Supporting Information

Pd(II)-Catalyzed Alkoxylation of Unactivated C(sp^3)–H and C(sp^2)–H Bonds Using a Removable Directing Group: Efficient Synthesis of Alkyl Ethers

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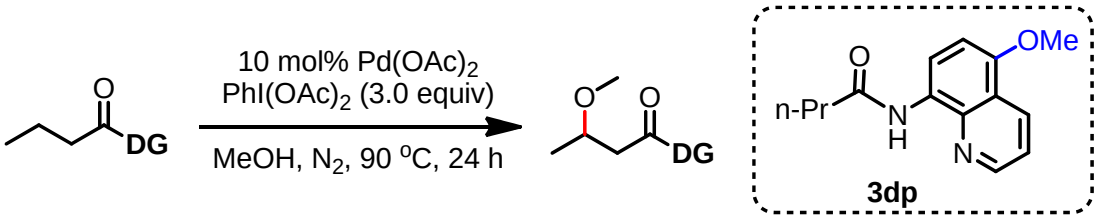
General Information: MeOH were dried by sodium and freshly distilled. *m*-Xylene was used without further purification. $PhI(OAc)_2$ was used after recrystallization from Hex and HOAc. The other materials and solvents were purchased from Aladdin and other commercial suppliers and used without additional purification. NMR spectra were recorded on a Bruke Avance operating for ^1H NMR at 400 MHz, and ^{13}C NMR at 100 MHz using TMS as internal standard. Chemical shifts were given relative to $CDCl_3$ (7.26 ppm for ^1H NMR, 77.16 ppm for ^{13}C NMR). The following abbreviations (or combinations thereof) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, m = multiplet, b = broad. Mass spectroscopy data of the products were collected on an HRMS-TOF instrument or a low-resolution MS instrument using EI ionization.

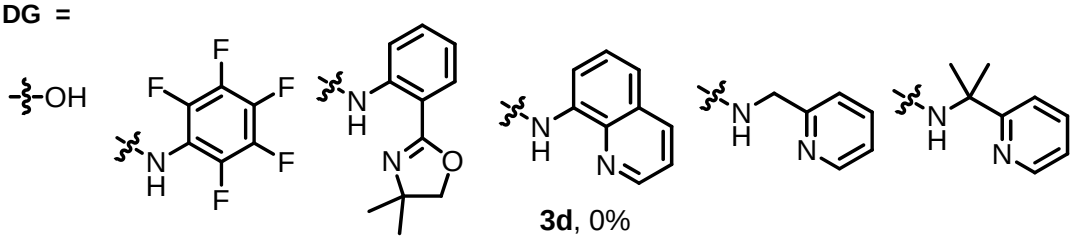
Experimental Procedures

Effect of the Directing Groups:

Table S1. Effect of the Directing Groups



DG =



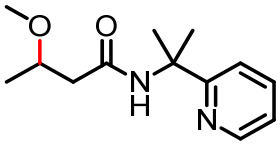
3a, 0% **3b**, 0% **3c**, 0% **3d**, 0% (**3dp**: 22%) **3e**, 0% **1**, 50%

Reaction conditions : Substrate (0.2 mmol), $Pd(OAc)_2$ (10 mol%),$PhI(OAc)_2$ (3.0 equiv) in MeOH (2.0 ml) at 90 °C under N_2 for 24 h. Yields are given for isolated products after chromatography.

A mixture of substrate (0.2 mmol), $Pd(OAc)_2$ (4.5 mg, 10 mmol%) and $PhI(OAc)_2$ (194 mg, 0.6 mmol), MeOH (1.0 mL) and *m*-xylene (1.0 mL) in a 50 mL Schlenk

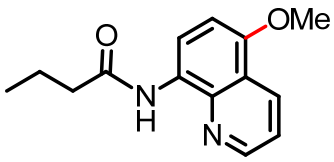
tube (purged with N_2) was heated at 90 ^oC for 24 hours. The reaction mixture was cooled to RT, and concentrated *in vacuo*. The resulting residue was purified by flash chromatography.

3-Methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 2



The title compound **2** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **2** was obtained as a colorless liquid (23.6 mg, 50%). R_f = 0.46 (1/2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (d, *J* = 4.4 Hz, 1H), 7.96 (s, 1H), 7.65 (td, *J* = 8.0, 1.4 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.13 (dd, *J* = 6.9, 5.3 Hz, 1H), 3.80 – 3.68 (m, 1H), 3.35 (s, 3H), 2.44 (dd, *J* = 14.6, 7.7 Hz, 1H), 2.28 (dd, *J* = 14.6, 4.5 Hz, 1H), 1.71 (s, 3H), 1.70 (s, 3H), 1.18 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 164.7, 147.8, 136.9, 121.8, 119.5, 74.4, 56.6, 56.4, 44.9, 27.7, 27.6, 19.2. HRMS (EI-TOF) calc. for $C_{13}H_{20}N_2O_2$ (M^+): 236.1525, found: 236.1531.

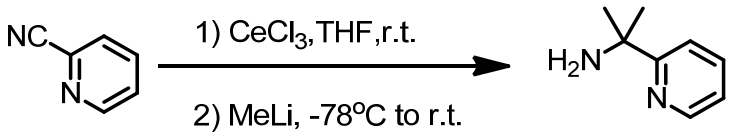
***N*-(5-Methoxyquinolin-8-yl)butyramide 3dp**



The title compound **3dp** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 5 : 1). **3dp** was obtained as a colorless oil (11.4 mg, 22%). R_f = 0.50 (3/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.56 (s, 1H), 8.81 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.71 (d, *J* = 8.5 Hz, 1H), 8.57 (dd, *J* = 8.4, 1.7 Hz, 1H), 7.44 (dd, *J* = 8.4, 4.2 Hz, 1H),

6.84 (d, J = 8.6 Hz, 1H), 3.99 (s, 3H), 2.52 (t, J = 7.6 Hz, 2H), 1.85 (m, 2H), 1.06 (t, J = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 150.3, 148.7, 139.2, 128.2, 120.8, 120.6, 116.7, 104.6, 55.9, 40.3, 19.4, 14.0. HRMS (EI-TOF) calc. for $C_{14}H_{16}N_2O_2$ (M^+): 244.1212, found: 244.1206.

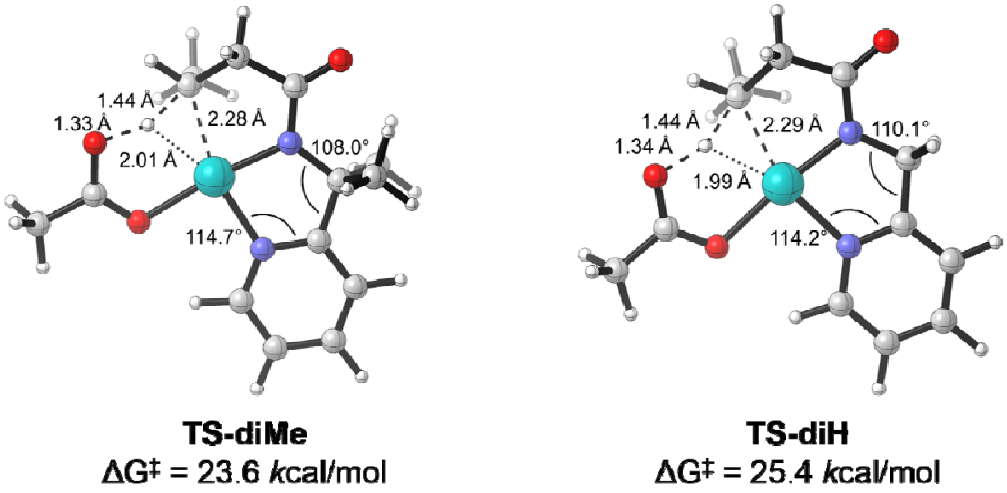
Preparation of 2-(pyridin-2-yl)propan-2-amine (Directing Group): [S1]



THF (400 mL) was added to anhydrous cerium (III) chloride (73.9 g, 300 mmol) and stirred vigorously under nitrogen. After cooling the mixture to -78 °C, 1.6 M methyl lithium in diethyl ether (200 mL, 320 mmol) was added dropwise over 2h. Then 2-cyanopyridine (9.6 mL, 100 mmol) was added slowly. After the addition was completed, the mixture was allowed to warm to room temperature and stirred overnight. The reaction was cooled to -78 °C, quenched with ammonium hydroxide (200 mL) and filtered through a pad of Celite which was washed thoroughly with THF. The solvent was removed under reduce pressure and the residue was suspension in CH_2Cl_2, a filtration give the 2-(pyridin-2-yl)propan-2-amine as yellow solid (11.0 g, 82 %). R_f = 0.51 (10/ 1 dichloromethane/ methanol). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.54 (d, J = 4.2 Hz, 1H), 7.66 (td, J = 7.8, 1.7 Hz, 1H), 7.45 (d, J = 8.0 Hz, 1H), 7.14 (dd, J = 7.3, 4.9 Hz, 1H), 4.21 (bs, 2H), 1.53 (s, 6H).

[S1] Tucker, R., Craig et al. PCT/US2009/039254. 2009

DFT Calculations on the Activation Ability of the designed DGs



TS-diMe
ΔG‡ = 23.6 *kcal*/mol

TS-diH
ΔG‡ = 25.4 *kcal*/mol

Figure S1. Selected geometries of the transition states (TS) for the concerted metallation deprotonation (CMD) C-H activation, **TS-diMe** for **1**, **TS-diH** for **3**.

To shed some light on the design of the directing groups (DGs), we conducted some preliminary density functional theory (DFT) calculations on the activation ability of the designed DGs. All calculations were performed with the Gaussian 09 program.[S2] Geometry optimizations and frequency calculations were performed with B3LYP[S3] functional using LANL2DZ[S4] basis set for Pd combined with 6-31G(d)[S5] basis set for all the other atoms. Single point energy calculations were also performed to

[S2] Gaussian 09, Revision B.01, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. ontgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, T. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2010. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. ontgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, T. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, O. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2010.

[S3] A. D. Becke, *J.Chem. Phys.*, **1993**, *98* **,**5648-5652.

[S4] a) P. J. Hay, W. R. Wadt, *J. Chem. Phys.*, **1985**, *82*, 270-283. b) W. R. Wadt, P. J. Hay, *J. Chem. Phys.*, **1985**, *82*, 284-298. c) P. J. Hay, W. R. Wadt, *J. Chem. Phys.*, **1985**, *82*, 299-310.

[S5] Hehre, W. J.; Ditchfeld, R.; Pople, *J. A. J. Chem. Phys.* **1972**, *56*, 2257.

further refine the results with M06-2x[S6] functional using Stuttgart/Dresden (SDD)[S7] basis set for Pd combined with 6-311++G(d,p)[S8] basis set for all the remaining atoms. Imaginary frequency in frequency calculations were used to confirm whether the geometries were intermediates or transition states. For transition states, the intrinsic reaction coordination (IRC)[S9] calculations were performed to determine the reactants and products.

As shown in Figure **S1**, the Gibbs free energy of the CMD process for the dimethyl-DG is lowered by 1.8 *k*cal/mol compared to the dihydro-DG. The relative Gibbs free energy of intermediates with AcOH as a neutral ligand for diMe-DG and diH-DG is 8.5 *k*cal/mol and 9.6 *k*cal/mol respectively. This is consistent with the gem-dimethyl effect (Thorpe–Ingold effect), which facilitates cyclisation by decreasing the bond angle on the carbon in the DG. The subtle difference in geometry might manipulate the coordination between DG and Pd, and therefore shows better reactivity for C-H activation. Moreover, the dihydro-DG might be labile in the strong oxidation condition, namely $PhI(OAc)_2$, during the reaction, which led to the failure of the alkoxylation.

Table S2. Calculated energy values

Species	E[a,b]	ZPE[a,c]	H_{298}[a,d]	G_{298}[a,e]	E'[a,f]	G'_{298}[e,f]
Substrate-diMe	-1007.521408	-1007.193499	-1007.170618	-1007.247346	-1008.46371	-1008.189648
TS-diMe	-1007.477284	-1007.1543	-1007.132491	-1007.204069	-1008.425307	-1008.152092
Intermediate-diMe	-1007.502944	-1007.175659	-1007.153175	-1007.227037	-1008.452019	-1008.176112
Substrate-diH	-928.896677	-928.625381	-928.605041	-928.677753	-929.8549767	-929.6360527
TS-diH	-928.848897	-928.582508	-928.563275	-928.630569	-929.8139124	-929.5955844
Intermediate-diH	-928.875559	-928.604938	-928.58496	-928.654784	-929.8415874	-929.6208124
AcOH	-229.07761	-229.015594	-229.010062	-229.042981	-229.0719924	-229.0373634

[S6] Y. Zhao and D. G. Truhlar, *Theor. Chem. Acc.*, **2008**, *120*, 215-241.

[S7] a) Szentpaly, L. V.; Fuentealba, P.; Preuss, H.; Stoll, H. *Chem. Phys. Lett.*, **1982**, *93*, 555. b) Dolg, M.; Wedig, U.; Stoll, H.; Preuss, H. *J. Chem. Phys.*, **1987**, *86*, 866. c) Schwerdtfeger, P.; Dolg, M.; Schwarz, W. H. E.;Bowmaker, G. A.; Boyd, P. D. W. *J. Chem. Phys.*, **1989**, *91*, 1762.

[S8] a) A. D. McLean, G. S. Chandler, *J. Chem. Phys.*, **1980**, *72*, 5639-5648. b) K. Raghavachari, J. S. Binkley, R. Seeger, and J. A. Pople, *J. Chem. Phys.*, **1980**, *72*, 650-654.

[S9] a) K. Fukui, *Acc. Chem. Res.*, **1981**, *14*, 363-368. b) H. P. Hratchian, H. B. Schlegel, *J. Chem. Phys.*, **2004**, *120*, 9918-9924. c) H. P. Hratchian, H. B. Schlegel, *J. Chem. Theory and Comput.*, **2005**, *1*, 61-69.

[a] With B3LYP functional; 6-31G(d) for C,H,O,N and LANL2DZ for Pd
[b] Electronic energies
[c] Sum of electronic and zero-point energies
[d] Sum of electronic and thermal enthalpies
[e] Sum of electronic and thermal free energies
[f] With M06-2X functional; 6-31G(d) for C,H,O,N and SDD for Pd; solvation with CPCM in methonal

Cartesian coordinates

Substrate-diMe

Pd	-0.068621	0.863208	-0.004071
O	-1.231385	-3.159722	0.005938
N	-0.122740	-1.155589	0.002002
N	1.901773	0.499089	-0.001989
C	-1.256963	-1.920057	0.002375
C	1.195968	-1.846702	0.004461
C	1.369875	-2.713486	-1.271445
H	0.585580	-3.469678	-1.289762
H	2.346779	-3.210535	-1.282546
H	1.287341	-2.088702	-2.167387
C	1.368406	-2.707400	1.284631
H	1.284859	-2.078420	2.177538
H	2.345299	-3.204386	1.299225
H	0.584103	-3.463514	1.305618
C	2.286332	-0.792665	0.002271
C	3.655684	-1.093051	0.003954
H	3.973622	-2.129188	0.007428
C	4.592723	-0.067764	0.001041
H	5.653743	-0.300058	0.002304
C	4.159523	1.262305	-0.003648
H	4.859722	2.090448	-0.006211
C	2.796961	1.508557	-0.005075
H	2.375735	2.508258	-0.008827
O	-0.178113	2.983724	-0.011673
C	-1.457167	2.930680	-0.008763
O	-2.012171	1.784855	-0.009679
C	-2.280724	4.187369	0.025690
H	-3.197345	4.051699	-0.553918
H	-2.565578	4.401534	1.062704
H	-1.704355	5.031731	-0.358887
C	-2.597202	-1.185819	-0.001934
H	-2.646274	-0.517917	0.866984
H	-2.642867	-0.521952	-0.874117

C	-3.793934	-2.142350	-0.001955
H	-3.731069	-2.799855	0.872787
H	-3.727849	-2.803818	-0.873472
C	-5.132947	-1.397767	-0.006146
H	-5.232280	-0.757465	-0.891868
H	-5.976111	-2.098750	-0.006138
H	-5.235598	-0.753452	0.876285

TS-diMe

Pd	-0.638254	0.016093	-0.015368
C	-1.798549	-1.931395	0.208001
H	-2.458262	-0.767539	-0.321333
O	-3.538999	-0.037848	-0.580618
C	-3.267863	1.161267	-0.263867
O	-2.110839	1.532182	0.101176
C	-0.810903	-2.893999	-0.475117
H	-1.012261	-2.922137	-1.555000
C	0.669949	-2.561349	-0.315411
C	2.261564	-0.672446	-0.095289
C	3.052892	-1.019303	-1.383116
H	3.101035	-2.104934	-1.479196
H	4.072177	-0.618246	-1.344599
H	2.548018	-0.605283	-2.262449
C	3.000724	-1.212876	1.155554
H	2.458453	-0.939256	2.067046
H	4.019273	-0.813667	1.222953
H	3.049119	-2.300686	1.083803
C	2.146109	0.843821	0.012275
C	3.265608	1.683207	0.082931
H	4.259415	1.250701	0.061550
C	3.097989	3.059639	0.179407
H	3.965189	3.711614	0.234249
C	1.806774	3.593234	0.204914
H	1.633475	4.661426	0.279952
C	0.732607	2.719250	0.133327
H	-0.300074	3.049658	0.150475
N	0.895777	-1.229511	-0.166575
O	1.546131	-3.431993	-0.369163
N	0.907946	1.387013	0.039888
H	-0.964401	-3.916583	-0.106371
H	-2.785737	-2.253928	-0.158668
C	-1.848417	-2.069414	1.737012
H	-2.563356	-1.364257	2.176543
H	-2.167186	-3.084135	2.018220

H	-0.873194	-1.890591	2.200240
C	-4.378907	2.183867	-0.323000
H	-4.782502	2.221909	-1.339960
H	-5.193598	1.872271	0.338563
H	-4.019795	3.171143	-0.029137

Intermediate-diMe

Pd	0.672540	-0.115383	-0.192055
C	1.637515	-1.931630	-0.306991
H	2.162932	0.144498	1.536461
O	3.024465	0.642570	1.624055
C	3.257246	1.207126	0.447440
O	2.483590	1.100429	-0.508975
C	0.715007	-2.961450	0.364171
H	0.984301	-3.084152	1.422740
C	-0.765182	-2.561405	0.346216
C	-2.257904	-0.581588	0.211164
C	-2.926592	-0.831243	1.588581
H	-3.015383	-1.908002	1.741279
H	-3.924058	-0.380128	1.640676
H	-2.310852	-0.407201	2.389408
C	-3.148183	-1.137331	-0.931177
H	-2.688200	-0.930752	-1.903691
H	-4.149607	-0.691762	-0.918382
H	-3.237800	-2.217434	-0.803475
C	-2.100746	0.932006	0.016043
C	-3.218485	1.781516	0.012085
H	-4.209402	1.365410	0.153378
C	-3.055039	3.148702	-0.174050
H	-3.919694	3.806648	-0.177692
C	-1.769184	3.663201	-0.358186
H	-1.595183	4.723283	-0.511255
C	-0.705884	2.772288	-0.343454
H	0.318384	3.104488	-0.487357
N	-0.929538	-1.217566	0.173064
O	-1.674240	-3.379890	0.519767
N	-0.865554	1.449705	-0.158751
H	0.805820	-3.957753	-0.090063
H	2.595900	-1.863748	0.224795
C	1.899816	-2.225522	-1.784542
H	2.509902	-1.445412	-2.255476
H	2.437897	-3.181077	-1.899109
H	0.965497	-2.308950	-2.351642
C	4.543020	1.979617	0.381713

H	4.579354	2.715037	1.191487
H	5.385456	1.294244	0.524704
H	4.630601	2.474846	-0.585332

Substrate-diH

Pd	0.180530	0.652480	0.001647
O	-1.517776	-3.178303	0.065223
N	-0.254042	-1.307882	0.019812
N	2.065077	-0.087339	-0.013005
C	-1.462502	-1.942509	0.028612
C	0.895647	-2.205567	0.060216
C	2.177106	-1.432179	0.016824
C	3.444109	-2.025437	0.009891
H	3.522401	-3.107758	0.033536
C	4.578503	-1.222998	-0.027342
H	5.566778	-1.673393	-0.033392
C	4.435071	0.168497	-0.057414
H	5.295764	0.827680	-0.086731
C	3.156183	0.703262	-0.049216
H	2.964310	1.770898	-0.070574
O	0.389344	2.773195	-0.017262
C	-0.882799	2.893745	0.007215
O	-1.589062	1.830966	0.020168
C	-1.532133	4.247938	0.051473
H	-2.474559	4.231947	-0.501729
H	-1.755931	4.504876	1.093733
H	-0.859134	5.005203	-0.356799
C	-2.719332	-1.080706	-0.009755
H	-2.721330	-0.415874	0.863829
H	-2.668868	-0.410305	-0.877094
C	-4.005628	-1.911349	-0.049791
H	-4.028815	-2.581375	0.817352
H	-3.984804	-2.566624	-0.928638
C	-5.263057	-1.036592	-0.075091
H	-5.274937	-0.378451	-0.953258
H	-6.172295	-1.648544	-0.105720
H	-5.322299	-0.396455	0.814377
H	0.866818	-2.919942	-0.776766
H	0.878206	-2.832910	0.965703

TS-diH

Pd	0.370656	-0.062594	0.003448
C	2.157682	1.359434	0.191442
H	2.338182	0.017997	-0.303290

O	3.085745	-1.064482	-0.547301
C	2.392745	-2.085508	-0.249588
O	1.175069	-2.015619	0.100612
C	1.589002	2.608225	-0.513583
H	1.801891	2.554289	-1.590018
C	0.084117	2.819593	-0.371480
C	-2.022292	1.651449	-0.066667
C	-2.527609	0.233254	0.002350
C	-3.888166	-0.088619	0.032546
H	-4.623998	0.708991	0.002030
C	-4.277847	-1.422171	0.099272
H	-5.332067	-1.683067	0.121150
C	-3.300004	-2.420310	0.136972
H	-3.564128	-3.471114	0.189728
C	-1.964940	-2.042195	0.106064
H	-1.148145	-2.755383	0.133372
N	-0.573308	1.661543	-0.121326
O	-0.479929	3.906684	-0.507882
N	-1.597350	-0.749242	0.040264
H	2.097280	3.509092	-0.145241
H	3.190989	1.286190	-0.181831
C	2.262721	1.499062	1.717140
H	2.666036	0.587440	2.172810
H	2.937980	2.327470	1.978072
H	1.292987	1.705867	2.179846
C	3.057051	-3.441608	-0.313233
H	3.449753	-3.606295	-1.321835
H	3.909178	-3.460179	0.374182
H	2.354815	-4.235048	-0.053283
H	-2.451208	2.159721	-0.944123
H	-2.396955	2.210930	0.806425

Intermediate-diH

Pd	0.450052	0.045208	0.189466
C	1.981539	1.433962	0.273339
H	1.763032	-0.752241	-1.517763
O	2.397264	-1.520905	-1.588665
C	2.410023	-2.111709	-0.401586
O	1.717802	-1.724452	0.544233
C	1.485791	2.710670	-0.432122
H	1.805172	2.716241	-1.483489
C	-0.042662	2.835333	-0.444888
C	-2.077852	1.522159	-0.262360
C	-2.531089	0.088900	-0.083850

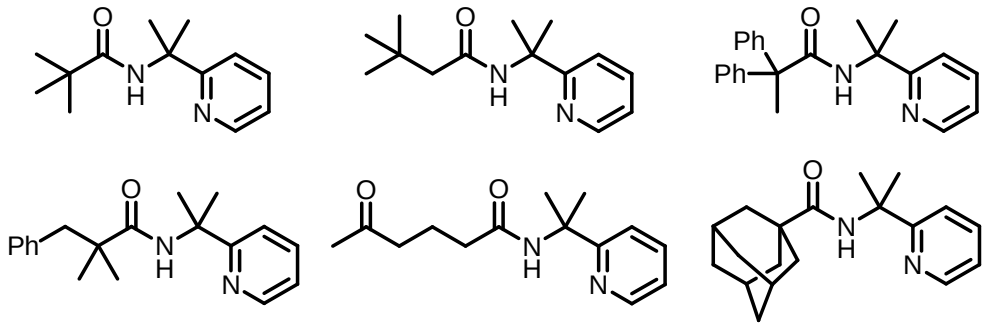
C	-3.890697	-0.246930	-0.132186
H	-4.625573	0.533281	-0.307868
C	-4.281110	-1.568883	0.045936
H	-5.332457	-1.840483	0.010072
C	-3.302813	-2.541199	0.272997
H	-3.562208	-3.584470	0.420818
C	-1.974464	-2.138508	0.308238
H	-1.170157	-2.847113	0.485069
N	-0.637320	1.636121	-0.206388
O	-0.659934	3.873544	-0.684702
N	-1.594536	-0.860710	0.132767
H	1.907354	3.618985	0.019730
H	2.853758	1.017436	-0.247084
C	2.325364	1.653022	1.746610
H	2.626526	0.721891	2.241146
H	3.160831	2.365683	1.845855
H	1.475940	2.068457	2.300847
C	3.342065	-3.285468	-0.311183
H	3.115972	-4.004262	-1.105053
H	4.371801	-2.944350	-0.463052
H	3.250121	-3.758664	0.666427
H	-2.465357	1.907450	-1.219221
H	-2.559763	2.141126	0.512879

AcOH

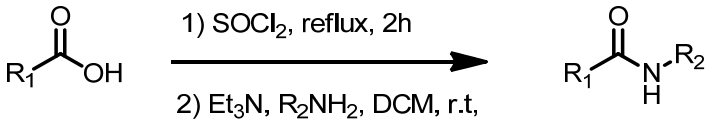
C	-0.092494	0.125638	0.000029
O	-0.645677	1.202013	-0.000040
O	-0.778686	-1.046655	-0.000028
H	-1.723731	-0.802927	-0.000080
C	1.397548	-0.109836	0.000048
H	1.685450	-0.691502	0.882016
H	1.685465	-0.691591	-0.881856
H	1.917402	0.848347	0.000006

Ineffective Substrate

Scheme S1. Ineffective Substrate

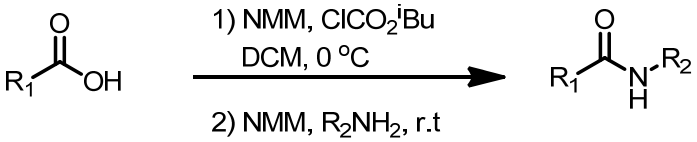


General Procedure for the Preparation of Starting Materials (Method A):



A solution of an acid (5 mmol) was refluxed in 5 mL $SOCl_2$ for 2h and cooled to RT. The excess of $SOCl_2$ was removed under vacuum to give corresponding acid choloride. The acid choloride was then re-dissolved in 5 mL dry CH_2Cl_2 and added dropwise to a 20 mL dry CH_2Cl_2 solution containing amine (5 mmol) and Et_3N (10 mmol) at 0 ^oC. After stirring for 6h at ambient temperature, the resulting mixture was washed with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography to give the desired product.

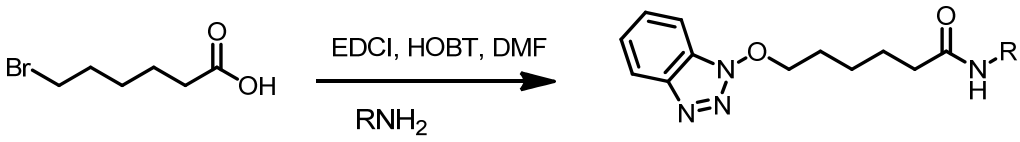
General Procedure for the Preparation of Starting Materials (Method B):



To an round bottom flask was added the acid (5 mmol) and dry CH_2Cl_2 (20 mL). Then the flask was submerged in a brine ice bath, N-methylmorpholine (6 mmol) was

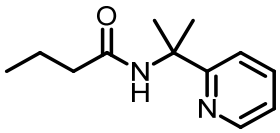
added via syringe and the solution stirred for 15 min. After that, isobutylchloroformate (5.5 mmol) was added dropwise over 20 minutes. After stirring for 6h at ambient temperature, the resulting mixture was washed with sat. Na_2CO_3, and brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography to give the desired product.

General Procedure for the Preparation of Starting Materials (Method C):



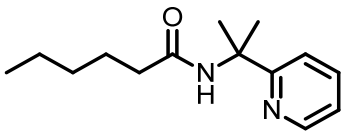
A mixture of amine (5 mmol), 6-bromohexanoic acid (5 mmol), EDCI (5.5 mmol) and HOBT (5.5 mmol) in anhydrous DMF (20 mL) was stirred at room temperature overnight. Water was added and the mixture was extracted with diethyl ether. The combined organic layer was washed with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography to give the desired product.

***N*-(2-(Pyridin-2-yl)propan-2-yl)butyramide 1**



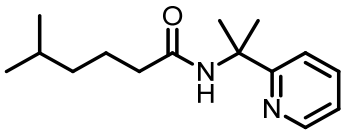
The title compound **1** was prepared according to the general procedure (Method B). R_f = 0.50 (1/2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.4 Hz, 1H), 7.72 – 7.69 (m, 2H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.19 – 7.16 (m, 1H), 2.23 (t, *J* = 7.2 Hz, 2H), 1.75 (s, 6H), 1.72 - 1.66 (m, 2H), 0.96 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 164.8, 147.7, 137.2, 121.9, 119.6, 56.5, 39.9, 27.7, 19.3, 13.9. HRMS (EI-TOF) calc. for $C_{12}H_{18}N_2O$ (M^+): 206.1419, found: 206.1418.

_N_-(2-(Pyridin-2-yl)propan-2-yl)hexanamide 4s



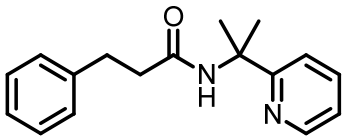
The title compound **4s** was prepared according to the general procedure (Method B). R_f = 0.49 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.8 Hz, 1H), 7.72 – 7.68 (m, 2H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.17 (dd, *J* = 7.2, 5.2 Hz, 1H), 2.23 (t, *J* = 7.2 Hz, 2H), 1.73 (s, 6H), 1.68 - 1.61 (m, 2H), 1.33 – 1.30 (m, 4H), 0.88 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 164.8, 147.7, 137.2, 121.9, 119.6, 56.4, 37.8, 31.5, 27.6, 25.5, 22.5, 14.1. HRMS (EI-TOF) calc. for $C_{14}H_{22}N_2O$ (M^+): 234.1732, found: 234.1729.

5-Methyl-_N_-(2-(pyridin-2-yl)propan-2-yl)hexanamide 5s



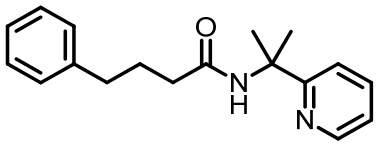
The title compound **5s** was prepared according to the general procedure (Method B). R_f = 0.55 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.4 Hz, 1H), 7.73 – 7.69 (m, 2H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.17 (dd, *J* = 7.6, 4.8 Hz, 1H), 2.23 (t, *J* = 7.6Hz, 2H), 1.75 (s, 6H), 1.66 – 1.55 (m, 3H), 1.25 – 1.20 (m, 2H), 0.88 (d, *J* = 6.4 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 164.8, 147.7, 137.2, 121.9, 119.6, 56.4, 38.6, 38.1, 28.0, 27.6, 23.7, 22.7. HRMS (EI-TOF) calc. for $C_{15}H_{24}N_2O$ (M^+): 248.1889, found: 248.1886.

3-Phenyl-_N_-(2-(pyridin-2-yl)propan-2-yl)propanamide 6s



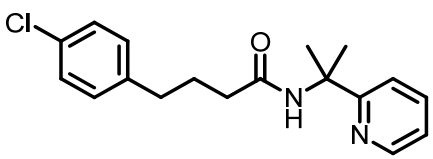
The title compound **6s** was prepared according to the general procedure (Method B). R_f = 0.49 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (d, *J* = 4.8 Hz, 1H), 7.71 – 7.67 (m, 1H), 7.63 (s, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.29 – 7.22 (m, 4H), 7.20 – 7.16 (m, 2H), 2.99 (t, *J* = 7.6 Hz, 2H), 2.56 (t, *J* = 8.4, 2H), 1.72 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 164.6, 147.7, 141.3, 137.2, 128.6, 128.5, 126.2, 121.9, 119.6, 56.6, 39.5, 31.8, 27.6. HRMS (EI-TOF) calc. for $C_{17}H_{20}N_2O$ (M^+): 268.1576, found: 268.1578.

4-Phenyl-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 7s



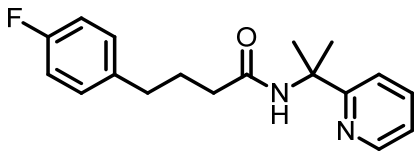
The title compound **7s** was prepared according to the general procedure (Method B). R_f = 0.65 (1/2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.4 Hz, 1H), 7.73 – 7.69 (m, 2H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.29 – 7.26 (m, 2H), 7.21 – 7.17 (m, 4H), 2.67 (t, *J* = 7.6 Hz, 2H), 2.27 (t, *J* = 6.4 Hz, 2H), 2.03 – 1.96 (m, 2H), 1.75 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.1, 164.7, 147.7, 141.9, 137.2, 128.7, 128.4, 125.9, 122.0, 119.6, 56.5, 37.0, 35.3, 27.7, 27.4. HRMS (EI-TOF) calc. for $C_{18}H_{22}N_2O$ (M^+): 282.1732, found: 282.1732.

4-(4-Chlorophenyl)-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 8s



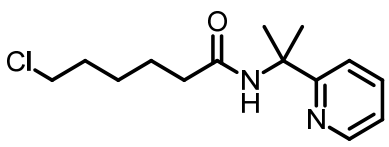
The title compound **8s** was prepared according to the general procedure (Method B). R_f = 0.44 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.4 Hz, 1H), 7.72 (t, *J* = 6.0 Hz, 2H), 7.40 (d, *J* = 8.4 Hz, 1H), 7.25 – 7.18 (m, 3H), 7.13 (d, *J* = 8.0 Hz, 2H), 2.64 (t, *J* = 7.2 Hz, 2H), 2.26 (t, *J* = 7.6 Hz, 2H), 2.01 – 1.95 (m, 2H), 1.75 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.8, 164.6, 127.7, 140.4, 137.3, 130.0, 128.5, 122.0, 119.6, 56.5, 36.8, 34.6, 27.6, 27.2. HRMS (EI-TOF) calc. for $C_{18}H_{21}ClN_2O$ (M^+): 316.1342, found: 316.1344.

4-(4-Fluorophenyl)-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 9s



The title compound **9s** was prepared according to the general procedure (Method B). R_f = 0.51 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 4.4 Hz, 1H), 7.72 – 7.69 (m, 2H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.20 – 7.13 (m, 3H), 6.95 (t, *J* = 8.8 Hz, 2H), 2.64 (t, *J* = 7.6 Hz, 2H), 2.25 (t, *J* = 7.6 Hz, 2H), 2.00 – 1.93 (m, 2H), 1.75 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 164.7, 161.4 (d, J_{C-F} = 242.0 Hz), 147.7, 137.5 (d, J_{C-F} = 3.0 Hz), 137.2, 130.0 (d, J_{C-F} = 8.0 Hz), 122.0, 119.6, 115.1 (d, J_{C-F} = 21.0 Hz), 56.5, 36.9, 34.5, 27.6, 27.5. HRMS (EI-TOF) calc. for $C_{18}H_{21}FN_2O$ (M^+): 300.1638, found: 300.1638.

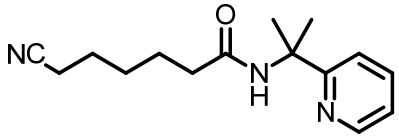
6-Chloro-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 10s



The title compound **10s** was prepared according to the general procedure (Method B). R_f = 0.54 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d,

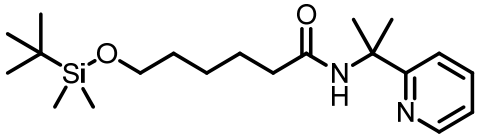
J = 4.4 Hz, 1H), 7.71 – 7.66 (m, 2H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.16 (dd, *J* = 5.2, 7.2 Hz, 1H), 3.51 (t, *J* = 6.8 Hz, 2H), 2.25 (t, *J* = 7.2 Hz, 2H), 1.82 – 1.64 (m, 10H), 1.51 – 1.43 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 164.7, 147.7, 137.2, 121.9, 119.6, 77.5, 77.2, 76.8, 56.5, 45.0, 37.5, 32.5, 27.6, 26.5, 25.0. HRMS (EI-TOF) calc. for $C_{14}H_{21}ClN_2O$ (M^+): 268.1342, found: 268.1348.

6-Cyano-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 11s



The title compound **11s** was prepared according to the general procedure (Method B). R_f = 0.65 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, *J* = 4.4 Hz, 1H), 7.74 – 7.69 (m, 2H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.18 (dd, *J* = 7.2, 5.6 Hz, 1H), 2.33 (t, *J* = 7.2 Hz, 2H), 2.26 (t, *J* = 7.2 Hz, 2H), 1.73 (s, 6H), 1.69 – 1.64 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.8, 164.5, 147.6, 137.3, 122.0, 119.8, 119.6, 77.5, 77.2, 76.8, 56.5, 37.2, 28.3, 27.6, 25.3, 24. 8, 17.1. HRMS (EI-TOF) calc. for $C_{15}H_{21}N_3O$ (M^+): 259.1685, found: 259.1682.

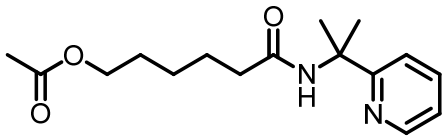
6-((*tert*-Butyldimethylsilyl)oxy)-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 12s



The title compound **12s** was prepared according to the general procedure (Method B). R_f = 0.58 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.8 Hz, 1H), 7.73 – 7.69 (m, 2H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.20 – 7.17 (m, 1H), 3.60 (t, *J* = 6.8 Hz, 2H), 2.26 (t, *J* = 7.6, 2H), 1.75 (s, 6H), 1.70 – 1.66 (m, 2H), 1.57 – 1.53 (m, 2H), 1.40 – 1.36 (m, 2H), 0.88 (s, 9H), 0.03 (s, 6H). ^{13}C NMR (100 MHz,

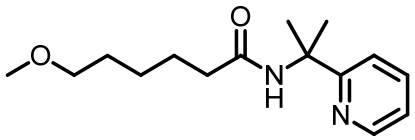
$CDCl_3$) δ 172.4, 164.8, 147.7, 137.2, 122.0, 119.6, 77.5, 77.2, 76.8, 63.3, 56.5, 38.0, 32.8, 27.7, 26.1, 25.8, 26.0, 18.5, 0.1, -5.1. HRMS (EI-TOF) calc. for $C_{20}H_{36}N_2O_2Si$ (M^+): 364.2546, found: 364.2547.

6-Oxo-6-((2-(pyridin-2-yl)propan-2-yl)amino)hexyl acetate 13s



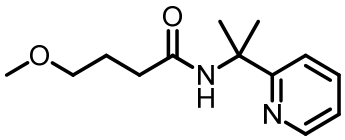
The title compound **13s** was prepared according to the general procedure (Method B). R_f = 0.65 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.8 Hz, 1H), 7.73 – 7.69 (m, 2H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.20 – 7.17 (m, 1H), 4.05 (t, *J* = 6.4 Hz, 2H), 2.26 (t, *J* = 7.2 Hz, 2H), 2.02 (s, 3H), 1.74 (s, 6H), 1.71 – 1.62 (m, 4H), 1.44 – 1.37 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.1, 171.2, 164.5, 147.3, 137.6, 122.0, 119.8, 77.5, 77.2, 76.8, 64.4, 56.3, 37.5, 28.5, 27. 6, 25.6, 25.3, 21.0. HRMS (EI-TOF) calc. for $C_{16}H_{24}N_2O_3$ (M^+): 292.1787, found: 292.1790.

6-Methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 14s



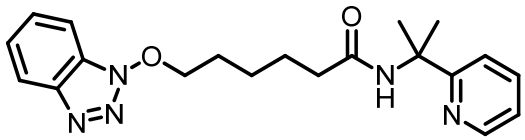
The title compound **14s** was prepared according to the general procedure (Method B). R_f = 0.45 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.4 Hz, 1H), 7.74 (t, *J* = 7.6 Hz, 1H), 7.68 (s, 1H), 7.42 (d, *J* = 8.0 Hz, 1H), 7.29 – 7.20 (m, 1H), 3.37 (t, *J* = 6.4 Hz, 2H), 3.31 (s, 3H), 2.26 (t, *J* = 7.2 Hz, 2H), 1.75 (s, 6H), 1.72 – 1.56 (m, 4H), 1.43 – 1.37 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 164.6, 147.3, 137.7, 122.1, 119.9, 77.5, 77.2, 76.8, 72.8, 58.7, 56.4, 37.7, 29.5, 27.7, 25.9, 25.7. HRMS (EI-TOF) calc. for $C_{15}H_{24}N_2O_2$ (M^+): 264.1838, found: 264.1837.

4-Methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 15s



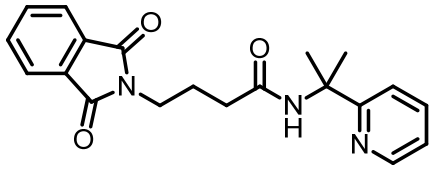
The title compound **15s** was prepared according to the general procedure (Method A). R_f = 0.50 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 5.2 Hz, 1H), 7.72 -7.68 (m, 2H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.19 – 7.16 (m, 1H), 3.43 (t, *J* = 6.0 Hz, 2H), 3.33 (s, 3H), 2.34 (t, *J* = 7.2 Hz, 2H), 1.96 – 1.89 (m, 2H), 1.74 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 164.7, 147.7, 137.1, 121.9, 119.6, 77.5, 77.2, 76.8, 72.0, 58.7, 56.5, 34.3, 27.7, 25.7. HRMS (EI-TOF) calc. for $C_{13}H_{20}N_2O_2$ (M^+): 236.1525, found: 236.1526.

6-((1H-Benzo[d][1,2,3]triazol-1-yl)oxy)-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 16s



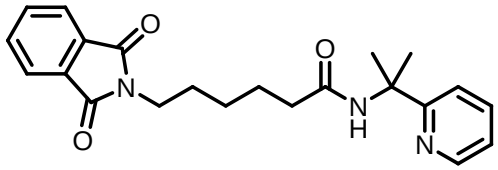
The title compound **16s** was prepared according to the general procedure (Method C). R_f = 0.44 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.0 Hz, 1H), 8.00 (d, *J* = 8.4 Hz, 1H), 7.83 (s, 1H), 7.72 – 7.68 (m, 1H), 7.59 (d, *J* = 8.4 Hz, 1H), 7.50 (t, *J* = 7.2 Hz, 1H), 7.42 – 7.35 (m, 2H), 7.18 (dd, *J* = 6.4, 5.3 Hz, 1H), 4.55 (t, *J* = 6.4 Hz, 2H), 2.34 (t, *J* = 7.2 Hz, 2H), 1.94 – 1.87 (m, 2H), 1.82 – 1.76 (m, 8H), 1.67 – 1.61 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 164.4, 147.5, 143.3, 137.0, 127.9, 127.3, 124.5, 121.7, 120.0, 119.4, 108.6, 80.7, 77.5, 77.2, 76.8, 56.3, 37.1, 27.8, 27.5, 25.1. HRMS (EI-TOF) calc. for $C_{20}H_{25}N_5O_2$ (M^+): 367.2008, found: 367.2009.

4-(1,3-Dioxoisoindolin-2-yl)-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 17s



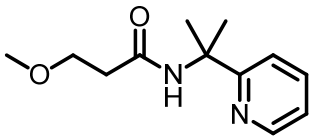
The title compound **17s** was prepared according to the general procedure (Method A). R_f = 0.65 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 4.4 Hz, 1H), 7.84 – 7.82 (m, 2H), 7.71 – 7.67 (m, 4H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.16 (dd, *J* = 6.4, 5.2 Hz, 1H), 3.78 (t, *J* = 6.8 Hz, 2H), 2.31 (t, *J* = 7.6 Hz, 2H), 2.08 – 2.03 (m, 2H), 1.71 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 164.6, 147.7, 137.7, 137.0, 128.5, 128.0, 127.9, 121.9, 119.3, 77.5, 77.3, 77.2, 76.8, 72.2, 56.1, 27.7, 27.4, 18.9. HRMS (EI-TOF) calc. for $C_{20}H_{21}N_3O_3$ (M^+): 351.1583, found: 351.1587.

6-(1,3-Dioxoisoindolin-2-yl)-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 18s



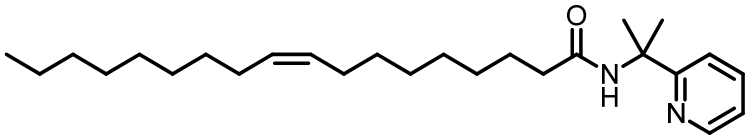
The title compound **18s** was prepared according to the general procedure (Method A). R_f = 0.58 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 4.8 Hz, 1H), 7.82 – 7.80 (m, 2H), 7.71 – 7.68 (m, 4H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.19 – 7.16 (m, 1H), 3.68 (t, *J* = 7.2 Hz, 2H), 2.25 (t, *J* = 7.2 Hz, 2H), 1.74 – 1.66 (m, 10H), 1.43 – 1.37 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.0, 168.5, 164.7, 147.7, 137.1, 133.9, 132.2, 123.2, 121.9, 119.5, 56.4, 37.9, 37.5, 28.5, 27.6, 26.5, 25.3. HRMS (EI-TOF) calc. for $C_{22}H_{25}N_3O_3$ (M^+): 379.1896, found: 379.1895.

3-Methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)propanamide 19s



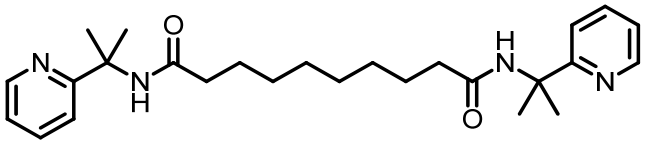
The title compound **19s** was prepared according to the general procedure (Method B). R_f = 0.35 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.0 Hz, 1H), 7.97 (s, 1H), 7.69 (t, *J* = 3.6 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.19 – 7.16 (m, 1H), 3.69 (t, *J* = 5.6 Hz, 2H), 3.40 (s, 3H), 2.51 (t, *J* = 6.0 Hz, 2H), 1.75 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 164.7, 127.8, 137.1, 121.9, 119.5, 69.0, 58.9, 56.8, 38.2, 27.7. HRMS (EI-TOF) calc. for $C_{12}H_{18}N_2O_2$ (M^+): 222.1368, found: 222.1371.

***N*-(2-(Pyridin-2-yl)propan-2-yl)oleamide 20s**



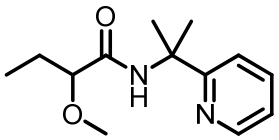
The title compound **20s** was prepared according to the general procedure (Method A). R_f = 0.65 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.8 Hz, 1H), 7.73 – 7.69 (m, 2H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.18 (dd, *J* = 7.2, 6.0 Hz, 1H), 5.35 - 5.33 (m, 2H), 2.24 (t, *J* = 7.6 Hz, 2H), 2.04 – 1.99 (m, 4H), 1.74 (s, 6H), 1.67 – 1.63 (m, 2H), 1.31 – 1.26 (m, 22H), 0.87 (t, *J* = 6.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 164.8, 147.7, 137.2, 130.1, 123.0, 121.9, 119.6, 77.5, 77.2, 76.8, 56.4, 37.9, 32.0, 29.9, 29.8, 29.8, 29.6, 29.4, 29.4, 29.4, 27.7, 27.3, 25.9, 22.8. HRMS (EI-TOF) calc. for $C_{26}H_{44}N_2O$ (M^+): 400.3454, found: 400.3452.

***N*^{1},*N*^{10}-Bis(2-(pyridin-2-yl)propan-2-yl)decanediamide 21s**



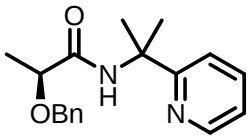
The title compound **21s** was prepared according to the general procedure (Method A). R_f = 0.70 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.8 Hz, 2H), 7.73 – 7,67 (m, 4H), 7.39 (d, *J* = 8.4 Hz, 2H), 7.18 (dd, *J* = 7.2, 5.2 Hz, 2H), 2.24 (t, *J* = 7.2 Hz, 4H), 1.74 (s, 12H), 1.66 – 1.63 (m, 4H), 1.32 (s, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 164.8, 147.7, 137.2, 121.9, 119.6, 56.5, 38.0, 29.4, 29.3, 27.7, 25.9. HRMS (EI-TOF) calc. for $C_{26}H_{38}N_4O_2$ (M^+): 438.2995, found: 438.2994.

2-Methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 22s



The title compound **22s** was prepared according to the general procedure (Method B). R_f = 0.61 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, *J* = 4.4 Hz, 1H), 8.44 (s, 1H), 7.67 (dt, *J* = 8.0, 1.6 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.15 (dd, *J* = 7.2, 5.6 Hz, 1H), 3.54 (dd, *J* = 6.0, 4.8 Hz, 1H), 3.43 (s, 3H), 1.85 – 1.70 (m, 8H), 0.93 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 164.6, 148.0, 136.9, 121.8, 119.4, 84.0, 58.1, 56.2, 27.6, 27.6, 25.5, 9.0. HRMS (EI-TOF) calc. for $C_{13}H_{20}N_2O_2$ (M^+): 236.1525, found: 236.1526.

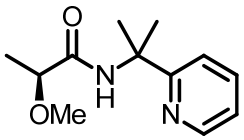
(R)-2-(Benzyloxy)-*N*-(2-(pyridin-2-yl)propan-2-yl)propanamide 23s



The title compound **23s** was prepared according to the general procedure (Method B). R_f = 0.66 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.94 (s, 1H), 8.49 (d, *J* = 4.8 Hz, 1H), 7.70 -7.66 (m, 1H), 7.48 (d, *J* = 7.6 Hz, 2H), 7.37 –

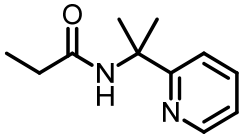
7.27 (m, 3H), 7.30 (t, *J* = 7.2 Hz, 1H), 7.16 (dd, *J* = 7.2, 5.2 Hz, 1H), 4.66 (AB, 1H), 4.59 (AB, 1H), 3.96 (q, *J* = 6.8 Hz, 1H), 1.78 (d, *J* = 2.8 Hz, 6H), 1.47 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 164.6, 147.8, 137.7, 137.0, 128.5, 128.0, 127.9, 121.9, 119.3, 77.5, 77.3, 77.2, 76.8, 72.2, 56.1, 27.7, 27.4, 18.9. HRMS (EI-TOF) calc. for $C_{18}H_{22}N_2O_2$ (M^+): 298.1681, found: 298.1689.

(R)-2-Methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)propanamide 24s



The title compound **24s** was prepared according to the general procedure (Method B). R_f = 0.55 (1/2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 – 8.53 (m, 1H), 8.43 (s, 1H), 7.69 (dt, *J* = 8.0, 2.0 Hz, 1H), 7.38 (td, *J* = 8.0, 0.8 Hz, 1H), 7.19 – 7.15 (m, 1H), 3.71 (q, *J* = 6.8 Hz, 1H), 3.45 (s, 3H), 1.76 (d, *J* = 1.6 Hz, 6H), 1.39 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 164.7, 148.1, 137.0, 121.9, 119.4, 79.2, 77.5, 77.2, 76.8, 57.7, 56.2, 27.7, 27.6, 18.4. HRMS (EI-TOF) calc. for $C_{12}H_{18}N_2O_2$ (M^+): 222.1368, found: 222.1371.

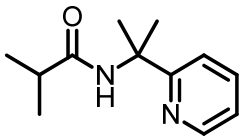
***N*-(2-(Pyridin-2-yl)propan-2-yl)propionamide 25s**



The title compound **25s** was prepared according to the general procedure (Method B). R_f = 0.50 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 4.4 Hz, 1H), 7.71 – 7.67 (m, 2H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.17 (dd, *J* = 6.8, 4.8 Hz, 1H), 2.28 (q, *J* = 7.6 Hz, 2H), 1.73 (s, 6H), 1.16 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ

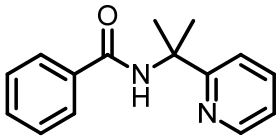
173.1, 164.8, 147.7, 137.2, 121.9, 119.6, 56.4, 30.9, 27.7, 10.0. HRMS (EI-TOF) calc. for $C_{11}H_{16}N_2O$ (M^+): 192.1263, found: 192.1258.

_N_-(2-(Pyridin-2-yl)propan-2-yl)isobutyramide 27s



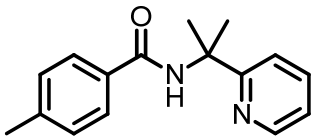
The title compound **27s** was prepared according to the general procedure (Method B). R_f = 0.47 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.8 Hz, 1H), 7.78 (s, 1H), 7.71 (t, *J* = 8.0 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.19 (t, *J* = 6.0 Hz, 1H), 2.48 – 2.42 (m, 1H), 1.75 (s, 6H), 1.19 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.4, 164.9, 147.7, 137.2, 121.9, 119.6, 56.2, 36.6, 27.6, 19.9. HRMS (EI-TOF) calc. for $C_{12}H_{18}N_2O$ (M^+): 206.1419, found: 206.1419.

_N_-(2-(Pyridin-2-yl)propan-2-yl)benzamide 28s



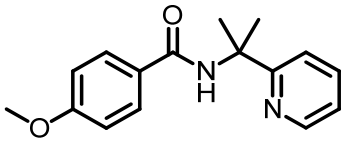
The title compound **28s** was prepared according to the general procedure (Method A). R_f = 0.75 (1/2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.88 (s, 1H), 8.54 (d, *J* = 4.0 Hz, 1H), 7.90 (dd, *J* = 7.6, 0.8 Hz, 2H), 7.72 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.47 – 7.41 (m, 4H), 7.19 (dd, *J* = 7.2, 5.6 Hz, 1H), 1.86 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.3, 164.8, 147.7, 137.4, 136.1, 131.2, 128. 6, 127.1, 122.1, 119.7, 77.5, 77.2, 76.8, 56.7, 27.6. HRMS (EI-TOF) calc. for $C_{15}H_{16}N_2O$ (M^+): 240.1263, found: 240.1259.

4-Methyl-_N_-(2-(pyridin-2-yl)propan-2-yl)benzamide 29s



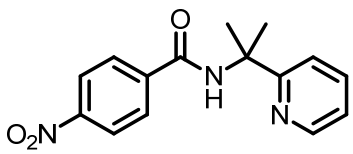
The title compound **29s** was prepared according to the general procedure (Method A). R_f = 0.79 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.78 (s, 1H), 8.56 (d, *J* = 4.4 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 2H), 7.75 (dt, *J* = 8.0, 1.6 Hz, 1H), 7.47 (d, *J* = 8.0 Hz, 1H), 7.26 – 7.21 (m, 3H), 2.40 (s, 3H), 1.86 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 164.9, 147.7, 141.5, 137.4, 133.3, 127.1, 122.0, 119.7, 56.7, 27.7, 21.6. HRMS (EI-TOF) calc. for $C_{16}H_{18}N_2O$ (M^+): 254.1419, found: 254.1419.

4-Methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)benzamide 30s



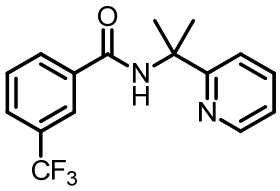
The title compound **30s** was prepared according to the general procedure (Method A). R_f = 0.40 (2/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.75 (s, 1H), 8.56 (d, *J* = 4.8 Hz, 1H), 7.87 (d, *J* = 8.8 Hz, 2H), 7.74 (dt, *J* = 8.0, 1.6 Hz, 1H), 7.46 (d, *J* = 8.4 Hz, 1H), 7.21 (dd, *J* = 7.2, 5.6 Hz, 1H), 6.94 (d, *J* = 8.8 Hz, 2H), 3.85 (s, 3H), 1.86 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9, 164.9, 162.0, 147.7, 137.3, 128.8, 128.5, 122.0, 119.7, 113.7, 55.6, 55.5, 27.7. HRMS (EI-TOF) calc. for $C_{16}H_{18}N_2O_2$ (M^+): 270.1368, found: 270.1363.

4-Nitro-*N*-(2-(pyridin-2-yl)propan-2-yl)benzamide 31s



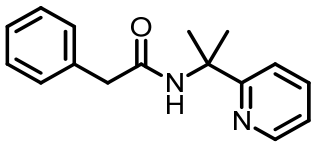
The title compound **31s** was prepared according to the general procedure (Method A). R_f = 0.71 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.22 (s, 1H), 8.56 (d, *J* = 4.4 Hz, 1H), 8.30 (d, *J* = 8.8 Hz, 2H), 8.06 (d, *J* = 8.8 Hz, 2H), 7.79 (dt, *J* = 9.2, 8.0 Hz, 1H), 7.48 (d, *J* = 8.0 Hz, 1H), 7.28 – 7.25 (m, 1H), 1.88 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 164.1, 149.5, 147.6, 141.8, 137.7, 128.3, 123.8, 122.4, 119.7, 77.5, 77.2, 76.8, 57.0, 27.4. HRMS (EI-TOF) calc. for $C_{15}H_{15}N_3O_3$ (M^+): 285.1113, found: 285.1111.

_N_-(2-(Pyridin-2-yl)propan-2-yl)-3-(trifluoromethyl)benzamide 32s



The title compound **32s** was prepared according to the general procedure (Method A). R_f = 0.57 (3/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.04 (s, 1H), 8.57 (d, *J* = 4.4 Hz, 1H), 8.18 (s, 1H), 8.07 (d, *J* = 7.6 Hz, 1H), 7.80 – 7.73 (m, 2H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.48 (d, *J* = 8.0, 1H), 7.27 – 7.24 (m, 1H), 1.88 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.9, 164.4, 147.6, 137.6, 136.9, 131.1 (q, $J_{C\text{-}F}$ = 32.0 Hz), 130.3, 129.1, 127.8 (q, $J_{C\text{-}F}$ = 3.0 Hz), 124.4 (q, $J_{C\text{-}F}$ = 4.0 Hz), 124.0 (q, $J_{C\text{-}F}$ = 271.0 Hz), 122.3, 119.7, 56.9, 27.5. HRMS (EI-TOF) calc. for $C_{16}H_{15}F_3N_2O$ (M^+): 308.1136, found: 308.1134.

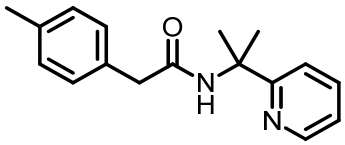
2-Phenyl-_N_-(2-(pyridin-2-yl)propan-2-yl)acetamide 33s



The title compound **33s** was prepared according to the general procedure (Method A). R_f = 0.48 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (d,

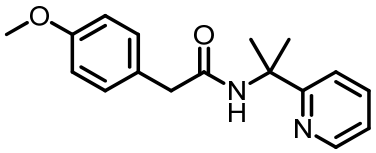
J = 4.8 Hz, 1H), 7.81 (s, 1H), 7.67 (t, *J* = 8.0 Hz, 3H), 7.38 – 7.28 (m, 6H), 7.14 (dd, *J* = 7.2, 5.6 Hz, 1H), 3.61 (s, 2H), 1.72 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 164.6, 147.6, 137.1, 135.7, 129.6, 128.9, 127.0, 121.9, 119.5, 77.5, 77.2, 76.8, 56.6, 45.1, 27.6. HRMS (EI-TOF) calc. for $C_{16}H_{18}N_2O$ (M^+): 254.1419, found: 254.1418.

_N_-(2-(Pyridin-2-yl)propan-2-yl)-2-(p-tolyl)acetamide 34s



The title compound **34s** was prepared according to the general procedure (Method A). R_f = 0.49 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (d, *J* = 4.4 Hz, 1H), 7.72 (s, 1H), 7.67 (t, *J* = 7.6 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.23 – 7.21 (m, 1H), 7.17 – 7.13 (m, 3H), 3.56 (s, 2H), 2.35 (s, 3H), 1.71 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 164.9, 147.7, 141.5, 137.4, 133.3, 129.2, 127.1, 122.0, 119.7, 77.5, 77.2, 76.8, 56.7, 27.7, 21.6. HRMS (EI-TOF) calc. for $C_{17}H_{20}N_2O$ (M^+): 268.1576, found: 268.1573.

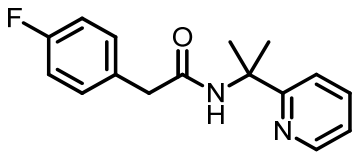
2-(4-Methoxyphenyl)-_N_-(2-(pyridin-2-yl)propan-2-yl)acetamide 35s



The title compound **35s** was prepared according to the general procedure (Method A). R_f = 0.51 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, *J* = 4.8 Hz, 1H), 7.74 (s, 1H), 7.67 (dt, *J* = 9.2, 8.0 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.25 (d, *J* = 8.8 Hz, 1H), 7.16 – 7.13 (m, 1H), 6.90 (d, *J* = 8.4 Hz, 1H), 3.82 (s, 3H), 3.55 (s, 2H), 1.71 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6, 164.6, 158.7, 147.6,

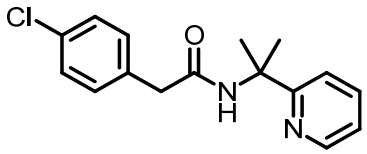
137.1, 130.6, 127.8, 119.4, 114.3, 56.6, 55.4, 44.2, 27.6. HRMS (EI-TOF) calc. for $C_{17}H_{20}N_2O_2$ (M^+): 284.1525, found: 284.1527.

2-(4-Fluorophenyl)-*N*-(2-(pyridin-2-yl)propan-2-yl)acetamide 36s



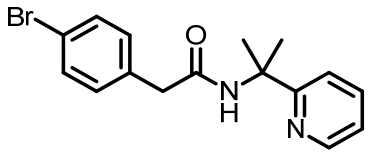
The title compound **36s** was prepared according to the general procedure (Method A). R_f = 0.39 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, *J* = 4.4 Hz, 1H), 7.90 (s, 1H), 7.69 (t, *J* = 8.0 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.31 – 7.28 (m, 2H), 7.18 – 7.15 (m, 1H), 7.04 (t, *J* = 8.4 Hz, 2H), 3.58 (s, 2H), 1.72 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 164.5 (d, J_{C-F} = 3.0 Hz), 162.1 (d, J_{C-F} = 242.0 Hz), 147.6, 137.2 (d, J_{C-F} = 3.0 Hz), 131.5, 131.1 (d, J_{C-F} = 8.0 Hz), 122.0 (d, J_{C-F} = 3.0 Hz), 119.5, 115.6 (d, J_{C-F} = 21.0 Hz), 56.6, 44.2, 27.5. HRMS (EI-TOF) calc. for $C_{16}H_{17}FN_2O$ (M^+): 272.1325, found: 272.1326.

2-Phenyl-*N*-(2-(pyridin-2-yl)propan-2-yl)acetamide 37s



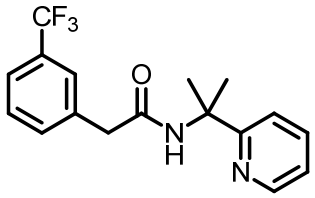
The title compound **37s** was prepared according to the general procedure (Method A). R_f = 0.51 (1/2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.41 (d, *J* = 4.8 Hz, 1H), 7.94 (s, 1H), 7.69 (dt, *J* = 8.0, 1.2 Hz, 1H), 7.36 – 7.31 (m, 3H), 7.28 – 7.26 (m, 2H), 7.17 (dd, *J* = 6.8, 5.2 Hz, 1H), 3.57 (s, 2H), 1.72 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.6, 164.4, 147.6, 137.3, 134.2, 132.9, 130.9, 128.9, 122.0, 119.5, 56.6, 44.31, 27.5. HRMS (EI-TOF) calc. for $C_{16}H_{17}ClN_2O$ (M^+): 288.1029, found: 288.1026.

2-(4-Bromophenyl)-*N*-(2-(pyridin-2-yl)propan-2-yl)acetamide 38s



The title compound **38s** was prepared according to the general procedure (Method A). R_f = 0.38 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.41 (d, *J* = 4.4 Hz, 1H), 7.93 (s, 1H), 7.69 (t, *J* = 7.6 Hz, 1H), 7.47 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.22 – 7.16 (m, 3H), 3.56 (s, 2H), 1.72 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.5, 164.4, 147.6, 137.3, 134.7, 131.9, 131.3, 122.0, 121.0, 119.5, 56.6, 44.4, 27.5. HRMS (EI-TOF) calc. for $C_{16}H_{17}BrN_2O$ (M^+): 332.0524, found: 332.0525.

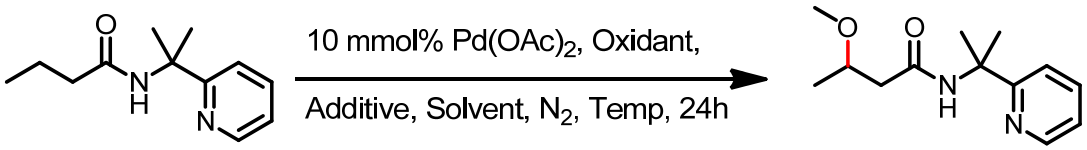
***N*-(2-(Pyridin-2-yl)propan-2-yl)-2-(3-(trifluoromethyl)phenyl)acetamide 39s**



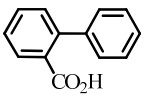
The title compound **39s** was prepared according to the general procedure (Method A). R_f = 0.59 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (d, *J* = 4.0 Hz, 1H), 7.96 (s, 1H), 7.83 (s, 1H), 7.59 (s, 1H), 7.55 – 7.52 (m, 2H), 7.47 – 7.43 (m, 2H), 7.31 – 7.28 (m, 1H), 3.70 (s, 2H), 1.76 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.5, 163.7, 146.3, 138.3, 136.5, 133.0, 130.7 (q, $J_{C\text{-}F}$ = 32.0 Hz), 139.0, 126.1 (q, $J_{C\text{-}F}$ = 4.0 Hz), 124.2 (q, $J_{C\text{-}F}$ = 271.0 Hz), 123.7 (q, $J_{C\text{-}F}$ = 4.0 Hz), 122.4, 120.1, 56.3, 43.9, 27.3. HRMS (EI-TOF) calc. for $C_{17}H_{17}F_3N_2O$ (M^+): 322.1293, found: 322.1293.

Optimization of Reaction Conditions:

Table S3. Optimization of Reaction Conditions

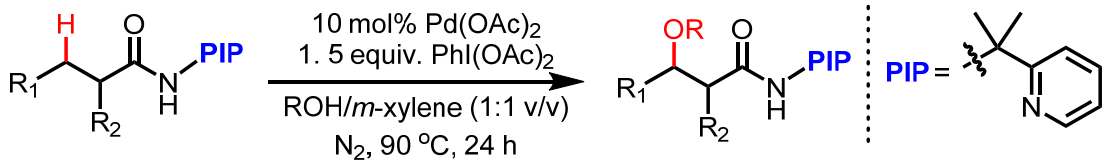


1 **2**

Entry	Oxidant (equiv)	Additive	Solvent	T/℃	Yield/%
1	$PhI(OAc)_2$ (3.0)	Ac_2O 10eq	MeOH	90	63
2	$PhI(OAc)_2$ (3.0)	-	MeOH	90	50
3	$PhI(OAc)_2$ (3.0)	-	MeOH	120	51
4	$PhI(OAc)_2$ (3.0)	-	MeOH :toluene =1:1	90	74
5	$PhI(OAc)_2$ (3.0)	-	MeOH : *o*-xylene=1:1	90	73
6	$PhI(OAc)_2$ (3.0)	-	MeOH : *m*-xylene=1:1	90	79
7	$PhI(OAc)_2$ (3.0)	-	MeOH : *p*-xylene=1:1	90	72
8	$PhI(OAc)_2$ (3.0)	-	MeOH : *o*-xylene=1:2	90	73
9	$PhI(OAc)_2$ (3.0)	-	MeOH : *o*-xylene=1:4	90	61
10	$PhI(OAc)_2$ (3.0)	-	MeOH : *o*-xylene=1:6	90	52
11	$PhI(OAc)_2$ (3.0)	-	MeOH : *o*-xylene=1:10	90	49
12	$PhI(OAc)_2$ (3.0)	-	MeOH : *o*-xylene=1:100	90	42
13	$PhI(OAc)_2$ (3.0)	-	MeOH : *o*-xylene=100:1	90	43
14	$PhI(OAc)_2$ (3.0)	-	MeOH : *o*-xylene=10:1	90	54
15	$PhI(OAc)_2$ (3.0)	-	MeOH : *o*-xylene=6:1	90	60
16	$PhI(OAc)_2$ (3.0)	-	MeOH : *o*-xylene=4:1	90	69
17	$PhI(OAc)_2$ (3.0)	-	MeOH : *o*-xylene=2:1	90	71
18	$PhI(TFA)_2$ (3.0)	-	MeOH : *o*-xylene=1:1	90	0
19	$PhI(OPiv)_2$ (3.0)	-	MeOH : o-xylene=1:1	90	67
20	$PhI(OPiv)_2$ (3.0)	-	MeOH : *m*-xylene=1:1	90	64
21	$PhI(OAc)_2$ (3.0)	1-AdCOOH	MeOH : *m*-xylene=1:1	90	76
22	$PhI(OAc)_2$ (3.0)		MeOH : *m*-xylene=1:1	90	66
23	$PhI(OAc)_2$ (3.0)	$KHCO_3$	MeOH : *m*-xylene=1:1	90	62
24	$KHSO_6$ (3.0)	-	MeOH : *m*-xylene=1:1	90	0
25	$K_2S_2O_8$ (3.0)	-	MeOH : *m*-xylene=1:1	90	0
26	Oxone (3.0)	-	MeOH : *m*-xylene=1:1	90	0
27	$NaIO_3$ (3.0)	-	MeOH : *m*-xylene=1:1	90	0
28	DDQ (3.0)	-	MeOH : *m*-xylene=1:1	90	0
29	Selectfluor (3.0)	-	MeOH : *m*-xylene=1:1	90	0
30	**$PhI(OAc)_2$ (3.0)**	**-**	**MeOH : *m*-xylene=1:1**	**90**	**89[a]**
31	$PhI(OAc)_2$ (3.0)	-	MeOH : *m*-xylene=1:1	90	70[b]
32	**$PhI(OAc)_2$ (1.5)**	**-**	**MeOH : *m*-xylene=1:1**	**90**	**80[a]**

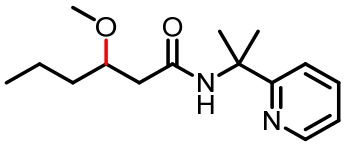
[a]$PhI(OAc)_2$ was used after recrystallized from HOAc and Hex. [b]the reaction was carried on under the atmosphere of O_2.

General Procedure for the Alkoxylation of C(sp^3)–H bonds:



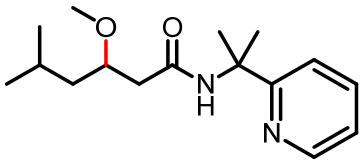
A mixture of substrate (0.2 mmol), $Pd(OAc)_2$ (4.5 mg, 10 mmol%), $PhI(OAc)_2$ (97 mg, 0.3 mmol), alcohol (1.0 mL) and *m*-xylene (1.0 mL) in a 30 mL Schlenk tube (purged with N_2) was heated at 90 °C for 24 hours. The reaction mixture was cooled to RT, and concentrated *in vacuo*. The resulting residue was purified by flash chromatography to give the desired product.

3-Methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 4



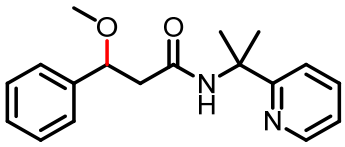
The title compound **4** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 2 : 1). **4** was obtained as a yellow liquid (43.9 mg, 83%). R_f = 0.25 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, *J* = 4.5 Hz, 1H), 7.91 (s, 1H), 7.73 (t, *J* = 7.8 Hz, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.20 (t, *J* = 5.6 Hz ,1H), 3.67-3.57 (m, 1H), 3.39 (s, 3H), 2.42 (dd, *J* = 14.6, 7.6 Hz, 1H), 2.35 (dd, *J* = 14.6, 4.4 Hz, 1H), 1.74 (s, 3H), 1.74 (s, 3H), 1.59-1.43 (m, 2H), 1.43-1.29 (m, 2H), 0.91 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 164.5, 147.4, 137.6, 122.0, 119.8, 78.4, 57.2, 56.6, 42.6, 36.1, 27.7, 27.6, 18.5, 14.3. HRMS (EI-TOF) calc. for $C_{15}H_{24}N_2O_2$ (M^+): 264.1838, found: 264.1834.

3-Methoxy-5-methyl-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 5



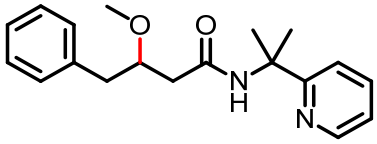
The title compound **5** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **5** was obtained as a colorless liquid (40.5 mg, 73%). R_f = 0.52 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.1 Hz, 1H), 7.89 (s, 1H), 7.69 (t, *J* = 7.7 Hz, 1H), 7.40 (d, *J* = 8.1 Hz, 1H), 7.17 (m, 1H), 3.69 (m, 1H), 3.40 (s, 3H), 2.39 (m, 2H), 1.75 (s, 6H), 1.53 (m, 1H), 1.29 (m, 2H), 0.92 (d, *J* = 5.3 Hz, 3H), 0.91 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 164.8, 147.9, 137.0, 121.9, 119.6, 57.2, 56.7, 43.7, 43.1, 27.7, 24.8, 23.1, 22.9. .HRMS (EI-TOF) calc. for $C_{16}H_{26}N_2O_2(M^+)$: 278.1994, found: 278.2002.

3-Methoxy-3-phenyl-*N*-(2-(pyridin-2-yl)propan-2-yl)propanamide 6



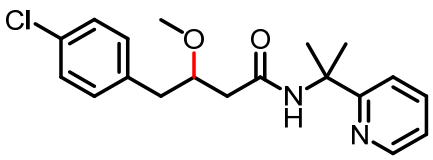
The title compound **6** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **6** was obtained as a light yellow liquid (59.1 mg, 89%). R_f = 0.57 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.7 Hz, 1H), 7.98 (s, 1H), 7.68 (t, *J* = 7.7 Hz, 1H), 7.36 (m, 5H), 7.28 (dd, *J* = 10.0, 5.8 Hz, 1H), 7.16 (m, 1H), 4.63 (dd, *J* = 9.3, 3.7 Hz, 1H), 3.30 (s, 3H), 2.75 (dd, *J* = 14.7, 9.4 Hz, 1H), 2.47 (dd, *J* = 14.8, 3.6 Hz, 1H), 1.75 (s, 3H), 1.70 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7, 164.7, 147.8, 141.2, 137.0, 128.7, 128.0, 126.6, 121.8, 119.5, 80.7, 57.0, 56.8, 46.8, 27.7, 27.6. HRMS (EI-TOF) calc. for $C_{18}H_{22}N_2O_2$ (M^+): 298.1681, found: 298.1681.

3-Methoxy-4-phenyl-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 7



The title compound **7** was prepared according to the general procedure with 3.0 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **7** was obtained as a yellow liquid (55.9 mg, 90%). R_f = 0.61 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 4.7 Hz, 1H), 7.84 (s, 1H), 7.70 – 7.63 (m, 1H), 7.37 (d, *J* = 8.1 Hz, 1H), 7.29 – 7.17 (m, 5H), 7.14 (dd, *J* = 7.1, 5.4 Hz, 1H), 3.86 (p, *J* = 6.1 Hz, 1H), 3.37 (s, 3H), 2.88 (dd, *J* = 13.7, 5.9 Hz, 1H), 2.80 (dd, *J* = 13.7, 6.2 Hz, 1H), 2.34 (d, *J* = 6.1 Hz, 2H), 1.72 (s, 3H), 1.71 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 164.7, 147.8, 138.1, 137.0, 129.7, 128.5, 126.4, 121.8, 119.5, 79.7, 57.7, 56.7, 42.5, 40.1, 27.7. HRMS (EI-TOF) calc. for $C_{19}H_{24}N_2O_2$ (M^+): 312.1838, found: 312.1829.

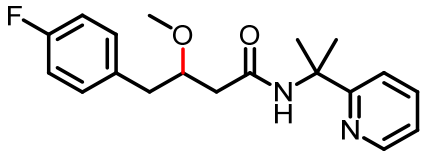
4-(4-Chlorophenyl)-3-methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 8



The title compound **8** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **8** was obtained as a light yellow liquid (62.2 mg, 90%). R_f = 0.42 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 4.3 Hz, 1H), 7.87 (s, 1H), 7.69 (t, *J* = 7.7 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.24 (d, *J* = 8.1 Hz, 2H), 7.16 (m, 3H), 3.90 – 3.80 (m, 1H), 3.36 (s, 3H), 2.82 (d, *J* = 5.8 Hz, 2H), 2.36 (d, *J* = 7.2 Hz, 2H), 1.73 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 164.5, 147.8, 137.1, 136.6,

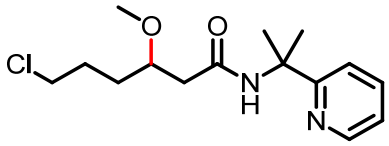
131.1, 128.5, 121.9, 119.5, 79.4, 57.7, 56.7, 42.3, 39.4, 27.6. HRMS (EI-TOF) calc. for $C_{19}H_{23}ClN_2O_2$ (M^+): 346.1448, found: 346.1454.

4-(4-Fluorophenyl)-3-methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 9



The title compound **9** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **9** was obtained as a colorless liquid (55.5 mg, 83%). R_f = 0.49 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, J = 4.6 Hz, 1H), 7.85 (s, 1H), 7.72 (t, J = 7.6 Hz, 1H), 7.42 (d, J = 7.9 Hz, 1H), 7.18 (m, 3H), 6.96 (t, J = 8.5 Hz, 2H), 3.84 (m, 1H), 3.37 (s, 3H), 2.82 (m, 2H), 2.35 (m, 2H), 1.74 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 164.4, 161.7 (d, $J_{C\text{-}F}$ = 247.2 Hz), 147.5, 137.5, 133.8 (d, $J_{C\text{-}F}$ = 4.0 Hz), 131.1 (d, $J_{C\text{-}F}$ = 7.4 Hz), 122.0, 119.8, 115.2 (d, $J_{C\text{-}F}$ = 21.0 Hz), 79.5, 57.7, 56.6, 42.2, 39.2, 27.7, 27.6. HRMS (EI-TOF) calc. for $C_{19}H_{23}FN_2O_2$ (M^+): 330.1744, found: 330.1748.

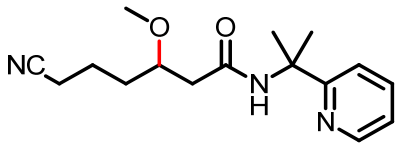
6-Chloro-3-methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 10



The title compound **10** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **10** was obtained as a colorless liquid (41.8 mg, 70%). R_f = 0.53 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, J = 4.4 Hz, 1H), 7.93 (s, 1H), 7.68 (t, J = 7.6 Hz, 1H), 7.38 (d, J = 8.0 Hz, 1H), 7.20 – 7.11 (m, 1H), 3.66 (m, 1H), 3.53 (t,

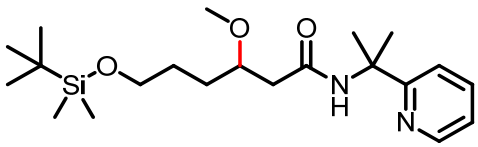
J = 6.5 Hz, 2H), 3.38 (s, 3H), 2.47 (dd, *J* = 14.5, 7.3 Hz, 1H), 2.32 (dd, *J* = 14.5, 4.4 Hz, 1H), 1.89 – 1.78 (m, 2H), 1.72 (s, 6H), 1.69 – 1.57 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 164.7, 147.9, 137.1, 121.9, 119.6, 77.9, 57.3, 56.8, 45.2, 42.7, 31.3, 28.4, 27.6. HRMS (EI-TOF) calc. for $C_{15}H_{23}ClN_2O_2$ (M^+): 298.1448, found: 298.1450.

6-Cyano-3-methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 11



The title compound **11** was prepared according to the general procedure and was purified by flash chromatography (ethyl acetate). **11** was obtained as a colorless liquid (40.2 mg, 67%). R_f = 0.53 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.6 Hz, 1H), 7.96 (s, 1H), 7.72 (t, *J* = 7.7 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.19 (m, 1H), 3.69 (m, 1H), 3.40 (s, 3H), 2.51 (dd, *J* = 14.5, 6.9 Hz, 1H), 2.38 (t, *J* = 6.8 Hz, 2H), 2.33 (dd, *J* = 14.6, 5.2 Hz, 1H), 1.80 (m, 2H), 1.75 (s, 6H), 1.69 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9, 164.5, 147.8, 137.3, 122.0, 119.7, 119.6, 77.5, 57.4, 56.7, 42.3, 33.0, 27.6, 21.5, 17.4. HRMS (EI-TOF) calc. for $C_{16}H_{23}N_3O_2$ (M^+): 289.1790, found: 289.1796.

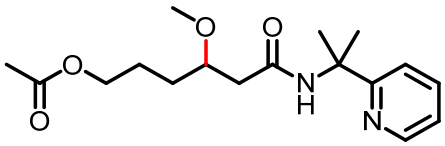
6-((*tert*-Butyldimethylsilyl)oxy)-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 12



The title compound **12** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 2 : 3). **12** was obtained as a yellow liquid (61.8 mg, 78%). R_f = 0.43 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.4 Hz, 1H), 7.93 (s, 1H), 7.69 (t,

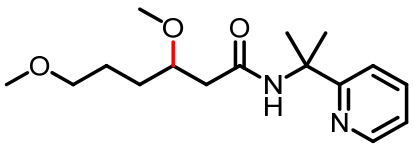
J = 7.6 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.17 (m, 1H), 3.66 (m, 1H), 3.61 (t, *J* = 5.2 Hz, 2H), 3.40 (s, 3H), 2.44 (dd, *J* = 14.6, 7.8 Hz, 1H), 2.35 (dd, *J* = 14.5, 4.1 Hz, 1H), 1.75 (s, 3H), 1.74 (s, 3H), 1.61 (m, 4H), 0.88 (s, 9H), 0.05 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 164.8, 147.9, 137.0, 121.9, 119.6, 78.4, 63.2, 57.2, 56.7, 42.9, 30.1, 28.4, 27.7, 27.7, 26.1, 18.5, -5.2. HRMS (EI-TOF) calc. for $C_{21}H_{38}N_2O_3Si$ (M^+): 394.2652, found: 394.2646.

4-Methoxy-6-oxo-6-((2-(pyridin-2-yl)propan-2-yl)amino)hexyl acetate 13



The title compound **13** was prepared according to the general procedure and was purified by flash chromatography (ethyl acetate). **13** was obtained as a colorless liquid (44.1 mg, 70%). R_f = 0.63 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.0 Hz, 1H), 7.94 (s, 1H), 7.69 (t, *J* = 7.6 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.17 (m, 1H), 4.06 (t, *J* = 6.3 Hz, 2H), 3.66 (m, 1H), 3.39 (s, 3H), 2.46 (dd, *J* = 14.4, 7.3 Hz, 1H), 2.33 (dd, *J* = 14.5, 4.4 Hz, 1H), 2.01 (s, 3H), 1.73 (s, 6H), 1.68 (m, 2H), 1.61 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 170.2, 164.6, 147.8, 137.1, 121.9, 119.5, 78.1, 64.5, 57.3, 56.7, 42.6, 30.4, 27.6, 24.5, 21.1. HRMS (EI-TOF) calc. for $C_{17}H_{26}N_2O_4$ (M^+): 322.1893, found: 322.1886.

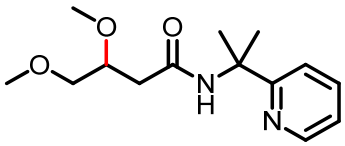
3,6-Dimethoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 14



The title compound **14** was prepared according to the general procedure and was purified by flash chromatography (ethyl acetat). **14** was obtained as a colorless liquid (42.0 mg, 71%). R_f = 0.43 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* =

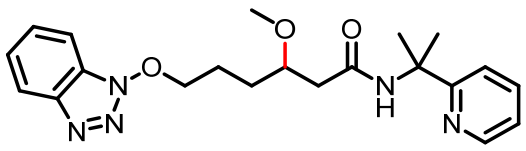
4.3 Hz, 1H), 7.91 (s, 1H), 7.69 (t, *J* = 7.7 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.17 (m, 1H), 3.65 (m, 1H), 3.39 (s, 3H), 3.37 (t, *J* = 5.9 Hz, 2H), 3.31 (s, 3H), 2.44 (dd, *J* = 14.5, 7.7 Hz, 1H), 2.34 (dd, *J* = 14.6, 4.2 Hz, 1H), 1.74 (s, 6H), 1.62 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 164.7, 147.9, 137.0, 121.9, 119.5, 78.3, 72.8, 58.6, 57.2, 56.7, 42.8, 30.4, 27.7, 27.7, 25.3. HRMS (EI-TOF) calc. for $C_{16}H_{26}N_2O_3$ (M^+): 294.1943, found: 294.1935.

3,4-Dimethoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 15



The title compound **15** was prepared according to the general procedure and was purified by flash chromatography (ethyl acetate). **15** was obtained as a light yellow liquid (46.7 mg, 88%). R_f = 0.49 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.4 Hz, 1H), 7.87 (s, 1H), 7.68 (t, *J* =7.5Hz, 1H), 7.39 (d, *J* = 8.1 Hz, 1H), 7.16 (dd, *J* = 6.9, 5.4 Hz, 1H), 3.80 (m, 1H), 3.52 (dd, *J* = 10.6, 4.7 Hz, 1H), 3.45 (s, 3H), 3.42 (m, 1H), 3.37 (s, 3H), 2.51 (dd, *J* = 14.5, 6.7 Hz, 1H), 2.43 (dd, *J* = 14.5, 5.1 Hz, 1H), 1.74 (s, 3H), 1.73 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 164.6, 147.8, 137.1, 121.9, 119.5, 73.9, 59.4, 57.9, 56.8, 40.1, 27.7, 27.6. HRMS (EI-TOF) calc. for $C_{14}H_{22}N_2O_3$ (M^+): 266.1630, found: 266.1626.

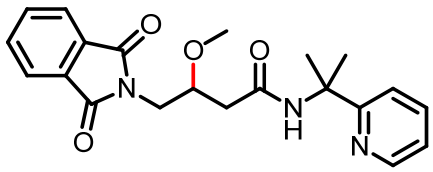
6-((1H-Benzo[*d*][1,2,3]triazol-1-yl)oxy)-3-methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 16



The title compound **16** was prepared according to the general procedure and was purified by flash chromatography (ethyl acetate). **16** was obtained as a light yellow

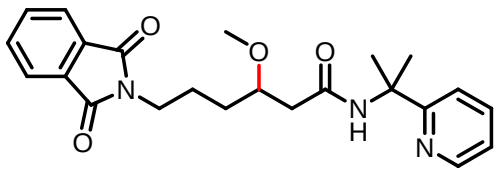
liquid (39.3 mg, 58%). R_f = 0.42 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.5 Hz, 1H), 7.99 (d, *J* = 8.4 Hz, 1H), 7.95 (s, 1H), 7.68 (dt, *J* = 8.6, 2.0 Hz, 1H), 7.57 (d, *J* = 8.3 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.42-7.33 (m, 2H), 7.16 (dd, *J* = 6.8, 5.3 Hz, 1H), 4.56 (t, *J* = 6.4 Hz, 2H), 3.74 (m, 1H), 3.41 (s, 3H), 2.54 (dd, *J* = 14.5, 7.0 Hz, 1H), 2.37 (dd, *J* = 14.5, 5.1 Hz, 1H), 1.97 (m, 2H), 1.85 (m, 2H), 1.74 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 164.5, 147.8, 143.6, 137.1, 128.1, 127.5, 124.7, 121.9, 120.3, 119.5, 108.8, 80.9, 77.9, 57.3, 56.7, 42.4, 30.1, 27.6, 27.6, 24.1. HRMS (EI-TOF) calc. for $C_{21}H_{27}N_5O_3$ (M^+): 397.2114, found: 397.2116.

4-(1,3-Dioxoisoindolin-2-yl)-3-methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 17



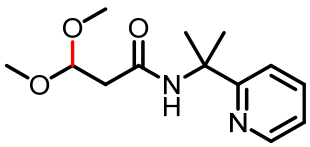
The title compound **17** was prepared according to the general procedure and was purified by flash chromatography (ethyl acetate). **17** was obtained as a colorless liquid (45.8 mg, 60%). R_f = 0.64 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.4 Hz, 1H), 7.85 (m, 3H), 7.72 (dd, *J* = 5.3, 3.2 Hz, 2H), 7.68 (t, *J* = 8.1 Hz, 1H), 7.39 (d, *J* = 7.9 Hz, 1H), 7.16 (dd, *J* = 7.4, 5.2 Hz, 1H), 3.98 (m, 1H), 3.87 (d, *J* = 5.1 Hz, 2H), 3.49 (s, 3H), 2.46 (m, 1H), 1.73 (s, 3H), 1.71(s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.4, 168.6, 164.6, 147.9, 137.0, 134.2, 132.2, 123.5, 121.9, 119.5, 76.6, 58.1, 56.9, 41.7, 40.1, 27.7. HRMS (EI-TOF) calc. for $C_{21}H_{23}N_3O_4$ (M^+): 381.1689, found:381.1691.

6-(1,3-Dioxoisoindolin-2-yl)-3-methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)hexanamide 18



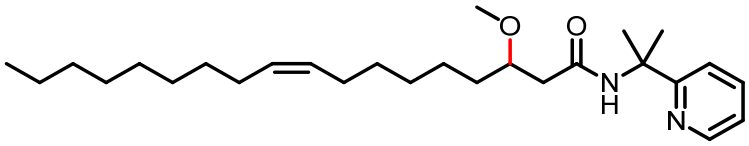
The title compound **18** was prepared according to the general procedure and was purified by flash chromatography (ethyl acetat). **18** was obtained as a yellow liquid (54.1 mg, 66%). R_f = 0.55 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 4.5 Hz, 1H), 7.87 (s, 1H), 7.81 (dd, *J* = 4.9, 3.1 Hz, 2H), 7.72 – 7.64 (m, 3H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.19 – 7.13 (m, 1H), 3.74 – 3.63 (m, 3H), 3.38 (s, 3H), 2.43 (dd, *J* = 14.5, 7.5 Hz, 1H), 2.32 (dd, *J* = 14.5, 4.4 Hz, 1H), 1.78 (m, 2H), 1.71 (s, 3H), 1.70 (s,3H), 1.59 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 168.5, 164.6, 147.8, 137.0, 134.0, 132.2, 123.3, 121.9, 119.5, 78.0, 57.5, 56.7, 42.6, 38.0, 31.2, 27.6, 24.5. HRMS (EI-TOF) calc. for $C_{23}H_{27}N_3O_4$ (M^+): 409.2002, found: 409.2008.

3,3-Dimethoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)propanamide 19



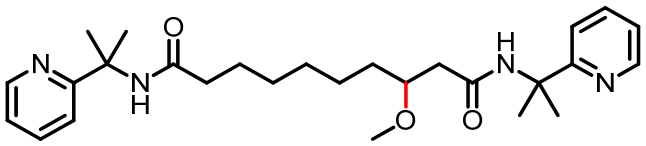
The title compound **19** was prepared according to the general procedure and was purified by flash chromatography (ethyl acetate). **19** was obtained as a light yellow liqiud (40.8 mg, 81%). R_f = 0.35 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.7 Hz, 1H), 8.00 (s, 1H), 7.69 (m, 1H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.17 (dd, *J* = 7.3, 5.0 Hz, 1H), 4.77 (t, *J* = 5.5 Hz, 1H), 3.41 (s, 6H), 2.58 (d, *J* = 5.5 Hz, 2H), 1.74 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.4, 164.6, 147.7, 137.2, 121.9, 119.6, 102.5, 56.8, 54.1, 42.2, 27.7. HRMS (EI-TOF) calc. for $C_{13}H_{20}N_2O_3$ (M^+): 252.1474, found: 252.1479.

(Z)-3-Methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)octadec-9-enamide 20



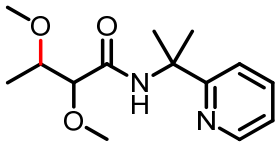
The title compound **20** was prepared according to the general procedure with 3.0 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 1). **20** was obtained as a yellow liqiud (63.2 mg, 73%). R_f = 0.60 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.2 Hz, 1H), 7.92 (s, 1H), 7.69 (t, *J* = 7.2 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.17 (m, 1H), 5.34 (m, 2H), 3.62 (m, 1H), 3.40 (s, 3H), 2.43 (dd, *J* = 14.6, 7.7 Hz, 1H), 2.35 (dd, *J* = 14.6, 4.3 Hz, 1H), 2.01 (m, 4H), 1.75 (s, 3H), 1.74(s, 3H), 1.53 (m, 2H), 1.32 (m, 6H), 1.26 (m, 12H), 0.87 (t, *J* = 6.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6, 164.8, 147.9, 137.0, 129.8, 121.8, 119.5, 78.7, 57.2, 56.7, 42.8, 29.7, 29.5, 27.7, 27.3, 22.8, 14.3. HRMS (EI-TOF) calc. for $C_{27}H_{46}N_2O_2$ (M^+): 430.3559, found: 430.3555.

3-Methoxy-*N*^{1},*N*^{10}-bis(2-(pyridin-2-yl)propan-2-yl)decanediamide 21



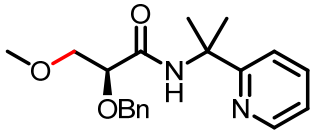
The title compound **21** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : acetone = 1 : 1). **21** was obtained as a colorless liquid (40.8 mg, 44%). R_f = 0.58 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.0 Hz, 2H), 7.90 (s, 1H), 7.70 (m, 3H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.16 (m, 2H), 3.60 (m, 1H), 3.38 (s, 3H), 2.41 (dd, *J* = 14.6, 7.6 Hz, 1H), 2.34 (dd, *J* = 14.4, 4.9 Hz, 1H), 2.23 (t, *J* = 7.5 Hz, 3H), 1.73 (s, 12H), 1.64 (m, 1H), 1.53 (m, 2H), 1.33 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 170.6, 164.8, 164.7, 147.9, 147.7, 137.2, 137.0, 121.9, 121.8, 119.6, 119.5, 78.6, 57.2, 56.7, 56.4, 42.7, 37.9, 22.8, 29.6, 29.3, 27.7, 25.8. HRMS (EI-TOF) calc. for $C_{27}H_{40}N_4O_3$ (M^+): 468.3100, found: 468.3101.

2,3-Dimethoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 22



The title compound **22** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 1). **22** was obtained as a colorless liquid (26.0 mg, 48%, dr = 2:1). R_f = 0.41 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (ddd, *J* = 4.8, 1.7, 0.9 Hz, 1H), 8.26 (s, 1H), 7.67 (dt, *J* = 7.8, 1.8 Hz, 1H), 7.43 (d, *J* = 8.1 Hz, 1H), 7.16 (ddd, *J* = 7.4, 4.9, 1.0 Hz, 1H), 3.75 (dq, *J* = 6.5, 2.8 Hz, 1H), 3.55 (s, 3H), 3.46 (d, *J* = 2.8 Hz, 1H), 3.35 (s, 3H), 1.78 (s, 3H), 1.75 (s, 3H), 1.27 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 164.6, 148.2, 136.9, 121.8, 119.4, 86.5, 77.4, 60.2, 57.6, 56.8, 28.2, 27.5, 15.5. HRMS (EI-TOF) calc. for $C_{14}H_{22}N_2O_3$ (M^+): 266.1630, found: 266.1637.

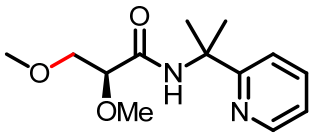
(S)-2-(Benzyloxy)-3-methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)propanamide 23



The title compound **23** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 2 : 1). **23** was obtained as a yellow liquid (50.4 mg, 77%). R_f = 0.60 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.80 (s, 1H), 8.49 (d, *J* = 4.8 Hz, 1H), 7.68 (t, *J* = 7.8 Hz, 1H), 7.48 (d, *J* = 7.2 Hz, 2H), 7.33 (dt, *J* = 14.2, 8.1 Hz, 4H), 7.17 (dd, *J* = 7.3, 5.0 Hz, 1H), 4.74 (s, 2H), 4.02 (dd, *J* = 5.6, 2.4 Hz, 1H), 3.78 (dd, *J* = 10.5, 2.3 Hz, 1H), 3.72 (dd, *J* = 10.6, 5.7 Hz, 1H), 3.39 (s, 3H), 1.75 (s, 3H), 1.73 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.2, 164.5, 147.9, 137.5, 137.0, 128.6, 128.3, 128.1,

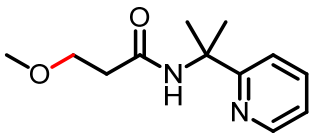
121.9, 119.4, 80.5, 73,7 73.4, 59.4, 56.7, 27.9, 27.4. HRMS (EI-TOF) calc. for $C_{19}H_{24}N_2O_3$ (M^+): 328.1787, found: 328.1781.

(S)-2,3-Dimethoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)propanamide 24



The title compound **24** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **24** was obtained as a yellow oil (37.2 mg, 73%). R_f = 0.45 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (d, *J* = 4.4 Hz, 1H), 8.41 (s, 1H), 7.68 (td, *J* = 8.0, 1.5 Hz, 1H), 7.41 (d, *J* = 8.1 Hz, 1H), 7.16 (dd, *J* = 6.9, 5.3 Hz, 1H), 3.81 – 3.77 (m, 1H), 3.78 – 3.73 (m, 1H), 3.67 (dd, *J* = 10.4, 5.3 Hz, 1H), 3.55 (s, 3H), 3.39 (s, 3H), 1.76 (s, 3H), 1.74 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.2, 164.4, 148.1, 137.0, 121.9, 119.4, 82.6, 72.8, 59.4, 58.8, 56.7, 28.0, 27.5. HRMS (EI-TOF) calc. for $C_{13}H_{20}N_2O_3$ (M^+): 252.1474, found: 252.1466.

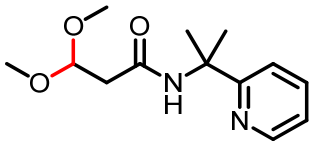
3-Methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)propanamide 25



The title compound **25** was prepared according to the general procedure with 1.0 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (petroleum ether : acetone = 1 : 1). **25** was obtained as a yellow liquid (41.0 mg, 92%). R_f = 0.49 (ethyl acetate). ^{1}H NMR (400 MHz,$CDCl_3$) δ 8.51 (d, *J* = 4.3 Hz, 1H), 7.97 (s, 1H), 7.69 (t, *J* = 7.7 Hz, 1H), 7.40 (d, *J* = 8.1 Hz, 1H), 7.17 (m, 1H), 3.69 (t, *J* = 6.0 Hz, 2H), 3.40 (s, 3H), 2.51 (t, *J* = 6.0 Hz, 2H), 1.74 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.0,

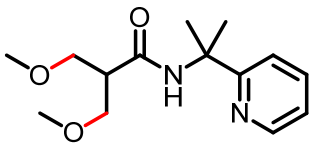
165.3, 148.4, 137.6, 122.4, 120.1, 69.6, 59.5, 57.3, 38.8, 28.3. HRMS (EI-TOF) calc. for $C_{12}H_{18}N_2O_2$ (M^+): 222.1368, found: 222.1371. .

3,3-Dimethoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)propanamide 26



The title compound **26** was prepared according to the general procedure with 3.0 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (petroleum ether : acetone = 1 : 1). **26** was obtained as a yellow liquid (35.1 mg, 70%). R_f = 0.46 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.7 Hz, 1H), 8.00 (s, 1H), 7.69 (m, 1H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.17 (dd, *J* = 7.3, 5.0 Hz, 1H), 4.77 (t, *J* = 5.5 Hz, 1H), 3.41 (s, 6H), 2.58 (d, *J* = 5.5 Hz, 2H), 1.74 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.4, 164.6, 147.7, 137.2, 121.9, 119.6, 102.5, 56.8, 54.1, 42.2, 27.7. HRMS (EI-TOF) calc. for $C_{13}H_{20}N_2O_3$ (M^+): 252.1474, found: 252.1479.

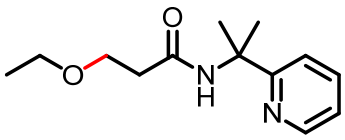
3-Methoxy-2-(methoxymethyl)-*N*-(2-(pyridin-2-yl)propan-2-yl)propanamide 27



The title compound **27** was prepared according to the general procedure with 2.0 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (ethyl acetate). **27** was obtained as a colorless liquid (39.9 mg, 75%). R_f = 0.34 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.2 Hz, 1H), 8.09 (s, 1H), 7.67 (t, *J* = 7.7 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.16 (m, 1H), 3.61 (m, 4H), 3.38 (s, 6H), 2.76 (m, 1H), 1.73 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 164.8, 148.0, 136.9,

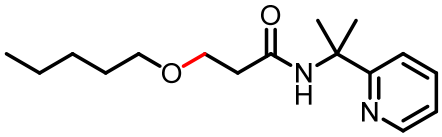
121.8, 119.5, 71.2, 59.1, 57.0, 48.1, 27.8. HRMS (EI-TOF) calc. for $C_{14}H_{22}N_2O_3$ (M^+): 266.1630, found: 266.1629.

3-Ethoxy-N-(2-(pyridin-2-yl)propan-2-yl)propanamide 28



The title compound **28** was prepared according to the general procedure with 1.5 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (ethyl acetate). **28** was obtained as a colorless liquid (33.3 mg, 71%). R_f = 0.60 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.3 Hz, 1H), 7.94 (s, 1H), 7.67 (td, *J* = 7.9, 1.8 Hz, 1H), 7.39 (d, *J* = 8.1 Hz, 1H), 7.16 (m, 1H), 3.71 (t, *J* = 6.0 Hz, 2H), 3.54 (q, *J* = 7.0 Hz, 2H), 2.50 (t, *J* = 6.0 Hz, 2H), 1.73 (s, 6H), 1.22 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 164.8, 147.9, 137.0, 121.8, 119.5, 66.9, 66.6, 56.9, 38.4, 27.8, 15.2. HRMS (EI-TOF) calc. for $C_{13}H_{20}N_2O_2$ (M^+): 236.1525, found:236.1527.

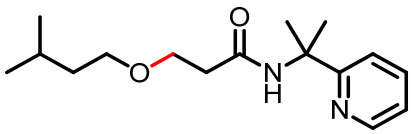
3-(Pentyloxy)-N-(2-(pyridin-2-yl)propan-2-yl)propanamide 29



The title compound **29** was prepared according to the general procedure with 1.5 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (ethyl acetate). **29** was obtained as a light yellow liquid (40.8 mg, 72%). R_f = 0.55 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (m, 1H), 7.87 (s, 1H), 7.68 (td, *J* = 7.8, 1.8 Hz, 1H), 7.39 (d, *J* = 8.1 Hz, 1H), 7.16 (ddd, *J* = 7.4, 4.9, 0.9 Hz, 1H), 3.70 (t, *J* = 6.0 Hz, 2H), 3.47 (t, *J* = 6.7 Hz, 2H), 2.50 (t, *J* = 6.0 Hz, 2H), 1.73 (s, 6H), 1.59 (m, 2H), 1.31 (td, *J* = 7.1, 3.7 Hz, 4H), 0.86 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 164.9,

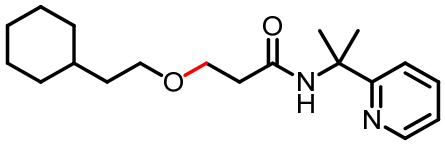
147.9, 137.0, 121.8, 119.5, 71.5, 67.2, 56.9, 38.5, 29.5, 28.5, 27.8, 22.7, 14.1. HRMS (EI-TOF) calc. for $C_{16}H_{26}N_2O_2$ (M^+): 278.1994, found:278.2000.

3-(Isopentyloxy)-N-(2-(pyridin-2-yl)propan-2-yl)propanamide 30



The title compound **30** was prepared according to the general procedure with 1.5 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (ethyl acetate). **30** was obtained as a light yellow liquid (41.0 mg, 74%, mono: di = 10:1). R_f = 0.50 (1/1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (m, 1H), 7.80 (s, 1H), 7.61 (td, *J* = 7.8, 1.8 Hz, 1H), 7.33 (d, *J* = 8.1 Hz, 1H), 7.09 (ddd, *J* = 7.4, 4.9, 0.9 Hz, 1H), 3.64 (t, *J* = 6.0 Hz, 2H), 3.44 (t, *J* = 6.8 Hz, 2H), 2.43 (t, *J* = 6.0 Hz, 2H), 1.67 (s, 6H), 1.61 (m, 1H), 1.43 (m, 2H), 0.82 (d, *J* = 6.6 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 164.7, 147.8, 147.8, 136.8, 128.8, 121.7, 119.4, 69.7, 67.1, 56.7, 38.5, 38.4, 27.6, 25.1, 22.6. HRMS (EI-TOF) calc. for $C_{16}H_{26}N_2O_2$ (M^+): 278.1994, found:278.1996.

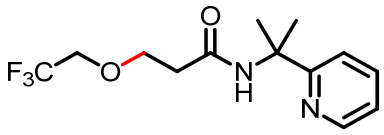
3-(2-Cyclohexylethoxy)-N-(2-(pyridin-2-yl)propan-2-yl)propanamide 31



The title compound **31** was prepared according to the general procedure with 1.5 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (ethyl acetate). **31** was obtained as a light yellow liquid (32.0 mg, 53%). R_f = 0.65 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, *J* = 4.4 Hz, 1H), 7.88 (s, 1H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.39 (d, *J* = 7.6 Hz, 1H), 7.16 (dd, *J* = 6.4, 4.8 Hz, 1H), 3.70 (t, *J* = 5.6 Hz, 2H), 3.51 (t, *J* = 6.8 Hz, 2H), 1.74 (s, 6H), 1.70 – 1.63 (m, 6H), 1.49 (dd, *J* = 13.6, 6.8 Hz, 2H),

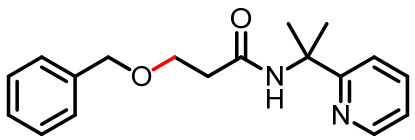
1.38 – 1.33 (m, 1H), 1.19 – 1.14 (m, 2H), 0.93 – 0.85 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 164.9, 147.9, 137.0, 121.8, 119.5, 69.4, 67.2, 56.9, 38.5, 37.3, 34.7, 33.5, 27.8, 26.7, 26.4. HRMS (EI-TOF) calc. for $C_{19}H_{30}N_2O_2$ (M^+): 318.2307, found: 318.2305.

_N_-(2-(Pyridin-2-yl)propan-2-yl)-3-(2,2,2-trifluoroethoxy)propanamide 32



The title compound **32** was prepared according to the general procedure with 1.5 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (ethyl acetate). **32** was obtained as a light yellow liquid (37.5 mg, 65%). R_f = 0.70 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 – 8.49 (m, 1H), 7.80 (s, 1H), 7.70 (dt, J = 7.6, 1.6 Hz, 1H), 7.39 (d, J = 8.0 Hz, 1H), 7.19 – 7.16 (m, 1H), 3.93 (t, J = 6.0 Hz, 2H), 3.88 (t, J = 8.8 Hz, 2H), 2.55 (d, J = 5.6 Hz, 2H), 1.74 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.6, 164.5, 147.8, 137.2, 124.1 (q, $J_{C\text{-}F}$ = 279.9 Hz), 122.0, 119.5, 69.2, 69.0, 68.8 (q, $J_{C\text{-}F}$ = 34.1 Hz), 56.9, 38.3, 27.7.HRMS (EI-TOF) calc. for $C_{13}H_{17}F_3N_2O_2$ (M^+): 290.1242, found: 290.1236.

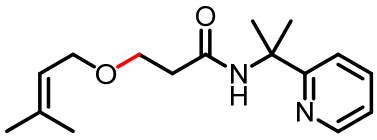
3-(Benzyloxy)-_N_-(2-(pyridin-2-yl)propan-2-yl)propanamide 33



The title compound **33** was prepared according to the general procedure with 1.5 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (ethyl acetate). **33** was obtained as a light yellow liquid (45.2 mg, 75%). R_f = 0.69 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (m, 1H), 7.91 (s, 1H), 7.65 (td, J = 7.8, 1.8 Hz, 1H), 7.38 (d,

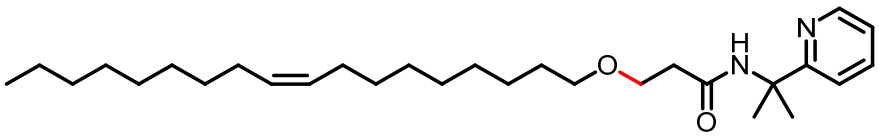
J = 8.1 Hz, 1H), 7.30 (m, 5H), 7.14 (ddd, *J* = 7.4, 4.9, 0.9 Hz, 1H), 4.57 (s, 2H), 3.79 (t, *J* = 6.0 Hz, 2H), 2.55 (t, *J* = 6.0 Hz, 2H), 1.73 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6, 164.7, 147.9, 138.2, 137.0, 128.5, 127.8, 127.8, 121.8, 119.5, 73.4, 66.9, 56.9, 38.5, 27.7. HRMS (EI-TOF) calc. for $C_{18}H_{22}N_2O_2$ (M^+): 298.1681, found: 298.1685.

3-((3-Methylbut-2-en-1-yl)oxy)-*N*-(2-(pyridin-2-yl)propan-2-yl)propanamide 34



The title compound **34** was prepared according to the general procedure with 1.5 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (ethyl acetate). **34** was obtained as a light yellow liquid (35.9 mg, 65%). R_f = 0.57 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (m, 1H), 7.90 (s, 1H), 7.67 (td, *J* = 7.9, 1.7 Hz, 1H), 7.39 (d, *J* = 8.1 Hz, 1H), 7.16 (dd, *J* = 7.2, 5.0 Hz, 1H), 5.36 (t, *J* = 6.9 Hz, 1H), 4.02 (d, *J* = 6.9 Hz, 2H), 3.71 (t, *J* = 6.0 Hz, 2H), 2.51 (t, *J* = 6.0 Hz, 2H), 1.73 (s, 9H), 1.66 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 164.8, 147.9, 137.2, 137.0, 121.8, 121.0, 119.5, 67.7, 66.4, 56.9, 38.5, 27.8, 25.9, 18.2.HRMS (EI-TOF) calc. for $C_{16}H_{24}N_2O_2$ (M^+): 276.1838, found: 276.1829.

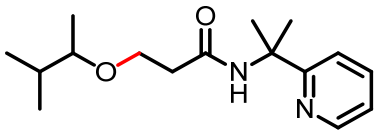
(Z)-3-(Octadec-9-en-1-yloxy)-*N*-(2-(pyridin-2-yl)propan-2-yl)propanamide 35



The title compound **35** was prepared according to the general procedure with 1.5 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 1). **35** was obtained as a light yellow liquid (65.6 mg, 71%). R_f = 0.50 (1/1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* =

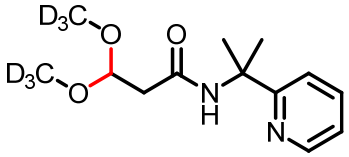
4.7 Hz, 1H), 7.88 (s, 1H), 7.67 (td, *J* = 7.9, 1.6 Hz, 1H), 7.39 (d, *J* = 8.1 Hz, 1H), 7.15 (dd, *J* = 7.3, 5.0 Hz, 1H), 5.33 (m, 2H), 3.70 (t, *J* = 5.9 Hz, 2H), 3.46 (t, *J* = 6.7 Hz, 2H), 2.49 (t, *J* = 5.9 Hz, 2H), 1.99 (m, 4H), 1.73 (s, 6H), 1.58 (m, 2H), 1.27 (m, 22H), 0.86 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 164.9, 147.91, 136.9, 130.1, 129.9, 121.8, 119.5, 71.5, 67.1, 56.9, 38.5, 32.0, 29.9, 29.8, 29.8, 29.7, 29.6, 29.6, 29.4, 29.4, 27.8, 27.3, 27.3, 26.3, 22.8, 14.2. HRMS (EI-TOF) calc. for $C_{29}H_{50}N_2O_2$ (M^+): 458.3872, found: 458.3874.

3-((3-Methylbutan-2-yl)oxy)-*N*-(2-(pyridin-2-yl)propan-2-yl)propanamide 36



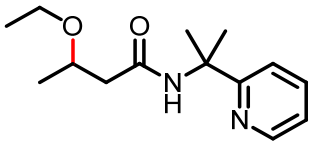
The title compound **36** was prepared according to the general procedure with 1.5 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (ethyl acetate). **36** was obtained as a light yellow liquid (30.6 mg, 55%). R_f = 0.61 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 6.8 Hz, 1H), 7.82 (s, 1H), 7.68 (dt, *J* = 8.0, 2.0 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.17 – 7.15 (m, 1H), 3.81 – 3.76 (m, 1H), 3.66 – 3.61 (m, 1H), 3.53 – 3.48 (m, 1H), 2.50 – 2.46 (m, 2H), 1.73 (d, *J* = 2.0 Hz, 6H), 1.53 – 1.47 (m, 1H), 1.14 (d, *J* = 6.0 Hz, 3H), 0.87 (dd, *J* = 11.2, 6.4 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9, 164.9, 148.0, 137.0, 121.8, 119.5, 74.2, 64.6, 56.9, 46.3, 38.8, 27.8, 27.8, 24.8, 23.1, 22.9, 19.9.HRMS (EI-TOF) calc. for $C_{16}H_{26}N_2O_2$ (M^+): 278.1994, found: 278.2000.

3,3-Di(*d_3*-methoxyl)-N-(2-(pyridin-2-yl)propan-2-yl)propanamide 37



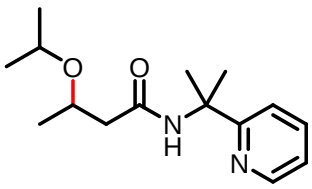
The title compound **37** was prepared according to the general procedure with 1.5 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (ethyl acetate). **37** was obtained as a light yellow liquid (40.1 mg, 77%). R_f = 0.35 (ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.8 Hz, 1H), 7.97 (s, 1H), 7.69 (td, *J* = 7.8, 1.8 Hz, 1H), 7.39 (d, *J* = 8.1 Hz, 1H), 7.17 (dd, *J* = 7.4, 4.9 Hz, 1H), 4.76 (t, *J* = 5.5 Hz, 1H), 2.58 (d, *J* = 5.5 Hz, 2H), 1.74 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.4, 164.7, 147.8, 137.1, 121.9, 119.5, 102.4, 56.9, 53.4 (q, *J* = 21.3 Hz), 42.4, 27.7. HRMS (EI-TOF) calc. for $C_{13}H_{14}D_6N_2O_3$ (M^+): 258.1851, found: 258.1854.

3-Ethoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 38



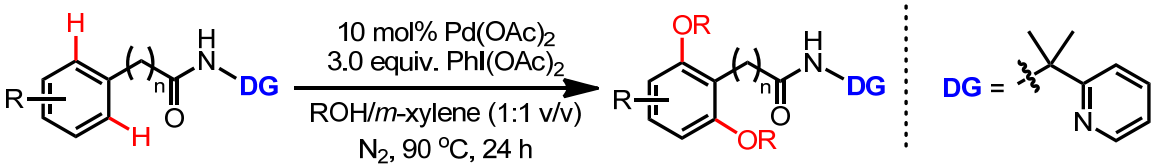
The title compound **38** was prepared according to the general procedure with 3.0 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (petroleum ether: ethyl acetate = 1:2). **38** was obtained as a colorless liquid (27.5 mg, 55%). R_f = 0.33 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.1 Hz, 1H), 7.90 (s, 1H), 7.68 (t, *J* = 7.7 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.16 (m, 1H), 3.85 (m, 1H), 3.62 (m, 1H), 3.49 (m, 1H), 2.45 (dd, *J* = 14.6, 7.8 Hz, 1H), 2.33 (dd, *J* = 14.5, 4.2 Hz, 1H), 1.74 (s, 6H), 1.21 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 164.8, 147.9, 137.0, 121.8, 119.5, 72.8, 64.2, 56.8, 45.4, 27.8, 27.7, 19.9, 15.7.HRMS (EI-TOF) calc. for $C_{14}H_{22}N_2O_2$ (M^+): 250.1681, found: 250.1685.

3-Isopropoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)butanamide 39



The title compound **39** was prepared according to the general procedure with 3.0 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (petroleum ether: ethyl acetate = 1:2). **39** was obtained as a colorless liquid (10.0 mg, 19%). R_f = 0.33 (1/ 2 petroleum ether/ ethyl acetate). ^1H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.2 Hz, 1H), 7.86 (s, 1H), 7.68 (t, *J* = 7.7 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.16 (m, 1H), 3.96 (m, 1H), 3.73 (m, 1H), 2.40 (dd, *J* = 14.2, 7.5 Hz, 1H), 2.33 (dd, *J* = 14.3, 4.3 Hz, 1H), 1.75 (s, 3H), 1.74 (s, 3H), 1.20 (d, *J* = 6.2 Hz, 3H), 1.17 (d, *J* = 6.1 Hz, 3H), 1.13 (d, *J* = 6.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 164.8, 148.0, 136.9, 121.8, 119.6, 70.0, 69.5, 56.9, 46.0, 27.8, 27.7, 23.3, 22.5, 20.8. HRMS (EI-TOF) calc. for $C_{15}H_{24}N_2O_2$ (M^+): 264.1838, found: 264.1830.

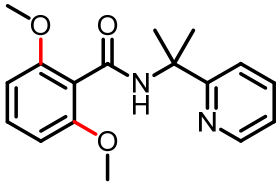
General Procedure for the Alkoxylation of C(sp^2)–H bonds:



n = 0 or 1, *β* or *γ* *Ortho*-C(sp^2)-H

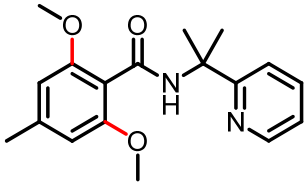
A mixture of substrate (0.2 mmol), $Pd(OAc)_2$ (4.5 mg, 10 mmol%), $PhI(OAc)_2$ (194 mg, 0.6 mmol), alcohol (1.0 mL) and *m*-xylene (1.0 mL) in a 50 mL Schlenk tube (purged with N_2) was heated at 90 °C for 24 hours. The reaction mixture was cooled to RT, and concentrated *in vacuo*. The resulting residue was was purified by flash chromatography to give the desired product.

2,6-Dimethoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)benzamide 40



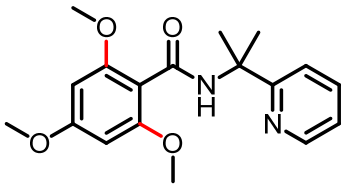
The title compound **40** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **40** was obtained as a yellow solid (47.0 mg, 78%). R_f = 0.50 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 4.3 Hz, 1H), 7.70 (t, *J* = 7.6 Hz, 1H), 7.57 (d, *J* = 8.1 Hz, 1H), 7.49 (s, 1H), 7.26 (t, *J* = 8.4 Hz, 1H), 7.15 (dd, *J* = 6.6, 5.3 Hz, 1H), 6.57 (d, *J* = 8.4 Hz, 2H), 3.82 (s, 6H), 1.87 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.2, 164.8, 157.6, 147.8, 136.9, 130.2, 121.7, 119.7, 117.6, 104.2, 57.7, 56.2, 28.0. HRMS (EI-TOF) calc. for $C_{17}H_{20}N_2O_3$ (M^+): 300.1474, found: 300.1472.

2,6-Dimethoxy-4-methyl-*N*-(2-(pyridin-2-yl)propan-2-yl)benzamide 41



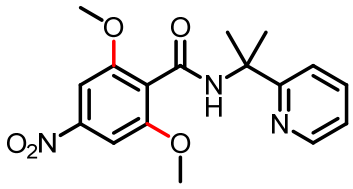
The title compound **41** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 4). **41** was obtained as a yellow solid (60.2 mg, 95%). R_f = 0.33 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (d, *J* = 4.6 Hz, 1H), 7.68 (t, *J* = 7.7 Hz, 1H), 7.56 (d, *J* = 8.0 Hz, 1H), 7.45 (s, 1H), 7.14 (t, *J* = 6.4Hz, 1H), 6.37 (s, 2H), 3.79 (s, 6H), 2.35 (s, 3H), 1.85 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.4, 164.8, 157.4, 147.8, 140.6, 136.9, 121.6, 119.7, 114.8, 104.9, 57.6, 56.1, 28.0, 22.3. HRMS (EI-TOF) calc. for $C_{18}H_{22}N_2O_3$ (M^+): 314.1630, found: 314.1631.

2,4,6-Trimethoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)benzamide 42



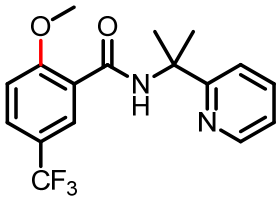
The title compound **42** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **42** was obtained as a colorless liquid (65.7 mg, 99%). R_f = 0.21 (2/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (d, *J* = 4.4 Hz, 1H), 7.67 (td, *J* = 8.0, 1.6 Hz, 1H), 7.54 (d, *J* = 8.1 Hz, 1H), 7.44 (s, 1H), 7.12 (dd, *J* = 6.7, 5.2 Hz, 1H), 6.10 (s, 2H), 3.80 (s, 3H), 3.78 (s, 6H), 1.83 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.1, 164.8, 161.7, 158.5, 147.8, 136.9, 121.6, 119.7, 110.6, 90.6, 57.5, 56.0, 55.5, 27.9. HRMS (EI-TOF) calc. for $C_{18}H_{22}N_2O_4$ (M^+): 330.1580, found: 330.1582.

2,6-Dimethoxy-4-nitro-*N*-(2-(pyridin-2-yl)propan-2-yl)benzamide 43



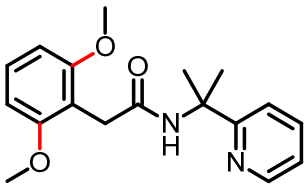
The title compound **43** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 1). **43** was obtained as a light yellow solid (36.6 mg, 53%). R_f = 0.49 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (d, *J* = 4.7 Hz, 1H), 7.95 (s, 1H), 7.72 (t, *J* = 7.8 Hz, 1H), 7.50 (d, *J* = 8.1 Hz, 1H), 7.45 (s, 2H), 7.18 (t, *J* = 6.4 Hz, 1H), 3.89 (s, 6H), 1.88 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 163.0, 157.6, 149.4, 147.6, 137.3, 122.8, 122.0, 119.6, 99.9, 57.7, 56.6, 27.7. HRMS (EI-TOF) calc. for $C_{17}H_{19}N_3O_5$ (M^+): 345.1325, found: 345.1328.

2-Methoxy-*N*-(2-(pyridin-2-yl)propan-2-yl)-5-(trifluoromethyl)benzamide 44



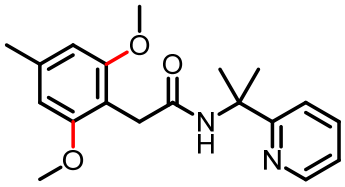
The title compound **44** was prepared according to the general procedure with 1.5 equiv of $PhI(OAc)_2$ and was purified by flash chromatography (petroleum ether : ethyl acetate = 3 : 1). **44** was obtained as a white solid (47.8 mg, 71%). R_f = 0.55 (3/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 8.58 (d, *J* = 4.3 Hz, 1H), 8.50 (s, 1H), 7.74 (t, *J* = 7.7 Hz, 1H), 7.66 (d, *J* = 8.5 Hz, 1H), 7.47 (d, *J* = 8.0 Hz, 1H), 7.21 (m, 1H), 7.05 (d, *J* = 8.6 Hz, 1H), 4.08 (s, 3H), 1.87 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.8, 162.7, 159.9, 147.9, 137.3, 129.7 (q, J_{C-F} = 3.8 Hz), 129.4 (q, J_{C-F} = 3.6 Hz), 124.1 (q, J_{C-F} = 274.3 Hz), 123.6 (q, J_{C-F} = 33.5 Hz), 123.3, 122.0, 119.8, 111.6, 57.6, 56.4, 27.4. HRMS (EI-TOF) calc. for $C_{17}H_{17}F_3N_2O_2$ (M^+): 338.1242, found: 388.1250.

2-(2,6-Dimethoxyphenyl)-*N*-(2-(pyridin-2-yl)propan-2-yl)acetamide 45



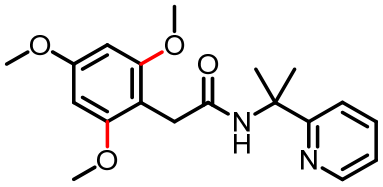
The title compound **45** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ether = 1 : 8). **45** was obtained as a colorless liquid (42.9 mg, 76%). R_f = 0.46 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, *J* = 4.5 Hz, 1H), 7.62 (t, *J* = 8.3 Hz, 1H), 7.58 (s, 1H), 7.33 (d, *J* = 8.1 Hz, 1H), 7.22 (t, *J* = 8.3 Hz, 1H), 7.12 (dd, *J* = 7.6, 5.2 Hz, 1H), 3.84 (s, 6H), 3.66 (s, 2H), 1.68 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9, 165.1, 158.6, 147.7, 136.9, 128.3, 119.5, 112.9, 104.0, 56.6, 56.0, 32.6, 27.7. HRMS (EI-TOF) calc. for $C_{18}H_{22}N_2O_3$ (M^+): 314.1630, found: 314.1641.

2-(2,6-Dimethoxy-4-methylphenyl)-*N*-(2-(pyridin-2-yl)propan-2-yl)acetamide 46



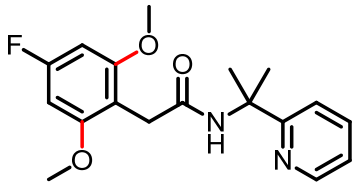
The title compound **46** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ether = 1 : 4). **46** was obtained as a yellow solid (51.8 mg, 87%). R_f = 0.50 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.41 (d, *J* = 4.2 Hz, 1H), 7.63 (t, *J* = 7.5 Hz, 1H), 7.51 (s, 1H), 7.33 (d, *J* = 8.0 Hz, 1H), 7.11 (m, 1H), 6.41 (s, 2H), 3.82 (s, 6H), 3.61 (s, 2H), 2.36 (s, 3H), 1.67 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 156.2, 160.5, 159.2, 147.8, 136.9, 121.6, 119.5, 105.4, 90.8, 56.6, 55.9, 55.5, 32.3, 27.8. HRMS (EI-TOF) calc. for $C_{19}H_{24}N_2O_3$ (M^+): 328.1787, found: 328.1791.

_N_-(2-(Pyridin-2-yl)propan-2-yl)-2-(2,4,6-trimethoxyphenyl)acetamide 47



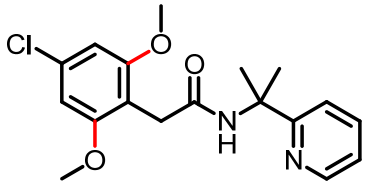
The title compound **47** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ether = 1 : 8). **47** was obtained as a light yellow liquid (43.5 mg, 70%). R_f = 0.50 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (d, *J* = 4.2 Hz, 1H), 7.65 (t, *J* = 7.7 Hz, 1H), 7.53 (s, 1H), 7.35 (d, *J* = 8.1 Hz, 1H), 7.13 (m, 1H), 6.19 (s, 2H), 3.85 (s, 3H), 3.84(s, 6H) 3.58 (s, 2H), 1.69 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 165.2, 160.5, 159.2, 147.8, 136.9, 121.6, 119.5, 105.4, 90.8, 56.6, 55.9, 55.5, 32.3, 27.8. HRMS (EI-TOF) calc. for $C_{19}H_{24}N_2O_4$ (M^+): 344.1736, found: 344.1732.

2-(4-Fluoro-2,6-dimethoxyphenyl)-_N_-(2-(pyridin-2-yl)propan-2-yl)acetamide 48



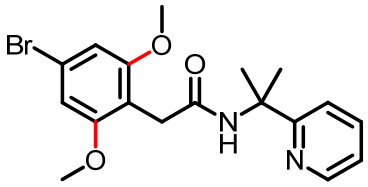
The title compound **48** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **48** was obtained as a yellow solid (37.4 mg, 59%). R_f = 0.41 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, *J* = 3.8 Hz, 1H), 7.71 (s, 1H), 7.65 (t, *J* = 7.5 Hz, 1H), 7.33 (d, *J* = 8.1 Hz, 1H), 7.13 (m, 1H), 6.32 (d, *J* = 10.8 Hz, 2H), 3.80 (s, 6H), 3.59 (s, 2H), 1.68 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 165.0, 163.5 (d, J_{C-F} = 242.4 Hz), 159.2 (d, J_{C-F} = 12.9 Hz), 147.7, 137.0, 121.7, 119.5, 108.4 (d, J_{C-F} = 4.0 Hz), 92.1 (d, J_{C-F} = 26.6 Hz), 56.5, 56.0, 32.2, 27.6. HRMS (EI-TOF) calc. for $C_{18}H_{21}FN_2O_3$ (M^+): 332.1536, found: 332.1535.

2-(4-Chloro-2,6-dimethoxyphenyl)-*N*-(2-(pyridin-2-yl)propan-2-yl)acetamide 49



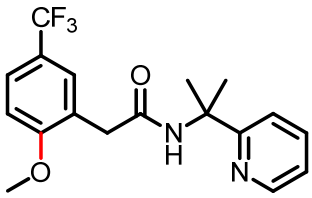
The title compound **49** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **49** was obtained as a light yellow solid (36.7 mg, 53%). R_f = 0.57 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.41 (d, *J* = 4.2 Hz, 1H), 7.75 (s, 1H), 7.65 (t, *J* = 7.2 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.17 – 7.10 (m, 1H), 6.59 (s, 2H), 3.82 (s, 6H), 3.61 (s, 2H), 1.69 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 164.9, 158.8, 147.6, 137.0, 133.8, 121.7, 119.5, 111.4, 104.8, 56.5, 56.1, 32.3, 27.6. HRMS (EI-TOF) calc. for $C_{18}H_{21}ClN_2O_3$ (M^+): 348.1241, found: 348.1243.

2-(4-Bromo-2,6-dimethoxyphenyl)-*N*-(2-(pyridin-2-yl)propan-2-yl)acetamide 50



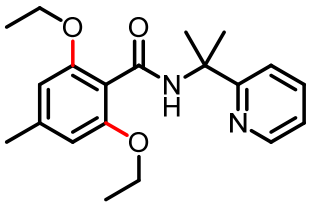
The title compound **50** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **50** was obtained as a yellow solid (39.9 mg, 51%). R_f = 0.40 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, *J* = 4.1 Hz, 1H), 7.75 (s, 1H), 7.65 (t, *J* = 7.0 Hz, 1H), 7.33 (d, *J* = 8.0 Hz, 1H), 7.13 (dd, *J* = 7.1, 5.1 Hz, 1H), 6.73 (s, 2H), 3.81 (s, 6H), 3.59 (s, 2H), 1.68 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 165.0, 158.9, 147.6, 137.0, 121.7, 121.4, 119.5, 112.0, 107.8, 56.5, 56.2, 32.4, 27.6. HRMS (EI-TOF) calc. for $C_{18}H_{21}BrN_2O_3$ (M^+): 392.0736, found: 392.0742.

2-(2-Methoxy-5-(trifluoromethyl)phenyl)-*N*-(2-(pyridin-2-yl)propan-2-yl)acetamide 51



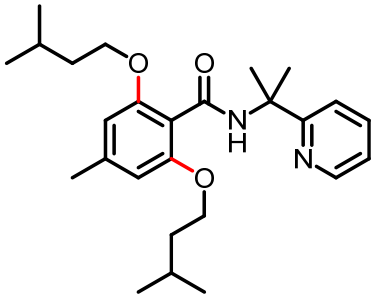
The title compound **51** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **51** was obtained as a yellow solid (68.1 mg, 97%). R_f = 0.64 (1/ 2 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (d, *J* = 4.3 Hz, 1H), 8.00 (s, 1H), 7.67 (t, *J* = 7.6 Hz, 1H), 7.54 (s, 1H), 7.52 (s, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.15 (m, 1H), 6.94 (d, *J* = 8.5 Hz, 1H), 3.90 (s, 3H), 3.62 (s, 2H), 1.71 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.6, 164.7, 160.0, 147.6, 137.2, 128.3 (q, J_{C-F} = 3.4 Hz), 126.0 (q, J_{C-F} = 4.0 Hz), 125.4, 124.5 (q, J_{C-F} = 270.8 Hz), 123.0 (q, J_{C-F} = 32.7 Hz), 121.9, 119.5, 110.3, 56.6, 55.8, 39.9, 29.8, 27.5. HRMS (EI-TOF) calc. for $C_{18}H_{19}F_3N_2O_2$ (M^+): 352.1399, found: 352.1406.

2,6-Diethoxy-4-methyl-*N*-(2-(pyridin-2-yl)propan-2-yl)benzamide 52



The title compound **52** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 1). **52** was obtained as a white solid (55.7 mg, 81%). R_f = 0.61 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 4.5 Hz, 1H), 7.65 (d, *J* = 3.6 Hz, 2H), 7.12 (dd, *J* = 8.7, 4.3 Hz, 1H), 7.08 (s, 1H), 6.34 (s, 2H), 4.02 (q, *J* = 6.9 Hz, 4H), 2.31 (s, 3H), 1.82 (s, 6H), 1.37 (t, *J* = 6.9 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.3, 164,9, 156.7, 147.9, 136.6, 119.8, 115.5, 106.1, 64.4, 57.7, 28.1, 22.2, 14.9. HRMS (EI-TOF) calc. for $C_{20}H_{26}N_2O_3$ (M^+): 342.1943, found: 342.1945.

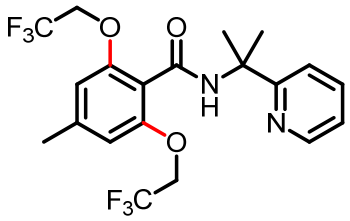
2,6-Bis(isopentyloxy)-4-methyl-*N*-(2-(pyridin-2-yl)propan-2-yl)benzamide 53



The title compound **53** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 1). **53** was obtained as a white solid (78.4 mg, 92%). R_f = 0.57 (3/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (d, *J* = 4.0 Hz, 1H), 7.67 (t, *J* = 7.3 Hz, 1H), 7.60 (d, *J* = 8.0 Hz, 1H), 7.25 (d, *J* = 11.1 Hz, 2H), 7.14 (m, 1H), 6.36 (s, 2H), 3.99 (t, *J* = 6.4 Hz, 4H), 2.33 (s, 3H), 1.82 (s, 9H), 1.79 (m, 1H), 1.64 (dd, *J* = 13.1, 6.5 Hz, 4H), 0.88 (d, *J* = 6.6 Hz, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.1, 165.0,

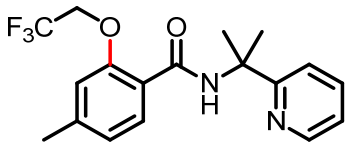
157.0, 147.9, 140.4, 136.7, 121.5, 119.8, 115.5, 105.8, 67.0, 57.6, 38.2, 28.0, 24.9, 22.7, 22.3. HRMS (EI-TOF) calc. for $C_{26}H_{38}N_2O_3$ (M^+): 426.2882, found: 426.2879.

4-Methyl-*N*-(2-(pyridin-2-yl)propan-2-yl)-2,6-bis(2,2,2-trifluoroethoxy)benzamide 54



The title compound **54** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 2 : 1). **54** was obtained as a light yellow solid (42.1 mg, 47%). R_f = 0.41 (3/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (d, J = 4.3 Hz, 1H), 7.68 (t, J = 7.7 Hz, 1H), 7.53 (d, J = 8.0 Hz, 1H), 7.46 (s, 1H), 7.14 (m, 1H), 6.48 (s, 2H), 4.37 (q, J = 8.0 Hz, 4H), 2.34 (s, 3H), 1.81 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.3, 163.0, 155.3, 147.8, 141.5, 137.0, 123.3 (q, $J_{C\text{-}F}$ = 277.3 Hz), 121.7, 119.7, 117.7, 109.5, 67.3 (q, $J_{C\text{-}F}$ = 35.3 Hz), 57.8, 27.7, 22.0. HRMS (EI-TOF) calc. for $C_{20}H_{20}F_6N_2O_3$ (M^+): 450.1378, found: 450.1381.

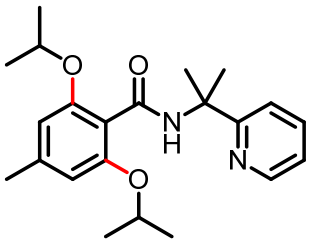
4-Methyl-*N*-(2-(pyridin-2-yl)propan-2-yl)-2-(2,2,2-trifluoroethoxy)benzamide 54a



The title compound **54a** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 2 : 1). **54a** was obtained as a colorless oil (21.7 mg, 26%). R_f = 0.44 (3/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (s, 1H), 8.54 (t, J = 4.0 Hz), 7.99 (d, J

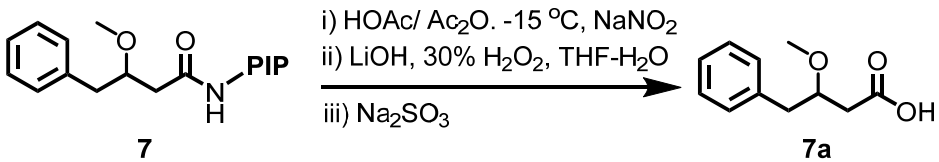
= 8.0 Hz, 1H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.45 (d, *J* = 8.1 Hz, 1H), 7.14 (m, 1H), 6.97 (d, *J* = 7.9 Hz, 1H), 6.72 (s, 1H), 4.53 (q, *J* = 8.0 Hz, 2H), 2.39 (s, 3H), 1.82 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.0, 163.7, 155.0, 148.4, 143.5, 136.7, 132.6, 124.1, 123.4 (q, $J_{C\text{-}F}$ = 273.2 Hz), 121.6, 119.5, 113.7, 66.8 (q, $J_{C\text{-}F}$ = 36.4 Hz), 57.6, 29.8, 28.1, 21.7. HRMS (EI-TOF) calc. for $C_{18}H_{19}F_3N_2O_2$ (M^+): 352.1399, found: 352.1395.

2,6-Diisopropoxy-4-methyl-*N*-(2-(pyridin-2-yl)propan-2-yl)benzamide 55



The title compound **55** was prepared according to the general procedure and was purified by flash chromatography (petroleum ether : ethyl acetate = 2 : 1). **55** was obtained as a yellow solid (50.0 mg, 68%). R_f = 0.47 (2/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 4.4 Hz, 1H), 7.65 (d, *J* = 3.7 Hz, 2H), 7.13 (dd, *J* = 8.6, 4.3 Hz, 1H), 7.00 (s, 1H), 6.36 (s, 2H), 4.53 (m, 2H), 2.31 (s, 3H), 1.81 (s, 6H), 1.31 (d, *J* = 6.0 Hz, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.4, 165.0, 156.0, 148.0, 140.1, 136.6, 121.5, 119.9, 118.0, 107.9, 71.3, 57.6, 28.1, 22.4, 22.3. HRMS (EI-TOF) calc. for $C_{22}H_{30}N_2O_3$ (M^+): 370.2256, found: 370.2265.

Removal of Directing Group:

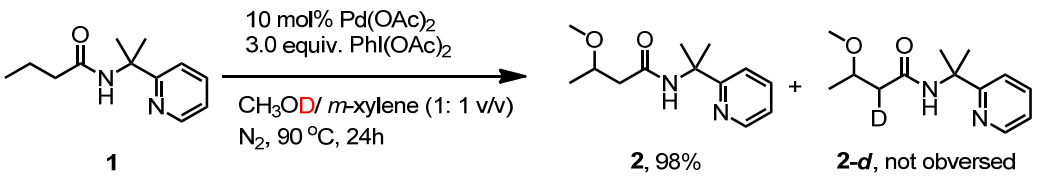


7 **7a**

A solution of substrate **7** (118 mg, 0.37 mmol) in a mixture of acetic acid (0.5 mL) and acetic anhydride (2.5 mL) was cooled to -15 °C and 555 mg of granular sodium

nitrite (22 equiv) was added slowly in portions. After being stirred for 37 hours at -15 ^{o}C and the mixture poured into a mixture of ice and water. (Caution! The nitrosoamide is unstable and the subsequent work-up should be carried out at 0 ^{o}C) The nitrosoamide was extracted with cold ether, and the organic phase was washed with ice water, with an aqueous solution of sodium carbonate (5%), with ice water, and then dried with anhydrous sodium sulfate under ice bath. The solvent was removed under reduce pressure under ice bath. The resident was dissolved in THF (6 mL)/ H_2O (2 mL) and cooled to -15 ^{o}C. Then 30% H_2O_2 (0.85 mL) was added followed by lithium hydroxide monohydrate (155 mg, 3.7 mmol). The mixture was stirred at -15 ^{o}C for 2 hours and at 0 ^{o}C for another 2 hours, and then quenched with an aqueous solution of Na_2SO_3. The mixture was basified with 1N NaOH and washed with EtOAc. The aqueous phase was acidied with 1M HCl and extracted with ether. The organic layer was washed with brine, dried over anhydrous sodium sulfate and concentrated *in vacuo*. The resulting residue was purified by flash chromatography (petroleum ether : ethyl acetate : acetic acid = 2 : 1 : 0.01). **7a** was obtained as a colorless liquid (62.4 mg, 87%). R_f = 0.55 (1/ 1 petroleum ether/ ethyl acetate). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (m, 2H), 7.21 (m, 3H), 3.86 (m, 1H), 3.39 (s, 3H), 2.97 (dd, *J* = 13.7, 5.9 Hz, 1H), 2.77 (dd, *J* = 13.7, 6.9 Hz, 1H), 2.49 (d, *J* = 6.3 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.1, 137.7, 129.6, 128.6, 126.7, 78.9, 57.6, 39.9, 38.9. HRMS (EI-TOF) calc. for $C_{11}H_{14}O_3$ (M^+): 194.0943, found:194.0950.

Mechanistic Investigation



A mixture of substrate **1** (41.2 mg, 0.2 mmol), $Pd(OAc)_2$ (4.5 mg, 10 mmol%) and $PhI(OAc)_2$ (194 mg, 0.6 mmol), MeOD (1.0 mL) and *m*-xylene (1.0 mL) in a 50 mL Schlenk tube (purged with N_2) was heated at 90 ^{o}C for 24 hours. The reaction mixture

was cooled to RT, and concentrated *in vacuo*. The resulting residue was purified by flash chromatography (petroleum ether : ethyl acetate = 1 : 2). **2** was obtained as a colorless liquid (46.3 mg, 98%). **2-*d*** was not obversed.

Supporting Information

NMR Spectra

Pd(II)-Catalyzed Alkoxylation of Unactivated C(sp^3)–H and C(sp^2)–H Bonds Using a Removable Directing Group: Efficient Synthesis of Alkyl Ethers

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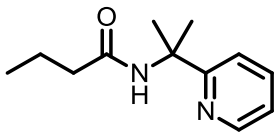
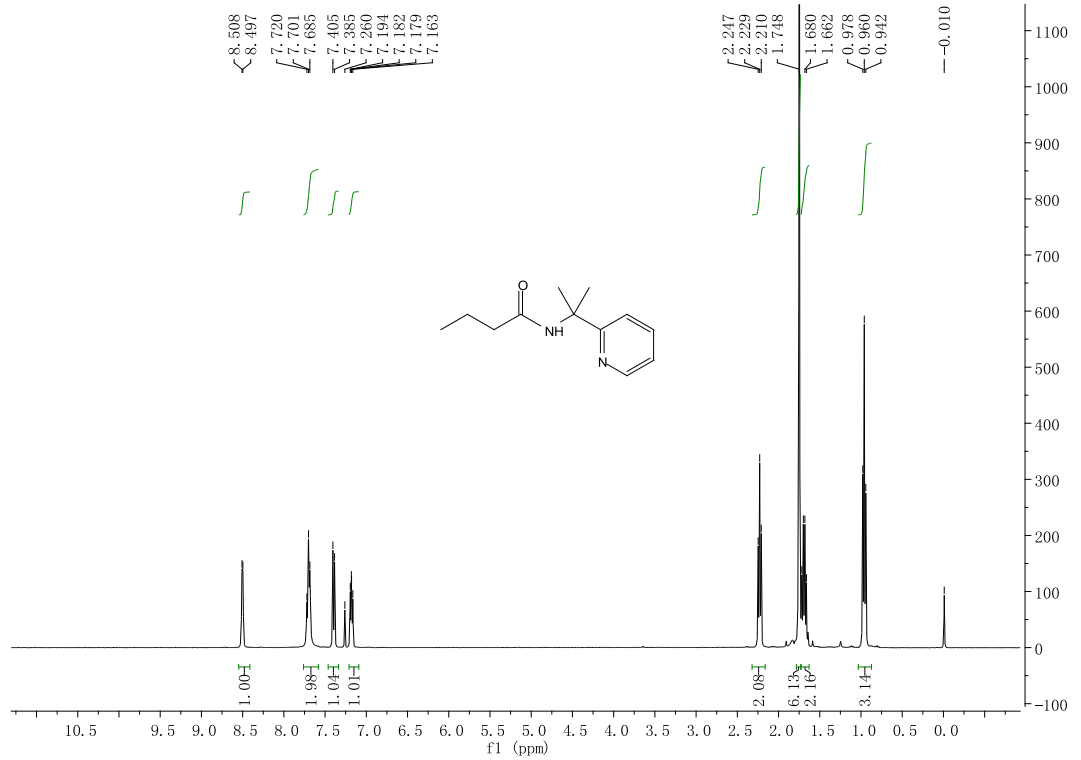
[b]State Key Laboratory of Bioorganic and Natural Products Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, China

*To whom correspondence should be addressed. Email: bfshi@zju.edu.cn

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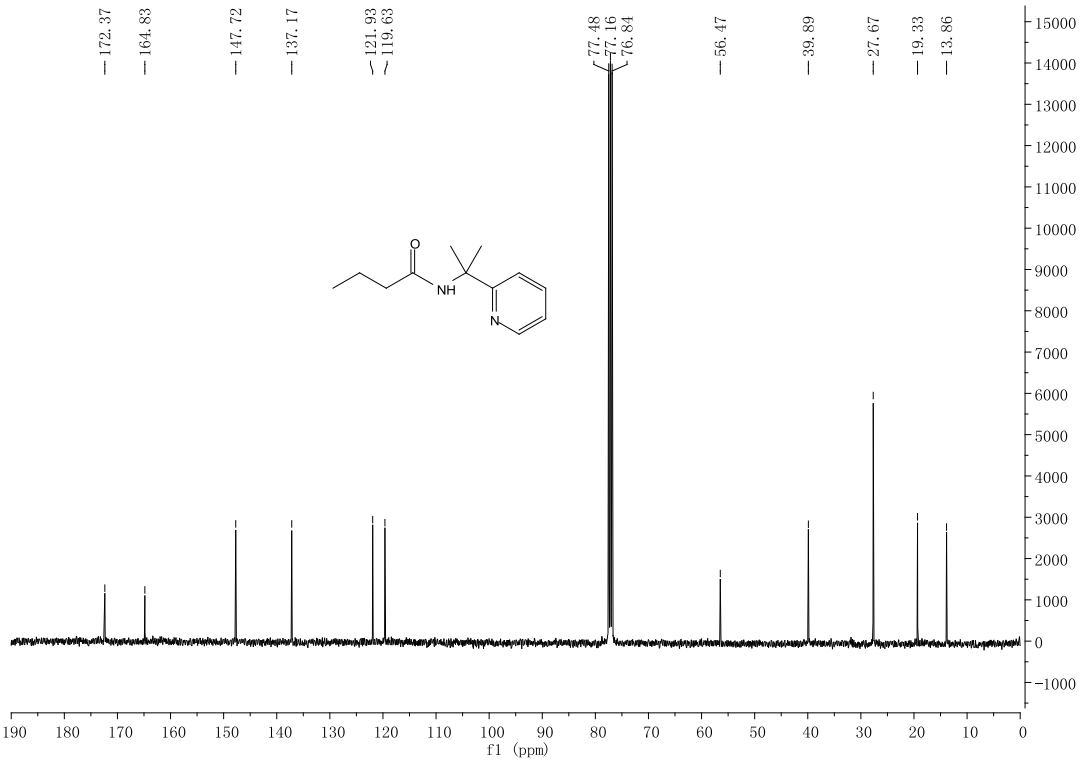
8.497
7.720
7.701
7.685
7.405
7.385
7.260
7.194
7.182
7.179
7.163
2.247
2.229
2.210
1.748
1.680
1.662
0.978
0.960
0.942
-0.010
1.00
1.98
1.04
1.01
2.08
6.13
2.16
3.14
f1 (ppm)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164.83
147.72
137.17
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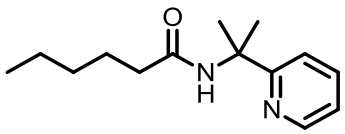
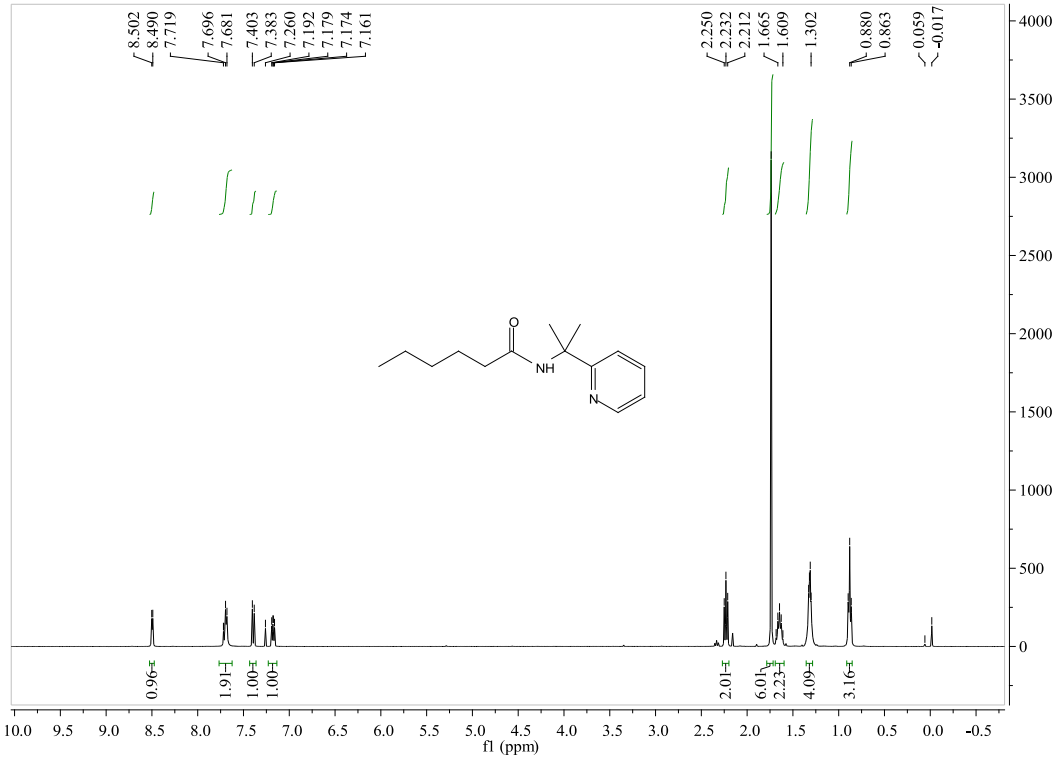
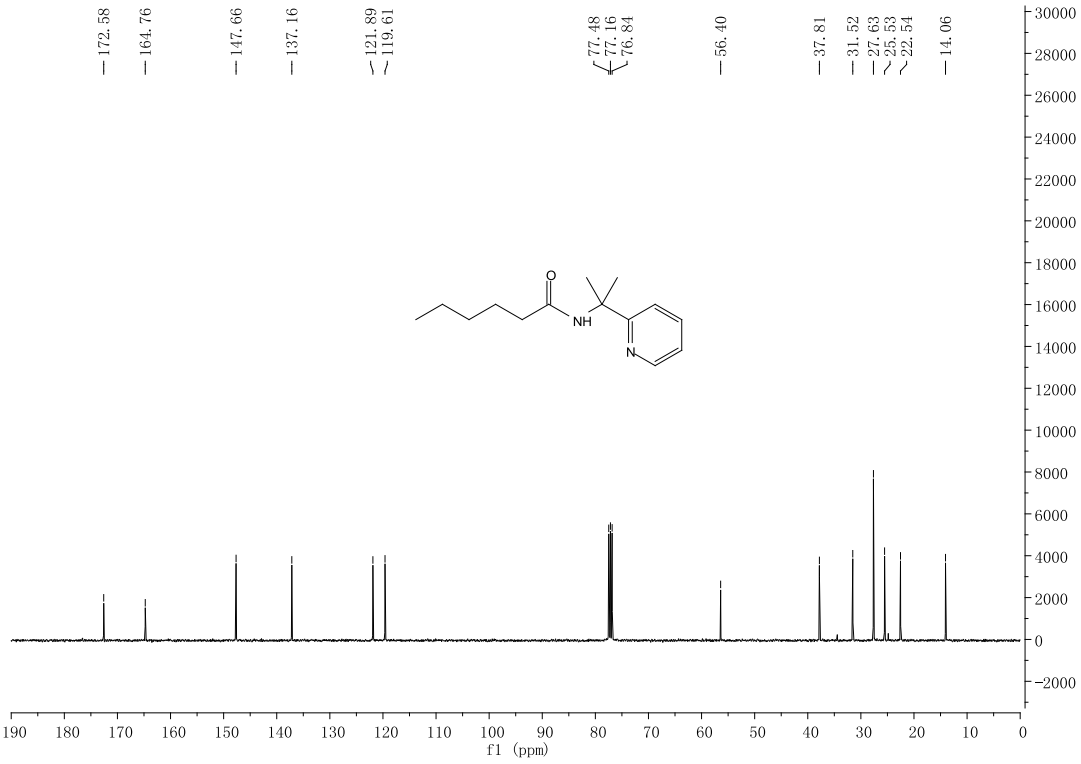
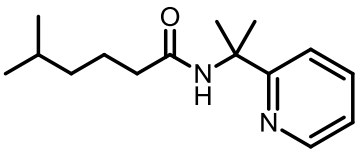
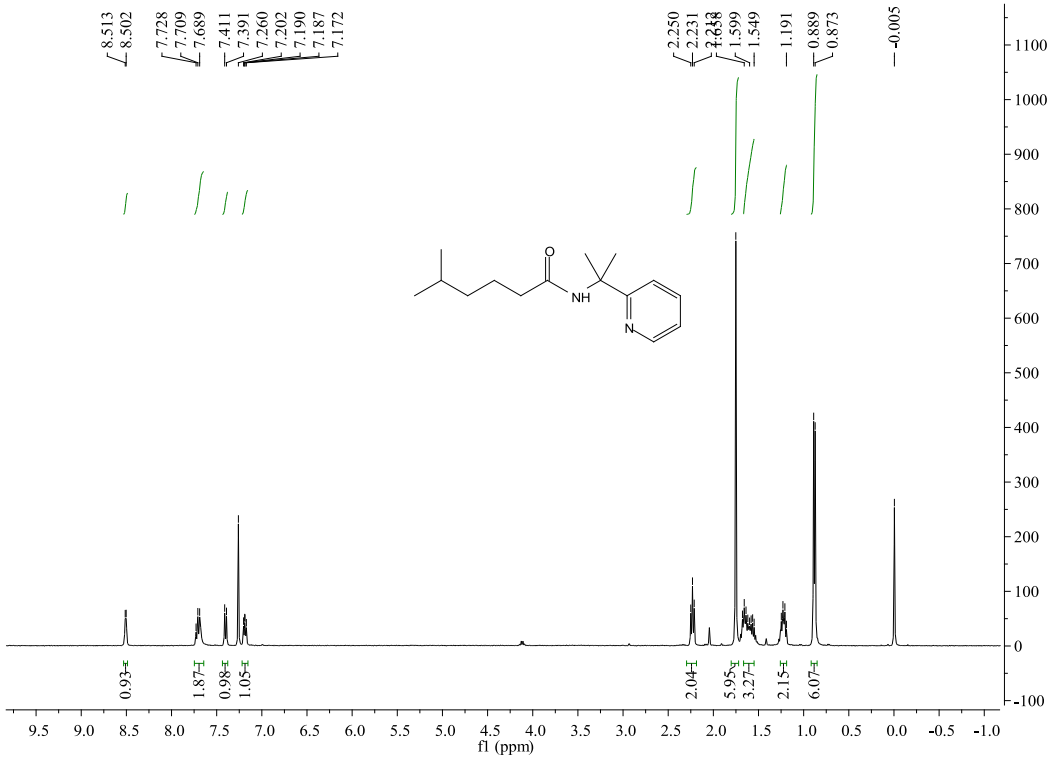
77.16
76.84
56.47
39.89
27.67
19.33
13.86
f1 (ppm)




8.502
8.490
7.719
7.696
7.681
7.403
7.383
7.260
7.192
7.179
7.174
7.161
2.250
2.232
2.212
1.665
1.609
1.302
0.880
0.863
0.059
-0.017
0.96
1.91
1.00
1.00
2.01
6.01
2.23
4.09
3.16
f1 (ppm)


172.58
164.76
147.66
137.16
121.89
119.61
77.48
77.16
76.84
56.40
37.81
31.52
27.63
25.53
22.54
14.06
f1 (ppm)

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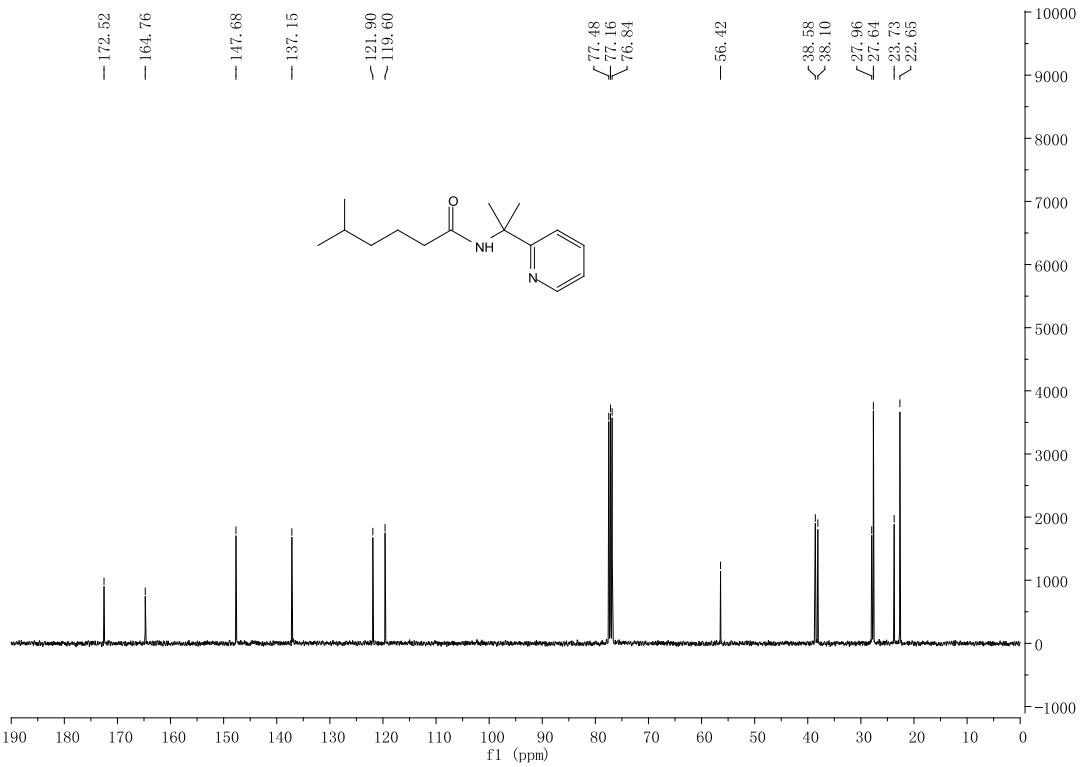
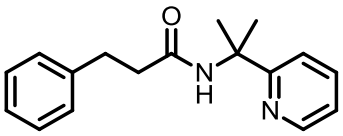
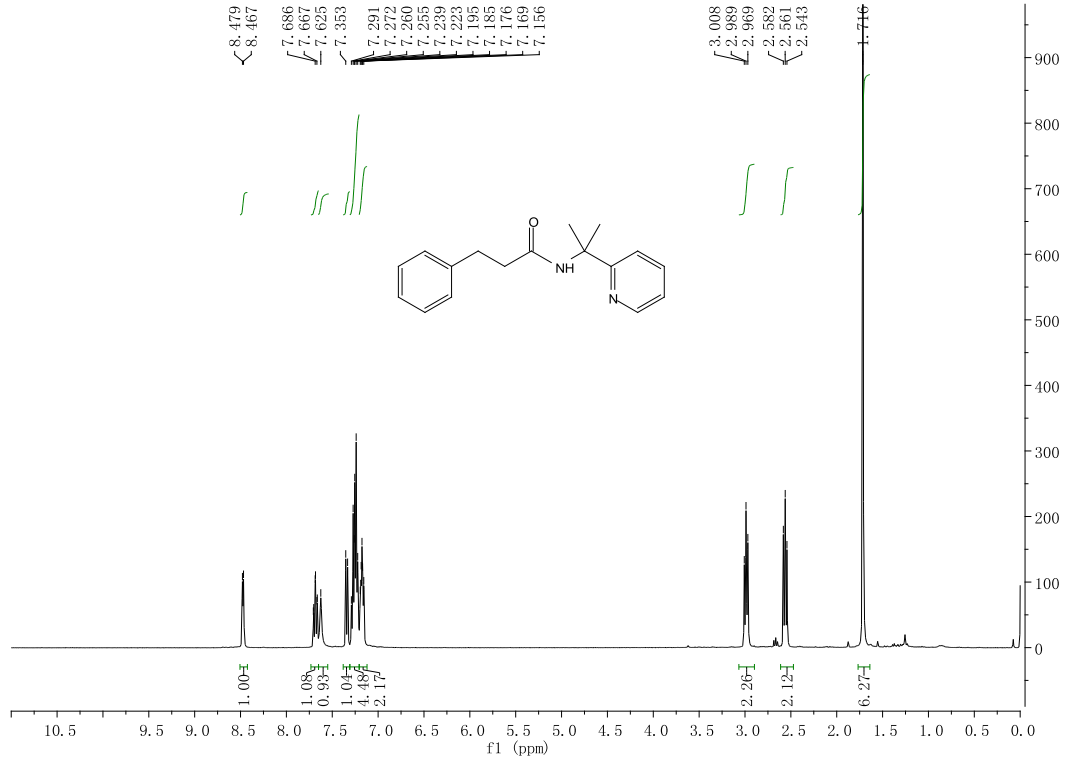
8.502
7.728
7.709
7.689
7.411
7.391
7.260
7.202
7.190
7.187
7.172
2.250
2.231
1.599
1.549
1.191
0.889
0.873
-0.005
0.93
1.87
0.98
1.05
2.04
5.95
3.27
2.15
6.07
f1 (ppm)


172.52
164.76
147.68
137.15
121.90
119.60
77.48
77.16
76.84
56.42
38.58
38.10
27.96
27.64
23.73
22.65
f1 (ppm)

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7.176
7.169
7.156
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2.969
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2.561
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1.04
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2.26
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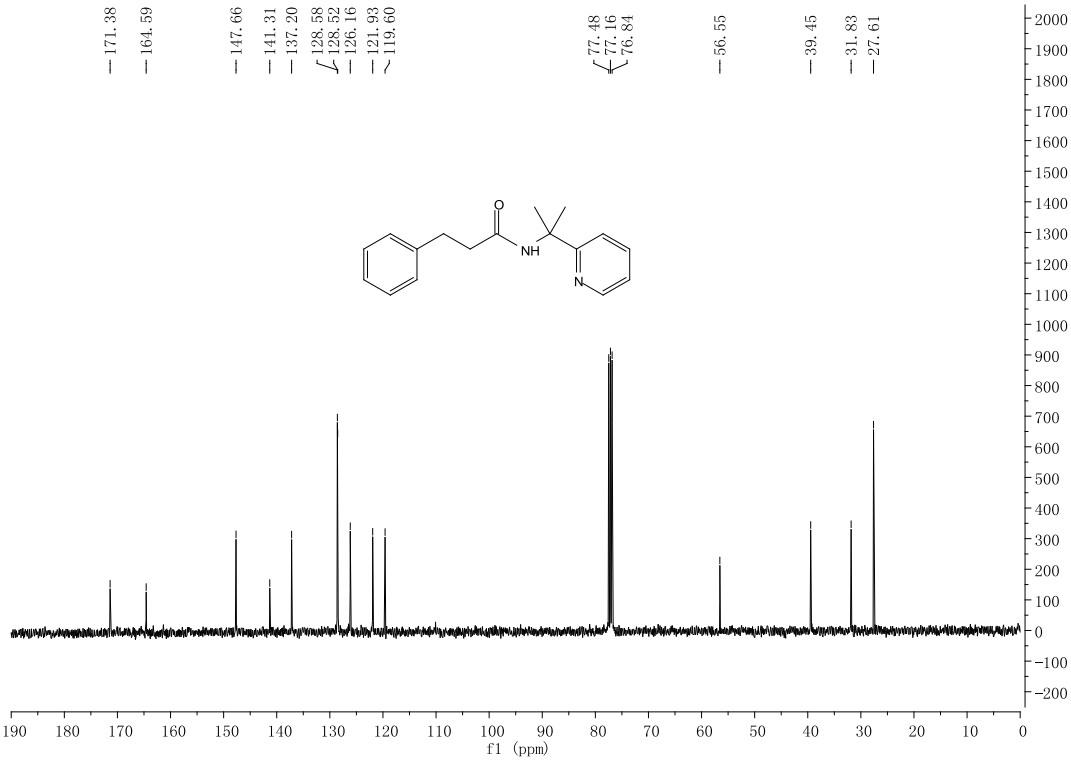
f1 (ppm)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77.16
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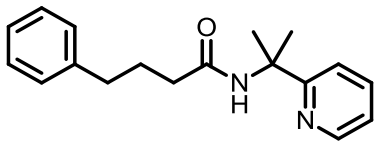
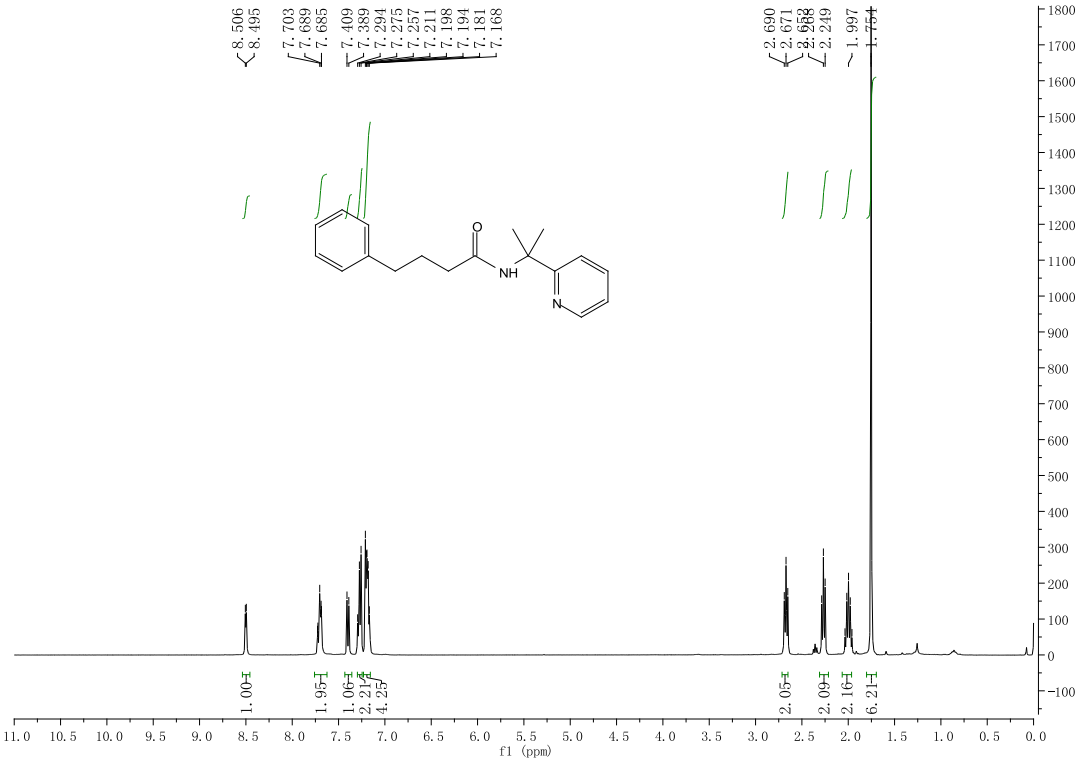
27.61
f1 (ppm)

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7.168
2.690
2.671
2.249
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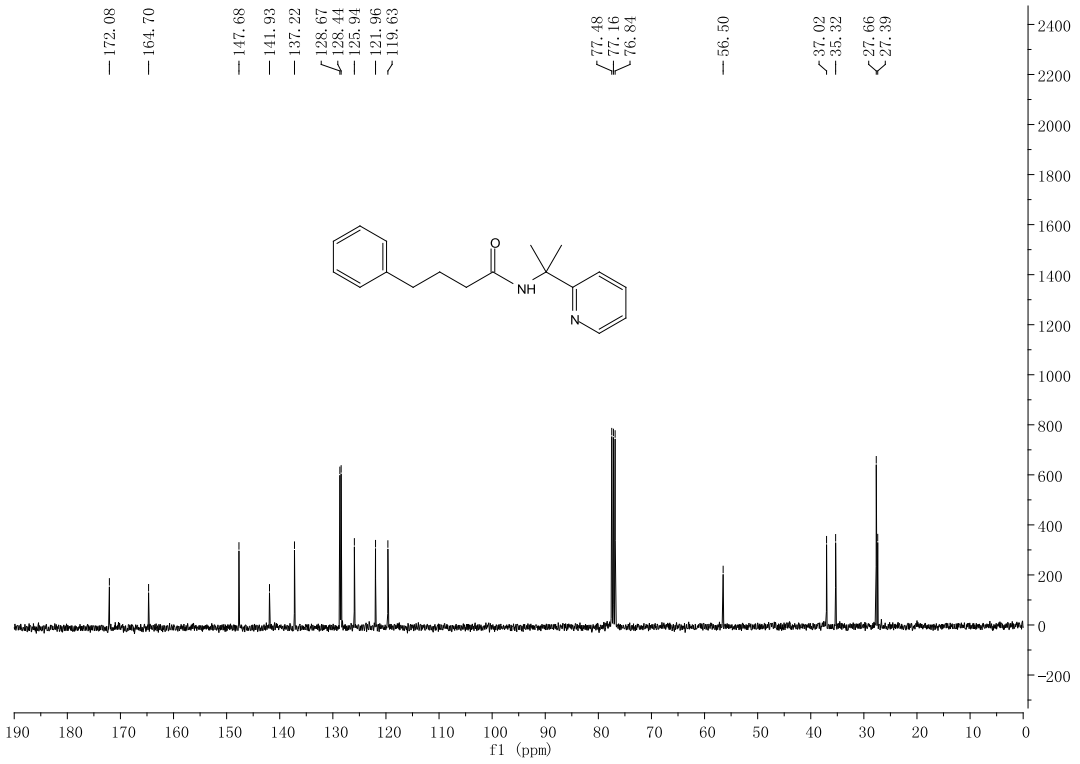
6.21
f1 (ppm)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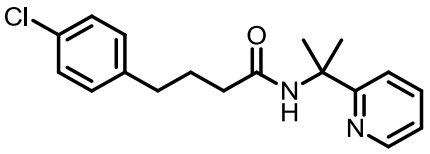
35.32
27.66
27.39
f1 (ppm)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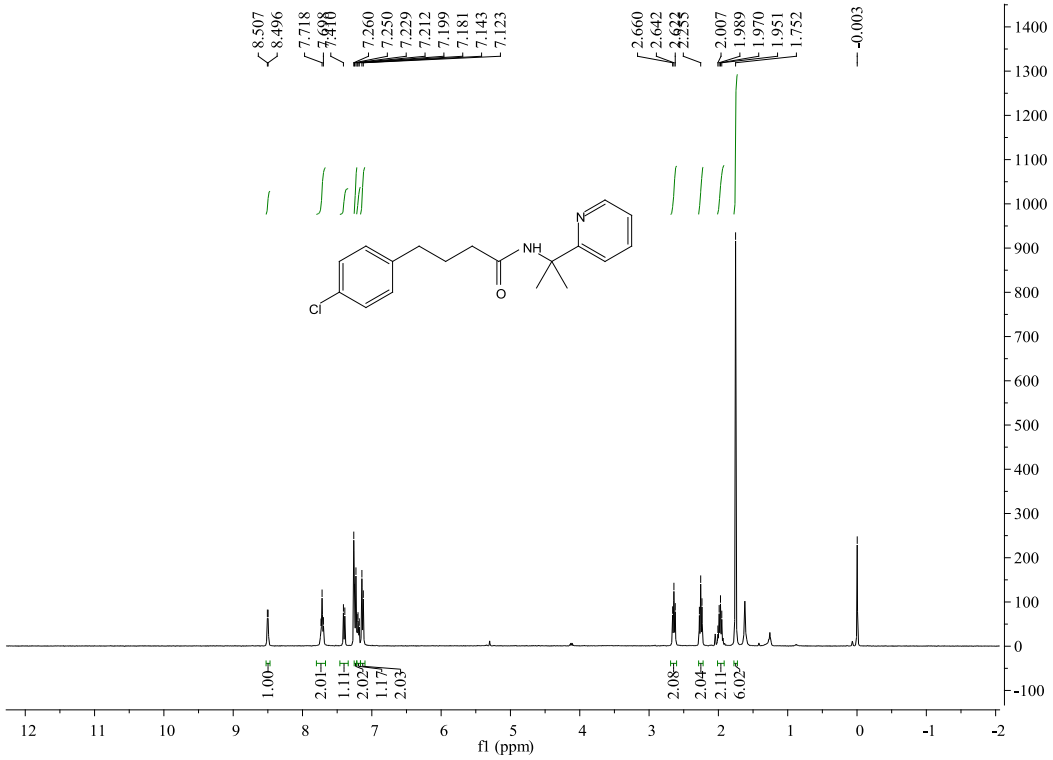
O
N
H
N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1.752
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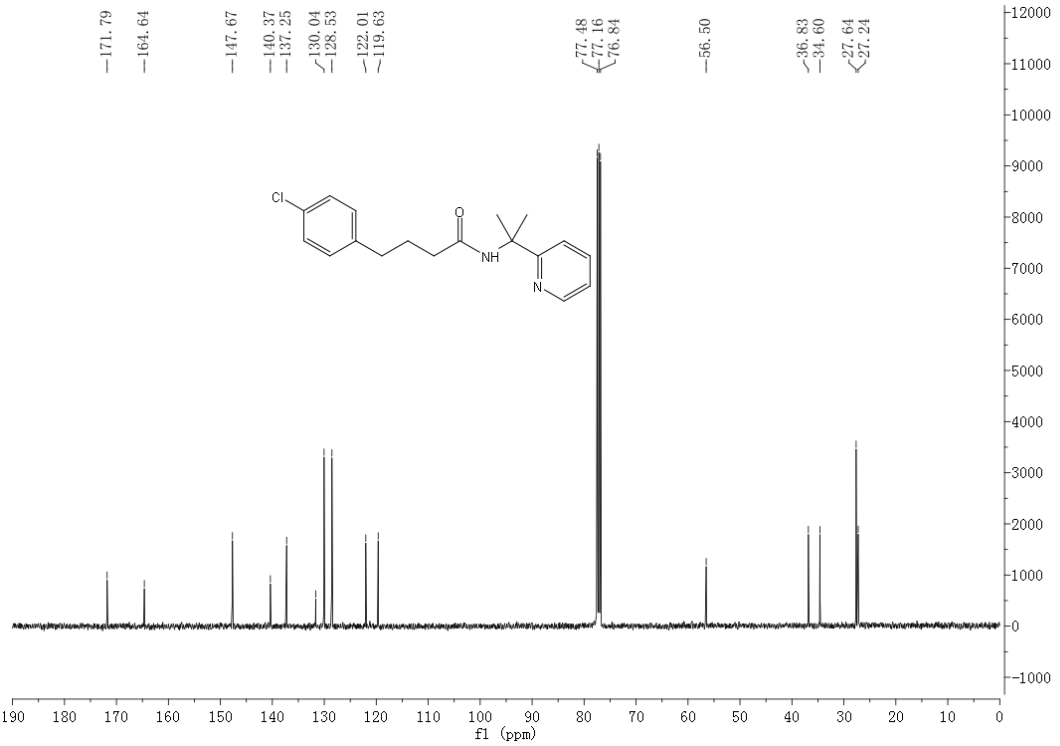
f1 (ppm)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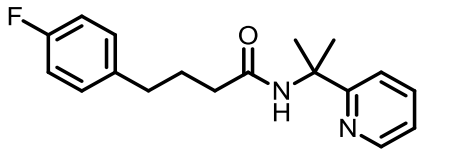
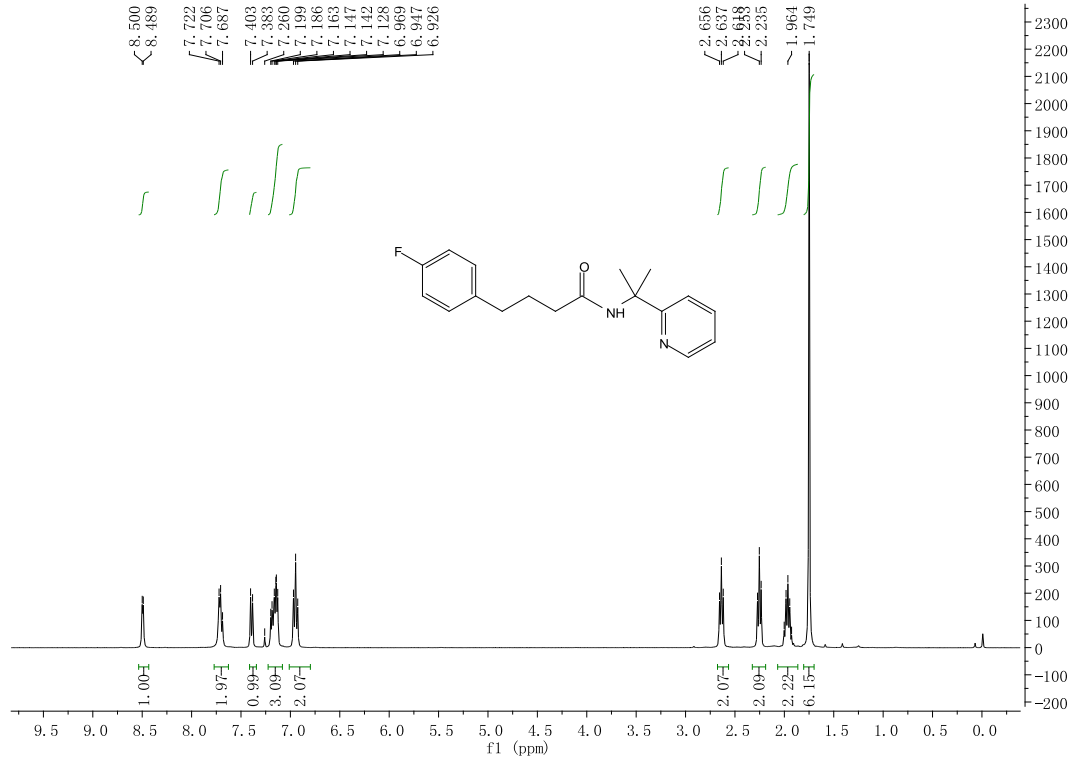
f1 (ppm)


F
O
N
H
N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7.260
7.199
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7.163
7.147
7.142
7.128
6.969
6.947
6.926
2.656
2.637
2.618
2.253
2.235
1.964
1.749
F
O
NH
N
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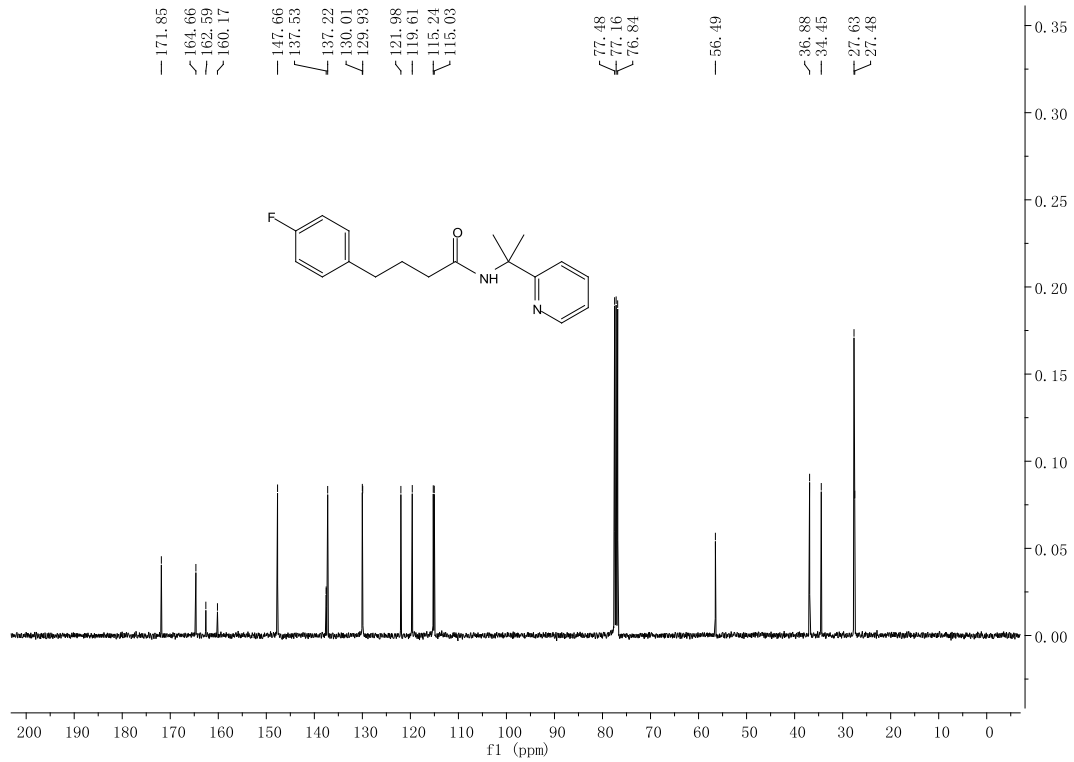
0.99
3.09
2.07
2.07
2.09
2.22
6.15
f1 (ppm)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137.53
137.22
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129.93
121.98
119.61
115.24
115.03
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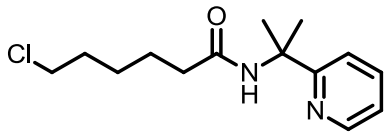
56.49
36.88
34.45
27.63
27.48
F
O
NH
N
f1 (ppm)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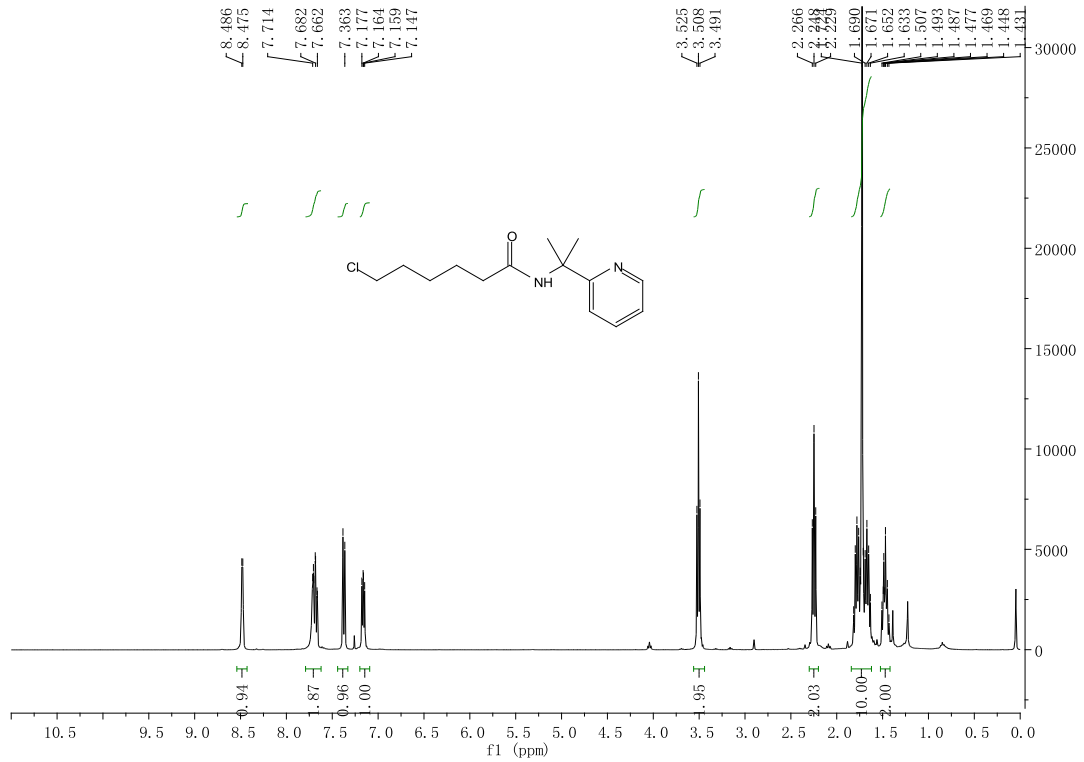
O
N
H
N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2.266
1.690
1.671
1.652
1.633
1.507
1.493
1.487
1.477
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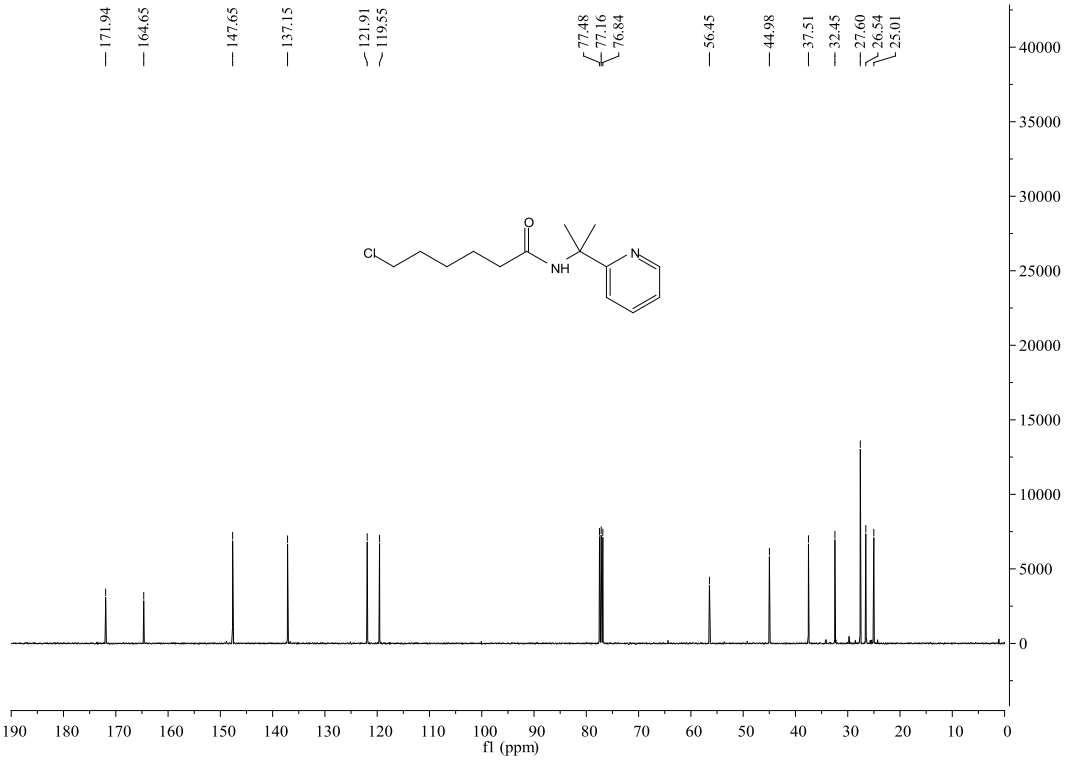
1.87
0.96
1.00
1.95
2.03
10.00
2.00
f1 (ppm)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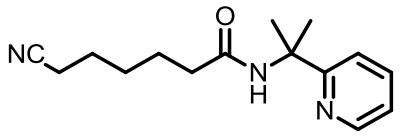
32.45
27.60
26.54
25.01
f1 (ppm)


NC
O
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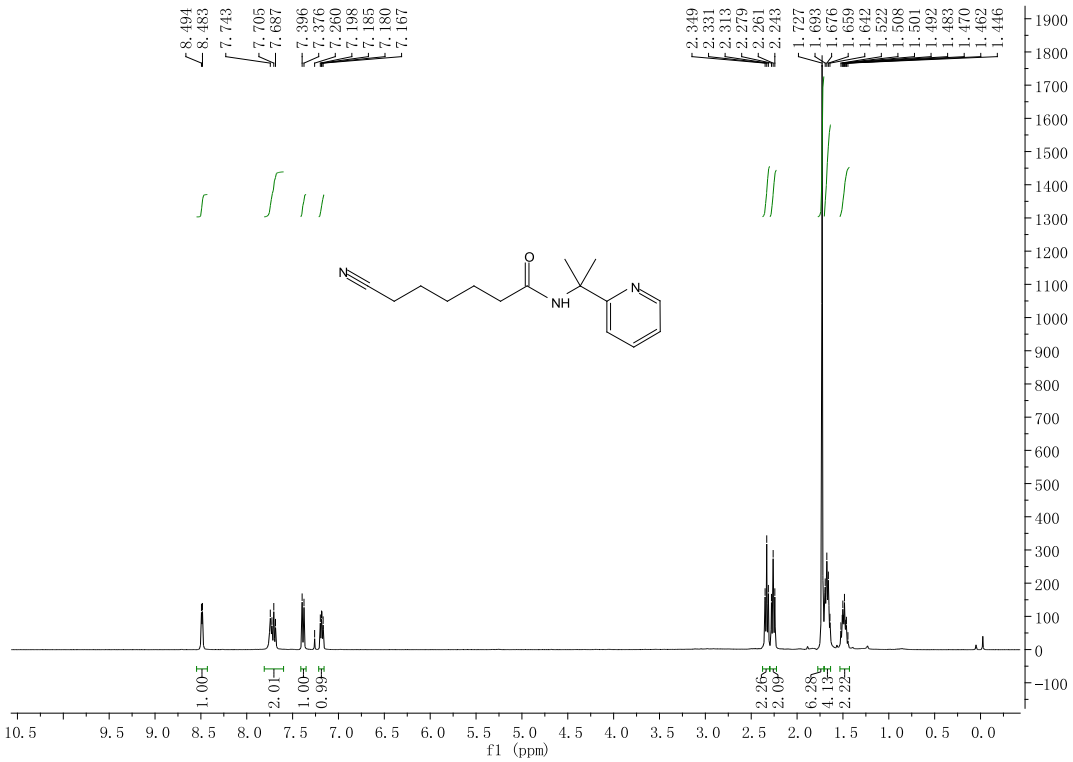
H
N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2.331
2.313
2.279
2.261
2.243
1.727
1.693
1.676
1.659
1.642
1.522
1.508
1.501
1.492
1.483
1.470
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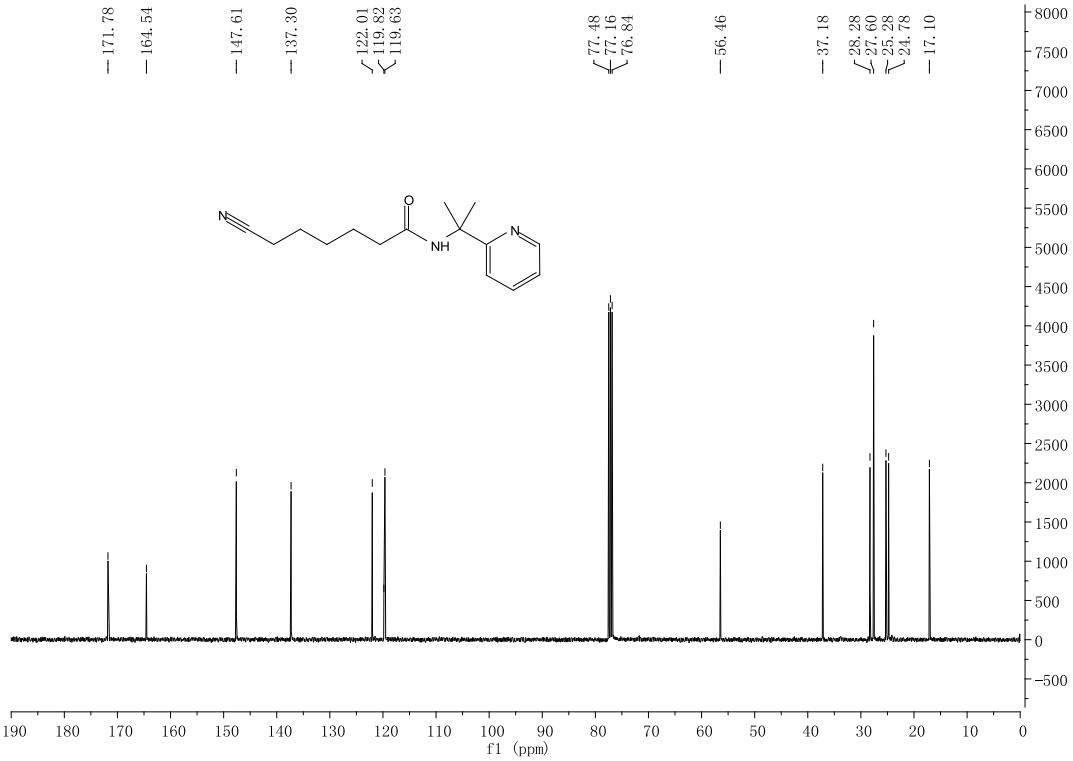
2.09
6.28
4.13
2.22
f1 (ppm)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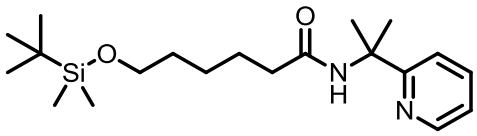
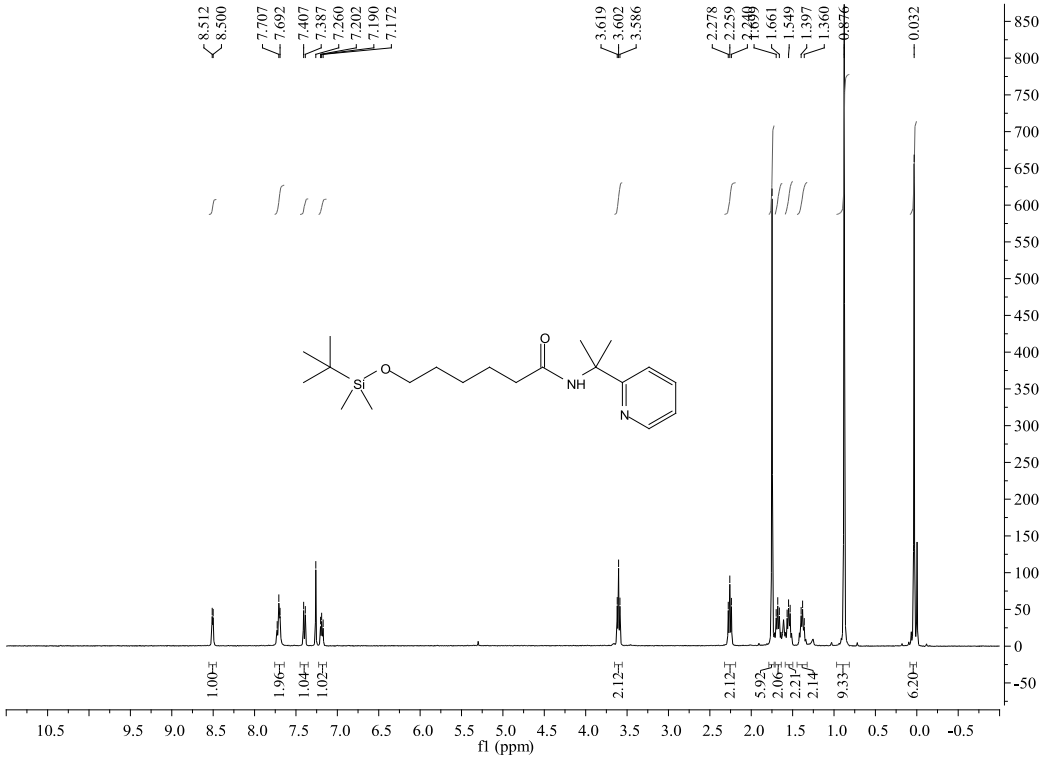
27.60
25.28
24.78
17.10
f1 (ppm)

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3.586
2.278
2.259
1.661
1.549
1.397
1.360
0.876
0.032
1.00
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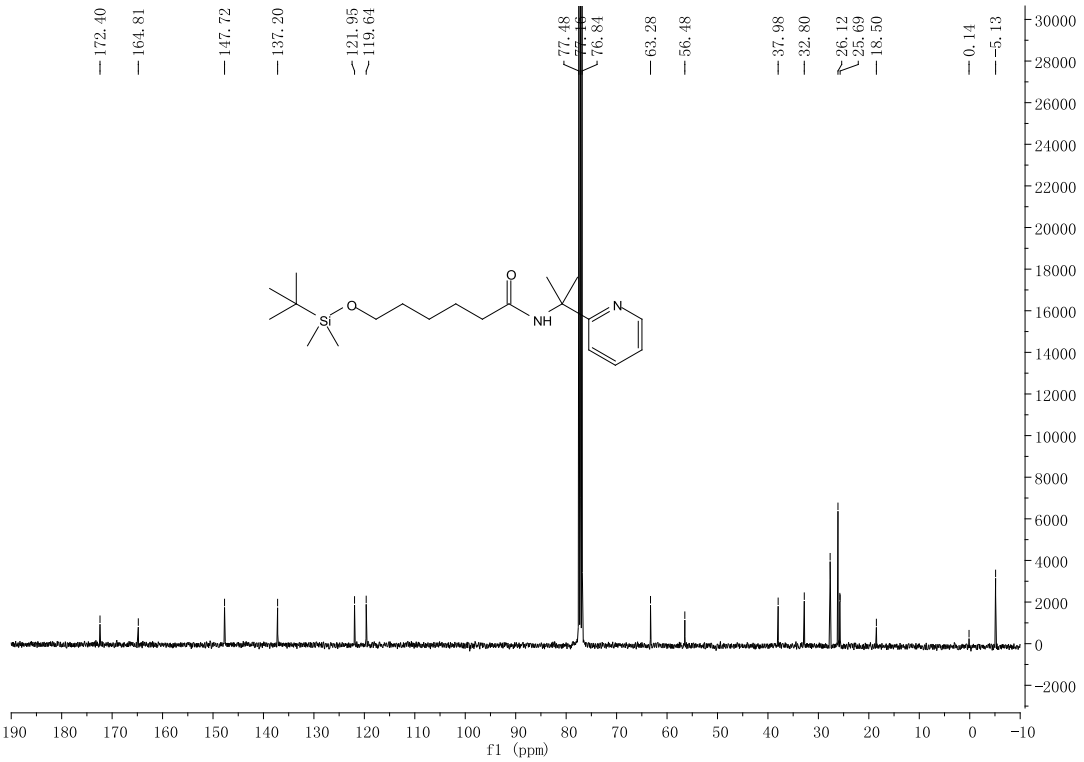
2.12
2.12
5.92
2.06
2.21
2.14
9.33
6.20
f1 (ppm)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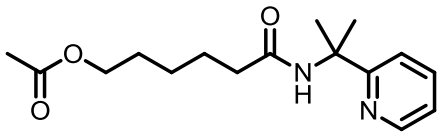
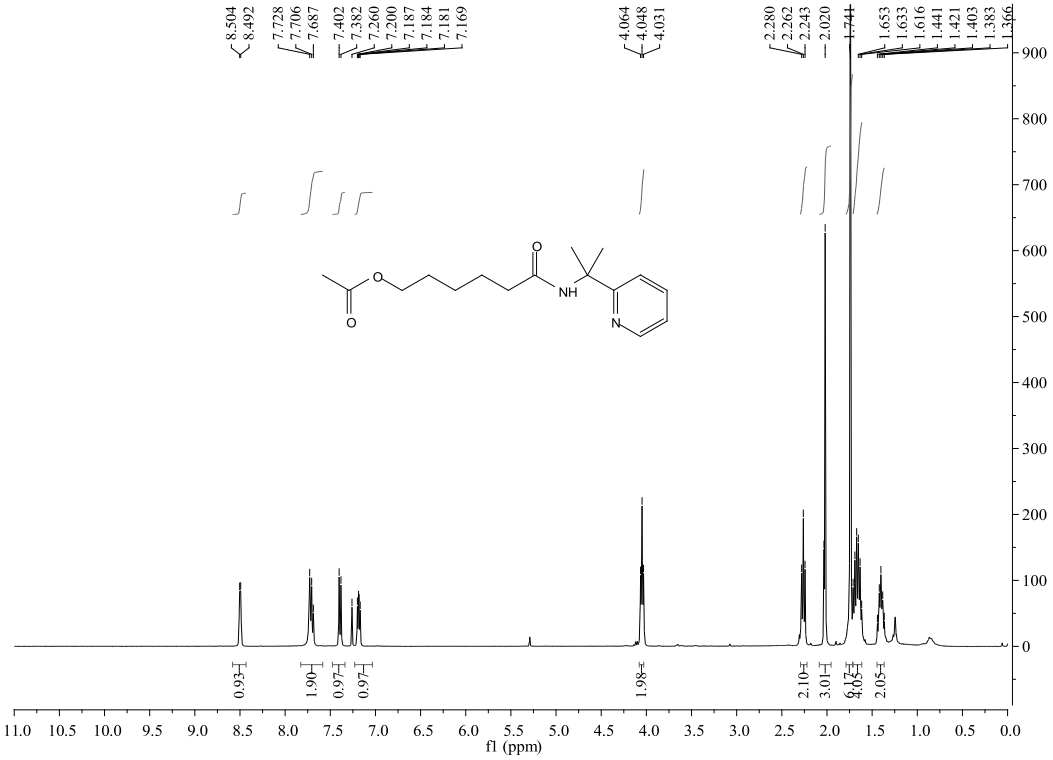
-5.13
f1 (ppm)

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7.181
7.169
4.064
4.048
4.031
2.280
2.262
2.243
2.020
1.741
1.653
1.633
1.616
1.441
1.421
1.403
1.383
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1.98
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3.01
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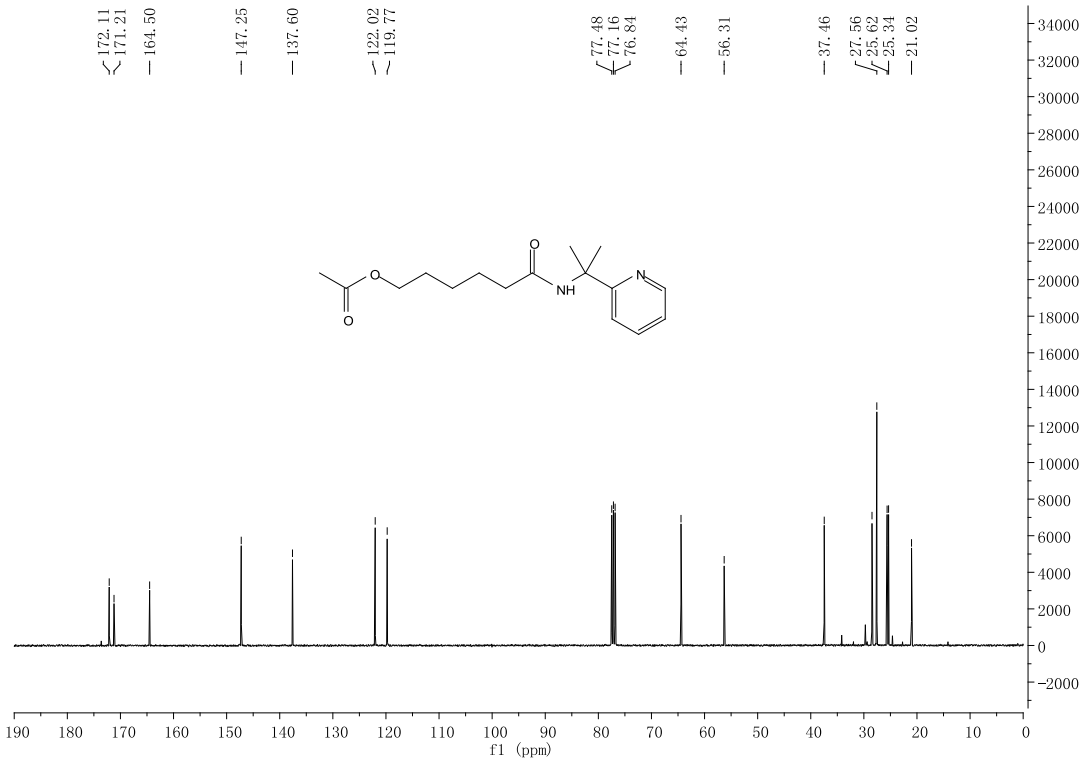
fl (ppm)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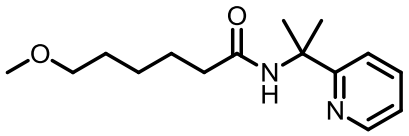
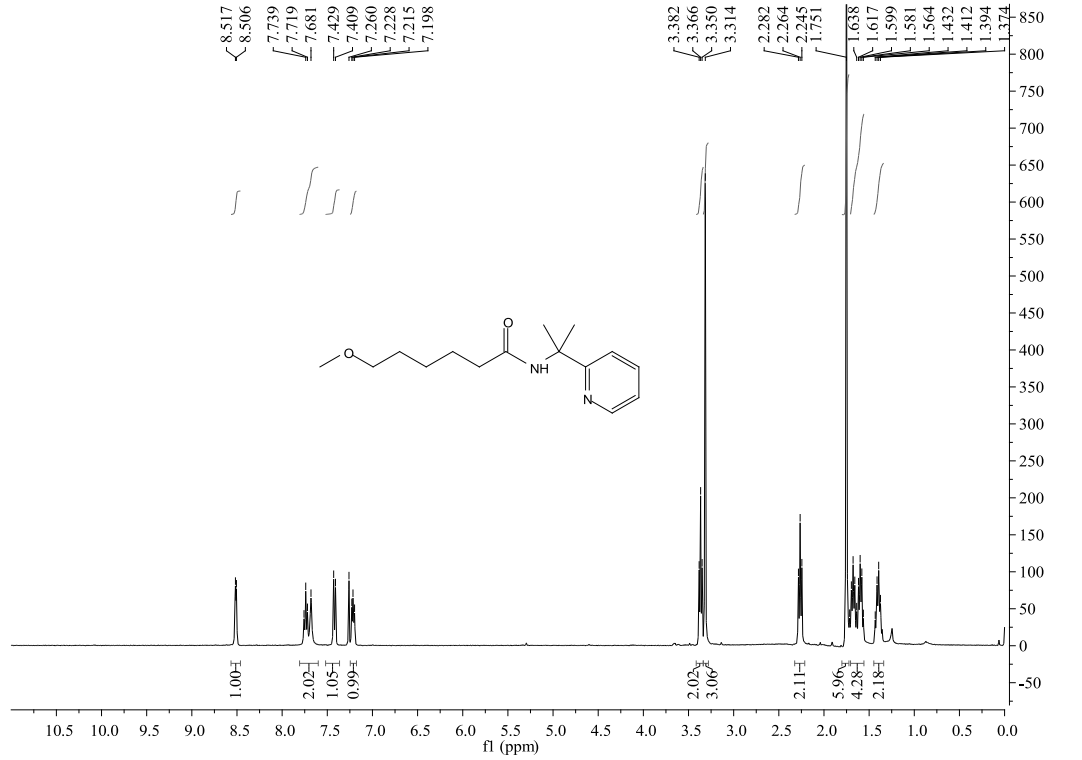
21.02
f1 (ppm)

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3.366
3.350
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2.282
2.264
2.245
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1.564
1.432
1.412
1.394
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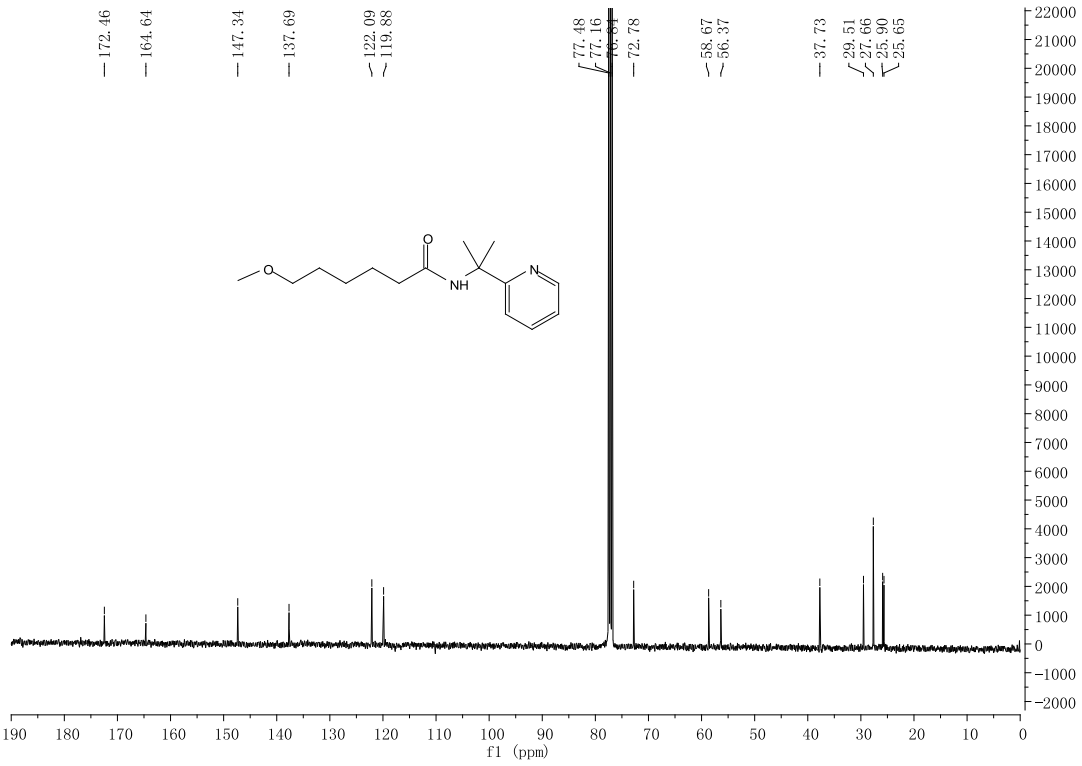
3.06
2.11
5.96
4.28
2.18
f1 (ppm)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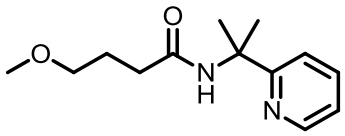
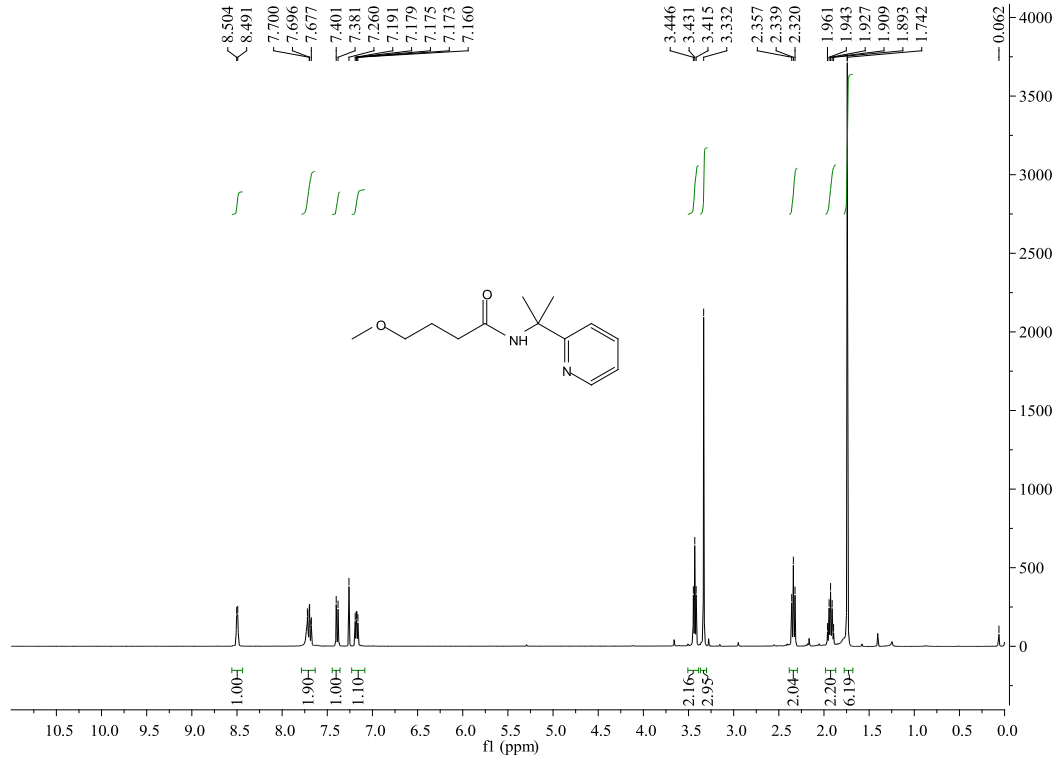
25.65
f1 (ppm)

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7.175
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7.160
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3.431
3.415
3.332
2.357
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1.927
1.909
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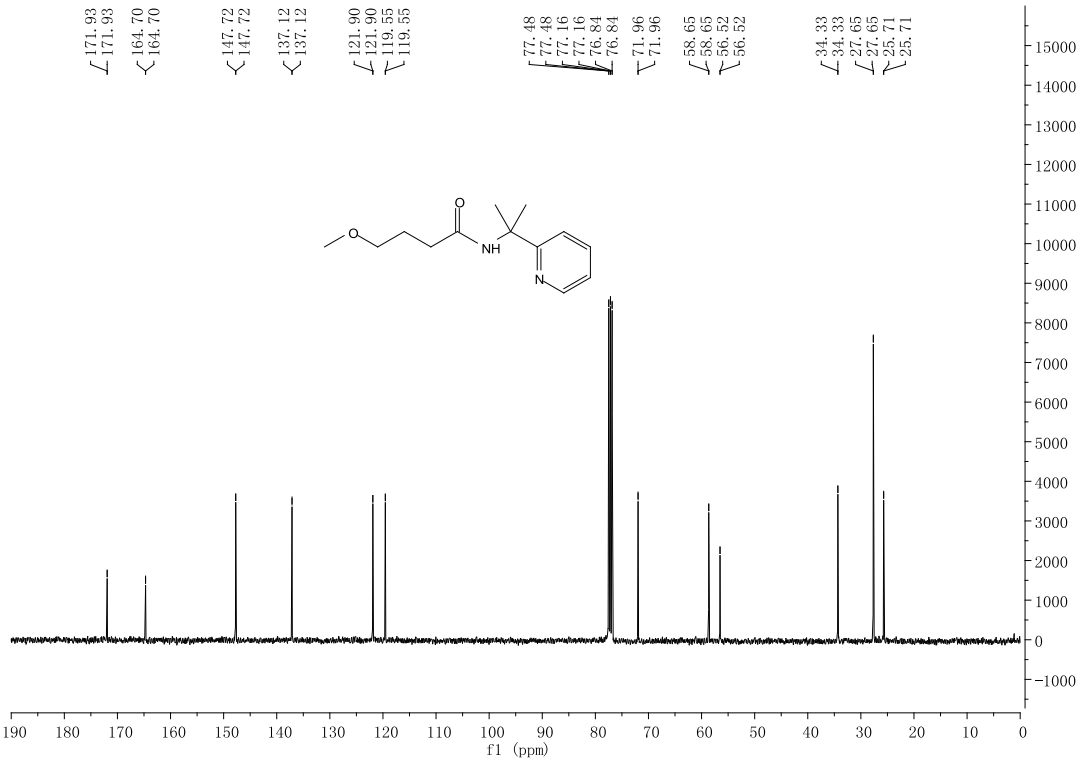
f1 (ppm)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77.16
76.84
76.84
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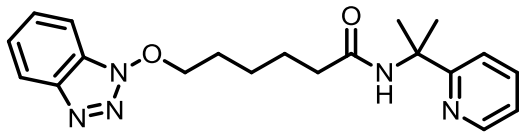
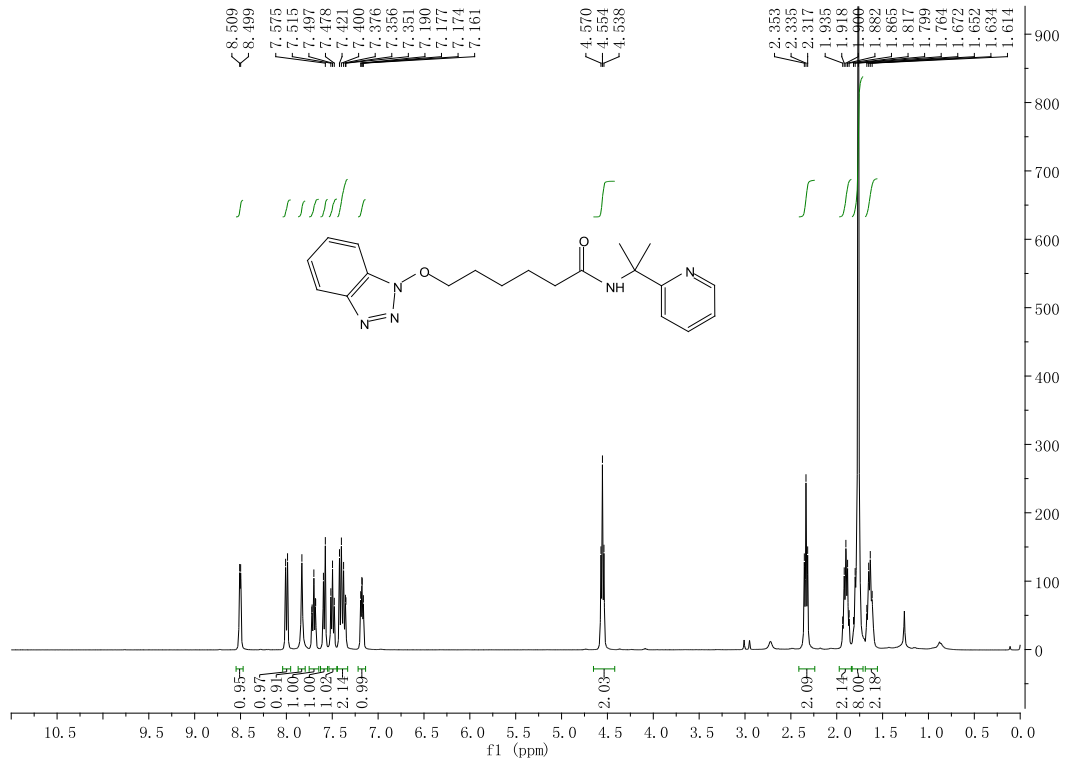
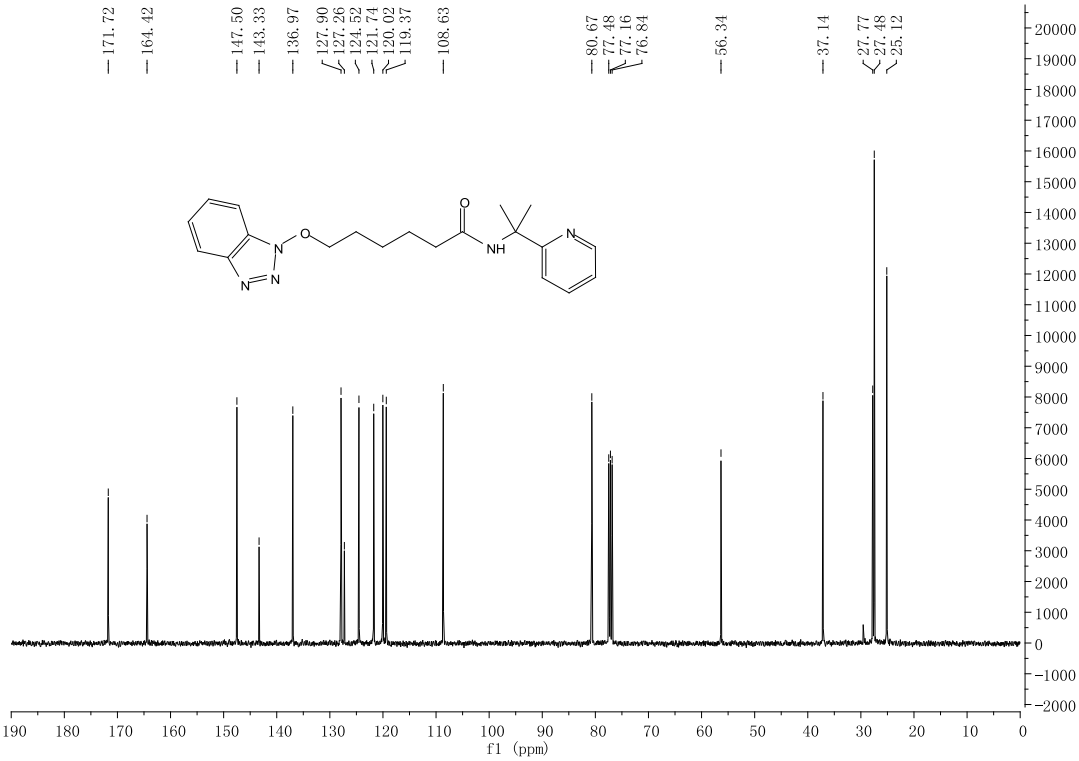
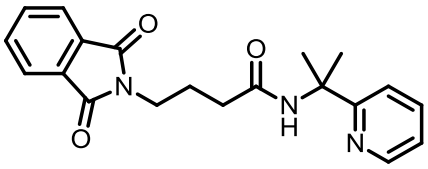
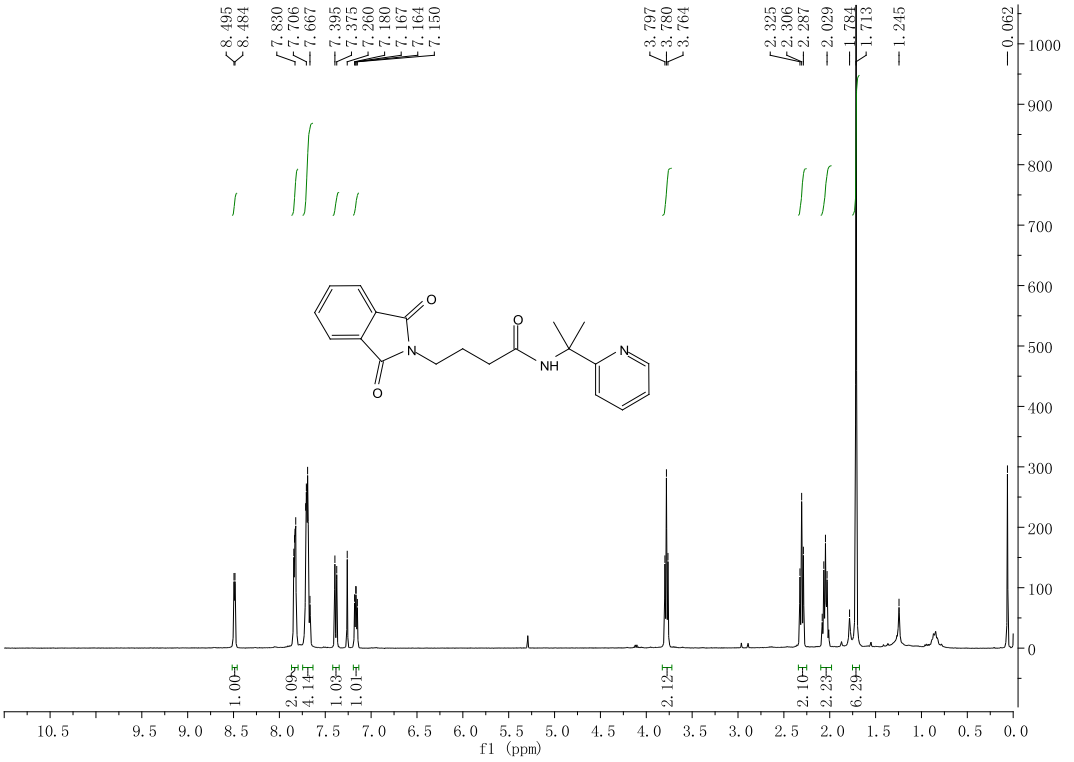
f1 (ppm)







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3.780
3.764
2.325
2.306
2.287
2.029
1.784
1.713
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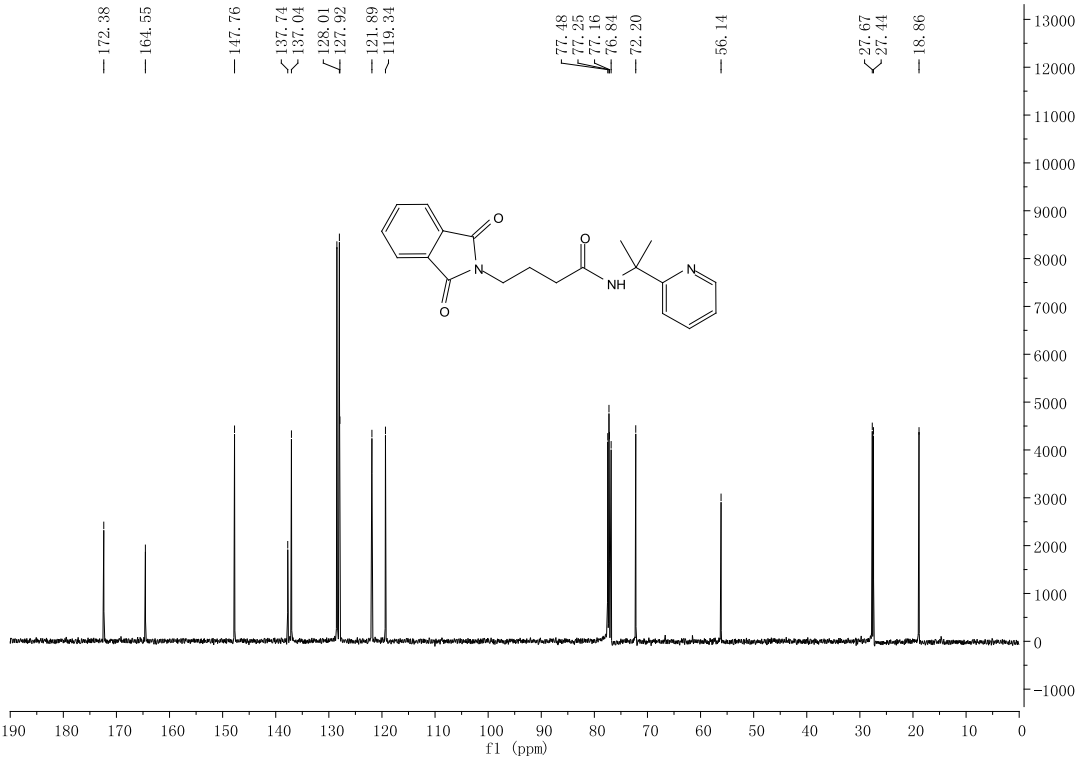
1.03
1.01
2.12
2.10
2.23
6.29
f1 (ppm)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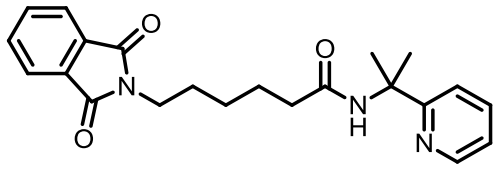
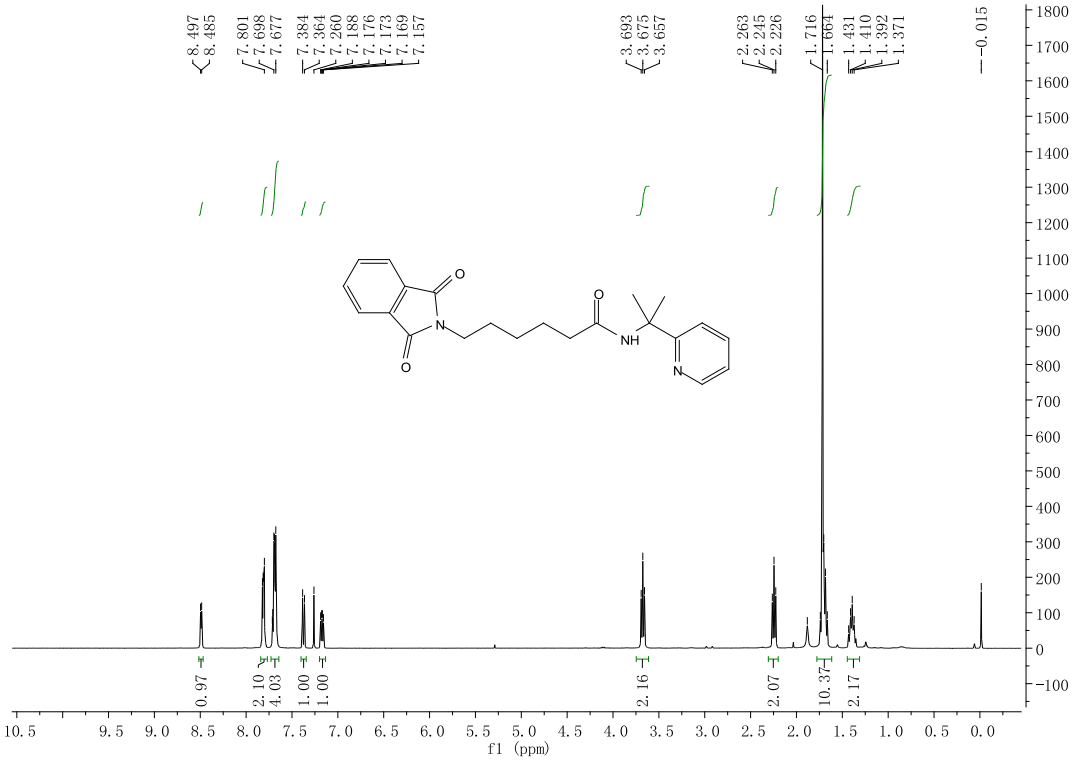
f1 (ppm)

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7.364
7.260
7.188
7.176
7.173
7.169
7.157
3.693
3.675
3.657
2.263
2.245
2.226
1.716
1.664
1.431
1.410
1.392
1.371
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4.03
1.00
1.00
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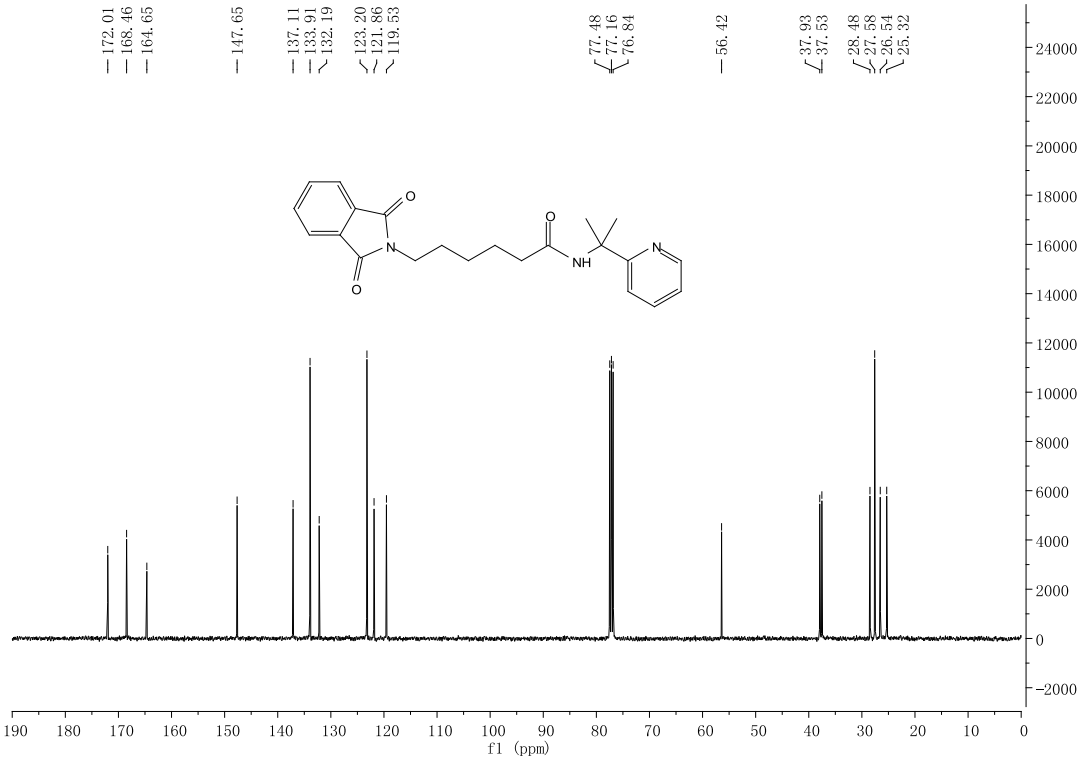
2.07
10.37
2.17
f1 (ppm)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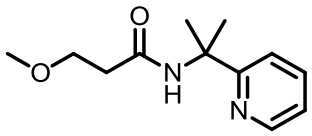
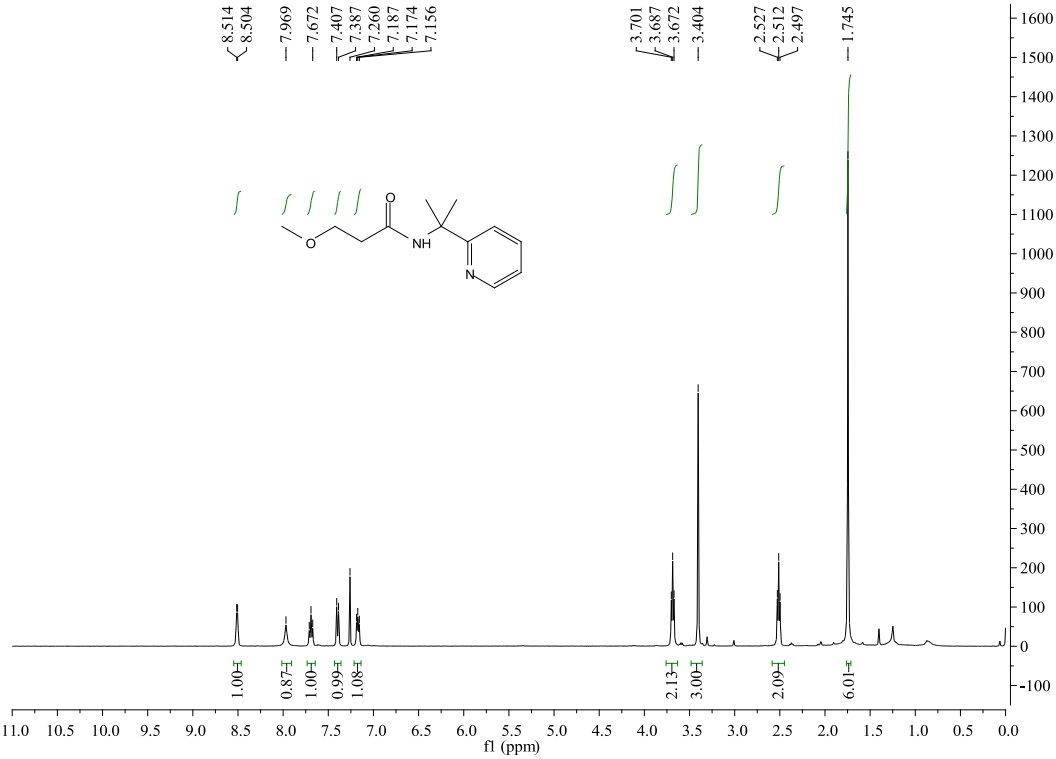
164.65
147.65
137.11
133.91
132.19
123.20
121.86
119.53
77.48
77.16
76.84
56.42
37.93
37.53
28.48
27.58
26.54
25.32
f1 (ppm)

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7.387
7.260
7.187
7.174
7.156
3.701
3.687
3.672
3.404
2.527
2.512
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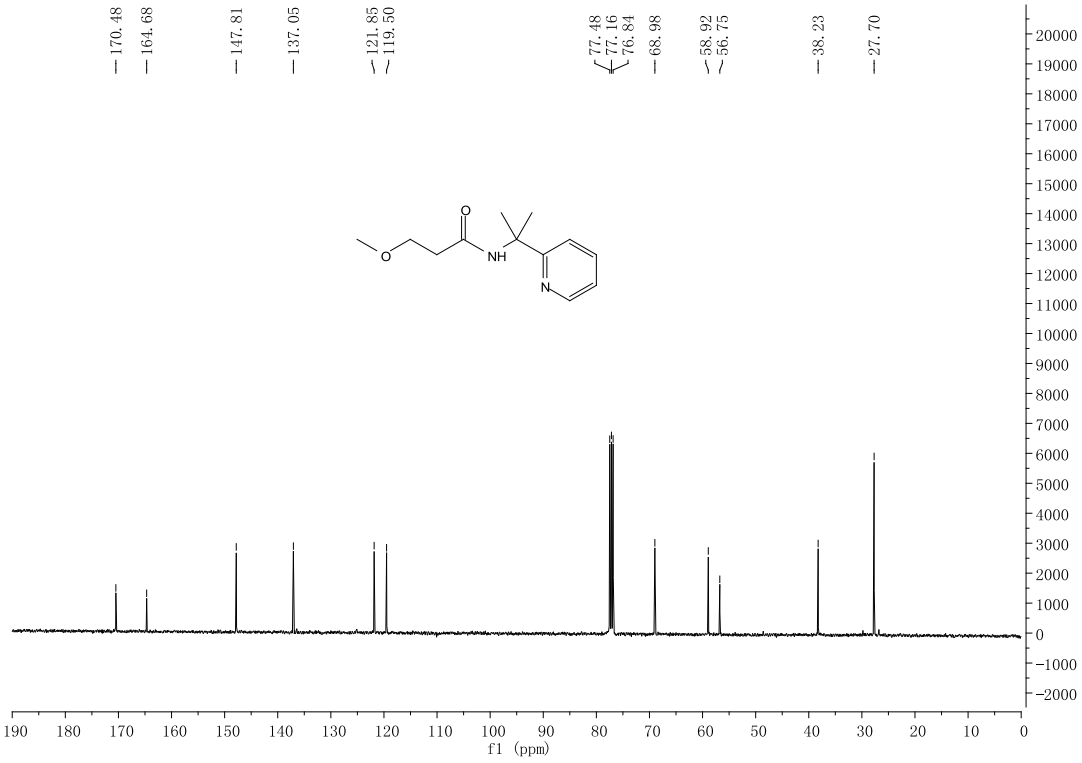
0.87
1.00
0.99
1.08
2.13
3.00
2.09
6.01
f1 (ppm)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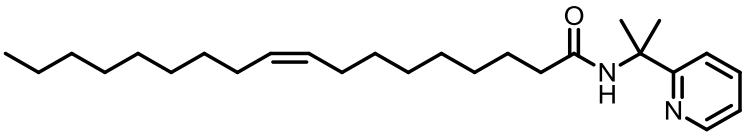
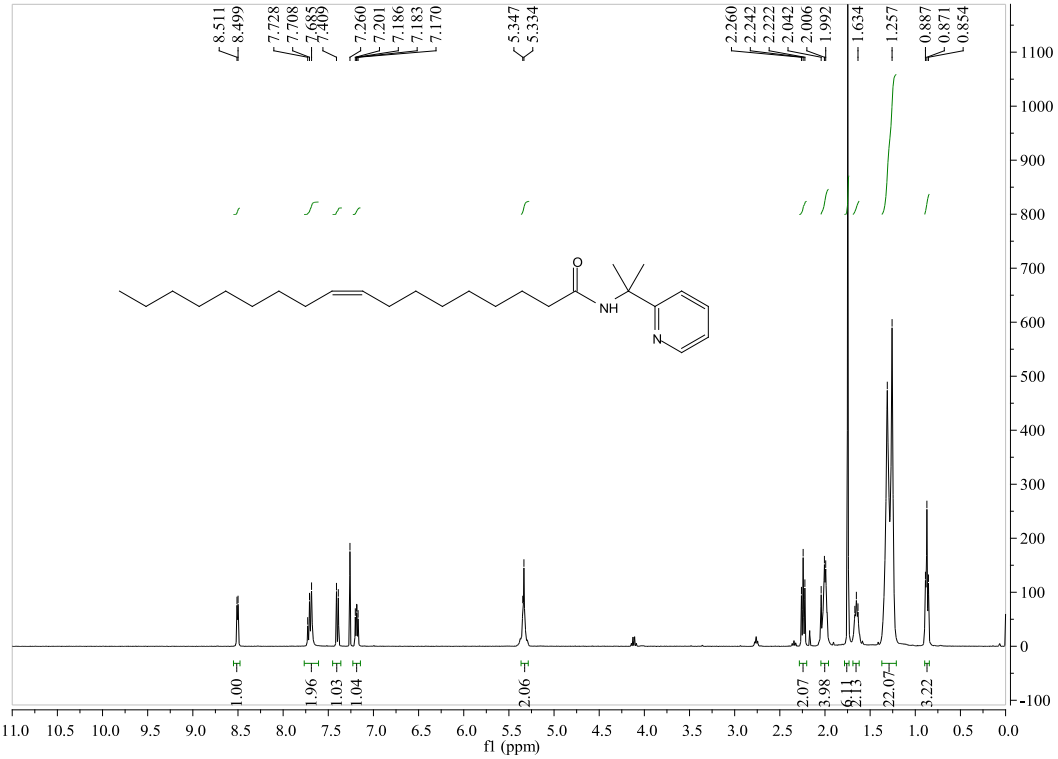
77.16
76.84
68.98
58.92
56.75
38.23
27.70
f1 (ppm)

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7.201
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7.170
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5.334
2.260
2.242
2.222
2.042
2.006
1.992
1.634
1.257
0.887
0.871
0.854
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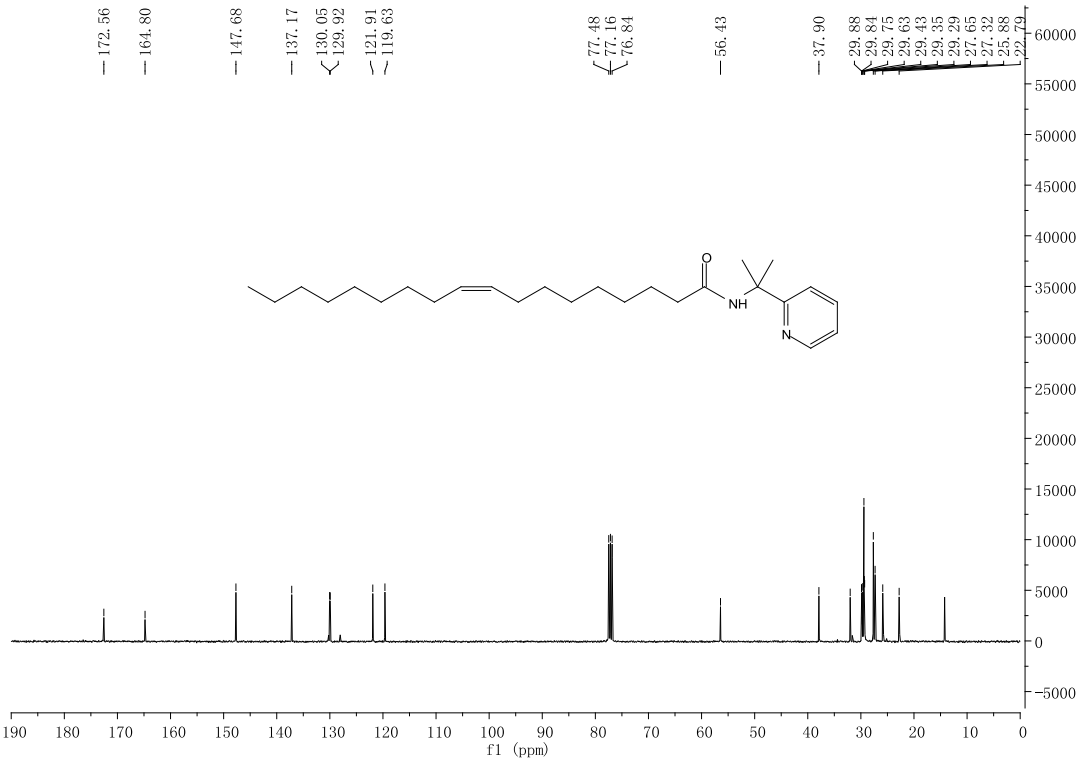
1.96
1.03
1.04
2.06
2.07
3.98
6.11
2.13
22.07
3.22
fl (ppm)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119.63
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77.16
76.84
56.43
37.90
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29.63
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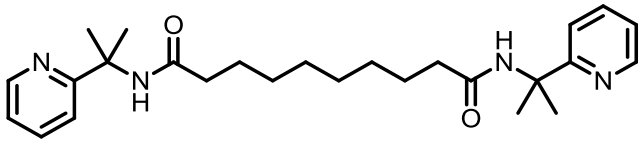
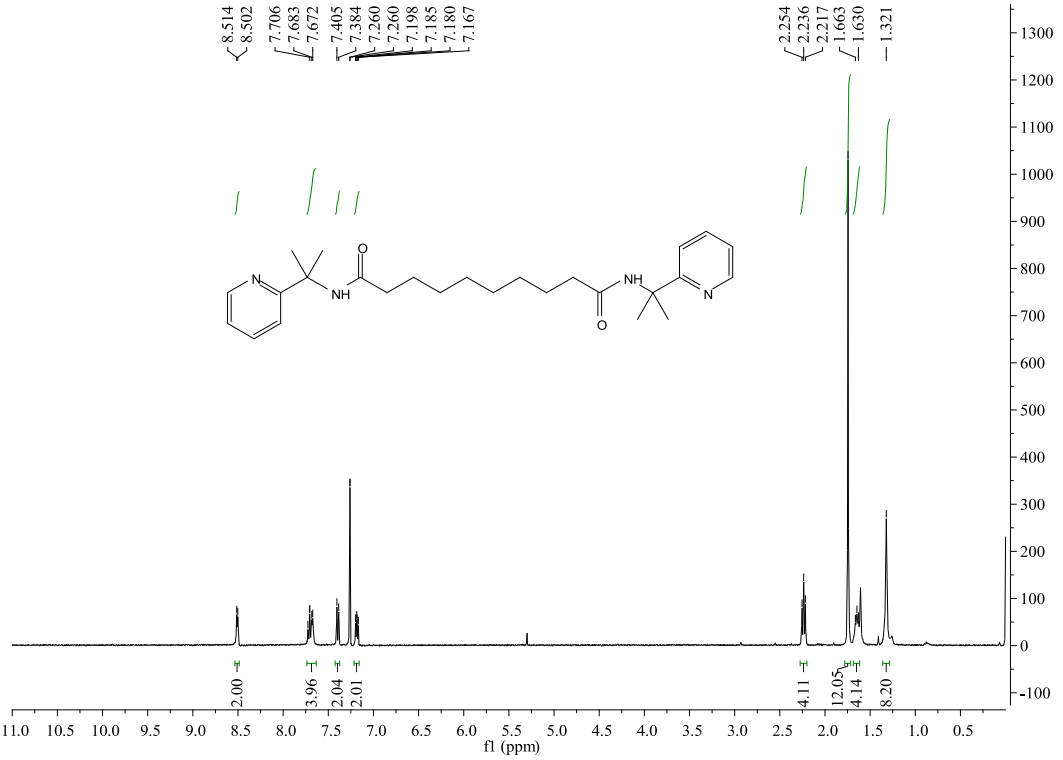
29.35
29.29
27.65
27.32
25.88
22.79
f1 (ppm)

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7.405
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7.260
7.260
7.198
7.185
7.180
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2.236
2.217
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1.630
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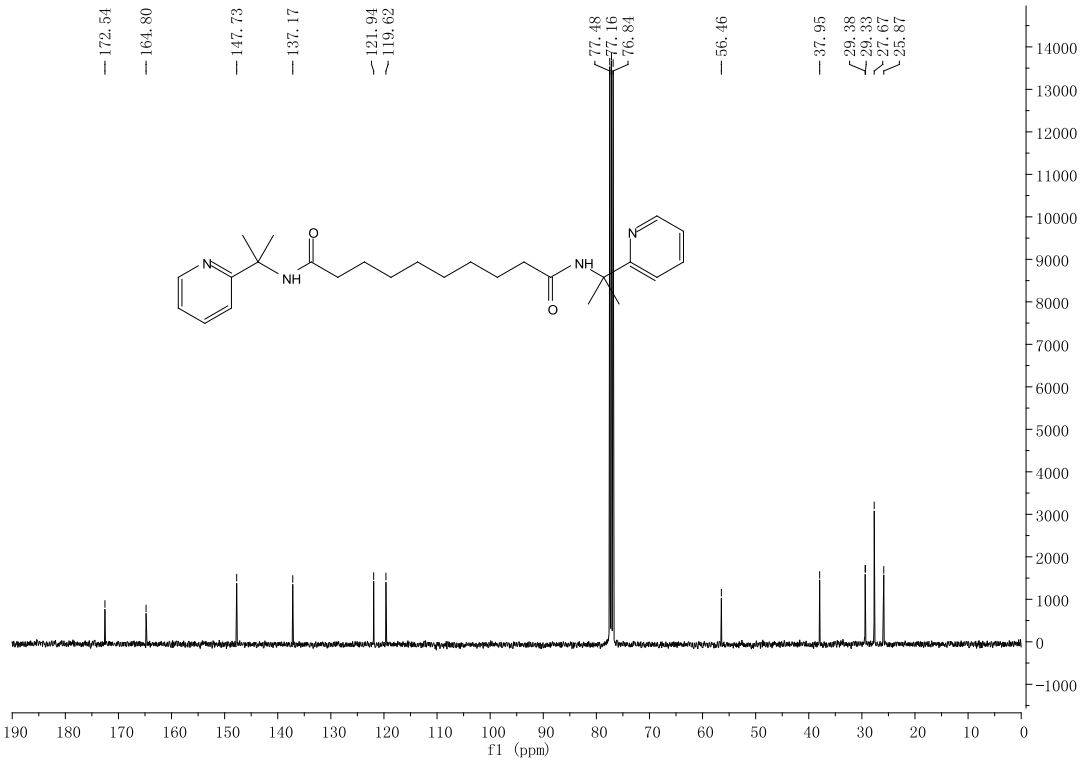
2.04
2.01
4.11
12.05
4.14
8.20
f1 (ppm)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77.16
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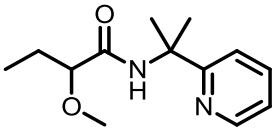
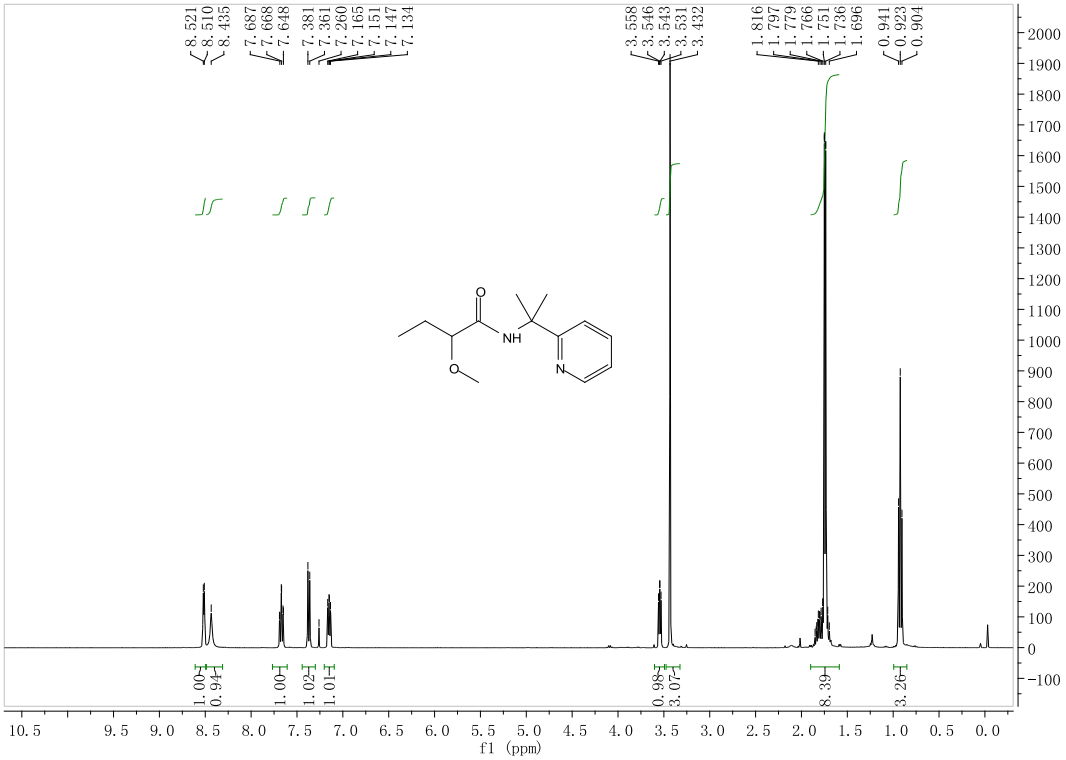
29.33
27.67
25.87
f1 (ppm)

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7.648
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7.361
7.260
7.165
7.151
7.147
7.134
3.558
3.546
3.543
3.531
3.432
1.816
1.797
1.779
1.766
1.751
1.736
1.696
0.941
0.923
0.904
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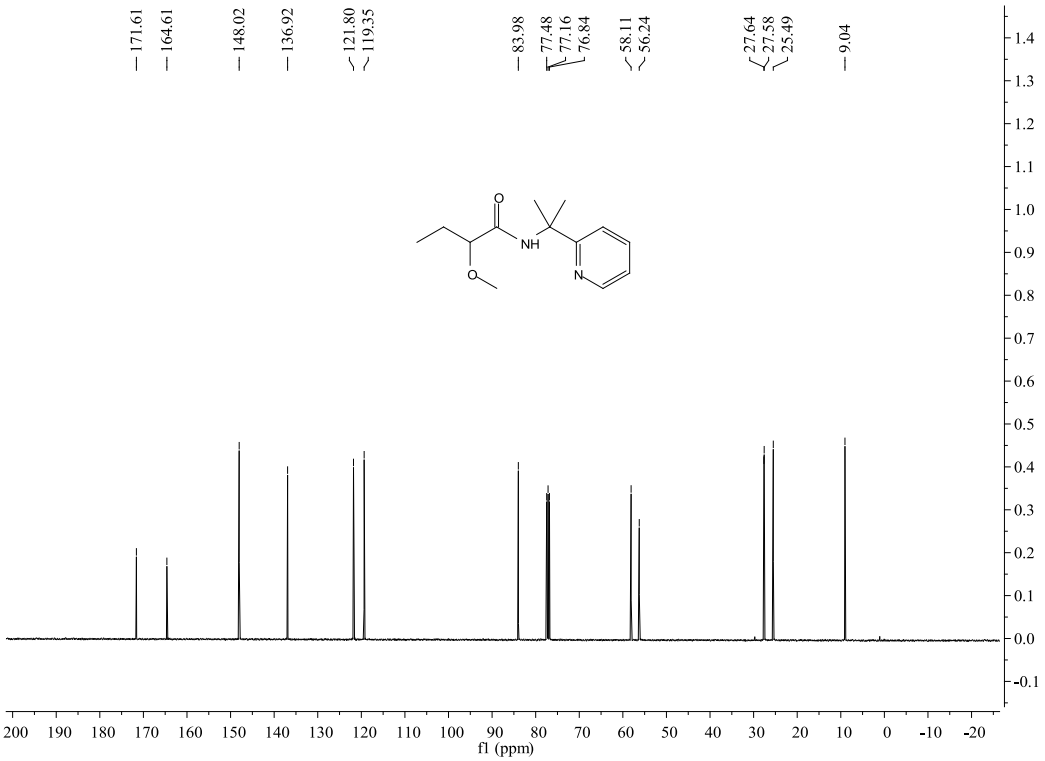
0.94
1.00
1.02
1.01
0.98
3.07
8.39
3.26
f1 (ppm)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164.61
148.02
136.92
121.80
119.35
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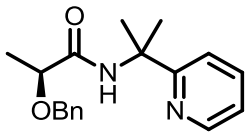
77.48
77.16
76.84
58.11
56.24
27.64
27.58
25.49
9.04
f1 (ppm)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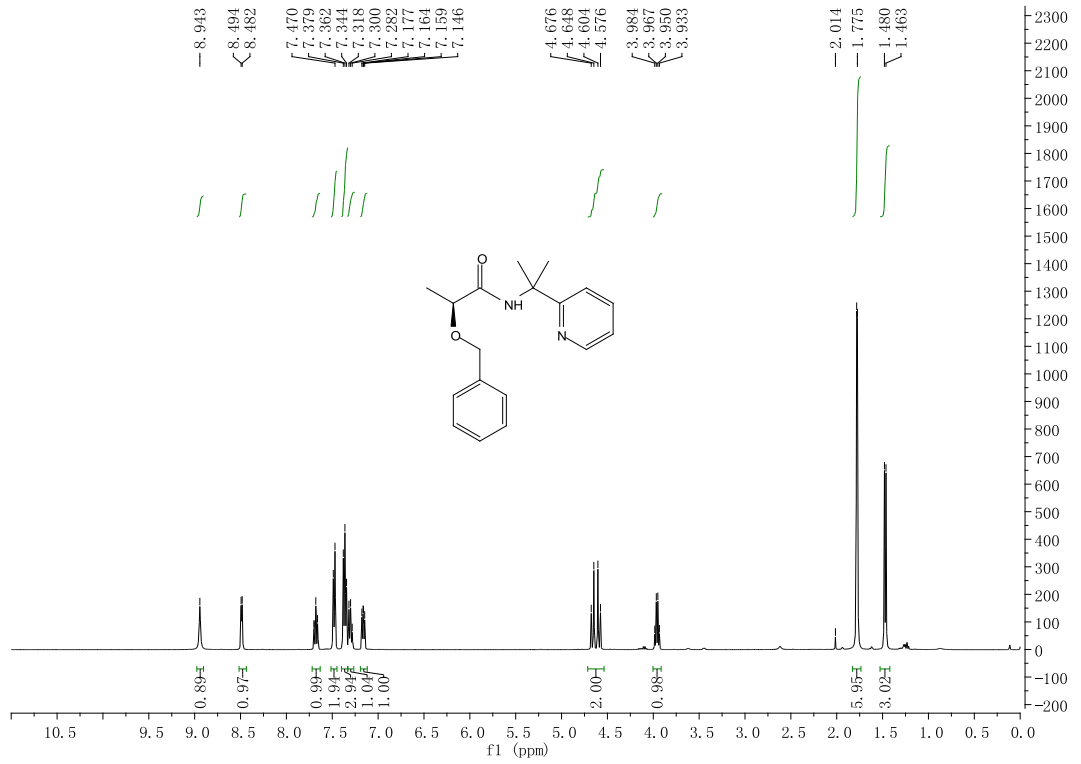
N
H
OBn
N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7.300
7.282
7.177
7.164
7.159
7.146
4.676
4.648
4.604
4.576
3.984
3.967
3.950
3.933
2.014
1.775
1.480
1.463
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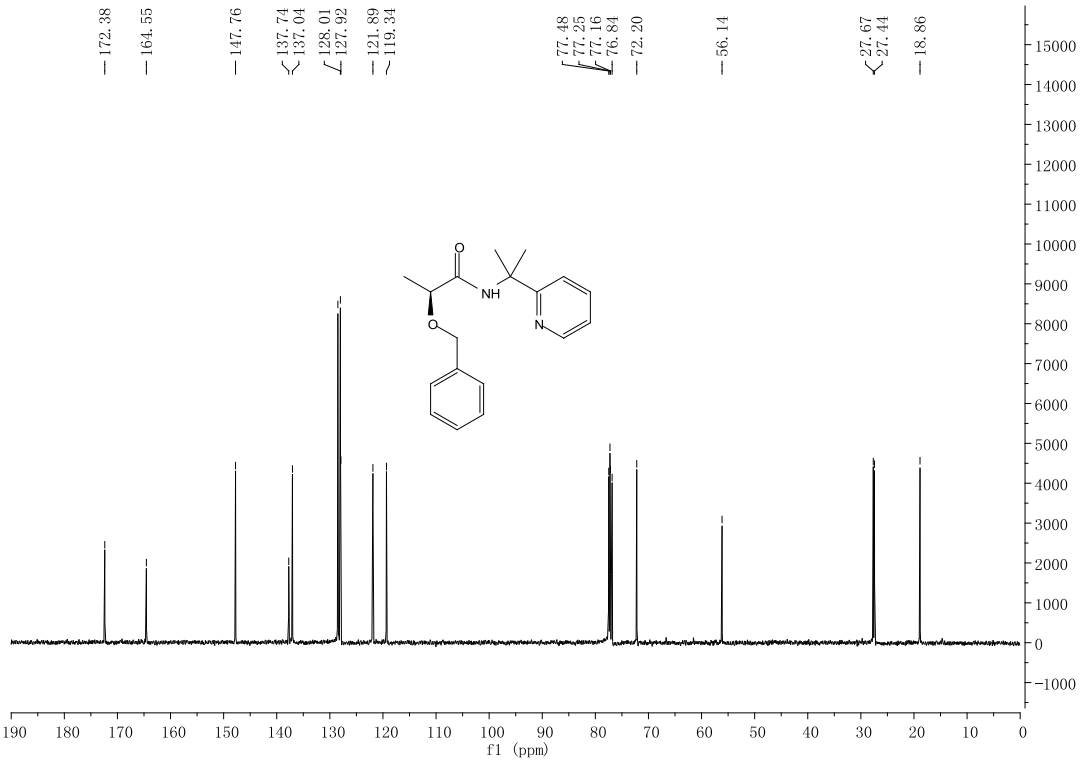
0.97
0.99
1.94
2.94
1.04
1.00
2.00
0.98
5.95
3.02
f1 (ppm)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137.04
128.01
127.92
121.89
119.34
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77.25
77.16
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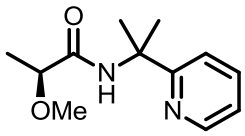
56.14
27.67
27.44
18.86
f1 (ppm)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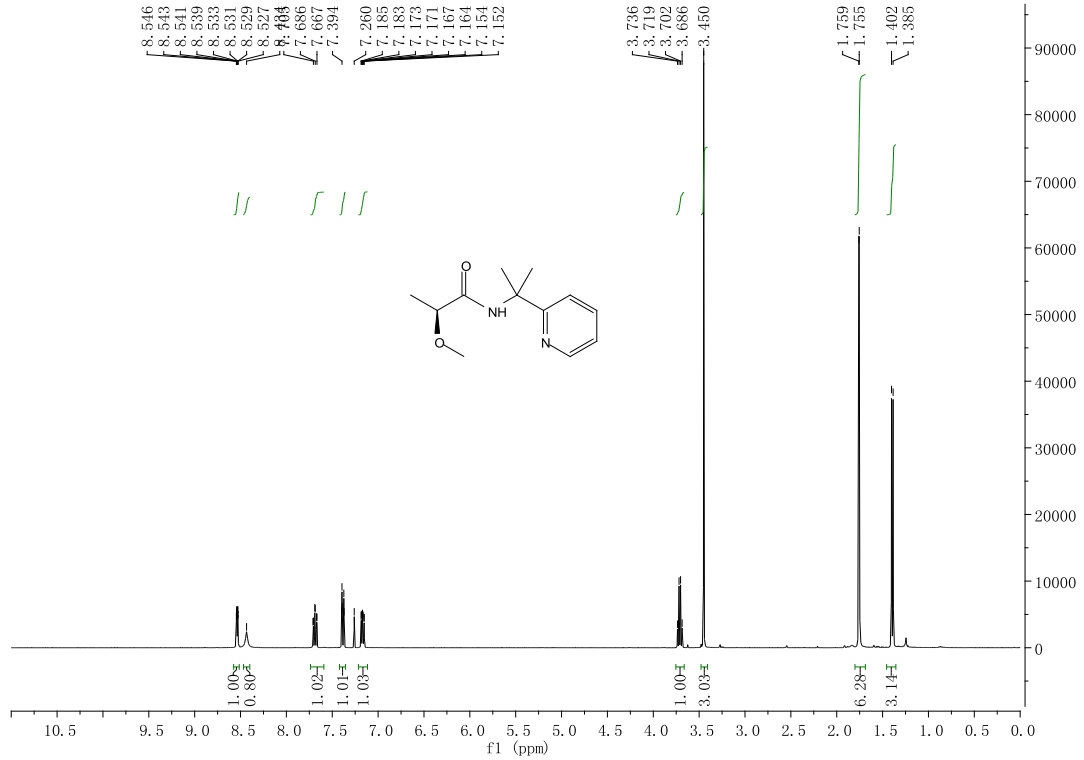
N
H
OMe
N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8.533
8.531
8.529
8.527
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7.667
7.394
7.260
7.185
7.183
7.173
7.171
7.167
7.164
7.154
7.152
3.736
3.719
3.702
3.686
3.450
1.759
1.755
1.402
1.385
NH
N
O
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1.02
1.01
1.03
1.00
3.03
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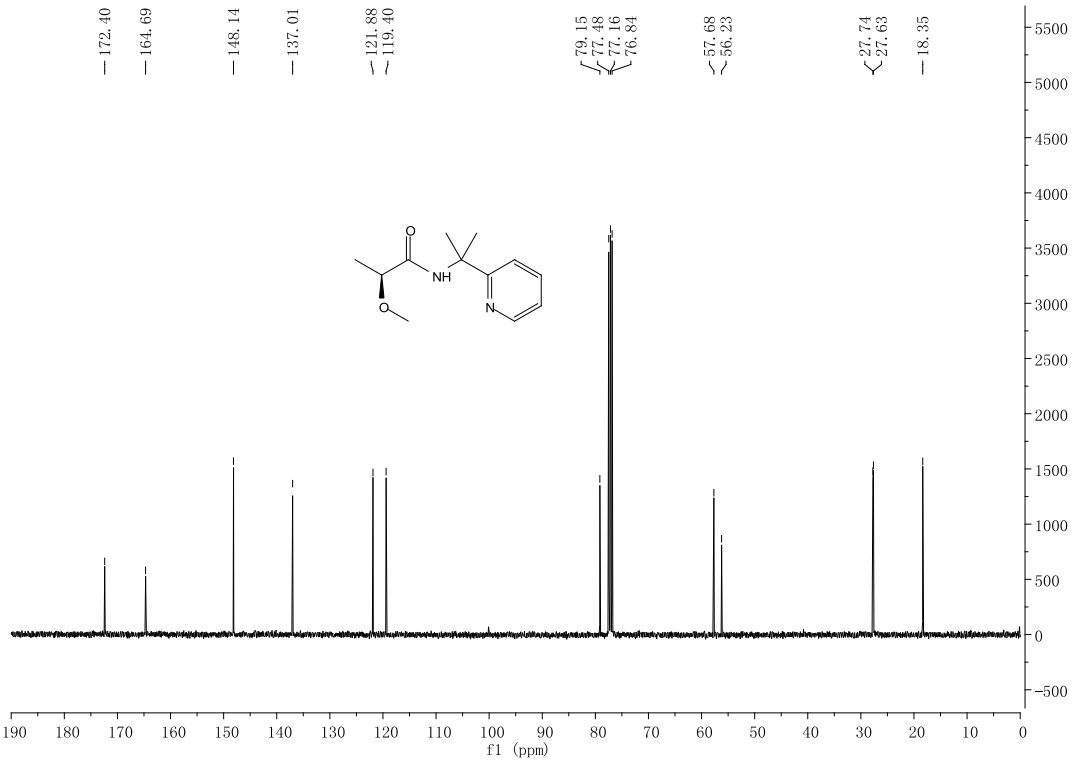
3.14
f1 (ppm)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119.40
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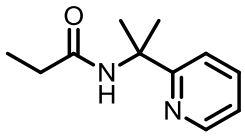
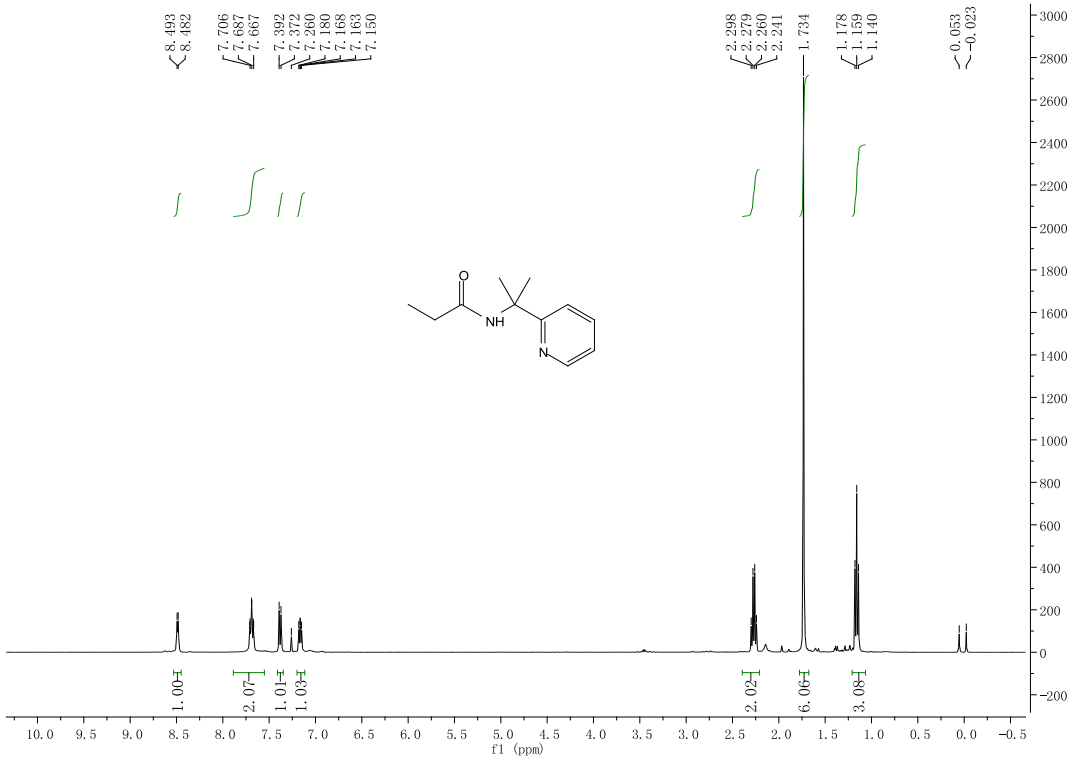
NH
N
O
f1 (ppm)

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7.150
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2.279
2.260
2.241
1.734
1.178
1.159
1.140
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-0.023
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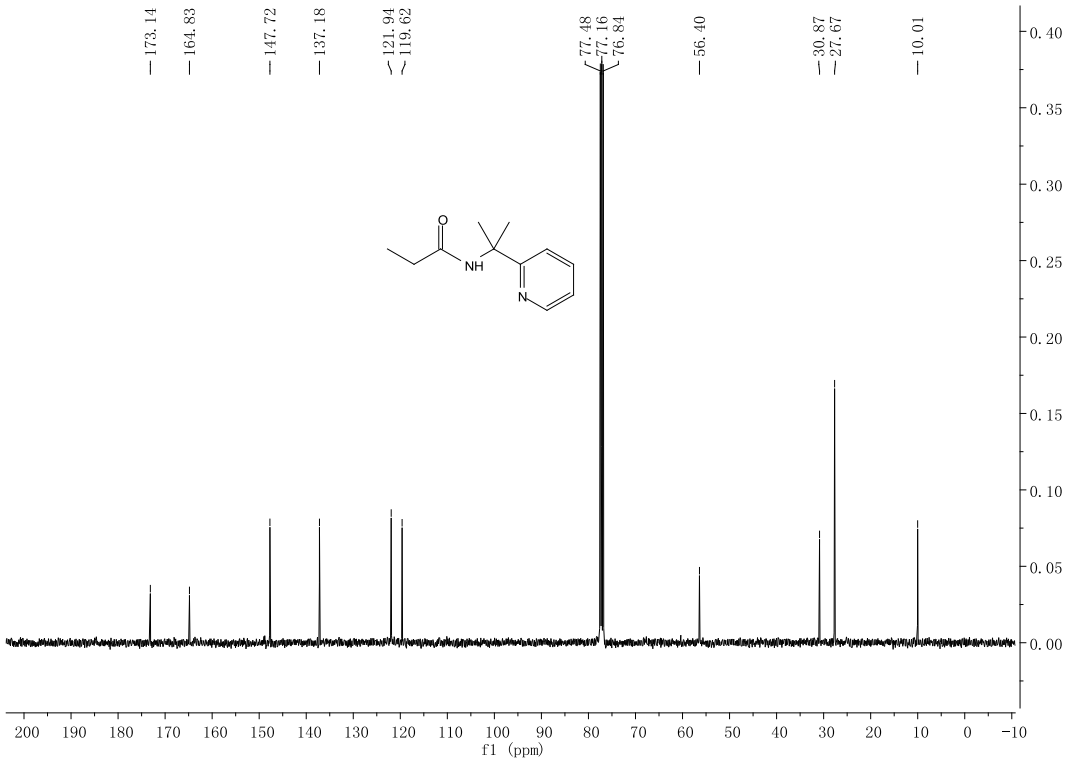
2.07
1.01
1.03
2.02
6.06
3.08
f1 (ppm)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77.16
76.84
56.40
30.87
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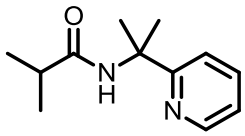
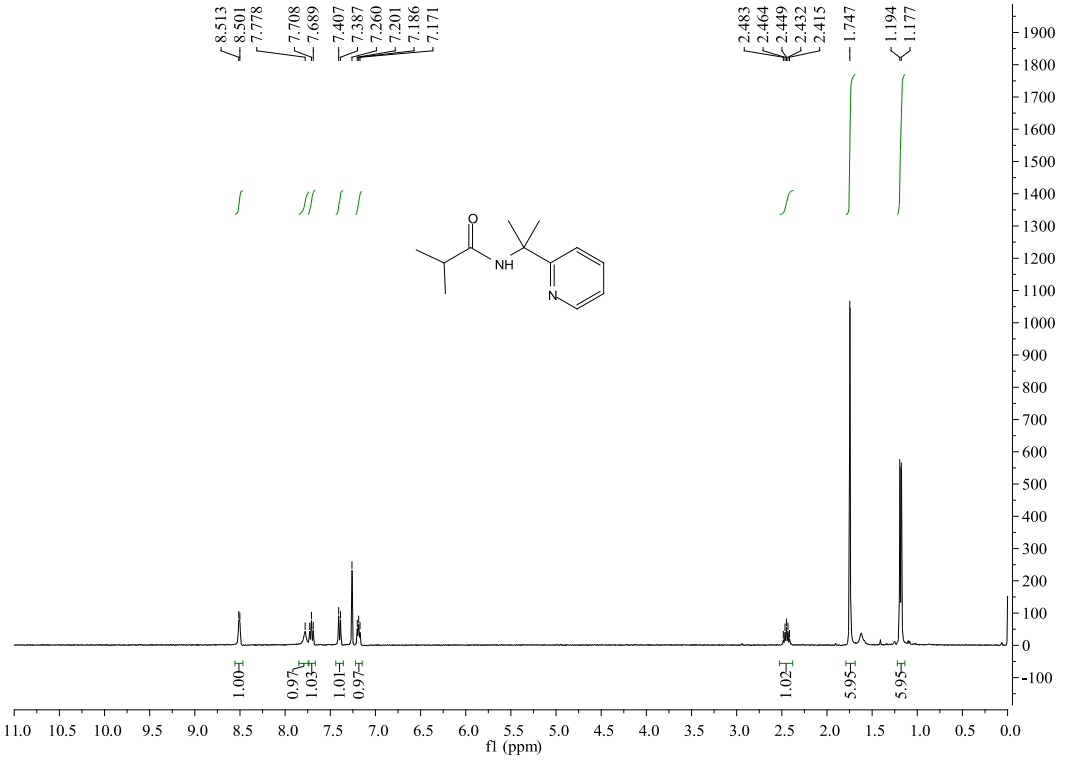
10.01
f1 (ppm)

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7.387
7.260
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7.186
7.171
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2.464
2.449
2.432
2.415
1.747
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1.03
1.01
0.97
1.02
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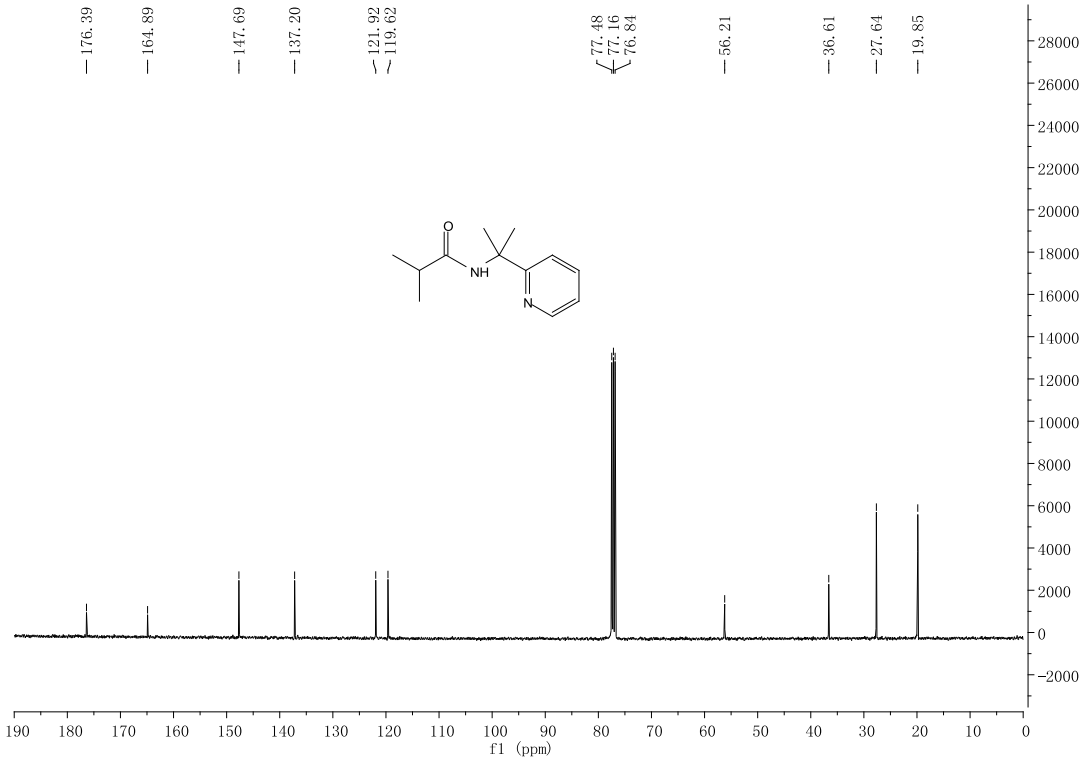
5.95
f1 (ppm)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77.16
76.84
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36.61
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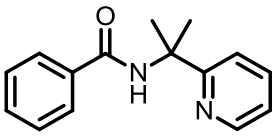
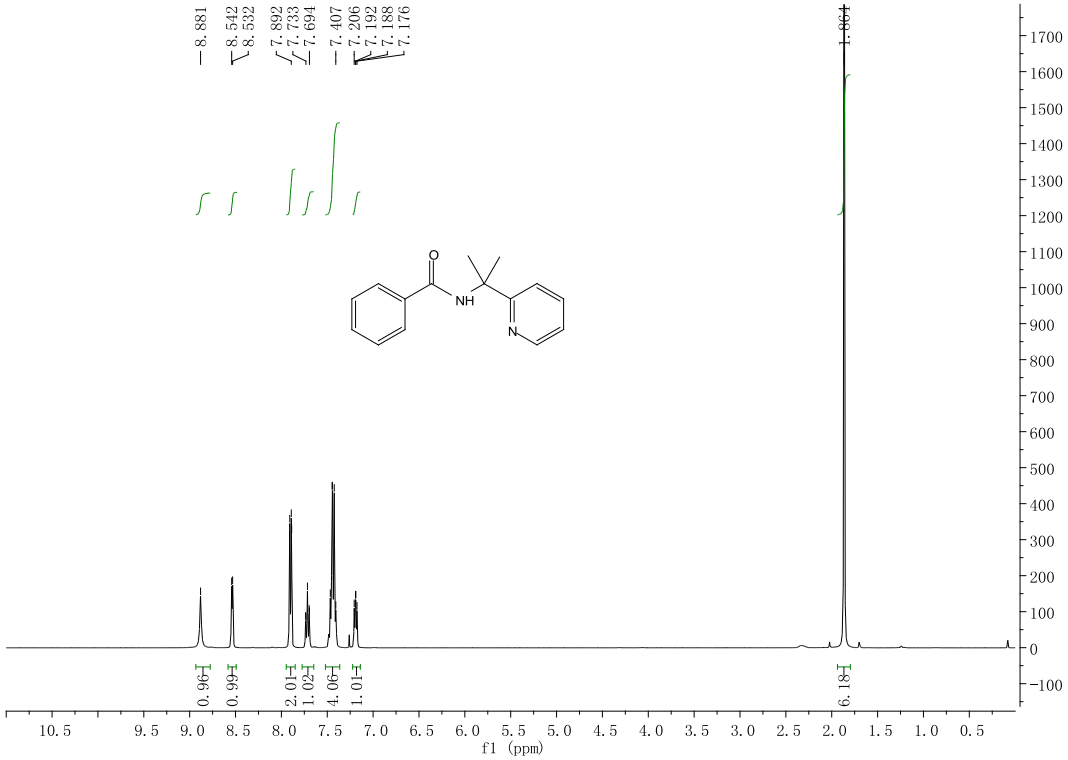
19.85
f1 (ppm)

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7.206
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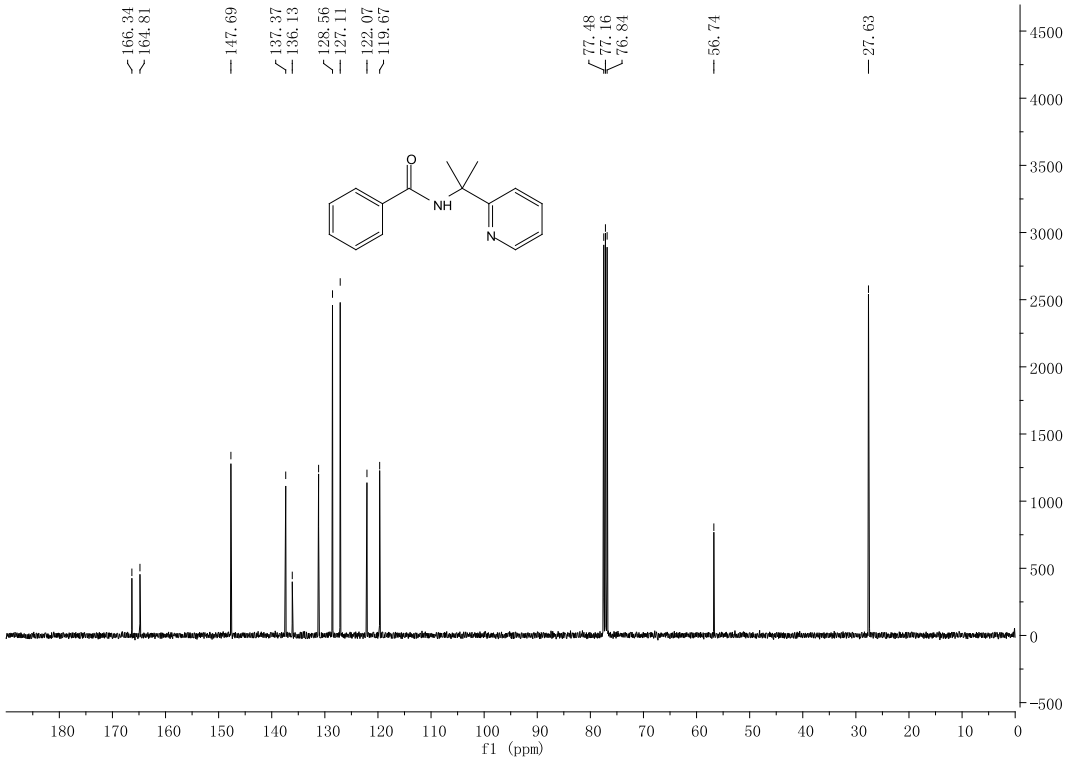
0.99
2.01
1.02
4.06
1.01
6.18
f1 (ppm)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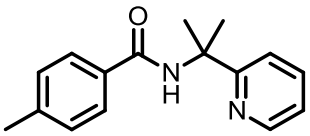
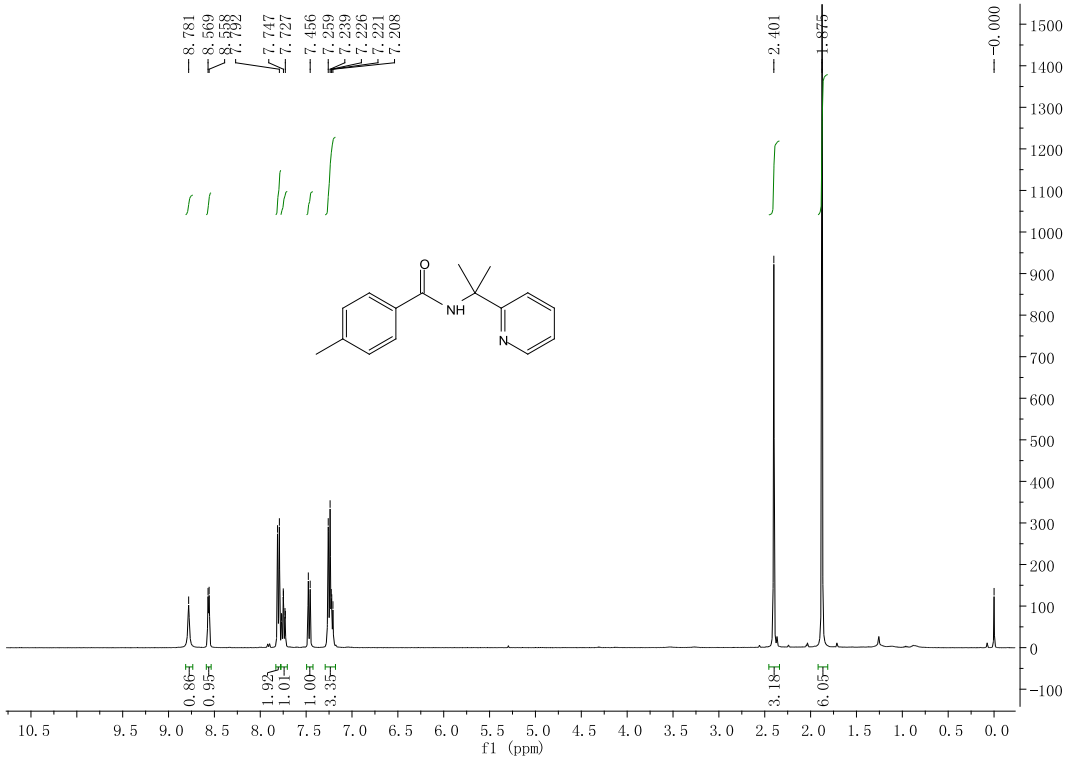
119.67
77.48
77.16
76.84
56.74
27.63
f1 (ppm)

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7.259
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7.226
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7.208
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0.86
0.95
1.92
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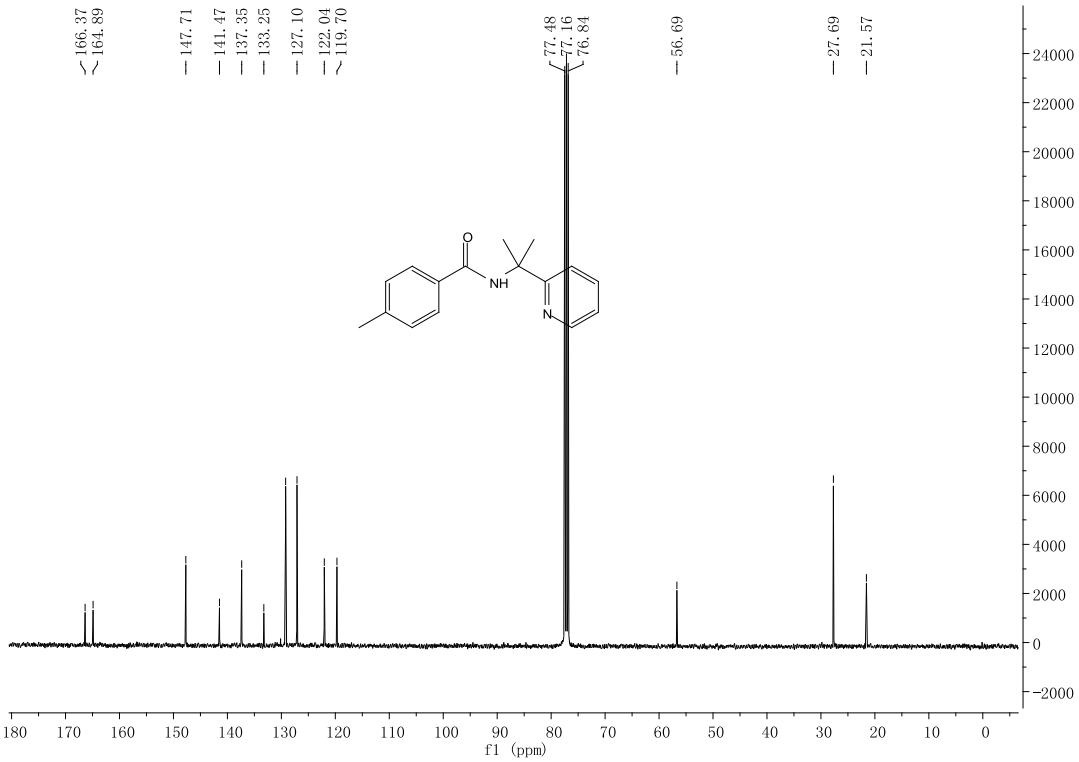
6.05
f1 (ppm)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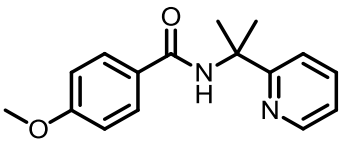
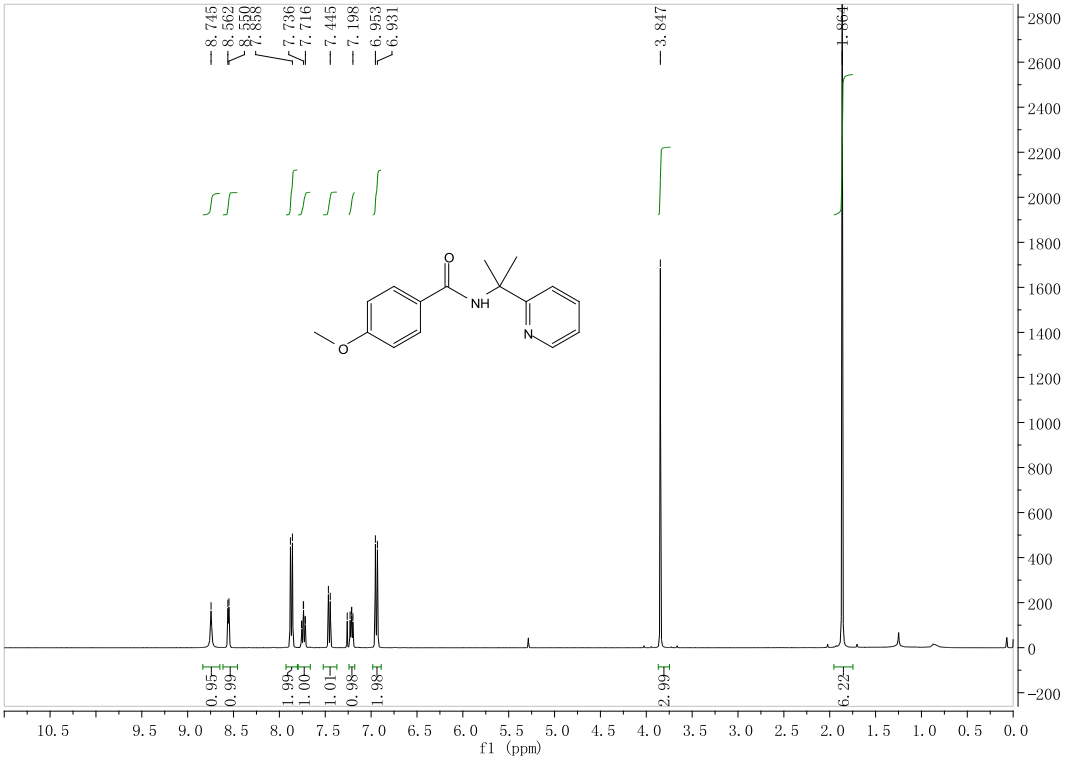
77.48
77.16
76.84
56.69
27.69
21.57
f1 (ppm)

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7.445
7.198
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6.931
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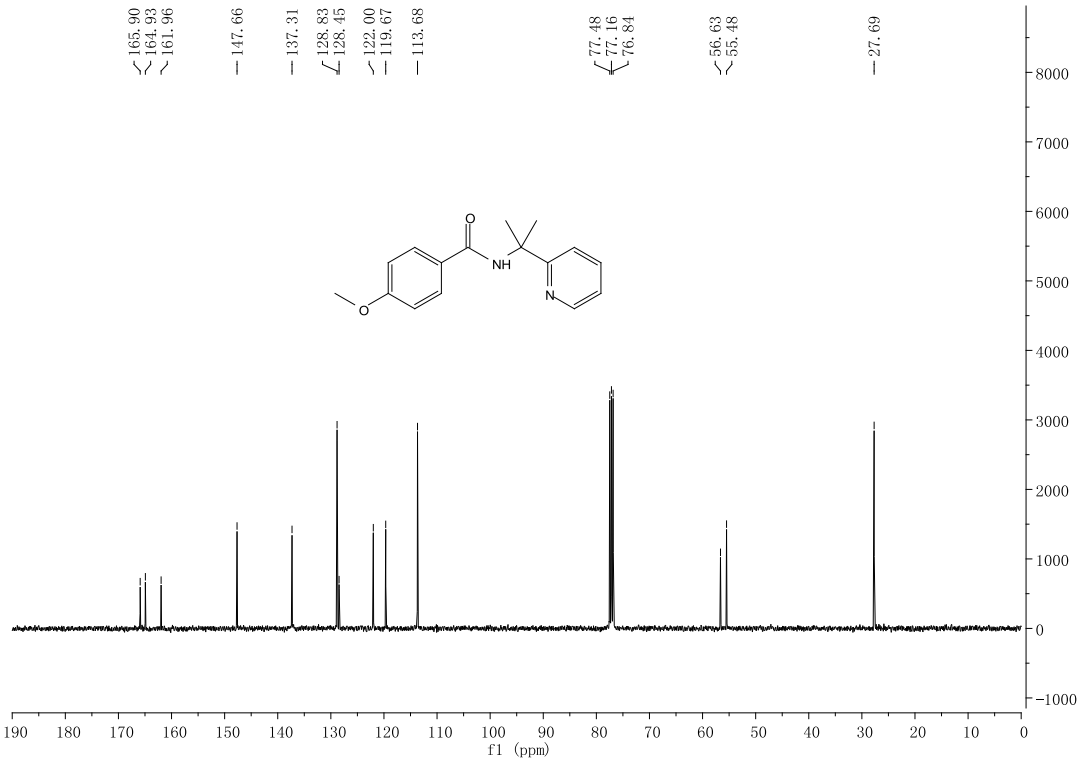
0.99
1.99
1.00
1.01
0.98
1.98
2.99
6.22
f1 (ppm)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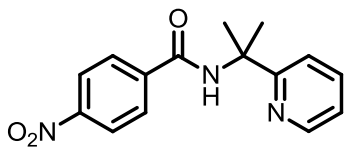
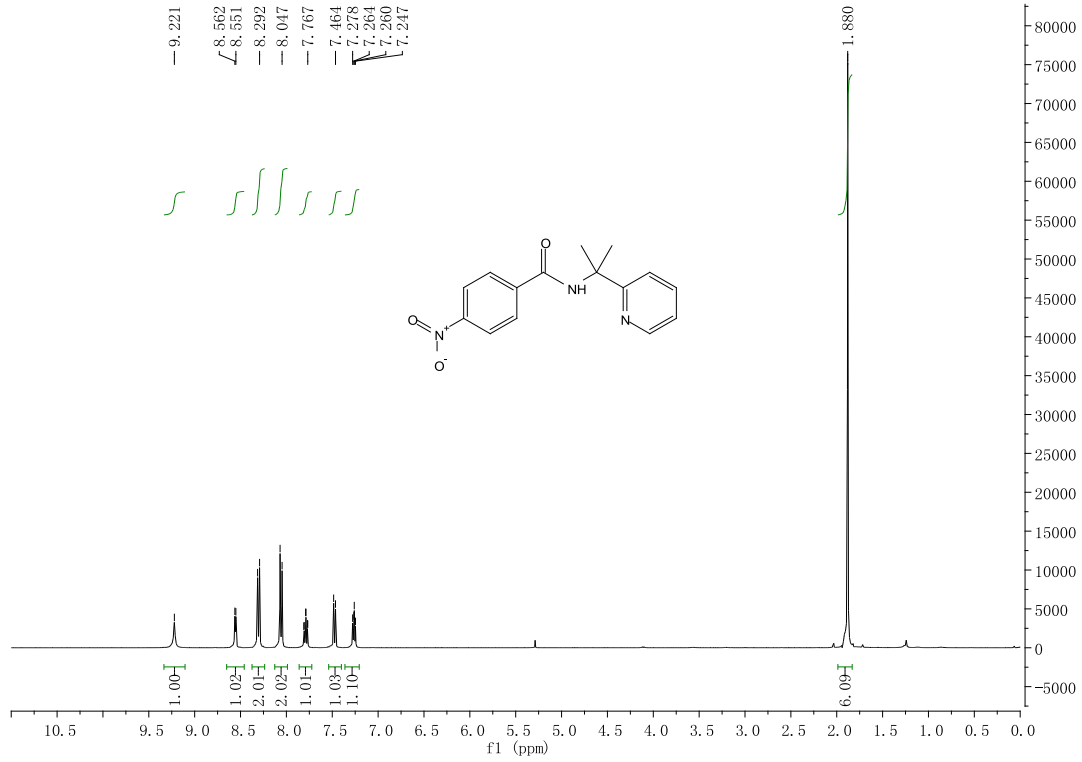
113.68
77.48
77.16
76.84
56.63
55.48
27.69
f1 (ppm)

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7.278
7.264
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7.247
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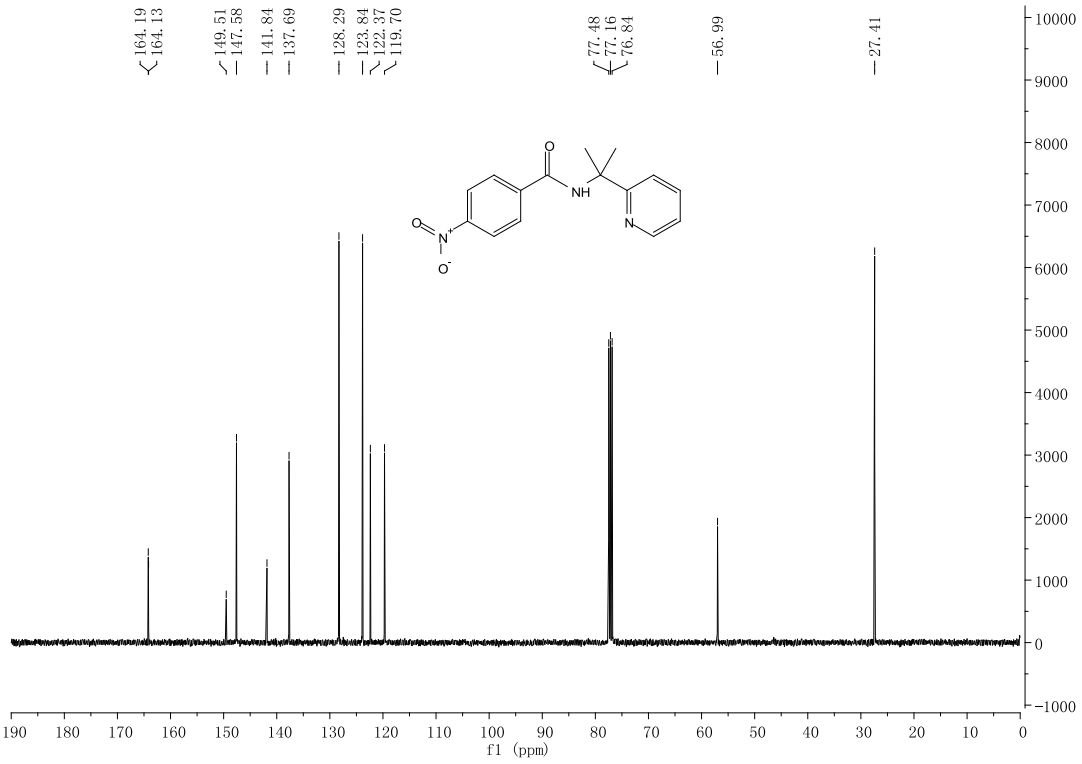
2.02
1.01
1.03
1.10
6.09
f1 (ppm)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122.37
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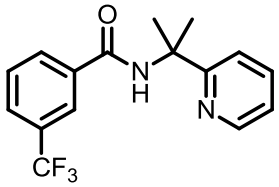
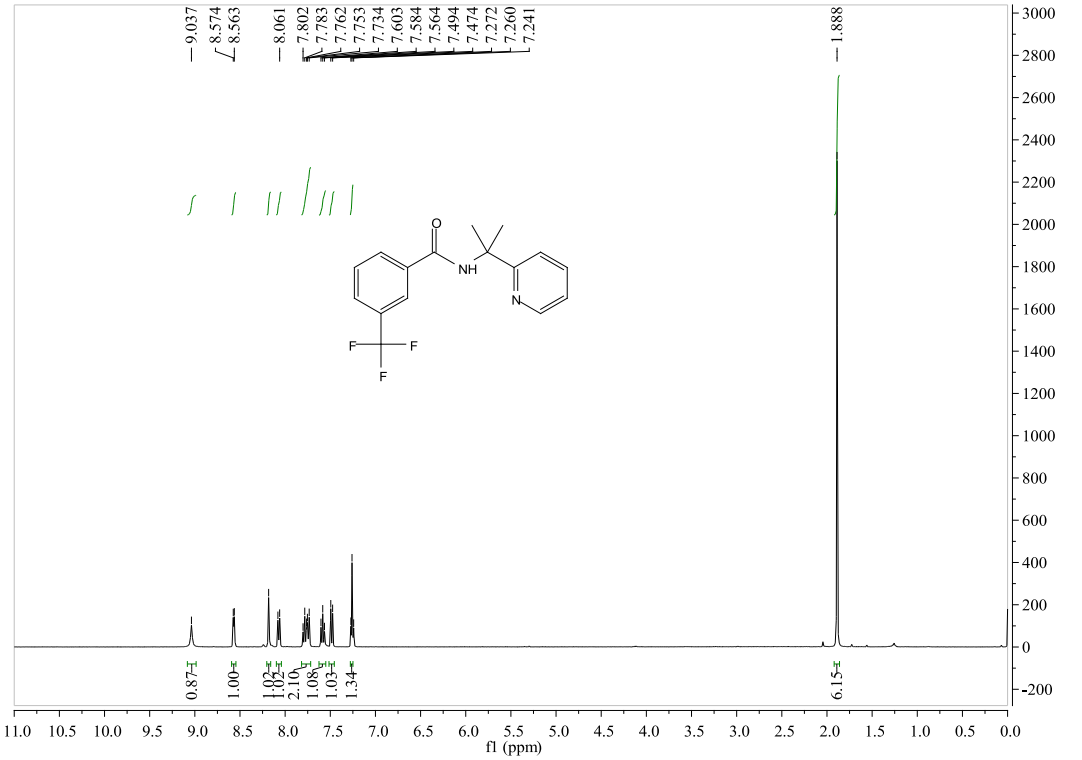
76.84
56.99
27.41
f1 (ppm)

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7.783
7.762
7.753
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7.584
7.564
7.494
7.474
7.272
7.260
7.241
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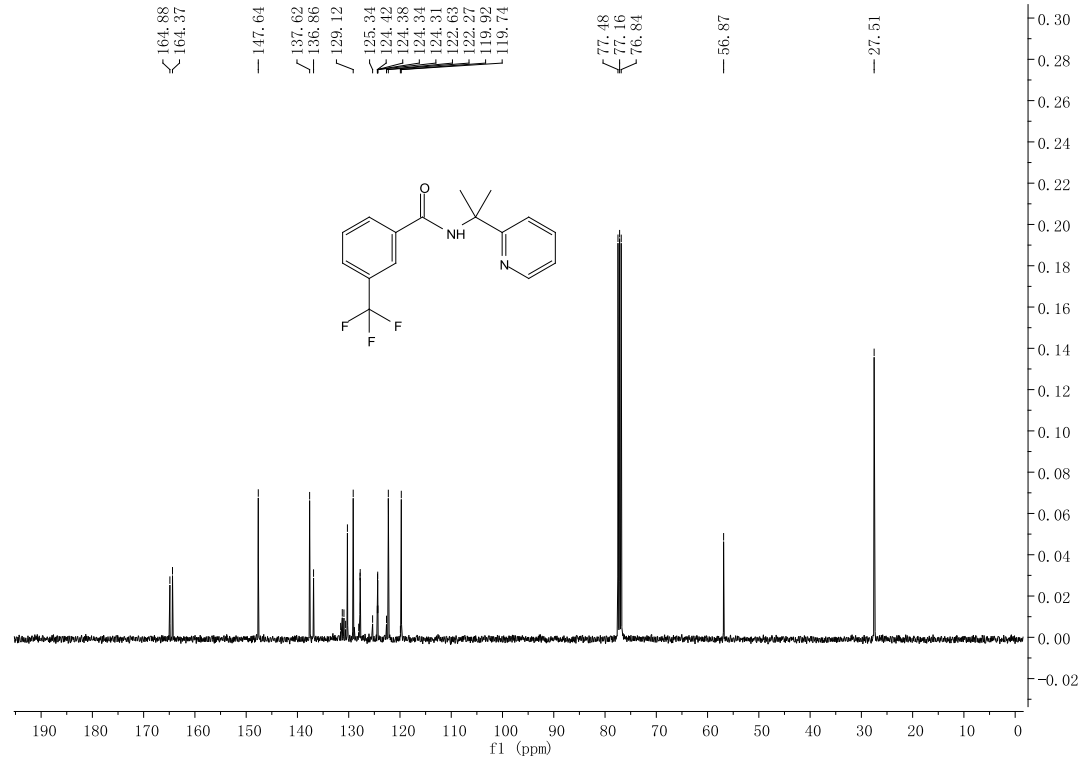
1.00
1.02
1.02
2.10
1.08
1.03
1.34
6.15
f1 (ppm)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129.12
125.34
124.42
124.38
124.34
124.31
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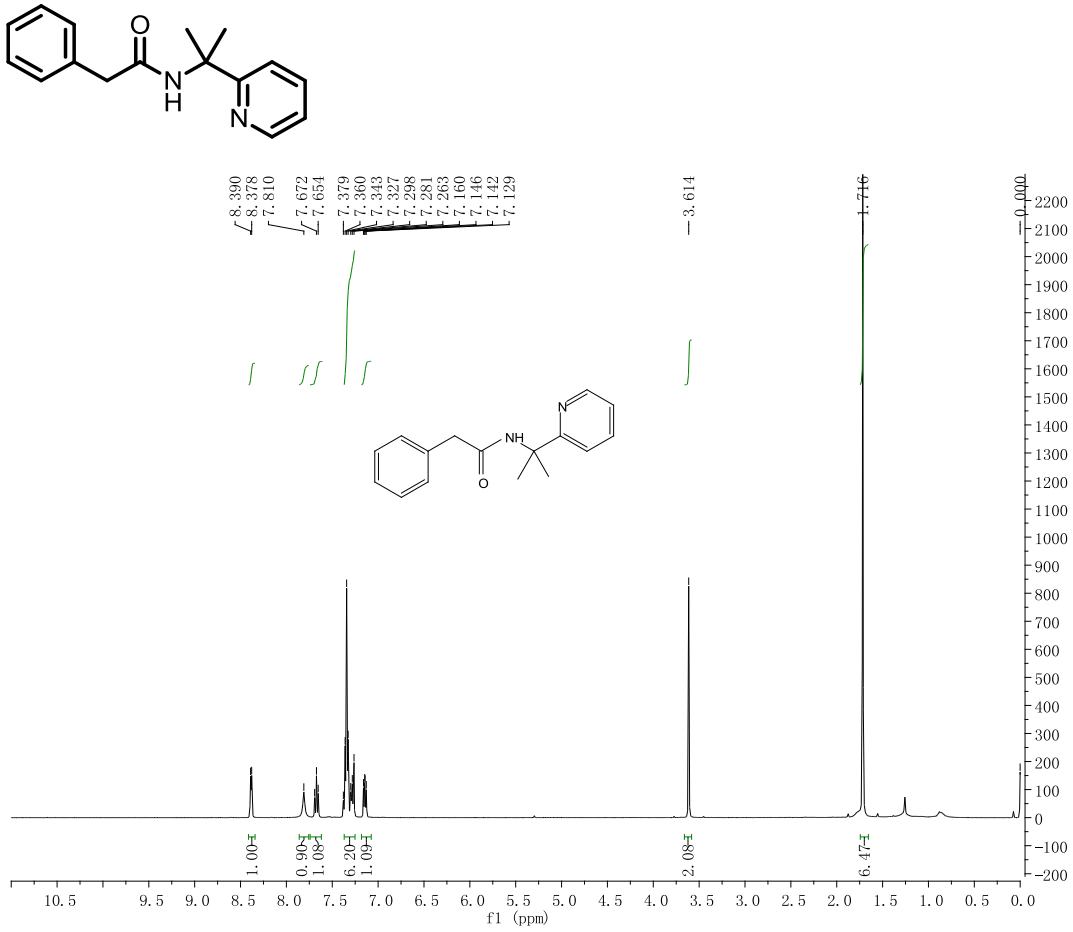
122.27
119.92
119.74
77.48
77.16
76.84
56.87
27.51
f1 (ppm)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7.343
7.327
7.298
7.281
7.263
7.160
7.146
7.142
7.129
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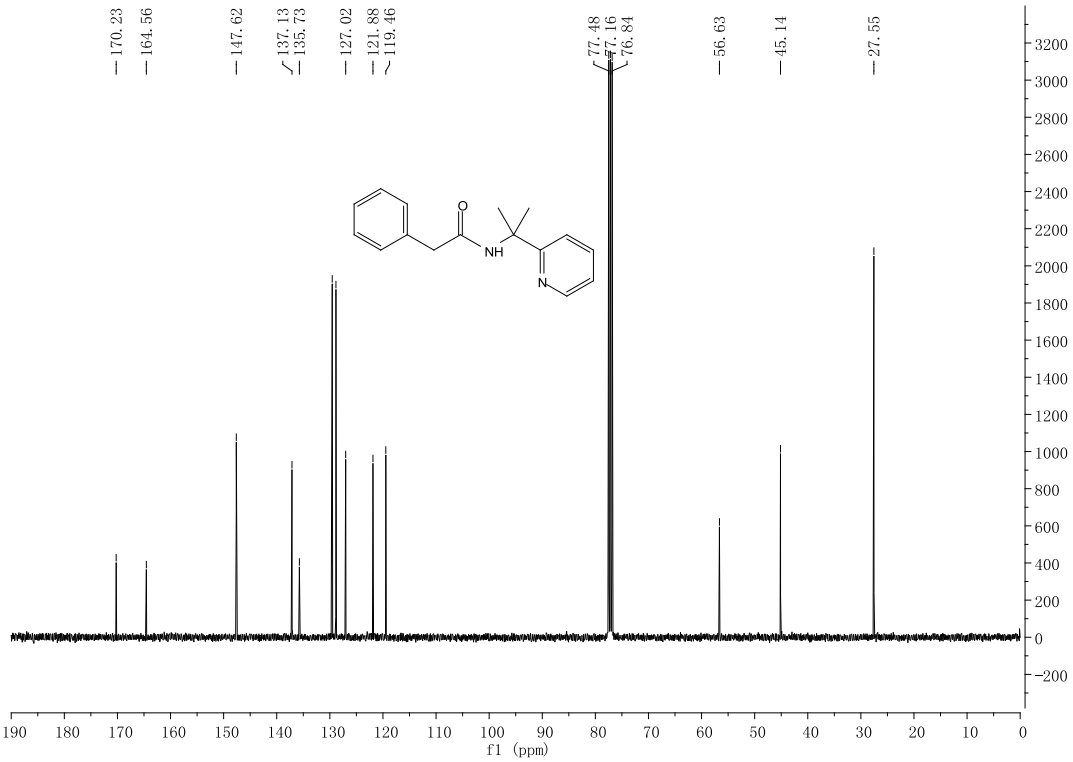
0.000
1.00
0.90
1.08
6.20
1.09
2.08
6.47
f1 (ppm)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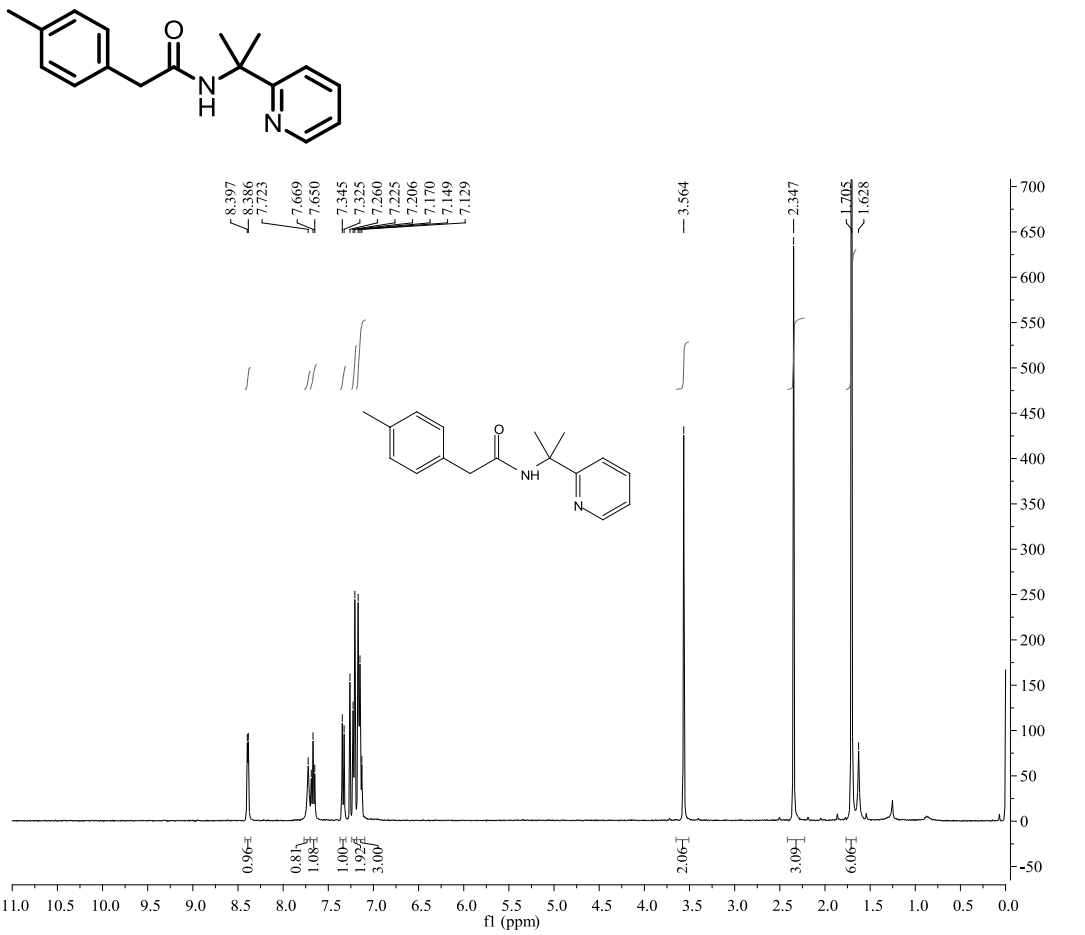
77.16
76.84
56.63
45.14
27.55
f1 (ppm)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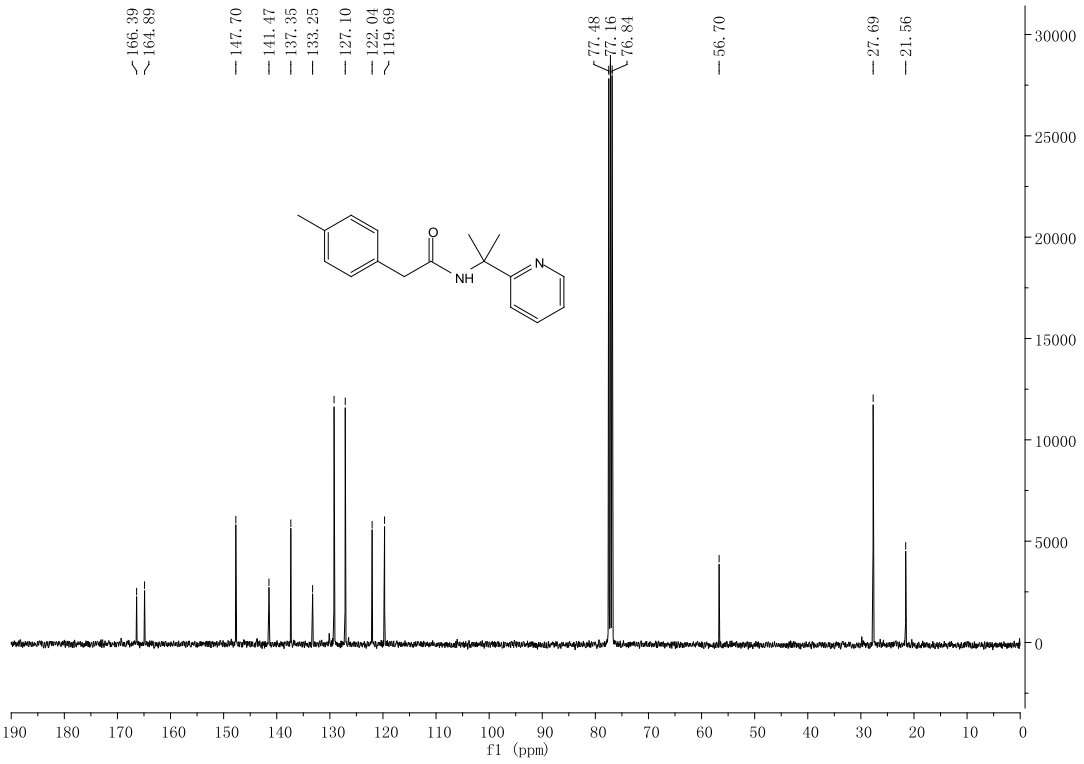
7.325
7.260
7.225
7.206
7.170
7.149
7.129
3.564
2.347
1.705
1.628
0.96
0.81
1.08
1.00
1.92
3.00
2.06
3.09
6.06
fl (ppm)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147.70
141.47
137.35
133.25
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122.04
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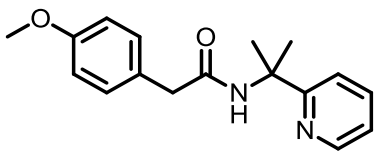
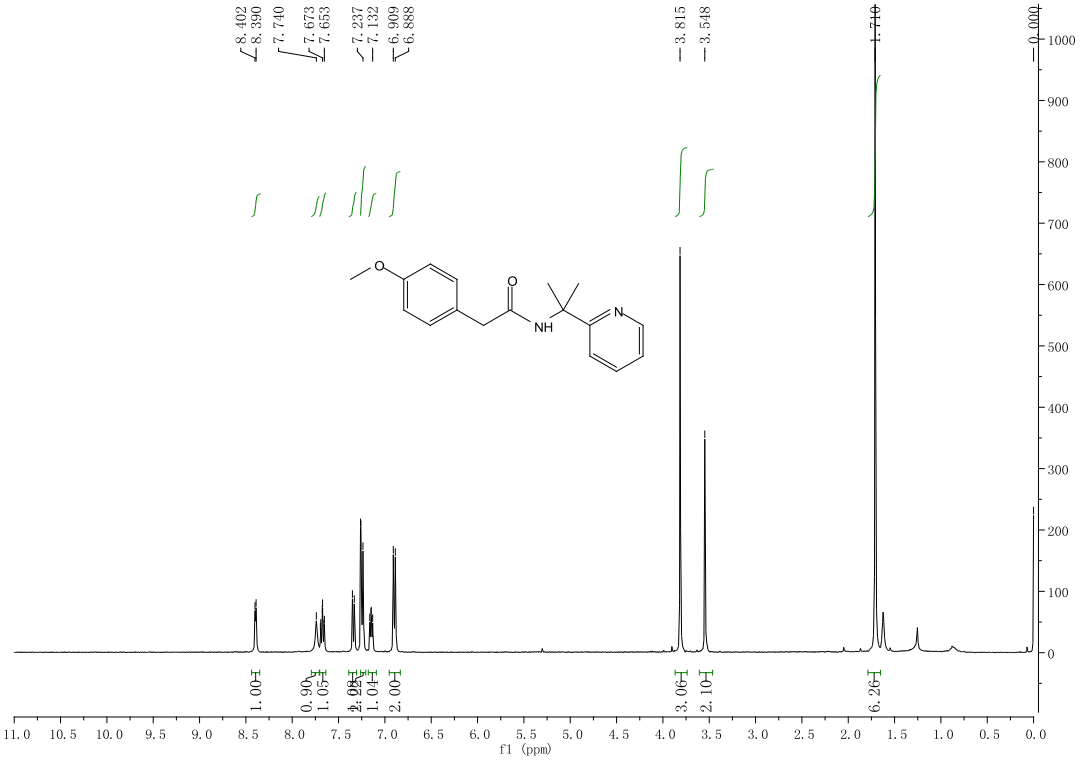
77.48
77.16
76.84
56.70
27.69
21.56
f1 (ppm)

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7.132
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6.888
3.815
3.548
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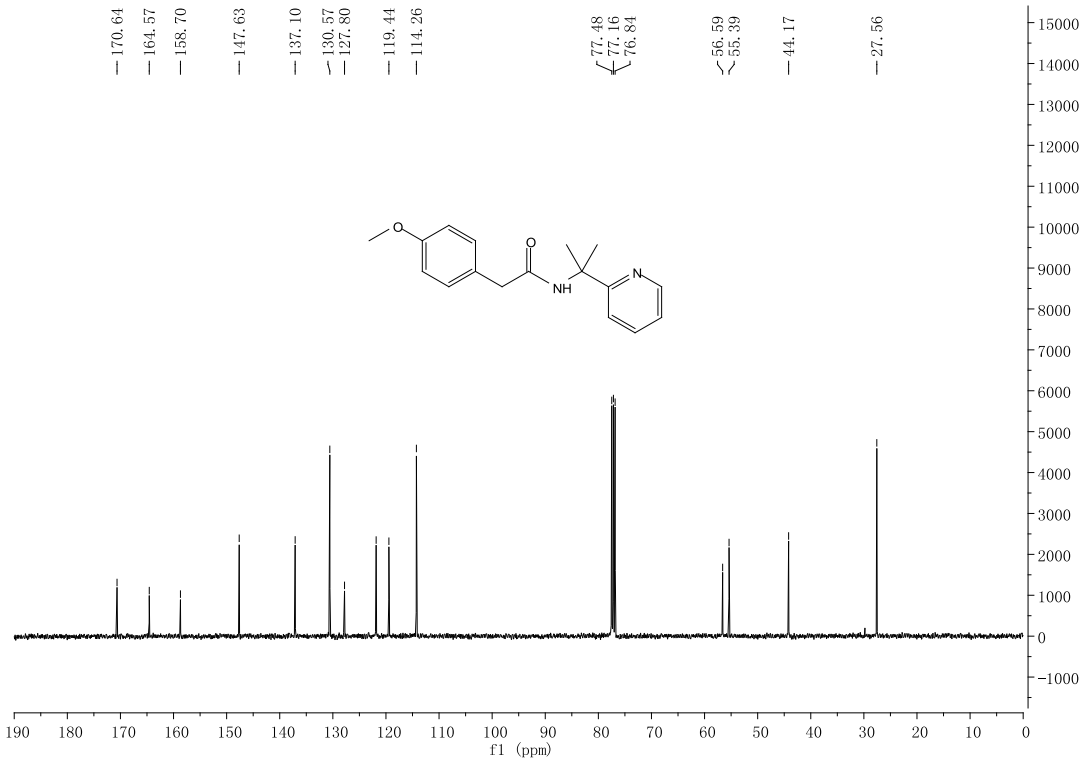
0.000
1.00
0.90
1.05
2.08
1.04
2.00
3.06
2.10
6.26
f1 (ppm)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130.57
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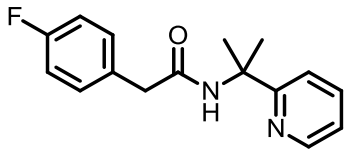
77.16
76.84
56.59
55.39
44.17
27.56
f1 (ppm)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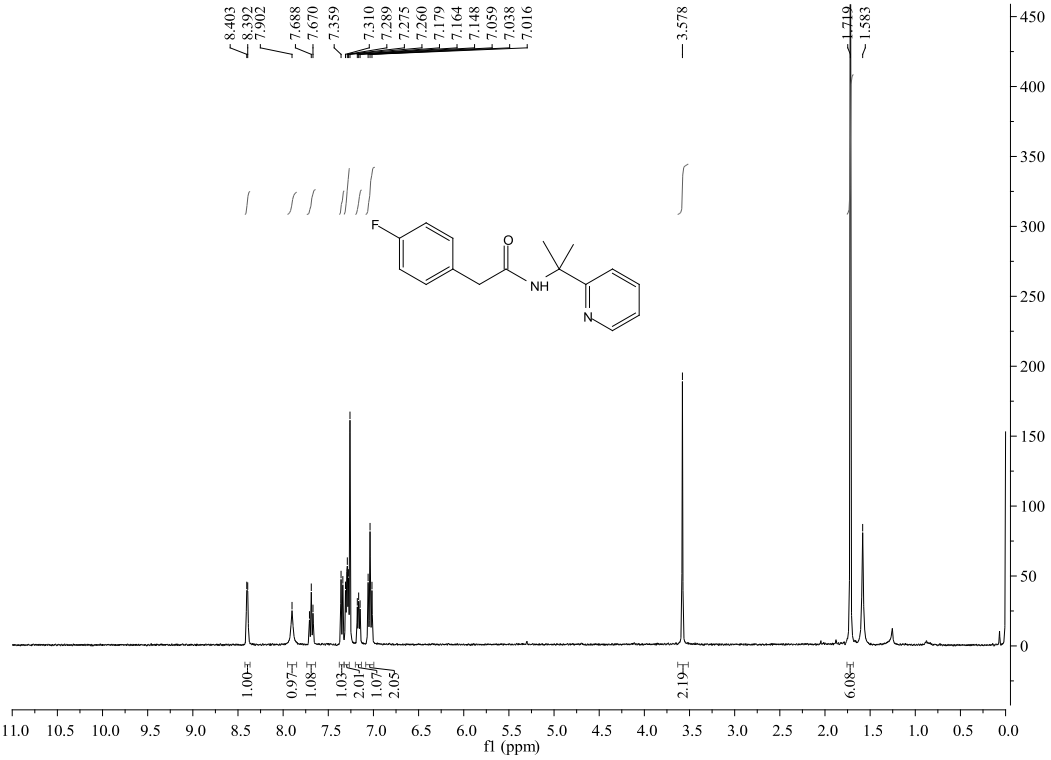
O
N
H
N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7.902
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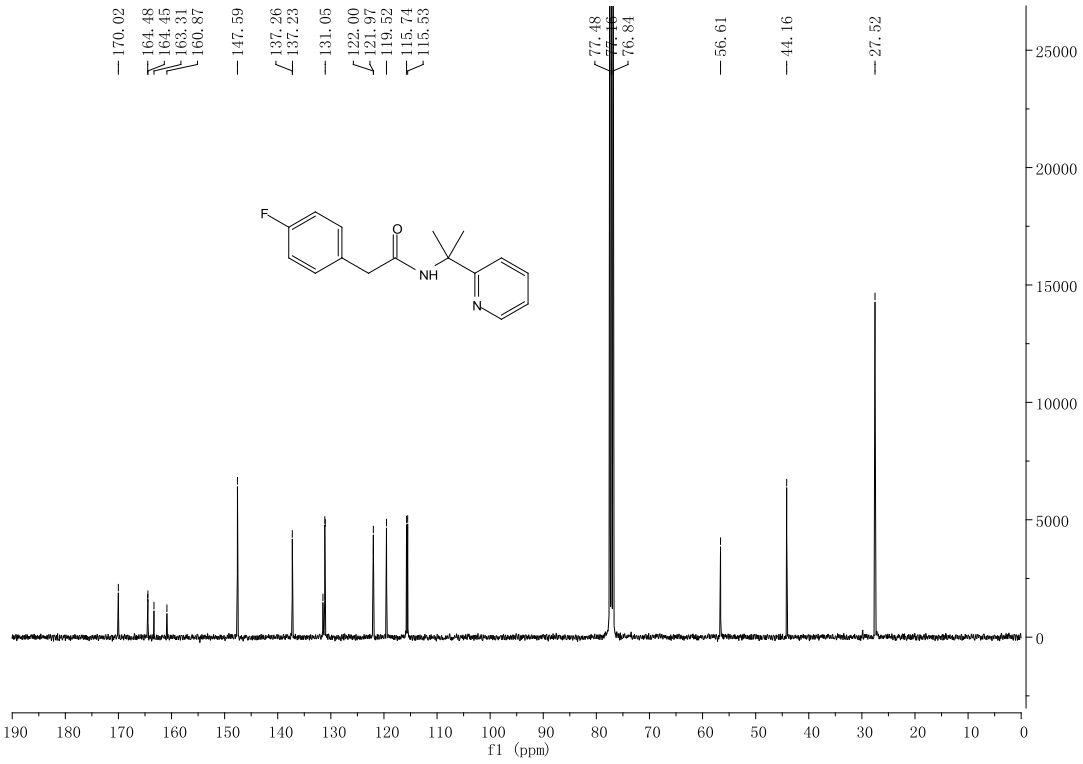
7.670
7.359
7.310
7.289
7.275
7.260
7.179
7.164
7.148
7.059
7.038
7.016
3.578
1.719
1.583
1.00
0.97
1.08
1.03
2.01
1.07
2.05
2.19
6.08
f1 (ppm)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163.31
160.87
147.59
137.26
137.23
131.05
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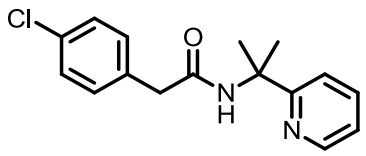
77.48
77.16
76.84
56.61
44.16
27.52
f1 (ppm)


Cl
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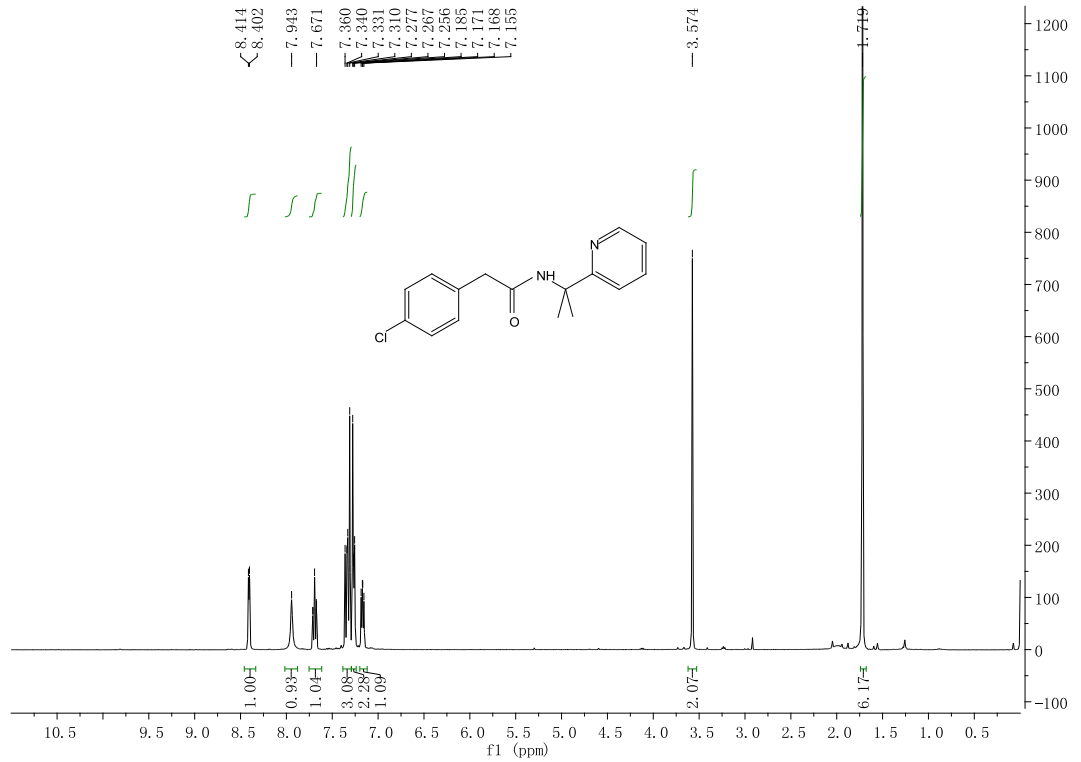
N
H
N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7.185
7.171
7.168
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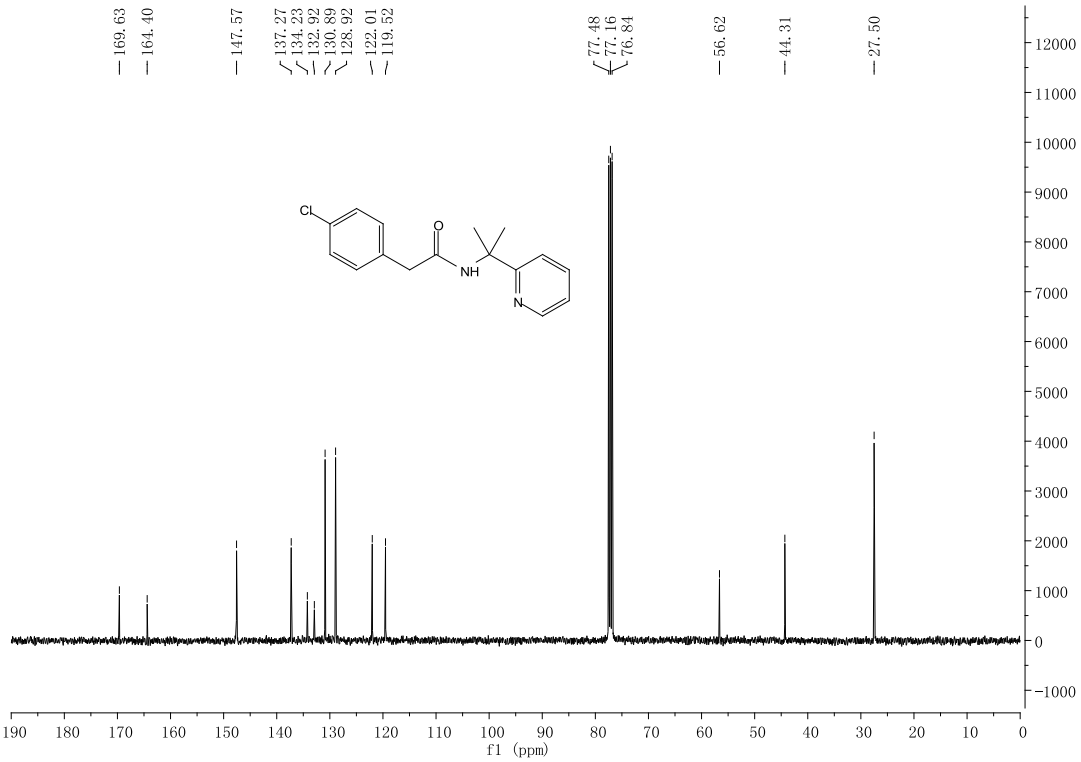
1.00
0.93
1.04
3.08
2.28
1.09
2.07
6.17
f1 (ppm)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134.23
132.92
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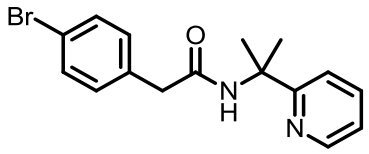
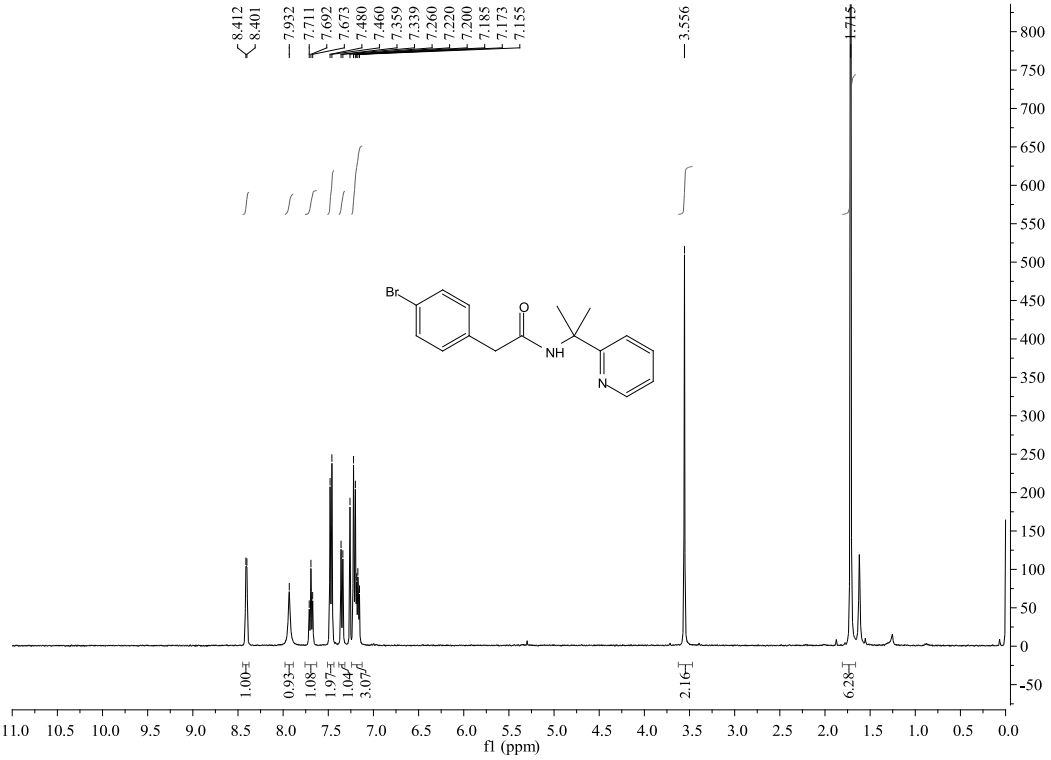
77.48
77.16
76.84
56.62
44.31
27.50
f1 (ppm)


Br
O
N
H
N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7.673
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7.460
7.359
7.339
7.260
7.220
7.200
7.185
7.173
7.155
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1.00
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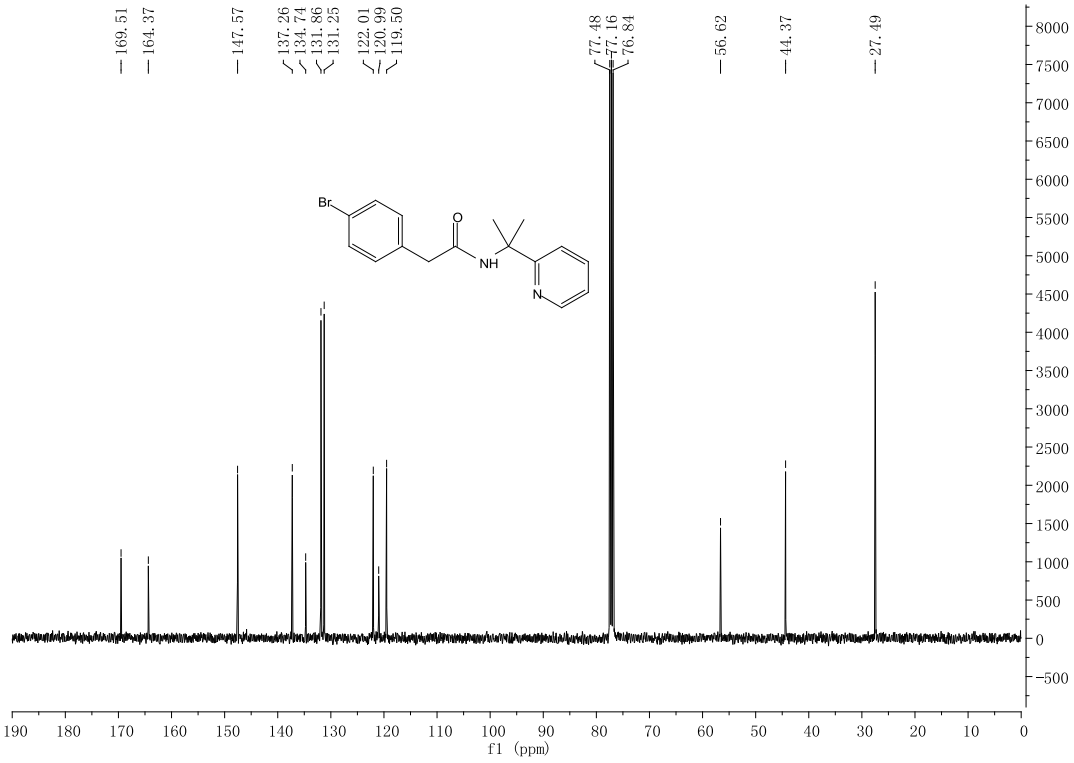
1.08
1.97
1.04
3.07
2.16
6.28
f1 (ppm)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131.25
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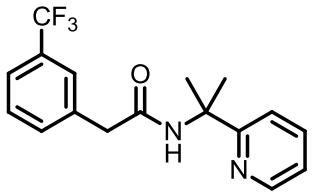
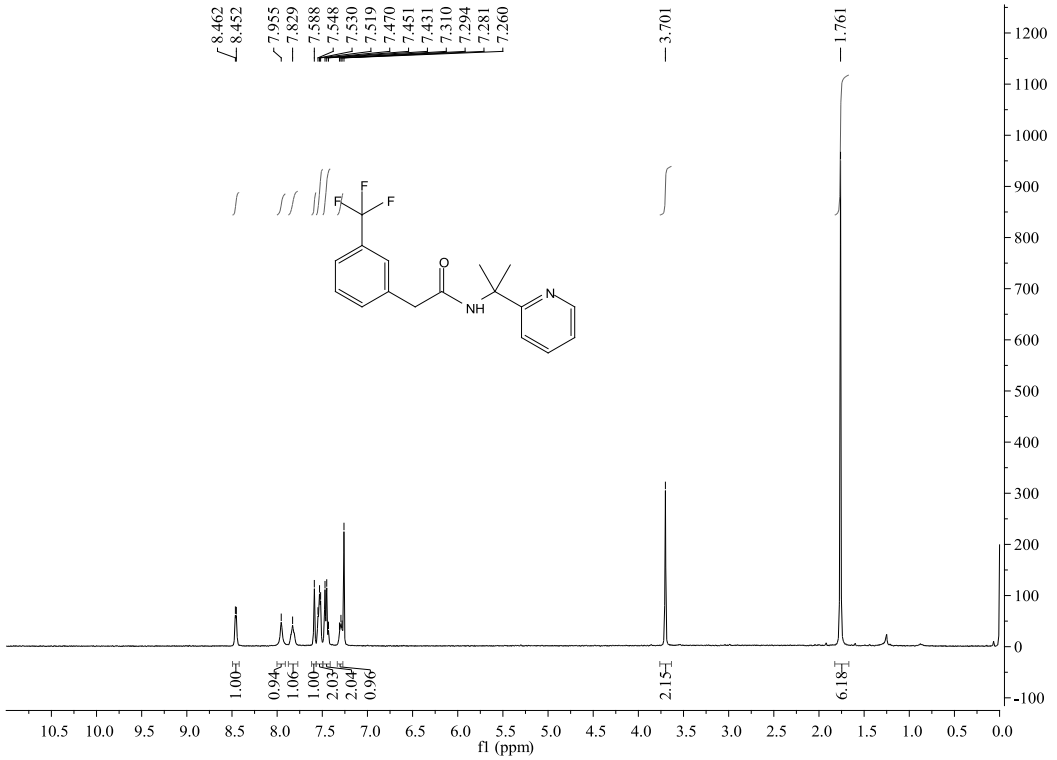
77.48
77.16
76.84
56.62
44.37
27.49
f1 (ppm)


CF3
O
N
H
N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7.310
7.294
7.281
7.260
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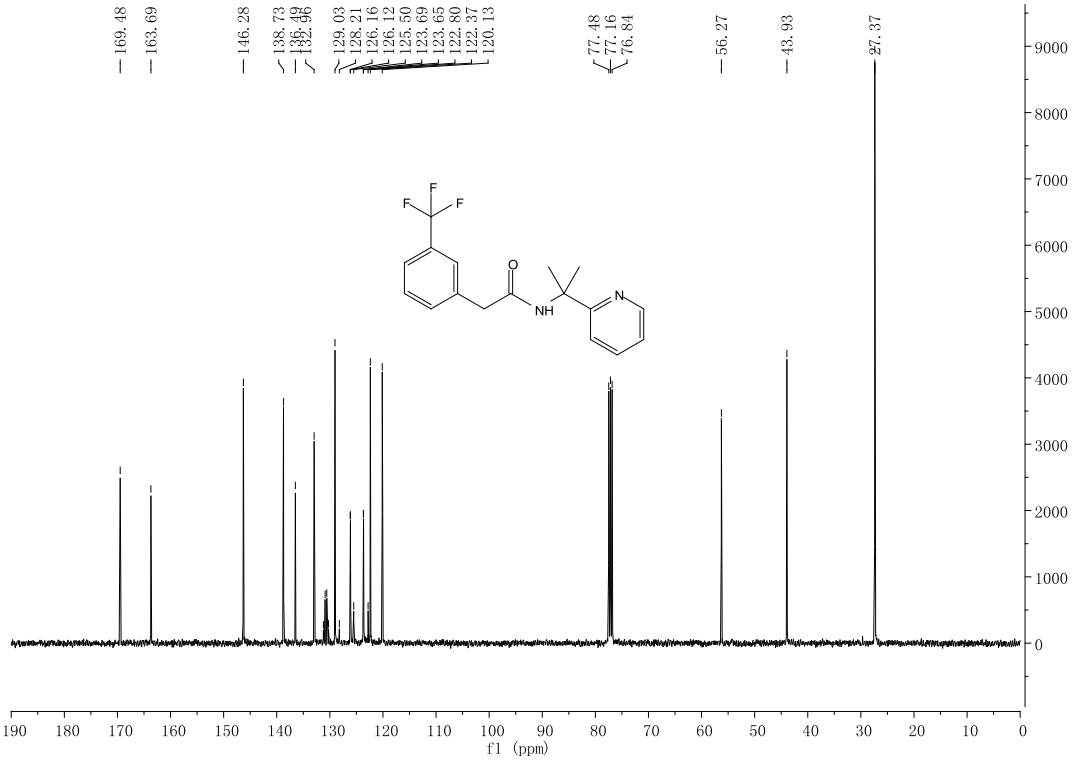
1.00
2.05
2.04
0.96
2.15
6.18
f1 (ppm)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132.96
129.03
128.21
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126.12
125.50
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122.37
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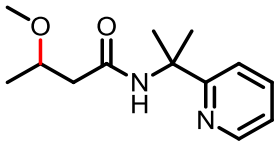
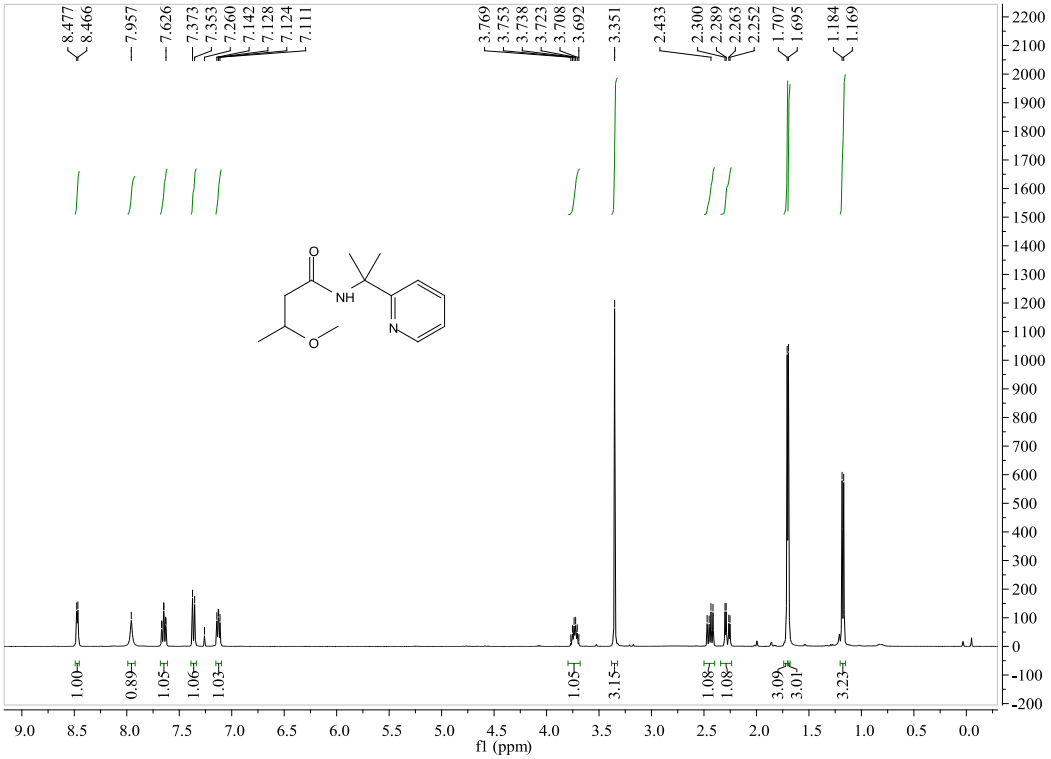
77.48
77.16
76.84
56.27
43.93
27.37
f1 (ppm)

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7.124
7.111
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3.753
3.738
3.723
3.708
3.692
3.351
2.433
2.300
2.289
2.263
2.252
1.707
1.695
1.184
1.169
1.00
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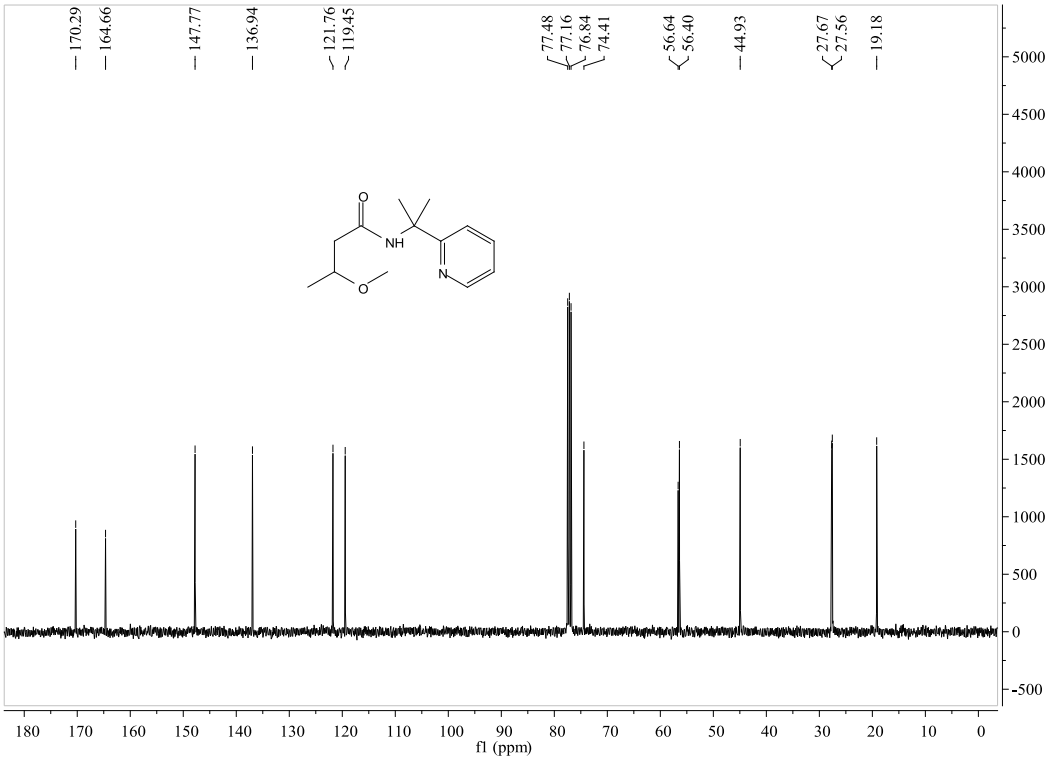
1.06
1.03
1.05
3.15
1.08
1.08
3.09
3.01
3.23
f1 (ppm)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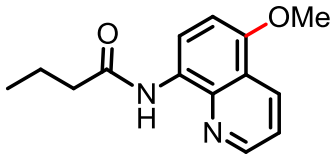
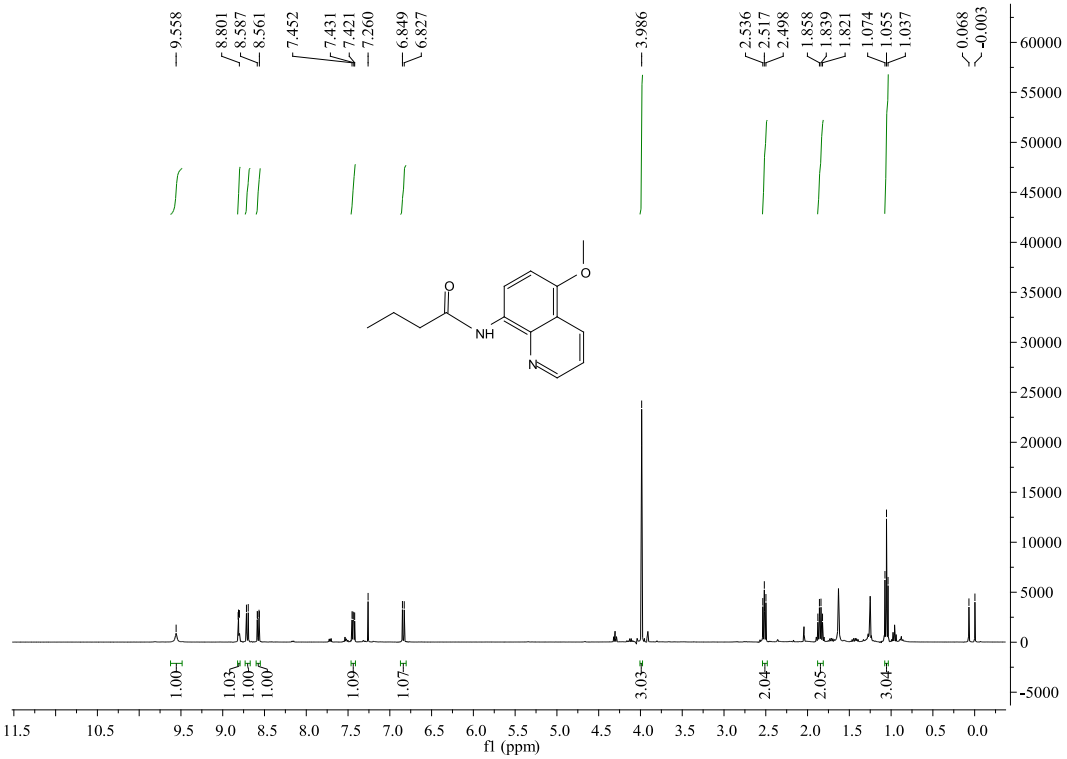
27.56
19.18
f1 (ppm)


OMe
O
N
H
N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7.431
7.421
7.260
6.849
6.827
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2.536
2.517
2.498
1.858
1.839
1.821
1.074
1.055
1.037
0.068
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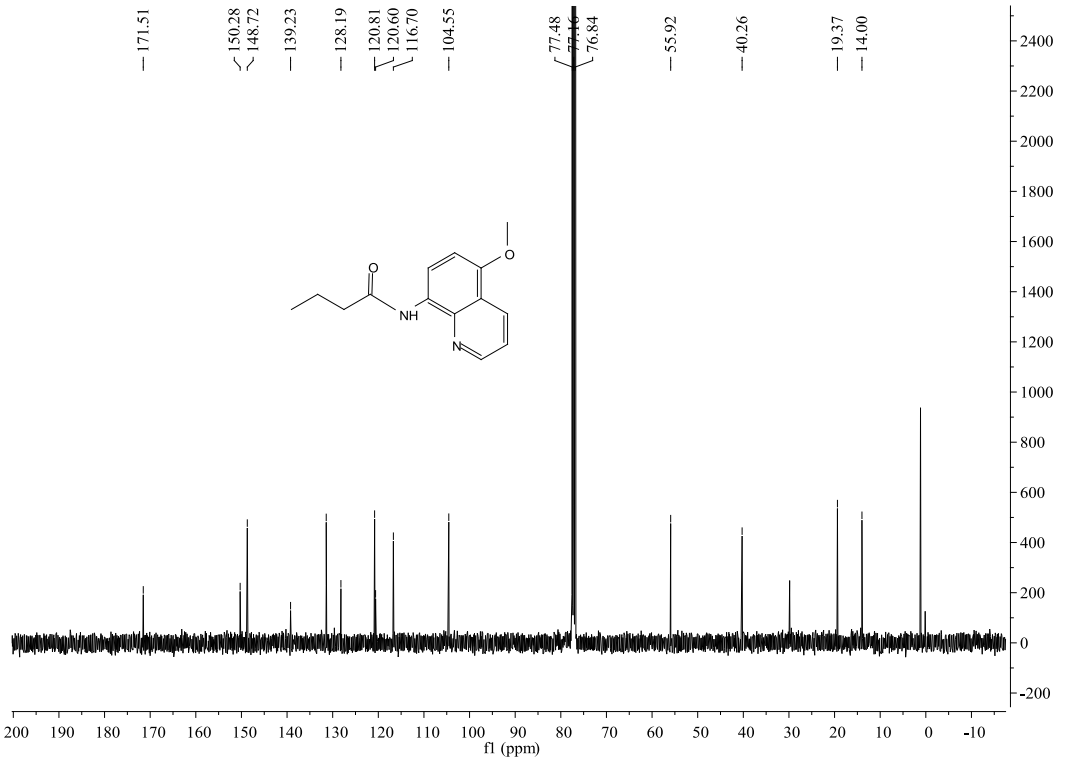
1.03
1.00
1.00
1.09
1.07
3.03
2.04
2.05
3.04
fl (ppm)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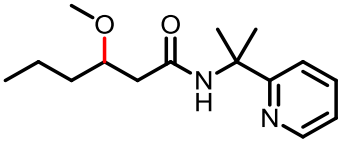
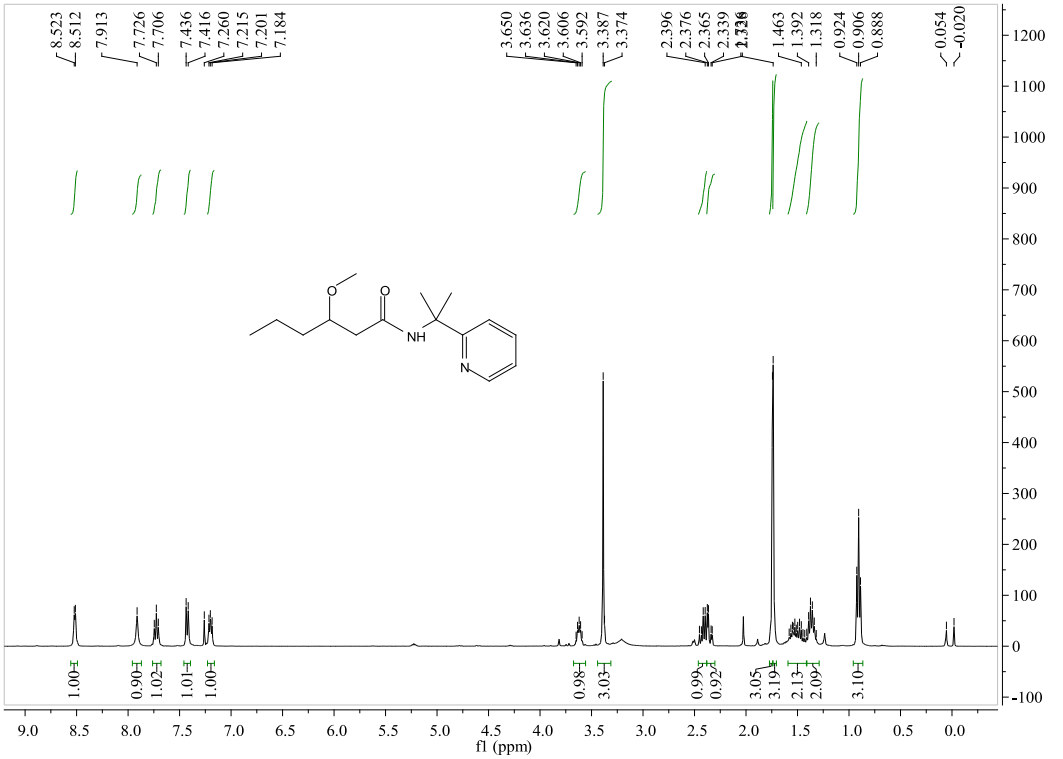
77.16
76.84
55.92
40.26
19.37
14.00
fl (ppm)

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7.260
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7.201
7.184
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3.636
3.620
3.606
3.592
3.387
3.374
2.396
2.376
2.365
2.339
1.463
1.392
1.318
0.924
0.906
0.888
0.054
-0.020
1.00
0.90
1.02
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1.00
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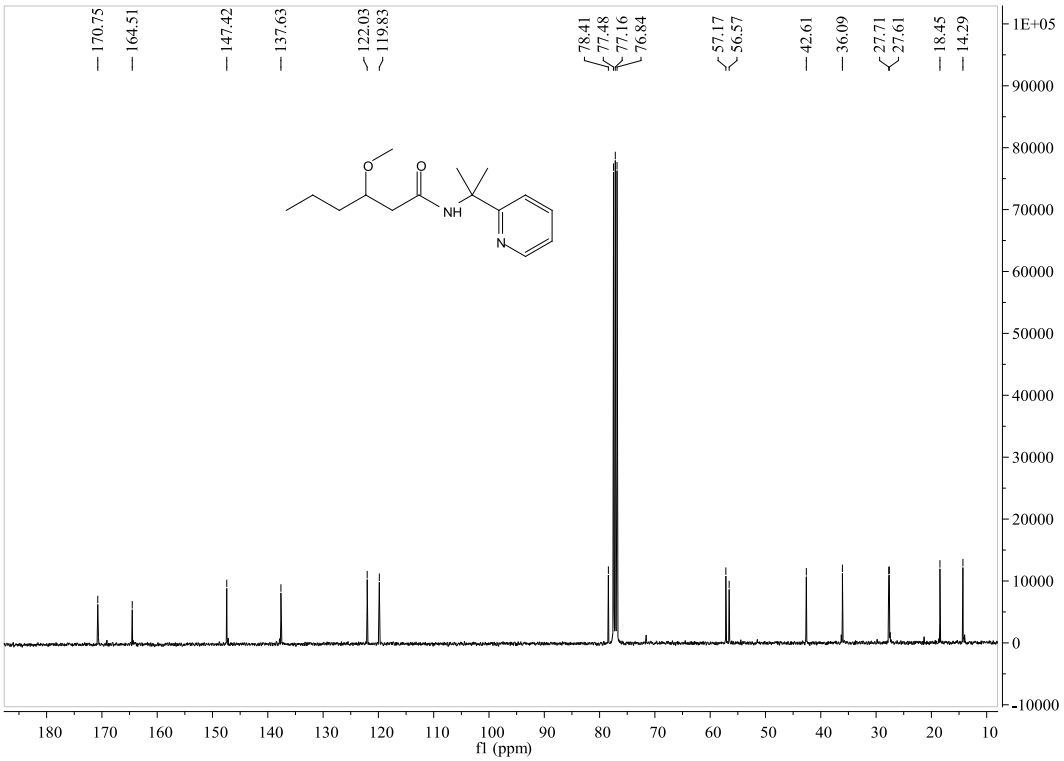
3.03
0.99
0.92
3.05
3.19
2.13
2.09
3.10
f1 (ppm)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77.16
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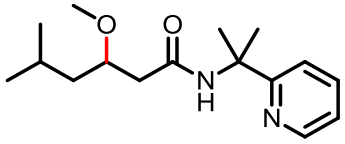
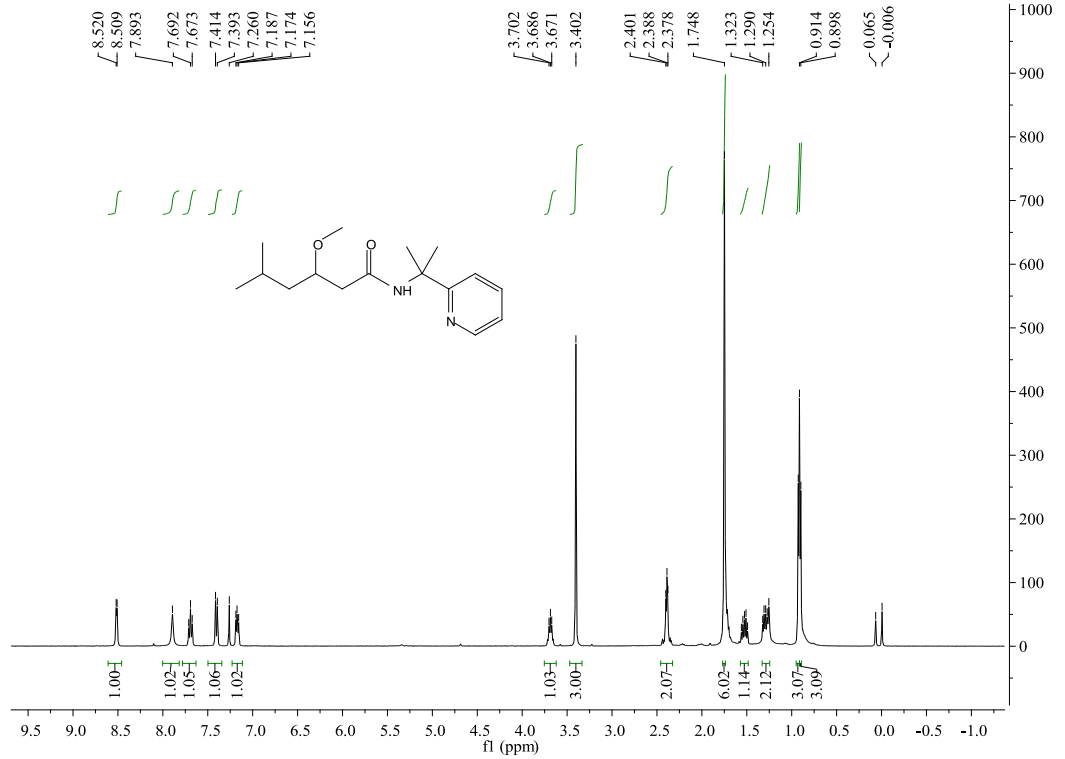
42.61
36.09
27.71
27.61
18.45
14.29
f1 (ppm)

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3.671
3.402
2.401
2.388
2.378
1.748
1.323
1.290
1.254
0.914
0.898
0.065
-0.006
1.00
1.02
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1.03
3.00
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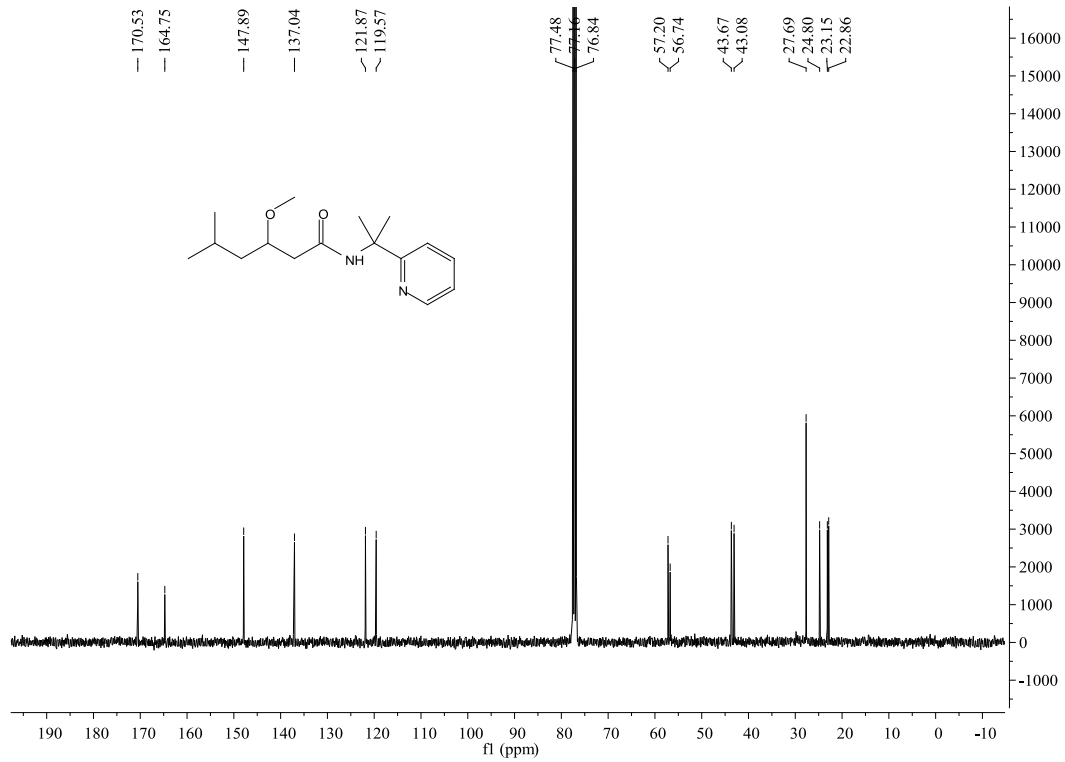
6.02
1.14
2.12
3.07
3.09
f1 (ppm)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57.20
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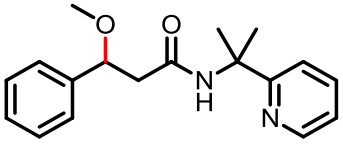
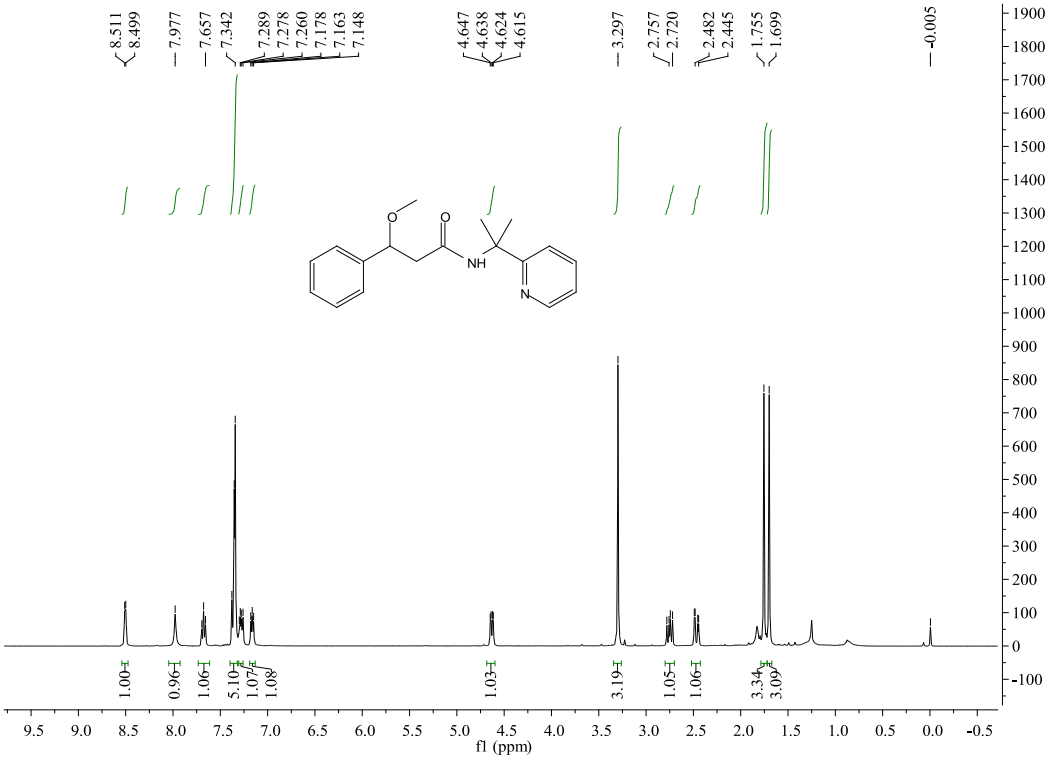
27.69
24.80
23.15
22.86
f1 (ppm)

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7.260
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7.148
4.647
4.638
4.624
4.615
3.297
2.757
2.720
2.482
2.445
1.755
1.699
-0.005
1.00
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1.07
1.08
1.03
3.19
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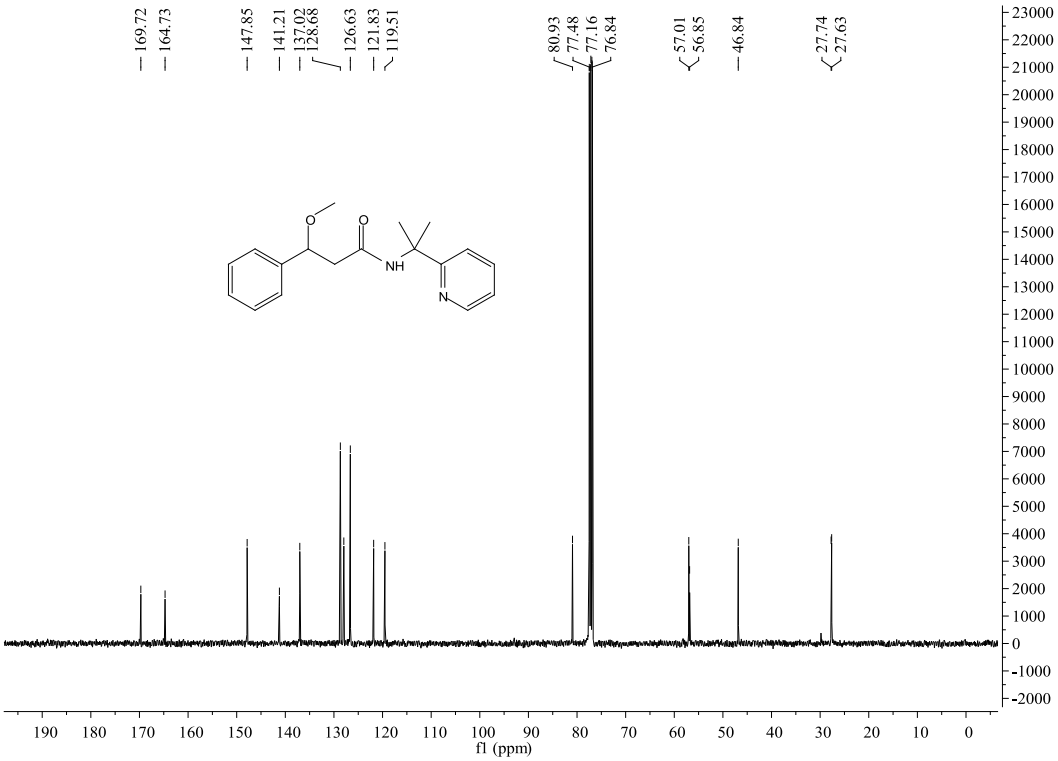
1.06
3.34
3.09
f1 (ppm)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137.02
128.68
126.63
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76.84
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46.84
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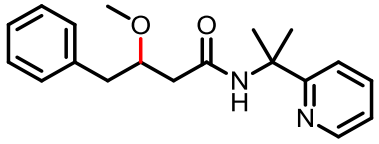
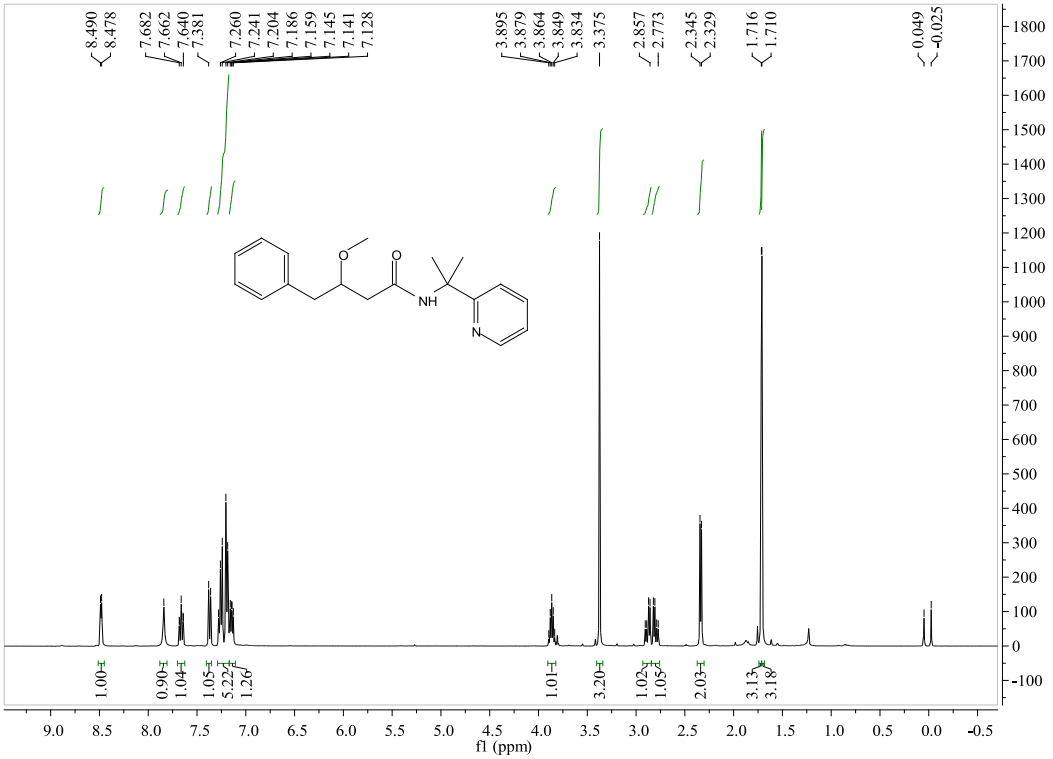
27.63
f1 (ppm)

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3.849
3.834
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2.773
2.345
2.329
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1.710
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0.90
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1.05
2.03
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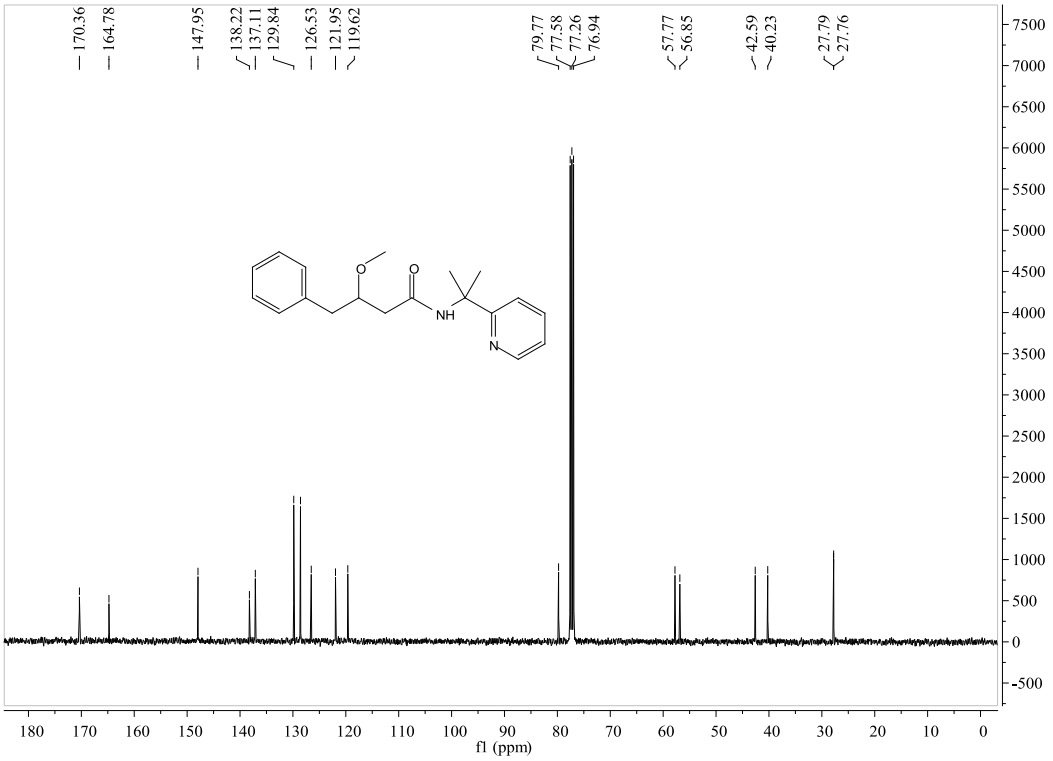
f1 (ppm)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40.23
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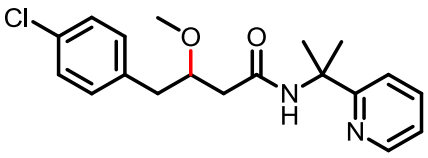
27.76
f1 (ppm)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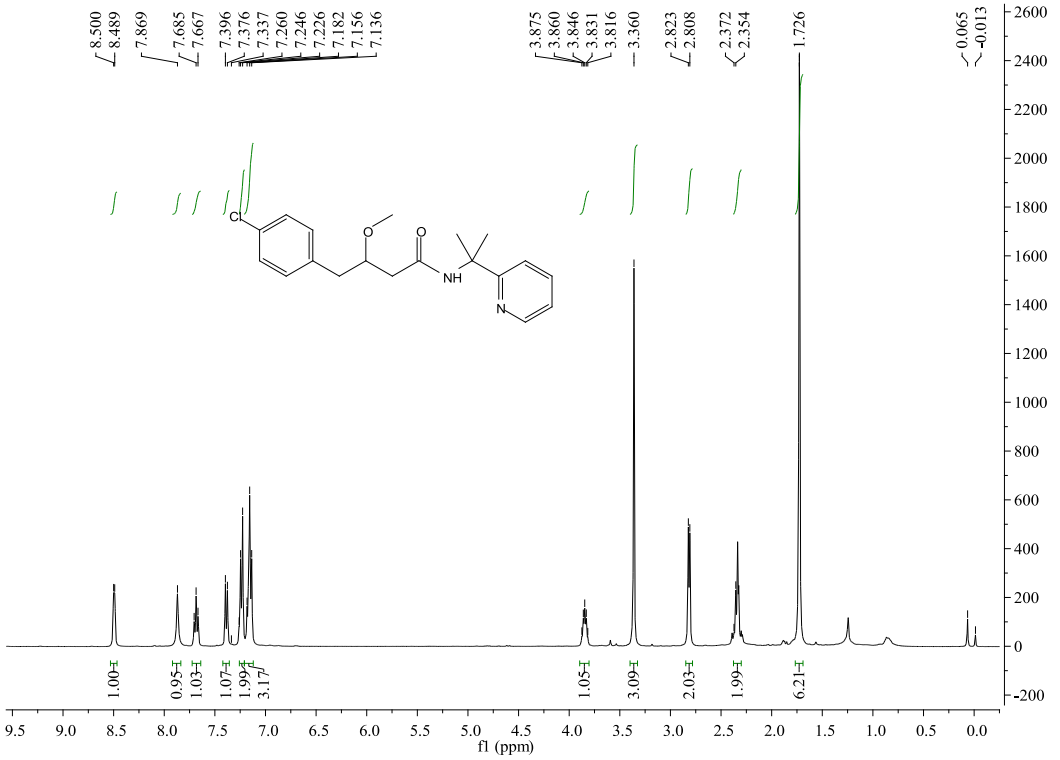
O
N
H
N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7.156
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3.860
3.846
3.831
3.816
3.360
2.823
2.808
2.372
2.354
1.726
0.065
-0.013
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0.95
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1.05
3.09
2.03
1.99
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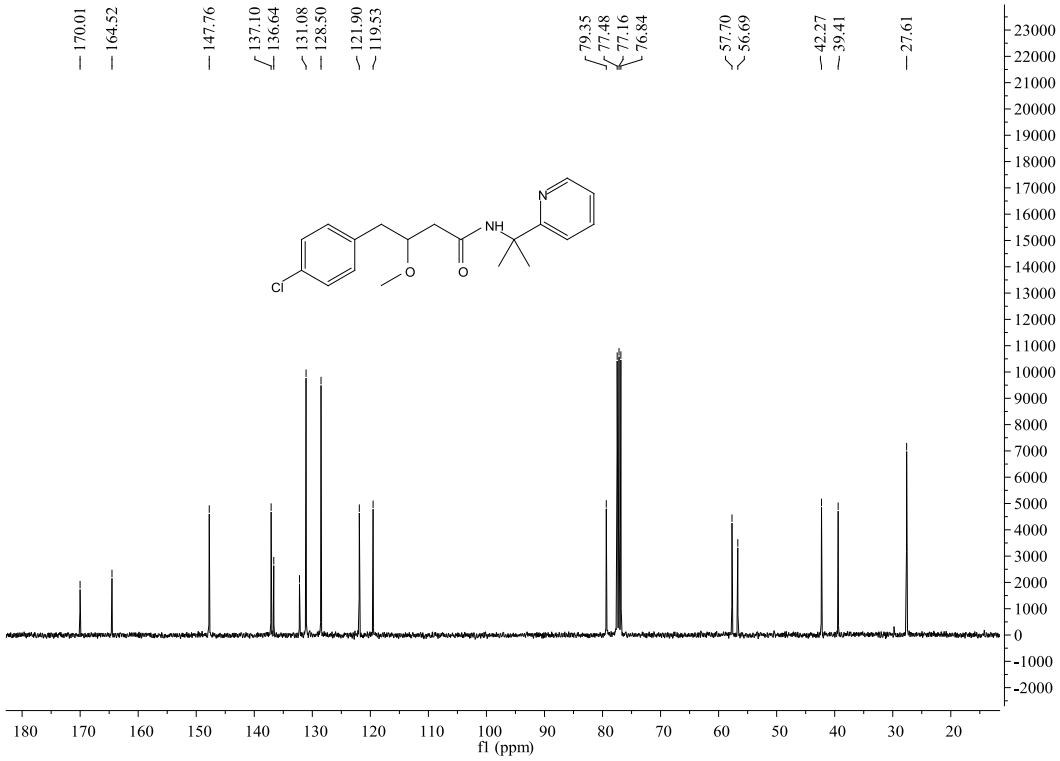
f1 (ppm)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42.27
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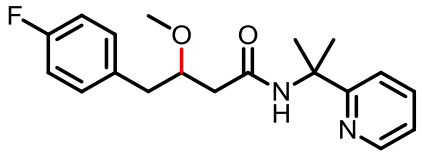
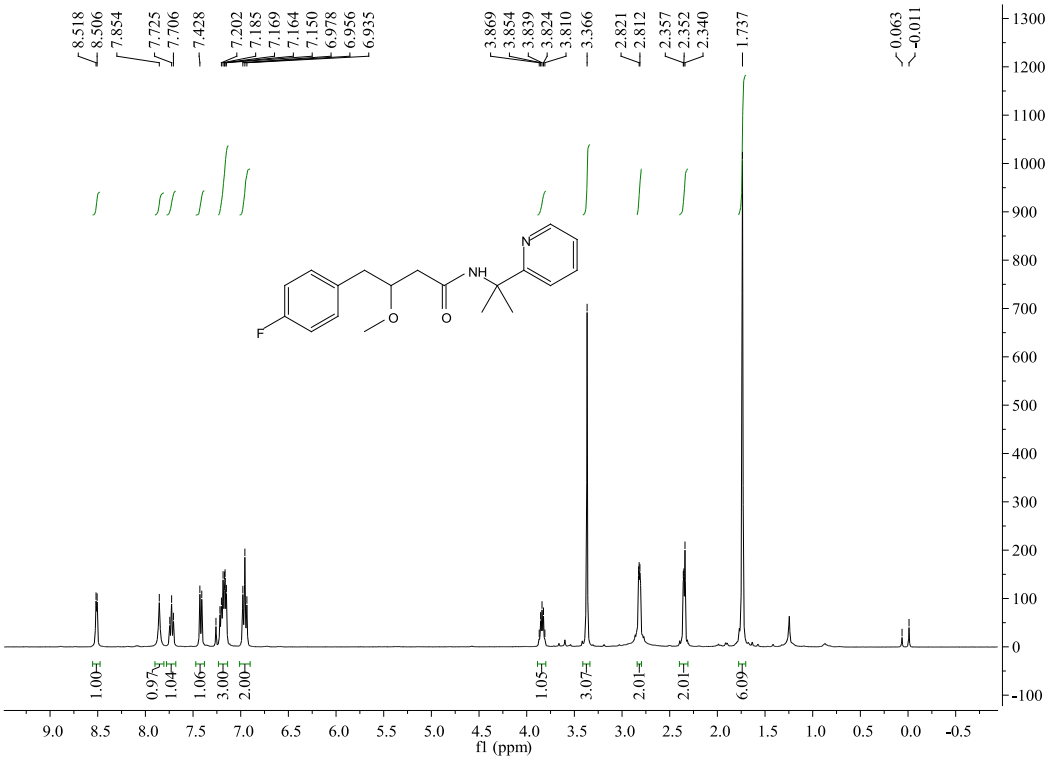
27.61
f1 (ppm)


F
O
O
N
H
N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7.164
7.150
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3.869
3.854
3.839
3.824
3.810
3.366
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2.812
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2.352
2.340
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3.07
2.01
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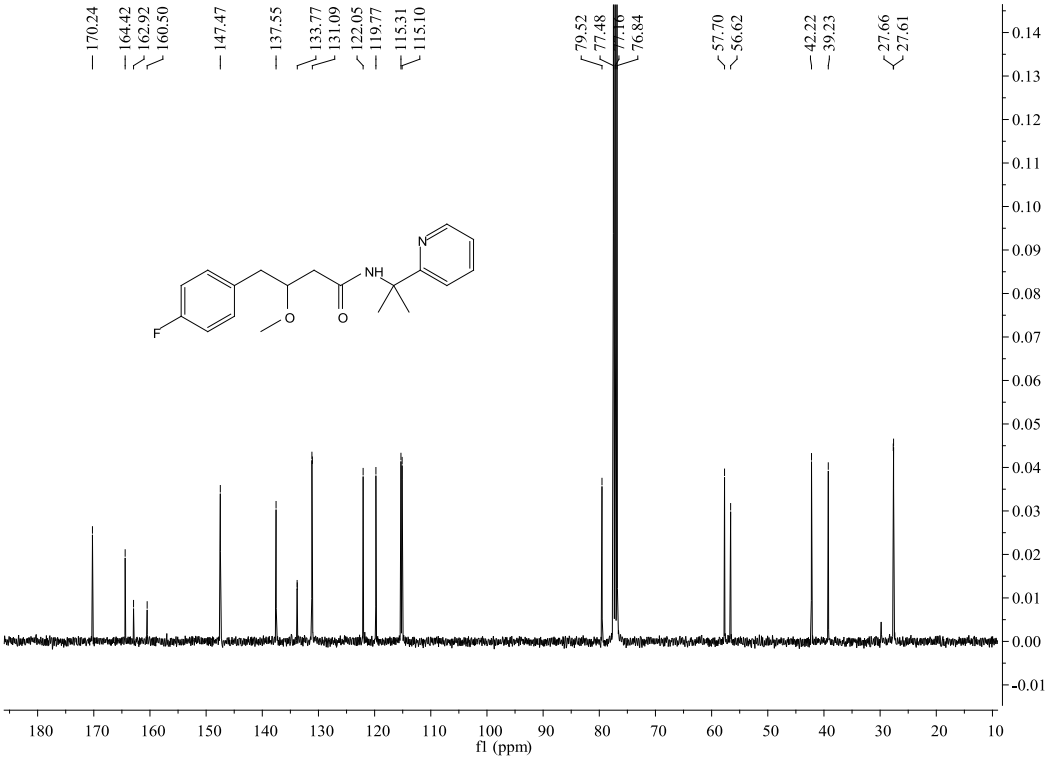
6.09
f1 (ppm)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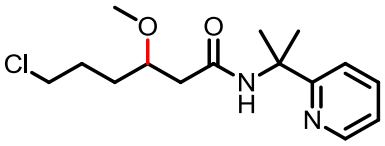
42.22
39.23
27.66
27.61
f1 (ppm)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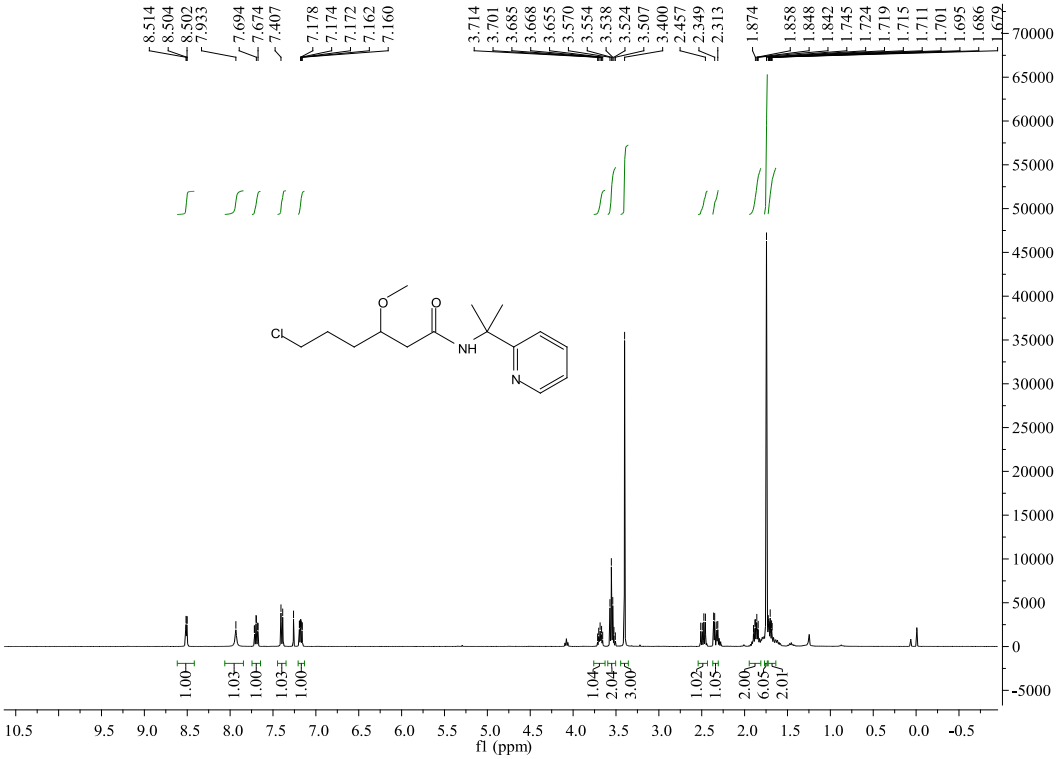
O
N
H
N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3.685
3.668
3.655
3.570
3.554
3.538
3.524
3.507
3.400
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2.313
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1.858
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1.724
1.719
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1.701
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1.686
1.679
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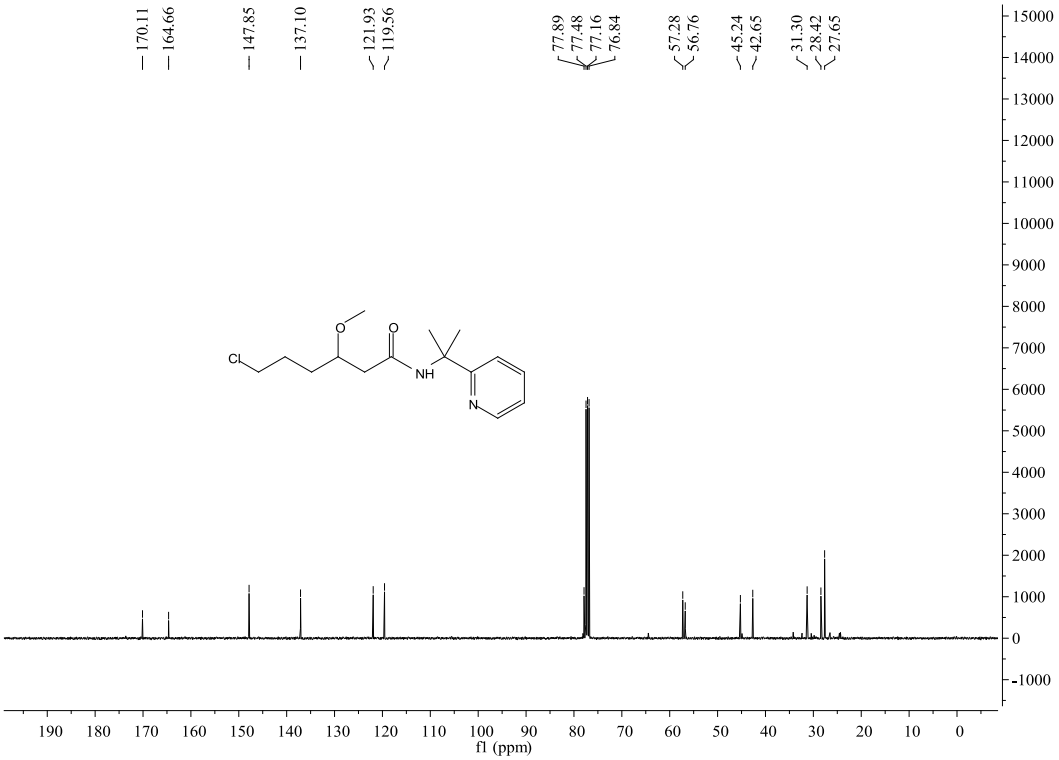
f1 (ppm)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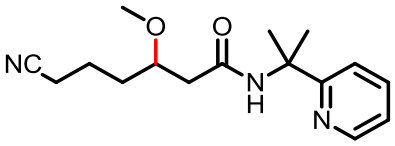
27.65
f1 (ppm)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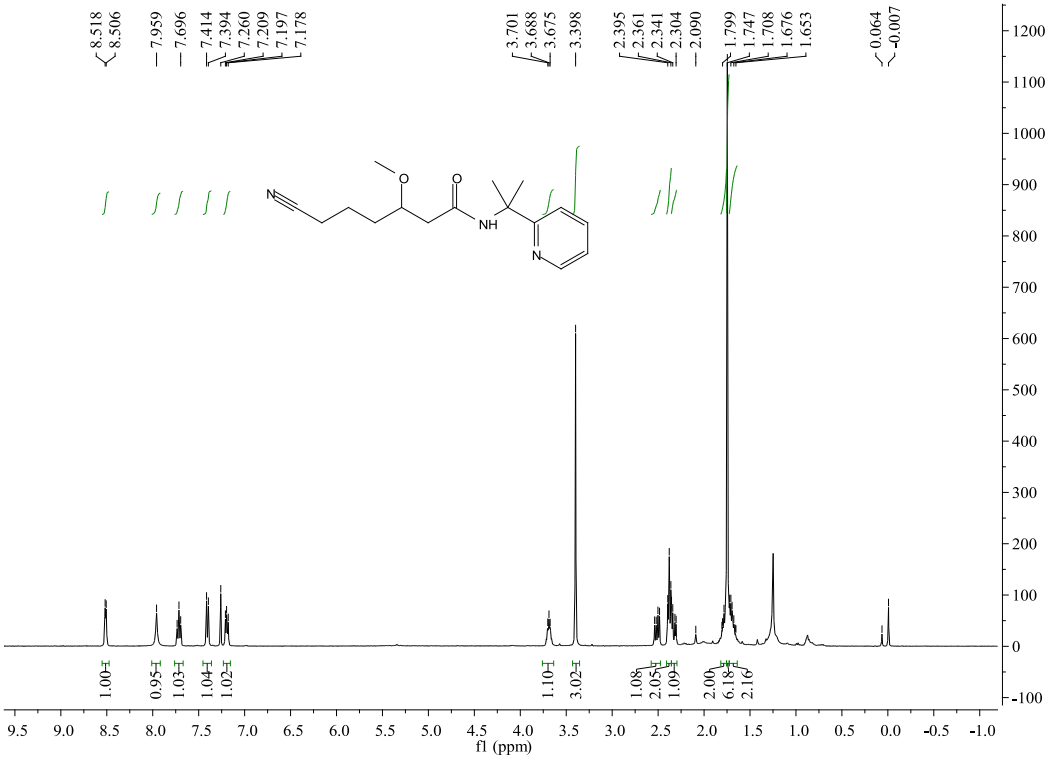
O
N
H
N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7.209
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7.178
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3.398
2.395
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2.341
2.304
2.090
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1.747
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1.653
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-0.007
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0.95
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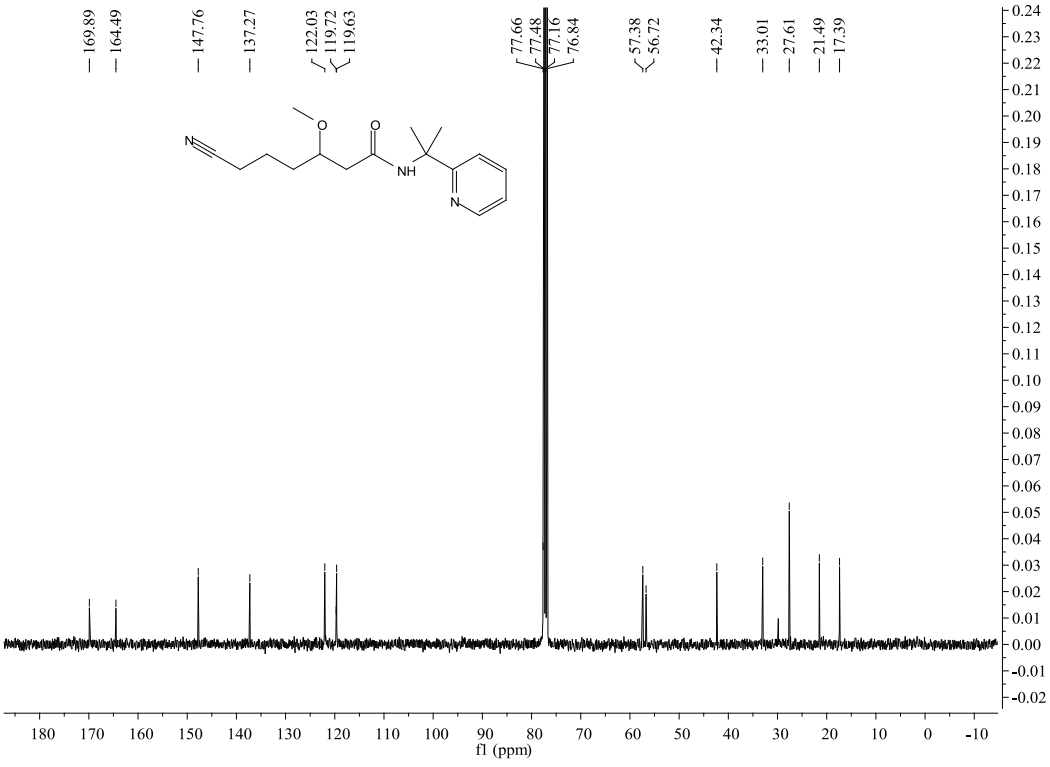
1.04
1.02
1.10
3.02
1.08
2.05
1.09
2.00
6.18
2.16
f1 (ppm)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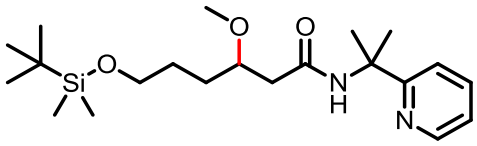
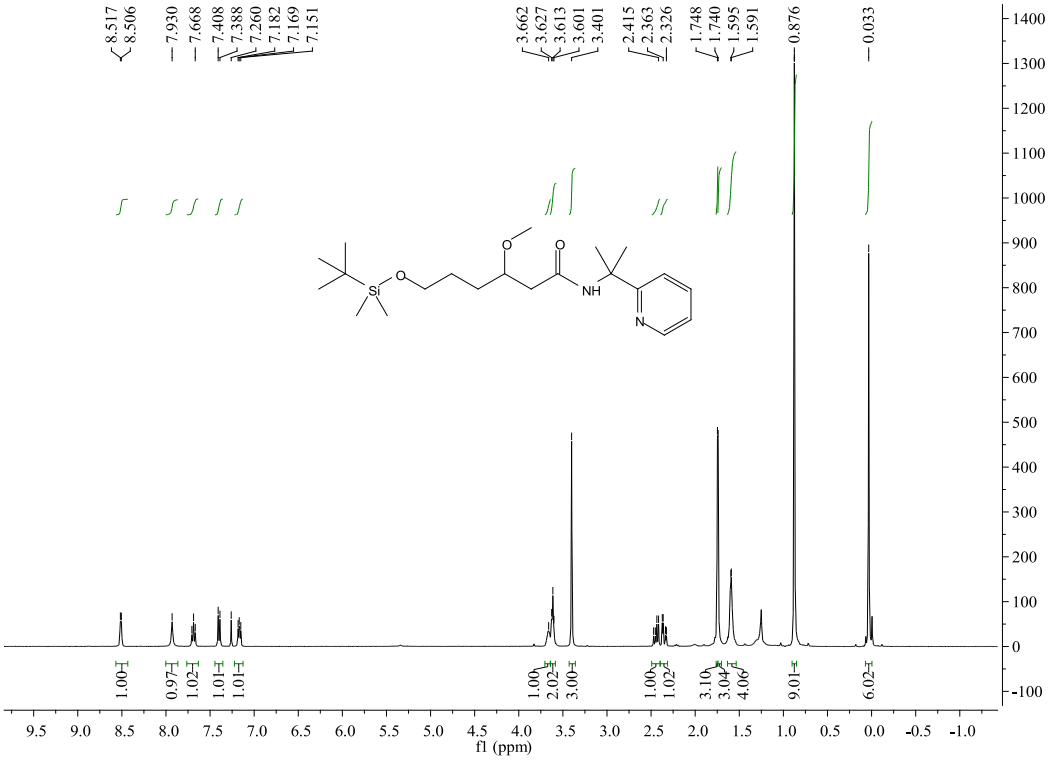
33.01
27.61
21.49
17.39
f1 (ppm)

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7.151
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3.601
3.401
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2.363
2.326
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1.591
0.876
0.033
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0.97
1.02
1.01
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1.02
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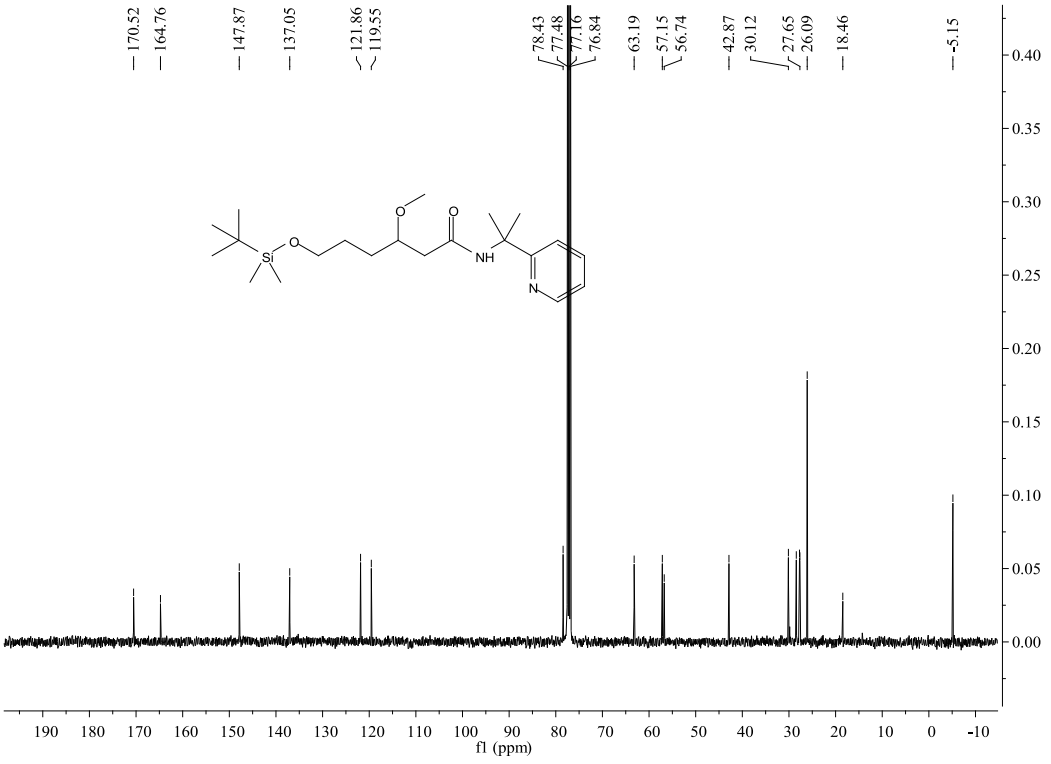
3.04
4.06
9.01
6.02
f1 (ppm)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26.09
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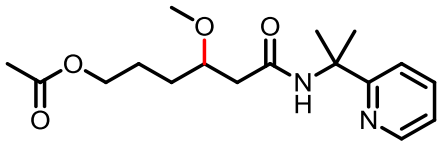
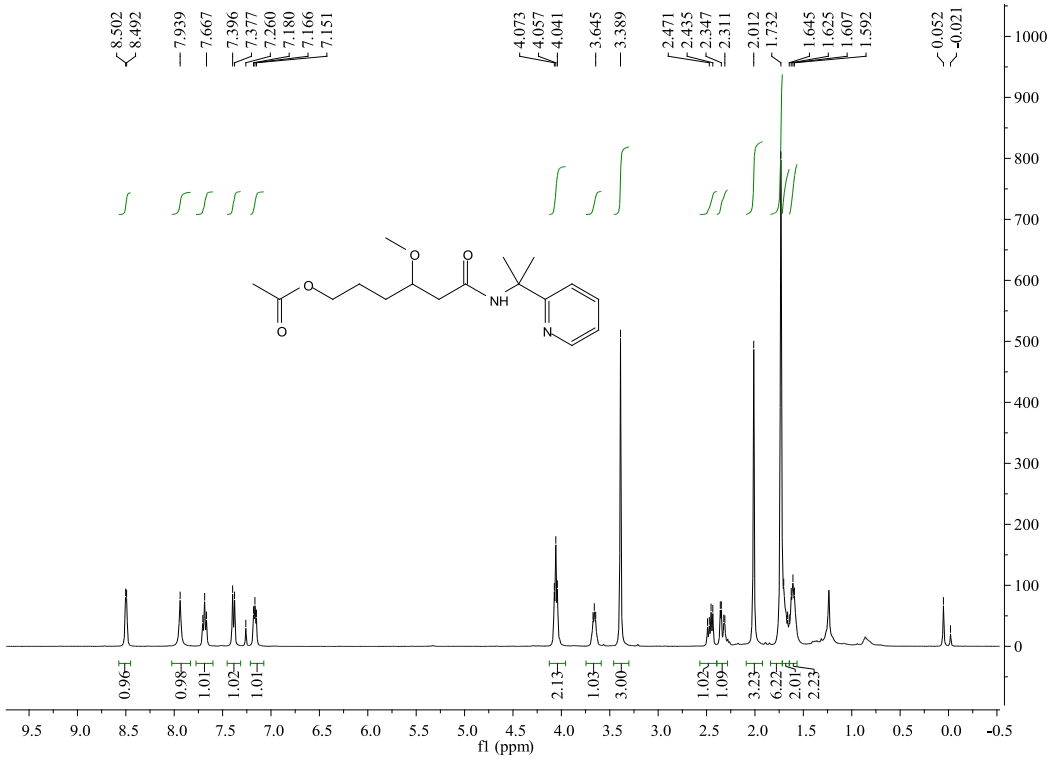
f1 (ppm)

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1.592
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0.98
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6.22
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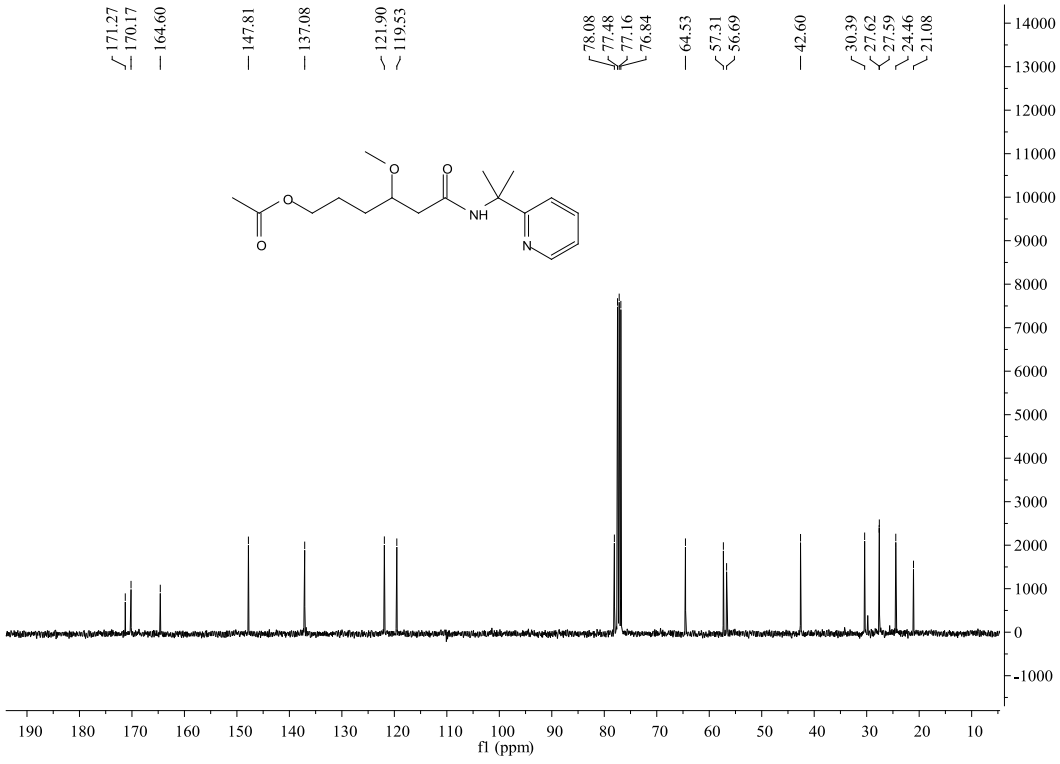
2.23
f1 (ppm)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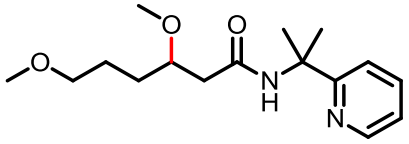
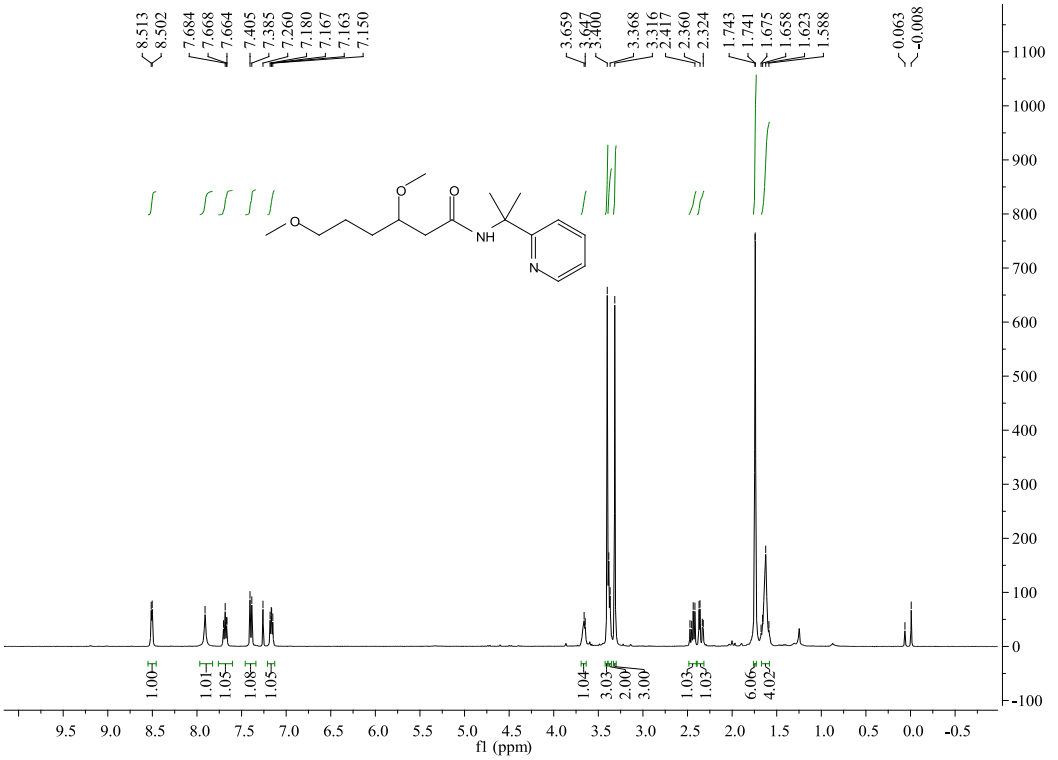
42.60
30.39
27.62
27.59
24.46
21.08
f1 (ppm)

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3.647
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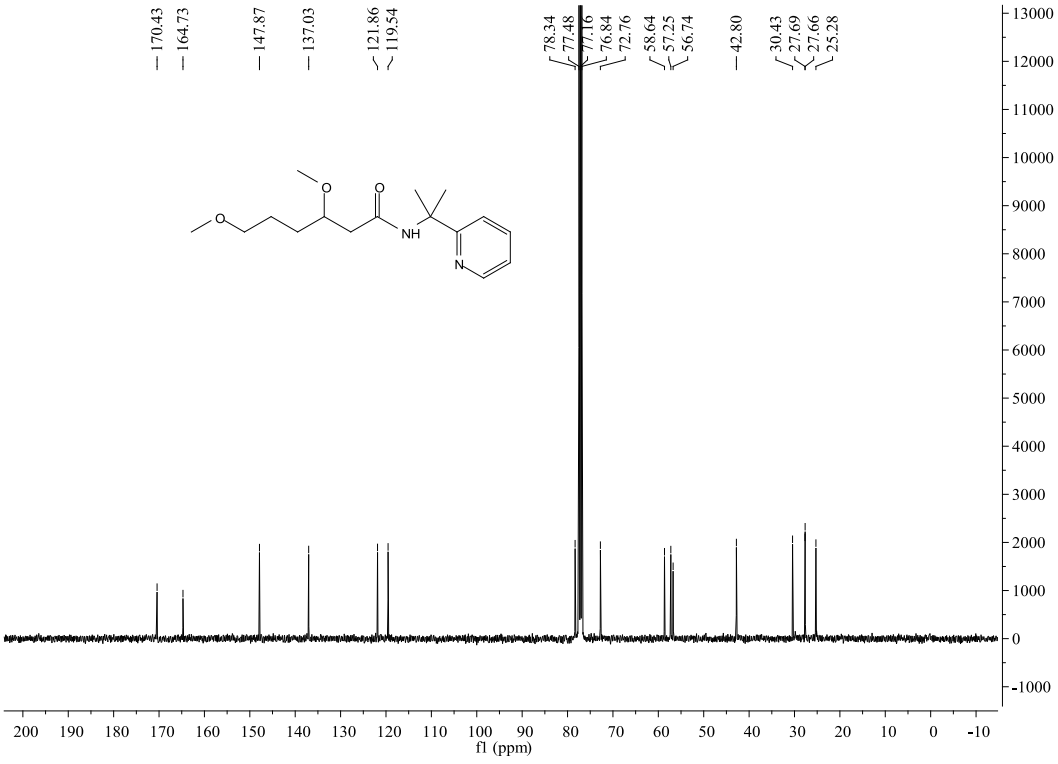
2.00
3.00
1.03
1.03
6.06
4.02
f1 (ppm)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27.69
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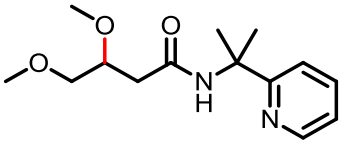
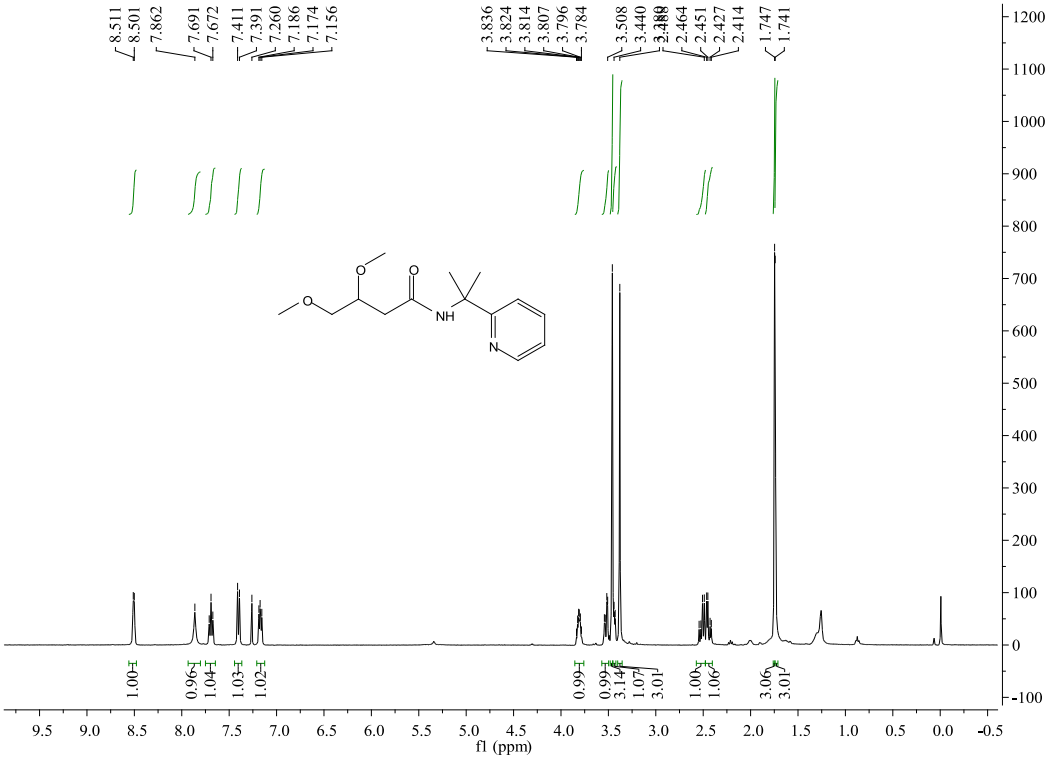
25.28
f1 (ppm)

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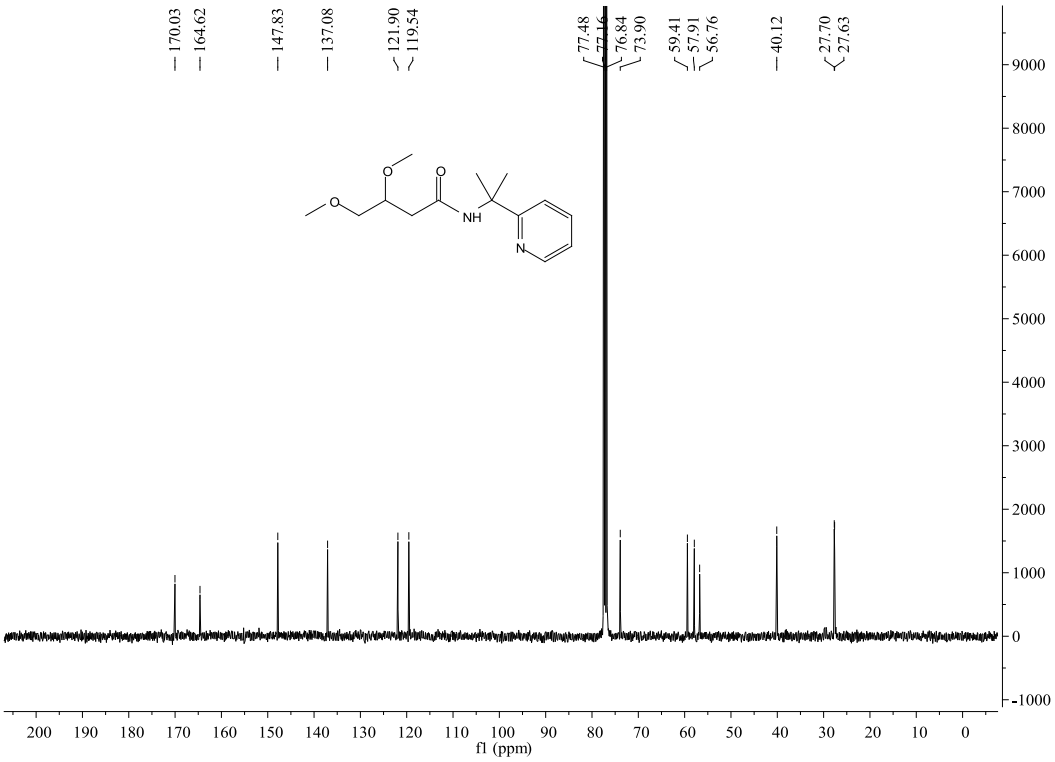
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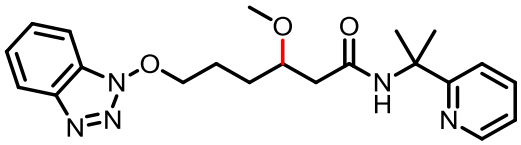
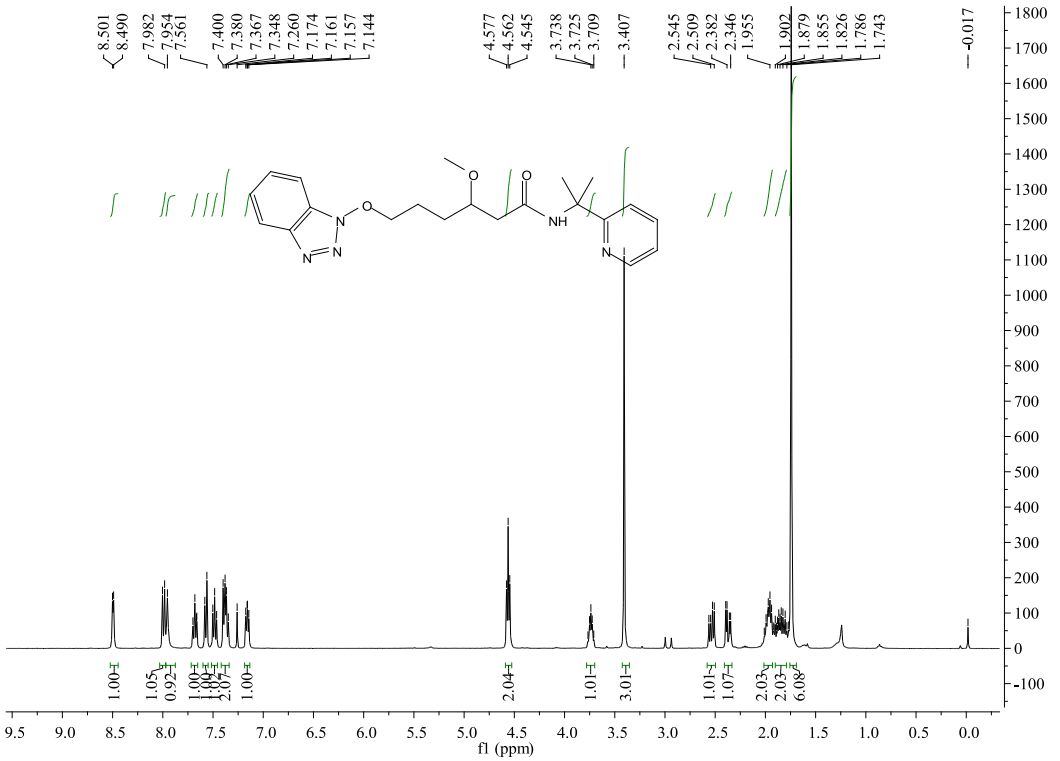
f1 (ppm)

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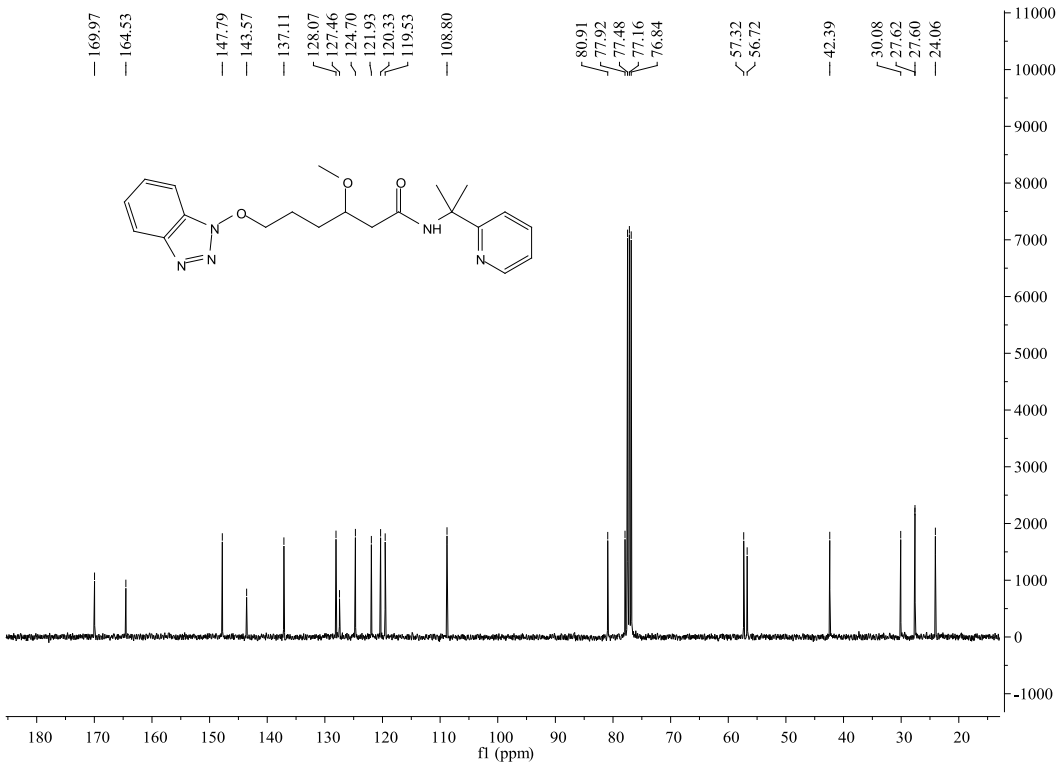
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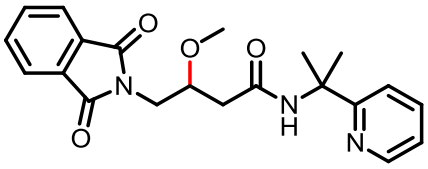
f1 (ppm)



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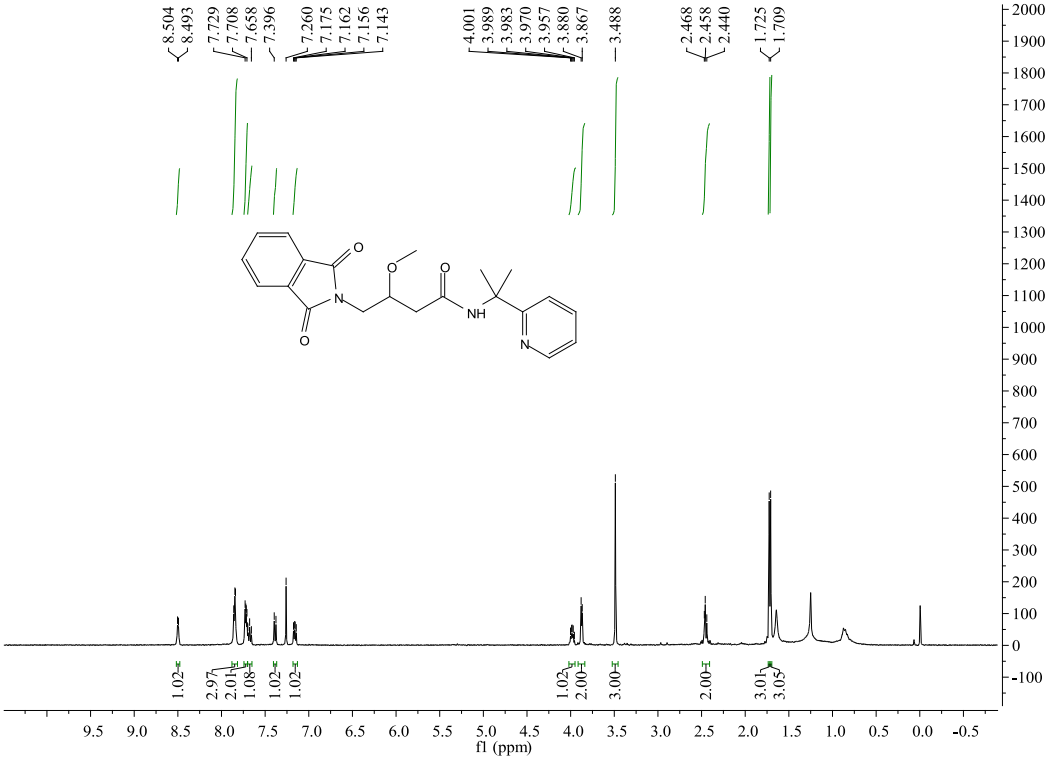

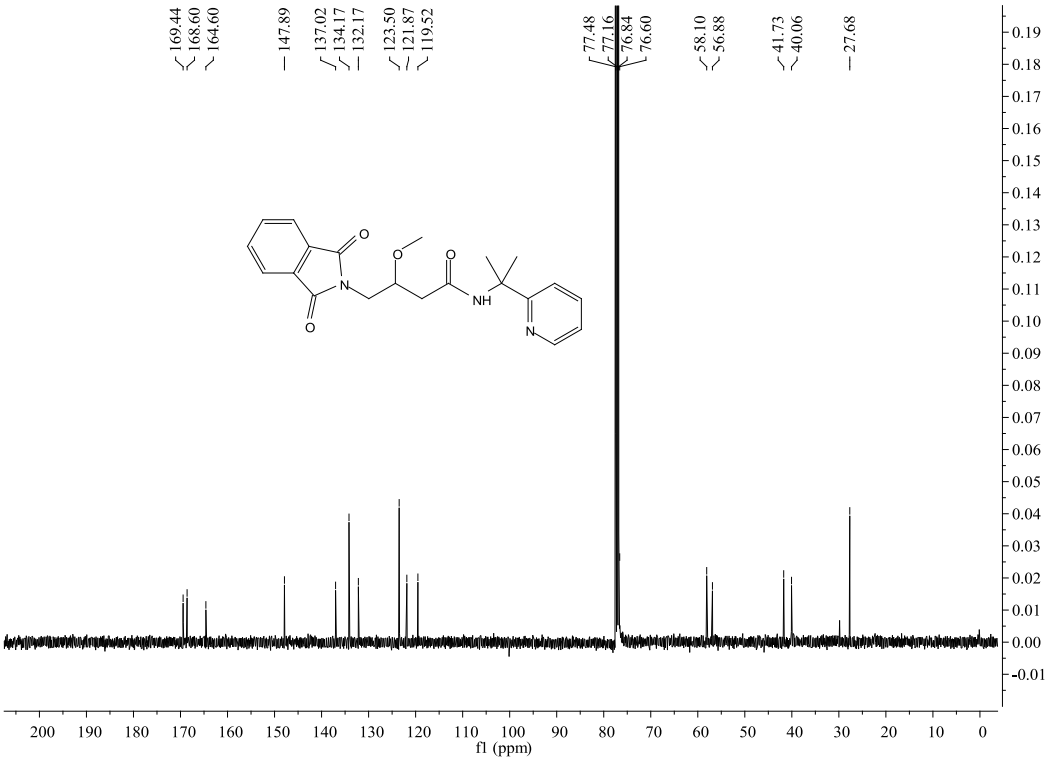

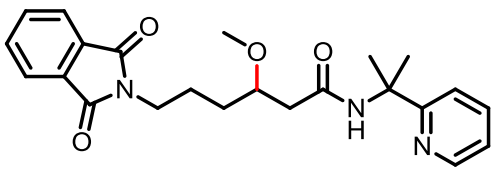

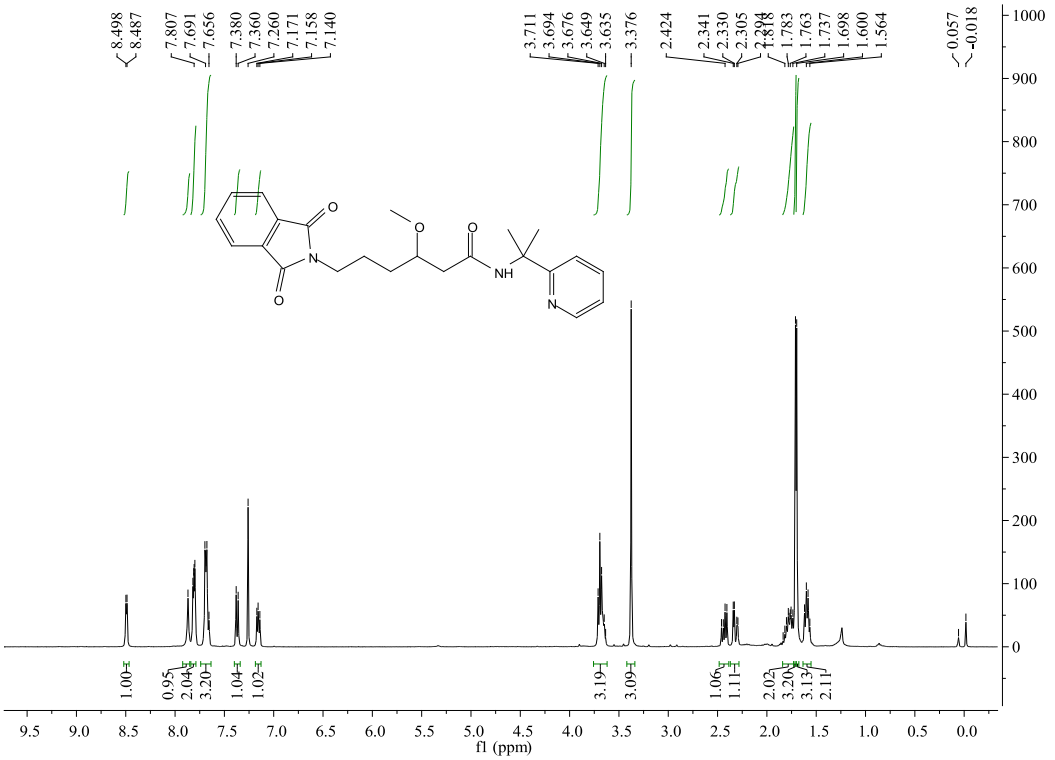
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3.19
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1.06
1.11
2.02
3.20
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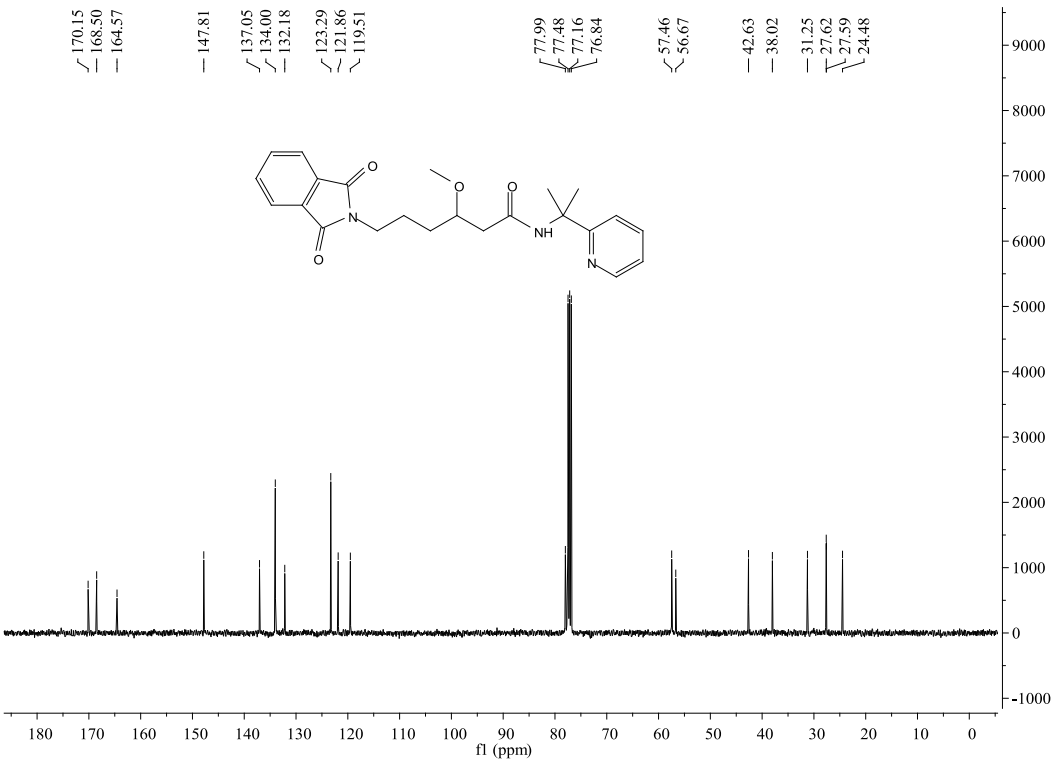
2.11
fl (ppm)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31.25
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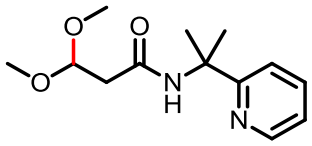
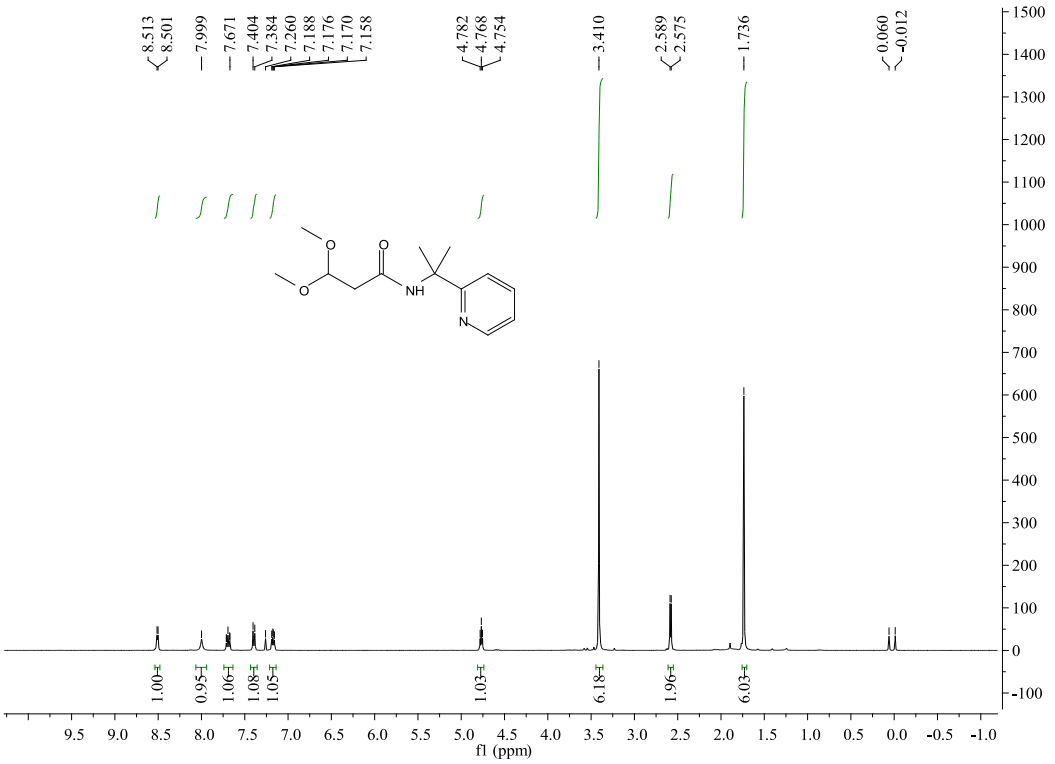
24.48
fl (ppm)

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7.188
7.176
7.170
7.158
4.782
4.768
4.754
3.410
2.589
2.575
1.736
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0.95
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1.08
1.05
1.03
6.18
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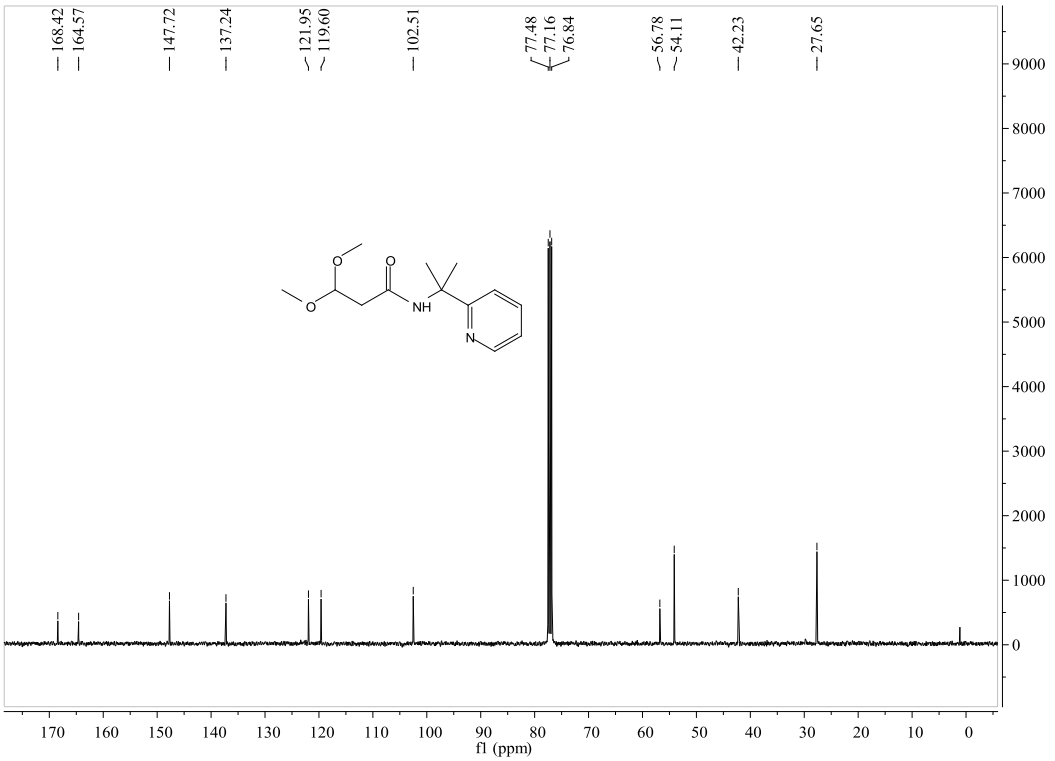
6.03
f1 (ppm)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77.16
76.84
56.78
54.11
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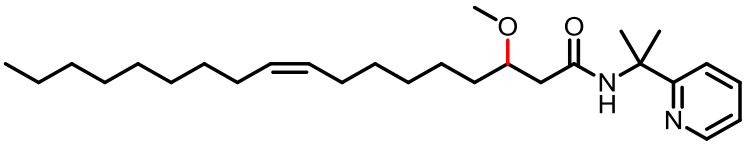
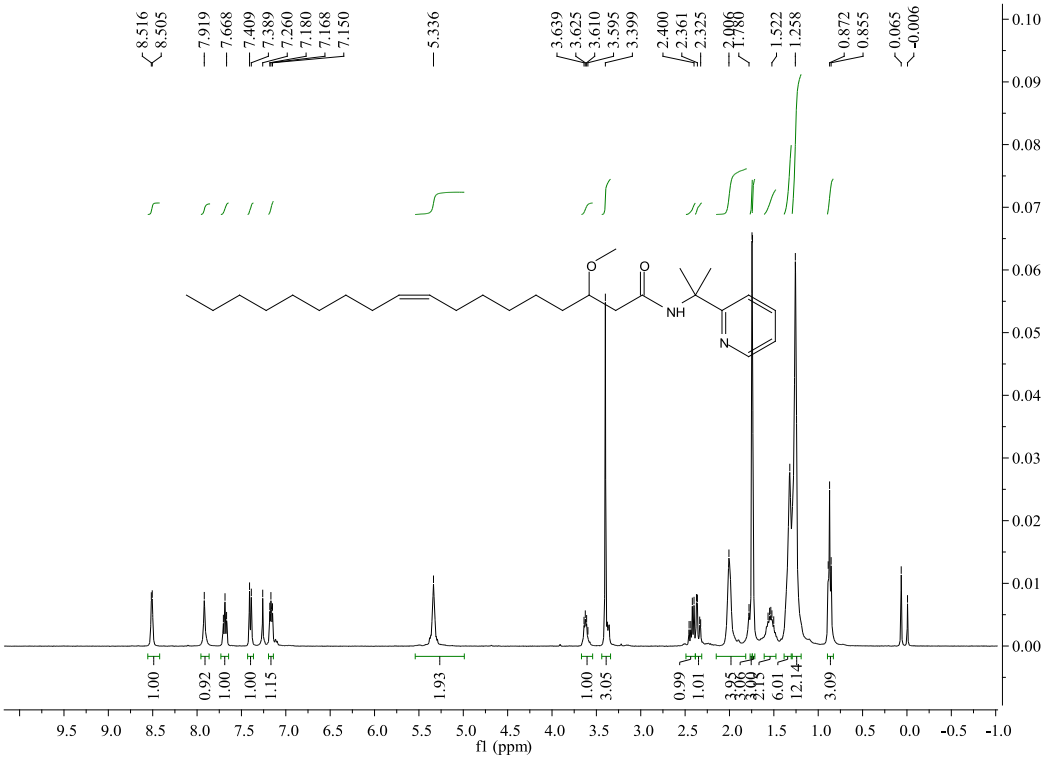
27.65
f1 (ppm)

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7.180
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7.150
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3.639
3.625
3.610
3.595
3.399
2.400
2.361
2.325
2.006
1.780
1.522
1.258
0.872
0.855
0.065
-0.006
1.00
0.92
1.00
1.00
1.15
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0.99
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3.95
3.06
2.10
6.01
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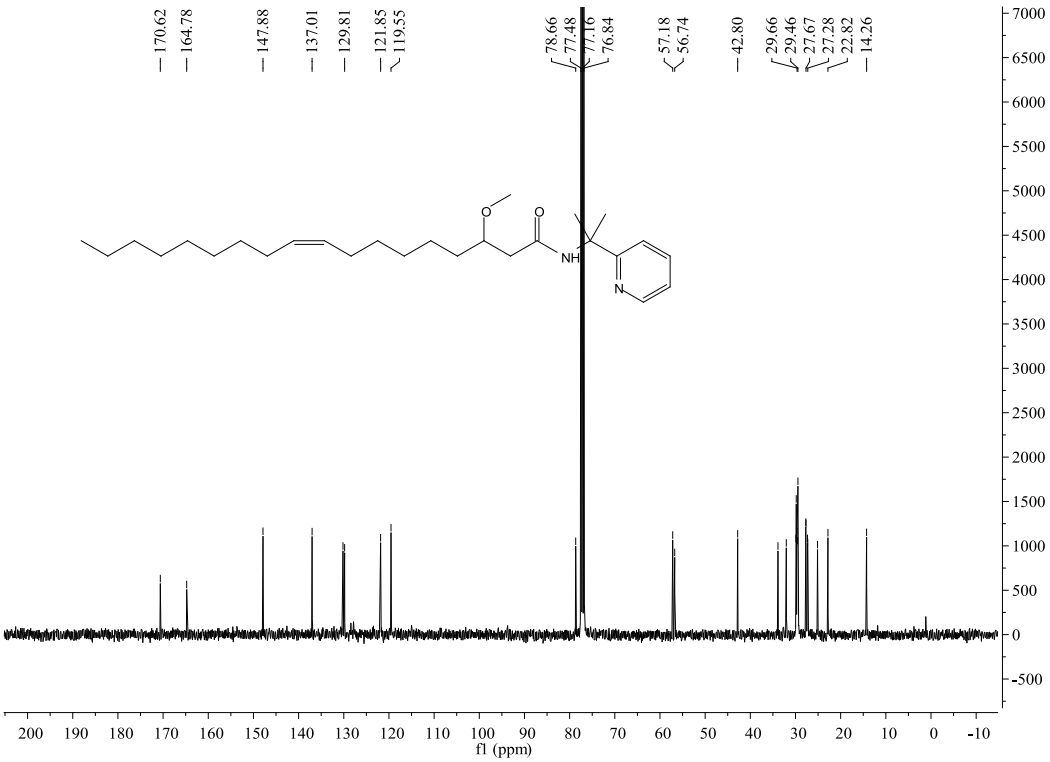
3.09
f1 (ppm)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78.66
77.48
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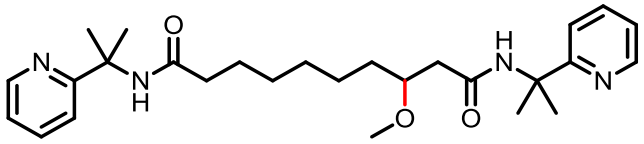
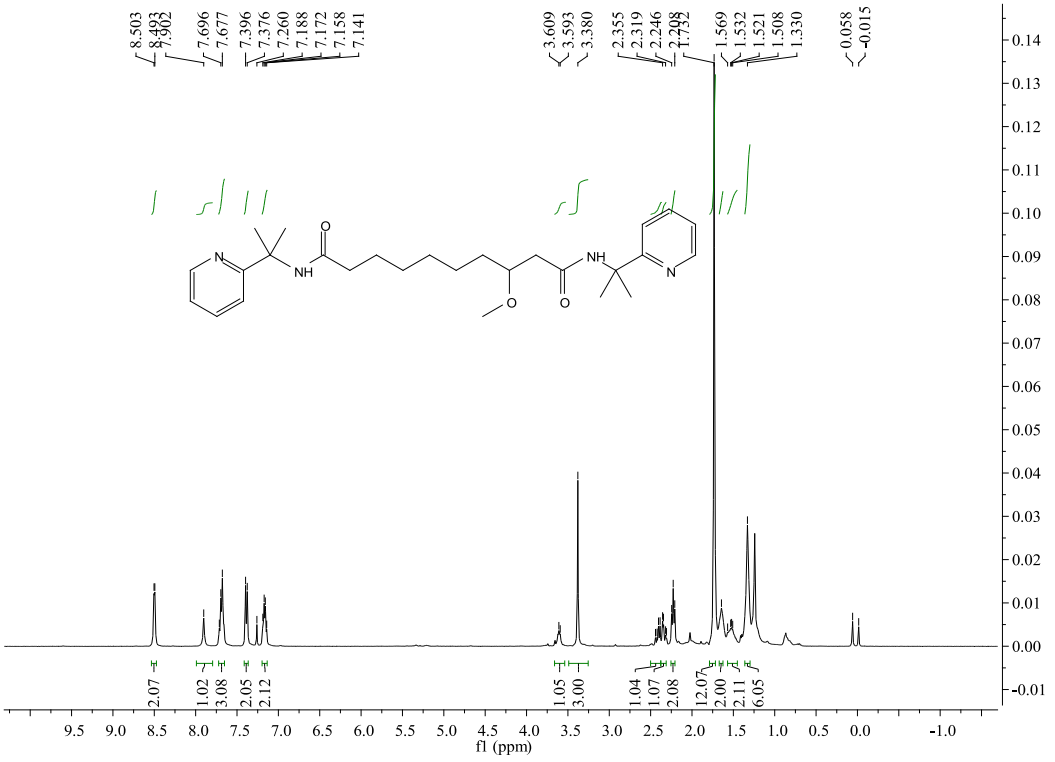
29.66
29.46
27.67
27.28
22.82
14.26
f1 (ppm)

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7.260
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7.172
7.158
7.141
3.609
3.593
3.380
2.355
2.319
2.246
2.208
1.732
1.569
1.532
1.521
1.508
1.330
0.058
-0.015
2.07
1.02
3.08
2.05
2.12
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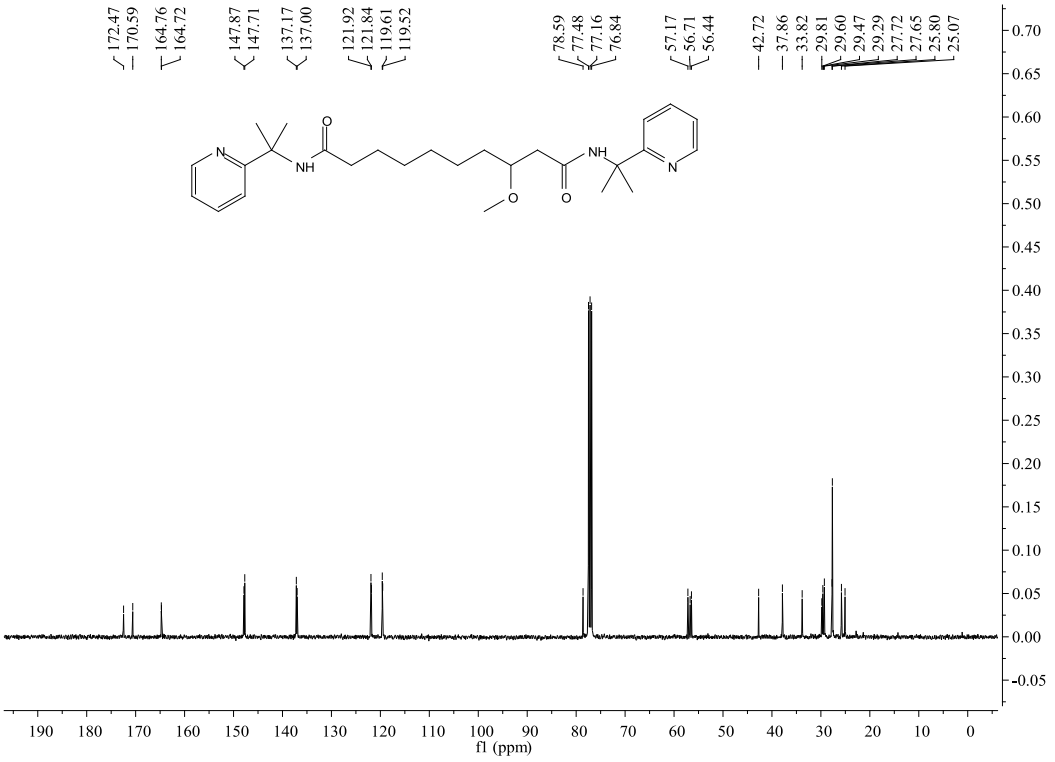
3.00
1.04
1.07
2.08
12.07
2.00
2.11
6.05
f1 (ppm)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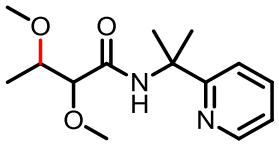
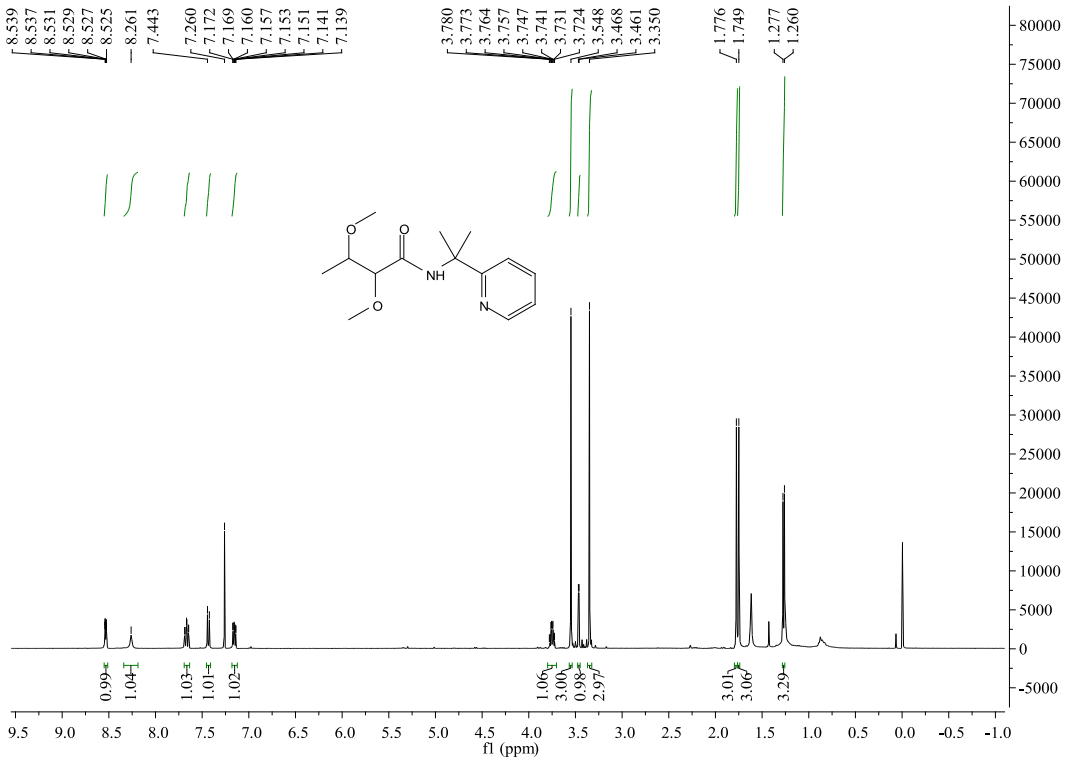
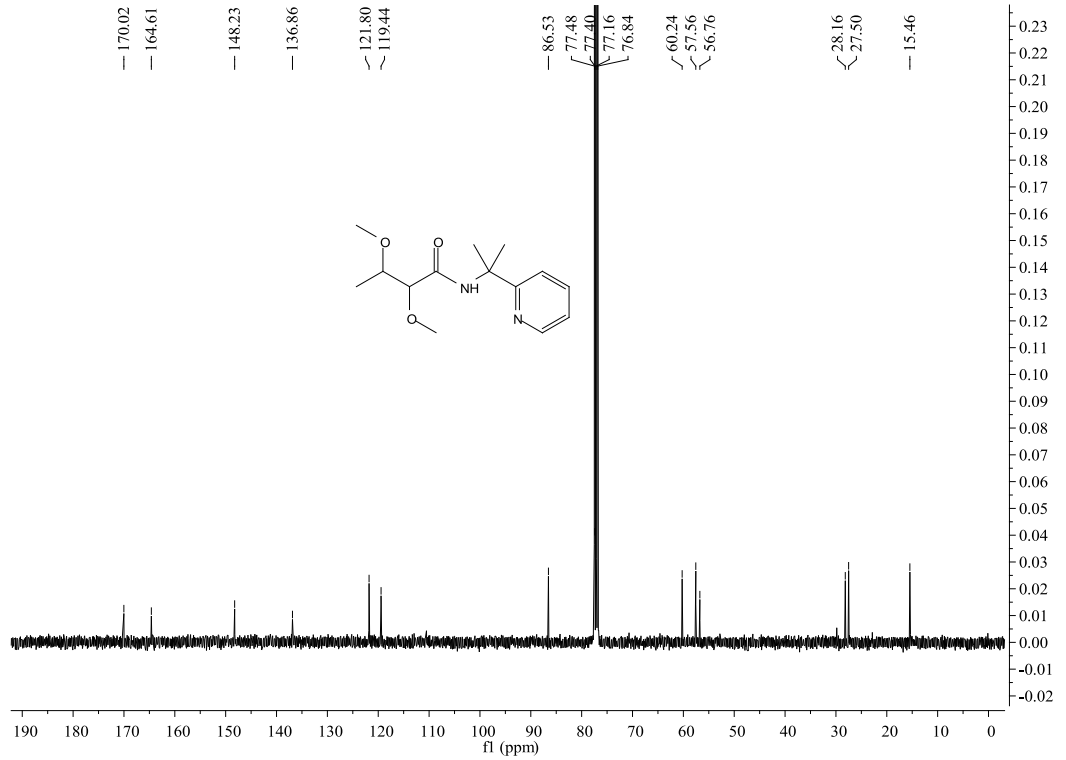
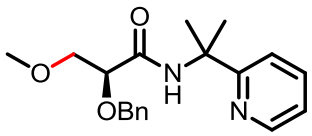
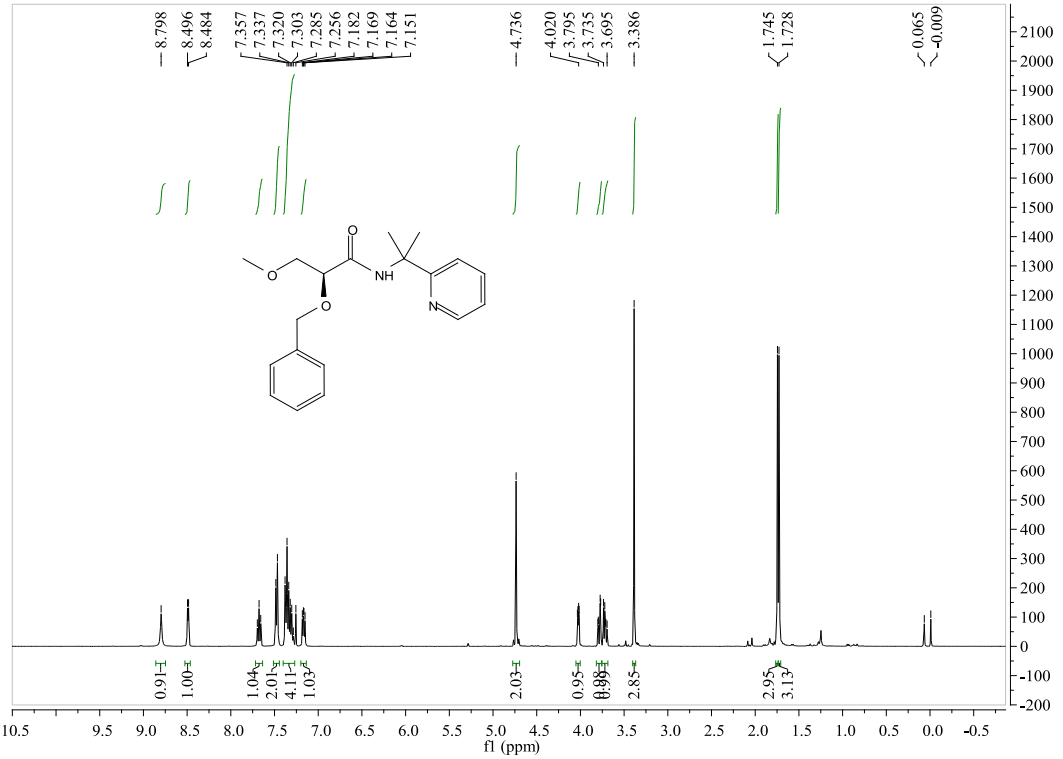
147.71
137.17
137.00
121.92
121.84
119.61
119.52
78.59
77.48
77.16
76.84
57.17
56.71
56.44
42.72
37.86
33.82
29.81
29.60
29.47
29.29
27.72
27.65
25.80
25.07
f1 (ppm)








O
N
H
O
OBn
N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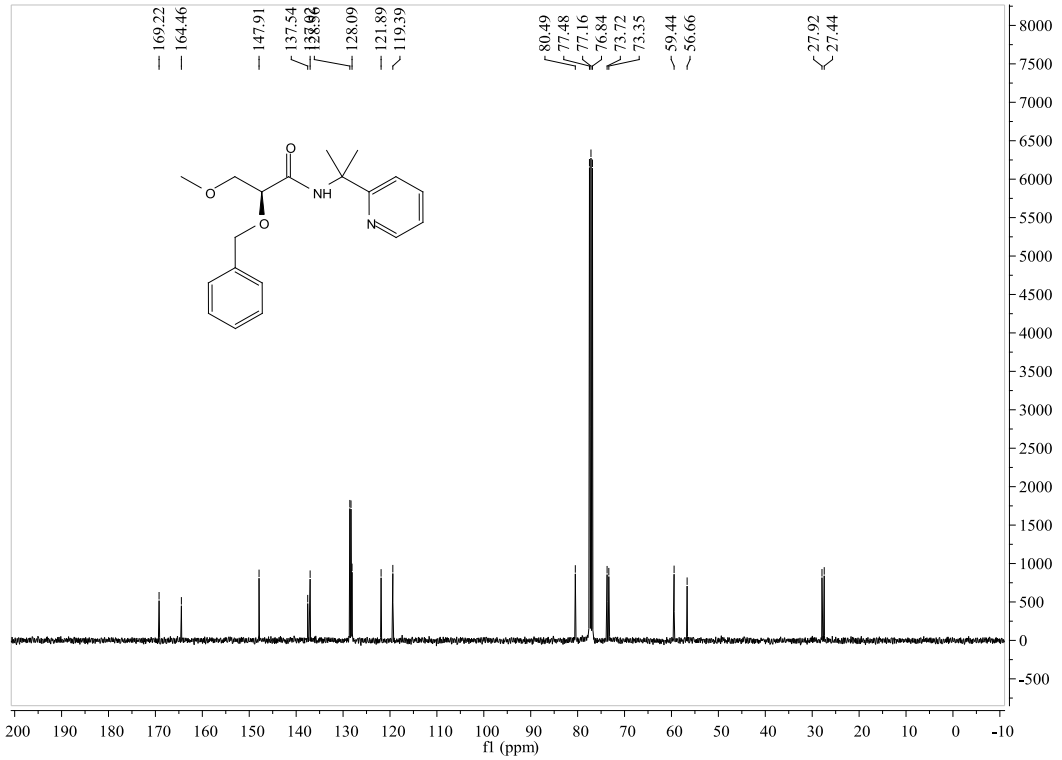
7.303
7.285
7.256
7.182
7.169
7.164
7.151
4.736
4.020
3.795
3.735
3.695
3.386
1.745
1.728
0.065
-0.009
0.91
1.00
1.04
2.01
4.11
1.03
2.03
0.95
0.98
2.85
2.95
3.13
f1 (ppm)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77.16
76.84
73.72
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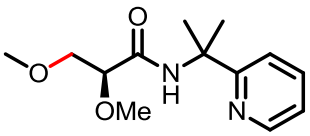
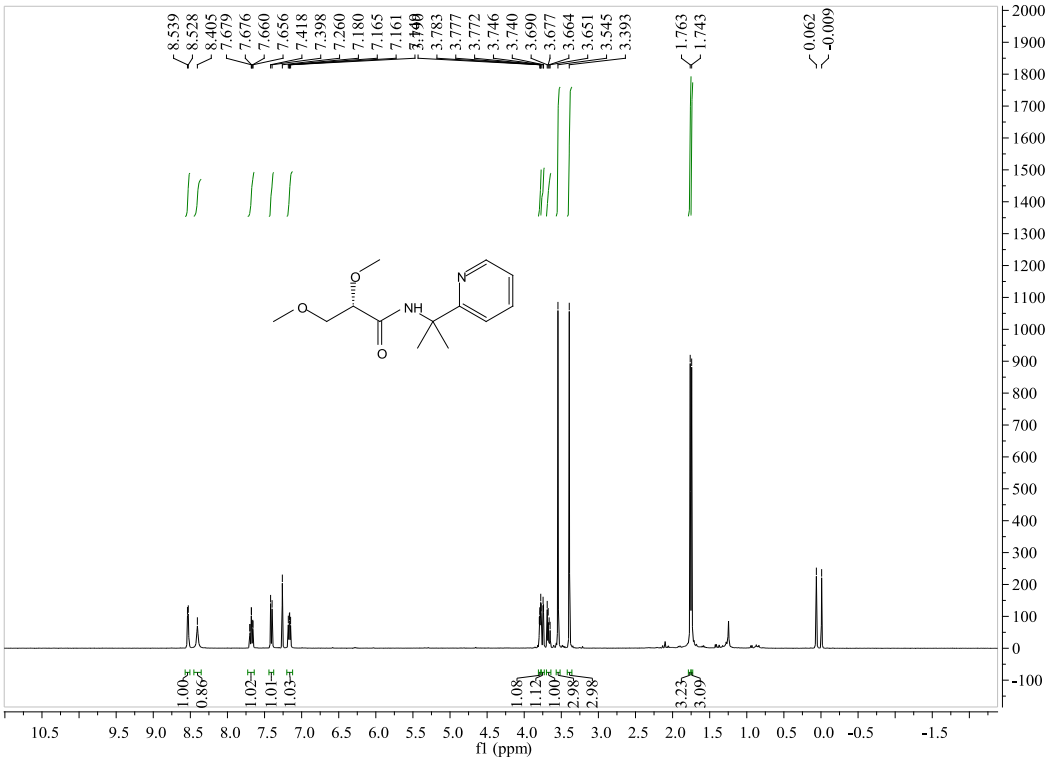
56.66
27.92
27.44
f1 (ppm)


O
O
N
H
OMe
N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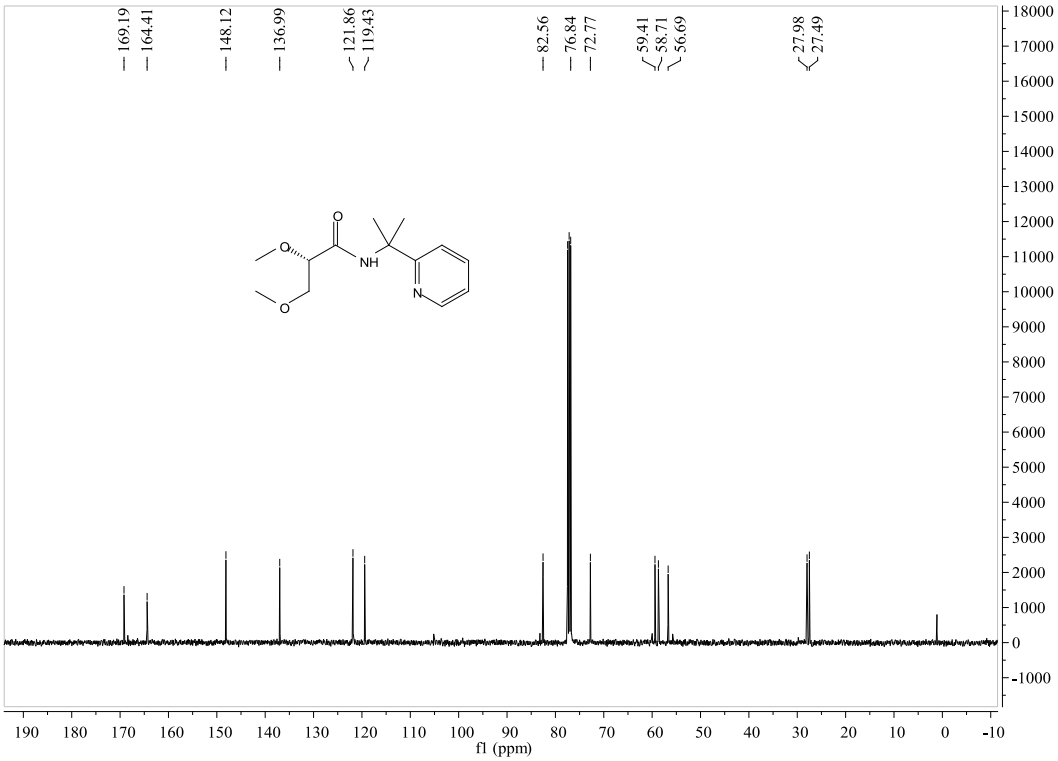
7.418
7.398
7.260
7.180
7.165
7.161
3.783
3.777
3.772
3.746
3.740
3.690
3.677
3.664
3.651
3.545
3.393
1.763
1.743
0.062
-0.009
1.00
0.86
1.02
1.01
1.03
1.08
1.12
1.00
2.98
2.98
3.23
3.09
f1 (ppm)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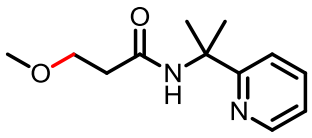
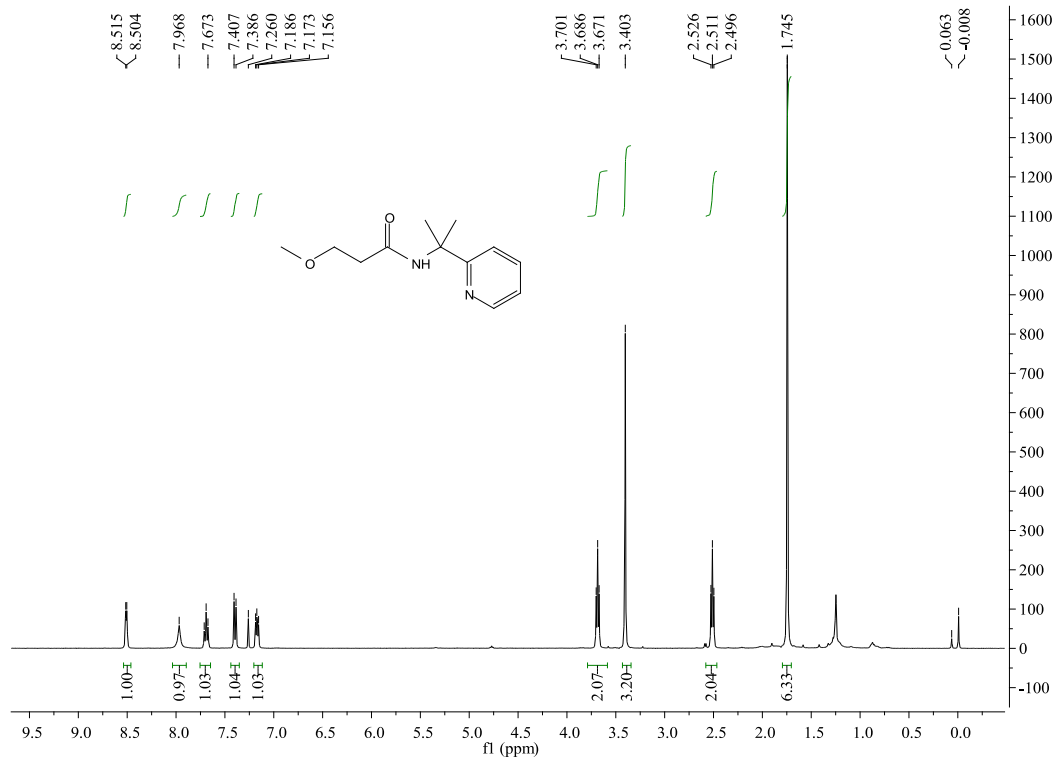
76.84
72.77
59.41
58.71
56.69
27.98
27.49
f1 (ppm)

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7.260
7.186
7.173
7.156
3.701
3.686
3.671
3.403
2.526
2.511
2.496
1.745
0.063
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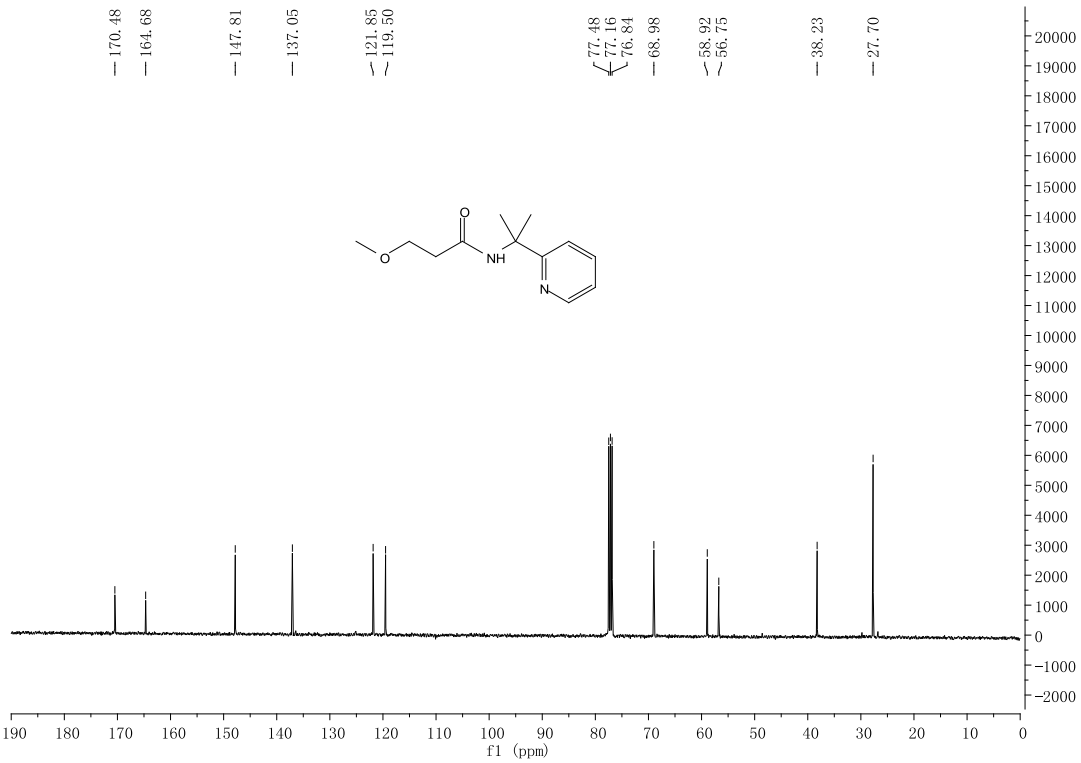
0.97
1.03
1.04
1.03
2.07
3.20
2.04
6.33
f1 (ppm)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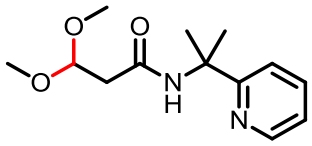
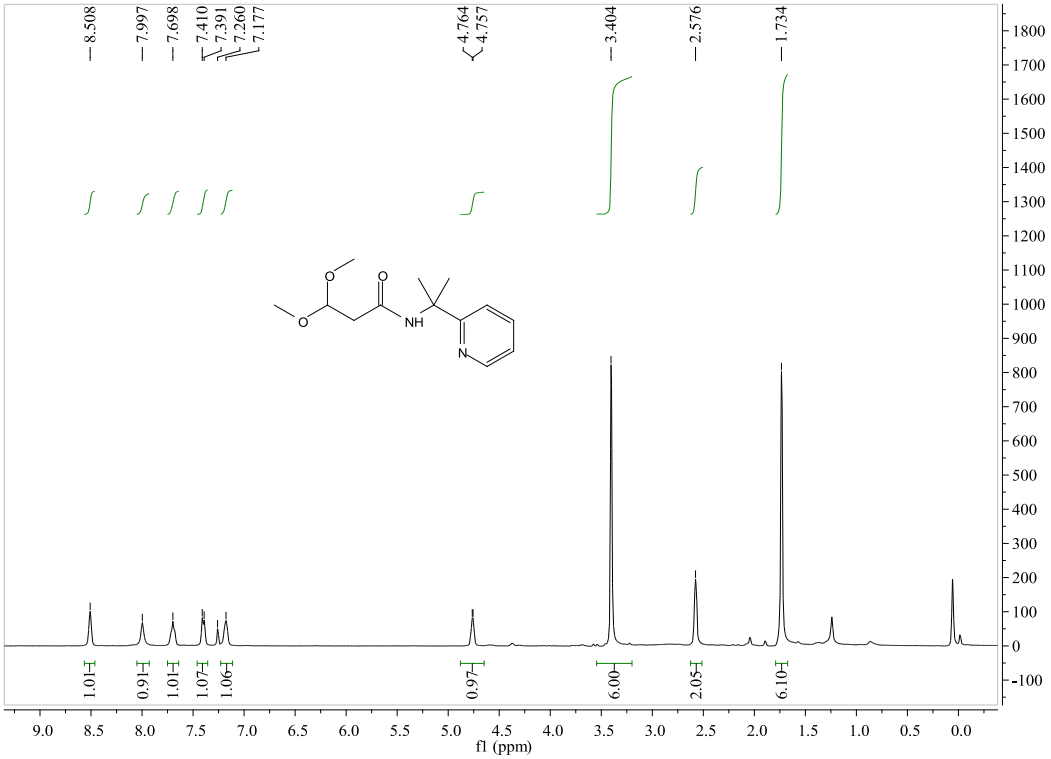
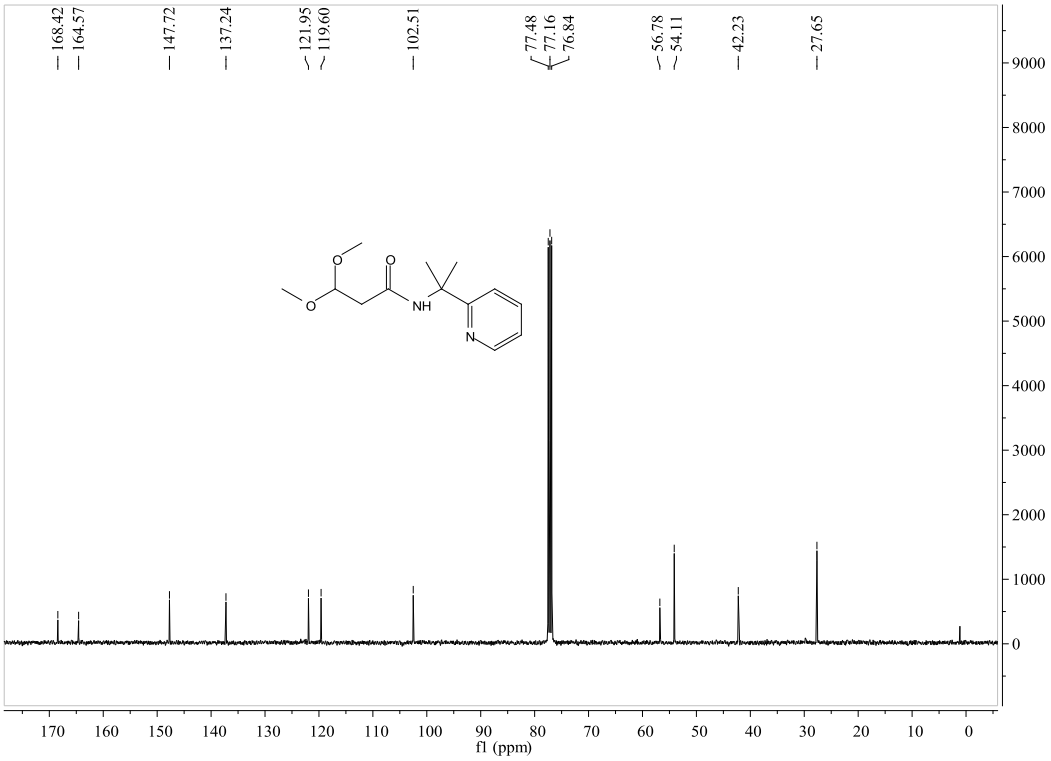
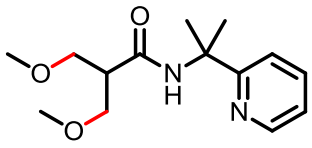
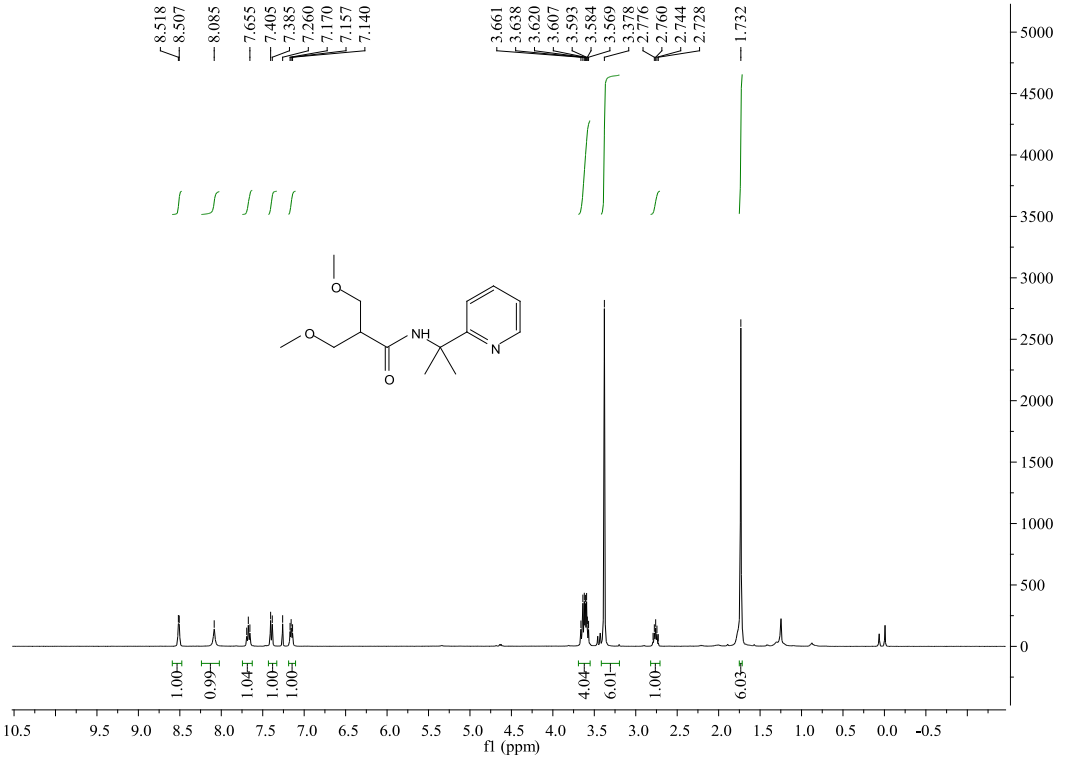
27.70
f1 (ppm)







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3.638
3.620
3.607
3.593
3.584
3.569
3.378
2.776
2.760
2.744
2.728
1.732
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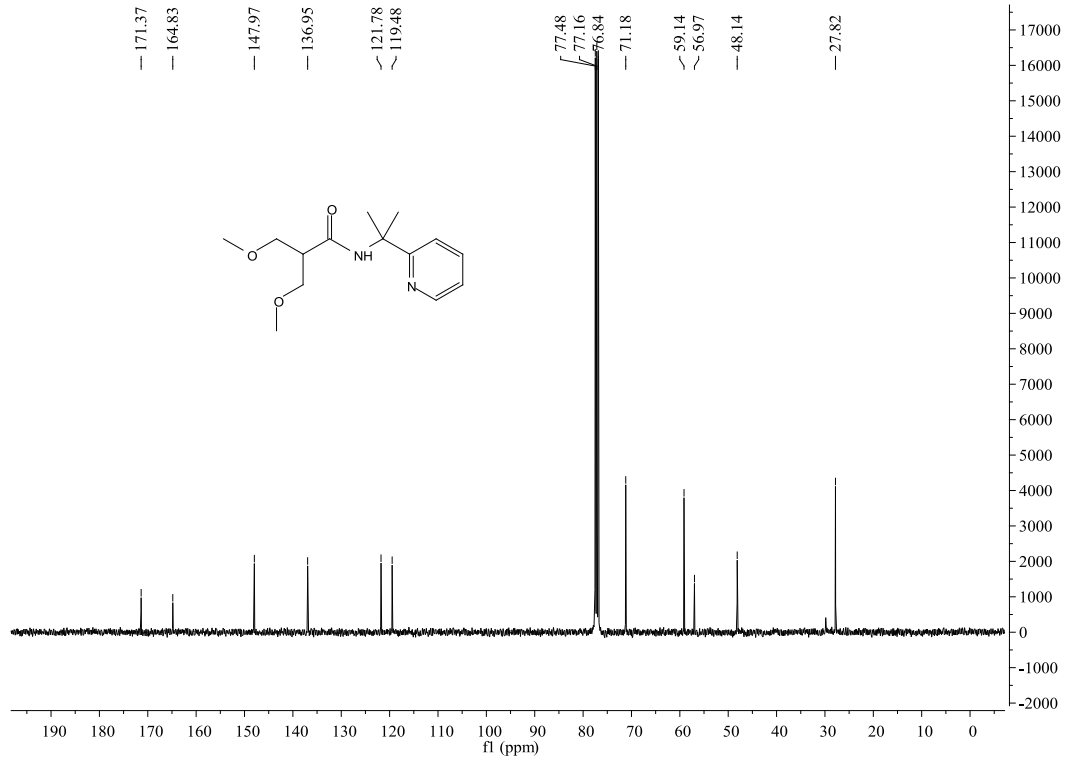
0.99
1.04
1.00
1.00
4.04
6.01
1.00
6.03
fl (ppm)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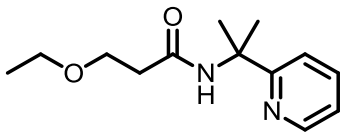
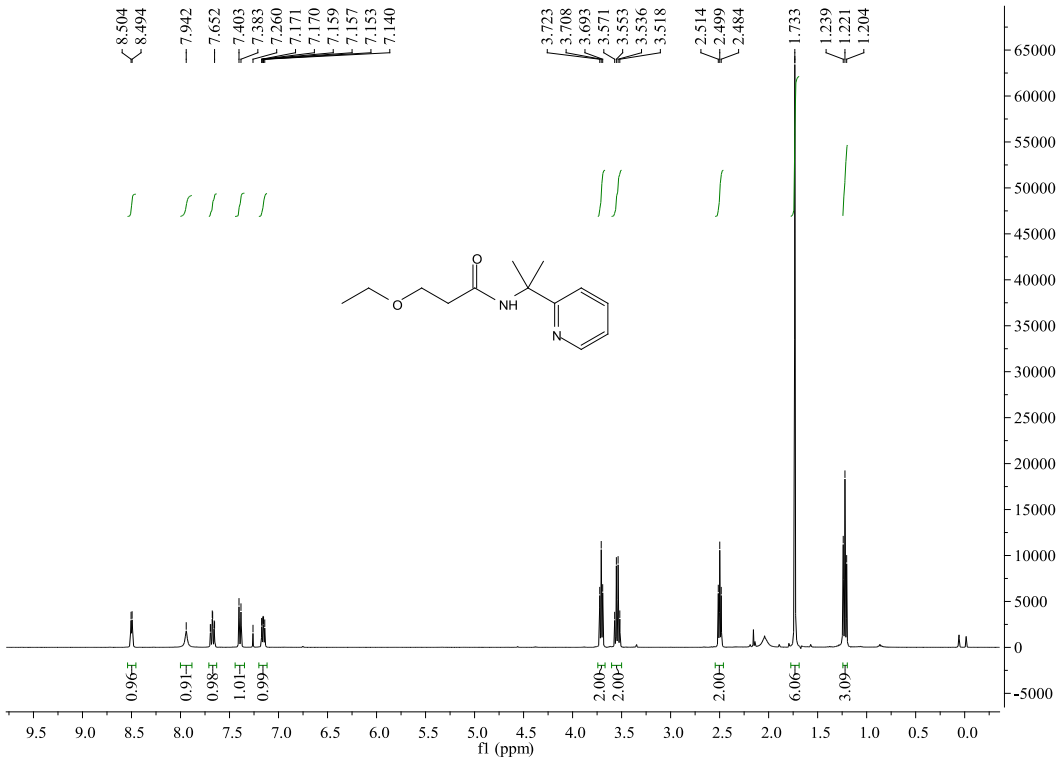
77.16
76.84
71.18
59.14
56.97
48.14
27.82
fl (ppm)

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7.383
7.260
7.171
7.170
7.159
7.157
7.153
7.140
3.723
3.708
3.693
3.571
3.553
3.536
3.518
2.514
2.499
2.484
1.733
1.239
1.221
1.204
0.96
0.91
0.98
1.01
0.99
2.00
2.00
2.00
6.06
3.09
f1 (ppm)


170.80
164.83
147.89
136.99
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77.16
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66.90
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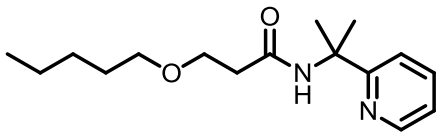
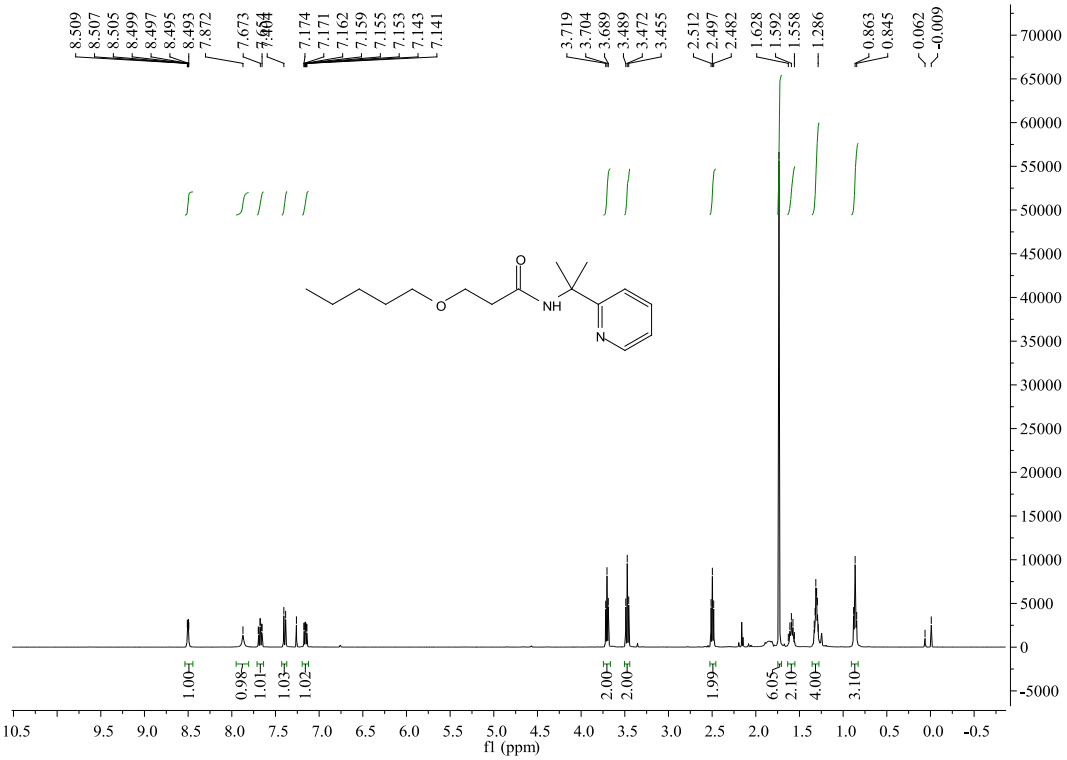
27.75
15.23
f1 (ppm)

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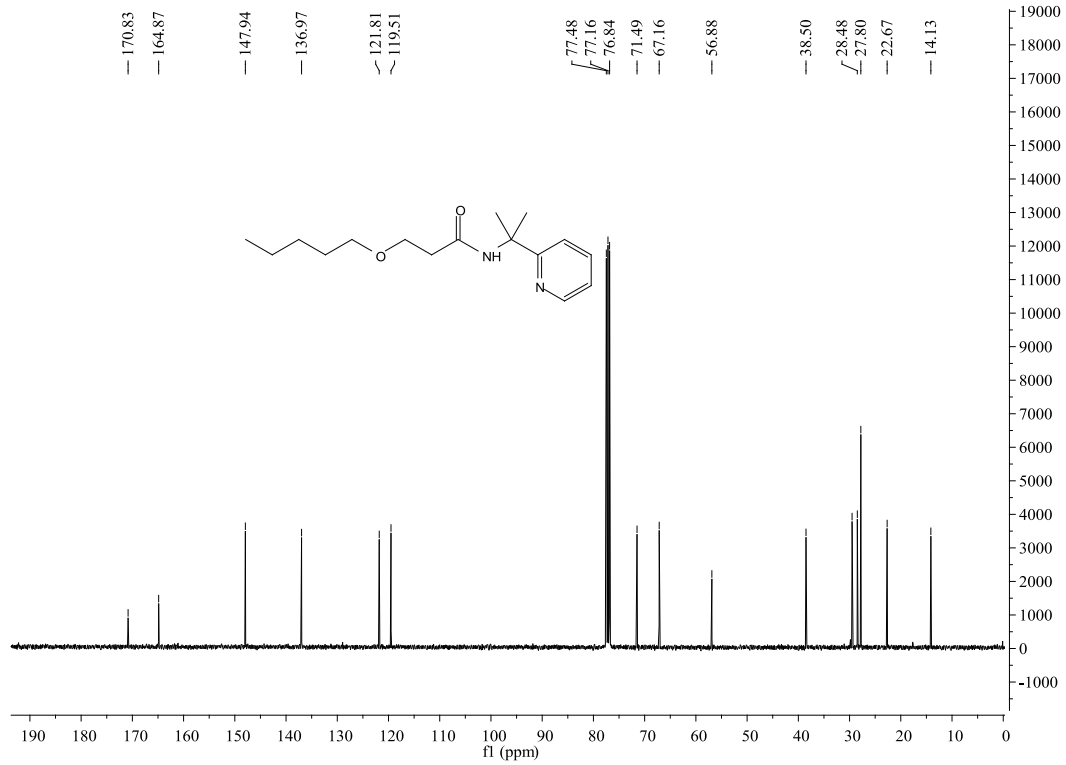
7.171
7.162
7.159
7.155
7.153
7.143
7.141
3.719
3.704
3.689
3.489
3.472
3.455
2.512
2.497
2.482
1.628
1.592
1.558
1.286
0.863
0.845
0.062
-0.009
1.00
0.98
1.01
1.03
1.02
2.00
2.00
1.99
6.05
2.10
4.00
3.10
f1 (ppm)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136.97
121.81
119.51
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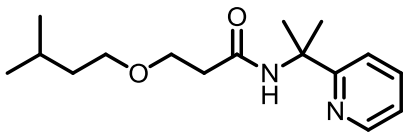
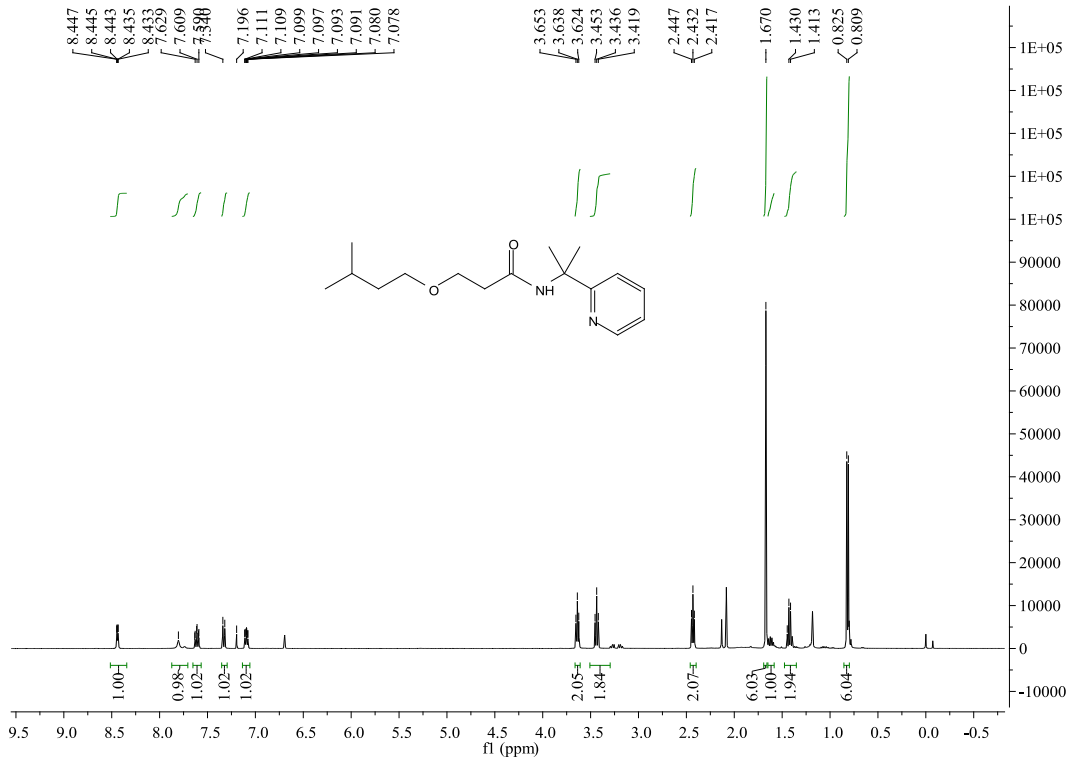
28.48
27.80
22.67
14.13
f1 (ppm)

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3.638
3.624
3.453
3.436
3.419
2.447
2.432
2.417
1.670
1.430
1.413
0.825
0.809
1.00
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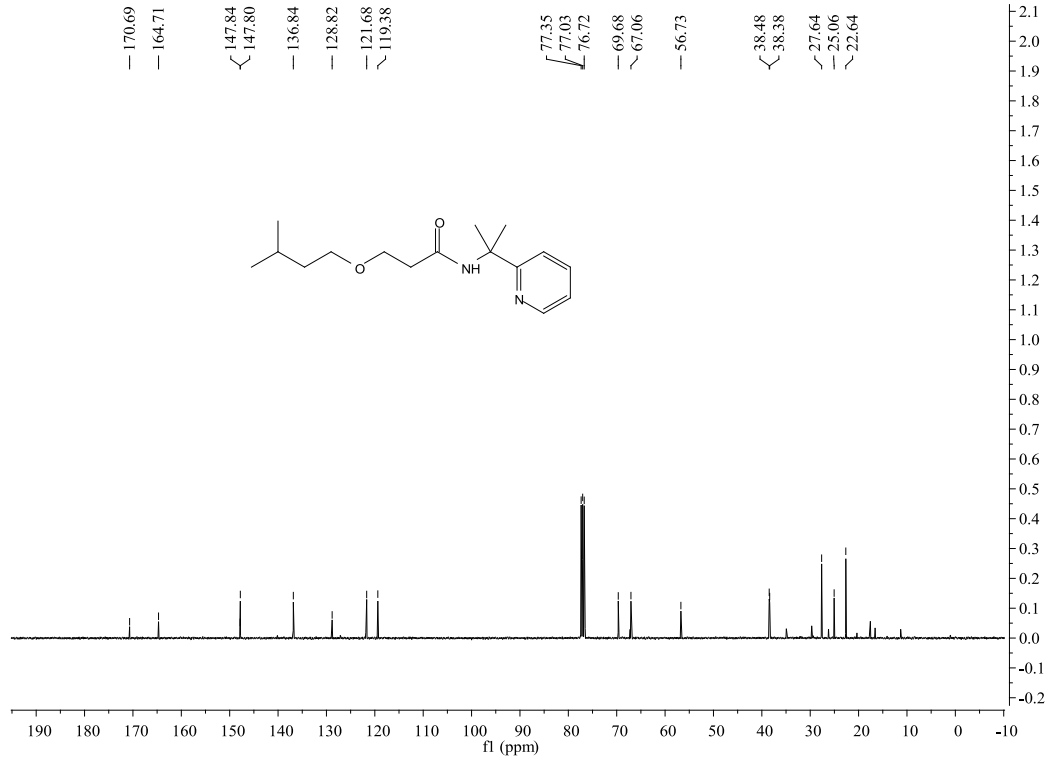
1.02
2.05
1.84
2.07
6.03
1.00
1.94
6.04
f1 (ppm)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128.82
121.68
119.38
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67.06
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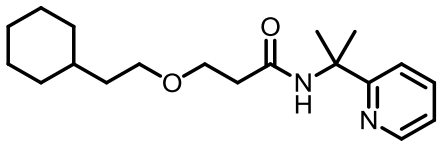
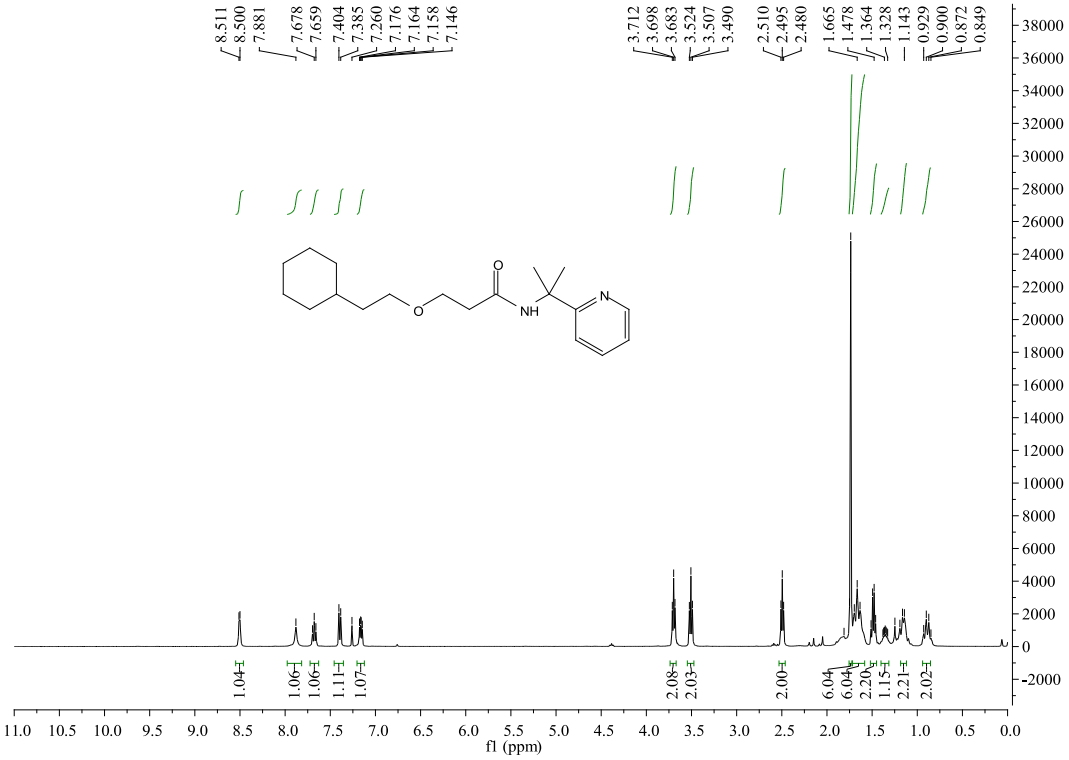
27.64
25.06
22.64
f1 (ppm)

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7.404
7.385
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7.164
7.158
7.146
3.712
3.698
3.683
3.524
3.507
3.490
2.510
2.495
2.480
1.665
1.478
1.364
1.328
1.143
0.929
0.900
0.872
0.849
1.04
1.06
1.06
1.11
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2.03
2.00
6.04
6.04
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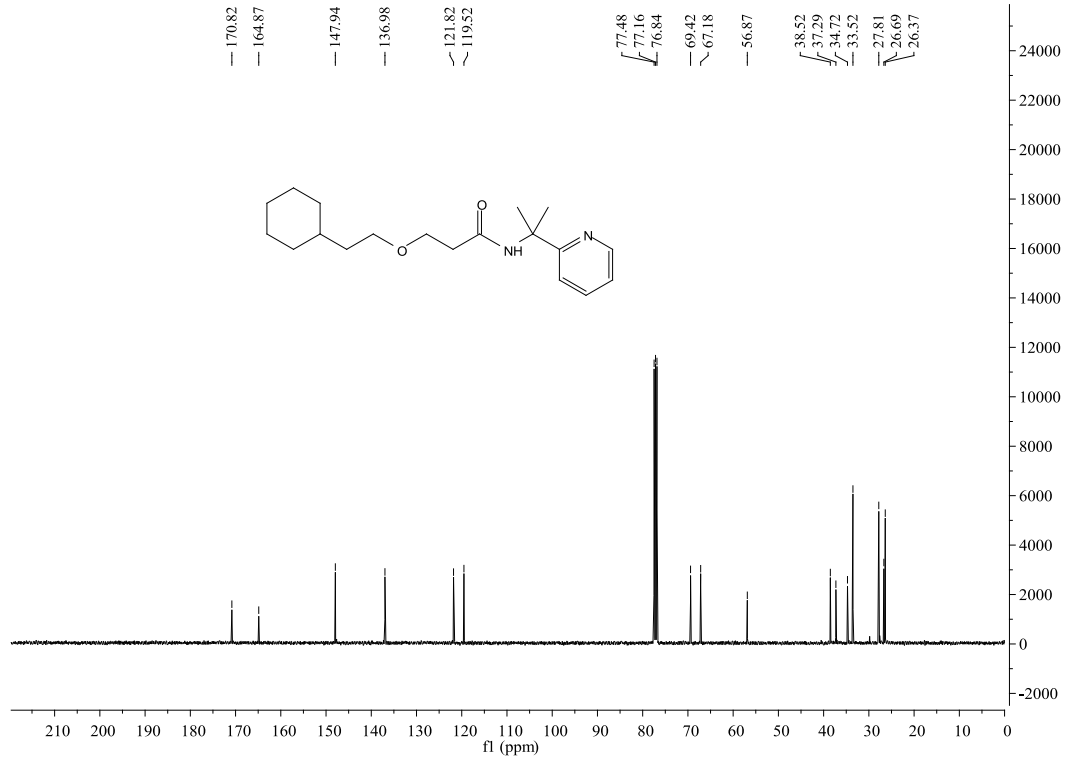
1.15
2.21
2.02
f1 (ppm)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77.16
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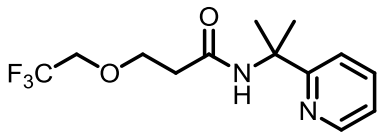
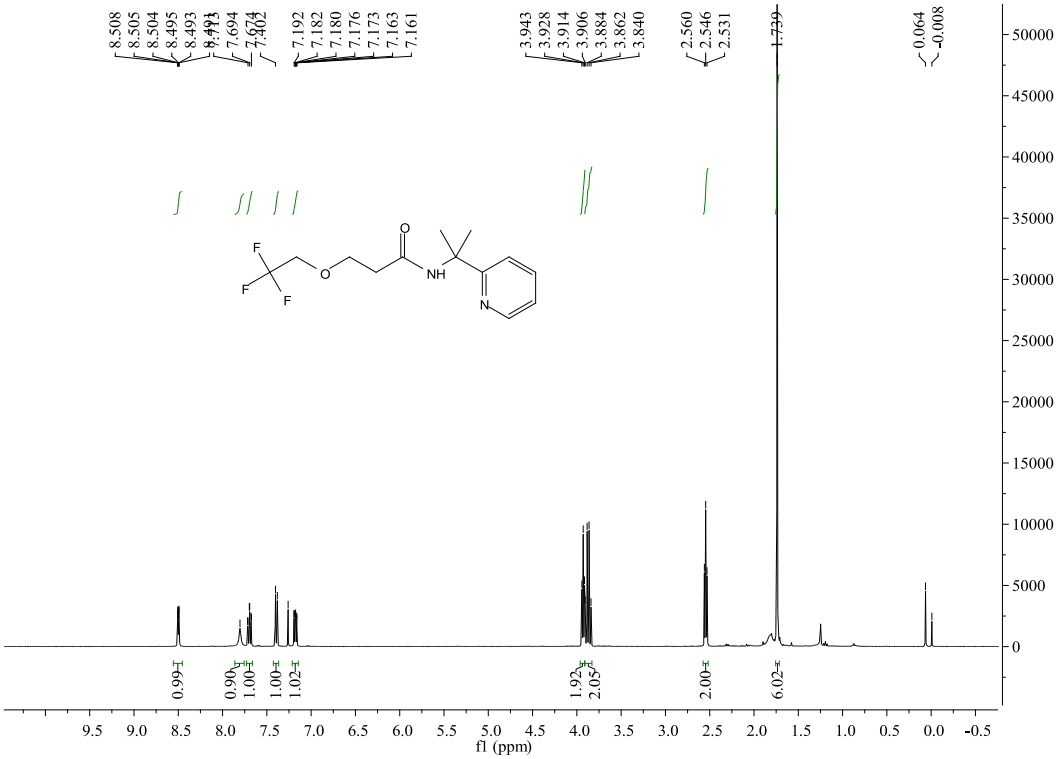
33.52
27.81
26.69
26.37
f1 (ppm)

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7.173
7.163
7.161
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3.914
3.906
3.884
3.862
3.840
2.560
2.546
2.531
1.739
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0.90
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1.00
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2.05
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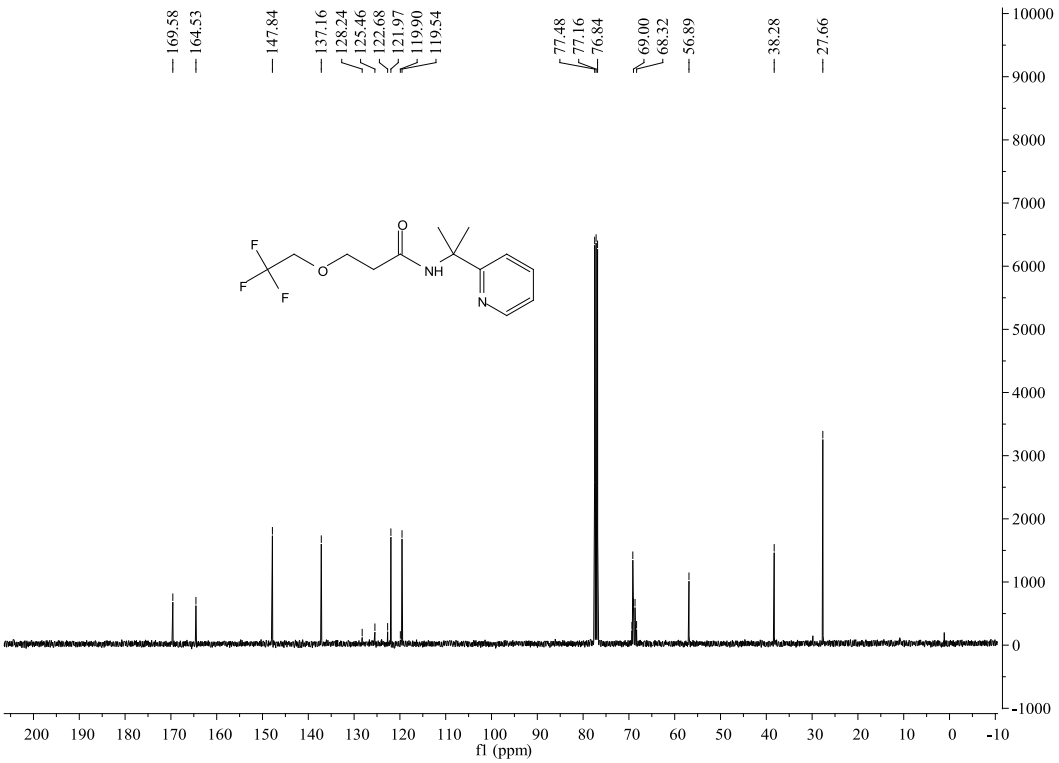
6.02
f1 (ppm)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122.68
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76.84
69.00
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56.89
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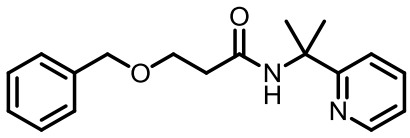
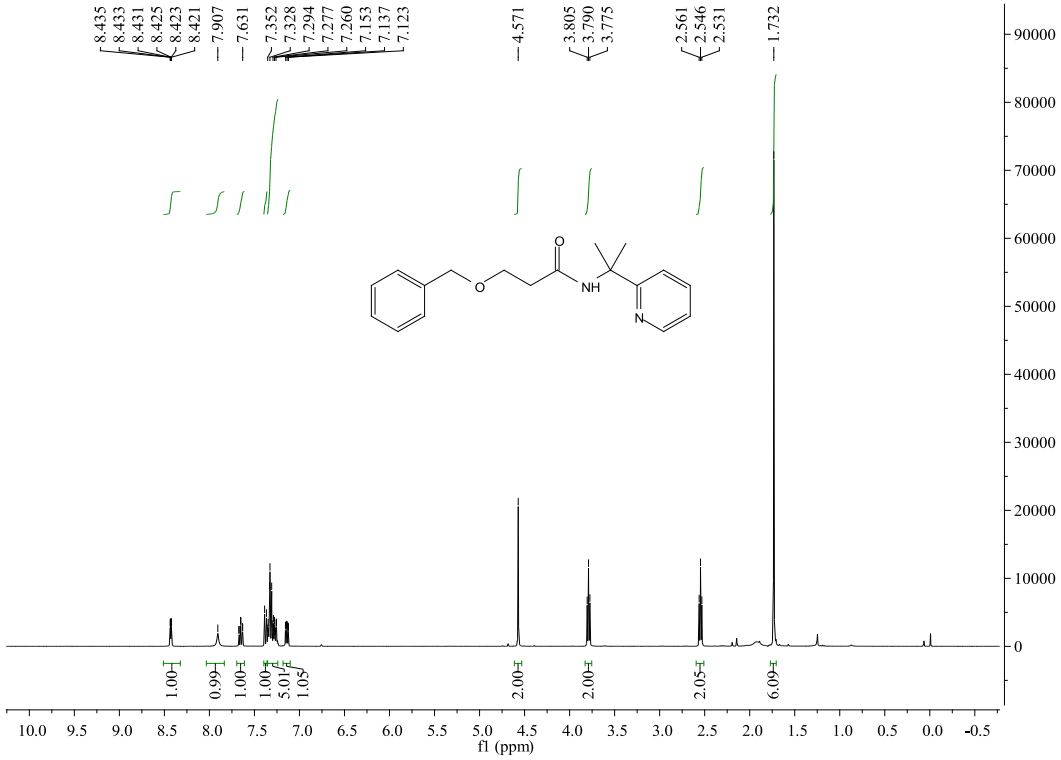
27.66
f1 (ppm)

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7.352
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7.260
7.153
7.137
7.123
4.571
3.805
3.790
3.775
2.561
2.546
2.531
1.732
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5.01
1.05
2.00
2.00
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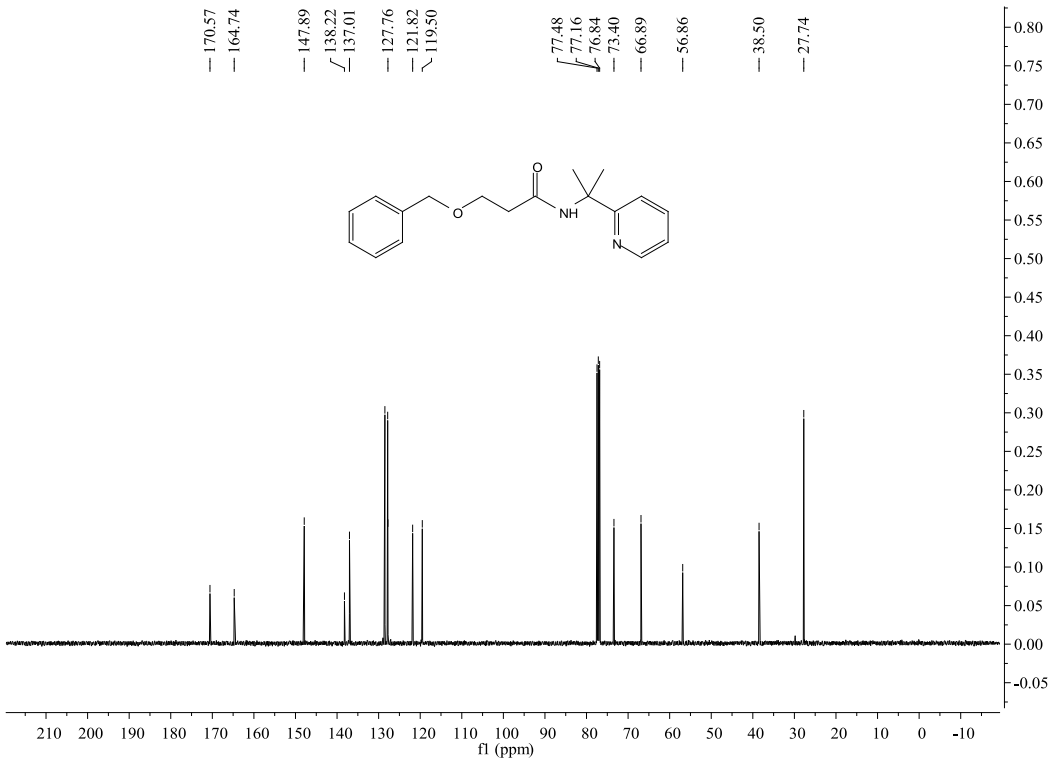
6.09
f1 (ppm)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66.89
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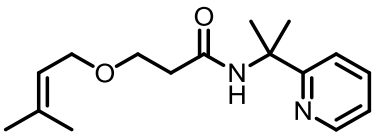
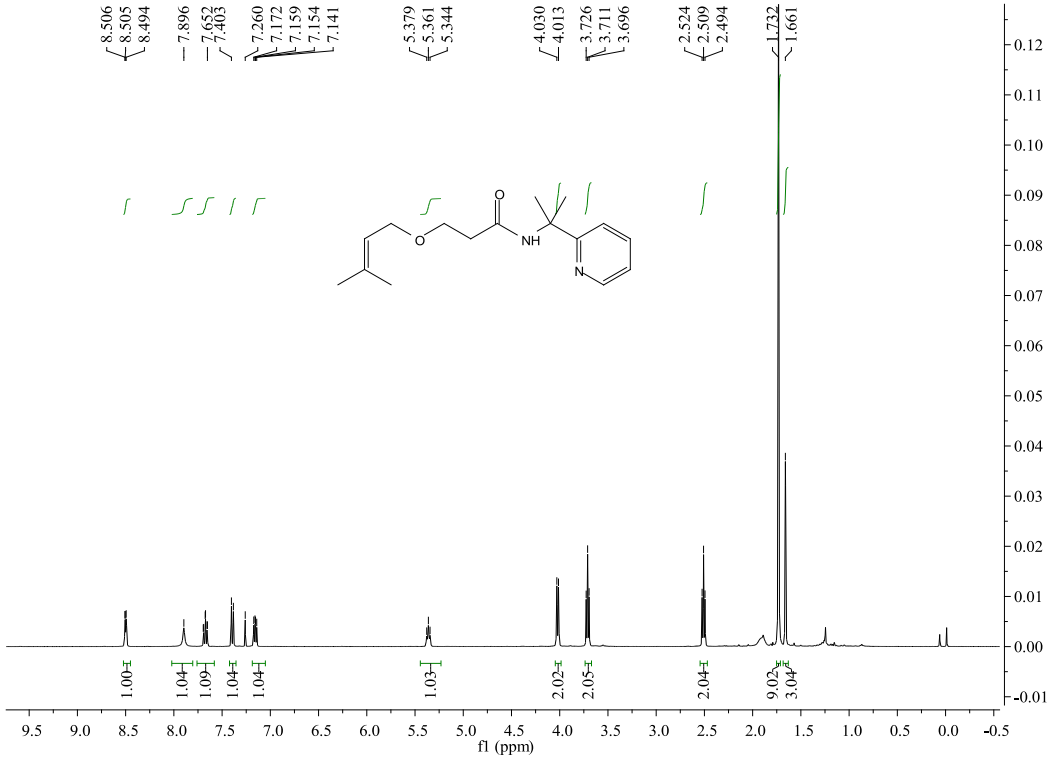
38.50
27.74
f1 (ppm)

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7.172
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7.141
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5.361
5.344
4.030
4.013
3.726
3.711
3.696
2.524
2.509
2.494
1.732
1.661
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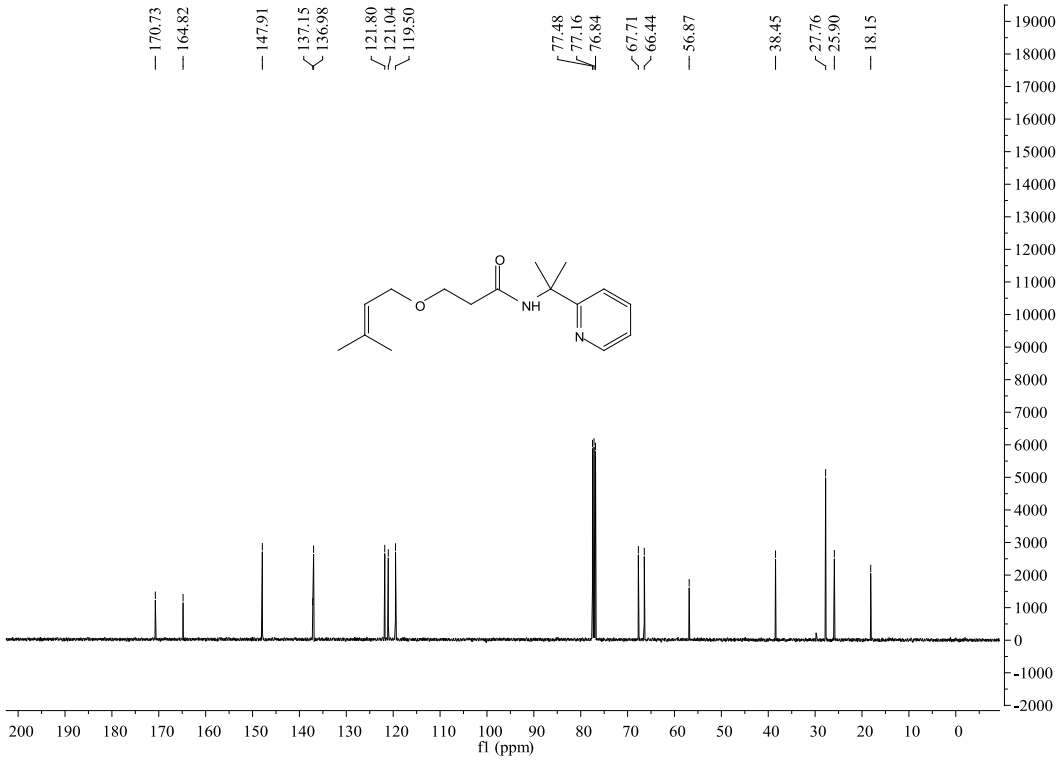
1.03
2.02
2.05
2.04
9.02
3.04
f1 (ppm)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66.44
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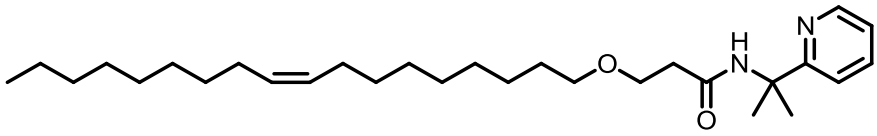
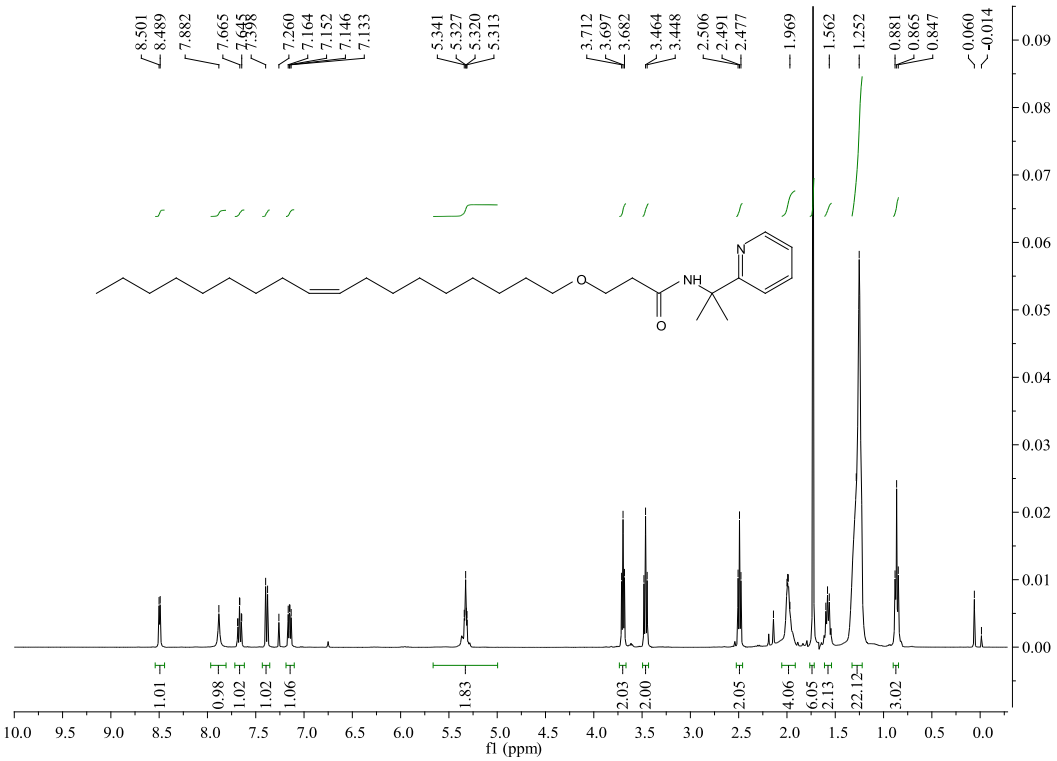
38.45
27.76
25.90
18.15
f1 (ppm)

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7.146
7.133
5.341
5.327
5.320
5.313
3.712
3.697
3.682
3.464
3.448
2.506
2.491
2.477
1.969
1.562
1.252
0.881
0.865
0.847
0.060
-0.014
1.01
0.98
1.02
1.02
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2.03
2.00
2.05
4.06
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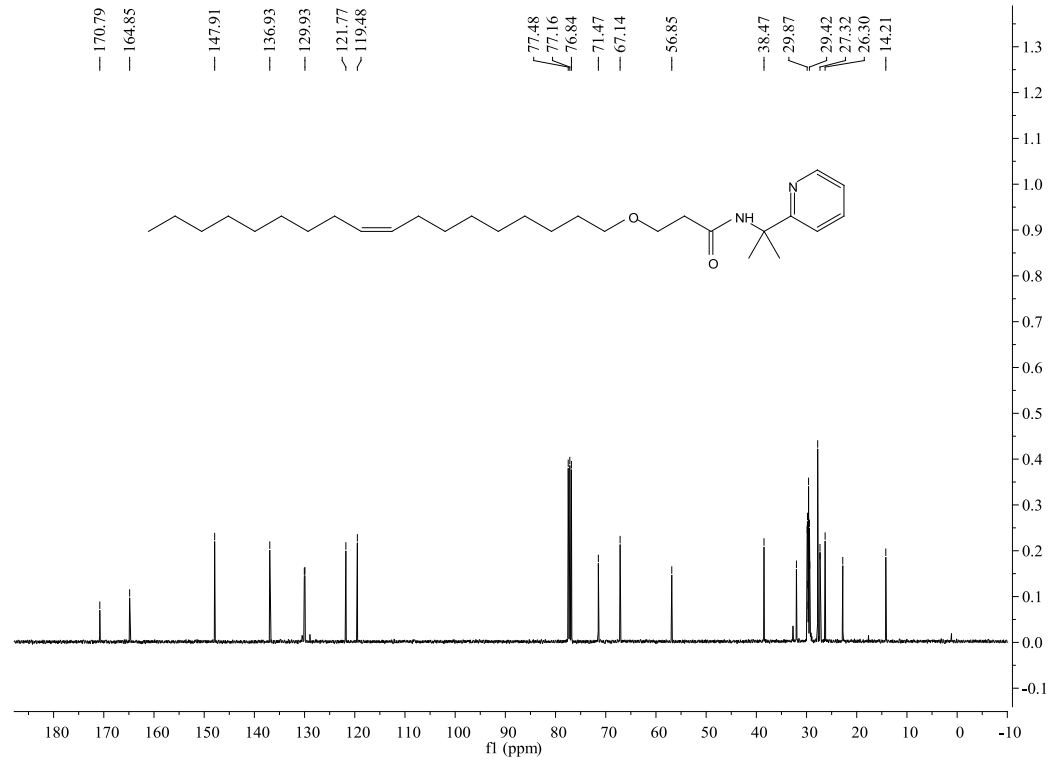
2.13
22.12
3.02
f1 (ppm)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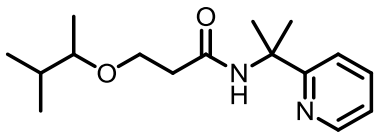
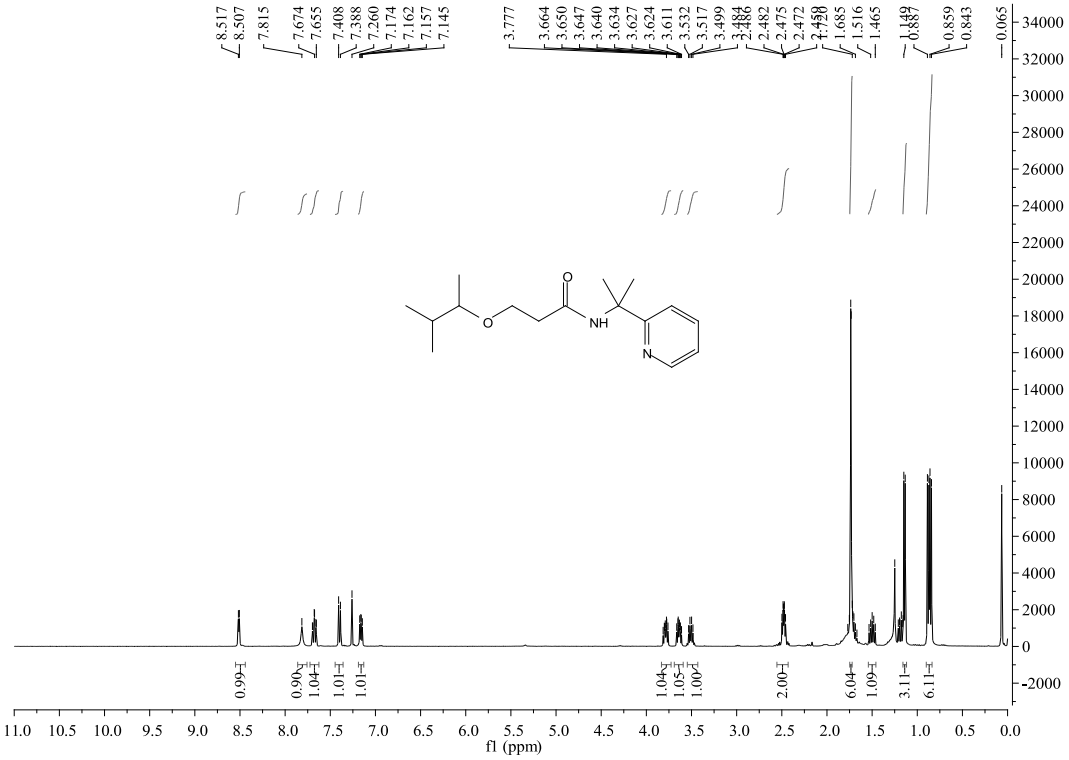
38.47
29.87
29.42
27.32
26.30
14.21
f1 (ppm)

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7.145
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3.664
3.650
3.647
3.640
3.634
3.627
3.624
3.611
3.532
3.517
3.499
2.482
2.475
2.472
1.685
1.516
1.465
1.149
0.887
0.859
0.843
0.065
0.99
0.90
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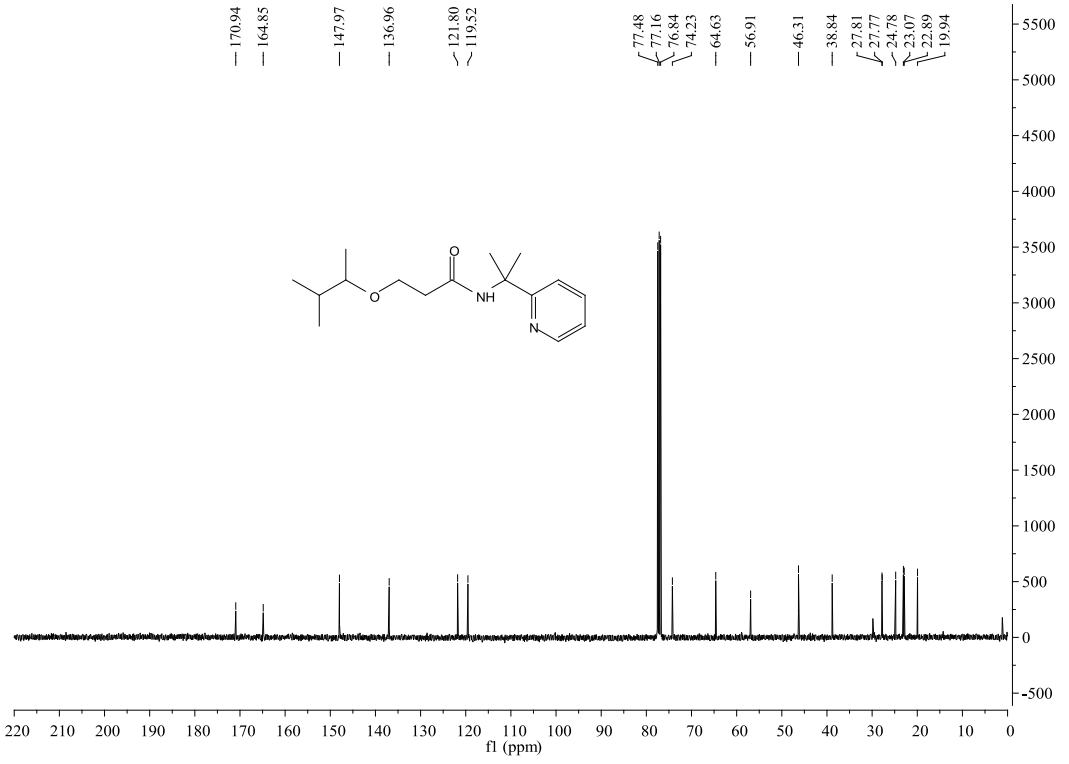
1.01
1.01
1.04
1.05
1.00
2.00
6.04
1.09
3.11
6.11
f1 (ppm)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77.16
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64.63
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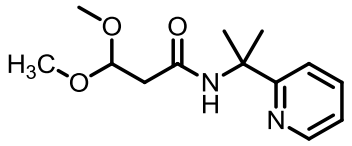
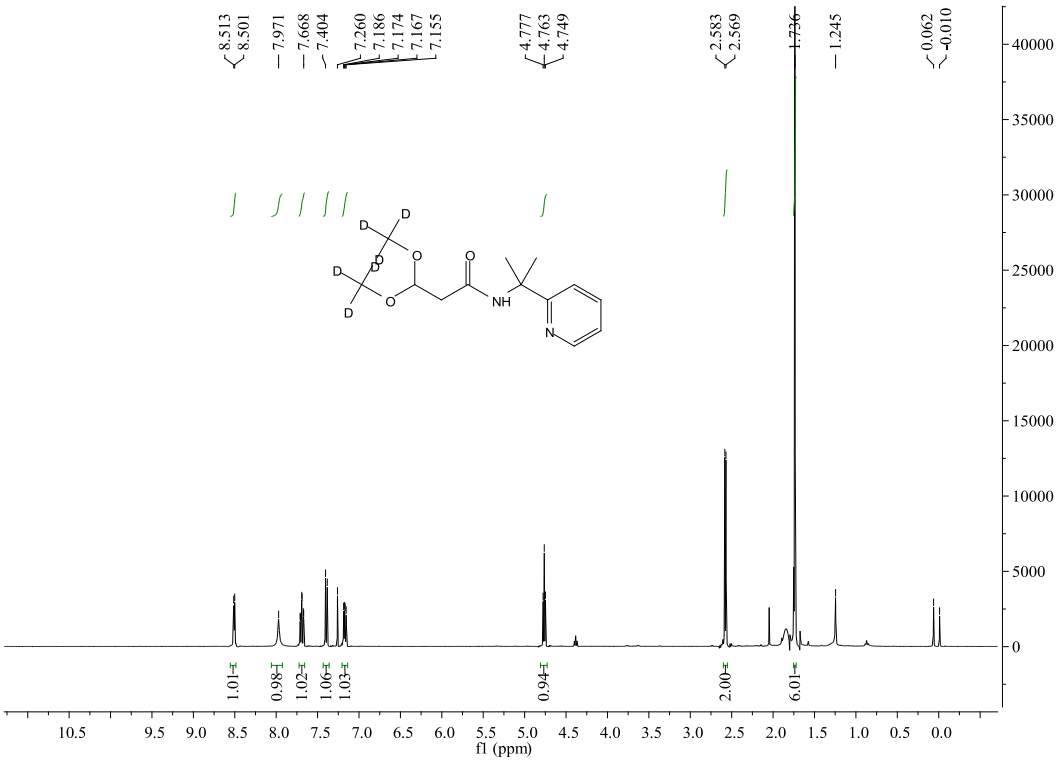
24.78
23.07
22.89
19.94
f1 (ppm)

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4.763
4.749
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2.569
1.736
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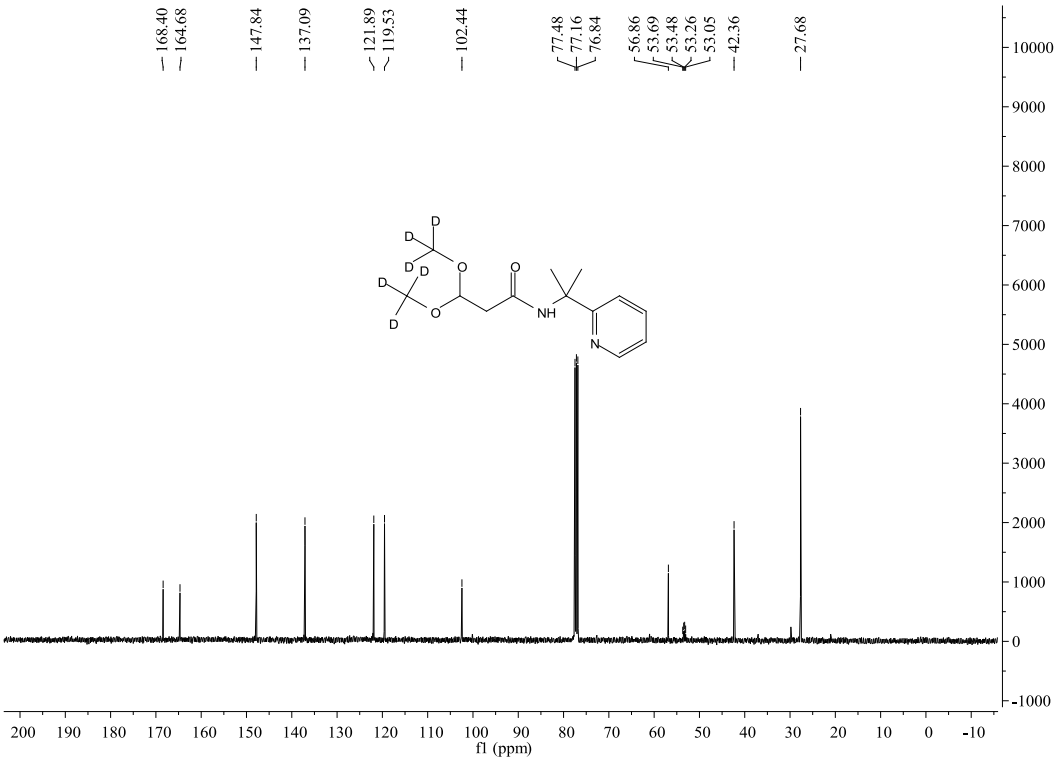
0.98
1.02
1.06
1.03
0.94
2.00
6.01
f1 (ppm)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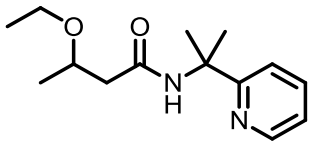
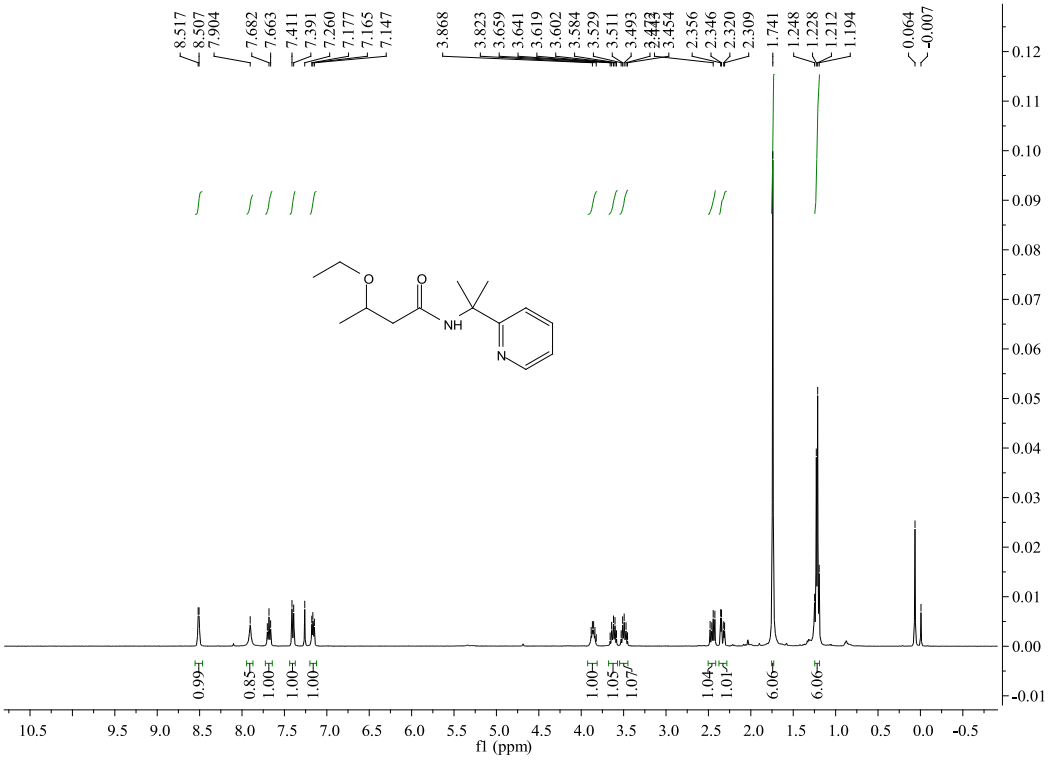
76.84
56.86
53.69
53.48
53.26
53.05
42.36
27.68
f1 (ppm)

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3.823
3.659
3.641
3.619
3.602
3.584
3.529
3.511
3.493
3.454
2.356
2.346
2.320
2.309
1.741
1.248
1.228
1.212
1.194
0.064
-0.007
0.99
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1.00
1.00
1.05
1.07
1.04
1.01
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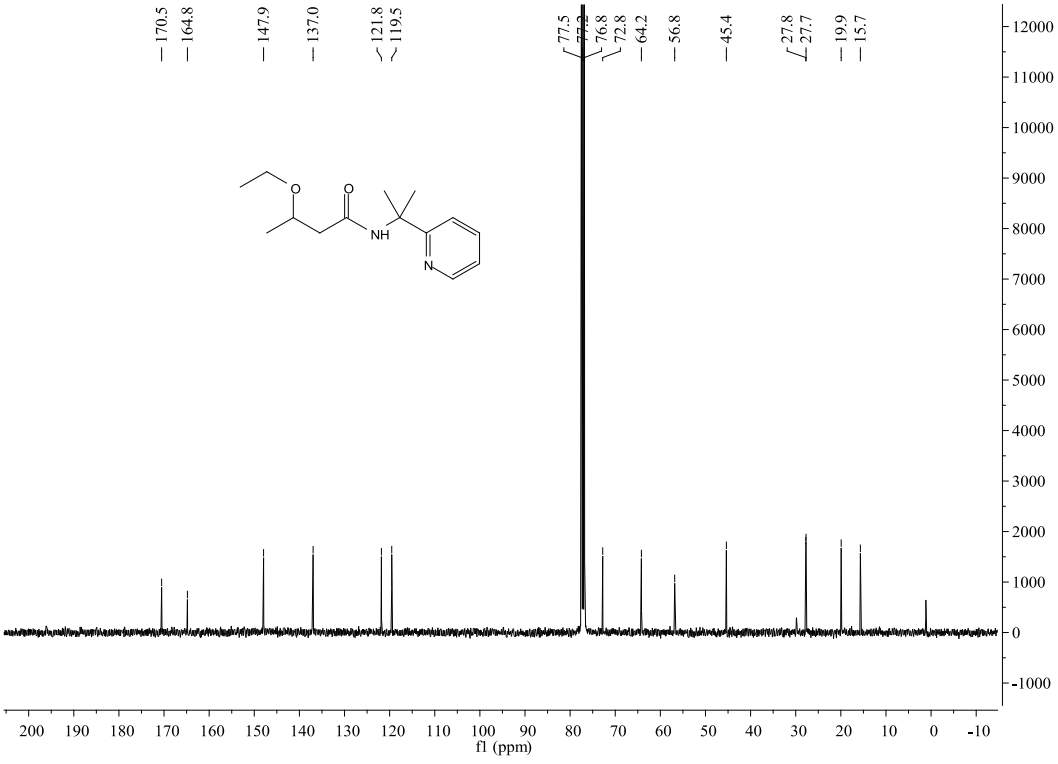
6.06
fl (ppm)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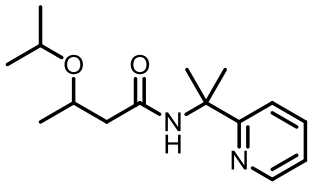
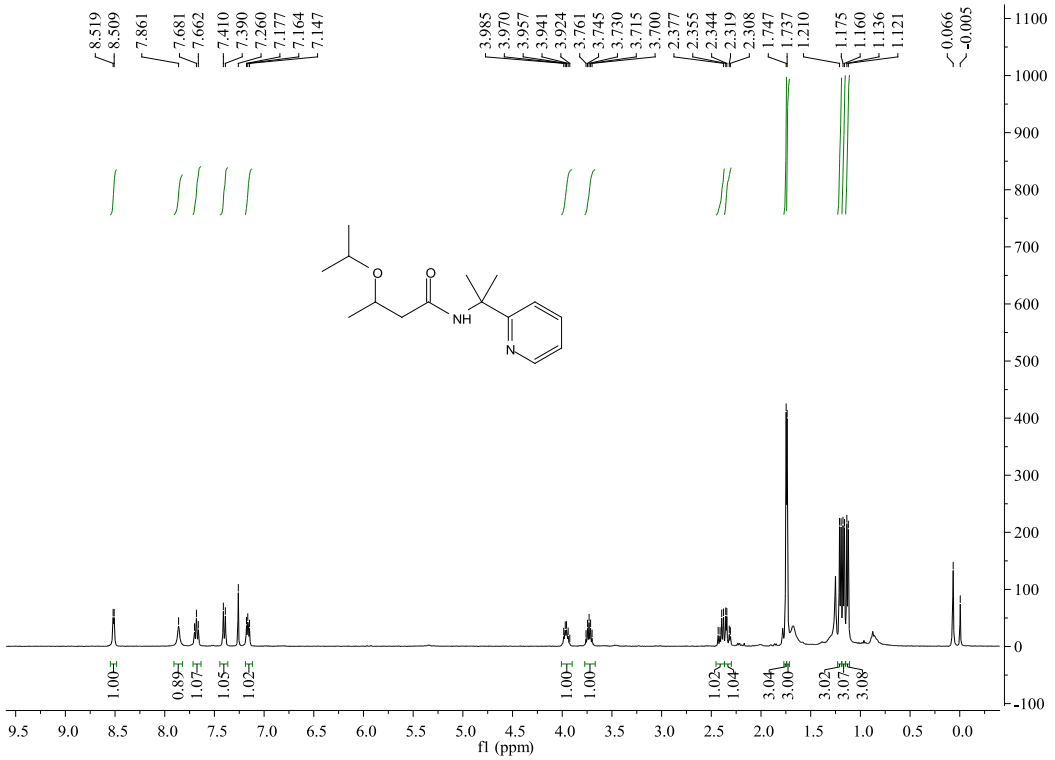
fl (ppm)

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3.970
3.957
3.941
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3.761
3.745
3.730
3.715
3.700
2.377
2.355
2.344
2.319
2.308
1.747
1.737
1.210
1.175
1.160
1.136
1.121
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0.89
1.07
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1.04
3.04
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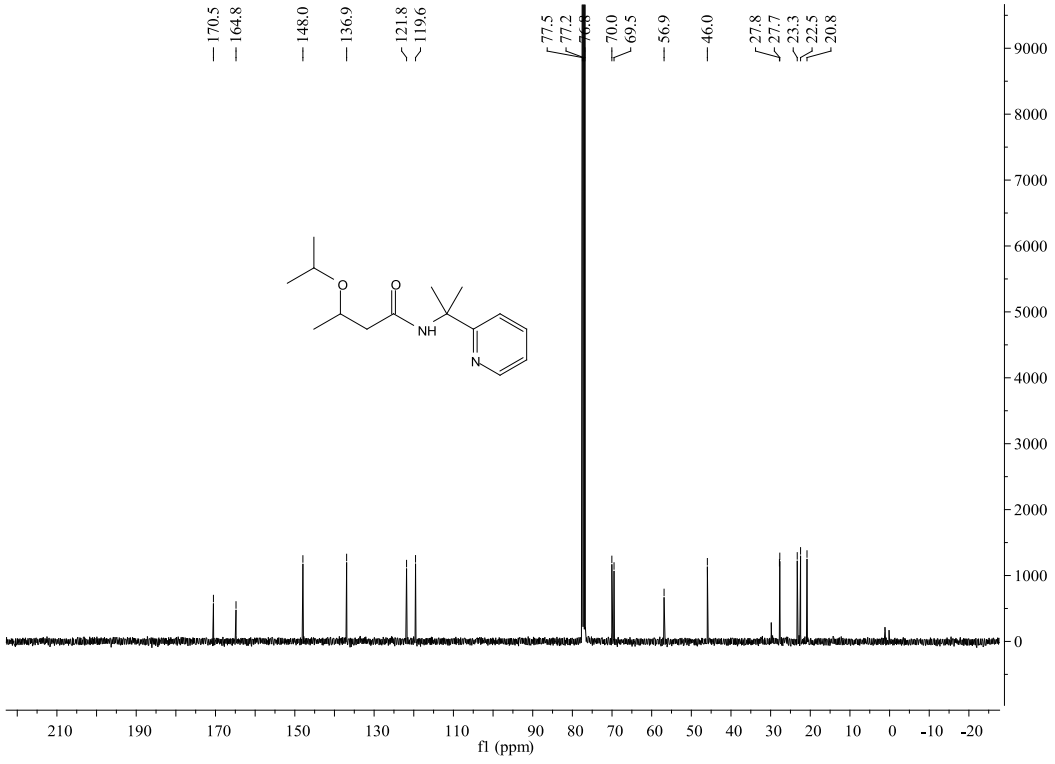
3.02
3.07
3.08
f1 (ppm)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136.9
121.8
119.6
77.5
77.2
76.8
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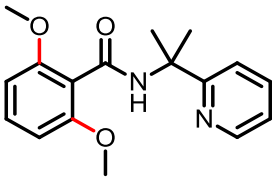
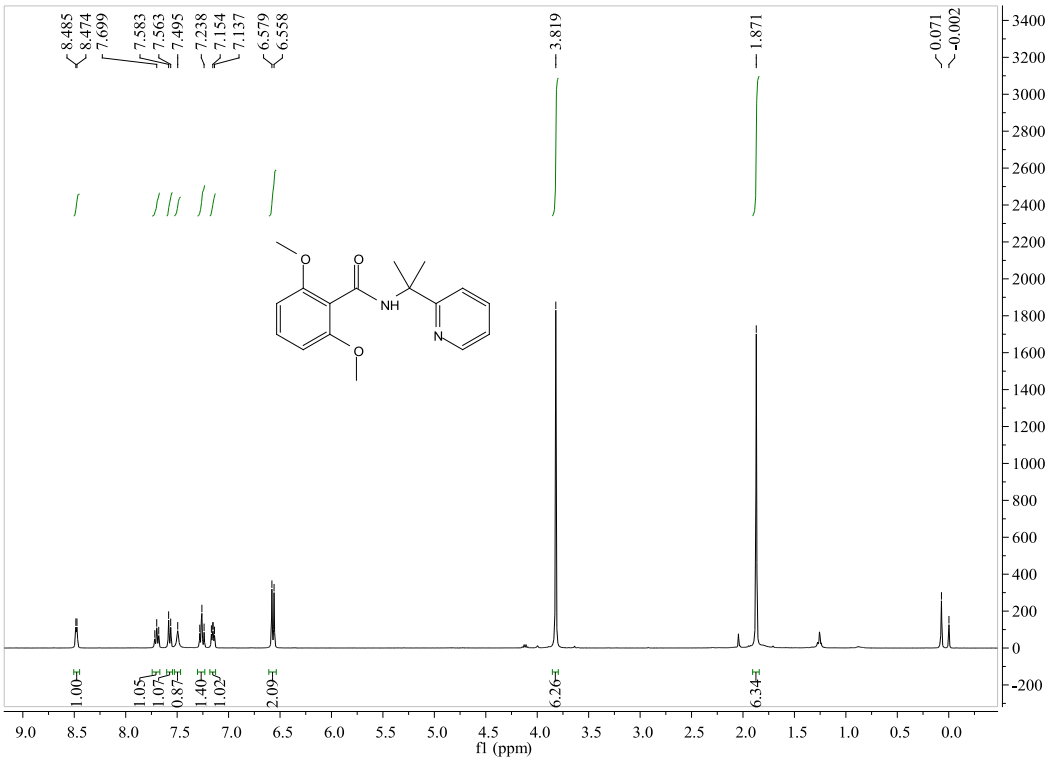
69.5
56.9
46.0
27.8
27.7
23.3
22.5
20.8
f1 (ppm)

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7.154
7.137
6.579
6.558
3.819
1.871
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1.05
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0.87
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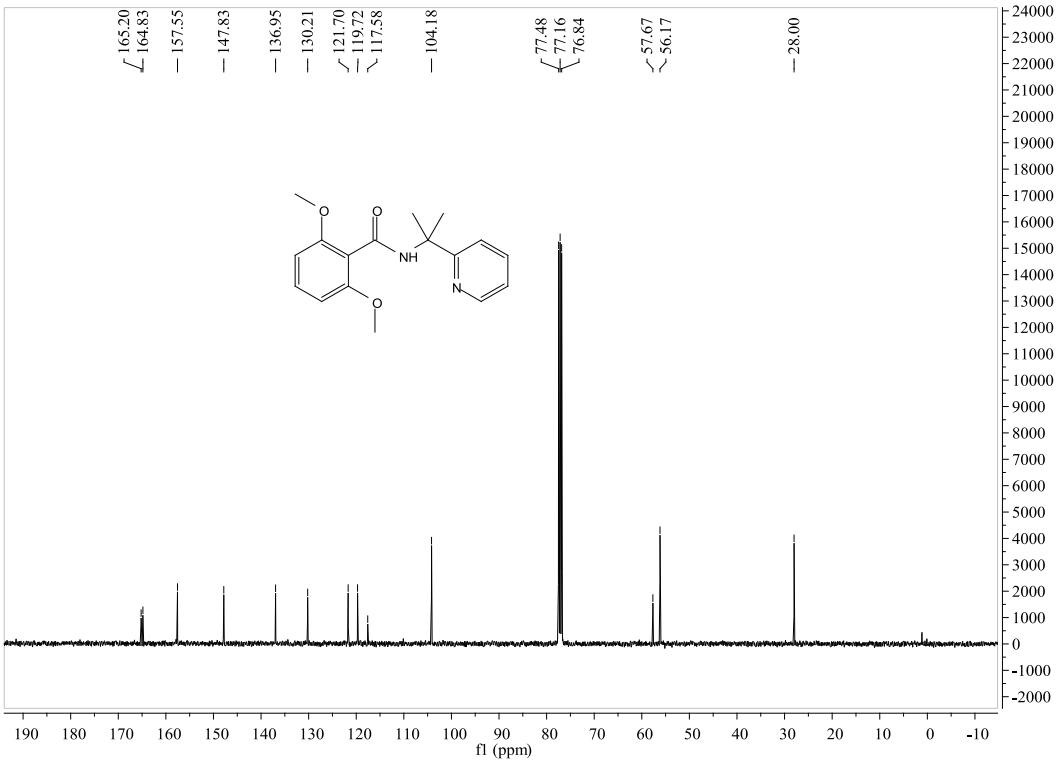
1.02
2.09
6.26
6.34
f1 (ppm)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57.67
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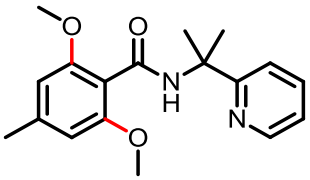
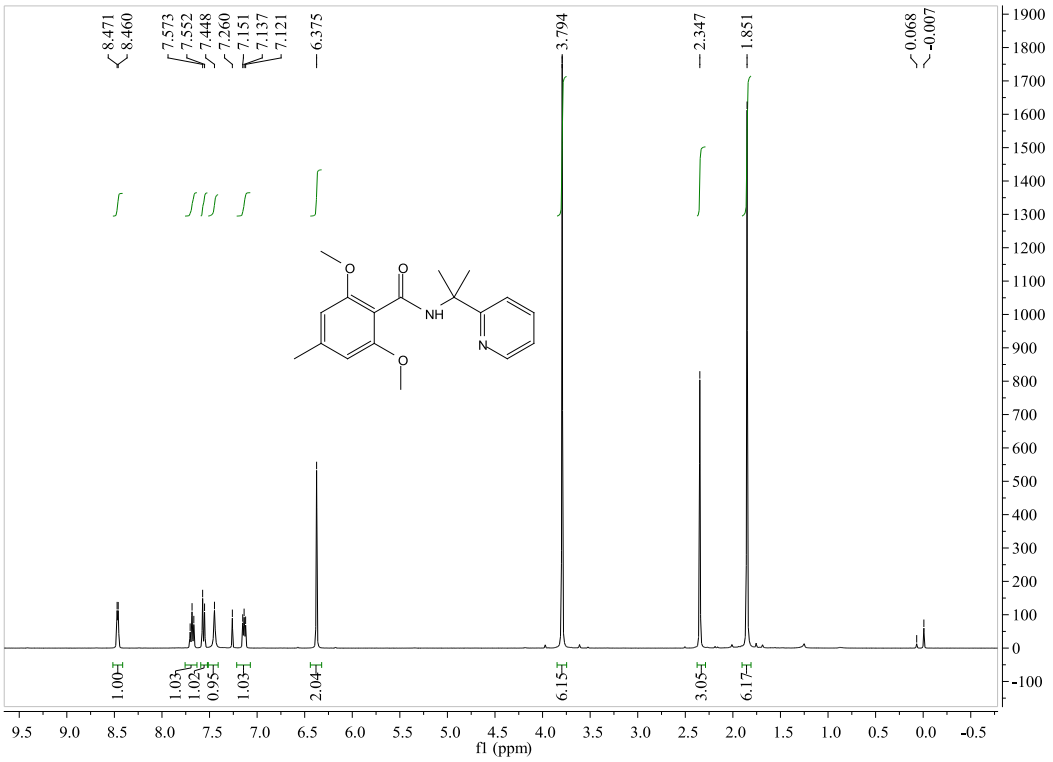
28.00
f1 (ppm)

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7.121
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2.04
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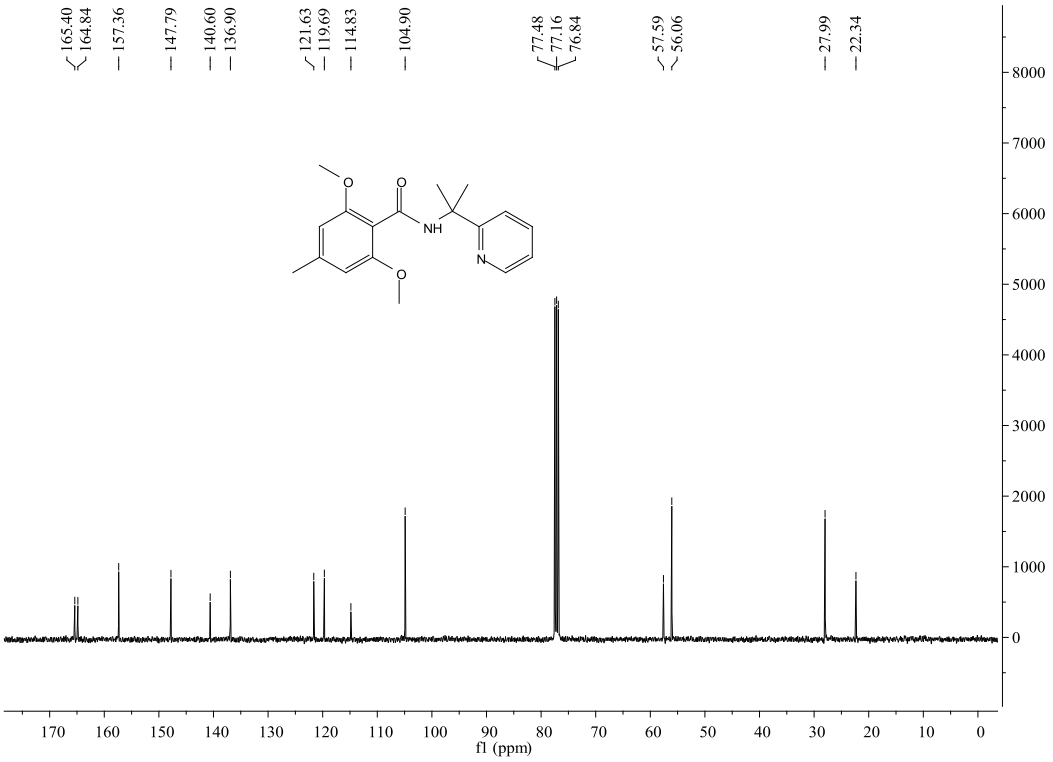
3.05
6.17
fl (ppm)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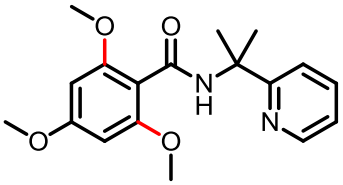
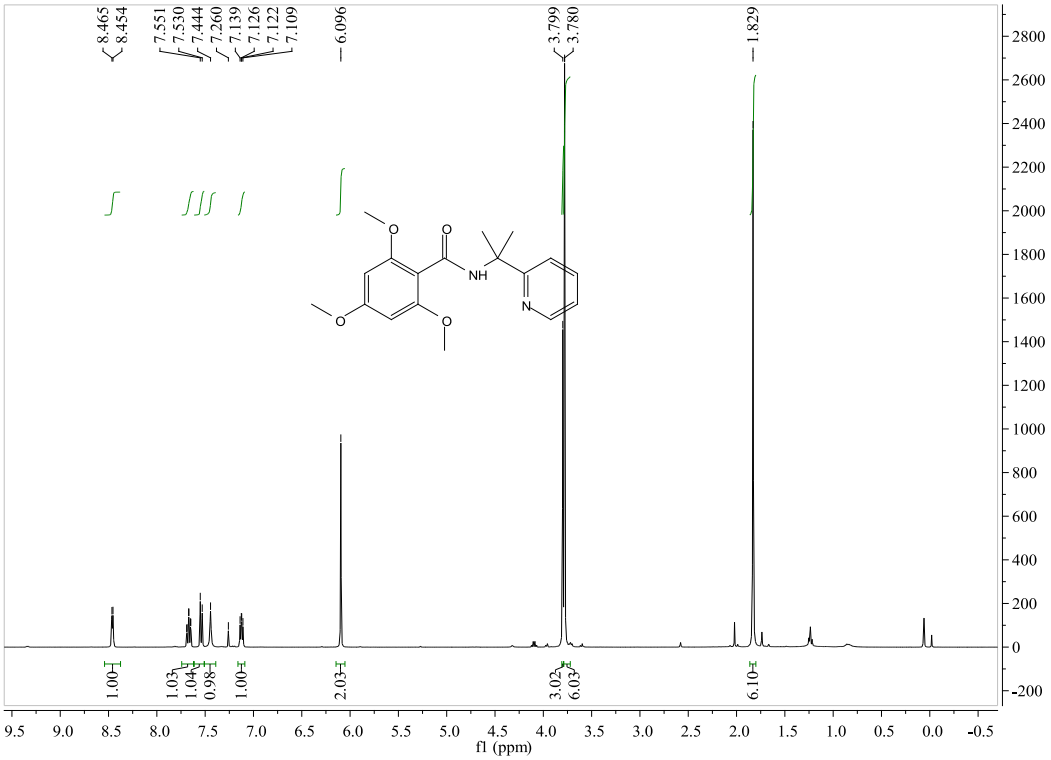
22.34
fl (ppm)

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7.139
7.126
7.122
7.109
6.096
3.799
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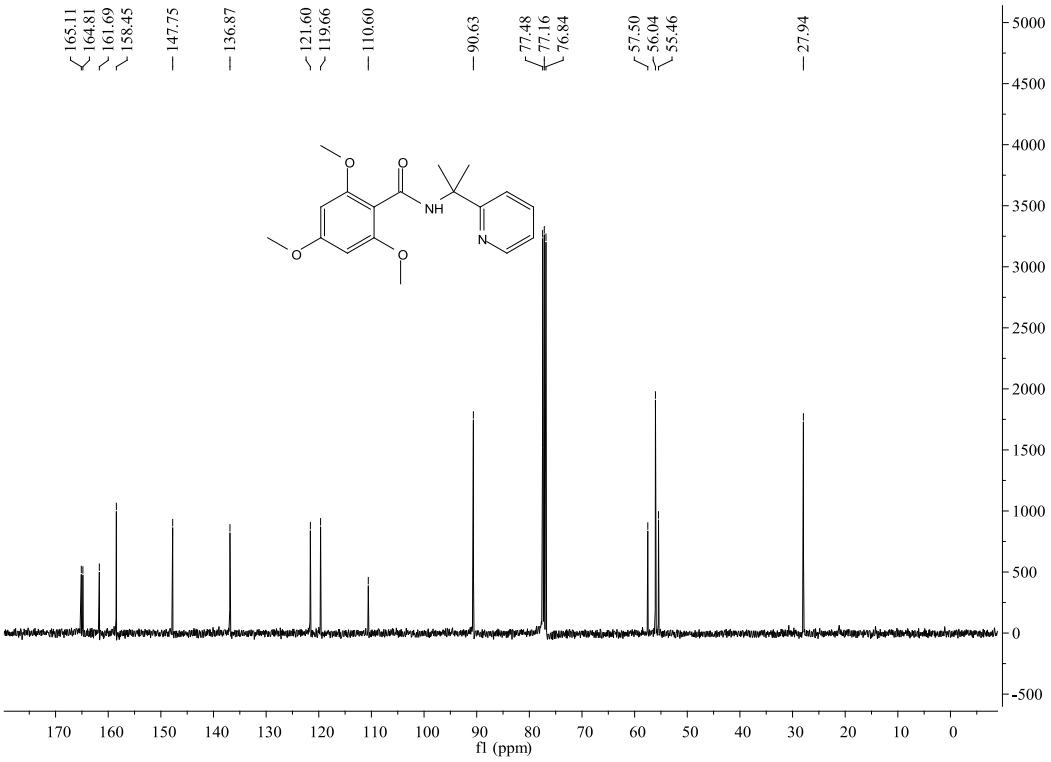
1.03
1.04
0.98
1.00
2.03
3.02
6.03
6.10
fl (ppm)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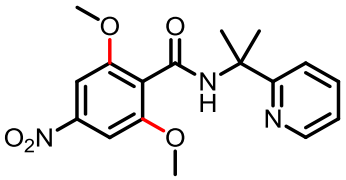
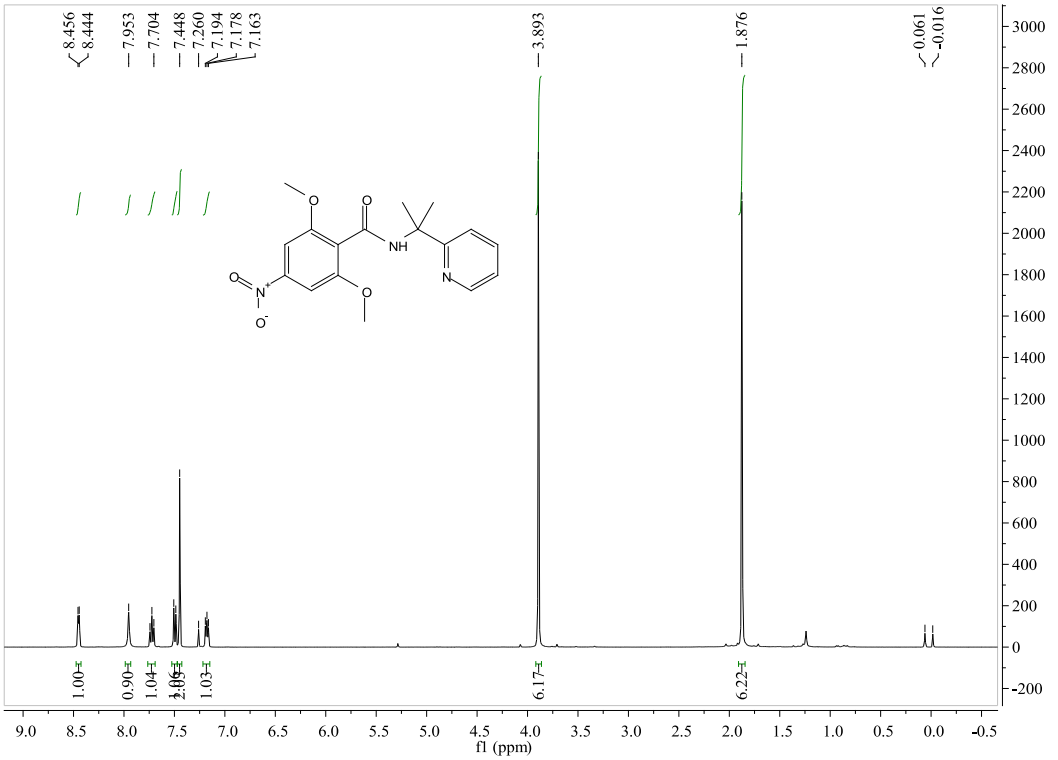
fl (ppm)

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-0.016
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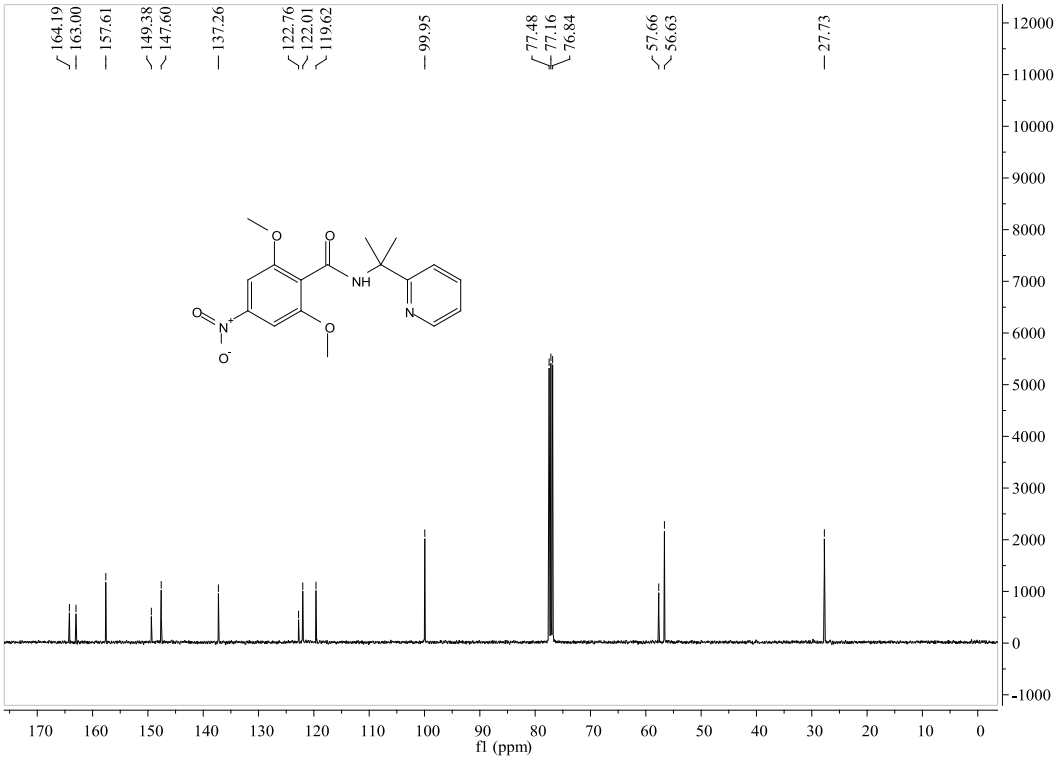
0.90
1.04
2.05
1.03
6.17
6.22
f1 (ppm)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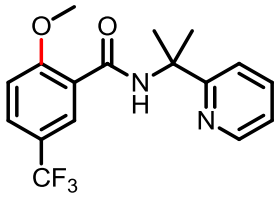
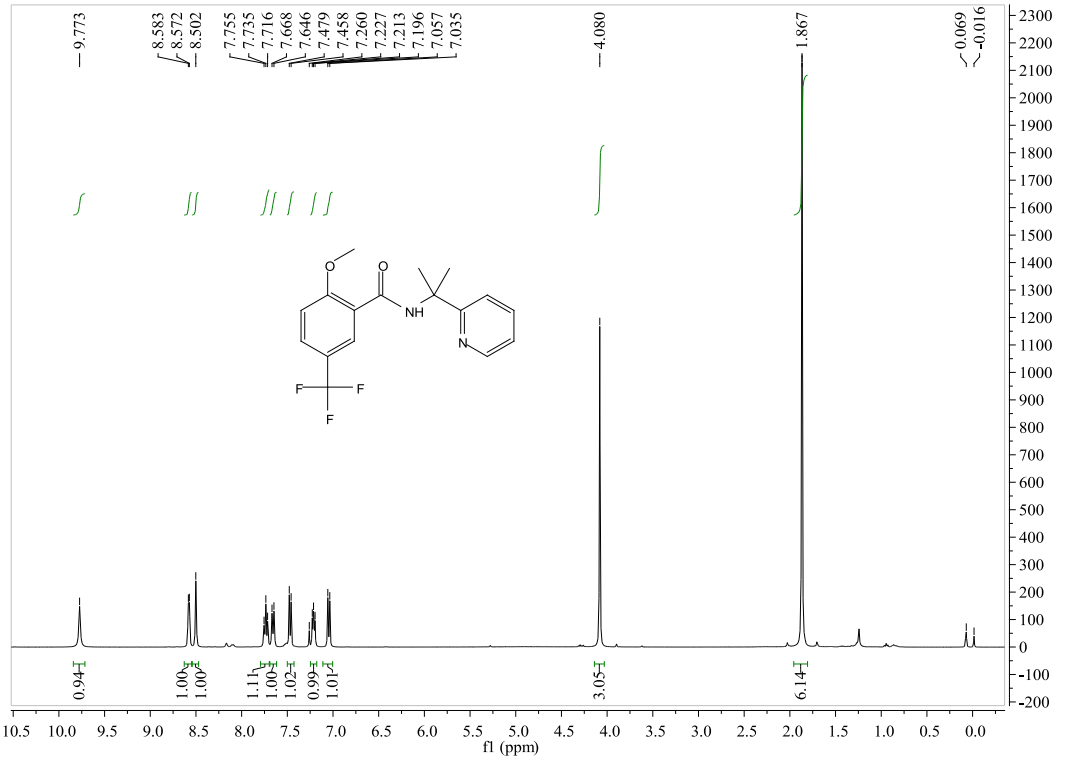
147.60
137.26
122.76
122.01
119.62
99.95
77.48
77.16
76.84
57.66
56.63
27.73
f1 (ppm)

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8.572
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7.735
7.716
7.668
7.646
7.479
7.458
7.260
7.227
7.213
7.196
7.057
7.035
4.080
1.867
0.069
-0.016
0.94
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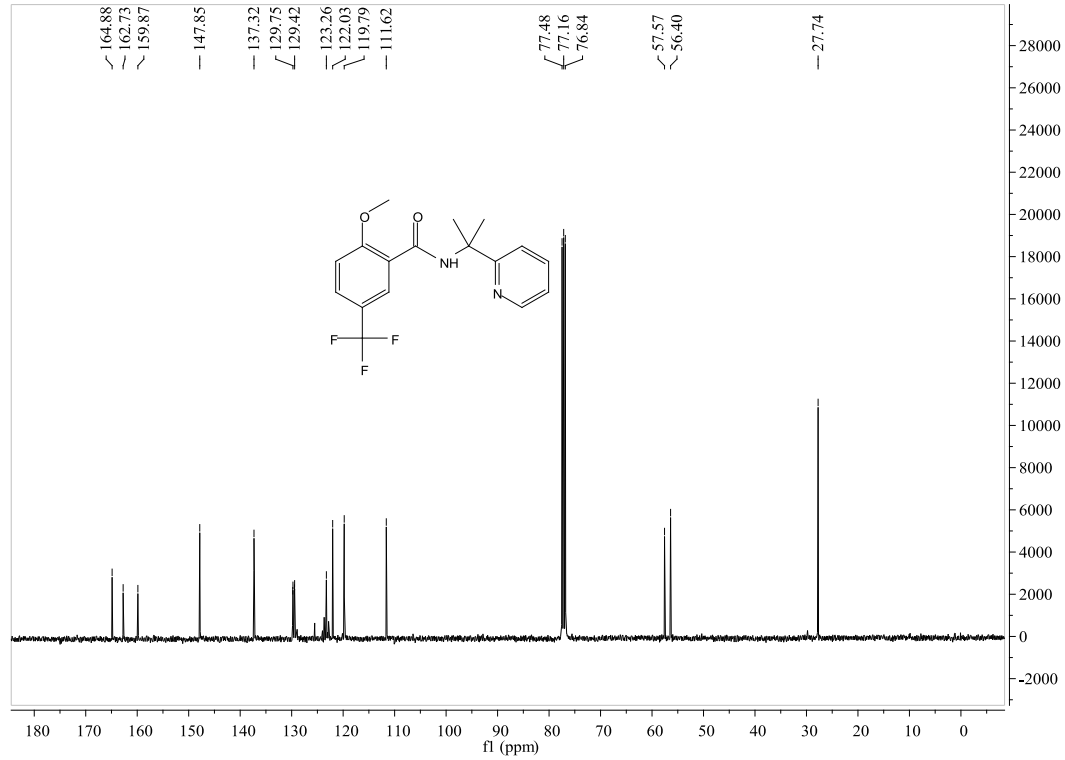
1.00
1.11
1.00
1.02
0.99
1.01
3.05
6.14
f1 (ppm)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159.87
147.85
137.32
129.75
129.42
123.26
122.03
119.79
111.62
77.48
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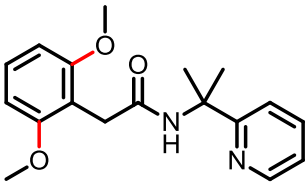
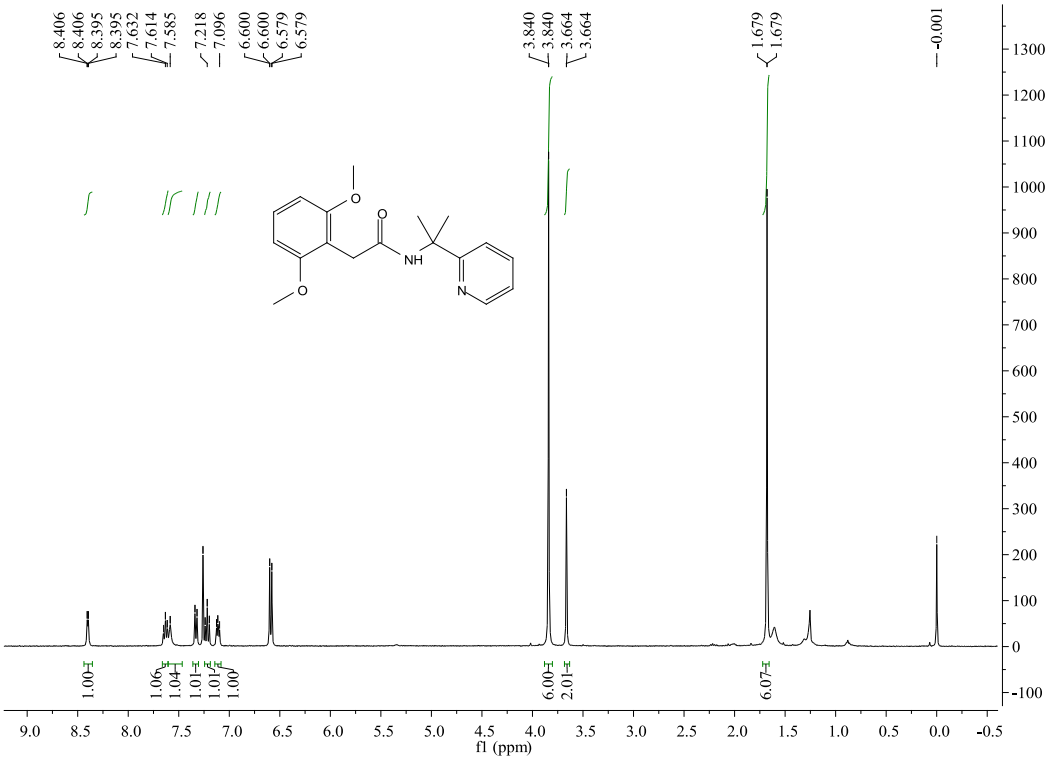
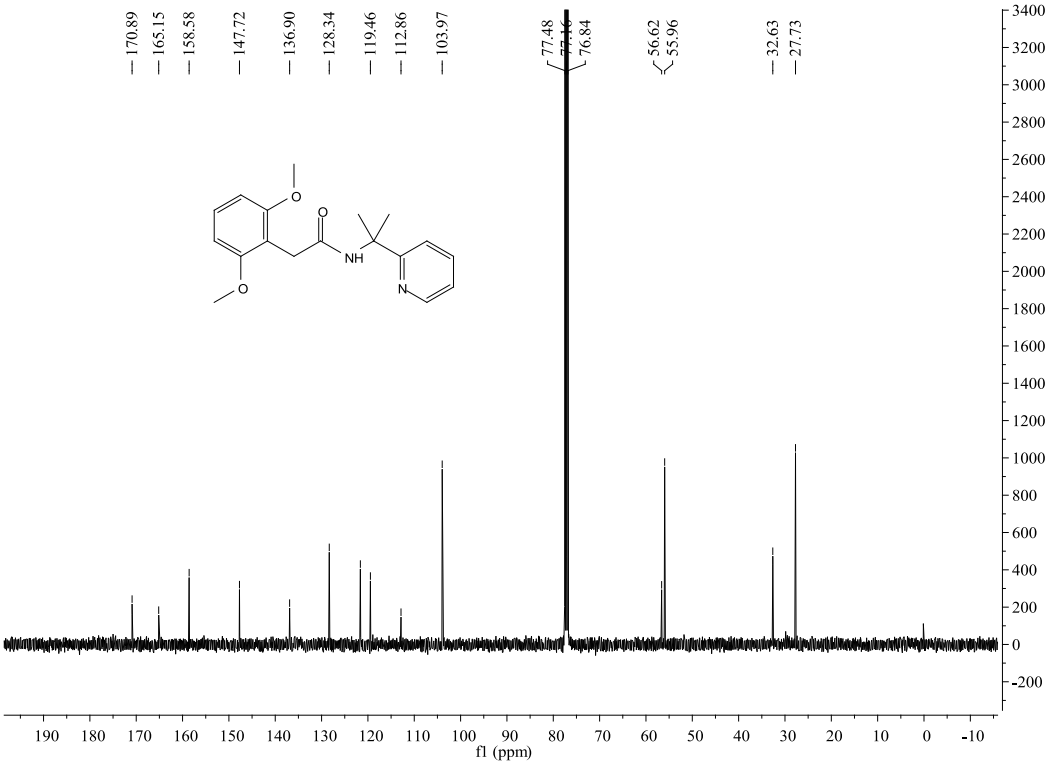
76.84
57.57
56.40
27.74
f1 (ppm)




8.406
8.406
8.395
8.395
7.632
7.614
7.585
7.218
7.096
6.600
6.600
6.579
6.579
3.840
3.840
3.664
3.664
1.679
1.679
-0.001
1.00
1.06
1.04
1.01
1.01
1.01
1.00
6.00
2.01
6.07
f1 (ppm)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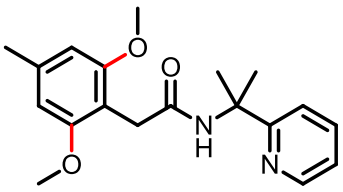
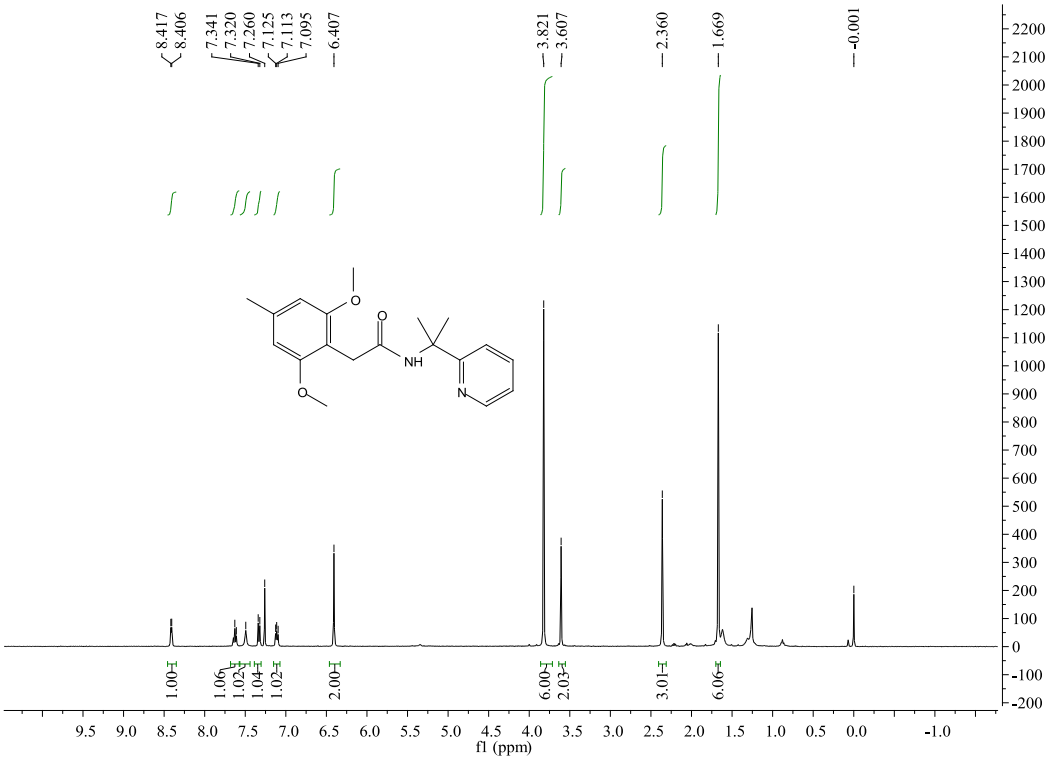
165.15
158.58
147.72
136.90
128.34
119.46
112.86
103.97
77.48
77.16
76.84
56.62
55.96
32.63
27.73
f1 (ppm)

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8.406
7.341
7.320
7.260
7.125
7.113
7.095
6.407
3.821
3.607
2.360
1.669
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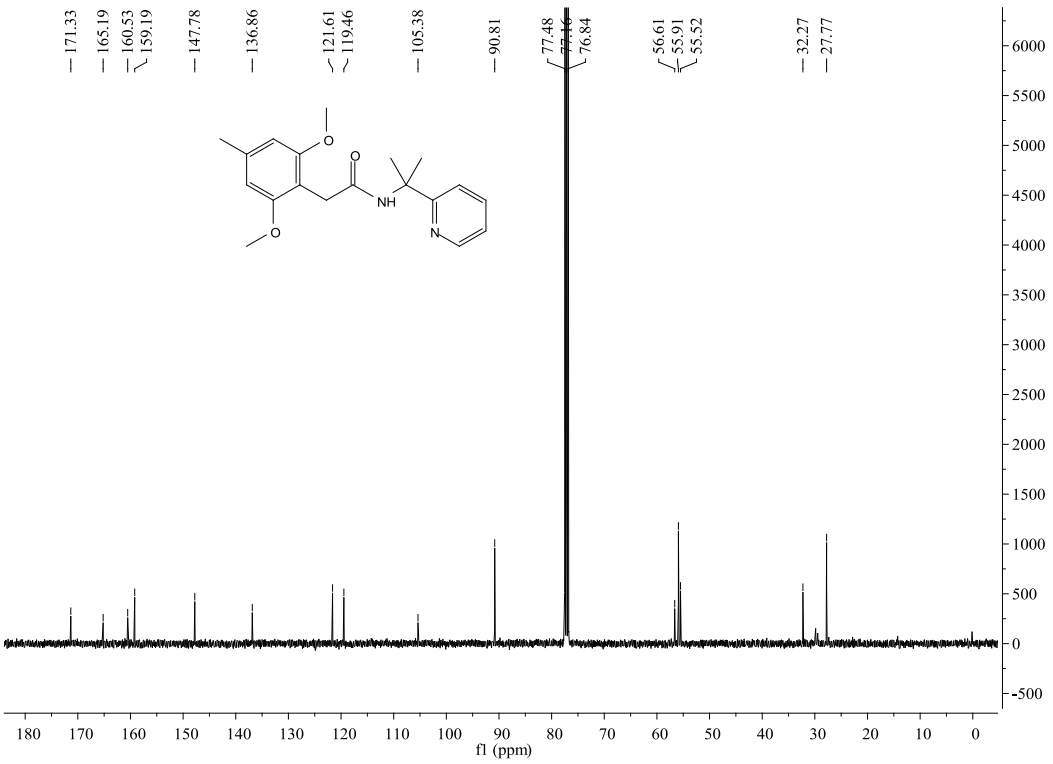
1.00
1.06
1.03
1.04
1.02
2.00
6.00
2.03
3.01
6.06
f1 (ppm)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160.53
159.19
147.78
136.86
121.61
119.46
105.38
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77.48
77.16
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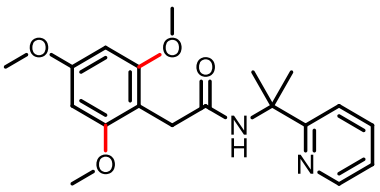
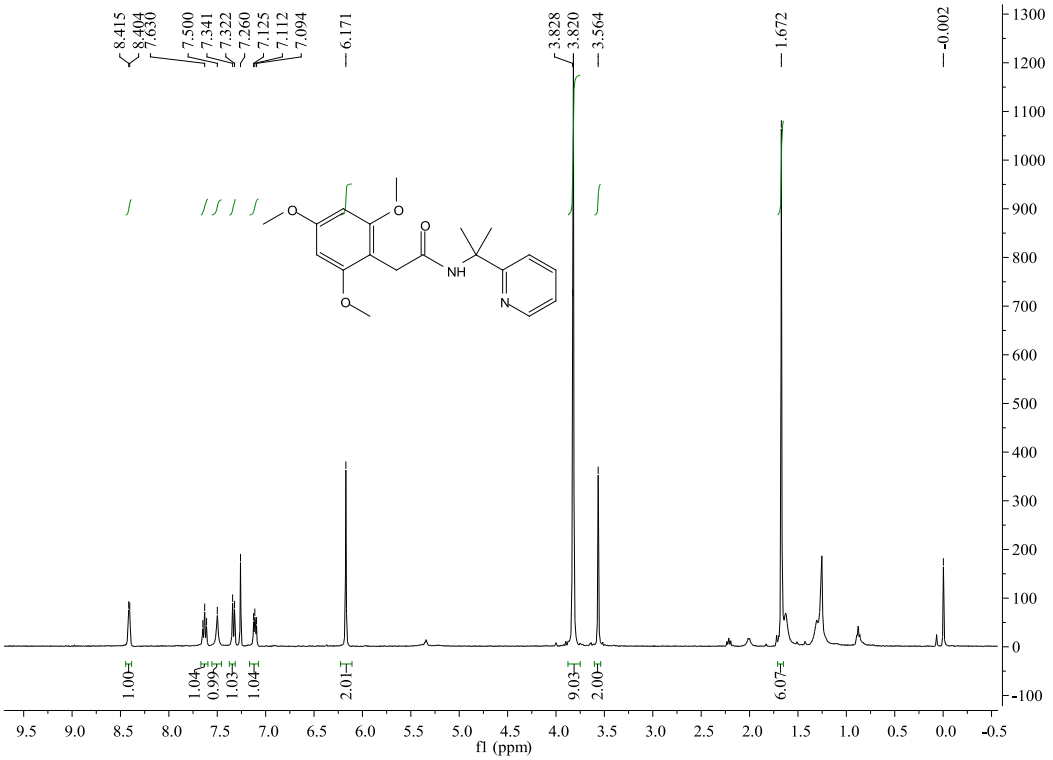
55.91
55.52
32.27
27.77
f1 (ppm)

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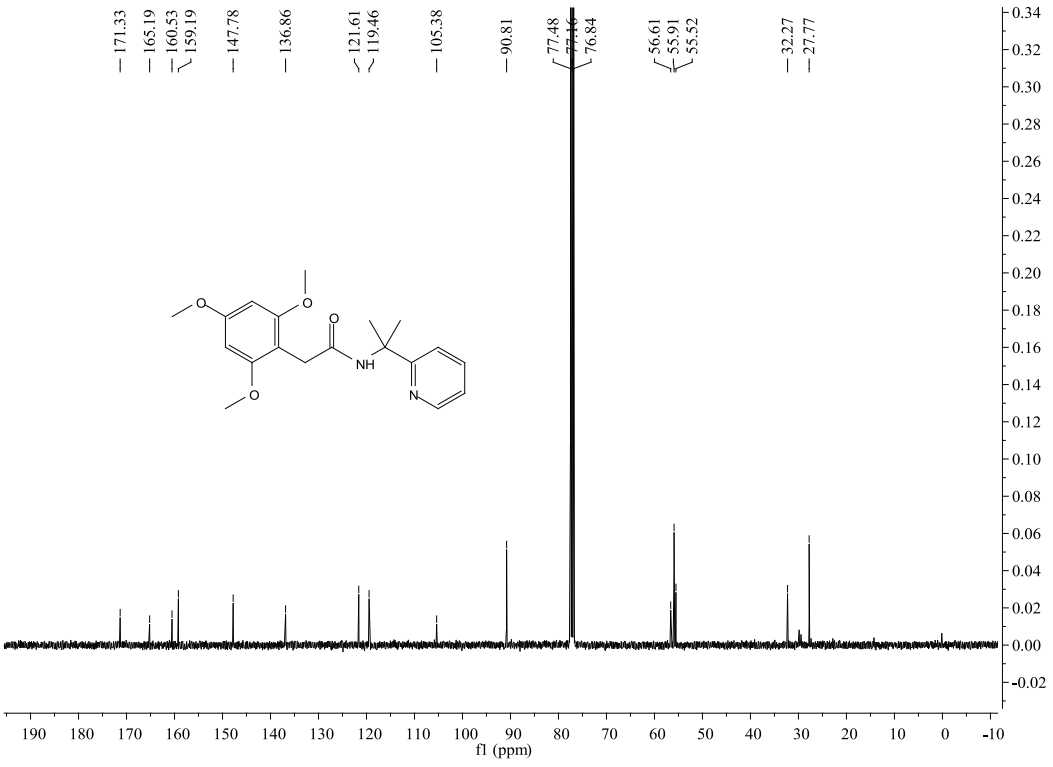
8.404
7.630
7.500
7.341
7.322
7.260
7.125
7.112
7.094
6.171
3.828
3.820
3.564
1.672
-0.002
1.00
1.04
0.99
1.03
1.04
2.01
9.03
2.00
6.07
f1 (ppm)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165.19
160.53
159.19
147.78
136.86
121.61
119.46
105.38
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77.48
77.16
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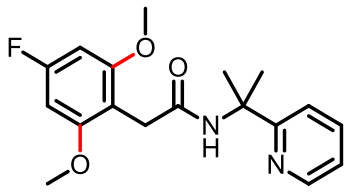
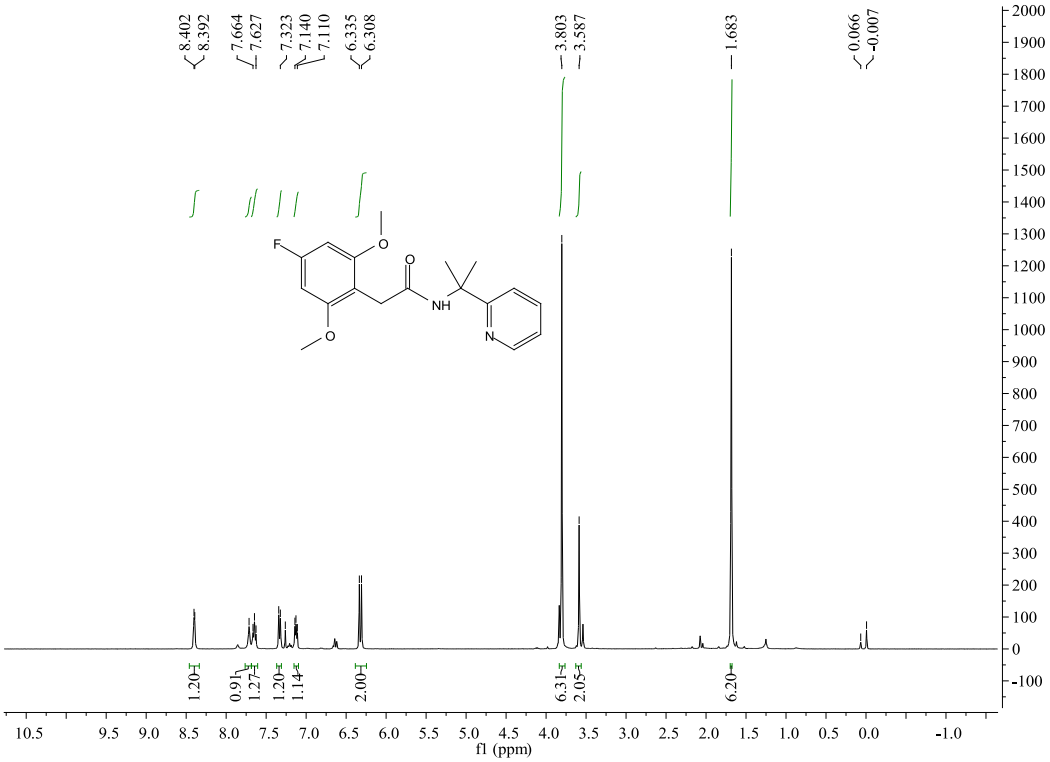
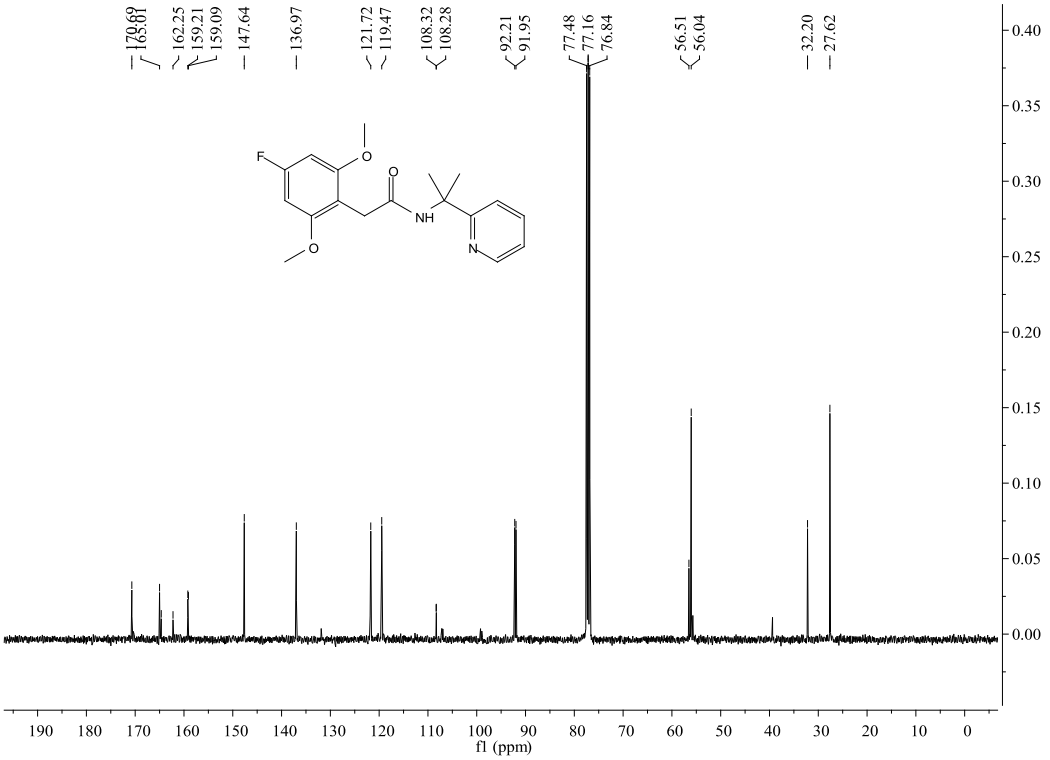
55.91
55.52
32.27
27.77
f1 (ppm)


F
O
O
N
H
O
N


8.402
8.392
7.664
7.627
7.323
7.140
7.110
6.335
6.308
3.803
3.587
1.683
0.066
-0.007
1.20
0.91
1.27
1.20
1.14
2.00
6.31
2.05
6.20
f1 (ppm)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165.01
162.25
159.21
159.09
147.64
136.97
121.72
119.47
108.32
108.28
92.21
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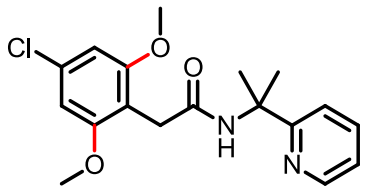
56.51
56.04
32.20
27.62
f1 (ppm)


Cl
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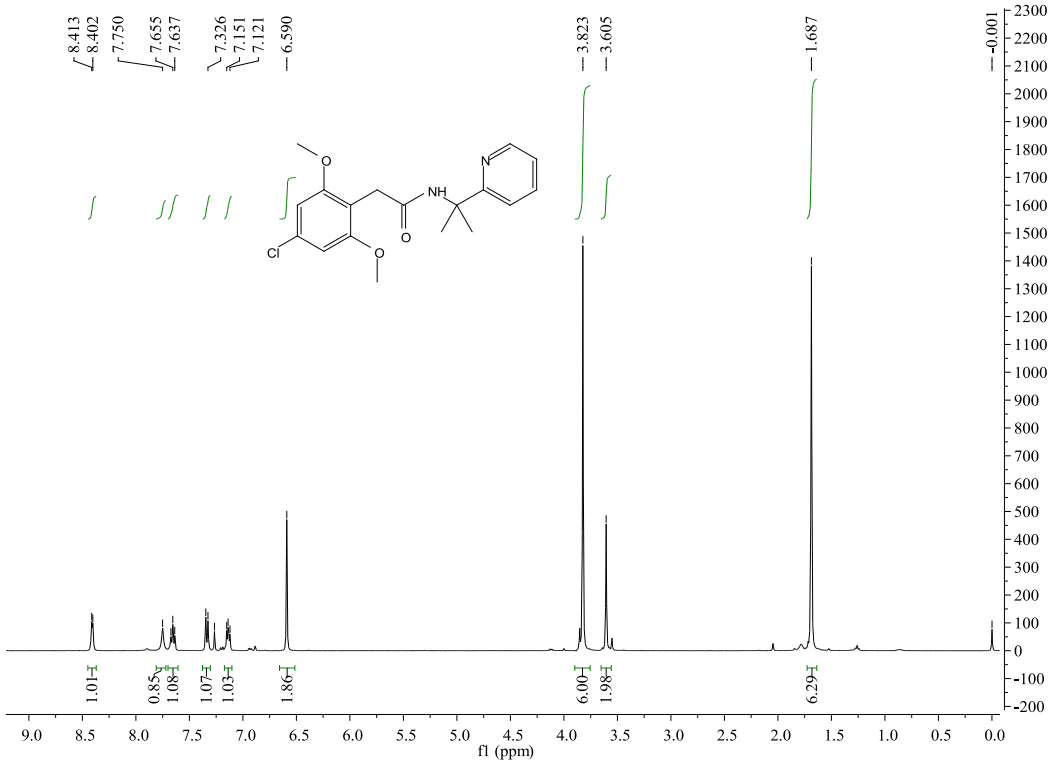
O
N
H
O
N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8.402
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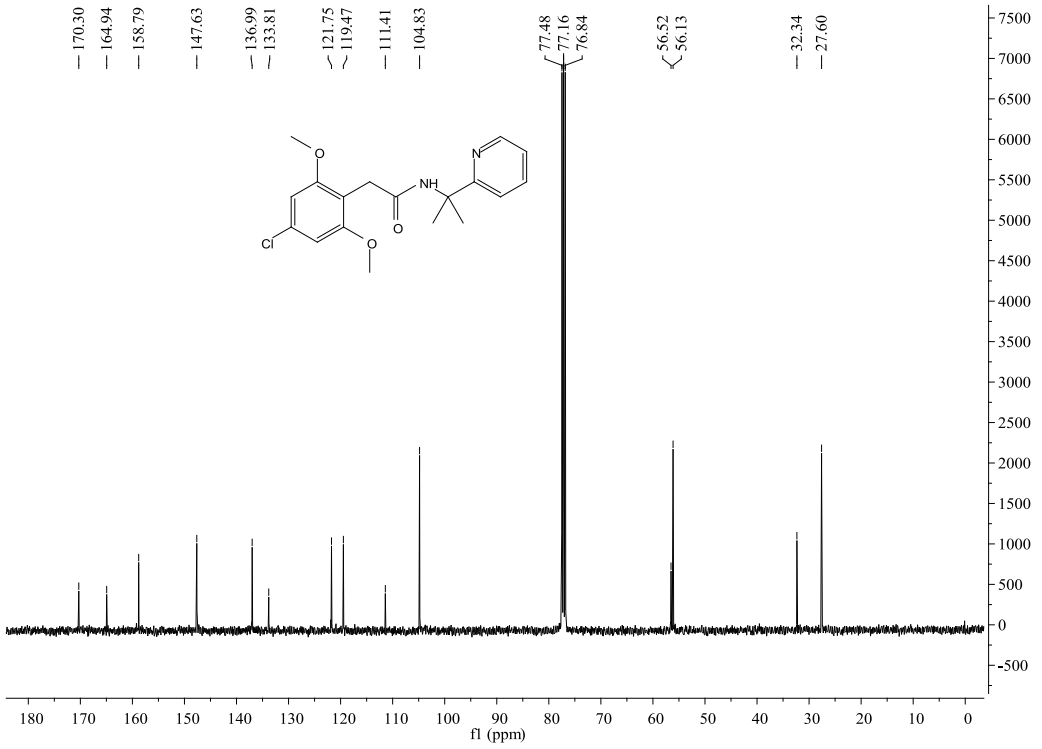
7.655
7.637
7.326
7.151
7.121
6.590
3.823
3.605
1.687
-0.001
1.01
0.85
1.08
1.07
1.03
1.86
6.00
1.98
6.29
fl (ppm)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147.63
136.99
133.81
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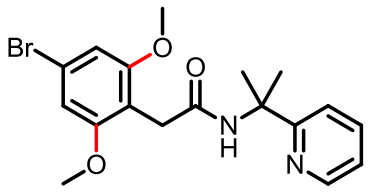
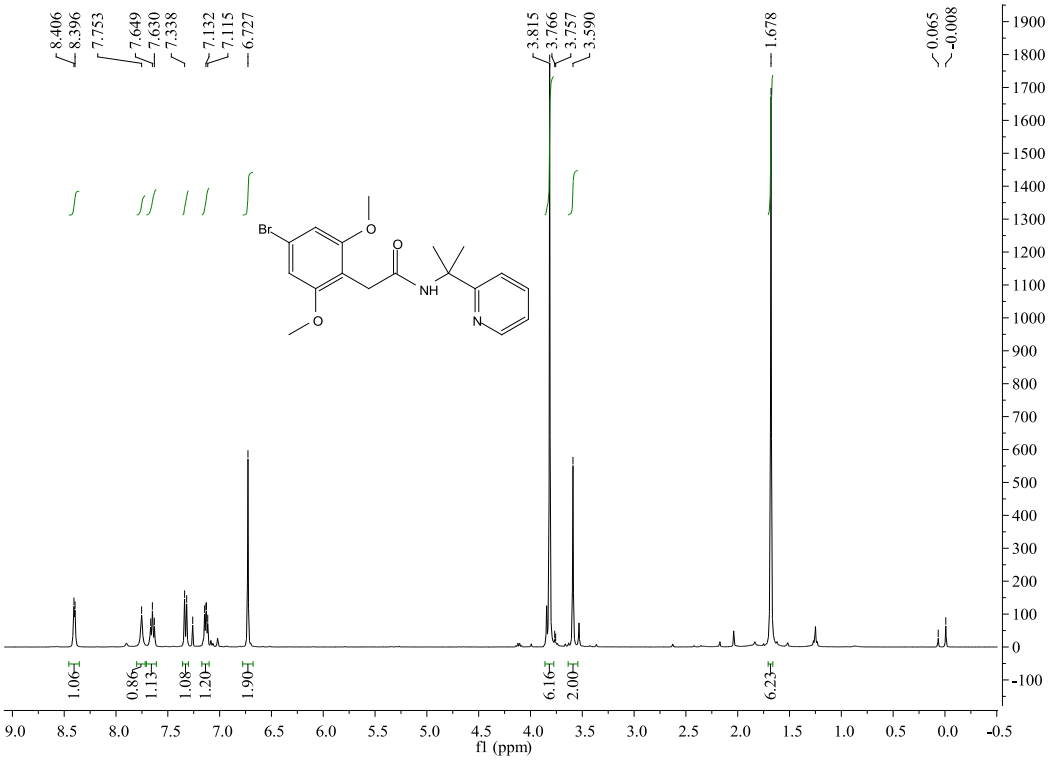
119.47
111.41
104.83
77.48
77.16
76.84
56.52
56.13
32.34
27.60
fl (ppm)


Br
O
O
N
H
O
N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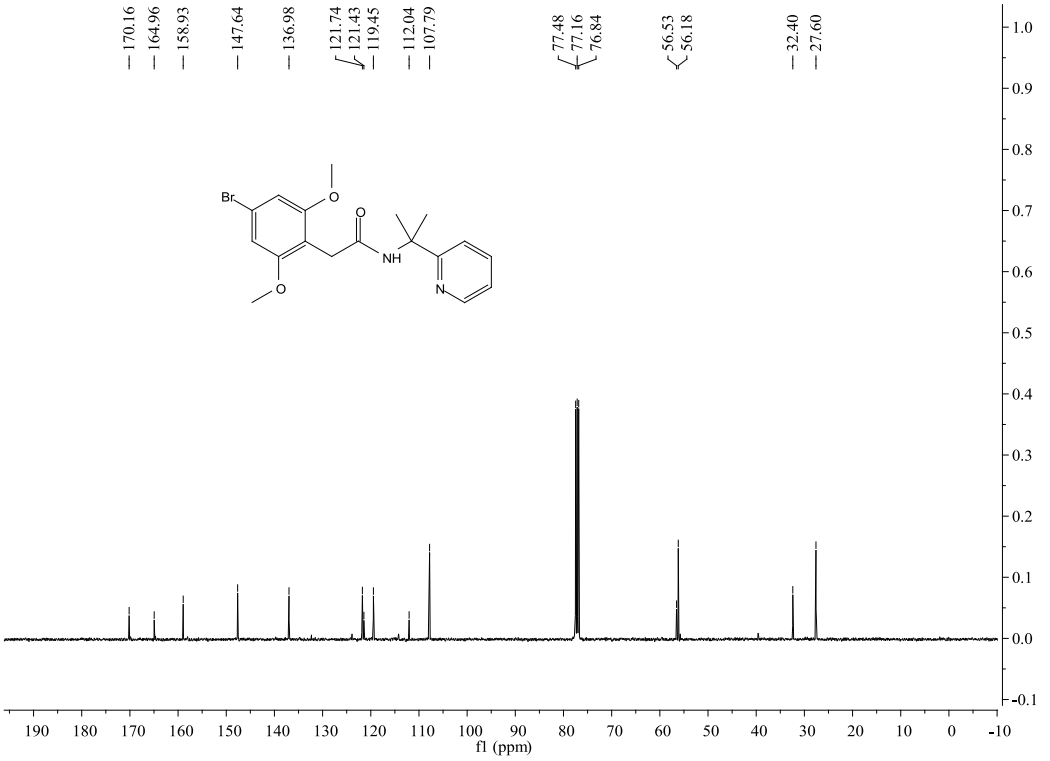
7.630
7.338
7.132
7.115
6.727
3.815
3.766
3.757
3.590
1.678
0.065
-0.008
1.06
0.86
1.13
1.08
1.20
1.90
6.16
2.00
6.23
fl (ppm)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136.98
121.74
121.43
119.45
112.04
107.79
77.48
77.16
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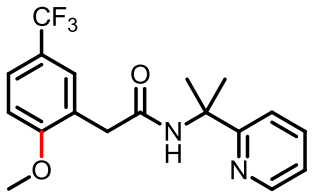
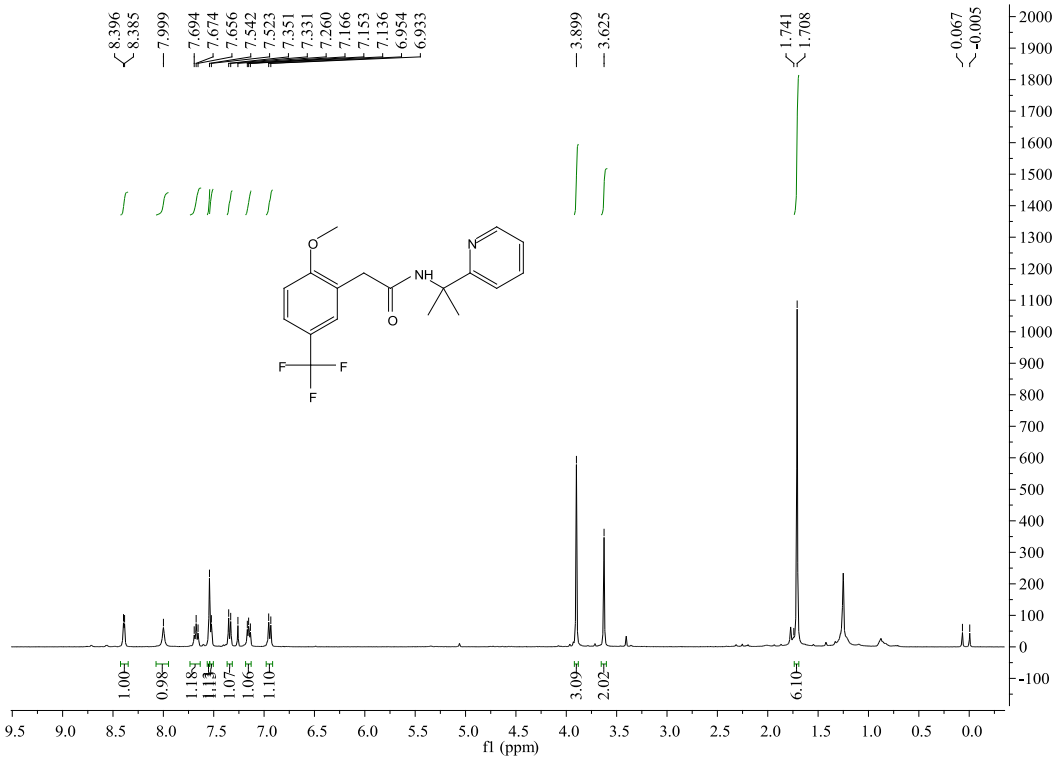
56.53
56.18
32.40
27.60
fl (ppm)


CF3
O
N
H
O
N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7.656
7.542
7.523
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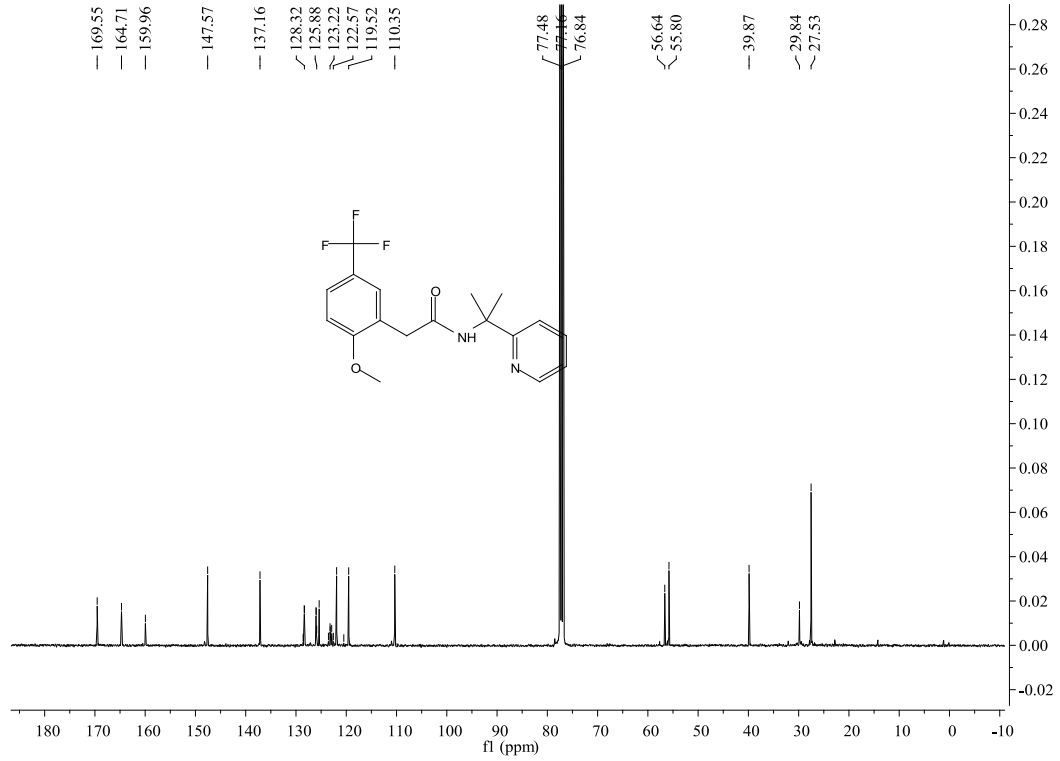
7.331
7.260
7.166
7.153
7.136
6.954
6.933
3.899
3.625
1.741
1.708
0.067
-0.005
1.00
0.98
1.18
1.13
1.07
1.06
1.10
3.09
2.02
6.10
f1 (ppm)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128.32
125.88
123.22
122.57
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77.48
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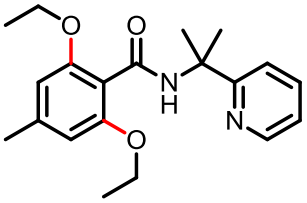
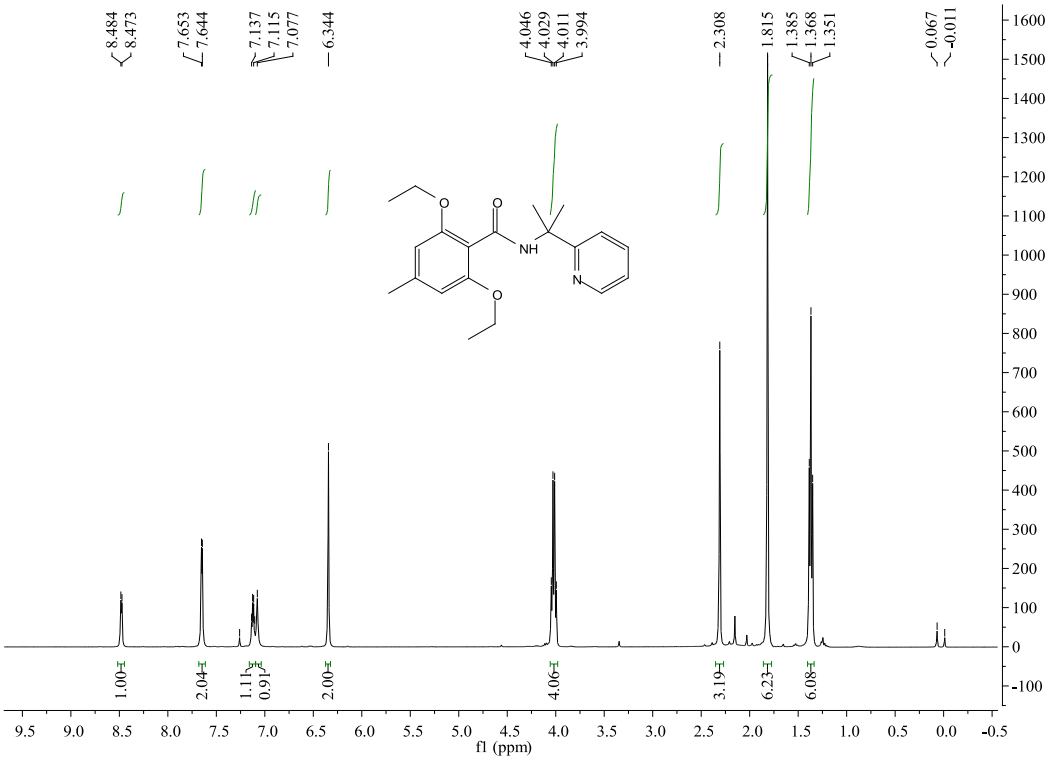
76.84
56.64
55.80
39.87
29.84
27.53
f1 (ppm)

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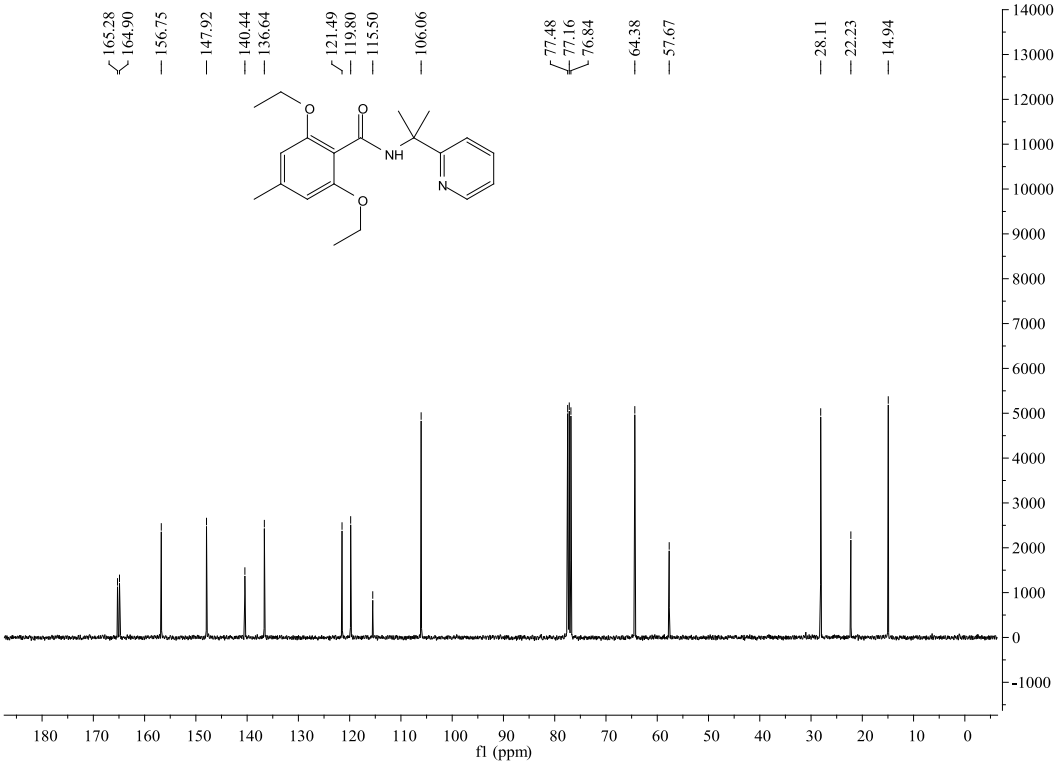
7.077
6.344
4.046
4.029
4.011
3.994
2.308
1.815
1.385
1.368
1.351
0.067
-0.011
1.00
2.04
1.11
0.91
2.00
4.06
3.19
6.23
6.08
f1 (ppm)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77.16
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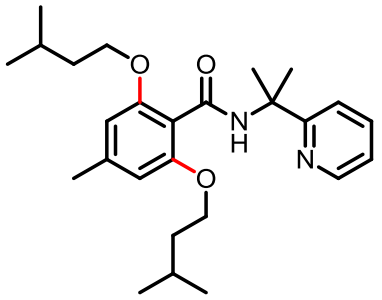
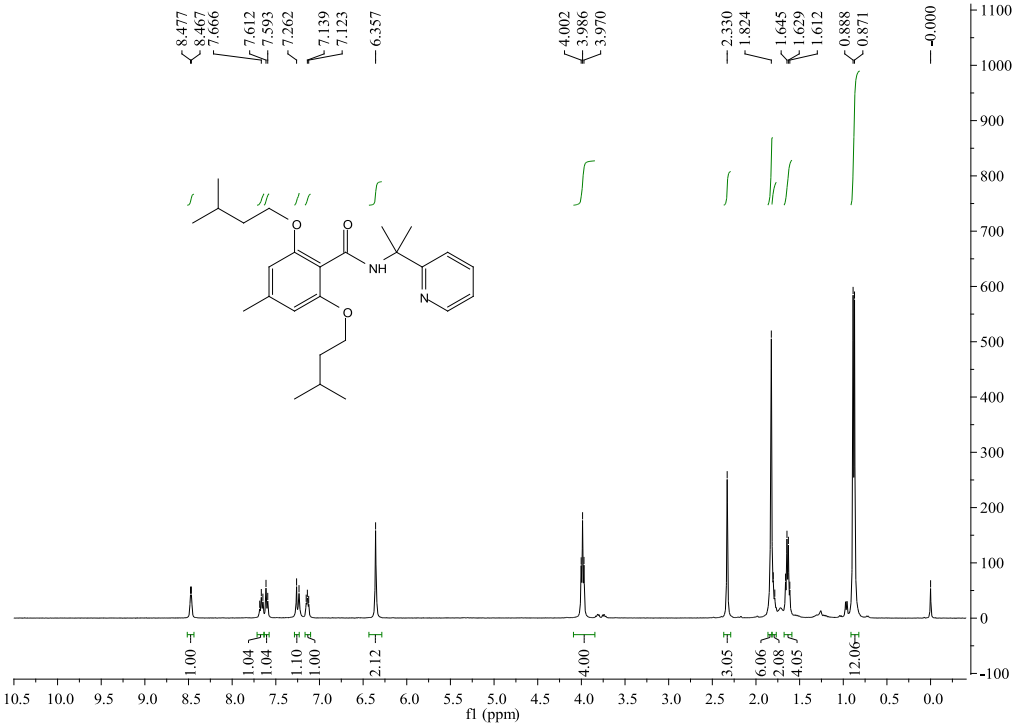
57.67
28.11
22.23
14.94
f1 (ppm)

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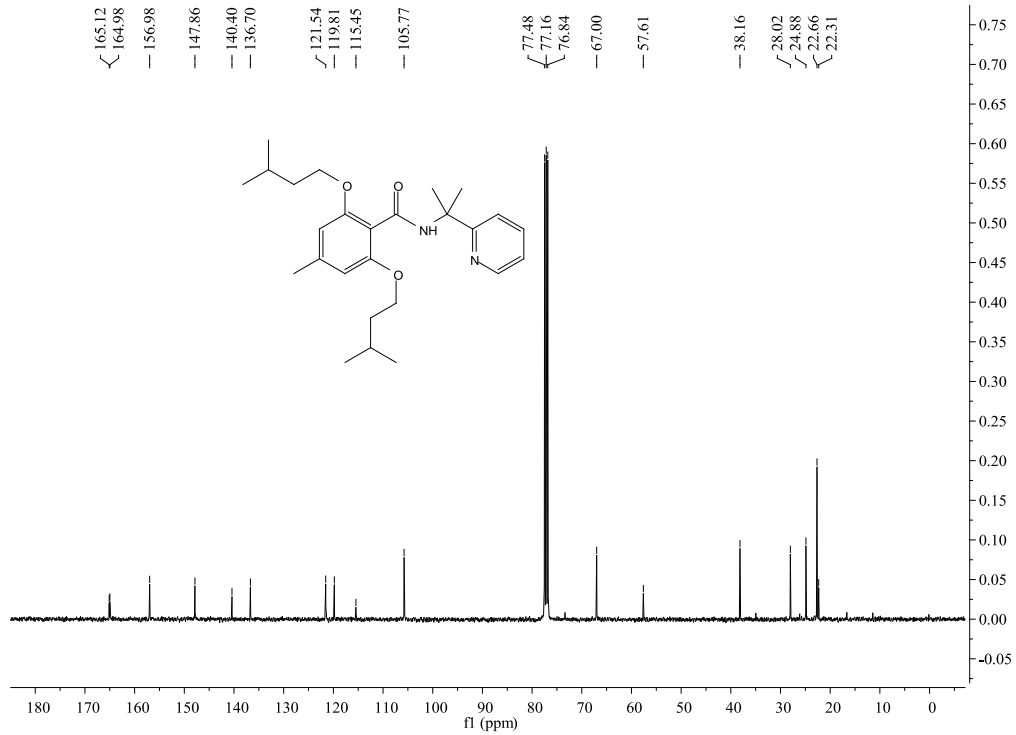
7.612
7.593
7.262
7.139
7.123
6.357
4.002
3.986
3.970
2.330
1.824
1.645
1.629
1.612
0.888
0.871
0.000
1.00
1.04
1.04
1.10
1.00
2.12
4.00
3.05
6.06
2.08
4.05
12.06
f1 (ppm)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24.88
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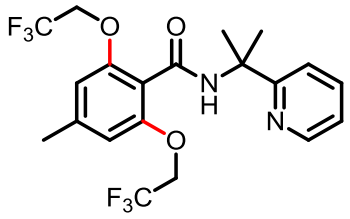
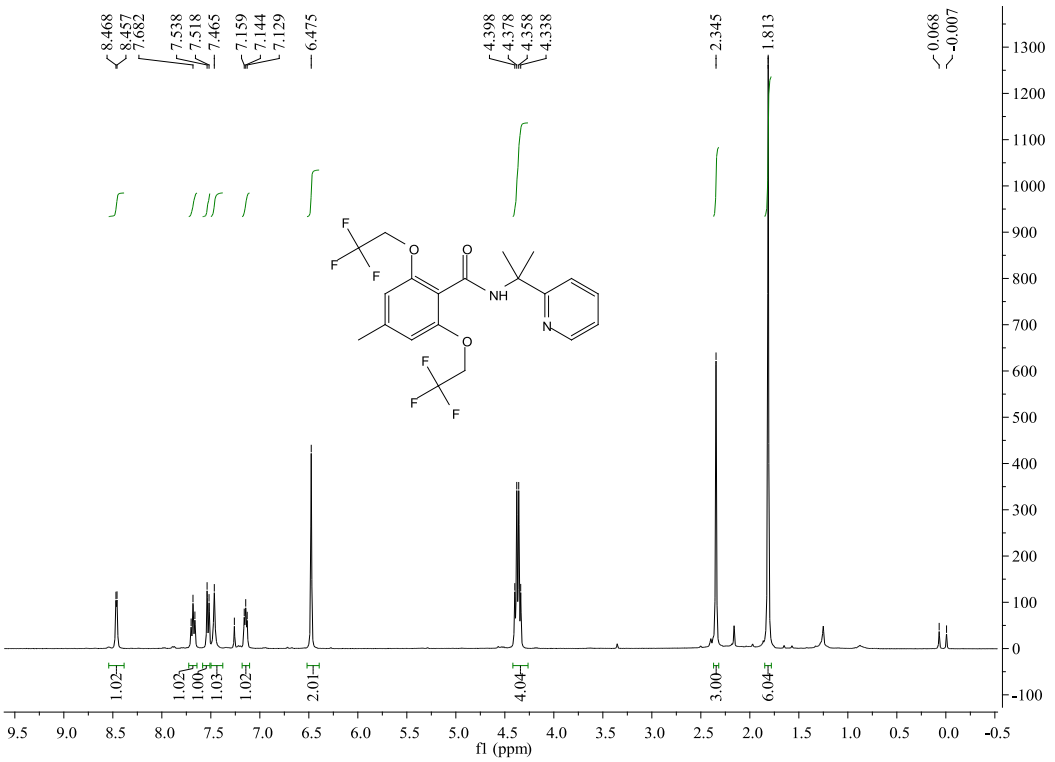
22.31
f1 (ppm)

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7.129
6.475
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4.378
4.358
4.338
2.345
1.813
0.068
-0.007
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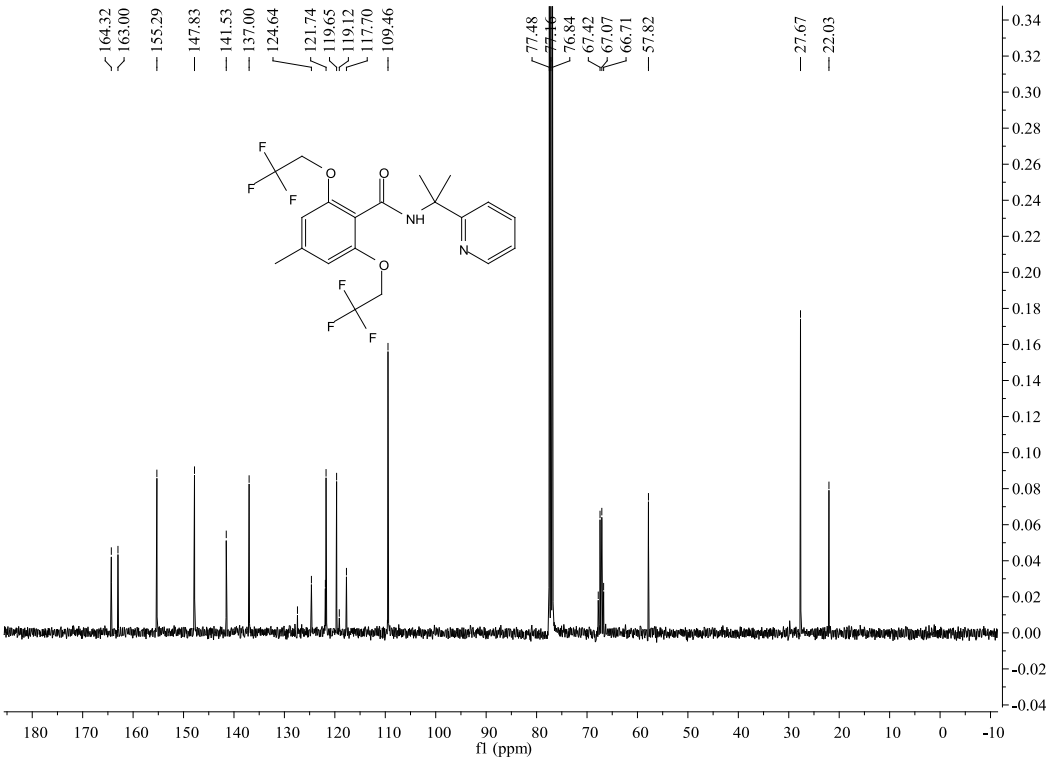
1.02
1.00
1.03
1.02
2.01
4.04
3.00
6.04
f1 (ppm)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121.74
119.65
119.12
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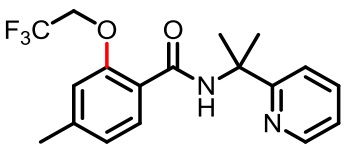
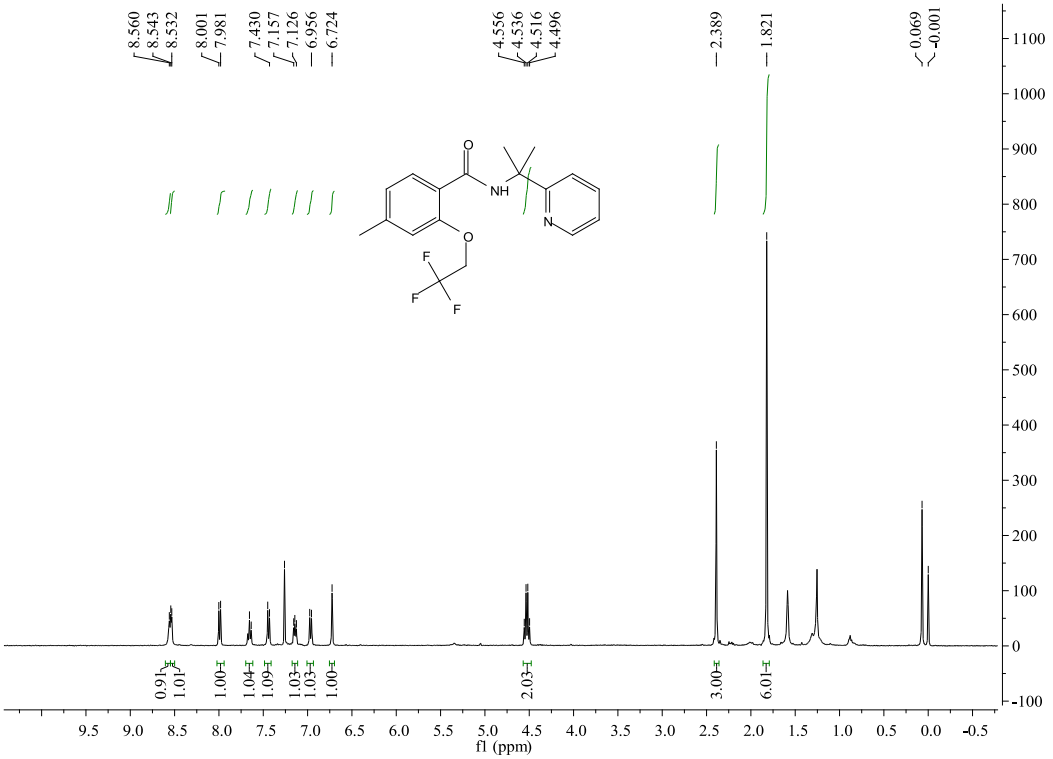
66.71
57.82
27.67
22.03
f1 (ppm)


F3C
O
O
N
H
N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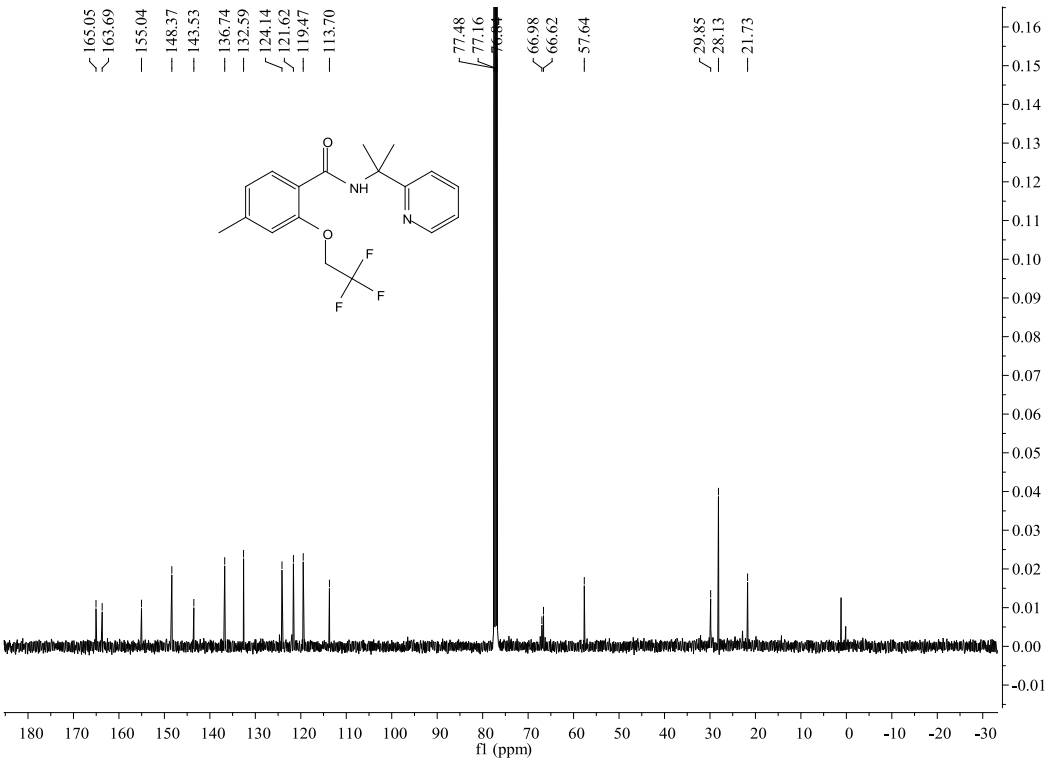
8.543
8.532
8.001
7.981
7.430
7.157
7.126
6.956
6.724
4.556
4.536
4.516
4.496
2.389
1.821
0.069
-0.001
0.91
1.01
1.00
1.04
1.09
1.03
1.03
1.00
2.03
3.00
6.01
f1 (ppm)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124.14
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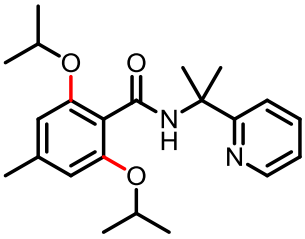
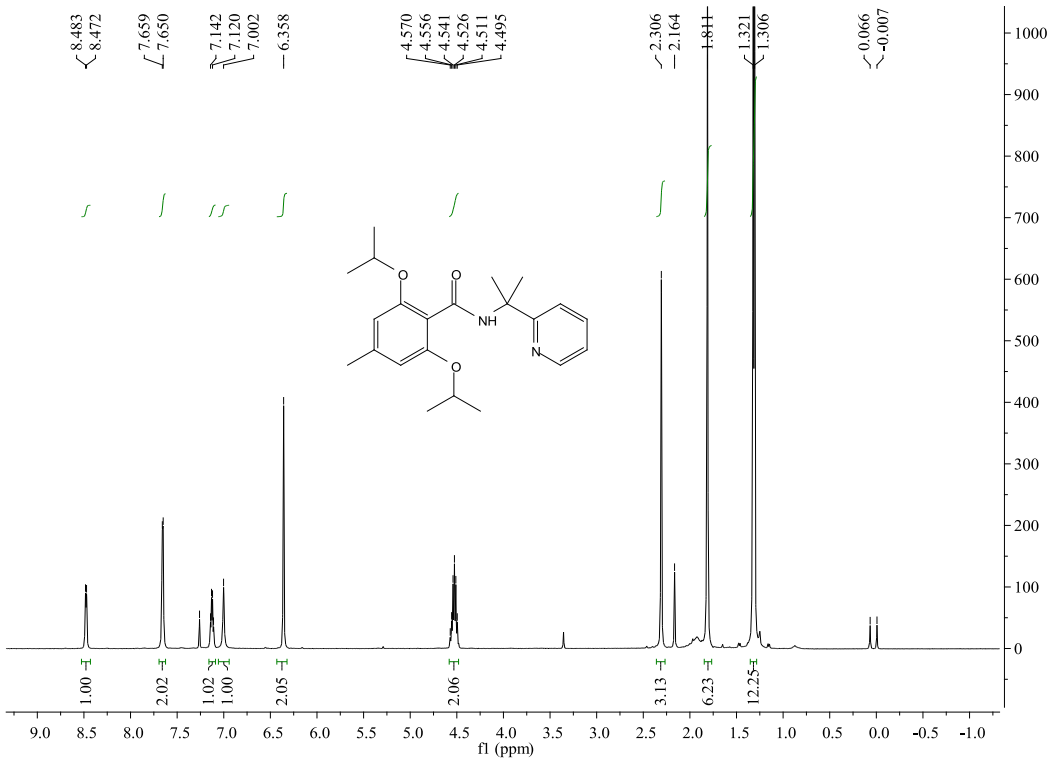
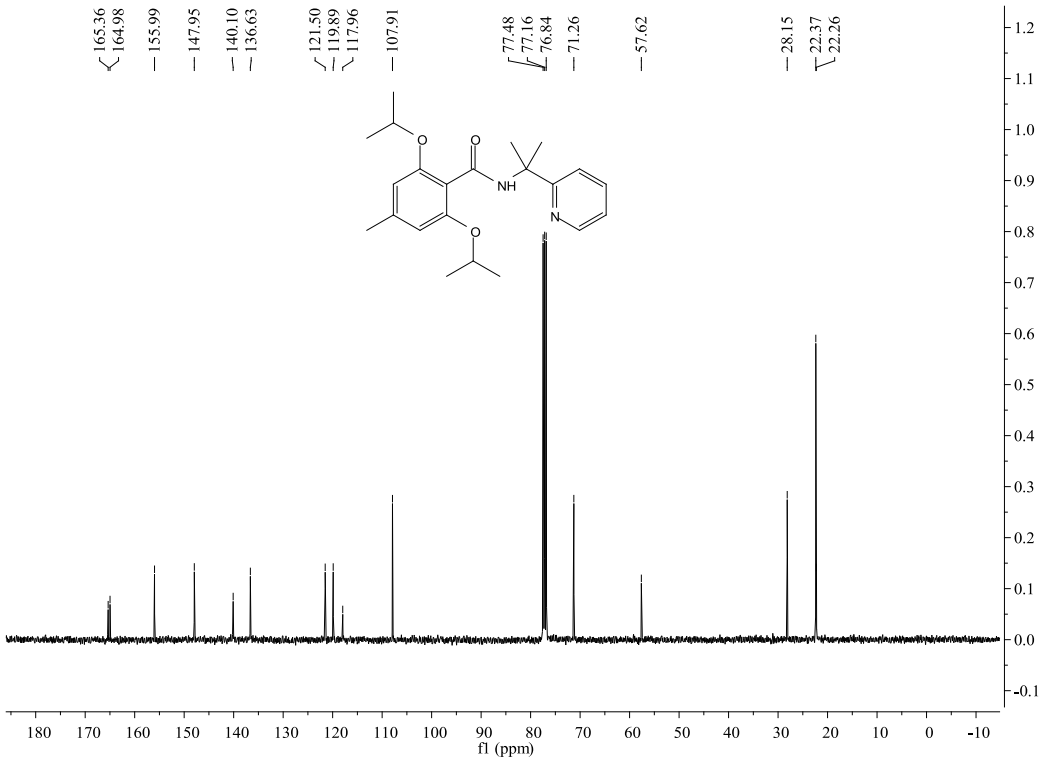
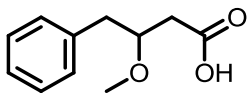
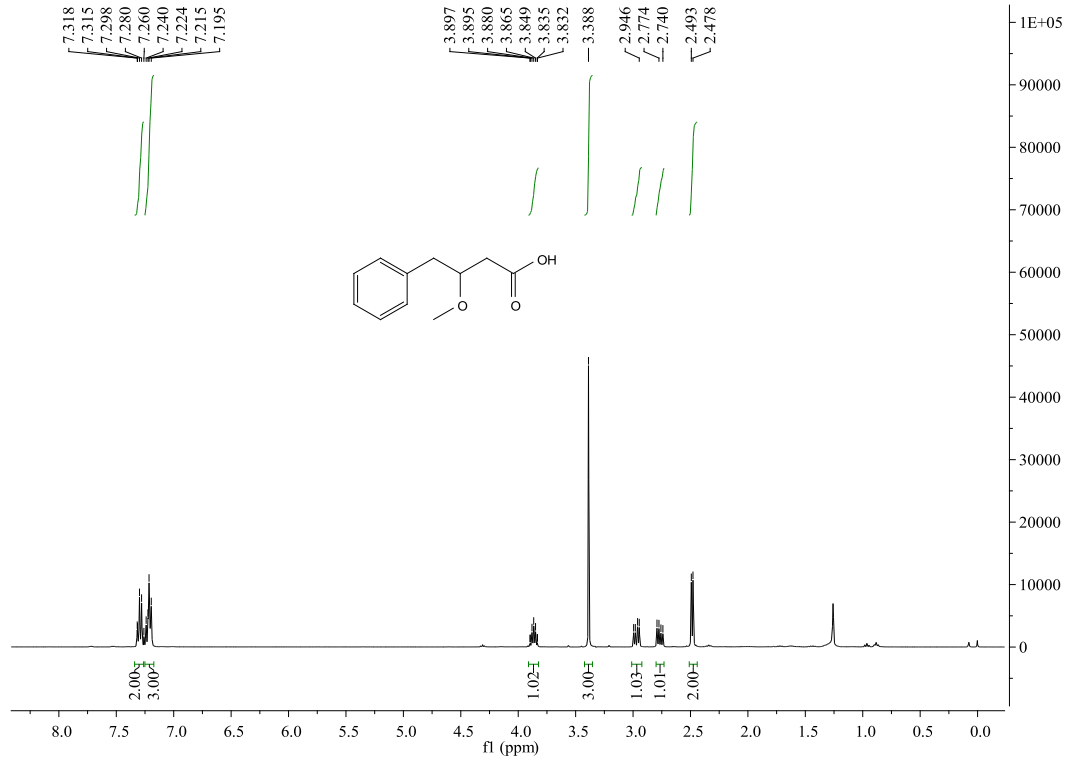
119.47
113.70
77.48
77.16
76.84
66.98
66.62
57.64
29.85
28.13
21.73
f1 (ppm)

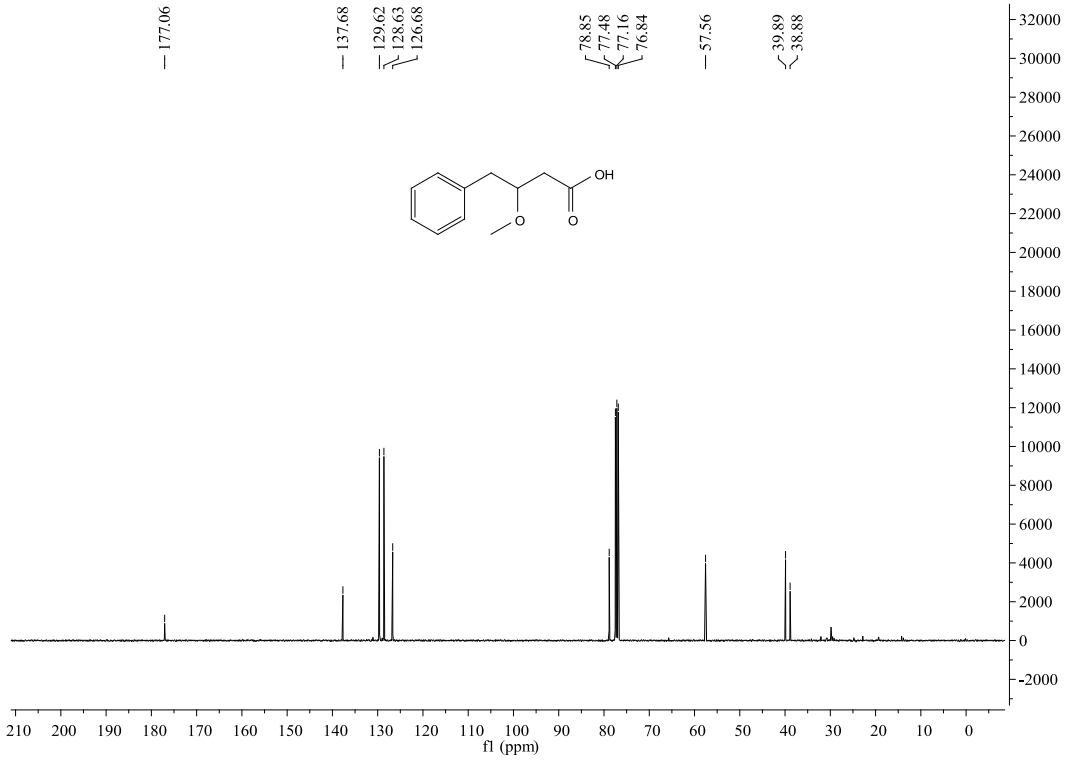



8.483
8.472
7.659
7.650
7.142
7.120
7.002
6.358
4.570
4.556
4.541
4.526
4.511
4.495
2.306
2.164
1.811
1.321
1.306
0.066
-0.007
1.00
2.02
1.02
1.00
2.05
2.06
3.13
6.23
12.25
f1 (ppm)


165.36
164.98
155.99
147.95
140.10
136.63
121.50
119.89
117.96
107.91
77.48
77.16
76.84
71.26
57.62
28.15
22.37
22.26
f1 (ppm)


O
O
OH


7.318
7.315
7.298
7.280
7.260
7.240
7.224
7.215
7.195
3.897
3.895
3.880
3.865
3.849
3.835
3.832
3.388
2.946
2.774
2.740
2.493
2.478
OH
O
O
2.00
3.00
1.02
3.00
1.03
1.01
2.00
fl (ppm)


177.06
137.68
129.62
128.63
126.68
78.85
77.48
77.16
76.84
57.56
39.89
38.88
OH
O
O
fl (ppm)