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Photoreduction of ethyl phenylglyoxylate

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Abstract

In the presence of electron donors, ethyl phenylglyoxylate generally forms adducts as the result of cross-coupling between the radical pair after a proton transfer. Quenching rate constants of triplet methyl phenylglyoxylate by hydrogen and electron donors were measured. Electron donors quench the excited state more than three orders of magnitude more rapidly than hydrogen donors. Quantum yields of the starting material's disappearance were shown to be very different in the reaction with a hydrogen donor to that with an electron donor. Photoreduction by dithiane (dithiolane) situated intramolecularly was also studied.

Keywords: Photoreduction; Ethyl phenylglyoxylate; Electron donor; Quantum yield

1. Introduction

In some of the earliest systematic studies of organic photochemical reactions, Schönberg and his co-workers in Cairo studied the photoreductive dimerization of phenylglyoxylic acid, suggesting it to be a unique way of synthesizing dihydroxydiphenylsuccinic acid [1]. The reduction of alkyl esters of phenylglyoxylic acid alcohols to dihydroxydiphenylsuccinates has been previously reported [2]. In a hydrogen-donating solvent, the mechanism proposed was analogous to that of the well-studied reductive dimerization of benzophenone [2–5]. As part of an attempt to utilize the photochemistry of alkyl phenylglyoxylates to selectively modify a polymer surface, we have systematically studied phenylglyoxylates bearing various functionalities in the alkyl moiety [6]. We report here recent studies on the photoreduction of alkyl phenylglyoxylates in the presence of hydrogen and electron donors. While dihydroxydiphenylsuccinates are the only products resulting from reactions with hydrogen donors, additional adducts are produced in reactions with most electron donors. The quenching rate constant of triplet ethyl phenylglyoxylate by a typical electron donor is more than three orders of magnitude higher than that by a typical hydrogen donor. The quantum efficiency of starting material disappearance in a hydrogen-donating solvent differs sharply from that in an electron-donating solvent.

2. Experimental

2.1. Materials

Benzene was distilled from sodium benzophenone ketyl under argon immediately before use. Ethyl phenylglyoxylate (**1**) was obtained from Aldrich and purified by column chromatography before use. Other chemicals were the highest grade from commercial sources and used as received. The general procedures for irradiating samples and isolating products were detailed in a recent publication [7].

2.2. Time-resolved laser flash photolysis

Nanosecond laser flash photolyses were carried out on a setup described by Ford and Rodgers [8] using a Q-switched Nd:YAG laser as a pump light. Argon was passed through the sample cuvette for 10 min before and during the experiments.

2.3. Quantum yields

The photolyses of valerophenone in benzene ($\Phi_{\text{acetophenone}} = 0.33$) [9] as the actinometer were carried out in parallel with sample irradiations. De-aeration was achieved by purging dry argon for 15 min before irradiation. Quantities of product produced in the actinometer were monitored by gas chromatography (GC). Chlorobenzene was used as the internal standard for GC calibrations and quantitative measure-

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ments. The disappearance of **1** was monitored by nuclear magnetic resonance (NMR).

After a certain period of irradiation, a known volume of reaction mixture was sampled and a certain amount of mesitylene was added as an internal standard. Both the aromatic and aliphatic signals of the standard were used and the results averaged. The relaxation delay was set to 3 s in all NMR measurements.

2.4. Diethyl 2,3-dihydroxy-2,3-diphenylsuccinate (2)

This has been characterized in earlier studies [2,7].

2.5. Ethyl α-hydroxyl-α-(3′-pyridyl)-benzeneacetate (3)

^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 1.27 (t, J=7.2 Hz, 3H), 4.33 (qd, J_1=7.2 Hz, J_2=1.6 Hz, 2H), 7.31–7.40 (m, 3H), 7.42–7.46 (m, 1H), 7.49–7.51 (m, 2H), 8.11–8.14 (m, 1H), 8.59–8.64 (m, 2H). ^{13}C NMR (APT, 50 MHz, $CDCl_3$), δ (ppm): 13.91, 63.26, 80.04, 122.55, 126.86, 128.71, 128.43, 129.79, 132.54, 141.09, 148.79, 151.37, 172.86. Mass Spectrum (MS): 51 (7.1), 78 (31), 106 (74), 184 (100), 257 (M^+, 2.0). High Resolution Mass Spectrum (HRMS) *m/e* calculated for $C_{15}H_{15}NO_3$: 257.1052, measured: 257.1051.

2.6. Ethyl α-hydroxyl-α-methylthiomethyl-benzeneacetate (4)

^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 1.28 (t, J=7.2 Hz, 3H), 2.20 (s, 3H), 2.99 (d, J=14 Hz, 1H), 3.40 (dd, J_1=14 Hz, J_2=0.8 Hz, 1H), 4.01 (d, J=0.8 Hz, 1H), 4.21–4.31 (m, 2H), 7.29–7.35 (m, 3H), 7.60–7.63 (m, 2H). ^{13}C NMR (APT, 50 MHz, $CDCl_3$), δ (ppm): 13.97, 17.66, 44.34, 62.53, 79.71, 125.53, 127.99, 128.23, 140.71, 173.59. MS: 62 (35), 77 (24), 91 (18), 105 (100), 167 (9.0), 179 (8.8), 240 (M^+, 0.5). HRMS *m/e* calculated for $C_{12}H_{16}SO_3$: 240.0820, measured: 240.0816.

2.7. Ethyl α-(1,3-dithiolan-2-yl)-α-hydroxyl-benzeneacetate (5)

^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 1.28 (t, J=7.2 Hz, 3H), 3.09–3.12 (m, 1H), 3.19–3.24 (m, 1H), 3.36–3.45 (m, 2H), 3.95 (s, 1H), 4.27–4.36 (m, 2H), 5.51 (s, 1H), 7.29–7.38 (m, 3H), 7.65–7.69 (m, 2H). ^{13}C NMR (APT, 50 MHz, $CDCl_3$), δ (ppm): 13.91, 39.28, 39.57, 61.28, 62.62, 82.15, 125.88, 127.99, 128.24, 139.67, 172.79. MS: 61 (35), 77 (9.0), 105 (100), 284 (M^+, 0.01). HRMS *m/e* calculated for $C_{13}H_{16}S_2O_3$: 284.0541, measured: 284.0539.

2.8. 2-(3-Hydroxypropyl)-1,3-dithiolane (16a)

^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 1.68–1.75 (m, 2H), 1.88–1.93 (m, 2H), 2.19 (s, 1H), 3.17–3.29 (m, 4H), 3.66 (t, J=6.4 Hz, 2H), 4.51 (t, J=7.2 Hz, 1H). ^{13}C NMR (APT, 50 MHz, $CDCl_3$), δ (ppm): 31.98, 35.67, 38.31, 53.36, 61.99, 62.02. MS: 44 (19), 61 (12), 71 (11), 105 (100), 118 (7.9), 164 (M^+, 22). HRMS *m/e* calculated for $C_6H_{12}S_2O$: 164.0330, measured: 164.0330.

2.9. 2-(4-Hydroxybutyl)-1,3-dithiolane (16b)

^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 1.49–1.62 (m, 4H), 1.82–1.88 (m, 2H), 1.96 (s, 1H), 3.17–3.28 (m, 4H), 3.63 (t, J=6.4 Hz, 2H), 4.48 (t, J=6.8 Hz, 1H). ^{13}C NMR (APT, 50 MHz, $CDCl_3$), δ (ppm): 25.36, 32.11, 38.26, 39.02, 53.53, 62.39. MS: 45 (4.5), 61 (6.8), 85 (9.2), 105 (100), 132 (2.6), 178 (M^+, 15). HRMS *m/e* calculated for $C_7H_{14}S_2O$: 178.0486, measured: 178.0485.

2.10. 2-(3-Hydroxypropyl)-1,3-dithiane (16a′)

^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 1.75–1.91 (m, 6H), 2.06 (s, 1H), 2.81–2.92 (m, 4H), 3.66 (t, J=6.4 Hz, 2H), 4.08 (t, J=6.4 Hz, 1H). ^{13}C NMR (APT, 50 MHz, $CDCl_3$), δ (ppm): 25.83, 29.58, 30.29, 31.80, 47.16, 62.12. MS: 45 (9.8), 71 (45), 85 (10), 119 (100), 178 (M^+, 39). HRMS *m/e* calculated for $C_7H_{14}S_2O$: 178.0486, measured: 178.0485.

2.11. 2-(4-Hydroxybutyl)-1,3-dithiane (16b′)

This is obtained according to a reported procedure and its spectroscopic data is in agreement with the literature [Ref. [17]].

2.12. (1,3-Dithiolan-2-yl)propyl phenylglyoxylate (13a)

^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 1.93–1.96 (m, 4H), 3.16–3.27 (m, 4H), 4.41 (t, J=6.4 Hz, 2H), 4.51 (t, J=6.4 Hz, 1H), 7.49–7.54 (m, 2H), 7.64–7.68 (m, 1H), 7.99–8.01 (m, 2H). ^{13}C NMR (APT, 50 MHz, $CDCl_3$), δ (ppm): 27.69, 35.64, 38.37, 52.80, 65.45, 128.81, 129.88, 132.25, 134.85, 163.66, 186.09. MS: 44 (47), 77 (29), 87 (34), 105 (100), 145 (44), 296 (M^+, 2.9). HRMS *m/e* calculated for $C_{14}H_{16}S_2O_3$: 296.0541, measured: 296.0541.

2.13. (1,3-Dithiolan-2-yl)butyl phenylglyoxylate (13b)

^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 1.56–1.62 (m, 2H), 1.78–1.90 (m, 4H), 3.16–3.27 (m, 4H), 4.39 (t, J=6.4 Hz, 1H), 4.47 (t, J=6.4 Hz, 2H), 7.50–7.54 (m, 2H), 7.64–7.68 (m, 1H), 7.99–8.02 (m, 2H). ^{13}C NMR (APT, 50 MHz, $CDCl_3$), δ (ppm): 25.36, 28.01, 38.45, 38.88, 53.27, 65.85, 128.84, 129.95, 132.36, 134.85, 163.82, 186.27. MS: 77 (18), 105 (100), 132 (7.3), 177 (12), 310 (M^+, 6.2). HRMS *m/e* calculated for $C_{15}H_{18}S_2O_3$: 310.0697, measured: 310.0695.

*2.14. (1,3-Dithian-2-yl)propyl phenylglyoxylate (**14a**)*

^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 1.81–1.94 (m, 2H), 1.99–2.06 (m, 2H), 2.08–2.15 (m, 2H), 2.80–2.91 (m, 4H), 4.08 (t, J = 7.2 Hz, 1H), 4.42 (t, J = 6.4 Hz, 2H), 7.50–7.54 (m, 2H), 7.64–7.68 (m, 1H), 7.99–8.02 (m, 2H). ^{13}C NMR (APT, 50 MHz, $CDCl_3$), δ (ppm): 25.54, 25.78, 30.25, 31.74, 46.70, 65.52, 128.88, 129.99, 132.34, 134.90, 163.71, 186.15. MS: 77 (45), 87 (70), 105 (100), 119 (67), 159 (67), 177 (17), 310 (M^+, 18). HRMS *m/e* calculated for $C_{15}H_{18}S_2O_3$: 310.0697, measured: 310.0695.

*2.15. (1,3-Dithian-2-yl)butyl phenylglyoxylate (**14b**)*

^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 1.64–1.71 (m, 2H), 1.80–1.91 (m, 4H), 2.10–2.16 (m, 2H), 2.76–2.92 (m, 4H), 4.06 (t, J = 6.8 Hz, 1H), 4.41 (t, J = 7.2 Hz, 2H), 7.51–7.55 (m, 2H), 7.65–7.70 (m, 1H), 8.00–8.03 (m, 2H). ^{13}C NMR (APT, 50 MHz, $CDCl_3$), δ (ppm): 22.97, 25.90, 28.05, 30.38, 34.89, 47.18, 65.85, 128.88, 129.99, 132.39, 134.87, 163.84, 186.29. MS: 77 (36), 105 (91), 119 (100), 191 (30), 324 (M^+, 17). HRMS *m/e* calculated for $C_{16}H_{20}S_2O_3$: 324.0854, measured: 324.0853.

3. Results and discussion

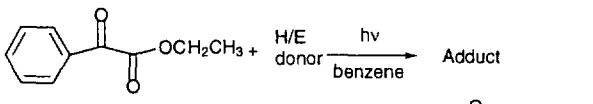
3.1. Photoreaction

Ethyl phenylglyoxylate (**1**) was irradiated in de-aerated benzene solution [0.05 M] together with 5 equivalents of either an electron or hydrogen donating component, Table 1, and the progress of the photolysis monitored by thin-layer chromatography (TLC). After ethyl phenylglyoxylate was consumed, the resulting mixture was sampled for an NMR measurement to assess the relative yields of products by repeated integrations of a representative signal from each product. The remainder of the solution was evaporated in vacuo and subjected to column chromatography on silica gel to obtain products, Table 1.

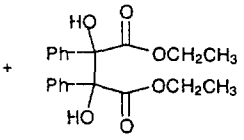
Diethyl 2,3-dihydroxy-2,3-diphenylsuccinate (**2**) was the only photoproduct isolated when the reaction was carried out in the presence of a hydrogen donor (isopropyl alcohol or ether). In addition to **2**, adducts in various yields resulted from the reactions when sulfides were added (entries 5 and 6 of Table 1). However, **2** was the only product when triethylamine was used as the electron donor. Adduct **3** was isolated from the reaction with pyridine.

Compounds **3**, **4**, and **5** were fully characterized by spectroscopic methods. The substitution position on the pyridyl ring in **3** was deduced from its NMR spectrum. In the aromatic region of its ^{1}H NMR spectrum, the two hydrogens ortho to N showed up as a very distinct signal at 8.59–8.64 ppm. The single hydrogen at the para position was at 8.11–8.14 ppm. The signal of the meta hydrogen appeared in the same region as that of the phenyl aromatic hydrogens and was less distinctive. However, the existence of two ortho and one para (with respect to N) hydrogens on the pyridyl ring of **3** left only the meta positions for substitution.

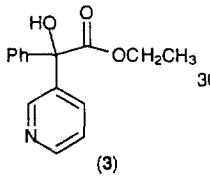
Table 1
Yields [a] of photoproducts



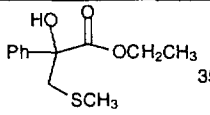
(1)



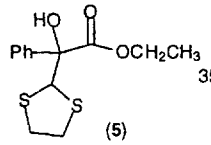
(2)

Entry	H/E donor	Adduct and Yield[b] (%)	Yield[b] (%) of **2**
1		/	86
2		/	79
3	$N(Et)_3$	/	73
4		 30 (54)	(46)

(3)

5		 35 (60)	(40)

(4)

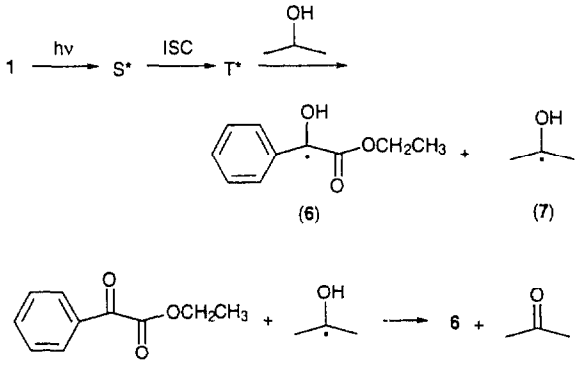
6		 35 (64)	(35)

(5)

[a] Isolated yields unless otherwise noted.
[b] Estimated yields from NMR are shown in parentheses.

3.2. Proposed mechanism

Mandelate radicals have been observed on a nanosecond timescale by laser flash photolyses of alkyl phenylglyoxylates in isopropyl alcohol [7]. If a good hydrogen donor is available, the mandelic radical (**6**) is produced from hydrogen abstraction by the triplet excited state of **1**, Scheme 1. A second radical **6** is derived from the reduction of a ground state molecule of **1** by the hydroxyalkyl radical **7**. The coupling of the two molecules of **6** produces **2**. The coupling of **6** with **7** does not occur to any significant degree.



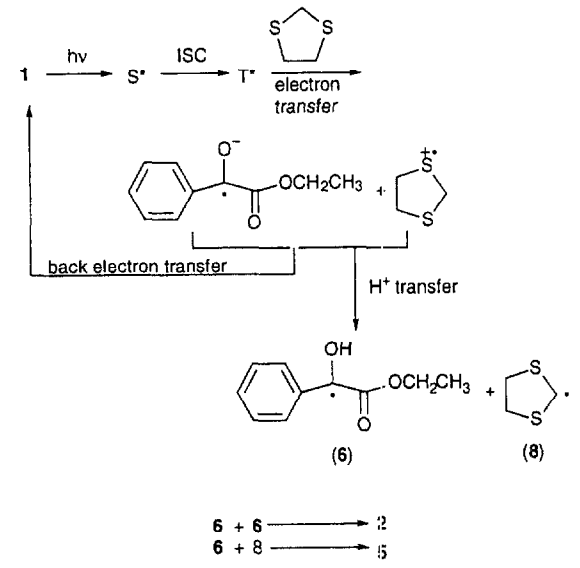
Scheme 1.

Electron transfer to the excited state carbonyl group followed by proton transfer results in the presence of an electron donor, Scheme 2.

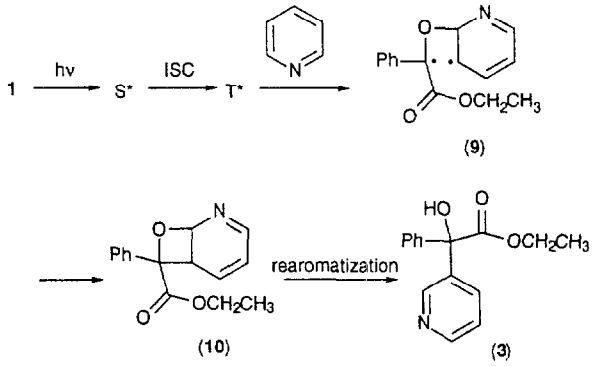
Regioselective proton transfer from the methylene group between the two S atoms in the dithiolane radical cation to the ketyl radical anion produces **6** and **8**. It is known that the protons on the methylene group between the two S atoms in dithiolane are more acidic than other protons [10]. Coupling of two mandelate radicals produces **2** and the reaction between **6** and **8** furnishes adduct **5**. It is noteworthy that though cross coupling between **6** and **8** results in significant product, radical **7** does not couple with **6**. This can be understood, in part, by considering that **7** is reactive with ground state **1** while **8** is not. As a result, the concentration of **7** in solution remains too low to produce a cross-coupling product.

With triethylamine the reaction expected is an electron transfer process similar to that outlined in Scheme 2. No adduct was observed.

Formation of **3** is explained by a 2 + 2 photocycloaddition process, Scheme 3. Electron rich alkenes undergo cycloaddition with alkyl phenylglyoxylates producing oxetanes as the only product [11]. Oxetane **10** is the immediate precursor of **3**. The regioselectivity observed in the formation of **10** is



Scheme 2.



Scheme 3.

the opposite of what would have been predicted from 'the most stable biradical' mechanism, suggesting other intermediates (such as an exciplex) before biradical **9**, the immediate intermediate to oxetane **10** [12].

We also noted that the yield of **2** is relatively high if the added pyridine participates only in the cycloaddition process. Since even benzene can serve as a hydrogen source for dimer formation [7], added pyridine also provides an additional hydrogen source and promotes the formation of **2**.

3.3. Quenching rate constants

The rates at which the triplet of **1** is quenched by a typical hydrogen donor (isopropyl alcohol) and a typical electron donor (methyl sulfide) were obtained by measuring the triplet lifetimes in the presence of differing concentrations of quencher in benzene. The quenching rate constant is obtained from the slope of a plot of k_{obs} against quencher concentrations, Fig. 1. The triplet is deactivated much faster with an electron donating quencher than with a hydrogen donating quencher. The quenching constant so obtained for isopropyl alcohol is 3.6×10^{6} M^{-1} s^{-1} and that for methyl sulfide is 3.9×10^{9} M^{-1} s^{-1}.

3.4. Quantum yields

Quantum yields for disappearance of **1** were measured in isopropyl alcohol and in methyl sulfide. Because **2** is not stable, decomposing to **1** and ethyl mandelate under GC analysis conditions, NMR was chosen to monitor the disappearance of **1**. A known amount of mesitylene was added to the NMR sample as the internal standard. At regular intervals after the beginning of the irradiation, samples were obtained for NMR analyses. [**1**] was determined by integrating its representative signal (~8.00 ppm) relative to the signals of the internal standard. The results are shown in Table 2 and are averages of three measurements.

A quantum yield higher than unity is observed in isopropyl alcohol due to reduction of **1** by **7**, Scheme 1. The observed quantum yield in methyl sulfide is much lower. This is attributed to the back electron transfer process between the radical ion pair, Scheme 2, which regenerates starting material **1** and dissipates photons.

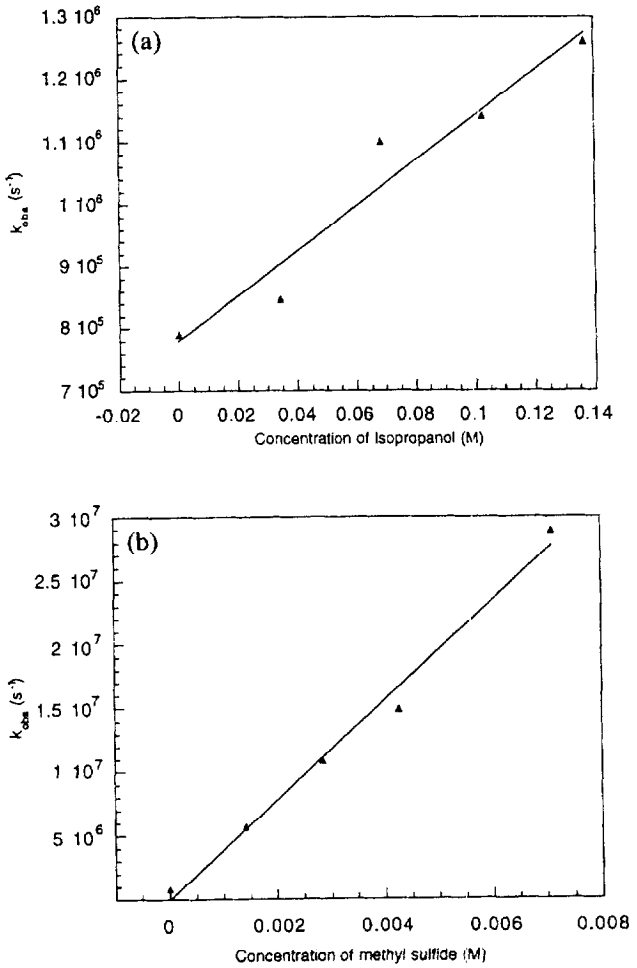


Fig. 1. Plots of decay rate constants of triplet of **1** (0.013 M in benzene) against quencher concentration. A: quencher is isopropyl alcohol, B: quencher is methyl sulfide.

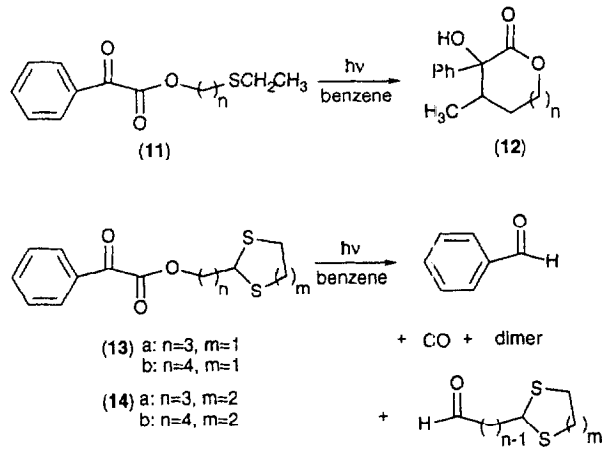
Table 2
Quantum yields of the disappearance of **1** in different solvents

Solvent	Isopropanol	Methyl sulfide
Φ	1.41 ± 0.21	0.23 ± 0.046

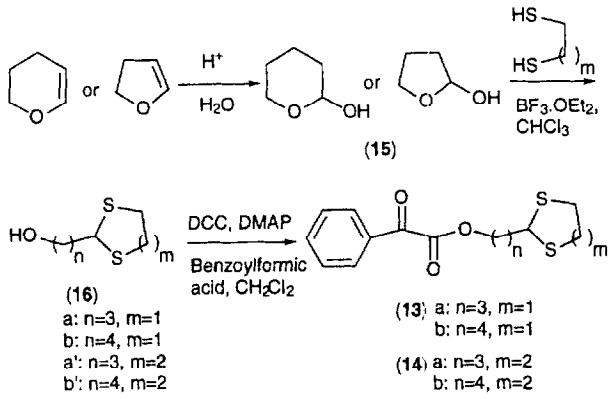
3.5. Intramolecular reduction

We have studied the photochemistry of sulfide substituted alkyl phenylglyoxylates (**11**) and found that an electron transfer induced cyclization results in cyclols **12** in good yields when $n = 2$–5, Scheme 4 [13,14].

Having observed that similar yields of adducts are formed from the photoreductions of **1** by methyl sulfide and by 1,3-dithiolane (Table 1), we surmised that **13** and **14** will react in an analogous fashion to **11** producing cyclols as products. Compounds **13** and **14** were synthesized, Scheme 5. 2,3-Dihydropyran (or 2,3-dihydrofuran) on treatment with aqueous hydrochloric acid afforded *n*-hydroxyl aldehyde **15** which exists in equilibrium with the cyclic hemiacetal form shown in Scheme 5 [15]. On reaction with dithiol in dry



Scheme 4.

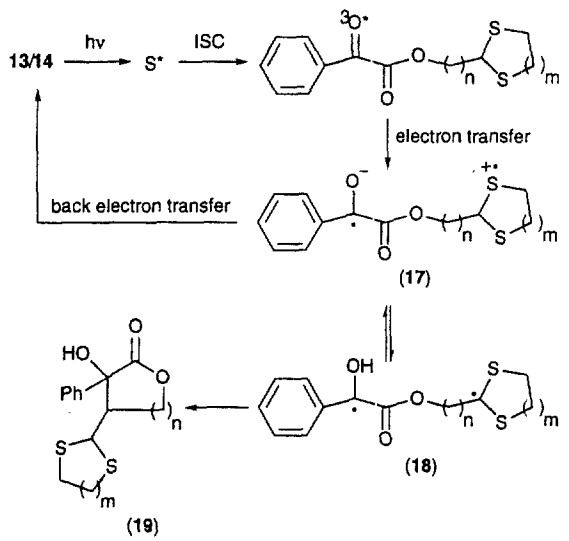


Scheme 5.

chloroform in the presence of boron trifluoride-etherate it gave alcohols **16** [16]. Esterifications using 1,3-dicyclohexylcarbodiimide (DCC) activation [7] furnished **13** and **14** in 65% and 40% overall yields (3 steps) respectively. However, GC and GC/MS analyses of the reaction mixtures resulting from irradiations of **13** or **14** in benzene evidenced none of the expected cyclols (**19**). Instead, products derived from Norrish Type II and intermolecular hydrogen abstraction induced reactions were observed, Scheme 4.[2]

The lifetimes of triplet excited states of **13** and **14** are short on the nanosecond time scale and escaped detection by the laser flash photolysis instrument employed in this study. That the lifetimes of triplets **13** and **14** are much shorter than that of the triplet of methyl phenylglyoxylate (microseconds) indicates that an intramolecular electron transfer occurs in these triplets [7,17]. As shown in Scheme 6, this produces radical anion **17** from the triplet excited state. Proton transfer in **17** is expected to be fast and regioselective due to the higher acidity of the proton on the carbon between the two S atoms [13,14]. The absence of **19** as a photoproduct suggests that cyclization of biradical **18** does not occur, due likely to the steric hindrance imposed by the dithianyl (dithiolanyl)

[2] Products were not isolated independently because the Norrish Type II and intermolecular reaction products of alkyl phenylglyoxylates have been adequately studied, see [7,17].



Scheme 6.

ring. Back electron transfer in **17** is a facile process as indicated by the short lifetime of the triplets **13/14**. Back electron transfer regenerates ground state **13/14** and consumes biradical **18**. When back electron transfer becomes the dominating deactivation pathway for radical anion **17**, the electron transfer process becomes non-productive and products from other reactions (Norrish Type II and intermolecular H abstraction) from the triplet state are derived. This phenomenon is in agreement with observations from earlier studies of other substituted alkyl phenylglyoxylates [17].

4. Conclusion

Photoreductions of ethyl phenylglyoxylate in the presence of different quenchers were studied. Diethyl 2,3-dihydroxy-2,3-diphenylsuccinate (**2**) is the only product isolated when a good hydrogen donor is available. Adducts are isolated in addition to **2** in the presence of several electron donating quenchers. Reaction mechanisms are proposed and quantum yields are measured. The quenching constant by an electron donor is significantly higher than that by a hydrogen donor. The photolyses of dithiane (dithiolane)-substituted alkyl phenylglyoxylates are also reported.

Acknowledgements

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