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A new synthesis strategy for acridine derivatives to constructing novel host for phosphorescent organic light-emitting diodes



PIGMENTS

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ABSTRACT

A new intermediate, 3-bromo-9,9-diphenyl-9,10-dihydroacridine, based on 9,10-dihydroacridine was synthesized and thereby a new host material, 2,6-bis(9,9,10-triphenyl-9,10-dihydroacridin-3-yl)pyridine, via the intermediate was designed and prepared. This intermediate directed a meta-derivation and could make the resulted host retain triplet energy of 2.68 eV. Besides, the rigid backbone along with the substituted phenyl rings at C9 position could benefit the thermal and morphological stabilities of host. According to these features arise from the key intermediate, blue and red phosphorescent organic light-emitting diodes were fabricated and external quantum efficiencies above 10% were achieved with relative flat roll-off.

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1. Introduction

Acridine, identified in 1870 by Graebe and Caro [1,2], continuously draws increasingly attention due to many attractive features. Conventional and important applications of acridine are mainly on biologically active drugs, dyes or sensors [3–7]. Recently, acridine and its derivatives are gradually utilized as new blocks for optoelectronic application stems from its rigid conjugated structure, shedding a new light on this old molecule [8–24]. For this purpose, various synthetic methods have been developed to prepare various acridine derivatives. Ring closure reaction, for example, is the most effective strategy applied in the synthesis of acridine derivatives. Starting from suitable precursors and positions, several types of acridine derivative can be obtained by different ring closure reactions, which are summarized in Fig. 1. In addition to the ring closure reaction, rearrangement, oxidation and reduction can also be used to synthesize acridine [25,26].

A noteworthy derivative of acridine is 9,10-dihydroacridine, a reduced acridine, which is converted to be an electron-rich ring from the electron-deficient acridine itself. This feature could potentially be exploited as hole-transport materials, host materials and emitters in organic light-emitting diodes (OLEDs) [8-24]. This 9,10-dihydroacridine can also be seen as a closed diphenylamine, and indeed, it commonly follows electrophilic reaction to form this core. Notice that there are three parts for further modification on 9,10-dihydroacridine: the phenyl backbone, nitrogen atom and bridged carbon atom. Interestingly, though there are many reports on modifying the 9,10-dihydroacridine, the derivatization from the backbone is rare. For instance, Lee et al. developed new holetransport materials for organic light emitting diodes (OLEDs) by introducing some hole-transport moieties on the C9-position in 9,10-dihydroacridine [12,13]. Recently, Adachi et al. utilized one of the acridine derivative, named 9,9-dimethyl-10-phenyl-9,10dihydroacridine (PhDMAc), to construct new emitters via thermally activated delayed fluorescence (TADF) [14-21]. And our group has explored the strategy on modification from the nitrogen atom in 9,10-dihydroacridine block, which can be applied to prepare host materials for phosphorescent OLEDs (PHOLEDs) [22]. But restricted by the active sites of 9,10-dihydroacridine, the subsequent substituent groups will be limited to the para-positions of acridine moiety [23,24]. To develop new position on 9,10dihydroacridine, improved methods should be figure out to get more possibilities of derivative work on the 9,10-dihydroacridine.

In this manuscript, a new intermediate of 3-bromo-9,9diphenyl-9,10-dihydroacridine, is designed and successfully



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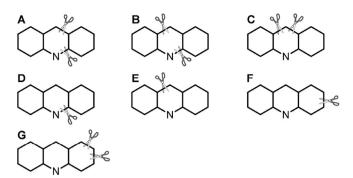


Fig. 1. Classification of synthetic strategy to prepare acridines.

prepared (Scheme 1). This intermediate remains a pre-occupied bromine atom at C3 position, and from this position, we prepared a new compound 2,6-bis(9,9,10-triphenyl-9,10-dihydroacridin-3-yl)pyridine (**DPhAcPy**) as host material for blue & red phosphorescent OLEDs. Good thermal stability, suitable HOMO/LUMO level and high triplet energy makes it achieve around 10% external quantum efficiency with relative flat roll-off for blue & red PHOLEDs.

2. Experimental

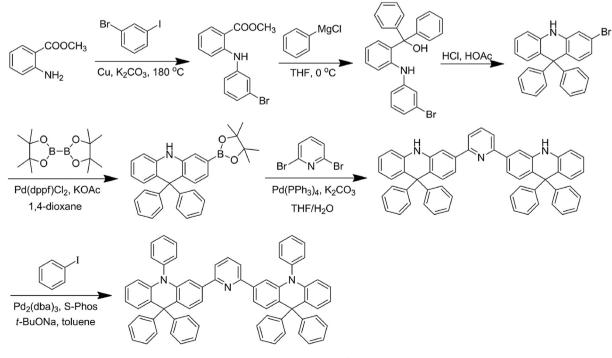
2.1. Chemicals and instruments

All chemicals and reagents were used as received from commercial sources without further purification. THF was purified by PURE SOLV (Innovative Technology) purification system. ¹H NMR and ¹³C NMR spectra were recorded on a Bruker 400 spectrometer at room temperature. Mass spectra were recorded on a Thermo ISQ mass spectrometer using a direct exposure probe. UV–vis absorption spectra were recorded on a Perkin Elmer Lambda 750 spectrophotometer. PL spectra and phosphorescent spectra were recorded on a Hitachi F-4600 fluorescence spectrophotometer. Differential scanning calorimetry (DSC) was performed on a TA DSC 2010 unit at a heating rate of 10 °C min⁻¹ under nitrogen. The glass transition temperatures (T_g) were determined from the second heating scan. Thermogravimetric analysis (TGA) was performed on a TA SDT 2960 instrument at a heating rate of 10 °C min⁻¹ under nitrogen. Temperature at 5% weight loss was used as the decomposition temperature (T_d). Cyclic voltammetry (CV) was carried out on a CHI600 voltammetric analyzer at room temperature with ferrocenium-ferrocene (Fc⁺/Fc) as the internal standard. DFT calculations were performed using B3LYP/6-31 G(d) basis set using Gaussian 09.

2.2. Preparation of 3-bromo-9,9-diphenyl-9,10-dihydroacridine

Methyl anthranilate (20 g, 132 mmol), 1-bromo-3-iodobenzene (37.3 g, 132 mmol), K₂CO₃ (36.4 g, 264 mmol), and copper powder (0.17 g, 2.64 mmol) was scattered in 1,2-dichlorobenzene (o-DCB) and stirred for 10 min at room temperature under argon, and then heats up to 180 °C for 24 h. After the reaction cooling to room temperature, filtered and washed with dichloromethane (DCM). Remove the solvent and the crude product was purified by column chromatography using petroleum ether (PE)/DCM (5/1, v/v) to afford the pure product methyl 2-[(3-bromophenyl)amino]-benzoate as a chartreuse oil (32 g, 80%). This product was used in the next step needn't to further purification. ¹H NMR (400 MHz, CDCl₃): δ 9.49 (s, 1H), 7.98–7.96 (m, 1H), 7.41 (s, 1H), 7.35 (m, 1H), 7.29 (d, I = 0.9 Hz, 1H), 7.19–7.13 (m, 3H), 6.79 (m, 1H), 3.90 (s, 3H) ppm. ¹³C NMR (101 MHz, CDCl₃): δ 168.83, 146.75, 142.63, 134.15, 131.71, 130.63, 126.06, 124.42, 122.93, 120.28, 118.07, 114.48, 112.76, 51.91 ppm. MS *m*/*z*: 305.01. Anal. calcd for C₁₄H₁₂BrNO₂ (%): C 54.92, H 3.95, N 4.58; found: C 54.71, H 4.08, N 4.63.

The chartreuse oil (15 g, 49 mmol) was dissolved in 150 mL tetrahydrofuran (THF) in a 500 mL boiling flask-3-neck under argon and cooled to 0 $^{\circ}$ C, and then phenylmagnesium chloride (PhMgCl, 2 M in THF, ~74 mL, 147 mmol) was added dropwise under stirred



Scheme 1. Synthetic routes of DPhAcPy.

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