Spectroscopic Studies of Benzimidazole, Quinoxaline and Quinoline Derivatives

J. Chem. Research (S), 1995, 10–11
J. Chem. Research (M), 1995, 0201–0220

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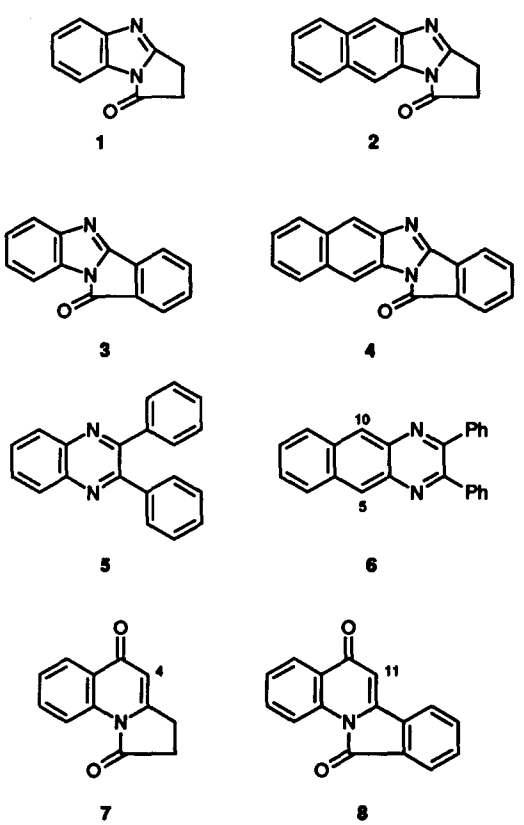
Four fused benzimidazoles, two quinoxalines and two fused quinoline derivatives were studied with respect to their mass, NMR and IR spectra; the compounds were chosen in order to compare the effect of the size of the hetero ring and the presence of C=N and C=O on their spectral behaviour.

In continuation of our work on the synthesis and identification of different classes of heterocyclic compounds,[1–4] we report here a detailed mass, NMR and IR spectral study of eight heterocyclic compounds: 2,3-dihydropyrrolo[1,2-*a*]-benzimidazol-1-one **1**, 2,3-dihydronaphtho[2,3-*d*]pyrrolo-[1,2-*a*]imidazol-1-one **2**, isoindolo[2,1-*a*]benzimidazol-1-one **3**, naphtho[2′,3′:4,5]imidazo[2,1-*a*]isoindol-13-one **4**, 2,3-diphenylquinoxaline **5**, 2,3-diphenylbenzo[*g*]quinoxaline **6**, 2,3-dihydropyrrolo[1,2-*a*]quinoline-1,5-dione **7** and isoindolo[2,1-*a*]quinoline-6,12-dione **8**. These compounds were chosen in order to compare the effect of the hetero ring and the presence of the C=N and C=O groups on their spectral behaviour.



In the HRMS study all eight compounds show very stable molecular ions (M^+) which, in fact, are the base peaks in almost all of them. In addition those compounds that originate from the same acid anhydride have almost the same types of fragmentation and bond cleavage. The carbonyl-containing compounds **1**–**4** and **7**–**8** lose in their first fragmentation carbon monoxide (M − 28) or formaldehyde (M − 29) followed by loss of a cyanide radical [(M − 28) − 26] or hydrogen cyanide [(M − 28) − 27].

Compounds **1** and **2** being analogous have the same type of fragmentation. They each lose a C_2H_4 molecule (M − 28) to give fragment peaks at *m/z* 144 and 194 respectively. The loss of a CN radical followed by loss of a CO molecule gives ions at *m/z* 90 and 140 for **1** and **2** respectively. Compounds **3** and **4** have very stable molecular ions which represent the base peaks, the intensities of the second most abundant peaks being not more than 10%. They also lose CO fragments followed by their breakdown into two main fragments, at *m/z* 90 and 102 for **3** and at *m/z* 102 and 140 for **4**. The quinoxalines **5** and **6** in their mass fragmentations lose phenyl radicals or benzonitrile radicals to produce peaks at *m/z* 205 and 179, respectively, for **5** and at *m/z* 255 and 229, respectively, for **6**. Further loss of benzonitrile fragments gives peaks at *m/z* 76 and 126 for **5** and **6** respectively. Previous studies on analogous compounds have reported somewhat similar type of fragmentations.[5,6] The quinoline derivatives **7** and **8** originate from different acid anhydrides and have different fragmentations in their high-resolution spectra. Compound **7** in three consecutive fragmentations loses C_2H_4 and 2 CO to give peaks at *m/z* 171, 143 and 115. Compound **8**, on the other hand, loses a CO fragment to give a peak at *m/z* 219. In contrast to **7**, compound **8** at this stage does not follow the same fragmentation pathway (loss of CO and C_6H_4): instead its next fragmentation involves some rearrangement to give peaks at *m/z* 190 and 164.

The ^{1}H NMR spectra of the eight compounds studied were in general simple and easy to interpret. The aliphatic protons in compounds **1** and **2** absorb as multiplets at δ 3.25 and 3.4 respectively. In contrast, the two CH_2 groups in **7** absorb as an A_2B_2 system. In **1** and **2** the C=C and C=N bonds appear to have the same effect on the two CH_2 groups while in **7**, the C=C and C=O bonds have different effects. The aromatic protons in **1**–**5** absorb in the normal range (δ 7.0–8.0). 5-H and 10-H in **6** are equivalent and appear as one singlet at δ 9.7. Compounds **7** and **8** have in their ^{1}H NMR spectra two doublets each at δ 8.2 and 9.1, while 4-H in **7** and 11-H in **8** absorb as singlets at δ 6.3 and 6.8, respectively.

The ^{13}C NMR spectra of compounds **1**–**8** show sharp and clear signals for the non-quarternary carbons while the quaternary ones need longer pulse delay times (15–20 s). The assignment of each signal to a particular carbon was achieved through the comparison of each compound with analogous compounds that have previously been studied (when available) and by comparison of the data for each set of compounds with each other. The C=O and C=N signals were identified first. Compounds **7** and **8** showed in their spectra two signals for the carbonyl groups which were assigned on the basis that the one at lower field is for the ketonic group. The non-quaternary carbons were next identified followed by the quaternary ones. It could be seen that there is a similarity between each set of compounds, and even between analogous positions in different compounds.

The IR spectra of the carbonyl containing compounds **1**–**4** and **7**, **8** show strong absorbances for those groups. The increase in the frequency of the absorbance of the lactamic carbonyl groups in **1**–**4** from the normal range for γ-lactams is due to their fusion to additional rings. Once again, compounds **7** and **8** have two carbonyl absorbances in their IR

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