methylamine on the protonated substrate is rate-determining and the second-order rate constants are linear in the concentration of hydrogen ion. As a significant fraction of the imidate is converted into the conjugate acid, these rate constants will tend to become independent of this parameter; the dotted line in Figure 5 exhibits this behavior. The measured second-order rate constants clearly deviate from linearity in a fashion that cannot be explained on the basis of protonation of the substrate. This behavior is interpreted as a transition to rate-determining decomposition of the tetrahedral intermediate. This behavior has been thoroughly treated both qualitatively and quantitatively[6] and need not be reexplained here. Experiments at more acidic conditions would strongly buttress the conclusion of a change in rate-determining step since the deviations from calculated behavior would be magnified. Unfortunately, reliable data are difficult to obtain below pH 6, since the concomitant hydrolysis reaction becomes rapid thus masking the aminolysis.

Registry No.—*syn*-Phenol N-methylacetimidate, 13758-81-1; *anti*-phenyl N-methylacetimidate, 13758-82-2.

Photocyclization of Methyl-*o*-benzyloxyphenylglyoxylate

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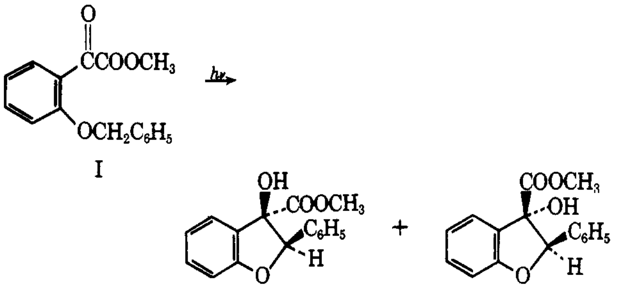
Methyl-*o*-benzyloxyphenylglyoxylate undergoes light-induced, intramolecular cyclization to afford an isomeric mixture of 2-phenyl-3-carboxymethyl-3-hydroxyl-2,3-dihydrobenzofurans in high yield. The stereochemistry of photocyclization is significantly dependent on both solvent and temperature, one of the isomers being formed almost exclusively in nonpolar solvents at low temperature. The results of photosensitization and quenching experiments indicate that reaction occurs predominately *via* the triplet state, suggesting that the stereochemical fate of the triplet is both solvent and temperature dependent. Some implications of the observed stereospecificity are discussed.

Recently, we reported that *o*-benzyloxybenzaldehyde undergoes light-induced cyclization to afford *cis*-2-phenyl-3-hydroxyl-2,3-dihydrobenzofuran and a closely related compound, possibly the corresponding *trans* isomer.[1] Further work has demonstrated that the major product resulting from intramolecular cyclization is the *cis* isomer.[2] While the over-all extent of intramolecular reaction was found to be modest, the resulting stereoselectivity was intriguing particularly since the major product was the less stable isomer. Clearly, studies on the stereospecificity of photocyclization of this system are of interest from both mechanistic and synthetic points of view.

Comprehensive studies on the photochemistry of *o*-benzyloxybenzaldehyde are encumbered, however, primarily because of the resulting complex product mixture, and we have been investigating closely related *o*-alkoxyaromatic carbonyl compounds, as well, with the hope of uncovering a more efficient system. To this end, we were gratified to find that methyl-*o*-benzyloxyphenylglyoxylate (I) undergoes intramolecular photocyclization cleanly to afford isomeric products in greater than 90% yield. Herein we report the effects of solvent and temperature as well as of photosensitization and quenching on the stereochemistry of photocyclization of I.[3]

Results

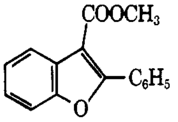
On solution irradiation, methyl-*o*-benzyloxyphenylglyoxylate (I) is transformed into an isomeric mixture of 2-phenyl-3-hydroxyl-3-carboxymethyl-2,3-dihydrobenzofurans (IIa and IIb). The isomers, separated by careful chromatography on neutral alumina, may be quantitatively dehydrated into 2-phenyl-3-carboxymethylbenzofuran (III).[4]



I

IIa

IIb



III

The relative positions of the carboxymethyl hydrogens in the nmr spectra[5] of the isomers provide compelling evidence for the stereochemical assignments. In addition to aromatic hydrogen resonances, the isomers exhibit two sharp singlets (area ratio 3:1) for the carboxymethyl and benzylic hydrogens, respectively.[6]

[1] S. P. Pappas and J. E. Blackwell, Jr., *Tetrahedron Letters*, 1171 (1966).

[2] Gas chromatography of the product mixture derived from *o*-benzyloxybenzaldehyde after irradiation in acetonitrile at 0° indicated that two major products with similar retention times were produced in the ratio of 2:1 to the extent of about 30%. It was easily demonstrated that the major product was *cis*-2-phenyl-3-hydroxyl-2,3-dihydrobenzofuran by chromatography of the mixture with added pure *cis* material.

[3] Kinetic studies are in progress.

[4] J. N. Chatterjea, *J. Indian Chem. Soc.*, **33**, 175 (1956).

[5] The nmr spectra were obtained in deuteriochloroform solution on a Varian A-60 spectrometer. All resonances are reported in parts per million using TMS as an internal reference.

[6] The isomer IIb exhibits hydroxyl hydrogen resonance, as well, which disappears on the addition of deuterium oxide; in contrast, such resonance has not been observed in the nmr spectrum of either IIa or mixtures of IIa and IIb.

Table I

Solvent, Temperature, Quenching, and Sensitization Results

Expt[a]	Solvent (temp, °C)	Quencher (concn, M)	Sensitizer (concn, M)	IIa:IIb	Extent of reacn[b]
1	CH_3CN (−35)			2.5:1	
2	CH_3CN (0)			2.0:1	
3	CH_3CN (0)	Piperylene (0.1)[d]		3.0:1	10
4	CH_3CN (80)			1.5:1	
5	Heptane (0)			>20:1	
6	Heptane (0)	Piperylene (0.1)[d]		>20:1	20
7	Heptane (100)			3:1	
8	Benzene[c] (15)			>20:1	
9	Benzene[c] (15)		Triphenylene (0.05)[d]	>20:1	100
10	*t*-Butyl alcohol (80)			1:1	

[a] The concentration of I was 4.0×10^{-3} M in all of the experiments except 8 and 9 where it was 2.0×10^{-3} M. Photocyclization was complete after 20 to 60 min in all of the unquenched reactions. [b] Per cent completion in the time required for complete reaction in the absence of added quencher or sensitizer. These values represent qualitative estimates from infrared spectra. [c] In view of the light utilized (>290 mμ) in these experiments, it is not expected that excited states of benzene will participate efficiently. Photosensitization experiments were conducted in benzene because of the insolubility of triphenylene in heptane. [d] It is assumed that energy transfer resulting in sensitization or quenching of the excited singlet state of I will be unimportant at the concentrations utilized in these reactions (see ref 7c).

In IIa the carboxymethyl and benzylic hydrogens appear at 3.73 and 5.75 ppm, respectively; the corresponding resonances in IIb are at 3.03 and 5.54 ppm. The unusually high-field position of the carboxymethyl hydrogens of IIb is postulated to arise from anisotropic shielding by the adjacent *cis*-phenyl ring. This assignment is further corroborated by the finding that elution from alumina of IIb, possessing the less hindered hydroxyl group, requires a solvent mixture of higher polarity than does IIa.

Many attempts to crystallize IIa and IIb have, as yet, been unsuccessful. However, both isomers provide crystalline derivatives in good yield (IIa 3,5-dinitrobenzoate, mp 168–169°, carboxymethyl hydrogens at 3.82 ppm; IIb *p*-nitrobenzoate, mp 153° dec, carboxymethyl hydrogens at 3.13 ppm).

Distillation of the crude photoproduct, derived from I, provides 90–95% distillate, the nmr spectrum of which is a simple composite of the two isomers. By integration of nmr spectra, we have determined that the ratio of isomeric photoproducts is significantly dependent on both solvent and temperature. The results are presented in Table I. In view of the striking dependence of stereospecificity on solvent polarity (compare, for example, expt 2 with 5 and 8), sensitization and quenching experiments were carried out to determine whether photocyclization proceeds *via* different multiplicities of the excited state in the polar and nonpolar media. The pertinent results appear in Table I, as well.[7]

Discussion

The photochemistry of α-keto esters has been investigated by several groups.[8] It is of interest that the reported phototransformations of phenylglyoxylate esters did not occur to any significant extent in our study. In particular, formation of the isomeric dihydrobenzofurans from I, presumably by intramolecular hydrogen abstraction *via* a seven-membered ring,[9] proceeded to the apparent exclusion of either decarbonylation[8b] or intramolecular hydrogen abstraction from the alkoxyl portion of the ester *via* a six-membered ring.[8b] Of obvious interest, in this regard, would be the photochemical behavior of methyl-*o*-methoxyphenylglyoxylate and benzyl-*o*-benzyloxyphenylglyoxylate.

The stereospecificity of photocyclization of I is both solvent and temperature dependent as evidenced by the photoproduct distribution (IIa:IIb) resulting from reaction at 0° in both acetonitrile (2:1) and heptane (20:1), and in refluxing heptane (3:1). In both solvents isomer IIa is the kinetically favored product. In refluxing *t*-butyl alcohol the reaction is completely nonstereospecific affording the isomers in equal amount.

The possibility that reaction occurs *via* different multiplicities of the excited state in the polar and nonpolar systems was considered. This possibility, at least with regard to the major reaction course, was clearly excluded, however, from the results of triplet-state photosensitization and quenching which appear in Table I. It is seen that photoreaction is largely inhibited in both acetonitrile and heptane when conducted in the presence of 25 M equiv of piperylene. Whether the minor amounts of photoproducts produced in these reactions arise *via* the excited singlet state or the triplet, as a result of incomplete quenching, has not been determined, although the sensitization results, *vide infra*, favor the latter course. This aspect of the reaction is under investigation.[3] In agreement with the extent of quenching, photosensitization by triphenylene, which absorbed more than 90% of the utilized light, resulted in no significant change in either the rate or stereospecificity of photocyclization relative to the unsensitized reaction. The results[7] indicate that photocyclization in both polar and nonpolar solvents occurs predominately *via* the triplet

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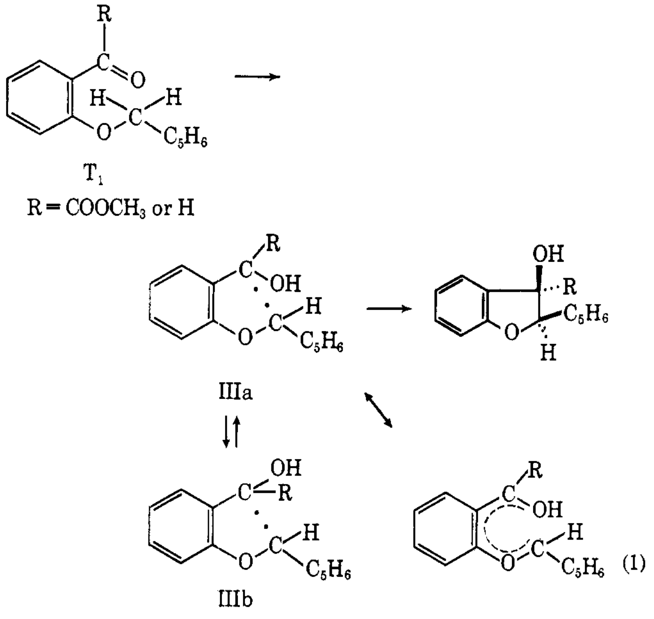
(7) (a) The use of piperylene as a triplet-state quencher is well documented; for example, see P. J. Wagner and G. S. Hammond, *J. Am. Chem. Soc.*, **88**, 1245 (1966). (b) The use of triphenylene as a photosensitizer has been recommended, W. G. Herkstroeter, A. A. Lamola, and G. S. Hammond, *ibid.*, **86**, 4537 (1964). (c) For a discussion of photosensitization, relevant to the concentrations of I utilized in these experiments, see H. E. Zimmerman, R. G. Lewis, J. J. McCullough, A. Padwa, S. W. Staley, and M. Semmelhack, *ibid.*, **88**, 1965 (1966).

(8) For example, see (a) G. S. Hammond, P. A. Leermakers, and N. J. Turro, *ibid.*, **83**, 2395 (1961); (b) P. A. Leermakers, P. C. Warren, and G. F. Vesley, *ibid.*, **86**, 1768 (1964); (c) P. A. Leermakers, M. E. Ross, G. F. Vesley, and P. C. Warren, *J. Org. Chem.*, **30**, 914 (1965); (d) E. S. Huyser and D. C. Neckers, *ibid.*, **29**, 276 (1964); (e) N. C. Yang and A. Morduchowitz, *ibid.*, **29**, 1654 (1964).

(9) D. J. Coyle, R. V. Peterson, and J. Heicklen, *J. Am. Chem. Soc.*, **86**, 3850 (1964).

state.[10] Since the stereospecificity of photocyclization varies significantly in the polar and nonpolar media, it follows that the stereochemical fate of the triplet is solvent dependent.[11]

A very high degree of stereospecificity is observed in the almost exclusive formation of IIa when photocyclization of I is conducted in nonpolar solvents at low temperatures. Photocyclization of *o*-benzyloxybenzaldehyde affords, predominantly, the isomer in which the phenyl and hydroxyl groups are *cis*, as well. This is of particular interest since the favored isomer is the more stable in the former case, the less stable in the latter. The results suggest that the same mechanism for stereochemical control is operating in both systems. Consideration of possible stereospecfic routes leads us to suggest the one depicted in eq 1.



Intramolecular hydrogen abstraction by the triplet, T_1, is assumed to occur *via* the rotational conformer in which the phenyl ring is farthest removed from the reaction site. This assumption is clearly supported by inspection of molecular models. Hydrogen abstraction followed by intersystem crossing is envisioned to give rise to the singlet biradical species IIIa. Assuming that IIIa maintains its geometric integrity, possibly because of double-bond character resulting from electron overlap, the favored isomers would result from a process of conrotatory cyclization. This mode of cyclization follows from the favored geometry for hydrogen abstraction and the stereochemistry of the products, rather than from theoretical considerations. The mode of cyclization is of theoretical interest, however, with regard to whether the process follows the Woodward–Hoffmann rules for electrocyclic reaction of a $4n + 2\ \pi$ electron system.[12] Of related interest is the electronic state (excited or ground)[13] of the biradical species IIIa. Lacking this information, however, conrotatory cyclization of IIIa, regardless of state, appears to be a reasonable postulate for steric reasons. The scheme (eq 1) depicts stereochemical control as arising from a favored geometry for hydrogen abstraction, nonrotational equilibration of the biradical intermediates, and conrotatory cyclization. Although speculative, these conclusions satisfactorily account for the available results. Rotational equilibration, for example, would be expected to favor IIIa for R = $COOCH_3$ and IIIb for R = H, thereby requiring different modes of cyclization for the two systems. Other means of stereochemical control are conceivable, however, and the scheme is presented, primarily, as a working hypothesis for the direction of further studies.

The marked dimunition in the stereospecificity of photocyclization of I which results from a change in solvent from heptane to acetonitrile is as remarkable as the high degree of stereoselectivity observed in the nonpolar media. Unfortunately, neither the available information on solvent effects in photocyclizations nor the results of this study, to date, allow us to suggest an attractive explanation for this effect. Clearly, further studies are necessary. Possibly, kinetic and stereochemical consequences of deuterium[14] substitution for the benzylic hydrogens, as well as the introduction of *para* substituents on the benzyl ring, may provide information on this and other aspects of the photocyclization. This work is in progress.

Finally, the high yields of cyclized products obtained in this study make the process potentially attractive for synthetic applications.[15] It seems reasonable that indole derivatives, for example, may be readily prepared by this method.

Experimental Section[16]

Methyl-*o*-benzyloxyphenylglyoxylate (I).—Methyl-*o*-benzyloxymandelate[17] was prepared from *o*-benzyloxybenzaldehyde by a known procedure[18] and, subsequently, was oxidized to I by the Jones reagent, as previously applied.[19] The resulting crude oil crystallized on trituration with ether. Several recrystallizations from ether–hexane resulted in a good yield of pure I: mp 84–85°; infrared spectrum (chloroform), 5.73 and 5.95 μ (ester and ketone carbonyl bands, respectively); ultraviolet spectra, $\lambda_{max}^{methanol}$ 322 mμ (ϵ 3.8 × 10^3), 258 (7.6 × 10^3), and $\lambda_{max}^{heptane}$ 370 mμ (70), 355 (140), 312 (4.1 × 10^3), 251 (8.4 × 10^3); nmr spectrum, δ 3.35 (carboxymethyl hydrogens), 5.05 (benzylic hydrogens).

Anal. Calcd for $C_{16}H_{14}O_4$: C, 71.1; H, 5.2. Found: C, 71.0; H, 5.2.

Irradiation Experiments.—The solution to be irradiated was added to a Pyrex cylindrical vessel, equipped with nitrogen inlet.

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(10) Evidence has been presented that the triplet state is predominantly responsible for (a) light-induced decarbonylation of ethyl pyruvate;[8a] (b) photocyclization of 2-hexanone, D. R. Coulson and N. C. Yang, *J. Am. Chem. Soc.*, **88**, 4511 (1966); (c) photoenolization of *o*-benzylbenzophenone, E. Zwicker, L. I. Grossweiner, and N. C. Yang, *ibid.*, **85**, 2671 (1963); and (d) photocyclization of α-diketones, W. H. Urry, D. J. Trecker, and D. A. Winey, *Tetrahedron Letters*, 609 (1962).

(11) In this regard, see P. J. Wagner, C. A. Stout, S. Sarles, Jr., and G. S. Hammond, *J. Am. Chem. Soc.*, **88**, 1242 (1966), in which evidence is presented that solvent polarity influences the fate of the excited singlet state of tetramethyloxetanone. In particular, a biradical intermediate is postulated to partition itself between two paths with solvent dependent, relative rates.

(12) R. B. Woodward and R. Hoffmann, *ibid.*, **87**, 395 (1965). See also H. C. Longuet-Higgens and E. W. Abrahamson, *ibid.*, **87**, 2045 (1965), and H. E. Zimmerman, *ibid.*, **88**, 1564, 1566 (1966).

(13) For a discussion on the intermediacy of vibrationally excited ground-state species in photochemical reactions, see E. F. Ullman and W. A. Henderson, Jr., *ibid.*, **88**, 4942 (1966).

(14) For the interesting effects of deuterium substitution on the photochemistry of 2-hexanone, see ref 10b.

(15) For an alternative photochemical route to benzofurans, see J. C. Sheehan and R. M. Wilson, *J. Am. Chem. Soc.*, **86**, 5277 (1964).

(16) All reagents and solvents utilized were reagent grade, recrystallized or distilled. Melting points are corrected. Elemental analyses were carried out by Spang, Ann Arbor, Mich.

(17) P. B. Russell and S. J. Childress, U. S. Patent 3,048,633 (1962); *Chem. Abstr.*, **57**, P16488*g* (1962).

(18) A. F. Wagner, U. S. Patent 2,892,847 (1959); *Chem. Abstr.*, **54**, P570*e* (1960).

(19) C. Djerassi, R. R. Engle, and A. Bowers, *J. Org. Chem.*, **21**, 1547 (1956).

serum capped opening for sample removal, and water-cooled condenser. A Vycor immersion well-condenser combination (available from Hanovia), in which were placed a Pyrex filter sleeve and light source (a high-pressure Hanovia 450-w, type L lamp), was placed into the solution through which nitrogen was passed for about 30 min before and also during the irradiation. Low temperatures were achieved by pumping ice-cooled water or Dry Ice cooled methanol through the immersion condenser. Solvents were maintained at reflux by alternately passing steam and water through the condenser. The reactions were monitored by examination of infrared spectra. The relative ratio of products was determined by nmr spectroscopy after distillation in a sublimation apparatus at 100° (0.05 mm) except in experiments with triphenylene. In these experiments, the bulk of triphenylene was removed by crystallization of the total product from benzene-heptane. The supernatants were concentrated, and nmr spectra were obtained following distillation, as above.

Separation of the Isomers of 2-Phenyl-3-hydroxyl-3-carboxymethyl-2,3-dihydrobenzofuran (IIa and IIb).—A 2:1 mixture (500 mg) of the isomers IIa and IIb, dissolved in a 1:1 benzene-hexane solution, was applied to a column wet packed with 25 g of neutral alumina (Woelm activity 1). The dimensions of the packed portion were 1.5 × 13.5 cm. Solvent mixtures employed consecutively were hexane-benzene, benzene, benzene-ether, ether, and ether-ethyl acetate. IIa (pure by nmr) was obtained on elution with an ether solution containing 1% ethyl acetate, and was followed by mixtures of IIa and IIb. After the amount of additional eluted material had become negligible, the polarity of eluting solvent was increased and IIb (pure by nmr) was obtained. The nmr spectrum of IIa exhibited carboxymethyl and benzylic hydrogen resonances at 3.73 and 5.75 ppm, respectively; for IIb the corresponding resonances appeared at 3.03 and 5.54 ppm. The isomers exhibited similar infrared and ultraviolet spectra: infrared (chloroform), 2.78 and 5.76 μ (hydroxyl and ester carbonyl bands, respectively); ultraviolet, λ_{max}^{ether} 288 (ϵ 3.3 × 10^3) and 280 mμ (ϵ 3.6 × 10^3).

2-Phenyl-3-carboxymethylbenzofuran (III).—To a solution of IIa, 70 mg, in 5 ml of ether were added thionyl chloride (0.1 ml) followed by pyridine (0.2 ml). After standing at room temperature for three days, the mixture was poured onto ice-water, which was subsequently extracted with ether. The ether layer was washed, in turn, with dilute aqueous solutions of sodium bicarbonate and hydrochloric acid, dried and concentrated. Crystalline product was obtained directly which, after recrystallization from hexane, provided III in near-quantitative yield: mp and mmp (with product obtained similarly from IIb) 80–81° (lit.[4] mp 80°); infrared spectrum (chloroform), 5.81 μ (ester carbonyl band); ultraviolet spectrum, $\lambda_{max}^{methanol}$ 302 mμ (ϵ 1.6 × 10^4); nmr spectrum, δ 3.88 (carboxymethyl hydrogens).

Anal. calcd for $C_{16}H_{12}O_3$: C, 76.2; H, 4.8. Found: C, 76.1; H, 4.9.

cis-**2-Phenyl-3-hydroxyl-3-carboxymethyl-2,3-dihydrobenzofuran-3,5-Dinitrobenzoate (IIa 3,5-Dinitrobenzoate).**—Solid 3,5-dinitrobenzoyl chloride (one molar excess) was added to an ice-cold solution of IIa in pyridine. After the solution was allowed to warm to room temperature, ice-water was added and the mixture was extracted with benzene. The benzene layer was washed, in turn, with dilute aqueous solutions of sodium bicarbonate and hydrochloric acid, dried and concentrated to an oil which solidified on trituration with ether. Several recrystallizations from ether-hexane resulted in a good yield of pure product: mp 168–169°; nmr spectrum, δ 3.82 (carboxymethyl hydrogens), 6.04 (benzylic hydrogen), a doublet centered at 8.47 ppm, J = 2 cps (*ortho* aromatic hydrogens on 3,5-dinitrobenzoyl ring), a triplet centered at 8.90 ppm, J = 2 cps (*para* aromatic hydrogen on 3,5-dinitrobenzoyl ring).

Anal. Calcd for $C_{23}H_{16}N_2O_9$: C, 59.5; H, 3.5; N, 6.0. Found: C, 59.9; H, 3.6; N, 5.9.

trans-**2-Phenyl-3-hydroxyl-3-carboxymethyl-2,3-dihydrobenzofuran *p*-Nitrobenzoate (IIb *p*-Nitrobenzoate).**—Treatment of IIb with *p*-nitrobenzoyl chloride and work-up, as above, resulted in a good yield of this compound: mp 153° dec; nmr spectrum, δ 3.13 (carboxymethyl hydrogens), 5.95 (benzylic hyrogen), 8.07 (aromatic hydrogens on *p*-nitrobenzoyl ring).

Anal. Calcd for $C_{23}H_{17}NO_7$: C, 65.9; H, 4.1; N, 3.3. Found: C, 65.7; H, 4.2; N, 3.3.

Registry No.—I, 13448-92-5; IIa, 13448-93-6; IIa 3,5-dinitrobenzoate, 13448-94-7; IIb, 13448-95-8; IIb *p*-nitrobenzoate, 13448-96-9; III, 13448-97-0.

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Aziridines. XVI. Isomerization of Some 1-Aroylaziridines

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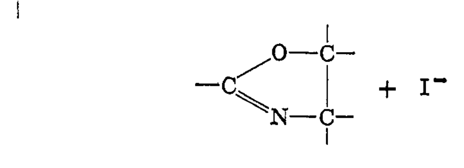
The iodide ion catalyzed isomerizations of 1-*p*-nitrobenzoyl-2-phenylaziridine and 1,3-diaroyl-2-arylaziridines are described. The thermolyses of *cis*- and *trans*-1-*p*-nitrobenzoyl-2,3-diphenylaziridines and 1,3-diaroyl-2-arylaziridines are also reported. The latter reaction represents a novel pyrolytic rearrangement of 1-aroylaziridines to α-benzamidobenzalacetophenones.

The isomerization of 1-acylaziridines into 2-aryl- or 2-alkyl-2-oxazolines by various nucleophiles has been investigated extensively in recent years.[1-9] The mechanism[10] proposed for the isomerization involves as a first step an attack by the nucleophile, such as iodide ion, on an aziridinyl carbon to form an N-β-iodoethylbenzamido ion. In a subsequent step the ion cyclizes to the oxazoline and regenerates the iodide ion (eq 1).



(1)

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[10] H. W. Heine, *Angew. Chem. Intern. Ed. Engl.*, **1**, 528 (1962).