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Photodecarboxylative Cyclizations of ω-Phthalimido-*para*-phenoxy Carboxylates

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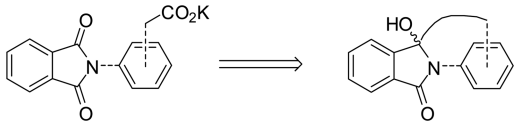
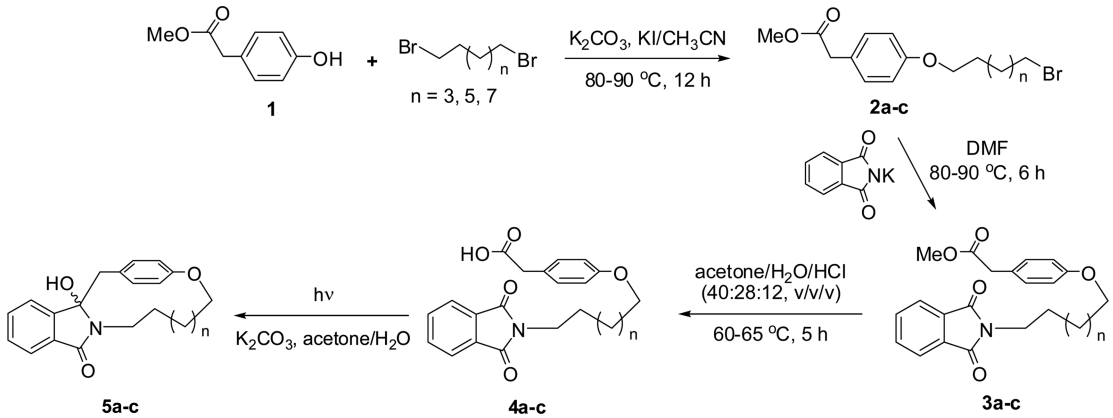
The chemistry of electronically-excited phthalimides is dictated by electron and/or hydrogen transfer reactions.[1,2] The photochemistry of phthalimides has been intensively studied, and numerous synthetically useful transformations with high chemical and quantum yields have been developed.[3] Among the synthetic applications, intra- and intermolecular photodecarboxylation (PDC) of ω-phthalimidoalkyl carboxylates has been developed by Griesbeck and coworkers as a versatile pathway to medium- and large-ring heterocycles.[4] Model reactions were further realized on macro- and micro-scales.[5] We recently described PDC cyclizations of ω-phthalimidoalkynoates to produce macrocyclic alkynes with ring-sizes up to 17.[6] In recent study, we expanded the portfolio of this reaction and investigated the photochemistry of related aryl-linked phthalimides in Scheme 1. Based on these approaches, we demonstrated that ω-phthalimido-*ortho/meta*-phenoxy carboxylates undergo efficient PDC cyclizations.[7,8] While the yields of ω-phthalimido-*ortho*-phenoxy carboxylates steadily decreased with increasing chain-length and the maximum yield of the 6-membered product was obtained in 75%, the yields of *meta*-phenoxy carboxylates steadily increased with increasing chain-length and the extended 16-membered product was subsequently obtained in 48% yield.



Scheme 1. Photocyclization of arene-linked phthalimides.

As an extension of our work, we were interested in using *para*-substituted aryl carboxylates with a linker between the phthalimide chromophore and terminal phenyl carboxylic acid. The U-shaped geometry of the central skeleton should favor close contact geometry between the two active ends of the molecule, preferably for large ring systems. In this paper, we describe the photochemistry of several ω-phthalimido-*para*-alkoxy phenyl carboxylic acids (**4a-c**) differing in internal carbon-chain lengths (Scheme 2).

Syntheses of ω-Phthalimido-*para*-alkoxy Phenyl Acetic Acids. ω-Phthalimido-*para*-alkoxy phenyl acetic acids (**4a-c**) containing an alkyl chain were prepared by the formation of an ether-linkage to investigate the relationship between ring size and yield through intramolecular PDC cyclization. To compensate for the *para*-substitution pattern in the ring closure step, longer carbon linkers were specifically introduced. The derivatives **4a-c** were prepared from 4-hydroxyphenyl acetate as described in Scheme 2. Coupling of 4-hydroxyphenyl acetate and the corresponding 1,ω-dibromoalkanes produced **2a-c** in moderate yields of 52-56%.[9] Treatment of **2a-c** with potassium phthalimide in DMF yielded compound **3a-c** in good yields of 72-89%. Subsequent



Scheme 2. Synthesis and photocyclization of **4a-c**.

hydrolysis with conc. HCl/H_2O/acetone at reflux afforded the desired ω-phthalimido-*para*-alkoxy phenyl acetic acids (**4a-c**) as colorless solids in 68-83% yield.

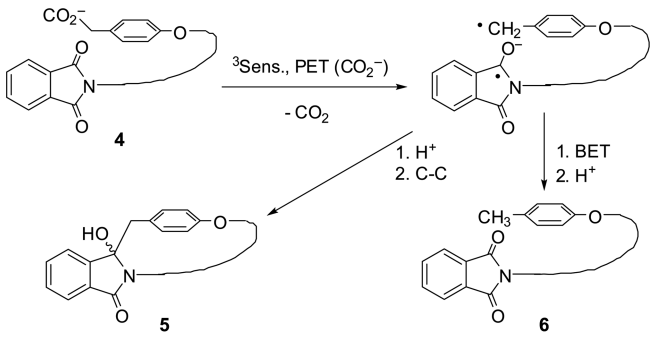
Photolyses of ω-Phthalimido-*para*-alkoxy Phenyl Acetic Acids. ω-Phthalimido-*para*-alkoxy phenyl acetic acids (**4a-c**) were deprotonated with potassium carbonate (K_2CO_3) prior to photolysis. Photolyses were performed with the corresponding potassium salts of **4a-c** in acetone/water mixtures (9:1,v/v) using a Rayonet photoreactor equipped with low pressure mercury lamps (phosphor coated with an emission maximum at *ca.* 300 nm; 800 W). Irradiations were stopped after 4 h and thin layer chromatography (TLC) analyses of the crude reaction mixtures indicated conversion rates greater than 90%.

Photolysis of ω-phthalimido-*para*-alkoxy phenyl acetic acid (**4a**) resulted in a the cyclization product **5a** in 19% isolated yield (Scheme 2). In $CDCl_3$, the ^{1}H-NMR spectra showed multi-signals for the N-CH_2 group between 4.14 and 4.37 ppm. The benzylic proton attached beside hydroxyl carbon gave a singlet at 3.48 ppm, which was unambiguously assigned. In the ^{13}C-NMR spectrum, the newly formed C-OH group showed a characteristic resonance at 90.7 ppm. Although the simple decarboxylation product was not observed in the crude product, NMR and TLC analysis indicated several other by-products in a total amount of *ca.* 5-10%. However, none of these by-products could be isolated in a sufficient amount and purity. Likewise, compounds **4b** and **4c** produced the corresponding cyclization products **5b** and **5c**, respectively. The isolated yield for the 18-membered ring **5c** was high with 46%, whereas the smaller ring-system **5b** had a decreased isolated yield of 37%, as summarized in Table 1. The amounts of unreactive starting material were estimated below 5-10%. Additionally, several by-products were detected in total amounts of *ca.* 5-10% in the crude reaction mixtures by TLC or NMR analysis. Yields of **5** steadily increased with increasing chain-length and following this extension strategy, the 18-membered product **5c** was subsequently obtained in 46% yield. The structural assignments of the photoproducts were based on the spectroscopic data. The complex multiplet between 6.8 and 7.8 ppm further revealed the asymmetry of the aromatic ring, corroborating the structure of the cyclization product. While the ^{1}H-NMR spectra were rather complex, all cyclization products **5a-c** showed the characteristic C-OH signal in their ^{13}C-NMR spectra at approximately 90 ppm. In all three cases, the ^{13}C-NMR spectra showed clear resonances at 90.65 (**5a**), 90.63 (**5b**) and 90.38 ppm (**5c**), respectively, corresponding to quaternary C-OH carbons. The GC/MS spectrum showed molecular ion peaks. The spectral data of

Table 1. Experimental details for the PDC cyclizations of **4a-c**[a]

Entry	*n*	Ring size	Yield of **5** (%)
4a	3	14	19
4b	5	16	37
4c	7	18	46

[a]Isolated yields.



Scheme 3. Photochemical decarboxylation of ω-phthalimido-*para*-phenoxy carboxylates: mechanistic scenario.

the cyclization products are consistent with carbon-carbon bond formation between the phthalimide carbonyl carbon and α-carbon of potassium carboxylates.

Mechanistic Interpretations. When the geometric disadvantages of the linking *para*-linked long chain compounds were cooperative by an elongated chain, photocyclization products were obtained in good yields (19-46%). The efficiency of the cyclization increases with increasing carbon-chain length. The key-step in the mechanistic scenario (Scheme 3) is an intramolecular electron transfer from the respective donor moiety to the triplet excited phthalimide, populated by sensitization with acetone.[10,11] For carboxylate **4**, electron transfer generates an unstable carboxy radical that undergoes rapid decarboxylation to the analogous carbon radical. Subsequently, protonation and biradical combination yields the desired cyclization product **5**. When cyclization is not possible, back electron transfer (BET) provides a carbanion,[12] which is protonated by water to produce the decarboxylation product **6**.[13]

In conclusion, ω-phthalimido-*para*-phenoxy carboxylates (**4a-c**) underwent photodecarboxylative macrocyclizations in reasonable yields of 19-46%. The optimal yield of intramolecular cyclization was obtained from the substrate (**4c**) to form an 18-membered ring. The photocyclization efficiency increases with increasing carbon-chain length, probably due to the good chance that they would collide. Therefore, we concluded that the efficiency of PDC cyclizations depended critically on the substitution pattern of the arene and the linking carbon-chain lengths between electron-donor and acceptor. The extended carbon linker in the *para*-substitution arene must compensate for the unfavorable *para*-substitution to allow for close contact for electron transfer and cyclization.

Experimental Section

General Procedures. All starting materials and reagents were purchased from Aldrich Chemical Co. and used without further purification. Solvents used for synthesis (acetonitrile, DMF, hexane, and ethyl acetate) were purified *via* literature methods.[14] Twice-distilled water and reagent grade acetone were used for the photoreactions.

Melting points were obtained on a Buchi 510 melting

point apparatus and were uncorrected. NMR spectra were recorded on a Brüker Avance 400 spectrometer (400 MHz ^{1}H- and 100 MHz ^{13}C-frequencies) using tetramethylsilane (TMS) or the solvent residual peak as an internal standard. $CDCl_3$ was stored over K_2CO_3 to remove trace amounts of acid. Chemical shifts δ are given in ppm and coupling constants *J* in Hz. The chemical shifts for the acid protons of **1a-c** were not observed (> 10 ppm). MS spectra were determined on a V. G. Autospec-Ultima (EI, 70 eV) instrument. Fourier-transform infrared (FT-IR) spectra were recorded on a Bomem MB-100 series FT-IR spectrophotometer (KBr disc or film). Photochemical reactions were performed in Pyrex vessels (λ > 280 nm) using a Rayonet photochemical reactor equipped with 3000 Å lamps (λ = 300 ± 10 nm; *ca.* 800 W).

Synthesis of Methyl Bromohexyloxy Phenyl Acetate (2a): The mixture of methyl 4-hydroxyphenyl acetate (**1**) (1.00 g, 6.02 mmol), K_2CO_3 (1.00 g, 7.22 mmol), and KI (catalyst) in dry acetonitrile (25 mL) were added to 1,6-dibromohexane (1.1 mL, 7.22 mmol) and stirred in a 80-90 °C oil bath for 12 h. After completing the reaction by monitoring on thin layer chromatography (TLC), the solvent was evaporated. The resulting mixture was diluted with ethyl acetate, washed with water, dried over $MgSO_4$ and the solvent was evaporated. The residue was purified by column chromatography on silica gel (hexane/ethyl acetate = 5:1) to produce **2a** (1.08 g, 55%) as a colorless oil. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.49-1.50 (m, 4H), 1.78 (t, 2H, *J* = 6.7 Hz), 1.89 (t, 2H, *J* = 7.2 Hz), 3.41 (t, 2H, J = 6.6 Hz), 3.56 (s, 2H), 3.68 (s, 3H), 3.94 (t, 2H, *J* = 6.3 Hz), 6.83 (d, 2H, *J* = 8.6 Hz), 7.17 (d, 2H, *J* = 8.1 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 25.3, 27.9, 29.1, 32.7, 33.7, 40.2, 51.9, 67.7, 114.5, 125.8, 130.1, 158.0, 172.1.

Methyl Bromooctyloxy Phenyl Acetate (2b): The reaction of methyl 4-hydroxyphenyl acetate (**1**) (1.00 g, 6.02 mmol), K_2CO_3 (1.00 g, 7.22 mmol), KI (catalyst), and 1,8-dibromooctane (1.32 mL, 7.22 mmol) in dry acetonitrile (25 mL) was performed as described for the preparation of **2a** to produce **2b** (1.13 g, 52%) as a colorless oil. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.35-1.45 (m, 8H), 1.76 (t, 2H, *J* = 7.2 Hz), 1.85 (t, 2H, *J* = 7.3 Hz), 3.40 (t, 2H, *J* = 6.8 Hz), 3.55 (s, 2H), 3.67 (s, 3H), 3.92 (t, 2H, *J* = 6.5 Hz), 6.83 (d, 2H, *J* = 8.1 Hz), 7.16 (d, 2H, *J* = 8.3 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 25.9, 28.1, 28.7, 29.1, 29.2, 32.8, 33.9, 40.3, 51.9, 67.9, 114.5, 125.7, 130.05, 158.1, 172.1.

Methyl Bromodecyloxy Phenyl Acetate (2c): The reaction of methyl 4-hydroxyphenyl acetate (**1**) (1.00 g, 6.02 mmol), K_2CO_3 (1.00 g, 7.22 mmol), KI (catalyst), and 1,8-dibromooctane (1.63 mL, 7.22 mmol) in dry acetonitrile (25 mL) was performed as described for the preparation of **2a** to produce **2c** (1.30 g, 56%) as a colorless oil. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.30-1.44 (m, 12H), 1.74-1.86 (m, 4H), 3.40 (t, 2H, *J* = 6.8 Hz), 3.55 (s, 2H), 3.67 (s, 3H), 3.92 (t, 2H, *J* = 6.6 Hz), 6.83 (d, 2H, *J* = 8.6 Hz), 7.16 (d, 2H, *J* = 8.8 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 25.6, 27.8, 28.1, 28.3, 28.9, 29.0, 29.1, 32.4, 33.2, 33.6, 39.9, 51.5, 67.5, 114.1, 125.3, 129.7, 157.7, 171.8.

Synthesis of ω-Phthalimido-*para*-hexyloxy Phenyl Acetate (3a): The mixture of **2a** (1.08 g, 3.28 mmol) and potassium phthalimide (0.73 g, 3.94 mmol) in dry *N,N*-dimethylformamide (DMF, 20 mL) was stirred in a 80-90 °C oil bath for 6 h. After completing the reaction by monitoring on TLC, the solvent was evaporated. The resulting mixture was diluted with ethyl acetate, washed with water, dried over $MgSO_4$ and the solvent was evaporated. The residue was purified by column chromatography on silica gel (hexane/ethyl acetate = 2:1) to produce **3a** (1.14 g, 89%) as a white solid. mp 37-38 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.38-1.52 (m, 4H), 1.67-1.78 (m, 4H), 3.55 (s, 2H), 3.67 (s, 3H), 3.69 (t, 2H, *J* = 7.4 Hz), 3.92 (t, 2H, *J* = 6.7 Hz), 6.81 (d, 2H, *J* = 8.5 Hz), 7.15 (d, 2H, *J* = 8.3 Hz), 7.70 (dd, 2H, *J* = 3.4, 3.4 Hz), 7.83 (dd, 2H, *J* = 3.4, 3.4 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 25.7, 26.6, 28.6, 29.1, 37.9, 40.3, 52.0, 67.7, 114.5, 123.1, 125.7, 130.1, 132.0, 133.7, 158.0, 168.3, 172.2.

ω-Phthalimido-*para*-octyloxy Phenyl Acetate (3b): The reaction of **2b** (1.10 g, 3.08 mmol) and potassium phthalimide (0.69 g, 3.70 mmol) in dry DMF (20 mL) was performed as described for the preparation of **3a** to produce **3b** (1.09 g, 84%) as a white solid. mp 43-44 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.35-1.76 (m, 12H), 3.55 (s, 2H), 3.66 (s, 3H), 3.67 (t, 2H, *J* = 7.1 Hz), 3.91 (t, 2H, *J* = 6.6 Hz), 6.82 (d, 2H, *J* = 8.9 Hz), 7.16 (d, 2H, *J* = 8.6 Hz), 7.70 (dd, 2H, *J* = 2.8, 2.8 Hz), 7.83 (dd, 2H, *J* = 2.8, 2.8 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 26.8, 28.6, 29.1, 29.2, 29.4, 29.7, 38.1, 40.3, 51.9, 67.94, 114,5, 123.0, 125.7, 130.1, 132.0, 133.7, 158.1, 168.3, 172.2.

ω-Phthalimido-*para*-decyloxy Phenyl Acetate (3c): The reaction of **2c** (1.29 g, 3.35 mmol) and potassium phthalimide (0.74 g, 4.02 mmol) in dry DMF (20 mL) was performed as described for the preparation of **3a** to produce **3c** (1.08 g, 72%) as a white solid. mp 48-49 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.29-1.76 (m, 16H), 3.55 (s, 2H), 3.65 (s, 3H), 3.67 (t, 2H, *J* = 5.4 Hz), 3.92 (t, 2H, *J* = 6.3 Hz), 6.83 (d, 2H, *J* = 8.9 Hz), 7.16 (d, 2H, *J* = 8.4 Hz), 7.69 (dd, 2H, *J* = 3.4, 3.4 Hz), 7.83 (dd, 2H, *J* = 2.8, 2.8 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 26.0, 26.9, 28.6, 29.2, 29.3, 29.4, 29.4, 29.5, 38.1, 40.3, 52.0, 68.0, 114.5, 123.0, 125.7, 130.1, 132.1, 133.7, 158.1, 168.3, 172.2.

Synthesis of ω-Phthalimido-*para*-hexyloxy Phenyl Acetic Acid (4a): A solution of **3a** (1.14 g, 2.90 mmol) in acetone/H_2O/HCl (40:28:12, v/v/v) (30 mL) was stirred in a 60-65°C oil bath for 5 h. After completing the reaction by monitoring on TLC, the solvent was evaporated. The resulting mixture was diluted with CH_2Cl_2, washed with water, dried over $MgSO_4$ and the solvent was evaporated. The residue was purified by column chromatography on silica gel (hexane/ethyl acetate = 1:1) to produce **4a** (0.92 g, 83%) as a white solid. mp 116-118 °C; FT-IR (KBr, cm^{-1}) 3462, 3227, 2940, 1773, 1613, 1362, 800; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.40-1.78 (m, 8H), 3.57 (s, 2H), 3.69 (t, 2H, *J* = 7.3 Hz), 3.92 (t, 2H, *J* = 6.4 Hz), 6.82 (d, 2H, *J* = 8.3 Hz), 7.15 (d, 2H, *J* = 8.9 Hz), 7.70 (dd, 2H, *J* = 3.4, 3.4 Hz), 7.83 (dd, 2H, *J* = 3.4, 3.4 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 25.7, 26.7, 28.6, 29.1, 38.0, 40.0, 67.8, 114.6, 123.1, 125.1, 130.2, 132.1, 133.8,

158.2, 168.3, 176.2; Mass (*m/z*) 381 (M^+), 281, 246, 202, 179, 160, 73.

ω-Phthalimido-*para*-octyloxy Phenyl Acetic Acid (4b): The reaction of **3b** (1.09 g, 2.58 mmol) in acetone/H_2O/HCl (40:28:12, v/v/v) (30 mL) was performed as described for the preparation of **4a** to produce **4b** (0.78 g, 74%) as a white solid. mp 110-112 °C; FT-IR (KBr, cm^{-1}) 3462, 3194, 2943, 1725, 1614, 1360, 792; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.34-1.76 (m, 12H), 3.57 (s, 2H), 3.67 (t, 2H, *J* = 7.3 Hz), 3.91 (t, 2H, *J* = 6.3 Hz), 6.83 (d, 2H, *J* = 8.6 Hz), 7.16 (d, 2H, *J* = 8.9 Hz), 7.69 (dd, 2H, *J* = 2.8, 2.8 Hz), 7.83 (dd, 2H, *J* = 3.0, 3.0 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 26.0, 26.8, 28.6, 29.1, 29.7, 31.3, 38.1, 40.0, 68.0, 114.6, 123.1, 125.1, 130.2, 132.1, 133.7, 158.3, 168.3, 176.6; Mass (*m/z*) 410 (M^+), 281, 246, 202, 179, 160, 73.

ω-Phthalimido-*para*-decyloxy Phenyl Acetic Acid (4c): The reaction of **3c** (1.08 g, 2.39 mmol) in acetone/H_2O/HCl (40:28:12, v/v/v) (30 mL) was performed as described for the preparation of **4a** to produce **4c** (0.71 g, 68%) as a white solid. mp 106-108 °C; FT-IR (KBr, cm^{-1}) 3464, 3115, 2942, 1726, 1615, 1365, 792; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 1.29-1.76 (m, 16H), 3.58 (s, 2H), 3.67 (t, 2H, *J* = 7.0 Hz), 3.92 (t, 2H, *J* = 6.5 Hz), 6.83 (d, 2H, *J* = 8.3 Hz), 7.16 (d, 2H, *J* = 8.7 Hz), 7.69 (dd, 2H, *J* = 3.4, 3.4 Hz), 7.83 (dd, 2H, *J* = 2.9, 2.9 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 26.0, 26.9, 28.6, 29.2, 29.26, 29.33, 29.4, 29.5, 38.1, 40.0, 67.97, 114.6, 123.1, 125.0, 130.2, 132.1, 133.7, 158.3, 168.4, 176.4; Mass (*m/z*) 438 (M^+), 363, 281, 207, 160, 73.

Photolysis of 4a: Potassium carbonate (K_2CO_3) (17.9 mg, 0.13 mmol) was dissolved in 2 mL of water. A solution of **4a** (100 mg, 0.26 mmol) in 100 mL of an acetone/H_2O (9:1, v/v) solution was added. The mixture was bubbled with N_2 gas for 30 min and irradiated (300 nm) for 4 h while purging with a slow stream of N_2 gas. The solution was evaporated and extracted with chloroform, washed with water, dried over $MgSO_4$ and evaporated. The residue was purified by column chromatography on silica gel (hexane/ethyl acetate = 1:1) to produce **5a** (17.0 mg, 19%). mp 182-184 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.31-1.69 (m, 8H), 2.56-2.76 (m, 2H), 3.48 (s, 2H), 4.l4-4.37 (m, 2H), 6.94 (d, 2H, *J* = 3.8 Hz), 6.99 (d, 1H, *J* = 8.3 Hz), 7.10 (d, 2H, *J* = 5.8 Hz), 7.46 (t, 1H, *J* = 7.1 Hz), 7.59 (d, 2H, *J* = 5.9 Hz), 7.68 (d, 1H, *J* = 7.1 Hz), 7.79 (d, 1H, *J* = 8.1 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 22.3, 25.6, 26.5, 28.0, 41.3, 44.4, 67.3, 90.7, 116.3, 117.7, 121.4, 123.2, 127.9, 129.5, 130.4, 131.3, 131.5, 132.2, 148.7, 157.5, 168.0; Mass (*m/z*) 337 (M^+), 319, 230, 160, 108.

Photolysis of 4b: The reaction of K_2CO_3 (16.9 mg, 0.12 mmol) in 2 mL of water and **4b** (100 mg, 0.24 mmol) in 100 mL of an acetone/H_2O (9:1, v/v) solution was performed as described for the photolysis of **4a** to produce **5b** (33.0 mg, 37%). mp 171-173 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.78-1.37 (m, 8H), 1.53-1.76 (m, 4H), 2.70-2.81 (m, 2H), 3.48 (s, 2H), 4.23-4.33 (m, 2H), 6.84-7.10 (m, 3H), 7.48 (t, 1H, *J* = 7.1 Hz), 7.57 (d, 2H, *J* = 7.3 Hz), 7.62 (d, 1H, *J* = 6.8 Hz), 7.72 (d, 1H, *J* = 7.2 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 22.3, 25,6, 26.5, 26.6, 26.8, 28.0, 41.3, 44.4, 67.3, 90.6, 116.3, 117.7, 121.4, 123.2, 127.9, 129.5, 130.4, 131.3, 131.5, 132.2, 148.6, 157.4, 168.0; Mass (*m/z*) 365 (M^+), 281, 207, 107.

Photolysis of 4c: The reaction of K_2CO_3 (15.7 mg, 0.11 mmol) dissolved in 2 mL of water and **4c** (100 mg, 0.22 mmol) in 100 mL of an acetone/H_2O (9:1, v/v) solution was performed as described for the photolysis of **4a** to produce **5c** (41.0 mg, 46%). mp 182-184 °C; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 0.87-1.76 (m, 16H), 2.77-2.85 (m, 2H), 3.08-3.15 (m, 2H), 3.48 (s, 2H), 3.59-4.17 (m, 2H), 6.80 (d, 2H, *J* = 3.8 Hz), 7.11 (d, 2H, *J* = 8.1 Hz), 7.31(d, 1H, *J* = 7.9 Hz), 7.47 (t, 1H, *J* = 7.1 Hz), 7.59 (d, 2H, *J* = 7.3 Hz), 7.62 (d, 1H, *J* = 6.8 Hz), 7.72 (d, 1H, *J* = 7.2 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 24.0, 25.8, 27.1, 27.5, 27.6, 27.7, 27.8, 28.6, 40.6, 43.8, 66.7, 90.4, 116.3, 117.7, 121.4, 123.2, 127.9, 129.5, 130.4, 131.3, 131.5, 132.2, 148.6, 157.4, 168.0; Mass (*m/z*) 393 (M^+), 305, 207, 107.

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