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ElectronTransfer Induced Ring Opening of α-Epoxyketones: Spirodioxolane Formation

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Abstract: Stereospecific formation of spirodioxolanes has been observed on electron transfer induced ring opening of α-epoxyketones by 2,4,6-triphenylpyrylium tetrafluoroborate in the presence of cyclohexanone.

**Keywords:** Dioxolanes; Electron transfer; α-Epoxyketones; Photochemistry; Spiro compounds.

Introduction

Ring opening of epoxides and α-epoxyketones induced by electron transfer to suitable electron acceptors in the presence of various nucleophiles leads to the formation of some interesting compounds such as ethers, dioxolanes and trioxolanes. Such reactions have been recognized as important processes not only in thermal but also in photochemical reactions.

Single electron transfer (SET) induced ring opening reactions of epoxides [1-4], α-epoxyketones [5-8] and α-epoxyesters [9] have demonstrated C_α-C_β, C_α-O and C_β-O bond cleavage through photosensitized electron transfer by dicyanoanthracene [1,2], tetracyanoethylene [3,4] and 2,4,6-triphenylpyrylium tetrafluoroborate [5-8] or thermally induced electron transfer to ceric ammonium nitrate [10,11], 2,3-dichloro-5,6-dicyano-*p*-benzoquinone [12] and iron (III) chloride [13].

Previously we investigated the formation of charge-transfer complexes between some α-epoxy-ketones as donor molecules and tetracyanoethylene (TCNE) as the acceptor molecule [14]. These results indicated that the amount of CT-complex formation is dependent on the extent of electron donating character of the α-epoxyketones, which is directly related to the nature and location of the additional substituent on the parent molecule. Recently we reported on the electron transfer induced ring opening of some α-epoxyketones of interest (**1a-f**) using 2,4,6-triphenylpyrylium tetrafluoroborate (**2**) as photocatalyst in methanol [8] and acetone solutions [15] (Scheme 1). The aim of this work was to explain the effects of the type of the additional substituent on the parent molecule (**1a**) and the nature of the nucleophile used as solvent on the rate of the ring opening process.

Scheme 1

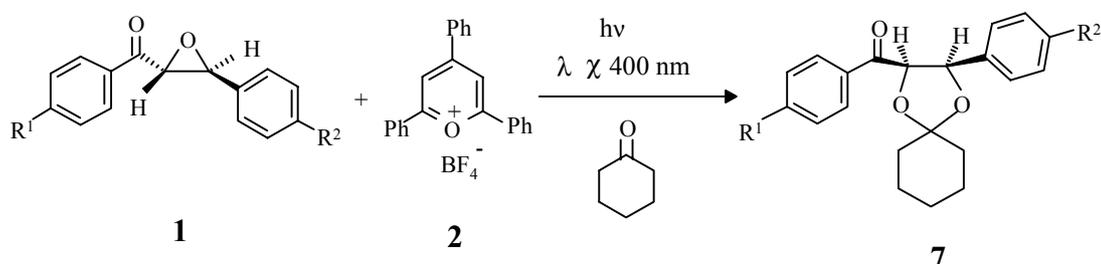| 1	R^1	R^2
a	H	H
b	H	CH_3
c	CH_3	H
d	H	OCH_3
e	OCH_3	H
f	OCH_3	OCH_3
g	Br	H
h	H	Br
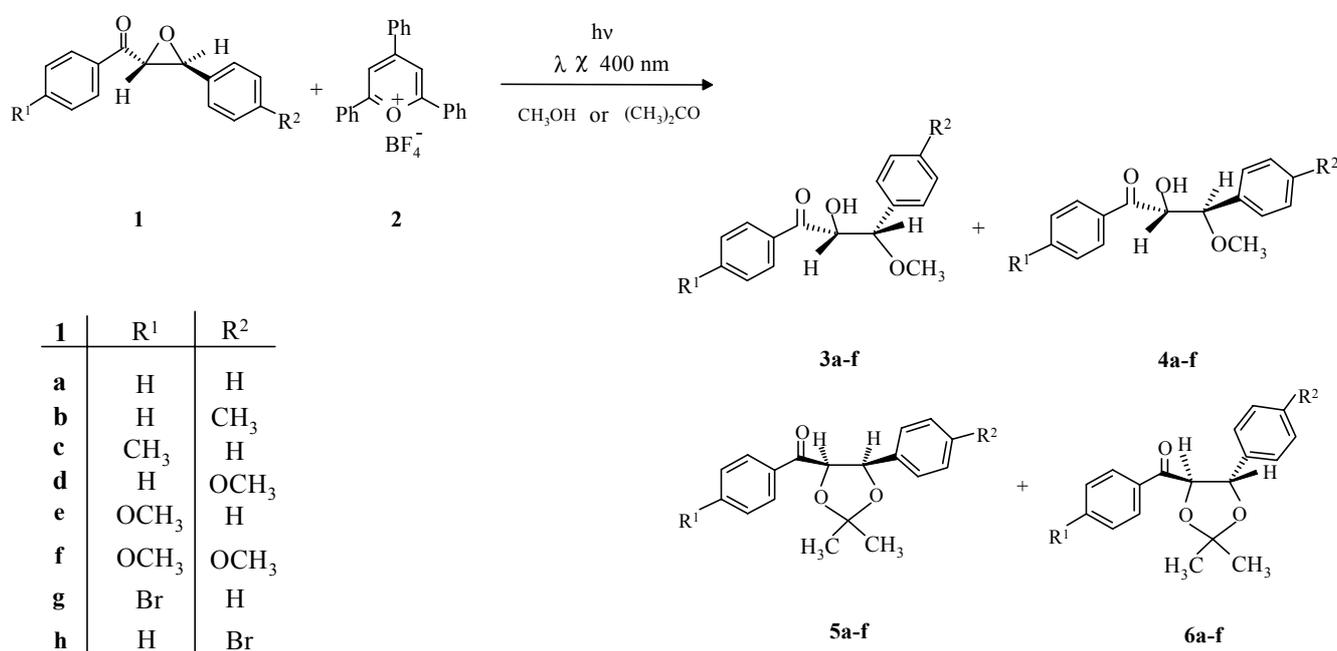
Although the rate of the ring opening in methanol was faster than in acetone, a diastereoselective reaction has been observed in the latter solutions. As a continuation of this work we have now investigated this reaction in the presence of cyclohexanone as nucleophile. By also using a cyclic ketone in place of acetone, we have attempted to elucidate the effect of the steric hindrance of the nucleophile on the diastereoselectivity of reaction and also the effect of the extent of its nucleophilicity on the rate of the ring opening.

Results and discussion

Irradiation (λ ≥ 400 nm) of each α-epoxyketones (**1a-e**, **1g**) and (**2**) in molar ratio of 10:1 in cyclo-hexanone / acetonitrile (19ml / 1ml) resulted in the opening of the epoxide ring and formation of a sole

product of type **7** in each reaction (Scheme 2). It should be mentioned that the light has been passed through a filter solution to prevent the absorption of light by compounds **1a-e** and **1g**, therefore selective excitation of the photocatalyst **2** has been achieved.

Scheme 2

1	R¹	R²
a	H	H
b	H	CH₃
c	CH₃	H
d	H	OCH₃
e	OCH₃	H
g	Br	H
h	H	Br

IR and ^{1}H-NMR data provided useful information for the structural assignment of the products (**7a-e**, **7g**). Monitoring of the photoreaction/conversion of **1a-e**, **1g** and formation of **7a-e**, **7g** was carried out using IR by following the appearance of the CH-stretching of the cyclohexane ring at ~ 2800-2980 and a shift of the CO bond to higher frequency due to opening of the epoxide ring and the resulting release of the ring strain. The results are summarized in Table 1.

Table1. Comparison of the CO-stretching in the IR spectra of
compounds **1a-e**, **1g** with those of **7a-e**, **7g**

| Compd.	CO (cm^{-1})	Prod.	CO (cm^{-1})
1a	1670	**7a**	1695
1b	1660	**7b**	1680
1c	1665	**7c**	1685
1d	1660	**7d**	1690
1e	1665	**7e**	1680
1g	1670	**7g**	1690

The ^{1}H-NMR spectra showed a multiplet at ~1.1-2.4 ppm with an integration corresponding to 10H for the cyclohexyl moiety and an AB-system for the 3-H and 4-H of the dioxolane ring. A *cis*

orientation of the hydrogens of the dioxolane ring was confirmed by comparison of their chemical shifts and coupling constants with those of known dioxolanes [16]. Semiempirical PM3 calculations on dioxolanes **7a-e**, **7g**, showed that, for example for **7a**, in the optimized structure (Figure 1) 4-H lies in the same plane as the phenyl ring. Since the anisotropic effect of the phenyl ring causes a deshielding on this proton, therefore the resonance of 4-H should appear at a lower field in comparison to the resonance for the benzylic protons [17,18]. On the other hand, the inductive effect of the benzoyl group on 3-H leads to absorption of this proton at lower field, therefore, the chemical shifts of 3-H and 4-H must be closer to each other. (Table 2)

Table 2. Structurally relevant ^{1}H-NMR chemical shifts
(δ values) and *J* ^{1}H-^{1}H coupling constants [Hz] of (**7a-e**, **7g**)

| Compd.	δ_A 4-H	δ_B 3-H	J_{AB}
7a	5.52	5.80	8.0
7b	5.46	5.78	7.6
7c	5.49	5.74	7.6
7d	5.59	5.77	7.6
7e	5.50	5.74	7.4
7g	5.60	5.70	7.4

Figure 1. Optimized structure of **7a** using PM3 calculations
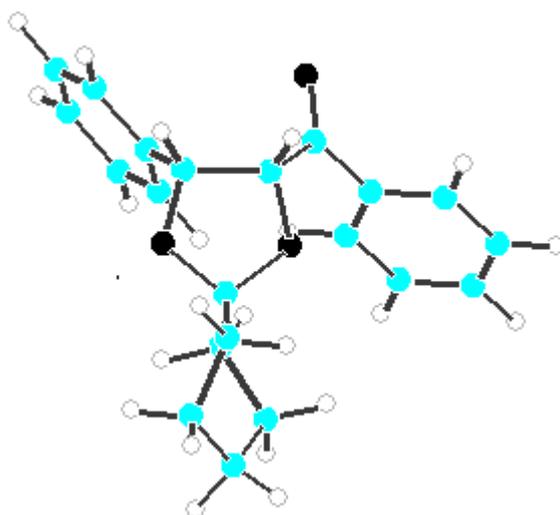
The mechanism of the ring opening can be explained as follows: due to electron transfer from **1a-e** and **1g** to photoexcited **2**, the radical cations **8** and **9** could be formed, which are then trapped by

nucleophilic attack of cyclohexanone. The observed high diastereospecificity of reaction supports the involvement of the intermediate **8** rather than **9**, which should be formed better in apolar cyclohexanone solution and would be attacked preferably by cyclohexanone acting as a nucleophilic reagent at the carbon atom at the rear of the bond comprising the leaving group oxygen due to the steric hindrance of the nucleophile. This suggestion is also supported by the formation of single products, namely the *cis*-products **7a-e, 7g**.

8   **9**
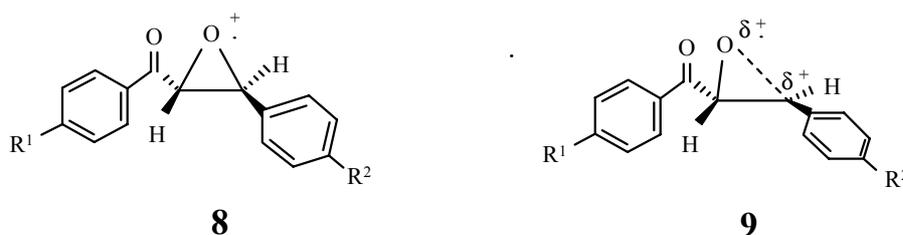
The electron transfer induced ring opening and the involvement of the intermediates such as **8** or **9** are supported by the following basis:

1. Irradiation of **1a-e** and **1g** in the absence of the photocatalyst **2** at $\lambda \geq 400$ nm (blank experiment) does not result the formation of any product.

2. Upon irradiation of **1h** and **2** in the presence of cyclohexanone unchanged starting materials were obtained even after 15 hours irradiation. This indicates that the formation of **8** or **9** will be destabilized by the electron withdrawing character of the bromine on the phenyl ring directly attached to the epoxide ring. As we show in Table 3, the ring opening will be facilitated, when the phenyl group bears the electron donor group such as methyl or methoxy group (**1b**, **1d**).

Table 3. Photochemical reactions of **1a-e**, **1g** catalyzed by
(**2**) in cyclohexanone solution[a]

Compd.	Irrad. Time[b] (hr)	Product	Yield %[c]
1a	5	**7a**	27
1b	2.5	**7b**	31
1c	5	**7c**	26
1d	2	**7d**	33
1e	5	**7e**	30
1g	15	**7g**	24

[a] Molar ratio of (**1a-e**, **1g**) : (**2**) (10:1), [**1a-e**, **1g**] = 0.04 M and [**2**] = 0.004 M. [b] Times are given after maximum progression of the reaction. [c] Isolated yields, based on consumed **1**.

It should be mentioned that some unidentified products were formed, which could not be isolated in pure form for spectroscopic analysis. The formation of the *trans* isomer was ruled out based on comparison of the TLC of the *cis* isomer with the TLC of the reaction mixture. The formation of the *cis*-products obtained in cyclohexanone solution compared to the results obtained in acetone can be attributed due to the steric hindrance of nucleophile.

Acknowledgments

Experimental

General

Melting points were determined on a Stuart Scientific SMP-2 capillary apparatus and are uncorrected. IR spectra were recorded using KBr pellets on a Shimadzu IR-435 spectrometer. ^{1}H-NMR spectra were recorded on a Bruker AW 80 spectrometer in $CDCl_3$ with TMS as internal standard. Mass spectra were measured by electron impact at 70 eV on a Micromass Platform instrument.. Elemental analyses were measured on a CHN-O-RAPID instrument. Preparative thin layer chromatography (PTLC) was carried out on 20 x 20 cm^2 plates, coated with a 1 mm layer of Merck silica gel PF_{254}, prepared by applying the silica as a slurry and drying in air. All irradiations were carried out using a 400 W high-pressure Hg-vapor lamp from NARVA in Duran glass equipment with sample cooling by cold running water. The light has been passed through a filter solution (75 gL^{-1} of $NaNO_2$ and 4.4 gL^{-1} of $CuSO_4$ in 2.7 M NH_4OH) [19] to obtain $\lambda \geq 400$ nm for the selective excitation of the photocatalyst **2** and also to prevent the absorption of the light by **1a-e**, **1g-h**.

General experimental procedure for the conversions of **1a-e***,* **1g-h** *by photoexcited* **2** *in the presence of cyclohexanone.*

To a solution of 0.08 mmol of **2** in dry acetonitrile (1 mL) and freshly distilled cyclohexanone (19 mL, c = 0.004 M) was added 0.8 mmol of each of compounds **1a-e, 1g-h** (c = 0.04 M) and the mixtures were irradiated until maximum progression of the reaction was observed. The excess of cyclohexanone was evaporated under reduced pressure and the residue was purified by preparative TLC (PTLC).

Reaction of 1,3-diphenyl-2,3-epoxy-1-propanone (**1a**). PTLC (12: 1 petroleum ether-ethyl acetate, three times): Zone 1, recovered **1a** (35 mg, 20%); Zone 2, 69 mg (27%) of *cis-3-benzoyl-4-phenyl-2,5-dioxaspiro[4,5]decane* (**7a**), recrystallized from cyclohexane, m.p. 95-98 ºC; IR cm^{-1}: 3055, 2880-

2990, 1695, 1590, 1220, 1110, 1080; ^{1}H-NMR (δ): 1.10-2.42 (m, 10H, cyclohexane H), AB-system (δ_A = 5.52, δ_B = 5.80, $^3J_{AB}$= 8.0 Hz, 2H, 4-H, 3-H), 7.03 (s, 5H, phenyl ring H), 7.15-7.44 ppm (m, 5H, benzoyl group); EI-MS *m/z* (%): 322 (M$^+$, 1), 224 (M$^+$-C$_6$H$_{10}$O, 15), 217 (M$^+$-C$_6$H$_5$CO, 9), 119 (M$^+$-C$_6$H$_{10}$O, - C$_6$H$_5$CO, 30), 105 (C$_6$H$_5$CO$^+$, 100), 77 (C$_6$H$_5$$^+$, 28); Anal. Calcd. for C$_{21}$H$_{22}$O$_3$ (322.40): C, 78.23; H, 6.88. Found: C, 77.98; H, 6.93.

Reaction of 3-p-methylphenyl-1-phenyl-2,3-epoxy-1-propanone (**1b**). PTLC (12:1 petroleum ether-ethyl acetate, four times): Zone 1, recovered **1b** (19 mg, 10%); Zone 2, 84 mg (31%) of *cis-3-benzoyl-4-(p-methylphenyl)-2,5-dioxaspiro[4,5]decane* (**7b**), recrystallized from cyclohexane, m.p. 114-116 ºC; IR cm^{-1}: 3045, 2880-2990, 1680, 1590, 1200, 1150, 1080; ^{1}H-NMR (δ): 1.15-2.20 (m, 10H, cyclohexane H), 2.14 (s, 3H, CH$_3$), AB-system (δ_A = 5.46, δ_B = 5.78, $^3J_{AB}$ = 7.6 Hz, 2H, 4-H, 3-H), 6.72-7.58 ppm (m, 9H, aromatic H); EI-MS *m/z* (%): 336 (M$^+$, 4), 238 (M$^+$-C$_6$H$_{10}$O, 75), 231 (M$^+$-C$_6$H$_5$CO, 43), 216 (M$^+$-CH$_3$C$_6$H$_4$CHO, 68), 133 (M$^+$-C$_6$H$_5$CO, -C$_6$H$_{10}$O, 84), 119 (CH$_3$C$_6$H$_4$CO$^+$, 48), 105 (C$_6$H$_5$O$^+$, 100), 91 (CH$_3$C$_6$H$_5$$^+$, 55), 77 (C$_6H_5$$^+$, 77); Anal. Calcd. for C$_{22}$H$_{24}$O$_3$ (336.43): C, 78.54; H, 7.19. Found: C, 77.94; H, 7.21.

Reaction of 1-p-methylphenyl-3-phenyl-2,3-epoxy-1-propanone (**1c**). PTLC (12:1 petroleum ether-ethyl acetate, three times): Zone 1, recovered **1c** (38 mg, 20%); Zone 2, 70 mg, (26%) of *cis-3-(p-methylbenzoyl)-4-phenyl-2,5-dioxaspiro[4,5]decane* (**7c**), recrystallized from cyclohexane, m.p. 116-118 ºC; IR cm^{-1}: 3045, 2880-2990, 1685, 1610, 1250, 1105; ^{1}H-NMR (δ): 1.15-2.18 (m, 10H, cyclohexane H), 2.22 (s, 3H, CH$_3$), AB-system (δ_A = 5.49, δ_B = 5.74, $^3J_{AB}$ = 7.6 Hz, 2H, 4-H, 3-H), AB-system (δ_A = 6.95, δ_B = 7.37, $^3J_{AB}$ = 7.6 Hz, 4H, phenyl ring H), 7.05 ppm (s, 5H, aromatic H); EI-MS: *m/z* (%): 336 (M$^+$, 5), 238 (M$^+$-C$_6$H$_{10}$, 32), 230 (M$^+$-C$_6$H$_5$CHO, 19), 217 (M$^+$-CH$_3$C$_6$H$_4$CO, 35), 119 (CH$_3$C$_6$H$_4$CO$^+$, 100), 105 (C$_6$H$_4$CO$^+$, 46), 91 (CH$_3$C$_6$H$_4$$^+$, 74), 77 (C$_6H_5$$^+$, 35); Anal. Calcd. for C$_{22}$H$_{24}$O$_3$ (336.43): C, 78.54; H, 7.19. Found: C, 78.01; H, 7.15.

Reaction of 3-p-methoxyphenyl-1-phenyl-2,3-epoxy-1-propanone (**1d**). PTLC (10:1 petroleum ether-ethyl acetate, three times): Zone 1, recovered **1d** (20 mg, 10%); Zone 2, 95 mg (33%) of *cis-3-benzoyl-4-(p-methoxyphenyl)-2,5-dioxaspiro[4,5]decane* (**7d**), recrystallized from cyclohexane, m.p. 77-78 ºC; IR cm^{-1}: 3040, 2880-2990, 1690, 1610, 1580, 1240, 1170, 1100; ^{1}H-NMR (δ): 1.10-2.15 (m, 10H, cyclohexane H), 3.64 (s, 3H, OCH$_3$), AB-system (δ_A = 5.49, δ_B = 5.77, $^3J_{AB}$ = 7.6 Hz, 2H, 4-H, 3-H), AB-system (δ_A = 6.53, δ_B = 7.02, $^3J_{AB}$ = 8.2 Hz, 4H, phenyl ring H), 7.20-7.58 ppm (m, 5H, aromatic H); EI-MS *m/z* (%): 352 (M$^+$, 2), 254 (M$^+$-C$_6$H$_{10}$O, 94), 216 (M$^+$-CH$_3$OC$_6$H$_4$CO, 100), 149 (M$^+$-C$_6$H$_5$CO, -C$_6$H$_{10}$O, 90), 135 (CH$_3$OC$_6$H$_4$CO$^+$, 52), 119 (M$^+$ - CH$_3$OC$_6$H$_4$CO, 93), 105 (C$_6$H$_5$CO$^+$, 97), 77 (C$_6$H$_5$$^+$, 88); Anal. Calcd. for C$_{22}$H$_{24}$O$_4$ (352.43): C, 74.97; H, 6.86. Found: C, 74.69; H, 6.89.

Reaction of 1-p-methoxyphenyl-3-phenyl-2,3-epoxy-1-propanone (**1e**). PTLC (10:1 petroleum ether-ethyl acetate, four times): Zone 1, recovered **1e** (20 mg, 10%); Zone 2, 86 mg (30%) of *cis-3-(p-methoxy-benzoyl)-4-phenyl-2,5-dioxaspiro[4,5]decane* (**7e**), recrystallized from cyclohexane, m.p.

129-130 ºC; IR cm^{-1}: 3045, 2800-2980, 1680, 1600, 1250, 1120, 1020; ^{1}H-NMR (δ): 1.10-2.21 (m, 10H, cyclohexane H), 3.74 (s, 3H, OCH$_3$), AB-system (δ_A = 5.50, δ_B = 5.74, ^{3}J$_{AB}$ = 7.4 Hz, 2H, 4-H, 3-H), AB-system (δ_A = 6.65, δ_B = 7.44, ^{3}J$_{AB}$ = 8.0 Hz, 4H, phenyl ring H), 7.10 ppm (s, 5H, aromatic H); EI-MS *m/z* (%): 352 (M$^+$, 3), 254 (M$^+$-C$_6$H$_{10}$O, 17), 217 (M$^+$-CH$_3$C$_6$H$_4$CO, 12), 149 (M$^+$-C$_6$H$_{10}$O, -C$_6$H$_5$CO, 4), 135 (CH$_3$OC$_6$H$_4$CO$^+$, 100), 119 (M$^+$ - C$_6$H$_{10}$O, - CH$_3$OC$_6$H$_4$CO, 36), 107 (CH$_3$OC$_6$H$_4$$^+$, 6), 105 (C$_6H_5CO^+$, 3), 77 (C$_6H_5$$^+$, 16); Anal. Calcd. for C$_{22}$H$_{24}$O$_4$ (352.43): C, 74.97; H, 6.86. Found: C, 75.07; H, 6.79.

Reaction of 1-p-bromophenyl-3-phenyl-2,3-epoxy-1-propanone (**1g**). PTLC (12:1 petroleum ether-ethyl acetate, three times): Zone 1, recovered **1g** (72 mg, 30%); Zone 2, 76 mg (24%) of *cis-3-(p-bromobenzoyl)-4-phenyl-2,5- dioxaspiro[4,5]decane* (**7g**), recrystallized from cyclohexane**,** m.p. 122-124 ºC; IR cm^{-1}: 3040, 2880-2985, 1690, 1580, 1240, 1170, 1110, 1050; ^{1}H-NMR (δ): 1.12-2.20 (m, 10H, cyclohexane H), AB-system (δ_A = 5.60, δ_B = 5.70, ^{3}J$_{AB}$ = 7.4 Hz, 2H, 4-H, 3-H), 7.05 (s, 5H, phenyl ring H), 7.30 ppm (s, 4H, aromatic H); EI-MS *m/z* (%): 402 [M$^+$ (^{81}Br), 6], 400 [M$^+$ (^{79}Br), 7], 304 [M$^+$ (^{81}Br)-C$_6$H$_{10}$O, 40], 302 [M$^+$ (^{79}Br)-C$_6$H$_{10}$O, 40], 296 [M$^+$ (^{81}Br)-C$_6$H$_5$CHO, 33], 294 [M$^+$ (^{79}Br)-C$_6$H$_5$CHO, 34], 217 (M$^+$-BrC$_6$H$_4$CO, 73), 185 (^{81}BrC$_6$H$_4$CO$^+$, 98), 183 (^{79}BrC$_6$H$_4$CO$^+$, 100), 157 (^{81}BrC$_6$H$_4$$^+$, 54), 155 (^{79}BrC$_6H_4$$^+$, 54), 119 (M$^+$ - C$_6$H$_{10}$O, - BrC$_6$H$_4$CO, 97), 77 (C$_6$H$_5$$^+$, 55); Anal. Calcd. for C$_{21}$H$_{21}$BrO$_3$ (401.29): C, 62.85; H, 5.27. Found: C, 62.91; H, 5.24.

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Sample Availability: Samples are available from MDPI