18 g, 7.31 mmol) were added to a mixture of 2-ethoxyethanol and water (80 mL, 3:1 v/v). The resulting mixture was refluxed at 140 °C for 20 h under an N<sub>2</sub> atmosphere and cooled to room temperature. The formed yellow solid was filtered, washed with water followed by methanol. Subsequently, the solid was dried in a vacuum oven at 120 °C to afford cyclometalated Ir(III) μ-chloride bridged dimer (3) as a yellow solid (4.8 g, 60 %). A solution of 3 (1 g, 0.57 mmol), 5 equivalents of ancillary ligand, and Na<sub>2</sub>CO<sub>3</sub> (0.60 g, 5.7 mmol) in 2-ethoxyethanol (20 mL) was refluxed under N<sub>2</sub> for 12 h. After cooling to room temperature, the mixture was poured into water and extracted with MC. The organic layer was dried over anhydrous MgSO<sub>4</sub>, filtered and removed the solvent under reduced pressure. The crude product was purified by silica gel column chromatography using EA:MC:hexane (2:4:4, v/v/v) as an eluent. In the case of  $(\text{Et-Cz-BTz})_2\text{Ir}(\text{EO}_2\text{-pic})$  and  $(\text{Et-Cz-BTz})_2\text{Ir}(\text{EO}_2\text{-pic-N-O})$ , the chlorine substituent in 4-chloropicolinic acid and 4-chloropicolinic acid N-oxide was replaced by ethoxyethanol by tandem reaction during the synthesis of Ir(III) complexes.

### 2.4. Bis[2-(9-ethyl-9H-carbazol-3-yl)benzothiazole]iridium picolinic acid $(\text{Et-Cz-BTz})_2\text{Ir}(\text{pic})$

Yield: 0.31 g, 56 %, m.p. 331 °C. <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>): δ (ppm): 8.57 (d, 1H), 8.43 (d, 2H), 8.21 (d, 1H), 8.05-7.97 (m, 2H), 7.88-7.82 (m, 4H), 7.47 (t, 1H), 7.42-7.30 (m, 4H), 7.27-7.08 (m, 5H), 6.94 (t, 1H), 6.51 (s, 1H), 6.21-6.15 (m, 2H), 3.82-3.68 (m, 4H), 0.96 (t, 3H), 0.84 (t, 3H). <sup>13</sup>C-NMR (CDCl<sub>3</sub>): δ (ppm) 182.3, 179.8, 173.3, 153.6, 150.5, 148.9, 147.0, 145.9, 143.3, 143.0, 139.9, 137.9, 133.0, 132.4, 131.7, 131.0, 128.3, 128.0, 126.9, 125.3, 125.2, 125.0, 124.4, 124.0, 123.8, 123.1, 122.1, 121.2, 119.8, 119.5, 119.4, 119.3, 119.2, 118.7, 118.5, 113.1, 112.9, 108.8, 108.5, 100.1, 37.4, 37.2, 13.1, 12; Anal. Calcd. for C<sub>48</sub>H<sub>34</sub>IrN<sub>5</sub>O<sub>3</sub>S<sub>2</sub>: C, 59.49; H, 3.54; N, 7.23; S, 6.62. Found: C, 59.27; H, 3.49; N, 7.04; S, 6.36. HRESI-MS [M+H]<sup>+</sup>: m/z calcd for 969.1996, found 969.1964.

### 2.5. Bis[2-(9-ethyl-9H-carbazol-3-yl)benzothiazole]iridium picolinic acid N-oxide (Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O)

Yield: 0.32 g, 57 %, m.p. 325 °C. <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>): δ (ppm) 8.57 (d, 1H), 8.43 (d, 2H), 8.21 (d, 1H), 8.05-7.97 (m, 2H), 7.89-7.82 (m, 4H), 7.47 (t, 1H), 7.41-7.28 (m, 5H), 7.25-7.09 (m, 4H), 6.94 (t, 1H), 6.51 (s, 1H), 6.22-6.15 (m, 2H), 3.82-3.68 (m, 4H), 0.95 (t, 3H), 0.84 (t, 3H). <sup>13</sup>C-NMR (CDCl<sub>3</sub>): δ (ppm) 182.3, 179.8, 173.3, 153.6, 150.6, 150.5, 148.9, 147.1, 146.0, 143.3, 143.0, 140.0, 137.9, 133.0, 132.5, 131.7, 131.0, 128.3, 128.0, 126.9, 125.3, 125.2, 125.0, 124.4, 123.9, 123.8, 123.1, 122.2, 121.2, 119.8, 119.5, 119.3, 119.2, 118.7, 118.5, 113.1, 112.9, 108.8, 108.5, 37.4, 37.2, 13.1, 12.9; Anal. Calcd. for C<sub>48</sub>H<sub>34</sub>IrN<sub>5</sub>O<sub>3</sub>S<sub>2</sub>: C, 58.52; H, 3.48; N, 7.11; S, 6.51. Found: C, 58.72; H, 3.48; N, 7.21; S, 6.25. HRESI-MS [M+H]<sup>+</sup>: m/z calcd for 985.1687, found 985.1643.

### 2.6. Bis[2-(9-ethyl-9H-carbazol-3-yl)benzothiazole]iridium-4-(2-ethoxyethoxy)picolinic acid (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic)

Yield: 0.26 g, 43 %, m.p. 282 °C. <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>): δ (ppm) 8.54-8.38 (m, 3H), 8.04-7.94 (m, 2H), 7.90-7.83 (m, 2H), 7.73-7.70 (m, 1H), 7.60-7.55 (m, 1H), 7.46-7.28 (m, 5H), 7.25-7.08 (m, 3H), 7.06-7.01 (m, 2H), 6.86-6.85 (m, 1H), 6.50 (s, 1H), 6.36 (t, 1H), 6.24 (s, 1H), 4.20-4.17 (m, 2H), 3.80-3.65 (m, 6H), 3.54 (q, 2H), 1.19 (t, 3H), 0.95 (t, 3H), 0.84 (t, 3H). <sup>13</sup>C-NMR (CDCl<sub>3</sub>): δ (ppm) 182.3, 179.6, 173.3, 166.5, 155.3, 150.6, 149.1, 147.4, 146.1, 143.3, 143.0, 139.9, 133.2, 132.5, 131.6, 131.0, 128.2, 127.1, 125.3, 125.1, 124.9, 124.4, 124.0, 123.8, 123.0, 122.2, 121.2, 119.7, 119.5, 119.3, 119.2, 119.1, 118.6, 117.9, 116.4, 113.1, 113.0, 112.5, 108.8, 108.5, 68.5, 68.3, 67.1, 37.4, 37.2, 15.2, 13.1, 12.9; Anal. calcd for C<sub>52</sub>H<sub>42</sub>IrN<sub>5</sub>O<sub>4</sub>S<sub>2</sub>: C, 59.07; H, 4.00; N, 6.62; S, 6.07. Found: C, 58.34; H, 3.99; N, 6.28; S, 5.91. HRESI-MS [M+H]<sup>+</sup>: m/z calcd for 1057.2754, found 1057.2723.

### 2.7. Bis[2-(9-ethyl-9H-carbazol-3-yl)benzothiazole]iridium-4-(2-ethoxyethoxy)picolinic acid N-oxide (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O)

Yield: 0.30 g, 49 %, m.p. 277 °C. <sup>1</sup>H-NMR (300 MHz, CDCl<sub>3</sub>): δ (ppm) 8.52 (d, 1H), 8.43 (d, 2H), 8.04-7.97 (m, 2H), 7.91-7.87 (m, 2H), 7.73 (d, 1H), 7.60 (d, 1H), 7.45 (t, 1H), 7.41-7.28 (m, 4H), 7.20-7.11 (m, 4H), 7.08-7.04 (m, 1H), 6.89-6.86 (m, 1H), 6.50 (s, 1H), 6.38 (d, 1H), 6.23 (s, 1H), 4.21-4.18 (m, 2H), 3.80-3.68 (m, 6H), 3.54 (q, 2H), 1.20 (t, 3H), 0.95 (t, 3H), 0.84 (t, 3H). <sup>13</sup>C-NMR (CDCl<sub>3</sub>): δ (ppm) 182.3, 179.6, 173.3, 166.5, 166.5, 155.3, 150.5, 149.1, 147.4, 146.1, 143.3, 143.1, 139.9, 133.2, 132.5, 131.6, 131.0, 128.2, 127.1, 125.3, 124.9, 124.4, 124.0, 123.8, 123.0, 122.2, 121.2, 119.7, 119.5, 119.3, 119.2,

119.1, 118.6, 117.9, 116.4, 113.1, 113.0, 112.5, 108.8, 108.5, 68.3, 67.1, 15.2, 13.1, 12.9; Anal. Calcd. For C<sub>52</sub>H<sub>42</sub>IrN<sub>5</sub>O<sub>5</sub>S<sub>2</sub>: C, 58.19; H, 3.94; N, 6.53; S, 5.98. Found: C, 58.36; H, 4.11; N, 6.54; S, 5.81. HRESI-MS [M+H]<sup>+</sup>: m/z calcd for 1073.2386, found 1073.2372.

## 2.8. Device Fabrication and Characterization

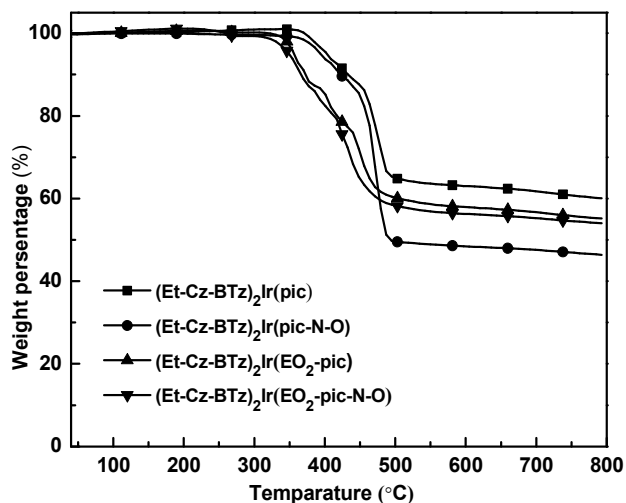
A patterned indium tin oxide (ITO) glass substrate (10 Ω sq<sup>-1</sup>) was UV-ozone treated for 20 min. A high WF GraHIL was spin-cast onto the substrate and immediately baked in air at 150 °C for 30 min, giving a 40 nm thick film. The EML solutions composed of each dopant and TCTA:TPBI (mixed host) (15:100 by wt.) dissolved in tetrahydrofuran:CF co-solvent (8:2 by wt.) or tetrahydrofuran solvent were spin coated on top of the GraHIL in a glove box and baked to give the ~40 nm film. Then, TPBI (50 nm), LiF (1 nm), and Al (90 nm) were thermally deposited under high vacuum (< 5x10<sup>-7</sup> Torr). The current density-voltage-luminance characteristics were measured using a Keithley 236 power source measurement unit and a Minolta CS-2000 Spectroradiometer.

## 3. Results and discussion

The solution-processed yellow PhOLEDs have garnered much attention in the recent years due to their easy process, cost effective, and large area lighting applications. For this purpose, four Ir(III) complexes, (Et-Cz-BTz)<sub>2</sub>Ir(pic), (Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O), (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic), and (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O) were synthesized according to the synthetic route as shown in Scheme 1. The boronic ester derivative of Cz (1) was prepared according to our previous report with quantitative yields. Then, it was reacted with 2-bromobenzothiazole by Suzuki coupling to get the desired donor-acceptor type of 2-(9-ethyl-9H-carbazol-3-yl)benzothiazole (Et-Cz-BTz) based ligand with 62% yield. The resultant Et-Cz-BTz was reacted with iridium trichloride hydrate (IrCl<sub>3</sub>·H<sub>2</sub>O) to get the cyclometalated Ir(III) μ-chloride bridged dimer [(Et-Cz-BTz)<sub>4</sub>Ir<sub>2</sub>Cl<sub>2</sub>] with quantitative yield. It can be easily converted to heteroleptic Ir(III) complexes, (Et-Cz-BTz)<sub>2</sub>Ir(pic), (Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O), (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic), and (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O) by replacing two bridge chlorides with bidentate monoanionic ancillary ligands such as picolinic acid (pic), picolinic acid N-oxide (pic-N-O), 4-chloropicolinic acid, and 4-chloropicolinic acid N-oxide with good yield, respectively. To enhance the solubility of the Ir(III) complexes, the ethoxyethanol group was introduced into (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic) and (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O) through the tandem reaction, which has been applied for Ir(III) complexes in our previous report for the first time.<sup>13</sup> It can reduce the synthetic steps with good yields. Here, the reaction of 4-chloropicolinic acid or 4-chloropicolinic acid N-oxide and cyclometalated Ir(III) μ-chloride bridged dimer, and replacement of chlorine in 4-chloropicolinic acid or 4-chloropicolinic acid N-oxide by the ethoxyethanol group were occurring simultaneously in one step. All the Ir(III) complexes are readily soluble in common organic solvents such as methylene chloride (MC), chloroform (CF), and ethylacetate (EA), etc. The chemical structure of the Ir(III) complexes were confirmed by the <sup>1</sup>H-, <sup>13</sup>C-NMR and HR-ESI mass spectral techniques.

The thermal stability of Ir(III) complexes was identified by thermo gravimetric analysis (TGA) and differential scanning calorimeter (DSC) analysis under N<sub>2</sub> atmosphere at a heating

rate of 10 °C/min and the corresponding TGA thermograms are shown in Fig. 1. The thermal decomposition temperatures ( $T_d$ ) at 5% weight loss of (Et-Cz-BTz)<sub>2</sub>Ir(pic), (Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O), (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic), and (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O) were 403, 394, 356, and 349 °C, respectively, revealing its high thermal stability, which is more favourable for long term stability of OLEDs.

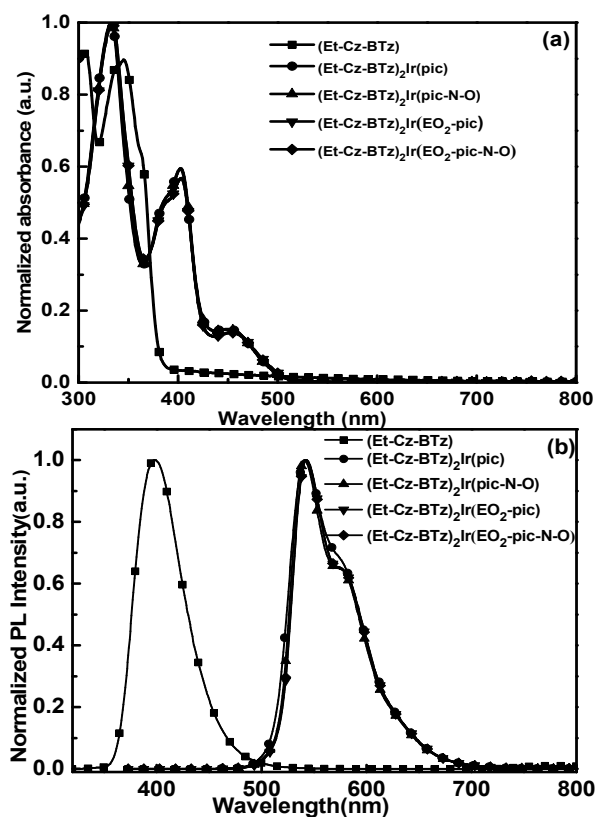


**Fig 1.** TGA of (Et-Cz-BTz)<sub>2</sub>Ir(pic), (Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O), (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic) and (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O) measured at a scan rate of 10°C/min under N<sub>2</sub> atmosphere.

Among the Ir(III) complexes, the pic containing Ir(III) complexes shows more thermal stability than the pic-N-O ancillary ligand. On one hand, the ethoxyethanol solubilizing group linked Ir(III) complexes, (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic) and (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O), shows less  $T_d$  values than their counterparts, but on the other hand, it increases the solubility of the Ir(III) complexes, which aids to fabricate the PhOLEDs under solution-processed techniques. The DSC thermograms of all Ir(III) complexes show the glass transition temperatures ( $T_g$ ), which were in the range of 161–247 °C, without any crystalline and melting peaks. It suggests that all synthesized Ir(III) complexes will not be easily distorted under high temperatures that produced during the operation of PhOLEDs.

The photophysical properties of main ligand, (Et-Cz-BTz) and their Ir(III) complexes, (Et-Cz-BTz)<sub>2</sub>Ir(pic), (Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O), (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic), and (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O) were measured using UV–visible absorption and photoluminescence (PL) spectra at room temperature in CF solution. Fig. 2a shows the UV–visible absorption spectra of main ligand, (Et-Cz-BTz) and their Ir(III) complexes in CF solution. The Et-Cz-BTz shows intense absorption between 320–375 nm with a peak at 345 nm attributed to the spin-allowed <sup>1</sup>π–π\* transitions of Cz and BTz moieties. All the Ir(III) complexes show three absorption maxima in three different regions as shown in Fig. 2a. The absorption bands in higher energy region below 360 nm are corresponding to the spin-allowed <sup>1</sup>π–π\* transitions of the cyclometalated ligands, which closely resembles to the absorption spectrum of the main ligand. The weak absorption bands between 370–500 nm are likely due to metal-to-ligand charge-transitions (<sup>1</sup>MLCT and <sup>3</sup>MLCT) and the spin-orbit coupling enhanced <sup>3</sup>π–π\* states. The absorption pattern of all

Ir(III) complexes are almost similar to each other, indicating that the introduction of ethoxyethanol group on the 4-position of the ancillary ligands does not induce much effect on the absorption spectra.



**Fig 2.** (a) UV–visible absorption and (b) PL spectra of (Et-Cz-BTz)<sub>2</sub>Ir(pic), (Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O), (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic), and (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O) in Chloroform at 25°C.

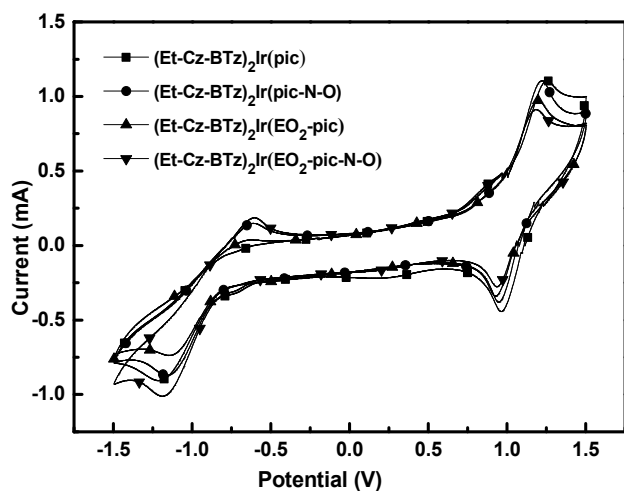
The PL spectra of the ligand and their Ir(III) complexes in CF solution are shown in Fig. 2b. All the Ir(III) complexes show very similar PL pattern with a maxima at 541 nm and a shoulder at 582 nm irrespective of nature of substituent on the ancillary ligand. It indicates that the presence of an ethoxyethanol solubilizing group in ancillary ligands will increase only the solubility of the Ir(III) complexes rather than any of the photophysical properties. When compared to the PL maxima of Tz-based Ir(III) complexes,<sup>13</sup> the BTz-based Ir(III) complexes show only 5 nm red shift in the maxima in CF solution. Under photoexcitation at its absorption maximum, all Ir(III) complexes emit yellow light in PL spectra (Fig. 2b). In comparison to Et-Cz-BTz, which emits deep blue fluorescence at 398 nm, all the Ir(III) complexes display a strong red shifted PL band ( $\lambda_{em}$ : ~ 541–543 nm) at room temperature upon excitation at 386 nm, which is associated predominantly with a ligand-centred <sup>3</sup>π–π\* excited state.

To investigate the charge carrier injection properties of the Ir(III) complexes, the highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) were determined by using cyclic voltammetry (CV). The cyclic voltammogram of all the Ir(III) complexes are shown in Fig. 3. All Ir(III) complexes display reversible redox waves over both the anodic and cathodic ranges, suggesting that the stable Ir(II) and Ir(IV) radicals are formed from the Ir(III) complexes.

**Table 1.** Photophysical, electrochemical and thermal properties of Ir(III) complexes

Compound	T <sub>d</sub> [°C] <sup>a</sup>	T <sub>g</sub> [°C] <sup>b</sup>	λ <sub>em</sub> [nm] <sup>c</sup>	Φ <sub>pl</sub> <sup>d</sup>	HOMO/ LUMO [eV] <sup>e</sup>
(Et-Cz-BTz) <sub>2</sub> Ir(pic)	403	161	541, 578	0.10	-5.26/ -2.75
(Et-Cz-BTz) <sub>2</sub> Ir (pic-N-O)	394	247	541, 582	0.11	-5.26/ -2.81
(Et-Cz-BTz) <sub>2</sub> Ir (EO <sub>2</sub> -pic)	356	233	543, 582	0.12	-5.24/ -2.79
(Et-Cz-BTz) <sub>2</sub> Ir (EO <sub>2</sub> -pic-N-O)	349	241	542, 582	0.11	-5.26/ -2.80

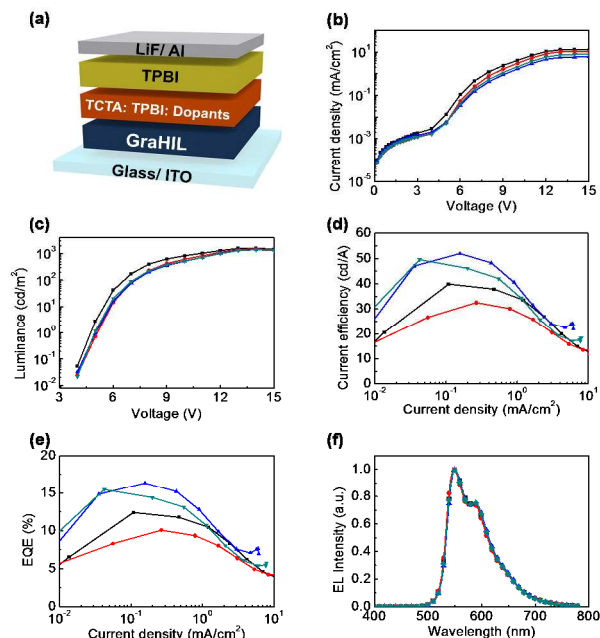
<sup>a</sup>Temperature with 5 % mass loss measure by TGA with a heating rate of 10 °C/min under N<sub>2</sub>. <sup>b</sup>Glass transition temperature, determined by DSC with a heating rate of 10 °C/min under N<sub>2</sub>. <sup>c</sup>Maximum emission wavelength, measured in CF solution at 1.0x10<sup>-5</sup> M. <sup>d</sup>Measured in 1x10<sup>-5</sup> M CHCl<sub>3</sub> solution relative to Ir(piq)<sub>2</sub>(acac) (Φ<sub>pl</sub>=0.20) with 455 nm excitation. <sup>e</sup>Determined from the onset of CV for oxidation and UV-visible absorption edge.

**Fig 3.** Cyclic voltammograms of (Et-Cz-BTz)<sub>2</sub>Ir(pic), (Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O), (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic), and (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O) in tetra-butylammoniumtetrafluoroborate (TBABF<sub>4</sub>) at a scan rate of 100 mVs<sup>-1</sup>.

The onset oxidation potentials were in the range of 1.00-1.02 eV and the corresponding HOMO energy levels were found to be -5.24 to -5.26 eV with ferrocene/ferrocenium as a reference redox system (4.8 eV below vacuum). The band gaps were calculated using the UV-visible absorption edge and were in the range of ~2.45 eV, which is within the error limits (0.1-0.7 eV) of E<sub>g</sub><sup>opt</sup> and the corresponding LUMO energy levels were found to be -2.75 to -2.81 eV.

We fabricated yellow emitting solution-processed PhOLEDs with the device structure shown in Fig. 4a. To realize the efficient solution-processed PhOLEDs with a simple structure, efficient blocking of electrons and quenching of excitons to increase the radiative decay as well as the efficient hole

injection to the EML at the HIL/EML interface is very important.<sup>27</sup>

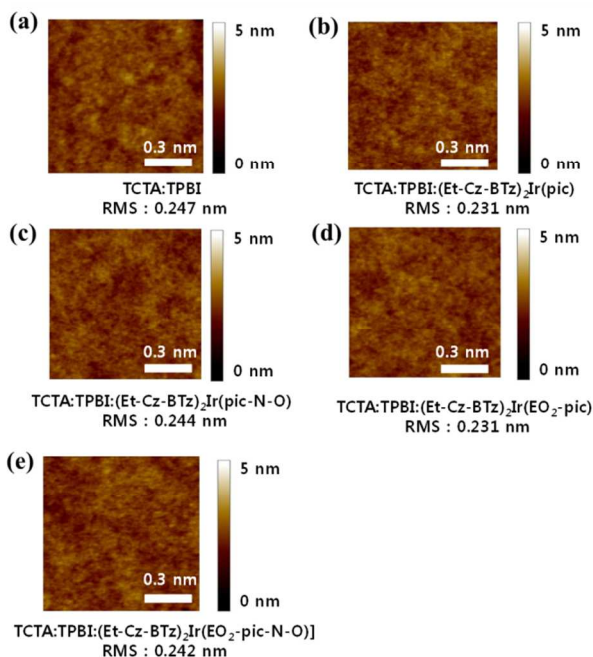
**Fig 4.** a) Device structure of yellow emitting PhOLEDs fabricated by spin coating the EML dissolved in tetrahydrofuran:CF cosolvent, b) current density-voltage of PhOLEDs with various yellow-emitting dopants ((—■—) [(Et-Cz-BTz)<sub>2</sub>Ir(pic)], (—●—) [(Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O)], (—▲—) [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic)] and (—▼—) [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O)]), c) luminance-voltage, d) current efficiency-current density e) external quantum efficiency-current density, and f) EL spectra.

We exploited a self-organized polymeric HIL that are composed of a conventional conducting polymer, poly(3,4-ethylenedioxythiophene):poly(styrene sulfonate) (PEDOT:PSS), and a perfluorinated polymeric acid with a low surface energy, tetrafluoroethylene-perfluoro-3,6-dioxo-4-methyl-7-octene-sulfonic acid copolymer (PFI), in 1:1 weight ratio (GraHIL).<sup>27-31</sup> These polymers develop gradient WF, which gradually increases from bottom surface (~4.8 eV) to top surface (~5.95 eV) due to self-organization of PFI, then, facilitate the hole injection and transport to EML by reducing the hole injection barrier between the WF of HIL and the HOMO energy level of EML. The large quantity of the insulating PFI on HIL surface also blocks electron injection and blocks quenching of excitons. Thus, the PhOLEDs with GraHIL do not require any overlying hole transport layer (HTL) or electron blocking layer (EBL).

We fabricated the EML by spin coating the solution of a yellow emitting phosphorescence dopant, a mixed host system consisting of a hole transporting host, 4,4',4''-tris(N-carbazolyl)triphenylamine (TCTA), and an electron transporting host, 1,3,5-tris(N-phenylbenzimidazol-2-yl)benzene (TPBI), in 1:1 weight ratio using tetrahydrofuran:CF (8:2) co-solvent on top of the GraHIL (Fig. 4a). The solution-processed PhOLEDs were fabricated with the configuration of ITO/GraHIL (40 nm)/TCTA:TPBI:Ir(III) complexes (15 wt%) (40 nm)/TPBI (50 nm)/LiF (1 nm)/Al (90 nm). The bipolar characteristics of the mixed host are advantageous to improve the device efficiency by broadening the recombination zone.<sup>32</sup>

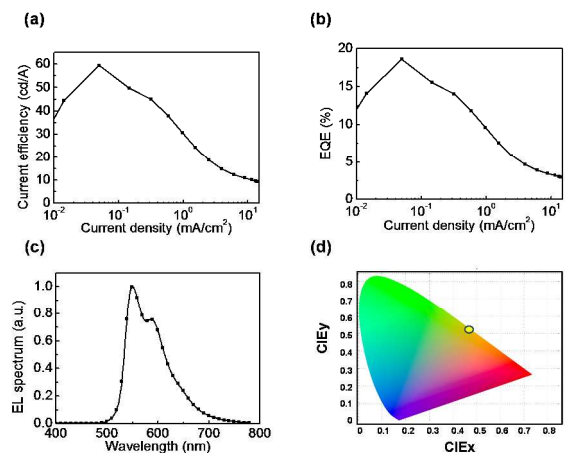
**Table 2.** Performances of solution-processed OLEDs with four different yellow emitting Ir(III) complexes

Compound	Solvent	CE <sub>max</sub> (cd/A)	EQE <sub>max</sub> (%)
(Et-Cz-BTz) <sub>2</sub> Ir(pic)	THF:CF (8:2)	40	12.5
(Et-Cz-BTz) <sub>2</sub> Ir(pic-N-O)	THF:CF (8:2)	32.5	10
(Et-Cz-BTz) <sub>2</sub> Ir(EO <sub>2</sub> -pic)	THF:CF (8:2)	52	16.5
(Et-Cz-BTz) <sub>2</sub> Ir(EO <sub>2</sub> -pic-N-O)	THF:CF (8:2)	50	15.5
(Et-Cz-BTz) <sub>2</sub> Ir(EO <sub>2</sub> -pic)	THF	60	19

**Fig 5.** Atomic force microscopy (AFM) images of a) TCTA:TPBI, b) TCTA:TPBI:(Et-Cz-BTz)<sub>2</sub>Ir(pic), c) TCTA:TPBI:(Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O), d) TCTA:TPBI:(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic) and e) TCTA:TPBI:(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O).

The current density-voltage and luminance-voltage of PhOLEDs with 4 different dopants are given in Fig. 4b and Fig. 4c, respectively. The maximum current efficiencies (CE) and EQE of PhOLEDs with [(Et-Cz-BTz)<sub>2</sub>Ir(pic)], [(Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O)], [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic)], and [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O)] were found to be (40 cd/A, 12.5%), (32.5 cd/A, 10%), (52 cd/A, 16.5%), and (50 cd/A, 15.5%), respectively (Fig. 4d and Fig. 4e). PhOLEDs with 4 different dopants showed same yellow spectrum with a maximum electroluminescence (EL) peak at ~550 nm and shoulder at ~590 nm, which correspond well with PL spectrum (Fig. 2b) at 1000 cd/m<sup>2</sup> (Fig. 4f). One of the major observations in solution processed PhOLEDs is the morphology, hence, the morphology of EML is ascertained the efficiency of the device performance. We also measured the surface roughness of neat TCTA:TPBI film and TCTA:TPBI with [(Et-Cz-BTz)<sub>2</sub>Ir(pic)], [(Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O)], [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic)], and [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O)] and shown in Fig. 5. The Root-mean-

square (RMS) roughness values of neat TCTA:TPBI film (Fig 5a.) and TCTA:TPBI with [(Et-Cz-BTz)<sub>2</sub>Ir(pic)] (Fig. 5b), [(Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O)] (Fig. 5c), [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic)] (Fig. 5d), and [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O)] (Fig. 5e) were found to be (0.247 nm), (0.231 nm), (0.244 nm), (0.231 nm), and (0.242 nm), respectively. This smooth surface roughness of the EML reduces the leakage current in device and induces the high luminous efficiency.

**Fig 6.** a) Current efficiency-current density of yellow-emitting PhOLEDs with (Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic) fabricated by spin coating the EML dissolved in tetrahydrofuran solvent, b) external quantum efficiency-current density, c) EL spectrum and d) CIE coordinate.

We further optimized the solution-processed PhOLEDs, which EML were fabricated by spin coating the solution dissolved only in THF solvent, and then finally achieved higher device efficiencies (CE: 60 cd/A, EQE: 19%) (Fig. 6a and Fig. 6b). The EL spectrum of optimized device was same with PL spectrum (Fig. 6c). To the best of our knowledge, these values are the best efficiencies for yellow EML solution-processed PhOLEDs using heteroleptic Ir(III) complexes reported so far. These high efficiencies can be attributed to the good organic solubility of dopants arising from benzothiazole linked ethylcarbazole in Ir(III) complexes and good electron-hole balance in the EML. The Commission Internationale de l'Éclairage (CIE) coordinates of device is (0.466, 0.525) at 1000 cd/m<sup>2</sup>, which corresponds well with yellow region in the CIE chromaticity diagram (Fig. 6d).

#### 4. Conclusions

In summary, we have successfully synthesized four BTz linked Et-Cz based Ir(III) complexes, [(Et-Cz-BTz)<sub>2</sub>Ir(pic)], [(Et-Cz-BTz)<sub>2</sub>Ir(pic-N-O)], [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic)], and [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic-N-O)], for solution-processed yellow PhOLEDs and fabricated devices with the configuration of ITO/GraHIL (40 nm)/TCTA:TPBI:Ir(III) complexes (15 wt%) (40 nm)/TPBI (50 nm)/LiF (1 nm)/Al (90 nm). Both Tz and BTz based Ir(III) complexes emit same bright yellow phosphorescence at room temperature, indicates that the introduction of BTz into the Et-Cz main ligand instead of Tz does not show significant effect on the phosphorescence emission of the Ir(III) complexes.<sup>13</sup> Among the four Ir(III) complexes, the captain Ir(III) complex [(Et-Cz-BTz)<sub>2</sub>Ir(EO<sub>2</sub>-pic)] showed the best luminous efficiency of 60 cd/A and EQE of 19% with CIE coordinates (0.467, 0.524), which is more

than three times higher than that of the captain Ir(III) complex of Tz counterparts.<sup>13</sup> To the best of our knowledge, this is one of the best solution-processed EML yellow PhOLEDs reported so far. This phenomenon paves an effective way towards the efficiency increment of particular PhOLEDs without changing the color by small modifications in the structure of main ligand of Ir(III) complexes.

## Acknowledgements

This work was supported by grant fund from the National Research Foundation (NRF) (2011-0028320), Pioneer Research Center Program through the NRF (NRF-2013M3C1A3065522) and (NRF-2013R1A2A2A01068753) by the Ministry of Science, ICT & Future Planning (MSIP) of Korea.

## Notes and references

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### Graphical abstract

A simple amendment on thiazole to benzothiazole based heteroleptic Iridium(III) complexes enhanced the efficiency in solution-processed yellow phosphorescent organic light-emitting diodes

