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Synthesis and Fluorescence Properties of 5,7-Diphenylquinoline and 2,5,7-Triphenylquinoline Derived from *m*-Terphenylamine

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Abstract: Synthesis of 5,7-phenylquinoline from the Skraup reaction of *m*-terphenylamine and glycerol in the presence of acid is reported. Further reaction of 5,7-diphenylquinoline with phenyl lithium prepared *in situ* led to the formation of 2,5,7-triphenylquinoline. All of the products and their intermediates were characterized and the UV-Vis and photo-luminescence (PL) spectra of *m*-terphenylamine, 5,7-diphenylquinoline and 2,5,7-triphenylquinoline are also reported.

Keywords: Synthesis**,** *m*-terphenylamine, Skraup reaction, 5,7-diphenylquinoline, 2,5,7-triphenylquinoline.

Introduction

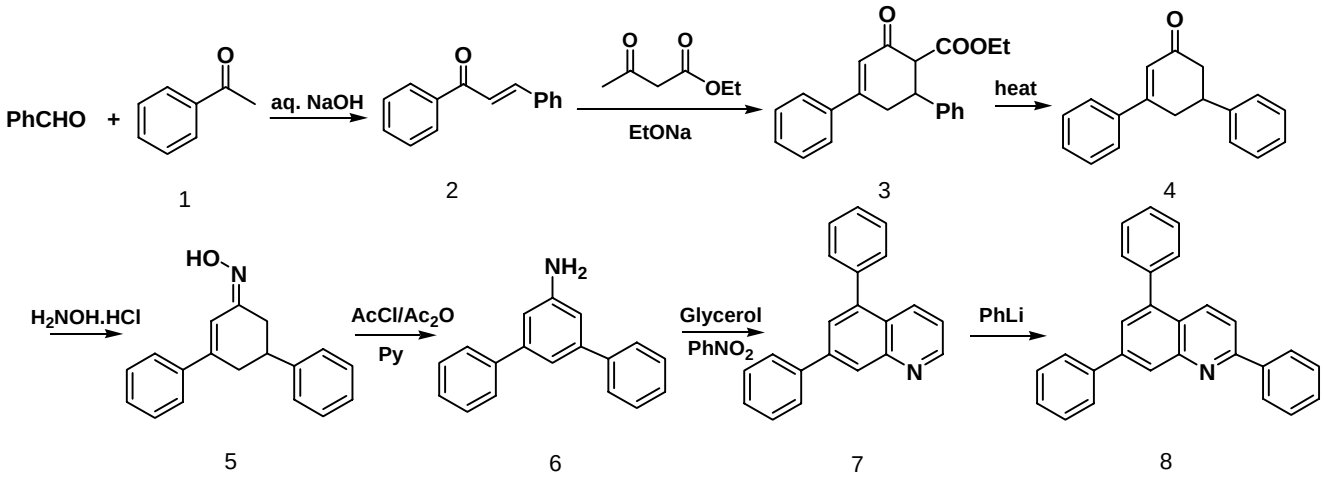
The exploitation of functional heterocyclic molecules is worthwhile due to their unique biological properties in drug evaluation and possible utilization in organic electroluminescent diodes (OLEDs) when coordinated with transition metal centers to form the corresponding organometallic compounds. As the research into and development of OLEDs has advanced, more and more international companies, for example: Philips, Siemens, Pioneer, Toyota, NEC, Kodak, HP, IBM, DuPont, Dow Chemical, Samsung, Sanyo and so on, have paid considerable attention to this topic. Luminescent materials include small organic molecules, organometallic compounds and polymers. Most of them are

heterocyclic compounds and polymers containing heterocycles. 8-Hydroxyquinoline aluminum (Alq_3) [1] and poly-*p*-phenylacelylene (PPV) [2] are universal OLED materials. Quinoline, isoquinoline and their derivatives are among the most important heterocyclic precursors [3]. For example $Almq_3$ [tris(4-methyl-8-quinolinolato)aluminum(III)] [4] and $Zn(BTZ)_2$ [bis(2-(2-hydroxyphenyl)benzothiazole)-zinc(II)] [5] are both excellent luminescent materials and good electron transmission materials formed from quinoline derivatives coordinated with the metals Al and Zn, respectively. The synthesis of quinolines and their derivatives has been of considerable interest to organic and medicinal chemists for many years as a large number of natural products [6] and drugs [7] contain this heterocyclic nucleus, e.g. 6-aminochrysene, discovered simultaneously in 1890 by Abegg [8] and Bamberger and Burgdorf [9], which has recently acquired importance as a chemical inhibitor of the growth of spontaneous adenocarcinoma of the breast. Acetylcholinesterase is the target of drugs that inhibit the hydrolysis of acetylcholine and alleviate the cholinergic deficit associated with Alzheimer's disease [10]. The classical methods for quinoline synthesis involve Skraup's procedure and the Doebner-Von Miller synthesis. The mechanism of these procedures and the synthesis of numerous quinoline derivatives have been studied in many papers [11]. In order to explore their synthetic utility and application, we considered exploiting the functionality of *m*-terphenylamine as a precursor for the synthesis of functional quinoline derivatives for further utilization, both in medicinal applications and optical-electric devices, and we report herein the synthesis and properties of *m*-terphenylamine, 5,7-diphenyl-quinoline and 2,5,7-triphenylquinoline.

Results and Discussion

Current research has focused on new synthetic routes to quinoline derivatives by multiple synthetic methods starting from simple starting materials such as acetophenone and benzaldehyde. To prepare the target molecule, *m*-terphenylamine was synthesized following a literature report [12]. Further reaction of 5,7-diphenylquinoline with phenyl lithium prepared *in situ*, adopting a literature method used for the synthesis of 2-substituted derivatives of 8-hydroxyquinoline compounds, led to the formation of 2,5,7-triphenylquinoline [13] (Scheme 1).

Scheme 1.



Padmavathi *et al*. reported synthesis of diphenylquinoline or diphenyltetrahydroquinoline derivatives from the *o*-allyl ethers of diarylcyclohexenone and diarylcyclohexanone by sigmatropic rearrangement and cyclization [14], while Dufour *et al*. reported preparation of a series of carcinogenic nitrogen compounds such as 2,7-, 4,7-disubstituted or 2,4,7-trisubstitued quinoline derivatives by the Bayer-Combes reactions or the Doebner reaction of 3-aminobiphenyl [15]. The key intermediates 6-carbethoxy-3,5-diphenylcyclohex-2-en-1-one (**3**) and 3,5-diphenylcyclohex-2-en-1-one (**4**) were used as effective synthons by Padmavathi *et al*. for synthesis of a range of heterocyclic derivatives [14b].

The intermediate **2** can also be prepared by using another method [16]. As for intermediate **4**, it was found that this product could be prepared in fairly good yield under either acidic or basic conditions. When compound **3** was added to an aqueous solution of sodium hydroxide and the mixture was subsequently refluxed for 3 hours to ensure completion of the reaction (as monitored by TLC) **4** could be obtained in 90% yield after workup and recrystallization from ethanol [17]. Alternative attempts were initially conducted under basic reaction conditions (e.g. in aqueous alcoholic KOH solution), and the resulting solution was refluxed overnight, then acidified by addition of 33 % sulfuric acid to pH = 7 and refluxed for a further 90 minutes. After workup and recrystallization, product **4** was obtained in 78% yield [18].

The Skraup reaction from **6** to **7** is usually vigorous and sometimes violent, as described by Clarke and Davis in *Organic Syntheses*: “In the Skraup synthesis of quinoline, the principal difficulty has always been the violence with which the reaction generally takes place; it occasionally proceeds relatively smoothly, but in the majority of cases gets beyond control” [19a]. Experiments proved that the violence of the ordinary Skraup reaction is due to the sudden liberation of acrolein, resulting from the action of sulfuric acid upon the glycerol. In our case, we have succeeded in avoiding this problem by the addition of acetic acid to dilute the reaction mixture [19b]. The acetic acid was introduced in an effort to form a glycerol mono- or di-acetate and thereby remove a large proportion of the glycerol from the reaction sphere. Additionally, it is surprising to find that not many side products were formed when nitrobenzene was used as solvent and oxidant for preparation of quinolines from different terphenylamines while the corresponding nitroarenes should be used as solvents and oxidants in general. The decreased reaction yield might be due to product losses during decolorization in ethanol over activated charcoal. Expected pure crystalline 5,7-diphenylquinoline was obtained in each run in better than 35% yield. The violent reaction could be avoided and the toxicity of acrolein could also be avoided. Efforts were also made to synthesize the title compounds using the Doebner-Miller synthesis in a two-phase solvent system [20]. Good yields are obtained by this method (the isolated yield was more than 50%) and the isolation is also easier, but in view of the toxicity and problems of acrolein and from a scale-up point of view, the Skraup reaction was preferred and studied in detail. Certainly, many other novel methods have been reported in synthesizing quinolines and their derivatives in good yield. For instance, Bose and others reported preparations of quinoline and dihydroquinoline derivatives under solvent-free conditions promoted with microwaves [21] or in the presence of metal halides [11b].

The preparation of the final product 2,5,7-triphenylquinoline was initially conducted in ethyl ether by adopting literature method as described for substituted 8-hydroxyquinolines [13]. Poor yields resulted in several attempts, which might be due to the poor solubility of 5,7-diphenylquinoline in

ethyl ether. Better results could be obtained when a mixture of solvents was used (phenyl lithium was prepared in Et_2O while the 5,7-diphenylquinoline was dissolved in THF), although there were still some by-products and yield was still low. Further attempts give better results (ca. 35% yield) when THF was used as the only solvent.

In order to study the properties of *m*-terphenylamine, 5,7-diphenylquinoline and 2,5,7-triphenyl quinoline, a preliminary investigation of their UV-Vis and photoluminescence (PL) spectra recorded for solutions of these compounds in CH_2Cl_2 (concentration: 2.5×10^{-5} mol/L) was undertaken. The results are shown in Figures 1-4. The UV-Vis spectra of 5,7-diphenylquinoline showed three absorptions at 210, 255 and 335 nm; whereas that of 2,5,7-triphenylquinoline showed three bands at 210, 275 and 350 nm, which are comparable to those of *m*-terphenylamine at 210, 250 and 320 nm, respectively. One can see that the absorptions shift from 255 nm to 275 nm as the result of red shift resulting from the increased conjugation.

Figure 1. The UV-Vis spectra of *m*-terphenylamine.

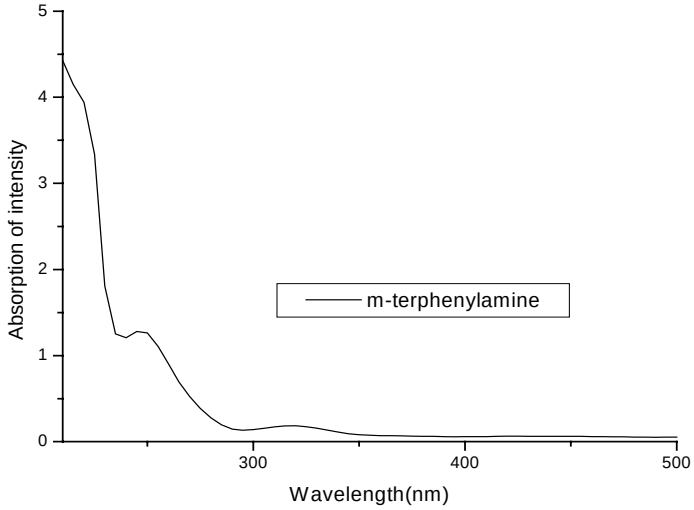
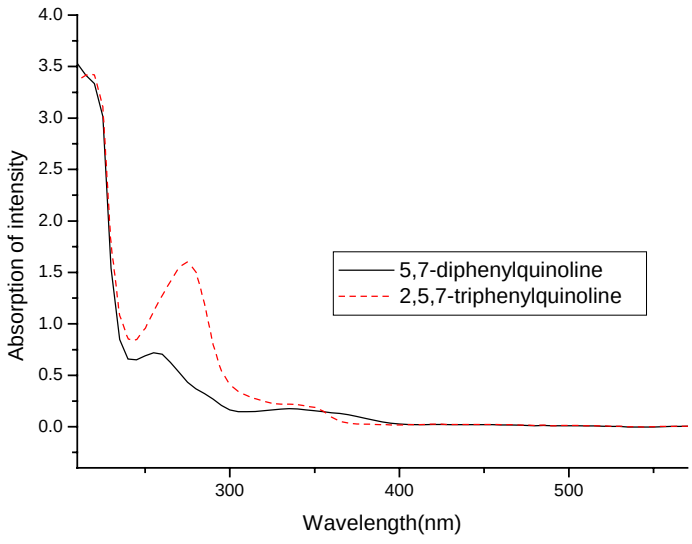


Figure 2. The UV-Vis spectra of 5,7-diphenylquinoline and 2,5,7-triphenylquinoline.



As for the photoluminescence (PL) spectra, the PL spectrum of 2,5,7-triphenylquinoline shows an absorption at 393.6 nm, while 5,7-diphenylquinoline shows one at 382.4 nm, that is, the absorption shifts *ca.*11 nm, which is comparable to that of *m*-terphenylamine at 400.4 nm (Figure 3). This might be the result of the replacement of the benzene ring of 2,5,7-triphenylquinoline causing a decrease in the HOMO-LUMO orbital energy gap. The energy needed for the transition state electrons is decreased, and the photoluminescent absorption of the molecules' spectra shifts to longer wavenumbers. It was obvious that 2,5,7-triphenylquinoline had better fluorescence intensity compared to that of 5,7-diphenylquinoline (see Figure 4), so we expect that 2,5,7-triphenylquinoline might be a promising precursor to develop new OLED materials, used either as single component or as a ligand for further synthesis of metal complexes (e.g. with iridium, palladium and platinum, etc.).

Figure 3. The PL spectra of *m*-terphenylamine.

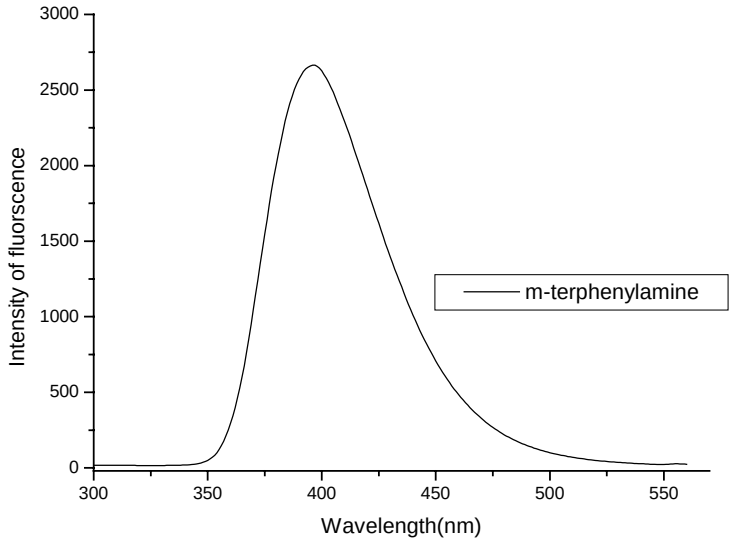
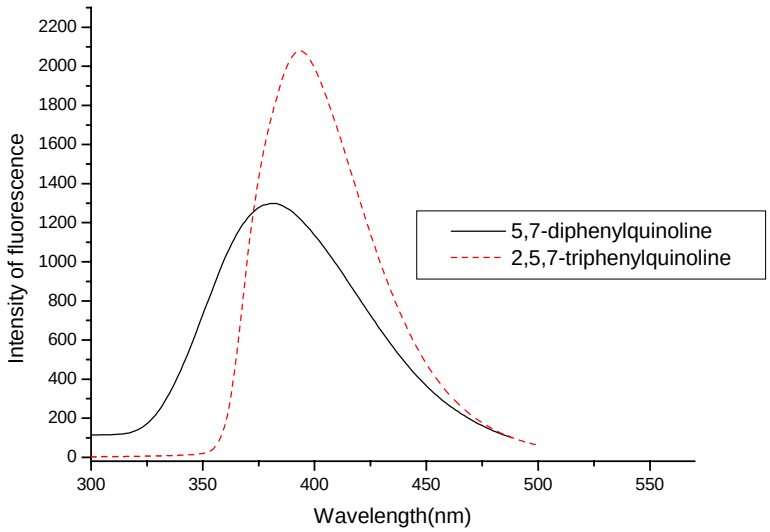


Figure 4. The PL spectra of 5, 7-diphenylquinoline and 2, 5, 7-triphenylquinoline.



Experimental

General

All of reactions were conducted under an inert atmosphere. Melting points were measured in open glass capillaries on a Temperature Apparatus and are uncorrected. The purification of compounds was accomplished by chromatography (Silica Gel HFG 254) with petroleum ether and ethyl acetate as eluents. The ^{1}H- and ^{13}C-NMR spectra were recorded in $CDCl_3$ on a Bruker AMX-500MHz spectrometer with TMS as internal standard (chemical shift in ppm), UV-Vis and photoluminescence (PL) spectra were measured by the Micro-analytical Chemistry Division, Department of Chemistry, East China Normal University, Shanghai 200062, P.R. China.

1,3-Diphenyl-2-propen-1-one (**2**): M.p.: 57 °C; ^{1}H-NMR δ = 8.03 (d, 2H, H-2, H-6), 7.84 (d, 1H, *J*= 16 Hz, -C = CH-Ph), 7.42-7.67 (m, 9H).

6-Carbethoxy-3,5-diphenylcyclohex-2-en-1-one (**3**): M.p.: 111 - 113 °C; ^{1}H-NMR δ = 7.26-7.56 (m, 10H, Ph-H), 6.57 (d, 1H, *J* = 4Hz, H-2), 4.03 (q, 2H, *J* = 7Hz, -CH_2CH_3), 3.81 (m, 2H, H-5, H-6), 3.13 (m, 2H, H-4), 1.05 (t, 3H, *J* = 7 Hz, -CH_2CH_3).

3,5-Diphenylcyclohex-2-en-1-one (**4**): M.p.: 81 - 83°C; ^{1}H-NMR δ = 7.26 - 7.57 (m, 10H, Ph-H), 6.52 (d, 1H, *J* = 3Hz, H-2), 3.36 (m, 1H, H-5), 3.08 (m, 2H, H-6), 2.78 (m, 2H, H-4).

3,5-Diphenylcyclohex-2-en -1-one oxime (**5**): M.p.: 162 - 165 °C; ^{1}H-NMR δ = 7.26 - 7.58 (m, 10H, Ph-H), 6.65 (s, 1H, H-2), 3.40 (m, 1H, H-5), 3.15 (m, 2H, H-4), 2.45 (m, 2H, H-6), 1.63 (br, 1H, -OH).

m-Terphenylamine (**6**): M.p.:107 - 109 °C; ^{1}H-NMR δ = 7.62 (m, 4H, H-2´6´, H-2´´6´´), 7.43 (m, 6H, H-3´4´5´, H-3´´4´´5´´), 7.20 (s, 1H, H-4), 6.89 (s, 2H, H-2, H-6), 4.0 (br, 2H, -NH_2).

Preparation of 5,7-diphenylquinoline (**7**) [22].

A round bottle was charged $FeSO_4$ (2.72 g, 11 mmol), *m*-terphenylamine (0.560 g, 3.7 mmol), glycerol (5.55 g, 60 mmol), conc. H_2SO_4 (3 mL) and nitrobenzene (2.78 mL), then glacial acetic acid (3.33 mL) was added and the mixture was heated to 145 °C for 4 h, then water (5 mL) was added. After steam distillation, the dark viscous oil was extracted with CH_2Cl_2, the combined organic phase was washed twice with water and brine and dried over $MgSO_4$. After filtration, the filtrate was evaporated to dryness. The residue was chromatographed (silica gel, eluent petroleum ether-EtOAc = 8:1) to give pale-yellow rhomboidal crystals of the title compound (1.02 g, yield: 33%); M.p.: 116 °C; ^{1}H-NMR δ = 8.94 (d, 1H, *J* = 1Hz, H-2), 8.36 (s, 1H, H-8), 8.23 (d, 1H, *J* = 8Hz, H-4), 7.80 (s, 1H, H-6), 7.43-7.80 (m, 10H, Ph-H), 7.41 (t, 1H, *J* = 7Hz, H-3); ^{13}C-NMR δ = 150.7 (C-2), 149 (C-9), 141.6 (C-7), 140.1 (C-5), 140.19 (C-1´´), 139.41 (C-1´), 134.18 (C-4), 130.03 (C-3´5´), 128.99 (C-

3´´5´´), 128.52 (C-2´6´), 127.96 (C-4´),127.78 (C-4´´), 127.52 (C-2´´6´´), 126.97 (C-6), 126.52 (C-10), 125.92 (C-3), 120.95 (C-8).

Preparation of 2,5,7-triphenylquinoline (**8**)

Freshly distilled bromobenzene (1.8 mmol) was added dropwise via syringe to a stirring suspension of lithium metal (28 mg, 4 mmol) in THF (4 mL) under an inert atmosphere. The reaction mixture was slowly warmed to ensure the formation of phenyl lithium during which the color changed to dark red and the stirring was continued at ambient temperature for further 40 minutes. Then to the mixture was added carefully a solution of 5,7-diphenylquinoline (200 mg, 0.6 mmol) in THF (4 mL) and the resulting mixture was refluxed for 4 hours. After cooling, air was bubbled into the mixture to remove all volatiles, then water (4 mL) and dichloromethane (4 mL) were added and the suspension was neutralized with conc. HCl. After workup the residue was chromatographed (silica gel, petroleum ether-EtOAc = 20:1) to give the title compound as a white solid (85 mg, yield 34.8 %); M.p.: 130 - 132 °C; ^{1}H-NMR δ = 8.43 (s, 1H, H-8), 8.30 (d, 1H, *J* = 9Hz, H-4), 8.18 (d, 2H, *J* = 8Hz, H-2´´´6´´´), 7.78 (s, 1H, H-4), 7.39-7.56 (m, 14H); ^{13}C-NMR δ = 157.6 (C-2), 149.2 (C-9), 141.7 (C-7), 140.7 (C-5), 140.3 (C-1´´), 139.6 (C-1´), 134.9 (C1), 130(C-1´´´), 129.4 (C-3´5´3´´5´´3´´´5´´´), 127.9 (C-2´6´2´´6´´), 127.6(C-2´´´6´´´), 128.5 (C-4´4´´4´´´), 124.8 (C-6) ,118.9 (C-3,10), 112.0 (C-8).

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Sample Availability: Samples of the compounds *m*-terphenylamine, 5,7-diphenylquinoline and 2,5,7-triphenylquinoline are available from the authors.