Syntheses and Spectral Properties of β-Iodoureas and 2-Amino-4,4-diphenyl-2-oxazolines

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The syntheses of *N*-(*trans*-2-iodocyclohexyl)- **1-4**, *N*-(2-iodo-3,3-dimethylbutyl)- **5**, and *N*-(2-iodo-1,1-diphenylethyl)ureas **6**, **7** and the cyclization of **6** and **7** into 2-amino-2-oxazoline derivatives **8**, **9** are reported. The structures of prepared compounds are based on analytical and spectroscopic data.

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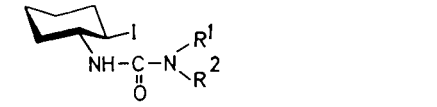
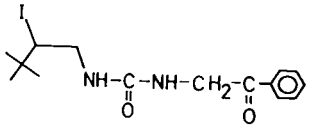
The β-iodoureas are useful intermediates in the syntheses of derivatives of imidazole and oxazole [1-5]. Hitherto, only a few β-iodoureas have been identified and we have recently described the preparation of β-iodourea derivatives of carbohydrates [6]. Now we have carried out a study on the synthesis of simple β-iodoureas from β-iodoisocyanates, and on their spectroscopic (uv, ir, pmr, cmr and ms) properties. We have used *trans*-2-iodocyclohexyl- [2], 2-iodo-3,3-dimethylbutyl- [3] and 2-iodo-1,1-diphenylethyl-[7] isocyanates, which cover isocyanates with an -NCO function bound to a secondary, primary or tertiary carbon atom respectively. Also ammonium hydroxide and a variety of amines were tried out. Thus, compounds **1-7** were obtained.



	1	2	3	4
R^1	H	Me	H	H
R^2	^nBu	Bn	CH_2COPh	$CH_2COC_6H_4CH_3$



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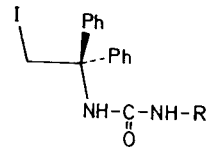
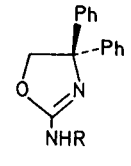


6 7

R H CH_2COPh



8 9

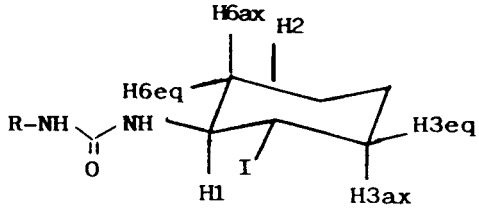
R H CH_2COPh

Formula

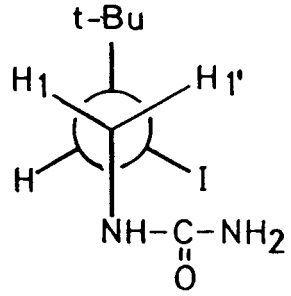
The β-iodoureas can undergo intramolecular S_N2 displacement of the iodine atom by the neighbouring urea group [4,6]. The resulting 2-amino-2-oxazolines are compounds with a variety of potential pharmaceutical applications such as central nervous system regulators, blood pressure regulators, antinociceptives, *etc.* [2,8-10]. We have studied this reactions on **6** and **7** to achieve the 2-amino-2-oxazoline **8** and **9** respectively.

Results and Discussion.

The *N*-(*trans*-2-iodocyclohexyl)-, *N*-2-iodo-3,3-dimethylbutyl)- and *N*-(2-iodo-1,1-diphenylethyl)ureas **1-7** have been prepared by reaction of the corresponding *vic*-iodoisocyanate with ammonium hydroxide, amines or phenacyl-amine hydrochlorides. The structures of **1-7** were assigned on the basis of analytical, uv, ir, pmr, cmr and ms data (see Experimental). The $^3J_{H,H}$ values let us assign a definite conformation for β-iodoureas **1-5**. Thus, in compounds **1-4**, $J_{1,2}$, $J_{1,6ax}$, and $J_{2,3ax}$ (9.6-10.9 Hz) were in the range for antiperiplanar protons in a chair conformer with the iodine atom and the urea group at equatorial position.



1-4



5 > 90 %

Figure 1

For the *N*-(2-iodo-3,3-dimethylbutyl)-*N'*-phenacylurea (**5**), $J_{1,2}$ (10.5 Hz), and $J_{1',2}$ (2.5 Hz) values showed that the major conformation in chloroformic solution was a staggered one with 2-H *anti* and *gauche* respectively to 1-H and 1'-H. The application of Altona's equation [11] to this system proved that the *tert*-butyl and urea groups were *anti* in the preferred conformation (Figure 1), although angles would be slightly distorted distancing the iodine atom from urea group.

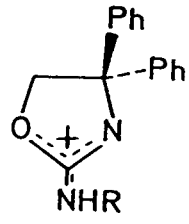
No antecedents on cmr data for β-iodoureas have been found. A signal at 156-157 ppm was assigned to the urea group [12]. The rest of the signals agreed with those expected according to the deshielding effect of the N atom and the shielding effect of the I atom.

The mass spectra for the *trans*-2-iodocyclohexylureas **2-4** showed the molecular peak and the characteristic [4] losses of I (M^+-127) and HI (M^+-128, peak A). For **2** further fragmentations implied the losses of 43 (C_3H_7) and 56 (C_4H_8). However, the β-iodo-β'-oxoureas **3** and **4** decomposed mainly by successive losses of HI and H_2O to give an oxazole derivative according to the chemistry of α-acylaminocarbonyl compound [13] and β-oxothioureas [14-18]. This fragment underwent McLafferty rearrangement to provide 2-amino-5-aryloxazole (fragment B) as it is described for related compounds [19]. The *N*-(2-iodo-3,3-dimethylbutyl)-*N'*-phenacylurea (**5**) had a similar fragmentation pathway. The formation of an aminooxazole cation was also supported by the presence of the fragment m/z 173, characteristic of 2-alkylaminooxazole derivatives [19]. The 2-iodo-4,4-diphenylureas **6,7** did not give molecular ion, but readily eliminated I, HI and IPh. These data agreed with the facility of **6** and **7** to eliminate the I atom *via* a phenonium cation, which is discussed later.

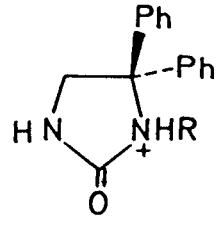
By heating of **6** and **7** in water the corresponding 2-amino- and 2-phenacylamino-4,4-diphenyl-2-oxazolines **8** and **9** were readily obtained (70-90%). The cyclization of **7** also took place after 12 hours in methyl sulphoxide. This behaviour contrast with that for other iodoalkylureas [2,6] which are transformed into aminooxazoles with difficulty. We attribute this difference to the anchimeric assistance of the neighbouring phenyl groups with formation of the above cited phenonium cation. The shorter reaction times diminish the thermolysis of starting β-iodourea and the hydrolysis and polimerisation of the protonated oxazolines [9,20], increasing the yield.

The structures of **8** and **9** were confirmed by analytical and spectroscopic data. The frequency ν C=N is dependent on the substituent at position 2 (amino 1695-1688 cm^{-1}, alkylamino 1665-1650 cm^{-1}) [10,21]. The ν C=N for **8** (1695 cm^{-1}) and **9** (1660 cm^{-1}) were in the expected range for the proposed structures. The cmr data supported the formation of an oxazoline ring when they were compared with data for **6** and **7**. Thus, the signal for C-5 in **8**, **9**, is shifted downfield (≅ −58 ppm) compared to that for C-2 (C-2') in **6**, **7**. These results accord with the substitution of the I atom by the O atom of the urea group, ruling out a possible substitution by the N atom. The chemical shift of C-2 (≅ 160 ppm) also accorded with data for related compounds [4]. The mass spectra of **8** and **9** showed the loss of H_2CO characteristic for 5-unsubstituted oxazolines [19]. Elimination of a phenyl radical led to the base peaks.

The displacement of the iodine atom in **6** and **7** by the oxygen atom rather than the nitrogen atom can be explained by the stability of the cations which would be formed. Aminooxazolinium cations **10** are stabilized by participation of π-bonding and nonbonding electrons of heteroatoms [20]. Such participation would not be possible in **11**.



10



11

EXPERIMENTAL

Melting points are uncorrected. The ir spectra were recorded as potassium bromide disk. Assignments of the pmr (200.13 MHz) spectra were confirmed by decoupling and H/D exchange experiments. Cmr (50.3 MHz) spectra were obtained for solutions in deuteriochloroform and DMSO-d_6. Proton-decoupled APT (attached proton test) [22] spectra were used to assist in signals assignments. The EI mass spectra were recorded on a Kratos MS-80RFA instrument operated at an ionizing energy of 70 eV, ionizing current 100 μA, accelerating voltage 4 KV, resolution 1000 (10% valley definition). The elemental composition of the ions was determined with a resolution of 10000 (10% valley definition).

Procedures for the Preparation of Compounds **1-7**.

The β-iodoureas **1-7** were obtained from *vic*-iodoisocyanates and amino compounds. Starting iodoisocyanates were prepared from cyclohexene, 3,3-dimethyl-1-butene or 1,1-diphenylethene, and silver isocyanate [23]. Storage of *vic*-iodoisocyanates was undertaken as dried solids at −5° in darkness. Depending on the solubility of the amines, one of the three following procedures was used.

(a) A solution of amine (4.7 mmoles) in ethyl ether (5 ml) was added to a solution of β-iodoalkyl isocyanate (4.7 mmoles) in ethyl ether (15 ml). The reaction mixture was kept at room temperature for t hours and then at 0° overnight. The crystalline β-iodoalkylurea was filtered off and recrystallized from an appropriate solvent.

(b) A solution of the amine hydrochloride (4.7 mmoles) in water (5 ml) was neutralized with sodium bicarbonate (4.7 mmoles) and added to a solution of β-iodoalkyl isocyanate (4.7 mmoles) in acetone (15 ml). This reaction mixture was kept at room temperature for t hours and the resulting solid product

crystallized from an appropriate solvent.

(c) A solution of the amine (4.7 mmoles) in water (5 ml) was added to a solution of β-iodoalkyl isocyanate (4.7 mmoles) in acetone (15 ml). After t hours at room temperature the reaction mixture was evaporated to dryness and the resulting syrup crystallized from a proper solvent. According to one of these methods, the following products were prepared.

N-(*n*-Butyl)-*N'*-(*trans*-2-iodocyclohexyl)urea (**1**).

This compound was obtained from *trans*-2-iodocyclohexyl isocyanate [24] and *n*-butylamine, method (a), t = 0.5 hour, yield 1.29 g (85%), mp 72-73° (from ethyl ether); uv (dichloromethane): λ max 230 nm (ε 4,000); ir: ν 3340, 3300 (NH), 1625 (CO) and 1575 cm^{-1} (NH); pmr (deuteriochloroform): δ 0.92 (3H, t, J = 7.0 Hz, CH_3), 1.10-2.60 (12H, m, 6 CH_2), 3.18 (2H, q, J = 7.0 Hz, N-CH_2), 3.73 (1H, qd, $J_{1',N'H} = J_{1',2'} = J_{1',6'ax} = 9.6$, $J_{1',6'eq} = 4.1$ Hz, 1'-H), 4.07 (1H, td, $J_{2',3'ax} = 9.6$, $J_{2',3'eq} = 4.1$ Hz, 2'-H), 4.97 (1H, t, $J_{NH,CH_2} = 7.0$ Hz, NH) and 5.11 (1H, d, N'H); cmr (deuteriochloroform): δ 13.7 (C-4), 20.0 (C-3), 24.5 (C-5'), 27.4 (C-4'), 32.2 (C-2), 33.7 (C-6'), 37.7 (C-2'), 38.9 (C-3'), 40.1 (C-1), 56. 0 (C-1'), and 157.6 (C=O).

Anal. Calcd. for $C_{11}H_{21}IN_2O$: C, 40.75; H, 6.53; N, 8.64. Found: C, 40.81; H, 6.55; N, 8.75.

N-Benzyl-*N*-methyl-*N'*-(*trans*-2-iodocyclohexyl)urea (**2**).

This compound was obtained from *trans*-2-iodocyclohexyl isocyanate and benzyl methyl amine, method (a), t = 0.5 hour, yield 1.60 g (91%), crystallized from ethanol keeping the temperature between 50° and 60° had mp 113-114°; uv (dichloromethane): λ max 254 and 227 nm (ε 2,700 and 4,600); ir: ν 3325 (NH), 1625 (CO), 1595 (C=C aromatic), 1535 (NH), 730 and 700 cm^{-1} (CH aromatic); pmr (deuteriochloroform): δ 1.10-2.60 (8H, m, 4CH_2), 2.91 (3H, s, CH_3), 3.85 (1H, tdd, $J_{1',6'ax} = J_{1',2'} = 10.9$, $J_{1',N'H} = 6.8$, $J_{1',6'eq} = 4.0$ Hz, 1'-H), 4.05 (1H, td, $J_{2',3'ax} = 10.9$, $J_{2',3'eq} = 4.0$ Hz, 2'-H), 4.48, 4.60 (each 1H, each d, $^2J = 16.0$ Hz, CH_2Ph), and 7.25-7.35 (6H, m, N'H, Ph); cmr (deuteriochloroform): δ 24.6 (C-5'), 27.7 (C-4'), 33.9 (C-6'), 34.2 (CH_3), 37.4 (C-2'), 39.3 (C-3'), 51.9 (CH_2Ph), 56.7 (C-1'), 127.0-137.6 (6C, Ph) and 157.5 (C=O); ms: m/z 372.0682 (M^+, 6%), 245.1580 (M^+-I, 48), 244.1531 (M^+-HI, peak A, 70), 229.1331 (A-CH_3, 29), 201.1012 (A-C_3H_7, 60), 188.1009 (A-C_4H_8, 6), 153.0995 (A-PhCH_2, 34), 120.0798 (52), 91.0532 (100), and 65 (24).

Anal. Calcd. for $C_{15}H_{21}IN_2O$: C, 48.39; H, 5.68; N, 7.52. Found: C, 48.27; H, 5.71; N, 7.65.

N-(*trans*-2-Iodocyclohexyl)-*N'*-*phenacylurea* (**3**).

This compound was obtained from *trans*-2-iodocyclohexyl isocyanate and phenacylamine hydrochloride, method (b), t = 0.5 hour, yield 1.84 g (94%), crystallized from ethanol (50-60°) had mp 131-133°; uv (dichloromethane): λ max 242 nm (ε 16,500); ir: ν 3335, 3305 (NH), 1685 (CO ketone), 1640 (CO urea), 1600 (C=C aromatic), 1570 (NH), 760 and 690 cm^{-1} (CH aromatic); pmr (deuteriochloroform): δ 1.10-2.70 (8H, m, 4CH_2), 3.83 (1H, qd, $J_{1,2} = J_{1,6ax} = J_{1,NH} = 10.0$, $J_{1,6eq} = 4.1$ Hz, 1-H), 4.02 (1H, td, $J_{2,3ax} = 10.0$, $J_{2,3eq} = 4.1$ Hz, 2-H), 4.81 (2H, m, CH_2COPh), 4.91 (1H, d, NH), 5.66 (1H, t, $J_{N'H,CH_2} = 3.4$ Hz, N'H) and 7.50-8.10 (5H, m, Ph); cmr (DMSO-d_6): δ 23.9 (C-5), 26.7 (C-4), 32.7 (C-6), 38.2 (C-3), 38.7 (C-2), 47.0 (CH_2COPh), 55.0 (C-1), 128.5-135.0 (6C, Ph), 156.8 (CO urea), and 196.5 (CH$_2$COPh); ms: m/z 386 (M^+, 1%), 259 (M^+-I, 1), 258 (M^+-HI, peak A, 2), 240 (A-H_2O, 20), 160 (peak B, 43), 105 (PhCO$^+$, 100), 81 (65), 79 (37), 77 (Ph$^+$, 85), 55 (30), 53 (33) and 51 (33).

Anal. Calcd. for $C_{15}H_{19}IN_2O_2$: C, 46.55; H, 4.96; N, 7.25. Found: C, 46.72; H, 4.93; N, 6.99.

N-(*trans*-2-Iodocyclohexyl)-*N'*-(*p*-methylphenacyl)urea (**4**).

This compound was prepared from *trans*-2-iodocyclohexyl isocyanate and *p*-methylphenacylamine hydrochloride, method (b), t = 0.5 hour, yield 1.71 g (90%), crystallized from ethanol (50-60°) had mp 137-139°; uv (dichloromethane): λ max 250 nm (ε 15,000); ir: ν 3340, 3295 (NH), 1685 (CO ketone), 1640 (CO urea), 1605 (C=C aromatic), 1570 (NH), and 820 cm^{-1} (CH aromatic); pmr (deuteriochloroform): δ 1.10-2.70 (8H, m, 4 CH_2), 3.83 (1H, qd, $J_{1,2} = J_{1,NH} = J_{1,6ax} = 9.6$, $J_{1,6eq} = 4.1$ Hz, 1-H), 4.02 (1H, td, $J_{2,3ax} = 9.6$, $J_{2,3eq} = 4.1$ Hz, 2-H), 4.81 (2H, m, CH_2COC$_6$H$_4$-), 5.11 (1H, m, N'H), 5.81 (1H, d, NH) and 7.25-7.93 (4H, m, C_6H_4); cmr (deuteriochloroform): δ 24.5 (C-5), 27.5 (C-4), 33.9 (C-6), 37.1 (C-2), 39.1 (C-3), 47.4 (CH_2COC$_6$H$_4$-), 56.1 (C-1), 131.9-144.8 (6C, C_6H_4), 156.9 (CO urea) and 195.5 (CH$_2$COC$_6$H$_4$-); ms: m/z 400.0596 (M^+, 1%), 273.1552 (M^+-I, 2), 272.1505 (M^+-HI, peak A, 6), 254.1407 (A-H_2O, 44), 174.0780 (peak B, 100), 128 (HI, 78), 127 (I^+, 42), 119 ($CH_3C_6H_4CO^+$, 70), 118 (45), 91 (20), and 81 (25).

Anal. Calcd. for $C_{16}H_{21}IN_2O_2$: C, 48.01; H, 5.29; N, 7.00. Found: C, 48.30; H, 5.24; N, 7.18.

N-(2-Iodo-3,3-dimethylbutyl)-*N'*-phenacylurea (**5**).

This compound was prepared from 3,3-dimethyl-2-iodobutyl isocyanate [3] and phenacylamine hydrochloride, method (b), t = 1 hour, yield 1.51 g (83%), mp 129-131° (from ethyl ether); uv (dichloromethane): λ max 244 nm (ε 21,400); ir: ν 3310 (NH), 1695 (CO ketone), 1620 (CO urea), 1595 (C=C aromatic), 1570 (NH), 755 and 685 cm^{-1} (CH aromatic); pmr (deuteriochloroform): δ 1.14 (9H, s, 3CH_3), 3.25 (1H, ddd, $J_{1a,1b} = 15.4$, $J_{1b,2} = 10.5$, $J_{1b,NH} = 4.4$ Hz, 1b-H), 3.92 (1H, ddd, $J_{1a,2}$ 2.5, $J_{1a,NH} = 6.6$ Hz, 1a-H), 4.26 (1H, dd, 2-H), 4.70, 4.89 (each 1H, each dd, $^2J_{H,H} = 23.1$, $J_{CH_2,N'H} = 4.6$ Hz, CH_2COPh), 5.46 (1H, dd, NH), 5.96 (1H, t, N'H) and 7.40-8.05 (5H, m, Ph); cmr (deuteriochloroform): δ 28.5 (3C, 3CH_3), 35.0 (C-3), 45.7 (C-1), 47.4 (CH_2COPh), 58.9 (C-2), 127.8-134.4 (6C, Ph), 157.3 (CO urea), and 195.6 (CH$_2$COPh); ms: m/z 388.0646 (M^+, 4%), 261.1580 (M^+-I, 38), 260.1504 (M^+-HI, peak A, 9), 242.1449 (A-H_2O, 10), 229.1481 (M^+-ICH$_3$-OH, 16), 173.0701 (11), 160.0614 (peak B, 45), 105 (PhCO$^+$, 100), 77 (60), 60 (40), and 55 (35).

Anal. Calcd. for $C_{15}H_{21}IN_2O_2$: C, 46.39; H, 5.45; N, 7.21. Found: C, 46.40; H, 5.67; N, 7.21.

N-(2-Iodo-1,1-diphenylethyl)urea (**6**).

This compound was obtained from 1,1-diphenyl-2-iodoethyl isocyanate [7] and ammonium hydroxide, method (c), t = 0.5 hour, yield 1.61 g (98%), crystallized from ethanol (50-60°) the product had mp 102-103°; uv (dichloromethane): λ max 229 nm (ε 6,900); ir: ν 3490, 3360, 3320 (NH), 1650 (CO), 1590 (C=C aromatic), 1530 (NH), 742 and 708 cm^{-1} (CH aromatic); pmr (deuteriochloroform): δ 4.37 (2H, br s, NH_2), 4.43 (2H, s, CH_2), 5.88 (1H, br s, NH), and 7.20-7.48 (10H, m, 2Ph); cmr (deuteriochloroform): δ 19.0 (C-2), 63.5 (C-1), 125.9-139.3 (12C, 2Ph), and 157.4 (CO urea); ms: m/z 306 (M^+-H_2NCONH_2, 4%), 239 (M^+-I, 2), 238 (M^+-HI, peak A, 8), 161 (A-Ph, 100), 180 (20), 165 (fluorenyl cation, 35), 128 (HI, 1), 127 (I, 1), 105 (19), 104 (18), 77 (11), and 60 (H_2NCONH_2, 21).

Anal. Calcd. for $C_{15}H_{15}IN_2O$: C, 49.20; H, 4.13; N, 7.65. Found: C, 49.45; H, 4.21; N, 7.42.

N-(2-Iodo-1,1-diphenylethyl)-*N'*-phenacylurea (**7**).

This compound was obtained from 1,1-diphenyl-2-iodoethyl isocyanate and phenacylamine hydrochloride, method (b), t = 0.5 hour, yield 2.16 g (95%), crystallized from ethanol (50-60°) the product had mp 124-125°; uv (dichloromethane): λ max 236 nm (ε 19,100); ir: ν 3305 (NH), 1695 (CO ketone), 1625 (CO urea), 1600, 1580 (C=C aromatic), 1550 (NH), 830, 745, and 700 cm^{-1} (CH aromatic); pmr (deuteriochloroform): δ 4.54 (2H, s, CH_2I), 4.67 (2H, d, $J_{CH_2,N'H}$ = 4.4 Hz, CH_2COPh), 5.76 (1H, t, N'H), 5.84 (1H, s, NH), and 7.22-7.92 (10H, m, 2Ph); cmr (deuteriochloroform): δ 19.2 (C-2), 47.5 (CH_2COPh), 63.5 (C-1), 125.7-142.6 (12C, 2Ph), 156.3 (CO urea), and 195.1 (CH_2COPh); ms: m/z 357.1593 (M^+-I, 2%), 356.1543 (M^+-HI, peak A, 9), 280.1163 (M^+-IPh, 22), 279.1073 (A-Ph, 100), 211.0823 (18), 180 (30), 165 (fluorenyl cation, 35), 105 ($PhCO^+$, 21), and 77 (21).

Anal. Calcd. for $C_{23}H_{21}IN_2O_2$: C, 57.03; H, 4.37; N, 5.78. Found: C, 57.01; H, 4.64; N, 5.95.

General Procedure for the Preparation of Compounds **8** and **9**.

A suspension of the appropriate *N*-(1,1-diphenyl-2-iodoethyl)-urea (**6** or **7**, 1.3 mmoles) in water (150 ml) was refluxed until solution was completed (3 hours). After cooling to room temperature, 50% potassium hydroxide was added to obtain a white solid, which was filtered off and crystallized from a proper solvent. According to this procedure, the products **8** (0.28 g, 90%) and **9** (0.36 g, 70%) were prepared.

2-Amino-4,4-diphenyl-2-oxazoline (**8**).

This compound had mp 176-177° (from ethanol); uv (dichloromethane): λ max 231 nm (ε 1,600); ir ν 3450 (NH), 1695 (C=N), 1590, 1580 (C=C aromatic), 1445 (NH), 1240 (C-O-C), 760 and 700 cm^{-1} (CH aromatic); pmr (deuteriochloroform): δ 3.80 (2H, br s, NH_2), 4.76 (2H, s, 5-H, 5'-H) and 7.14-7.36 (10H, m, 2Ph); cmr (deuteriochloroform): δ 80.1 (2C, C-4, C-5), 126.9-147.2 (12C, 2 Ph), and 160.6 (C-2); (DMSO-d_6): δ 76.6 (C-4), 77.9 (C-5), 126.1-148.3 (12C, 2 Ph), and 159.9 (C-2); ms: m/z 238.1084 (M^+, 45%), 237.1053 (M^+-H, 18), 208.0951 (M^+-H_2CO, 33), 207.0869 (30), 196.0897 (33), 194.0919 (43), 165.0692 (fluorenyl cation, 69), 161.0701 (M^+-Ph, 100), 119.0487 (32), 105.0379 ($PhCO^+$, 32), 104 (38), 91 (53), and 77 (17).

Anal. Calcd. for $C_{15}H_{14}N_2O$: C, 75.61; H, 5.92; N, 11.76. Found: C, 75.70; H, 5.94; N, 11.52.

2-Phenacylamino-4,4-diphenyl-2-oxazoline (**9**).

This compound had mp 125-126° (from 1:1 ether-hexane); uv (dichloromethane): λ max 242 and 229 nm (ε 16,300 and 14,300); ir: ν 3425 (NH), 1690 (CO), 1660 (C=N), 1590, 1580 (C=C aromatic), 1500 (NH), 1230 (C-O-C), 760, 750 and 690 cm^{-1} (CH aromatic); pmr (DMSO-d_6): δ 4.79 (2H, s, CH_2COPh), 4.79 (2H, s, 5-H, 5'-H), 7.00 (1H, br s, NH), and 7.15-8.13 (15H, m, 3Ph); cmr (DMSO-d_6): δ 49.5 (CH_2COPh), 76.2 (C-4), 79.3 (C-5), 126.8-148.1 (18C, 3Ph), 160.0 (C-2) and 196.4 (CH_2COPh); ms: m/z 356 (M^+, 35%), 326 (M^+-H_2CO, 4), 279 (M^+-Ph, 100), 237 (M^+-CH_2COPh, 5), 194 (20), 165 (fluorenyl cation, 45), 119 (9), 105 ($PhCO^+$, 45), 91 (47), and 77 (42).

Anal. Calcd. for $C_{23}H_{20}N_2O_2$: C, 77.51; H, 5.66; N, 7.86. Found: C, 77.48; H, 5.82; N, 7.71.

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