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Laser flash photolysis study of the hydrogen abstraction properties of acenaphthenequinone and 1-acenaphthenone

N.C. de Lucas, J.C. Netto-Ferreira *

Departamento de Química, Universidade Federal Rural do Rio de Janeiro, Rio de Janeiro, 23851-970, Brazil

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Abstract

Nanosecond laser flash photolysis has been used to study the kinetics of hydrogen abstraction by the triplets of acenaphthenequinone (**3**) and 1-acenaphthenone (**4**) from 1,4-cyclohexadiene, 2-propanol and phenols. Diketone **3** was found to be more reactive than monoketone **4**. Hammett plots for the reaction of these triplets with phenols gave similar reaction constants ($\rho = -1.5$) in acetonitrile solution. An Arrhenius plot for the reaction of diketone **3** with 4-methoxyphenol gave $E_a = 1.2$ kcal mol^{-1} and log $A = 10.2$.

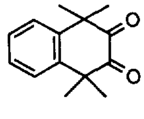
Keywords: Laser photolysis; Carbonyl compounds; Hydrogen abstraction; Triplet

1. Introduction

Hydrogen abstraction by the triplet state of aromatic ketones is the most thoroughly studied photochemical reaction [1]. It is well known that ketones having the lowest triplet state as a $\pi\pi^*$ configuration show little reactivity towards aliphatic or benzylic hydrogen abstraction [1]. Phenolic hydrogen abstraction is known to be faster than benzylic hydrogen abstraction. Surprisingly, rates for *para*-methoxypropiophenone (a ketone having lower triplet energy with $\pi\pi^*$ configuration) were found to be faster than from benzophenone [2].

Unlike these carbonyl compounds, α-diketones such as biacetyl have a low lying $n\pi^*$ triplet state and show slow hydrogen abstraction rate constants when in the presence of suitable hydrogen donors. Thus, hydrogen abstraction from 2-propanol occurs with a rate constant of 3.0×10^3 M^{-1} s^{-1} [3], three orders of magnitude slower than the value obtained for benzophenone, i.e., $k_r = 3.2 \times 10^6$ M^{-1} s^{-1} [4]. This difference in reactivity probably reflects differences in the ΔH of the reaction and is a consequence of the lower triplet energy of biacetyl ($E_T = 56$ kcal mol^{-1}) when compared with that of benzophenone ($E_T = 69$ kcal mol^{-1}).

Despite its low triplet energy, 1,1,4,4-tetramethyl-1,4-dihydro-2,3-naphthalendione (**1**), $E_T = 55$ kcal mol^{-1}, shows remarkably fast hydrogen abstraction rate constants for 2-propanol (2.6×10^7 M^{-1} s^{-1}) [5], and other hydrogen donors, which exceed that of triplet benzophenone [4]. This was attributed to a considerable degree of stabilization of the resulting ketyl radical by intramolecular hydrogen bonding, with such stability already being present in the transition state [5]. A similar explanation can be used to justify the considerably faster hydrogen abstraction observed for cyclic vicinal tricarbonyl compounds, such as 1,2,3-indanetrione (**2**) in the presence of hydrogen donors. Thus, **2** ($E_T = 42$ kcal mol^{-1}) shows hydrogen abstraction rate constant from 1,4-cyclohexadiene of 2.6×10^7 M^{-1} s^{-1} [6]. However, it has been questioned if a partial hydrogen bond would be sufficient to explain the increased reactivity given that the triplet energy of benzophenone is only 14 kcal mol^{-1} greater than that observed for **1** (or 25 kcal mol^{-1}, for **2**) [1].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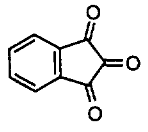
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In this paper we report the results of a laser flash photolysis study of the reaction of acenaphthenequinone (**3**) and 1-acenaphthenone (**4**) in the presence of 2-propanol, 1,4-cyclohexadiene and phenols as hydrogen donors. The very similar characteristics of these two carbonyl compounds

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* Corresponding author.

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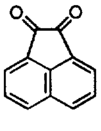
makes them an ideal pair for the comparison of the reactivity between mono and diketones.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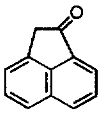
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2. Materials and methods

2.1. Material

The solvents, acetonitrile, *iso*-propanol, deuterium oxide and benzene (Aldrich spectrograde), were used as received. The acetonitrile used for the isotope effect study was refluxed with CaH_2 and distilled. Phenol, 3-methoxyphenol, 4-methoxyphenol, 4-cyanophenol, 4-chlorophenol, 4-bromophenol, 3-methylphenol, 4-methylphenol, 4-*tert*-butylphenol, 4-hydroxyphenol, 4-phenylphenol and methyl 4-hydroxybenzoate (all phenols were purchased from Aldrich) were purified when necessary (purity $<99\%$). Acenaphthenequinone (**3**) (Aldrich) was recrystallized from acetic acid. 1-Acenaphthenol and methyl viologen (Aldrich) were used as received.

1-Acenaphthenone (**4**) was synthesized by oxidation of 1-acenaphthenol [7] and recrystallized from ethanol, m.p. = 121–122°C (lit. [8] 121–121.5°C). Physical and spectral data for this compound were found to be in agreement with the proposed structure.

2.2. General techniques

UV–visible spectra were recorded with a Varian Cary 3E. GC analysis were performed on a HP 5890 with a RTX-5 capillary column.

2.3. Laser flash photolysis

The laser flash photolysis (LFP) experiments were carried out on an Edinburgh Analytical Instruments LP900. Samples were contained in a 10×10 mm^2 cell made from Suprasil quartz tubing and were deaerated by bubbling with oxygen-free nitrogen for 20 min. The samples were irradiated with a Nd/YAG Surelite laser, using its third harmonic ($\lambda = 355$ nm, ~ 5 ns, ≥ 40 mJ $pulse^{-1}$). The concentrations of the ketones were chosen in order to give an absorption in the wavelength of excitation (355 nm) of ~ 0.3 (concentration ~ 0.5–1 mM). Stock solutions of quenchers were prepared so that it was only necessary to add microliter volumes to the sample cell in order to obtain appropriate concentrations of the quencher. The temperature control experiments were performing using a cryostat from Oxford Instruments model DN-1704.

3. Results

3.1. Laser flash photolysis studies

3.1.1. Triplet characterization

Irradiation of a degassed acetonitrile solution of **3** led to the formation of a readily detectable transient with absorptions at 570 and 610 nm (Fig. 1). This transient decays by mixed first and second order kinetics with a lifetime around 1.5 μs (see insert Fig. 1) and is quenched by 1,3-cyclohexadiene with a rate constant of $(1.6 \pm 0.05) \times 10^9$ M^{-1} s^{-1} in this solvent. With β-carotene, this transient was quenched at a rate controlled by diffusion and the formation of triplet β-carotene observed at 535 nm. To the former transient we assign the triplet **3**.

Laser irradiation of a degassed solution of 1-acenaphthenone (**4**) in ACN resulted in the formation of an intense signal. The transient absorption spectrum of **4** is peculiar due to the fact that it shows some vibrational structure with maxima at 370, 410, 430, 460 and 530 nm (Fig. 2). That these absorptions belong to the same species is clearly demonstrated by following the decay at each of these maxima. In all cases, mixed first and second order kinetics with a lifetime around 2.2 μs were observed (see insert Fig. 2). This species was assigned to the triplet state of **4** due to the fact that it is quenched by 1,3-cyclohexadiene with a rate constant of $(2.3 \pm 0.08) \times 10^9$ M^{-1} s^{-1}, and at a diffusion controlled rate by β-carotene with concomitant formation of its triplet. Second order contributions are not unusual in this type of experiment and are normally due to triplet–triplet annihilation under conditions of laser excitation [5]. This was easily confirmed by reducing laser power or using a lower concentration of the ketone.

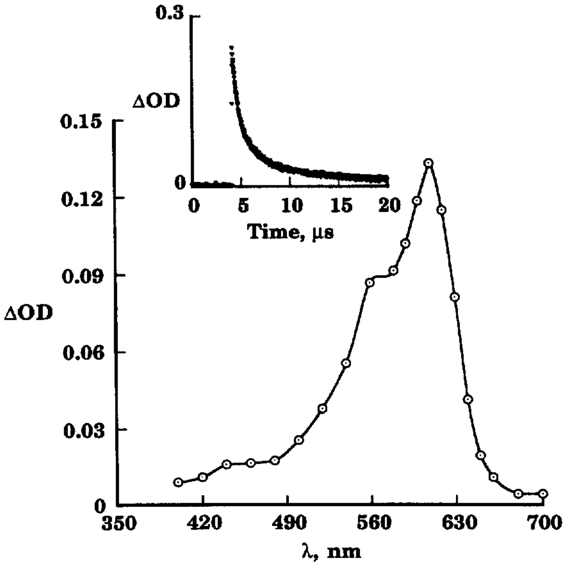

Fig. 1. Transient absorption spectrum recorded after 355 nm laser excitation of **3** (1.5 μs after laser pulse) in ACN solution. Insert: decay of the transient at 610 nm.

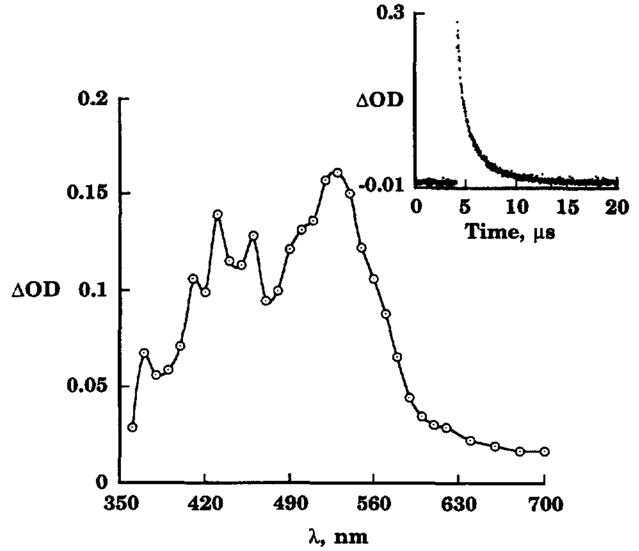

Fig. 2. Transient absorption spectrum recorded after 355 nm laser excitation of **4** (0.75 μs after laser pulse) in ACN solution. Insert: decay of the transient at 520 nm.

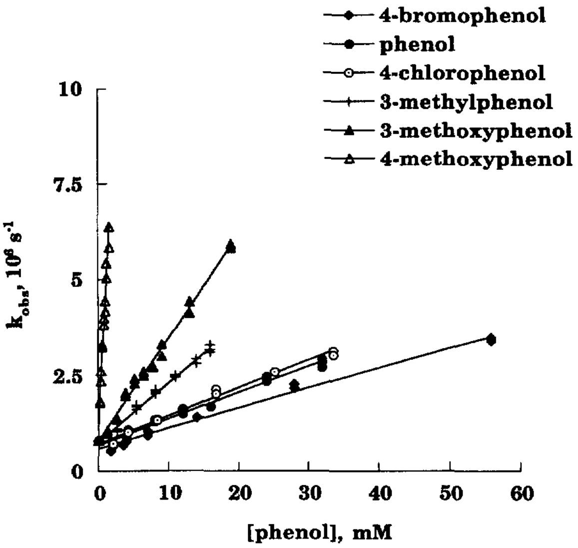

Fig. 3. Quenching plot of the triplet of **3** by Phenol, 4-chlorophenol, 4-bromophenol, 3-methylphenol, 3-methoxyphenol and 4-methoxyphenol, $\lambda_{exc} = 355$ nm; $\lambda_{mon} = 610$ nm; solv = ACN.

The energy transfer process for both of the triplets, **3** and **4**, with 1,3-cyclohexadiene is slower than diffusion control. This can be attributed to the low triplet energy (~50 kcal mol^{-1}) of both, **3** [9,10] and **4** [11].

3.1.2. Triplet reaction with hydrogen donors

Addition of typical hydrogen donors, such as 2-propanol and 1,4-cyclohexadiene, or of quenchers that could act as an electron or hydrogen donors, such as phenols, led to a shortening of the triplet lifetime of the ketones **3** and **4**. The triplet decay of these ketones followed pseudo-first-order kinetics in the presence of a hydrogen donor. The experimentally observed pseudo-first-order kinetic rate constant, k_{obs} is related to the quenching rate constant, k_q, according to Eq. (1),

$$k_{obs} = k_0 + k_q[Q] \tag{1}$$

where k_0 is the decay rate constant of the triplet in the absence of quencher, and $[Q]$ the quencher concentration.

Plots based on this equation for the triplets of **3** and **4** being quenched by various hydrogen donors were found to be linear, from which one can determine the value of k_q. Fig. 3 shows some quenching plots for **3** by various phenols. Rate constants obtained for the quenching of triplets **3** and **4** by various hydrogen donors, in acetonitrile, are shown in Table 1. This table also contains some rate constants obtained in benzene solution.

The reaction of ketone triplets with 1,4-cyclohexadiene, a well-known hydrogen atom donor [12], led to the formation of the cyclohexadienyl radical, that does not absorb strongly above 350 nm, and the ketone-derived ketyl radical. The transient absorption spectrum registered in 0.053 M of 1,4-cyclohexadiene obtained after laser excitation of **3** showed a very weak signal with a maximum around 450 nm. The same experiment was done with monoketone **4**. In this case, a transient with a maximum at 400 nm was observed. These transients were assigned to the ketyl radical of the ketones **3** and **4** respectively, based on experiments with methyl viologen. Ketyl radicals are known to be excellent electron donors [13]. Their characterization has been done using methyl viologen, since its radical cation has a strong absorption band at 603 nm and is easily detectable [13]. Laser flash photolysis experiments performed with **3** or **4** in 1,4-cyclohexadiene, in the presence of methyl viologen, lead to the formation of an intense absorption at around 600 nm, which was assigned to the methyl viologen cation radical.

Table 1
Second-order rate constants for the quenching of the triplets of **3** and **4** by various hydrogen donors in acetonitrile

Quencher	k_q[a], M^{-1} s^{-1}	
	3	**4**
2-propanol	6.2×10^5	1.3×10^5
1,4-cyclohexadiene	7.7×10^7	3.2×10^6
4-methoxyphenol	3.1×10^9	3.6×10^7
	5.7×10^{9}[b]	9.9×10^{8}[b]
4-hydroxyphenol	2.3×10^9	8.3×10^7
4-*tert*-butylphenol	2.9×10^8	4.4×10^6
3-methoxyphenol	2.8×10^8	1.7×10^6
	3.3×10^{9}[b]	1.8×10^{8}[b]
4-phenylphenol	2.4×10^8	2.9×10^6
3-methylphenol	1.5×10^8	1.0×10^6
	3.1×10^{9}[b]	1.1×10^{8}[b]
4-methylphenol	9.3×10^7	3.3×10^6
4-chlorophenol	7.4×10^7	9.7×10^5
phenol	6.9×10^7	1.0×10^6
4-bromophenol	5.3×10^7	1.2×10^6
methyl 4-hydroxybenzoate	1.5×10^7	5.2×10^5
4-cyanophenol	1.5×10^7	4.0×10^5

[a] Estimated to be accurate to ± 10%.
[b] In benzene.

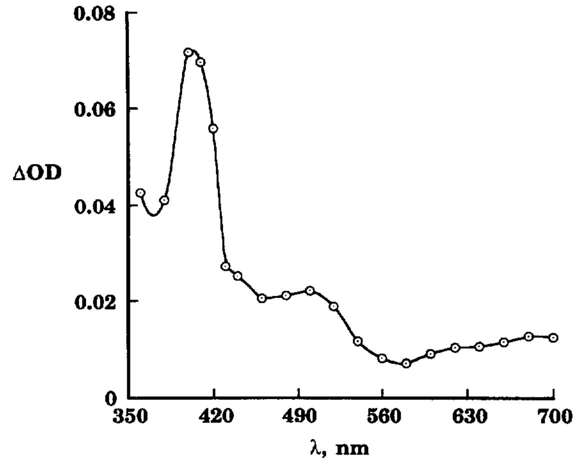

Fig. 4. Transient absorption spectrum obtained after 1.5 μs excitation (355 nm) of **3** in 4.7 × 10^{-3} M of 4-methoxyphenol in acetonitrile solution.

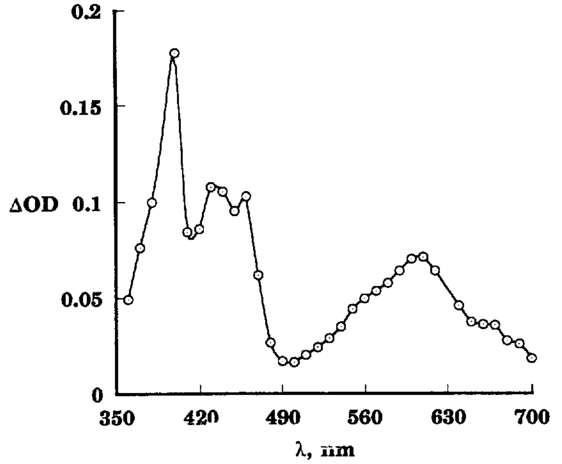

Fig. 5. Transient absorption spectrum obtained after 1.5 μs excitation (355 nm) of **3** in 0.9 mM of *para*-dimethoxybenzene in acetonitrile solution.

Phenoxyl radicals were easily detected when laser flash photolysis experiments were performed in the presence of phenols. For example, Fig. 4 shows the transient spectra of **3** in 4.7 × 10^{-3} M 4-methoxyphenol recorded 1.5 μs after the laser pulse. Phenoxyl radicals usually have strong absorption bands in the 370–505 nm region (depending upon substitution) [14]. In this spectrum the ketyl radical is probably hidden by the phenoxyl radical absorption.

In order to determine the role of electron transfer and possible implications of the presence of reaction sites other than phenolic hydrogen abstraction we recorded the transient absorption spectrum in 0.9 mM of *para*-dimethoxybenzene (Fig. 5). This spectrum shows a long lived transient with absorption at 620 nm that was attributed to the acenaphthenequinone radical anion. The benzophenone radical anion absorbs at 640 nm [15].

3.1.3. Isotope effect

The rates of reaction of the triplet of **3** and **4** with 4-methoxyphenol were also examined in 9:1 acetonitrile–water and in 9:1 acetonitrile–D_2O. The results are given in Table 2.

Table 2
Second-order rate constants for the quenching of the triplet of **3** and **4** by 4-methoxyphenol

Solvent	k_q[a], $M^{-1} s^{-1}$	
	3	**4**
Acetonitrile-H_2O (9:1v/v)	(4.21 ± 0.50) × 10^9	(2.42 ± 0.05) × 10^7
Acetonitrile-D_2O (9:1v/v)	(3.25 ± 0.50) × 10^9	(1.02 ± 0.04) × 10^7

[a] Estimated to be accurate to ± 10%.

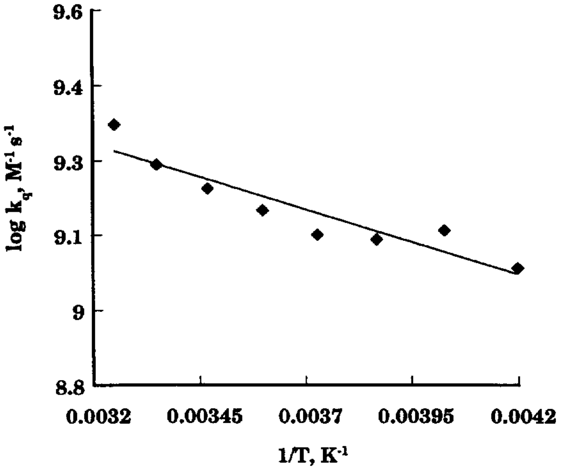

Fig. 6. Arrhenius plot for the quenching of **3** triplet in acetonitrile by various phenols.

3.1.4. Temperature dependence

The rate constants for the quenching of acenaphthenequinone (**3**) triplet by 4-methoxyphenol has been determined in acetonitrile in the temperature range −35 to 35°C. The Arrhenius plot is shown in Fig. 6 and gave $E_a = 1.2 \pm 0.2$ kcal mol^{-1} and $\log A = 10.2 \pm 0.2$.

4. Discussion

The diketone **3** has a triplet energy of 52 kcal mol^{-1} [9,10], which is slightly larger than that for the monoketone **4** ($E_T = 50$ kcal mol^{-1}) [11]. Both ketones are known to have the lowest triplet state as being $n\pi^*$ [11,16] with a triplet of $\pi\pi^*$ configuration relatively close in energy. Thus it would seem unreasonable to correlate the reduced reactivity of **4**, when compared to **3**, to a mixing of different states, as the same behaviour would be expected for both ketones, due to their configurational similarity. The data in Table 1 lead to the conclusion that, in spite of similar triplet energies, **3** shows a greater reactivity in the presence of a hydrogen donor when compared with the monoketone **4**.

The difference of reactivity of ketones **3** and **4** with 2-propanol and 1,4-cyclohexadiene is smaller than with phenols, which demonstrated the involvement of different mechanisms in these two hydrogen abstraction reactions. 2-Propanol and 1,4-cyclohexadiene are typical hydrogen atom donors and the magnitude of the rate constant obtained for the quenching of the triplets of **3** and **4** by these two compounds are in agreement with a mechanism similar to the one observed for the reaction of alkoxyl radicals [17,18].

Phenolic hydrogen abstraction by a carbonyl $n\pi^*$ triplet has long been known to be faster than from allylic or benzylic positions [2,14]. This can be attributed to the low bond dissociation and low oxidation potential of phenols. The reaction of the ketone triplets with *para*-dimethoxybenzene must involve an electron transfer process, since *para*-dimethoxybenzene has no easily abstractable hydrogen. This was evidenced by the formation of an absorption at ~610 nm due to the corresponding radical anion (Fig. 5). Evidence that the triplets of **3** and **4** were being quenched by phenolic hydrogen abstraction was the observation of the phenoxyl radical formation (Fig. 4). However, a hydrogen atom abstraction mechanism, similar to the one described above, seems unlikely since the reaction with phenols is faster than with 1,4-cyclohexadiene and 2-propanol (typical hydrogen atom donors).

Fig. 7 shows the Hammett plots for the reaction of the triplets **3** and **4** with phenols. The best correlation was obtained using σ^+ values [19], rather than σ values. Values of -1.50 (±0.17) and -1.48 (±0.15) were obtained for the reaction constant ρ for **3** and **4**, respectively. No changes in the mechanism were observed for phenol with donor or acceptor substituent. The similarity between the behavior of $n\pi^*$ carbonyl triplets and alkoxyl radicals toward hydrogen abstraction has been emphasized in several studies [20,21]. These ρ values are also in agreement with the reported value for the hydrogen abstraction by *tert*-butoxy from phenols [14,22]. The negative values of ρ for diketone **3** and monoketone **4** suggests that the transition states are electrophilic in nature, consistent with the known electrophilic character of carbonyl triplets. Most surprisingly, the similarity of the ρ values for the hydrogen abstraction reactions would indicate that the transition states are more or less equally electrophilic. This was unexpected as the characteristics of the $n\pi^*$ triplet of the α-diketone are considerably different to those of the monoketone [23]. The more negative value obtained in our experiments reflects the increased importance of polar structures in the transition state in polar solvents and it is also in agreement with a similar value of -1.4 reported by Das and Bhattacharyya [15] for the reaction of the benzophenone triplet with phenols in water:acetonitrile (1:1). Another study of the reaction of benzophenone triplet with phenols in benzene yielded a ρ of -0.6 [2]. It may well be that in this case the reaction constant values merely reflect solvation effects (a more polar environment in acetonitrile, when compared with results in benzene), and not a characteristic of the excited $n\pi^*$ triplet of the mono and diketones.

The mechanism of phenolic hydrogen abstraction by the carbonyl triplet is not completely understood, but our data can be rationalized in terms of a mechanism that involves initial formation of a triplet exciplex (ketone acceptor/arene donor) with substantial charge transfer, similar to the quenching of the benzophenone triplet by substituted phenols in benzene and aqueous acetonitrile [2]. In this mechanism it is expected that not only the lower oxidation potential of the phenols dominate the reactivity but also the larger reduction potential of the ketone.[1]

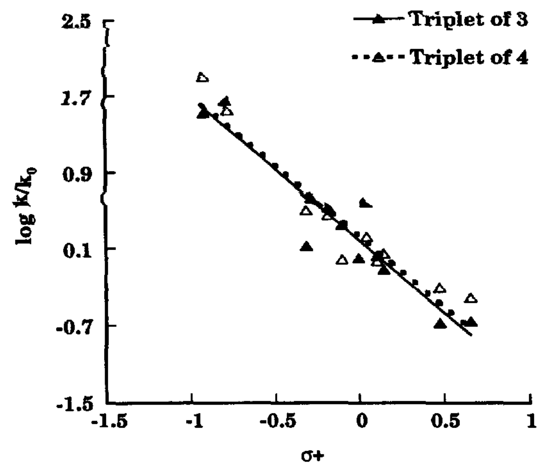

Fig. 7. Hammett plot for the hydrogen abstraction of the triplet of **3** ($r=0.94$) and **4** ($r=0.96$), by phenols, in ACN solution.

Recently Leigh et al. suggested that hydrogen bonding between the phenolic hydroxyl group and the carbonyl group apparently plays a crucial role in carbonyl triplet reactions of electron donor-substituted benzophenones, and under certain conditions proton transfer may be the driving force behind hydrogen atom transfer [24]. In this case the carbonyl triplets reveal nucleophilic character, this is contrary to the case of the ketones **3** and **4** where electrophilic character was clearly observed in the Hammett plots. Further, the authors suggested that the electron donor substituents reduce the ($n\pi^*$)–($\pi\pi^*$) energy gap of the substituted benzophenones and that the higher energy $\pi\pi^*$ triplet, through vibronic mixing, was responsible for the observed nucleophilicity. In this case our results are in agreement with a $n\pi^*$ triplet configuration for ketones **3** and **4** and we may also rule out the lack of reactivity of monoketone **4** as being due to the proximity of their $n\pi^*$ and $\pi\pi^*$ states.

It has been reported previously that the reactivity of carbonyl triplets with respect to phenolic hydrogen abstraction is reduced in hydrogen-bond accepting solvents compared to benzene [2,3,14]. This difference in reactivity between polar and apolar solvents was attributed to the higher concentration of free reactive phenol in the latter [2,14,15]. However this has been contested by the fact that the ratio between two given solvents is very dependent on the ketone used [25]. Our results are in agreement with this last statements as the ratios for quenching of the triplets in benzene and acetonitrile are different for the diketone **3** and the monoketone **4**.

The isotope effect observed in the case of 4-methoxyphenol is 1.3 for **3** and 2.4 for **4**. The magnitude of the isotope

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[1] A comparison between the reduction potential of diketone **3** (-0.9 V) [26] and monoketone **4** was not possible as the value for **4** is not available in the literature.

effect for reaction of **3** with 4-methoxyphenol obtained in this work is the same as that observed for the quenching of the triplet of benzophenone with phenols [2]. Comparison of the isotope effect of **3** with that of **4** indicates that hydrogen bond stretching in the transition state for the reaction of **3** with phenol is less important than for monoketone **4**. This could be the result of a more organized transition state for the former.

Despite the smaller triplet energy of diketone **3** in comparison to benzophenone, these triplets show similar reactivity. A comparison of the thermodynamic parameters for the interaction of the triplets of benzophenone [2] ($E_a = 3.7$ kcal mol^{-1}; log $A = 12.5$) and diketone **3** ($E_a = 1.2$ kcal mol^{-1}; log $A = 10.2$) with 4-methoxyphenol reveals that reaction of the triplet of **3** has a lower activation energy and smaller pre-exponential factor. Perhaps a more complete study should be done with diketones, as the results may indicate that an entropic factor has some contribution to the reactivity of diketone **3** in comparison to monoketone **4**, and should reflect a more organized transition state for α-diketones with a *cisoid* conformation, with the neighbouring carbonyl group assisting the hydrogen transfer.

5. Conclusions

The triplets of acenaphthenequinone (**3**) and 1-acenaphthenone (**4**) were detected by laser flash photolysis experiments. Hydrogen abstraction kinetic experiments in acetonitrile, using 1,4-cyclohexadiene, 2-propanol and phenols, revealed the triplet of **3** to be more reactive than the triplet of **4**. Hammett plots for the reaction of substituted phenols with the triplets of **3** and **4** confirmed the electrophilic nature of the triplets and the observation of similar reaction constants was attributed to a polar solvent effect. However, for both triplets phenolic hydrogen abstraction occurs with rate constants two orders of magnitude faster than with 2-propanol or 1,4-cyclohexadiene. The faster phenolic hydrogen abstraction is in agreement with a mechanism that involves an initial charge transfer.

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