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Synthetic Communications

An International Journal for Rapid Communication of Synthetic Organic Chemistry

ISSN: (Print) (Online) Journal homepage: https://www.tandfonline.com/loi/lsyc20

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To cite this article: Vadym Syngaevsky , Andrew Karkhut , Sviatoslav Polovkovych , Andrzej Gzella , Roman Lesyk & Volodymyr Novikov (2020): Study of 1,3-dipolar cycloaddition of amino-acid azomethines and Juglone, Synthetic Communications, DOI: 10.1080/00397911.2020.1795880

To link to this article: https://doi.org/10.1080/00397911.2020.1795880

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https://doi.org/10.1080/00397911.2020.1795880

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Study of 1,3-dipolar cycloaddition of amino-acid azomethines and Juglone

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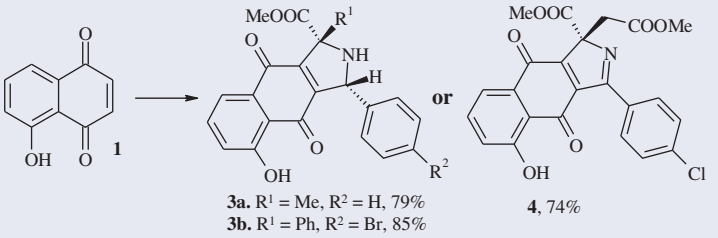
ABSTRACT

Novel 2,3,4,9-tetrahydro- and 4,9-dihydro-1*H*-benzo[*f*]isoindole derivatives were synthesized from Juglone and amino-acid azomethines in 74–85% yields via 1,3-dipolar cycloaddition. The stereo- and regioselectivity of cycloaddition was confirmed by NMR spectra and single-crystal X-ray diffraction analysis.

GRAPHICAL ABSTRACT



ARTICLE HISTORY
Received 9 May 2020

KEYWORDS
13-cycloaddition; amino acids; azomethines; Juglone; X-ray

Introduction

The cycloaddition reactions occupy a prominent place in synthetic organic chemistry. For many years, the reaction of 1,3-dipolar cycloaddition has evolved into a generally applicable method for the synthesis of five-membered heterocyclic rings, since many 1,3-dipoles are readily available and react with a wide variety of dipolarophils.[1–11] Moreover, this approach enables to generate rings containing several adjacent stereocenters in one synthetic operation. The configurations of these new stereocenters are due to the geometry of the dipole and dipolarophile as well as the mechanism of cycloaddition. In recent years, numerous natural and synthetic products have been prepared by synthetic routes *via* 1,3-dipolar cycloadditions.[12–14]

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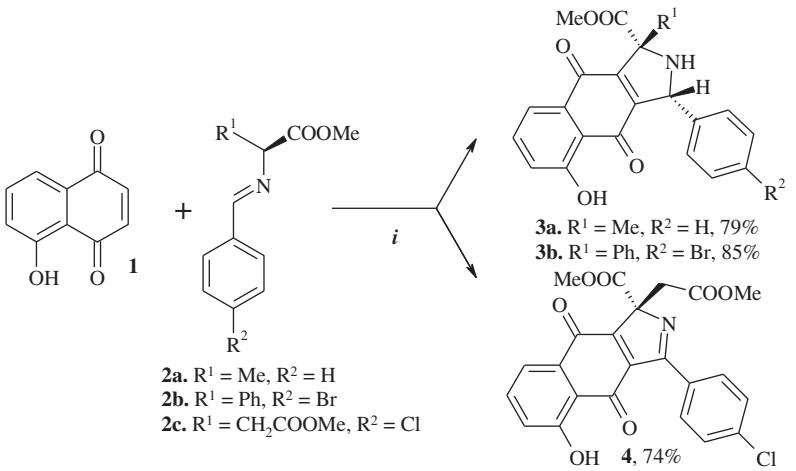


Scheme 1. Reaction of Schiff's bases of amino acids with 5-hydroxy-1,4-naphthoquinone. Reagents and conditions: *i* – appropriate imine (1.0 equiv.), 5-hydroxy-1,4-naphthoquinone (2.0 equiv.), AcOAg (0.1 equiv.), TEA (0.5 equiv.), PPh_3 (0.1 equiv.), toluene, 12–24 h 60 °C/24 h rt.

The cycloaddition of azomethine ylides as 1,3-dipoles with alkenes is a useful tool for the construction of the pyrroline or pyrrolidine ring contained in many biologically active compounds.[15,16] Pharmacologically important pyrrole derivatives possesses antibacterial,[17–19] antifungal,[17] anti-inflammatory,[17,20] hypolipidemic,[21–23] anticancer,[24–26] antiviral[27–31] activity, etc. Pyrrole and pyrrolidine systems are used as effective intermediates in the synthesis of natural alkaloids[32,33] and synthetic nitrogen-containing heterocycles.[34] In addition, the issue of diastereo- and enantioselectivity of such processes remain key for further research, especially for the design of potential biologically active compounds.[10,34–41] The high activity of 1,4-naphthoquinones (as the synthetic analogs of electron-deficient alkenes) is explained by the strong electrophilic nature of the C=C bond due to the electron-withdrawing effects of the adjacent carbonyl groups.[10,11,39,41] Its global electrophilicity $\omega = 3.34$ eV makes it an active dipolarophile in the reaction of [3 + 2]-cycloaddition.

Results and discussion

This paper covers the reactions of azomethines (Schiff's bases) of amino acids (**2a**–c) with the cyclic dipolarophile 5-hydroxy-1,4-naphthoquinone (Juglone). Azomethines **2a**–c were obtained with 82–90% yields and sufficient purity *via* reaction of amino acid methyl esters and aromatic aldehydes in dichloromethane in the presence of magnesium sulfate according to the known method.[42] Heating the imines (**2a**–c) with Juglone in toluene for varying periods of time gave high yields of substituted pyrrolines **3a**,**b** and **4** (Scheme 1).

According to the ^{1}H NMR spectra and X-ray data (ESI) mentioned cycloadditions were regio- and diastereoselective. Juglone has a strong hydrogen bond between the adjacent hydroxyl and carbonyl groups. Therefore, the hydroxyl substituent acts as electron acceptor and the LUMO is highly polarized with the larger coefficient at the C2-position.[43] It explains the regioselectivity of the process. In turn, the diastereoselectivity of this reaction may be due to the use of silver acetate as a catalyst.[35,36,44–47]

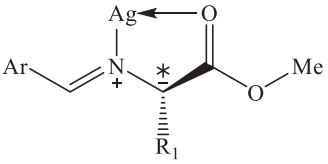
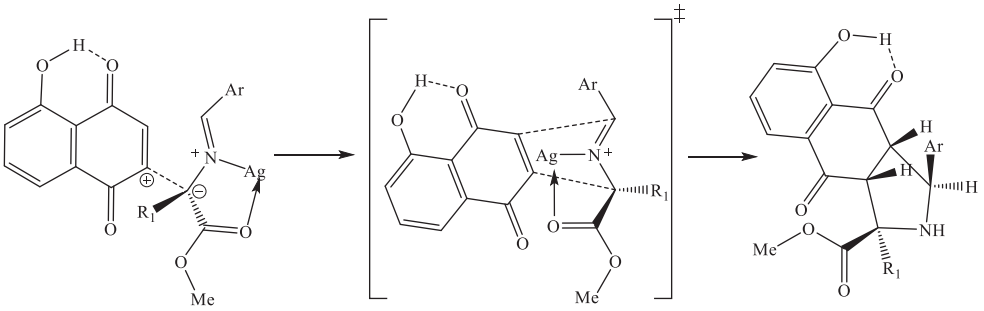


Figure 1. Chelating silver complex of azomethine ylides.



Scheme 2. Plausible mechanism of 1,3-dipolar cycloaddition of amino-acid azomethines and Juglone.

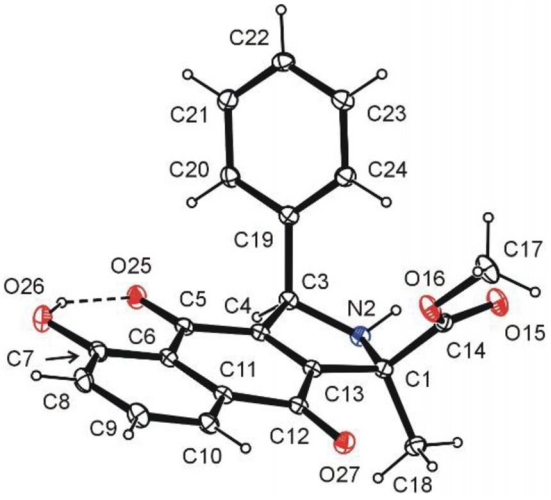


Figure 2. ORTEP view of **3a** showing the atomic labeling scheme. Non-H atoms are drawn as 30% probability displacement ellipsoids and H atoms are drawn as spheres of an arbitrary radius.

Thus, it is known that the use of monovalent metal salts as catalysts leads to the formation of exclusively *cis*-adducts.[35] This is possible due to the stereocoordination of the reactive ylide in the transition state. The reaction between the chelating complexes of azomethine ylides (Figure 1) begins with the approximation of the most electrophilic and nucleophilic sites of the reacting molecules, namely, the C2 atom of the activated C=C bond of Juglone and the α-Carbon atom of azomethine ylide, which is consistent with the possible canonical forms of azomethine ylides (Scheme 2). Mechanistically, the dehydrogenation to compounds **3a**,**b** and **4** could be proposed *via* tautomeric aromatization or enolization of intermediates followed by the oxidation by the excess of Juglone or by air.[47–52]

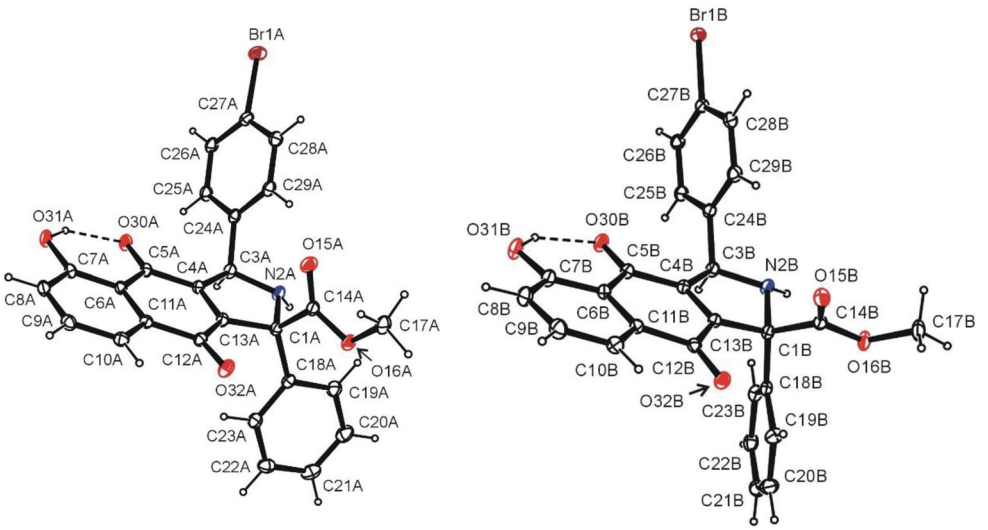


Figure 3. ORTEP view of two symmetry-independent molecules of 3b, showing the atomic labeling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are depicted as small spheres of an arbitrary radius.

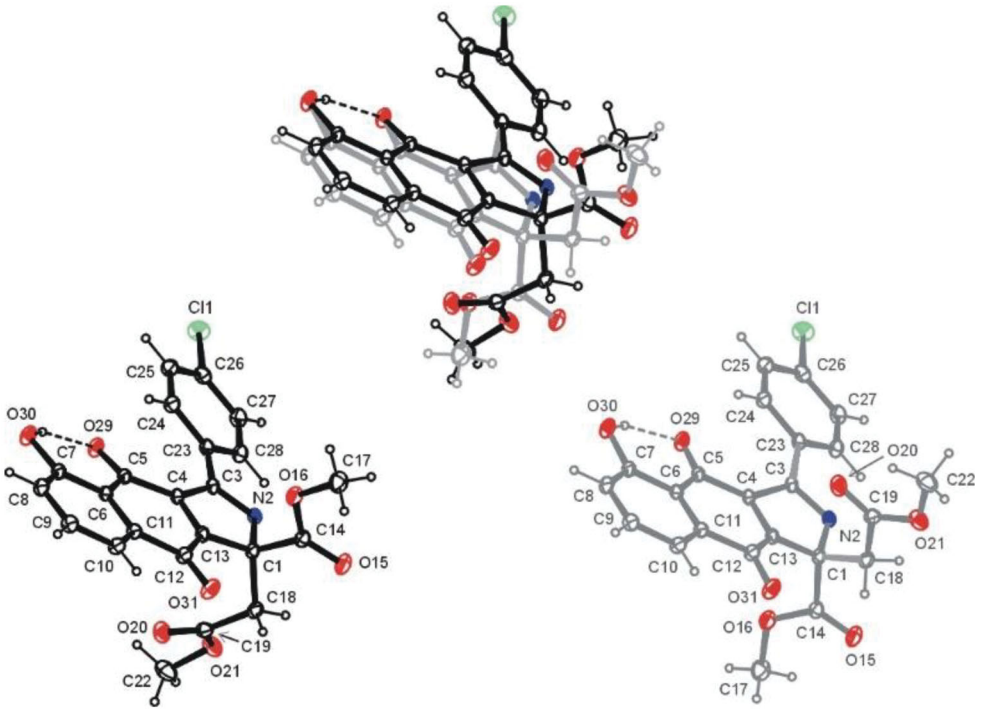


Figure 4. ORTEP view of 4. Non-H atoms are drawn as 30% probability displacement ellipsoids, and H atoms are drawn as spheres of an arbitrary radius. The disordered part of the molecule is colored gray. Two enantiomers are presented separately.

Structural features of **3a**,**b** and **4** were confirmed by X-ray diffraction study (ESI). The ORTEP drawing of **3a**,**b** structures and atomic numberings are shown in Figures 2 and 3. Compound **3a** crystallizes in the space group P-1 (DMF) with one molecule in the asymmetric unit while compound **3 b** crystallizes in the space group P21/c (AcOMe) with two symmetry independent molecules in the asymmetric unit, denoted A and B (Figure 3), which differ to a rather moderate extent in conformation. The differences concern primarily the angular arrangement of the substituents present in the C1 and C3 positions of the 4,9-dioxo-2,3,4,9-tetrahydro-*1H*-benzo[*f*]isoindole system.

The spatial geometry of the molecule of **4** is presented in Figure 4. Structural investigations have shown that in the crystal, most atoms of the molecule occupy two alternative positions marked a and b. The positions of atoms are related to two alternative enantiomeric forms, in which the methoxycarbonyl and methoxycarbonylmethyl groups are located at C1 stereogenic center on the opposite sites (Figure 4).

Conclusions

In summary, we have successfully developed an efficient regio- and diastereoselective method for the construction of 1*H*-benzo[*f*]isoindole derivatives via 1,3-dipolar cycloaddition of amino-acid azomethines and Juglone.

Experimental

All the chemicals were purchased from Aldrich Chemical Company (USA) and were used without further purification. Melting points (m.p.) were determined using an SRS-EZMelt automated melting point instrument without correction. The elemental analyses were performed using the Perkin–Elmer 2400 CHN analyzer. The ^{1}H and ^{13}C NMR spectra were recorded on AVANCE (Bruker) spectrometer (^{1}H at 400 and ^{13}C at 100 MHz) instrument in $CDCl_3$. Chemical shifts (δ) are given in ppm units relative to tetramethylsilane as reference (0.00). HRMS spectra were obtained on an MicrOTOF-Q III (Bruker) spectrometer. The purity of all obtained compounds was checked by TLC on Silufol-254 plates (eluent hexane/EtOAc 1:1).

General procedure of 1,3-cycloaddition affording 3a,b and 4

AcOAg (0.032 mg, 0.19 mmol) and PPh_3 (0.049 mg, 0.19 mmol) were dissolved in toluene (2 mL) and stirred at room temperature for about 1 h Under argon atmosphere. Then, imine substrate (1.93 mmol), and Juglone (3.86 mmol) were added sequentially, after that the mixture was cooled to 0 °C and TEA (0.091 mg, 0.095 mmol) was added. Then it was heated to 60 °C and maintained for 24 h. Once starting material was consumed (monitored by TLC), the resulting mixture was evaporated in a vacuum. The mixture was purified by chromatography on SiO_2 hexanes/EtOAc gradient elution from 1:99 to 50:50 to give target product.

rel-(1R,3R)-5-Hydroxy-1-methyl-4,9-dioxo-3-phenyl-2,3,4,9-tetrahydro-1H-benzo[f]isoindole-1-carboxylic acid methyl ester (**3a**). Yield 79%, red-brown plate crystal, mp 143-145 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ, ppm: 11.71 (s, 1H, OH); 7.64 (dd, 1H, J_1 = 7.6 Hz, J_2 = 1.5 Hz, CH); 7.60 (*t*, 1H, $J = 7.9 \times 2$ Hz, CH); 7.44–7.40 (m, 2H, 2CH);

7.38-7.27 (m, 4H, 3CH, 1NH); 7.22 (dd, 1H, J_1 = 8.2 Hz, J_2 = 1.5 Hz, CH); 5.66 (s, 1H, CH); 3.78 (s, 3H, OCH_3), 1.78 (s, 3H, CH_3). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 24.6, 52.9, 66.5, 71.2, 115.9, 119.4, 124.9, 127.7, 128.3, 128.8, 133.2, 136.5, 141.1, 148.9, 149.9, 161.8, 172.9, 181.1, 187.6. HRMS (ESI): calcd. for $C_{21}H_{18}NO_5$ $[M+H]^+$ 364.1179; found: 364.1175. Anal. calcd for $C_{21}H_{17}NO_5$: C, 69.41, H, 4.72, N, 3.85. Found: C, 69,50; H, 4.80; N, 3.70.

X-ray diffraction studies

Crystallographic data for 3a

Empirical formula: $C_{21}H_{17}NO_5$, formula weight 363.36, triclinic, space group *P*-1, $a = 7.3424(4)$, $b = 8.1185(3)$, $c = 14.4387(6)$ Å, $\alpha = 101.308(3)$, $\beta = 93.859(4)$, $\gamma = 94.273(4)^\circ$, $V = 838.67(6)$ Å^3, $Z = 2$, D_{calc} = 1.439 g/cm^3, μ = 0.857 mm^{-1}, $T = 130.0(1)$ K. A red-brown plate crystal (DMF) of 0.25 × 0.20 × 0.05 mm was used to record 16180 (Cu K*a*-radiation, $\theta_{max} = 75.96^\circ$) intensities on a Rigaku SuperNova Dual Atlas diffractometer using mirror monochromatized Cu Kα radiation from a high-flux microfocus source ($\lambda = 1.54178$ Å). The supplementary crystallographic data of **3a** have been deposited at the Cambridge Crystallographic Data Center (CCDC) as a supplementary publication CCDC 1900363. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union ROAD, Cambridge CB2 1EZ (UK) (fax: +44-(0)1223-336033 or e-mail: deposit@ccdc.cam.ac.uk).

Crystallographic data for 3b

Empirical formula: $C_{26}H_{18}BrNO_5$, formula weight 504.32, monoclinic, space group $P2_1/c$, $a = 19.35586(17)$, $b = 16.30452(16)$, $c = 13.34255(10)$ Å, $\beta = 91.0173(7)^\circ$, $V = 4210.08(6)$ Å^3, $Z = 8$, D_{calc} = 1.591 g/cm^3, μ = 3.010 mm^{-1}, T =130.0(1) K. A red lath crystal (AcOEt) of 0.22 × 0.14 × 0.05 mm was used to record 44382 (Cu K*a*-radiation, $\theta_{max} = 76.55^\circ$) intensities on a Rigaku SuperNova Dual Atlas diffractometer using mirror monochromatized Cu Kα radiation from a high-flux microfocus source ($\lambda = 1.54178$ Å). The supplementary crystallographic data of **3b** have been deposited at the Cambridge Crystallographic Data Center (CCDC) as a supplementary publication CCDC 1900364. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union ROAD, Cambridge CB2 1EZ (UK) (fax: +44-(0)1223-336033 or e-mail: deposit@ccdc.cam.ac.uk).

Crystallographic data for **4**. Empirical formula: $C_{23}H_{16}ClNO_7$, formula weight 453.82, triclinic, space group *P*-1, $a = 7.8945(3)$, $b = 10.6545(4)$, $c = 13.0942(5)$ Å, $\alpha = 70.282(4)$, $\beta = 81.576(3)$, $\gamma = 74.872(4)^\circ$, V = 998.80(7) Å^3, $Z = 2$, D_{calc} = 1.509 g/cm^3, μ = 2.126 mm^{-1}, T =130.0(1) K. A red lath crystal (AcOEt) of 0.20 × 0.15 × 0.05 mm was used to record 18505 (Cu K*a*-radiation, $\theta_{max} = 76.38^\circ$) intensities on a Rigaku SuperNova Dual Atlas diffractometer using mirror monochromatized Cu Kα radiation from a high-flux microfocus source ($\lambda = 1.54178$ Å). The supplementary crystallographic data of **4** have been deposited at the Cambridge Crystallographic Data Center (CCDC) as a supplementary publication CCDC 1900365. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union ROAD, Cambridge CB2 1EZ (UK) (fax: +44-(0)1223-336033 or e-mail: deposit@ccdc.cam.ac.uk).

Supplementary information

Supplementary data (spectral and analytical data for compounds **3a**,**b** and **4**) associated with this article can be found, in the online version, at http://dx.doi.org/10.1080/00397911.2020.1795880.

Acknowledgments

We would like to express the warmest appreciation to Erasmus + program aims to support actions in the fields of Education, Training, Youth and Sport for the period 2014–2020 and to Professor Arnaud Martel (Le Mans University, France) for support and assistance in conducting research.

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