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Indandione-terminated Quinoids: Facile Synthesis by Alkoxidemediated Rearrangement Reaction and Semiconducting Properties

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Dedication ((optional))

Abstract: A series of 1,3-indandione-terminated π -conjugated quinoids were synthesized by alkoxide-mediated rearrangement reaction of the respective alkene precursors, followed by simultaneous air oxidation. This new protocol allows to access quinoidal compounds with variable termini and cores. The resulting quinoids all show LUMO levels below -4.0 eV and molar extinction coefficients above 10⁵ L mol⁻¹ cm⁻¹. The optoelectronic properties of these compounds can be regulated by tuning the central cores as well as the aryl termini ascribed to the delocalized frontier molecular orbitals over the entire molecular skeleton involving aryl termini. n-Channel organic thin-film transistors with electron mobility of up to 0.38 cm² V⁻¹ s⁻¹ were fabricated, showing the potential of this new class of quinoids as organic semiconductors.

Quinoidal compounds are of great interest because of their unique electronic, optical, and magnetic properties.^[1] Most of the reported quinoidal compounds are terminated bv dicyanomethylene moieties, which stabilize the quinoidal scaffold by virtue of the strong electron-withdrawing ability of the cyano groups.^[1b, 2] A commonly used method for accessing such compounds involves palladium-catalyzed Takahashi coupling reaction between aromatic halides and dicyanomethanide anion followed by oxidation (Figure 1a),^[3] and various π -conjugated quinoidal compounds have been synthesized by this approach. However, their termini are difficult to functionalize, which are known to be as important as the quinoidal core in regulating the electronic properties of these compounds.^[1b, 3a, 3b, 4] Another type of quinoidal compounds is end capped by the aryl groups, [2b, 4b-d, ^{5]} which can be synthesized by nucleophilic addition to a carbonyl group followed by reduction (Figure 1b). The terminal aryl groups of the resulting compounds can serve as handles for the construction of guinoidal compounds with modifiable termini. However, these guinoids suffer from limited central core units and poor stability.^[2b, 4b, 6] Additionally, their lowest unoccupied molecular orbital (LUMO) energy levels are rather high owing to

Joint School of National University of Singapore and Tianjin University, International Campus of Tianjin University, Binhai New City, Fuzhou 350207, China Supporting information for this article is given via a link at the end of the document.((Please delete this text if not appropriate)) Previous work



Figure 1. Reported methods and our new approach for the synthesis of quinoidal compounds. Ar = aryl, Q = quinoid.

the lack of strong electron-withdrawing end groups.^[4c, 5b, 5c]

Obviously, the limitations of the above synthetic routes have created the need for new synthetic strategies to develop quinoidal compounds whose center cores and termini can be modified concurrently. In the current paper, we report a new approach, *i.e.*, alkoxide-mediated rearrangement reaction of alkene precursors followed by air oxidation, to access terminiand core-modified quinoids (Figure 1c). This protocol is operationally simple and has a wide substrate scope. The resulting compounds showed excellent stability and promising semiconducting properties. Importantly, their properties can be regulated by tuning the central core and the aryl termini.

The key intermediates in the synthesis of quinoidal compounds are the aromatic moieties bearing a tertiary-carbon at each end. It is known that the reaction between benzaldehyde and phthalide in the presence of sodium methoxide (CH₃ONa) give 2-phenyl-1,3-indandione in high yield, via rearrangement of 1-benzylidene-1,3-dihydroisobenzofuran intermediate а (Scheme 1a).^[7] We hypothesized that this reaction could enable the synthesis of di-tertiary-carbon-substituted aromatic compounds, which could then be converted to quinoids by oxidation. Therefore, we carried out the reaction of 4,4didodecyl-4H-cyclopenta[2,1-b:3,4-b']dithiophene-2,6-dicarboxal dehyde (1a) and phthalide under the conditions shown in Scheme 1b (Route A). Subsequent air oxidation did afford the target compound 5a, but the yield was low (14%). Attempts to improve the yield by optimizing the reaction conditions including solvent, base, and oxidation reagent were unsuccessful (Tables

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S1-S3). The formation of 5a should involve three steps: condensation, alkoxide-mediated rearrangement, and oxidation (Scheme 1b, Route A).^[7b] We inferred that the condensation reaction to form 3 might be the limiting step for the synthesis of 5a. Then we synthesized compound 3 separately by Wittig-Horner reaction between 1a and phthalide-3-phosphonate (2a) in 91% yield (Scheme 1b, Route B). Rearrangement reaction of 3 in the presence of sodium methoxide followed by simultaneous air oxidation afforded 5a in 65% yield. The process of rearrangement was confirmed by density functional theory (DFT) calculations, which revealed that the rearrangement of 3 was kinetically feasible (22.5 kcal/mol) and thermodynamically favorable (42.7 kcal/mol) (Figure S1). We later found that the isolation of 3 was unnecessary, and 5a could be readily obtained from 1a and 2a in 70% yield over three steps (Scheme 1b, Route B). Sodium hydride (NaH) was proved to be the best base for the Wittig-Horner reaction of 1a and 2a after extensive screening (Table S4).



 $\label{eq:scheme-1} \begin{array}{l} \mbox{Scheme-1. Synthesis of 2-phenyl-1,3-indandione and 5a. Isolated yields are reported.} \end{array}$

Table 1. Substrate scope^a

with different sizes were selected to react with 2a under the optimal reaction condition (Table 1). All the dialdehydes were readily converted to the desired quinoids 5b-5f in reasonable yields, and the yield was still as high as 44% (5f) when a substrate with five-ring polycyclic core was used. The protocol was also applicable for the synthesis of quinoidal compounds with various substituents on the terminal phenyl rings. As shown in Table 1, dimethoxyphenyl- and naphthyl-terminated compounds 5g and 5h were obtained in yields of 56% and 41%, respectively. However, the introduction of an electronwithdrawing substituent, fluorine (F), chlorine (Cl), or bromine (Br), on the terminal phenyl rings resulted in low yields (~10%) of the corresponding products (5i-5k). We ascribed these inferior results to the chemical instability of the F-, Cl-, and Brsubstituted phosphonates. However, by using stable phosphonium salts 2d-2f instead, we were able to synthesize 5i-5k in yields of >40%. Note that 5i and 5k were obtained as mixtures of two isomers, owing to the asymmetry of the terminal groups.

To explore the scope of this new protocol, dialdehydes 1b-1f

The chemical structures of all the guinoidal compounds were verified by ¹H NMR and ¹³C NMR spectroscopy, high-resolution matrix-assisted laser desorption ionization time-of-flight (MALDI-TOF) mass spectrometry, and elemental analysis. Single-crystal structure analysis can be used for unambiguous identification of quinoidal compounds. Fortunately, single crystals of 5b and 5f qualified for X-ray diffraction analysis were obtained by slow diffusion of methanol into benzene or toluene solution. As shown in Figure 2, the bond-lengths of C9-C10 and C11-C12 in 5b and C6-C7 and C8-C9 in **5f** are shorter than that of a typical $C(sp^2)$ - $C(sp^2)$ single-bond (ca. 1.45 Å) but are close to the length of a typical $C(sp^2)$ - $C(sp^2)$ double-bond (ca. 1.34 Å). While the bondlengths of C10-C11 and C12- C13 in 5b and C1-C6 and C7-C8 in **5f** are longer than that of a typical $C(sp^2)$ - $C(sp^2)$ double-bond. This alternation in bond lengths unequivocally confirms the quinoidal framework of these compounds.[5f, 8]



^alsolated yields are reported. **5b-5h** were synthesized with phosphonates **2a-2c** as the starting materials, and **5i-5k** were obtained using phosphonium salts **2d-2f** as the starting materials. The yields in parentheses for **5i-5k** were synthesized from phosphonates.





Figure 2. ORTEP front view of **5b** (a) and **5f** (b) obtained from single-crystal structures analysis. Alkyl chains and hydrogen atoms are omitted for clarity. The numbers in red indicate bond lengths (Å).

The UV-vis-NIR absorption spectra of 5a-5k in toluene are shown in Figure 3, and the related data are summarized in Table S5. All the compounds showed well-structured absorption spectra along with remarkably high molar extinction coefficients (ε) of up to 2.4 × 10⁵ L mol⁻¹ cm⁻¹, which can be attributed to the rigid and planar nature of the quinoidal structure.^[9] The absorption maxima (λ_{max}) were gradually bathochromic-shifted from 610 nm for 5a and 5b to 790 nm for 5f upon extending the π -conjugation of quinoidal core (Figure 3a). The spectra of **5a** and 5b, which have the same central core, are almost identical, indicating that the alkyl side chains had little effect on the electronic structures of the molecules.^[10] The absorption spectra of compounds with the same indaceno[1,2-b:5,6-b']dithiophene (IDT) core but different termini are depicted in Figure 3b. Introducing dimethoxy groups and halogen atoms or fusing benzene ring on the terminal phenyl rings all gave rise to redshifted spectra. Compound 5h, which has the largest termini (naphthyl), showed the reddest spectrum with a λ_{max} of 835 nm, which is red-shifted by 45 nm compared with that of 5f. In their dicyanomethylene-substituted comparison to counterparts, $^{[11]}$ 5c and 5d showed λ_{max} values that were redshifted by at least 75 nm, indicating that the present quinoidal system has the potential for the construction of low-bandgap conjugated molecules. This can be ascribed to the extended π conjugation involving the terminal aromatic moieties. In fact, DFT calculations indicated that both the highest occupied molecular orbital (HOMO) and the LUMO were delocalized over the entire conjugated framework (Figure S2). Notably, these new quinoidal compounds were environmentally stable. Their UV-vis-NIR absorption spectra remained unchanged when their solutions were irradiated with UV light for 30 min (Figure S3), and their ¹H NMR spectra did not change after their solutions being stored in air for two months (Figures S4-S14). From solution to film state, the λ_{max} of **5a-5e** were blue-shifted by 24 to 91 nm, suggesting the formation of H-type aggregations.^[11b, 12] In contrast, those of 5f-5k showed noticeable red-shifts due to the formation of J-type aggregations (Figure S15 and Table S5).^{[11b,} ^{12b]} This core-dependent aggregation behavior is also supported by the single-crystal structure analysis of 5b and 5f (Figures S16 and S17). By tuning the central cores and the termini, the thinfilm optical bandgaps of these guinoids could be varied over a wide range of 1.93-1.13 eV.

The electrochemical properties of **5a-5k** were investigated by solution cyclic voltammetry (CV) (Figure S18). All the

compounds showed a reversible reduction process, indicating that the anion species can be well stabilized by the quinoidal skeleton. All the compounds showed low-lying LUMO levels of < -4.0 eV, which are comparable to those of the dicyanomethylene-substituted quinoids.^[1d, 3a] Compounds 5a and 5b exhibited identical frontier molecular orbital (FMO) energy levels, suggesting that the influence of the alkyl chains on the FMOs was negligible.^[10, 13] As the π -conjugation length of the central core increased, the LUMO levels decreased from -4.06 eV for 5a to -4.27 eV for 5f, whereas the HOMO levels increased from -5.70 eV for 5a to -5.27 eV for 5f (Figure 3c). Compared with the HOMO level, the LUMO level is less influenced by the central core, indicating that the bandgap reduction is mainly caused by the increase of HOMO level as the π -conjugation extending. The FMO levels could also be modulated by introducing various substituents on the terminal phenyl rings (Figure 3d). Incorporating electron-rich methoxyl groups increased the HOMO and LUMO levels, whereas introducing electron-deficient F. Cl and Br groups decreased the HOMO and LUMO levels. Compound 5i, which bears four F atoms at the termini, had the lowest HOMO and LUMO levels at -5.43 and -4.38 eV, respectively. Interestingly, fused benzene ring on the end groups (5h) gave rise to low LUMO level (-4.34 eV) and intact HOMO level (-5.28 eV), suggesting that increasing the π -conjugation length of the termini mainly influenced the LUMO level. Clearly, the electronic properties of the present quinoids could be finely tuned by modifying the central cores and the termini. This feature is distinct from the dicyanomethylene-terminated guinoidal systems, where these properties can only be adjusted by turning the central core. In addition, polymerization groups such as Br could also be incorporated into these compounds, making them potentially serve as building blocks for novel conjugated systems.^[1c, 14]



Figure 3. Solution UV-vis-NIR absorption spectra of **5a-5k** in toluene (a, b) and HOMO/LUMO levels of **5a-5k** (c, d).

Top-gate and bottom-contact (TGBC) organic thin-film transistors (OTFTs) were fabricated to investigate the semiconducting properties of these quinoids. No transistor characteristics were observed for the devices based on **5a**, **5b**, **5d** or **5g**, whereas other compounds showed unipolar electron-transport behavior as revealed by the clear off-regimes in their transfer curves (Figures 4, S19 and S20).^[15] No hole injection and transport were observed for the devices of **5c**, **5e**, **5f**, **5h**, **5i**, **5j** and **5k** although they showed relatively high-lying HOMO

levels (Figure S21). Similar phenomenon was observed in other quinoidal compounds,^[4a] and the actual reason for the absence of hole transport is unclear. Representative output and transfer curves for the devices are shown in Figures 4, S19 and S20, and their performance parameters are summarized in Table S6. All the transfer and output characteristics showed negligible hysteresis between forward and reverse sweeps, indicating low trap density levels for electron transport.^[16] The saturation electron mobilities (μ_e) of 5c, 5e and 5f were 0.032, 0.0041 and 0.28 cm² V⁻¹ s⁻¹, respectively. Fluorination of the terminal phenyl groups enhanced the device performance and the μ_e of 5i reached 0.38 cm² V⁻¹ s⁻¹. In contrast, 5h, 5j and 5k showed inferior device performances with μ_e of 0.13, 0.16 and 0.15 cm² V^{-1} s⁻¹, respectively. The reliability factors (*r*) of the mobility values were calculated according to the method proposed by Choi,^[17] which are 67%, 76%, 80%, 86%, 81%, 82% and 73% for 5c, 5e, 5f, 5h, 5i, 5j and 5k, respectively (Table S6). Additionally, the device performance data in linear regime are summarized in Table S6. The saturation and linear mobility values were very close for these compounds. To evaluate the device stability, electrical characteristics of 5i-based devices were measured after storage for 30 days under ambient conditions (Figure S22). The devices still operated effectively with a μ_e of 0.11 cm² V⁻¹ s⁻¹. Given that **5i** contains bulky 4hexylphenyl side chains, which are detrimental to molecular packing, further engineering of the side chains can be expected to improve the electron mobility of the present quinoidal compounds.^[18]



Figure 4. Output (a) and transfer (b) characteristics of the TGBC OTFT devices based on $\mathbf{5i}.$

In conclusion, we have demonstrated a facile method for the synthesis of 1,3-indandione-terminated quinoidal compounds from readily accessible aryl dialdehydes and phthalide derivatives. This new approach allows the access of termini- and core-tunable quinoidal compounds and the feasible regulation of their optoelectronic properties *via* central core and termini engineering. These termini functionalized quinoids can also be used as building blocks for novel conjugated systems. Furthermore, OTFTs based on these compounds exhibited n-channel characteristics with the highest electron mobility of up to 0.38 cm² V⁻¹ s⁻¹. This study not only demonstrates a new protocol for the synthesis of quinoidal compounds but also provides a new class of organic semiconductors and new building blocks for organic semiconductors.

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Keywords: Indandione • quinoidal compound • aromatic dialdehydes • electron transport • organic thin-film transistor

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es adjustable via termini and core; Unipolar n-type semiconductors.

1,3-Indandione-terminated quinoids with variable termini and central cores were synthesized by alkoxide-mediated rearrangement reaction. The properties of these quinoids can be regulated by tuning the central cores as well as the aryl termini. These compounds displayed unipolar n-type semiconductor characteristics with electron mobility of up to 0.38 cm² V⁻¹ s⁻¹.

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