Two-dimensional spectroscopy in the analysis of the reaction products of 1,3-dipolar cycloaddition of some azomethine ylides and 3-phenyl-1-(pyrrol-2-yl)propen-2-ones

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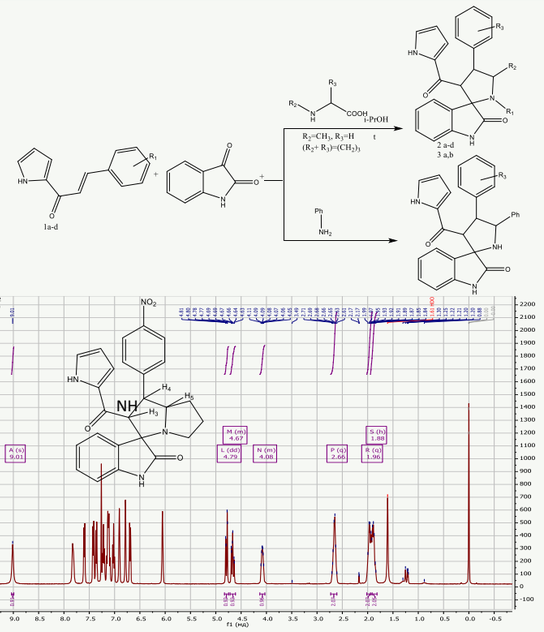
Abstract

Approaches have been suggested to establish, using various NMR spectroscopy techniques (1H, 13C, HSQC, HMBC, NOESY-2D), the structural features of the products and the mechanism of the 1,3-dipolar cycloaddition reaction of some azomethine ylides generated in situ and 3-phenyl-1-(pyrrol-2-yl)propen-2-ones carried out in the multicomponent interaeion mode

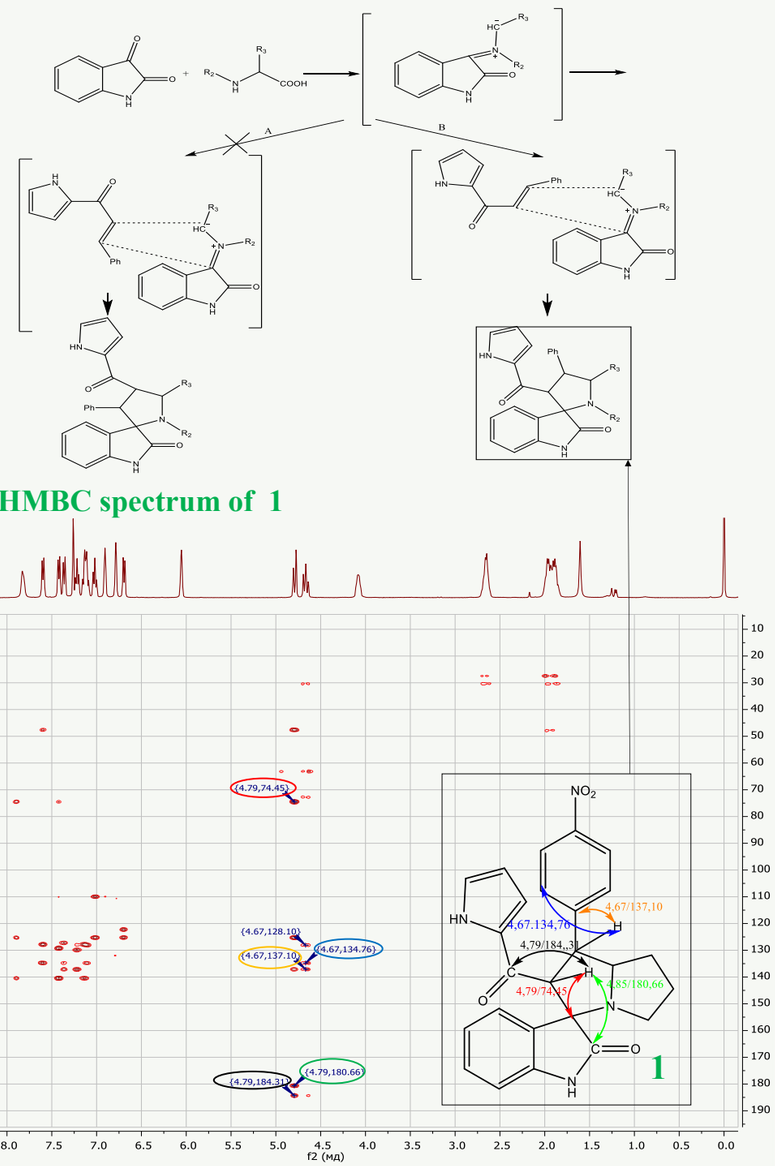
Instrumental

^{1}H NMR, HSQC, HMBC, NOESY-2d spectra were obtained on a Varian 400 spectrometer (400 MHz, CDCl3, DMSO-d6). Internal standard - tetramethylsilane.

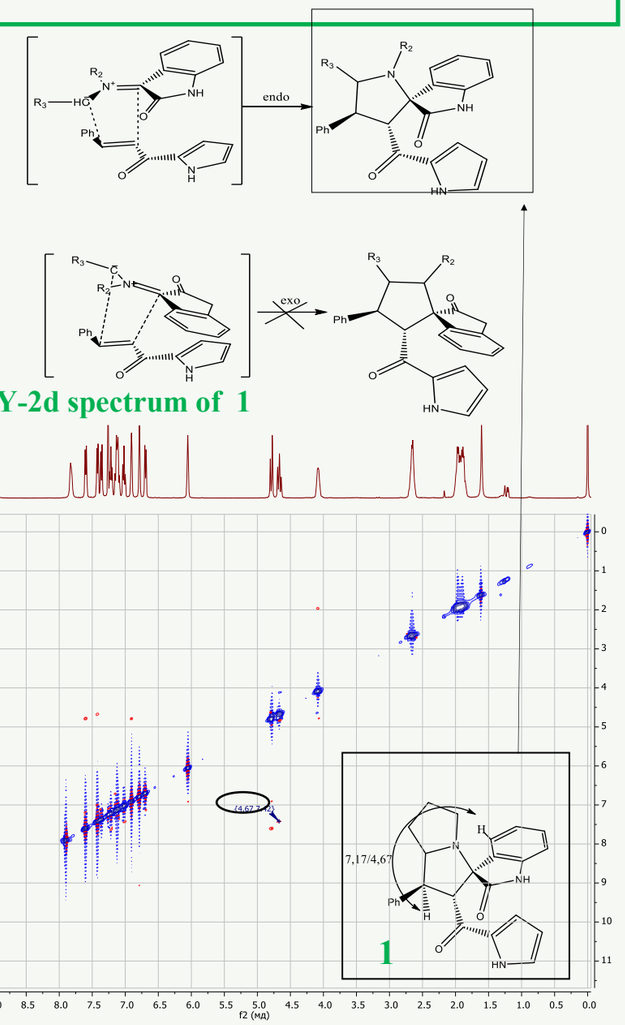


R	$t_{кип.}$, °C	yield,%	NMR ^{1}H	NMR ^{13}C
2-Cl	150-155	65	5,06(q,1H, H-4), 4,39 (d,1H,H-3.), 3,55(t.), 3,43(t.)(2H, H-5), 2,20(s,3H, N(CH_3)	185.39(C=O), 179,85(-NH-C=O), 74.40 C2(spiro), 61.10(C3), 40,32(C4), 60,54(C5),
4-Cl	156-160	65	5,00(q,1H, H-4), 4,33 (d,1H,H-3.), 3,54(t.), 3,46 (t.)(2H, H-5), 2,23(s,3H, N(CH_3)	187.91(C=O), 179,83(-NH-C=O), 77.91 C2(spiro), 61.83(C3), 41,25(C4), 61,54(C5),
4-NO_2	153-159	65	4,44(q,1H, H-4), 4,22 (d,1H,H-3.), 3,59(t.), 3,43(t.)(2H, H-5), 2,22(s,3H, N(CH_3)	186.40(C=O), 178,89(-NH-C=O), 79.99 C2(spiro), 60.87(C3), 40,38(C4), 60,56(C5),
4-OCH_3	155-158	63	4,59(q,1H, H-4), 4,20 (d,1H,H-3.), 3,65(t.), 3,59(t.)(2H, H-5), 2,23(s,3H, N(CH_3)	188.37(C=O), 179,85(-NH-C=O), 77.92 C2(spiro), 60.88(C3), 41,05(C4), 61,00(C5),
4-NO_2	162-159	49	4,79(dd,1H, H-3), 4,67 (m,1H,H-4.), 4,08(m, 1H, H-5),2,66(q,2H,H-8), 1,96(q, 2H),1,88(q, 2H)(H-6, H-7)	184.31(C=O), 177,54(-NH-C=O), 74.45 C2(spiro), 63.14(C3), 47,62(C4), 72,76(C5),30,35 (C-6), 27,19 (C-7), 48,05 (C-8).
2-Cl	165-168	50	4,43(s,1H, H-3), 4,85 (d,1H,H-4.), 4,42(t, 1H, H-5),2,26(s,2H,H-8), 2,09(s, 1H, H-6, H-7)	182.28(C=O), 171,19(-NH-C=O), 74.34 C2(spiro), 61.28(C3), 51,39(C4), 71,97(C5),28,37 (C-6), 25,55 (C-7), 51,39 (C-8).
4-NO_2	175-177	41	5,05 (d, 1H, H-3), 4,90 (t., 1H, H-4), 4,54 (d,1H, H-5)	184.89(C=O), 173,54(-NH-C=O), 68.95C2(spiro), 61.96(C3), 51,18(C4), 68,73(C5)
2-Cl	168-169	55	5,08 (d, 1H, H-3), 4,59 (m., 1H, H-4), 4,32 (d,1H, H-5)	184.63(C=O), 181,82(-NH-C=O), 69.15 C2(spiro), 68.14(C3), 62,04(C4), 55,03(C5)

The different arrangement of the dipole and dipolarophile reaction centers at the moment of interaction leads to the possibility of the formation of two regioisomenres



Different spatial positions of the dipole and dipolarophile at the moment of interaction leads to the possibility of endo- and exocyclic addition



Conclusion:

Analysis of NMR spectroscopy data proves that the mechanism of the studied reaction includes the synchronous interaction of azomethine ylides obtained during the reaction and dipolaro-philes.