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REACTION OF BENZOFURAZAN 1-OXIDE WITH 1-(2-NAPHTHYL)- AND 1-HETARYL-BUTANE-1,3-DIONES; PREPARATION OF ARYL AND HETARYL 2-QUINOXALINYL KETONES

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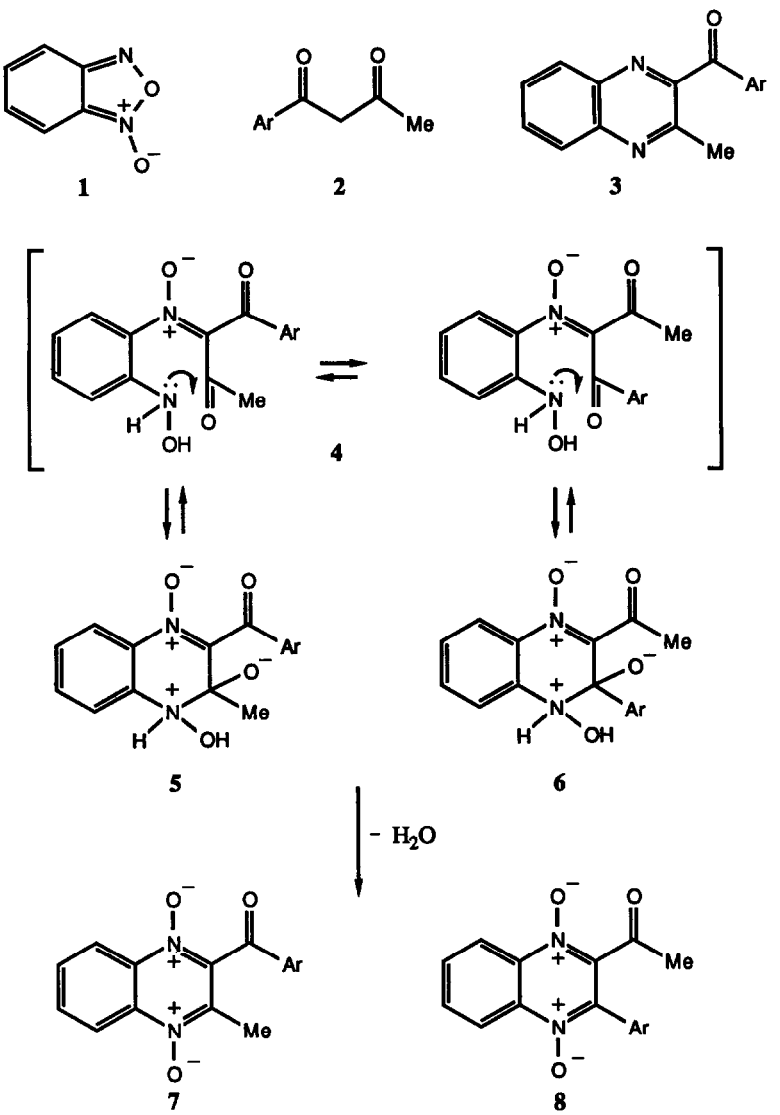
Abstract: Aryl (4-methoxyphenyl and 2-naphthyl) and hetaryl (2-furyl, 3-pyridyl, and 2-thienyl) 3-methyl-2-quinoxalinyl ketones were prepared *via* the reaction of benzofurazan 1-oxide (BFO) with 1-aryl- and 1-hetaryl-butane-1,3-diones, followed by reduction of the resulting aryl or hetaryl 3-methyl-2-quinoxalinyl ketone 1,4-dioxides with sodium dithionite. 1-(2-Furyl)butane-1,3-dione and 1-(2-pyridyl)butane-1,3-dione yielded 2-acetyl-3-(2-furyl)-quinoxaline 1,4-dioxide and 2-acetyl-3-(2-pyridyl)quinoxaline 1,4-dioxide respectively as minor products on reaction with BFO. The effect of the structure of the hetaryl (or aryl) group in the 1,3-diketone on the reaction with BFO is reported.

As part of a study into the photochemistry of 2-quinoxalinyl ketones, the 2-aroyl-3-methylquinoxalines **3a**, **3b**, **3c**, **3e** and **3f** were prepared by sodium dithionite reduction of the corresponding quinoxaline 1,4-dioxides **7a**, **7b**, **7c**, **7e** and **7f**. The latter compounds were synthesised by the well known Beirut reaction[1] between benzofurazan 1-oxide (BFO) (**1**) and the appropriate 1,3-diketone **2** in diethylamine or triethylamine. The 1,3-diketones were prepared according to the literature methods cited in the experimental section.

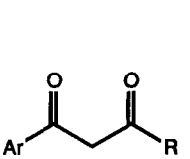
The reaction of BFO (**1**) with unsymmetrical diketones **2** could, in theory, yield either or both of the two isomeric 2,3-disubstituted quinoxaline 1,4-dioxides **7** and **8**. Formation of these products may result from cyclisation of intermediate **4*** *via* nucleophilic addition of the hydroxylamine nitrogen at either of the two carbonyl groups, followed by dehydration. In the absence of other factors, cyclisation at the more electrophilic acetyl carbonyl group, leading to quinoxaline dioxide **7**, should be favoured. Haddadin, Issidorides, and co-workers showed the influence of steric and electronic effects on the regiospecificity of the reaction of BFO with a series of 1,3-diketones **9**.[3] Smaller alkyl groups (R = Me and Et) and electron-donating aryl groups (Ar = 4-methoxyphenyl and *p*-tolyl) in the diketone **9** favoured the formation of 3-alkyl-

*An intermediate of type **4** was proposed by Mason and Tennant.[2]



Ar = 2-naphthyl (**a**), 2-thienyl (**b**), 2-furyl (**c**), 2-pyridyl (**d**), 3-pyridyl (**e**), 4-methoxyphenyl (**f**)

2-aroylquinoxaline 1,4-dioxides **10**, whereas larger alkyl groups (R = i-Pr and t-Bu) and electron-withdrawing aryl groups (Ar = 4-bromophenyl and 4-nitrophenyl) favoured the formation of 2-alkanoyl-3-arylquinoxaline 1,4-dioxides **11**. Steric effects dominated when R was methyl or t-butyl, when quinoxaline dioxides **10** and **11** respectively were the sole products irrespective of the nature of the aryl group. In our studies, however, the structure of the aryl group (in diketone 2) was of greater significance in determining the outcome of the reaction with BFO. Several new hetaroyl and aroyl quinoxalines and quinoxaline 1,4-dioxides, and a modification to the deoxygenation of the latter, are described.



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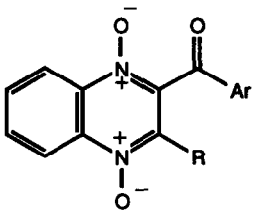

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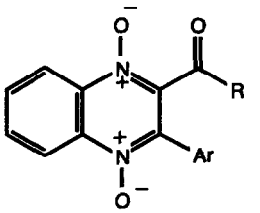

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BFO (**1**) reacted with diketones **2a - 2e** to give 2-aroylquinoxaline 1,4-dioxides **7a - 7e** respectively as the major or sole product (see Table). With the furyl- and 2-pyridyl-1,3-diketones **2c** and **2d**, 2-acetyl-quinoxaline 1,4-dioxides **8c** and **8d** respectively were minor products. With the exception of quinoxalines **7c**, **7d** (isolated by column chromatography), **8c**, and **8d** (isolated by preparative t.l.c.), the products crystallised from the reaction mixtures in a fairly pure state. Isomers **7** were readily distinguished from **8** on the basis of their carbonyl stretching frequencies in the infrared spectra; ν_{CO} (**7**) 1665-1640 cm^{-1} and ν_{CO} (**8**) 1720-1700 cm^{-1}.

In the reaction of BFO with diketones **2**, diethylamine was the solvent and base catalyst, except in the case of diketone **2c** when triethylamine was preferred. However, diethylamine itself reacts slowly with BFO to give a complex mixture of products[4] and, in some cases,[3] deacylation has been shown to occur in diethylamine. As these side-reactions may occur to a lesser extent in triethylamine, this amine was compared with diethylamine for use as the base solvent in the reaction of BFO with 2-thenoylacetone (**2b**). The yields of quinoxaline dioxide **7b** were 65% (crude product, m.p. 200-201°C) and 48% (crude product, m.p. 199-201°C) after 18 hours using diethylamine and triethylamine respectively (see Table). In the latter case, 10% more of dioxide **7b** was collected after a further 6 days. After filtering off the product **7b** from the reaction in diethylamine, the residual reaction mixture was shown (t.l.c.) to contain a complex mixture of compounds. None of them were present in significant amounts and no deacylation product (2-methylquinoxaline 1,4-dioxide) could be detected. Although side-reactions appear (t.l.c.) more significant in diethylamine, the use of

triethylamine (in which the reaction is much slower*) does not offer any advantage in this instance.

Unlike aryl-butane-1,3-diones **9** (R = Me), which react with BFO to yield solely 2-aroyl-3-methyl-quinoxaline 1,4-dioxides **10**, hetaryl-butane-1,3-diones **2c** and **2d** form 2-acetyl-3-hetarylquinoxaline 1,4-dioxides **8c** and **8d** as minor products. This difference in regiospecificity may be connected with the ability

Table: Reaction of BFO (**1**) with 1,3-Diketones **2**

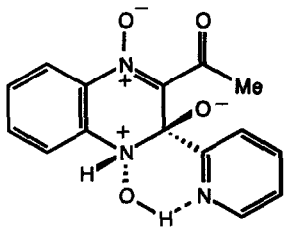
1,3-Diketone	Base solvent	% Yield (Quinoxaline 1,4-dioxide)[a]
2a	Et_2NH	70 (**7a**)
2b	Et_2NH	65 (**7b**)
2b	Et_3N	48 (**7b**) or 58 (**7b**)[b]
2c	Et_3N	52 (**7c**), 8 (**8c**)
2d	Et_2NH	33 (**7d**), 14 (**8d**)
2e	Et_2NH	60 (**7e**)

[a] Reaction time approx. 18 hours. [b] Reaction time 7 days.

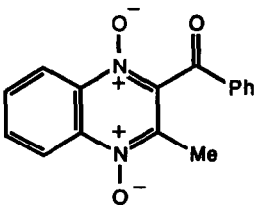
of the hetero atom in the 2-furyl or 2-pyridyl substituent to take part in hydrogen-bonding at a crucial stage of the reaction. For example, reversible intramolecular nucleophilic attack by the hydroxylamino nitrogen at the aroyl carbonyl group in **4d** leads to intermediate **6d** which is stabilised by hydrogen-bonding (see diagram; such hydrogen-bonding is not possible in the alternative cyclisation intermediate **5d**). Subsequent dehydration yields quinoxaline dioxide **8d**. Thus a combination of hydrogen-bonding and relatively high electrophilic reactivity of the 2-pyridine-carbonyl CO group leads to a significant proportion (30%) of intermediate **4d** cyclising to 2-acetylquinoxaline dioxide **8d**. Similarly, hydrogen-bonding should be present in intermediate **6c** from the furyl-diketone **2c** (and to a lesser extent in the thienyl intermediate **6b**) but, in this case, the lower electrophilic reactivity of the furoyl carbonyl group results in a lower yield of the 2-acetylquinoxaline dioxide **8c**. The reaction of BFO with diketones **2a** and **2e**, in which hydrogen-bonding as described above is not a factor, leads as expected only to 2-aroylquinoxaline dioxides **7a** and **7e** respectively.

Quinoxalines **3a**, **3b**, **3c**, **3e**, and **3f** were prepared from the corresponding 1,4-dioxides **7a**, **7b**, **7c**, **7e**, and **7f** by reduction with aqueous sodium dithionite using dimethylformamide (DMF) as the solvent. Addition of water causes the quinoxaline to precipitate from the reaction mixture. Methanol and, less frequently, ethanol have been used previously as solvents in similar reductions of quinoxaline 1,4-dioxides,[5]

*In the reaction of BFO with diketone **2b** in diethylamine at room temperature, crystals of product **7b** first appeared after 1-2 minutes and a considerable amount of product was present after 5 minutes. In triethylamine, the corresponding times for a similar appearance of product were 45 minutes and 4-5 hours respectively.



6d



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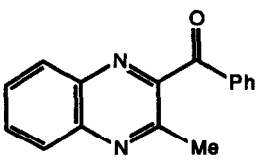

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but the low solubility of many such dioxides, in particular **7a**, necessitates the use of inconveniently large quantities of hot alcohol and the later addition of a large volume of water. Quinoxaline dioxides **7** form 5-10% solutions in DMF and the reduction in this solvent is rapid at room temperature. Prior to the above work, several solvents were tested for use in the dithionite reduction of quinoxaline dioxide **12**.[6] A 3.3% solution of dioxide **12** in a 5:1 mixture of solvent and water at 100-120°C was treated with aqueous sodium dithionite. Quinoxaline **13** was obtained in 46-47% yield when the solvent was DMF or dimethyl sulphoxide. With dioxan as the solvent, only partial reduction took place. Examination of the product (t.l.c. and i.r.) indicated that the product contained quinoxalines **12** and **13** along with two further compounds, probably mono-oxides. No product was isolated from the reaction mixture when 2-ethoxyethanol was used as the solvent. In neat DMF, quinoxalines **3a**, **3b**, **3c**, **3e**, and **3f** were produced in 56-80% yield.

Experimental Section

Melting points, determined using an electrothermal melting point apparatus, are uncorrected. I.r. spectra were measured as KBr discs or Nujol mulls, on Pye Unicam SP3-100 or Perkin Elmer 297 spectrophotometers. ^{1}H N.m.r. spectra were recorded for solutions in DMSO-d_6 or $CDCl_3$ with TMS as internal reference on Brucker WP80 SY or Perkin Elmer R32 spectrometers. Mass spectra were run on VG Analytical 7070E or Kratos MS30 mass spectrometers. 2-(4-Methoxybenzoyl)-3-methylquinoxaline 1,4-dioxide (**7f**), benzofurazan 1-oxide (**1**), and 1,3-diketones **2a**, **2b**, **2c**, **2d**, and **2e** were synthesised according to literature methods in references 3 and 7-12 respectively.

General Method for the Preparation of Quinoxaline 1,4-Dioxides 7 and 8. - A warm solution of benzofurazan 1-oxide (25 mmol) in diethylamine (20 ml) was added to a warm solution of the 1,3-diketone **2** (25 mmol) in diethylamine (10-15 ml). The reaction mixture was left to stand at room-temperature overnight, although the product usually crystallised from the solution after a few minutes. The mixture was thinned with a little diethylamine, cooled and filtered, and the product was washed with a little cold methanol and recrystallised from methanol. In the reaction of diketone **2c**, triethylamine was used instead of diethylamine. Yield, m.p., and spectroscopic data are quoted for the following quinoxaline 1,4-dioxides.

3-*Methyl*-2-(2-*naphthoyl*)*quinoxaline* 1,4-*dioxide* (**7a**) (5.8 g, 70%), m.p. 199-201°C (decomp.) (Found: C, 72.45; H, 4.3; N, 8.6. $C_{20}H_{14}N_2O_3$ requires C, 72.7; H, 4.3; N, 8.5%); $\nu_{max.}$ (Nujol) 1665

(CO) and 1330 cm^{-1} (NO); *m/z* 330 (M^+, 6%), 269 (25), 159 (29), 155 (52), 143 (88), 127 (100), 115 (21), 102 (36), 77 (26), 75 (20), and 51 (21). *3-Methyl-2-(2-thenoyl)quinoxaline 1,4-dioxide* (**7b**) (4.55 g, 65%), m.p. 202-203°C (decomp.) (Found: C, 58.8; H, 3.3; N, 9.6. $C_{14}H_{10}N_2O_3S$ requires C, 58.4; H, 3.5; N, 9.8%); $\nu_{max.}$ (Nujol) 1640 (CO) and 1340 cm^{-1} (NO); δ (DMSO-d_6) 2.4 (3H, s, Me), 7.18-7.28 (1H, m, ArH), 7.85-8.3 (4H, m, ArH), and 8.35-8.7 (2H, m, ArH); *m/z* 286 (M^+, 14%), 253 (28), 187 (36), 159 (65), 143 (100), 111 (96), 102 (52), 90 (58), 83 (32), 76 (34), 75 (29), 55 (65), and 51 (40). *3-Methyl-2-nicotinoylquinoxaline 1,4-dioxide* (**7e**) (4.2 g, 60%), m.p. 208-209°C (decomp.) (Found:, C, 63.8; H, 3.9; N, 14.75. $C_{15}H_{11}N_3O_3$ requires C, 64.05; H, 3.9; N, 14.9%); $\nu_{max.}$ (KBr) 1665 (CO) and 1330 cm^{-1} (NO); δ ($CDCl_3$) 2.5 (3H, s, Me), 7.48 (1H, dd, *J* 5 and 8 Hz, ArH), 7.75-8.05 (2H, m, ArH), 8.14-8.24 (1H, m, ArH), 8.4-8.8 (2H, m, ArH), 8.8-8.9 (1H, m, ArH), and 9.0-9.05 (1H, m, ArH); *m/z* 281 (M^+, 30%), 266 (26), 250 (27), 220 (32), 219 (25), 159 (25), 143 (23), 106 (25), 102 (22), 90 (24), 78 (100), 51 (42), and 43 (33).

Quinoxaline 1,4-dioxides **7c** and **8c** were isolated by chromatographic separation of the crude solid product over silica gel, eluting with suitable mixtures of toluene and ethyl acetate. Quinoxaline 1,4-dioxides **7d** and **8d** were obtained from the crude solid product by preparative t.l.c. (using Merck silica gel G), eluting with toluene/ethyl acetate (55:45).

2-(2-Furoyl)-3-methylquinoxaline 1,4-dioxide (**7c**) (3.5 g, 52%), m.p. 196-198°C (decomp.) (Found: C, 62.1; H, 3.7; N, 10.2. $C_{14}H_{10}N_2O_4$ requires C, 62.2; H, 3.7; N, 10.4%); $\nu_{max.}$ (Nujol) 1650 (CO) and 1330 cm^{-1} (NO); δ (DMSO-d_6) 2.35 (3H, s, Me), 6.84 (1H, dd *J* 2 and 4 Hz, furoyl 4-H), 7.8 (1H, dd *J* ca. 1 and 4 Hz, furoyl 3-H), 7.9-8.15 (2H, m, ArH), 8.2-8.5 (1H, m, furoyl 5-H), and 8.35-8.65 (2H, m, ArH); *m/z* 270 (M^+, 5%), 187 (52), 159 (100), 143 (56), 102 (37), 95 (46), 90 (58), 76 (22), 51 (20), and 43 (55). *2-Acetyl-3-(2-furyl)quinoxaline 1,4-dioxide* (**8c**) (0.52 g, 8%), m.p. 179-180°C (decomp.) (Found: M^+ 270.0615. $C_{14}H_{10}N_2O_4$ requires M^+ 270.0640); $\nu_{max.}$ (Nujol) 1720 (CO) and 1340 cm^{-1} (NO); δ (DMSO-d_6) 2.72 (3H, s, Me), 6.73-6.93 (1H, m, furyl 4-H), 7.79-8.22 (4H, m, ArH), and 8.22-8.65 (2H, m, ArH); *m/z* 270 (M^+, 50%), 253 (25), 237 (26), 236 (61), 209 (33), 129 (35), 107 (82), 102 (55), 95 (45), 90 (31), 79 (42), 77 (34), 76 (68), 75 (42), 69 (48), 64 (32), 63 (46), 55 (57), 53 (23), 52 (23), 51 (100), and 50 (69). *3-Methyl-2-(2-pyridinecarbonyl)quinoxaline 1,4-dioxide* (**7d**) (2.3 g, 33%), m.p. 202-203°C (decomp.) (Found: M^+, 281.0781. $C_{15}H_{11}N_3O_3$ requires M^+ 281.0800); $\nu_{max.}$ (KBr) 1685 (CO) and 1330 cm^{-1} (NO); δ ($CDCl_3$) 2.52 (3H, s, Me) and 7.27-8.77 (8H, m, ArH); *m/z* 281 (M^+, 15%), 264 (25), 248 (40), 247 (96), 102 (30), 90 (33), 78 (100), 76 (28), 52 (20), 51 (53), and 50 (28). *2-Acetyl-3-(2-pyridyl)quinoxaline 1,4-dioxide* (**8d**) (1.0 g, 14%), m.p. 165-167°C (decomp.) (Found: C, 63.8; H, 3.8; N, 14.6. $C_{15}H_{11}N_3O_3$ requires C, 64.05; H, 3.9; N, 14.9%); $\nu_{max.}$ (KBr) 1710 (CO) and 1340 cm^{-1} (NO); δ (DMSO-d_6) 2.86 (3H, s, Me), 7.25-7.5 (1H, m, ArH), 7.75-8.05 (3H, m, ArH), and 8.5-8.8 (4H, m, ArH); *m/z* 281 (M^+, 17%), 266 (60), 250 (46), 79 (22), 78 (100), 51 (23) and 43 (23).

Deoxygenation of Quinoxaline 1,4-Dioxides **7**. - A concentrated aqueous solution of sodium dithionite (10 mmol) was added in portions to a solution of quinoxaline 1,4-dioxide **7** (2 mmol) in the minimum amount

of dimethylformamide (8-15 ml) heated on a water bath. The reaction was complete after a few minutes, when the solution has acquired a deep purple-blue colour which persists for several min. Sufficient water was added to precipitate the product **3** and the mixture was kept in the dark at ca. 4°C for 18 h. The product was collected and crystallised from methanol or aqueous methanol, avoiding exposure of the solution to direct bright daylight. Quinoxalines **3** prepared are as follows.

3-*Methyl*-2-(2-*naphthoyl*)*quinoxaline* (**3a**) (0.48 g, 80%), m.p. 163-164°C (Found: C, 80.7; H, 4.5; N, 9.6. $C_{20}H_{14}N_2O$ requires C, 80.5; H, 4.7; N, 9.4%); $\nu_{max.}$ (Nujol) 1650 cm^{-1} (CO); δ ($CDCl_3$) 2.82 (3H, s, Me) and 7.4-8.4 (11H, m, ArH); *m/z* 298 (M^+, 42%), 270 (15), 269 (24), 155 (100), 127 (81), 102 (19), 77 (10), 75 (11), 69 (20), and 51 (11). 3-*Methyl*-2-(2-*thenoyl*)*quinoxaline* (**3b**) (0.4 g, 79%), m.p. 138-139°C (Found: C, 66.4; H, 3.85; N, 10.9. $C_{14}H_{10}N_2OS$ requires C, 66.1; H, 4.0; N, 11.0%); $\nu_{max.}$ (Nujol) 1635 cm^{-1} (CO); δ ($CDCl_3$) 2.92 (3H, s, Me), 7.1-7.25 (1H, m, ArH), and 7.6-8.3 (6H, m, ArH); *m/z* 254 (M^+, 47%), 225 (23), 143 (17), 111 (100), 102 (37), 76 (17), 75 (19), and 51 (18). 3-*Methyl*-2-(2-*furoyl*)*quinoxaline* (**3c**) (0.355 g, 74%), m.p. 164-166°C (Found: C, 70.5; H, 4.3; N, 11.8. $C_{14}H_{10}N_2O_2$ requires C, 70.6; H, 4.2; N, 11.8%); $\nu_{max.}$ (KBr) 1640 cm^{-1} (CO); δ ($CDCl_3$) 2.9 (3H, s, Me), 6.63 (1H, dd *J* 2 and 4 Hz, furoyl 4-H), 7.46 (1H, dd, *J* ca. 1 and 4 Hz, furoyl 3-H), 7.7-8.0 (3H, m, ArH), and 8.0-8.25 (2H, m, ArH); *m/z* 238 (M^+, 77%), 210 (50), 209 (31), 182 (13), 181 (32), 143 (45), 117 (45), 102 (72), 95 (100), 76 (30), 75 (24), and 51 (22). 3-*Methyl*-2-*nicotinoylquinoxaline* (**3e**) (0.28 g, 56%), m.p. 122-123°C (Found: C, 72.05; H, 4.4; N, 16.7. $C_{15}H_{11}N_3O$ requires C, 72.3; H, 4.45; N, 16.9%); $\nu_{max.}$ (KBr) 1655 cm^{-1} (CO); δ ($CDCl_3$) 2.89 (3H, s, Me) and 7.3-9.3 (8H, m, ArH); *m/z* 249 (M^+, 82%), 248 (46), 221 (33), 220 (100), 143 (44), 106 (90), 102 (70), 78 (70), and 51 (41). 2-(4-*Methoxybenzoyl*)-3-*methylquinoxaline* (**3f**) (0.31 g, 56%), m.p. 113-114°C (Found: C, 73.4; H, 5.1; N, 10.1. $C_{17}H_{14}N_2O_2$ requires C, 73.0; H, 5.0; N, 10.0%); $\nu_{max.}$ (KBr) 1645 cm^{-1} (CO); δ ($CDCl_3$) 2.77 (3H, s, Me), 3.87 (3H, s, OMe), 6.96 (2H, half of A_2B_2 m, ArH), and 7.7-8.25 (6H, m, ArH); *m/z* 278 (M^+, 23%), 143 (8), 136 (10), 135 (100), 107 (8), 102 (15), 92 (19), 77 (26), 76 (10), 75 (9), 64 (10) and 51 (8).

Deoxygenation of 2-*Benzoyl*-3-*methylquinoxaline* 1,4-*Dioxide* (**12**).[6] - (a) A concentrated aqueous solution of sodium dithionite (6.75 g) was added in portions to a solution of 2-benzoyl-3-methyquinoxaline 1,4-dioxide[3] (1.0 g) in dimethylformamide (25 ml) and water (5 ml) at 120°C. After 10 min. the solution was cooled and water was added to precipitate the product. The mixture was kept at ca. 4°C for 18 h. Filtration gave 2-benzoyl-3-methylquinoxaline (**13**) (0.41 g, 46%), m.p. 89.5°C (after crystallisation from aqueous methanol, lit.[13] m.p. 88-89°C). (b) 2-Benzoyl-3-methylquinoxaline (47%) was obtained using the above procedure, but with dimethyl sulphoxide as the solvent.

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