Electronic Supplementary Information

Stereo Configuration-Activity Study of 3-Iodo-4-(2-Methylcyclohexyloxy)-6-Phenethylpyridin-2(2H)-One as Potency Inhibitors of HIV-1 Variants

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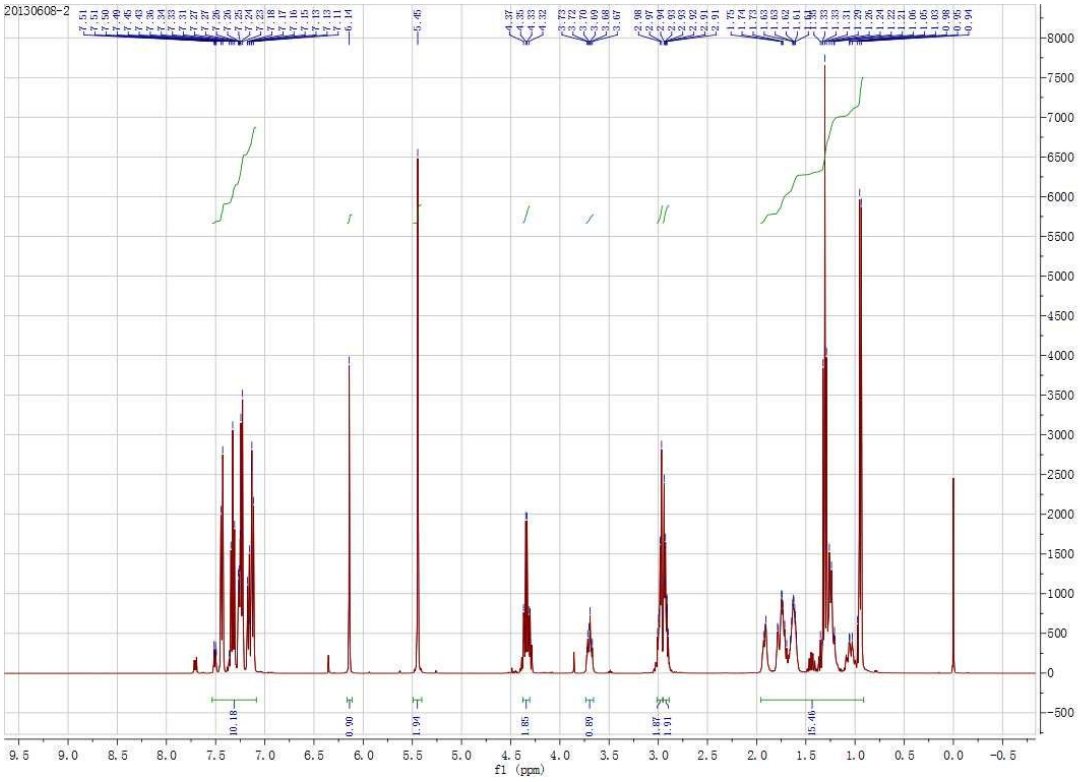
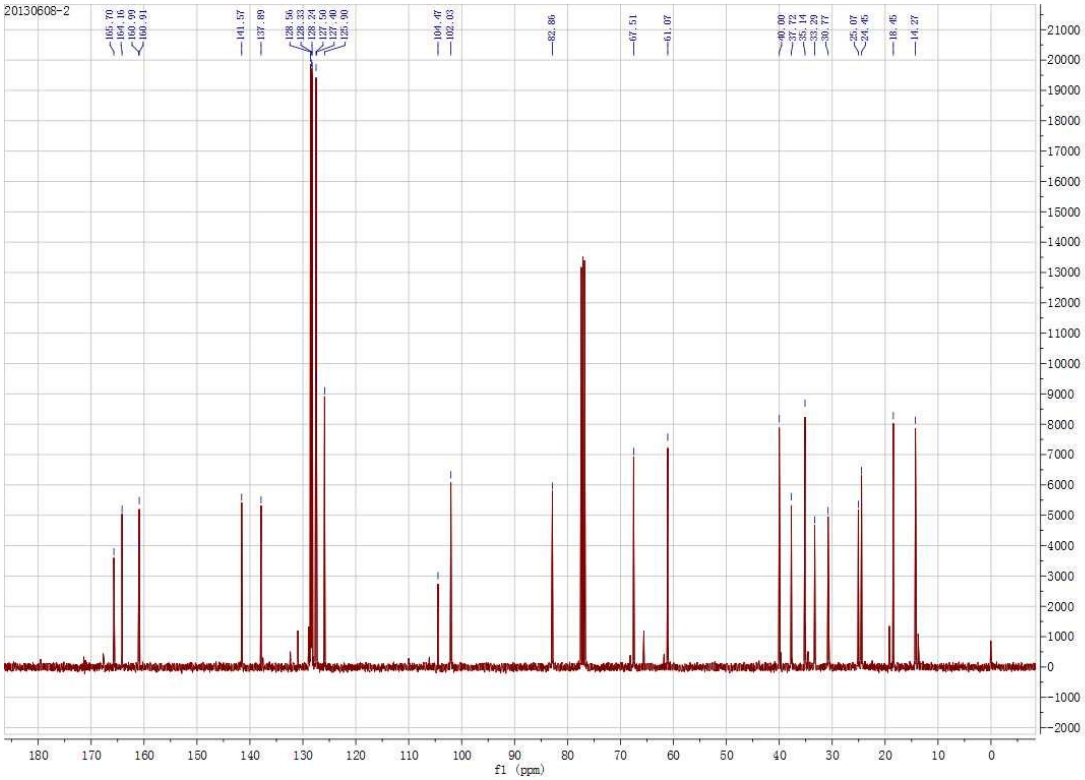
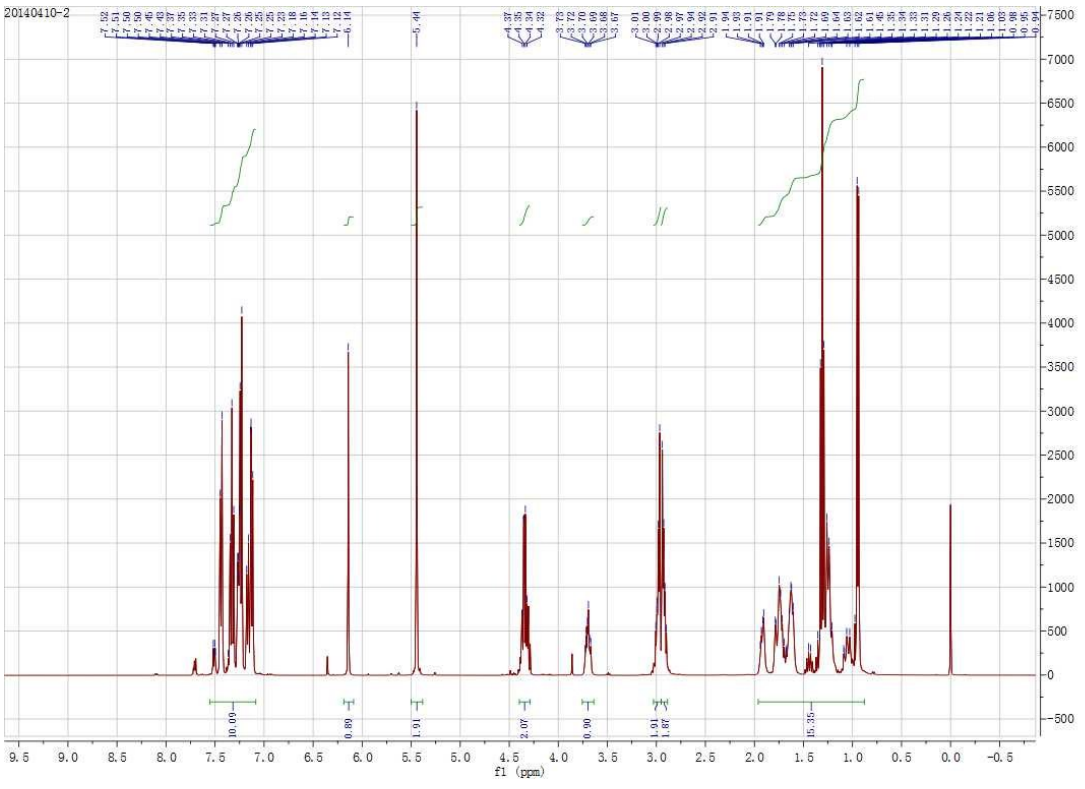
1. NMR spectra



^{1}H-NMR of **4a**



^{13}C-NMR of **4a**



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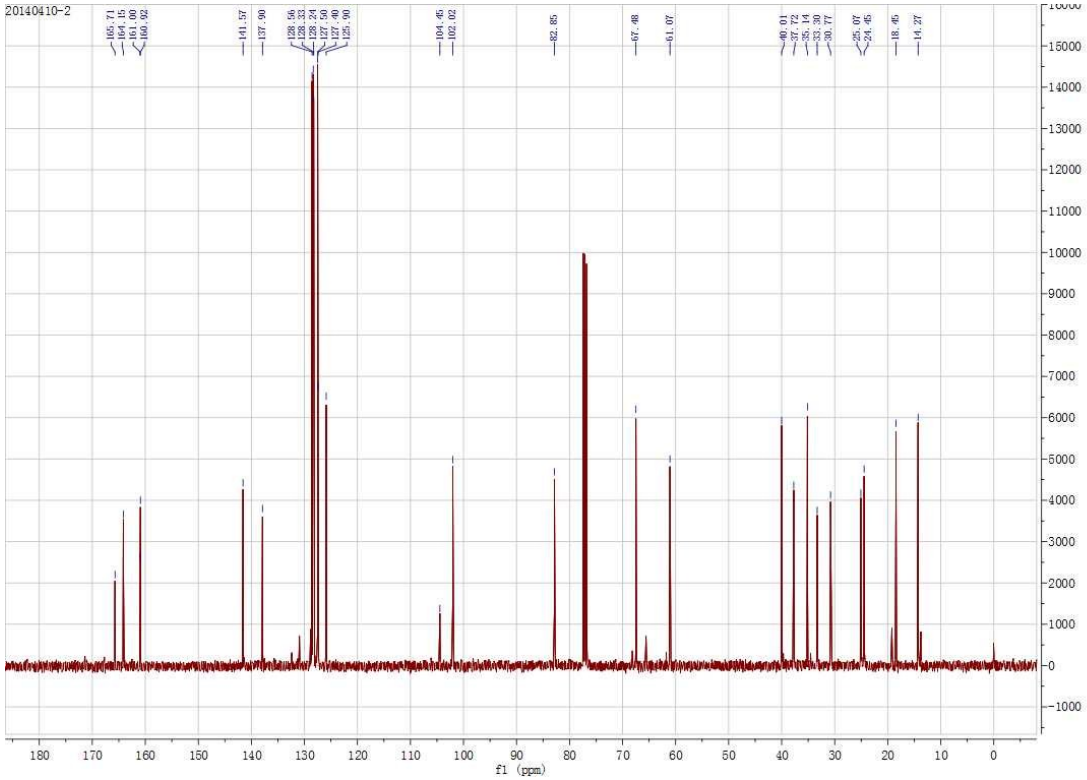

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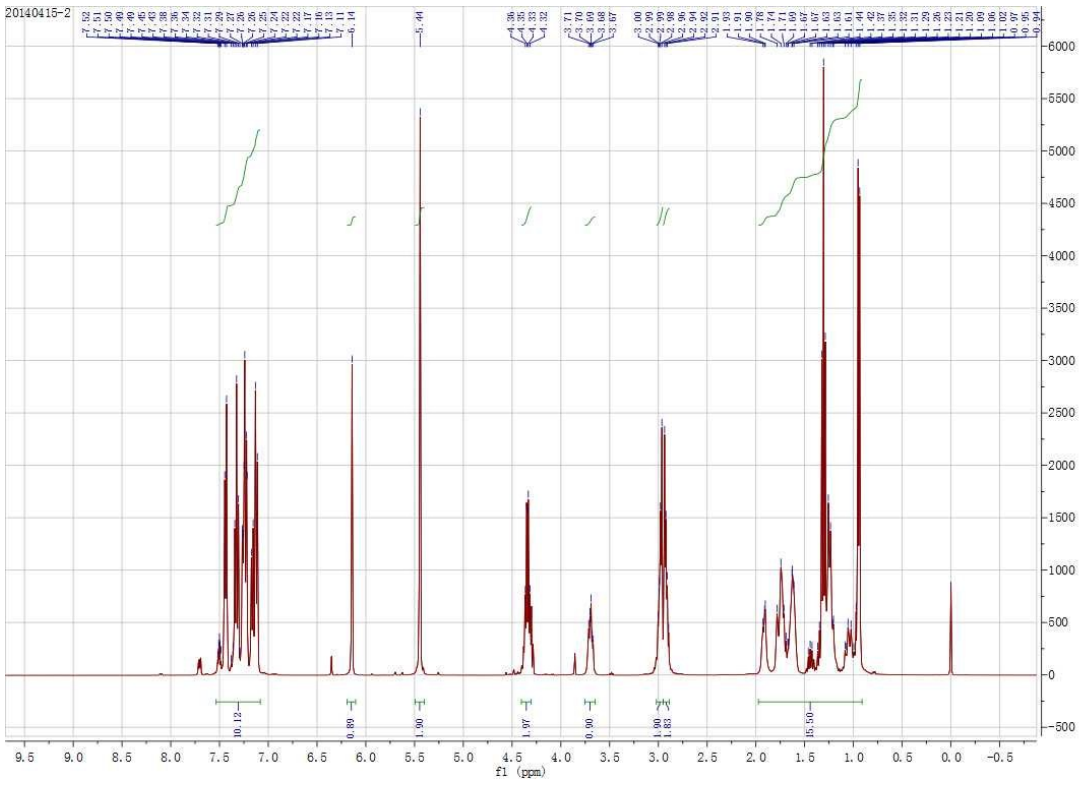

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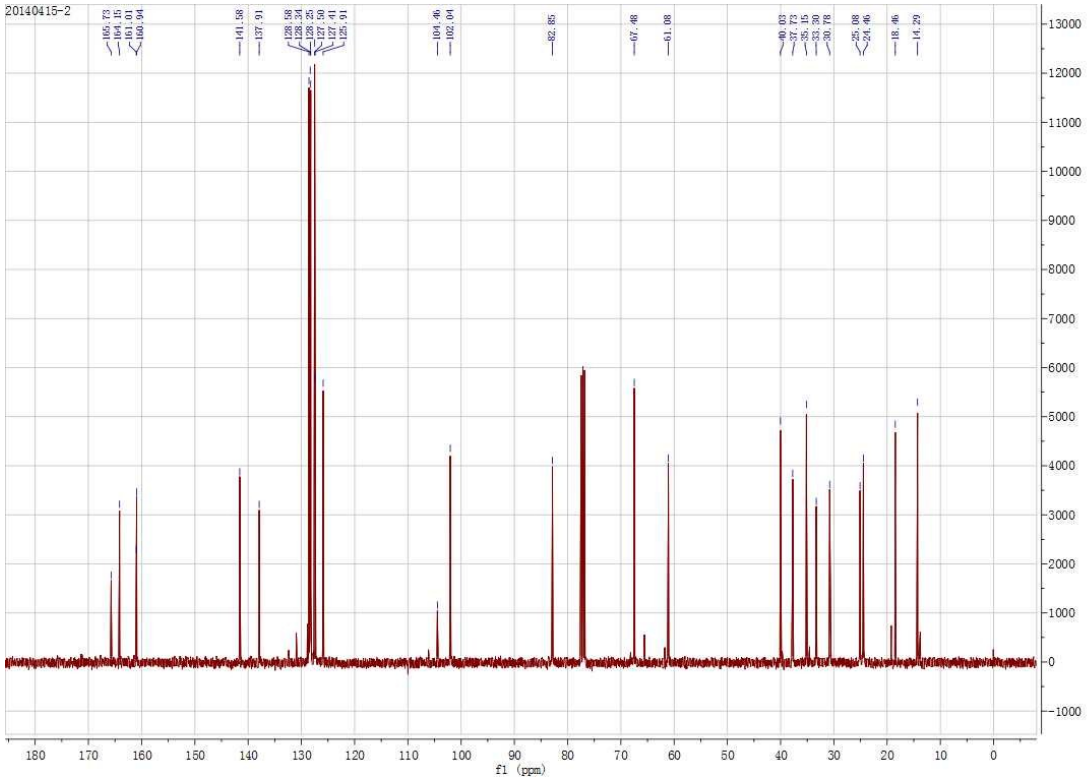

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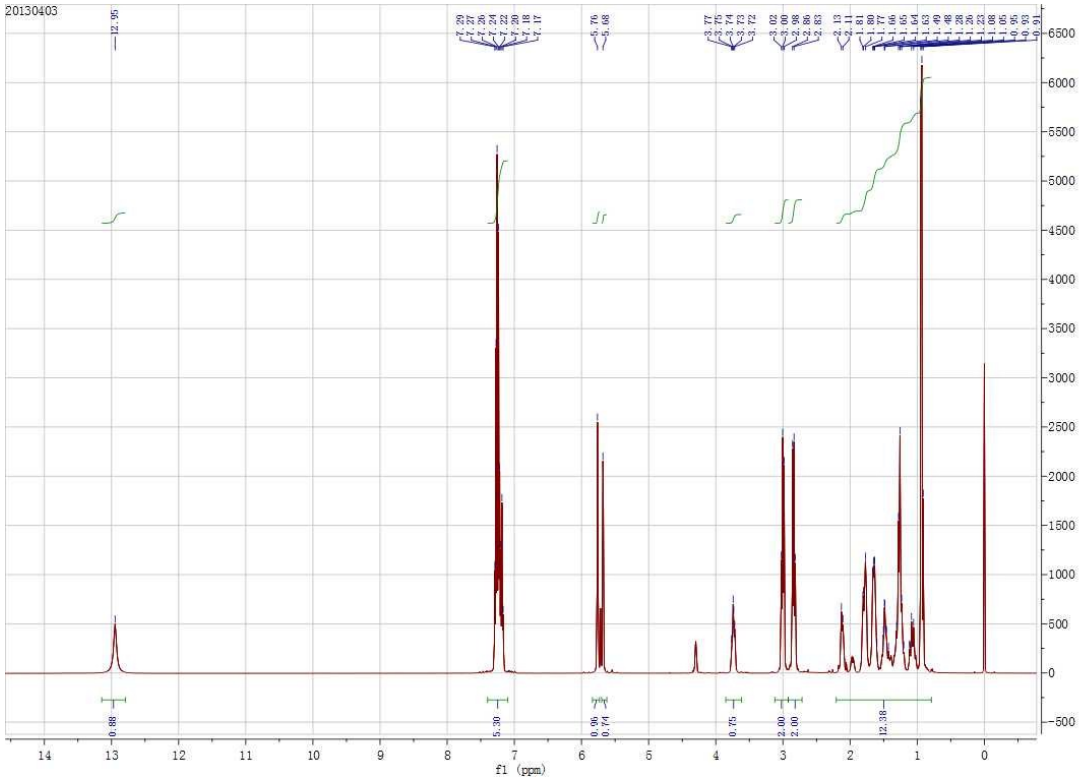

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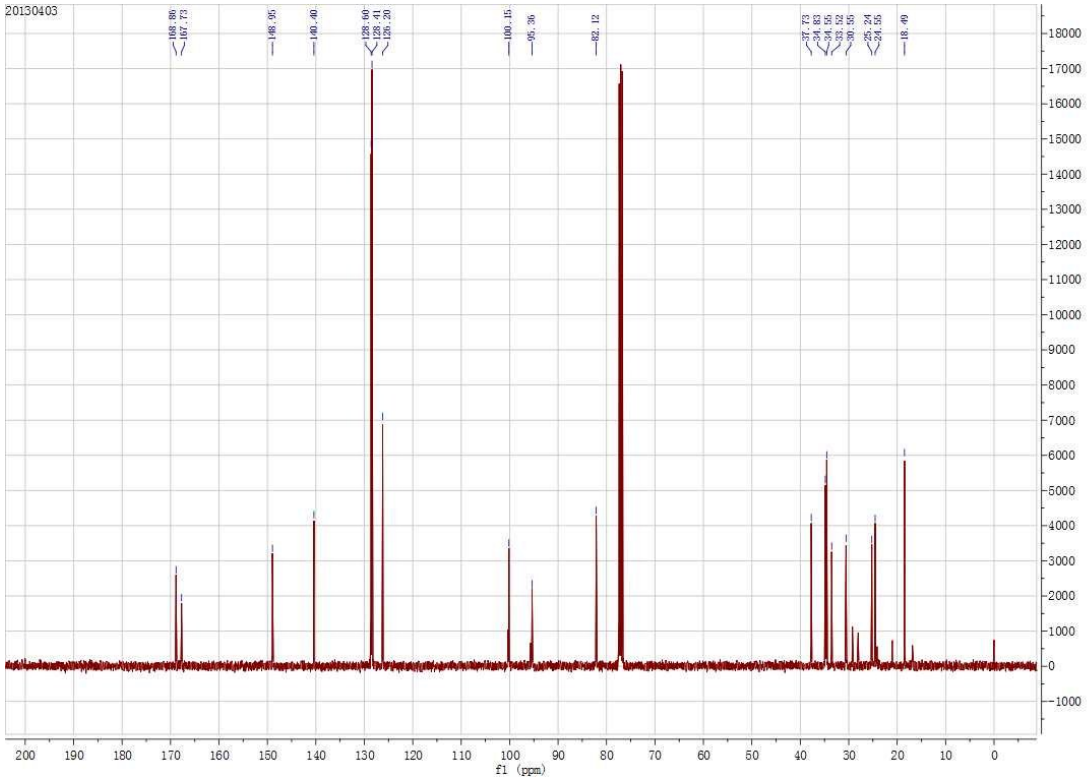

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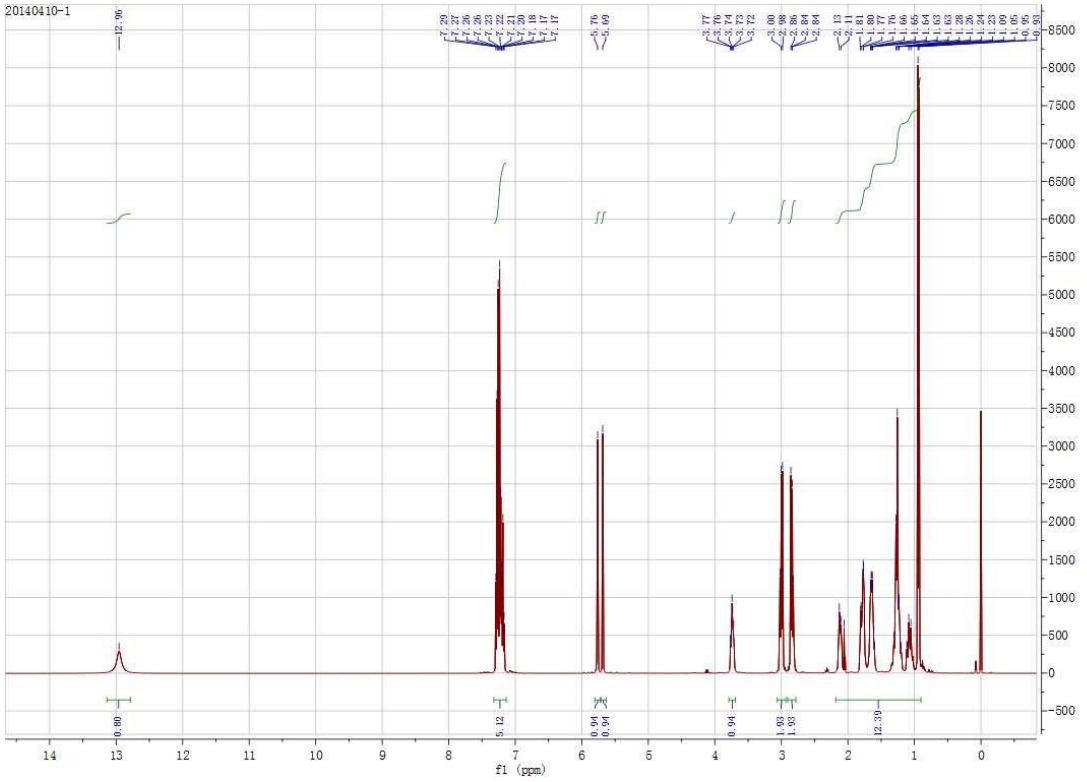

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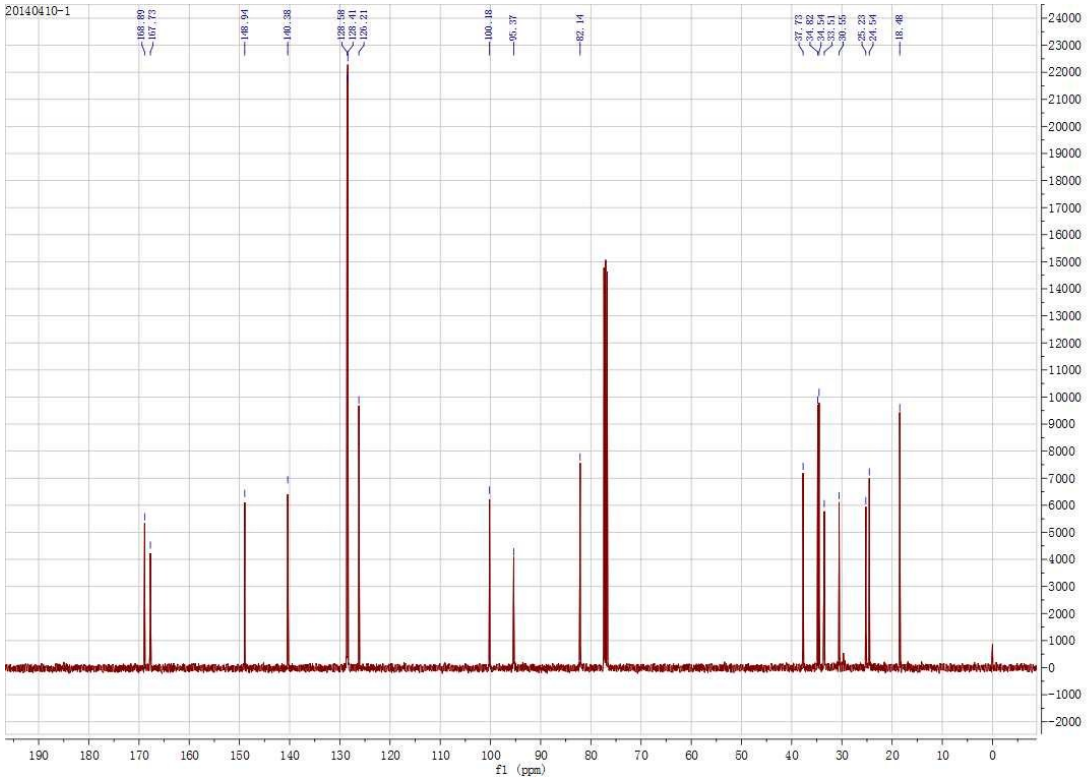

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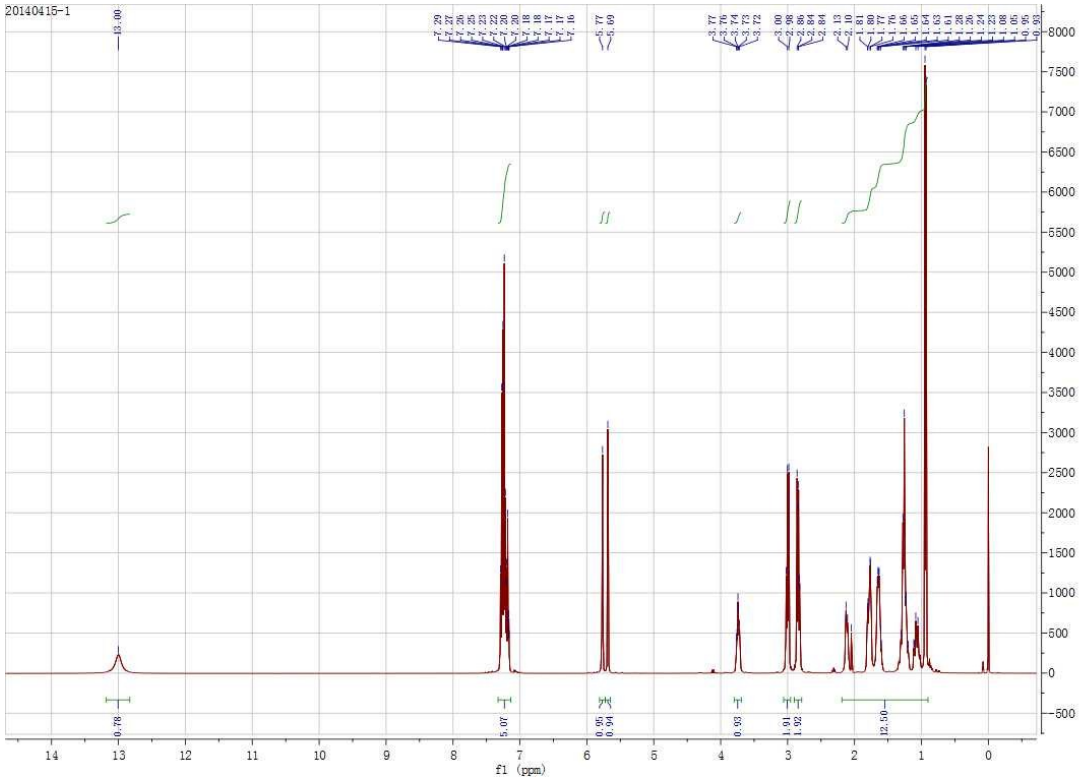

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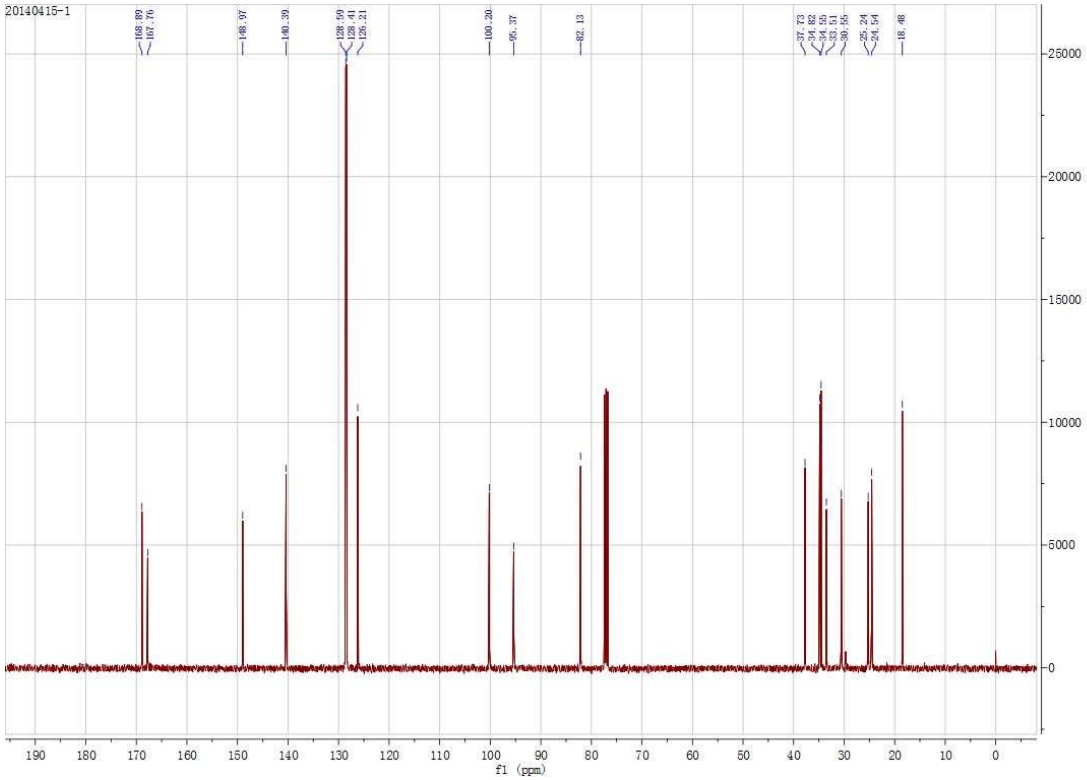

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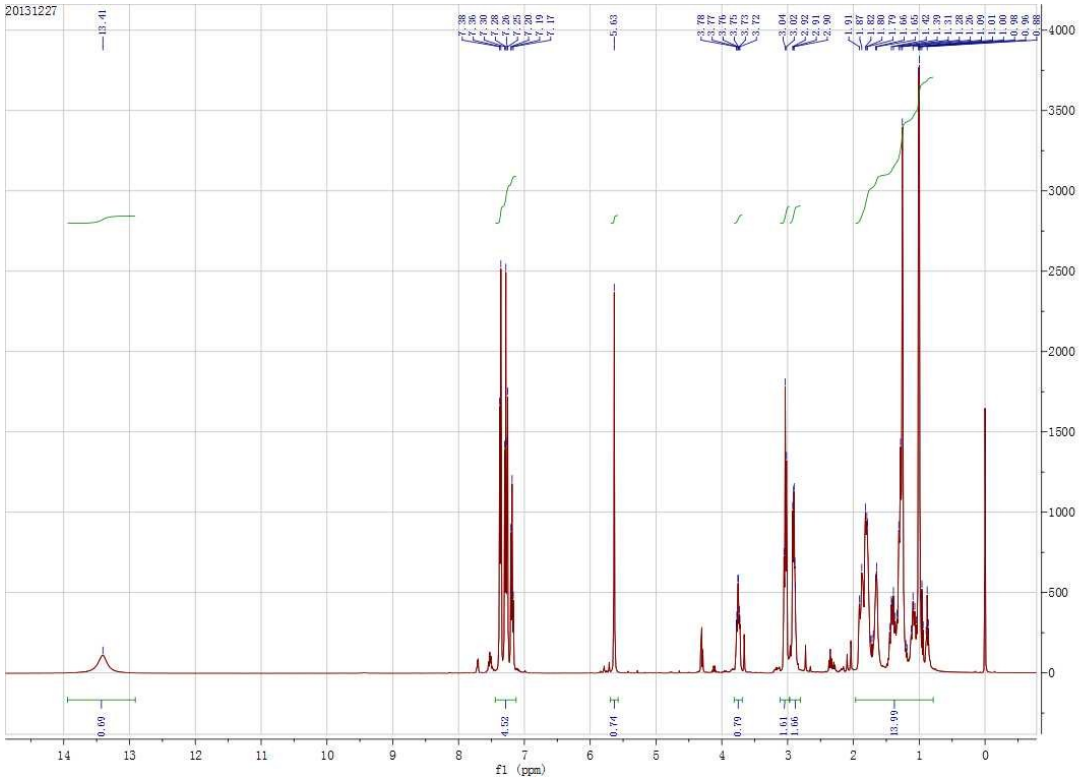

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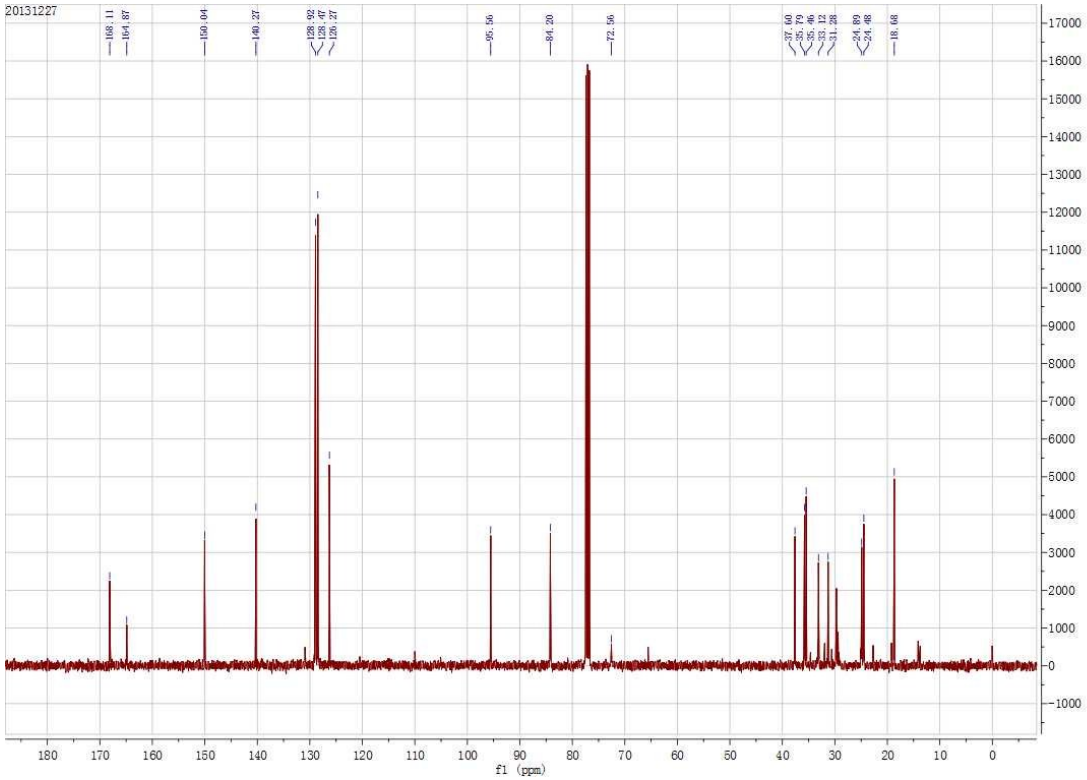
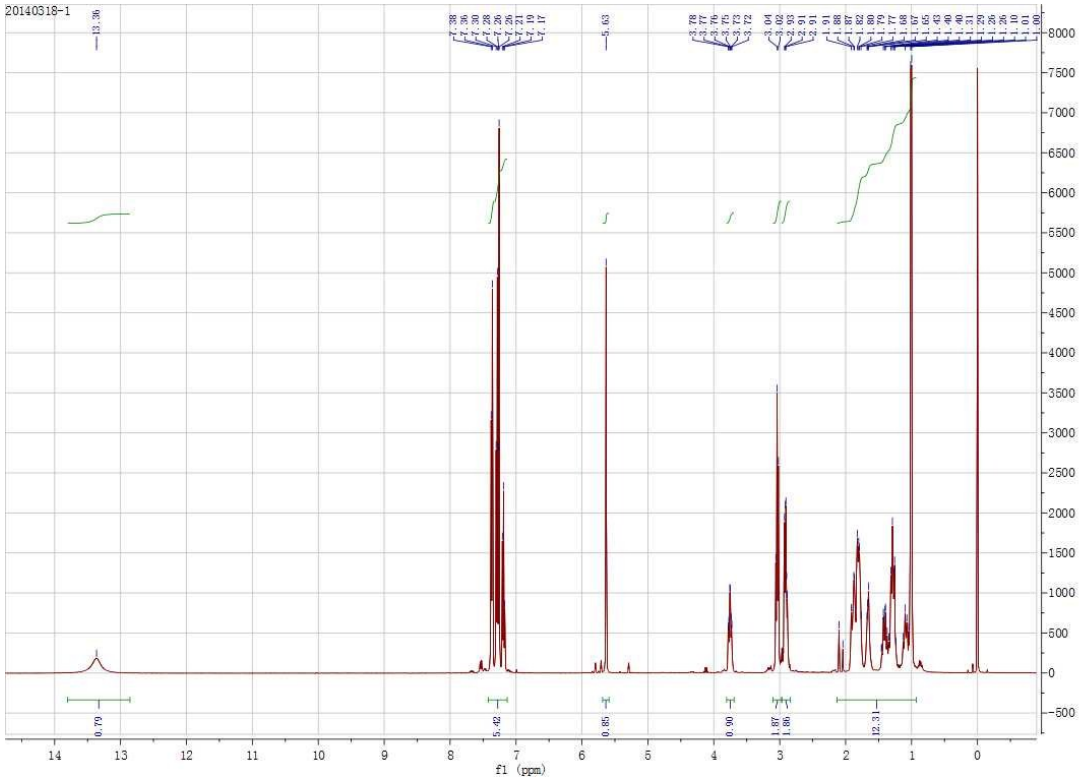


^{13}C-NMR of **2a**



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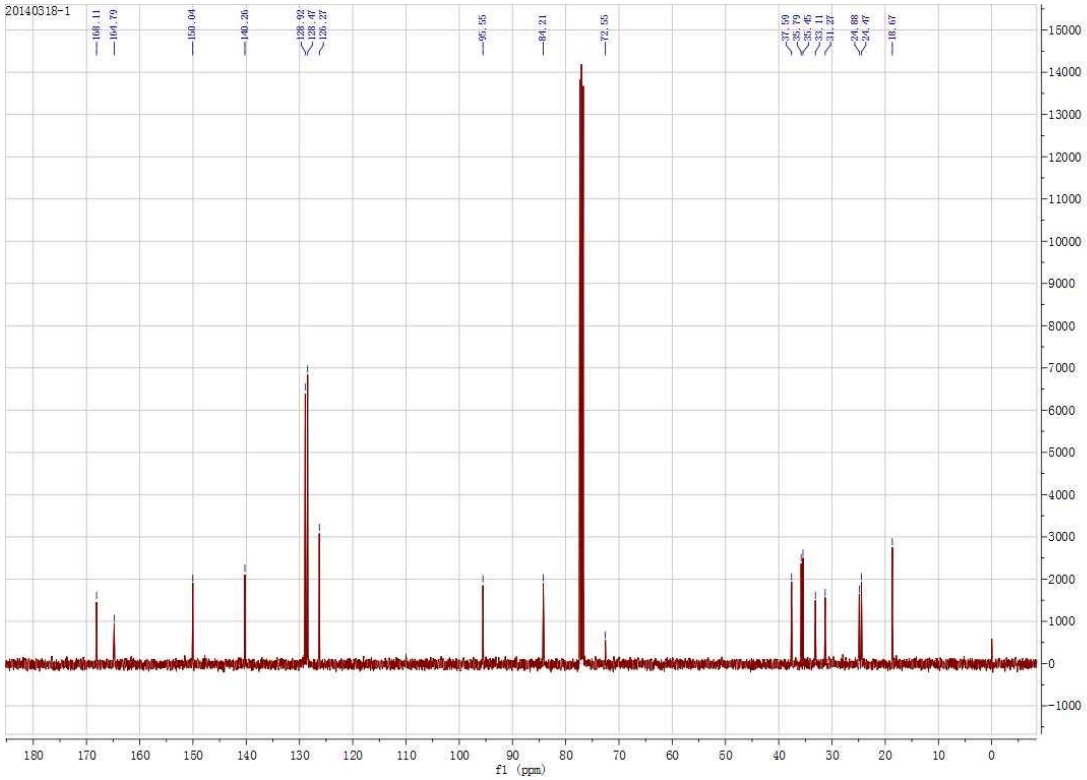

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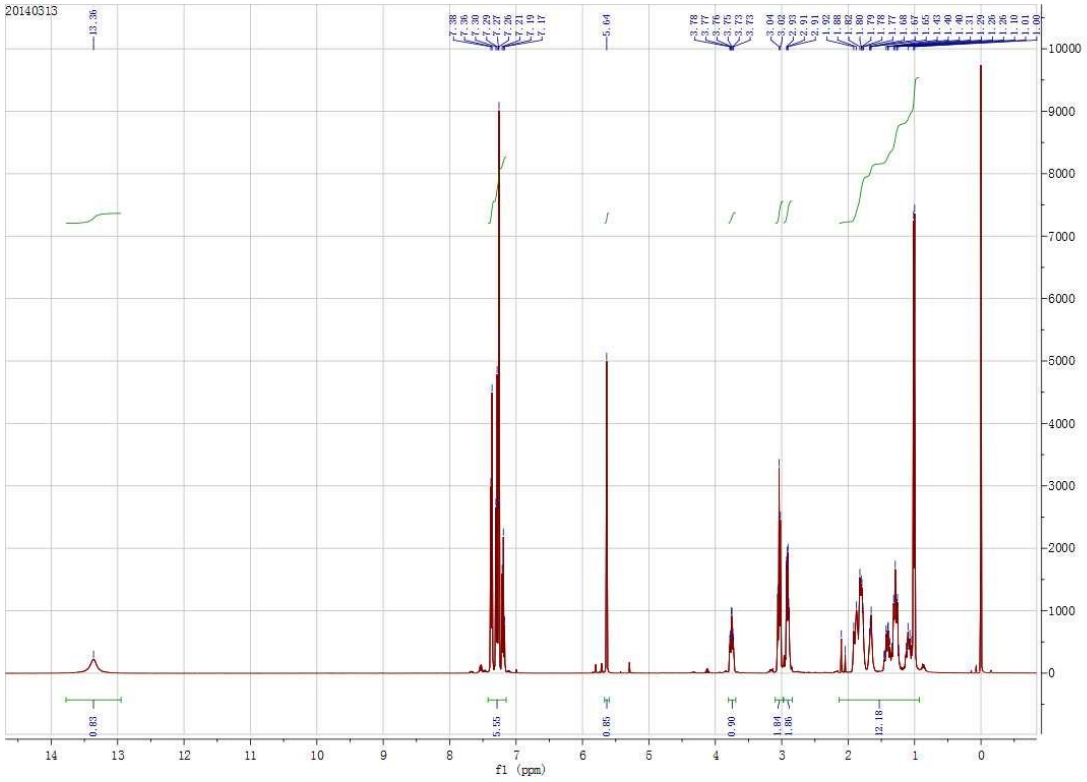

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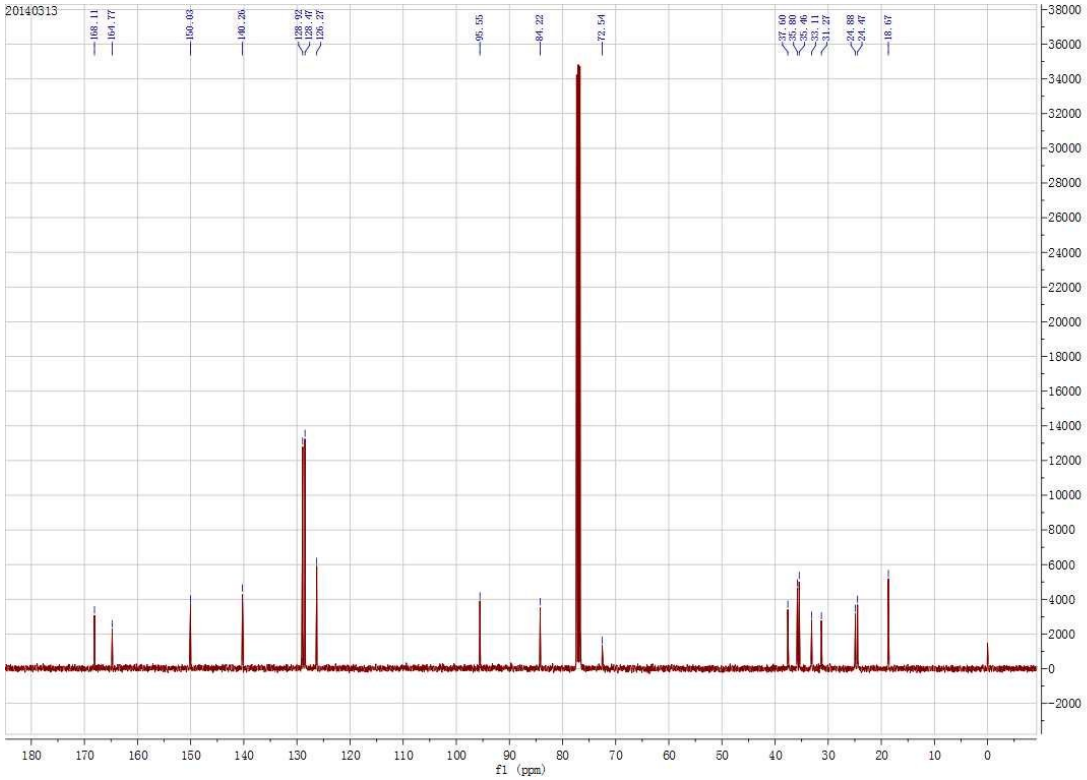

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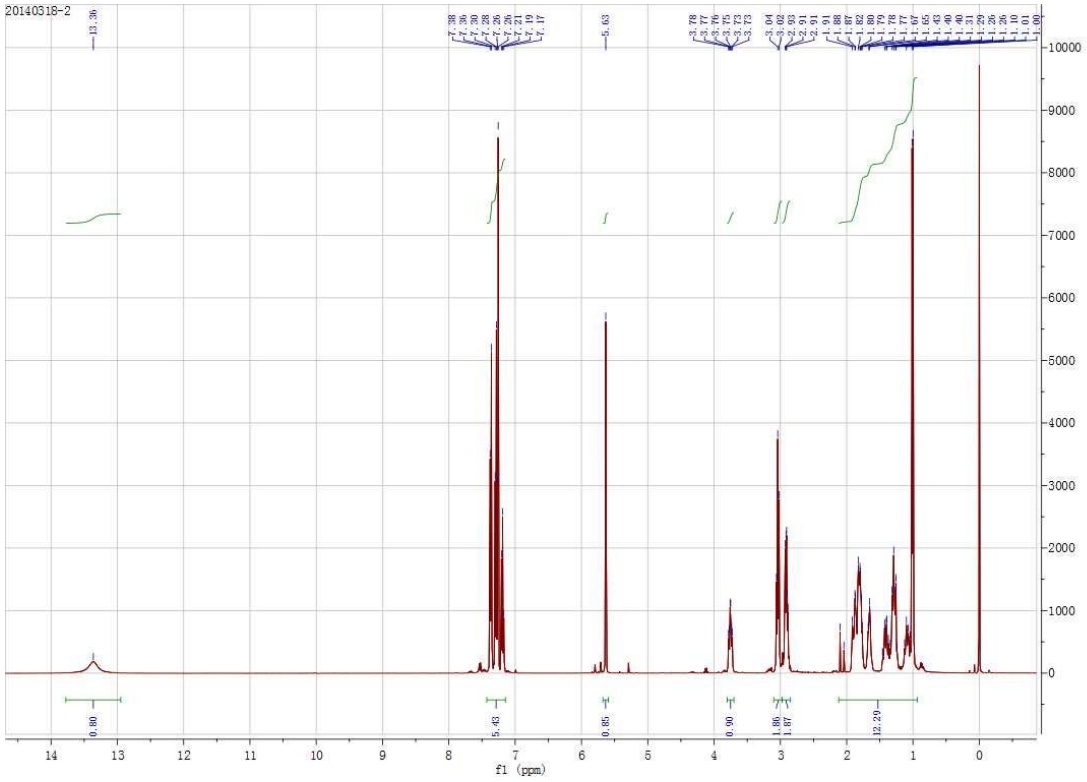
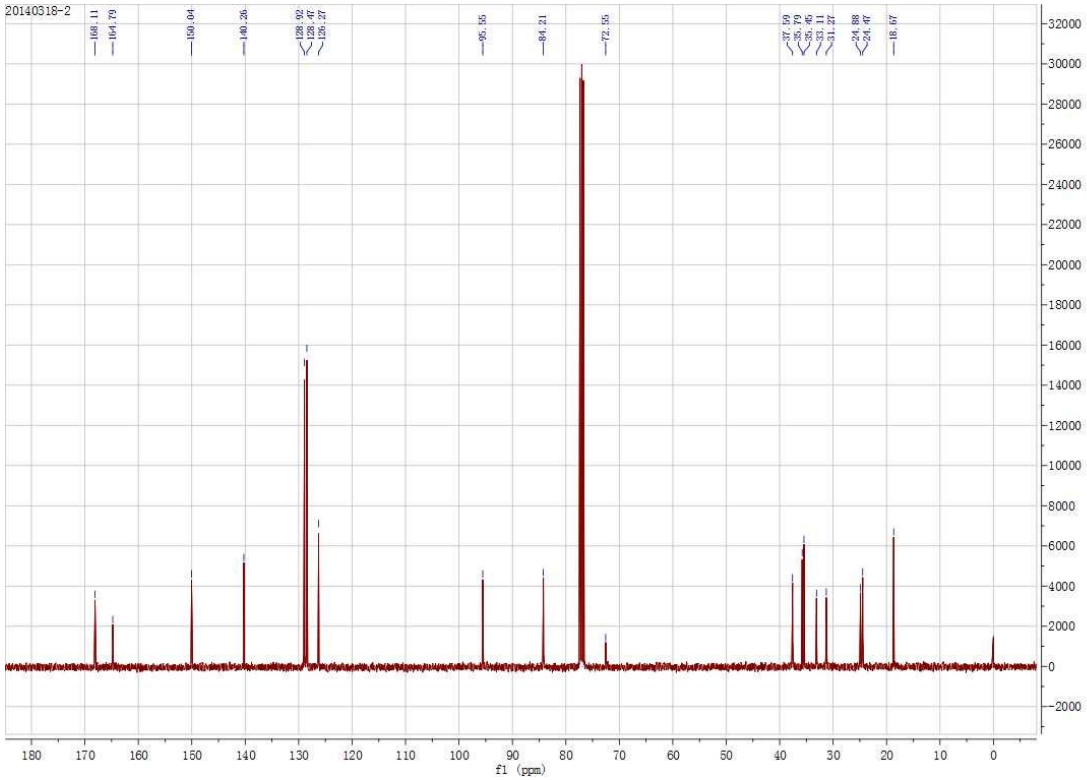


^{1}H-NMR of **2d**



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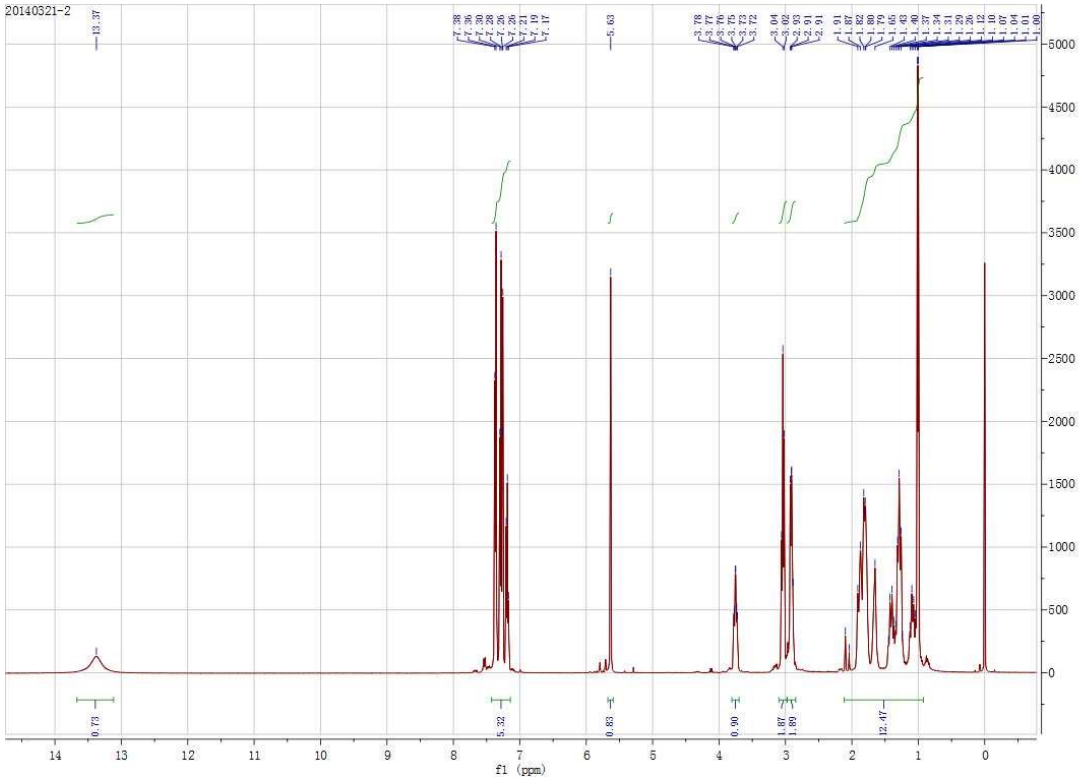

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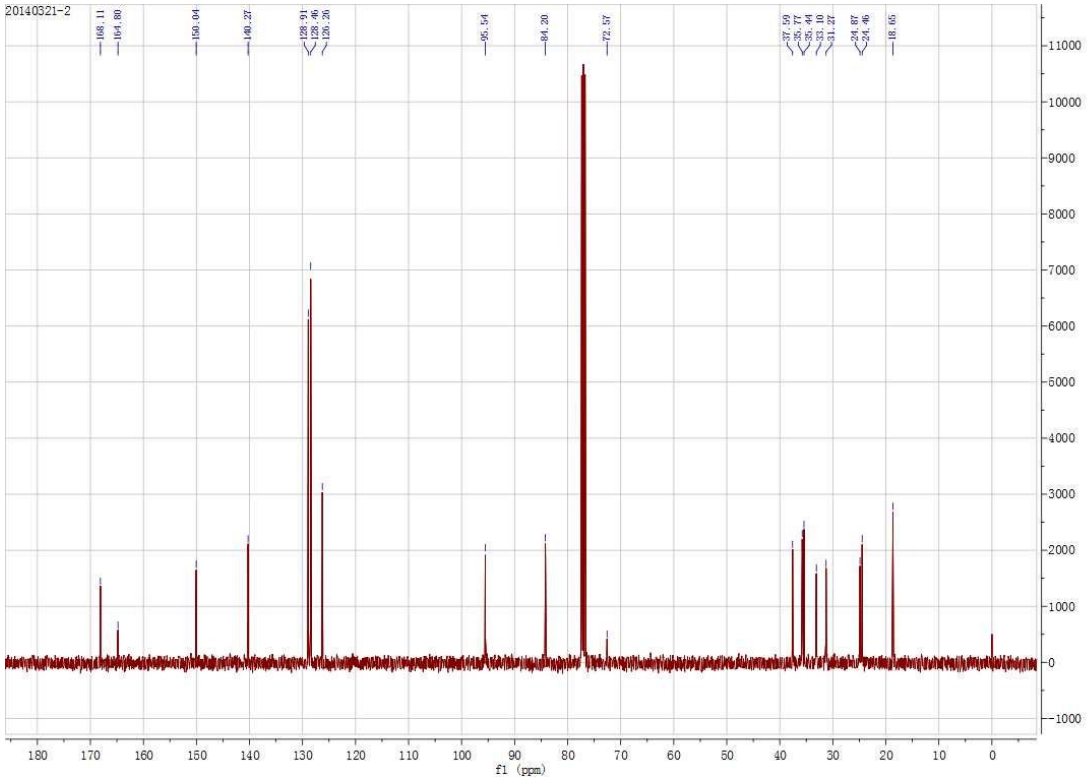

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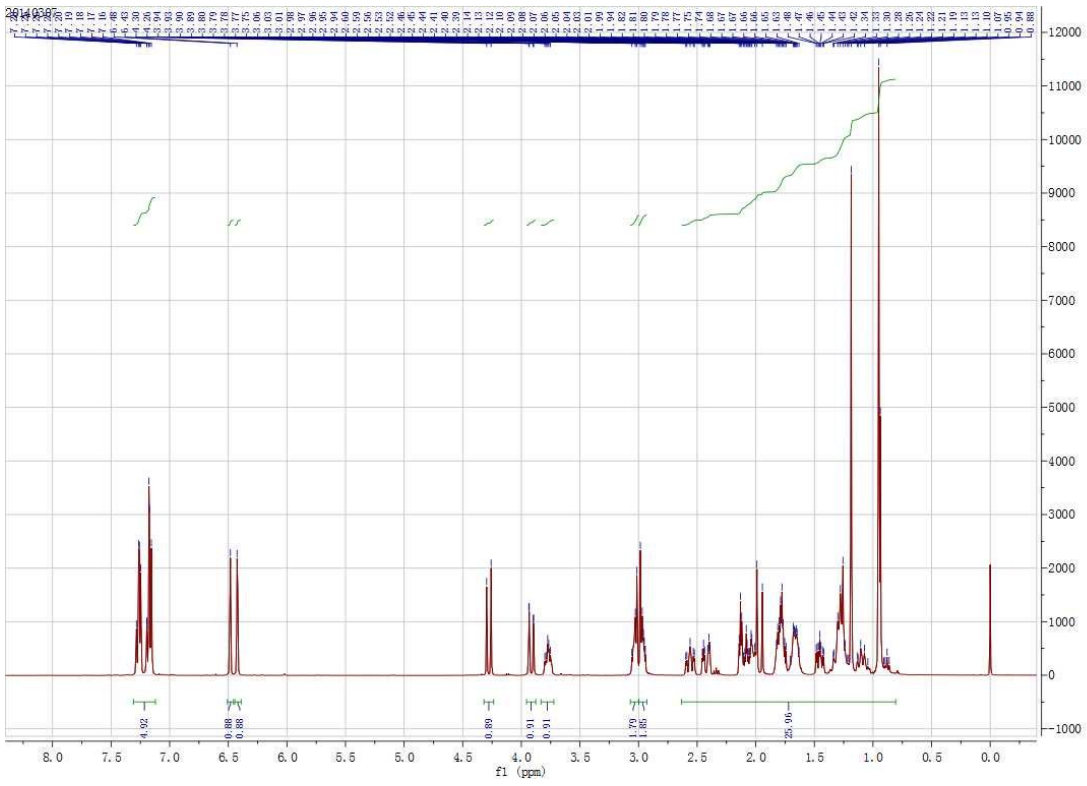
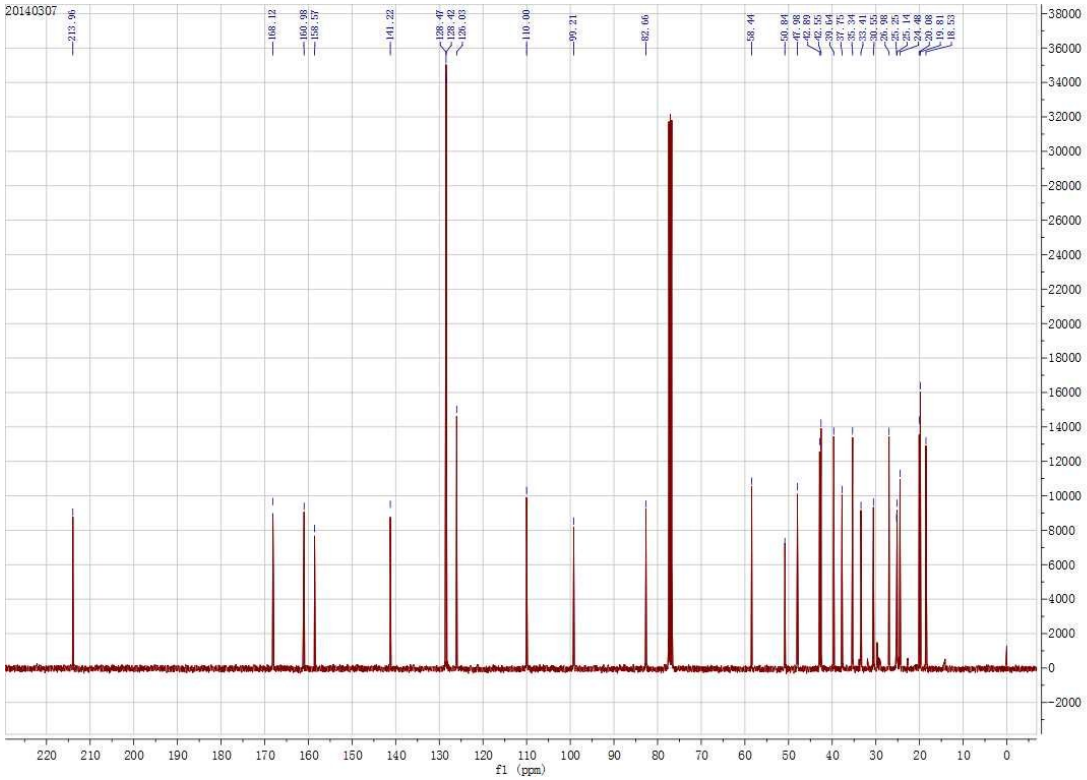


^{1}H-NMR of **6b**



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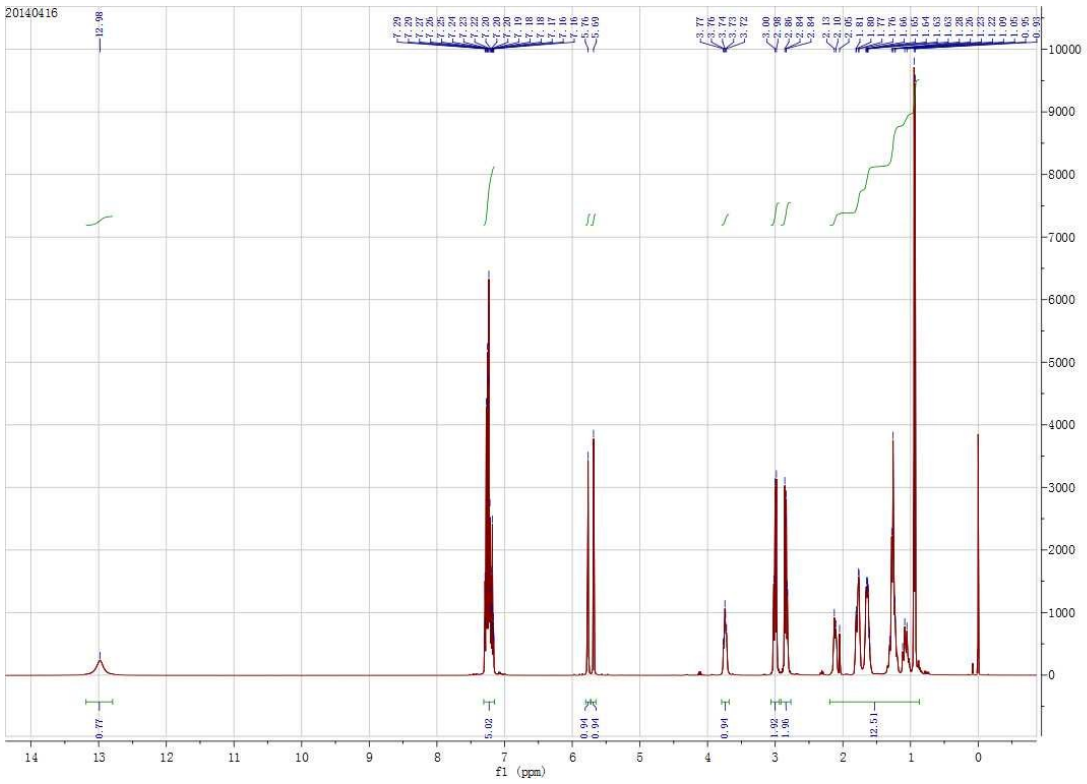

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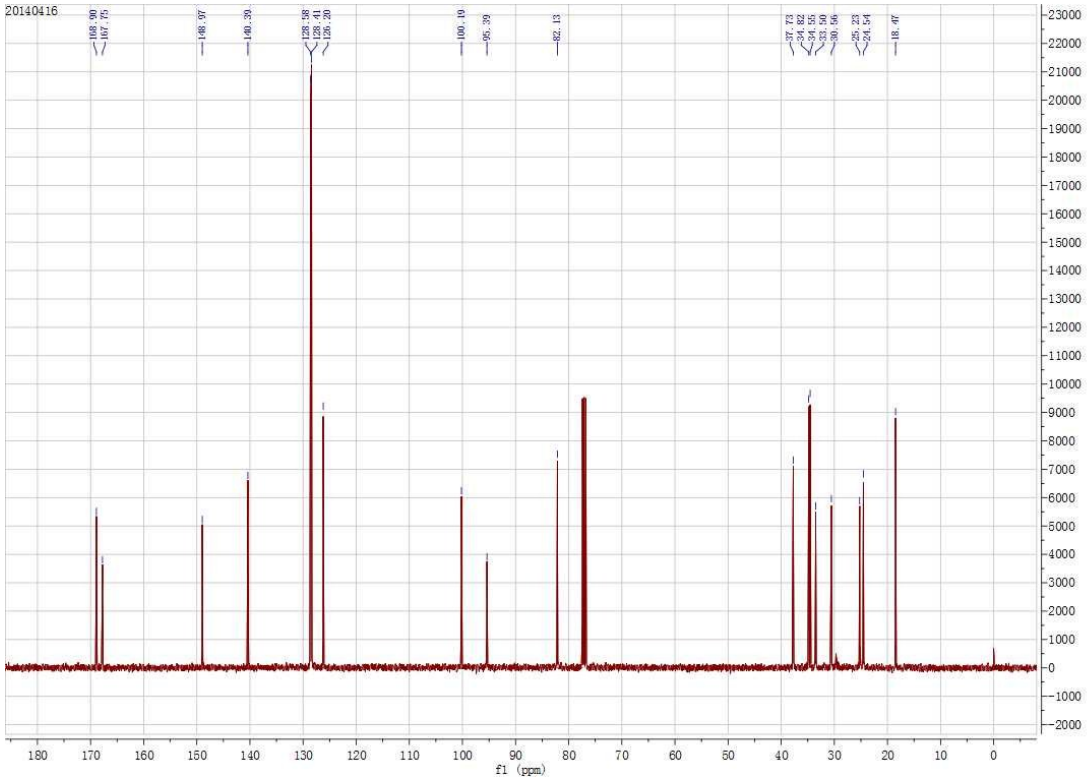

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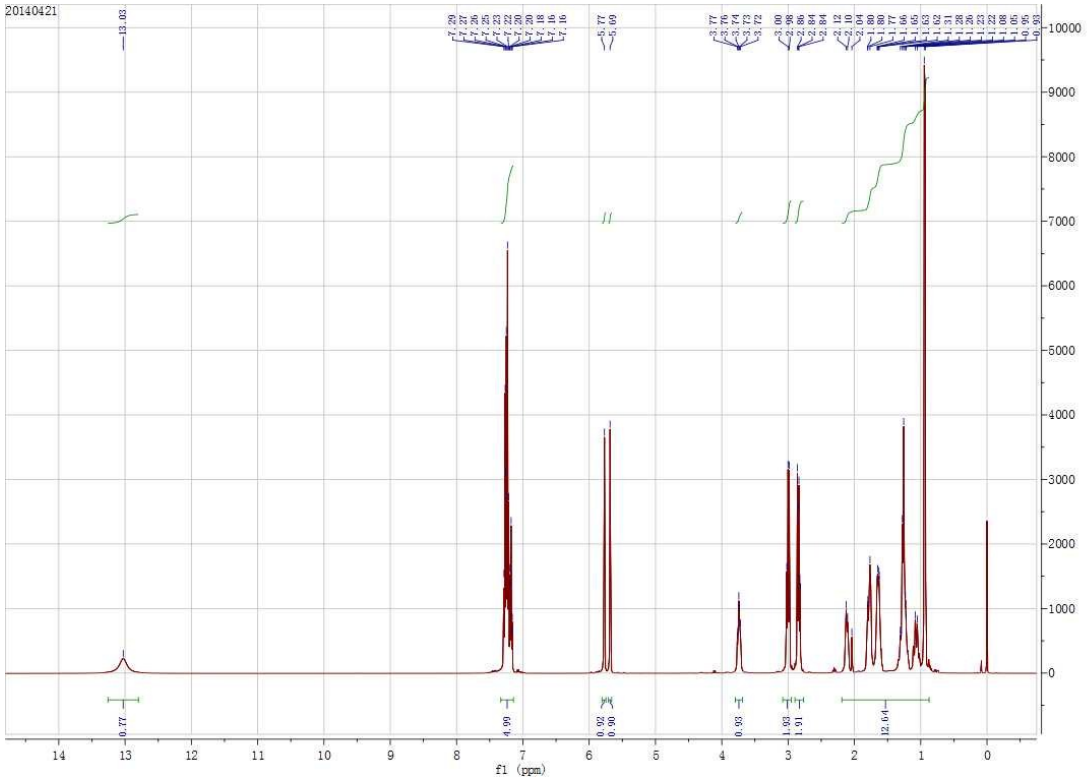

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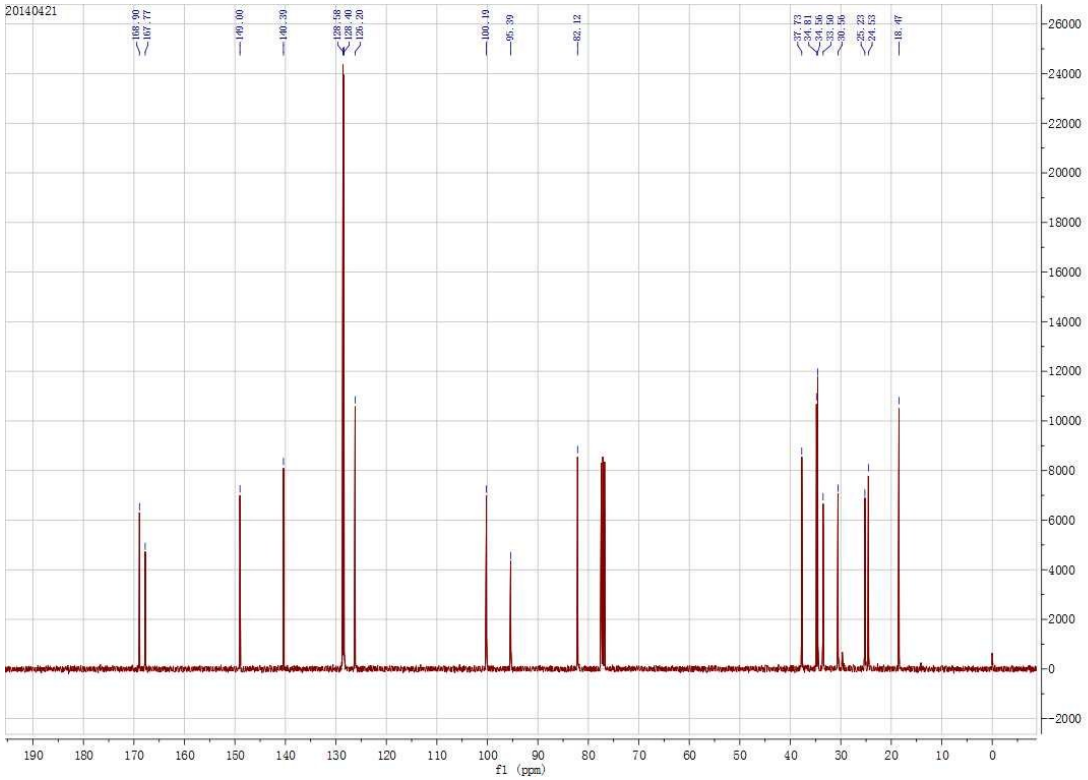

^{13}C-NMR of **5e**

2. Structural Data

Table 1 Crystal data and structure refinement for exp_4139

Identification code	exp_4139
Empirical formula	$C_{20}H_{25}NO_2$
Formula weight	311.41
Temperature/K	180.01(10)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	11.71049(13)
b/Å	11.80555(11)
c/Å	52.7036(5)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	7286.21(13)
Z	16
ρ_{calc}mg/mm^3	1.136
m/mm^{-1}	0.570
F(000)	2688.0

Crystal size/mm^3	0.2 × 0.1 × 0.1
2Θ range for data collection	7.674 to 147.666°
Index ranges	-13 ≤ h ≤ 14, -14 ≤ k ≤ 14, -65 ≤ l ≤ 64
Reflections collected	69569
Independent reflections	14519[R(int) = 0.0339]
Data/restraints/parameters	14519/0/849
Goodness-of-fit on F^2	1.044
Final R indexes [I>=2σ (I)]	R_1 = 0.0433, wR_2 = 0.1111
Final R indexes [all data]	R_1 = 0.0451, wR_2 = 0.1131
Largest diff. peak/hole / e Å^{-3}	0.17/-0.22
Flack parameter	0.05(5)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for exp_4139. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C1	7239.7(18)	3475.4(18)	218.0(4)	32.6(4)
C2	6913.2(19)	2429.5(17)	333.7(4)	35.3(5)
C3	5990(2)	1837.5(19)	246.5(5)	43.6(5)
C4	5325(2)	2279(2)	43.6(5)	46.9(6)
C5	5617(2)	3284.7(19)	-62.7(4)	38.3(5)
C6	4975(2)	3822(2)	-276.7(5)	42.9(5)
C7	5622(4)	3727(3)	-526.4(5)	73(1)
C8	4928(3)	4143(2)	-748.8(5)	56.1(7)
C9	4294(5)	3415(3)	-895.7(7)	96.2(16)
C10	3678(5)	3812(3)	-1104.1(7)	94.0(14)
C11	3690(3)	4923(3)	-1162.8(6)	65.6(8)
C12	4274(3)	5655(3)	-1012.4(6)	58.1(7)
C13	4898(3)	5272(2)	-807.3(5)	51.2(6)
C14	6049(2)	310.7(19)	560.6(5)	45.5(6)
C15	5208(3)	540(3)	775.0(7)	72.4(9)
C16	5535(3)	-108(3)	1013.9(6)	74.4(10)
C17	5610(3)	-1353(3)	960.0(6)	74.5(10)
C18	6443(3)	-1577(2)	747.8(5)	53.3(7)
C19	6161(2)	-940.4(19)	503.5(4)	38.8(5)
C20	7061(3)	-1179(3)	303.6(6)	70.7(9)
C21	3897.5(18)	4683.7(16)	2331.6(4)	29.7(4)
C22	4952.9(18)	4335.4(16)	2220.2(4)	30.0(4)
C23	5524.8(17)	3405.1(17)	2312.4(4)	29.5(4)
C24	5073.4(18)	2776.0(16)	2518.2(4)	30.1(4)
C25	4073.7(18)	3109.2(16)	2623.6(4)	29.3(4)

C26	3540.4(19)	2513.8(17)	2844.0(4)	32.7(4)
C27	3748(2)	3128(2)	3096.5(4)	41.3(5)
C28	3336(2)	2413(2)	3313.8(4)	38.9(5)
C29	3993(2)	1518(2)	3401.9(5)	46.1(6)
C30	3608(3)	802(3)	3591.5(5)	58.8(7)
C31	2550(3)	967(3)	3694.6(5)	62.0(8)
C32	1873(3)	1835(3)	3609.5(5)	61.6(8)
C33	2265(2)	2567(2)	3419.3(5)	48.8(6)
C34	7170.8(18)	3614.2(17)	2037.9(4)	31.4(4)
C35	7998.1(18)	2784.8(18)	1914.9(4)	34.4(4)
C36	8770(2)	3437(2)	1733.1(4)	40.6(5)
C37	9415(2)	4397(2)	1864.6(5)	44.2(5)
C38	8581(2)	5200(2)	1995.3(5)	41.6(5)
C39	7799.9(19)	4552.1(18)	2176.3(4)	34.4(4)
C40	7392(2)	1808(2)	1785.0(5)	51.6(6)
C41	10874.4(18)	5443.3(17)	2712.7(4)	30.9(4)
C42	9834.4(18)	5793.3(18)	2830.4(4)	33.2(4)
C43	9275.2(18)	6740.7(19)	2746.2(4)	34.2(4)
C44	9729.0(19)	7387.5(18)	2541.6(4)	34.7(4)
C45	10727.2(18)	7057.9(17)	2432.8(4)	30.8(4)
C46	11299(2)	7693.3(18)	2221.1(4)	34.4(4)
C47	11137(2)	7119(2)	1962.6(4)	47.7(6)
C48	11701(2)	7772(2)	1751.6(4)	41.1(5)
C49	11110(2)	8577(2)	1613.6(5)	48.8(6)
C50	11625(3)	9158(3)	1415.7(5)	56.8(7)
C51	12744(3)	8939(3)	1354.3(5)	56.7(7)
C52	13349(2)	8156(3)	1492.2(5)	52.3(6)
C53	12835(2)	7575(2)	1690.2(4)	45.2(6)
C54	7759(2)	6769(2)	3070.3(4)	44.2(6)
C55	6546(2)	6442(2)	3010.0(4)	40.8(5)
C56	5886(2)	6213(3)	3253.0(6)	53.2(6)
C57	5919(3)	7185(3)	3434.7(7)	77.2(11)
C58	7135(4)	7465(4)	3499.1(6)	91.3(14)
C59	7804(3)	7759(4)	3257.0(6)	80.1(12)
C60	6515(4)	5428(3)	2834.8(6)	82.9(11)
C61	7972.2(17)	6452.2(17)	-173.5(4)	30.5(4)
C62	8304.6(18)	7494.0(17)	-288.6(4)	31.3(4)
C63	9210.2(17)	8099.9(17)	-194.1(4)	30.1(4)
C64	9830.0(17)	7694.7(17)	18.8(4)	31.5(4)
C65	9517.6(17)	6697.0(18)	125.8(4)	30.9(4)

C66	10089.8(18)	6205.8(19)	354.7(4)	34.3(4)
C67	9373(2)	6359(2)	596.6(4)	40.0(5)
C68	10002(2)	5921.1(19)	825.9(4)	36.1(5)
C69	10853(2)	6555(2)	938.4(4)	43.2(5)
C70	11449(3)	6153(3)	1146.8(5)	56.6(7)
C71	11196(3)	5108(3)	1245.3(5)	62.0(8)
C72	10359(3)	4460(3)	1134.6(6)	59.9(8)
C73	9760(2)	4860(2)	925.9(5)	46.7(6)
C74	8991.8(18)	9673.9(18)	-483.7(4)	32.3(4)
C75	9819(2)	10492.5(18)	-610.6(4)	36.4(5)
C76	9166(3)	11154(2)	-814.0(5)	47.9(6)
C77	8139(3)	11778(2)	-706.6(6)	56.5(7)
C78	7344(2)	10953(2)	-572.0(6)	54.6(7)
C79	7987(2)	10291.7(19)	-368.1(5)	40.4(5)
C80	10863(3)	9901(2)	-717.2(6)	55.5(7)
N1	6553.4(15)	3850.1(15)	22.7(4)	33.8(4)
N2	3514.5(15)	4033.1(14)	2532.2(3)	30.1(3)
N3	11260.4(16)	6108.0(14)	2515.7(3)	30.6(3)
N4	8615.0(15)	6104.0(15)	30.7(3)	31.5(4)
O1	8089.8(13)	4051.6(13)	281.6(3)	39.8(4)
O2	5620(2)	830.0(17)	330.7(4)	72.2(7)
O3	3320.7(13)	5517.1(12)	2260.7(3)	36.4(3)
O4	6505.7(13)	2975.3(12)	2219.3(3)	36.1(3)
O5	11436.0(14)	4586.6(13)	2777.3(3)	38.9(3)
O6	8288.2(15)	7171.1(17)	2838.3(3)	52.9(5)
O7	7140.6(13)	5858.6(13)	-243.8(3)	37.5(3)
O8	9617.5(13)	9089.1(13)	-287.0(3)	36.3(3)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for exp_4139. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka×b×U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	28.4(10)	31.5(10)	38(1)	4.4(8)	2.9(8)	2.9(8)
C2	33.2(11)	28.9(10)	43.8(11)	7.1(8)	-3.9(9)	0.9(8)
C3	44.9(13)	29.1(11)	56.9(14)	10.6(10)	-10.7(11)	-4(1)
C4	44.6(13)	36.8(12)	59.3(15)	9.1(11)	-17.8(11)	-12(1)
C5	37.0(12)	33.9(10)	43.8(12)	5.0(9)	-6.7(9)	-0.9(9)
C6	37.9(12)	40.0(12)	50.6(13)	8.1(10)	-10.5(10)	-2.8(10)
C7	97(3)	75(2)	47.4(15)	-2.6(14)	-9.3(16)	51(2)

C8	75.8(19)	52.6(15)	39.9(13)	-4.8(11)	-4.7(13)	28.1(15)
C9	180(5)	43.0(16)	66(2)	-8.9(15)	-36(3)	21(2)
C10	150(4)	72(2)	60(2)	-9.5(17)	-40(2)	-5(3)
C11	70(2)	79(2)	48.5(15)	15.6(14)	-8.7(14)	11.8(17)
C12	62.6(18)	54.2(16)	57.4(16)	17.5(13)	3.2(14)	7.9(14)
C13	53.1(16)	50.9(14)	49.7(14)	4.4(11)	-0.3(12)	1.8(12)
C14	49.4(14)	29.9(11)	57.3(14)	10.5(10)	-12.0(12)	-7.3(10)
C15	66(2)	56.4(18)	94(2)	-23.6(17)	-3.9(18)	22.2(16)
C16	66(2)	100(3)	56.9(18)	-12.5(17)	19.1(16)	4.7(19)
C17	79(2)	84(2)	60.4(18)	26.5(17)	15.7(17)	-12.7(19)
C18	62.2(17)	35.6(12)	62.0(16)	11.4(11)	0.2(13)	-0.4(12)
C19	33.9(11)	36.0(11)	46.6(12)	0.4(9)	1.7(9)	-6.8(9)
C20	55.0(18)	103(3)	54.2(16)	-19.0(17)	10.2(14)	-3.3(18)
C21	29.3(10)	25.2(9)	34.7(10)	0.1(8)	3.2(8)	-1.5(8)
C22	29(1)	27.5(9)	33.7(10)	1.3(7)	6.0(8)	-1.4(8)
C23	26.4(10)	27.3(9)	34.9(10)	-1.8(8)	4.1(8)	-1.6(8)
C24	31.1(10)	24.9(8)	34.4(10)	1.2(8)	2.2(8)	0.4(8)
C25	30.5(10)	23.9(9)	33.4(10)	-0.8(7)	2.7(8)	-3.9(7)
C26	35.8(11)	27.6(9)	34.7(10)	0.8(8)	6.0(8)	-3.1(8)
C27	52.9(14)	36.2(11)	34.9(11)	-1.2(9)	3.9(10)	-8.8(10)
C28	44.7(13)	42.8(12)	29.3(10)	-6.2(9)	3.9(9)	-8.4(10)
C29	41.9(13)	57.9(15)	38.7(12)	6.2(11)	0.7(10)	-6.9(11)
C30	67.3(18)	67.6(18)	41.6(13)	14.4(12)	-9.6(13)	-14.3(15)
C31	73(2)	76(2)	36.2(13)	4.3(13)	7.0(13)	-28.4(17)
C32	59.2(17)	82(2)	43.3(13)	-21.2(14)	23.8(13)	-20.6(16)
C33	50.2(14)	52.0(14)	44.1(13)	-13.9(11)	10.1(11)	-1.9(12)
C34	28.2(10)	32.4(10)	33.5(10)	5.1(8)	6.3(8)	2.1(8)
C35	30.9(11)	35.3(10)	37(1)	-0.5(8)	4.6(8)	4.2(9)
C36	36.9(12)	48.7(13)	36.1(11)	0.2(9)	10.8(9)	4.5(10)
C37	34.5(12)	48.7(13)	49.5(13)	2.2(10)	13.1(10)	-5.6(10)
C38	41.6(13)	34.9(11)	48.4(13)	2.2(9)	9.7(10)	-6.2(10)
C39	32.8(11)	34.9(10)	35.4(10)	2.5(8)	7.6(8)	2.4(9)
C40	47.3(15)	49.0(14)	58.5(15)	-15.4(12)	10.4(12)	-2.5(12)
C41	33.1(11)	24.3(9)	35.3(10)	-2.9(8)	5.6(8)	-2.4(8)
C42	30.5(10)	33.2(10)	35.8(10)	0.3(8)	7.5(8)	-3.3(8)
C43	27.7(10)	40.3(11)	34.7(10)	-3.7(9)	5.7(8)	1.7(9)
C44	34.3(11)	35.4(10)	34.4(10)	2.0(8)	4.1(8)	5.4(9)
C45	32.3(10)	29.5(9)	30.6(9)	-2.4(8)	2.7(8)	-1.4(8)
C46	37.8(11)	30.8(10)	34.6(10)	2.6(8)	4.8(9)	-0.3(9)
C47	48.8(14)	58.5(15)	35.7(11)	-2.1(10)	2.6(10)	-24.7(12)

C48	41.1(12)	51.5(13)	30.7(10)	-3.3(9)	-0.2(9)	-18.1(11)
C49	35.7(12)	61.9(16)	48.9(13)	-1.2(12)	-2.6(11)	-11.3(11)
C50	52.7(16)	65.2(17)	52.6(14)	15.4(13)	-12.4(12)	-12.2(14)
C51	55.3(16)	74.8(19)	40.0(13)	14.1(13)	1.4(11)	-21.4(15)
C52	39.8(13)	74.2(18)	42.9(13)	5.4(12)	5.7(10)	-10.0(13)
C53	40.7(13)	57.1(15)	37.7(12)	5(1)	-1.5(10)	-8.1(11)
C54	31.3(11)	64.7(16)	36.7(11)	5.1(11)	8.0(9)	7.0(11)
C55	34.9(12)	42.9(12)	44.6(12)	-0.5(9)	-3(1)	4.5(9)
C56	33.9(13)	58.9(16)	66.9(17)	0.7(13)	10.9(12)	-3.3(12)
C57	79(2)	72(2)	81(2)	-15.2(18)	49.6(19)	-9.0(18)
C58	103(3)	114(3)	56.6(18)	-38(2)	28.1(19)	-49(3)
C59	68(2)	110(3)	62.4(18)	-26.8(19)	18.3(16)	-45(2)
C60	117(3)	70(2)	61.7(19)	-23.6(17)	0(2)	-20(2)
C61	24.3(10)	30.7(10)	36.5(10)	1.7(8)	1.1(8)	2.2(8)
C62	28.5(10)	31.4(10)	34.1(10)	4.8(8)	-0.3(8)	1.2(8)
C63	26.8(10)	28.2(10)	35.2(10)	3.5(8)	2.2(8)	1.7(8)
C64	26.2(10)	32.6(10)	35.7(10)	2.9(8)	-1.0(8)	-0.2(8)
C65	24.2(10)	33.6(10)	34.9(10)	3.7(8)	0.5(8)	3.7(8)
C66	26.7(10)	38.0(11)	38.3(11)	6.5(9)	-2.9(8)	3.2(9)
C67	33.6(11)	47.4(13)	39.1(11)	0.9(9)	-0.4(9)	5.9(10)
C68	40.1(12)	35.5(11)	32.8(10)	-1.2(8)	4.5(9)	7.4(9)
C69	53.4(15)	35.5(11)	40.7(12)	-2.9(9)	-5.8(10)	4.1(10)
C70	65.1(18)	63.0(17)	41.9(13)	-9.6(12)	-14.6(13)	11.1(14)
C71	76(2)	76(2)	34.3(13)	12.2(12)	-0.3(13)	21.4(17)
C72	70.0(19)	55.3(16)	54.3(15)	24.7(13)	17.5(14)	9.4(14)
C73	46.1(14)	45.9(13)	48.2(13)	5.6(11)	12.2(11)	1.8(11)
C74	33.6(11)	28.2(10)	35.1(10)	5.7(8)	-4.0(8)	-2.6(8)
C75	40.9(12)	30.6(10)	37.5(11)	1.1(8)	4.3(9)	-7.4(9)
C76	63.5(17)	37.2(12)	43.1(12)	10.9(10)	-1.2(11)	-9.8(11)
C77	61.1(17)	37.4(13)	71.0(17)	18.8(12)	-7.6(14)	1.2(12)
C78	39.7(13)	41.9(13)	82.2(19)	15.3(13)	-7.0(13)	6.0(11)
C79	35.4(12)	34.1(11)	51.6(13)	6.3(9)	3.8(10)	-1.2(9)
C80	52.7(16)	50.8(15)	63.1(16)	4.3(12)	21.2(13)	-0.9(12)
N1	30.0(9)	30.2(8)	41.3(9)	5.7(7)	-2.2(7)	-1.5(7)
N2	28.7(9)	26.7(8)	35.0(9)	1.1(7)	7.4(7)	-0.2(7)
N3	29.3(9)	28.4(8)	34.2(9)	0.0(7)	7.8(7)	-0.3(7)
N4	26.7(8)	29.6(8)	38.2(9)	5.7(7)	-0.7(7)	-0.4(7)
O1	31.8(8)	38.4(8)	49.3(9)	10.8(7)	-6.0(7)	-8.0(7)
O2	80.0(15)	45.4(10)	91.2(15)	34.2(11)	-46.4(13)	-31.4(11)
O3	32.7(8)	30.9(7)	45.6(8)	7.6(6)	8.4(6)	5.6(6)

O4	32.1(8)	31.5(7)	44.7(8)	8.7(6)	13.3(6)	5.5(6)
O5	40.0(9)	31.2(7)	45.4(8)	5.1(6)	12.0(7)	4.6(7)
O6	39.1(9)	70.4(12)	49.1(9)	16.3(9)	18.4(8)	21.1(9)
O7	30.7(8)	34.5(8)	47.1(8)	6.7(6)	-5.7(6)	-5.6(6)
O8	32.2(8)	33.0(7)	43.7(8)	10.5(6)	-5.9(6)	-5.6(6)

Table 4 Bond Lengths for exp_4139.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C1	C2	1.429(3)	C41	C42	1.428(3)
C1	N1	1.379(3)	C41	N3	1.378(3)
C1	O1	1.251(3)	C41	O5	1.253(3)
C2	C3	1.366(3)	C42	C43	1.370(3)
C3	C4	1.422(3)	C43	C44	1.424(3)
C3	O2	1.341(3)	C43	O6	1.353(3)
C4	C5	1.357(3)	C44	C45	1.359(3)
C5	C6	1.496(3)	C45	C46	1.502(3)
C5	N1	1.360(3)	C45	N3	1.356(3)
C6	C7	1.522(4)	C46	C47	1.533(3)
C7	C8	1.509(4)	C47	C48	1.505(3)
C8	C9	1.375(5)	C48	C49	1.383(4)
C8	C13	1.369(4)	C48	C53	1.387(4)
C9	C10	1.395(5)	C49	C50	1.386(4)
C10	C11	1.348(5)	C50	C51	1.375(4)
C11	C12	1.358(5)	C51	C52	1.373(4)
C12	C13	1.381(4)	C52	C53	1.387(3)
C14	C15	1.523(4)	C54	C55	1.506(3)
C14	C19	1.513(3)	C54	C59	1.529(4)
C14	O2	1.448(3)	C54	O6	1.451(3)
C15	C16	1.522(5)	C55	C56	1.520(3)
C16	C17	1.500(5)	C55	C60	1.512(4)
C17	C18	1.507(5)	C56	C57	1.495(4)
C18	C19	1.527(3)	C57	C58	1.502(6)
C19	C20	1.517(4)	C58	C59	1.537(4)
C21	C22	1.429(3)	C61	C62	1.426(3)
C21	N2	1.381(3)	C61	N4	1.376(3)
C21	O3	1.251(3)	C61	O7	1.256(3)
C22	C23	1.375(3)	C62	C63	1.373(3)
C23	C24	1.417(3)	C63	C64	1.419(3)
C23	O4	1.348(2)	C63	O8	1.353(2)

C24	C25	1.354(3)	C64	C65	1.356(3)
C25	C26	1.495(3)	C65	C66	1.497(3)
C25	N2	1.360(3)	C65	N4	1.363(3)
C26	C27	1.535(3)	C66	C67	1.537(3)
C27	C28	1.502(3)	C67	C68	1.507(3)
C28	C29	1.387(4)	C68	C69	1.381(3)
C28	C33	1.384(4)	C68	C73	1.388(3)
C29	C30	1.384(4)	C69	C70	1.385(4)
C30	C31	1.367(5)	C70	C71	1.371(4)
C31	C32	1.371(5)	C71	C72	1.373(5)
C32	C33	1.401(4)	C72	C73	1.388(4)
C34	C35	1.522(3)	C74	C75	1.523(3)
C34	C39	1.517(3)	C74	C79	1.513(3)
C34	O4	1.445(2)	C74	O8	1.445(2)
C35	C36	1.526(3)	C75	C76	1.531(3)
C35	C40	1.517(3)	C75	C80	1.516(4)
C36	C37	1.528(3)	C76	C77	1.519(4)
C37	C38	1.526(3)	C77	C78	1.522(4)
C38	C39	1.527(3)	C78	C79	1.527(3)

Table 5 Bond Angles for exp_4139.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
N1	C1	C2	116.09(19)	C42	C43	C44	120.30(19)
O1	C1	C2	124.6(2)	O6	C43	C42	126.8(2)
O1	C1	N1	119.29(18)	O6	C43	C44	112.89(19)
C3	C2	C1	120.7(2)	C45	C44	C43	119.13(19)
C2	C3	C4	119.9(2)	C44	C45	C46	123.63(19)
O2	C3	C2	126.7(2)	N3	C45	C44	119.81(19)
O2	C3	C4	113.4(2)	N3	C45	C46	116.56(18)
C5	C4	C3	119.5(2)	C45	C46	C47	112.58(18)
C4	C5	C6	123.7(2)	C48	C47	C46	112.07(19)
C4	C5	N1	119.7(2)	C49	C48	C47	121.4(2)
N1	C5	C6	116.53(19)	C49	C48	C53	118.1(2)
C5	C6	C7	111.7(2)	C53	C48	C47	120.5(2)
C8	C7	C6	112.3(3)	C48	C49	C50	121.2(3)
C9	C8	C7	121.7(3)	C51	C50	C49	119.9(3)
C13	C8	C7	120.4(3)	C52	C51	C50	119.6(2)
C13	C8	C9	117.9(3)	C51	C52	C53	120.5(3)
C8	C9	C10	120.8(3)	C52	C53	C48	120.6(3)

C11	C10	C9	120.2(4)	C55	C54	C59	111.4(2)
C10	C11	C12	119.4(3)	O6	C54	C55	108.0(2)
C11	C12	C13	121.0(3)	O6	C54	C59	106.1(2)
C8	C13	C12	120.6(3)	C54	C55	C56	110.3(2)
C19	C14	C15	112.1(2)	C54	C55	C60	110.8(3)
O2	C14	C15	108.8(2)	C60	C55	C56	111.2(2)
O2	C14	C19	106.1(2)	C57	C56	C55	113.0(2)
C16	C15	C14	111.2(2)	C56	C57	C58	109.8(3)
C17	C16	C15	110.5(3)	C57	C58	C59	110.2(3)
C16	C17	C18	110.5(3)	C54	C59	C58	110.1(3)
C17	C18	C19	113.5(2)	N4	C61	C62	116.17(18)
C14	C19	C18	109.4(2)	O7	C61	C62	124.60(19)
C14	C19	C20	112.3(2)	O7	C61	N4	119.22(18)
C20	C19	C18	110.1(2)	C63	C62	C61	120.42(19)
N2	C21	C22	115.80(18)	C62	C63	C64	120.41(18)
O3	C21	C22	124.81(19)	O8	C63	C62	126.22(19)
O3	C21	N2	119.39(18)	O8	C63	C64	113.36(18)
C23	C22	C21	120.40(18)	C65	C64	C63	118.91(19)
C22	C23	C24	120.48(19)	C64	C65	C66	123.4(2)
O4	C23	C22	125.95(18)	C64	C65	N4	120.15(19)
O4	C23	C24	113.53(17)	N4	C65	C66	116.39(18)
C25	C24	C23	118.96(18)	C65	C66	C67	112.23(18)
C24	C25	C26	122.92(19)	C68	C67	C66	110.98(18)
C24	C25	N2	120.25(18)	C69	C68	C67	120.8(2)
N2	C25	C26	116.82(18)	C69	C68	C73	118.3(2)
C25	C26	C27	112.68(17)	C73	C68	C67	121.0(2)
C28	C27	C26	110.12(18)	C68	C69	C70	121.2(2)
C29	C28	C27	120.4(2)	C71	C70	C69	120.0(3)
C33	C28	C27	121.5(2)	C70	C71	C72	119.7(3)
C33	C28	C29	117.9(2)	C71	C72	C73	120.5(3)
C30	C29	C28	121.8(3)	C72	C73	C68	120.3(3)
C31	C30	C29	119.7(3)	C79	C74	C75	111.44(18)
C30	C31	C32	120.0(3)	O8	C74	C75	107.21(17)
C31	C32	C33	120.4(3)	O8	C74	C79	109.62(17)
C28	C33	C32	120.2(3)	C74	C75	C76	108.27(19)
C39	C34	C35	111.42(18)	C80	C75	C74	112.54(19)
O4	C34	C35	106.79(16)	C80	C75	C76	112.2(2)
O4	C34	C39	108.93(16)	C77	C76	C75	112.4(2)
C34	C35	C36	108.66(17)	C76	C77	C78	110.3(2)
C40	C35	C34	112.55(19)	C77	C78	C79	110.7(2)

C40	C35	C36	112.16(19)	C74	C79	C78	110.3(2)
C35	C36	C37	112.47(18)	C5	N1	C1	124.02(18)
C38	C37	C36	110.5(2)	C25	N2	C21	124.09(18)
C37	C38	C39	110.73(19)	C45	N3	C41	124.25(18)
C34	C39	C38	110.86(17)	C65	N4	C61	123.93(18)
N3	C41	C42	116.25(18)	C3	O2	C14	122.7(2)
O5	C41	C42	124.27(19)	C23	O4	C34	120.23(16)
O5	C41	N3	119.48(19)	C43	O6	C54	122.98(19)
C43	C42	C41	120.23(19)	C63	O8	C74	119.54(16)

Table 6 Hydrogen Bonds for exp_4139.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
C4	H4	O1[1]	0.95	2.57	3.501(3)	166.7
C4	H4	O1[1]	0.95	2.57	3.501(3)	166.7
C24	H24	O3[2]	0.95	2.52	3.465(3)	174.6
C24	H24	O3[2]	0.95	2.52	3.465(3)	174.6
C44	H44	O5[3]	0.95	2.45	3.380(3)	165.0
C44	H44	O5[3]	0.95	2.45	3.380(3)	165.0
C64	H64	O7[4]	0.95	2.46	3.412(3)	176.9
C64	H64	O7[4]	0.95	2.46	3.412(3)	176.9
N3	H3	O3[5]	0.85(3)	2.00(3)	2.849(2)	179(3)
N3	H3	O3[5]	0.85(3)	2.00(3)	2.849(2)	179(3)
N2	H2A	O5[6]	0.87(3)	1.96(3)	2.832(2)	174(3)
N2	H2A	O5[6]	0.87(3)	1.96(3)	2.832(2)	174(3)
N4	H4A	O1	0.92(3)	1.91(3)	2.828(2)	175(3)
N4	H4A	O1	0.92(3)	1.91(3)	2.828(2)	175(3)
N1	H1	O7	0.91(3)	1.93(3)	2.840(2)	174(3)
N1	H1	O7	0.91(3)	1.93(3)	2.840(2)	174(3)

[1]-1/2+X,1/2-Y,-Z; [2]1-X,-1/2+Y,1/2-Z; [3]2-X,1/2+Y,1/2-Z; [4]1/2+X,3/2-Y,-Z; [5]1+X,+Y,+Z; [6]-1+X,+Y,+Z

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for exp_4139.

Atom	*x*	*y*	*z*	U(eq)
H2	7342	2143	473	42
H4	4680	1872	-17	56
H6A	4842	4631	-237	51
H6B	4222	3449	-294	51

H7A	6335	4176	-515	88
H7B	5836	2926	-555	88
H9	4275	2631	-855	115
H10	3250	3298	-1205	113
H11	3293	5191	-1308	79
H12	4253	6443	-1049	70
H13	5311	5797	-706	61
H14	6814	634	604	55
H15A	5194	1362	812	87
H15B	4432	311	721	87
H16A	6282	169	1077	89
H16B	4958	29	1148	89
H17A	4847	-1643	912	89
H17B	5861	-1758	1115	89
H18A	6453	-2400	712	64
H18B	7218	-1358	804	64
H19	5409	-1220	439	47
H20A	6883	-752	149	106
H20B	7070	-1991	265	106
H20C	7812	-948	367	106
H22	5261	4749	2082	36
H24	5466	2131	2581	36
H26A	3855	1737	2855	39
H26B	2708	2450	2815	39
H27A	4573	3284	3117	50
H27B	3337	3861	3096	50
H29	4726	1392	3330	55
H30	4077	199	3650	71
H31	2283	481	3825	74
H32	1134	1941	3680	74
H33	1794	3171	3362	59
H34	6657	3947	1906	38
H35	8490	2461	2052	41
H36A	8301	3756	1594	49
H36B	9328	2907	1657	49
H37A	9947	4076	1992	53
H37B	9870	4820	1738	53
H38A	8114	5593	1866	50
H38B	9013	5780	2091	50
H39A	8260	4222	2315	41

H39B	7240	5081	2252	41
H40A	6908	2101	1648	77
H40B	6918	1405	1909	77
H40C	7959	1286	1714	77
H42	9529	5368	2967	40
H44	9339	8041	2482	42
H46A	12126	7754	2258	41
H46B	10984	8471	2213	41
H47A	11462	6346	1969	57
H47B	10310	7051	1926	57
H49	10337	8736	1655	59
H50	11204	9706	1323	68
H51	13097	9327	1217	68
H52	14126	8012	1452	63
H53	13264	7037	1785	54
H54	8187	6104	3139	53
H55	6176	7093	2921	49
H56A	5081	6046	3210	64
H56B	6208	5535	3337	64
H57A	5497	6984	3591	93
H57B	5544	7853	3358	93
H58A	7497	6810	3584	110
H58B	7155	8117	3617	110
H59A	7472	8442	3177	96
H59B	8609	7926	3301	96
H60A	6983	5582	2685	124
H60B	5725	5282	2783	124
H60C	6816	4763	2924	124
H62	7897	7771	-432	38
H64	10453	8116	85	38
H66A	10841	6576	378	41
H66B	10226	5388	326	41
H67A	9197	7172	620	48
H67B	8642	5946	578	48
H69	11034	7280	872	52
H70	12033	6601	1222	68
H71	11597	4833	1389	74
H72	10190	3732	1201	72
H73	9181	4406	851	56
H74	8710	9113	-611	39

H75	10083	11045	-479	44
H76A	9688	11708	-894	58
H76B	8905	10623	-947	58
H77A	7722	12160	-846	68
H77B	8399	12364	-585	68
H78A	7017	10418	-697	66
H78B	6707	11376	-493	66
H79A	8261	10819	-235	48
H79B	7465	9739	-287	48
H80A	11274	9520	-580	83
H80B	11365	10460	-797	83
H80C	10624	9340	-844	83
H3	11870(20)	5930(20)	2439(5)	36(6)
H2A	2880(30)	4250(30)	2603(5)	51(8)
H4A	8410(20)	5420(20)	105(5)	43(7)
H1	6750(20)	4520(30)	-53(5)	50(8)

Experimental

Single crystals of $C_{20}H_{25}NO_2$ [exp_4139] were []. A suitable crystal was selected and [] on a SuperNova, Dual, Cu at zero, Atlas diffractometer. The crystal was kept at 180.01(10) K during data collection. Using Olex2 [1], the structure was solved with the Superflip [2] structure solution program using Charge Flipping and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard and H. Puschmann, OLEX2: a complete structure solution, refinement and analysis program. J. Appl. Cryst. (2009). 42, 339-341.
2. SUPERFLIP, J. Appl. Cryst. (2007) 40, 786-790
3. SHELXL, G.M. Sheldrick, Acta Cryst. (2008). A64, 112-122

Crystal structure determination of [exp_4139]

Crystal Data for $C_{20}H_{25}NO_2$ (M =311.41): orthorhombic, space group $P2_12_12_1$ (no. 19), a = 11.71049(13) Å, b = 11.80555(11) Å, c = 52.7036(5) Å, V = 7286.21(13) Å^3, Z = 16, T = 180.01(10) K, μ(Cu Kα) = 0.570 mm^{-1},*Dcalc* = 1.136 g/mm^3, 69569 reflections measured (7.674 ≤ 2Θ ≤ 147.666), 14519 unique (R_{int} = 0.0339) which were used in all calculations. The final R_1 was 0.0433 (I > 2σ(I)) and wR_2 was 0.1131 (all data).