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Tetrahedron Letters 45 (2004) 3663–3667
Tetrahedron Letters

Selective and efficient transformation of *N*-(4-substituted benzoyl)-α-dehydroarylalanine alkyl esters into 4,5-dihydrooxazole derivatives via photoinduced electron transfer

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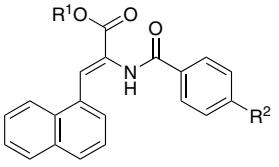
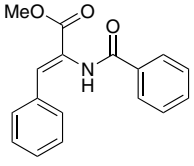
Received 19 January 2004; revised 4 March 2004; accepted 5 March 2004

Abstract—The irradiation of *N*-(4-substituted benzoyl)-α-dehydroarylalanine alkyl esters (**1**) in methanol containing triethylamine (TEA) was found to quantitatively give *cis*- and *trans*-4,5-dihydrooxazole derivatives (**2**), which were described as being formed via electron transfer from TEA to the excited-state (*E*)-**1** followed by kinetically-controlled cyclization of the (*E*)-**1**-derived anion radical. A product composition analysis showed that the *cis*-**2**/*trans*-**2** composition ratio is greatly varied depending on the stereoelectronic properties of the substituents, the polarity of protic solvents and the concentration of TEA.

Excited-state chemistry has continued to contribute to the development of new synthetic methods that enable the construction of a variety of hetero atom-containing rings.[1] In recent years much attention is being devoted to studies regarding photoinduced electron transfer reactions, owing to the fact that many of these reactions initiated by an electron transfer give products in high chemical and quantum yields.[1,2] In previous studies we found that *N*-acetyl-α-dehydrophenylalaninamides in polar solvents undergo a novel photocyclization giving isoquinoline and 1-azetine derivatives in satisfactory yields but with low efficiency.[3] The use of *N*-acetyl-α-dehydrophenylalanine alkyl esters as the starting α-dehydroamino acid derivatives enabled the selective formation of the corresponding isoquinolines by their photocyclization.[4] In addition, one-electron reduction of 1-naphthyl-substituted α-dehydroalaninamides was found to proceed efficiently affording 3,4-dihydrobenzoquinolinone derivatives in high selectivity.[5] The latter finding demonstrates the synthetic utility of electron transfer-initiated photocyclization of *N*-acyl-α-dehydroamino acids and, hence, it stimulated us to explore photoinduced electron transfer reactions of *N*-acyl-α-dehydroalanine alkyl esters, hoping to develop an efficient and highly selective phototransformation process of synthetic utility. To this end we designed and synthesized *N*-(4-substituted benzoyl)-α-dehydroarylalanine alkyl esters (**1a**–**g**) and examined the effects of the substituent and triethylamine (TEA) concentration on the reactivity of **1a** and the photoproduct composition.



(*Z*)-**1a** (R^1= Me, R^2= H); (*Z*)-**1b** (R^1= Et, R^2= H);
(*Z*)-**1c** (R^1= *i*-Pr, R^2= H); (*Z*)-**1d** (R^1= *t*-Bu, R^2= H);
(*Z*)-**1e** (R^1= Me, R^2= OMe); (*Z*)-**1f** (R^1= Me, R^2= CN)



(*Z*)-**1g**

Keywords: Amino acids and derivatives; Photochemistry; Electron transfer; Dihydrooxazoles; Substituent effects.
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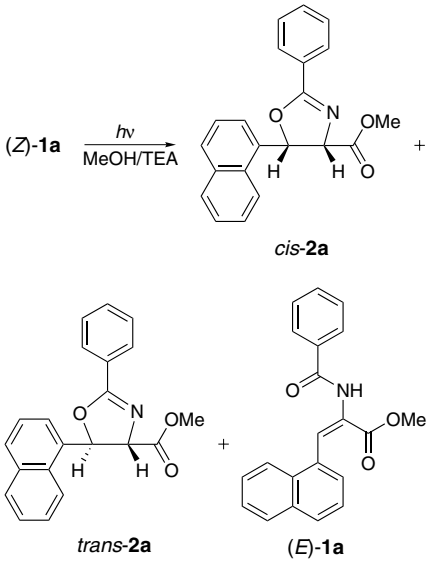
doi:10.1016/j.tetlet.2004.03.042

The starting (*Z*)-isomers [(*Z*)-**1a**–**g**] were prepared in good yields (42–71%) by the ring-opening reactions of (*Z*)-2-aryl-4-(1-arylmethylene)-5(4*H*)-oxazolones with the corresponding alcohols in the presence of TEA.[4,6] After a nitrogen-saturated methanol solution of (*Z*)-**1a** (4.0×10^{-3} mol dm^{-3}, 500 mL) containing TEA (0.10 mol dm^{-3}) was irradiated with Pyrex-filtered light (>280 nm) from a 400 W high-pressure Hg lamp for 5 h at room temperature (conversion, 100%), the reaction mixture obtained was subjected to preparative thin-layer chromatography over silica gel (eluent: EtOAc–hexane or EtOAc–$CHCl_3$), which allowed us to isolate *cis*- and *trans*-4-methoxycarbonyl-5-(1-naphthyl)-2-phenyl-4,5-dihydrooxazoles (*cis*-**2a**, 30%; *trans*-**2a**, 58% yield) having the vicinal coupling constants ($J_{4,5}$) of 10.3 and 6.9 Hz in DMSO-d_6, respectively, as shown in Scheme 1. The (*E*)-isomer of **1a** was isolated independently from the reaction mixture, which was irradiated for 0.5 h under the same conditions (conversion, 16.5%), by similar workup (Scheme 1). The structures of the isolated products were determined based on their spectroscopic and physical properties.[7] In addition, an X-ray analysis of *cis*-**2a**-derived single crystal provided definitive evidence for the structure of the *cis*-isomer with larger $J_{4,5}$ value (Fig. 1).[8]



Scheme 1.

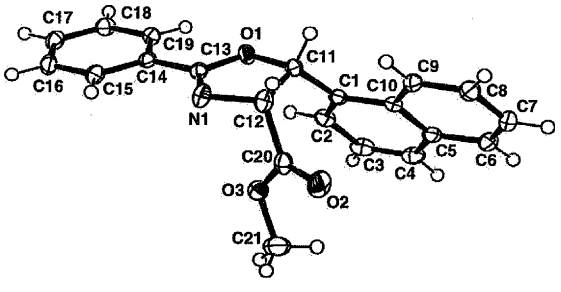


Figure 1. ORTEP drawing of *cis*-**2a**.

The finding that the photocyclization proceeds cleanly without forming any other products allows us to monitor the reaction by means of ^{1}H NMR spectroscopy and then to examine the effect of TEA concentration on the reactivity of **1a** and the composition ratio of *cis*-**2a** and *trans*-**2a**. For this examination, deaerated methanol solutions of (*Z*)-**1a** (4.0×10^{-3} mol dm^{-3}, 10 mL), which contain varying amounts of TEA were irradiated in parallel for 3 h on a merry-go-round apparatus using the same filter and light source. ^{1}H NMR spectral analysis of the reaction mixture obtained after usual workup gave the results collected in Table 1, which demonstrate that the reactivity is increased with an increase in TEA concentration while negligible amounts of the products are observed without TEA. This is consistent with the participation of an electron transfer mechanism. The data of Table 1 also show that the composition ratio of *cis*-**2a** to *trans*-**2a** decreases with increasing TEA concentration, suggesting the occurrence of TEA-catalyzed isomerization to the thermodynamically more stable *trans*-isomer. This suggestion was substantiated by the observation that on allowing a deuterated methanol solution of *cis*-**2a** (4.0×10^{-3} mol dm^{-3}, 1.0 mL) containing TEA (0.10 mol dm^{-3}) to stand at room temperature, the isomerization to the *trans*-isomer slowly proceeds to furnish a 9:1 equilibrium mixture of *trans*-**2a** and *cis*-**2a**, respectively, after 7 days. Additionally, a deuterium atom was incorporated at the 4-position on the oxazole ring when the equilibrium was established, confirming the existence of a carbanion intermediate in the isomerization process. Thus, we are allowed to preferentially obtain either of these two isomers by controlling the amine concentration and also to mainly generate the *trans*-isomer by allowing the reaction mixture to stand in the presence of a tertiary amine having high basicity.

In order to explore the substituent effects on the reactivity of the excited-state **1a** and the extent to which *cis*-**2a** is isomerized to *trans*-**2a**, an oxygen-free methanol or ethanol solution of **1b**–**g** (4.0×10^{-3} mol dm^{-3}, 500 mL) being irradiated in the presence of TEA (0.10 mol dm^{-3}) for a given period of time was subjected to ^{1}H NMR spectral analysis in DMSO-d_6, which gave the results collected in Table 2. Interestingly, the introduction of a bulky alkyl substituent such as an isopropyl or a *tert*-butyl group into the alkoxycarbonyl moiety of (*Z*)-**1** produced the corresponding *cis*-isomer in high selectivity and, in addition, considerably suppressed the isomerization induced by TEA. The suppression of this isomerization was also achieved by employing the less polar protic solvent, ethanol. It is very likely that the abstraction of the proton at the 4-position by TEA is subject to large steric and solvent polarity effects. On the other hand, the presence of an electron-donating methoxy or an electron-withdrawing cyano group at the *para*-position on the *N*-benzoyl benzene ring both lowers the excited-state reactivity of **1a**, suggesting that if we accept an electron transfer mechanism, the anion radical generated is delocalized into the *N*-acyl moiety. Furthermore, the finding that the isomer ratio of *cis*-**2** to *trans*-**2** decreases with increasing electron-withdrawing ability of the aryl group attached to the oxazole ring

Table 1. Effects of TEA concentration on the conversion of **1a** and the composition of each compound, obtained by the 3 h irradiation of (*Z*)-**1a** in methanol at room temperature

TEA (mol dm^{-3})	Conversion (%)	Composition (%)				*cis*-**2a**/*trans*-**2a**
		(*Z*)-**1**	(*E*)-**1**	*cis*-**2a**	*trans*-**2a**	
0	<0.1	>35.5	>65.4	<0.1	<0.1	—
0.01	48.7	17.8	33.5	38.8	9.9	3.9
0.05	70.4	10.2	19.5	48.2	22.2	2.2
0.10	74.6	8.8	16.6	43.9	30.7	1.4
0.20	80.2	7.0	12.8	36.4	43.8	0.8

Table 2. Substituent effects on the conversion of **1** and the composition of each compound, obtained by the irradiation of (*Z*)-**1** in methanol or ethanol at room temperature

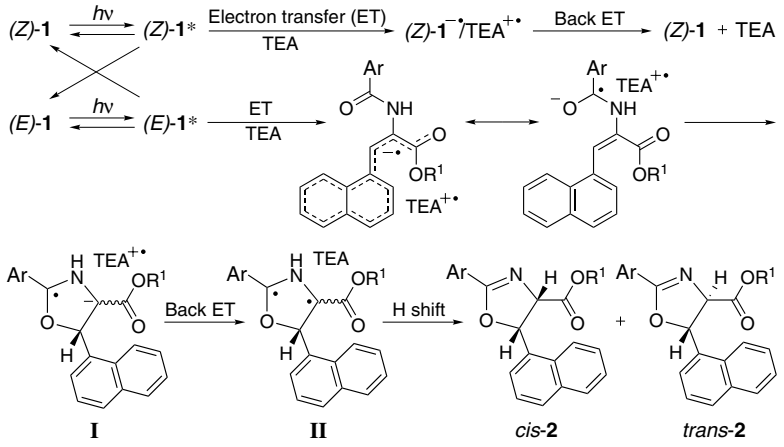
Compound	Irradiation time (h)	Conversion (%)	Composition (%)				*cis*-**2**/*trans*-**2**
			(*Z*)-**1**	(*E*)-**1**	*cis*-**2**	*trans*-**2**	
1a[a]	0.5	16.5	30.1	53.4	11.5	5.0	2.3
	5	100	0	0	36.9	63.1	0.6
1b[b]	0.5	18.9	29.8	51.3	13.2	5.7	2.3
	5	100	0	0	68.3	31.7	2.2
1c[a]	0.5	18.9	25.2	55.9	16.5	2.4	6.9
	5	100	0	0	80.9	19.1	4.2
1d[a]	0.5	19.7	25.5	54.8	18.1	1.6	11.3
	5	100	0	0	88.5	11.5	7.7
1e[a]	0.5	12.6	33.5	53.9	9.8	2.8	3.5
	5	92.7	2.9	4.4	60.6	32.1	1.9
1f[a]	0.5	9.0	47.9	43.1	4.6	4.4	1.0
	5	75.4	12.3	12.4	13.5	61.9	0.2
1g[a]	0.5	8.7	75.9	15.4	5.0	3.7	1.4
	5	73.1	18.1	8.8	20.6	52.5	0.4

[a] In methanol.
[b] In ethanol. This solvent was used because of the occurrence of TEA-catalyzed ester exchange reaction in methanol.

(**2e** > **2a** > **2f**) provides additional evidence for the occurrence of TEA-induced isomerization of the *cis*-isomer. An inspection of the data in Table 2 confirms that replacement of the 1-naphthyl group in **1a** by the phenyl (**1g**) still enables selective phototransformation into **2g**, though it is responsible for the decreased excited-state reactivity as well as for the enhanced isomerization rate.

In addition to the principle of least motion,[9] molecular modeling of (*Z*)-**1a** and (*E*)-**1a** reveals that the latter isomer adopts a most suitable conformation for the cyclization reaction that eventually gives **2a**. The previous findings that the triplet-sensitized reaction of (*Z*)-*N*-acyl-α-dehydrophenylalanine derivatives gives only the corresponding (*E*)-isomers without forming any cyclized products strongly suggest the involvement of singlet excited-state **1a** in the electron transfer process.[3,4] Although the very weak fluorescence of **1a** made it extremely difficult to measure emission quenching in the presence of TEA, the simplified Weller equation: $\Delta G_{et}/\mathrm{kJ\,mol^{-1}} = 96.5(E_{ox} - E_{red}) - E_S$,[10] where E_{ox}, E_{red} and E_S refer to the oxidation potential of TEA (0.76 V vs Ag/AgCl in MeCN), the reduction potential of **1a** (−2.26 V vs Ag/AgCl in MeCN) and the first singlet excitation energy of **1a** (368 kJ mol^{-1} in MeCN), respectively, allowed us to estimate the free energy change (ΔG_{et}) for electron transfer from TEA to singlet **1a** as −77 kJ mol^{-1}. Thus, taking into account the fact that the TEA concentration remains constant during the irradiation,[11] we were led to propose Scheme 2 that explains the formation of dihydrooxazole derivatives (**2a**–**g**).

Because the *para*-substituted benzoyl chromophores exhibit only very little absorption in the range of 300–400 nm, an electron from TEA should at first migrate to the naphthylmethylene moiety and then should be delocalized into the *N*-acyl moiety. This interpretation is substantiated by the finding that the fluorescence intensity of **1** is decreased in the following order: **1e** (R^2 = OMe) > **1a** (R^2 = H) > **1f** (R^2 = CN, virtually nonfluorescent). The observation of the TEA-catalyzed isomerization of **2** allows us to assume the anion radical intermediate **I** formed by the nucleophilic attack of *N*-acyl carbonyl oxygen upon the olefinic carbon in the (*E*)-**1**-derived anion radical (Scheme 2). Back electron transfer to the TEA cation radical followed by hydrogen shift in **II** affords *cis*-**2** and *trans*-**2**. The presence of a cyano group as the substituent R^2 is considered to lower the nucleophilicity of the acyl carbonyl oxygen through stabilization of the anion radical delocalized into the acyl moiety, as already described. It is very likely that the electron-donating methoxy substituent in **1e** suppresses delocalization of the anion radical into the acyl moiety to induce a decrease in reactivity of the carbonyl oxygen. The fact that the thermodynamically less stable *cis*-isomer is preferentially formed leads us to propose



Scheme 2.

that electrostatic attraction between the anion radical **I** and the TEA cation radical, as well as hydrogen bonding solvation of **I** (by methanol) and **II** (by methanol and TEA), renders an electron transfer-initiated cyclization a kinetically-controlled process. Since electrostatic and hydrogen bonding interactions in the *cis*-configurations may undergo less steric hindrance, hydrogen shift from *cis*-**II** should be accelerated to a much greater extent, as compared to that from *trans*-**II**, resulting in a preferential formation of the *cis*-isomers.

Although there are several synthetic routes to dihydrooxazole derivatives, no convenient photochemical route to these derivatives is known.[12] The procedure for preparing the starting α-dehydroamino acids (*Z*)-**1a**–**g** is very simple and easily applicable to their related compounds. The photoinduced electron transfer reaction of (*Z*)-*N*-(4-substituted benzoyl)-α-dehydroarylalanine alkyl esters described above, therefore, constitutes a novel photochemical method for the construction of a dihydrooxazole ring.

Acknowledgements

This research was partially supported by a 'High-Tech Research Project' from the Ministry of Education, Sports, Culture, Science and Technology, Japan.

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Data for (*Z*)-**1a**. Mp 118.0–118.5 °C. IR (KBr): 3200, 1732, 1632 cm^{-1}. ^1H NMR (600 MHz, DMSO-d_6): δ 3.80 (3H, s), 7.46 (2H, dd, $J = 7.6$, 7.6 Hz), 7.51 (1H, dd, $J = 7.6$, 8.9 Hz), 7.55 (1H, dd, $J = 7.6$, 7.6 Hz), 7.58 (1H, dd, $J = 6.9$, 6.9 Hz), 7.59 (1H, dd, $J = 6.9$, 7.6 Hz), 7.69 (1H, d, $J = 7.6$ Hz), 7.84 (2H, d, $J = 7.6$ Hz), 7.90 (1H, s), 7.94 (1H, d, $J = 8.9$ Hz), 7.97 (1H, d, $J = 6.9$ Hz), 8.05 (1H, d, $J = 7.6$ Hz), 10.0 (1H, s). ^{13}C NMR (150 MHz, DMSO-d_6) δ 52.3, 124.1, 125.4, 126.2, 126.62, 126.65, 127.6 (2C), 128.3 (2C), 128.5, 128.9, 129.2, 129.9, 130.5, 131.0, 131.8, 133.15, 133.22, 165.3, 166.4. Anal. Calcd (found) for $C_{21}H_{17}NO_3$: C, 76.12 (75.88); H, 5.17 (4.98); N, 4.23% (4.19%).
7. Data for (*E*)-**1a**. Mp 156.0–157.0 °C. IR (KBr): 3240, 1736, 1632 cm^{-1}. ^1H NMR (600 MHz, DMSO-d_6): δ 3.47 (3H, s), 7.32 (1H, d, $J = 7.4$ Hz), 7.34 (1H, s), 7.49 (1H, dd, $J = 7.4$, 8.0 Hz), 7.55–7.61 (2H, m), 7.57 (2H, dd, $J = 7.4$, 8.6 Hz), 7.64 (1H, dd, $J = 7.4$, 7.4 Hz), 7.90 (1H, d, $J = 8.0$ Hz), 7.95–7.98 (1H, m), 8.00 (2H, d, $J = 8.6$ Hz), 8.02–8.04 (1H, m), 10.7 (1H, s). ^{13}C NMR (150 MHz, DMSO-d_6) δ 51.7, 120.0, 124.3, 125.5, 125.7, 126.2, 126.5, 127.8 (2C), 128.1, 128.5, 128.6 (2C), 131.0, 131.1, 131.6, 132.2, 132.8, 133.1, 165.09, 165.10. Anal. Calcd (found) for $C_{21}H_{17}NO_3$: C, 76.12 (76.02); H, 5.17 (5.26); N, 4.23% (4.09%).
Data for *cis*-**2a**. Mp 133.0–134.0 °C. IR (KBr): 1742, 1646 cm^{-1}. ^1H NMR (600 MHz, DMSO-d_6): δ 2.72 (3H, s), 5.62 (1H, d, $J = 10.3$ Hz), 6.88 (1H, d, $J = 10.3$ Hz), 7.51–7.52 (2H, m), 7.57 (1H, dd, $J = 7.4$, 7.4 Hz), 7.59 (2H, dd, $J = 7.4$, 7.7 Hz), 7.60 (1H, dd, $J = 7.4$, 8.7 Hz), 7.67 (1H, dd, $J = 7.4$, 7.4 Hz), 7.90–7.92 (1H, m), 7.96 (1H, d, $J = 7.4$ Hz), 8.08 (2H, d, $J = 7.7$ Hz), 8.10 (1H, d, $J = 8.7$ Hz). ^{13}C NMR (150 MHz, DMSO-d_6) δ 50.7, 72.7, 80.0, 122.9, 123.6, 125.1, 125.9, 126.2, 126.4, 128.2 (2C), 128.3, 128.4, 128.9 (2C), 129.7, 132.0, 132.3, 132.8, 165.2,

169.0. Anal. Calcd (found) for $C_{21}H_{17}NO_3$: C, 76.12 (75.89); H, 5.17 (5.20); N, 4.23% (4.48%).
Data for *trans*-**2a**. Mp 108.0–109.0 °C. IR (KBr): 1742, 1640 cm^{-1}. ^1H NMR (600 MHz, DMSO-d_6): δ 3.81 (3H, s), 4.89 (1H, d, $J = 6.9$ Hz), 6.70 (1H, d, $J = 6.9$ Hz), 7.52 (1H, d, $J = 7.4$ Hz), 7.54 (1H, dd, $J = 6.9$, 7.4 Hz), 7.57 (2H, dd, $J = 7.4$, 7.4 Hz), 7.62 (1H, dd, $J = 8.6$, 9.2 Hz), 7.62 (1H, dd, $J = 6.3$, 9.2 Hz), 7.67 (1H, dd, $J = 7.4$, 7.4 Hz), 7.98 (1H, d, $J = 6.9$ Hz), 8.00 (1H, d, $J = 8.6$ Hz), 8.04 (1H, d, $J = 6.3$ Hz), 8.05 (2H, d, $J = 7.4$ Hz). ^{13}C NMR (150 MHz, DMSO-d_6) δ 53.3, 75.8, 81.2, 123.3, 123.7, 126.1, 126.8, 126.9, 127.4, 128.9 (2C), 129.5 (2C), 129.6, 129.7, 130.0, 133.0, 134.1, 135.0, 165.1, 171.5. Anal. Calcd (found) for $C_{21}H_{17}NO_3$: C, 76.12 (76.14); H, 5.17 (5.21); N, 4.23% (4.09%).

8. Crystal data for *cis*-**2a**: $C_{21}H_{17}NO_3$, fw = 331.36; colourless prism, $0.35 \times 0.23 \times 0.20$ mm, monoclinic, space group $P2_1/c$; $a = 8.2216(4)$ Å, $b = 18.288(1)$ Å, $c = 10.9309(7)$ Å, $\alpha = 90.00°$, $\beta = 94.889(3)°$, $\gamma = 90.00°$, $V = 1637.55(18)$ Å^3; $Z = 4$; $D_{calc} = 1.344$ g cm^{-3}; $R = 0.0468$, $wR(F^2) = 0.1573$. Crystallographic data of *cis*-**2a** have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 228004. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44(0)-1223-336033 or e-mail: deposit@ccdc.cam.ac.uk).
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11. A ^1H NMR spectral analysis of the reaction mixture obtained by the irradiation of a CD_3OD solution of (*Z*)-**1a** (4.0×10^{-3} mol dm^{-3}) containing TEA (1.0×10^{-2} mol dm^{-3}) and 1,4-dioxane (internal standard, 1.0×10^{-2} mol dm^{-3}) for a given period of time showed no sign of a change in this amine concentration during the reaction.
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