

Photoaddition of benzil to electron-rich olefins

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ABSTRACT

Benzil I was found to add photochemically to ethyl vinyl ether III, α -methylstyrene IV, furan V and 2,3-dimethyl-2-butene VI to give the corresponding oxetanes as the major photoproducts, (in the case of 2,3-dimethyl-2-butene, dioxene was also formed in equal amounts), whereas with furfural II, the photoproduct was found to be the corresponding keto ester.

INTRODUCTION

Oxetane formation from electronically excited carbonyl compounds and olefins is an elegant and flexible method and belongs to the most extensively studied photo-reactions (Yang 1964). The importance of oxetanes stems from the fact that they are used as intermediates in the synthesis of natural products (Koch *et al.* 1983; Carless & Fekaruruhobo 1984), commercially interesting polymers (Farthing 1955) and for their pharmacological properties (Searles, 1964).

The photochemical addition of olefins to 1,2-dicarbonyl compounds leading to the formation of ketooxetanes was discovered by Krauch (1965). Other products such as dioxenes, allylic ethers, and alcohols are also noticed (Schonberg 1968; Rubin, 1985; Monroe 1972). There are several publications describing the photochemicals reactions of 1,2-dicarbonyl compounds with olefins (Hara *et al.* 1967; Shima *et al.* 1974, 1977; Gream *et al.* 1970). Although benzil was one of the earliest 1,2-diketone investigated, its photoreactions with olefins have not been studied as much as those of biacetyl (Jones *et al.* 1980; Turro *et al.* 1980; Ryang *et al.* 1973).

Photoexcited benzil is known to give oxetanes with visnagin (Krauch 1965) and 4,5-dimethyl-1,3-dioxole (Gersdorf *et al.* 1987). But with other olefins such as stilbene, 1,1-diphenylethylene (Schonberg & Latif 1945) and 1,1-diphenyl-2-methylethylene (Shuping *et al.* 1987), benzil was reported to produce dioxene derivatives. However, R. J. C. Koster later reported that the product from the photoreaction in benzil with 1,1-diphenylethylene was actually an oxetane (Koster *et al.* 1974).

These observations indicate that unlike other non-cisoid 1,2-dicarbonyl compounds which give mainly oxetanes and allylic products (Shima *et al.* 1974; Ryang *et al.* 1973), benzil gives dioxenes also, like o-quinones (Schonberg 1968) and non-enolizable cyclic 1,2-diketones (Gream *et al.* 1970).

طريقة جديدة للتحليل الأولي المساعد وأقل تحقيق لمصفوفة دالة النقل

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خلاصة

يقدم هذا البحث خوارزم حسابي بسيط لحساب وصف كسر المصفوفة الأولي، وأقل عرض لحالة الفضاء الخاصة بالنظام الخطي متعدد المتغيرات، والذي يتم تعريفه بمصفوفة دالة النقل. وقد تم بناء الخوارزم على أساس نظرية تم إستنتاجها في هذه المقالة، وهي تميز حساب درجة أقل تحقيق وكذلك فهارس المراقبة (تدرج الأعمدة) مباشرة من معاملات مصفوفة دالة النقل المعطاة. وقد تم مراجعة ومقارنة الخوارزمات المعروفة لأقل تحقيق والتحليل الأولي بالطريقة المقترحة. وتم وضع مثال مفصل لعرض كفاءة الطريقة المقترحة.

In this contribution, we would like to describe further results of the photochemical reactions of benzil with other various electron-rich olefins (e.g. furfural, ethyl vinyl ether, α -methylstyrene, furan and 2,3-dimethyl-2-butene), and discuss whether or not such reactions might shed further support on the previous reported observations about benzil. We have observed that the photo-labilities of these olefins in the presence of benzil vary appreciably and we now report the details of these results.

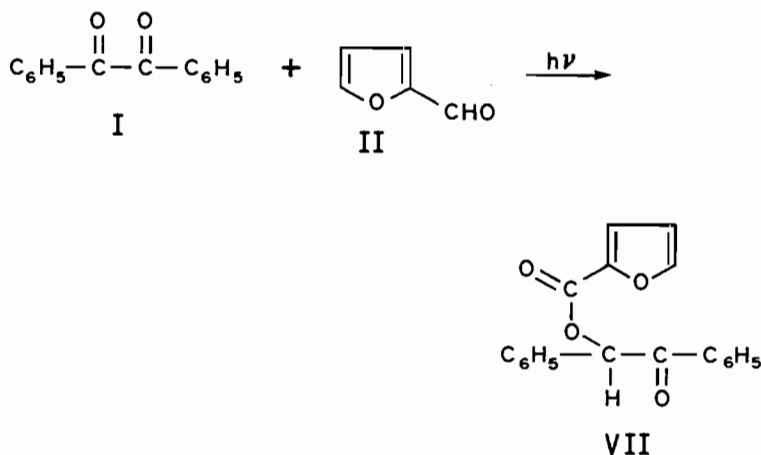
RESULTS AND DISCUSSION

Irradiation of benzil I and furfural II have produced only one major photoproduct which was isolated pure by flash chromatography. The ir spectrum of the photoproduct showed two absorption bands at 1685 and 1716 cm^{-1} (C=O, ester), characteristic of two different carbonyl groups. The ^1H nmr spectrum showed a

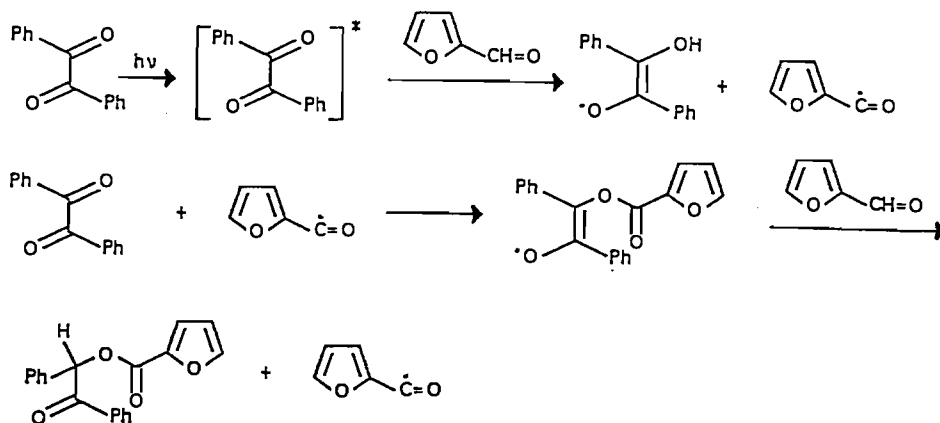
singlet at δ 6.0 ppm $\left(1\text{H}, \begin{array}{c} \text{O} \quad \text{H} \\ \parallel \quad | \\ -\text{C}-\text{C}-\text{O} \\ | \\ \text{Ph} \end{array} \right)$, two doublets at δ 7.10 and 7.35 (2 H ethylenic protons of furan) and a multiplet at δ 7.10–7.35 ppm (11 H, aromatic plus one ethylenic proton). These data are in total agreement with our assignment of the keto ester VII (Scheme 1) for the photoproduct.

The formation of the keto ester VII could be accounted for by a free radical mechanism similar to the one previously reported (Moore & Waters 1953) for the photoaddition of aromatic aldehydes to o-quinones, (Scheme 2).

Irradiation of benzil I and ethyl vinyl ether III produced two photoproducts (Scheme 3) in a 2:1 ratio (GC). The ^1H nmr spectrum of the major photoproduct showed a triplet of δ 5.30 ppm (1H, $-\text{O}-\text{CH}-\text{O}-$) and a multiplet at δ 3.30–3.70 ppm (2 H, oxetane ring), together with an ethyl and aromatic protons. Nuclear overhouse effect (NOE) measurements were not possible with our instrument. However, the assignment of the oxetane structure VIII for the major photoproduct, was supported



Scheme 1. Photoproduct of benzil I and furfural II.

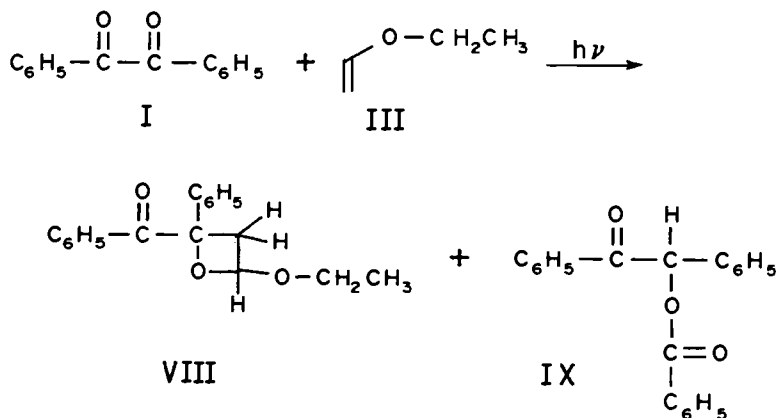


Scheme 2. Mechanism for the photochemical formation of the keto ester VII.

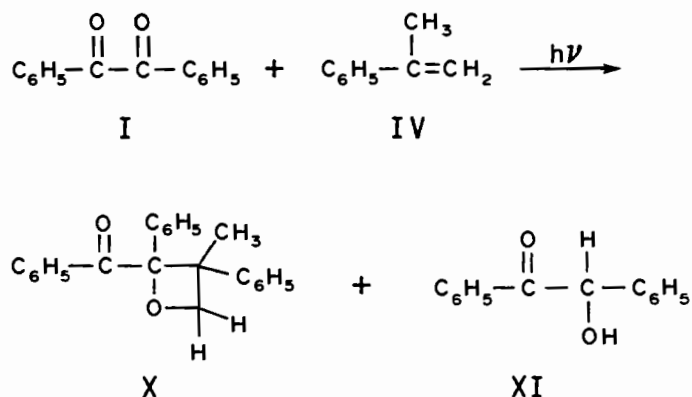
by the fact that the proton appearing at δ 5.30 ppm (triplet), could only be attributed to the ($-\text{O}-\text{CH}-\text{O}-$) proton, due to the strong deshielding effect of the two oxygens. The other possible regioisomer would necessary produce that triplet at a much higher field. Similar oxetanes reported by Ryang *et al.* (1973) and Mattay & Buchkremer (1988) give further support to this assignment. The ^1H nmr spectrum of the minor photoproduct, on the other hand, showed a singlet at δ 7.10 ppm (1 H, aliphatic) and a multiplet at δ 7.20–8.20 ppm (15 H, aromatic). These data are in full agreement with the known compounds, benzoin benzoate IX (Bunbury & Chuang 1969).

Formation of benzoin benzoate IX has only been shown to occur when benzil I is irradiated in proton donating solvents (Monroe 1971; Ogata *et al.* 1972 and Bunbury & Wang 1968). Hence, ethyl vinyl ether must have also acted as the hydrogen atom donating solvent in this system.

Irradiation of benzil I and α -methylstyrene IV gave two photoproducts (Scheme 4) in a 3 : 1 ratio (GC), which were separated pure by flash chromatography. The ^1H nmr spectrum of the major product showed two doublets at δ 4.60 and 4.80 ppm (oxetane ring) along with a methyl and aromatic protons. The ir spectrum indicated the



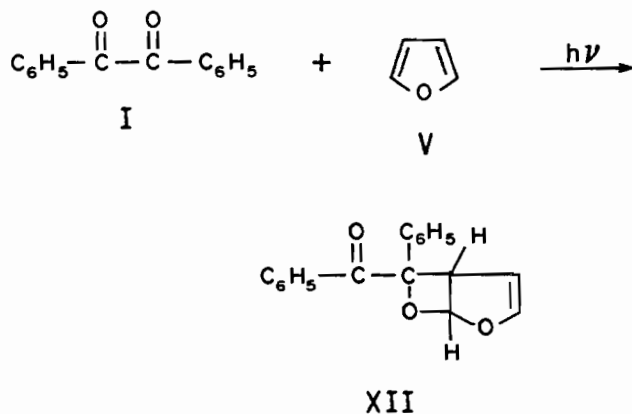
Scheme 3. Photoproducts of irradiation of benzil I and ethyl vinyl III.



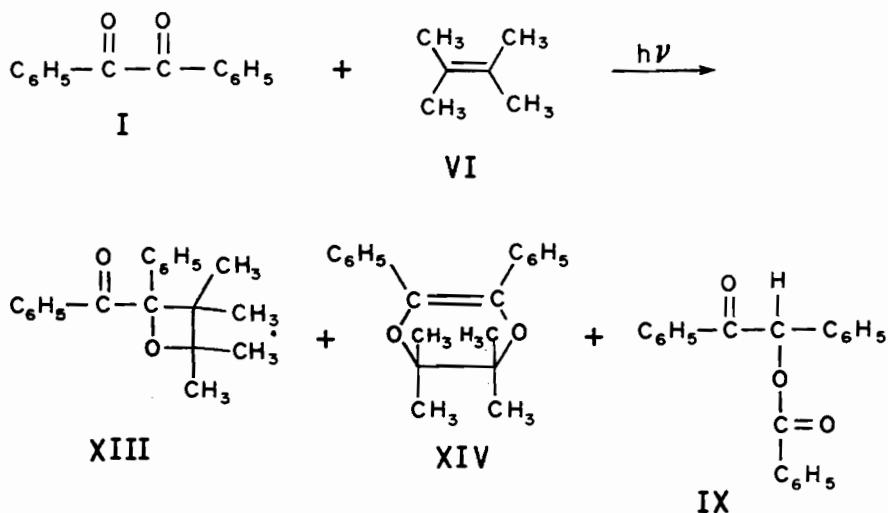
Scheme 4. Photoproducts of irradiation of benzil I and α -methylstyrene IV.

presence of a conjugated carbonyl group at 1685cm^{-1} . From the spectroscopic properties of this adduct (see Experimental), and from the comparison with similar oxetane compounds (Ryang *et al.* 1973), the photoproduct was assigned the oxetane structure X. Here again, the possibility of formation of the other regioisomer was excluded on the basis of the appearance of the oxetane ring protons at δ 4.60 and 4.80 ppm, due to the strong deshielding effect of this nearby oxygen atom. The ^1H nmr spectrum and the melting point of the minor product, on the other hand, were identical to those of authentic benzoin XI. Similarly, the formation of benzoin could be justified in this reaction if we consider that α -methylstyrene was also acting as a hydrogen atom donating species.

Irradiation of benzil I and furan V have also produced only one major photoproduct (Scheme 5). The ^1H nmr spectrum of this adduct showed four different multiplets at δ 4.90, δ 5.80, δ 6.15 and δ 6.50 ppm along with aromatic protons at δ 7.2–8.0 ppm. The ir spectrum again indicated the presence of a conjugated carbonyl group at 1685cm^{-1} . From the spectroscopic properties of this adduct (see Experimental) and from the comparison with those of similar oxetanes (Ryang *et al.* 1973), the photoproduct was assigned the oxetane structure XII. The other possible regioisomer was completely excluded due to the fact that it would necessarily show



Scheme 5. Photoproduct of irradiation of benzil I and furan V.



Scheme 6. Photoproduct of irradiation of benzil I and 2,3-dimethyl-2-butene VI.

the two oxetane ring protons in the region between (δ 4.50–5.00 ppm), because both of them would have been adjacent to only one oxygen atom.

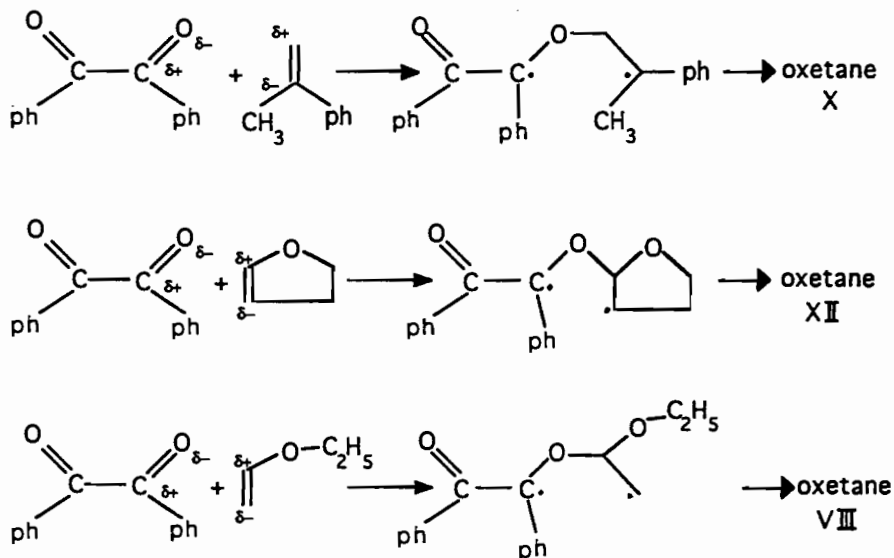
The irradiation of benzil I and 2,3-dimethyl-2-butene VI, on the other hand, produced three photoproducts (Scheme 6) in a 2 : 2 : 1 ratio (GC). The ir spectrum of the first major showed a carbonyl group at 1685 cm^{-1} . The ^1H nmr of the same compound showed two singlets at δ 1.20 and δ 14.0 ppm for the four methyl protons and a multiplet centered at δ 7.40 ppm for the aromatic protons. These data are in full agreement with the oxetane structure XIII.

The second major product showed no carbonyl stretching in the ir spectrum, but an absorption at 1640 cm^{-1} indicating the presence of $>\text{C}=\text{C}<$. The ^1H nmr spectrum showed a singlet at δ 1.35 ppm and a multiplet centered at δ 7.1 ppm (aromatic). These data suggest that the other major product was the dioxene XIV. The minor product, on the other hand, was identified as benzoin benzoate IX.

The regiochemistry observed with oxetanes VIII, X and XII is totally consistent with the preferential formation of the most stable 1,4 diradical with the carbonyl carbon adding to the more electron-rich end of the ethene (Scheme 7). It is striking that benzoin benzoate is formed only in the irradiations with ethyl vinyl ether and tetramethylethene, whereas benzoin is only formed in the case of α -methylstyrene. At this point, we do not clearly understand the reason behind these observations. No benzoin or benzoin benzoate is formed in the case of furan, which is understandable because furan does not contain an easily abstractable hydrogen atom.

In all of these cases, the formation of allylic ethers or allylic alcohols was not observed. A similar result was reported from the photoreactions of 1-phenyl-1,2-propanedione with olefins (Shima *et al.* 1977). To summarize, the photochemical reactions of benzil with electron-rich olefins yield oxetanes as the major product with the exception of 2,3-dimethyl-2-butene, where, in addition, to the oxetane the formation of dioxene was also observed. The photochemical reaction of benzil with furfural yielded only the corresponding keto ester.

Irradiation of benzil I in the presence of electron deficient olefins such as



Scheme 7. Possible explanation of the observed regioselectivity of the oxetane formation.

acrylonitrile and methacrylonitrile were also carried out in a similar manner, however, no photoproducts were observed. These results: a) further support the previously reported observations about the photochemical reactions of benzil with olefins (Schonberg & Mustafa 1945; Shaping *et al.* 1987), and b) suggest that the excited benzil, like biacetyl, is electrophilic in its reaction with olefins.

EXPERIMENTAL

INSTRUMENTATION

An annular model APQ40 (Applied Photophysics Limited) photoreactor fitted with a 400 Watt medium pressure mercury arc-lamp was used for the irradiations. The NMR spectra were obtained with a Bruker AM-300 MHz spectrometer. The chemical shifts are given as ppm (δ) and tetramethylsilane ($\delta = 0$) was used as the internal standard. Infrared and ultraviolet spectra were determined with a Perkin-Elmer 580 B IR Spectrometer and a Shimadzu UV-visible recording spectrophotometer (UV 160), respectively. Mass spectra were recorded on a Ribermag R10-10C (NERMAG). Gas chromatographic (GC) analyses were performed with a Varian 3300 instrument fitted with a flame ionization detector and a 8 m DB1-15 W (OV 17) bonded phase capillary column. Melting points were taken on an electrothermal melting point apparatus. Microanalysis were done by the Malissa Microanalytical Laboratory, West Germany.

MATERIALS

Benzil, furfural, ethylvinylether, α -methylstyrene, furan and 2,3-dimethyl-2-butene were all from Aldrich. Acetonitrile (Analar) was from BDH and was used without further purification. Ethyl acetate was purified by distillation before use. Flash chromatography was performed according to Still *et al.* (1978), using silica gel (E. Merck No. 9385, 40-60 μ m) and eluted with 5:1 petroleum ether (40-60°C).

E. Merck TLC sheets (No. 60, silica gel) were used to monitor the chromatographic separation.

The solutions were degassed by bubbling nitrogen for 1 hr before irradiation.

BENZIL—FURFURAL SYSTEM

Irradiation of benzil (2.5 g, 0.012 mol) and furfural (6.96 g, 0.072 mol) in acetonitrile (30 ml) for 24 hours gave 0.4 g of the keto ester VII (m.p. 125–7°C) photoproduct. The 1 : 1 adduct VII was isolated pure (GC, TLC one spot) by flash chromatography using a 5 : 1 (v/v) mixture of ethyl acetate and petroleum ether as eluting solvent. Elem. anal. found % (calcd. for $C_{19}H_{14}O_4$): C, 74.42 (74.50); H, 4.68 (4.61); IR: 1716 and 1685 cm^{-1} ; M^+ = 306 m.u.; 1H NMR ($CDCl_3$) δ : 7.95 (1 H, d, $J = 6$ Hz); 7.40 (1 H, d, $J = 4$ Hz); 7.10–7.35 (11 H, m); and 6.0 ppm (1 H, s).

BENZIL—ETHYL VINYL ETHER SYSTEM

Irradiation of benzil (2.5 g, 0.012 mol) and ethyl vinyl ether (4.52 g, 0.063 mol) in acetonitrile (30 ml) for 24 hours gave two photoproducts in a 2 : 1 ratio (GC). The major photoproduct was separated pure (GC, TLC one spot) by flash chromatography using a 3 : 1 (v/v) mixture of ethyl acetate and petroleum ether as eluting solvent, and was identified to be the oxetene VIII (0.50 g, m.p. = 111–3°C). Elem. anal. found % (calcd. for $C_{18}H_{18}O_3$): C, 76.49 (76.57); H, 6.48 (6.42); IR: 1685 cm^{-1} ; M^+ = 282 m.u.; 1H NMR (C_2D_6O), δ : 7.2–8.2 (10 H, m); 5.30 (1 H, t, $J = 3$ Hz); 4.15 (2 H, q); 3.3–3.7 (2 H, m); and 1.4 ppm (3 H, t, $J = 7$ Hz).

The minor photoproduct benzoin benzoate IX (0.30 g, m.p. = 123–5°C, The literature value = 125°C (Bunbury and Wang 1968)) was also isolated pure by flash chromatography (GC, TLC one spot) using the same column. Elem. anal. found % (calcd. for $C_{21}H_{16}O_3$): C, 79.68 (79.73); H, 5.15 (5.10); IR: 1715 and 1695 cm^{-1} ; M^+ = 316 m.u.; 1H NMR ($CDCl_3$), δ : 7.2–8.2 ppm (15 H, m) and 7.10 ppm (1 H, s).

BENZIL— α -METHYLSTYRENE SYSTEM

Irradiation of benzil (2.5 g, 0.012 mol) and α -methyl styrene (5.45 g, 0.046 mol) in acetonitrile (30 ml) for 24 hours gave two photoproducts in a 3 : 1 ratio (GC). The major photoproduct oxetane X, was obtained as an oil (0.50 g) and was isolated pure by flash chromatography (GC, TLC one spot) using a 5 : 1 (v/v) mixture of ethyl acetate and petroleum ether as eluting solvent. Elem. anal. found % (calcd. for $C_{23}H_{20}O_2$): C, 84.02 (84.11); H, 6.22 (6.14); IR: 1685 cm^{-1} ; M^+ = 328 m.u.; 1H NMR ($CDCl_3$), δ : 7.0–8.0 (10 H, m); 4.80 (1 H, d, $J = 5$ Hz); 4.60 (1H, d, $J = 5$ Hz) and 1.30 ppm (3 H, s).

The minor photoproduct benzoin XI (0.15 g, yellow crystals, m.p. = 133–5°C, The literature value = 135°C C^{25}) was also isolated pure by flash chromatography (GC, TLC one spot) using the same column. Elem. anal. found % (calcd. for $C_{14}H_{12}O_2$): C, 79.18 (79.22); H, 5.66 (5.70); IR: 3440 and 1680 cm^{-1} ; M^+ = 212 m.u. 1H NMR ($CDCl_3$): δ 7.2–8.2 (10 H, m) and 6.0 (1 H, d, $J = 2.5$ Hz) and 4.50 ppm (1 H, d, $J = 2.5$ Hz, OH group).

BENZIL—FURAN SYSTEM

Irradiation of benzil (2.5 g, 0.012 mol) and furan (5.62 g, 0.082 mol) in acetonitrile (30 ml) for 24 hours gave 0.4 g of oxetane XII (which was obtained as an oil). Oxetane

XII was isolated pure (GC, TLC one spot) by flash chromatography using a 4 : 1 (v/v) mixture of ethyl acetate and petroleum ether as eluting solvent. Elem. anal. found % (calcd. for $C_{18}H_{14}O_3$): C, 77.61 (77.68); H, 5.13 (5.07); IR: 1685 cm^{-1} ; $M^+ = 278\text{ m.u.}$; $^1\text{H NMR}$ (CDCl_3), δ : 7.0–8.2 (10 H, m); 6.50 (1 H, d, $J = 4\text{ Hz}$); 6.15 (1 H, m); 5.8 (1 H, d, $J = 3\text{ Hz}$) and 4.90 ppm (1 H, m).

BENZIL—2,3-DIMETHYL-2-BUTENE SYSTEM

Irradiation of benzil (2.50 g, 0.012 mol) and 2,3-dimethyl-2-butene (4.25 g, 0.050 mol) in acetonitrile (30 ml) for 24 hours gave three photoproducts in a 2 : 2 : 1 ratio (GC). The first major photoproduct oxetane XIII (which was obtained as an oil) was isolated pure by flash chromatography (GC, TLC one spot) using a 5 : 1 (v/v) mixture of ethyl acetate and petroleum ether as eluting solvent. Elem. anal. found % (calcd. for $C_{20}H_{22}O_2$): C, 81.52 (81.60); H, 7.56 (7.53); IR: 1685 cm^{-1} ; $M^+ = 294\text{ m.u.}$; $^1\text{H NMR}$ (CDCl_3), δ : 7.0–8.2 (10 H, m); 1.4 (6 H, d) and 1.20 ppm (6 H, s).

The second major product dioxene XIV (0.60 g, m.p. 54–56°C) was also isolated pure by flash chromatography from the same column (GC, TLC one spot). Elem. anal. found % (calcd. for $C_{20}H_{22}O_2$): C, 81.48 (81.60); H, 7.60 (7.53); IR 1640 cm^{-1} (C=C); $M^+ = 294\text{ m.u.}$; $^1\text{H NMR}$ (CDCl_3), δ : 7.1–7.3 (10 H, m) and 1.35 ppm (12 H, s).

The third and minor photoproduct benzoin benzoate IX (0.3 g, m.p. 123–125°C) was also separated by flash chromatography from same column. Elem. anal. found % (calcd. for $C_{21}H_{16}O_3$): C, 79.78 (79.73); H, 5.17 (5.10); IR, 1715 and 1695 cm^{-1} ; $M^+ = 316\text{ m.u.}$; $^1\text{H NMR}$ (CDCl_3), δ : 7.20–8.20 (15 H, m) and 7.10 ppm (1 H, s).

ACKNOWLEDGEMENT

This work is a part of Research Project SC 045 supported by the Research Management Unit at Kuwait University.

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(Received 20 October 1993, Revised 4 October 1994)

تفاعلات الاضافة الضوئية بين مركب البنزل والأولوفينات الغنية

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خلاصة

يتفاعل مركب البنزل I تفاعلا ضوئيا مع كل من المركبات التالية:
الايثيل فينيل اثير III والالقاميشيل ستيرين IV والفيوران V و ٢، ٣- ثنائي ميثيل -٢- بيوتين
VI ليعطي مركبات الاوكسي تانز المقابلة (في حالة مركب ٢، ٣- ثنائي ميثيل -٢- بيوتين وجد أن
مركب الدايكسين موجود ايضا وبكمية متساوية)، وفي حالة تفاعل مركب البنزل مع الفيرفيورل II
وجد ان الناتج هو مركب الكيتو أستر المقابل.