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TETRAHEDRON

Regioselectivity in Photocyclization of 2-(*N*,*N*-Dialkylamino)- and 2-(*N*-Alkylanilino)ethyl Benzoylformates *via* Remote Proton Transfer

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Abstracts: Upon irradiation 2-(*N*,*N*-dialkylamino)- and 2-(*N*-alkylanilino)ethyl benzoylformates **1** underwent photocyclization *via* ζ-hydrogen transfer to ketone carbonyl oxygen from charge-transfer states to give seven-membered azalactones **2** and/or **2'**. The ζ-hydrogen transfer occurred regioselectively. Neither Type II products produced *via* γ-hydrogen trasnfer nor five-membered aminolactones produced *via* δ-hydrogen transfer could be detected.

INTRODUCTION

The Norrish Type II reaction of carbonyl compounds is one of the best known organic photoreactions, and in its initial step the mechanism involves direct γ-hydrogen abstraction by excited carbonyl oxygen.[1] Nitrogen-substituted ketones such as *N*,*N*-dimethylaminoacetophenone[25] also undergo the Type II reaction though in this case the reaction occurs by way of electron transfer from nitrogen to the excited carbonyl group followed by proton transfer.[3-6] Intramolecular hydrogen transfer irrespective of forms of hydrogen, an atom or proton, is very specific in that γ-hydrogen transfer greatly predominates over other possible modes.[1,6,7] The great facility of the γ-hydrogen transfer is due to the favorable stereoelectronic[5] and geometric requirements.[9] Remote hydrogen transfer *via* medium-sized cyclic transition states has been observed in some carbonyl compounds but is not a common event.[10,11] The charge-transfer interaction occurring in ketones containing an amino[12,13] or sulfide group[14,15] facilitates the remote hydrogen transfer. The regioselectivity for the remote hydrogen transfer is not yet well established. The hydrogen transfer from an external methylene group α to a hetero atom may be predominate over that from an internal α methylene group.[1c,12,15,16] The regioselectivity may meet the requirement for accessibility of the external α methylene hydrogens to the carbonyl group.[5,17] However, Hu and Neckers recently reported the regioselective photocyclization of 2-(*N*,*N*-dimethylamino)ethyl benzoylformate to a five-membered aminolactone **3c**

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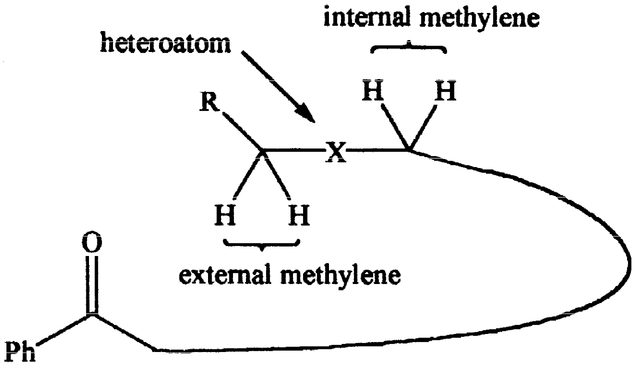
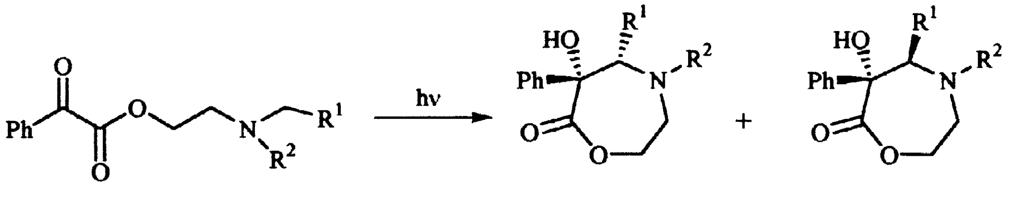
(Scheme 1) *via* δ-hydrogen transfer from its internal methylene group α to nitrogen.[18] They claimed that δ-proton transfer is preferred to possible ζ-proton transfer because of geometry of an intermediate amino cation radical. Their result seems to be quite different from ours obtained from remote photocyclization of β-[12,13,20] and γ-oxoesters[21-23] containing an ω-dialkylamino alkyl group. Our results provide examples of great predominance of external hydrogen transfer to ketone carbonyl oxygen. Then, we studied on regioselectivity in photoreactions of benzoylformates and found that the formates did not undergo photocyclization *via* δ-proton transfer but *via* ζ-proton transfer regioselectively to give seven-membered azalactones.



RESULTS AND DISCUSSION

Irradiation of 2-(*N*,*N*-dibenzylamino)ethyl benzoylformate (**1a**) in benzene under nitrogen with a 450 W high-pressure mercury lamp through a Pyrex filter gave seven-membered azalactones **2a** and **2a'** in 37 and



1 **2** **2'**

Table 1. Yields of the azalactones **2** and **2'**.

	R^1	R^2	Yield/%	
			2	**2'**
a	Ph	CH_2Ph	37	36
b	Ph	Ph	51	25
c	H	CH_3	44	
d	H	Ph	38	

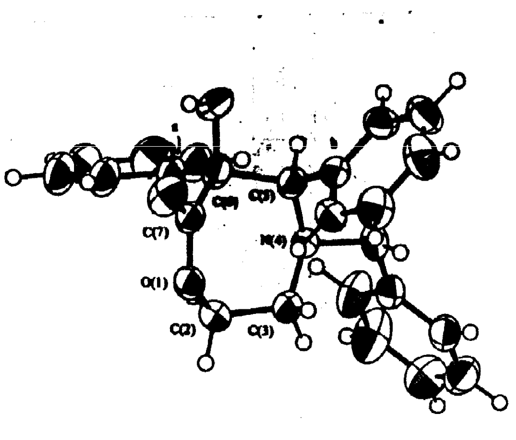


Figure 1. Crystal structure of **2a**.

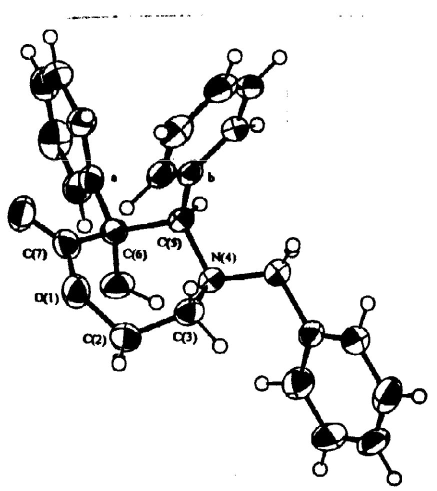


Figure 2. Crystal structure of **2a'**.

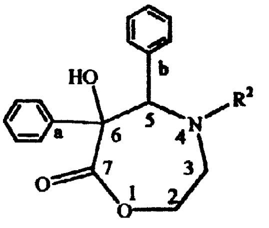


Table 2. Distances between Atoms in **2a**, **2a'** and **2b**.

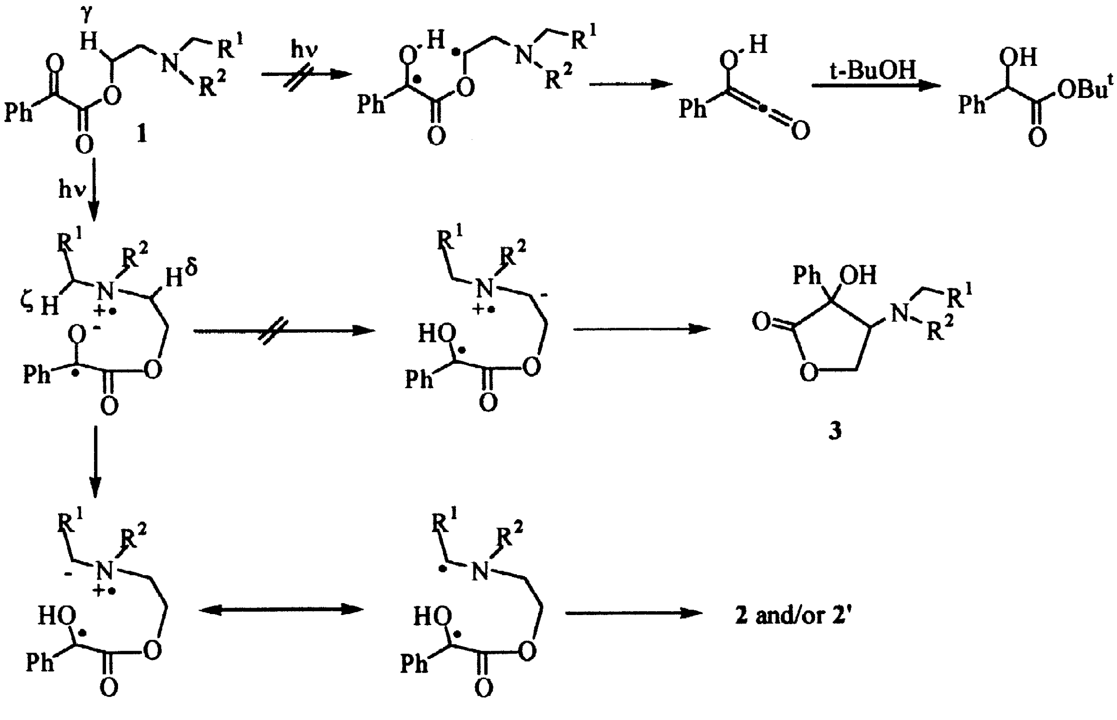
Atom	Atom	Distance / Å		
		2a	**2a'**	**2b**
1	4	3.193	3.189	3.213
2	7	2.453	2.471	2.439
3	7	3.148	3.142	3.137
4	6	2.551	2.477	2.568
4	7	3.235	3.250	3.260
5	7	2.584	2.622	2.579
a	b	3.988	3.275	3.943

36 % yield, respectively. The structures of **2a** and **2a'** were determined on the basis of spectral data, elemental analysis and the X-ray structure analysis. The IR spectra of **2a** and **2a'** showed characteristic absorptions of the carbonyl (both of **2a** and **2a'**: 1720 cm^{-1}) and hydroxyl groups (**2a**: 3460 and **2a'**: 3425 cm^{-1}). Their ^{1}H-NMR spectra showed singlets attributable to methine proton at δ 4.05 in **2a** and δ 3.89 in **2a'** and pairs of doublets attributable to N-benzyl protons at δ 3.16 and 3.81in **2a** and δ 3.29 and 3.70 in **2a'**. The ^{13}C-NMR spectra showed doublets attributable to methine carbon at δ 69.9 in **2a** and δ 74.4 in **2a'**. The results obtained from the X-ray structure analysis showed that **2a** and **2a'** have nearly the same chair-like seven-membered rings (Figure 1, 2 and Table 2). The distances between lactone carbonyl carbon and nitrogen in **2a** and **2a'** are 3.235 and 3.250 Å, respectively. This reflects the same frequency of the carbonyl

absorptions in their IR spectra. The dihedral angles of Ph-C(5)-C(6)-Ph in **2a** and **2a'** are 173.3 and 71.8°, respectively. Therefore, the phenyl groups locate to be *trans* to each other in **2a** and *cis* in **2a'**. Neither Type II photoproducts nor five-membered aminolactones could be detected. Alkyl benzoylformates have been reported to undergo selective Type II elimination to give ketenes, which can be trapped by alcohol.[24] Irradiation of **1a** was performed in a 1:1 mixture of benzene and *tert*-butanol. However, no products arising from ketenes could be detected. Therefore, we can safely conclude that the compound **1a**, which has γ-hydrogens, does not show the Type II photoreactivity.

The photoreaction of **1a** proceeded quite efficiently. The quantum yield for the disappearance of **1a** in benzene was determined to be unity. The photoreaction did not quenched with 2,5-dimethyl-2,4-hexadiene. These results and lack of the Type II photoreactivity of **1a** indicate that the rate of the charge-transfer interaction is much more faster than that of the direct γ-hydrogen abstraction by excited carbonyl oxygen. Upon irradiation 2-methoxyethyl-[18] and 3-benzyloxypropyl benzoylformate[19] undergo only the Type II reaction.

Scheme 1



Similarly, irradiation of 2-(*N*-benzylanilino)ethyl benzoylformate (**1b**) under the same conditions gave **2b** and **2b'** in 51 and 25 % yield, respectively. The structures of **2b** and **2b'** were determined on the basis of spectral data and elemental analysis. The IR spectra of these compounds showed the characteristic carbonyl (**2b**: 1730 and **2b'**: 1725 cm^{-1}) and hydroxyl absorptions (**2b**: 3460 and **2b'**: 3370 cm^{-1}). The X-ray structure analysis of **2b** showed that conformation of the seven-membered ring is chair-like (Figure 3) and the dihedral angle of Ph-(5)-C(6)-Ph is 171.8° indicating that these phenyl groups locate *trans* to each other. Although the X-ray structure analysis of **2b'** could not be achieved because of high viscous oil, **2b** should be the isomer having *cis*-configuration. Analysis using MOPAC was performed for **2b'** and the dihedral angle of Ph-(5)-C(6)-Ph was taken to be 88.7°.

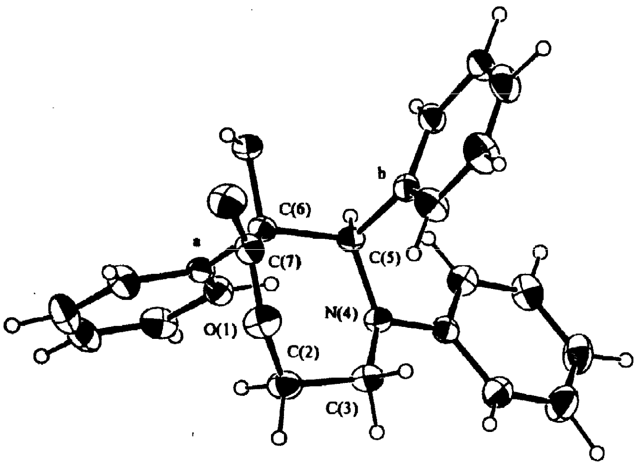


Figure 3. Crystal structure of **2b**.

These results are quite different from the result that Hu and Neckers obtained. They reported that 2-(*N,N*-dimethylamino)ethyl benzoylformate (**1c**) underwent photocyclization *via* regioselective δ-hydrogen transfer to give a five-membered aminolactone **3c**. The difference may be due to the substituents on nitrogen in the benzoylformates. Then, we re-examined the photoreaction of **1c**.

After irradiation of the 2-(*N,N*-dimethylamino)ethyl ester **1c** under the same conditions, a reaction mixture was concentrated and the residue was chromatographed on a silica-gel column. A product showing IR absorption peaks at 3450 and 1720 cm^{-1} was isolated. The ^{1}H-NMR spectrum of this compound showed a singlet attributable to N-methyl protons at δ 2.44 and an AB quartet attributable to C-5 methylene protons at δ 3.04, and the ^{13}C-NMR spectrum showed three triplets at δ 57.4, 61.7 and 68.5. These results indicate that this compound is not the five-membered aminolactone but the seven-membered azalactone **2c**. The yield of **2c** was 44 %. Hu and Neckers claimed that the aminolactone **3c** was obtained in 77 % yield by vacuum distillation from a reaction mixture of irradiating **1c** in benzene and assigned singlet peaks at δ 2.16 and 2.45 in its ^{1}H-NMR spectrum to two methyl groups on nitrogen.[18] The isolation of **3** by distillation appears doubtful because two singlets were observed at δ 2.18 and 2.44 in the ^{1}H-NMR spectrum of the reaction mixture from **1c** though the intensity of the former peak was one third of that of the latter. We attempted to isolate the product showing the peak at δ 2.18. However, it could not be isolated because of quite low mobility on silica gel.

Irradiation of the 2-(*N*-methylanilino)ethyl ester **1d** under the same conditions also gave the azalactone **2d** in 38 % yield. The yields of photoproducts from the *N*-methyl derivatives **1c** and **1d** are lower than those from the *N*-benzyl derivatives **1a** and **1b**. This indicates that the stability of radical centers in the biradical intermediate **4** is one of the important factors in photocyclization *via* remote hydrogen transfer.

In conclusion, the hydrogen transfer from an external methylene group α to a nitrogen atom is predominated over that from an internal α methylene group in the photoreaction of the benzoylformates **1**. This regioselectivity meets the requirement for accessibility of the external α methylene hydrogens to the carbonyl group.[5,17]

EXPERIMENTAL SECTION

General methods

The IR spectra were recorded with a JASCO IR Report-100 spectrometer, the ^{1}H and ^{13}C NMR spectra were measured with a JEOL JNM-LA-500 spectrometer using tetramethylsilane as an internal standard. An Ushio 450 W high-pressure mercury lamp was used as an irradiation source. GC analysis was performed with a Shimadzu GC-8A gas chromatograph. X-Ray data collection was carried out on a Mac Science MXC18 diffractometer and the computations were performed on SUN computer.

Synthesis of Benzoylformates. Benzoylformates (**1a-d**) were prepared by esterification of ethyl benzoylacetate and the corresponding aminoalcohols in the presence of dicyclohexylcarbodiimide.[18]

General Procedure for Preparative Photolysis of 1. A benzene solution of **1** (*ca.* 0.03 mol dm^{-1}) was irradiation under nitrogen with a 450 W high-pressure mercury lamp through a Pyrex filter. After removal of the solvent, the residue was chromatographed on a silica-gel column. Elution with a mixture of n-hexane-ethyl acetate for reaction mixtures from **1a-c** or n-hexane-acetone for a reaction mixture from **1d** gave **2** and/or **2'**.

***X-Ray Structure Determination of 2a, 2a' and / *** with a graphite crystal Mo Kα radiation (0.71073 Å) was used. Cell constants and an orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 22 reflection measured by the computer controlled diagonal slit method of centering. Intensity data with $2\theta \leqq 52.94°$ for **2a**, $2\theta \leqq 34.34°$ for **2a'** and $2\theta \leqq 34.57°$ for **2b** were collected with the ω-2θ scan technique (scan speed 5°/min) at 5337 reflections for **2a**, 5086 for **2a'** and 4710 for **2b**. As a check on crystal and electronic stability three representative reflections were measured every 100 reflections. The slope of the least-squares line through a plot of indensity versus time was -9 ± 1 counts/hour which corresponds to a total loss in intensity of 9.0 % for **2a**, 1.0 % for **2a'** and 6.0% for **2b**. Lorentz and polarization corrections were applied to the data. No absorption correction was made. An extinction correction was not necessary. The structures were solved using the direct methods program. The refinement was carried out by the full-matrix least-squares method. Scattering factors were taken from Cromer and Waber.[26] Anomalous dispersion effects were included in Fc[27]; the values for $\Delta f'$ and $\Delta f''$ were those of Cromer.[26] The final discrepancy factors were R =0.095 and R_w = 0.114 for **2a**, R = 0.062 and R_w = 0.076 for **2a'** and R = 0.055 and R_w = 0.084 for **2b**. The standard deviation of an observation of unit weight was 1.0. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.39 and −0.52 e/Å^3, respectively, for **2a**, 0.75 and −0.49 e/Å^3, respectively, for **2a** and 0.46 and −0.32 e/Å^3, respectively, for **2b**.[28] Plots of $\Sigma w(|Fo| - |Fc|)^2$ *versus* $|Fo|$, reflection order in data collection, sin θ/λ, and various classes of indices showed no unusual trends. All calculations were performed on a SUN computer.

Crystal Data of 2a. $C_{24}H_{23}O_3N$, M = 373.50. Monoclinic, a = 13.389(4), b = 14.794(5), c = 10.054(4) Å, β = 97.98(3)°, V = 1972.1(1) Å^3. Space group $P2_1/a$, Z = 4, D_x = 1.257 g/cm^3. Colorless prism. Crystal dimension: 0.50 x 0.40 x 0.30 mm^3, $\mu(MoH_\alpha)$ = 0.77 cm^{-1}. F(000) = 792 . T = 293K.

Crystal Data of 2a'. $C_{24}H_{23}O_3N$, M = 373.50. Monoclinic, a = 14.859(9), b = 16.114(7), c = 8.070(3) Å, β = 96.50(4)°, V = 1919.8(2) Å^3. Space group $P2_1/a$, Z = 4, D_x = 1.292 g/cm^3. Colorless prism. Crystal dimension: 0.80 x 0.30 x 0.10 mm^3, $\mu(MoH_\alpha)$ = 0.79 cm^{-1}. F(000) = 792 . T = 293K.

Crystal Data of 2b. $C_{23}H_{21}O_3N$, M = 360. Monoclinic, a = 10.12(1), b = 20.73(2), c = 9.189(4) Å, β = 108.17(5)°, V = 1832(2) Å^3. Space group $P2_1/a$, Z = 4, D_x = 1.305 g/cm^3. Colorless prism. Crystal dimension: 0.80 x 0.50 x 0.40 mm^3, $\mu(MoH_\alpha)$ = 0.80 cm^{-1}. F(000) = 760 . T = 293K.

Quantum Yield Determination. The benzoylformate **1a** (*ca.* 0.05 mol dm^{-3}) dissolved in benzene-d_6 containing 1% of toluene as a standard was placed in a test tube and. The tube was degassed, sealed and irradiated. Irradiation was performed on a "merry-go-round" apparatus with an Ushio 450-W high-pressure mercury lamp. The potassium chromate filter solution was used for isolation of the 313 nm line.[28] The amount of starting ester disappeared was determined by an NMR analysis of the irradiated mixture. Valerophenone was used as an actinometer.[29]

4-Benzyl-5,6-diphenyl-6-hydroxy-1-oxa-4-azacycloheptan-7-one **(2a)**. mp 148 - 149 °C (Found: C, 77.18; H, 6.11; N, 3.86. $C_{24}H_{23}O_3N$ requires C, 77.19; H, 6.21; N, 3.75%); ν_{max}(KBr)/cm^{-1} 3460 and 1720; δ_H 2.58 (1H, dd, *J* 15.0 and 4.0, NC*H*H), 3.16 (1H, d, *J* 13.7, C*H*HPh), 3.28 (1H, ddd, *J* 15.0, 13.15 and 2.3, NC*H*H), 3.81 (1H, d, *J* 13.7, C*H*HPh), 4.05 (1H, s, CHPh) 4.15 (1H, ddd, *J* 13.2, 4.0 and 2.3, OC*H*H), 4.22 (1H, dd, *J* 13.2 and 13.15, OC*H*H), 4.78 (1H, s, OH), 7.2 - 7.6 (15H, m, ArH); δ_C 47.8 (t), 60.2 (t), 69.9 (d), 70.1 (t), 81.1 (s), 127.0 (d, 2C), 127.5 (d), 128.2 (d), 128.4 (d, 2C), 128.5 (d, 2C), 128.8 (d, 2C), 128.9 (d, 2C), 129.0 (d, 2C), 130.4 (d), 134.1 (s), 137.9 (s), 138.7 (s), 176.9 (s).

4-Benzyl-5,6-diphenyl-6-hydroxy-1-oxa-4-azacycloheptan-7-one **(2a')**. mp 155 - 156 °C (Found: C, 77.02; H, 6.08; N, 3.75. $C_{24}H_{23}O_3N$ requires C, 77.19; H, 6.21; N, 3.75%); ν_{max}(KBr)/cm^{-1} 3425 and 1720; δ_H 2.69 (1H, dd, *J* 15.0 and 3.4, NC*H*H), 3.24 (1H, ddd, *J* 15.0, 11.9 and 1.8, NC*H*H), 3.29 (1H, d, *J* 13.4, C*H*HPh), 3.70 (1H, d, *J* 13.4, C*H*HPh), 3.89 (1H, s, CHPh) 4.37 (1H, ddd, *J* 12.2, 3.4 and 1.8, OC*H*H), 5.05 (1H, dd, *J* 12.2 and 11.9, OC*H*H), 6.41 (1H, s, OH), 7.1 - 7.5 (15H, m, ArH); δ_C 47.6 (t), 59.8 (t), 69.5 (t), 74.4 (d), 79.1 (s), 126.9 (d, 2C), 127.0 (d, 2C), 127.4 (d), 128.0 (d), 128.7 (d, 2C), 128.8 (d, 2C), 128.9 (d, 2C), 129.0 (d, 2C), 130.6 (s), 131.5 (d), 137.0 (s), 140.4 (s), 171.1 (s).

6-Hydroxy-4,5,6-triphenyl-1-oxa-4-azacycloheptan-7-one **(2b)**. mp 157 - 158 °C (Found: C, 76.93; H, 5.90; N, 3.88. $C_{23}H_{21}O_3N$ requires C, 76.86; H, 5.89; N, 3.90%); ν_{max}(KBr)/cm^{-1} 3460 and 1730; δ_H 3.37 (1H, dd, *J* 15.3 and 4.3, NC*H*H), 3.88 (1H, ddd, *J* 15.3, 10.4 and 2.0, NC*H*H), 4.17 (1H, s, CHPh), 4.30 (1H, ddd, *J* 13.1, 4.3 and 2.0, OC*H*H), 4.42 (1H, dd, *J* 13.1 and 10.4, OC*H*H) 5.71 (1H, s, OH), 6.9 - 7.6 (15H, m, ArH); δ_C 46.9 (t), 68.0 (d), 69.5 (t), 81.6 (s), 117.8 (d, 2C), 120.8 (d), 126.6 (d, 2C), 127.9 (d), 128.4 (d, 2C), 128.9 (d), 129.1 (d, 2C), 129.2 (d, 2C), 129.3 (d, 2C), 135.6 (s), 138.9 (s), 150.3 (s), 176.5 (s).

6-Hydroxy-4,5,6-triphenyl-1-oxa-4-azacycloheptan-7-one **(2b')**. viscous oil (Found: C, 76.74; H, 5.90; N, 3.89. $C_{24}H_{23}O_3N$ requires C, 76.86; H, 5.89; N, 3.90%); ν_{max}(neat)/cm^{-1} 3370 and 1725; δ_H 3.24 (1H, dd, *J* 14.6 and 3.4, NC*H*H), 3.80 (1H, ddd, *J* 14.6, 11.9 and 1.9, NC*H*H), 4.50 (1H, ddd, *J* 13.0, 3.4 and 1.9, OC*H*H), 4.54 (1H, s, CHPh), 5.17 (1H, dd, *J* 13.0 and 11.9, OC*H*H) 5.97 (1H, s, OH), 6.8 - 7.3 (15H, m, ArH); δ_C 47.0 (t), 69.3 (t), 77.3 (d), 79.1 (s), 122.0 (d, 2C), 123.9 (d), 127.0 (d, 2C), 127.2 (d, 2C), 127.6 (d), 128.4 (d, 2C), 128.7 (d), 129.2 (d, 2C), 130.5 (d, 2C), 130.8 (s), 140.1 (s), 150.7 (s), 171.3 (s).

6-Hydroxy-4-methyl-6-phenyl-1-oxa-4-azacycloheptan-7-one **(2c)**. viscous oil (Found: C, 65.16; H, 6.57; N, 6.12%. $C_{12}H_{15}O_3N$ requires C, 65.14; H, 6.83; N, 6.33%); ν_{max}(neat)/cm^{-1} 3450 and 1720; δ_H 2.44 (3H, s, CH_3), 2.61 (1H, ddd, *J* 1.3, 9.5 and 14.1, 5-H), 2.77 (1H, ddd, *J* 1.0, 5.2 and 14.1, 5-H), 3.04 (2H, ABq, *J* 13.4, 3-H), 4.01 (1H, ddd, *J* 1.0, 9.5 and 13.2, OC*H*H), 4.15 (1H, ddd, *J* 1.3, 5.2 and 13.2, OC*H*H), 4.53 (1H, s, OH), 7.3 - 7.5 (5H, m, ArH); δ_C 46.5 (q), 57.4 (t), 61.7 (t), 68.5 (t), 78.4(s), 126.4 (d, 2C), 128.6 (d), 128.8 (d, 2C), 138.8 (s), 176.4 (s).

6-Hydroxy-4,6-diphenyl-1-oxa-4-azacycloheptan-7-one **(2d)**. viscous oil (Found: C, 71,93; H, 6.02; N, 5.19%. $C_{17}H_{17}O_3N$ requires C, 72.07; H, 6.05; N, 4.94%); ν_{max}(neat)/cm^{-1} 3455 and 1720; δ_H 3.31 (1H, ddd, *J* 12.8, 2.2 and 2.5, 3-H), 3.60 (1H, ddd, *J* 12.8, 10.7 and 4.6, 3-H), 3.95 (2H, s, 5-H), 4.36 (1H, ddd, *J* 2.5, 10.7 and 13.4, OC*H*H), 4.39 (1H, s, OH), 4.41 (1H, ddd, *J* 2.2, 4.6 and 13.4, OC*H*H), 6.9 - 7.4 (10H, m, ArH); δ_C 50.2 (t), 59.1 (t), 66.9 (t), 82.4 (s), 116.2 (d, 2C), 120.4 (d), 125.7 (d, 2C), 128.9 (d), 129.1 (d, 2C), 129.3 (d, 2C), 139.8 (s), 149.9 (s), 175.8 (s).

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