Twisted imide bond in non-cyclic imides. Synthesis, structural and vibrational properties

N,N-bis(furan-2-carbonyl)-4-chloroaniline

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Supporting information available

Table S1. Elemental, mass and NMR spectroscopic data of compound (3).

^{1}H NMR (300 MHz) δ (ppm)	**^{13}C NMR(75.4 MHz) δ (ppm)**	**Elemental analysis (%)**	**GC-MS (m/z)**
6.47 (dd, J_1 =1.8, J_2 =1.8 Hz, 2H, H-4', furan H), 7.08 (d, J = 3.3 Hz, 2H, H-3', furan H), 7.19 (d, 2H, J = 8.7 Hz H-3, H-5, ArH), 7.38 (d, J = 8.0 Hz, 2H, ArH), 7.47 (d J =0.6 Hz, 1H, H-5', furan)	112.41 (C4' furan) 115.58 (C'3 furan)., 119.84 (C2, C6, Ar), 129.79 (C3, C5, Ar), 134.07 (C4, Ar), 137.40 (C1, Ar), 146.44 (C5' furan), 147.31 (C2' furan), 161.04 (C=O).	Calcd. For $C_{16}H_{10}ClNO_4$: C: 60.87; H: 3.19 N: 4.44; S: 11.28; Found: C: 60.94; H: 3.26, N: 4.39; S: 11.34	95 (100%, $C_4H_3OC(O)^+$), 39 (14%, $C_3H_3^+$), 204 (8%, $N(C_4H_3OC(O))_2)^+$, 287 (8%, M-28), 315 (6%, M^+)

Table S2. Natural Bond Orbital stabilization energies (kcal/mol) for the main orbital interactions around the imide group for relevant conformers of the title compound computed at the HF/6-311++G** level of approximation.

	Anti-anti [a]	**Syn-anti**	**Syn-syn** [a]
$\mathbf{lp_p(N) \rightarrow \pi^*(C{=}O)}$	42.1 (× 2)	32.3 / 44.4	33.4 (× 2)
$\mathbf{lp_\pi(O) \rightarrow \sigma^*(N{-}C)}$	36.6 (× 2)	38.5 / 36.1	37.8 (× 2)

[a] Due to the symmetric considerations the same values are computed for interactions in the equivalent 2-carbonyl furan group for anti-anti and syn-syn conformers (belonging to the C_2 point group)

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for the title compound. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	2331(1)	609(1)	6846(1)	40(1)
N(1)	2512(1)	6665(1)	4450(1)	21(1)
C(1)	3455(1)	6915(1)	3960(1)	21(1)
O(11)	3988(1)	5702(1)	3720(1)	30(1)
C(2)	1610(1)	7792(1)	4443(1)	20(1)
O(21)	1070(1)	7956(1)	5096(1)	27(1)
C(11)	3784(1)	8698(1)	3838(1)	21(1)
O(12)	4519(1)	8979(1)	3201(1)	29(1)
C(13)	4775(1)	10681(2)	3247(1)	36(1)
C(14)	4231(1)	11472(2)	3885(1)	41(1)
C(15)	3585(1)	10176(2)	4281(1)	31(1)
C(21)	2480(1)	5173(1)	5016(1)	20(1)
C(22)	3326(1)	4868(2)	5641(1)	27(1)
C(23)	3287(1)	3443(2)	6198(1)	29(1)
C(24)	2394(1)	2361(1)	6130(1)	25(1)
C(25)	1547(1)	2640(1)	5502(1)	25(1)
C(26)	1599(1)	4052(1)	4937(1)	23(1)
C(31)	1299(1)	8643(1)	3602(1)	20(1)
O(32)	1693(1)	7996(1)	2832(1)	24(1)
C(33)	1238(1)	8973(2)	2158(1)	30(1)
C(34)	586(1)	10200(2)	2479(1)	33(1)
C(35)	614(1)	9984(2)	3419(1)	26(1)

Table S3. Bond lengths [Å] and angles [°] for the title compound.

Cl(1)-C(24)	1.7376(11)
N(1)-C(2)	1.4087(13)
N(1)-C(1)	1.4134(12)
N(1)-C(21)	1.4383(13)
C(1)-O(11)	1.2103(13)
C(1)-C(11)	1.4552(14)
C(2)-O(21)	1.2177(12)
C(2)-C(31)	1.4588(14)
C(11)-C(15)	1.3547(15)
C(11)-O(12)	1.3623(12)
O(12)-C(13)	1.3586(15)
C(13)-C(14)	1.341(2)
C(13)-H(13)	0.9500
C(14)-C(15)	1.4281(18)
C(14)-H(14)	0.9500
C(15)-H(15)	0.9500
C(21)-C(22)	1.3851(14)
C(21)-C(26)	1.3876(14)
C(22)-C(23)	1.3888(16)
C(22)-H(22)	0.9500
C(23)-C(24)	1.3789(16)
C(23)-H(23)	0.9500
C(24)-C(25)	1.3852(15)
C(25)-C(26)	1.3892(15)
C(25)-H(25)	0.9500
C(26)-H(26)	0.9500
C(31)-C(35)	1.3571(15)
C(31)-O(32)	1.3712(12)
O(32)-C(33)	1.3629(14)
C(33)-C(34)	1.3479(18)
C(33)-H(33)	0.9500
C(34)-C(35)	1.4201(16)
C(34)-H(34)	0.9500
C(35)-H(35)	0.9500

C(2)-N(1)-C(1)	124.87(9)
C(2)-N(1)-C(21)	117.42(8)
C(1)-N(1)-C(21)	117.69(8)
O(11)-C(1)-N(1)	121.02(10)
O(11)-C(1)-C(11)	122.99(9)
N(1)-C(1)-C(11)	115.77(9)
O(21)-C(2)-N(1)	121.06(9)
O(21)-C(2)-C(31)	121.39(9)
N(1)-C(2)-C(31)	117.37(9)
C(15)-C(11)-O(12)	110.52(10)
C(15)-C(11)-C(1)	133.26(10)
O(12)-C(11)-C(1)	115.95(9)
C(13)-O(12)-C(11)	106.21(10)
C(14)-C(13)-O(12)	111.06(11)
C(14)-C(13)-H(13)	124.5
O(12)-C(13)-H(13)	124.5
C(13)-C(14)-C(15)	106.29(11)
C(13)-C(14)-H(14)	126.9
C(15)-C(14)-H(14)	126.9
C(11)-C(15)-C(14)	105.91(11)
C(11)-C(15)-H(15)	127.0
C(14)-C(15)-H(15)	127.0
C(22)-C(21)-C(26)	120.30(10)
C(22)-C(21)-N(1)	119.87(9)
C(26)-C(21)-N(1)	119.83(9)
C(21)-C(22)-C(23)	119.85(10)
C(21)-C(22)-H(22)	120.1
C(23)-C(22)-H(22)	120.1
C(24)-C(23)-C(22)	119.40(10)
C(24)-C(23)-H(23)	120.3
C(22)-C(23)-H(23)	120.3
C(23)-C(24)-C(25)	121.39(10)
C(23)-C(24)-Cl(1)	119.29(9)
C(25)-C(24)-Cl(1)	119.32(9)
C(24)-C(25)-C(26)	118.96(10)

C(24)-C(25)-H(25)	120.5
C(26)-C(25)-H(25)	120.5
C(21)-C(26)-C(25)	120.06(10)
C(21)-C(26)-H(26)	120.0
C(25)-C(26)-H(26)	120.0
C(35)-C(31)-O(32)	110.44(9)
C(35)-C(31)-C(2)	131.00(10)
O(32)-C(31)-C(2)	118.47(9)
C(33)-O(32)-C(31)	105.97(9)
C(34)-C(33)-O(32)	110.81(10)
C(34)-C(33)-H(33)	124.6
O(32)-C(33)-H(33)	124.6
C(33)-C(34)-C(35)	106.65(10)
C(33)-C(34)-H(34)	126.7
C(35)-C(34)-H(34)	126.7
C(31)-C(35)-C(34)	106.12(10)
C(31)-C(35)-H(35)	126.9
C(34)-C(35)-H(35)	126.9

Table S4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for the title compound. The anisotropic displacement factor exponent takes the form:$-2p^2[h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	40(1)	37(1)	43(1)	21(1)	2(1)	0(1)
N(1)	20(1)	20(1)	24(1)	4(1)	5(1)	1(1)
C(1)	20(1)	20(1)	22(1)	1(1)	5(1)	1(1)
O(11)	30(1)	21(1)	40(1)	1(1)	14(1)	4(1)
C(2)	19(1)	21(1)	21(1)	0(1)	2(1)	1(1)
O(21)	27(1)	33(1)	21(1)	2(1)	7(1)	6(1)
C(11)	19(1)	21(1)	24(1)	1(1)	5(1)	1(1)
O(12)	28(1)	29(1)	31(1)	5(1)	10(1)	0(1)
C(13)	29(1)	31(1)	47(1)	15(1)	2(1)	-6(1)
C(14)	36(1)	20(1)	66(1)	0(1)	0(1)	-5(1)
C(15)	29(1)	23(1)	42(1)	-8(1)	8(1)	-1(1)
C(21)	20(1)	20(1)	21(1)	2(1)	3(1)	1(1)
C(22)	21(1)	28(1)	30(1)	4(1)	-2(1)	-4(1)
C(23)	25(1)	32(1)	29(1)	8(1)	-4(1)	1(1)
C(24)	26(1)	24(1)	24(1)	6(1)	4(1)	3(1)
C(25)	23(1)	24(1)	27(1)	3(1)	2(1)	-3(1)
C(26)	21(1)	25(1)	23(1)	2(1)	-1(1)	-2(1)
C(31)	20(1)	22(1)	19(1)	0(1)	4(1)	0(1)
O(32)	26(1)	28(1)	18(1)	-1(1)	4(1)	2(1)
C(33)	31(1)	40(1)	20(1)	6(1)	3(1)	1(1)
C(34)	32(1)	40(1)	27(1)	11(1)	3(1)	7(1)
C(35)	25(1)	27(1)	25(1)	3(1)	4(1)	5(1)

Table S5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for the title compound.

	x	y	z	U(eq)
H(13)	5273	11238	2877	43
H(14)	4269	12657	4041	49
H(15)	3112	10321	4759	37
H(22)	3931	5632	5689	32
H(23)	3869	3215	6621	34
H(25)	940	1879	5459	30
H(26)	1031	4250	4496	28
H(33)	1363	8811	1544	36
H(34)	185	11045	2143	39
H(35)	230	10644	3835	31

Table S6. Torsion angles [°] for the title compound.

C(2)-N(1)-C(1)-O(11)	-151.94(11)
C(21)-N(1)-C(1)-O(11)	29.74(15)
C(2)-N(1)-C(1)-C(11)	33.33(14)
C(21)-N(1)-C(1)-C(11)	-144.99(10)
C(1)-N(1)-C(2)-O(21)	-148.83(11)
C(21)-N(1)-C(2)-O(21)	29.50(15)
C(1)-N(1)-C(2)-C(31)	36.00(14)
C(21)-N(1)-C(2)-C(31)	-145.68(9)
O(11)-C(1)-C(11)-C(15)	-152.96(13)
N(1)-C(1)-C(11)-C(15)	21.66(18)
O(11)-C(1)-C(11)-O(12)	20.36(16)
N(1)-C(1)-C(11)-O(12)	-165.02(9)
C(15)-C(11)-O(12)-C(13)	-0.48(13)
C(1)-C(11)-O(12)-C(13)	-175.30(10)
C(11)-O(12)-C(13)-C(14)	-0.02(14)
O(12)-C(13)-C(14)-C(15)	0.48(16)
O(12)-C(11)-C(15)-C(14)	0.76(14)
C(1)-C(11)-C(15)-C(14)	174.35(12)
C(13)-C(14)-C(15)-C(11)	-0.75(15)
C(2)-N(1)-C(21)-C(22)	-125.14(11)
C(1)-N(1)-C(21)-C(22)	53.31(14)
C(2)-N(1)-C(21)-C(26)	54.48(14)
C(1)-N(1)-C(21)-C(26)	-127.07(11)
C(26)-C(21)-C(22)-C(23)	-0.64(17)
N(1)-C(21)-C(22)-C(23)	178.98(10)
C(21)-C(22)-C(23)-C(24)	-1.06(18)
C(22)-C(23)-C(24)-C(25)	1.69(18)
C(22)-C(23)-C(24)-Cl(1)	-178.42(9)
C(23)-C(24)-C(25)-C(26)	-0.60(17)
Cl(1)-C(24)-C(25)-C(26)	179.51(9)
C(22)-C(21)-C(26)-C(25)	1.74(17)
N(1)-C(21)-C(26)-C(25)	-177.88(10)
C(24)-C(25)-C(26)-C(21)	-1.12(17)
O(21)-C(2)-C(31)-C(35)	17.68(18)

N(1)-C(2)-C(31)-C(35)	-167.16(11)
O(21)-C(2)-C(31)-O(32)	-158.62(10)
N(1)-C(2)-C(31)-O(32)	16.54(14)
C(35)-C(31)-O(32)-C(33)	0.17(12)
C(2)-C(31)-O(32)-C(33)	177.20(10)
C(31)-O(32)-C(33)-C(34)	0.37(13)
O(32)-C(33)-C(34)-C(35)	-0.74(15)
O(32)-C(31)-C(35)-C(34)	-0.61(13)
C(2)-C(31)-C(35)-C(34)	-177.14(11)
C(33)-C(34)-C(35)-C(31)	0.81(14)

Table S7. Atom Coordinates for optimized (B3LYP/6-311++G**) *EE*-syn-anti form

Cl	-0.33127	-4.72681	3.61784
N	-0.06148	0.01807	0.0038
C	1.12346	0.16898	-0.7788
O	1.74153	-0.78727	-1.18511
C	-1.18008	0.88535	-0.02092
O	-1.90323	1.01731	0.94771
C	1.60182	1.55082	-0.9274
O	2.50302	1.78188	-1.92609
C	2.89728	3.0704	-1.81609
H	3.60917	3.40877	-2.55053
C	2.2816	3.67794	-0.76228
H	2.41741	4.69754	-0.43925
C	1.4368	2.68845	-0.1811
H	0.80438	2.79878	0.68527
C	-0.12943	-1.12682	0.87735
C	0.81477	-1.28651	1.88856
H	1.59558	-0.54731	2.0215
C	0.76195	-2.39426	2.72855
H	1.49438	-2.52479	3.51444
C	-0.2523	-3.32986	2.55375
C	-1.20472	-3.17992	1.55066
H	-1.98423	-3.91998	1.42469
C	-1.13433	-2.07623	0.70737
H	-1.86353	-1.95572	-0.0844
C	-1.50462	1.55774	-1.28608
O	-0.91809	1.1561	-2.45689
C	-1.45232	1.90946	-3.44745
H	-1.09037	1.70449	-4.44114
C	-2.37152	2.78231	-2.9446
H	-2.95082	3.49517	-3.50916
C	-2.41037	2.55034	-1.53955
H	-3.02367	3.03905	-0.80023

Table S8. Atom Coordinates for optimized (B3LYP/6-311++G**) *EE*-syn-syn form

Cl	-5.962334	0.000127	-0.000052
N	0.007495	-0.000369	0.000044
C	0.679885	-1.025740	-0.723477
O	0.230575	-1.487671	-1.749405
C	0.679861	1.024963	0.723891
O	0.230822	1.486053	1.750291
C	1.890145	-1.549874	-0.075315
O	2.757093	-2.242185	-0.870760
C	3.733959	-2.723457	-0.069228
H	4.508598	-3.289198	-0.559669
C	3.513642	-2.370753	1.229334
H	4.127106	-2.627326	2.077973
C	2.312278	-1.605613	1.228162
H	1.819245	-1.164578	2.079900
C	-1.430593	-0.000290	-0.000001
C	-2.129787	-1.154347	0.349538
H	-1.591028	-2.053195	0.623580
C	-3.520485	-1.161572	0.342819
H	-4.066440	-2.057072	0.609749
C	-4.204910	-0.000030	-0.000033
C	-3.520272	1.161382	-0.342872
H	-4.066054	2.056983	-0.609813
C	-2.129568	1.153890	-0.349561
H	-1.590643	2.052637	-0.623620
C	1.889502	1.549954	0.075375
O	2.756260	2.242881	0.870564
C	3.732572	2.724694	0.068739
H	4.506991	3.290989	0.558896
C	3.512161	2.371774	-1.229754
H	4.125281	2.628607	-2.078560
C	2.311264	1.605917	-1.228216
H	1.818233	1.164574	-2.079789

Table S9. Atom Coordinates for optimized (B3LYP/6-311++G**) *EE*-anti-anti form

Cl	-4.777691	3.502895	0.628566
N	0.011313	-0.008313	-0.001500
C	-0.095629	-1.317120	0.536705
O	-1.171155	-1.872156	0.628546
C	1.123942	0.563156	-0.671983
O	1.323953	1.760112	-0.648603
C	1.110940	-1.940586	1.095684
O	2.241334	-1.197168	1.310805
C	3.152125	-2.017926	1.887940
H	4.103318	-1.572046	2.126181
C	2.634399	-3.268467	2.053001
H	3.138680	-4.115532	2.489776
C	1.304965	-3.217488	1.543065
H	0.573521	-4.008104	1.506956
C	-1.149141	0.842513	0.151176
C	-1.378326	1.490976	1.360650
H	-0.680863	1.364581	2.179734
C	-2.494122	2.308491	1.513874
H	-2.679803	2.819371	2.449656
C	-3.367568	2.469021	0.443043
C	-3.147335	1.827689	-0.771678
H	-3.840657	1.961278	-1.591814
C	-2.030984	1.008639	-0.912127
H	-1.850721	0.491506	-1.846690
C	1.962757	-0.312995	-1.500075
O	1.548894	-1.581739	-1.809471
C	2.486037	-2.115809	-2.629722
H	2.297553	-3.120879	-2.968312
C	3.489231	-1.221192	-2.858633
H	4.358539	-1.381191	-3.476125
C	3.146818	-0.046442	-2.128746
H	3.689115	0.882848	-2.067735

Table S10. Atom Coordinates for optimized (B3LYP/6-311++G**) *EZ*-syn-anti form

Cl	-6.101674	-0.032718	-0.670311
N	-0.175918	-0.004190	0.052819
C	0.537561	-1.101606	-0.541140
O	0.062253	-1.736255	-1.453277
C	0.506845	1.201532	0.338086
O	1.576340	1.478054	-0.170002
C	1.799863	-1.461669	0.111886
O	2.630890	-2.279197	-0.598708
C	3.680597	-2.570303	0.199629
H	4.438668	-3.202671	-0.231583
C	3.543542	-1.972398	1.417958
H	4.229299	-2.039359	2.247190
C	2.318896	-1.249228	1.361021
H	1.879402	-0.651133	2.142542
C	-1.604959	-0.004341	-0.125542
C	-2.380256	-0.967367	0.518236
H	-1.903680	-1.701054	1.157046
C	-3.759117	-0.985473	0.347076
H	-4.366558	-1.731412	0.842934
C	-4.355954	-0.024275	-0.464981
C	-3.595144	0.938304	-1.118468
H	-4.072655	1.668552	-1.758767
C	-2.212162	0.936432	-0.954344
H	-1.606840	1.663889	-1.482136
C	-0.117634	2.122446	1.299332
O	-1.119670	1.702057	2.133334
C	-1.445750	2.753454	2.921460
H	-2.227707	2.577246	3.641285
C	-0.679504	3.840206	2.618444
H	-0.730011	4.809191	3.088482
C	0.188504	3.429083	1.566979
H	0.946482	4.007012	1.063798

Table S11. Atom Coordinates for optimized (B3LYP/6-311++G**) *ZZ*-syn-anti form

Cl	-5.808965	0.238326	-0.394340
N	0.116728	-0.125212	0.239358
C	0.789298	-1.125633	-0.526250
O	0.395440	-1.459536	-1.619317
C	0.737777	0.796137	1.116537
O	0.146032	1.262250	2.071104
C	1.893162	-1.805076	0.166806
O	2.769249	-2.501451	-0.614379
C	3.624717	-3.137842	0.217011
H	4.390875	-3.727703	-0.258124
C	3.317157	-2.879883	1.519890
H	3.826763	-3.264212	2.388794
C	2.187880	-2.011355	1.489303
H	1.653497	-1.607278	2.334211
C	-1.314188	-0.035141	0.088289
C	-2.117605	-1.124796	0.414997
H	-1.666987	-2.036705	0.788003
C	-3.498240	-1.048879	0.261689
H	-4.127299	-1.893218	0.511685
C	-4.064318	0.131752	-0.207707
C	-3.273714	1.229374	-0.533148
H	-3.729681	2.138783	-0.902267
C	-1.893376	1.137974	-0.389642
H	-1.267349	1.982235	-0.651342
C	2.095643	1.252249	0.790673
O	2.632274	1.009443	-0.445930
C	3.853700	1.594040	-0.470496
H	4.404391	1.493584	-1.390871
C	4.114646	2.211421	0.717100
H	5.009600	2.756841	0.970190
C	2.969344	1.994470	1.536070
H	2.795109	2.338377	2.542519

Figure S1 UV spectrum for the title compound in $CHCl_3$

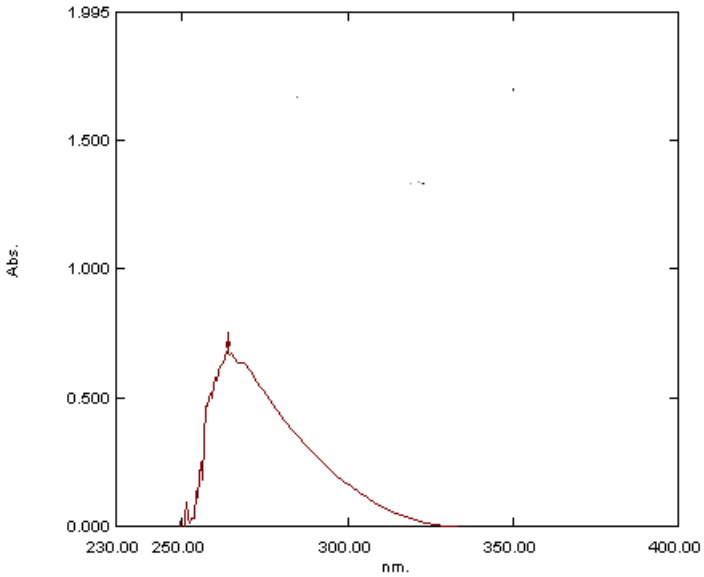
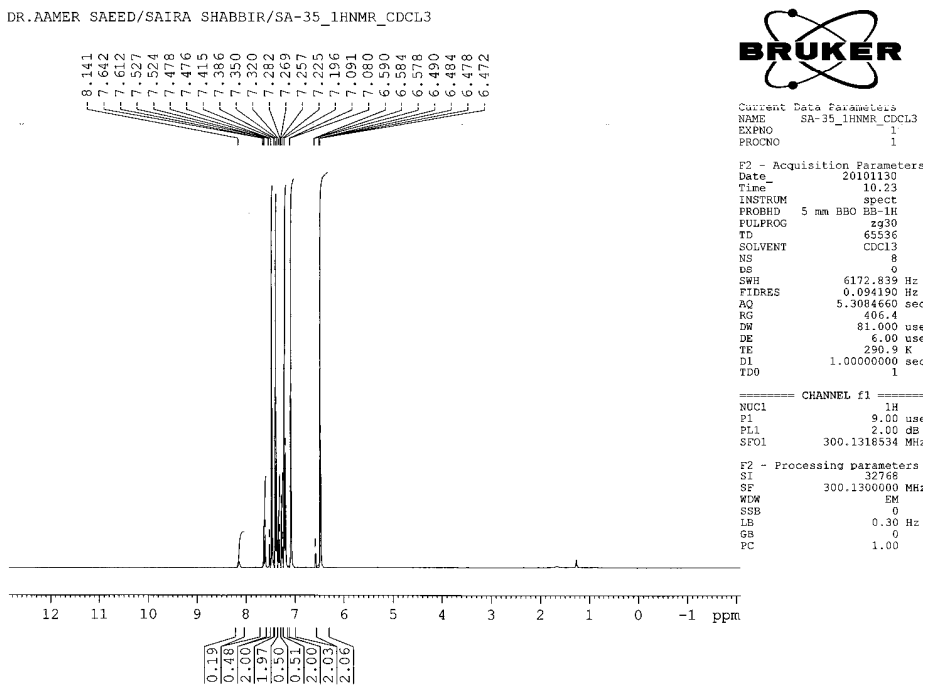


Figure S2. ^{1}HNMR spectrum for the title compound in $CDCl_3$



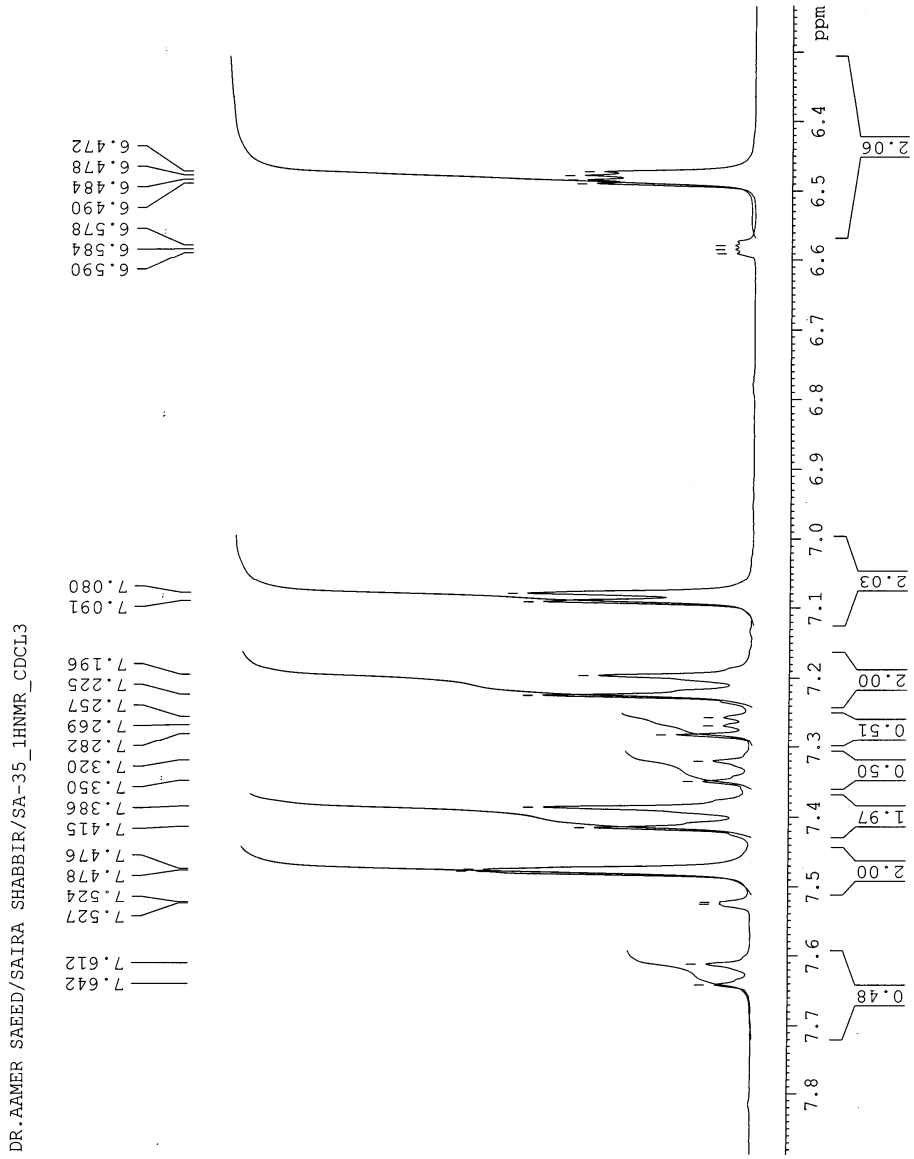
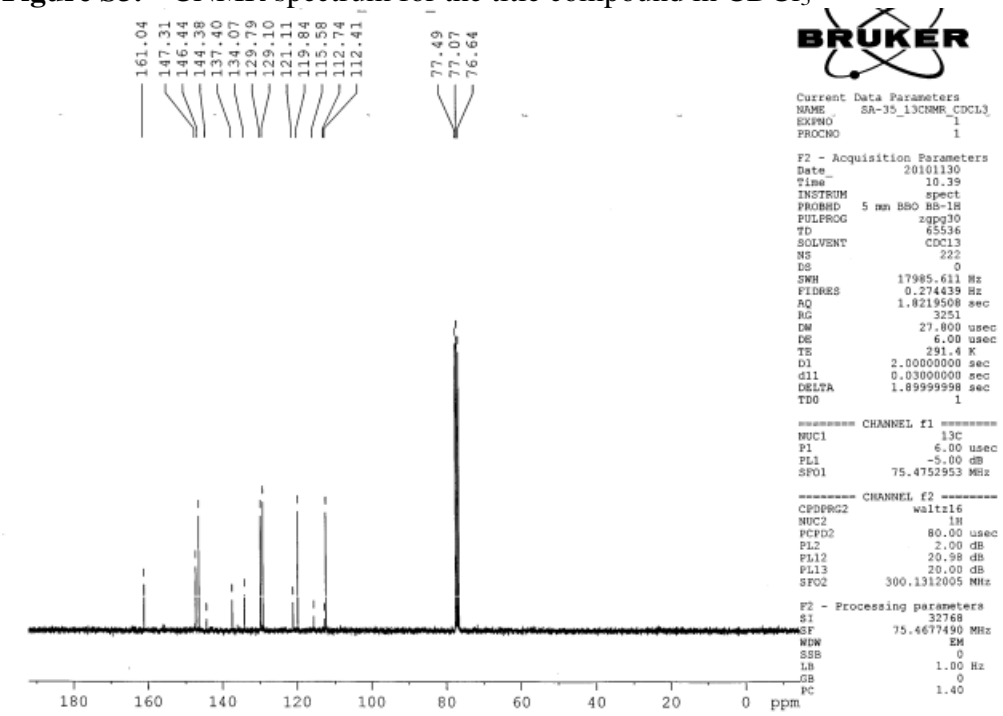
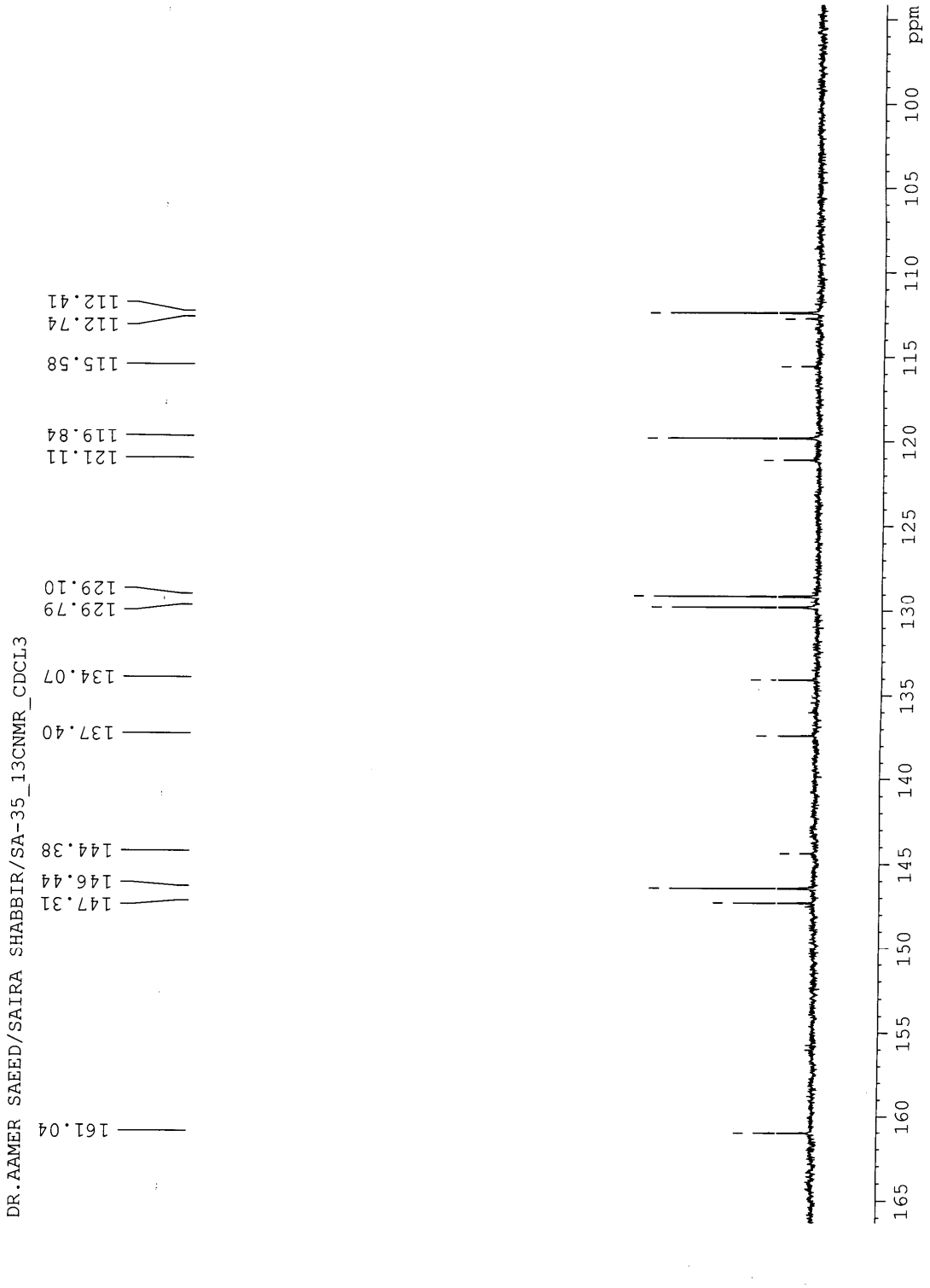

DR.AAMER SAEED/SAIRA SHABBIR/SA-35_1HNMR_CDCL3
7.642
7.612
7.527
7.524
7.478
7.476
7.415
7.386
7.350
7.320
7.282
7.269
7.257
7.225
7.196
7.091
7.080
6.590
6.584
6.578
6.490
6.484
6.478
6.472
ppm
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
6.8
6.7
6.6
6.5
6.4
0.48
2.00
1.97
0.50
0.51
2.00
2.03
2.06

Figure S3. ^{13}CNMR spectrum for the title compound in $CDCl_3$




DR.AAMER SAEED/SAIRA SHABBIR/SA-35_13CNMR_CDCL3
161.04
147.31
146.44
144.38
137.40
134.07
129.79
129.10
121.11
119.84
115.58
112.74
112.41
165
160
155
150
145
140
135
130
125
120
115
110
105
100
ppm