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Journal of Photochemistry and Photobiology A: Chemistry 162 (2004) 579–584

Journal of Photochemistry and Photobiology A:Chemistry

www.elsevier.com/locate/jphotochem

Photolysis of 4-azidodiphenyl in solution studied by UV absorption spectroscopy

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Received 3 May 2003; received in revised form 4 October 2003; accepted 7 October 2003

Abstract

4-Azidodiphenyl was photolysed with low intensity radiation in oxygen-free and oxygen containing dichloromethane solutions. It was found that the main product is azodiphenyl when the concentration is greater than about 10^{-2} mol/l and oxygen is absent. In the case of oxygen containing diluted azide solutions, the triplet azide molecules are trapped by oxygen to form, probably, a nitroazodiphenyl intermediate. In both cases, the light-induced decomposition of azide can be treated approximately as a unidirectional first order (UFO) reaction, where the reactant is the only light absorbent. The deviation from such kinetics appearing at high azide conversions is mainly caused by the nitrene—solvent reaction. The concentration of unreacted azide was measured by the absorbance at 276 nm.

Keywords: 4-Azidodiphenyl; Photolysis; 4,4′-Azodiphenyl; Photokinetics

1. Introduction

As known, the final products resulted from the decomposition of azides are rather numerous because each of the primary products (excited molecules and nitrene radicals in singlet (s) and triplet (t) state) shows specific reactions. The main light-induced reactions occurring with an aromatic azide ArN_3 in a solvent RH are the following [1–4]:

$$\mathrm{Ar{-}N_3} + h\nu \xrightarrow{k_{\Phi}} \mathrm{Ar{-}N_3(s)} + \mathrm{Ar{-}N_3(t)} \xrightarrow[-\mathrm{N_2}]{} \mathrm{Ar{-}N:(s)} + \mathrm{Ar{-}N:(t)} \quad (1)$$

$$\mathrm{Ar{-}N:}\binom{\mathrm{s}}{\mathrm{t}} + \mathrm{Ar{-}N_3} \xrightarrow{k_{\mathrm{na}}} \mathrm{Ar{-}N{=}N{-}Ar} + \mathrm{N_2} \quad (2)$$

$$\mathrm{Ar{-}N:}\binom{\mathrm{s}}{\mathrm{t}} + \mathrm{Ar{-}N:}\binom{\mathrm{s}}{\mathrm{t}} \xrightarrow{k_{\mathrm{nn}}} \mathrm{Ar{-}N{=}N{-}Ar} \quad (3)$$

$$\mathrm{Ar{-}N:}\binom{\mathrm{s}}{\mathrm{t}} + \mathrm{RH} \xrightarrow{k_{\mathrm{ns}}} \frac{\mathrm{ArNHR}}{\mathrm{ArNH_2}} \quad (4)$$

$$\mathrm{Ar{-}N:(t)} + \mathrm{O_2} \xrightarrow{k_{\mathrm{no}}} \text{nitro, nitroso, and azoxyarenes} \quad (5)$$

The reaction rates are, evidently, dependent on the reaction conditions and we tried to find particular cases when one or two of the reactions were favoured. The rate constant $k_{\Phi} = \Phi \times \varepsilon \times I \times \ln 10$, where Φ is the quantum yield of azide photomodification, ε is the molar extinction coefficient of azide, and I is the light intensity, must be associated to the primary processes of irreversible transformation. It is to be mentioned that k_{Φ} usually differs from the rate constant of azide consumption due to reaction 2.

On the other hand, the kinetic treatment of photochemical reactions involves equations that can be exactly solved only for the unidirectional first order (UFO) photoreactions and only if the reactant absorbs light [5]. Even in this simple case, the momentary concentration of species is dependent on the irradiation depth and time, except in the case of fluid media where diffusion and Brownian motion spatially average the concentration. Zimmerman et al. [6] introduced the equation of the rate for a reversible first-order photoreaction in solution. Although its integration is not possible, this equation has been successfully used to determine photokinetic parameters by means of ingenious methods of approximation. It was applied especially to the photoisomerization of azoaromatic compounds, both in solution and polymer matrix [7,8].

Most of the measurements have been made in rigid solutions at low temperature where the nitrenes have long life times and, consequently, the secondary reactions 2–5 can be monitored on-line [4,9]. Studies performed in fluid solutions are few in number and mostly refer to strong irradiation experiments when the nitrene recombination (Eq. (3)) is the predominant secondary reaction ([4d,9,10]). On the other hand, studies of azide photolysis kinetics

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doi:10.1016/S1010-6030(03)00422-2

where the Zimmerman's equation is used are not available. Such a treatment is necessary because the reaction mixtures are optically dense media.

This article presents photolysis experiments carried out with 4-azidodiphenyl in such conditions so that the decomposition can be approximated by an UFO reaction. As the Zimmerman's equation is exactly solvable in this case, the quantum yield of azide decomposition was obtained from graphic plots of experimental data. 4-Azidodiphenyl was chosen from a number of aromatic azides previously studied [11] because its electronic absorption band seems to be less affected by reaction products. It is known that the strong irradiation of this azide in concentrated solutions gives good yield of azodiphenyl [12,13]. We used low levels of irradiation so that the reaction 3 is negligible relative to reactions 2, 4 and 5. Special attention was given to the influence of oxygen. Relative to this subject, it is accepted that nitro, nitroso and azoxy compounds are the characteristic arylazide photolysis products in the presence of oxygen, but the reaction mechanisms of their formation are not well clarified [3,14–16].

2. Experimental section

4-Azidodiphenyl was prepared by reacting the diazonium salt of 4-aminodiphenyl with sodium azide [17,18]. IR (KBr, cm^{-1}): 2110 (doublet, $-N_3$ group), 1590, 1475, 1130, 835, 760, 690; ^{1}H-NMR ($CDCl_3$, TMS, 400 MHz): δ 7.096 (H_2, $J_{2,3} = 8.4$ Hz), 7.556 (H_3), 7.567 (H_8, $J_{8,9} = 7.6$ Hz), 7.434 (H_9, $J_{9,10} = 7.2$ Hz) and 7.340 (H_{10}); UV-Vis (dichloromethane, $\lambda_{max} = 276$ nm; [ε] [27000 l mol^{-1} cm^{-1}]); mp 77–79 °C.

trans-4,4′-Azodiphenyl was obtained from 4-nitrodiphenyl in according with a literature method [19]. IR (KBr, cm^{-1}): 1510 (–N=N– group), 1480, 1445, 1400, 840, 760, 720, 685; ^{1}H-NMR ($CDCl_3$, TMS, 400 MHz): δ 7.756 (H_2, $J_{2,3} = 8.8$ Hz), 8.016 (H_3), 7.762 (H_8, $J_{8,9} = 7.8$ Hz and $J_{8,10} = 1.2$ Hz), 7.457 (H_9, $J_{9,10} = 7.2$ Hz), and 7.387 (H_{10}); UV-Vis (dichloromethane, $\lambda_{max} = 362$ nm); mp 184–187 °C. This product is changed to its *cis* form ($\lambda_{max} = 260$ nm) by irradiation with 365 nm in dichloromethane. It was used to confirm the chemical structure of the product that results by photolysis of 4-azidodiphenyl in concentrated solutions.

Other characteristics of electronic absorption spectra in dichloromethane of interest for the present study are the followings: $\lambda_{max} = 305$ nm for 4-nitrodiphenyl and $\lambda_{max} = 278$ nm for 4-aminodiphenyl.

The irradiation and spectral measurements were performed at room temperature in spectrophotometric grade dichloromethane solutions. Dichloromethane is considered to be rather inert towards nitrene (Ref. [2], p. 451). Two kinds of solutions, de-aerated and oxygen containing, were used. The de-aeration was carried by bubbling argon. Alternatively, the bubbling method was used to introduce oxygen (about 10^{-2} mol/l, according to literature [20]). A water layer was used to avoid solvent evaporation.

Two kinds of irradiation cells were used: standard cells, with optical path length of 10, 2 and 1 mm, and assembled cells with spacers of 0.1 and 0.03 mm thickness. The irradiation was performed at 280 nm using a 350 W medium pressure mercury lamp and the incident irradiance was about 0.4 W m^{-2} that means 2×10^{-5} einstein m^{-2} s^{-1}. The nitrene recombination (reaction 3) is altogether negligible for such radiation intensity because, for example, in the most favourable case (10^{-5} mol/l azide concentration, 0.03 mm optical path, unitary yield of nitrene formation and 10^{-6} s lifetime), the momentary density of photons is 10^4 times smaller than that of azide.

The electronic absorption spectra were recorded in dark with a Specord M42 spectrometer. The "interval method" [21], where the irradiation was interrupted after a specific time and the sample was transferred to the spectrometer, was used. The spectra recorded in time were identical meaning that the reaction products are stabilised fast after irradiation. The concentration of unreacted azide was measured by the absorbance at 276 nm.

A special experiment was carried out to obtain more information about the product that results by irradiation of aerated solutions. A quantity of 0.4 l solution of 5×10^{-5} mol/l was irradiated at 280 nm until the azide band completely disappeared (about 3 h). It was observed the appearance of a new absorption band with maximum at 342 nm. Then, the solvent was evaporated by using an air stream at room temperature. Now the absorption of the product (a pale brown solid) in dichloromethane was found shifted at 336 nm. After then the product was processed to obtain a KBr pellet. Rather strange, it was found that the recorded IR spectrum quite well coincides with that of 4-nitrodiphenyl, namely: 1600, 1580, 1480 and 1445 cm^{-1} from benzene –C=C– stretching, 1515 and 1395 cm^{-1} from $-NO_2$ stretching, 1105 cm^{-1} from C–N stretching, and 855, 765, 745 and 695 cm^{-1} from C–H deformations of mono and 1,4 disubstituted benzene rings. Additional bands were observed at 1620 and 1125 cm^{-1}, the both of medium amplitude. It is also to be mentioned that the optical absorption of the product extracted with dichloromethane from the KBr pellet was at about 310 nm, therefore rather close to the absorption of pure 4-nitrodiphenyl.

3. Results and discussion

Two kinds of experiments were found to be of interest for establishing the reaction products and azide decomposition kinetics: (i) irradiation of oxygen-free of relatively concentrated azide solutions and (ii) irradiation of oxygen containing and diluted azide solutions. Hereafter, the initial concentration of azide will be noted a_0.

The UV absorption spectra recorded after various irradiation times show a new absorption band at longer wavelengths than that of azide (Fig. 1). Another characteristic of

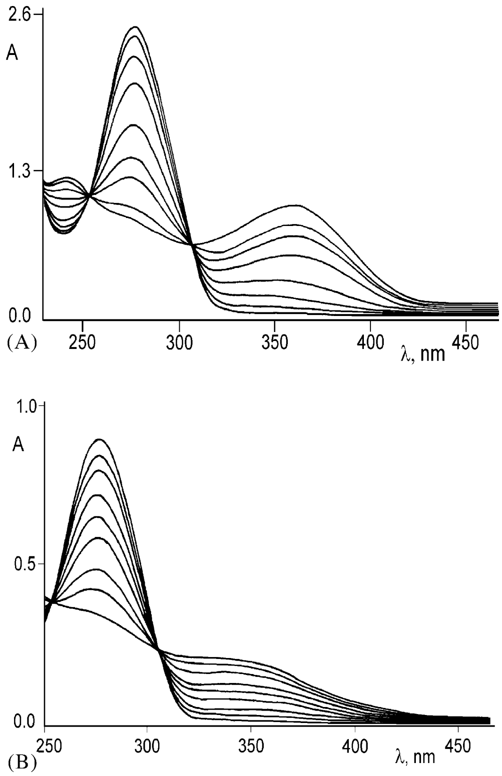


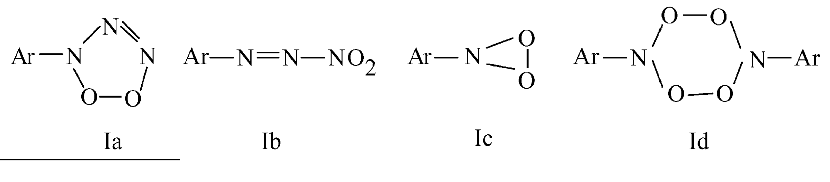
Fig. 1. Changes in the electronic absorption spectra of dichloromethane solutions of 4-azidodiphenyl during irradiation with 280 nm light; (A)—de-aerated solution, 0.1 mm cell, irradiation times: 0, 15, 30, 60, 120, 180, 240, 360 s; (B)—oxygen containing solution, 10 mm cell, irradiation times: 0, 15, 30, 60, 90, 120, 180, 240, 300 s.

the spectra is the presence of two isosbestic points placed on both sides of the azide band and the absence of any other crossing of spectra. This means that the reaction is uniform [22] for both kinds of samples and that the steps are elementary reactions.

3.1. *Products resulted from azide photolysis*

3.1.1. *Case of oxygen-free and concentrated azide media ($a_0 > 10^{-3}$ mol/l)*

The absorption band appearing at about 360 nm (Fig. 1A) is due to the formation of *trans*-4,4′-azodiphenyl ($\lambda_{max} = 362$ nm). This assignment is based on the rather good similitude between the IR and ^{1}H-NMR spectra of the global photolysis product and of the synthesised *trans*-4,4′-azodiphenyl. In addition, the following observation led to the conclusion that the azodiphenyl product is only in *trans* form: by irradiation at 365 nm, its absorption decreased in intensity concurrently with the formation of the *cis*-form absorption band at 260 nm. This band disappeared when the solution was warmed in dark at 35 °C for 4–5 h and the absorption at 360 nm returned to its initial intensity.



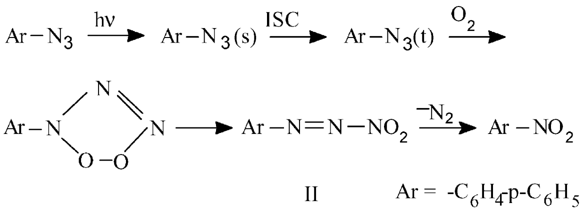
(6)

Taking into account the low radiation intensity used in these experiments, we consider that azodiphenyl results mainly from reaction 2.

3.1.2. *Case of oxygen containing and diluted azide media ($a_0 < 10^{-4}$ mol/l)*

The absorption band appearing has a maximum at 335–345 nm and is an overlapping of several bands because its width remains unusually large even at high azide conversions (Fig. 1B). Certainly, this absorption band mainly arises from oxygen containing products because its intensity diminishes with the degree of de-aeration.

According to the observation presented in the experimental section, it can be said that the primary product which results from the reaction of excited azide molecules or nitrenes and oxygen shows an optical absorption maximum at about 342 nm and turns into 4-nitrodiphenyl at a rate that is much lower in the reaction medium than when the product is isolated. The following chemical structures are proposed in literature for such intermediates: an oxygen-azide adduct with dioxatriazoline ring (Ia), nitroazoarene (Ib), dioxazirane (Ic) and its dimer (Id) (Scheme 6). The first two were suggested by Abramovich and Challand [23] and introduced by Boyer [3] as possible intermediates for nitroarene formation during photooxygenation of aryl azides (reaction 7). The other two were proposed by Brinen and Singh [24] on the base of ESR spectra observed for the products of *p*-diazidobenzene photolysis in aerated media at 77 K. Taking into account the chemical stability and the position of the optical absorption maximum, we consider that the product in discussion is probably 4-nitroazodiphenyl (II, reaction 7). Its stability could be explained by the resonance structures similar those of β-nitrostyrene [25]. Also, this resonance stabilisation would be the reason we failed to evidence any effect of *trans–cis* photoisomerization, the absorption band in discussion remaining unchanged after irradiation at 330 nm.



(7)

A charge transfer complex, $C_6H_5C_6H_4N^+HR\ldots O_2^-$, [3] of oxygen and the amines resulting from the nitrene–solvent reaction (Eq. (4)) should be considered. Its formation would be favoured by the fact that such amines (λ_{max} = 275–285 nm), if present, can be in their excited state during the irradiation of azide. Nevertheless, we suppose that such complexes are not present in the reaction mixture as main components. An argument for this assumption is the observation made when 4-aminodiphenyl was irradiated in dichloromethane in the presence of oxygen, namely the appearance of a broad absorption band with maximum at 350–360 nm and the formation of a precipitate the absorption of which in methanol is at 250 nm. No trace of precipitate was observed in the case of azide photolysis. In addition, the kinetics of azide decomposition (Section 3.2) does not indicate amine formation as a significant reaction, at least at low and medium azide conversions. The amine formation was found to be practically exclusive when 4-azidodiphenyl is irradiated in acetic acid.

3.2. Kinetics of light-induced chemical reactions of azide

For the UFO photochemical reaction 8, where the reactant R is the only UV absorber, the local rate equation is evidently $\mathrm{d}a/\mathrm{d}t = -k\Phi \times a$, with $a = [R]$. In this case the Zimmerman equation takes the form 9, where l is the optical path length and $\bar{a} = \bar{a}(t)$ is the spatially averaged reactant concentration resulted by integrating the momentary local concentration a (x, t) over the depth x between 0 and l. I_0 is the incident radiation intensity which is considered as constant during irradiation.

$$R + h\nu \overset{k_\Phi}{\rightarrow} P \tag{8}$$

$$\frac{\mathrm{d}\bar{a}}{\mathrm{d}t} = \Phi \times \frac{I_0[1 - \exp(-\varepsilon \times \bar{a} \times l \times \ln 10)]}{l} \tag{9}$$

The solution of Eq. (9) can be written under the form 10 where $A_0 = \varepsilon \times a_0 \times l$ and $A = \varepsilon \times \bar{a} \times l$. The Eq. (10) takes the approximate forms 11 and 12 for $A_0 \ll 1$ and $A_0 > 2$, respectively.

$$\log\left[\frac{(10^{A_0} - 1)}{(10^{A} - 1)}\right] = \Phi \times \varepsilon \times I_0 \times t \tag{10}$$

$$\log\left(\frac{A_0}{A}\right) \cong \Phi \times \varepsilon \times I_0 \times t \quad \text{when } A_0 \ll 1 \tag{11}$$

$$A_0 - A \cong \Phi \times \varepsilon \times I_0 \times t \quad \text{when } A_0 > 2 \tag{12}$$

Eqs. (10)–(12) lead to simple methods of quantum yield determination from measurements of absorbance carried out during irradiation.

3.2.1. Kinetics of azide photodecomposition

The experimental values (A, t) obtained for azide diluted and oxygen containing solutions are plotted according to Eq. (10) in Fig. 2A. One observed that, with the exception of the portion of high conversion, the points are rather well placed on a straight line, regardless of A_0 (and a_0). This means that, for low and medium conversions, (i) the photodecomposition of azide is an UFO reaction, and (ii) the absorption at 276 nm corresponds indeed to unreacted azide. So, it can be said that reaction 7 is the predominant one in the considered samples and, therefore, oxygen is an efficient quencher of the excited molecules of azide. As a consequence, the generation of nitrene species (at least by the triplet azide) becomes too low for the reactions 2–4 to be significant.

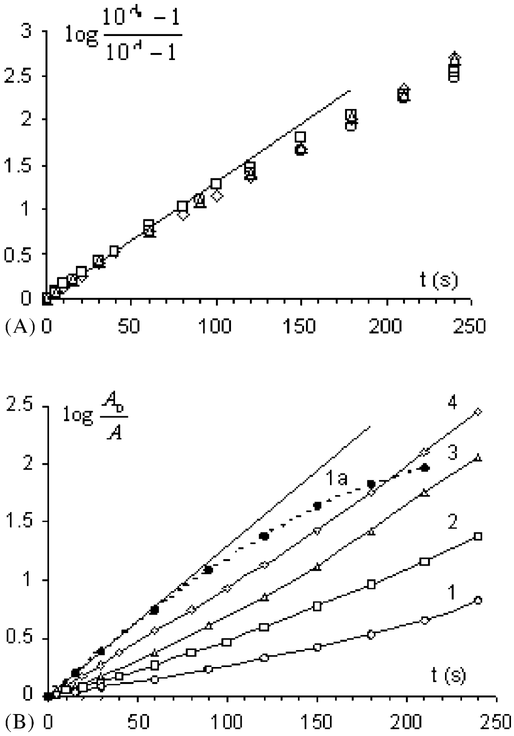


Fig. 2. Plots using (A) Eq. (10) and (B) Eq. (11) for oxygen-saturated and azide diluted solutions irradiated in cells of 10 mm thickness. The values of A_0, a_0 (mol/l) and azide conversion (%) at 120 and 210 s were: 1 (2.63, 9.7×10^{-5}, 53, 78), 2 (1.80, 6.7×10^{-5}, 74, 93), 3 (0.90, 3.3×10^{-5}, 85, 98) and 4 (0.44, 1.6×10^{-5}, 93, 99). Additional plots: straight lines with $0.013\,\mathrm{s}^{-1}$ slopes, and the plot 1a (B) where the data used for the curve 1 were processed with Eq. (12).

However, there is a visible deviation from linearity at high azide conversion. We explain this deviation by amines that are produced by the nitrene–solvent reaction (Eq. (4)). By their absorption band at 275–285 nm, these amines lead both to an overestimation of the azide absorbance and to a non-observance of the validity condition for Eq. (9), namely that the reactant (azide) to be the only radiation absorbent.

The value of $0.013\,\mathrm{s}^{-1}$ for the initial slope of the plot 2A leads to $\Phi = 0.24$, that is in agreement with values reported for aromatic azides [26].

The plots 1–4 in Fig. 2B show the error of quantum yield determination, which is caused by the non-observance of the condition $A_0 \ll 1$imposed by the application of Eq. (11). On the other hand, the plot 1a illustrates that the simple Eq. (12)

can be successfully used for optically dense samples at low azide conversions.

3.2.2. *Azide mainly consumed by formation of azoproduct*

As presented in Section 3.1.1., the prevalent product is azodiphenyl when azide-concentrated and oxygen-free solutions are irradiated. In this case, the excited azide molecules decompose in nitrene intermediates that react only with azide molecules. A necessary condition is that the reaction 2 should be predominant in comparison with the reactions 3–5. The nitrene recombination is negligible because $k_{\text{na}} \approx k_{\text{nn}} \approx 10^9\,\text{l}\,\text{mol}^{-1}\,\text{s}^{-1}$ [4d] and $n \ll a$ (see experimental section) where n is the momentary concentration of nitrene. Since it was not possible to do predictions about the condition $k_{\text{na}} \times a \gg k_{\text{ns}}$, we used dichloromethane as solvent because it is considered relatively inert toward nitrene species [2].

The rate of azide consumption results by subtracting the rate of reactions 1 and 2, namely $\mathrm{d}a/\mathrm{d}t = -k_{\Phi} \times a - k_{\text{na}} \times a \times n$ and $\mathrm{d}n/\mathrm{d}t = k_{\Phi} \times a - k_{\text{na}} \times a \times n$, and assuming $a \gg n$. The obtained equation $\mathrm{d}a/\mathrm{d}t \cong -2k_{\Phi} \times a$ is similar to that of the UFO photochemical reaction 8 with the difference that the quantum yield is apparently two times greater. Of course, the corresponding Zimmerman equation will have the solution 13.

$$\log\left[\frac{(10^{A_0}-1)}{(10^{A}-1)}\right] = 2\Phi \times \varepsilon \times I_0 \times t \qquad (13)$$

The doubling of Φ is normal because one molecule of azide is decomposed by Φ^{-1} photons and the appearing nitrene will decompose another molecule due to reaction 2.

The experimental values (A, t) are plotted using Eq. (13) in Fig. 3. This time the points do not follow a straight line and are placed on curves dependent on the concentration a_0. However, the curves become a straight line with a slope of $2\Phi\varepsilon I_0 \ln 10$ when a_0 increases and this fact supports the theory developed previously. The deviation from the equation $\mathrm{d}a/\mathrm{d}t \cong 2k_{\Phi} \times a$ is most probable due to reaction 4, the irradiated samples being still diluted to correspond to the condition $k_{\text{na}} \times a \gg k_{\text{ns}}$. Unfortunately, it was not possible to do measurements with more concentrated solutions ($a_0 > 3 \times 10^{-2}\,\text{mol}\,\text{l}^{-1}$) because such solutions show tendency of precipitation for rather low conversion of azide.

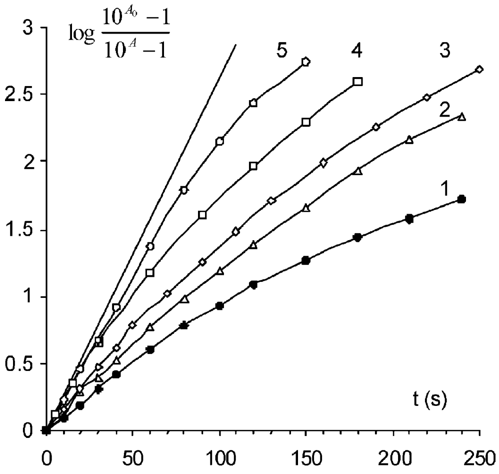


Fig. 3. Plots using Eq. (13) for oxygen-free solutions containing 4-azidodiphenyl of various concentrations irradiated in cells of different thickness in order to have about the same absorbance A_0. The values of A_0, a_0 (mol/l), l (mm) and azide conversion (%) at the end point were: 1 (2.6, 9.6×10^{-5}, 10, 75), 2 (2.7, 4.9×10^{-4}, 2, 81), 3 (2.5, 9.2×10^{-4}, 1, 93), 4 (2.58, 8.7×10^{-3}, 0.1, 87) and 5 (2.42, 3×10^{-2}, 0.03, 93). A straight line with slope two times larger than those in Fig. 2A is drawn for reference.

4. Conclusions

It was found that 4-azidodiphenyl is predominantly consumed by a proper photodecomposition when oxygen-containing diluted azide solutions in dichloromethane are irradiated. The predominant reaction in this case seems to be the trapping of triplet azide molecules by oxygen, a quite probable structure of the adduct being 4-nitroazodiphenyl. On the other hand, in the absence of oxygen, the nitrene–azide reaction consumes the nitrene intermediates producing 4,4′-azodiphenyl the more so as the azide concentration is greater.

In both cases, the light-induced decomposition of azide can be approximately treated as a unidirectional first order reaction when the reactant is the only UV absorbent.

The deviation from such kinetics is mainly caused by the nitrene–solvent reaction. The same value results for the quantum yield of proper azide photodecomposition in the two cases.

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