Russian Journal of Organic Chemistry, Vol. 39, No. 3, 2003, pp. 430–435. From Zhurnal Organicheskoi Khimii, Vol. 39, No. 3, 2003, pp. 457–462.

Dedicated to Full Member of the Russian Academy of Sciences I.P. Beletskaya on Her Jubilee

Unexpected Products from Carbonylation of Lithiated Quinazolin-4(3*H*)-one Derivatives*

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Received October 23, 2002

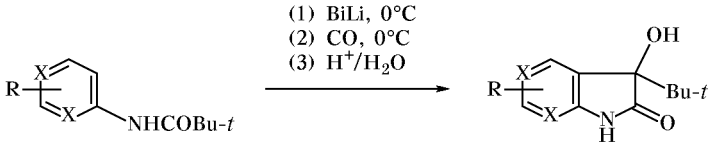
Abstract—Doubly lithiated 3-pivaloylaminoquinazolin-4(3*H*)-one reacts with carbon(II) oxide at 0°C to give 77% of a mixture of azetidinone and indole derivatives, each incorporating a diisopropylamide unit from lithium diisopropylamide used for lithiation. No analogous reaction occurs with doubly lithiated 3-acetylaminoquinazolin-4(3*H*)-one and 3-acyl-2-alkylquinazolin-4(3*H*)-one. Carbonylation of doubly lithiated 2-alkyl-3-aminoquinazolin-4(3*H*)-ones at 0°C results in deamination to give 2-alkylquinazolin-4(3*H*)-ones in good yields.

Reactions of organolithium compounds with carbon monoxide are rarely used in synthetic chemistry, presumably because of the instability and high reactivity of acyllithium intermediates which can lead to a variety of products [1]. To circumvent this problem, organic chemists have devised a number of acyl carbanion equivalents which are effective in nucleophilic acylation reactions [2]. Seyferth *et al.* [3] have shown that it is possible to trap acyllithium intermediates formed by carbonylation of alkyllithium reagents provided that the temperature is kept very low and that the trapping electrophile can be used *in situ*. However, the conditions are rather restrictive, and the method has rarely been used with aryllithium derivatives [4].

Better results are obtained when the incipient aryllithium is trapped intramolecularly [5]. For example, we have previously shown that doubly lithiated *N*-pivaloylanilines and *N*-pivaloylaminopyridines **I** smoothly react with carbon monoxide at 0°C under atmospheric pressure to give 3-*tert*-butyldioxindoles and 3-*tert*-butylazadioxindoles **II** in good yields as a result of intramolecular trapping [6] (Scheme 1). Carbon(II) oxide reacts in a similar way with doubly lithiated *N′*-aryl-*N*,*N*-dimethylthioureas to give indigotins [7] and with lithiated *N′*-aryl-*N*,*N*-dimethylureas to give isatins [8].

We have also shown that doubly lithiated 3-(acylamino)quinazolin-4(3*H*)-ones can be successfully trapped with a variety of electrophiles [9–11]. Compounds possessing this ring system exhibit versatile biological activity [12]. Therefore, it was reasonable to extend the range of electrophiles to include carbon monoxide with a view to obtain some interesting

Scheme 1.



I **II**

X = CH, N.

* The original article was submitted in English.

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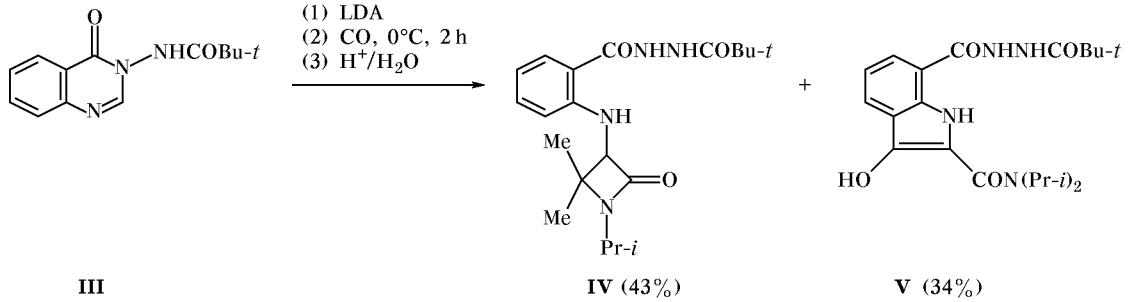
Scheme 2.



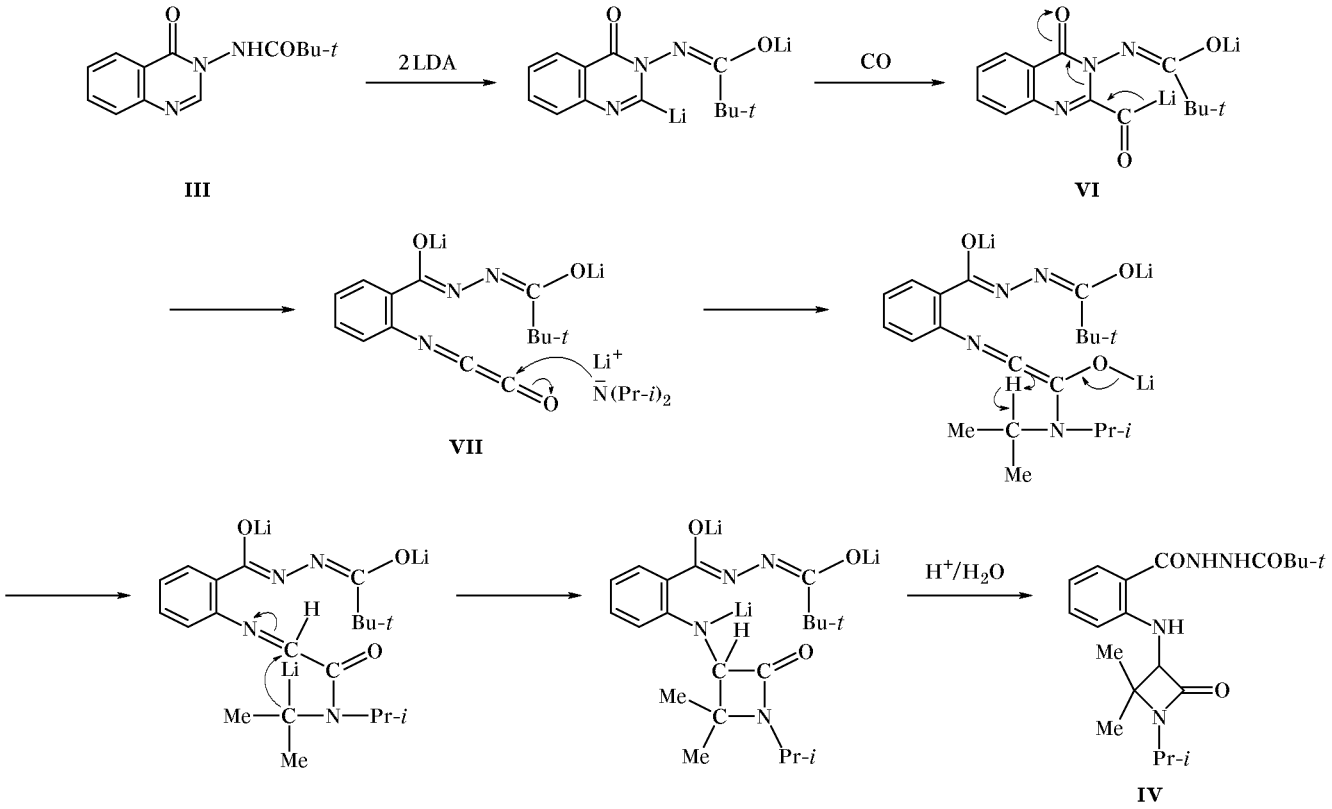
III **IV** (43%) **V** (34%)

fused quinazolinone derivatives by analogy with Scheme 1. The present article reports on our attempts at such reactions, which gave unexpected results.

3-Pivaloylaminoquinazolin-4(3*H*)-one (**III**) was doubly lithiated with LDA and was then brought into reaction with carbon(II) oxide at 0°C under atmospheric pressure. The subsequent protonation gave two new products. They were purified by column chromatography and were identified as azetidinone derivative **IV** and substituted indole **V** (Scheme 2).

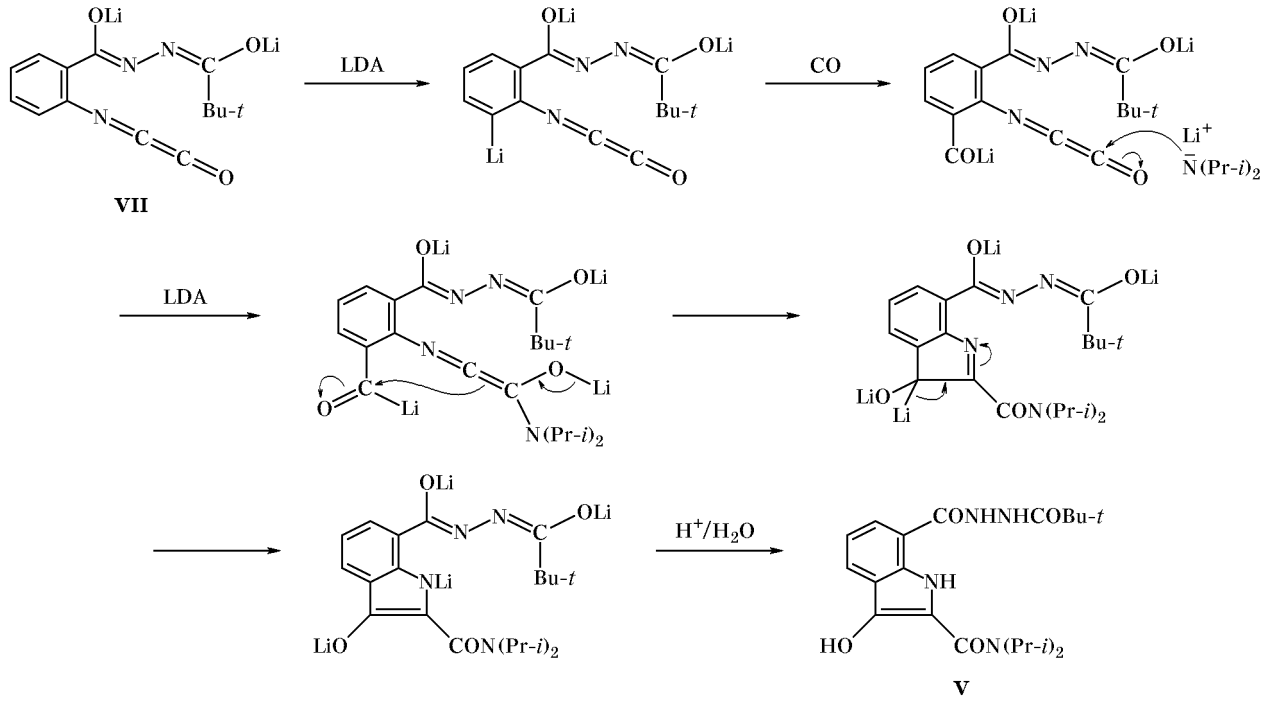
The ^{1}H NMR spectrum of compound **IV** showed three exchangeable doublets resonating in the δ 7.92–9.24 ppm region. Two of these signals were coupled to each other ($J \approx 5$ Hz), and the third was coupled to a CH doublet resonating at δ 4.19 ppm ($J \approx 7$ Hz). The spectrum clearly showed an isopropyl group and two separate methyl groups in addition to the *tert*-butyl group, as well as four aromatic protons of an *ortho*-substituted benzene ring. The IR spectrum showed two carbonyl groups, and the accurate mass of the molecular ion in the mass spectrum indicated a formula of $C_{20}H_{30}N_4O_3$. The ^{13}C NMR spectrum of **IV** contained 18 different signals, only the three methyl groups of the *tert*-butyl group being degenerate. These data are consistent with the proposed structure.

Scheme 3.



III **VI** **VII** **IV**

Scheme 4.



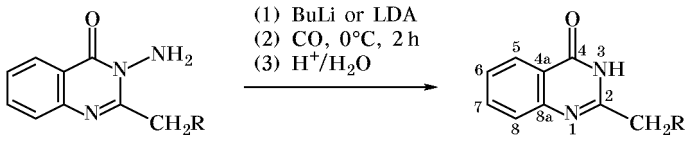
The ^{1}H NMR spectrum of compound **V** showed four exchangeable singlets resonating in the δ 9.24–10.16 ppm region. It also showed the presence of two identical isopropyl groups in addition to the *tert*-butyl group and only three aromatic protons corresponding to a 1,2,3-trisubstituted benzene ring. The accurate mass of the molecular ion in the mass spectrum indicated a molecular formula of $C_{21}N_{30}N_4O_4$ which could be accounted for by incorporation of a second carbon(II) oxide unit over and above that required for formation of **IV**. Apart from the signals due to the *tert*-butyl and isopropyl groups, all carbons resonated at separate positions in the ^{13}C NMR spectrum. The observed chemical shifts (^{1}H and ^{13}C) for compounds **IV** and **V** were in reasonable agreement with those estimated using ChemDraw.™

We have not studied the mechanisms of the reactions leading to compounds **IV** and **V** which are formed via incorporation of one or two carbon(II) oxide molecules, repectively. However, Schemes 3 and 4 show plausible pathways for their formation. Here, the key step which causes the reaction to deviate from the path predominating in reactions with pivaloylaminobenzenes is ring opening of the lithiated quinazoline ring of intermediate **VI**. The subsequent carbon(II) oxide uptake gives intermediate **VII**.

An attempt was made to extend the reaction to 3-acetylaminoquinazolin-4(3*H*)-one. However, its doubly lithiated derivative failed to take up carbon(II) oxide under analogous conditions, and the starting material was recovered from the reaction mixture. Presumably, initial deprotonation of the acetylamino group yields a relatively unreactive enolate anion. No further attempts were made to find conditions under which this carbonylation could be successful.

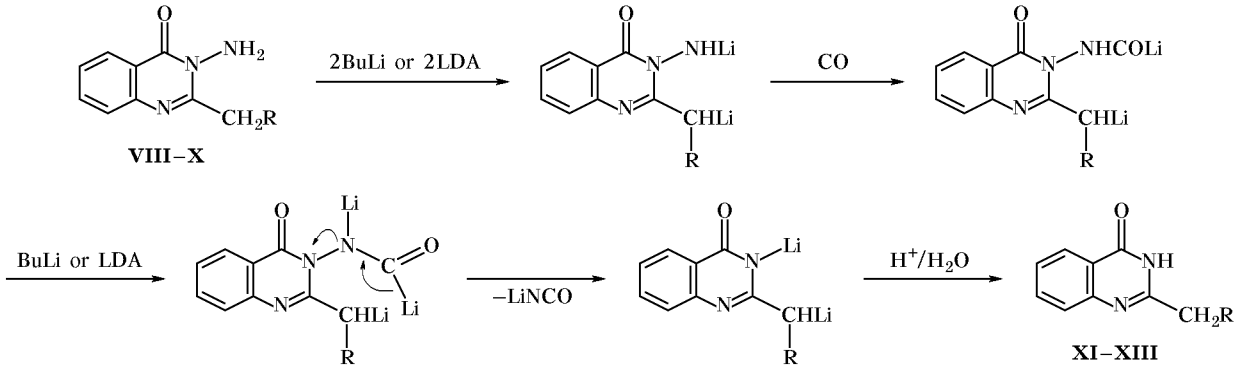
We have previously shown that 2-alkyl-3-acylaminoquinazolin-4(3*H*)-ones [9, 10] and 2-alkyl-3-aminoquinazolin-4(3*H*)-ones [11] are readily doubly

Scheme 5.



VIII, **XI**, R = H; **IX**, **XII**, R = Me; **X**, **XIII**, R = Et.

Scheme 6.



lithiated and that their dilithium derivatives are capable of successfully reacting with a variety of electrophiles. Attention was therefore turned to the carbonylation of these doubly lithiated compounds. Doubly lithiated 3-acylamino-2-alkylquinazolin-4(3*H*)-ones failed to take up carbon(II) oxide. However, doubly lithiated 2-alkyl-3-aminoquinazolin-4(3*H*)-ones **VIII**–**X** did react with carbon(II) oxide at 0°C, surprisingly giving the corresponding 2-alkylquinazolin-4(3*H*)-ones **XI**–**XIII** in 61–69% yield (isolated product; Scheme 5).

The ^{1}H NMR spectra of compounds **XI**–**XIII** were characterized by the presence of an exchangeable singlet resonating at δ 12.2 ppm, which was assigned to the quinazolinone NH proton. Compounds **XI**–**XIII** were found to be identical in all respects to authentic samples prepared as described in [13]. A plausible mechanism of the reaction leading to products **XI**–**XIII** is shown in Scheme 6. The mechanism presumes elimination of isocyanate ion.

Thus the carbonylation of doubly lithiated quinazolin-4(3*H*)-one derivatives occurs in a way different from the carbonylation of doubly lithiated *N*-pivaloylanilines or *N*-pivaloylaminopyridines. Unexpected products were isolated from the reactions of doubly lithiated 3-pivaloylaminoquinazolin-4(3*H*)-one and 2-alkyl-3-aminoquinazolin-4(3*H*)-ones with carbon(II) oxide.

EXPERIMENTAL

The melting points were determined on an electrothermal digital melting point apparatus and are uncorrected. The IR spectra were recorded on a Perkin–Elmer 1725X spectrometer. The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker spectrometer operating at 400 MHz for ^{1}H and 100 MHz for ^{13}C. The chemical shifts are reported in parts per million relative to tetramethylsilane. Assignments of signals are based on coupling patterns and expected chemical shift values and have not been rigorously confirmed. Signals with similar characteristics might be interchanged. The low-resolution mass spectra were recorded on a VG 12-253 spectrometer under electron impact (EI) at 70 eV and chemical ionization (CI) using ammonia as ionizing gas. The accurate mass data were obtained on a VG ZAB-E instrument. The elemental analyses were obtained from the laboratories of the University of Wales, Cardiff. Column chromatography was carried out using Merck Kieselgel 60 (230–400 mesh). Butyllithium and lithium diisopropylamide were obtained from Aldrich; butyllithium was estimated prior to use by the method of Watson and Eastham [14]. Tetrahydrofuran was distilled over sodium diphenylketyl. The other chemicals were obtained from Aldrich and were used without further purification. The solvents were purified by standard techniques [15, 16].

Carbonylation of 3-pivaloylaminoquinazolin-4(3*H*)-one (III). A solution of 0.245 g (1.0 mmol) of quinazolinone **III** and 2.06 ml (3.3 mmol) of lithium diisopropylamide in 10 ml of THF was stirred for 1 h at –78°C under argon. The temperature was slowly allowed to rise to 0°C. A balloon filled with carbon(II) oxide (~500 ml) was fitted to a needle, and CO was allowed to bleed into the reaction mixture. After 2 h, the solution was poured into a saturated solution of NH_4Cl (10 ml), and the mixture was extracted with ethyl acetate (20 ml). The organic layer was washed with 10 ml of water, dried over $MgSO_4$, and evaporated under reduced pressure. The residue was purified by column chromatography using ether–hexane (80:20 by volume) to isolate 0.162 g (43%) of azetidinone **IV** and 0.137 g (34%) of indole **V**.

4,4-Dimethyl-1-(1-methylethyl)-3-(2-pivaloylhydrazinocarbonylphenylamino)azetidin-2-one (IV). mp 200–201°C. IR spectrum (KBr), ν, cm^{-1}: 3305, 1725, 1678, 1641, 1602, 1581, 1509. ^{1}H NMR

spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 9.24 br.d (1H, CON**H**NH, *J* = 4.8), 8.91 br.d (1H, CONHN**H**CO-Bu-*t*, *J* = 4.8), 7.93 br.d (1H, N**H**CH, *J* = 6.6), 7.57 d.d (1H, 3′-H, *J* = 1.4, 7.9), 7.27 d.t (1H, 5′-H, *J* = 1.4, 7.9), 6.67 d.t (1H, 4′-H, *J* = 1.4, 7.9), 6.46 d (1H, 6′-H, *J* = 7.9), 4.19 d (1H, 3-H, *J* = 6.6), 3.58 sept [1H, C**H**$(CH_3)_2$, *J* = 6.9], 1.56 s (3H, CH_3), 1.34 d and 1.32 d [6H, CH(C**H**$_3)_2$, *J* = 6.9], 1.28 s [9H, C$(CH_3)_3$], 1.25 s (3H, CH_3). ^{13}C NMR spectrum (CDCl3), δ_C, ppm: 175.6 s (COBu-*t*); 166.6 s (C^2); 165.1 s (CONHNH); 148.3 s ($C^{1'}$); 133.4 d ($C^{5'}$); 128.0 d ($C^{3'}$); 116.5 d ($C^{4'}$); 113.4 s ($C^{2'}$); 111.9 d ($C^{6'}$); 68.3 d (C^3); 62.7 s (C^4); 44.4 d [**C**H$(CH_3)_2$]; 38.2 s [**C**$(CH_3)_3$]; 27.2 q, 25.5 q, 21.9 q, 21.7 q, and 21.1 q [C(**C**$H_3)_2$]. Mass spectrum, *m*/*z* (I_{rel}, %): EI: 374 M^+ (4), 347 (2), 289 (17), 173 (30), 100 (100), 57 (45); CI: 375 MH^+ (100), 347 (5), 276 (12), 100 (22). Found: M^+ 374.2318; calculated for $C_{20}H_{30}N_4O_3$: 374.2318. Found, %: C 64.0; H 8.1; N 15.1. Calculated, %: C 64.17; H 8.02; N 14.97.

2-Diisopropylaminocarbonyl-3-hydroxy-7-pivaloylhydrazinocarbonylindole (V). mp 223–224°C. IR spectrum (KBr), ν, cm^{-1}: 3437, 1692, 1662, 1600, 1575, 1544. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 10.16 br.s (1H, OH), 9.83 br.s (1H, NH), 9.50 br.s (1H, CON**H**NH), 9.24 br.s (1H, CONHN**H**-COBu-*t*), 7.89 d (1H, 4-H, *J* = 7.9), 7.86 d (1H, 6-H, *J* = 7.9), 7.03 t (1H, 5-H, *J* = 7.9), 4.08 br {2H, [C**H**$(CH_3)_2]_2$}, 1.41 d {12H, [CH(C**H**$_3)_2]_2$, *J* = 6.7} and 1.29 s [9H, C$(CH_3)_3$]. ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 177.7 s (**C**OBu-t), 166.7 s (CONHNH), 163.5 s (**C**OPr-*i*), 138.3 s (C^{7a}), 132.6 s (C^2), 123.6 d (C^4), 123.2 d (C^5), 120.4 s (C^{3a}), 117.6 d (C^6), 114.7 s (C^3), 114.2 s (C^7), 47.8 d {[**C**$H(CH_3)_2]_2$}, 38.0 s [**C**$(CH_3)_3$], 27.4 q [C(**C**$H_3)_3$] and 21.1 q [CH(**C**$H_3)_2$]. Mass spectrum, *m*/*z* (I_{rel}, %): EI: 402 M^+ (35), 360 (10), 244 (30), 186 (17), 102 (55), 57 (100); CI: 403 MH^+ (100), 304 (20), 130 (30). Found: M^+ 402.2267; calculated for $C_{21}H_{30}N_4O_4$: 402.2267. Found, %: C 62.6; H 7.6; N 13.9. Calculated, %: C 62.68; H 7.46; N 13.93.

Carbonylation of 2-alkyl-3-aminoquinazolin-4(3*H*)-ones VIII–X. A solution of BuLi (in the case of compound **VIII**) or LDA (**IX** and **X**) (1.6 M, 2.06 ml, 3.3 mol) was added dropwise with stirring under nitrogen to a solution of 1.0 mmol of the corresponding 2-alkyl-3-aminoquinazolin-4(3*H*)-one in 20 ml of THF. The mixture was stirred for 45 min at –78°C and was allowed to slowly warm up to 0°C. Carbon(II) oxide was bled into the mixture by attachment of a balloon filled with the gas through a needle. After 2 h, the solution was poured into a saturated solution of NH_4Cl (10 ml), and the mixture was extracted with ethyl acetate (20 ml). The organic layer was washed with 10 ml of water, dried over $MgSO_4$, and evaporated under reduced pressure. The residue was purified by column chromatography using ether–hexane (80 : 20 by volume) as eluent to isolate compounds **XI**–**XIII** as white crystals in 61–69% yields.

2-Methylquinazolin-4(3*H*)-one (XI). mp 239°C; published data [13]: mp 238–239°C. IR spectrum (KBr), ν, cm^{-1}: 3318, 1677, 1610, 1566, 1504. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 12.21 br.s (1H, NH), 8.08 d.d (1H, 5-H, *J* = 1.0, 7.9), 7.74 d.t (1H, 7-H, *J* = 1.0, 7.9), 7.55 d (1H, 8-H, *J* = 7.9), 7.43 d.t (1H, 6-H, *J* = 1.0, 7.9), 2.36 s (3H, CH_3). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 161.7 s (C^4), 154.1 s (C^2), 149.0 s (C^{8a}), 134.1 d (C^7), 126.5 d (C^5), 125.8 d (C^6), 125.7 d (C^8), 120.7 s (C^{4a}), 21.43 q (CH_3). Mass spectrum, *m*/*z* (I_{rel}, %): EI: 160 M^+ (100), 147 (20), 119 (60), 92 (65), 64 (40). Found: M^+ 160.0637. Calculated for $C_9H_8N_2O$: 160.0637. Found, %: C 67.3; H 5.2; N 17.7. Calculated, %: C 67.50; H 5.00; N 17.50.

2-Ethylquinazolin-4(3*H*)-one (XII). mp 236°C; published data [13]: mp 235°C. IR spectrum (KBr), ν, cm^{-1}: 3313, 1681, 1611, 1565, 1505. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 12.17 br.s (1H, NH), 8.08 d.d (1H, 5-H, *J* = 1.0, 7.9), 7.75 d.t (1H, 7-H, *J* = 1.0, 7.9), 7.59 d (1H, 8-H, *J* = 7.9), 7.44 d.t (1H, 6-H, *J* = 1.0, 7.9), 2.36 q (2H, CH_2, *J* = 7.5), 1.28 t (3H, CH_3, *J* = 7.5). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 161.8 s (C^4), 158.2 s (C^2), 148.9 s (C^{8a}), 134.0 d (C^7), 126.7 d (C^5), 125.7 d (C^6), 125.6 d (C^8), 120.7 s (C^{4a}), 26.5 t (CH_2), 11.3 q (CH_3). Mass spectrum, *m*/*z* (I_{rel}, %): EI: 175 MH^+ (40), 174 M^+ (100), 146 (12), 119 (16); CI: 175 MH^+, (100). Found: M^+ 174.0793. Calculated for $C_{10}H_{10}N_2O$: 174.0793. Found, %: C 68.7; H 5.8; N 16.1. Calculated, %: C 68.96; H 5.75; N 16.09.

2-Propylquinazolin-4(3*H*)-one (XIII). mp 207°C; published daa [13]: mp 206–207°C. IR spectrum (KBr), ν, cm^{-1}: 3400, 1668, 1609, 1565, 1504. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 12.16 br.s (1H, NH), 8.08 d.d (1H, 5-H, *J* = 1.0, 7.9), 7.75 d.t (1H, 7-H, *J* = 1.0, 7.9), 7.58 d (1H, 8-H, *J* = 7.9), 7.44 d.t (1H, 6-H, *J* = 1.0, 7.9), 2.58 t (2H, C**H**$_2CH_2CH_3$, *J* = 7.5), 1.77 sext (2H, C**H**$_2CH_3$, *J* = 7.5), 0.96 t (3H, CH_3, *J* = 7.5). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 161.8 s (C^4), 157.1 s (C^2), 148.9 s (C^{8a}), 134.0 d (C^7), 126.7 d (C^5), 125.7 d (C^6), 125.6 d (C^8), 120.8 s (C^{4a}), 36.3 t (**C**$H_2CH_2CH_3$), 20.2 t (**C**H_2CH_3), 13.4 q (CH_3). Mass spectrum, *m*/*z*

(I_{rel}, %): EI: 189 MH^+ (100), 160 (12). Found: MH^+ 189.1028. Calculated for $C_{11}H_{13}N_2O$: 189.1028. Found, %: C 70.2; H 6.4; N 14.9. Calculated, %: C 70.21; H 6.38; N 14.89.

G.A. El-Hiti thanks the University of Wales Swansea for financial support and the Royal Society of Chemistry for an international author grant. We thank the EPSRC Mass Spectrometry Service, University of Wales Swansea, for recording mass spectra for us. We also thank EPSRC and the University of Wales for grants that enable the purchase of NMR equipment used in the course of this work.

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