candidates for dynamic resolution, yet to our knowledge this is the first general synthesis of a class of atropisomeric compounds by a dynamic resolution using a recyclable resolving agent.

Received: March 12, 1999 [Z13152IE]
German version: *Angew. Chem.* **1999**, *111*, 2755 – 2757

Keywords: amides · asymmetric synthesis · atropisomerism · dynamic resolution

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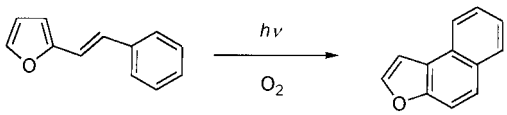
Novel Photochemical Rearrangement of Styrylfurans**

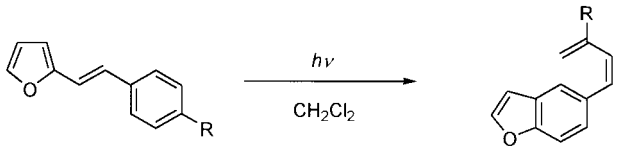
Tong-Ing Ho,* Jin-Yi Wu, and Shun-Li Wang

Rearrangement reactions are ubiquitous and they can occur both in the ground state as well as in the excited state.[1] The photochemical properties of stilbene-type compounds are well-documented[2–4] and major reactions include *cis*–*trans* isomerization, exciplex reactions, addition, and oxidative cyclization to phenanthrene. The mechanism for the oxidative cyclization involves a photochemically allowed six-electron conrotatory process to form a *trans*-dihydrophenanthrene intermediate;[4b] oxidation of this intermediate then affords the phenanthrene product. Styrylfuran is also known to undergo photochemical isomerization, and in the presence of oxygen or iodine it affords cyclized products [Eq. (1)].[5]



(1)

We have prepared several styrylfurans **1**–**11** with substituents in the *para* position of the benzene ring and now report a novel skeletal rearrangement of the styrylfurans (Scheme 1). The starting materials were prepared from 2-furaldehyde and



1 – **11** **12** – **19**

1, **12**, R = CH_3
2, **13**, R = CH_2CH_3
3, **14**, R = $CH(CH_3)_2$
4, **15**, R = $CH_2CH_2CH_3$
5, **16**, R = $C(CH_3)_3$
6, **17**, R = CH_2Ph
7, **18**, R = F
8, **19**, R = Cl
9, R = CN
10, R = NO_2
11, R = $N(CH_3)_2$

Scheme 1. Photochemical rearrangement of the styrylfuran derivatives **1**–**11**.

the corresponding benzyl chloride through a Wittig reaction. When N_2-degassed *p*-methylstyrylfuran **1** (1×10^{-2} M in CH_2Cl_2) was irradiated (350 nm) in a Rayonet reactor for 4 h the only product isolated was the 5-(3-methylbuta-1,3-dienyl)benzo[*b*]furan **12** (96% yield).[6] The presence of a 3-substituted 1,3-butadienyl group could be clearly identified

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[**] We are grateful to the National Science Council of the Republic of China (Taiwan) for financial support.

Supporting information for this article is available on the WWW under http://www.wiley-vch.de/home/angewandte/ or from the author.

  1433-7851/99/3817-2558 $ 17.50+.50/0

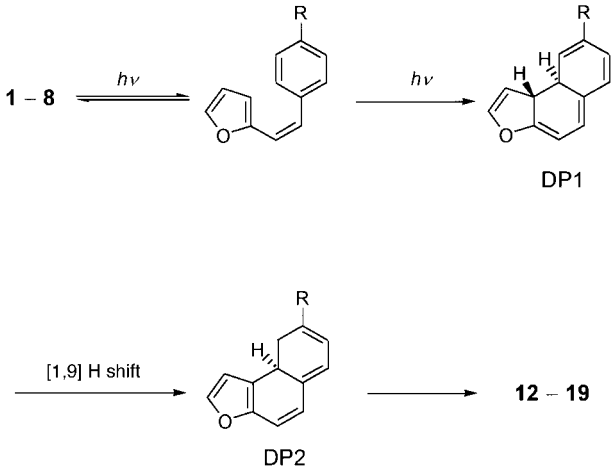
in the ^{1}H NMR spectrum of compound **12** by signals at $\delta =$ 6.53 (doublet, $J = 12.2$ Hz, $-$CH=), 6.17 (doublet, $J = 12.2$ Hz, =CH$-$), and 5.00 (multiplet, =CH_2). The yields for this new type of photochemical rearrangement are summarized in Table 1[7] for a series of styrylfurans. The yields of the isolated products are pretty good. For most cases only the *Z* isomer is obtained. Prolonged photolysis of the *Z* isomer produces a mixture of the *Z* and *E* isomers in which the *Z* isomer is still the major product. Hence, the *Z* form is the primary photoproduct. The reaction cannot be quenched by *trans*-1,3-pentadiene, a triplet quencher.[8] For both strong electron-withdrawing substituents (**9**, **10**) and strong electron-donating substituents (**11**) the reaction does not occur in dichloromethane even after prolonged photolysis (64 h). The reaction yield for the fluoro derivative (**18**) is moderate and for the chloro derivative (**19**) it is only 52%.

Table 1. The chemical yields for the photochemical reactions of **1**–**8** at 350 nm in dichloromethane.

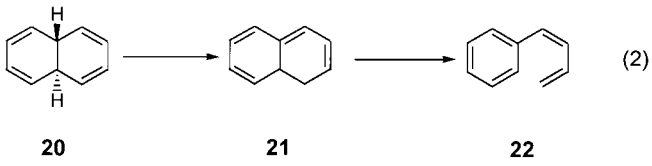
Reactant	*t* [h]	Product	Conversion [%]	Yield [%]
1	4	**12**	84	96[b]
2	3	**13**	81	81[a]
3	4	**14**	94	85[a]
4	3	**15**	90	82[a]
5	4	**16**	89	88[c]
6	3	**17**	81	95[d]
7	4	**18**	63	67[a]
8	64	**19**	62	52[e]

[a] Only the *Z* isomer. [b] With a *Z*:*E* ratio of 89:11. [c] With a *Z*:*E* ratio of 84:16. [d] With a *Z*:*E* ratio of 95:5. [e] With a *Z*:*E* ratio of 86:14.

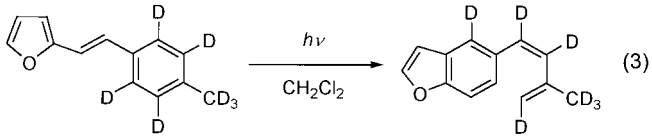
Irradiation of **1**–**11** in the presence of oxygen or iodine led to the isolation of the oxidative cyclization products [Eq. (1)]. The skeletal rearrangement occurred only when photolysis was carried out in the absence of oxygen. A reasonable explanation for this photoreaction is provided in Scheme 2. The reaction starts from the most stable helical conformers of *cis*-**1**–*cis*-**8** which absorb light to effect a conrotatory six-electron photocyclization reaction to yield the *trans*-fused dihydrophenanthrene-type[10] intermediate (DP1). This is followed by a [1,9] hydrogen shift to give the dihydrophenanthrene-type intermediate (DP2). A six-electron electrocyclic ring opening then follows,[9] which is initiated by the re-aromatization of the benzofuran from DP2 to afford the final products **12**–**19**. Attempts to isolate these intermediates (DP1, DP2) were not successful, even though there are successful examples[10] of trapping the dihydrophenanthrene intermediate. There is one example[11] of a homologous transformation from a dihydronaphthalene (**21**) to *cis*-1-phenylbuta-1,3-diene (**22**) from the thermal reaction of *trans*-9,10-dihydronaphthalene (**20**) [Eq. (2)].



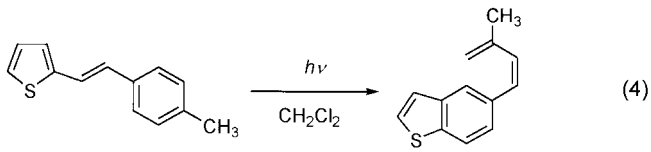
Scheme 2. Mechanism for the photochemical rearrangement of the styrylfurans **1**–**8**.



A semiempirical calculation (PM3) of the geometry of DP1 has indicated that the transferred hydrogen and the carbon atoms of DP1 are close spatially (2.68 Å) in the [1,9] hydrogen shift. The distance for the corresponding dihydrophenanthrene intermediate is slightly longer (2.71 Å). The presence of the oxygen atom in the furan ring might cause the greater acidity of the hydrogen atom needed for the [1,9] hydrogen shift to occur. To study this process we have prepared 2-{2-[2,3,5,6-tetradeutero-4-(trideuteromethyl)phenyl]vinyl}furan (**23**).[12] Photolysis of **23** in dichloromethane with light at 350 nm afforded 4-deutero-5-[1,2,4-trideutero-3-(trideuteromethyl)buta-1,3-dienyl]benzo[*b*]furan (**24**)[13] in 95% yield (*Z*:*E* = 19:1) [Eq. (3)]. The stereochemistry of **24** has not been investigated, but if the electrocyclic ring opening occurs by a photochemical conrotatory process then the terminal deuterium atom will be *cis* to the CD_3 group.



In conclusion, this novel and efficient photochemical rearrangement is both mechanistically and synthetically[14] interesting. The mechanism consists of a conrotatory photocyclization, a novel [1,9] hydrogen shift, and a lateral ring opening. Compared to the stilbene system the presence of the furan oxygen atom might cause the higher acidity of the hydrogen atom that initiates the [1,9] hydrogen shift. We have also investigated the photolysis of *p*-methylstyrylthiophene **25** and isolated 5-(3-methylbuta-1,3-dienyl)benzo[*b*]thiophene **26**[15] [Eq. (4)] in 63% yield. We belive that this novel



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rearrangement is initiated by the presence of a heteroatom and there must be other reactions similar to this one.

Received: January 11, 1999 [Z12888IE]
German version: *Angew. Chem.* **1999**, *111*, 2753 – 2755

Keywords: heterocycles · isomerizations · photochemistry · rearrangements

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[6] The photochemical reaction was carried out in degassed dichloromethane solution in a Pyrex tube using a Rayonet reactor (350 nm) at room temperature. The product **12** was isolated by column chromatography on silica gel, no other by-products were observed in the crude ^{1}H NMR spectra. **12**: ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 7.59$ (d, $J = 2.2$ Hz, 1H), 7.52 (d, $J = 0.8$ Hz, 1H), 7.40 (d, $J = 8.5$ Hz, 1H), 7.27 (dd, $J = 1.7$, 8.5 Hz, 1H), 6.72 (dd, $J = 0.8$, 2.2 Hz, 1H), 6.53 (d, $J = 12.2$ Hz, 1H), 6.17 (d, $J = 12.2$ Hz, 1H), 5.02 – 4.97 (m, 2H), 1.70 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): $\delta = 154.0$, 145.2, 142.1, 132.7, 132.1, 129.5, 127.1, 125.4, 121.3, 116.9, 110.6, 106.6, 22.1; MS (70 eV, EI): *m*/*z* (%): 184 (67, $[M^+]$), 169 (100, $[M^+ - CH_3]$), 155 (59), 141 (89), 115 (39), 105 (41), 91 (26), 77 (26); HR-MS: calcd for $C_{13}H_{12}O$: 184.0888; found: 184.0882.

[7] Spectral data for compound **13**: ^{1}H NMR (200 MHz, $CDCl_3$): $\delta =$ 7.58 – 7.57 (m, 2H), 7.39 (d, $J = 8.5$ Hz, 1H), 7.32 (dd, $J = 1.6$, 8.5 Hz, 1H), 6.71 (d, $J = 2.2$ Hz, 1H), 6.50 (d, $J = 12.3$ Hz, 1H), 6.10 (d, $J =$ 12.3 Hz, 1H), 4.98 (s, 2H), 2.10 (q, $J = 7.4$ Hz, 2H), 1.01 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): $\delta = 154.0$, 147.7, 145.1, 132.6, 131.2, 129.7, 127.2, 125.4, 121.3, 113.5, 110.8, 106.6, 28.8, 12.8. **14**: ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 7.63$ (s, 1H), 7.58 (d, $J = 2.2$ Hz, 1H), 7.38 (s, 2H), 6.71 (d, $J = 2.2$, Hz, 1H), 6.50 (d, $J = 12.4$ Hz, 1H), 6.07 (d, $J =$ 12.4 Hz, 1H), 4.95 (s, 2H), 2.41 (sept, $J = 6.8$ Hz, 1H), 1.12 (d, $J =$ 6.8 Hz, 6H); ^{13}C NMR (50 MHz, $CDCl_3$): $\delta = 154.0$, 151.6, 145.1, 132.4, 130.4, 130.0, 127.3, 125.4, 121.3, 111.5, 110.8, 106.6, 34.0, 21.7. **15**: ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 7.59 – 7.56$ (m, 2H), 7.39 (d, $J =$ 8.5 Hz, 1H), 7.33 (dd, $J = 1.6$, 8.5 Hz, 1H), 6.71 (dd, $J = 0.7$, 2.2 Hz, 1H), 6.49 (d, $J = 12.3$ Hz, 1H), 6.07 (d, $J = 12.3$ Hz, 1H), 5.00 – 4.96 (m, 2H), 2.08 (t, $J = 7.6$ Hz, 2H), 1.54 – 1.35 (m, 2H), 0.83 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): $\delta = 154.0$, 146.0, 145.1, 132.5, 131.1, 129.7, 129.5, 127.2, 125.4, 121.3, 114.7, 110.7, 106.6, 38.2, 21.5, 13.8. **16**: ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 7.66$ (m, 1H), 7.55 (d, $J = 2.2$ Hz, 1H), 7.45 (dd, $J = 1.8$, 8.6 Hz, 1H), 7.35 (d, $J = 8.6$ Hz, 1H), 6.64 (dd, $J = 0.8$, 2.2 Hz, 1H), 6.50 (d, $J = 12.4$ Hz, 1H), 6.17 (dd, $J = 1.3$, 12.4 Hz, 1H), 4.97 – 4.93 (m, 2H), 1.18 (s, 9H); ^{13}C NMR (50 MHz, $CDCl_3$): $\delta = 153.9$, 153.7, 145.0, 132.3, 129.9, 129.8, 127.2, 125.5, 121.4, 111.3, 110.7, 106.6, 35.9, 29.3. **17**: ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 7.57$ (d, $J = 2.2$ Hz, 1H), 7.50 (s, 1H), 7.38 (d, $J = 8.5$ Hz, 1H), 7.17 – 7.29 (m, 4H), 7.09 (d, $J = 8.3$ Hz, 2H), 6.69 (d, $J = 2.2$ Hz, 1H), 6.49 (d, $J =$ 12.3 Hz, 1H), 6.07 (d, $J = 12.3$ Hz, 1H), 5.09 (s, 1H), 4.96 (s, 1H), 3.40 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 154.1$, 145.2, 145.1, 139.4, 132.4, 130.5, 130.3, 129.0, 128.2, 127.3, 126.1, 125.3, 121.3, 116.6, 110.8, 106.6, 42.5. **18**: ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 7.61 – 7.58$ (m, 2H), 7.42 (d, $J = 8.5$ Hz, 1H), 7.28 (td, $J = 2.1$, 8.5 Hz, 1H), 6.73 (dd, $J = 0.9$, 2.1 Hz, 1H), 6.64 (d, $J = 12.6$ Hz, 1H), 5.93 (dd, $J = 12.6$, 26.2 Hz, 1H), 4.75 (ddd, $J = 1.2$, 2.7, 16.4 Hz, 1H), 4.56 (dd, $J = 2.7$, 47.4 Hz, 1H). (*E*)-**19**: ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 7.66$ (s, 1H), 7.61 (d, $J = 2.1$ Hz, 1H), 7.46 (d, $J = 8.7$ Hz, 1H), 7.41 (dd, $J = 1.5$, 8.7 Hz, 1H), 7.09 (d, $J =$ 15.3 Hz, 1H), 6.80 (d, $J = 15.3$ Hz, 1H), 6.75 (d, $J = 2.1$ Hz, 1H), 5.46 (s, 1H), 5.42 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 155.1$, 145.7, 138.8, 133.7, 131.1, 127.9, 124.5, 123.4, 120.0, 115.2, 111.7, 106.7.

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[12] **23**: ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 7.39$ (d, $J = 1.9$ Hz, 1H), 7.02 (d, $J = 16.2$ Hz, 1H), 6.84 (d, $J = 16.2$ Hz, 1H), 6.41 (dd, $J = 1.9$, 3.1 Hz, 1H), 6.32 (d, $J = 3.1$ Hz, 1H).

[13] **24**: ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 7.59$ (d, $J = 1.9$ Hz, 1H), 7.39 (d, $J = 8.6$ Hz, 1H), 7.26 (d, $J = 8.6$ Hz, 1H), 6.72 (d, $J = 1.9$ Hz, 1H), 5.01 (s, 1H).

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[15] **26**: ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 7.74$ (d, $J = 8.3$ Hz, 1H), 7.72 (s, 1H), 7.37 (d, $J = 5.6$ Hz, 1H), 7.32 (dd, $J = 1.6$, 8.3 Hz, 1H), 7.25 (d, $J =$ 5.6 Hz, 1H), 6.52 (d, $J = 12.3$ Hz, 1H), 6.19 (d, $J = 12.3$ Hz, 1H), 5.03 (s, 1H), 4.98(s, 1H), 1.71 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta =$ 142.0, 139.4, 138.2, 134.1, 132.7, 129.3, 126.5, 125.4, 123.8, 123.7, 121.7, 117.0, 22.2.

Cryo-TEM Snapshots of Ferritin Adsorbed on Small Zeolite Crystals**

Daniel Klint, Gunnel Karlsson, and Jan-Olov Bovin*

The development of cryo techniques in combination with transmission electron microscopy (TEM) has increased the number of biological systems that can be studied.[1] Cryo-TEM has become a common tool for the investigation of water/surfactant systems[2] and nucleation of inorganic crystals in solution.[3,4] We present for the first time direct imaging

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[**] The Swedish Natural Science Research Council is acknowledged for financial support. The Knut and Alice Wallenberg Foundation is acknowledged for funding the equipment at the Biomicroscopy Unit.

  1433-7851/99/3817-2560 $ 17.50+.50/0