Supporting Information

A Simple Method for the Electrophilic Cyanation of Secondary Amines

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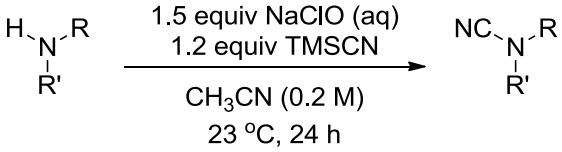
1. General Information

All reactions were performed in glassware without argon protection. Commercially available reagents were used without further purification. Organic solutions were concentrated by rotary evaporator at ca. 30 mmHg. Flash column chromatography was performed as described by Still (Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923), employing EMD silica gel 60 (230–400 mesh ASTM). TLC analyses were performed on EMD 250 μm Silica Gel 60 F_{254} plates and visualized by quenching of UV fluorescence (λ_{max}= 254 nm), or by staining ceric ammonium molybdate. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Inova-500 or Inova-400. Chemical shifts for ^{1}H and ^{13}C NMR spectra are reported in ppm (δ) relative to the ^{1}H and ^{13}C signals in the solvent ($CDCl_3$: 7.26, 77.00 ppm; C_6D_6: δ 7.16, 128.06 ppm; CD_3CN: δ 1.94 ppm) and the multiplicities are presented as follows: s = singlet, d = doublet, t = triplet, q = quartet, hept = heptet, m = multiplet. Mass spectra were acquired on an Agilent 1200 LC-MS or VG 70-VSE. Data collection on 70-VSE (purchased in part with a grant from the Division of Research Resources, National Institutes of Health RR 04648) was serviced by the Mass Spectrometry Laboratory at the University of Illinois at Urbana-Champaign.

2. General Procedures

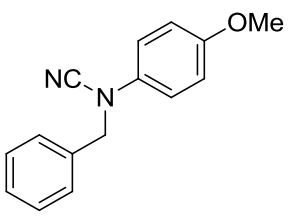


Secondary amine (0.2 mmol) was loaded in a reaction vial. Acetonitrile (1 mL) followed by TMSCN (0.24 mmol, 30 uL) were syringed into the mixture. Afterwards, bleach (0.3 mmol, ~ 200 uL) were added dropwise to the reaction which was then stirred at rt for 4 h or until the starting material had been consumed as determined by TLC. The reaction was extracted with 3 x 10 mL EtOAc. The organic layer was combined and washed with 5 mL H_2O and 5 mL brine.

After drying with anhydrous Na_2SO_4 and concentration, the residue was purified by column chromatography to give product.

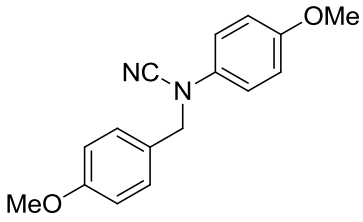
3. Compound Characterization

Compound **2**



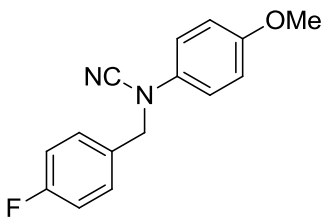
Yield 70%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.75 (s, 3H), 4.71 (s, 2H), 6.82-6.86 (m, 2H), 7.02-7.06 (m, 2H), 7.30-7.37 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 54.6, 55.5, 114.7, 114.8, 118.3, 127.5, 128.5, 129.0, 133.1, 134.5, 156.3. MS $(ESI)^+$ calcd for $C_{15}H_{15}N_2O$ $(M+H)^+$ 239.1, found 239.1.

Compound **3**



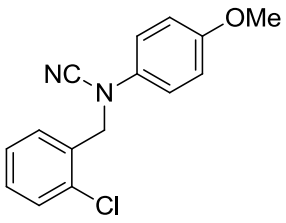
Yield 70%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.75 (s, 3H), 3.78 (s, 3H), 4.62 (s, 2H), 6.82-6.86 (m, 2H), 6.86-6.90 (m, 2H), 7.01-7.06 (m, 2H), 7.23-7.28 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 54.2, 55.3, 55.5, 114.3, 114.8, 118.5, 126.3, 129.2, 133.2, 156.3, 159.7. MS $(ESI)^+$ calcd for $C_{16}H_{16}N_2O_2Na$ $(M+Na)^+$ 291.1, found 291.1.

Compound **4**



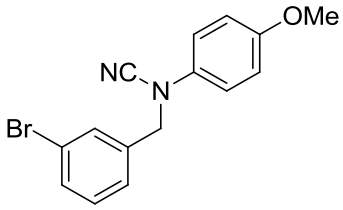
Yield 67%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.75 (s, 3H), 4.66 (s, 2H), 6.82-6.87 (m, 2H), 7.00-7.07 (m, 4H), 7.26-7.34 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 54.1, 55.5, 114.6, 114.9, 116.0 (d, J_{C-F} = 21.7 Hz), 118.5, 129.5 (d, J_{C-F} = 8.3 Hz), 130.2 (d, J_{C-F} = 3.1 Hz), 132.9, 156.4, 162.7 (d, J_{C-F} = 246.2 Hz). MS $(ESI)^+$ calcd for $C_{15}H_{14}FN_2O$ $(M+H)^+$ 257.1, found 257.2.

Compound **5**



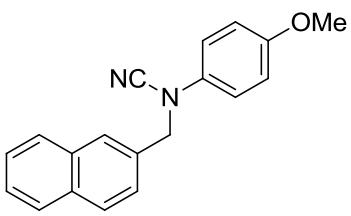
Yield 58%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.75 (s, 3H), 4.82 (s, 2H), 6.83-6.88 (m, 2H), 7.01-7.06 (m, 2H), 7.23-7.30 (m, 2H), 7.35-7.38 (m, 1H), 7.40-7.43 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 52.0, 55.5, 114.3, 114.9, 118.0, 127.3, 129.0, 129.8, 129.9, 131.8, 132.9, 133.3, 156.4. MS $(ESI)^+$ calcd for $C_{15}H_{14}ClN_2O$ $(M+H)^+$ 273.1, found 273.1.

Compound **6**



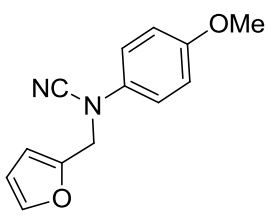
Yield 64%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.75 (s, 3H), 4.68 (s, 2H), 6.82-6.87 (m, 2H), 6.98-7.03 (m, 2H), 7.21-7.29 (m, 2H), 7.43-7.48 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 54.0, 55.6, 114.5, 114.9, 118.3, 123.1, 125.9, 130.4, 130.6, 131.7, 132.7, 136.8, 156.4. MS $(ESI)^+$ calcd for $C_{15}H_{14}BrN_2O$ $(M+H)^+$ 317.0, found 317.1.

Compound **7**



Yield 60%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.73 (s, 3H), 4.88 (s, 2H), 6.80-6.86 (m, 2H), 7.05-7.11 (m, 2H), 7.45 (dd, *J* = 1.6, 8.4 Hz, 1H), 7.47-7.51 (m, 2H), 7.78 (s, 1H), 7.78-7.83 (m, 2H), 7.85 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 54.9, 55.5, 114.8, 114.9, 118.3, 124.8, 126.4, 126.5, 126.6, 127.8, 127.9, 129.0, 131.9, 133.0, 133.1, 133.2, 156.3. MS $(ESI)^+$ calcd for $C_{19}H_{17}N_2O$ $(M+H)^+$ 289.1, found 289.2.

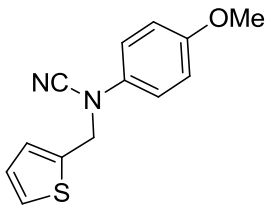
Compound **8**



Yield 69%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.76 (s, 3H), 4.64 (s, 2H), 6.34 (dd, *J* = 2.0, 3.2 Hz, 1H), 6.39 (dd, *J* = 0.8, 3.2 Hz, 1H), 6.84-6.89 (m, 2H), 7.08-7.13 (m, 2H), 7.41 (dd, *J* = 0.8, 2.0 Hz, 1H); ^{13}C NMR (100

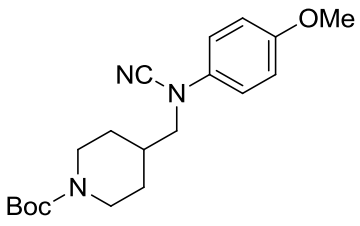
MHz, $CDCl_3$) δ 47.8, 55.5, 110.3, 110.7, 114.3, 114.8, 118.8, 132.9, 143.4, 147.8, 156.6. MS $(ESI)^+$ calcd for $C_{13}H_{13}N_2O_2$ $(M+H)^+$ 229.1, found 229.1.

Compound **9**



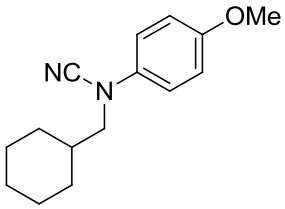
Yield 71%. ^1H NMR (400 MHz, $CDCl_3$) δ 3.76 (s, 3H), 4.84 (s, 2H), 6.84-6.88 (m, 2H), 6.97 (dd, *J* = 3.6, 5.2 Hz, 2H), 7.05-7.10 (m, 3H), 7.28 (dd, *J* = 3.6, 5.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 49.9, 55.5, 114.3, 114.9, 119.1, 126.6, 127.2, 127.8, 132.7, 136.3, 156.6. MS $(ESI)^+$ calcd for $C_{13}H_{13}N_2OS$ $(M+H)^+$ 245.1, found 245.0.

Compound **10**



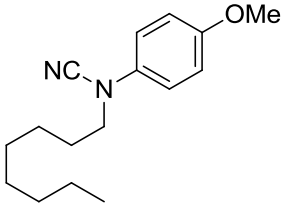
Yield 73%. ^1H NMR (400 MHz, $CDCl_3$) δ 1.12-1.22 (m, 2H), 1.42 (s, 9H), 1.77 (d, *J* = 12.0 Hz, 2H), 1.90-2.00 (m, 1H), 2.66 (dd, *J* = 12.0, 12.0 Hz, 2H), 3.38 (d, *J* = 7.2 Hz, 2H), 3.76 (s, 3H), 4.11 (s, 2H), 6.82-6.92 (m, 2H), 7.00-7.10 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.4, 29.6, 35.0, 55.6, 55.9, 79.6, 114.7, 114.9, 118.2, 133.2, 154.6, 156.3. MS $(ESI)^+$ calcd for $C_{19}H_{28}N_3O_3$ $(M+H)^+$ 346.2, found 346.3.

Compound **11**



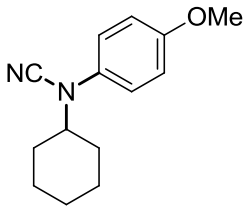
Yield 66%. ^1H NMR (400 MHz, $CDCl_3$) δ 0.92-1.04 (m, 2H), 1.08-1.28 (m, 3H), 1.62-1.86 (m, 6H), 3.33 (d, *J* = 6.8 Hz, 2H), 3.77 (s, 3H), 6.85-6.89 (m, 2H), 7.01-7.05 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.5, 26.2, 30.6, 36.3, 55.6, 56.7, 114.8, 115.0, 118.0, 133.5, 156.0. MS $(ESI)^+$ calcd for $C_{15}H_{21}N_2O$ $(M+H)^+$ 245.1, found 245.2.

Compound **12**



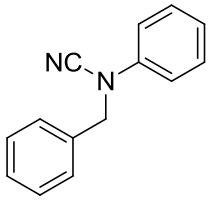
Yield 56%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.86 (t, *J* = 6.8 Hz, 3H), 1.20-1.35 (m, 8H), 1.35-1.45 (m, 2H), 1.72-1.80 (m, 2H), 3.48 (t, *J* = 7.2 Hz, 2H), 3.77 (s, 3H), 6.85-6.90 (m, 2H), 7.01-7.05 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 22.6, 26.5, 27.5, 29.0, 29.1, 31.7, 50.3, 55.6, 114.5, 114.8, 118.0, 133.4, 156.1. MS $(ESI)^+$ calcd for $C_{16}H_{25}N_2O$ $(M+H)^+$ 261.2, found 261.2.

Compound **13**



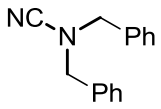
Yield 68%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.12-1.25 (m, 1H), 1.25-1.37 (m, 2H), 1.48-1.60 (m, 2H), 1.62-1.70 (m, 1H), 1.82-1.90 (m, 2H), 2.00-2.08 (m, 2H), 3.38 (tt, *J* = 4.0, 11.6 Hz, 1H), 3.77 (s, 3H), 6.84-6.89 (m, 2H), 7.05-7.10 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.1, 25.2, 31.0, 55.5, 59.1, 113.7, 114.8, 120.2, 133.2, 156.6. MS $(ESI)^+$ calcd for $C_{14}H_{19}N_2O$ $(M+H)^+$ 231.1, found 231.2.

Compound **14**



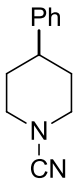
Yield 63%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.80 (s, 2H), 7.07-7.15 (m, 3H), 7.32-7.41 (m, 7H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 53.6, 113.8, 115.9, 123.6, 127.2, 128.4, 129.0, 129.5, 134.2, 139.7; MS $(ESI)^+$ calcd for $C_{14}H_{12}N_2Na$ $(M+Na)^+$ 231.1, found 231.1.

Compound **15**



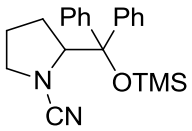
Yield 72%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.10 (s, 3H), 7.27-7.31 (m, 4H), 7.33-7.41 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 54.2, 118.3, 128.6, 128.7, 128.9, 134.3. MS $(ESI)^+$ calcd for $C_{15}H_{15}N_2$ $(M+H)^+$ 223.1, found 223.2.

Compound **16**



Yield 88%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.80-1.88 (m, 4H), 2.54-2.63 (m, 1H), 3.09-3.17 (m, 2H), 3.49-3.55 (m, 2H), 7.16-7.20 (m, 2H), 7.20-7.25 (m, 1H), 7.29-7.34 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 32.0, 41.3, 50.1, 118.3, 126.6, 126.8, 128.7, 144.5. MS $(ESI)^+$ calcd for $C_{12}H_{15}N_2$ $(M+H)^+$ 187.1, found 187.1.

Compound **17**



Yield 82%. ^{1}H NMR (400 MHz, $CDCl_3$) δ -0.15 (s, 9H), 0.63-0.73 (m, 1H), 1.45-1.55 (m, 1H), 1.82-1.92 (m, 1H), 2.03-2.13 (m, 1H), 2.75 (ddd, *J* = 5.2, 8.0, 9.2 Hz, 1H), 3.18 (ddd, *J* = 7.6, 7.6, 9.2 Hz, 1H), 4.75 (dd, *J* = 4.0, 9.2 Hz, 1H), 7.30-7.38 (m, 6H), 7.40-7.48 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 1.65, 23.8, 27.9, 52.7, 68.3, 82.9, 118.3, 127.4, 127.7, 127.8, 127.9, 129.2, 129.4, 141.9, 142.0. MS $(ESI)^+$ calcd for $C_{21}H_{27}N_2OSi$ $(M+H)^+$ 351.2, found 351.2.

X-ray Experimental for $C_{15}H_{13}N_2OCl$: Crystals grew as colorless prisms by slow evaporation from a mixture of ethyl acetate and hexanes. The data crystal was cut from a larger crystal and had approximate dimensions; 0.24 x 0.17 x 0.14 mm. The data were collected at room temperature on a Rigaku R-Axis Spider diffractometer with an image plate detector using a graphite monochromator with CuKα radiation (λ = 1.5418Å). A total of 191 images of data were collected using ω-scans with a scan range of 5° and a counting time of 360 seconds per image. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using the Rigaku Americas Corporation's Crystal Clear version 1.40.[S1] The structure was solved by direct methods using SIR97[S2] and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.[S3] The absolute configuration was determined by the method of Flack.[S4] The Flack x parameter refined to 0.04(3). The assignment of absolute configuration was corroborated by the Hooft analysis.[S5] The Hooft y-parameter was determined to by 0.020(8). The hydrogen atoms were calculated in ideal positions with isotropic displacement parameters set to 1.2xUeq of the attached atom (1.5xUeq for methyl hydrogen atoms).

The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.0437*P)^2 + (0.9407*P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.166, with R(F) equal to 0.0926 and a goodness of fit, S, = 1.1257. Definitions used for calculating R(F), $R_w(F^2)$ and the goodness of fit, S, are given below.[S6] The data were checked for secondary extinction effects but no correction was necessary. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[S7] All figures were generated using SHELXTL/PC.[S8] Tables of positional and thermal parameters, bond lengths and angles, torsion angles and figures are given below.

[S1] DENZO-SMN. (1997). Z. Otwinowski and W. Minor, Methods in Enzymology, **276**: Macromolecular Crystallography, part A, 307 – 326, C. W. Carter, Jr. and R. M. Sweets, Editors, Academic Press.

[S2] SIR97. (1999). A program for crystal structure solution. Altomare A., Burla M.C., Camalli M., Cascarano G.L., Giacovazzo C. , Guagliardi A., Moliterni A.G.G., Polidori G.,Spagna R. J. Appl. Cryst. 32, 115-119.

[S3] Sheldrick, G. M. (1994). SHELXL97. Program for the Refinement of Crystal Structures. University of Gottingen, Germany.

[S4] Flack, H. D. (1983). Acta Cryst. A39, 876-881.

[S5] Hooft, R. W. W., Straver, L. H. and Spek, A. L. (2008). J. Appl. Cryst. 41, 96-103.

[S6] $R_W(F^2) = \{\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w(|F_o|)^4\}^{1/2}$ where w is the weight given each reflection.

$R(F) = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|\}$ for reflections with $F_o > 4(\sigma(F_o))$.

$S = [\Sigma w(|F_o|^2 - |F_c|^2)^2/(n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.

[S7] International Tables for X-ray Crystallography (1992). Vol. C, Tables 4.2.6.8 and 6.1.1.4, A. J. C. Wilson, editor, Boston: Kluwer Academic Press.

[S8] Sheldrick, G. M. (1994). SHELXTL/PC (Version 5.03). Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

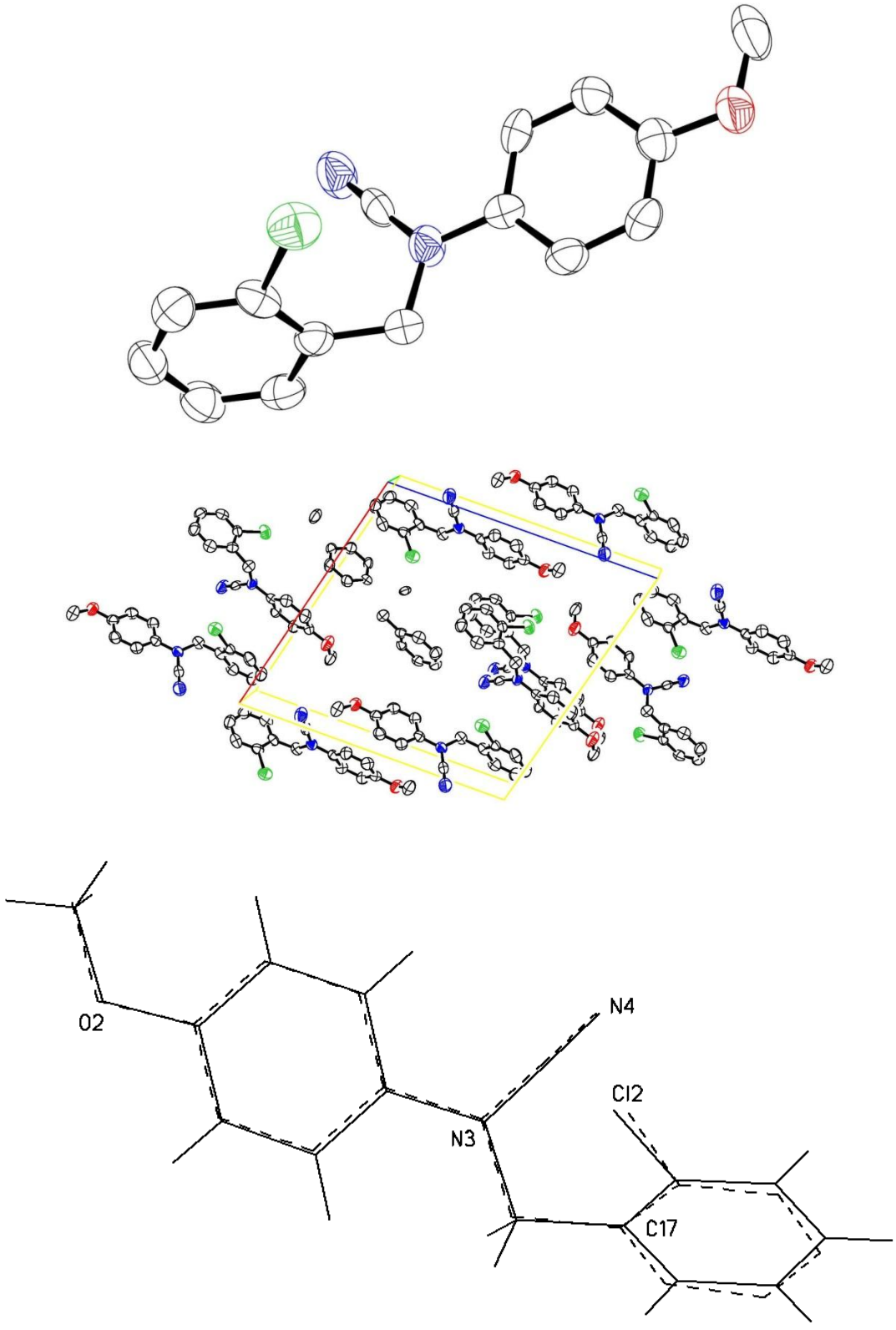


Figure S1. X-ray structures of **5**.

Table S1. Crystal data and structure refinement for **5**.

Empirical formula	C15 H13 Cl N2 O	
Formula weight	272.72	
Temperature	298(2) K	
Wavelength	1.54180 Å	
Crystal system	Monoclinic	
Space group	P21	
Unit cell dimensions	a = 15.5380(13) Å	α= 90°.
	b = 5.5510(6) Å	β= 104.854(5)°.
	c = 16.2790(15) Å	γ = 90°.
Volume	1357.2(2) $Å^3$	
Z	4	
Density (calculated)	1.335 Mg/m^3	
Absorption coefficient	2.431 mm^{-1}	
F(000)	568	
Crystal size	0.24 x 0.17 x 0.14 mm	
Theta range for data collection	6.99 to 66.58°.	
Index ranges	-17<=h<=18, -6<=k<=6, -19<=l<=19	
Reflections collected	15994	
Independent reflections	4704 [R(int) = 0.0470]	
Completeness to theta = 66.58°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00 and 0.794	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4704 / 1 / 346	
Goodness-of-fit on F^2	1.249	
Final R indices [I>2sigma(I)]	R1 = 0.0926, wR2 = 0.1590	
R indices (all data)	R1 = 0.1079, wR2 = 0.1664	
Absolute structure parameter	0.04(3)	
Largest diff. peak and hole	0.283 and -0.379 $e.Å^{-3}$	

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **5**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl1	7479(1)	8375(3)	7812(1)	62(1)
O1	8573(3)	11217(9)	3530(3)	61(1)
N1	9009(3)	11118(9)	7038(3)	46(1)
N2	10135(4)	8463(13)	7930(3)	81(2)
C1	8561(4)	12191(10)	8347(4)	43(1)
C2	8148(3)	10261(11)	8591(4)	46(2)
C3	8210(4)	9705(12)	9430(4)	58(2)
C4	8715(4)	11179(15)	10051(4)	65(2)
C5	9134(4)	13119(15)	9837(4)	65(2)
C6	9071(4)	13606(12)	8994(4)	56(2)
C7	8500(4)	12811(11)	7432(3)	48(2)
C8	9617(4)	9718(13)	7521(4)	54(2)
C10	8890(3)	11093(11)	6136(3)	40(1)
C11	9272(4)	9321(12)	5762(3)	51(2)
C12	9180(4)	9286(13)	4895(4)	53(2)
C13	8694(4)	11044(13)	4397(4)	50(2)
C14	8311(4)	12805(11)	4762(4)	52(2)
C15	8409(4)	12862(11)	5633(3)	48(2)
C16	8851(4)	9266(16)	3112(4)	79(2)
Cl2	6382(1)	9201(3)	2848(1)	62(1)
O2	3226(3)	7448(9)	-1412(2)	66(1)
N3	4473(3)	6601(9)	2099(3)	45(1)
N4	3804(3)	9143(12)	3017(3)	66(2)
C17	5590(3)	5397(10)	3394(3)	42(1)
C18	6144(4)	7313(11)	3635(3)	43(1)
C19	6537(3)	7880(12)	4474(4)	50(2)
C20	6359(4)	6431(12)	5097(4)	54(2)
C21	5815(4)	4489(14)	4882(4)	57(2)
C22	5435(4)	3943(12)	4046(4)	52(2)
C23	5156(4)	4813(10)	2489(3)	46(2)
C24	4113(4)	7959(12)	2597(3)	43(1)

C26	4156(3)	6840(10)	1197(3)	37(1)
C27	4372(4)	5158(10)	659(4)	47(2)
C28	4045(4)	5465(11)	-214(4)	52(2)
C29	3515(4)	7357(12)	-542(4)	46(2)
C30	3309(4)	9061(12)	-3(3)	51(2)
C31	3633(3)	8797(11)	864(3)	47(2)
C32	2604(4)	9238(16)	-1775(4)	73(2)

Table S3. Bond lengths [Å] and angles [°] for **5**.

Cl1-C2	1.764(6)	O2-C29	1.373(6)
O1-C13	1.378(6)	O2-C32	1.407(8)
O1-C16	1.405(8)	N3-C24	1.330(7)
N1-C8	1.316(7)	N3-C26	1.430(6)
N1-C10	1.433(6)	N3-C23	1.472(6)
N1-C7	1.476(7)	N4-C24	1.140(7)
N2-C8	1.141(7)	C17-C18	1.361(7)
C1-C2	1.359(7)	C17-C22	1.403(7)
C1-C6	1.389(7)	C17-C23	1.491(7)
C1-C7	1.508(7)	C18-C19	1.382(7)
C2-C3	1.379(7)	C19-C20	1.376(8)
C3-C4	1.379(9)	C19-H19	0.93
C3-H3	0.93	C20-C21	1.359(9)
C4-C5	1.350(9)	C20-H20	0.93
C4-H4	0.93	C21-C22	1.371(7)
C5-C6	1.376(8)	C21-H21	0.93
C5-H5	0.93	C22-H22	0.93
C6-H6	0.93	C23-H23A	0.97
C7-H7A	0.97	C23-H23B	0.97
C7-H7B	0.97	C26-C27	1.380(7)
C10-C11	1.369(8)	C26-C31	1.381(7)
C10-C15	1.370(7)	C27-C28	1.391(7)
C11-C12	1.382(7)	C27-H27	0.93
C11-H11	0.93	C28-C29	1.357(8)
C12-C13	1.366(8)	C28-H28	0.93
C12-H12	0.93	C29-C30	1.383(8)
C13-C14	1.359(8)	C30-C31	1.379(7)
C14-C15	1.387(7)	C30-H30	0.93
C14-H14	0.93	C31-H31	0.93
C15-H15	0.93	C32-H32A	0.96
C16-H16A	0.96	C32-H32B	0.96
C16-H16B	0.96	C32-H32C	0.96
C16-H16C	0.96	C13-O1-C16	117.7(5)
Cl2-C18	1.766(6)	C8-N1-C10	119.4(5)

C8-N1-C7	120.0(5)	C14-C13-C12	119.4(6)
C10-N1-C7	120.5(5)	C14-C13-O1	116.0(6)
C2-C1-C6	116.4(5)	C12-C13-O1	124.6(6)
C2-C1-C7	123.7(5)	C13-C14-C15	121.2(6)
C6-C1-C7	120.0(5)	C13-C14-H14	119.4
C1-C2-C3	123.2(6)	C15-C14-H14	119.4
C1-C2-Cl1	119.5(5)	C10-C15-C14	119.6(6)
C3-C2-Cl1	117.3(5)	C10-C15-H15	120.2
C4-C3-C2	118.4(6)	C14-C15-H15	120.2
C4-C3-H3	120.8	O1-C16-H16A	109.5
C2-C3-H3	120.8	O1-C16-H16B	109.5
C5-C4-C3	120.3(6)	H16A-C16-H16B	109.5
C5-C4-H4	119.9	O1-C16-H16C	109.5
C3-C4-H4	119.9	H16A-C16-H16C	109.5
C4-C5-C6	119.9(7)	H16B-C16-H16C	109.5
C4-C5-H5	120.0	C29-O2-C32	117.8(5)
C6-C5-H5	120.0	C24-N3-C26	119.0(4)
C5-C6-C1	121.7(6)	C24-N3-C23	119.3(4)
C5-C6-H6	119.1	C26-N3-C23	121.7(4)
C1-C6-H6	119.1	C18-C17-C22	116.7(5)
N1-C7-C1	112.2(5)	C18-C17-C23	123.2(5)
N1-C7-H7A	109.2	C22-C17-C23	120.1(5)
C1-C7-H7A	109.2	C17-C18-C19	123.3(5)
N1-C7-H7B	109.2	C17-C18-Cl2	119.3(4)
C1-C7-H7B	109.2	C19-C18-Cl2	117.4(5)
H7A-C7-H7B	107.9	C20-C19-C18	118.3(6)
N2-C8-N1	178.5(8)	C20-C19-H19	120.8
C11-C10-C15	118.8(5)	C18-C19-H19	120.8
C11-C10-N1	120.3(5)	C21-C20-C19	120.2(6)
C15-C10-N1	120.8(5)	C21-C20-H20	119.9
C10-C11-C12	121.4(6)	C19-C20-H20	119.9
C10-C11-H11	119.3	C20-C21-C22	120.8(6)
C12-C11-H11	119.3	C20-C21-H21	119.6
C13-C12-C11	119.6(6)	C22-C21-H21	119.6
C13-C12-H12	120.2	C21-C22-C17	120.7(6)
C11-C12-H12	120.2	C21-C22-H22	119.7

C17-C22-H22	119.7	C27-C28-H28	119.2
N3-C23-C17	111.9(4)	C28-C29-O2	116.1(5)
N3-C23-H23A	109.2	C28-C29-C30	119.7(5)
C17-C23-H23A	109.2	O2-C29-C30	124.2(6)
N3-C23-H23B	109.2	C31-C30-C29	119.6(6)
C17-C23-H23B	109.2	C31-C30-H30	120.2
H23A-C23-H23B	107.9	C29-C30-H30	120.2
N4-C24-N3	179.2(6)	C30-C31-C26	120.6(5)
C27-C26-C31	119.8(5)	C30-C31-H31	119.7
C27-C26-N3	120.9(5)	C26-C31-H31	119.7
C31-C26-N3	119.3(5)	O2-C32-H32A	109.5
C26-C27-C28	118.7(5)	O2-C32-H32B	109.5
C26-C27-H27	120.6	H32A-C32-H32B	109.5
C28-C27-H27	120.6	O2-C32-H32C	109.5
C29-C28-C27	121.5(5)	H32A-C32-H32C	109.5
C29-C28-H28	119.2	H32B-C32-H32C	109.5

Table S4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **5**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl1	60(1)	52(1)	71(1)	-9(1)	13(1)	-9(1)
O1	67(3)	69(3)	45(2)	0(2)	11(2)	9(3)
N1	42(3)	51(3)	49(3)	3(3)	16(2)	7(2)
N2	82(4)	106(6)	60(4)	21(4)	26(3)	41(4)
C1	43(3)	34(3)	57(4)	2(3)	21(3)	-2(3)
C2	37(3)	46(4)	56(4)	-14(3)	15(3)	0(3)
C3	61(4)	51(4)	74(4)	7(4)	37(4)	0(4)
C4	73(5)	80(6)	45(4)	2(4)	21(4)	9(4)
C5	63(4)	77(6)	51(4)	-16(4)	10(3)	9(4)
C6	53(4)	46(4)	70(4)	-11(4)	20(3)	-3(3)
C7	51(3)	38(4)	58(4)	6(3)	20(3)	2(3)
C8	50(4)	75(5)	43(4)	0(3)	21(3)	11(4)
C10	38(3)	38(3)	47(3)	5(3)	14(3)	-1(3)
C11	55(4)	46(4)	50(3)	11(3)	13(3)	13(3)
C12	53(4)	49(4)	59(4)	-8(4)	16(3)	9(4)
C13	38(3)	62(4)	47(4)	4(3)	7(3)	-4(3)
C14	53(4)	46(4)	54(4)	12(3)	9(3)	15(3)
C15	54(3)	39(4)	52(4)	-3(3)	16(3)	3(3)
C16	87(5)	100(6)	48(4)	-6(5)	15(3)	1(5)
Cl2	67(1)	56(1)	68(1)	10(1)	29(1)	-8(1)
O2	86(3)	71(3)	38(2)	-3(2)	11(2)	22(3)
N3	49(3)	53(3)	35(3)	-2(2)	14(2)	6(3)
N4	68(3)	83(4)	44(3)	-4(3)	11(3)	24(4)
C17	37(3)	36(3)	49(3)	-1(3)	7(3)	6(3)
C18	41(3)	51(4)	43(3)	13(3)	18(3)	10(3)
C19	35(3)	53(4)	63(4)	-7(3)	11(3)	-4(3)
C20	51(4)	63(5)	43(3)	1(3)	4(3)	7(4)
C21	66(4)	60(4)	47(4)	12(4)	15(3)	7(4)
C22	55(4)	35(4)	64(4)	3(3)	15(3)	0(3)
C23	54(4)	36(4)	45(3)	0(3)	8(3)	5(3)
C24	36(3)	53(4)	33(3)	7(3)	-4(2)	0(3)

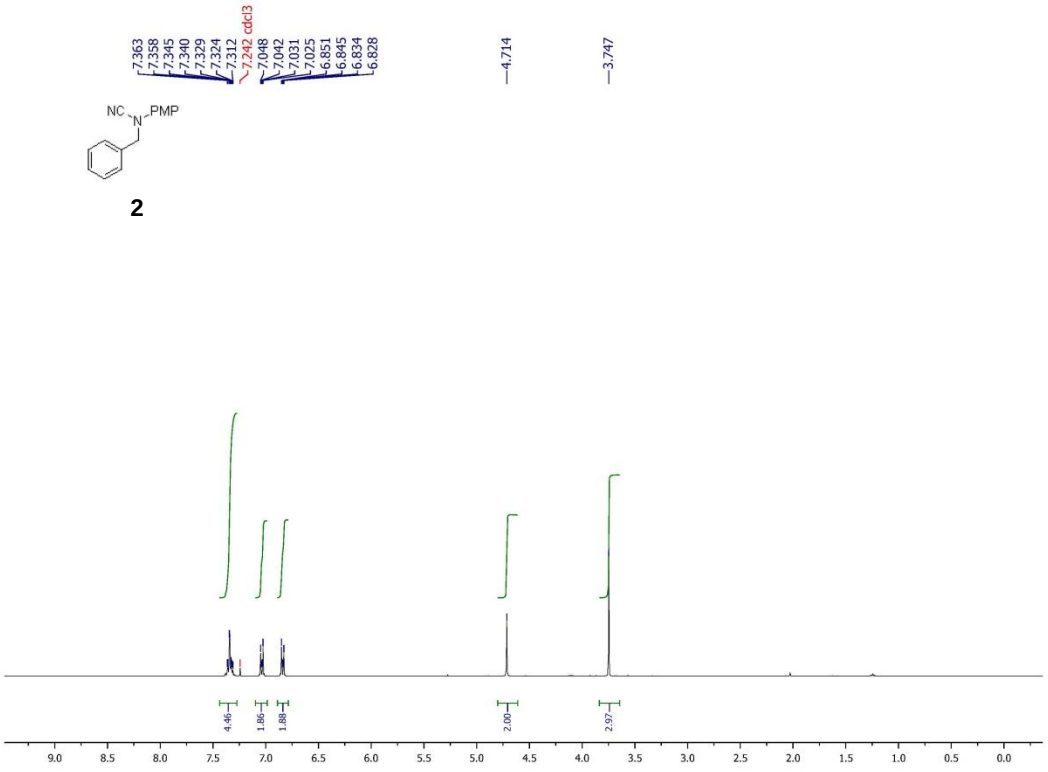
C26	31(3)	34(3)	49(3)	0(3)	13(2)	0(2)
C27	50(4)	40(4)	53(4)	-1(3)	16(3)	10(3)
C28	68(4)	45(4)	45(4)	-13(3)	20(3)	6(3)
C29	46(3)	54(4)	42(3)	3(3)	16(3)	-6(3)
C30	56(4)	48(4)	47(3)	3(3)	13(3)	10(3)
C31	54(3)	51(4)	37(3)	-17(3)	17(3)	-4(3)
C32	87(5)	93(6)	35(3)	6(4)	10(3)	-8(5)

Table S5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **5**.

	x	y	z	U(eq)
H3	7919	8367	9573	70
H4	8766	10836	10621	78
H5	9465	14124	10257	78
H6	9379	14917	8855	67
H7A	7880	12787	7112	58
H7B	8725	14430	7402	58
H11	9600	8115	6099	61
H12	9446	8073	4652	64
H14	7977	13995	4422	62
H15	8150	14094	5875	58
H16A	9486	9104	3302	118
H16B	8683	9545	2510	118
H16C	8573	7814	3237	118
H19	6911	9206	4615	61
H20	6611	6783	5666	65
H21	5700	3518	5307	69
H22	5071	2595	3911	62
H23A	5605	4757	2171	55
H23B	4883	3233	2457	55
H27	4730	3843	875	56
H28	4192	4346	-581	62
H30	2954	10377	-223	61
H31	3498	9947	1227	56
H32A	2097	9125	-1542	109
H32B	2417	9013	-2379	109
H32C	2873	10798	-1651	109

Table S6. Torsion angles [°] for **5**.

C6-C1-C2-C3	0.8(8)	C22-C17-C18-C19	-1.0(8)
C7-C1-C2-C3	179.8(5)	C23-C17-C18-C19	179.0(5)
C6-C1-C2-Cl1	179.5(4)	C22-C17-C18-Cl2	179.2(4)
C7-C1-C2-Cl1	-1.5(7)	C23-C17-C18-Cl2	-0.8(7)
C1-C2-C3-C4	0.1(9)	C17-C18-C19-C20	0.1(8)
Cl1-C2-C3-C4	-178.7(5)	Cl2-C18-C19-C20	179.9(4)
C2-C3-C4-C5	0.1(9)	C18-C19-C20-C21	0.5(9)
C3-C4-C5-C6	-1.1(10)	C19-C20-C21-C22	-0.1(9)
C4-C5-C6-C1	2.1(9)	C20-C21-C22-C17	-0.8(9)
C2-C1-C6-C5	-1.9(8)	C18-C17-C22-C21	1.4(8)
C7-C1-C6-C5	179.1(5)	C23-C17-C22-C21	-178.6(5)
C8-N1-C7-C1	-17.0(7)	C24-N3-C23-C17	-21.5(7)
C10-N1-C7-C1	167.1(5)	C26-N3-C23-C17	161.4(5)
C2-C1-C7-N1	-71.9(7)	C18-C17-C23-N3	-69.4(7)
C6-C1-C7-N1	107.0(6)	C22-C17-C23-N3	110.6(6)
C8-N1-C10-C11	12.7(8)	C24-N3-C26-C27	-166.6(5)
C7-N1-C10-C11	-171.4(5)	C23-N3-C26-C27	10.4(8)
C8-N1-C10-C15	-165.9(6)	C24-N3-C26-C31	14.0(7)
C7-N1-C10-C15	10.0(8)	C23-N3-C26-C31	-169.0(5)
C15-C10-C11-C12	-0.1(9)	C31-C26-C27-C28	-0.8(8)
N1-C10-C11-C12	-178.8(5)	N3-C26-C27-C28	179.8(5)
C10-C11-C12-C13	-0.4(9)	C26-C27-C28-C29	-0.6(9)
C11-C12-C13-C14	0.2(9)	C27-C28-C29-O2	-179.3(5)
C11-C12-C13-O1	178.6(5)	C27-C28-C29-C30	1.5(9)
C16-O1-C13-C14	-171.4(5)	C32-O2-C29-C28	173.6(5)
C16-O1-C13-C12	10.2(9)	C32-O2-C29-C30	-7.3(8)
C12-C13-C14-C15	0.5(9)	C28-C29-C30-C31	-1.0(8)
O1-C13-C14-C15	-178.1(5)	O2-C29-C30-C31	179.9(5)
C11-C10-C15-C14	0.8(8)	C29-C30-C31-C26	-0.4(8)
N1-C10-C15-C14	179.4(5)	C27-C26-C31-C30	1.3(8)
C13-C14-C15-C10	-1.0(9)	N3-C26-C31-C30	-179.2(5)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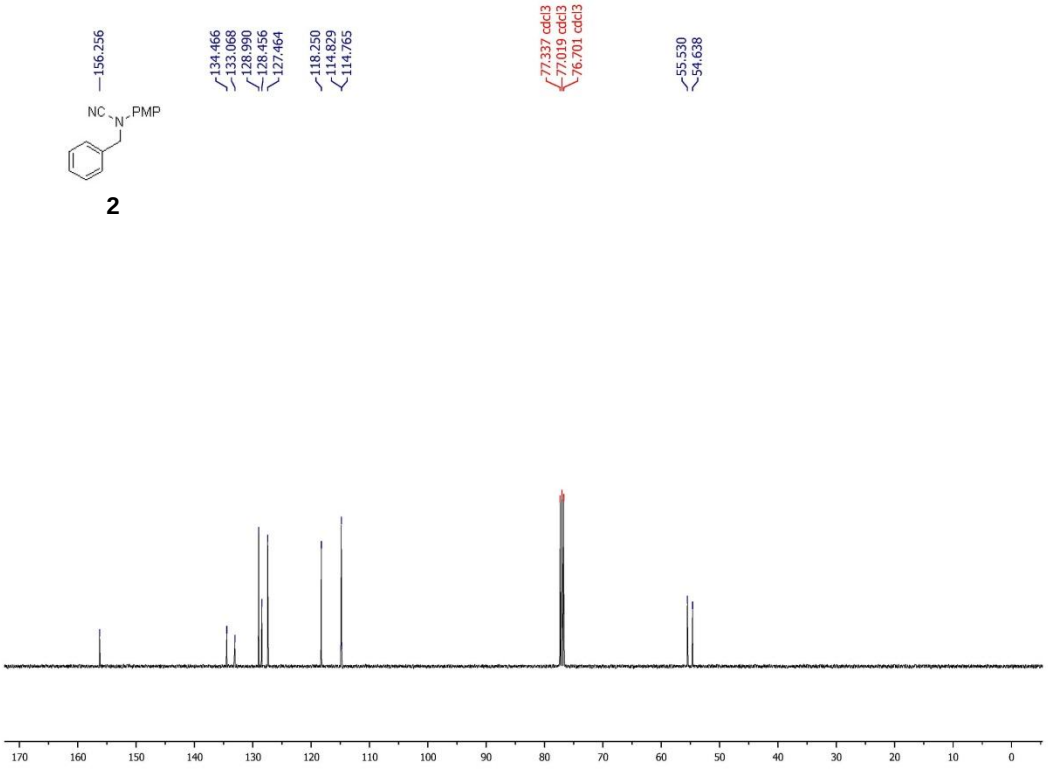
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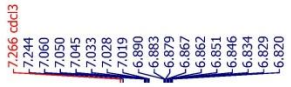
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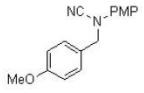
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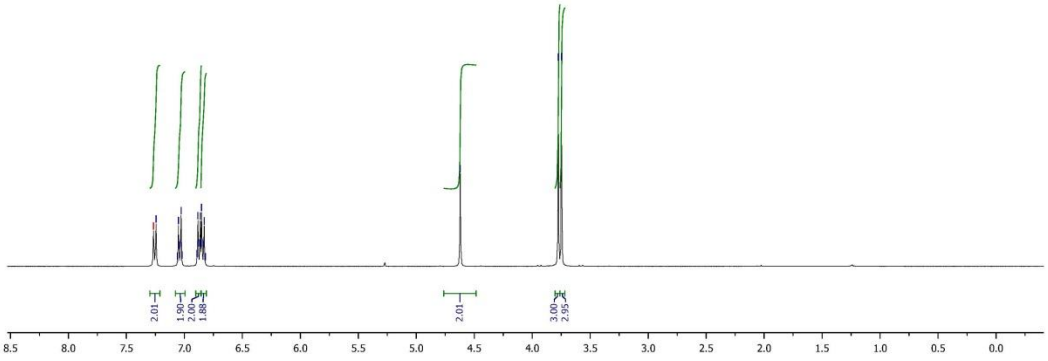
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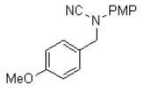
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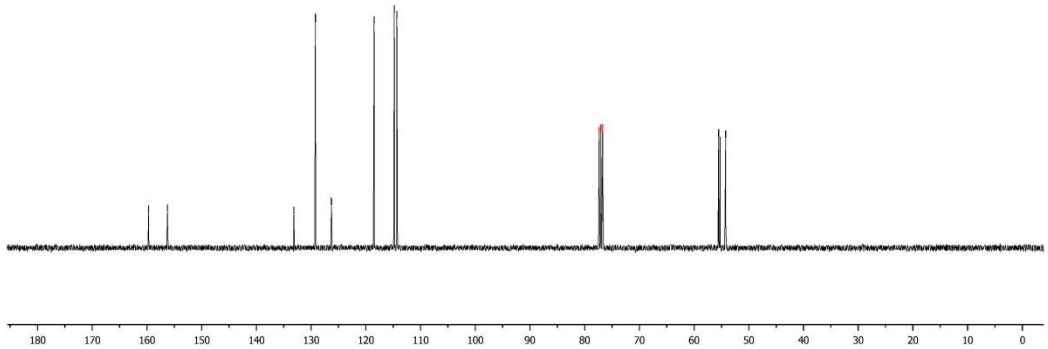
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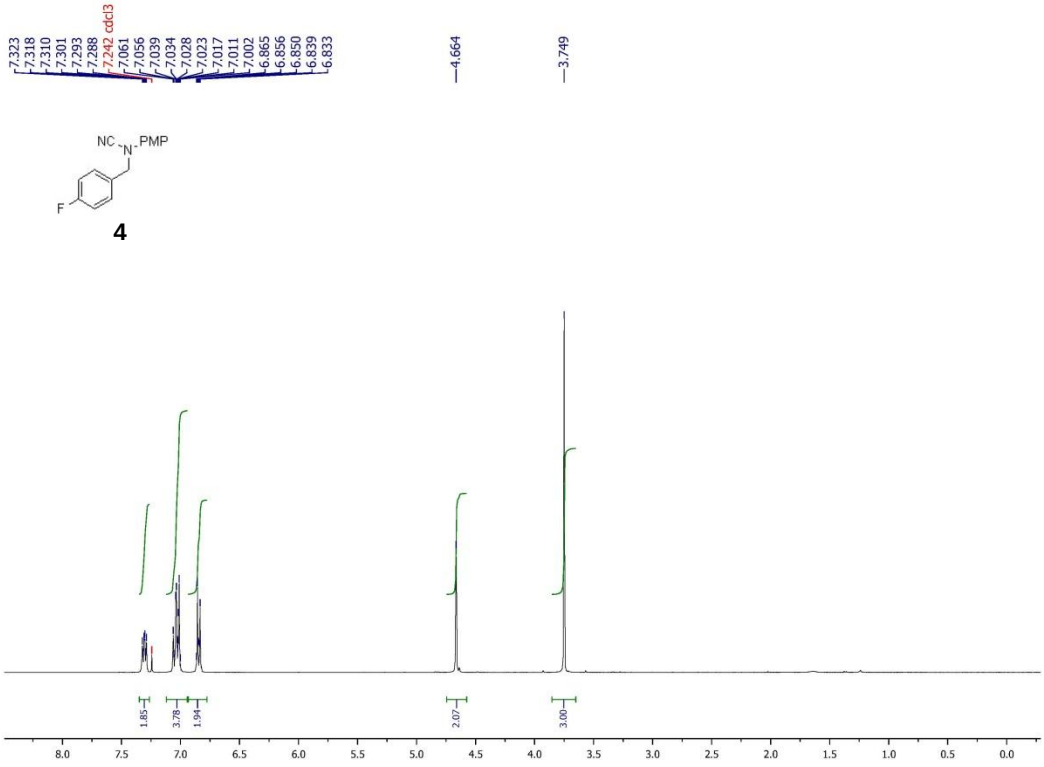
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7.039
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7.011
7.002
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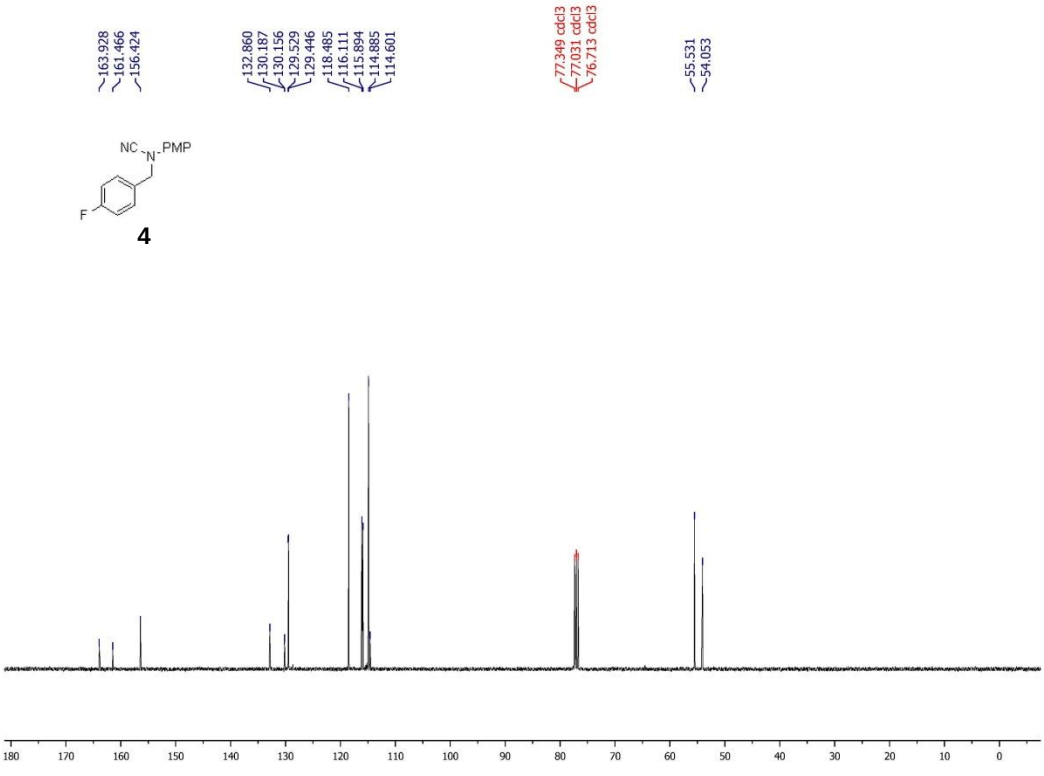
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PMP
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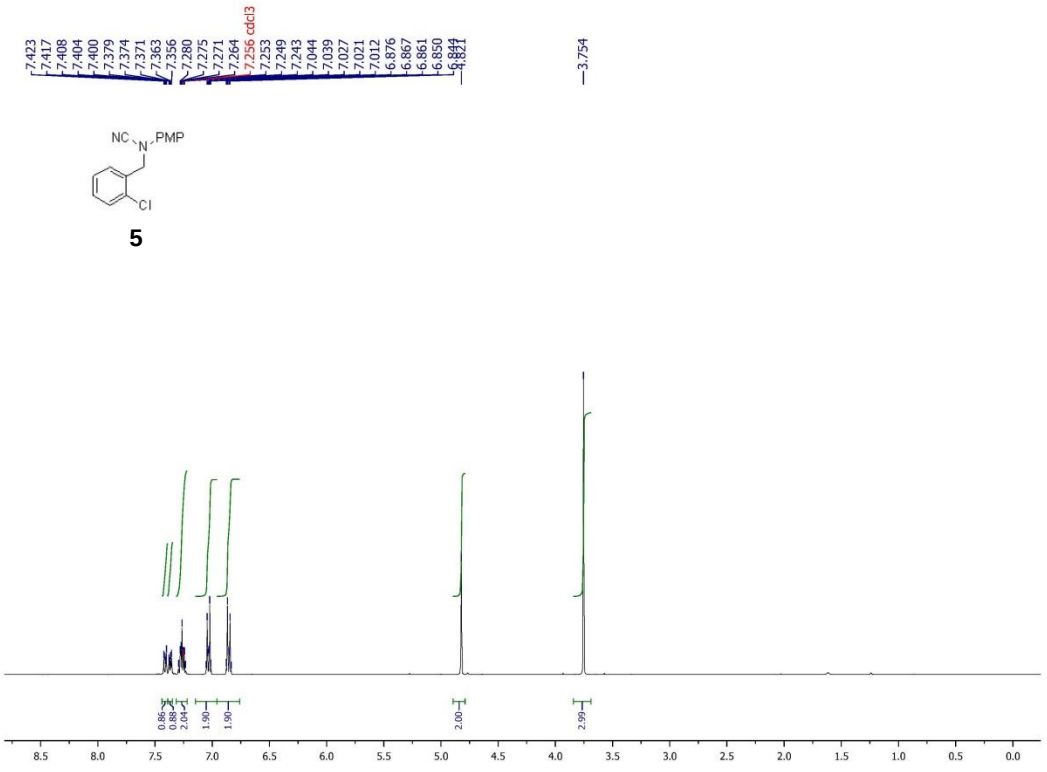
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7.243
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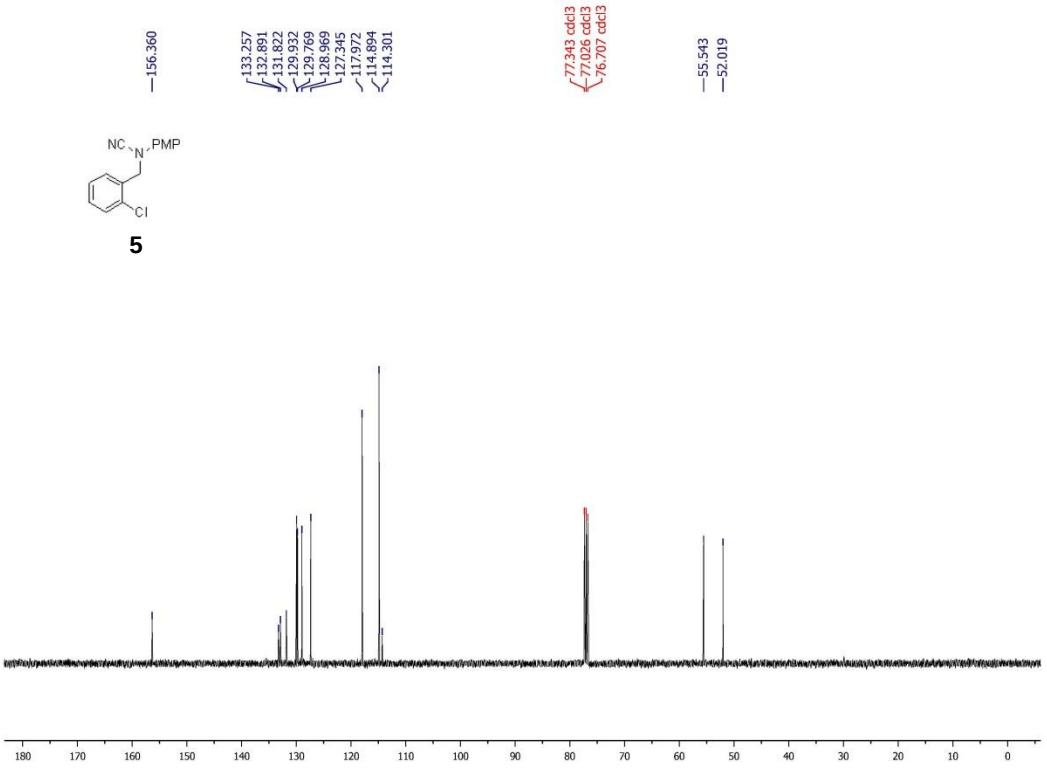
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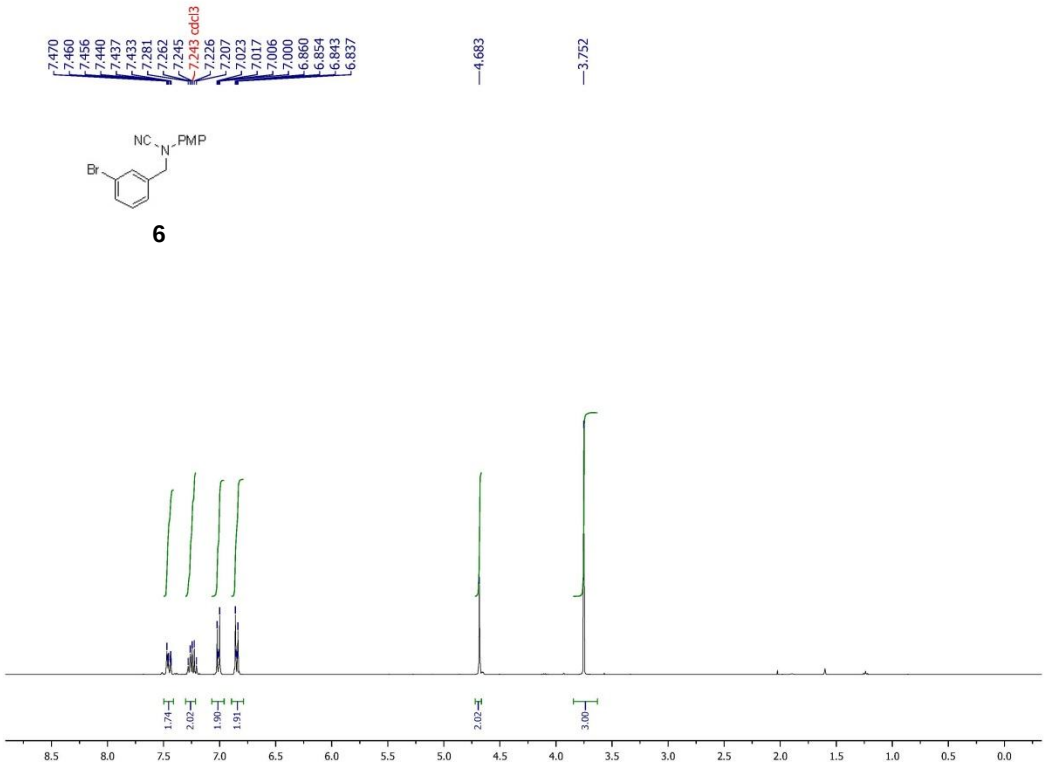
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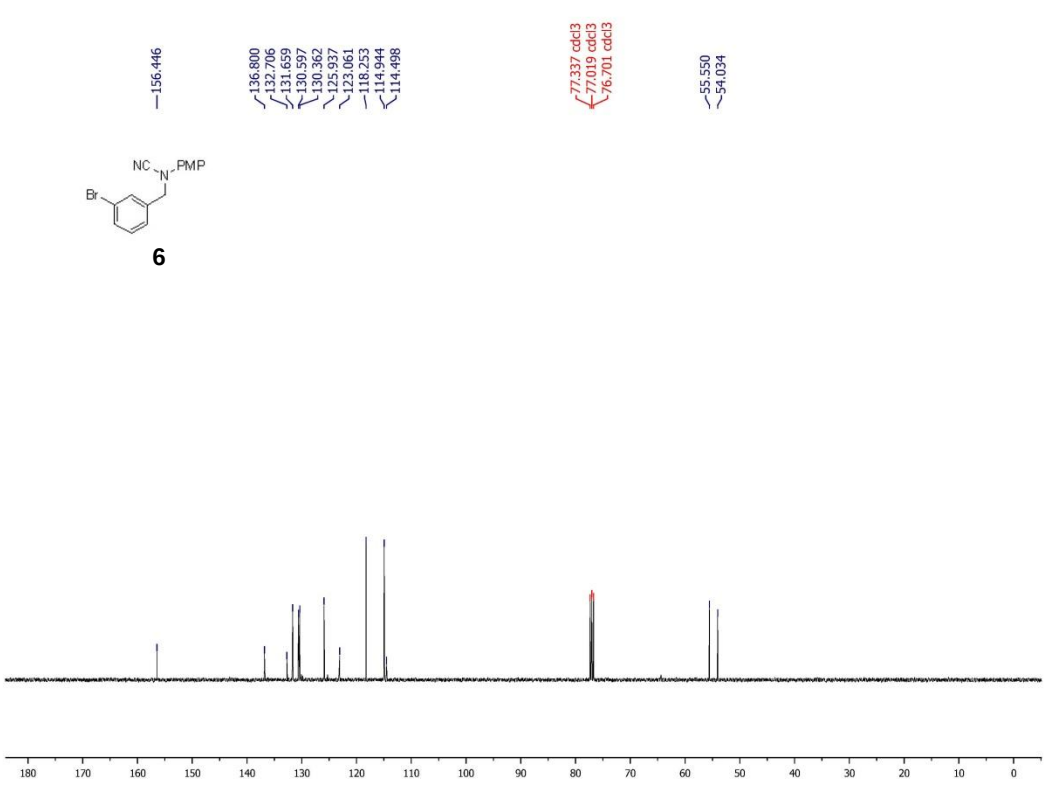
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76.701 cdcl3
55.550
54.034
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PMP
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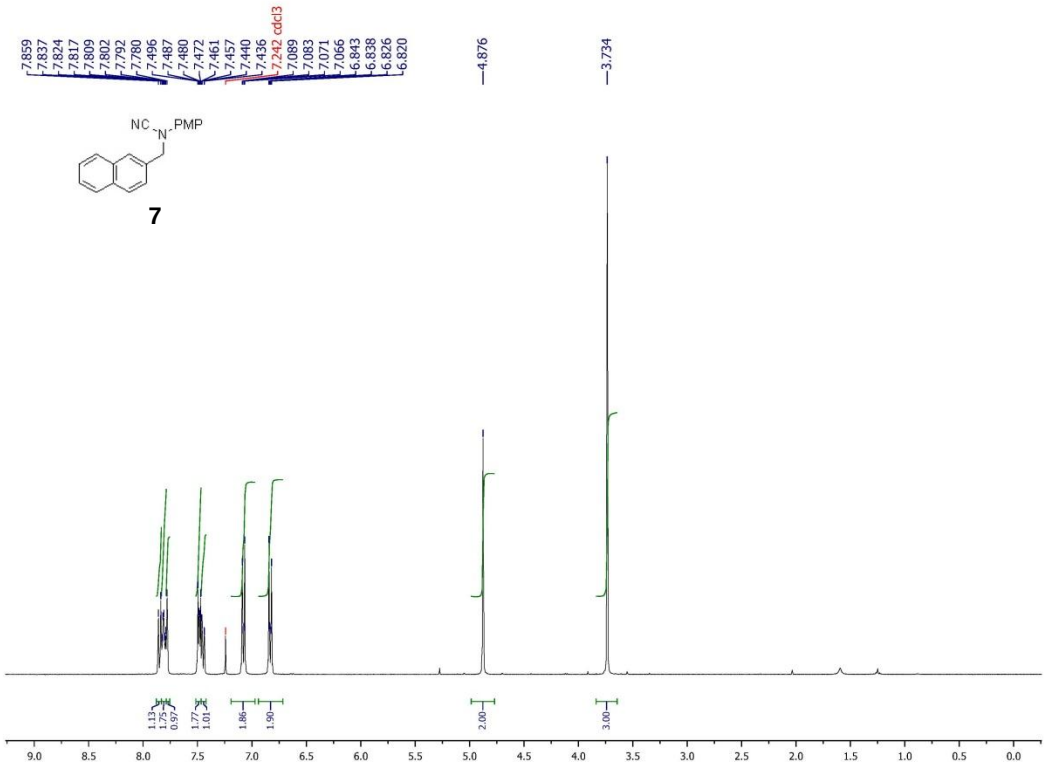
6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7.457
7.440
7.436
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7.066
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6.826
6.820
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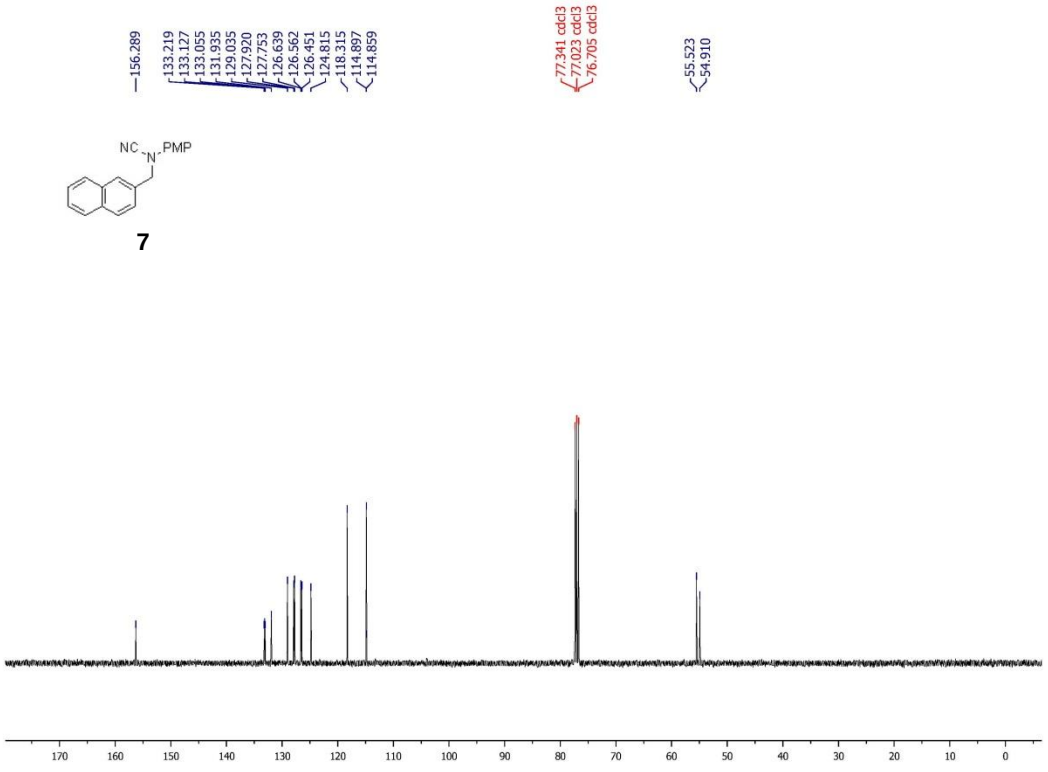
7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118.315
114.897
114.859
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77.023 cdcl3
76.705 cdcl3
55.523
54.910
NC
N
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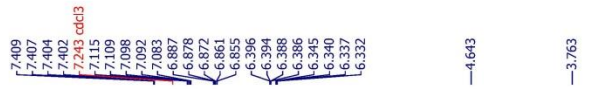
7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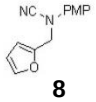
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PMP
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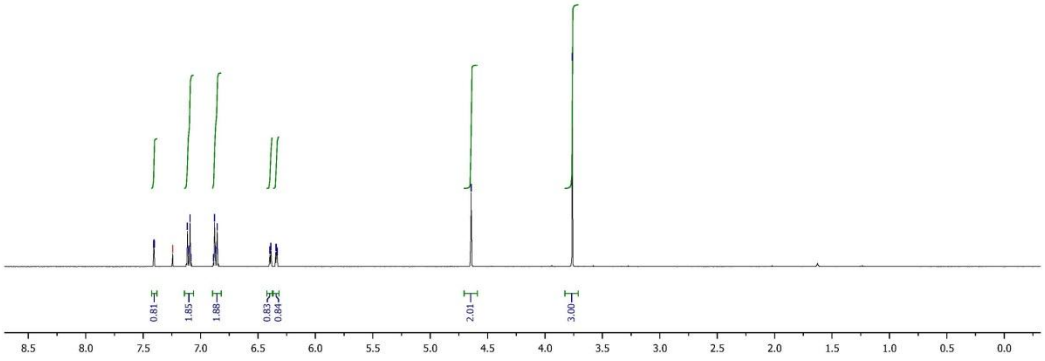
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4.0
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3.0
2.5
2.0
1.5
1.0
0.5
0.0


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PMP
O

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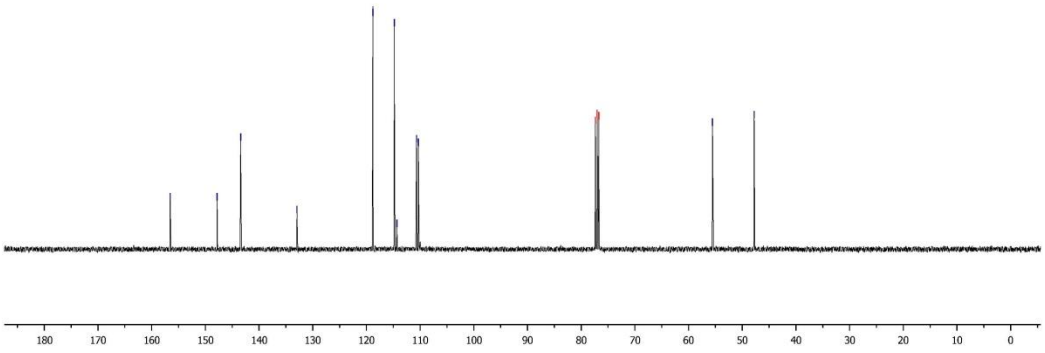
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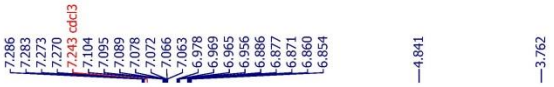
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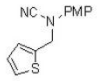
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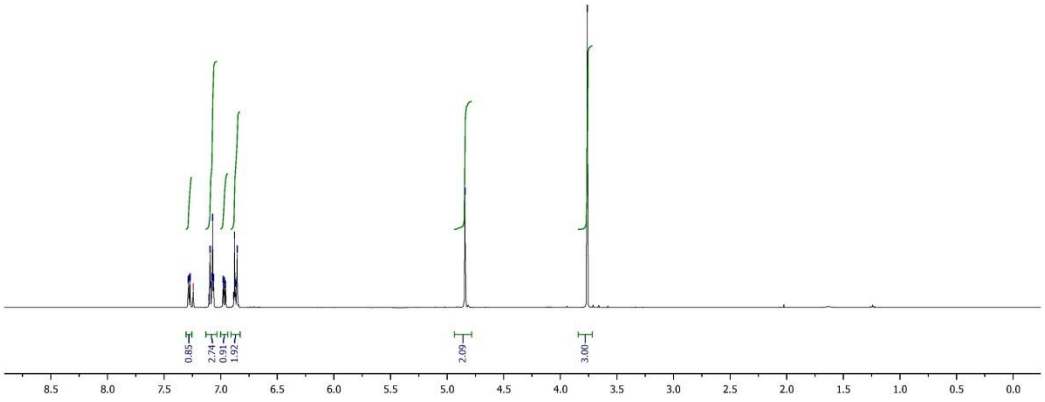
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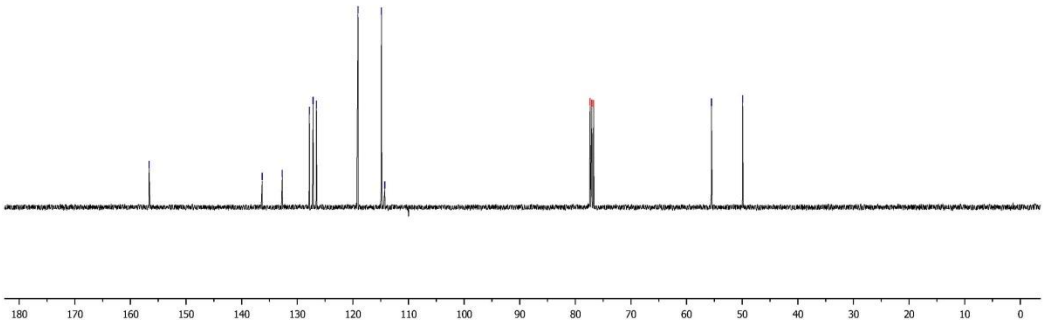
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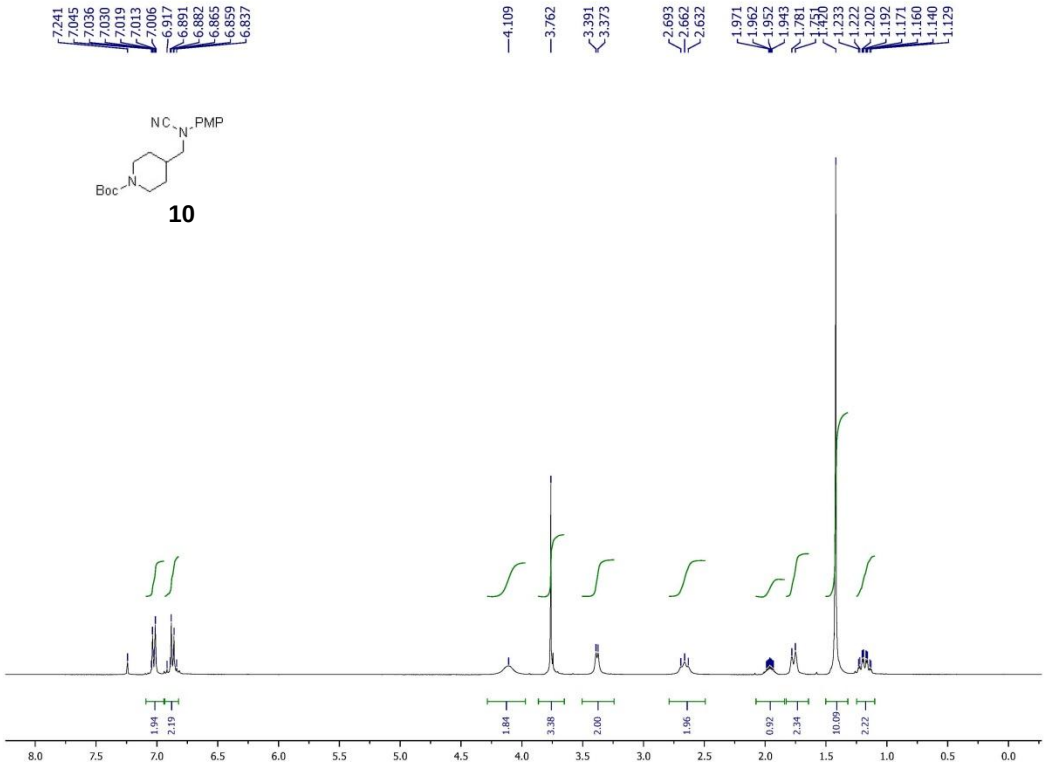
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PMP
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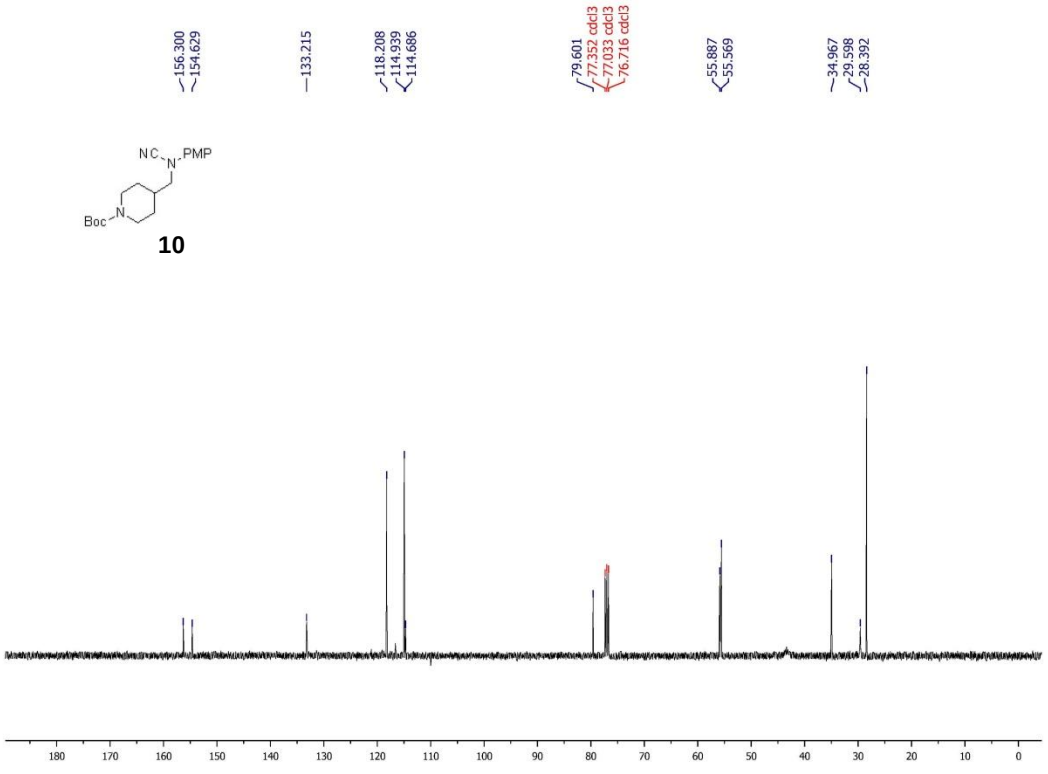
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PMP
Boc
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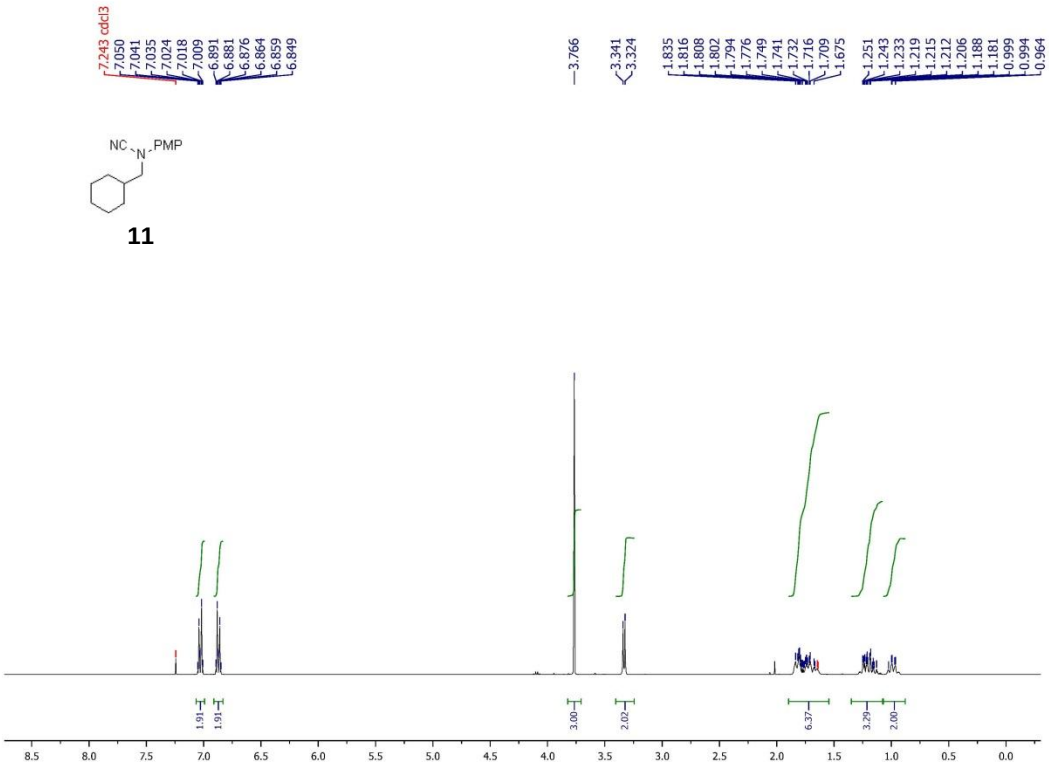
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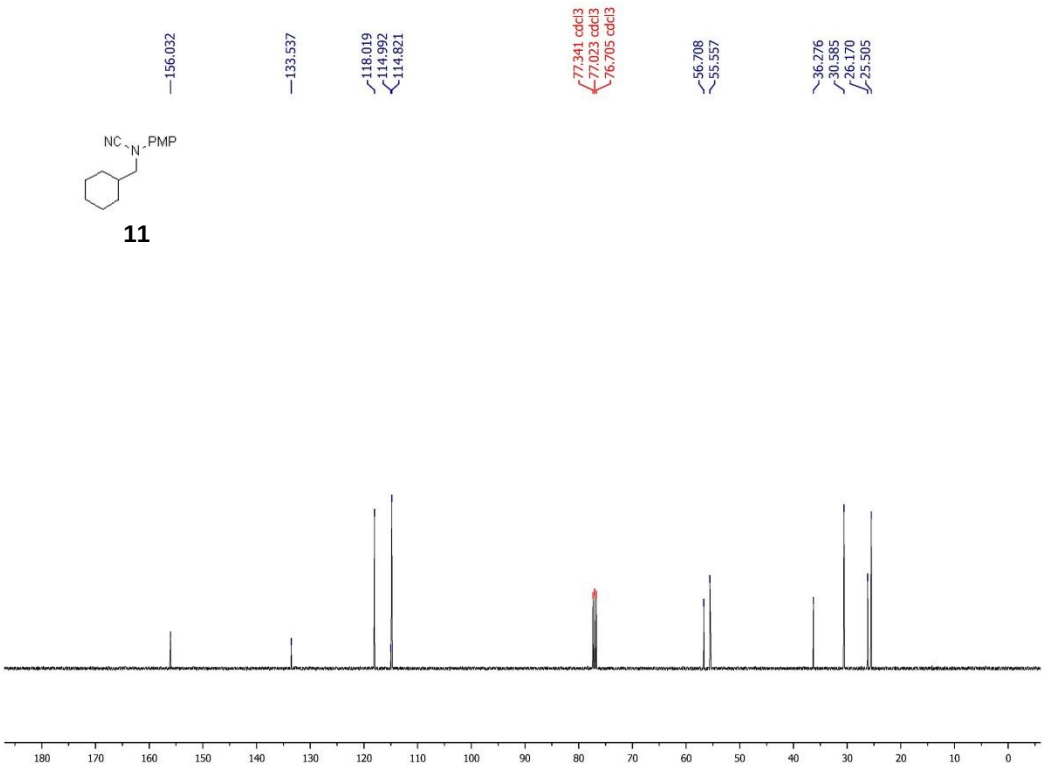
11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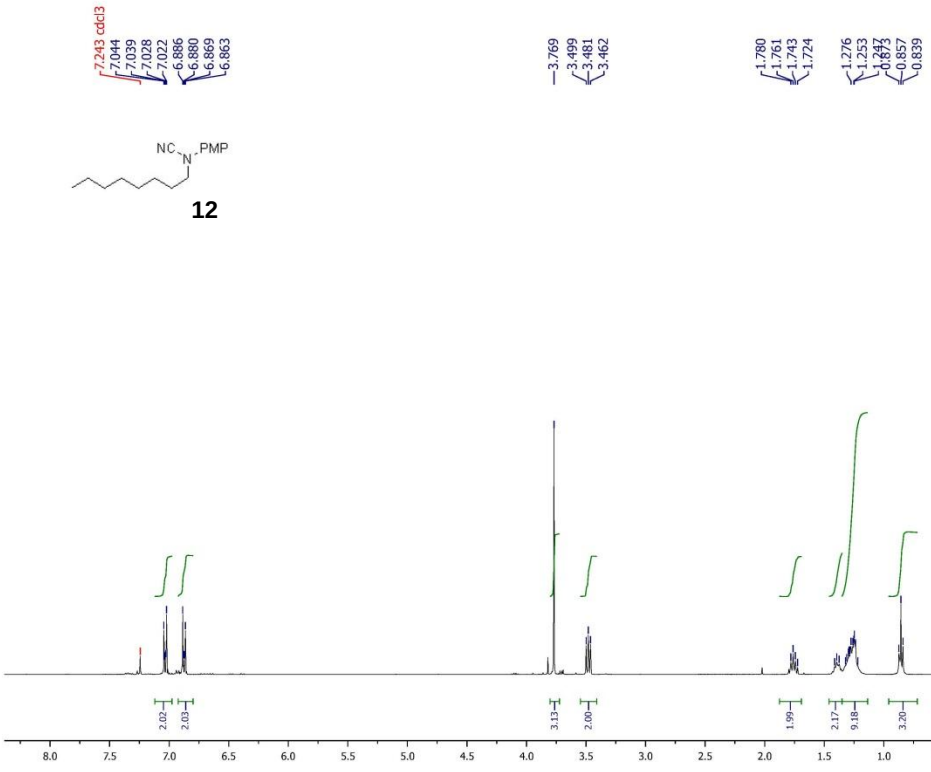
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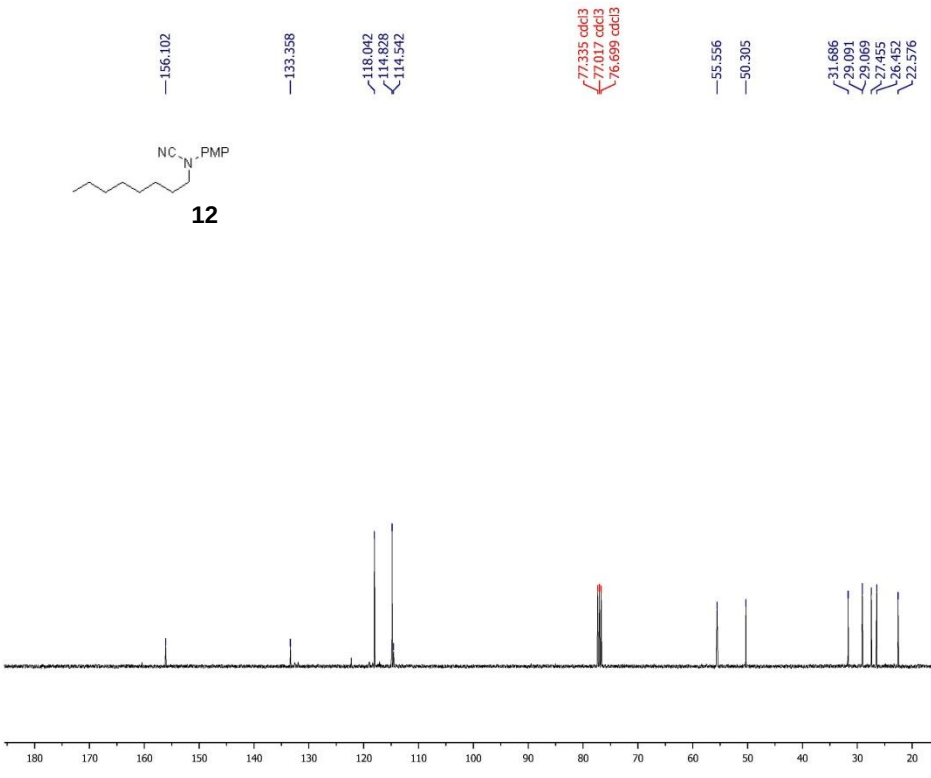
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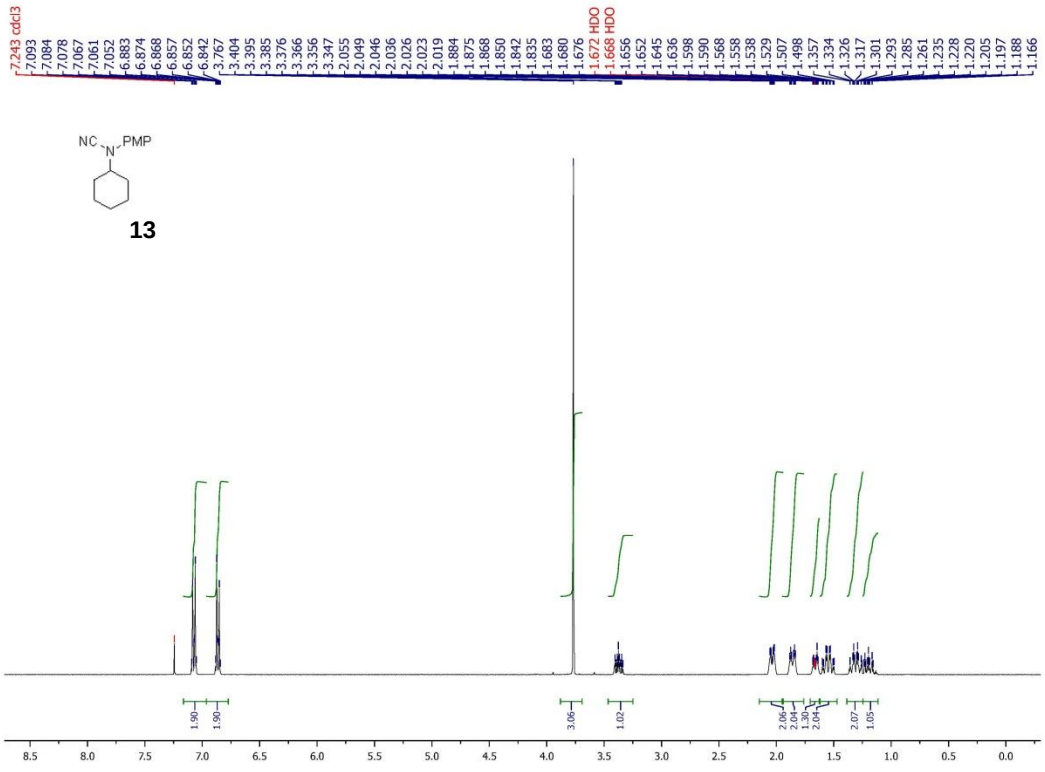
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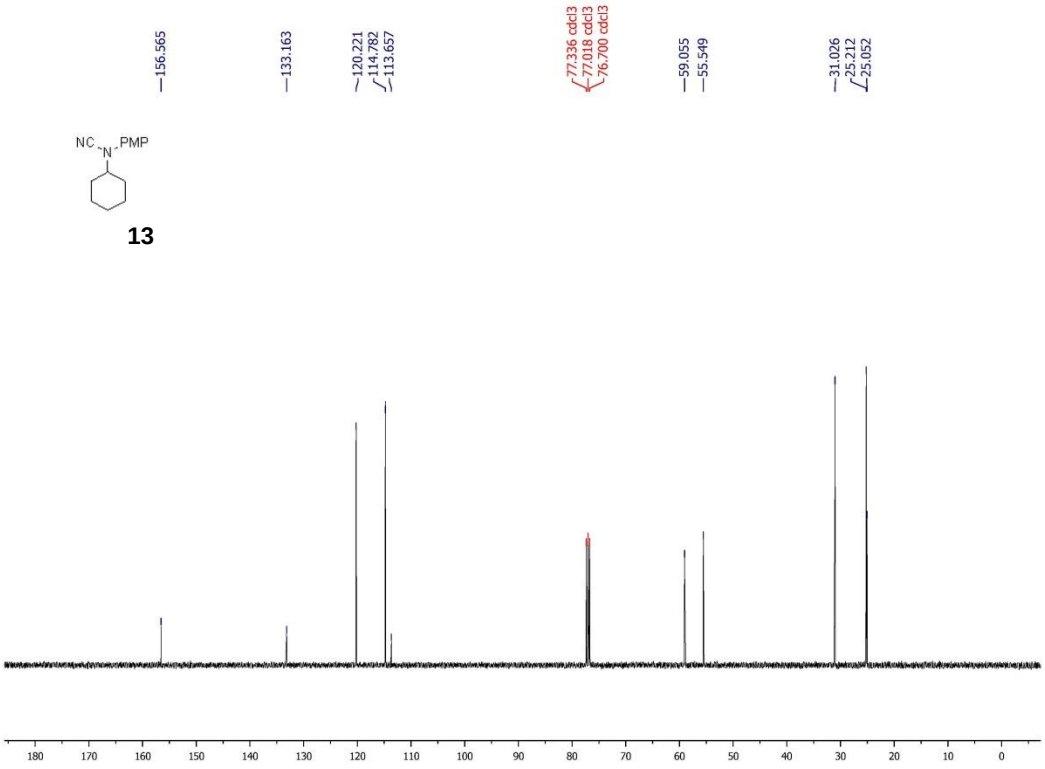
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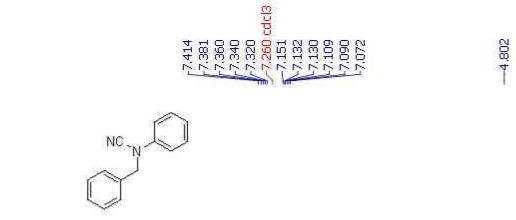
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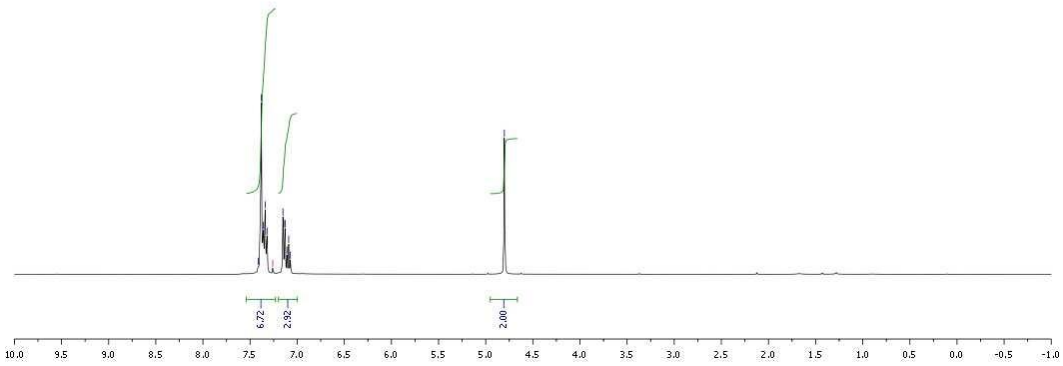
7.072
4.802
NC
N

14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5.5
5.0
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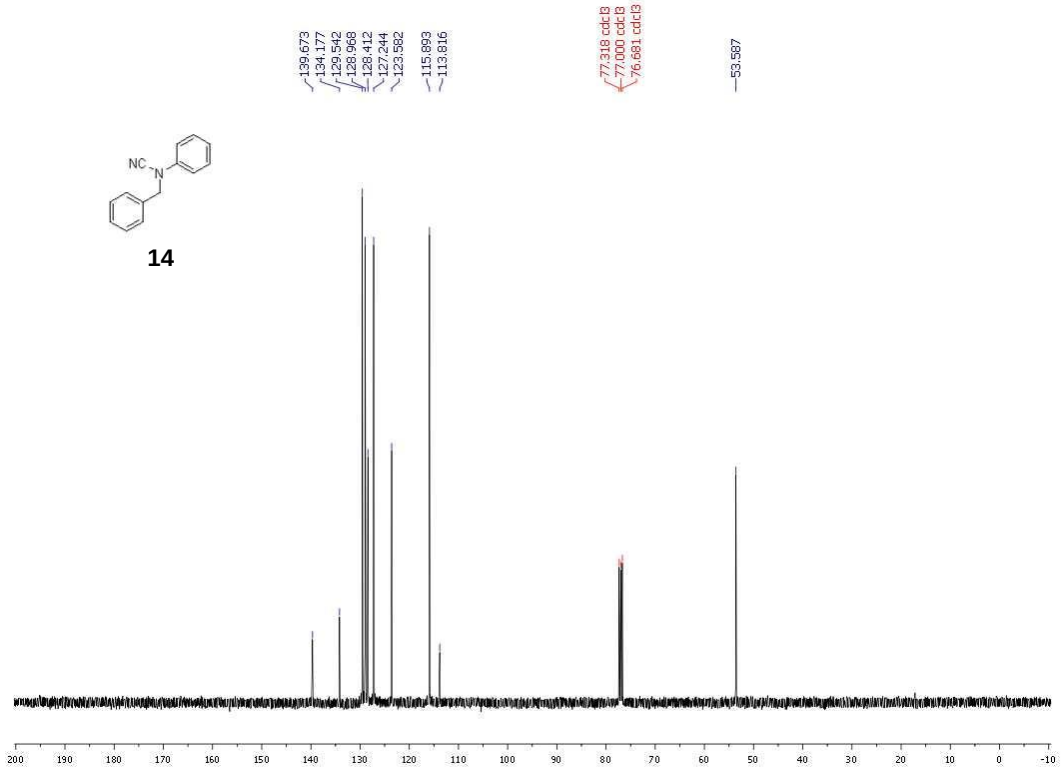
-0.5
-1.0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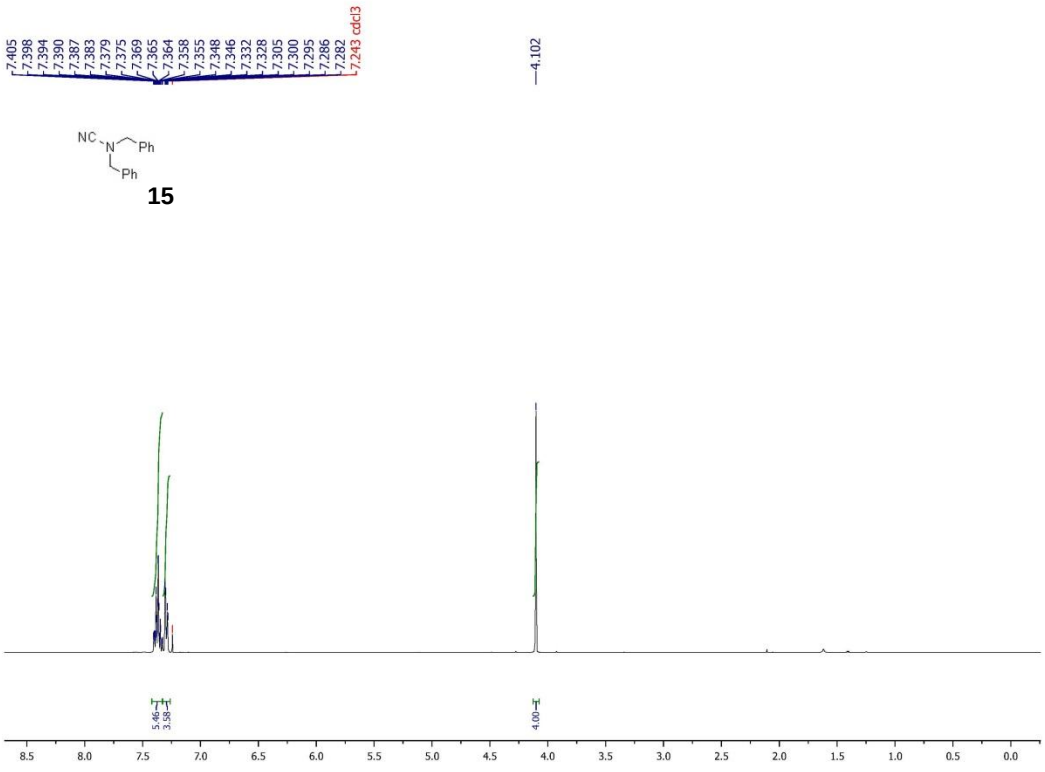
134.177
129.542
128.968
128.412
127.244
123.582
115.893
113.816
77.318 cdcl3
77.000 cdcl3
76.681 cdcl3
53.587
NC
N
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

14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7.383
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7.375
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7.355
7.348
7.346
7.332
7.328
7.305
7.300
7.295
7.286
7.282
7.243 cdcl3
4.102
NC
N
Ph
Ph
15
5.46
3.88
4.00
8.5
8.0
7.5
7.0
6.5
6.0
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4.5
4.0
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2.0
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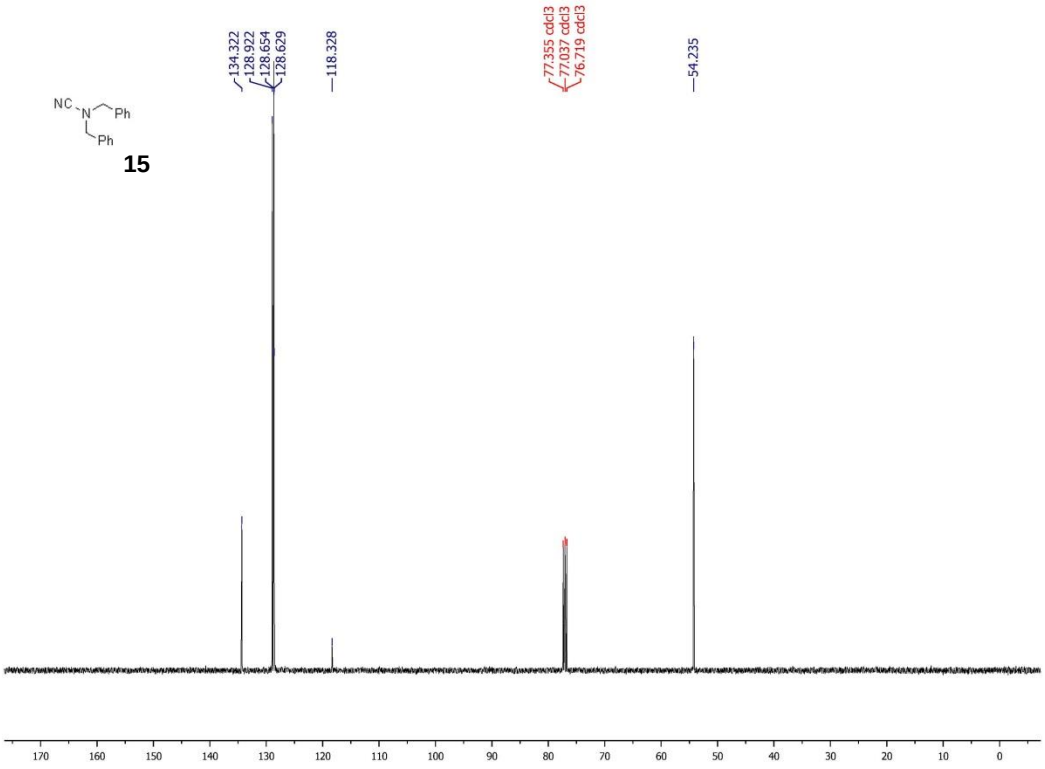
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128.922
128.654
128.629
118.328
77.355 cdcl3
77.037 cdcl3
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54.235
NC
N
Ph
Ph
15
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


7.331
7.327
7.318
7.314
7.298
7.295
7.290 cdcl3
7.243
7.240
7.236
7.226
7.221
7.216
7.206
7.203
7.200
7.192
7.187
7.183
7.171
7.170
7.168
7.162
3.546
3.543
3.537
3.530
3.514
3.512
3.504
3.498
3.495
3.170
3.137
3.131
2.606
2.600
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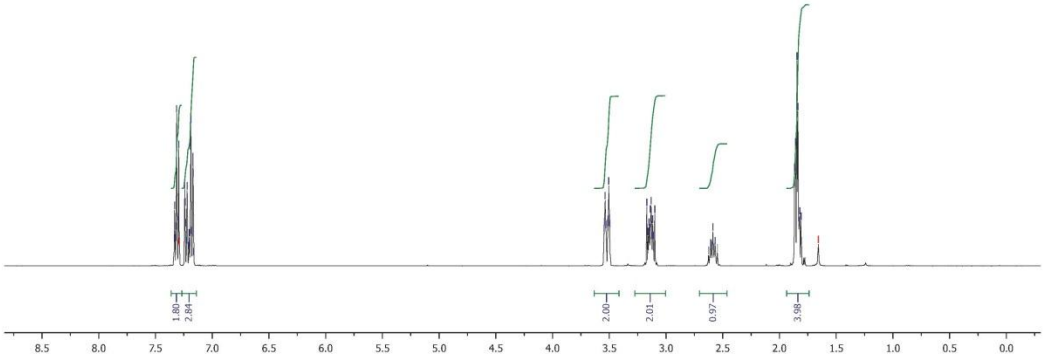
1.846
1.837
1.827
1.820
1.810
1.657 HDO


Ph
N
CN

16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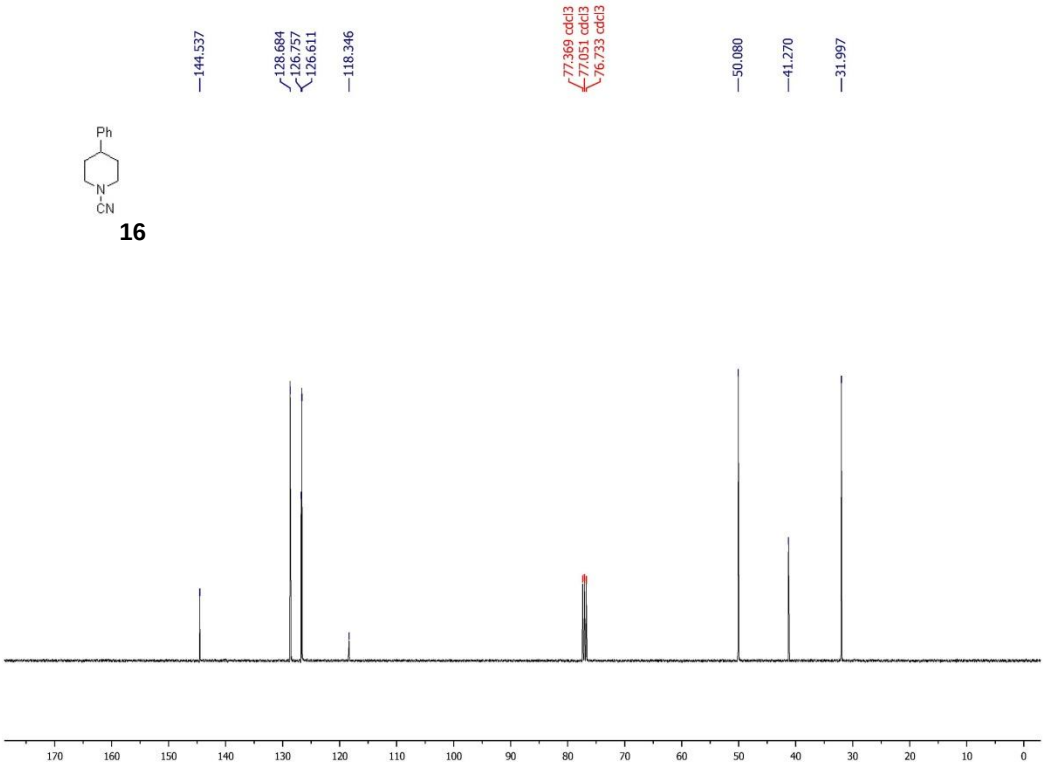
0.0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77.051 cdcl3
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170
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110
100
90
80
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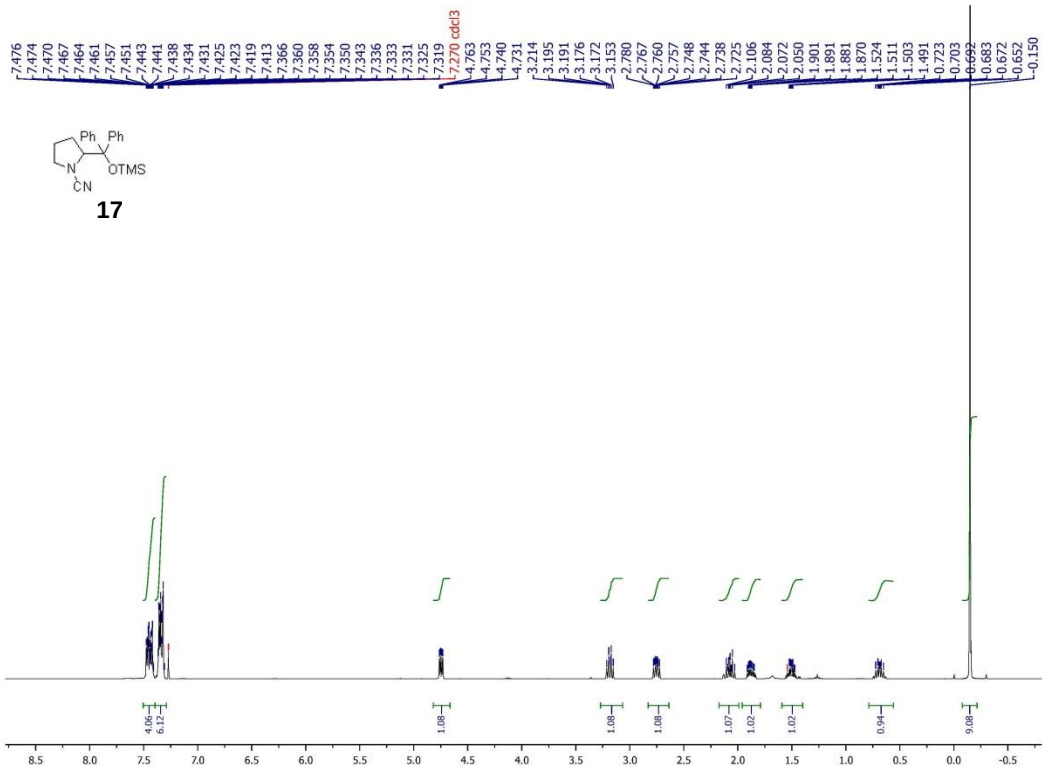
50
40
30
20
10
0

Ph
N
CN

16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7.470
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7.461
7.457
7.451
7.443
7.441
7.438
7.434
7.431
7.425
7.423
7.419
7.413
7.366
7.360
7.358
7.354
7.350
7.343
7.336
7.333
7.331
7.325
7.319
7.270 cdcl3
4.763
4.753
4.740
4.731
3.214
3.195
3.191
3.176
3.172
3.153
2.780
2.767
2.760
2.757
2.748
2.744
2.738
2.725
2.106
2.084
2.072
2.050
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1.891
1.881
1.870
1.524
1.511
1.503
1.491
0.723
0.703
0.692
0.683
0.672
0.652
0.150
Ph
Ph
OTMS
CN
17
4.06
6.12
1.08
1.08
1.08
1.08
1.07
1.02
1.02
0.94
9.08
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
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1.5
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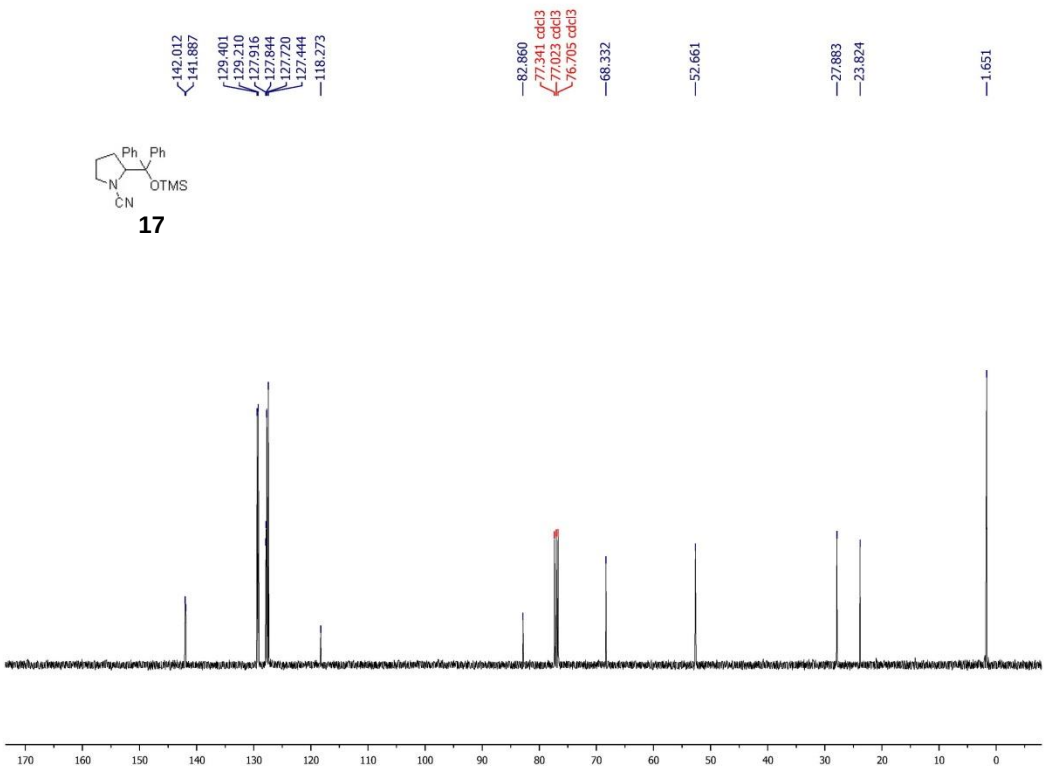
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129.210
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127.720
127.444
118.273
82.860
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77.023 cdcl3
76.705 cdcl3
68.332
52.661
27.883
23.824
1.651
Ph
Ph
OTMS
CN
17
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0