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Photochemistry of 2-dialkylamino-1,4-naphthoquinones

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Abstract. The review surveys the results of studies on the photolysis of 1,4-naphthoquinones with 2-dialkylamino- and heterocyclic amino-groups. New data on the structures of the primary photolysis products and the mechanisms of their subsequent dark reactions are presented in relation to the reaction conditions and structure. The bibliography includes 53 references.

I. Introduction

Derivatives of 1,4-naphthoquinone are highly reactive and have a wide variety of valuable properties. Among naphthoquinones, dyes, vitamins, antibiotics, and pesticides are known. The ease of their excitation by light has led to the use of naphthoquinone derivatives as sensitisers and photoreducing agents in photographic materials of various types. Quinones with an internal source of hydrogen are most favoured. 2-Dialkylamino-1,4-naphthoquinones occupy a special place among them. They have found applications as photosensitive components in nonsilver photographic materials,[1–5] in particular in materials with physical development[1,2] and also in photoresist compositions.[4] Furthermore, the photochemical reactions of 2-dialkyl (1, ω-alkylene) amino-1, 4-naphthoquinones make it possible to obtain relatively easily derivatives of heterocyclic compounds which are either difficult or impossible to obtain by other procedures.[6–8] Much attention has therefore been devoted in recent years to the development of methods for the synthesis and study of the properties of 2-dialkylaminonaphthoquinones with various physicochemical characteristics.[9–21]

The wide variety of photochemical reactions of quinones has been most fully presented in the reviews by El'tsov et al.[22] and Maruyama and Osuka,[23] which cover the studies up to 1984. The present review is devoted to the photolysis of only 2-dialkyl (1, ω-alkylene) amino-1, 4-naphthoquinones, a characteristic feature of which is the intramolecular photochemical reduction reaction involving the abstraction of a hydrogen atom from the 2-position in the side chain via a readily formed six-membered transition state.[24] This determines the structure of the radicals formed — the primary photolysis products — and also the nature of the products formed when they are stabilised in secondary processes. The stabilisation can become, in the first place both intramolecular and intermolecular, i.e. with participation of the solvent.[22] The secondary intramolecular photochemical reduction reactions of 2-dialkylamino-1,4-naphthoquinones are primarily considered in the present review. In speaking of the primary photolysis products, we have in mind the primary products formed as a result of the stabilisation of the radicals.

The knowledge of the structure of the primary products and of the mechanism of the photolysis of photosensitive quinones makes it possible to determine the influence of various factors both on the process itself and on the stability of the primary photolysis products, which makes it possible to control the photochemical behaviour of the photosensitive component and the subsequent reactions of its photoinduced form in solutions and in polymer films.

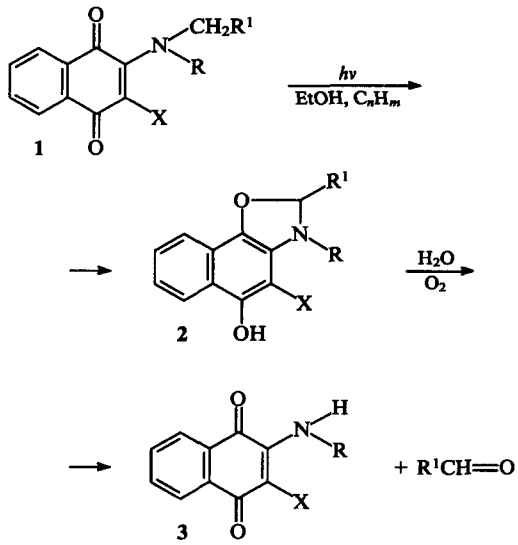
Recently published studies on the photolysis of 2-dialkyl-(1, ω-alkylene) amino-1, 4-naphthoquinones, using NMR spectroscopy as the main research method, made it possible not only to establish the structures of the primary photolysis products but also to distinguish the photochemical and thermal processes, which enables one to explain the formation of a wide variety of photolysis products. The data dealt with in the present review have been divided into two parts: studies concerning mainly the results of the photolysis of 2-dialkylamino-1,4-naphthoquinones, without a substituent in the quinone ring or having a substituent not involved in the photolysis, and subsequent reactions are discussed in the first part; the second part deals with studies on the photolysis of 2-dialkylamino-1,4-naphthoquinones with a substituent in the 3-position which participates in the formation of the photolysis product.

II. Photochemical reactions of 2-dialkyl (1, ω-alkylene) amino-1, 4-naphthoquinones

1,4-Naphthoquinones **1**, having a dialkyl- or an alkylarylamino-group in the 2-position, enter into an intramolecular photochemical reduction reaction on exposure to light with formation of relatively unstable naphtho [2, 1-*d*]-2, 3-dihydrooxazoles **2**,[25] which afford on decomposition the products **3** of the dealkylation of the initial quinones.[25–28]

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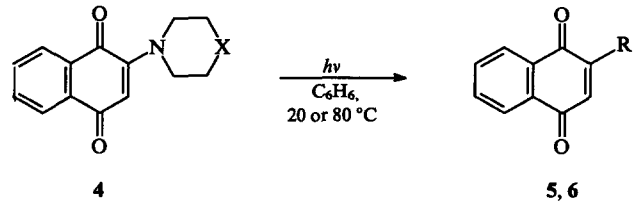


R = Me, Et, Ph; R^1 = H, Me; X = H, Cl.

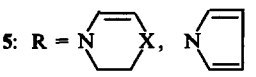
In contrast to 2-dialkylaminonaphthoquinones **1**, 1,4-naphthoquinones **4**, having a heterocyclic amino-group in the 2-position, give rise to dehydrogenation products **5**[29–31] and the products of the degradation of the heterocyclic ring **6**[30,31] on photolysis.

The study of the photolysis of 2-dialkylamino-1,4-naphthoquinones **1** (R = Me; R^1 = H, Me; X = H, Cl) and 2-morpholino-1,4-naphthoquinone **4** by measuring the optical spectra[32] showed that the primary photochemical stages of their photolysis are the same, so that also in the presence of a heterocyclic amino-group the primary photolysis products have the structure of naphthodihydrooxazole **7**, which was subsequently demonstrated by ^{1}H and ^{13}C NMR spectroscopy.[33,34]

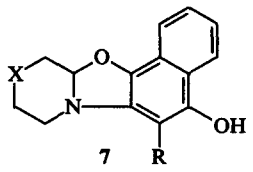
The conjunction of the amino-group with the π-system of the quinone is highly characteristic of 2-dialkylamino-1,4-naphthoquinones, which makes it possible to transfer the charge from the nitrogen atom to the oxygen atom directly during the



X = bond, CH_2, O.



6: R = $NH(CH_2)_3CHO$, $NH(CH_2)_4CHO$, $NH(CH_2)_2OCH_2CHO$, $NHCO(CH_2)_3CHO$, $NH(CH_2)_2OCHO$, $NH(CH_2)_2OH$, NH_2.

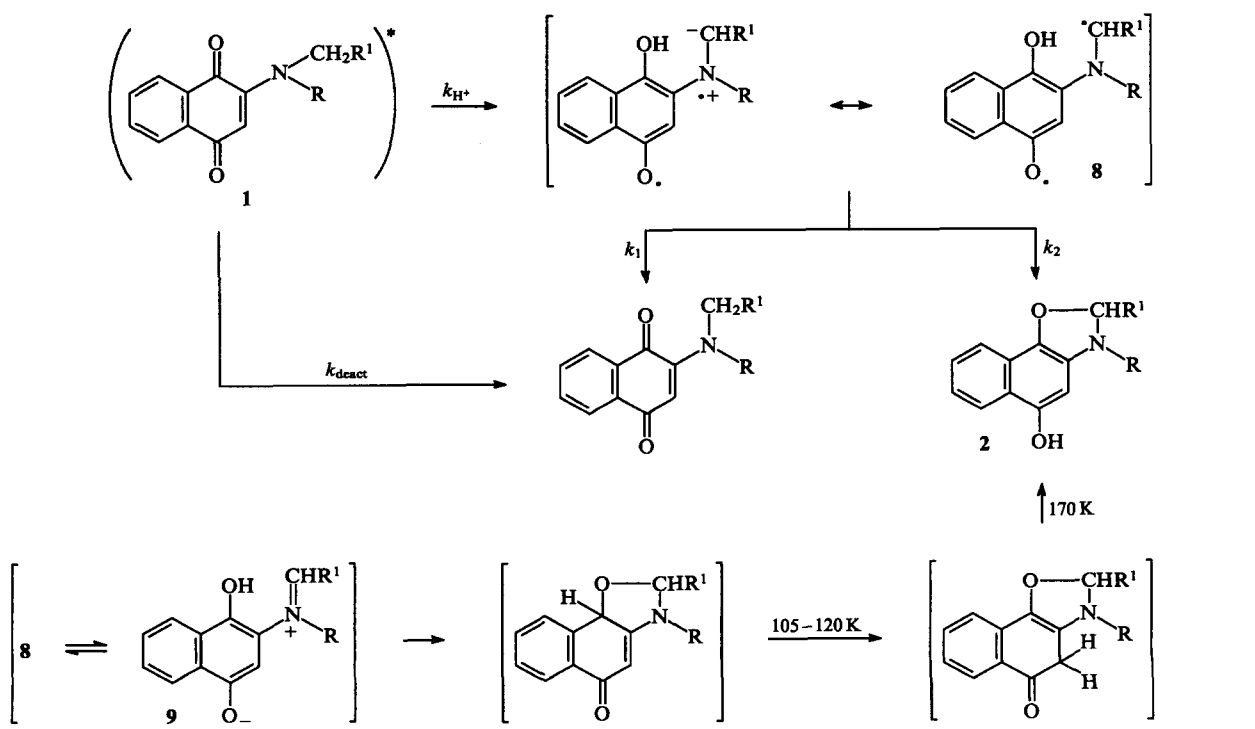


7: R = H, Me, OAlk; X = CH_2, O.

excitation process.[35] Quantum-chemical calculations of the electronic structures of 2-dialkylamino-1,4-naphthoquinones in the ground and excited triplet and singlet states showed that, on excitation of 2-aminonaphthoquinones, the electron density on the nitrogen atom falls appreciably[36,37] and increases on virtually all the atoms of the quinone ring, the increase being most significant not on the oxygen atom O(1) of the nearest carbonyl group but on the O(4) atom, which may be responsible for the subsequent migration of a hydrogen atom to the latter and this leads to cyclic structures of types **2** and **7**.[36]

The study of the influence of various factors on the quantum yields of the formation reaction of naphthodihydrooxazoles **2** is of interest both from the standpoint of the elucidation of the details of the mechanism and of the selection of the optimum

Scheme 1

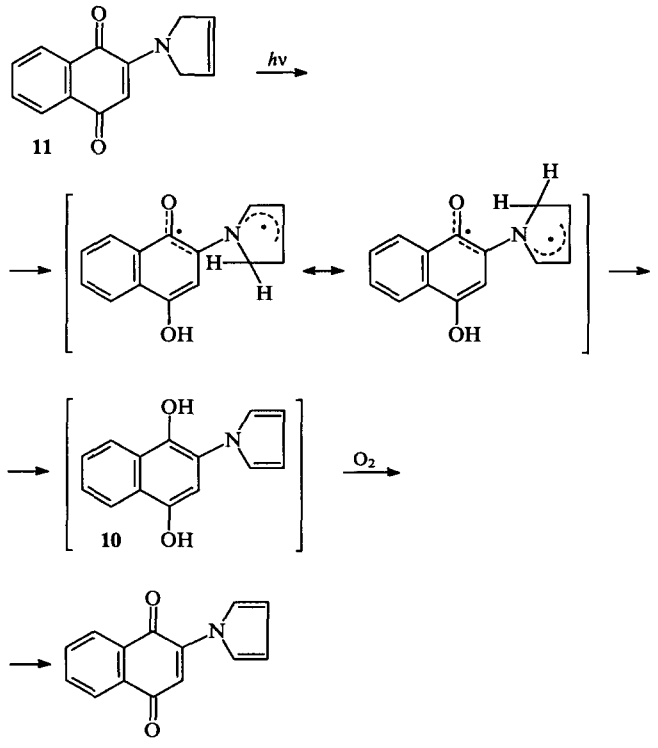


photolysis conditions. The nature of the solvent exerts a significant influence on the photolytic process. The photochemical reaction proceeds most effectively in nonpolar media such as hexane, benzene, and carbon tetrachloride; in alcohols, the effectiveness of the photolysis is appreciably lower owing to the formation of a hydrogen bond, which indicates the intramolecular abstraction of a hydrogen atom.[37] It has been established that this is a one-quantum and unimolecular process. A state of the $r_a\pi^*$ type is reactive, but the multiplet nature of the reactive state could not be established experimentally.[32,37] It has been shown that the nature of the photolysis product is independent of the irradiation wavelength (250–500 nm), the light intensity, and the concentration of the solutions ($0-2 \times 10^{-3}$ M). This permitted the assumption that the primary stage of the process is proton phototransfer and made it possible to put forward a hypothetical mechanism of the photolysis of 2-dialkylamino-1,4-naphthoquinones (Scheme 1).[32,35]

The multistage mechanism of the conversion of the photolysis products at 77 K into the product **2**, detected at room temperature, was put forward on the basis of low-temperature photolysis data. However, a biradical structure of type **8** could not be detected and its isomerisation to the zwitterion **9** was postulated.[35]

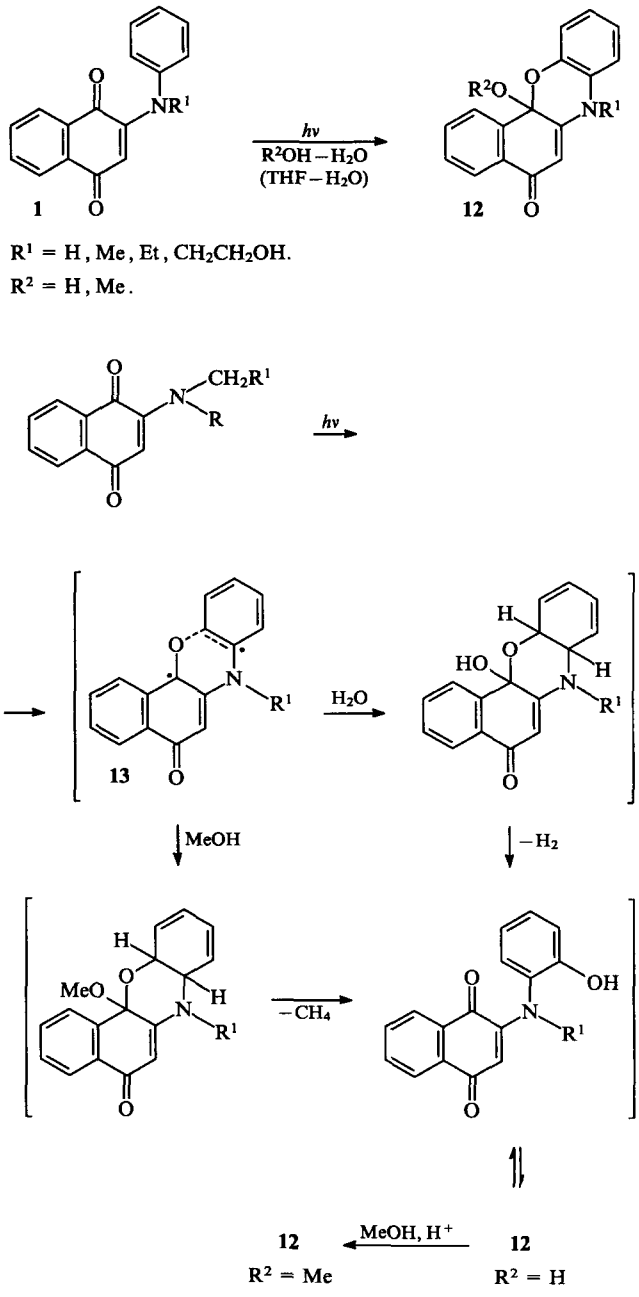
A somewhat different mechanism for the photolytic reaction—via the unstable 1,4-dihydroxy-2-(1-pyrrolyl)naphthalene **10**, which was detected as the diacetate—was proposed for the naphthoquinone **11** (Scheme 2).[30] The study of the photolysis of 2-(1-indolyl)-1,4-naphthoquinone in the presence of dienophiles may serve as its confirmation.[38]

Scheme 2



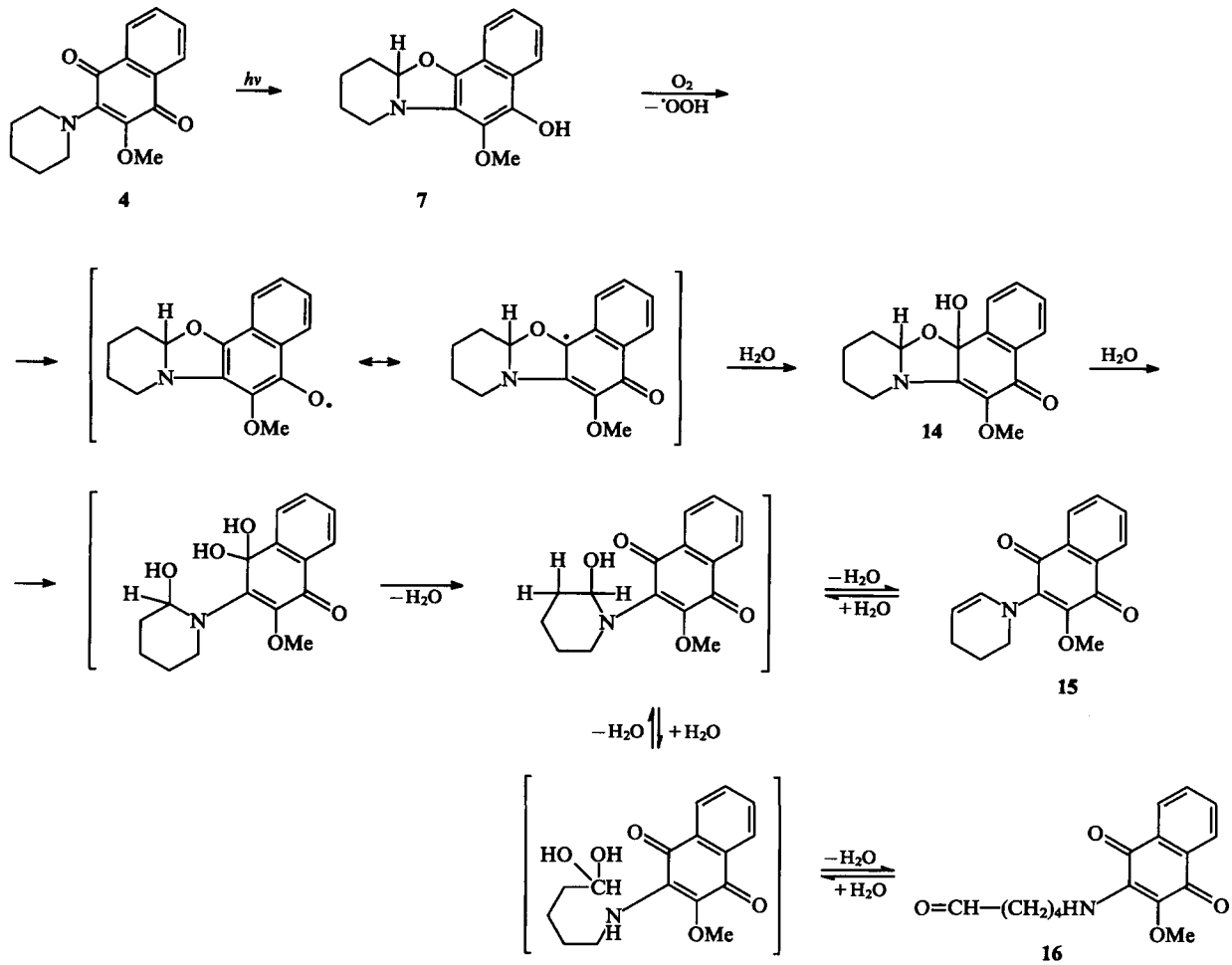
There are contradictory data as regards the products of the photolysis of 2-alkylphenylamino-1,4-naphthoquinones **1** (X = H, R = Ph). As already stated above, according to some data[25] irradiation results in the formation of naphthodihydrooxazoles **2**, whilst according to others[28] the products are benzophenoxazone derivatives **12**. It is noteworthy that the conditions of the photolysis in the above studies,[25,28] were different (water was present in the latter study[28]), which may partly alter the course of the reaction. It has been suggested[28] that the nonbonding orbital on the oxygen atom of the carbonyl group interacts in the excited state with the π electrons of the aromatic ring. There is then insufficient time for the occurrence of the intra- and intermolecular abstraction of a hydrogen atom by the biradical **13** formed and the latter is stabilised by combining with the solvent and cyclising (Scheme 3). Nevertheless, together with benzo[*c*]phenoxazone derivatives, dealkylation products (in some cases a product of this kind was the main one) were obtained with the preferential elimination of the alkyl residue compared with the aryl residue. This type of dealkylation is the result of the hydrolysis of the relatively unstable naphthodihydrooxazoles of type **2**.[25]

Scheme 3



According to the results of Gritsan and Bazhin,[32] the primary stage in the dark reactions of the naphthodihydrooxazoles **2** and **7** is the hydrolysis of the C–O bond, while according to Maruyama et al.[30] oxygen exerts the primary effect. The study of the properties of compounds **7** (R = Me, OMe, OPr^i; X = CH_2) showed that they are stable

Scheme 4

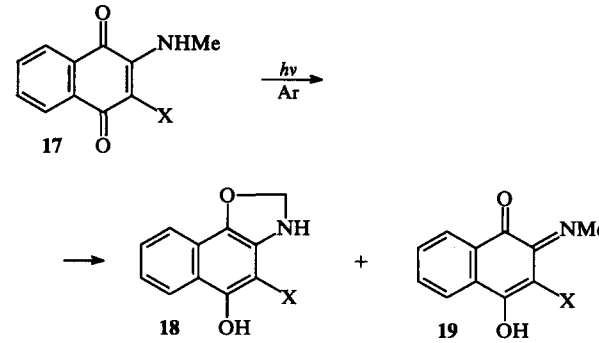


in the absence of oxygen for a long time at temperatures in the range from −70 to +23 °C.[33,39] The smallest traces of oxygen lead to the decomposition of the photolysis products; 6-methoxy-9,10,11,11*a*-tetrahydro-8*H*-naphtho[2′,1′:4,5]oxazolo-[3,2-*a*]pyridin-5-ol **7** gives rise to a mixture of compounds — the enaminoquinone **15**, the aminoaldehyde **16**, and compounds in which the oxazole ring is retained, namely 12*a*-hydroxy-6-methoxytetrahydroxazolopyridin-5-one **14**. The formation of compound **14** and the study of its properties enabled Shishkina et al.[33] to propose a mechanism of the dark reactions of the primary products of the photolysis of naphthodihydrooxazoles **7** including oxidation as the first stage (Scheme 4).

The methoxy-group as well as the more bulky isopropoxy-group[39] in the 3-position of 2-dialkylamino-1,4-naphthoquinones do not participate in photolysis on irradiation with light at $\lambda > 400$ nm,[33] although there are data showing that 2-alkoxy-1,4-naphthoquinones unsubstituted in the 3-position undergo photochemical reactions under more severe conditions.[40]

The methyl group is retained on irradiation of 2-dialkyl-(1,ω-alkylene)amino-3-methyl-1,4-naphthoquinones,[34] but its presence exerts a significant influence on the effectiveness of the photolysis: the quantum yields are 2–6 times higher than for 2-dialkylaminonaphthoquinones without a methyl group in the 3-position.[34,41,42] The greater effectiveness of the photochemical reaction of 2-dialkylamino-3-methyl-1,4-naphthoquinones made it possible to observe photochemical dealkylation also in the case of 2-monoalkylamino-1,4-naphthoquinones — a reaction which had not been known previously.

It has been found[45] that, on irradiation ($\lambda > 400$ nm) in organic solvents and under an inert atmosphere, compounds **17** are converted into naphthodihydrooxazoles **18** and 1,2-naphthoquinone 2-imines **19**. In the series of 1,4-benzoquinones, the stability of monoalkylamino-derivatives on irradiation has been explained by the presence of an intramolecular hydrogen bond between the NH and CO groups of the quinone.[43,44]



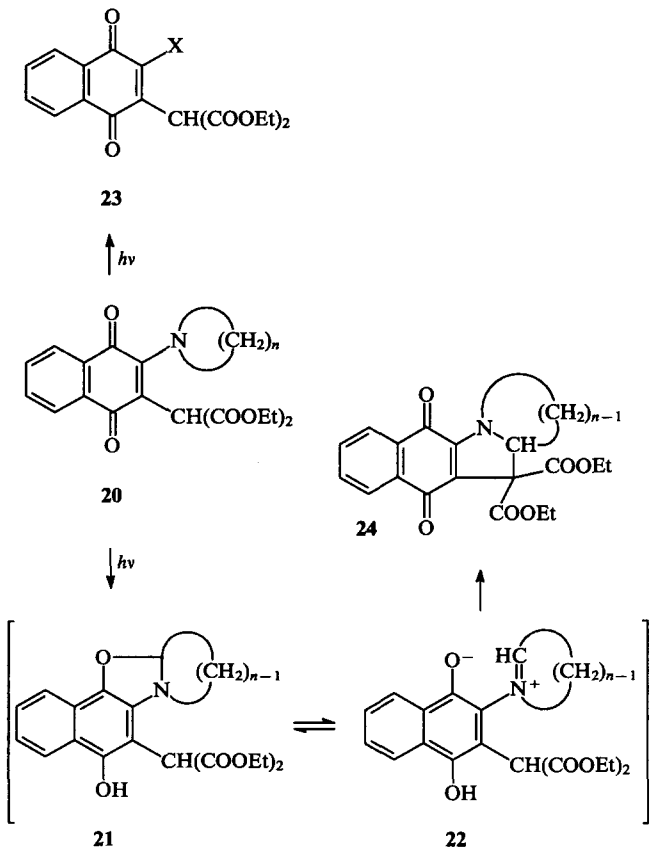
X = Me, OMe.

Since the methyl group gives rise to approximately the same bathochromic shift as the chlorine atom and the methoxy-group in the electronic absorption spectra of the corresponding 2-dialkylamino-1,4-naphthoquinones,[34] its activating effect is apparently not determined solely by the electronic effect of the substituent. It may be that the enhanced effectiveness of the photolysis of 2-dialkylamino-3-methyl-1,4-naphthoquinones is associated with their ability to form the quinone methide structure similar to that described by Serdobov.[46] However, one cannot rule out the additional involvement of the methyl group in the stabilisation of biradicals of type **8** as a result of the delocalisation of one of the unpaired electrons.[42]

III. *ortho*-Substituted 2-dialkyl(1,ω-alkylene)-amino-1,4-naphthoquinones

The results of the photochemical reactions of 2-dialkyl-(1,ω-alkylene)amino-1,4-naphthoquinones without substituents in the *ortho*-position or with substituents such as a halogen, an alkoxy-group, or a methyl group, which do not affect the nature of the photolysis products, were discussed in the previous section. At the same time, there have been studies indicating the possible involvement of the *ortho*-substituent in the overall photolytic process.

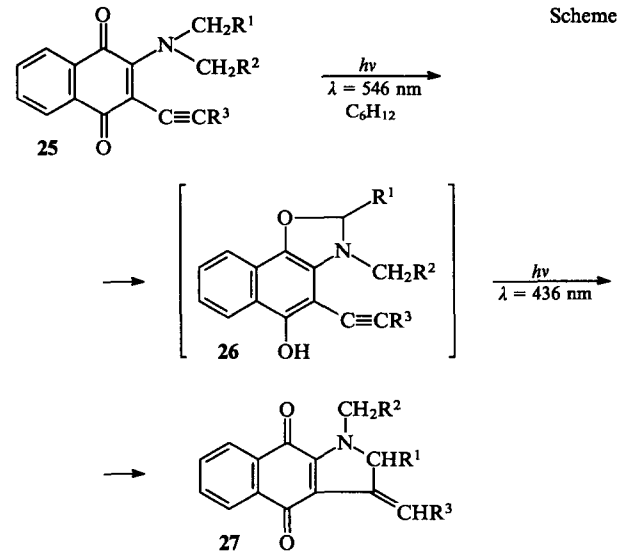
Thus irradiation of 2-piperidino- and 2-pyrrolidino-1,4-naphthoquinones **20**, having an active methylene group in the 3-position, leads both to the products **23** of the dehydrogenation and degradation of the heterocyclic ring (a situation similar to that indicated by the results of Fokin and Detsina[29] and Maruyama et al.[30]) and to the indoloquinones **24**, the yield of the latter increasing in polar solvents.[7] It has been suggested that the intermediate oxazole **21** is converted into the indoloquinone **24** via the zwitterionic intermediate **22** in a polar medium.



There exists an example of the photochemical activity of the naphthodihydrooxazoles **26** formed on photolysis of 2-dialkylamino-3-alkynyl-1,4-naphthoquinones **25**, but the mechanism of the formation of the indole derivatives **27** has not been discussed.[47] This fact merits special attention, since it had been shown earlier[38] that naphthodihydrooxazoles without a substituent in the 3-position are stable on irradiation with light at $\lambda > 300$ nm (Scheme 5).

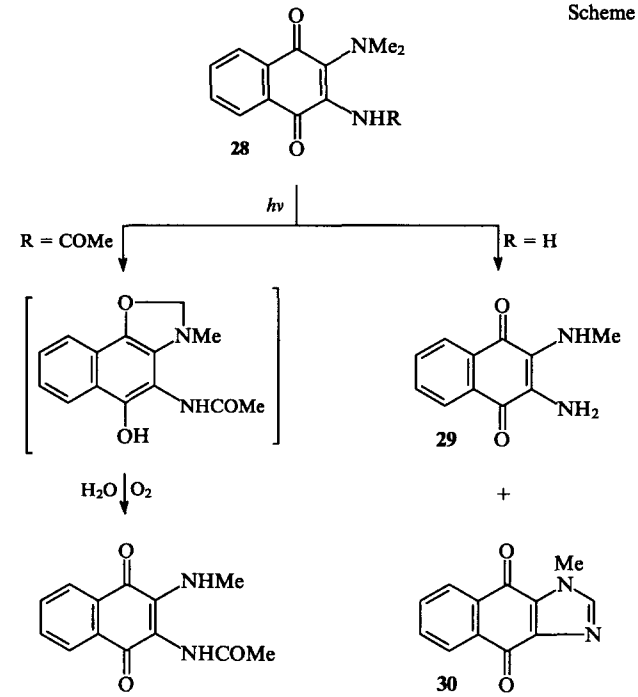
The products of the photolysis of 3-amino-2-dialkylamino-1,4-naphthoquinones are distinguished by the greatest variety. Thus irradiation of 3-amino-2-dimethylamino-1,4-naphthoquinone **28** (R = H) afforded, together with the dealkylation

Scheme 5



R^1 = H, Me; R^2 = Me, Et; R^3 = H, Ph, CH_2OPh, CH_2OCOMe.

Scheme 6

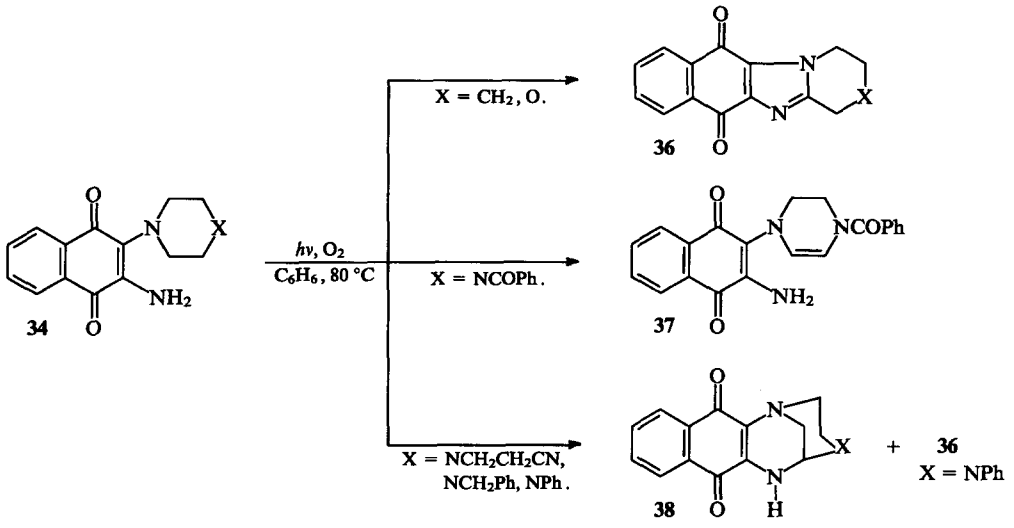


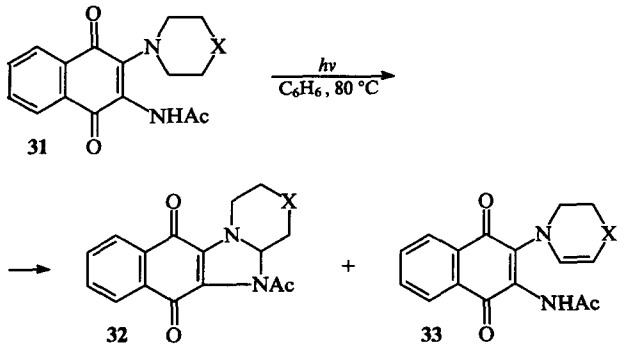
product **29**, 1-methylnaphtho[2,3-*d*]imidazole-4,9-dione **30**, whereas only the dealkylation product is formed on photolysis of the 3-acetamido-derivatives **28** (R = COMe) (Scheme 6).[48]

When the 2-dialkylamino-group is replaced by a heterocyclic group, 3-acetamidonaphthoquinones **31** form on irradiation the dihydroimidazole derivatives **32** as well as the ring dehydrogenation products **33**.[49]

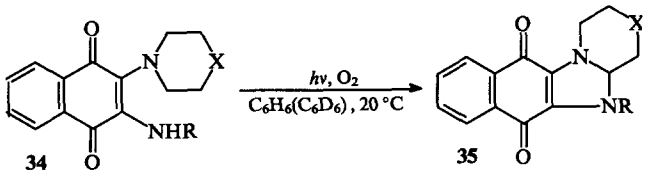
For the 3-aminonaphthoquinones with a cyclic alkylamino-group in the 2-position, the nature of the photolysis product is determined not only by the structure of the initial quinone but also by the reaction temperature. Thus the products of the photolysis of 2-cycloalkylamino-1,4-naphthoquinones with both primary[48,49] and secondary (alkyl,[39,50] and aryl[50,51]) amino-groups in the 3-position in the quinone at 20 °C are exclusively the unstable naphthodihydroimidazolediones **35**. Compounds **35** (R = Et) can be detected only spectroscopically,[39,51] since they are dealkylated with formation of naphthoimidazolediones **36** and, according to the data of Rubashko et al.,[50] the process takes place only under the influence of light. Depending on the

Scheme 7

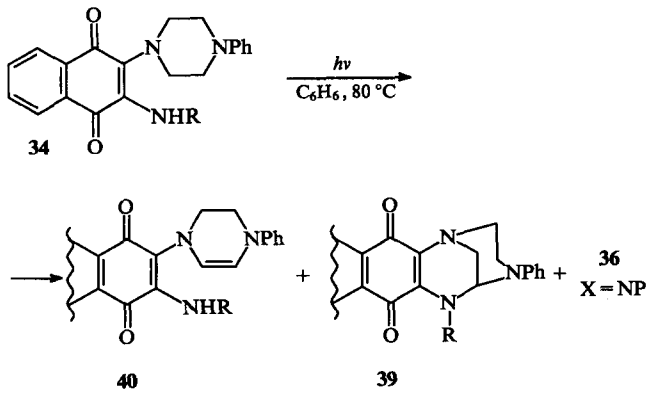




X = CH_2, O, NCOPh.



R = H, Et, C_6H_4Me-*p*, C_6H_4COMe-*p*;
X = CH_2, O, NCH_2CH_2CN, NPh, NCOPh.



R = Et, C_6H_4Me-*p*.

nature of the cyclic alkylamino-group, naphthoimidazole derivatives **36**,[49] enaminoquinones **33**,[49,51] as well as 1,2,3,4-tetrahydro-13-alkylphenyl)-3,1-(iminoethano)benzo[*g*]quinoxaline-5,10-diones **38** [51–53] are formed at 80 °C (Scheme 7).

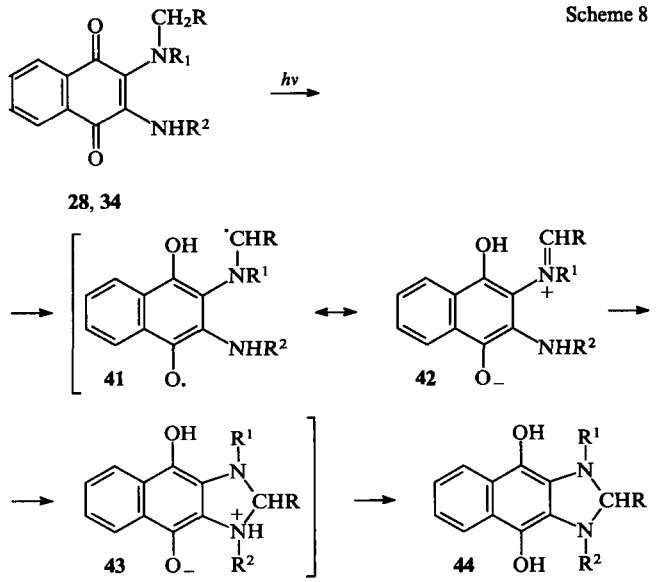
Tetrahydrobenzo[*g*]quinoxalinediones of type **38** are also the main products of the photolysis of 2-(4-alkylpiperazino)-1,4-naphthoquinones with a secondary amino-group in the 3-position; compound **34** (R^1 = C_6H_4Me-*p*), having a phenyl group in the 4-position in the piperazine ring, gives rise to the enaminoquinone **40** and the corresponding naphthoimidazoledione **36** (X = NPh) in addition to benzoquinoxalinedione **39**.[51–53]

The wide variety of the data obtained is due to the increased sensitivity of the primary photolysis product to atmospheric oxygen and heating. It has been established by NMR spectroscopy that this product has the structure of naphthodihydroimidazolediol **44**. In contrast to a polar medium where the spectrum is recorded directly, in a nonpolar medium the spectrum of the photolysis product cannot be recorded; it has been suggested that the photolysis stops at the stage involving the formation of the zwitterion **43**. This enabled the authors[39] to conclude that the *ortho*-amino-group participates in the reactions of the biradicals **41** arising in the intramolecular photochemical reduction process (Scheme 8).

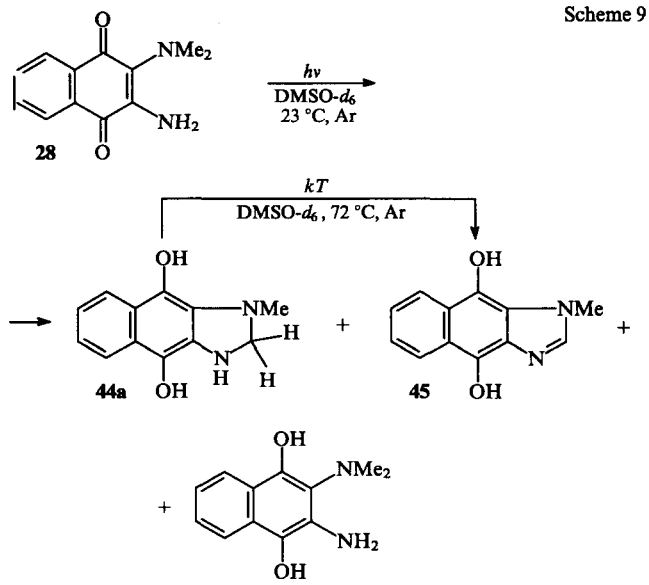
The naphthodihydroimidazolediols **44** are relatively unstable. In order to elucidate the pathway leading to the formation of a wide variety of final photolysis products from them (of the type of compounds **30** and **35**–**38**), a study was made of the influence of various factors (temperature, atmospheric oxygen, and the structure of the compounds) on their further reactions.[39,51,52] It was shown that naphthodihydroimidazolediols **44a**, which are unsubstituted in the 2-position, are oxidised under an inert atmosphere at a temperature in excess of 20 °C to the corresponding naphthoimidazolediols **45**, with the initial quinone or the solvent serving as the hydrogen acceptor.[39] In contrast to dihydroimidazolediol **44a**, which has no substituent in the 2-position in the imidazoline ring (Scheme 9), solutions of the products of the photolysis of 3-amino-2-piperidino-1,4-naphthoquinones **44** [R, R^1 = $(CH_2)_4$, R^2 = H, Et, *p*-MeC_6H_4] are stable at ~20 °C under an inert atmosphere. On exposure to atmospheric oxygen, they give rise to the products of the oxidation of the imidazoline and naphthalene rings of type **35** and **36**.[39,51]

A significant influence of the structure of naphthodihydroimidazolediols on their subsequent thermal reactions has been observed for compounds **44b**, c in which the imidazoline ring has a [2-*a*]-junction with a piperidine or 4-substituted piperazine ring. It was found that heating of compound **44b** — the product

Scheme 8

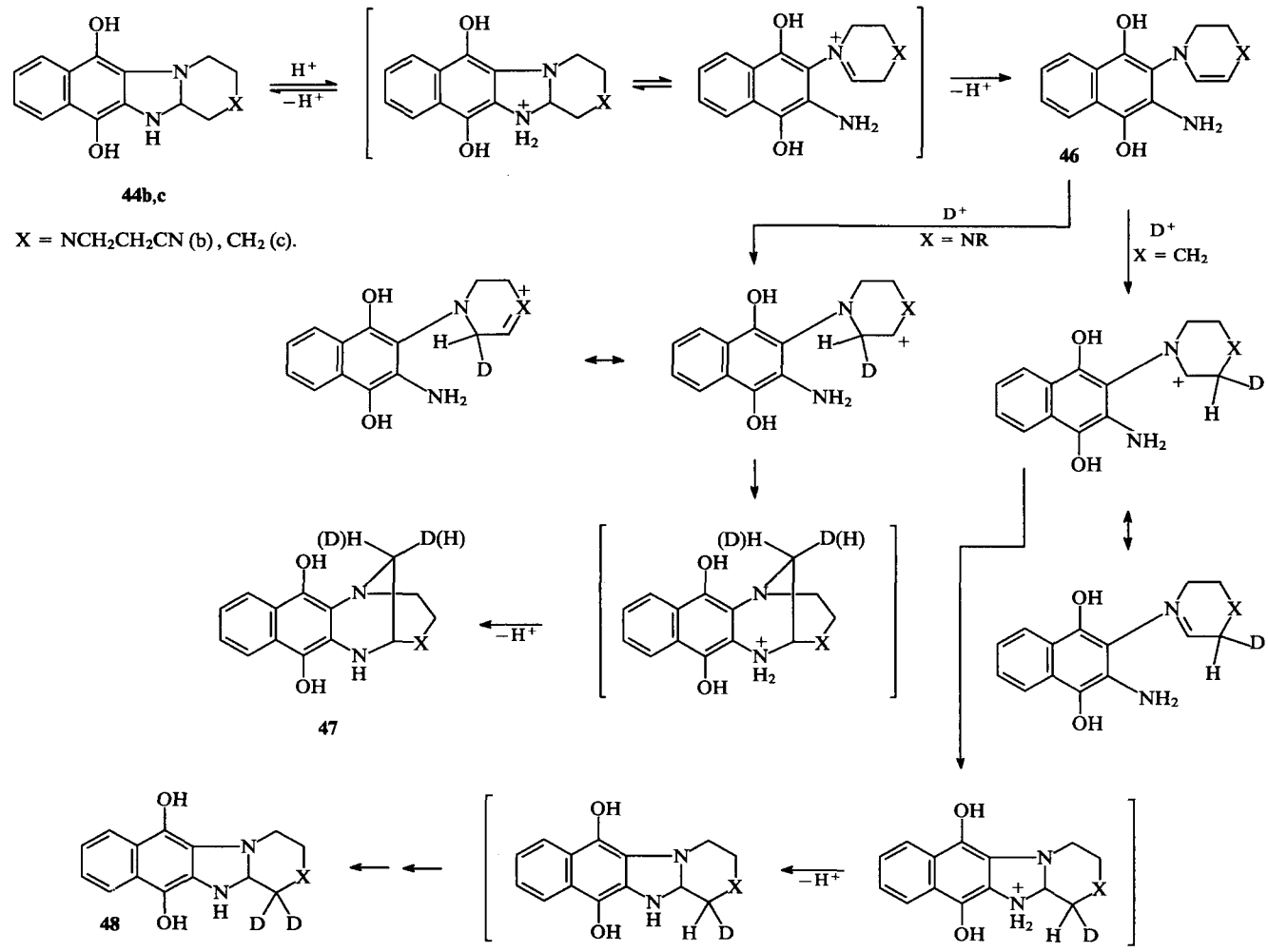


Scheme 9



of the photolysis of 2-[4-(2-cyanoethyl)piperazino]-1,4-naphthoquinone — leads to recylisation with formation of 1,2,3,4-tetrahydro-13-(2-cyanoethyl)-3,1-(iminoethano)benzo[*g*]quinoxaline-5,10-diol **47**, which is oxidised to the corresponding dione **38**. In the case of the [1,2-*a*]-junction between the imidazoline ring of naphthodihydroimidazolediol and a piperidine (compound **44c**) or piperazine ring having an electron-accepting substituent in the 4-position (compound **44b**), the thermolysis product has the structure of naphthodihydroimidazolediol **48**, i. e. the rearrangement is degenerate in character. On the basis of deuterium exchange data, a mechanism was proposed for the recyclisation of naphthodihydroimidazolediols, including the intermediate formation of the reduced form **46** of the enaminoquinone (Scheme 10).[51,52]

Scheme 10



Thus it has been shown[39,51,52] that, in the photolysis of 3-amino-2-dialkylamino-1,4-naphthoquinones, the *ortho*-substituent participates in the formation of the structure of the primary stable photolysis product—naphthodihydroimidazolediol, the subsequent reactions of which are determined by its structure and the photolysis conditions.

IV. Conclusion

It follows from the few available data on the mechanism of the photolysis of 2-dialkyl(1,ω-alkylene)amino-1,4-naphthoquinones that, regardless of the presence of other substituents in the naphthoquinone, the primary stage of the process is the phototransfer of a proton from the carbon atom in the α-position in the dialkylamino-group to the oxygen atom of the carbonyl group. The postulate that biradical structures of type **8** are formed and isomerise to the zwitterions **9**,[37] the subsequent cyclisation of which proceeds, depending on the structure, either with participation of the oxygen atom (formation of naphthodihydrooxazoles)[25,33,34] or with participation of the *ortho*-substituent,[7,39,47–52] is entirely logical. The structure of the final photolysis product depends both on the irradiation conditions (temperature, the presence of atmospheric oxygen and water) and on the structure of the initial quinone.

NMR spectroscopy proved to be useful for the establishment of the mechanism of the photolysis of various 2-dialkylamino-1,4-naphthoquinones. It makes it possible to determine reliably the structure of the primary photolysis products and the structure of the compounds formed as a result of further dark reactions.

The questions concerning the structure of the primary photolysis products when 2-dialkyl(cycloalkyl)amino-1,4-naphthoquinones contain in the 3-position substituents other than a hydrogen or halogen atom and alkyl, alkoxy- and amino-groups and their influence on the subsequent reactions remain obscure. The problem of the behaviour of the primary photolysis products on further irradiation requires resolution.

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