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PHOTOCHEMICAL DIMERIZATION OF STYRYLNAPHTHOFURANS-II[1][#]

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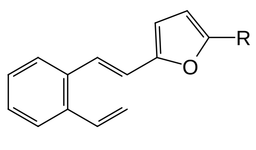
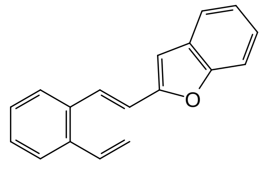
Abstract – Irradiation of styrylnaphthofurans (**3a**, **3b**) [10^{-1} M] (benzene) under anaerobic conditions gives mainly dimeric products (65-70%): [2+2] ethene-ethene cycloaddition products (**9a**, **9b**, **10a**, **10b** and **11a**, **11b**) (40-44%) and [2+2] ethene-furan cycloaddition products (**12a**, **12b**) (20-22%). Minor quantities of electrocyclization products (**7a**, **7b**) (3.5-4.5%) and dihydrocyclization products (**8a**, **8b**) (3-4.5%) are also isolated. The structures of the products are based on ^{1}H and ^{13}C NMR spectra, applying COSY, LR COSY, APT, HETCOR and NOESY techniques.

The photochemistry of *β*-heteroaryl substituted *o*-divinylbenzenes, heteroaromatic stilbene-type compounds,[2] was first reported by us[2a] in 1989 with *o*-vinylstyrylfuran (**1a**).



1 a: R = H
b: R = Ph



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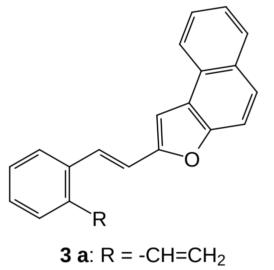

3 a: R = -CH=CH$_2$
b: R = -CH$_3$

Recent studies on the systems like phenyl substituted o-vinylstyrylfuran (**1b**),[2b] o-vinylstyrylbenzofuran (**2**)[1] and *o*-vinylstyrylnaphthofuran (**3a**)[1] in a sense of intramolecular cycloaddition reactions have shown distinctive influence of π delocalization mode on the reaction course. While 2-benzofuran derivative (**2**) reacts in the same way[1] as furan derivative (**1a**) giving **4a**, and bicyclo[3.2.1]octadiene (**5**) respectively, **1b** gives only traces of **4b**. No bicyclic structure (**6**) was found upon irradiation of naphthofuran

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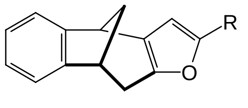
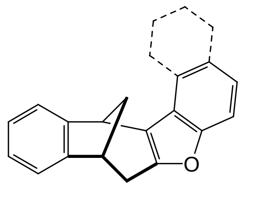
[#] This article is dedicated to Prof. Dr. W. H. Laarhoven, in gratitude, on the occasion of his 75th birthday.

derivative (**3a**) under the same conditions in dilute solution.[1] The lack of reactivity of naphthofuran derivatives in the sense of intramolecular cycloaddition, may be ascribed to the rapid formation of the excited state complex. In addition to *cis-trans* and *trans-cis* isomerization of **3a**, some traces of dimeric products, along with decomposed materials, were detected in ^{1}H NMR spectra.



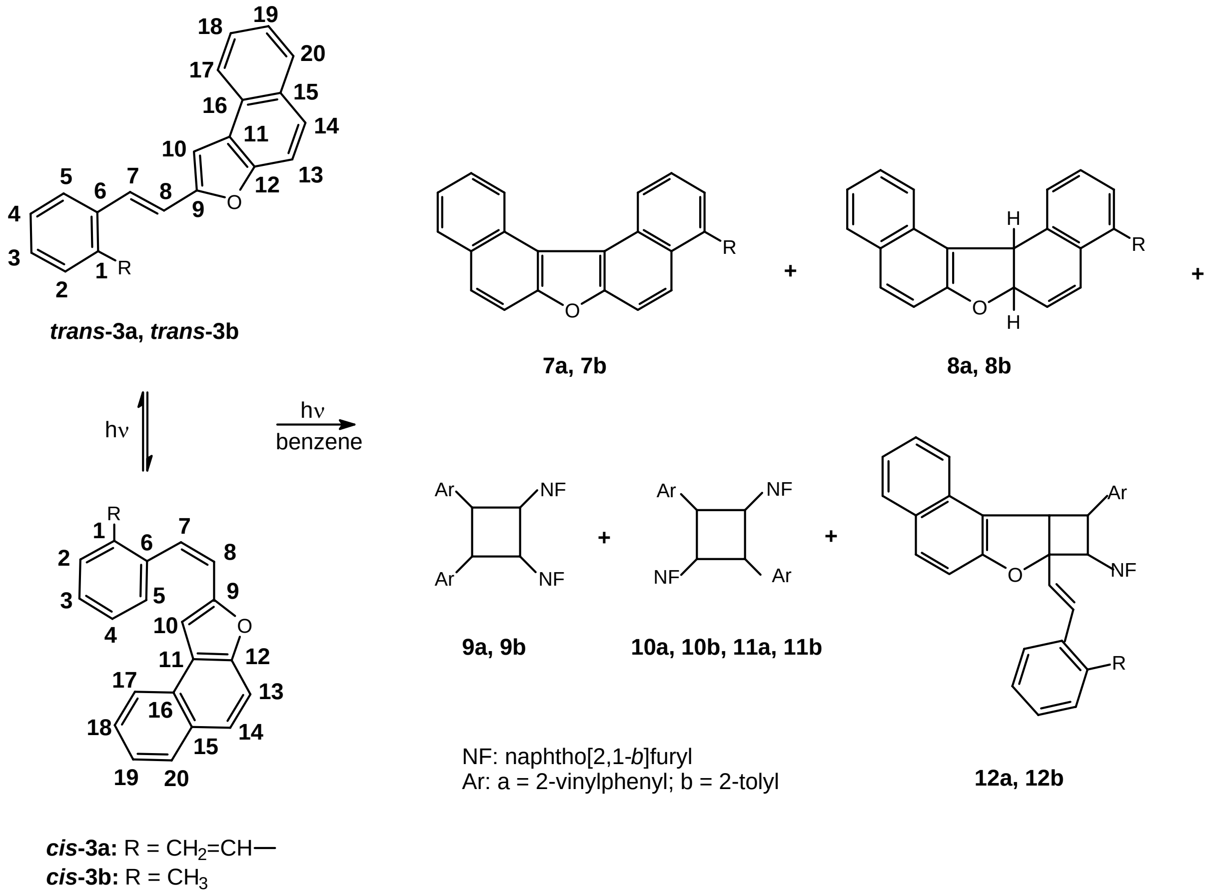
4a: R=H
4b: R=Ph



5: -benzofuran
6: -naphtho[2,1-b]furan

In this paper we report results on the photochemical reactions of styrylnaphthofurans (**3)** under conditions that enable us to isolate dimeric products and determine their structures. We were able to isolate, for the first time, electrocyclization product (**7**), rearranged dihydro-electrocyclization product (**8**), diverse cyclobutane (**9-11**) and fused cyclobutane (**12**) products formed by [2+2] ethene-ethene and [2+2] ethene-furan cycloaddition reactions.

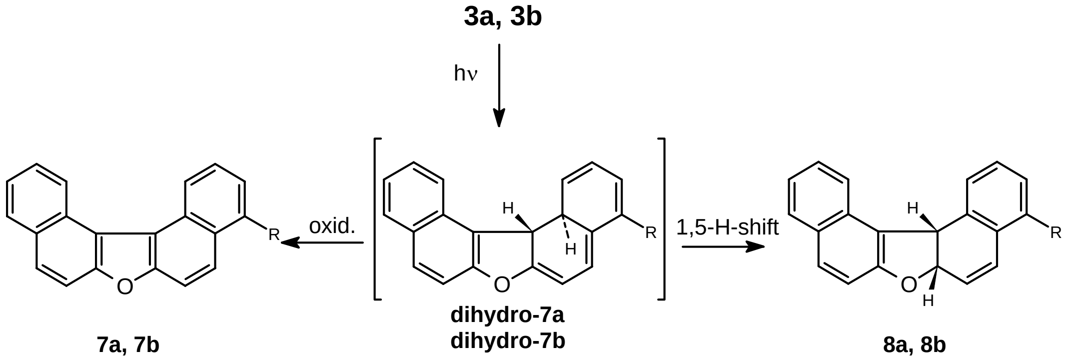
RESULTS AND DISCUSSION



Scheme 1

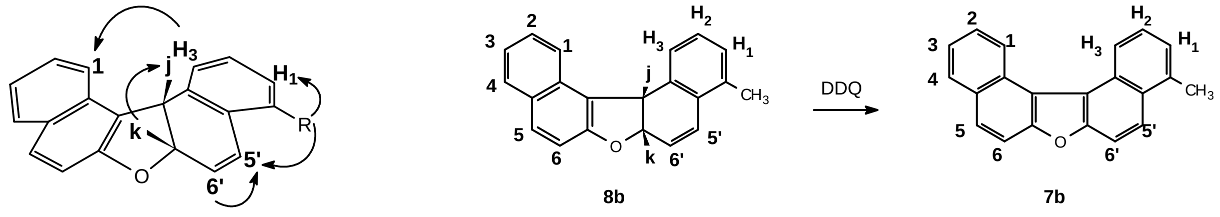
2-[2-(2-Vinylphenyl)ethenyl]naphtho[2,1-*b*]furan (**3a**) and 2-[2-(2-methylphenyl)ethenyl)]naphtho[2.1-*b*]furan (**3b**), were prepared by a Wittig reaction[3,4] and obtained as mixtures of *trans*- and *cis*-isomers that were separated by column chromatography. Their ^{1}H and ^{13}C NMR spectra were fully assigned.

Irradiations of **3a** were carried out in benzene. Best results, in the sense of the formation of dimeric products (**9a-12a**) (60% isolated yield), were obtained at the concentration of 1.6x10^{-1}M and under anaerobic conditions. From the NMR spectra of the isolated products it was obvious that no reactions with the vinyl group take place. Divinyl- and distyrylnaphthalenes give naphthalenophan derivatives[5] by [2+2] intermolecular photocycloaddition. To confirm that the vinyl group in **3** is not involved in the photoreaction, and to facilitate determination of the structure of the photoproducts, 2-[2-(2-methylphenyl)ethenyl)]naphtho[2,1-*b*]furan (**3b**) was irradiated under the same conditions as **3a** (Scheme 1). As in the case of **3a**, the main reactions were intermolecular photocycloaddition giving a mixture of dimeric cyclobutane products (**9b-12b**) (66% isolated yield), and *cis-trans* isomerization of **3b**. The other photoproducts (**7**) and (**8**), were obtained in 3-5% yield as the result of intramolecular electrocyclization reaction.[6] The dihydro derivative of **7** is formed from *cis*-styrylnaphthofurans (**3)** and this oxidizes to **7** or rearranges by 1,5-hydrogen shift to the more stable **8** (Scheme2).



Scheme2

The structures of the products (**8**) were determined spectroscopically. The MS spectra of **8a** and **8b** suggest monomeric structures with pronounced [M^{+}-2] fragments, indicating a structure in which facile hydrogen loss can occur. The ^{1}H NMR spectrum showed multiplet of eleven protons and two signals at 4.80 and 4.96 ppm ascribed to H-**k** and H-**j**. All the protons could be assigned by combining the results from spectra taken in CDCl$_3$ and in C$_6$D$_6$. The observed interaction of the protons **j** and **k** in the NOESY spectrum pointed to the *cis*-configuration of these two hydrogens. In ^{13}C NMR spectrum two doublets were found at δ 86.19 and 51.58 ppm for **8a** and δ 86.44 and 51.50 ppm for **8b**. These signals are assigned to C-**k** and C-**j** respectively. This is in good agreement with the influence of oxygen on the chemical shift.[7] The structure of **8** was confirmed by aromatization with DDQ to a product that was identical to structure **7b** (Scheme 3).



8a, 8b

Scheme 3

Dimeric products were isolated by column chromatography from the latter fractions. The ^{1}H NMR spectrum of the product mixture showed good relation of carbons and hydrogens compared to the theoretical values, suggesting strongly the formation of the isomeric products, dimers. **9**-**12** were isolated by repeated thin layer chromatography.

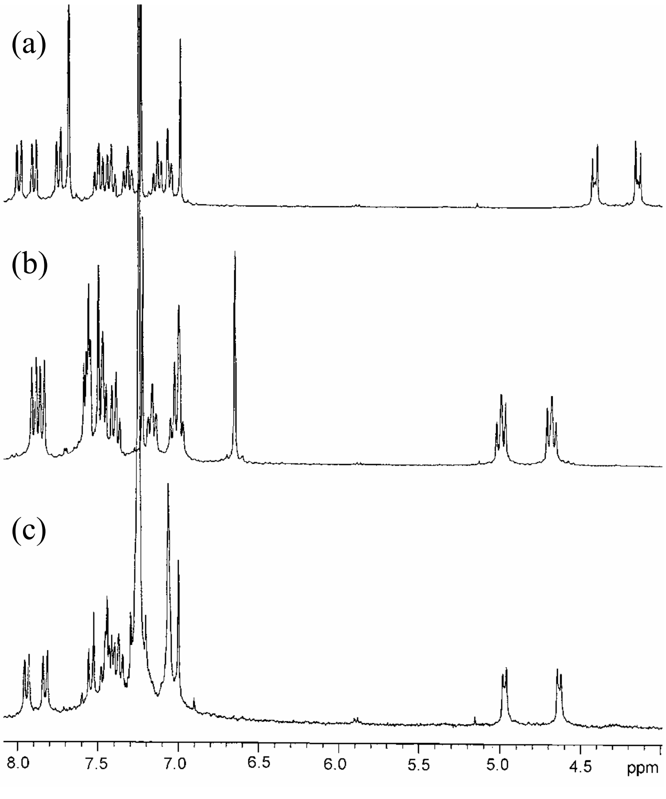
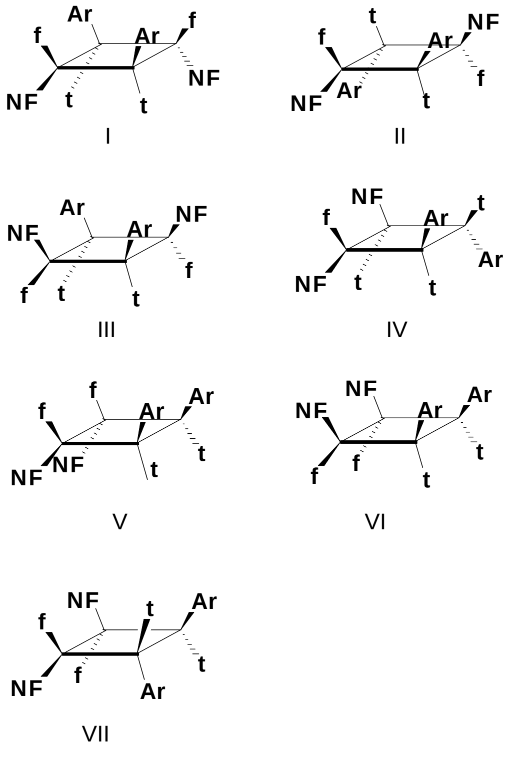
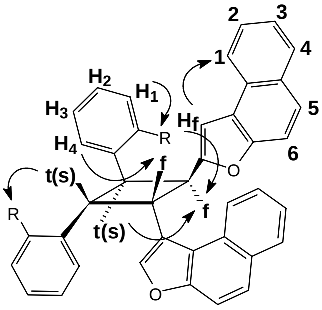


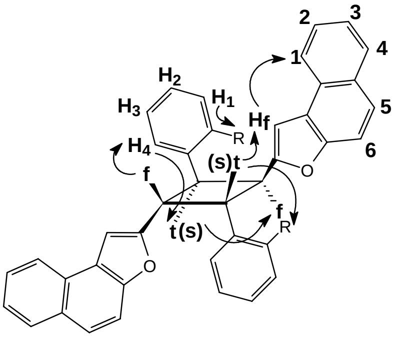
Figure 1. ^{1}H NMR spectra of (a) **9b**, (b) **10b** and (c) **11b** (CDCl$_3$)



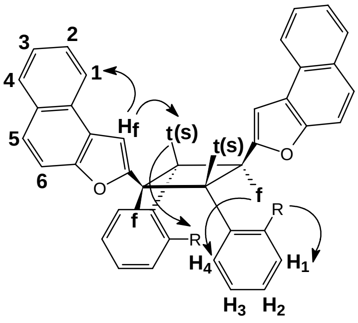
Three of the dimeric products were cyclobutane derivatives (**9**, **10** and **11**), formed from a [2+2] cycloaddition reaction of two ethylenic bonds. Each showed a very small molecular ion M$^+$ 568 and the base peak at *m/z* 284, M$^+$/2 so we assign **10** and **11** to be "head-to-tail" adducts whereas compound (**9**) is "head-to-head" adduct. Only dimer (**9**) shows a fragment at *m*/z 360 (14%) and *m/z* 208 (2%) characteristic for an unsymmetrical reversion, in addition to the M$^+$/2 signal. In regard to *cis-trans* isomerization of the starting compounds and the existence of both isomers in the photochemical mixture one can expect eleven isomers formed by *trans-trans*, *trans-cis* and *cis-cis* dimerization in a "head-to-head" and "head-to-tail" modes. There are two signals for four cyclobutane protons in a ratio 1:1 and one signal of six protons for two methyl groups in the ^{1}H NMR spectra of all three methyl-dimers (Figure 1). The ^{13}C NMR spectra of these compounds show twenty-one signals: at low magnetic field seven singlets, ten aromatic doublets and one furanic doublet and in aliphatic region two cyclobutane doublets and one quartet for the methyl group. These results also confirm the symmetrical structures of **9**, **10** and **11**. Based on these data the number of possible structures can be reduced.



9a, 9b

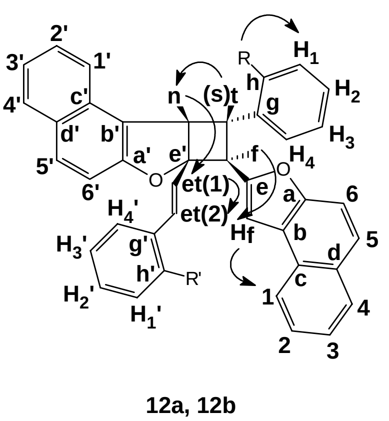


10a, 10b



11a, 11b

Compounds (**10**) and (**11**) could be I-III and compound (**9**) one structure from IV-VII. From the NOESY experiments on isomers (**9**, **10** and **11**) it is evident that only dimer (**11**) shows no interaction between cyclobutane protons **t** and **f**. This implies a structure with **t** and **f** protons on the opposite sides of the cyclobutane ring for **11**. Because of this the compound (**11**) is assigned the structure (I). The other important NOE shown on the compound of **11** confirm the structure. Compound (**9)** shows NOE between **t** and **f** protons and a very strong interaction of **f** and **H$_4$** protons. These data eliminated structures (V) and (VI) as possibilities for compound (**9**). Additional NOESY experiments, using a different mixing time,[8] were necessary to conclude that compound (**9**) has the structure VII with vicinal **f** and **t** protons and diagonally oriented **f** and **H$_4$** protons. Since compound (**10**) shows NOE between **t** and **H$_f$** as well as between **f** and **H$_4$** it has to have the structure (II).

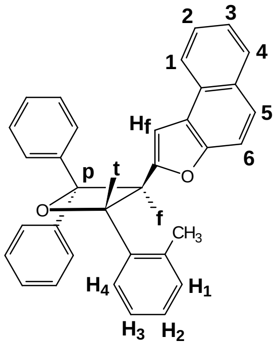


12a, 12b

One fused cyclobutane derivative (**12**) was isolated in the last fraction during the separation on the silica gel column in addition to cyclobutane products (**9**,**10** and **11**). This is a 2-oxabicyclo[3.2.0]hept-3-ene derivative, the only dimeric product obtained by intermolecular furan-ethene [2+2] photocycloaddition.

The structure of product **12a** and complete assignment of all hydrogen and carbon resonances were deduced based on different NMR techniques in various solvents using the model compound (**12b**).[2d] These have been described previously.

Irradiation of **3b** in benzene in the presence of benzophenone produces a benzophenone adduct, *trans*-2-(2-methylphenyl)-3-(2-naphtho[2,1-*b*]furyl)-4,4-diphenyl-1-oxetane (**13b**), as the main product. No energy transfer occurs. The orientation of addition of **3b** to the carbonyl group was determined from the characteristic fragmentation pattern in the MS spectrum while the *trans* configuration of the oxetane hydrogens was determined by NOESY experiment.



13b

ACKNOWLEDGMENT

We thank Prof. D. C. Neckers for critically reading the manuscript. Financial support from the Ministry of Science and Technology of the Republic of Croatia under contract 125004 is gratefully acknowledged.

EXPERIMENTAL

General. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Varian GEMINI 300 spectrometer at 300 and 75 MHz, respectively, in $CDCl_3$ and/or in C_6D_6 and Me_4Si as an internal standard. The assignment of the signals is based on 2D-CH correlation and 2D-HH-COSY and NOESY experiments. UV spectra were measured on Perkin Elmer LAMBDA 20 Spectrophotometer. HRMS spectra were measured on an Auto Spec Q (VG Analytical Manchester, GB). Elemental analyses were carried out in the Microanalytical Laboratory at The Rugjer Boskovic Institute. Irradiations were performed in a Quartz or Pyrex vessel in benzene solutions and in the Rayonet reactor equipped with RPR 3500 A lamps. Standard column chromatography was carried out on silica gel 60-230 mesh ASTM. Thin layer chromatography (TLC) was performed on Merck precouated silica gel 60 F_{254} plates. All irradiation experiments were carried out in degassed solutions made so by bubbling a stream of nitrogen or argon prior to irradiation. Solvents were purified by distillation. Melting points were determined on a Kofler micro hot-stage (Reichert, Wien) and are uncorrected. 2-Formylnaphtho[2,1-*b*]furan was prepared by the known procedure.[9]

2-[2-(2-Vinylphenyl)ethenyl]naphtho[2,1-*b*]furan (**3a**)[1] was prepared by a one-pot Wittig reaction[3] from α,α'-*o*-xylyl(ditriphenylphosphonium bromide), formaldehyde and 2-formylnaphtho[2,1-*b*]furan.

2-[2-(2-Methylphenyl)ethenyl]]naphtho[2,1-*b*]furan (**3b**) was prepared in 90% yield (1.43 g; *cis*:*trans*=1:2) from *o*-methylbenzyltriphenylphosphonium bromide (2.5 g, 5.6 mmol) and 2-formylnaphtho[2,1-*b*]furan (1.1 g, 5.6 mmol) with sodium ethoxide (0.4 g, 6 mmol) as a base according to the described procedure.[2d, 4] The reaction mixture was purified and separated by repeated column chromatography on silica gel using petroleum ether/ether mixture (9:1) as eluent. The first fractions yielded *cis*-isomer and the last fractions the *trans* product. Characterization data of new compounds are given bellow:

***cis*-3b**: light yellow oil; UV (EtOH, λ_{max}/nm, log ε): 212 (4.18), 244 (3.97), 344 (4.32); ^{1}H-NMR ($CDCl_3$): δ 7.89 (d, H-17, *J* 7.8 Hz,), 7.87 (d, H-20, *J* 7.8 Hz,), 7.63 (d, H-14, *J* 9.0 Hz), 7.49 (t, 1H, *J* 7.8 Hz), 7.48 (d, H-13, *J* 9.0 Hz), 7.44 (d, H-5, *J* 7.5 Hz), 7.43 (t, 1H, *J* 7.8 Hz), 7.31-7.18 (m, 3H, H-2,3,4), 6.76 (s, H-10), 6.74 (d, H-7, *J* 12.6 Hz), 6.68 (d, H-8, *J* 12.6 Hz), 2.32 (s, CH_3); ^{13}C-NMR ($CDCl_3$): δ 153.60 (s), 151.62 (s), 136.86 (s), 136.04 (s), 130.35 (d, C-7), 130.28 (s, C-16), 130.09 (d), 128.68 (2d, C-5,C-20), 127.85 (d), 127.38 (s, C-15), 126.11 (d), 125.74 (d), 125.31 (d, C-14), 124.46 (d), 124.03 (s, C-11), 123.36 (d, C-17), 119.19 (d, C-8), 112.10 (d, C-13), 104.40 (d, C-10), 19.65 (q, CH_3);

***trans*-3b**: yellow crystals, mp 118-120 °C (benzene /petroleum ether); UV (EtOH, λ_{max}/nm, log ε): 210 (4.62), 242 (4.10), 360 (4.57); ^{1}H-NMR ($CDCl_3$): δ 8.10 (d, H-17, *J* 8.1 Hz), 7.92 (d, H-20, *J* 8.1 Hz), 7.71 (d, H-14, *J* 8.8 Hz), 7.65 (d, H-13, *J* 8.8 Hz), 7.62 (d, H-5, *J* 7.8 Hz), 7.57 (t, H-18, *J* 7.8 Hz), 7.56 (d, H-7, *J* 15.9 Hz), 7.47 (t, H-19, *J* 7.8 Hz), 7.27-7.18 (m, 3H, H-2,3,4), 7.15 (s, H-10), 6.99 (d, 1H-8, *J* 15.9 Hz), 2.50 (s, CH_3); ^{13}C-NMR ($CDCl_3$): δ 154.90 (s), 152.42 (s), 136.21 (s), 135.60 (s), 130.60 (d), 130.35 (s, C-16), 128.77 (d, C-20), 127.91 (d), 127.43 (s, C-15), 127.00 (d, C-7), 126.26 (d, C-18), 126.23 (d), 125.52 (d, C-14), 124.97 (d, C-5), 124.54 (d, C-19), 124.48 (s, C-11), 123.45 (d, C-17), 117.47 (d, C-8), 112.04 (d, C-13), 104.15 (d, C-10), 19.80 (q, CH_3); MS *m/z*: 284 (M^+, 100%), 268 (19), 252 (13), 239 (18), 168 (17), 141 (19), 128 (30), 115 (11); Anal. Calcd for $C_{21}H_{16}O$: C, 88.69; H, 5.68. Found: C, 88.79; H, 5.89.

Irradiation experiments. A benzene solution (1.55×10^{-1} M) of **3a** (200 mg) and **3b** (220 mg), respectively, was irradiated under argon for 40 h in Rayonet (8 lamps) at 350 nm in a Pyrex tube. The solvent was removed in vacuum and the residue was chromatographed on a silica gel column as well as on TLC plates using petroleum ether-ether as the eluent in both cases.

Irradiation of 2-[2-(2-vinylphenyl)ethenyl]naphtho[2,1-*b*]furan (3a):

4-Vinyldinaphtho[2,1-*b*: 1',2'-*d*]furan (**7a**)[1] was eluted with petroleum ether in the first fractions from the column in 3.5% yield followed by a mixture (8%) of **3a** (*cis*:*trans*=1:3) and **8a** (6 mg, 3%). Eluting with the more polar petroleum ether/ether solution (5% to 50% ether) produced a mixture of dimeric products (131 mg, 65%) from which dimmers (**9a-12a**) were isolated. Some minor quantities of unidentified dimeric products (10 mg, 5%) were also obtained. Turry material (20%) remained on the column. Anal. Calcd for dimeric products $C_{44}H_{32}O_2$: C, 89.16; H, 5.44. Found: C, 89.21; H, 5.60.

6a,13c-Dihydro-4-vinyldinaphtho[2,1-*b*: 1',2'-*d*]furan (**8a**): 6 mg (3%); greenish oil; UV (EtOH, λ_{max}/nm, log ε): 212 (4.21), 231 (4.32), 328 (3.47), 344 (3.57), 360 (3.57); ^{1}H-NMR ($CDCl_3$): δ 8.05 (d, 1H, *J* 8.1 Hz), 7.88 (d, 1H, *J* 8.7 Hz), 7.85 (d, 1H, *J* 8.1 Hz), 7.81 (d, 1H, *J* 8.7 Hz), 7.54 (t, 1H, *J* 7.5 Hz), 7.46 (d, 1H, *J* 8.1 Hz), 7.39 (t, 2H, *J* 7.5 Hz), 7.25 (d, 1H, *J* 8.1 Hz), 7.11 (dd, 1H, *J* 17.1, 11.1 Hz, -$CH=CH_2$), 6.92 (s, 2H), 5.69 (dd, 1H, *J* 17.1, ~1 Hz, =CHH), 5.41 (dd, 1H, *J* 11.1, ~1 Hz, =CHH), 4.97 (d, 1H, *J* 17.7 Hz, H-j), 4.80 (dd, 1H, *J* 17.7, ~2 Hz, H-k); ^{13}C-NMR ($CDCl_3$): δ 160.51 (s), 138.85 (s), 135.74 (s), 134.45 (d), 132.88 (s), 132.26 (d), 130.55 (s), 130.33 (d), 129.39 (d), 129.05 (s), 127.10 (d), 126.76 (d), 125.10 (d), 124.82 (d), 124.76 (2d), 123.51 (d), 120.19 (s), 117.49 (t, =CH_2), 113.18 (d, C-6), 86.19 (d, C-k), 51.58 (d, C-j); MS *m/z*: 296 (M^+, 100%), 294 (63).

***r*-1,*t*-2-Di(2-naphtho[2,1-*b*]furyl)-*t*-3,*c*-4-di(2-vinylphenyl)cyclobutane [*r* -*ttc* hth dimer]** (**9a**):[10] 9 mg (4.5%); colourless crystals, mp 172-173 °C (benzene /petroleum ether); UV (EtOH, λ_{max}/nm, log ε): 217 (4.76), 230 (4.73, sh), 237 (4.70, sh), 244 (4.67, sh), 300 (4.15), 316 (4.08), 321 (4.00), 328 (4.23); ^{1}H-NMR ($CDCl_3$): δ 7.99 (d, 2H, *J* 8.1 Hz), 7.90 (d, 2H, *J* 7.8 Hz), 7.71 (d, 2H, *J* 8.1 Hz), 7.68 (s, 4H, H-5,H-6), 7.50 (t, 2H, *J* 7.5 Hz), 7.42 (t, 2H, *J* 7.5 Hz), 7.38 (t, 2H, *J* 7.5 Hz), 7.36 (d, 2H, *J* 7.8 Hz), 7.24 (t, 2H), 6.99 (s, 2H, H_f), 6.70 (dd, 2H, *J* 17.1, 10.8 Hz, -$CH=CH_2$), 5.31 (dd, 2H, *J* 17.1, ~1 Hz, =CHH), 4.87 (dd, 2H, *J* 10.8, ~1 Hz, =CHH), 4.49 (d, 2H, *J* 9.6 Hz), 4.13 (d, 2H, *J* 9.6 Hz); ^{13}C-NMR ($CDCl_3$): δ 156.33 (s), 152.28 (s), 138.04 (s), 137.70 (s), 134.60 (d), 130.14 (s), 128.52 (d), 127.95 (d), 127.40 (s), 126.91 (d), 126.34 (2d), 125.94 (d), 124.46 (d), 124.20 (d), 123.69 (s), 123.26 (d), 116.08 (t), 112.13 (d), 102.74 (d), 46.02 (d), 44.62 (d); MS (FAB) *m/z*: 592 (M^+, <1%), 360 (M^+-$CH_2=CHC_6H_4CH=CHC_6H_4CH=CH_2$, 5%), 296 ($M^+$/2, 30), 232 ($CH_2=CHC_6H_4CH=CHC_6H_4CH=CH_2$, 3%).

***r*-1,*t*-3-Di(2-naphtho[2,1-*b*]furyl)-*c*-2,*t*-4-di(2-vinylphenyl)cyclobutane [*r*-*ctt* htt dimer]** (**10a**): 32 mg (16%); colourless crystals, mp 70-71 °C (benzene /petroleum ether); UV (EtOH, λ_{max}/nm, log ε): 215 (4.42), 248 (4.31), 278 (3.77), 286 (3.83), 300 (3.70), 316 (3.63), 320 (3.52), 328 (3.77); ^{1}H-NMR ($CDCl_3$): δ 7.91 (d, 2H, *J* 8.1 Hz), 7.46 (d, 2H, *J* 8.1 Hz), 7.61-7.54 (m, 4H), 7.52-7.44 (m, 4H), 7.40 (t, 2H, *J* 7.5 Hz), 7.32-7.20 (m, 4H), 7.14-7.02 (m, 4H), 6.68 (s, 2H, H_f), 5.47 (dd, 2H, *J* 17.1, ~1 Hz, =CHH), 5.33 (dd, 2H, *J* 11.1, ~1 Hz, =CHH), 5.00 (dd, 2H, *J* 8.4, 7.8 Hz, H-f), 4.82 (dd, 2H, *J* 8.4, 7.8 Hz, H-s); ^{13}C-NMR ($CDCl_3$): δ 156.93 (s), 152.19 (s), 137.95 (s), 136.18 (s), 134.99 (d), 130.33 (s), 128.80 (d), 127.93 (d), 127.60 (s), 127.22 (d), 126.37 (d), 126.16 (d), 126.10 (d), 124.38 (2d), 123.79 (s), 123.58 (d), 116.56 (t), 112.30 (d), 103.26 (d), 44.36 (d), 40.89 (d); MS (FAB) *m/z*: 593 (MH^+, 2%), 296 ($M^+/2$, 64).

***r*-1,*c*-3-Di(2-naphtho[2,1-*b*]furyl)-*t*-2,*t*-4-di(2-vinylphenyl)cyclobutane [*r-tct* htt dimer]** (**11a**): 38 mg (19%); colourless crystals, mp 55-57 °C (benzene /petroleum ether); UV (EtOH, λ_{max}/nm, log ε): 225 (4.86), 233 (4.84), 243 (4.82), 255 (4.60, sh), 260 (4.50, sh), 287 (4.31), 295 (4.31), 314 (4.14), 322 (4.01), 328 (4.19); ^{1}H-NMR ($CDCl_3$): δ 7.95 (d, 2H, *J* 8.1 Hz), 7.83 (d, 2H, *J* 7.8 Hz), 7.55 (d, 2H, *J* 9.0 Hz), 7.50-7.28 (m, 10H), 7.18-7.06 (m, 4H), 7.00 (s, 2H, H_f), 6.99 (dd, 2H, *J* 17.1, 10.8 Hz, -CH=CH), 5.47 (dd, 2H, *J* 17.1, ~1 Hz, =CHH), 5.27 (dd, 2H, *J* 10.8, ~1 Hz, =CHH), 5.08 (d, 2H, *J* 6.6 Hz), 4.71 (d, 2H, *J* 6.6 Hz); ^{13}C-NMR ($CDCl_3$): δ 156.59 (s), 152 28 (s), 137.72 (s), 136.50 (s), 134.87 (d), 130.11 (s), 128.54 (d), 127 43 (d), 127.35 (s), 126.72 (d), 126.51 (d), 126.28 (d), 125.86 (d), 124.32 (d), 124.12 (d), 123.54 (s), 123.36 (d), 116.36 (t), 112.05 (d), 102.89 (d), 43.82 (d), 41.61 (d); MS (FAB) *m/z*: 592 (M^+, 2%), 296($M^+/2$, 100).

6-(2-Vinylphenyl)-1-[2-(2-vinylphenyl)ethenyl]-7-(2-naphtho[2,1-*b*]furyl)-3-[2,1]naphtho-2-oxabicyclo[3.2.0]hept-3-ene (**12a**): 42 mg (21%); colourless crystals, mp 70-71 °C (benzene/petroleum ether); UV (EtOH, λ_{max}/nm, log ε): 218 (4.76), 236 (4.86), 295 (4.09), 313 (3.98), 320 (3.89), 326 (4.05); ^{1}H-NMR ($CDCl_3$): δ 8.09 (d, 1H, *J* 8.1 Hz), 7.97 (d, 1H, *J* 8.1 Hz), 7.83 (d, 1H, *J* 9.0 Hz), 7.76 (d, 1H, *J* 8.1 Hz), 7.73-7.55 (m, 4H), 7.50 (t, 1H, *J* 7.5 Hz), 7.44-7.34 (m, 3H), 7.28-7.10 (m, 7H), 7.06 (t, 1H, *J* 7.2 Hz), 6.92-6.78 (m, 4H), 6.60 (d, 1H, *J* 15.6 Hz, H-et), 5.91 (dd, 1H, *J* 17.1, ~1Hz), 5.64 (dd, 1H, *J* 11.1, ~1 Hz), 5.50 (dd, 1H, *J* 17.1, ~1 Hz), 4.99 (dd, 1H, *J* 10.8, ~1 Hz), 4.91-4.79 (m, 3H, H-f,n,s); ^{1}H-NMR (C_6D_6): δ 7.76 (d, 1H, *J* 7.8 Hz), 7.67 (d, 1H, *J* 7.8 Hz), 7.57 (d, 1H, *J* 15.6 Hz, H-et), 7.52 (d, 1H, *J* 9.0 Hz), 7.46-7.21 (m, 10H), 7.10-6.99 (m, 3H), 6.96-6.71 (m, 7H, 1H_f), 6.51 (t, 1H, *J* 7.5 Hz), 5.76 (dd, 1H, *J* 17.1, ~1 Hz), 5.40 (dd, 1H, *J* 10.8, ~1 Hz), 5.38 (dd, 1H, *J* 17.1, ~1 Hz), 5.10 (d, 1H, *J* 11.4 Hz, H-f), 4.99 (dd, 1H, *J* 11.4, 8.1 Hz, H-s), 4.79 (dd, 1H, *J* 10.8, ~1 Hz), 4.71 (d, 1H, *J* 8.1 Hz, H-n); ^{13}C-NMR ($CDCl_3$): δ 157.85 (s), 154.54 (s), 152.68 (s), 137.16 (s), 136.24 (s), 134.95 (s), 134.80 (d), 134.77 (d), 134.46 (s), 132.09 (s), 130.42 (d), 130.23 (s), 129.62 (d), 129.31 (s), 128.65 (d), 128.11 (d), 127.80 (d), 127.71 (2d), 127.52 (d), 127.45 (s), 127.15 (2d), 126.40 (d), 126.23 (d), 126.12 (d), 126.08 (d), 125.99 (d), 124.69 (d), 124.32 (d), 123.56 (s), 123.36 (d), 123.05 (d), 122.96 (d), 118.60 (s), 117.34 (t), 116.21 (t), 112.88 (d), 112.24 (d), 103.47 (d, C-H_f), 91.49 (s, C-e'), 52.50 (d), 48.76 (d), 40.02 (d); MS (EI) *m/z*: 592 (M^+, 1%), 296 ($M^+/2$, 100), 181 (naphtho[2,1-*b*]furylmethyl, 90).

Irradiation of 2-[2-(2-methylphenyl)ethenyl]]naphtho[2,1-*b*]furan (**3b**): **7b** was eluted with petroleum ether in the first fractions from the column in 4.5% yield followed by a mixture (10.5%) of *cis*-**3b** (2.3%) and *trans*-**3b** (8.2%) and **8b** (10 mg, 4.5%). Eluting with the more polar petroleum ether/ether solution

(5→50% ether) yielded a mixture of dimeric products (154 mg, 70%) from which dimmers (**9b-12b**) were isolated besides some minor quantities of unidentified dimeric products (9.1 mg, 4.1%). Turry material (10.5%) remained on the column. Anal. Calcd for dimeric products $C_{42}H_{32}O_2$: C, 88.69; H, 5.68. Found: C, 88.75; H, 5.75.

4-Methyldinaphtho[2,1-*b*: 1',2'-*d*]furan (**7b**): 10 mg (4.5%); colourless crystals, mp 83-85 °C; UV (EtOH, λ_{max}/nm, log ε): 208 (4.64), 252 (4.28), 260 (4.33), 302 (3.90), 326 (3.81), 340 (4.23), 356 (4.43); ^{1}H-NMR ($CDCl_3$): δ 9.15 (d, 1H, *J* 8.1 Hz, H-1), 9.03 (d, 1H, *J* 8.1 Hz, H_3), 8.16 (d, 1H, *J* 9.3 Hz), 8.07 (d, 1H, *J* 8.1 Hz, H-4), 7.96 (d, 1H, *J* 9.0 Hz), 7.86 (d, 1H, *J* 9.3 Hz), 7.84 (d, 1H, *J* 9.0 Hz), 7.74 (t, 1H, *J* 7.8 Hz, H-2), 7.64 (t, 1H, *J* 7.8 Hz, H_2), 7.58 (t, 1H, *J* 7.8 Hz, H-3), 7.45 (d, 1H, *J* 7.8 Hz, H_1), 2.85 (s, 3H, CH_3); ^{13}C-NMR ($CDCl_3$): δ 154.72 (s), 154.45 (s), 135.86 (s), 131.46 (s), 130.39 (s), 129.70 (d, C-4), 129.08 (s), 128.91 (s), 128.64 (d), 126.37 (d, C-2), 126.05 (d, C-H_2), 125.88 (d, C-H_1), 125.84 (d, C-1), 124.62 (d, C-3), 124.41 (d), 124.34 (d, C-H_3), 120.18 (s), 119.68 (s), 112.93 (d), 112.44 (d), 20.70 (q, CH_3); MS (EI) *m/z*: 282 (M^+, 100%); Anal. Calcd for $C_{21}H_{14}O$: C, 89.34; H, 5.00. Found: C, 89.24; H, 4.99.

6a,13c-Dihydro-4-methyldinaphtho[2,1-*b*: 1',2'-*d*]furan (**8b**): 10 mg (4.5%); greenish oil; UV (EtOH, λ_{max}/nm, log ε): 226 (4.40), 260 (3.91), 284 (3.66), 328 (3.59), 340 (3.53), 356 (3.60), 390 (3.23), 414 (3.23); ^{1}H-NMR (C_6D_6): δ 8.06 (d, 1H, *J* 8.4 Hz, H-1), 7.82 (d, 1H, *J* 7.5 Hz, H_3), 7.66 (d, 1H, *J* 8.4 Hz, H-4), 7.48 (d, 1H, *J* 8.7 Hz, H-5), 7.29 (t, 1H, *J* 8.4 Hz, H-2), 7.20 (d, 1H, *J* 8.7 Hz, H-6), 7.18 (t, 1H, *J* 8.4 Hz, H-3), 6.93 (t, 1H, *J* 7.5 Hz, H_2), 6.88 (d, 1H, *J* 7.5 Hz, H_1), 6.72 (dd, 1H, *J* 9.9, 1.8 Hz, H-6'), 6.43 (dd, 1H, *J* 9.9, 2.4 Hz, H-5'), 4.98 (ddd, 1H, *J* 18.0, 2.4, 1.8 Hz, H-k), 4.73 (d, 1H, *J* 18.0 Hz, H-j), 2.06 (s, 3H, CH_3); ^{13}C-NMR (C_6D_6): δ 161.42 (s), 139.40 (s), 135.17 (s), 134.03 (s), 132.41 (s), 131.94 (d, C-6'), 131.26 (s), 130.80 (d, C-5), 129.85 (d, C-4), 129.40 (d, C-H_1), 127.25 (d, C-H_2), 127.00 (d, C-2), 125.62 (d, C-5'), 125.36 (d, C-1), 123.76 (d, C-3), 123.65 (d, C-H_3), 120.99 (s), 113.71 (d, C-6), 87.00 (d, C-k), 52.10 (d, C-j), 19.72 (q, CH_3); ^{1}H-NMR ($CDCl_3$): δ 8.07 (d, 1H, *J* 8.1 Hz, H-1), 7.87 (d, 1H, *J* 8.1 Hz, H_3), 7.78 (d, 1H, *J* 8.7 Hz, H-5), 7.76 (d, 1H, *J* 8.1 Hz, H-4), 7.52 (dd, 1H, *J* 8.1, 7.2 Hz, H-2), 7.38 (dd, 1H, *J* 8.1, 7.2 Hz, H-3), 7.24 (d, 1H, *J* 8.7 Hz, H-6), 7.17 (t, 1H, *J* 8.1 Hz, H_2), 7.16 (d, 1H, *J* 8.1 Hz, H_1), 6.88 (dd, 1H, *J* 10.2, ~1 Hz, H-6'), 6.80 (dd, 1H, *J* 10.2, 2.1 Hz, H-5'), 4.96 (dd, 1H, *J* 18.0, 2.1 Hz, H-k), 4.80 (d, 1H, *J* 18.0 Hz, H-j), 2.42 (s, 3H, CH_3); ^{13}C-NMR ($CDCl_3$): δ 160.48 (s), 138.68 (s), 135.06 (s), 135.06 (s), 133.59 (s), 131.56 (d, C-6'), 130.53 (s), 130.22 (d, C-5), 129.33 (d, C-4), 129.11 (d, C-H_1), 126.85 (d, C-H_2), 126.68 (d, C-2), 125.38 (d, C-5'), 124.90 (d, C-1), 123.47 (d, C-3), 123.18 (d, C-H_3), 120.38 (s), 113.17 (d, C-6), 86.44 (d, C-k), 51.50 (d, C-j), 19.78 (q, CH_3); MS (FAB) *m/z*: 284 (M^+, 45%), 282 (100);

_r_-1,_t_-2-Di(2-naphtho[2,1-_b_]furyl)-_t_-3,_c_-4-di(2-tolyl)cyclobutane [_r-ttc_ hth dimer] (**9b**): 10 mg (4.5%); colourless crystals, mp 67-68 °C (benzene /petroleum ether); UV (EtOH, λ_{max}/nm, log ε): 216 (4.70), 300 (4.14), 314 (4.08), 328 (4.18); ^{1}H-NMR (C_6D_6): δ 7.87 (d, 2H, *J* 7.8 Hz, H-1), 7.86 (d, 2H, *J* 7.5 Hz, H_4), 7.72 (d, 2H, *J* 7.8 Hz, H-4), 7.57 (d, 2H, *J* 8.7 Hz, H-6), 7.43 (d, 2H, *J* 8.7 Hz, H-5), 7.35 (t, 2H, *J* 7.8 Hz, H-2), 7.27 (t, 2H, *J* 7.8 Hz, H-3), 7.25 (t, 2H, *J* 7.5 Hz, H_3), 7.05 (t, 2H, *J* 7.5 Hz, H_2), 6.92 (d, 2H, *J* 7.5 Hz, H_1), 6.89 (s, 2H, H_f), 4.62 (d, 2H, *J* 9.6 Hz, H-t), 4.38 (d, 2H, *J* 9.6 Hz, H-f), 1.98 (s, 6H, CH_3); ^{13}C-NMR (C_6D_6): δ 157.63 (s), 153.36 (s), 140.41 (s), 137.18 (s), 131.28 (s), 131.23 (d, C-H_1), 129.45 (d, C-4), 127.55 (s), 127.51 (d, C-H_2), 127.26 (d, C-H_3), 126.91 (d, C-H_4), 126.71 (d, C-2), 125.60 (d, C-5), 125.06 (d, C-3), 124.79 (s), 124.28 (d, C-1), 112.82 (d, C-6), 103.49 (d, C-H_f), 47.65 (d, C-t), 45.10 (d, C-f), 20.17 (q, CH_3); ^{1}H-NMR ($CDCl_3$): δ 8.01 (d, 2H, *J* 7.8 Hz, H-1), 7.91 (d, 2H, *J* 7.8 Hz, H-4), 7.76 (d, 2H, *J* 7.5 Hz, H_4), 7.70 (s, 4H, H-5, H-6), 7.51 (dd, 2H, *J* 7.8, 6.9 Hz, H-2), 7.43 (dd, 2H, *J* 7.8, 6.9 Hz, H-3), 7.33 (t, 2H, *J* 7.5 Hz, H_3), 7.15 (t, 2H, *J* 7.5 Hz, H_2), 7.08 (d, 2H, *J* 7.5 Hz, H_1), 7.00 (s, 2H, H_f), 4.42 (d, 2H, *J* 9.6 Hz, H-t), 4.15 (d, 2H, *J* 9.6 Hz, H-f), 2.05 (s, 6H, CH_3); ^{13}C-NMR ($CDCl_3$): δ 156.85 (s), 152.36 (s), 139.56 (s), 136.57 (s), 130.34 (d, C-H_1), 130.24 (s), 128.63 (d, C-4), 127.48 (s), 126.69 (d, C-H_2), 126.34 (d, C-H_3), 126.20 (d, C-H_4), 126.08 (d, C-2), 124.54 (d, C-5, C-6), 124.32 (d, C-3), 123.77 (s), 123.37 (d, C-1), 112.25 (d, C-6), 102.52 (d, C-H_f), 46.31 (d, C-t), 44.47 (d, C-f), 19.73 (q, CH_3); MS *m/z*: 568 (M^+, ~1%), 360 (M^+-$CH_3C_6H_4CH=CHC_6H_4CH_3$, 14%), 284 ($M^+$/2, 100%), 208 ($CH_3C_6H_4CH=CHC_6H_4CH_3$, 2%).

_r_-1,_t_-3-Di(2-naphtho[2,1-_b_]furyl)-_c_-2,_t_-4-di(2-tolyl)cyclobutane [_r-ctt_ htt dimer] (**10b**): 37 mg (16.8%); colourless crystals, mp 213-215 °C (benzene/petroleum ether); UV (EtOH, λ_{max}/nm, log ε): 218 (4.79), 246 (4.63), 300 (4.16), 314 (4.10), 328 (4.21); ^{1}H-NMR (C_6D_6): δ 7.83 (d, 2H, *J* 7.8 Hz, H-1), 7.67 (d, 2H, *J* 7.5 Hz, H_4), 7.65 (d, 2H, *J* 8.4 Hz, H-4), 7.41 (d, 2H, *J* 9.0 Hz, H-6), 7.32 (d, 2H, *J* 9.0 Hz, H-5), 7.30 (dd, 2H, *J* 7.8, 7.5 Hz, H-2), 7.23 (dd, 2H, *J* 8.4, 7.5 Hz, H-3), 7.11 (t, 2H, *J* 7.5 Hz, H_3), 6.93 (t, 2H, *J* 7.5 Hz, H_2), 6.85 (d, 2H, *J* 7.5 Hz, H_1), 6.61 (s, 2H, H_f), 5.06 (dd, 2H, *J* 8.6, 7.8 Hz, H-f), 4.79 (dd, 2H, *J* 8.6, 7.8 Hz, H-t), 2.16 (s, 6H, CH_3); ^{13}C-NMR (C_6D_6): δ 157.99 (s), 152.90 (s), 137.89 (s), 137.58 (s), 131.17 (s), 130.83 (d, C-H_1), 129.38 (d, C-4), 127.28 (s), 127.49 (d, C-H_2), 126.66 (d, C-H_3), 126.57 (d, C-H_4), 126.57 (d, C-2), 125.23 (d, C-5), 124.83 (d, C-3), 124.39 (s), 124.05 (d, C-1), 112.56 (d, C-6), 103.36 (d, C-H_f), 45.39 (d, C-t), 40.84 (d, C-f), 20.02 (q, CH_3); ^{1}H-NMR ($CDCl_3$): δ 7.91 (d, 2H, *J* 7.8 Hz, H-1), 7.87 (d, 2H, *J* 8.1 Hz, H-4), 7.58 (d, 2H, *J* 8.7 Hz, H-5), 7.57 (d, 2H, *J* 7.5 Hz, H_4), 7.50 (d, 2H, *J* 8.7 Hz, H-6), 7.48 (dd, 2H, *J* 7.8, 7.0 Hz, H-2), 7.40 (dd, 2H, *J* 8.1, 7.0 Hz, H-3), 7.18 (t, 2H, *J* 7.5 Hz, H_3), 7.04 (t, 2H, *J* 7.5 Hz, H_2), 7.00 (d, 2H, *J* 7.5 Hz, H_1), 6.66 (s, 2H, H_f), 5.01 (dd, 2H, *J* 8.6, 7.8 Hz, H-f), 4.69 (dd, 2H, *J* 8.6, 7.8 Hz, H-t), 2.33 (s, 6H, CH_3); ^{13}C-NMR ($CDCl_3$): δ 157.17 (s), 151.93 (s), 137.06 (s), 136.90 (s), 130.12 (d, C-H_1), 130.11 (s), 128.57 (d, C-4), 127.34 (s), 126.71 (d, C-

H$_2$), 125.88 (d, C-H$_3$), 125.88 (d, C-2), 125.77 (d, C-H$_4$), 124.14 (d, C-3), 124.14 (d, C-5), 123.50 (s), 123.31 (d, C-1), 112.04 (d, C-6), 102.50 (d, C-H$_f$), 44.61 (d, C-t), 40.01 (d, C-f), 19.63 (q, CH$_3$); MS *m/z*: 568 (M$^+$, ~1%), 284 (M$^+$/2, 100%).

***r*-1,*c*-3-Di(2-naphtho[2,1-*b*]furyl)-*t*-2,*t*-4-di(2-tolyl)cyclobutane [*r-tct* htt dimer]** (**11b**): 49 mg (22.3%); colourless crystals, mp 61-63 °C (benzene/petroleum ether); UV (EtOH, λ_{max}/nm, log ε): 218 (4.61), 298 (4.01), 314 (3.94), 328 (4.00); ^{1}H-NMR (C$_6$D$_6$): δ 7.81 (d, 2H, *J* 7.5 Hz, H-1), 7.61 (d, 2H, *J* 7.5 Hz, H-4), 7.31 (d, 2H, *J* 6.9 Hz, H$_4$), 7.28 (d, 2H, *J* 8.7 Hz, H-6), 7.24 (t, 2H, *J* 7.5 Hz, H-2), 7.23 (d, 2H, *J* 8.7 Hz, H-5), 7.21 (t, 2H, *J* 7.5 Hz, H-3), 7.02 (t, 2H, *J* 6.9 Hz, H$_3$), 6.99 (d, 2H, *J* 6.9 Hz, H$_1$), 6.98 (s, 2H, H$_f$), 6.94 (t, 2H, *J* 6.9 Hz, H$_2$), 5.13 (d, 2H, *J* 6.6 Hz, H-t), 4.59 (d, 2H, *J* 6.6 Hz, H-f), 2.12 (s, 6H, CH$_3$); ^{13}C-NMR (C$_6$D$_6$): δ 157.83 (s), 153.30 (s), 138.87 (s), 137.01 (s), 131.15 (s), 131.01 (d, C-H$_2$), 129.37 (d, C-4), 127.65 (s), 127.29 (d, C-H$_1$), 127.06 (d, C-H$_4$), 126.62 (d, C-H$_3$), 126.57 (d, C-2), 125.36 (d, C-5), 124.82 (d, C-5), 124.45 (s), 124.09 (d, C-1), 112.61 (d, C-6), 103.56 (d, C-H$_f$), 44.65 (d, C-t), 42.64 (d, C-f), 20.19 (q, CH$_3$); ^{1}H-NMR (CDCl$_3$): δ 7.95 (d, 2H, *J* 8.1 Hz, H-1), 7.84 (d, 2H, *J* 8.1 Hz, H-4), 7.55 (d, 2H, *J* 9.0 Hz, H-5), 7.46 (t, 2H, *J* 7.5 Hz, H-2), 7.44 (d, 2H, *J* 9.0 Hz, H-6), 7.38 (t, 2H, *J* 7.5 Hz, H-3), 7.26 (H$_4$, under the signal of solvent), 7.07 (m, 6H, H$_1$,H$_2$,H$_3$), 7.01 (s, 2H, H$_f$), 4.98 (d, 2H, *J* 6.8 Hz, H-t), 4.64 (d, 2H, *J* 6.8 Hz, H-f), 2.26 (s, 6H, CH$_3$); ^{13}C-NMR (CDCl$_3$): δ 156.88 (s), 152.24 (s), 137.96 (s), 136.47 (s), 130.24 (d), 130.12 (s), 128.54 (d, C-4), 127.35 (s), 126.48 (d), 126.17 (d, C-H$_4$), 125.85 (d, C-2), 125.74 (d), 124.26 (d, C-5), 124.11 (d, C-3), 123.56 (s), 123.36 (d, C-1), 112.04 (d, C-6), 102.72 (d, C-H$_f$), 43.75 (d, C-t), 41.78 (d, C-f), 19.81 (q, CH$_3$); MS *m/z*: 568 (M$^+$, <1%), 284 (M$^+$/2, 100%).

6-(2-Methylphenyl)-1-[2-(2-methylphenyl)ethenyl]-7-(2-naphtho[2,1-*b*]furyl)-3-[2,1]naphtho-2-oxa-bicyclo[3.2.0]hept-3-ene (**12b**):[2d] 49 mg (22.3%); colourless crystals, mp 81-82 °C (benzene/petroleum ether); UV (EtOH, λ_{max}/nm, log ε): 212 (4.68), 236 (4.68), 284 (4.07), 292 (4.04), 314 (3.93), 328 (4.02); ^{1}H-NMR (CDCl$_3$): δ 8.02 (d, 1H, *J* 8.1 Hz, H-1), 7.89 (d, 1H, *J* 8.1 Hz, H-4), 7.78 (d, 1H, *J* 9.0 Hz, H-5'), 7.70 (d, 1H, *J* 8.1 Hz, H-1'), 7.66 (d, 1H, *J* 9.0 Hz, H-5), 7.63 (d, 1H, *J* 9.0 Hz, H-6), 7.51 (t, 1H, *J* 7.3 Hz, H-2), 7.43 (t, 1H, *J* 7.3 Hz, H-3), 7.36 (d, 1H, *J* 9.0 Hz, H-6'), 7.23 (d, 1H, *J* 7.3 Hz, H$_1$), 7.21 (d, 1H, *J* 16.2 Hz, H-et(2)), 7.20 (t, 1H, *J* 7.3 Hz, H$_2$'), 7.13 (t, 1H, *J* 7.5 Hz, H-2'), 7.06 (2d, 2H, *J* 7.3 Hz, H$_1$',H$_4$'), 7.05 (s, 1H, H$_f$), 7.01 (t, 1H, *J* 7.3 Hz, H$_3$'), 6.99 (t, 1H, *J* 7.5 Hz, H-3'), 6.96 (t, 1H, *J* 7.3 Hz, H$_2$), 6.69 (d, 1H, *J* 7.3 Hz, H$_4$), 6.65 (t, 1H, *J* 7.3 Hz, H$_3$), 6.65 (d, 1H, *J* 7.5 Hz, H-4'), 6.60 (d, 1H, *J* 16.2 Hz, H-et(1)), 4.87 (d, 1H, *J* 11.4 Hz, H-f), 4.84 (d, 1H, *J* 7.5 Hz, H-n), 4.78 (dd, 1H, *J* 11.4, 7.5 Hz, H-t), 2.77 (s, 3H, CH$_3$), 2.27 (s, 3H, CH$_3$'); ^{13}C-NMR (CDCl$_3$): δ 157.91 (s), 154.62 (s), 152.62 (s), 135.78 (s), 135.72 (2s), 135.57 (s), 132.14 (s), 130.35 (d, C-5'), 130.20 (s), 130.14 (d, C-H$_3$'), 130.06 (d, C-H$_2$'), 129.52 (d, C-et(2)), 129.29 (s), 128.62 (d, C-4), 128.15 (d, C-1'), 127.69 (d), 127.43 (s), 126.86 (d, C-H$_2$), 126.31 (d, C-H$_4$), 126.16 (d, C-et(1)), 126.05 (3d, C-2, C-3', C-H$_1$), 126.05 (d), 125.85 (d), 124.62 (d, C-5), 124.28 (d, C-3), 123.56 (s), 123.34 (d, C-1), 122.91 (d, C-2'), 122.74 (d), 118.73 (s), 112.92 (d, C-6'), 112.18 (d, C-6), 103.41 (d, C-H$_f$), 91.59 (s, C-e'), 51.76 (d, C-n), 48.99 (d, C-f), 40.13 (d, C-t), 20.06 (q, **C**H$_3$), 19.56 (q, **C**H$_3$'); MS *m/z*: 568 (M$^+$, 2%), 284 (M$^+$/2, 100%).

Irradiation of 3b in the presence of benzophenone: A solution of 22 mg (0.08 mmol) of **3b** in 0.5 ml of benzene and 62 mg (0.34 mmol) of benzophenone was purged with argon and irradiated at 350 nm (8 lamps) for 11 h. Subsequent the solvent was removed in vacuum and the residue chromatographed on TLC silica gel plate eluting with petroleum ether-dichloromethane (3:2). The main product, ***trans*-2-(2-methylphenyl)-3-(2-naphtho[2,1-*b*]furyl)-4,4-diphenyl-1-oxetane** (**13b**), was isolated.

13b: 11mg (36%); colourless crystals, mp 65-66 °C; UV (EtOH, λ_{max}/nm, log ε): 217 (4.67), 245 (4.40), 260 (4.09), 285 (3.96, sh), 299 (4.03), 308 (3.90, sh), 313 (3.96), 320 (3.83), 327 (4.04); ^{1}H-NMR (C$_6$D$_6$): δ 8.05 (d, 1H, *J* 7.5 Hz, H$_4$), 7.86 (d, 1H, *J* 8.1 Hz, H-1), 7.73 (d, 2H, *J* 7.5 Hz), 7.65 (d, 1H, *J* 8.1 Hz, H-4), 7.59 (d, 2H, *J* 7.5 Hz), 7.33 (s, 2H, H-5,H-6), 7.33 (t, 1H, *J* 7.5 Hz, H-2), 7.25 (t, 1H, *J* 7.5 Hz, H-3), 7.19 (t, 2H, *J* 7.5 Hz), 7.09 (t, 1H, *J* 7.5 Hz, H$_2$), 7.05 (t, 1H, *J* 7.5 Hz, H$_3$), 7.02 (t, 1H, *J* 7.5 Hz), 6.97 (t, 2H, *J* 7.5 Hz), 6.88 (d, 1H, *J* 7.5 Hz, H$_1$), 6.87 (t, 1H, *J* 7.5 Hz), 6.62 (s, 1H, H$_f$), 6.56 (d, 1H, *J* 8.1 Hz, H-t), 4.96 (d, 1H, *J* 8.1 Hz, H-f), 1.93 (s, 3H, CH$_3$); ^{13}C-NMR (C$_6$D$_6$): δ 153.87 (s), 153.17 (s), 148.13 (s), 143.35 (s), 140.84 (s), 134.78 (s), 131.21 (s), 130.75 (d), 129.48 (d, C-4), 129.05 (2d), 128.62 (3d), 127.85 (d, C-H$_3$), 127.58 (d, C-H$_1$), 127.17 (d, C-H$_2$), 126.82 (d, C-2), 126.46 (2d), 126.26 (d, C-H$_4$), 125.82 (d, C-5), 125.71 (2d), 125.09 (d, C-3), 124.26 (s), 124.05 (d, C-1), 112.65 (d, C-6), 105.27 (d, C-H$_f$), 88.99 (s, C-p), 78.99 (d, C-t), 54.54 (d, C-f), 19.36 (q, CH$_3$); MS *m/z*: 466 (M$^+$, <1%), 346 (30), 284 (100); Anal. Calcd for $C_{34}H_{26}O_2$: C, 87.53; H, 5.62. Found: C, 87.38; H, 5.69.

Reaction of compound (**8b**) **with DDQ**. To a solution of **8b** (10 mg, 0.004 mmol) in benzene (10 mL), was added an exscess of DDQ (25 mg). After 2 h stirring at rt, the benzene solution was triturated with water and dried over magnesium sulfate. The solvent was removed under reduced pressure, and the solid residue was purified by column chromatography using silica gel and petrol ether/ether (5%) as the eluent. This yielded 5 mg (44.3%) of **7b**.

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