

Short Note

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(7aR*,7bR*)-7a,7b-Dihydro-15H-dibenzo[f,f']cyclopenta[1,2-b:5,4-b']dichromene

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Short Note

(7aR*,7bR*)-7a,7b-Dihydro-15H-dibenzo[*f,f'*]cyclopenta[1,2-*b:5,4-b'*]dichromene

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Abstract

The reaction of a 2-naphthol-derived Mannich base with the push–pull 5-morpholinopenta-2,4-dienal under acidic conditions unexpectedly afforded (7aR*,7bR*)-7a,7b-dihydro-15H-dibenzo[*f,f'*]cyclopenta[1,2-*b:5,4-b'*]dichromene. The structure of this product was unambiguously confirmed by NMR spectroscopy and X-ray diffraction analysis. A plausible mechanism involves the in situ generation of 1,2-naphthoquinone-1-methide, followed by a [4 + 2] cycloaddition and a subsequent interrupted iso-Nazarov cyclization. In this process, the enol tautomer of the resulting fused cyclopentenone is trapped by a second equivalent of the 1,2-naphthoquinone-1-methide, leading to the observed polycyclic framework.

Keywords: chromenes; push-pull dienals; Diels-Alder reaction; interrupted iso-Nazarov reaction; cascade reaction; *ortho*-quinone methides; 2-naphthol Mannich bases

1. Introduction

4*H*-Chromenes and their benzo analogues are privileged structural motifs, fragments of which are present in a large number of natural compounds, pharmaceuticals, and functional organic materials [1–6]. Of particular interest are electron-deficient 4*H*-chromenes bearing an electron-withdrawing group at the β-position relative to the pyran oxygen atom [7]. The high degree of polarization of the double bond in the pyran ring makes it susceptible to attack by nucleophiles, ambiphilic reagents, and 1,3-dipoles, making such heterocycles valuable substrates for the Michael reaction and 1,3-dipolar cycloaddition. Finally, a large number of compounds with proven antitumor activity have been found among this type of chromenes. For example, crolibulin is a low-molecular-weight inhibitor of tubulin polymerization with an antineoplastic effect and an acceptable side effect profile [8]. Chromeceptin selectively reduces the viability and growth of hepatocellular carcinoma cells overexpressing IGF2 (insulin-like growth factor-2) and binds multifunctional protein 2 (MFP-2), which is involved in peroxisomal oxidation [9]. Compound MX58151 showed growth inhibition in highly drug-resistant MES-SA/DX-5 tumor cells [10]. Chromene LY294002 is a selective, cell-permeable, potent, and specific inhibitor of phosphatidylinositol 3-kinase PI3K [11]. Increased levels of PI3K products were observed in colorectal tumors and breast cancer. Additional examples of push-pull chromenes with anticancer activity include compounds SP-6-27 [12], LY290181 [13], and HA 14-1 [14] (Figure 1).

One of the effective approaches to the synthesis of electron-deficient 4*H*-chromenes is the Diels-Alder reaction between *ortho*-quinone methides (*o*-QM) [15] and push-pull olefins, for which we have previously studied a number of enaminketones [16], enaminals [17], nitroenamines [18] and some others [19].

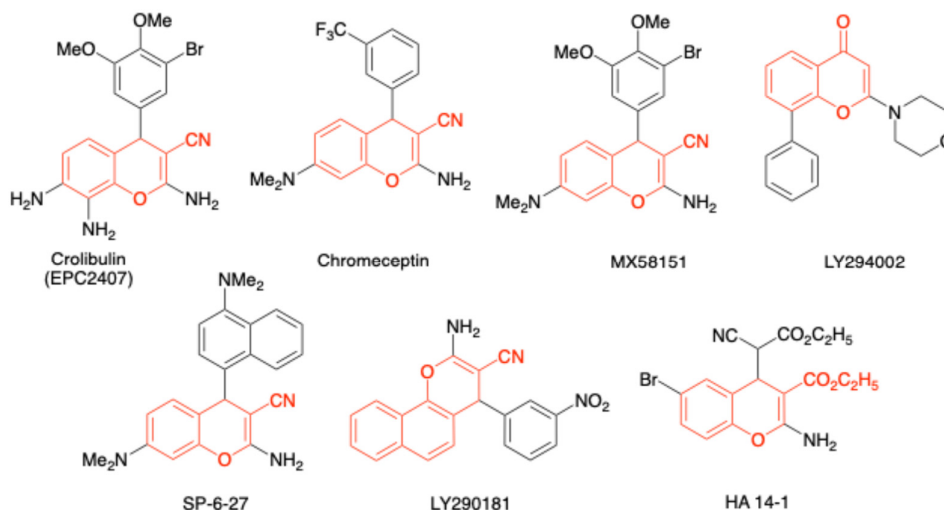
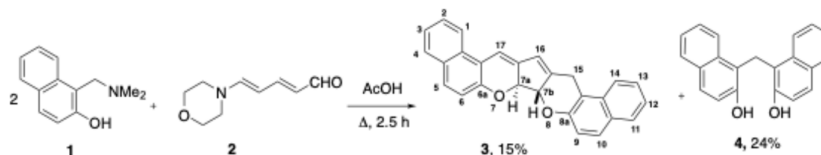


Figure 1. Highly polarized 4H-chromenes with anticancer activity.

2. Results and Discussion

2.1. Synthesis and Spectroscopy

Continuing our research into developing methods for the preparation of electron-deficient 4H-chromenes [16–19], we studied the interaction of 2-naphthol Mannich base **1** with push-pull dienal **2** (Zincke aldehyde), which contains a donor (morpholine fragment) and an acceptor (aldehyde) group at opposite ends of the conjugated 1,3-diene system. This resulted in a complex mixture of products, from which dichromene **3** and the literature-described bis(2-hydroxy-1-naphthyl)methane **4** were isolated in pure form and identified (Scheme 1). The reaction was carried out by refluxing compounds **1** and **2** in a 2:1 ratio in acetic acid for 2.5 h. The products were isolated by column chromatography on silica gel.



Scheme 1. Reaction of Mannich base **1** with Zincke aldehyde **2**.

In the ¹H NMR spectrum of compound **3**, the diastereotopic protons of the methylene group (H-15) appear as two doublet signals at 4.06 and 4.14 ppm with a geminal coupling constant ²J = 20.8 Hz. The protons H-7a and H-7b are observed in the region of 5.08–5.25 ppm, with a spin-spin coupling constant between them of 3.7 Hz. Two singlet signals in the region of 6.60–7.04 ppm correspond to the olefinic protons at positions 16 and 17. In the ¹³C NMR spectrum, the methylene carbon atom resonates at 25.3 ppm. The sp³-hybridized carbon atoms bonded to oxygen (C-7a, C-7b) appear at 81.5 and 83.4 ppm. The most deshielded signals belong to the aromatic carbon atoms attached to oxygen (C-6a, C-8a), which resonate at 151.2 and 152.0 ppm. In the DEPT spectra, the number of carbon atoms directly bonded to protons is consistent with the considered structure.

In addition, the structure of dichromene **3** was confirmed by X-ray diffraction data, according to which the protons at positions 7a and 7b occupy a *trans*-arrangement with respect to each other in the five-membered carbocycle (Figure 2).

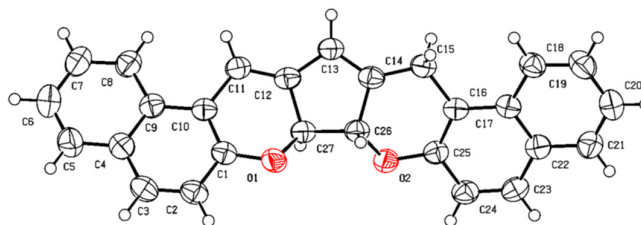
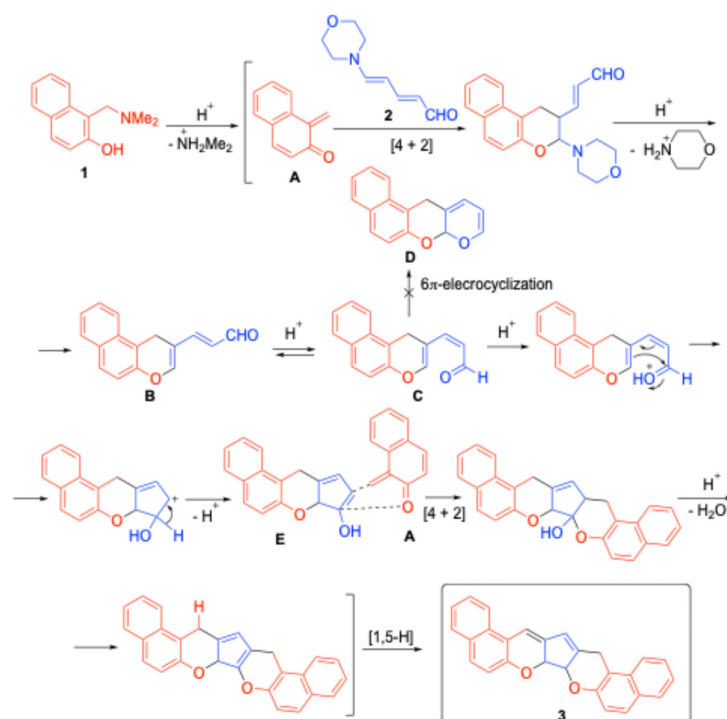


Figure 2. Molecular structure of compound **3** shown with thermal ellipsoids of atomic displacement parameters at the 50% probability level.

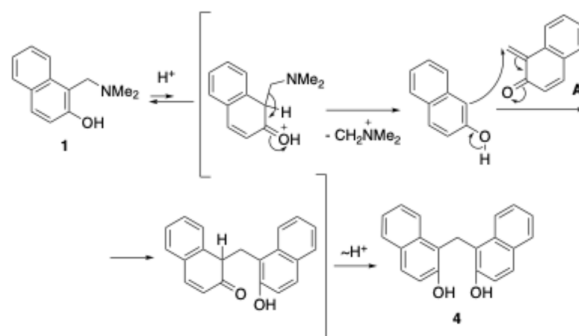
2.2. Proposed Mechanism

We initially hypothesized that the reaction of Mannich base **1** with dienal **2** would yield aldehyde **B** in the *E*-configuration *via* a Diels-Alder reaction between *in situ*-generated 1,2-naphthoquinone 1-methide **A** and the most electron-rich double bond of dienal **2**, followed by elimination of morpholine. Furthermore, due to the lower energy barrier of *E/Z* isomerization, especially in acidic media, we might expect the formation of a cycloaddition product **C** with the *Z*-configuration of the exocyclic double bond, which could isomerize to 7*aH*,12*H*-benzo[*f*]pyrano[2,3-*b*]chromene **D** *via* 6*π*-oxa-electrocyclization [20]. However, this reaction pathway was also not realized. Apparently, the aldehyde undergoes cyclization in an acidic medium to the enol **E**, followed by the addition of another 1 equivalent of *o*-QM **A** to the electron-rich double bond of the enol form of the ketone **E** (the intermolecular interrupted iso-Nazarov reaction) [21–25]. Subsequent dehydration and a 1,5-sigmatropic hydrogen shift lead to the formation of dichromene **3** (Scheme 2).



Scheme 2. Proposed mechanism for the formation of (7*aR**,7*bR**)-7*a*,7*b*-dihydro-15*H*-dibenzo[*f,f'*]cyclopenta[1,2-*b:5,4-b'*]dichromene **3**.

The formation of 1,1'-methylenebis(naphthalen-2-ol) **4** apparently occurs *via* deamination of Mannich base **1** under acidic conditions through a retro-Mannich reaction, followed by addition of the resulting 2-naphthol to *o*-QM **A** (Scheme 3).



Scheme 3. Proposed mechanism for the formation of 1,1'-methylenebis(naphthalen-2-ol) **4**.

3. Materials and Methods

All synthetic manipulations were performed in air. All reagents and solvents were purchased from commercial vendors and used as received. ^1H and ^{13}C (proton-decoupled) NMR spectra (at 400 and 100 MHz, respectively), as well as DEPT-135 spectrum, were registered on a JEOL JNM-ECX400 spectrometer (Japan) in CDCl_3 . Chemical shifts were referenced internally to the residual solvent signal (CDCl_3 : 7.26 ppm for ^1H nuclei, 77.2 ppm for ^{13}C nuclei). Elemental analysis was performed on an automated Euro Vector EA-3000 CHNS analyzer (Italy) using L-cystine as a standard. Melting point was determined by the capillary method on an SRS OptiMelt MPA100 instrument (USA). Reaction progress and purity of the obtained compounds were monitored by TLC on Merck Silica gel 60 F₂₅₄ plates (eluent – CH_2Cl_2). The starting dienal **2** was obtained by a method published previously [26].

3.1. Synthesis and Characterization of (7aR*,7bR*)-7a,7b-dihydro-15H-dibenzo[*f,f'*]cyclopenta[1,2-*b:5,4-b'*]dichromene (**3**)

A solution of 0.36 g (1.8 mmol) of 1-[(dimethylamino)methyl]naphthalen-2-ol **1** and 0.15 g (0.9 mmol) of (2*E*,4*E*)-5-morpholinopenta-2,4-dienal **2** in 10 mL of acetic acid was refluxed for 2.5 h. The mixture was cooled and poured with stirring into 30 mL of ice-cold water. The precipitated solid was filtered off, and the crude product was purified by column chromatography on silica gel (eluent – CH_2Cl_2). For further purification, the product was dissolved in CH_2Cl_2 upon heating, three volumes of methanol were added, and CH_2Cl_2 was evaporated until the onset of product crystallization. The mixture was then kept at $-30\text{ }^\circ\text{C}$ for 2 h. The precipitate was filtered off, washed with ice-cold methanol, and dried in air to afford **3** (50 mg, 15%).

Colorless crystals, mp 243–245 $^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3) δ , ppm: 8.03 (d, $J = 8.2$ Hz, 1H), 7.83–7.78 (m, 3H), 7.690 (d, $J = 8.9$ Hz, 1H), 7.685 (d, $J = 8.9$ Hz, 1H), 7.56–7.49 (m, 2H), 7.42–7.37 (m, 2H), 7.30 (d, $J = 8.9$ Hz, 1H), 7.21 (d, $J = 8.9$ Hz, 1H), 7.04 (s, 1H), 6.60 (br. s, 1H), 5.25 (d, $J = 3.7$ Hz, 1H), 5.08 (dd, $J = 3.7, 2.3$ Hz, 1H), 4.14 (d, $J = 20.8$ Hz, 1H), 4.06 (d, $J = 20.8$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ , ppm: 152.0 (C), 151.2 (C), 142.4 (C), 136.0 (C), 132.6 (C), 130.4 (C), 130.0 (C), 129.5 (C), 128.74 (CH), 128.69 (CH), 128.67 (CH), 128.5 (CH), 126.8 (CH), 126.7 (CH), 125.0 (CH), 124.2 (CH), 123.9 (CH), 122.1 (CH), 121.8 (CH), 119.5 (CH), 118.1 (CH), 117.7 (C), 111.6 (C), 109.6 (CH), 83.4 (CH), 81.5 (CH), 25.3 (CH_2). Calc. for $\text{C}_{27}\text{H}_{18}\text{O}_2$: C 86.61; H 4.85. Found: C 86.56; H 4.82.

3.2. X-Ray Crystallography

X-ray structural analysis of compound **3** was carried out on an Agilent SuperNova diffractometer equipped with a microfocus copper-anode X-ray source and an Atlas S2 CCD detector. Crystals suitable for X-ray diffraction were grown by slow evaporation from a CH_2Cl_2 –MeOH mixture at room temperature. Data collection, as well as determination and refinement of unit-cell parameters, were performed using the CrysAlisPro software package, version 1.171.38.41 [27]. The

structure was solved with ShelXT [28] and refined using ShelXL [29]. Molecular graphics and preparation of the manuscript were performed with the Olex2 software package, version 1.2.10 [30].

Selected crystallographic data: $C_{27}H_{18}O_2$, $M = 374.41$, monoclinic, $a = 6.8568(1)$, $b = 7.6873(1)$, $c = 34.9443(6)$ Å, $\alpha = 90^\circ$, $\beta = 92.552(2)^\circ$, $\gamma = 90^\circ$, $V = 1840.10(5)$ Å³, $T = 293$ K, space group $P2_1/c$, $Z = 4$, $d_{calc} = 1.352$ g/cm³. CCDC 2256754 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/structures.

4. Conclusions

In this work, we described the preparation of a new heterocyclic system, (7a*R**,7b*R**)-7a,7b-dihydro-15*H*-dibenzo[*f,f'*]cyclopenta[1,2-*b:5,4-b'*]dichromene. Structural elucidation by NMR spectroscopy and X-ray diffraction revealed a unique fused heptacyclic framework with *trans*-arrangement of hydrogen atoms in a five-membered carbocycle.

Supplementary Materials: The following supporting information can be downloaded at: Preprints.org, Figure S1: ¹H NMR spectrum of **3**; Figure S2: ¹³C NMR spectrum of **3**; Figure S3: DEPT-135 spectrum of **3**.

Author Contributions: D.V.O.— supervision, writing (original draft, review and editing); O.P.D.— investigation, data analysis; V.A.O.— funding acquisition, conceptualization, supervision, data analysis. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Data are contained within the article and Supplementary Materials.

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Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

NMR	Nuclear Magnetic Resonance
DEPT	Distortionless Enhancement by Polarization Transfer
<i>o</i> -QM	<i>ortho</i> -Quinone Methide

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