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Rhodium(II) catalysed decomposition of 3-diazo-4-hydroxycoumarin An easy access to furocoumarin ring system

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Abstract

Several furo[3,2-*c*]coumarins are produced efficiently and regioselectively by a formal [3+2] cycloaddition of 3-diazo-4-hydroxycoumarin and oxygenated alkenes, presumably via the intermediacy of rhodium carbenoid. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Rhodium(II); Furocoumarin; α-Diazocarbonyl

1. Introduction

Metal induced diazo compound decomposition, reported since 1906 [1], is a convenient method to generate transient carbenoid species; these react with a large variety of functional groups, giving either addition to multiple bonds or insertion into single bonds, making this protocol an useful tool in synthetic organic chemistry [2–9].

α-Diazocarbonyl compounds extend the preparative purposes of this reaction; the functional group α to the electrophilic carbenoid centre allows further evolution of the initial addition/insertion products ultimately giving interesting ring systems [10–13]; the larger (thermal) stability of α-diazocarbonyls with respect to unfunctionalized ones is another advantage.

A great number of catalytic systems based on transition metal derivatives have been investigated for the diazo-decomposition; between them rhodium(II), and particularly $Rh_2(OAc)_4$, is the most effective to generate reactive metal-carbenoid species.

In a precedent work [14], we explored the chemistry of the carbenoid system derived from Rh(II)-decomposition of 3-diazo-4-hydroxycoumarin **1** in cyclic ethers, obtaining coumarin-fused crown ethers. We report here a facile and efficient construction method of fused furocoumarins by the use of a Rh(II)-induced [3+2] cycloaddition of **1** and oxygenated alkenes **3** (Eq. (1)). Since many naturally derived coumarins embody furo[3,2-*c*]coumarin ring

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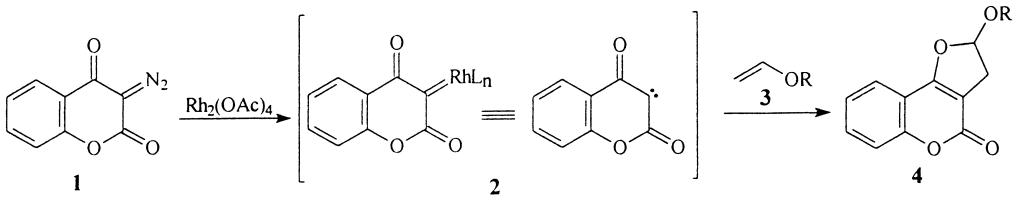
system as a common structural unit, our annulation method has considerable synthetic utility for a variety of naturally occurring compounds [15]. [1]

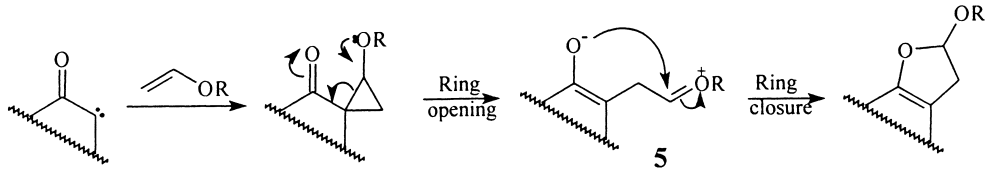


(1)

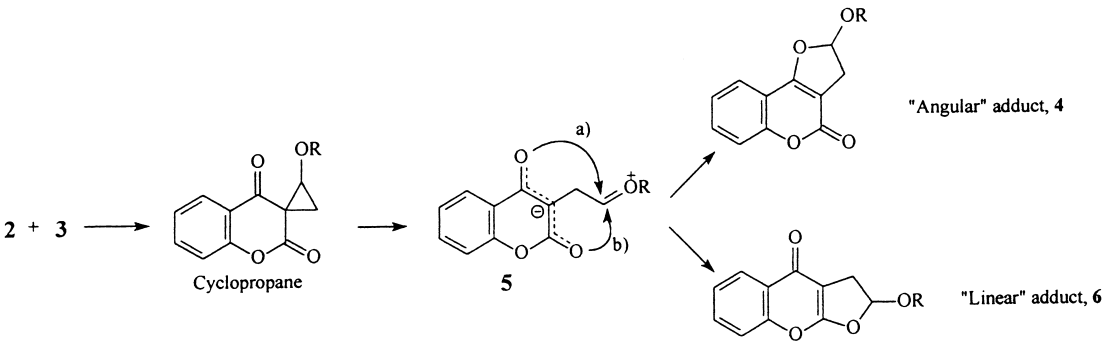
As reported previously [11,16], α-carbonylcarbenes react with vinyl ethers, giving either cyclopropanes (via [2+1]-cycloaddition) or dihydrofurans. The most probable mechanism, as proposed by Pirrung and Blume [11], involves an initial cycloaddition of metal-carbenoid to vinyl ether, giving an alkoxycyclopropane. Under these reaction conditions, cyclopropane ring opening follows to give dipolar intermediate **5**. Subsequent ring closure by intramolecular nucleophilic attack by the oxygen atom leads to the dihydrofuran system (Eq. (2)).

Fairly unstable spiro-fused cyclopropanes are seldom isolated, depending on structural features. For example, diazo compounds stabilised by ester groups give mainly cyclopropanes, while α-diazoketones provide almost exclusively dihydrofurans. This behaviour is generally ascribed, in the dipolar pathway, to the nucleophilic character of the oxygen atom, greater in ketones than in esters.

The rhodium-catalysed decomposition of 3-diazo-4-hydroxycoumarin with vinyl ethers is particularly representative: the molecule shows a doubly stabilised diazo-group, endowing either a ketone and a lactone moiety (Eq. (3)).



(2)



(3)

Assuming the initial formation of the cyclopropane and subsequent ring opening a dipolar intermediate **5** is formed, in which the negative charge is delocalised on the β-dicarbonyl system, involving either O–C(2) or O–C(4). Ring closure to dihydrofuran may occur

[1] Lee and coworkers have reported on the addition of a number of unsaturated compounds to diazocoumarin to give the corresponding five-membered heterocycles. Upon engaging in this chemistry, we became aware they had conducted a preliminary investigation into the addition of vinyl ethers (two examples).

via nucleophilic attack to oxonium ion by both oxygen atoms, leading to an 'angular' (**4**) and a 'linear' adduct (**6**), in our study only 'angular' products were isolated in good yields. According to the mechanism proposed, the products ensue from intramolecular attack by oxygen atom on C(4), as expected from its nucleophilic character, higher than O–C(2).

2. Results

The protocol has been applied to many vinyl ethers (including furans) and esters, producing the 'angular' adducts, in one case only accompanied by an insertion product. The reaction was tipically conducted by refluxing **1** with the metal catalyst in the vinyl compound as reagent-solvent. Many transition metal based catalysts were scrutinised in order to optimise yields and rates. Among them copper powder, metal salts [e.g. $Pd(OAc)_2$ and CuOTf] and metal complexes [e.g. $Rh_2(OAc)_4$, $Cu(acac)_2$, $PdCl_2(CH_3CN)_2$, $PdCl_2(PhCN)_2$, $Rh(PPh_3)_3Cl$, $Ru(PPh_3)_3Cl_2$ and $Pd(PPh_3)_2Cl_2$]. The best catalyst was found to be $Rh_2(OAc)_4$, the other complexes being less efficient and with copper powder or salts nearly or at all ineffective in diazo-decomposition. The choice of the experimental conditions was made on the reaction with *t*-butyl vinyl ether. The optimal catalyst $Rh_2(OAc)_4$, initially tried at 10% molar amount, was subsequently decreased up to 3% without affecting the yield or the rate. Lower ratios slowed or stopped at all the diazo-decomposition. The solvent was not scrutinised being directly involved. With low-boiling ethers (ethyl vinyl ether, methyl isopropenyl ether, furan) the refluxing temperature was not high enough to trigger the decomposition; the reaction was then run in a steel bomb under N_2 pressure ($\sim$10 atm). With benzofuran a co-solvent was needed and fluorobenzene [11] was used although the carbenoid system was known to react slowly with it. Along with traces of the undesired insertion product with fluorobenzene [i.e. 3-(4-fluorophenyl)-4-hydroxycoumarin], the adduct with benzofuran was uneventfully isolated. Conversely, the use of other aromatic solvents as well as alcohols under the same conditions induce chemoselective insertion to afford 3-aryl- and 3-alkoxy-4-hydroxycoumarins, respectively [17]. The time required for completion of the reaction was typically in the range 2–3 h for 'atmospheric' reactions (Method A) and 4–5 h for experiments under nitrogen pressure (Method B). The results are reported in Table 1.

In most cases, cycloadditions occurred in good to excellent yields, with the exception of benzofuran **3l**, which furnished **4l** in low yield. Accordingly, the reaction products obtained were substantially pure and the work-up was simple, requiring only evaporation of the reagent–solvent and a short chromatographic purification. In the case of methoxydihydropyran **3i**, both diastereomeric adducts **4i**/**a** and **4i**/**b** were obtained. A similar result was observed with 2-methylfuran (silvan), where two regioisomeric adducts **4k**/**a**–**b** were isolated accompanied by **4k**–**c**, arising from formal carbenoid insertion into heterocyclic α'-position.

The reaction described above provides another useful procedure for introducing regioselectively a furan subunit onto coumarin skeleton. Work is underway to obtain diversely functionalised furocoumarins and to apply it in the synthesis of natural products and analogues of biological interest.

3. Experimental

3-Diazo-4-hydroxycoumarin **1** was prepared from 4-hydroxycoumarin accordingly to Taber et al. [18]. Dirhodium(II) tetraacetate was purchased from Fluka. Fluorobenzene and all vinyl ethers were purchased from Aldrich and used without purification. Chromatographies were performed on silica gel (230–400 mesh). ^1H and ^{13}C NMR were recorded with Bruker AC200 or Bruker DRX300 instruments. Mass spectra were obtained from a VG7070EQ spectrometer.

3.1. Procedure A

Compound **1** (1 mmol) is dissolved in the vinyl compound (5 ml or 5 mmol + co-solvent for **4l**) and $Rh_2(OAc)_4$ (0.03 mmol) is added. The mixture is brought to reflux for 2–3 h, checking by TLC (CH_2Cl_2 eluent) the reaction progress. Solvents are removed by rotary evaporation and the residue is submitted to flash chromatography (CH_2Cl_2 eluent), obtaining pure furo[3,2-*c*]coumarins.

Table 1

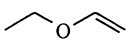
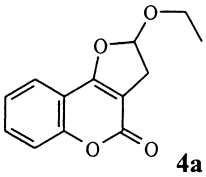
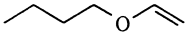
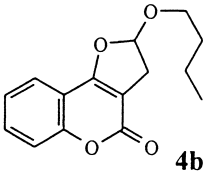
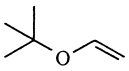
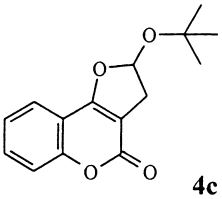
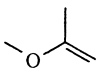
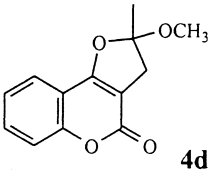
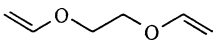
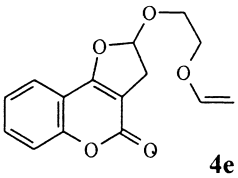
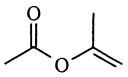
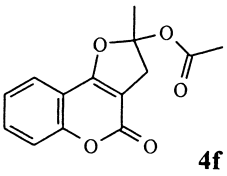
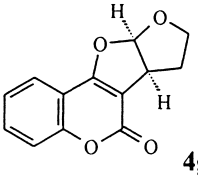
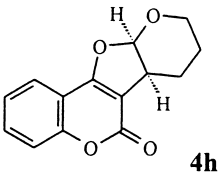
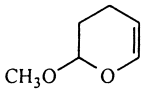
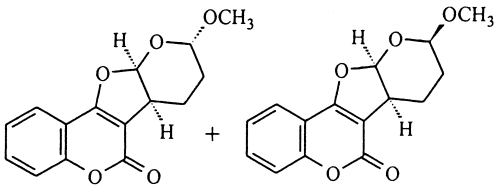
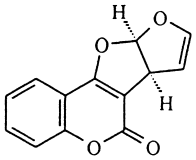
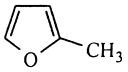
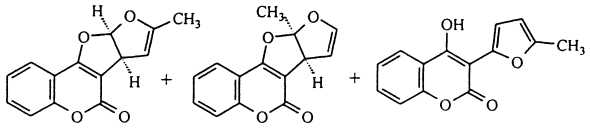
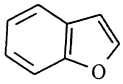
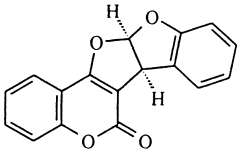
Vinyl compound	Product	Yield (%)	Method
 **3a**	 **4a**	73	B
 **3b**	 **4b**	75	A
 **3c**	 **4c**	70	A
 **3d**	 **4d**	47	B
 **3e**	 **4e**	86	A
 **3f**	 **4f**	67	A
 **3g**	 **4g**	78	A
 **3h**	 **4h**	61	A

Table 1 (*Continued*)

Vinyl compound	Product	Yield (%)	Method
 **3i**	 **4i/a** **4i/b**	78[a]	A
 **3j**	 **4j**	58	B
 **3k**	 **4k/a** **4k/b** **4k/c**	62[b]	A
 **3l**	 **4l**	27	A[c]

[a] Combined yield; diastereomeric ratio 1.75:1.0.
[b] Combined yield; ratio 1.0:1.1:2.0.
[c] Fluorobenzene added as a co-solvent.

3.2. Procedure B

Compound **1** (1 mmol) is dissolved in the vinyl compound (5 ml) and $Rh_2(OAc)_4$ (0.03 mmol) is added. The mixture is poured in a glass vessel and the latter introduced in a N_2-purged steel-bomb. The bomb is pressurised with 10 atm N_2 and then heated to 100°C for 4–5 h. The reaction mixture is then evaporated to dryness and the residue flash-chromatographed (CH_2Cl_2 eluent), giving pure furo[3,2-*c*]coumarins.

3.3. Compound ***4a***

Melting point: 108–111°C. ^{1}H NMR ($CDCl_3$) 7.68 (1H, d, J = 7.8 Hz), 7.56 (1H, td, J_1 = 7.8 Hz, J_2 = 1.1 Hz), 7.36 (1H, d, J = 8.2 Hz), 7.28 (1H, t, J = 7.5 Hz), 6.07 (1H, dd, J_1 = 7.0 Hz, J_2 = 3.3 Hz), 4.04 (1H, dq, J_1 = 9.6 Hz, J_2 = 7.0 Hz), 3.77 (1H, dq, J_1 = 9.6 Hz, J_2 = 7.1 Hz), 3.32 (1H, dd, J_1 = 16.4 Hz, J_2 = 7.1 Hz), 3.02 (1H, dd, J_1 = 16.4 Hz, J_2 = 3.3 Hz), 1.29 (3H, t, J = 7.1 Hz). ^{13}C NMR ($CDCl_3$)165.0, 160.2, 154.8, 132.2, 123.8, 122.6, 116.8, 112.5, 110.0, 101.2, 65.4, 33.7, 14.9. MS(EI) 232, 103, 175, 121.

3.4. Compound ***4b***

Melting point: 69–72°C. ^{1}H NMR ($CDCl_3$) 7.66 (1H, dd, J_1 = 7.8 Hz, J_2 = 1.6 Hz), 7.54 (1H, ddd, J_1 = 8.7 Hz, J_2 = 7.0 Hz, J_3 = 1.7 Hz), 7.35 (1H, d, J_1 = 8.2 Hz), 7.28 (1H, dd, J_1 = 7.8 Hz, J_2 = 7.2 Hz), 6.05 (1H, dd, J_1 = 7.1 Hz, J_2 = 3.3 Hz),

3.97 (1H, dt, $J_1 = 9.5$ Hz, $J_2 = 6.5$ Hz), 3.69 (1H, dt, $J_1 = 9.5$ Hz, $J_2 = 6.5$ Hz), 3.29 (1H, dd, $J_1 = 16.4$ Hz, $J_2 = 7.1$ Hz), 3.02 (1H, dd, $J_1 = 16.5$ Hz, $J_2 = 3.3$ Hz), 1.63 (2H, quint, $J = 6.5$ Hz), 1.40 (2H, sext, $J = 7.0$ Hz), 0.92 (3H, t, $J = 7.2$ Hz). ^{13}C NMR ($CDCl_3$) 165.0, 160.1, 154.8, 132.1, 123.8, 122.5, 116.8, 112.5, 110.2, 101.2, 69.6, 33.7, 31.3, 19.0, 13.6. MS(EI) 260, 204, 187, 176, 121, 57.

3.5. Compound **4c**

Melting point: 140°C (dec.). ^{1}H NMR ($CDCl_3$) 7.60 (1H, d, $J = 7.8$ Hz), 7.51 (1H, td, $J_1 = 7.8$ Hz, $J_2 = 1.4$ Hz), 7.31 (1H, d, $J = 8.2$ Hz), 7.24 (1H, t, $J = 7.7$ Hz), 6.30 (1H, dd, $J_1 = 7.3$ Hz, $J_2 = 3.6$ Hz), 3.28 (1H, dd, $J_1 = 16.2$ Hz, $J_2 = 7.3$ Hz), 2.92 (1H, dd, $J_1 = 16.2$ Hz, $J_2 = 3.7$ Hz), 1.35 (9H, s). ^{13}C NMR ($CDCl_3$) 165.0, 160.3, 154.8, 132.0, 123.7, 122.6, 116.7, 112.6, 105.4, 100.9, 77.0, 34.5, 28.5. MS(EI) 260, 204, 187, 176, 121, 92.

3.6. Compound **4d**

^{1}H NMR ($CDCl_3$) 7.70 (1H, dd, $J_1 = 7.6$ Hz, $J_2 = 1.5$ Hz), 7.57 (1H, td, $J_1 = 7.6$ Hz, $J_2 = 1.6$ Hz), 7.38 (1H, d, $J_1 = 8.2$ Hz), 7.30 (1H, d, $J_1 = 7.5$ Hz), 3.39 (3H, s), 3.23 (1H, d, $J = 16.7$ Hz), 3.06 (1H, d, $J = 16.7$ Hz), 1.79 (3H, s).

3.7. Compound **4e**

Melting point: 80–83°C. ^{1}H NMR ($CDCl_3$) 7.67 (1H, d, $J = 7.8$ Hz), 7.56 (1H, t, J = 7.8 Hz), 7.37 (1H, t, $J = 7.8$ Hz), 7.29 (1H, t, $J = 7.5$ Hz), 6.50 (1H, dd, $J_1 = 14.4$ Hz, $J_2 = 6.8$ Hz), 6.12 (1H, dd, $J_1 = 7.0$ Hz, $J_2 = 3.2$ Hz), 4.26–3.89 (6H, m), 3.33 (1H, dd, $J_1 = 16.6$ Hz, $J_2 = 7.0$ Hz), 3.09 (1H, dd, $J_1 = 16.6$ Hz, $J_2 = 3.2$ Hz). ^{13}C NMR ($CDCl_3$) 164.9, 162.6, 154.8, 151.4, 132.2, 123.8, 122.5, 116.9, 112.4, 110.0, 101.3, 86.9, 67.8, 66.6, 33.7. MS(EI) 274, 246, 175, 121, 73, 45.

3.8. Compound **4f**

Melting point: 103–106°C. ^{1}H NMR ($CDCl_3$) 7.67 (1H, d, $J_1 = 7.8$ Hz, $J_2 = 1.6$ Hz), 7.58 (1H, td, $J_1 = 7.9$ Hz, $J_2 = 1.5$ Hz), 7.40–7.26 (2H, m), 3.50 (1H, d, $J = 16.8$ Hz), 3.20 (1H, d, $J = 16.8$ Hz), 2.10 (3H, s), 1.96 (3H, s). ^{13}C NMR ($CDCl_3$) 168.2, 164.2, 159.8, 154.9, 132.4, 124.5, 123.9, 122.6, 116.9, 111.8, 101.9, 38.9, 26.0, 21.7. MS(EI) 218, 200, 176, 121, 92, 43.

3.9. Compound **4g**

Melting point: 150–152°C. ^{1}H NMR ($CDCl_3$) 7.70 (1H, d, $J = 7.7$ Hz), 7.58 (1H, t, $J = 7.7$ Hz), 7.37 (1H, d, $J = 8.4$ Hz), 7.30 (1H, t, $J = 7.3$ Hz), 6.58 (1H, d, $J = 5.8$ Hz), 4.07 (1H, t, $J = 7.0$ Hz), 3.75 (2H, m), 2.25 (2H, m). ^{13}C NMR ($CDCl_3$) 166.4, 159.9, 154.9, 132.6, 124.0, 123.1, 116.9, 114.4, 111.8, 102.4, 68.2, 44.8, 30.0. MS(EI) 230, 202, 187, 121, 92, 82.

3.10. Compound **4h**

Melting point: 136–139°C. ^{1}H NMR ($CDCl_3$) 7.72 (1H, dd, $J_1 = 7.8$ Hz, $J_2 = 1.7$ Hz), 7.58 (1H, ddd, $J_1 = 8.6$ Hz, $J_2 = 7.3$ Hz, $J_3 = 1.7$ Hz), 7.38 (1H, d, $J = 8.3$ Hz), 7.31 (1H, t, $J = 7.9$ Hz), 6.29 (1H, d, $J = 7.7$ Hz), 3.92 (1H, dd, $J_1 = 11.7$ Hz, $J_2 = 5.7$ Hz), 3.82 (1H, dd, $J_1 = 11.5$ Hz, $J_2 = 6.6$ Hz), 3.47 (1H, dt, $J_1 = 7.6$ Hz, $J_2 = 5.6$ Hz), 2.03 (2H, m), 1.72 (2H, m). ^{13}C NMR ($CDCl_3$) 165.6, 160.0, 154.9, 132.5, 123.9, 122.9, 116.9, 112.0,108.1, 104.5, 60.5, 36.0, 19.4, 18.8. MS(CI) 245, 121.

3.11. Compounds **4i**/**a** + **4i**/**b**

Major diastereomer: ^{1}H NMR ($CDCl_3$) 7.70 (1H, dd, $J_1 = 7.1$ Hz, $J_2 = 1.1$ Hz), 7.57 (1H, td, $J_1 = 7.7$ Hz, $J_2 = 1.2$ Hz), 7.38–7.24 (2H, m), 6.29 (1H, d, $J = 7.2$ Hz), 4.82 (1H, t, $J = 4.8$ Hz), 3.57–3.43 (1H, m), 3.50 (3H, s), 2.23–1.57 (4H, m). ^{13}C NMR ($CDCl_3$) 165.6, 159.9, 154.9, 132.5, 124.0, 122.7, 116.9, 112.0, 105.2, 104.0, 98.1, 56.1, 37.9, 26.0, 17.6. Minor diastereomer: ^{1}H NMR ($CDCl_3$) 7.70 (1H, dd, $J_1 = 7.1$ Hz, $J_2 = 1.2$ Hz), 7.57 (1H, td, $J_1 = 7.8$ Hz, $J_2 = 1.1$ Hz), 7.38–7.24 (2H, m), 6.37 (1H, d, $J = 8.5$ Hz), 4.88 (1H, t, $J = 6.4$ Hz), 3.57–3.43 (1H, m), 3.44 (3H, s), 2.25–1.53 (4H, m). ^{13}C NMR ($CDCl_3$) 165.5, 160.0, 154.9, 132.4, 123.8, 122.9, 116.9, 112.0, 107.0, 102.9, 99.1, 55.3, 36.4, 24.9, 17.2.

3.12. Compound **4j**

Melting point: 145–148°C. ^{1}H NMR ($CDCl_3$) 7.74 (1H, dd, $J_1 = 7.8$ Hz, $J_2 = 1.5$ Hz), 7.60 (1H, td, $J_1 = 7.6$ Hz, $J_2 = 1.6$ Hz), 7.40 (1H, d, $J = 8.2$ Hz), 7.32 (1H, t, $J = 7.7$ Hz), 6.92 (1H, d, $J = 7.4$ Hz), 6.52 (1H, t, $J = 2.4$ Hz), 5.54 (1H, t, $J = 2.8$ Hz), 4.66 (1H, dt, $J_1 = 7.4$ Hz, J_2=2.3 Hz), ^{13}C NMR ($CDCl_3$) 165.4, 159.7, 154.7, 145.2, 132.7, 124.1, 123.1, 116.9, 113.7, 111.9, 104.8, 102.5, 48.7. MS(EI) 229, 201, 187, 121, 81.

3.13. Compounds **4k/a + 4k/b**

Major diastereomer: ^{1}H NMR ($CDCl_3$) 7.72 (1H, dd, J_1=7.8 Hz, J_2=2.1 Hz), 7.58 (1H, td, $J_1 = 7.2$ Hz, $J_2 = 1.7$ Hz), 7.38 (1H, d, $J = 8.2$ Hz), 7.30 (1H, t, $J = 7.8$ Hz), 6.86 (1H, d, $J = 7.3$ Hz), 5.13 (1H, t, $J = 1.5$ Hz), 4.62 (1H, m), 1.90 (3H, t, $J = 1.6$ Hz). Minor diastereomer: ^{1}H NMR ($CDCl_3$) 7.72 (1H, dd, $J_1 = 7.8$ Hz, $J_2 = 2.0$ Hz), 7.58 (1H, td, $J_1 = 7.2$ Hz, $J_2 = 1.9$ Hz), 7.38 (1H, d, $J = 8.1$ Hz), 7.30 (1H, t, $J = 7.8$ Hz), 6.43 (1H, t, $J = 2.6$ Hz), 5.47 (1H, t, $J = 2.7$ Hz), 4.29 (1H, t, $J = 2.3$ Hz), 1.86 (3H, s).

3.14. Compound **4k/ c**

^{1}H NMR ($CDCl_3$) 9.52 (1H, bs), 7.96 (1H, dd, $J_1 = 7.5$ Hz, $J_2 = 1.5$ Hz), 7.55 (1H, t, $J = 7.7$ Hz), 7.35–7.28 (2H, m), 7.21 (1H, d, $J = 3.4$ Hz), 6.19 (1H, d, $J = 3.3$ Hz), 2.44 (3H, s). ^{13}C NMR ($CDCl_3$) 159.4, 157.5, 151.6, 150.6, 146.7, 132.0, 124.0, 123.3, 116.2, 115.4, 112.3, 108.2, 96.6, 13.6.

3.15. Compound **4l**

Melting point: 153–156°C. ^{1}H NMR ($CDCl_3$) 7.70 (1H, t, $J = 7.7$ Hz), 7.60 (1H, d, $J = 7.6$ Hz), 7.42–7.30 (4H, m), 7.03 (1H, t, $J = 7.5$ Hz), 6.99 (1H, d, $J = 8.3$ Hz), 6.66 (1H, d, $J = 7.3$ Hz), 6.39 (1H, d, $J = 7.5$ Hz). MS(CI) 279, 121.

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