

Synthesis and luminescent study of naphthalenedioxyboron compounds for photoelectronic applications

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Abstract

The coordinatively saturated naphthalenedioxyboron compounds were newly synthesized and characterized by ¹H-NMR and mass spectrometry. First, the dianion of 2,3-dihydroxynaphthalene (dhn) was coordinated to the boron center by the reaction of dhn and B(OEt)₃, resulting in (dhn)B(OEt). (dhn)B(OEt) was then reacted with 2-amino-3-hydroxynaphthalene (an), 8-hydroxyquinoline (q) or 10-hydroxybenzo[h]quinoline (bq) to obtain the coordinatively saturated boron compounds, (dhn)B(an) (**1**), (dhn)B(q) (**2**), or (dhn)B(bq) (**3**). The photophysical properties of the resulting boron compounds were investigated with UV–VIS spectroscopy and photoluminescence (PL) spectroscopy. Their luminescent properties and sublimability were tested for the photoelectronic applications. The absorption bands of these boron compounds **1–3** were observed at below 320, 400, 450 nm, respectively, in the UV–VIS spectra, showing typical ligand-based transitions. The solid films of **1–3** showed luminescence at 378, 485 and 510 nm, respectively. All of these boron compounds were sublimable under vacuum condition of 1.7×10^{-5} Torr. Further studies on these compounds have been attempted in our laboratory for the photoelectronic applications such as light-emitting layers of the light-emitting devices.

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1. Introduction

Recently, various boron compounds have been utilized as blue emitting materials of OLED. Useful and efficient organic or organometallic blue emitters are however still rare. Boron is strongly electrophilic by virtue of its tendency to fill the vacant orbital and complete the octet. Consequently, the properties of many boron

compounds have been explained by postulating coordination of an electron-donating atom to boron. Thus, boron compounds may provide extra stability as emitting materials.

The common feature of those previously reported boron compounds is that they are all chelated complexes and bind to the central boron atom through both nitrogen and/or oxygen donor atoms [1–3]. We have been interested in synthesis and application of new other boron compounds to EL device because the photophysical properties of this class of compounds can be modified readily by manipulating the coordination environment around the central boron atom.

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Herein, we report that coordinatively saturated naphthalenedioxyboron compounds were newly synthesized and characterized. Their thermal properties including sublimability were studied and their luminescent characteristics were investigated for the application to light-emitting devices. Finally, electroluminescent properties were also investigated.

2. Experimental

2.1. General method

Reagents were purchased from Aldrich Co. and used without further purification. The solvents for the synthesis of the boron compounds were the reagent grades and predried according to the purification methods freshly when needed. Anhydrous solvents packed under nitrogen were purchased if necessary and used with standard Schlenk techniques. $^1\text{H-NMR}$ spectra were obtained on Bruker 200 MHz spectrometer at Sogang University, Korea. Mass spectra were determined on JEOL, JMS-AX505WA, HP 5890 Series II Hewlett-Packard 5890A (capillary column) at Seoul National University, Korea. UV–VIS absorption spectra were measured on Hewlett-Packard 8425A spectrometer. The PL spectra were measured on Perkin Elmer LS 50B spectrometer.

2.2. Device fabrication

Commercial ITO-coated glass, with a sheet resistance of $30 \Omega/\square$, was used as the starting substrate. After the glass precleaning, the constituent organic layers of the OLEDs were deposited by a thermal vacuum evaporation. The structure of multi-layer OLEDs were shown as follows: and ITO/PEDOT (1000 Å)/boron compound (500 Å)/LiQ (30 Å)/Al (1000 Å) (device I). ITO/NPB (400 Å)/boron compound (500 Å)/LiQ (30 Å)/Al (1000 Å) (device II).

PEDOT/PSS (poly(3,4-ethylenedioxythiophene)/polystyrene sulfonate) for the device I was coated. NPB (4,4'-bis{*N*-(1-naphthyl)-*N*-phenylamino}biphenyl}) for device II was deposited as the hole injection layer (HIL) or the hole transport layer (HTL), respectively. The PEDOT/PSS films were spin-coated in air onto patterned ITO substrates from dispersion after filtration (0.45 μm) and dried in an oven at temperature between 60 and 70 °C in air. The boron compounds (1)–(3) were deposited as the light-emitting layer (500 Å), respectively. A 30-Å-thick Liq (lithium quinolate) as an electron injection layer were then deposited sequentially. Finally, Al (150 nm) was deposited as a cathode. The deposition rate of the organic thin film was 0.1 nm/s at the base pressure of 5×10^{-6} Torr. *I*–*V* curve was measured on a Keithley 236 Source meter in an ambient

environment. The CIE coordinates of devices was obtained by Minolta CS-100.

2.3. Synthesis of (dhn)B(OEt)

2,3-Dihydroxynaphthalene (0.300 g, 1.875 mmol) was placed in 2 mL anhydrous toluene in a 3-neck round bottom flask (rbf) equipped with N_2 supply and a reflux condenser. Triethoxyboron (0.4 mL, $d = 0.858$, 2.43 mmol) was added and the mixture was heated to reflux for 3 h. The mixture turned to a yellow solution. After it was cooled to room temperature, ethanol produced from the reaction and most of the solvent was removed by azeotropic vacuum distillation to yield the concentrated solution of (dhn)B(OEt). The volume of (dhn)B(OEt) solution was reduced to ≈ 10 mL by evaporation and used in situ for the next reaction.

2.4. Synthesis of (dhn)B(an) (1), (dhn)B(q) (2) and (dhn)B(bq) (3)

2.4.1. 2,3-Naphthalenedioxy-2'-amino-naphthalene-3'-oxyboron (dhn)B(An) (1)

2-Amino-3-hydroxynaphthalene (0.297 g, 1.875 mmol) was suspended in 20 mL of anhydrous toluene and its solution was added to the concentrated solution of (dhn)B(OEt). The resulting mixture was heated to reflux for 4 h and cooled to room temperature. The precipitate formed from the reaction was filtered and washed with acetonitrile to remove unreacted 2-amino-3-hydroxynaphthalene. The resulting solid was obtained as beige powder of (dhn)B(an) (1) (0.325 g, 53%).

mp. 270 °C (dec.) MS calcd for $\text{C}_{20}\text{H}_{14}\text{BNO}_3$ *m/e* 327.11, found *m/e* 328.

2.4.2. 2,3-Naphthalenedioxy-quinolin-8'-oxyboron (dhn)B(q) (2)

8-Hydroxyquinoline (q) was dissolved in 20 mL of anhydrous toluene and its solution was added to the concentrated solution of (dhn)B(OEt) via a syringe. The pale yellow precipitate was formed immediately upon mixing the both solutions. The reaction mixture was stirred for 4 h. The resulting precipitate was filtered and washed with toluene several times. The solid was dried to give (dhn)B(q) (2) as pale yellow powder (0.305 g, 52%).

mp. 220 °C MS calcd for $\text{C}_{19}\text{H}_{12}\text{BNO}_3$ *m/e* 313.09, found *m/e* 314.

2.4.3. 2,3-Naphthalenedioxy-benzoquinolin-10'-oxyboron (dhn)B(bq) (3)

10-Hydroxybenzo[*h*]-quinoline was dissolved in 20 mL of anhydrous toluene and its solution was added to the concentrated solution of (dhn)B(OEt) via a syringe. The yellow precipitate was formed immediately upon mixing the both solutions. The reaction mixture

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