

COMPARATIVE STUDIES OF PMR SPECTRA OF SOME SUBSTITUTED DIINDOLYL METHANE SERIES. PART IV

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Comparative PMR studies of diindolyl-3-methane (I), (3'-nitrophenyl)-diindolyl-3-methane (II), (4'-nitrophenyl)-diindolyl-3-methane (III), (4'-methoxyphenyl)-diindolyl-3-methane (IV), (3', 4'-dimethoxyphenyl)-diindolyl-3-methane (V), (2'-hydroxyphenyl)-diindolyl-3-methane (VI) and (α -naphthyl)-diindolyl-3-methane have been described here.

In earlier communications^{1,2,3} we have described the comparative PMR studies of different types of compounds, such as the isocoumarins, dihydropyridines and trithians. In continuation of these studies we are now describing the PMR spectra of diindolyl series.

The PMR spectrum of diindolyl-3-methane (I) showed a singlet at τ 5.6 (2H) for methylene protons. The benzenoid protons being under identical conditions appeared as a multiplet at τ 2.7 (8H). The α protons of indole nucleus (adjacent to NH) appeared as a multiplet at τ 2.4 (2H). The remaining two NH protons appeared as a broad peak at τ 2.25 (2H).

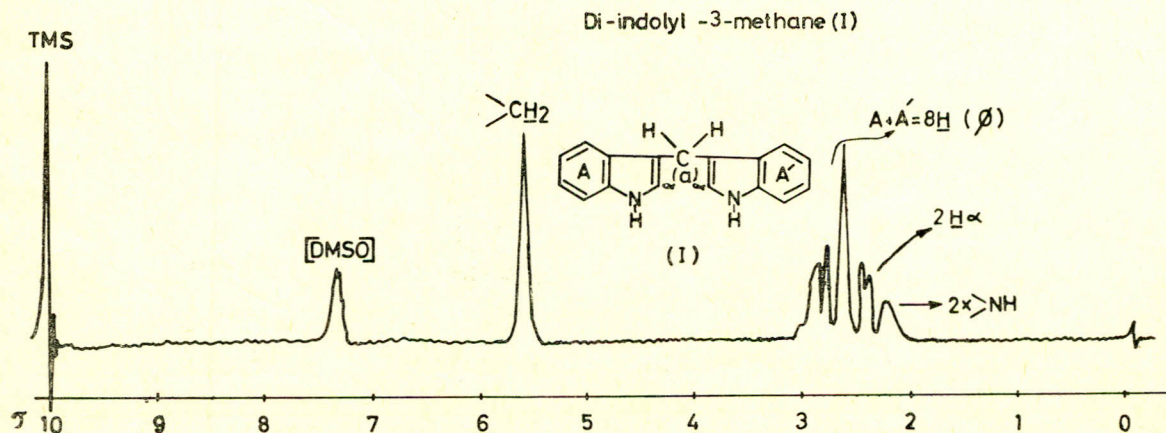
The PMR spectrum of (3'-nitrophenyl)-diindolyl-3-methane (II) showed a sharp singlet at τ 3.28 (1H) for methine proton. The benzenoid protons of the indolyl group showed a multiplet at τ 2.42 (8H). All the other benzenoid protons of the substituted nitrophenyl group appeared as a complex multiplet at τ 1.98 (4H). The two α protons adjacent to NH group showed an ill-defined multiplet, centred at τ 1.4 (2H). The remaining two protons of the NH groups gave a singlet at τ 0.75 (2H).

The PMR spectrum of (4'-nitrophenyl)-diindolyl-3-methane (III) showed a singlet for

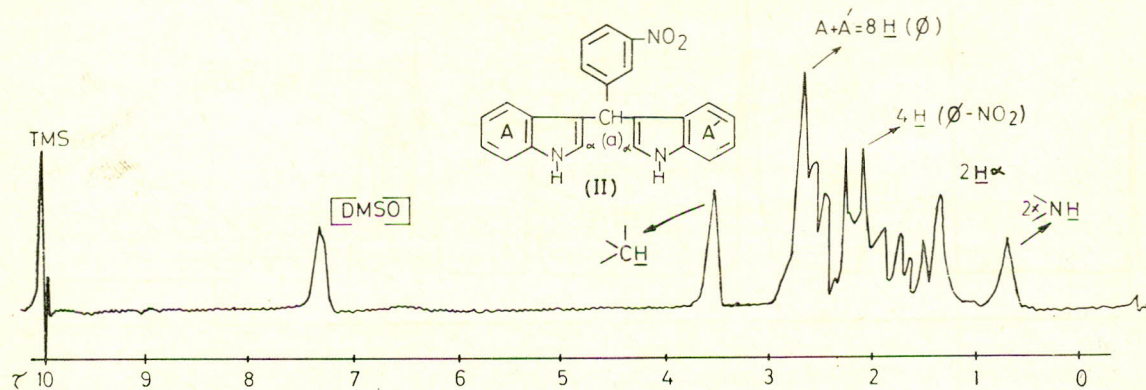
methine protons at τ 3.6 (1H). The benzenoid protons of indolyl group appeared as a multiplet centred at τ 2.78 (8H). The other benzenoid protons of the substituted nitrophenyl group appeared as two doublets, showing *ortho* coupling ($J=7$ Hz) centred at τ 2.1 (2H; a, a') and τ 2.55 (2H; b, b') respectively. The α protons of indole group appeared at τ 1.5 (2H) as a doublet. The remaining two protons of the NH group showed a broad singlet at τ 0.98 (2H).

The PMR spectrum of (4'-methoxyphenyl)-diindolyl-3-methane (IV) showed a singlet centred at τ 6.2 (3H) for $-\text{OCH}_3$ group. The proton of methine group gave a singlet at τ 3.98 (1H). The protons of the benzene rings of the indole groups appeared as multiplet at τ 2.86 (8H). The benzenoid protons of 4'-methoxyphenyl group appeared as double doublet at τ 2.75 (2H) and τ 2.38 (2H) showing *ortho* coupling ($J=7$ Hz) respectively. The two α protons of the indole rings appeared as doublet at τ 1.98 (2H). Finally, the protons of NH group showed a broad singlet at τ 0.8 (2H).

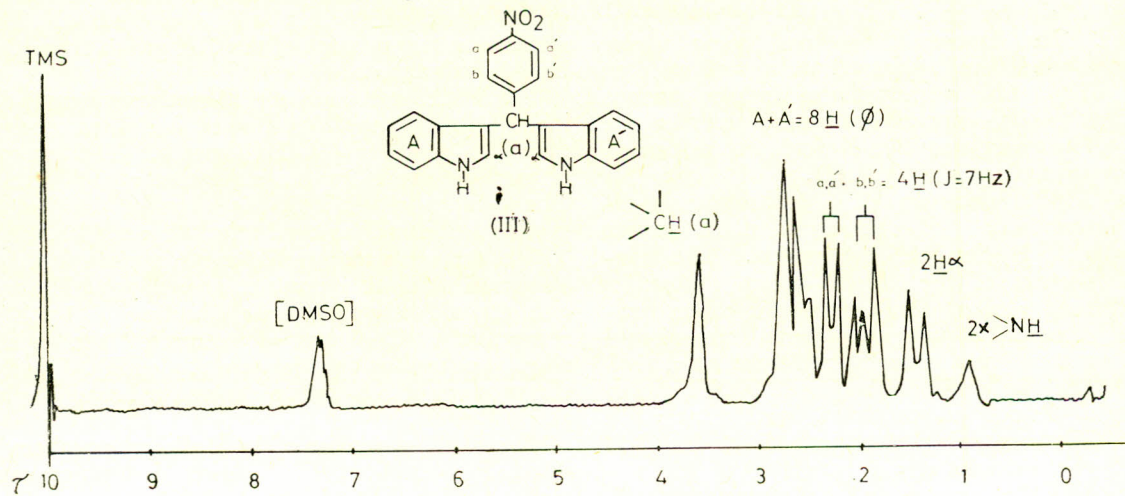
The PMR spectrum of (3',4'-dimethoxyphenyl)-diindolyl-3-methane (V) showed a doublet centred at τ 6.2 (6H) for two $-\text{OCH}_3$ groups. The proton of the methine group gave a singlet at



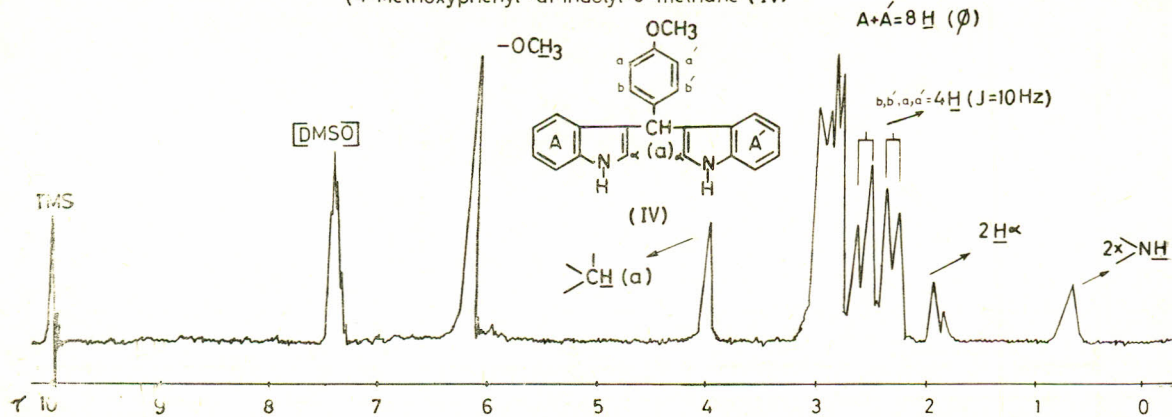
(3-Nitrophenyl)-di-indolyl-3-methane (II)



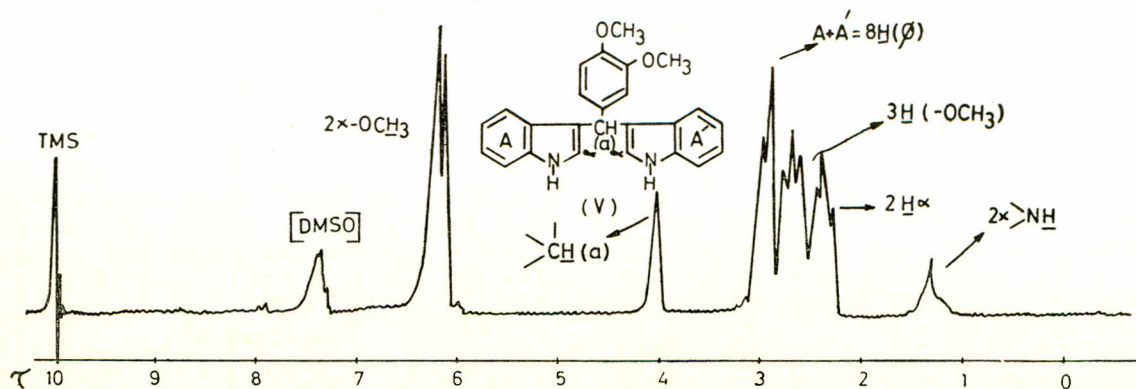
(4-Nitrophenyl)-di-indolyl-3-methane (III)



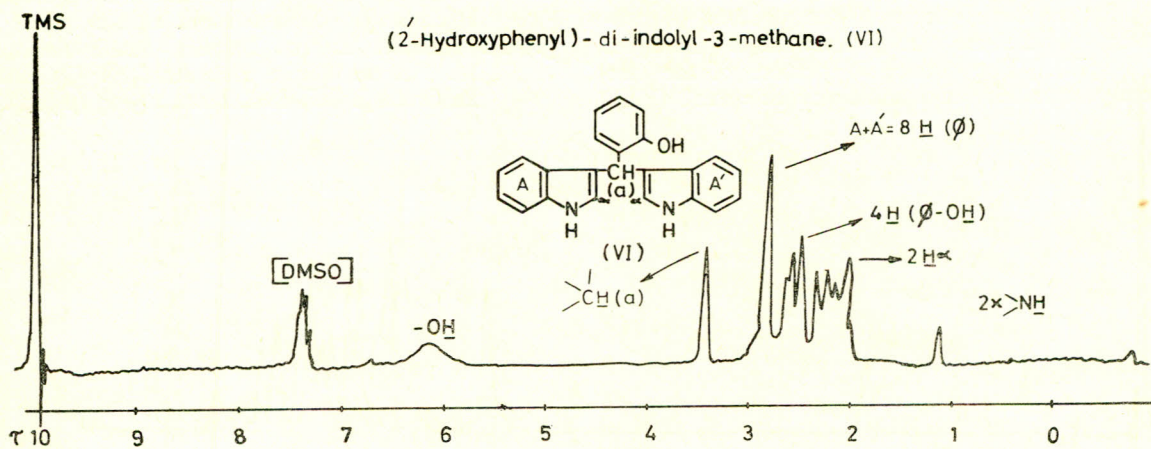
(4-Methoxyphenyl)-di-indolyl-3-methane (IV)



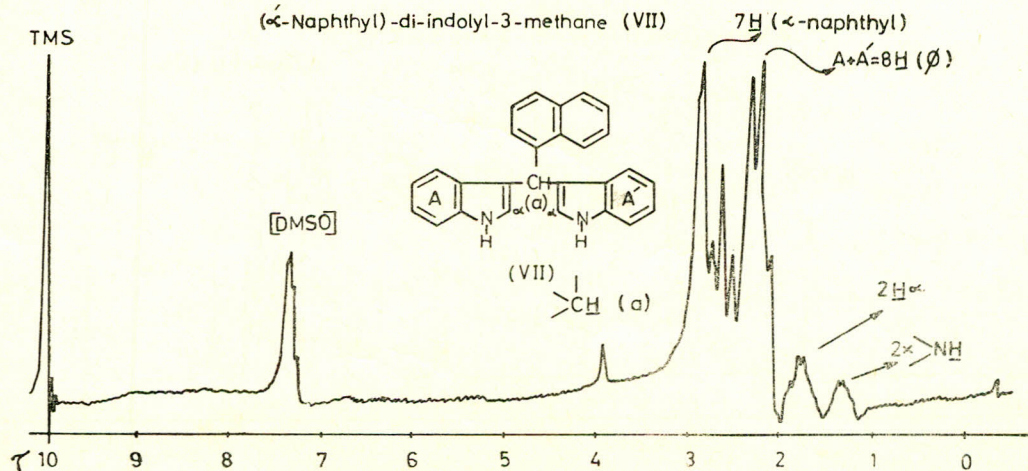
(3,4-Dimethoxyphenyl)-di-indolyl-3-methane (V)



(2-Hydroxyphenyl)-di-indolyl-3-methane (VI)



(α-Naphthyl)-di-indolyl-3-methane (VII)



τ 4.0 (1H). The benzenoid protons of the indolyl groups appeared as multiplet centred at τ 2.9 (8H). The benzenoid protons of the 3',4'-dimethoxyphenyl group appeared at τ 2.8. The two α -protons on the indole rings appeared as doublet centred at τ 2.5 (2H). The remaining two protons of the NH groups gave a broad singlet at τ 1.3 (2H).

The PMR spectrum of (2'-hydroxyphenyl)-diindolyl-3-methane (VI) showed broad hump at τ 6.15. Methine proton appeared at τ 3.45 (1H). The benzenoid protons of the indole groups and substituted hydroxy benzene ring showed multiplet centred at τ 2.8 (8H) and τ 2.45 (4H) respectively. The two α -protons of the indolyl groups showed an ill-defined doublet at τ 2.15 (2H). Finally, the protons of the two NH groups showed a singlet at τ 1.12 (2H).

PMR spectrum of (α -naphthyl)-diindolyl-3-methane (VII) showed up as a broad peak at τ 3.94 (1H) for methine proton. The protons of substituted naphthalene ring showed a multiplet τ 2.8 (7H). The benzenoid protons of the two indole groups showed a multiplet at τ 2.18 (8H).

The α -protons of the indole group showed an ill-defined doublet at τ 1.8 (2H). The remaining two protons of NH groups appeared at τ 1.3 (2H).

Experimental

All compounds were run on DP-60 Varian associate NMR machine in DMSO, using TMS as an internal standard. All the compounds were run in DMSO (d_6) due to their low solubility in other solvents, the concentration being 11~15%.

References

1. A. Kamal, Nilofer Kazi, Tahira Begum, M. Afroz Khan and Asaf A. Qureshi, Pakistan J. Sci. Ind. Res., **14**, 1 (1971).
2. A. Kamal, Rafia Akhtar, Tahira Begum, M. Afroz Khan and Asaf A. Qureshi, Pakistan J. Sci. Ind. Res., **14** 6(1971).
3. A. Kamal, Tahira Begum, Rafia Akhtar, M. Afroz Khan and Asaf A. Qureshi, Pakistan J. Sci. Ind. Res., **14**, 13 (1971).