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Synthesis of 2-[2-(Ethoxymethoxy)phenyl]spiro[cyclopropane-1,2′-indene]-1′,3′-dione

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Abstract: An 1,3-indanedione-derived donor–acceptor cyclopropane, bearing the ethoxymethyl-protected phenolic group at the *ortho*-position of the donor aryl substituent, has been synthesized using a reaction sequence involving the Knoevenagel condensation of 1,3-indanedione with the corresponding protected salicylaldehyde followed by the Corey–Chaykovsky cyclopropanation of the obtained adduct with dimethylsulfoxonium methylide. The structure of the synthesized cyclopropane was unambiguously proved by single-crystal X-ray diffraction data.

Keywords: donor–acceptor cyclopropanes; Knoevenagel condensation; Corey–Chaykovsky cyclopropanation

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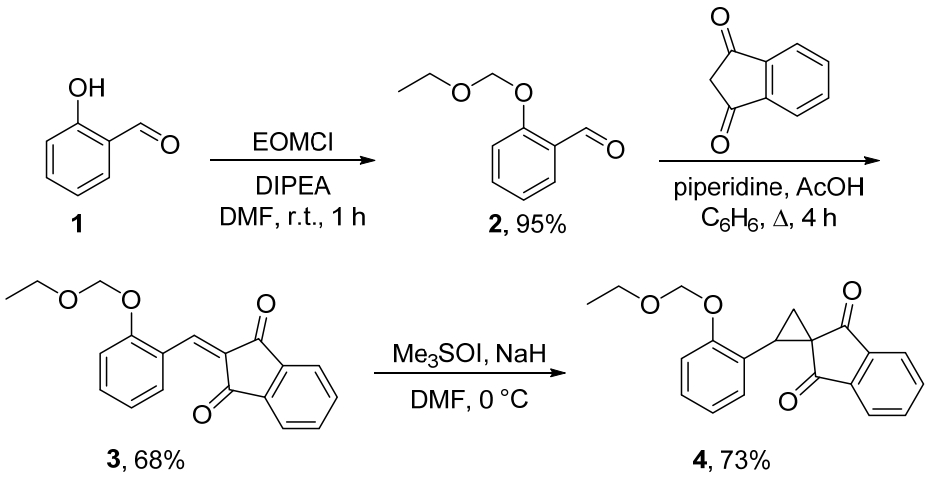
1. Introduction

Cyclopropanes, bearing donor and acceptor substituents at vicinal carbon atoms have attracted considerable attention from organic chemists in recent decades due to their high reactivity with respect to various classes of reagents, such as nucleophiles, electrophiles, diverse compounds with multiple carbon–carbon, carbon–heteroatom, and heteroatom–heteroatom bonds, 1,3-dipoles, 1,3-dienes, etc. Because of this unique reactivity, such substrates have become known as donor–acceptor (DA) cyclopropanes [1–7]. Typically, DA cyclopropanes react as synthetic equivalents of a 1,3-dipole, in which the carbon atom connected to the donor group serves as the electrophile, while the carbon atom bonded to the acceptor group(s) appears for the nucleophilic center. However, we and others have shown that DA cyclopropanes, in which the donor is an electron-rich (hetero)aromatic group, can also react with the involvement of the *ortho*-position of the aromatic group in the process as the nucleophile, affording various annulation products [5,8–12].

Further extension of the multifaceted reactivity of DA cyclopropanes can be achieved by using substrates in which the donor aryl substituent contains a reactive functional group at the *ortho*-position to the three-membered ring [13–25]. For example, domino transformations of 2-hydroxyaryl-derived DA cyclopropanes, including the small ring opening and a new ring closure with the participation of phenolic oxygen, afforded various heterocyclic products [13–15]. However, the study of the reactivity of such cyclopropanes has usually been limited to the corresponding 2-(2-hydroxyaryl)cyclopropane-1,1-diesters. Therefore, the synthesis of related substrates with other acceptor substituents should be important for the development of original processes leading to new polycyclic products. Herein, we describe a simple approach to 1,3-indanedione-derived DA cyclopropane bearing a protected phenolic moiety at the *ortho*-position of the donor phenyl group.

2. Results and Discussion

The title cyclopropane was synthesized from the commercial starting compound by simple procedures. Salicylaldehyde **1** was protected with ethoxymethyl chloride (EOMCl) according to the procedure previously described [14]. The Knoevenagel reaction of the resulting product **2** with 1,3-indanedione was carried out similarly to related condensations of other aldehydes, which were previously reported [26]. The cyclopropanation of the synthesized alkene **3** to obtain the cyclopropane **4** was performed by adding dimethylsulfoxonium methylide, generated by the treatment of trimethylsulfoxonium iodide with sodium hydride, to the solution of **3** in DMF at 0 °C (Scheme 1). It is worth noting that both the order of addition and the reaction temperature are crucial to the efficiency of this process. The addition of the solution of alkene **3** to the generated ylide led to the formation of the complex mixture of products. Similarly, the yield of the desired compound **4** was much lower due to the formation of various side products when cyclopropanation was performed at room temperature.



Scheme 1. Synthesis of cyclopropane **4**. DIPEA = *N*,*N*-diisopropylethylamine.

^{1}H NMR spectrum of compound **4** contains three doublets of doublets in the upfield region. These signals correspond to protons of the three-membered ring. Geminal coupling constant ^2J for CH_2 protons is 4.2 Hz, which is typical (from 4 to 5 Hz) for the methylene group of cyclopropane. The structure of indanedione-derived cyclopropane **4** was unambiguously confirmed by single-crystal X-ray analysis (Figure 1). This structure closely matches the structures of the related DA cyclopropane without *ortho*-substituent [26]. The small difference is in some rotation of the phenyl group, providing the most efficient interaction of ether oxygen with the closest carbonyl group. This interaction is possibly responsible for some shortening of the C(1)–C(2) bond between the atoms linked to donor and acceptor substituents in **4** (1.542 Å) compared to the analogue without the 2-ethoxymethoxy group (1.561 Å, [26]). NMR and IR spectral data for compound **4** resemble the corresponding data for other 1,3-indanedione-derived DA cyclopropanes [26–28]. For copies of spectra, see Supplementary Materials.

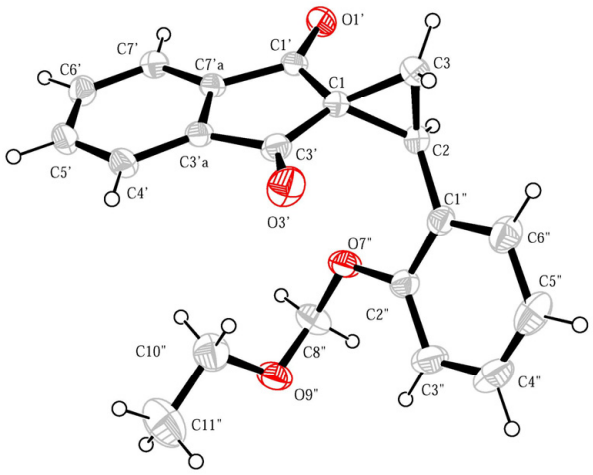


Figure 1. Molecular structure (ORTEP-3) from single-crystal X-ray study of **4**.

3. Materials and Methods

NMR spectra were acquired on a Bruker Avance 400 spectrometer (Bruker, Billerica, MA, USA) at room temperature; the chemical shifts δ were measured in ppm with respect to the solvent (^{1}H: $CDCl_3$, δ = 7.26 ppm; ^{13}C: $CDCl_3$, δ = 77.00). The splitting patterns were designated as s, singlet; d, doublet; m, multiplet; dd, double doublet; br., broad. The coupling constants (*J*) were in Hertz. Infrared spectra were recorded on an Infralum FT-801 spectrometer (Simex, Novosibirsk, Russian Federation) and a Thermo Nicolet IR-200 FT-IR spectrometer (Thermo Fisher Scientific, Waltham, MA, USA). High resolution and accurate mass measurements were carried out using a micrOTOF-Q^{TM} ESI-TOF (Electro Spray Ionization/Time of Flight, Bruker, Billerica, MA, USA). The melting points (m.p.) were determined using a 9100 capillary melting point apparatus (Electrothermal, Stone, UK). Analytical thin layer chromatography (TLC) was carried out with silica gel plates (silica gel 60, F_{254}, supported on aluminum); the revelation was conducted by UV lamp (365 nm). Column chromatography was performed on silica gel 60 (230–400 mesh, Merck, Darmstadt, Germany). All reactions were carried out using freshly distilled and dry solvents. Commercial reagents employed in the synthesis were analytical grade, obtained from Aldrich (St. Louis, MI, USA) or Alfa Aesar (Ward Hill, MO, USA). CCDC 1889833 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html (accessed on 9 September 2019) (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk). Compound **2** was synthesized by the reported procedure [14].

3.1. 2-[2-(Ethoxymethoxy)benzylidene]indene-1,3(2H)-dione (**3**)

To the solution of 2-(ethoxymethoxy)benzaldehyde **2** (2.0 g, 11.1 mmol) and indane-1,3-dione (1.76 g, 12.0 mmol) in benzene (12 mL), piperidine (0.11 mL, 1.1 mmol) and acetic acid (126 μL, 2.2 mmol) were added. The mixture was refluxed with the Dean–Stark trap until water separation was finished (4 h). Upon cooling, the precipitate was formed. It was filtered and recrystallized by the dissolution of product in the minimal quantity of the boiling mixture of petroleum ether and ethyl acetate (4:1) followed by cooling the solution to between 0 and 5 °C. Product **3** was isolated as a brown solid in 68% yield (2.33 g); m.p. = 105–106 °C.

^{1}H NMR ($CDCl_3$, 400 MHz): δ = 1.25 (t, ^3J = 7.1 Hz, 3H, CH_3), 3.80 (q, ^3J = 7.1 Hz, 2H, CH_2), 5.38 (s, 2H, OCH_2O), 7.13–7.16 (m, 1H, Ar), 7.25 (dd, ^3J = 8.3 Hz, ^4J = 0.8 Hz, 1H, Ar), 7.49–7.53 (m, 1H, Ar), 7.80–7.82 (m, 2H, Ar), 8.00–8.03 (m, 2H, Ar), 8.51 (s, 1H, CH=), 8.89 (dd, ^3J = 8.0 Hz, ^4J = 1.6 Hz, 1H, Ar). ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 15.3 (CH_3), 65.0 (CH_2O), 93.7 (OCH_2O), 114.5 (CH), 121.6 (CH), 122.8 (C), 123.3 (CH), 123.4 (CH), 128.7 (C), 134.0 (CH), 135.2 (CH), 135.36 (CH), 135.38 (CH), 140.3 (C), 141.6 (CH), 142.6 (C), 158.8 (C), 189.3 (CO), 190.7 (CO). IR (KBr): ν = 3088, 2985, 2908, 1726, 1687, 1608, 1582, 1480,

1459, 1373, 1347, 1216, 1199, 1171, 1150, 1114, 1075, 1017, 974 cm^{-1}. HRMS ESI-TOF: m/z = 309.1126 $[M + H]^+$ (309.1121 calculated for $C_{19}H_{17}O_4^+$).

*3.2. 2-[2-(Ethoxymethoxy)phenyl]spiro[cyclopropane-1,2′-indene]-1′,3′-dione (**4**)*

To the suspension of NaH (60% suspension in mineral oil, 62 mg, 1.55 mmol) in dry DMF (2 mL), trimethylsulfoxonium iodide (341 mg, 1.55 mmol) was added in a single portion under argon atmosphere at room temperature. The reaction mixture was stirred for 45 min. The obtained solution was added dropwise to the solution of alkene **3** (400 mg, 1.30 mmol) in DMF (4 mL) at 0 °C under argon atmosphere. The reaction mixture was stirred at 0 °C for 50 min and poured into the mixture of the saturated aq. NH_4Cl solution and ice (10 mL). The product was extracted with ethyl acetate (5 × 5 mL) and the combined organic layers were dried with Na_2SO_4. The solvent was evaporated under the reduced pressure. The residue was purified by column chromatography on silica gel using the mixture of petroleum ether and ethyl acetate (1:1) as an eluent. Cyclopropane 4 was isolated as yellow crystals in 73% yield (305 mg). Rf = 0.44 (petroleum ether-ethyl acetate, 4:1); m.p. = 85–86 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 0.93 (t, ^3J = 7.3 Hz, 3H, CH_3), 2.30 (dd, ^2J = 4.2 Hz, ^3J = 8.7 Hz, 1H, CH_2), 2.37 (dd, ^2J = 4.2 Hz, ^3J = 9.0 Hz, 1H, CH_2), 3.11 (dq, ^2J = 9.6 Hz, ^3J = 7.3 Hz, 1H, CH_2), 3.17 (dq, ^2J = 9.6 Hz, ^3J = 7.3 Hz, 1H, CH_2), 3.31 (dd, ^3J = 9.0 Hz, ^3J = 8.7 Hz, 1H, CH_2), 4.76 (d, ^2J = 7.0 Hz, 1H, OCH_2O), 4.86 (d, ^2J = 7.0 Hz, 1H, OCH_2O), 6.98 (br. d, ^3J = 8.3 Hz, 1H, Ar); 7.01–7.04 (m, 1H, Ar), 7.22–7.26 (m, 1H, Ar), 7.36 (br. d, ^3J = 7.3 Hz, 1H, Ar); 7.72–7.80 (m, 3H Ar), 7.99 (br. d, ^3J = 7.4 Hz, 1H, Ar). ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 14.9 (CH_3), 21.7 (CH_2), 36.9 (CH), 41.7 (CH), 63.8 (CH_2O), 92.7 (OCH_2O), 113.1 (CH), 121.3 (CH), 122.2 (CH), 122.4 (CH), 123.6 (C), 129.3 (CH), 130.0 (CH), 134.4 (CH), 134.6 (CH), 141.7 (C), 142.7 (C), 156.3 (C), 195.7 (CO), 198.9 (CO). IR (KBr): ν = 2970, 2904, 2864, 1732, 1703, 1599, 1491, 1454, 1429, 1379, 1335, 1313, 1234, 1163, 1120, 1097, 1051, 995 cm^{-1}. HRMS ESI-TOF: m/z = 323.1277 $[M + H]^+$. (323.1278 calculated for $C_{20}H_{19}O_4^+$). Crystal Data for $C_{20}H_{18}O_4$ (M = 322.34 g/mol): triclinic, space group P-1 (no. 2), a = 7.9274(4) Å, b = 8.4765(4) Å, c = 14.3237(8) Å, α = 77.297(4)°, β = 75.805(4)°, γ = 63.850(4)°, V = 830.66(8) $Å^3$, Z = 2, T = 295 K, μ(CuKα) = 0.729 mm^{-1}, D_{calc} = 1.289 g/cm^3, 8,517 reflections measured (3.930° ≤ Θ ≤ 70.282°), 2988 unique (R_{int} = 0.0315, R_{sigma} = 0.0252), which were used in all calculations. The final R1 was 0.0355 (I > 2σ (I)) and wR^2 was 0.0989 (all data).

4. Conclusions

Here, we have described the synthesis of donor–acceptor cyclopropane, which may participate in various domino reactions due to the presence of the 1,3-indanedione moiety as an acceptor group, the effect of spiro activation, as well as the possible participation of an additional functionality in the donor substituent. The reactivity of this substrate is under investigation.

Supplementary Materials: The following supporting information can be downloaded. Figure S1: ^{1}H NMR ($CDCl_3$, 400 MHz) spectrum of compound **3**; Figure S2: ^{13}C NMR ($CDCl_3$, 100 MHz) spectrum of compound **3**; Figure S3: HRMS spectrum of compound **3**; Figure S4: IR spectrum of compound **3**; Figure S5: ^{1}H NMR ($CDCl_3$, 400 MHz) of compound **4**; Figure S6: ^{13}C NMR ($CDCl_3$, 100 MHz) of compound **4**; Figure S7: HRMS spectrum of compound **4**; Figure S8: IR spectrum of **4**.

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