

Microwave-assisted and light-induced catalytic ring opening of α -epoxyketones using DDQ

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Abstract

Ring opening reaction of α -epoxyketones in the presence of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone in methanol solution under microwave and UV irradiations resulted in the formation of α -hydroxy- β -methoxyketones through C_{β} -O bond cleavage in excellent yields. Whereas the nature and location of the additional substituent affect the rate of ring opening, microwave irradiation remarkably raised the rate of reactions compared with UV-light. Cyclic voltammetric and conductometric studies supported these experimental results.
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Keywords: Ring opening; α -Epoxyketones; Microwave; Cyclic voltammetric; Conductometric

1. Introduction

In recent years, application of microwave irradiation to the optimization and acceleration of organic reactions has rapidly increased. A variety of reactions such as pericyclic reaction [1], cyclization [2], aromatic substitution [3], oxidation [4], alkylation [5], decarboxylation [6], radical reactions [7], condensation [8], peptide synthesis [9] and heterocycle synthesis [10] could be facilitated by using of microwave irradiation. The application of microwave techniques in organic synthesis especially leads to shorter reaction times, higher yields, easier work-up and environmentally friendliness.

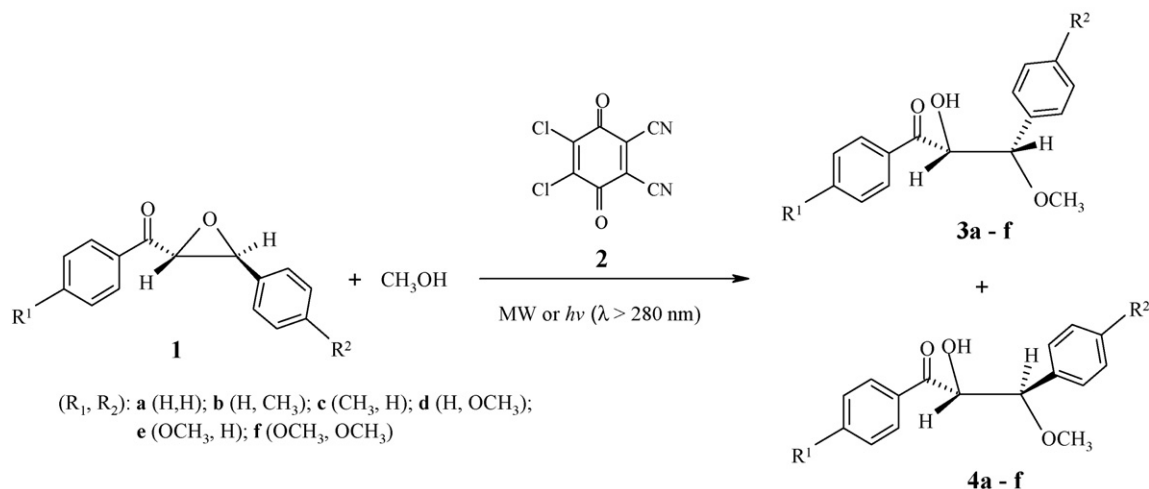
2,3-Dichloro-5,6-dicyano-1,4-benzoquinone known as DDQ is a versatile reagent for organic synthesis. Due to the ability of this reagent for oxidation reactions and also its relative stability under various reaction conditions this reagent has been used for several organic transformations such as dehydrogenation [11–15], oxidation [16–20] and deprotection [21–23]. DDQ is also a well-known electron-acceptor species and its interaction with variety of electron donors has been the subject of several investigations concerning the formation of charge-transfer (CT) complexes [24–29]. The photochemical reactions using DDQ as

photocatalyst have not been well documented. To the best of our knowledge a few works are reported for deprotection of thioacetals, ketals and benzyl ethers [30,31]. DDQ has also been used for several organic transformations under microwave irradiation [32–35].

Ring opening reactions of α -epoxyketones have also attracted considerable interest from both synthetic and mechanistic standpoints. Such reactions have been recognized as an important process not only in thermal but also photochemical transformations. Single electron transfer (SET) induced ring opening reactions of α -epoxyketones have caused C_{α} -O, and C_{β} -O bond cleavages. These reactions occur through photochemical [36–47], thermal [44,48–51] or electrochemical [52] processes.

Recently, we have investigated the photocatalytic ring opening reactions of α -epoxyketones by 2,4,6-triphenylpyrilium tetrafluoroborate (TPT) as photocatalyst in methanol [36], cyclohexanone [37], acetone [38] and acetic acid [39] solutions to clarify the effect of the substituent and also the nucleophilicity of solvent on the rate and diastereoselectivity of reaction. The use of DDQ as catalyst and photocatalyst in various electron-transfer induced reactions leads us to investigate the ring opening of α -epoxyketones in the presence of DDQ in methanol solution under microwave and UV irradiations to elucidate the effect of both irradiation sources and also the electron-accepting ability of DDQ on the rate and diastereoselectivity of reactions.

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Scheme 1.

2. Results and discussion

In order to investigate the effects of the microwave and UV-irradiations on catalytic ring opening of α -epoxyketones, in an optimized reaction conditions, a mixture of α -epoxyketones **1a–f** and DDQ in a molar ratio of 10:1 in methanol solution was irradiated with microwave or UV-light, followed by check with TLC until total disappearance of **1a–f** (Scheme 1). The results are summarized in Table 1.

The presented data showed that the rate of the ring opening of α -epoxyketones **1a–f** by DDQ is dependent on the nature and location of the additional substituent on the parent molecule **1a** and that the use of microwave irradiation greatly increases the rate of ring opening compared with UV irradiation.

According to the following observations:

1. The use of the catalytic amounts of DDQ in this reaction.

Table 1

The results of ring opening reactions of **1a–f** catalyzed by **2** in methanol solution^a under microwave and UV irradiations

Compound	Time (h)	Yield (%) (% based on consumed 1a–f)	4/3 ^b	3%	4%
1a (MW)	0.25	97	2.69:1	27.10	72.90
1a (hv)	12	95	3.03:1	24.81	75.19
1b (MW)	0.06	97	1:1.27	55.95	44.05
1b (hv)	0.75	96	1:1.29	56.33	43.67
1c (MW)	0.18	96	2.56:1	28.09	71.91
1c (hv)	10	94	3.69:1	21.32	78.68
1d (MW)	0.05	96	1:1.06	51.46	48.54
1d (hv)	0.75	96	1:1.02	50.50	49.50
1e (MW)	0.17	96	3.45:1	22.47	77.53
1e (hv)	8.5	94	5.00:1	16.67	83.33
1f (MW)	0.04	98	1.06:1	48.54	51.46
1f (hv)	0.5	97	1.05:1	48.78	51.22

MW-irradiations were carried out in 10 s time interval to prevent splashing of solvent. UV-irradiations were carried out at 25 °C by running cold water.

^a [**1a–f**] = 0.04 M, [**2**] = 0.004 M, corresponding to molar ratio of 10:1.

^b The ratios have been determined by comparison of the integral ratios of the hydrogen on C-2 in ¹H NMR.

2. The better light absorption by DDQ on comparison of the UV spectra of **1a–f** and DDQ under our UV condition ($\lambda \geq 280$ nm) (Table 2).
3. The failure of any kinds of photoreactions on irradiation ($\lambda \geq 280$ nm) of **1b** (45 min) as the test experiment in the absence of DDQ.
4. The photocatalytic ring opening (~15–20%) of **1b** as the test experiment on irradiation ($\lambda \geq 400$ nm) of **1b** in the presence of DDQ.
5. The formation of charge transfer (CT) complex between **1b** and DDQ as the test experiment (Fig. 1).
6. The effect of the additional substituent on the rate and on the observed distereoselectivity of reaction.

We propose the following mechanism:

(i) Single electron transfer (SET) from **1a–f** to DDQ (electron affinity of DDQ ~ 3 eV) [53], or to its excited species DDQ* and/or SET in the charge transfer (CT) complex in the ground state or by excitation of CT-complex leads to the formation of **1a–f**^{•+} and DDQ^{•-} (path 1); (ii) nucleophilic attack of methanol to **1a–f**^{•+} forms **1a–f**-CH₃OH^{•+} adduct (path 2); (iii) back electron-transfer (BET) from DDQ^{•-} to **1a–f**-CH₃OH^{•+} adduct followed by a proton transfer accomplishes the reaction (path 3, Scheme 2).

Regarding the proposed mechanism, due to electron transfer from **1a–f** as electron-donor molecules to DDQ as electron-acceptor molecule, **1a–f**^{•+} could produce three different

Table 2

UV absorption spectra of **1a–f** and DDQ in CH₃OH

Compound	λ_{\max} , nm (ϵ_{\max} , l mol ⁻¹ cm ⁻¹)
1a	238 (17867), 320 (sh, 558)
1b	240 (17046), 320 (sh, 1215)
1c	241 (15067), 320 (sh, 1360)
1d	240 (17908), 326 (8754)
1e	243 (16857), 330 (sh, 4807)
1f	225 (13192), 258 (17138), 340 (sh, 3446)
DDQ	240 (14588), 350 (1713), 460 (4538)

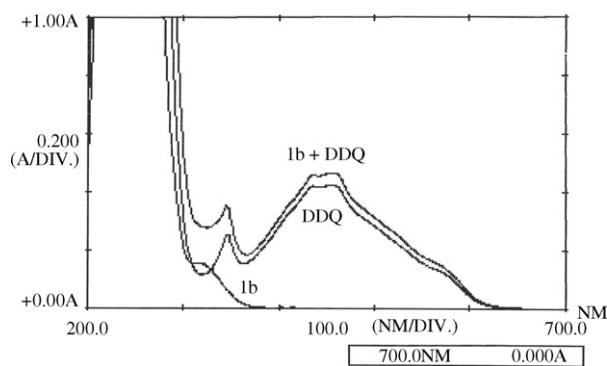
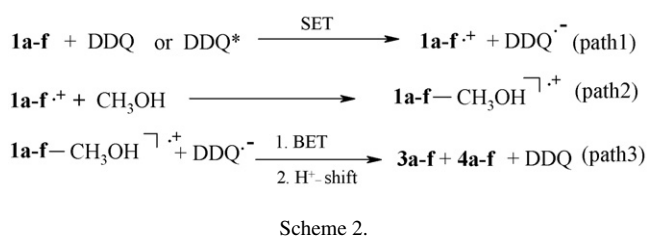
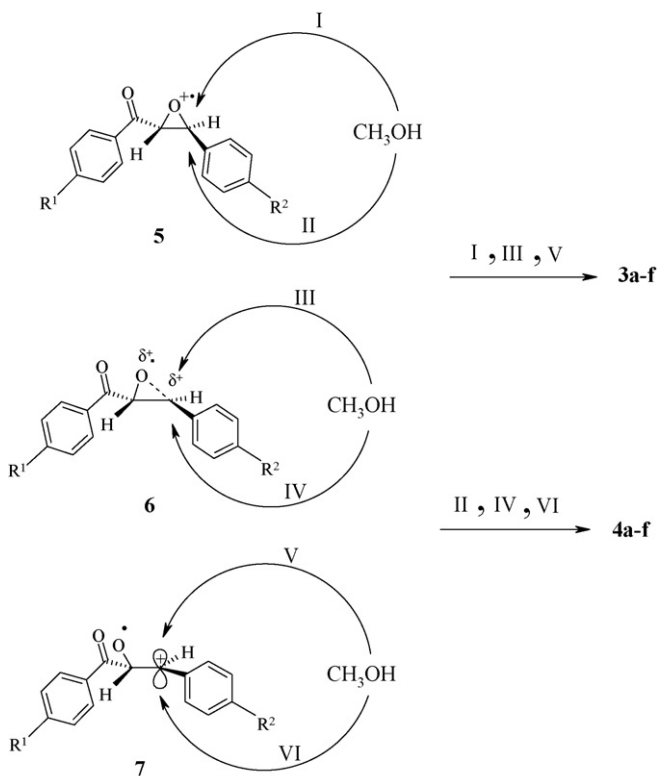


Fig. 1. Electronic absorption spectra of 1b-DDQ reaction in CH_3OH . $[\mathbf{1b}] = 2.5 \times 10^{-4} \text{ M}$, $[\text{DDQ}] = 2.5 \times 10^{-4} \text{ M}$, $\mathbf{1b} + \text{DDQ}$ mixture $[\mathbf{1b}] = [\text{DDQ}] = 2.5 \times 10^{-4} \text{ M}$.



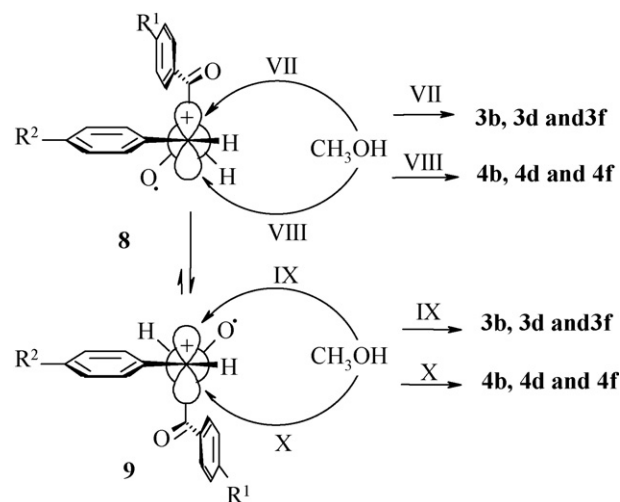
intermediates **5–7** which are trapped by nucleophilic attack of methanol (Scheme 3). Whereas the electron-donor groups such as *p*-methyl or *p*-methoxy on the phenyl ring directly attached to the epoxide ring (**1b**, **1d** and **1f**) facilitate the ring opening, the same substituents on the phenyl ring of the benzoyl group



(**1c** and **1e**) have a less remarkable effect. This observation indicates that the epoxide ring is the active site of electron-donating species and would support our suggestion that the rate determining step (rds) is path 1. The preferred participation of one of these intermediates in the reaction should be dependent on the extent of the electron-donating ability of the additional substituent on the stabilization of the involved intermediate. This suggestion is supported by the formation of different diastereomeric ratios of **4:3** when these groups change their positions. In this case it is better to compare these ratios obtained by reactions of **1b** with **1c** or **1d** with **1e**.

The interesting point is that in the cases of **1b** and **1d**, the ratios of the diastereomeric products are reversed when we compared these ratios with those obtained by reaction of **1a**, **1c**, **1e** and **1f**. This leads us to assume that the inductive effect of the *p*-methyl group (**1b**) and also the resonance effect of the *p*-methoxy group (**1d** and **1f**) on the phenyl ring directly attached to the epoxide ring would increase the contribution of the intermediates **6** or **7** and especially **7** because the more stable conformer of the intermediate **7** can also be formed through interaction of oxygen lone pair of the carbonyl group with carbocation center (the intermediates **8** and **9** in Scheme 4). Considering the steric hindrance of the substituents in both conformations **8** and **9**, participation of the conformation **9** in the reaction is more possible than that of the conformation **8**. On the basis of Cram's rule [54], the involvement of these intermediates leads to the preferred nucleophilic attack of methanol to the carbon atom at the less hindered site (VIII and IX) to form the diastereomeric products (Scheme 4). On the other hand, the intermediates, e.g. **5** and **6** are proposed for **1a**, **1c** and **1e**, in which the phenyl ring attached to the epoxide ring is not bearing such electron donating groups on the phenyl ring directly attached to the epoxide ring.

Cyclic voltammertic studies of DDQ at 15°C , 33°C and under UV-irradiation support our experimental results obtained by the ring opening with DDQ under microwave and UV irradiations. These studies showed that DDQ at 33°C is a better electron acceptor than under UV-light and at 15°C , because the



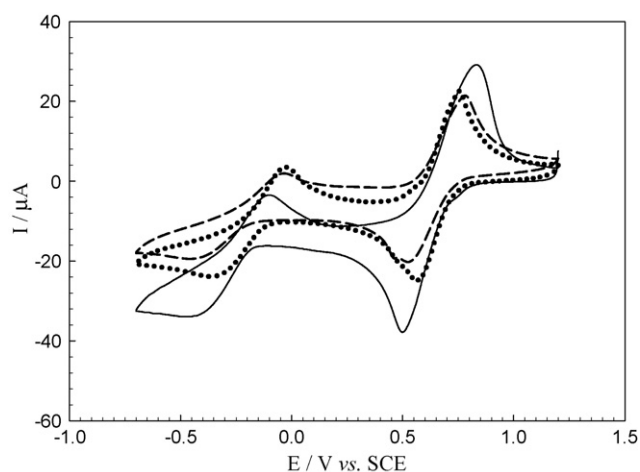


Fig. 2. Cyclic voltammograms of DDQ (1×10^{-3} M) under UV-light irradiation (---), at 33 °C (···) and at 15 °C (—) in argon saturated dichloromethane solutions containing tetrabutylammonium perchlorate (0.05 M) as the supporting electrolyte at a scan rate of 200 mV s^{-1} . (SCE = standard calomel electrode).

reduction potential of DDQ at 33 °C (0.57 V) is more positive than the reduction potential of DDQ under UV-light (0.52 V) and at 15 °C (0.50 V) (Fig. 2). This clearly indicates that with increasing temperature, the rate of reduction of DDQ increases. It should be noted that the obtained value of 0.52 V is not the real value for the reduction potential of DDQ in the excited state, but this refers to the obtained reduction potential of DDQ by UV irradiation of its solution, which supports the increased electron-accepting ability of DDQ by UV irradiation. The value of 3.16 V is found by calculation ($E^{\text{red}} + E_{0,0}$) for the reduction potential, E^{red} , of the excited DDQ. $E_{0,0}$ indicates the singlet excitation energy obtained from the fluorescence 0,0 band (470 nm) and is about $254.5 \text{ kJ mol}^{-1}$ [55].

The oxidation potentials of **1a–f** are between 1.10 and 1.32 V [40]. By using the simplified Rehm–Weller Equation ($\Delta G = 96.5 (E_{\text{ox}} - E_{\text{red}}) - E_s$) [56], ΔG values in the same range are obtained. This means that the rate of photocatalytic ring opening of **1a–f** by DDQ should be also in the same range. Actually the presented times of the UV irradiation (Table 1) do not support this suggestion, but this clearly supports the proposed

mechanism (Scheme 2) according to our observation, namely the occurrence of dark reaction (room temperature reaction) during our UV-irradiation, SET in the CT-complex in the ground state or on excitation of the CT-complex.

In order to get more information about the formation of charge-transfer complexes between DDQ and α -epoxyketones **1a–f**, conductance measurement, as a very sensitive method, was performed. Conductance measurements were carried out for solutions of α -epoxyketones **1a–f**, DDQ, and their mixtures at three mole ratios (1:1, 1:3 and 1:5) at 23 °C, 55 °C and under UV irradiation in methanol solution. The results are summarized in Table 3.

In all cases, the addition of DDQ to α -epoxyketones **1a–f** causes a sharp increase in the conductances. The conductances of the mixtures of **1a–f** with DDQ are more than the sum of conductivities of the separate compounds. The results clearly indicate that the charge-transfer complexes are more mobile than the individual compounds [57]. The difference in conductances between the mixture of **1a–f** with DDQ and the sum of conductances of the individual compounds at the same concentration could be related to the extent of CT-formation which is directly related to the extent of the electron-donating abil-

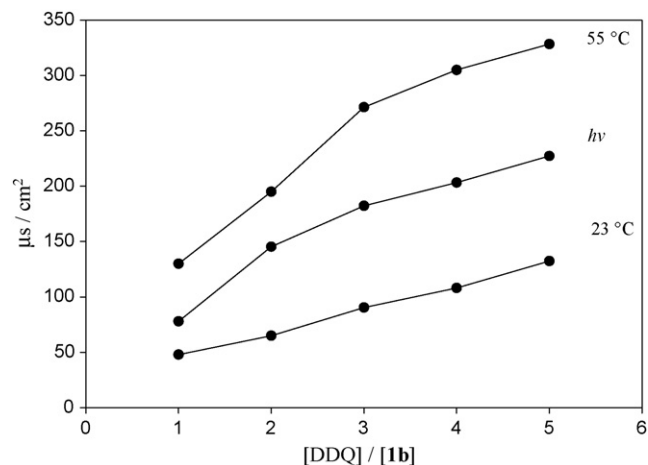


Fig. 3. A plot of conductances vs. DDQ/**1b** at 23 °C, 55 °C and under UV irradiation.

Table 3

Observed conductances ($\mu\text{s}/\text{cm}^2$) of charge-transfer complexes **1a–f** with DDQ at different mole ratios and at 23 °C, 55 °C and under UV irradiation in methanol solution^a

	[α -EP]:[DDQ] = 0.01 M:0 M			[α -EP]:[DDQ] = 0.01 M:0.01 M			[α -EP]:[DDQ] = 0.01 M:0.03 M			[α -EP]:[DDQ] = 0.01 M:0.05 M					
	23 °C	55 °C	$h\nu$	23 °C	55 °C	$h\nu$	23 °C	55 °C	$h\nu$	23 °C	55 °C	$h\nu$			
1a	3.311	4.021	3.520	41.300	109.900	71.800	82.100	220.400	162.800	121.900	301.300	202.900			
1b	5.040	5.987	5.160	47.900	130.000	77.900	90.400	271.300	182.200	132.400	328.200	227.200			
1c	4.182	4.931	4.291	43.200	112.800	73.400	85.900	268.200	172.900	127.400	309.400	212.800			
1d	5.158	6.138	5.273	49.700	132.900	77.700	91.300	275.300	195.300	138.200	339.900	230.300			
1e	5.345	6.453	5.532	48.700	127.700	76.900	89.400	281.400	192.700	139.400	327.300	219.800			
1f	7.001	8.209	7.189	51.800	141.500	80.900	95.900	310.200	201.300	151.900	359.800	245.500			
[α -EP]:[DDQ]							0:0.01 M			0:0.03 M			0:0.05 M		
DDQ				34.000	100.000	58.200	67.100	166.000	83.600	98.700	230.000	115.500			

^a Conductances of methanol at 23 °C and 55 °C are 2.227 and 2.400 $\mu\text{s}/\text{cm}^2$, respectively.

Table 4
Catalytic ring opening of **1b** in methanol solution at different temperature^a

Reaction temperature (K)	Reaction time (s)	$\ln(1/t_{\infty})$	$1/T$ (10^{-3})
299	13,560	-9.515	3.34
306	8,100	-9.000	3.27
316	3,000	-8.006	3.16
326	2,100	-7.650	3.07
333	1,800	-7.495	3.00

^a The reaction is carried out until total disappearance of **1b**.

ity of the additional substituent on the right position of the molecule, since the epoxide ring is the active site. A plot of conductance versus DDQ/**1b** ratios of 1:1, 2:1, 3:1, 4:1, and 5:1 at 23 °C, 55 °C and under UV irradiation in methanol solution clearly indicates that the electron transfer is facilitated with increasing temperature compared with under UV-light (Fig. 3).

The electronic absorption spectra of 2.5×10^{-4} M solutions of **1b**, DDQ and **1b**+DDQ shown in Fig. 1 clearly indicate the formation of charge transfer complex between the two compounds in the ground state, supporting the results obtained by the conductivity measurements.

The acceleration of ring opening under microwave irradiation is not only due to increased temperature of the reaction medium as expected, although the temperature of methanol as solvent can be raised up to 84 °C under microwave exposure [58], above its normal boiling point 65 °C. As a control experiment, the ring opening reaction of **1b** with DDQ in methanol is carried out at

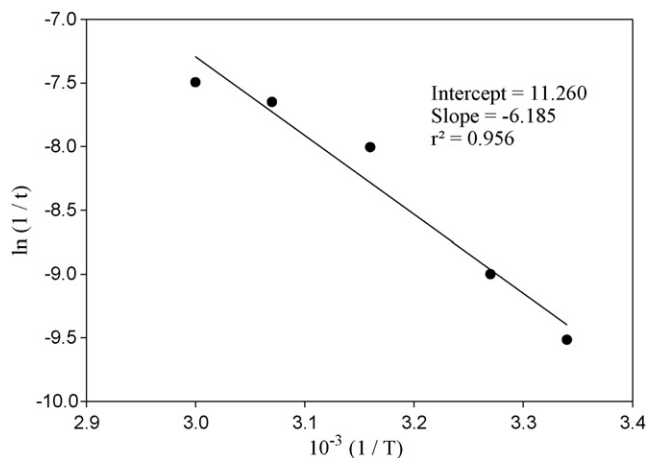


Fig. 4. A plot of $\ln(1/t_{\infty})$ vs. $1/T$ for the ring opening of **1b** by DDQ.

different temperatures until completion of the reaction. The data are presented in Table 4.

The value of 51.4 kJ mol^{-1} was obtained for activation energy of the catalytic ring opening of **1b** by DDQ (Fig. 4).

As the temperature of methanol as solvent is raised up to 84 °C under microwave exposure, the expected time for completion of reaction under microwave irradiation can be estimated from the following equation:

$$\ln\left(\frac{1/t_{\infty 2}}{1/t_{\infty 1}}\right) = \frac{E_a}{R} \left(\frac{1}{T_1} - \frac{1}{T_2}\right)$$

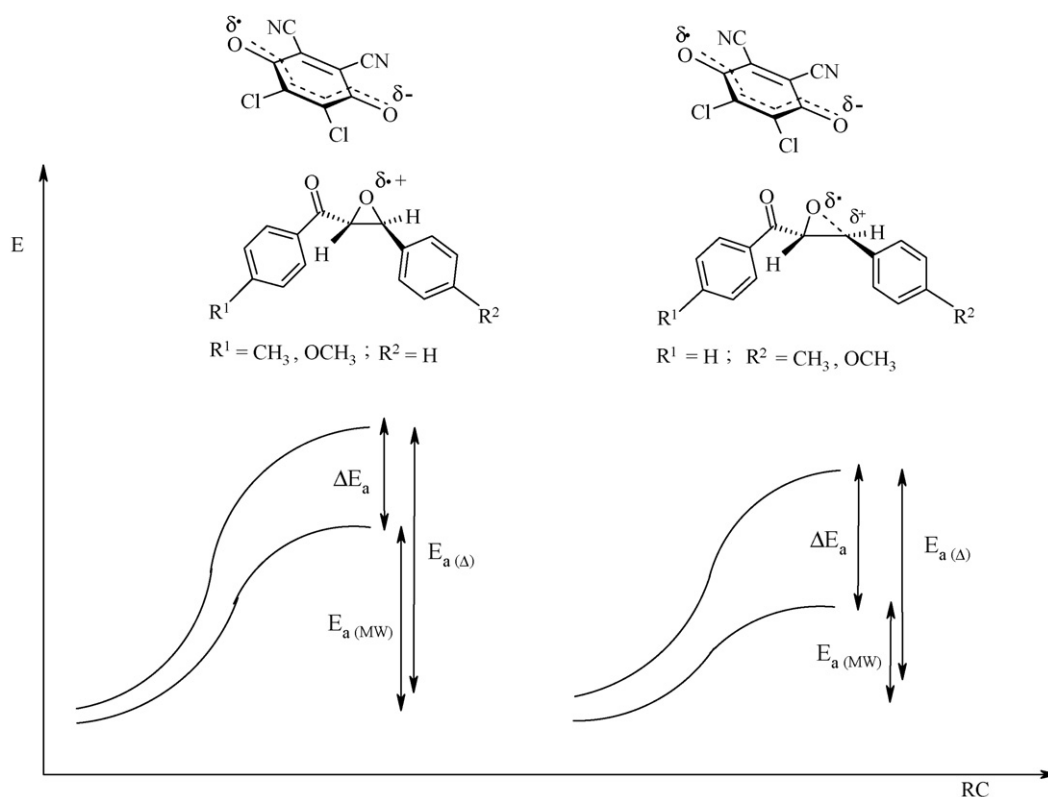


Fig. 5. Relative stabilization of the more polar transition state (TS) compared to the less polar ground state (GS) depending on the location of the additional substituent (polar mechanism).

where $t_{\infty 1}$ and $t_{\infty 2}$ are the time of reaction at 60 and 84 °C, which corresponds to microwave irradiation temperature, respectively. The value of 101.7 s is obtained for $t_{\infty 2}$. This means that if the reaction of **1b** is carried out at constant temperature of 84 °C, the reaction should be completed after 101.7 s, but as shown in Table 1, this reaction is finished after 0.06 h, namely 216 s. The reason for this difference may be due to experimental restriction of continuously applying microwave irradiation (the continuous microwave irradiation causes the splashing of solvent and we have to stop the microwave irradiation in 10 s time interval). This leads to a decrease in the reaction temperature after every 10 s, consequently increasing the reaction time.

It is true that the rapid heating by microwave exposure could be the reason of the increased rate of reaction under microwave conditions, but the specific effect derived from the microwave field, recognized as “microwave effect” should also be considered for our polar mechanism, especially when the polarity is increased during the reaction from the ground state (GS) towards the transition state (TS) [59]. Since the stabilization by dipole–dipole electrostatic interactions of the transition state is more effective than that of the ground state, an enhancement of the reactivity by a decrease in the activation energy should be expected for microwave irradiation. This may also be an explanation of the increased rate of ring opening under microwave irradiation compared with under UV irradiation and also contribution of the effect of the location of the additional substituent on the activation energy. This phenomenon is clearly shown in Fig. 5.

Another interesting point is that the diastereomeric ratios in some cases under microwave irradiation are closer to each other in comparison to the reaction under UV irradiation. We assume that the interaction of the intermediates with counter ion is decreased with increasing reaction temperature; therefore the possibility of the nucleophilic attack of methanol from both sides comes closer to each other.

3. Conclusion

In conclusions, the rate of ring opening of α -epoxyketones **1a–f** in the presence of DDQ as catalyst under microwave irradiation is faster than under UV-light irradiation. The rate of the disappearance of the α -epoxyketones **1a–f** was also accelerated depending on their nature of the additional substituents on the phenyl rings. The diastereomeric ratios of products in some cases obtained under microwave irradiation are closer to each other than under UV irradiation.

4. Experimental

α -Epoxyketones **1a–f** were prepared by epoxidation of the corresponding chalcones using alkaline hydrogen peroxide [60,61]. ^1H NMR spectra of the isolated mixture of products were measured in CDCl_3 solutions containing tetramethylsilane (TMS) as internal standard on a Bruker drx-500 (500 MHz). The cyclic voltammetric experiments were performed on an Autolab 30 potentiostat galvanostat. The electrochemical studies were

conducted by using dichloromethane solution containing tetrabutylammonium perchlorate under argon. A three electrode with a saturated calomel electrode (SCE) as the reference, a platinum foil as the counter electrode and a platinum disk as the working electrode was used. The conductance measurements were carried out with an Orion Model 180 conductivity meter. A dip-type cell with a cell constant of 0.723 cm^{-1} of platinum blank was used. Stock solutions of the α -epoxyketones **1a–f** and DDQ were prepared in methanol. Varying concentrations of DDQ were added to a fixed concentration of the related α -epoxyketones to prepare the desired DDQ to **1a–f** mole ratios of 1:1, 3:1, and 5:1 for conductance measurements at 23 °C, 55 °C and under UV irradiation. All irradiations were carried out using a 400 W high-pressure Hg vapor lamp from Narva. The samples were placed in Duran glass, which were cooled by running cold water. Preparative layer chromatography (PLC) was carried out on $20\text{ cm} \times 20\text{ cm}$ plates coated with 1 mm layer of Merck silica gel PF₂₅₄ prepared by applying the silica as a slurry and drying in air. A mixture of petroleum ether:ethyl acetate = 10:1 was used as eluent for PLC.

4.1. General procedure for ring opening of α -epoxyketones **1a–f** under microwave irradiation

A solution of 0.8 mmol of **1a–f** in 20 cm^3 methanol ($c = 0.04\text{ M}$) and 0.08 mmol of **2** ($c = 0.004\text{ M}$) in a beaker was placed in a microwave oven (National, 900 W, 2450 MHz) and irradiated for the time given in Table 1. The solvent was evaporated and the products were isolated by PLC.

4.2. General procedure for ring opening of α -epoxyketones **1a–f** under UV-light irradiation

A solution of 0.8 mmol of **1a–f** in 20 cm^3 methanol ($c = 0.04\text{ M}$) and 0.08 mmol of **2** ($c = 0.004\text{ M}$) was irradiated ($\lambda \geq 280\text{ nm}$) at 25 °C by running cold water for the time given in Table 1. The solvent was evaporated and the products were isolated by PLC.

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