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Oxidation of 1-acyl-2-naphthol oximes: *peri*- and *o*-cyclisation and spiro cyclodimerisation of naphthoquinone nitrosomethide intermediates

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Abstract—The oxidation of 2-hydroxynaphthaldehyde oxime with lead(IV) acetate (LTA) gave a mixture of naphtho[1,8-*de*][1,2]oxazine and a spiro dimer. LTA oxidation of 6-bromo (or nitro)-2-hydroxynaphthaldehyde oximes provided only spiro dimers. Similar treatment of (2-hydroxy-1-naphthyl)keto oximes with LTA gave naphtho[1,8-*de*][1,2]oxazines and benzo[*cd*]indol-3(1*H*)-ones. Low temperature oxidation of 1-(2-hydroxy-1-naphthyl)propan-1-one oxime furnished 2-ethylbenzo[*cd*]indol-3(1*H*)-one and 1-ethylnaphtho[1,2-*d*]isoxazole-2-oxide. *peri*- and *o*-Naphthoquinone nitrosomethides are invoked as intermediates that undergo *peri*- and *o*-cyclisation and intermolecular cyclodimerisation.

Compounds that incorporate the 1,2,5-oxadiazole nucleus have been reported to possess a wide-range of interesting and varying biochemical and pharmacological properties. They are useful herbicides, plant-growth regulators and pesticides, and in analytical chemistry fluorigenic reagents. Certain nitro-furazans and furoxans can act as propellants and explosives.[1] Monocyclic and fused isoxazoles have found diverse applications in medicine and agriculture. Some derivatives are also used in batteries, photographic emulsion and fibre dyes.[2]

1. Results and discussion

In a recent communication, we described the simultaneous *o*- and *peri*-cyclisation of 2-hydroxy-1-naphthaldehyde oxime **1** with lead(IV) acetate (LTA) that gave a mixture of the isomeric naphtho[1,8-*de*][1,2]oxazine **2** and naphtho[1,2-*d*]isoxazole-2-oxide **7** (Scheme 1).[3] In order to study the chemistry of compounds **2** and **7** scaled-up preparations were required and so the reaction was repeated several times. Thorough chromatographic analysis in all cases revealed, each time, a mixture of two major components. The compounds isolated were naphtho[1,8-*de*][1,2]-oxazine **2** and spiro{naphthalene-1(2*H*),4′-(naphtho[2′,1′:2,3]-pyrano[4,5-*c*]furazan)}-2-one-11′-oxide **3** in 17 and 14% yield on average, respectively. Compound **2** was identical in all respects to the respective compound isolated initially.[3] Compound **3** was fully characterised by ^{1}H and ^{13}C NMR spectroscopy, mass spectrometry and with the aid of DQF-COSY, DEPT, HMQC and HMBC spectra. Furthermore the structure of **3** was unambiguously assigned by X-ray crystallography (Fig. 1).

Some characteristic structural features of **3** are given below. The tetracyclic system and the bicyclic system are almost planar with the sp^3-hybridised carbon deviating from the mean plane by 0.2 Å from the former and by 0.1 Å from the latter. The angles between the mean planes of the two ring systems are 94°. The arrangement of atoms around the sp^3-hybridised carbon is that of a distorted tetrahedron. The three sp^3–sp^2 bond distances are typical of single bonds. The C22–C13–C14 bond angle of 114.7° is slightly larger than 109° due to the extended conjugated system. For the same reason the C21–C22 bond distance of 1.431 (3) Å and C19–C20 bond distance of 1.447 (3) Å have partial double bond character. Also the C10–O2 bond distance of 1.373 (2) Å is, as expected, shorter than a typical C–O single bond. The bond lengths and angles of the furoxan ring are similar to those observed in the non-fused furoxan ring.[1b,e] An intramolecular hydrogen bond of the type C2–H2···N1 is probably formed as evidenced by the C2···N1 bond distance of 2.981 (2) Å, the H2···N1 bond distance of 2.281 (5) Å and a C2–H2···N1 bond angle of 130°.

Keywords: naphthalenes; oximes; oxidation; *N*-oxides; spiro compounds.
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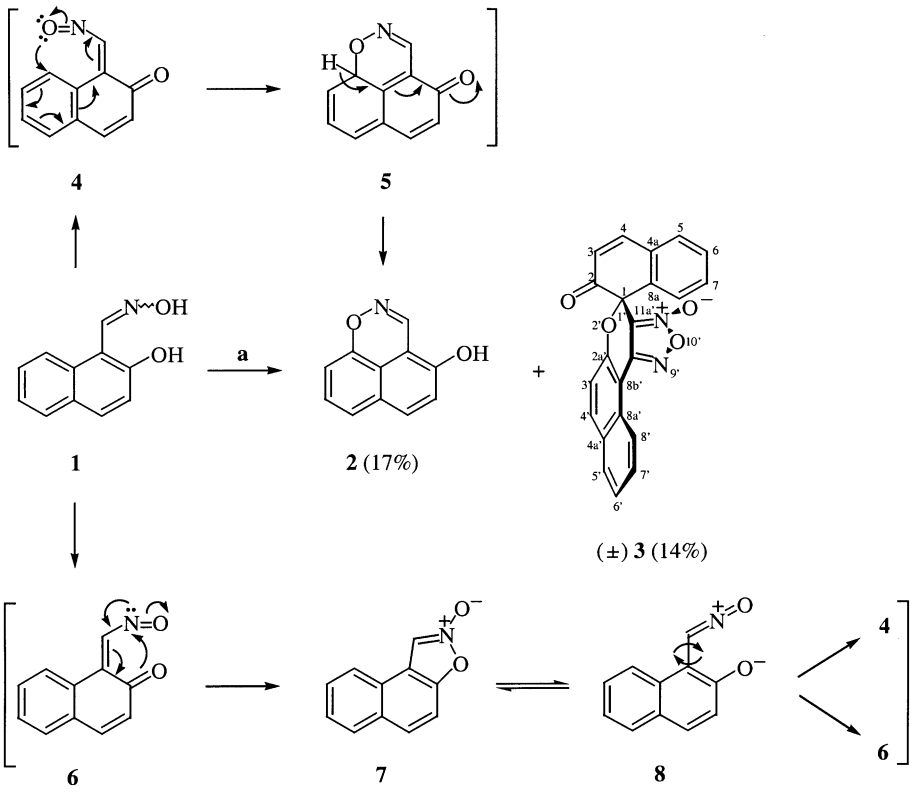


Scheme 1. *Reagents*: (a) $Pb(OAc)_4$, THF, 0°C 2 h, room temperature 12 h.

According to our earlier proposal, compounds **2** and **7** originate from an organolead intermediate formed from the reaction of **1** with LTA. The intermediate decomposes into the corresponding *peri*- and *o*-naphthoquinone nitrosomethides **4** and **6**, which then undergo *peri*- and *o*-cyclisation, respectively.[3] The new findings suggest that the *N*-oxide **7** appears to be very sensitive and quite unstable to the reaction conditions. A similar difficulty was encountered in the synthesis of the benzo analogue of **7**.[4] Furthermore, spiro compound **3** is a dimer of naphthoquinone nitrosomethides **4/6**. An analogous dimer has been synthesised from in situ generated *o*-benzoquinone methide.[5] Taking these facts into consideration it was imperative to explain the formation of **3** and the instability of **7**.

To this end, aldoximes **10** and **14** and ketoximes **21**, **25** and **31** were selected as appropriate precursors. Oximes **10** and **14** were prepared from the corresponding aldehydes **9**[6] and **12**[7] by standard methods. Aldehyde **12** and 2-hydroxy-1,6-dinitronaphthalene **13** were obtained as a mixture by treating 2-hydroxy-1-naphthaldehyde **1** with 85% nitric acid. Column chromatography of the mixture gave **12** and **13** in 46 and 50% yield, respectively. Oxidation of oximes **10** and **14** with LTA, using the standard procedure of stirring first at 0°C for 2 h and then at room temperature for 12 h, provided the corresponding spiro dimers **11** and **15** in 25% yield, in each case. No traces of any other products were detected in these two reactions (Scheme 2). All these compounds were characterised by ^1H and ^{13}C NMR spectroscopy, low-resolution mass spectrometry, HRMS measurements or elemental analyses, and by comparison with the spectra of related compounds. Furthermore, the carbon atoms of spiro dimer

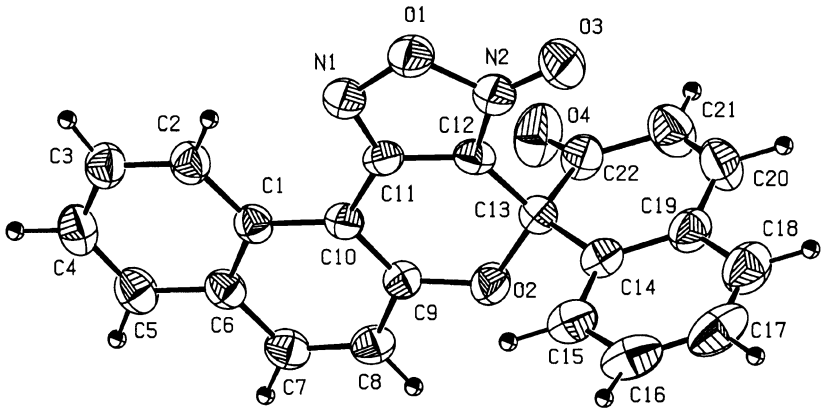
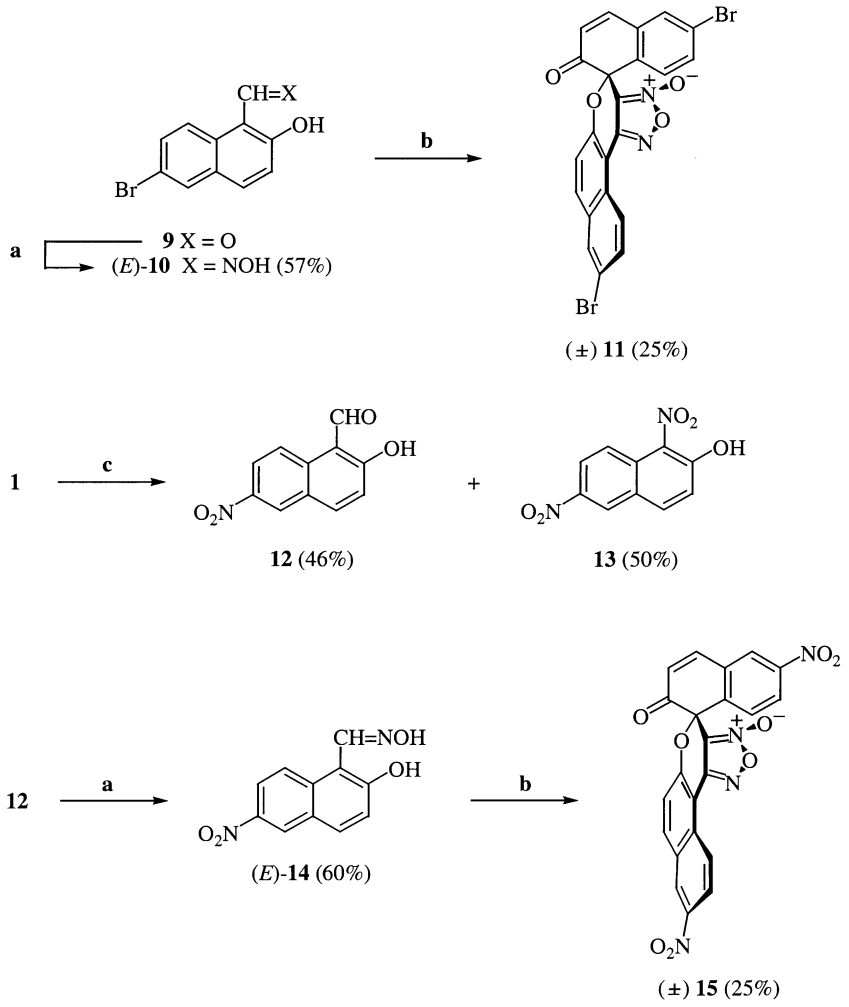


Figure 1. ORTEP diagram of molecule **3** (thermal ellipsoids at 40% probability).

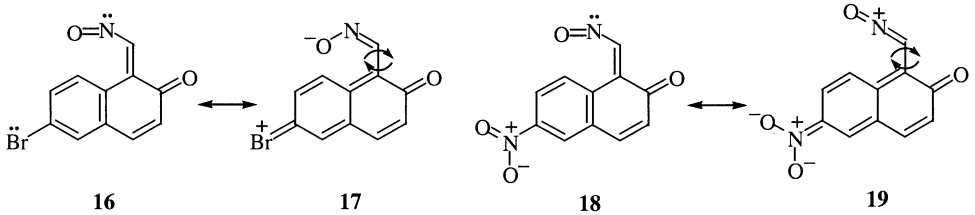


Scheme 2. *Reagents*: (a) $H_2NOH{\cdot}HCl$, pyridine, *n*-butanol, reflux, 1 h; (b) $Pb(OAc)_4$, THF, 0°C 2 h, room temperature 12 h; (c) 85% HNO_3, −5°C 20 min.

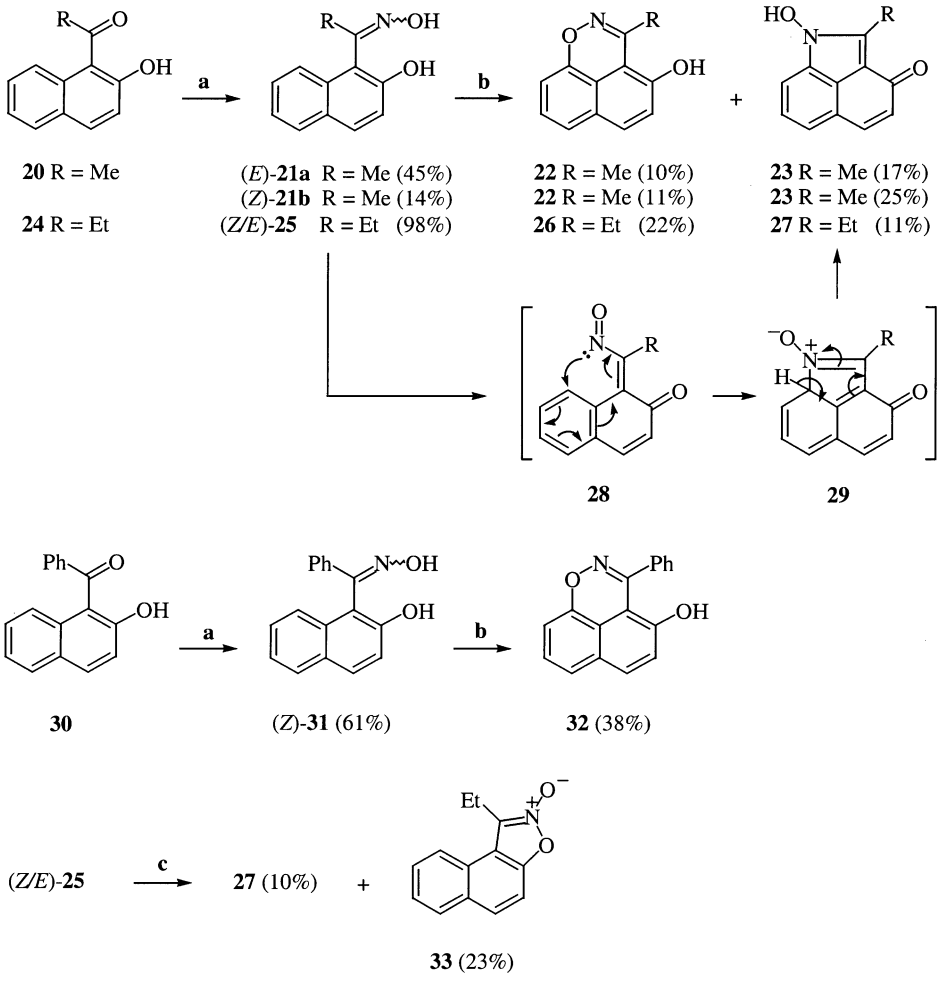
11 were fully assigned with the aid of DQF-COSY, DEPT, HMQC and HMBC spectra. Characteristic features in the ^1H and ^{13}C NMR spectra of spiro dimers **3**, **11** and **15** are the following. The *J* values 10.0, 10.1 and 10.2 Hz of the α,β-unsaturated protons, the downfield H-8′ protons at δ 8.96, 8.81 and 8.98, the sp^3-hybridised carbon atoms at δ 76.46, 76.21 and 77.28, and the carbonyl carbon atoms at δ 190.96, 190.30 and 189.64, respectively.

Oxidation of oximes **10** and **14** did not furnish the corresponding naphtho[1,8-*de*][1,2]oxazines. This can be adequately explained by the conjugative effects of the substituents at C-6 of the naphthalene nucleus. Thus it appears that *peri*-cyclisation is not likely through contribution of resonance structures **17** and **19**, which allow free rotation of the N═O moiety (Scheme 3).

Ketoximes **21**, **25** and **31** were synthesised by reacting the corresponding ketones **20**, **24** and **30**[8] with hydroxylamine hydrochloride. The reaction of **20** by this method resulted in the isolation of two isomeric products *E*-ketoxime **21a** (45%) and *Z*-ketoxime **21b** (14%) (Scheme 4). Vijayakumar et al.[9] have reported the synthesis of oxime **21** as an isomer mixture. The assignment of **21a** to the *E*-isomer was confirmed by NMR spectroscopy. The sharp exchangeable peak at δ 9.79 is characteristic of a hydrogen-bonded proton. Hydrogen bonding can occur between the hydroxyl proton of naphthol and the lone-pair of electrons of the nitrogen atom. In the NOESY spectrum of **21a** there is a cross peak between the methyl group at δ 2.14 and H-8 at δ 7.76, whereas no such cross peak is seen in the corresponding spectrum of **21b**. In the ^1H NMR spectrum of **21b** the chemical shift of H-8 is at δ 7.37. The deprotection of H-8 in



Scheme 3.



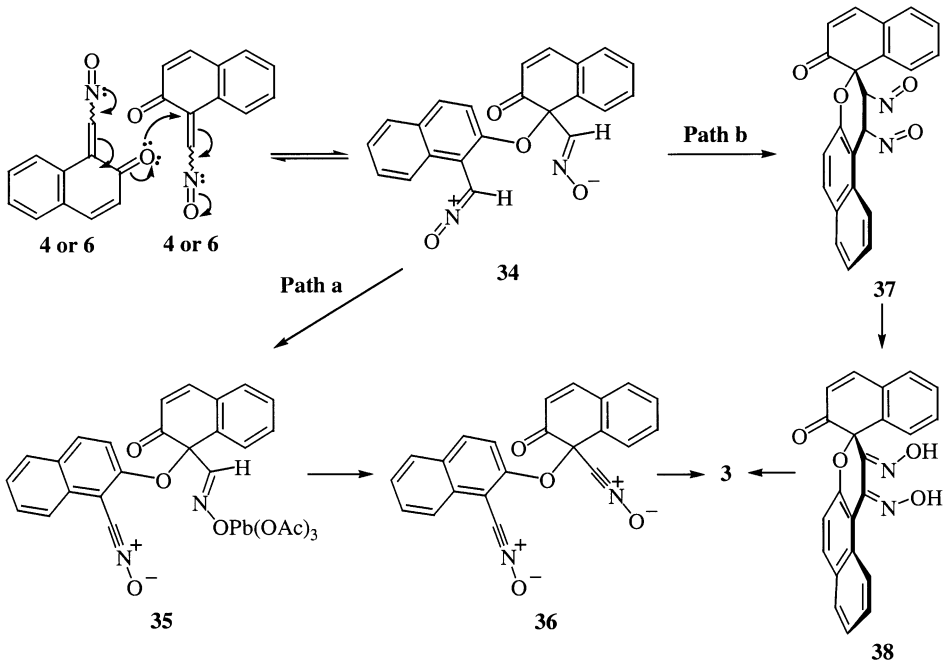
Scheme 4. *Reagents*: (a) $H_2NOH{\cdot}HCl$, aq. $MeCO_2Na$, *n*-butanol, reflux, 2 h; (b) $Pb(OAc)_4$, THF, 0°C 2 h, room temperature 12 h; (c) $Pb(OAc)_4$, THF, 0°C 2 h.

21a is therefore most likely due to steric hindrance from the methyl group. In *E*-isomer **21a** the methyl group is in the vicinity of H-8 because of the six-membered ring formed by hydrogen bonding. This six-membered ring is co-planar with the naphthol ring.

Treatment of ketoximes **21a**, **21b** or **25** with LTA by the standard procedure furnished oxazines **22** or **26** and benzo[*cd*]indol-3(1*H*)-ones **23** or **27**, respectively (Scheme 4). It is proposed that **23/27** originate from *peri*-attack by the nitrogen atom of the nitroso group of **28** to give **29**. The latter tautomerises into **23/27**. An analogous dual behaviour by a nitroso group was reported for the intramolecular cyclisation of nitroso intermediates, formed from *N*-aryl-*S*,*S*-dimethylsulfimides and benzonitrile oxides, that gave benzimidazole-3-oxides and benzoxadiazines.[10] A compound closely related to **23/27** is 1,2,5-trimethylbenzo[*cd*]-indol-3(1*H*)-one, which has been synthesised by heating 4-(1,2-dimethyl-1*H*-indol-3-yl)butan-2-one in PPA.[11] Oxidation of *Z*-oxime **31** with LTA gave oxazine **32** in 38% yield. When the reaction of **25** with LTA was stopped after 2 h stirring at 0°C benzo[*cd*]indol-3(1*H*)-one **27** (10%) and isoxazole-*N*-oxide **33** (23%) were isolated. The last reaction provides direct evidence that oxazine **26** is formed from isoxazole-*N*-oxide **33**. Reaction of **33** with LTA for 12 h at room temperature afforded **26** in near quantitative yield. A most likely route proposed for this transformation is ring opening of the isoxazole-*N*-oxide ring to give intermediate *peri*- and/or *o*-naphthoquinone nitrosomethides as shown for isoxazole-*N*-oxide **7** in Scheme 1. Cyclisation of the appropriate *peri*-naphthoquinone nitrosomethide would then give the more stable oxazine **32**.

All compounds in Scheme 4 were satisfactorily characterised by ^1H and ^{13}C NMR spectroscopy, low-resolution mass spectrometry and from HRMS measurements or elemental analyses. The carbon atoms in compounds **22**, **23** and **27** were fully assigned from their DQF-COSY, DEPT, HMQC and HMBC spectra. Characteristic features in the ^1H and ^{13}C NMR spectra of oxazines **2**,[3] **22**, **26** and **32** are the following. The protons of the 4-OH groups at δ 10.61, 10.39, 10.50 and 10.10, C-4 at δ 148.44, 149.97, 149.28 and 149.95, and C-9a at δ 150.72, 151.07, 151.10 and 150.97, respectively. On the other hand, the characteristic features in the ^1H and ^{13}C NMR spectra of benzo-[*cd*]indol-3(1*H*)-ones **23** and **27** are the *J* value 9.6 Hz of the α,β-unsaturated protons for both compounds and the carbonyl carbon atoms at δ 178.02 and 177.50, respectively.

The results in Scheme 4 fit very well into our initial proposal regarding naphthoquinone nitrosomethides intermediates **4** and **6** undergoing *peri*- and *o*-cyclisation and explain adequately the instability of isoxazole-*N*-oxide **7**.[4] Furthermore, oxidation of oximes *E*-**21a** and *Z*-**21b** confirms that



Scheme 5.

these cyclisation modes occur regardless of the *E*- or *Z*-oxime configuration.

Based on these experimental observations and according to molecular modelling studies, it is suggested that the formation of spiro dimer **3** is the result of intermolecular cyclodimerisation of **4**, **6** or/and **4** and **6**, as depicted by paths *a* and *b* (Scheme 5). The reaction is chemoselective in that the nitroso group can activate the exocyclic double bond. Addition of carbonyl oxygen of a second molecule gives intermediate oxime **34**. This initial step may be reversible. Intermediate **34** may react with LTA[12] to give the organolead complex **35** (path *a*), which is then converted to the dinitrile oxide **36**, ultimately leading to product **3** via carbon–carbon and nitrogen–oxygen bond formation. Alternatively (path *b*) intermediate **34** undergoes intramolecular cyclisation by carbon–carbon bond formation to furnish dinitroso pyran **37**. The latter tautomerises to dioxime **38** which oxidatively cyclises to **3**. It is interesting to note that LTA is involved in both rationales either prior to or after carbon–carbon bond formation. Both dimerisation of nitrile oxides[1] and oxidation of 1,2-dioximes[1] are well-documented precursors of 1,2,5-oxadiazole-*N*-oxides (furoxans). In the light of the evidence at hand, alternative plausible pathways involving free radical mechanisms or other organolead species cannot be ruled out.

The mechanism of Scheme 5 takes into account the fact that no spiro compound was detected after the oxidation of ketoximes *E*-**21a**, *Z*-**21b**, (*Z*/*E*)-**25** and (*Z*)-**31**.

2. Conclusion

In summary, it has been observed that the oxidation of 2-hydroxy-1-naphthaldehyde oximes leads to novel spiro compounds while the parent oxime also produced 4-hydroxynaphtho[1,8-*de*][1,2]oxazine. The spiro compounds are dimers of the proposed intermediate *peri*- and/or *o*-naphthoquinone nitrosomethides. The oxidation of (2-hydroxy-1-naphthyl)ethan-1-one(or propan-1-one) oximes at ambient temperature afforded benzo[*cd*]indol-3(1*H*)-ones and naphtho[1,8-*de*][1,2]oxazines, products of *peri*-intramolecular cyclisation of appropriate naphthoquinone nitrosomethides. In contrast, oxidation of 1-(2-hydroxy-1-naphthyl)(phenyl)methanone oxime gave only 4-hydroxy-3-phenylnaphtho[1,8-*de*][1,2]oxazine. Evidence for the instability of naphtho[1,2-*d*]isoxazole-2-oxides in solution is provided both directly and indirectly.

3. Experimental

3.1. General methods

Melting points were taken on a Büchi 510 apparatus and are uncorrected. Infrared spectra were recorded on a Perkin–Elmer 257 spectrometer, as Nujol mulls between sodium chloride discs. Elemental analyses were performed on a Carlo Erba 1106 elemental analyser. Nuclear magnetic resonance spectra were measured at 300 MHz on a Brüker AC 300 spectrometer or at 400 MHz on a Brüker AMX 400 spectrometer using tetramethylsilane as internal standard. Mass spectra were obtained by use of Finnigan 4500 (low resolution) or JEOL JMS-AX 505W (high resolution) instruments using EI. Analytical TLC was carried out on Fluka silica gel 60 F_{254}. Preparative flash chromatography was carried out using Merck 9385 silica gel. Solvents and reagents were used as received from the manufacturers except for dichloromethane, ethanol, ethyl acetate, hexane and methanol, that were purified and dried according to recommended procedures.[13]

3.1.1. Nitration of 2-hydroxy-1-naphthaldehyde. To an

aqueous solution of 85% nitric acid (10 mL) at −5°C **1** (1 g, 6 mmol) was added in small portions during 10 min. After the addition was complete stirring was continued for another 10 min at −5°C. The mixture was poured into ice water and the precipitate was filtered, washed with water and dried. The residue was subjected to column chromatography (20% ethyl acetate/hexane) to give two fractions. The first fraction gave **2-hydroxy-6-nitro-1-naphthaldehyde 12** and the second fraction gave **1,6-dinitro-2-naphthol 13**.

3.1.2. 2-Hydroxy-6-nitro-1-naphthaldehyde (12). 0.6 g, 46% as pale yellow powder, mp 235–237°C (lit.[7] mp 239°C); ν_{max} (Nujol) 1640, 1550, 1350 cm^{-1}; δ_H (400 MHz; DMSO-d_6) 7.43 (1H, d, *J*=9.1 Hz, H-3), 8.32 (1H, d, *J*=9.1 Hz, H-7), 8.43 (1H, d, *J*=9.1 Hz, H-8), 8.91 (1H, s, H-5), 9.17 (1H, d, *J*=9.1 Hz, H-4), 10.77 (1H, s, CHO), 12.21 (1H, s, br, OH); *m/z* (EI) 217 (100, M^+), 171 (31), 115.1 (67), 63 (36%); HRMS (EI): M^+, found 217.0248 $C_{11}H_7NO_4$ requires 217.0375.

3.1.3. 1,6-Dinitro-2-naphthol (13). 0.7 g, 50% as pale yellow powder, mp 190–193°C; ν_{max} (Nujol) 3330, 1535, 1345 cm^{-1}; δ_H (400 MHz; DMSO-d_6) 7.51 (1H, d, *J*=9.1 Hz, H-3), 7.76 (1H, d, *J*=9.3 Hz, H-8), 8.32 (1H, d, *J*=9.3 Hz, H-7), 8.37 (1H, d, *J*=9.1 Hz, H-4), 9.02 (1H, s, H-5), 12.26 (1H, s, br, OH); δ_C (100 MHz, DMSO-d_6) 120.74, 121.29, 122.29, 125.23, 125.29, 128.26, 133.08, 134.87, 143.48, 151.43; *m/z* (EI) 234 (100, M^+), 189 (41), 114 (69), 102 (33%); HRMS (EI): M^+, found 234.0279. $C_{10}H_6N_2O_5$ requires 234.0276.

3.2. Reaction of 6-bromo-2-hydroxy-1-naphthaldehyde and 2-hydroxy-6-nitro-1-naphthaldehyde with hydroxylamine hydrochloride:[9] general procedure A

A mixture of **9**[6] or **12**[7] (2 g, 8 mmol) and hydroxylamine hydrochloride (3 g, 40 mmol) in *n*-butanol (70 mL) containing pyridine (3 mL) was heated under reflux for 1 h. The solvents were removed in vacuo and to the residue was added cold water (150 mL). The resulting suspension was filtered, washed with water and dried to give either **6-bromo-2-hydroxy-1-naphthaldehyde oxime 10** or **2-hydroxy-6-nitro-1-naphthaldehyde oxime 14**.

3.2.1. (*E*)-6-Bromo-2-hydroxy-1-naphthaldehyde oxime (10). 1.2 g, 57% as pale green needles (toluene), mp 244–247°C; ν_{max} (Nujol) 3310, 1630 cm^{-1}; δ_H (400 MHz; DMSO-d_6) 7.24 (1H, d, *J*=9.0 Hz, H-3), 7.60 (1H, d, *J*=9.0 Hz, H-7), 7.83 (1H, d, *J*=9.0 Hz, H-8), 8.10 (1H, s, H-5), 8.49 (1H, d, *J*=9.0 Hz, H-4), 8.96 (1H, s, CH=N), 11.11 (1H, s, OH), 11.53 (1H, s, NOH); δ_C (100 MHz, DMSO-d_6) 109.52, 116.44, 119.83, 125.87, 129.72, 130.26 (2C), 130.50, 130.96, 147.25, 156.58; *m/z* (EI) 265 (11, M^+), 249 (100), 219 (8) 194 (9), 170 (21), 140 (25), 113 (32), 87 (9), 63 (12%); HRMS (EI): M^+, found 264.9727. $C_{11}H_8BrNO_4$ requires 264.9738.

3.2.2. (*E*)-2-Hydroxy-6-nitro-1-naphthaldehyde oxime (14). 1.12 g, 60% as yellow powder (propan-2-ol), mp 254–257°C; ν_{max} (Nujol) 3420, 1660 cm^{-1}; δ_H (250 MHz; DMSO-d_6) 7.39 (1H, d, *J*=9.0 Hz, H-3), 8.15–8.20 (2H, m, H-7, H-8), 8.76 (1H, d, *J*=9.0 Hz, H-4), 8.89 (1H, s, H-5) 8.94 (1H, s, CH=N) 11.57 (1H, s, OH) 11.64 (1H, s, NOH); δ_C (100 MHz, DMSO-d_6) 109.91, 120.20, 120.41, 125.02, 126.17, 126.53, 133.63, 134.45, 142.73, 146.43, 159.07; *m/z* (EI) 232 (100, M^+), 214 (51), 140 (68), 113 (40) 63 (18%); HRMS (EI): M^+, found 232.0478. $C_{11}H_8N_2O_4$ requires 232.0484.

3.3. Reaction of 1-(2-hydroxy-1-naphthyl)ethan-1-one, 1-(2-hydroxy-1-naphthyl)propan-1-one and (2-hydroxy-1-naphthyl)(phenyl)methan-1-one with hydroxylamine hydrochloride:[9] general procedure B

Hydroxylamine hydrochloride (3.1 g, 40 mmol) and sodium acetate (3.7 g, 40 mmol) were dissolved in water (10 mL) and added to a suspension of **20**, **24** or **30** (10 mmol) in *n*-butanol (70 mL). The resulting mixture was heated under reflux for 2 h. The solvent was removed in vacuo and to the residue was added cold water (150 mL) and extracted with dichloromethane (3×50 mL). The combined organic extracts were dried (Na_2SO_4), concentrated in vacuo, and the residue was purified by column chromatography (10, 20, 26, 50% ethyl acetate/hexane) to give in the first fraction **(*Z*)-1-(2-hydroxy-1-naphthyl)ethan-1-one oxime 21b** and in the second fraction **(*E*)-1-(2-hydroxy-1-naphthyl)ethan-1-one oxime 21a**, or chromatographed (20% ethyl acetate/hexane) to give **(*Z*)/(*E*)-1-(2-hydroxy-1-naphthyl)propan-1-one oxime 25** or recrystallised to give **(*Z*)-1-(2-hydroxy-1-naphthyl)(phenyl)-methanone oxime 31**.

3.3.1. (*E*)-1-(2-Hydroxy-1-naphthyl)ethan-1-one oxime (21a). 0.9 g, 45% as colourless powder, mp 130–132°C [Found: C, 71.60; H, 5.48; N, 6.98; $C_{12}H_{11}NO_2$ requires: C, 71.63; H, 5.51; N, 6.96%]; ν_{max} (Nujol) 3400, 3300, 1650 cm^{-1}; δ_H (360 MHz; DMSO-d_6) 2.14 (3H, s, Me), 7.21 (1H, d, *J*=8.9 Hz, H-3), 7.27 (1H, td, *J*=8.0, 1.2 Hz, H-7), 7.40 (1H, td, *J*=8.0, 1.2 Hz, H-6) 7.71 (1H, d, *J*=8.0 Hz, H-5), 7.76 (2H, m, H-4, H-8), 9.79 (1H, s, OH), 11.00 (1H, s, NOH); δ_C (90 MHz, DMSO-d_6) 14.07, 20.75, 116.82, 118.12, 127.57, 127.73 (2C), 130.47, 132.82, 150.68, 152.22, 152.57.

3.3.2. (*Z*)-1-(2-Hydroxy-1-naphthyl)ethan-1-one oxime (21b). 0.28 g, 14% as colourless powder, mp 140–141°C [Found: C, 71.59; H, 5.49; N, 7.01; $C_{12}H_{11}NO_2$ requires: C, 71.63; H, 5.51; N, 6.96%]; ν_{max} (Nujol) 3315, 1620 cm^{-1}; δ_H (360 MHz; DMSO-d_6) 2.11 (3H, s, Me), 7.19 (1H, d, *J*=8.8 Hz, H-3), 7.27 (1H, td, *J*=8.1, 1.5 Hz, H-7), 7.37–7.42 (2H, m, H-6, H-8), 7.74 (1H, d, *J*=8.8 Hz, H-4), 7.78 (1H, d, *J*=8.2 Hz, H-5), 9.73 (1H, s, br, OH), 10.27 (1H, s, br, NOH); δ_C (90 MHz, DMSO-d_6) 15.96, 20.48, 118.20, 122.57, 123.69, 124.10, 126.22, 126.33, 127.87 (2C), 128.96, 129.25.

3.3.3. (*Z*)/(*E*)-1-(2-Hydroxy-1-naphthyl)propan-1-one oxime (25). 2.1 g, 98% as colourless powder, mp 112–116°C [Found: C, 72.50; H, 6.12; N, 6.55; $C_{13}H_{13}NO_2$ requires: C, 72.54; H, 6.09; N, 6.51%]; ν_{max} (Nujol) 3320, 1620 cm^{-1}; δ_H [250 MHz, DMSO-d_6, data assigned from a mixture of (*E*)/(*Z*)=1:1, diastereomeric ratio] 1.38 (3H, t, *J*=7.6 Hz, CH_2CH_3, *Z*-isomer), 1.53 (3H, t, *J*=7.6 Hz, CH_2CH_3, *E*-isomer), 3.07 (2H, q, *J*=7.6 Hz, CH_2CH_3, *E*-isomer), 3.21 (2H, q, *J*=7.6 Hz, CH_2CH_3, *Z*-isomer), 7.68–7.94 (7H, m, benzenoid), 8.18–8.30 (5H, m,

benzenoid), 10.00–11.00 (3H, s, br, NOH, OH, *Z*-isomer, OH, *E*-isomer), 11.45 (1H, s, NOH, *E*-isomer); *m/z* (EI) 215 (43, M^+), 197 (35), 182 (28), 169 (100), 153 (8), 141 (17), 114 (25), 88 (6), 57 (8), 43 (27%).

3.3.4. (*Z*)-1-(2-Hydroxy-1-naphthyl)(phenyl)methanone oxime (31). 1.6 g, 61% as colourles powder (benzene/hexane), mp 205–206°C [Found: C, 77.58; H, 5.02; N, 5.29; $C_{17}H_{13}NO_2$ requires: C, 77.55; H, 4.98; N, 5.32%]; ν_{max} (Nujol) 3410, 1620 cm^{-1}; δ_H (250 MHz; DMSO-d_6) 7.24–7.44 (9H, m, benzenoid), 7.82–7.86 (2H, m, benzenoid), 9.97 (1H, s, br, OH), 11.29 (1H, s, br, NOH); δ_C (90 MHz; DMSO-d_6) 114.13, 118.26, 122.65, 123.89, 125.93 (2C), 126.42, 127.65, 127.99, 128.32, 128.56, 129.56, 131.69, 136.30, 151.70, 152.19, 152.37; *m/z* (EI) 263 (44, M^+), 245 (100), 231 (12), 217 (36), 202 (9), 140 (12), 114 (28), 103 (18), 78 (34), 51 (18), 44 (13%).

3.4. Oxidation of 2-hydroxy-1-naphthaldehyde oxime, (*E*)-6-bromo-2-hydroxy-1-naphthaldehyde oxime, (*E*)-2-hydroxy-6-nitro-1-naphthaldehyde oxime, (*E*)-1-(2-hydroxy-1-naphthyl)ethan-1-one oxime, (*Z*)-1-(2-hydroxy-1-naphthyl)ethan-1-one oxime, (*Z*/*E*)-1-(2-hydroxy-1-naphthyl)propan-1-one oxime or (*Z*)-1-(2-hydroxy-1-naphthyl)(phenyl)methanone oxime with lead(IV) acetate: general procedure

A stirred solution of **10**, **14**, **21a**, **21b**, **25** or **31** (5 mmol) in dry tetrahydrofuran (60 mL) under argon was cooled to 0°C. Lead(IV) acetate (4.43 g, 10 mmol) was added slowly over a period of 30 min. The mixture was stirred at this temperature for 1.5 h and then stirred for a further 12 h at room temperature. The reaction mixture was filtered, the solid was washed with tetrahydrofuran and the filtrate was evaporated in vacuo to leave a residue. The residue was chromatographed (25, 33, 50% dichloromethane/hexane, 33, 50% ethyl acetate/hexane) to give in the first fraction **(±)-spiro{naphthalene-1(2*H*),4′-(naphtho[2′,1′:2,3]pyrano-[4,5-*c*]furazan)}-2-one-11′-oxide 3** and in the second fraction **4-hydroxynaphtho[1,8-*de*][1,2]oxazine 2** or (25, 33%, dichloromethane/hexane) to give **(±)-6,6′-dibromospiro-{naphthalene-1(2*H*),4′-(naphtho[2′,1′:2,3]pyrano-[4,5-*c*]furazan)}-2-one-11′-oxide 11**, or (25, 33%, ethyl acetate/hexane) to give **(±)-6,6′-dinitrospiro{naphthalene-1(2*H*),4′-(naphtho-[2′,1′:2,3]pyrano[4,5-*c*]furazan)}-2-one-11′-oxide 15** or was triturated with acetonitrile and filtered to give **1-hydroxy-2-methyl-benzo[*cd*]indol-3(1*H*)-one 23**, the filtrate evaporated and the residue chromatographed (25% ethyl acetate/hexane) to give **4-hydroxy-3-methyl-naphtho[1,8-*de*][1,2]oxazine 22,** or chromatographed (25, 33, 50% ethyl acetate/hexane, ethyl acetate) to give **3-ethyl-4-hydroxynaphtho[1,8-*de*][1,2]oxazine 26 1-hydroxy-2-ethylbenzo[*cd*]indol-3(1*H*)-one 27** or was chromatographed (25% ethyl acetate/hexane) to give **4-hydroxy-3-phenylnaphtho[1,8-*de*][1,2]oxazine 32**.

3.4.1. 4-Hydroxynaphtho[1,8-*de*][1,2]oxazine (2). 0.15 g, 17% as pale green powder (benzene/hexane), mp 130–134°C (lit.[3] mp 130–134°C), identical in all respects to an authentic sample.

3.4.2. (±)-Spiro{naphthalene-1(2*H*),4′-(naphtho[2′,1′:2,3]-pyrano[4,5-*c*]furazan)}-2-one-11′-oxide (3). 0.23 g, 14% as yellow needles (acetonitrile), mp 211–214°C; ν_{max} (Nujol) 1670, 1220 cm^{-1}; δ_H (400 MHz; $CDCl_3$) 6.22 (1H, d, *J*=10.0 Hz, H-3), 7.23 (1H, d, *J*=9.0 Hz, H-3′), 7.46–7.56 (5H, m, H-5, H-6, H-7, H-8, H-6′), 7.60 (1H, d, *J*=10.0 Hz, H-4), 7.70 (1H, t, *J*=8.3 Hz, H-7′), 7.86 (1H, d, *J*=8.1 Hz, H-5′), 7.96 (1H, d, *J*=9.0 Hz, H-4′), 8.96 (1H, d, *J*=8.5 Hz, H-8′); δ_C (100 MHz; $CDCl_3$) 76.46 (C-1/1′), 104.02 (C-8b′), 108.92 (11a′), 118.00 (C-3′), 123.05 (C-3), 125.53 (C-6′), 125.70 (C-8′), 128.70 (C-5′), 128.78 (C-5), 129.17 (C-7′), 129.44, (C-8a′), 129.83 (C-4a′), 130.01 (C-8), 130.87, (C-4a), 130.92 (C-6), 131.03 (C-7), 134.06 (C-8a), 134.92 (C-4′), 146.54 (C-8c′), 147.24 (C-4), 155.17 (C-2a′), 190.96 (C-2); *m/z* (EI) 368 (73, M^+), 324 (27), 308 (30), 394 (41), 280 (63), 250 (100), 183 (63), 154 (26), 140 (30), 126 (45%); HRMS (EI): M^+, found 368.0795. $C_{22}H_{12}N_2O_4$ requires 368.0797.

Crystal data of (**3**). $C_{22}H_{12}N_2O_4$. *M*=368.34, monoclinic, space group $P2_1/c$, *a*=9.3953 (4) Å, *b*=13.9612 (5) Å, *c*=12.7471 (5) Å, β=90.945 (10)°, *U*=1671.81 (11) $Å^3$, *Z*=4, D_C=1.463 g cm^{-3}, μ=0.103 mm^{-1} (MoKα, λ=0.71073 Å), *T*=293 K. Of 7365 reflections measured on an Enraf–Nonius Kappa CCD diffractometer and corrected semiempirically for absorption, 3909 were unique (R_{int}=0.058). The structure was solved by direct methods and refined on F^2 values. Hydrogen atoms were located by difference Fourier maps and refined isotropically. *R*=0.0449 [*F* values, $I>2\sigma(I)$], *wR*2=0.1186 (F^2 values, all data), goodness-of-fit=0.882, final difference map extremes +0.145 and −0.184 e $Å^{-3}$. Software: SHELXS-97, SHELXL-97.

3.4.3. (±)-6,6′-Dibromospiro{naphthalene-1(2*H*),4′-(naphtho[2′,1′:2,3]pyrano[4,5-*c*]furazan)}-2-one-11′-oxide (11). 0.65 g, 25% as yellow powder, mp 243–245°C [Found: C, 50.19; H, 1.88; N, 5.35; $C_{22}H_{10}Br_2N_2O_4$ requires: C, 50.22; H, 1.92; N, 5.32%]; ν_{max} (Nujol) 1680, 1240 cm^{-1}; δ_H (400 MHz; $CDCl_3$) 6.25 (1H, d, *J*=10.1 Hz, H-3), 7.25 (1H, d, *J*=9.0 Hz, H-3′), 7.44 (1H, d, *J*=8.0 Hz, H-8), 7.53 (1H, d, *J*=10.1 Hz, H-4), 7.57–7.59 (2H, m, H-5, H-7), 7.76 (1H, d, *J*=9.0 Hz, H-7′), 7.87 (1H, d, *J*=9.0 Hz, H-4′), 8.02 (1H, s, H-5′), 8.81 (1H, d, *J*=9.0 Hz, H-8′); δ_C (100 MHz; $CDCl_3$) 76.21 (C-1/1′), 104.05 (C-8b′), 103.75 (C-11a′), 119.09 (C-3′), 119.56 (C-6′), 124.13 (C-3), 125.32 (C-6), 127.35 (C-8′), 127.88 (C-8a′), 130.18 (C-8), 130.69 (C-5′), 131.10 (C-4a′), 132.42 (C-7′), 132.65 (C-4a), 132.69 (C-5, C-8a), 133.80 (C-7), 133.86 (C-4′), 145.77 (C-4), 148.33 (C-8c′), 155.07 (C-2a′), 190.30 (C-2); *m/z* (EI) 524 (4, M^+), 508 (2), 440 (5), 371 (9), 286 (6), 249 (62), 217 (11), 165 (34), 140 (24), 113 (29) 44 (100%).

3.4.4. (±)-6,6′-Dinitrospiro{naphthalene-1(2*H*),4′-(naphtho[2′,1′:2,3]pyrano[4,5-*c*]furazan)}-2-one-11′-oxide (15). 0.58 g, 25% as yellow powder, mp 255–258°C [Found: C, 57.61; H, 2.17; N, 12.26; $C_{22}H_{10}N_4O_8$ requires: C, 57.65; H, 2.20; N, 12.22%]; ν_{max} (Nujol) 1690, 1230 cm^{-1}; δ_H (400 MHz; DMSO-d_6) 6.52 (1H, d, *J*=10.2 Hz, H-3), 7.64 (1H, d, *J*=9.1 Hz, H-3′), 8.16 (1H, d, *J*=10.2 Hz, H-4), 8.24 (1H, d, *J*=8.6 Hz, H-8), 8.38 (1H, dd, *J*=8.6, 2.3 Hz, H-7), 8.52–8.66 (3H, m, H-5, H-4′, H-7′), 8.98 (1H, d, *J*=9.3 Hz, H-8′), 9.16 (1H, d, *J*=2.4 Hz, H-5′); δ_C (100 MHz; DMSO-d_6) 77.28, 104.09,

118.50, 120.39, 122.74, 124.44 (3C), 125.26 (3C), 125.56, 126.29 (2C), 130.94, 131.26, 132.84, 137.40, 146.03, 149.55, 157.41 (C-2a′), 189.64 (C-2); *m/z* (EI) 458 (37, M^+), 442 (7), 414 (45), 384 (20), 353 (22), 324 (40), 279 (41), 239 (22), 230 (11), 214 (22), 165 (21), 98 (22), 44 (100%).

3.4.5. 4-Hydroxy-3-methylnaphtho[1,8-*de*][1,2]oxazine (22). 0.1 g, 10% as a pale green powder, mp 180–182°C; ν_{max} (Nujol) 1650 cm^{-1}; δ_H (400 MHz; DMSO-d_6) 2.37 (3H, s, Me), 6.63 (1H, d, *J*=7.4 Hz, H-9), 7.10–7.19 (3H, m, H-5, H-7, H-8), 7.69 (1H, d, *J*=9 Hz, H-6), 10.39 (1H, s, br, OH); δ_C (100 MHz; DMSO-d_6) 20.74 (Me), 100.92 (C-3a), 104.96, (C-9), 118.78 (C-7), 121.06 (C-5), 121.42 (C-9b), 125.12 (C-8), 127.45 (C-6a), 131.29 (C-6), 149.97 (C-4), 151.07 (C-9a), 153.92 (C-3); *m/z* (EI) 199 (100, M^+), 184 (57), 171 (80), 128 (46), 102 (89), 75 (48%); HRMS (EI): M^+, found 199.0617. $C_{12}H_9NO_2$ requires 199.0633.

3.4.6. 1-Hydroxy-2-methylbenzo[*cd*]indol-3(1*H*)-one (23). 0.17 g, 17% as pale yellow powder, mp 220–222°C; ν_{max} (Nujol) 3340, 1620 cm^{-1}; δ_H (400 MHz; DMSO-d_6) 2.75 (3H, s, Me), 6.51 (1H, d, *J*=9.6 Hz, H-4), 7.37 (1H, t, *J*= 7.6, Hz, H-7), 7.57 (1H, d, *J*=7.6 Hz, H-6), 7.66 (1H, d, *J*=7.6 Hz, H-8), 7.75 (1H, d, *J*=9.6 Hz, H-5), 12.23 (1H, s, br, OH); δ_C (100 MHz; DMSO-d_6) 10.75 (Me), 105.05 (C-2a), 111.69 (C-8), 120.90 (C-8b), 121.89 (C-5a), 122.53 (C-6), 122.98 (C-7), 130.12 (C-8a), 132.54 (C-4), 136.33 (C-4), 143.59 (C-2), 178.02 (C-3); *m/z* (EI) 199 (24, M^+), 183 (100), 169 (31), 154 (25), 140 (14), 127 (17), 114 (15), 102 (7), 77 (10), 63 (10), 41 (34%); HRMS (EI): M^+, found 199.0624. $C_{12}H_9NO_2$ requires 199.0633.

3.4.7. 3-Ethyl-4-hydroxynaphtho[1,8-*de*][1,2]oxazine (26). 0.23 g, 22% as a pale green powder, mp 180–183°C; ν_{max} (Nujol) 1630 cm^{-1}; δ_H (400 MHz; DMSO-d_6) 1.17 (3H, t, *J*=7.3 Hz, CH_2CH_3), 2.77–2.86 (2H, q, CH_2CH_3), 6.63 (1H, d, *J*=7.3 Hz, H-9), 7.04–7.17 (3H, m, H-5, H-7, H-8), 7.66 (1H, d, *J*=9 Hz, H-6), 10.50 (1H, s, br, OH); δ_C (100 MHz; DMSO-d_6) 12.18 (Me), 26.98 (CH_2), 99.98 (C-3a), 104.96, (C-9), 118.77 (C-7), 121.16 (C-5), 121.82 (C-9b), 125.05 (C-8), 127.65 (C-6a), 131.16 (C-6), 149.28 (C-4), 151.10 (C-9a), 157.77 (C-3); *m/z* (EI) 213 (58, M^+), 198 (32), 185 (100), 156 (15), 139 (22), 128 (12), 115 (12), 102 (54), 75 (38), 63 (25), 60 (15%); HRMS (EI): M^+, found 213.0789. $C_{13}H_{11}NO_2$ requires 213.0790.

3.4.8. 1-Hydroxy-2-ethylbenzo[*cd*]indol-3(1*H*)-one (27). 0.12 g, 11% as yellow powder, mp 180–182°C; ν_{max} (Nujol) 3400, 1620 cm^{-1}; δ_H (400 MHz; DMSO-d_6) 1.33 (3H, t, CH_2CH_3), 3.15–3.20 (2H, q, CH_2CH_3), 6.51 (1H, d, *J*= 9.6 Hz, H-4), 7.38 (1H, t, *J*=7.6 Hz, H-7), 7.57 (1H, d, *J*= 7.6 Hz, H-6), 7.67 (1H, d, *J*=7.6 Hz, H-8), 7.75 (1H, d, *J*=9.6 Hz, H-5), 12.23 (1H, s, br, OH); δ_C (100 MHz; DMSO-d_6) 12.04 (CH_2CH_3), 18.04 (CH_2CH_3), 106.78 (C-2a), 111.85 (C-8), 121.02 (C-8b), 122.06 (C-5a), 122.62 (C-6), 123.05 (C-7), 130.16 (C-8a), 132.61 (C-4), 136.31 (C-5), 148.79 (C-2), 177.50 (C-3); *m/z* 213 (19, M^+), 196 (100), 183 (15), 162 (21), 151 (20), 132 (12), 113 (10), 81 (4%); HRMS (EI): M^+, found 213.0841. $C_{13}H_{11}NO_2$ requires 213.0790.

3.4.9. 4-Hydroxy-3-phenylnaphtho[1,8-*de*][1,2]oxazine (32). 0.5 g, 38% as pale green powder, mp 148–150°C; ν_{max} (Nujol) 1640 cm^{-1}; δ_H (400 MHz; DMSO-d_6) 6.72 (1H, d, *J*=7.4 Hz, H-9), 7.00 (1H, d, *J*=9.1 Hz, H-5), 7.17 (1H, t, *J*=8.0 Hz, H-8), 7.24 (1H, d, *J*=8.0 Hz, H-7), 7.41 (5H, m, benzenoid) 7.71 (1H, d, *J*=9.1 Hz, H-6), 10.10 (1H, s, OH); δ_C (100 MHz; DMSO-d_6) 99.74, 105.19, 119.19, 121.46, 122.45, 125.05, 127.41 (2C), 128.25 (3C), 128.45, 131.80, 135.30, 149.95, 150.97, 157.75; *m/z* (EI) 261 (100, M^+), 244 (35), 204 (7), 156 (8), 78 (39%); HRMS (EI): M^+, found 261.0780. $C_{17}H_{11}NO_2$ requires 261.0790.

3.4.10. Oxidation of (*Z*/*E*)-1-(2-hydroxy-1-naphthyl)propan-1-one oxime with lead(IV) acetate. A stirred solution of **25** (5 mmol) in dry tetrahydrofuran (60 mL) under argon was cooled to 0°C. Lead(IV) acetate (4.43 g, 10 mmol) was added slowly over a period of 30 min. The reaction mixture was stirred at this temperature for 1.5 h and then filtered, the solid was washed with tetrahydrofuran and the filtrate was evaporated in vacuo to leave a residue. The residue was chromatographed (25% ethyl acetate/hexane) to give **1-ethylnaphtho[1,2-*d*]isoxazole-2-oxide 33** in the first fraction. The second fraction was evaporated and the residue triturated with acetonitrile to give **1-hydroxy-2-ethylbenzo[*cd*]indol-3(1*H*)-one 27**.

3.4.11. 1-Ethyl-9-hydroxynaphtho[1,2-*d*]isoxazole (27). 0.1 g, 10% as pale yellow powder, mp 181–182°C, identical in all respects to an authentic sample prepared above.

3.4.12. 1-Ethylnaphtho[1,2-*d*]isoxazole-2-oxide (33). 0.25 g, 28% as yellow powder, mp 97–100°C; ν_{max} (Nujol) 1200 cm^{-1}; δ_H (400 MHz; $CDCl_3$) 1.45 (3H, t, *J*=7.6 Hz, CH_2CH_3), 3.25–3.19 (2H, q, *J*=7.6 Hz, CH_2CH_3), 7.35 (1H, d, *J*=9.0 Hz, H-4), 7.55 (1H, t, *J*=7.2 Hz, H-7), 7.66 (1H, t, *J*=7.2 Hz, H-8), 7.90 (1H, d, *J*=9.0 Hz, H-5), 7.94 (1H, d, *J*=8.2 Hz, H-9), 8.11 (1H, d, *J*=8.2 Hz, H-6); δ_C (100 MHz; $CDCl_3$) 10.42, 19.53, 107.92, 108.04, 122.07, 123.40, 125.43, 125.50, 128.09, 129.69, 130.03, 130.70, 148.57; *m/z* (EI) 213 (100, M^+), 196 (21), 183 (29), 165 (79), 153 (48), 139 (29), 155 (34%); HRMS (EI): M^+, found 213.0818. $C_{13}H_{11}NO_2$ requires 213.0790.

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