

Supporting Information

Primary 1,2-diamine catalysis III: An unexpected domino reaction for the synthesis of multisubstituted cyclohexa-1,3-dienamine

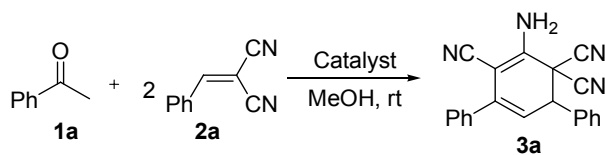
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General Methods. Unless otherwise stated, all reagents were purchased from commercial suppliers and were used without further purification. Reactions were monitored by thin layer chromatography (TLC) on GF₂₅₄ silica gel plates, using UV light as a visualizing agent. Flash column chromatography was performed using 200-300 mesh silica gel. ¹H NMR spectra and ¹³C NMR spectra were recorded on a Varian INOVA-500 (500 MHz) spectrometer in needful D-reagents with tetramethylsilane (TMS) as an internal reference. Data for ¹H NMR are reported as follows: chemical shift (ppm), and multiplicity (s = singlet, d = doublet, t = triplet, dd = double of doublet, br = broad, m = multiplet), coupling constants (Hz) and integration; Data for ¹³C NMR are reported as ppm. Melting points were measured on an X₄-type micro-melting point apparatus and were uncorrected. Electrospray ionization (ESI) mass spectrometry (MS) experiments were performed on an ABI PE-QSTAR mass spectrometer. High-resolution mass spectra (HRMS) were obtained on a Bruker APEX IV FT_MS (7.0) spectrometer for electrospray ionization (ESI).

Catalyst and Solvent Screening:

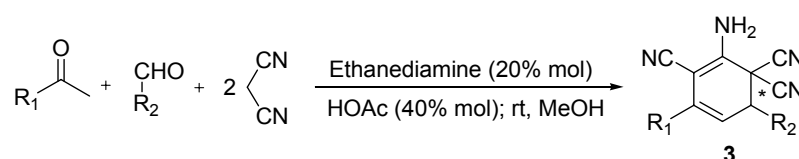


Entry	Catalyst	Additive	Media	Time (h)	3a Yield (%) ^a
1		HOAc ^b	MeOH	12	66
2		HOAc ^b	MeOH	48	63
3		-	MeOH	12	64
4		-	MeOH	48	42
5		HOAc ^c	MeOH	48	trace
6		-	MeOH	48	trace
7		HOAc ^c	MeOH	48	trace
8		-	MeOH	48	trace
9		HOAc ^c	MeOH	48	-
10		-	MeOH	48	-
11		HOAc ^b	CHCl ₃	48	-
12		HOAc ^b	THF	48	-
13		HOAc ^b	DMSO	48	Trace

^a Isolated yield of the corresponding product; ^b The additive loading is 40 mol%; ^c The additive loading is 20 mol%.

General procedure for multicomponent domino reactions:

To a mixture of ary ketone **1**, aromatic aldehyde **2** (1.0 mmol), malononitrile (2.2 mmol) and catalyst ethanediamine (0.2 mmol) in 1.0-1.5 ml of MeOH was added the acid additive HOAc (0.4 mmol). The resulting mixture was stirred under room temperature for the required time monitored by TLC (silica gel, pet ether: ethyl acetate, 4:1). The resulting mixture was then directly purified by filtration and/or recrystallization to afford the pure products **3**.

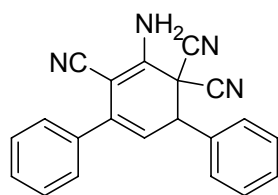


Entry ^a	R ₁	R ₂	Time(h)	Yield (%) ^b
1	Ph	Ph	15	3a -90
2	Ph	2-CIPh	12	3b -92
3	Ph	3-CIPh	12	3c -95
4	Ph	4-MePh	10	3d -89
5	Ph	Me(CH ₂) ₉	10	3e -91
6	4-MePh	Ph	15	3f -88
7	4-MePh	2-CIPh	18	3g -86
8	4-MePh	3-CIPh	18	3h -90
9	4-MePh	3-BrPh	14	3i -96
10	3-CIPh	4-CIPh	13	3j -93
11	3-CIPh	4-MePh	12	3k -93
12	4-FPh	Ph	18	3l -91
13	4-FPh	3-FPh	12	3m -89
14	3,4-diCIPh	Ph	12	3n -95
15	3,4-diCIPh	3-CIPh	12	3o -95
16	3,4-diMeOPh	Ph	32	3p -86

^a All the reactions were conducted at room temperature; ^b Isolated yields of the corresponding products.

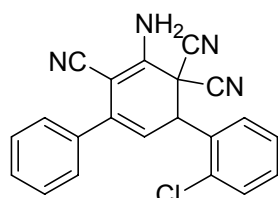
Scope of the Domino Reactions:

2-amino-4,6-diphenylcyclohexa-2,4-diene-1,1,3-tricarbonitrile 3a:¹ obtained in



90% yield; white powder; m.p. 158-160°C; ¹H NMR (500 MHz, CDCl₃): δ 7.50 (s, 1H), 7.46-7.43 (m, 1H), 7.42-7.39 (m, 2H), 7.32-7.31(m, 2H), 7.23 (d, *J* = 7.5 Hz, 2H), 5.72 (d, *J* = 4.0 Hz, 1H), 5.60 (s, 2H), 4.24 (d, *J* = 4.0 Hz, 1H), 2.39 (s, 3H); ESI (*m/z*): [(M+H)⁺] calcd. for C₂₁H₁₅N₄ 323.1297, found 323.1305.

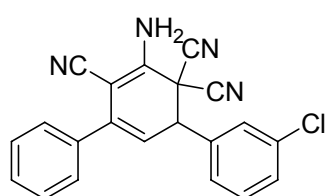
2-amino-6-(2-chlorophenyl)-4-phenylcyclohexa-2,4-diene-1,1,3-tricarbonitrile 3b:



obtained in 92% yield;² light yellow powder; m.p. 152-154°C; ¹H NMR (500 MHz, CDCl₃): δ 7.66 (dd, *J* = 2.5 Hz, 7.5 Hz, 1H), 7.54 (dd, *J* = 2.0 Hz, 7.5 Hz, 1H), 7.40-7.25 (m, 7H), 5.69 (d, *J* = 4.0 Hz, 1H), 5.67 (s, 2H), 5.11 (d, *J* = 4.0 Hz, 1H);

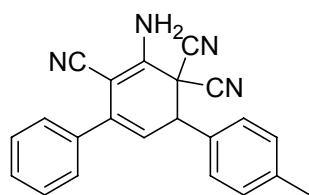
¹³C NMR (125 MHz, CDCl₃): δ 145.8, 137.9, 136.6, 135.2, 132.2, 131.3, 130.7, 130.3, 129.5, 129.0, 128.3, 127.7, 116.3, 115.9, 111.5, 110.9, 82.2, 44.2, 43.0.

2-amino-6-(3-chlorophenyl)-4-phenylcyclohexa-2,4-diene-1,1,3-tricarbonitrile 3c:



obtained in 95% yield; colorless crystals; m.p. 226-228°C; ¹H NMR (500 MHz, CDCl₃): δ 7.51 (m, 1H), 7.48-7.45 (m, 1H), 7.42-7.39 (m, 8H), 5.77 (d, *J* = 3.5 Hz, 1H), 5.24 (s, 2H), 4.25 (d, *J* = 4.0 Hz, 1H); ¹³C NMR (125 MHz,

CDCl₃): δ 145.1, 137.8, 136.1, 135.7, 135.3, 130.6, 130.3, 129.5, 129.4, 129.1, 128.8, 127.6, 127.5, 115.3, 111.6, 110.2, 82.6, 48.6, 43.9; ESI (*m/z*): [(M+Na)⁺] calcd. for C₂₁H₁₃ClN₄Na 379.0726, found 379.1204.

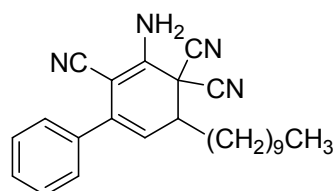


2-amino-4-phenyl-6-p-tolylcyclohexa-2,4-diene-1,1,3-tricarbonitrile 3d: obtained in 89% yield; yellow powder; m.p.

210-212°C; ¹H NMR (400 MHz, CDCl₃): δ 7.43 (m, 5H), 7.40 (d, *J* = 10.5 Hz, 2H), 7.27 (d, *J* = 9.5 Hz, 2H), 5.82 (d, *J* = 5.0 Hz, 1H), 5.59 (s, 2H), 4.26 (d, *J* = 4.5 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃):

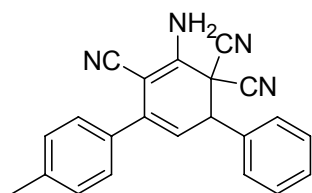
δ 145.6, 139.8, 137.0, 136.3, 129.8, 128.9, 127.3, 116.1, 115.6, 115.6, 111.8, 110.4, 81.3, 48.6, 44.1, 21.0; ESI (m/z): $[(M+Na)^+]$ calcd. for $C_{22}H_{16}N_4Na$ 359.1273, found 359.1270.

2-amino-6-decyl-4-phenylcyclohexa-2,4-diene-1,1,3-tricarbonitrile 3e: obtained in



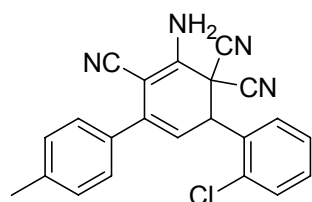
91% yield; colorless oil; 1H NMR (500 MHz, $CDCl_3$): δ 7.41-7.36 (m, 5H), 5.61 (d, $J = 3.5$ Hz, 1H), 5.45 (s, 2H), 3.01 (dt, $J = 3.5$ Hz, 1H), 1.93-1.87 (m, 1H), 1.87-1.78 (m, 1H), 1.66-1.60 (m, 1H), 1.56-1.26 (m, 19H), 0.89 (t, $J = 6.5$ Hz, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 145.3, 136.5, 136.3, 129.0, 128.6, 127.4, 115.8, 115.5, 112.4, 110.6, 82.3, 42.4, 42.3, 31.9, 29.9, 29.5, 29.5, 29.3, 29.3, 29.2, 26.4, 22.7, 14.1; HRMS-ESI (m/z): $[(M+H)^+]$ calcd. for $C_{25}H_{30}N_4Na$ 409.2368, found 409.2365.

2-amino-6-phenyl-4-p-tolylcyclohexa-2,4-diene-1,1,3-tricarbonitrile 3f:³ obtained



in 88% yield; light yellow powder; m.p. 162-164°C; 1H NMR (500 MHz, $CDCl_3$): δ 7.52-7.49 (m, 2H), 7.47-7.44 (m, 3H), 7.44-7.35 (m, 3H), 7.33-7.31 (m, 2H), 7.22 (d, $J = 7.5$ Hz, 2H), 5.79 (d, $J = 3.5$ Hz, 1H), 5.53 (s, 2H), 4.26 (d, $J = 3.5$ Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 145.3, 139.3, 137.2, 133.8, 133.5, 129.9, 129.7, 129.4, 129.4, 129.3, 129.1, 128.4, 127.3, 115.7, 115.6, 111.9, 110.5, 82.5, 49.1, 44.2, 21.3.

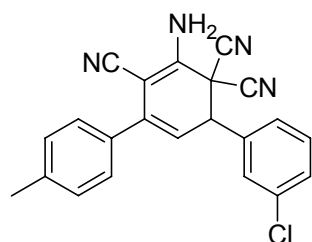
2-amino-6-(2-chlorophenyl)-4-p-tolylcyclohexa-2,4-diene-1,1,3-tricarbonitrile 3g:



obtained in 86% yield;⁴ yellow powder; m.p. 208-210°C; 1H NMR (500 MHz, $CDCl_3$): δ 7.66 (dd, $J = 2.0$ Hz, 7.5 Hz, 1H), 7.53 (dd, $J = 1.5$ Hz, 7.5 Hz, 1H), 7.41-7.35 (m, 2H), 7.31 (d, $J = 8.0$ Hz, 1H), 7.22 (d, $J = 8.0$ Hz, 1H), 5.66 (d, $J = 4.0$ Hz, 1H), 5.96 (s, 2H), 5.10 (d, $J = 3.5$ Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 145.2, 139.3, 137.5, 134.9, 133.4, 131.9, 130.9, 130.5, 130.0, 129.4,

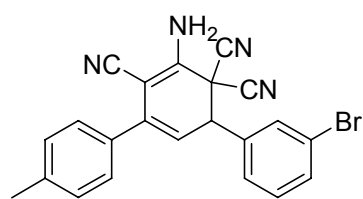
127.9, 127.3, 115.6, 115.4, 111.3, 110.6, 82.3, 43.9, 42.7, 21.3.

2-amino-6-(3-bromophenyl)-4-p-tolylcyclohexa-2,4-diene-1,1,3-tricarbonitrile 3h:



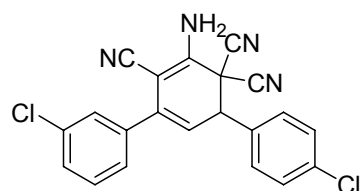
obtained in 90% yield;⁴ white powder; m.p. 98-99 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.49 (s, 1H), 7.47-7.43 (m, 1H), 7.40-7.38 (m, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 5.72 (d, *J* = 4.0 Hz, 1H), 5.63 (s, 2H), 4.23 (d, *J* = 4.0 Hz, 1H), 2.39 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 145.4, 139.4, 137.7, 135.8, 135.2, 133.3, 130.6, 130.2, 129.5, 129.4, 127.6, 127.3, 115.6, 114.5, 111.6, 110.3, 82.2, 48.6, 43.9, 21.3.

2-amino-6-(3-bromophenyl)-4-p-tolylcyclohexa-2,4-diene-1,1,3-tricarbonitrile 3i:



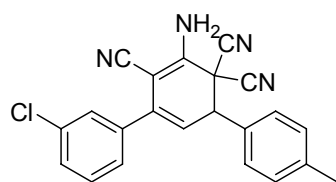
obtained in 96% yield; white powder; mp: 205-209 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.65 (m, 1H), 7.61-7.59 (m, 1H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.35-7.30 (m, 3H), 7.25-7.22 (m, 2H), 5.71 (d, *J* = 3.5 Hz, 1H), 5.63 (s, 2H), 4.22 (d, *J* = 3.5 Hz, 1H), 2.39 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 145.4, 139.4, 137.8, 136.1, 133.3, 133.1, 132.4, 130.8, 129.4, 128.1, 127.3, 123.2, 115.6, 114.5, 111.7, 110.3, 82.2, 48.5, 43.9, 21.3; HRMS-ESI (*m/z*): [(M+H)⁺] calcd. for C₂₂H₁₅BrN₄Na 437.0378, found 437.0377.

2-amino-4-(3-chlorophenyl)-6-(4-chlorophenyl)cyclohexa-2,4-diene-1,1,3-tricarbonitrile 3j:



obtained in 93% yield; white powder; mp: 172-174 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.51-7.43 (m, 4H), 7.41-7.35 (m, 3H), 7.32-7.30 (m, 1H), 5.78 (d, *J* = 4.0 Hz, 1H), 5.68 (s, 2H), 4.27 (d, *J* = 3.5 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 145.7, 137.9, 136.7, 136.3, 134.7, 131.8, 130.7, 130.0, 129.7, 129.4, 127.6, 125.7, 116.3, 115.2, 111.5, 110.2, 81.4, 48.4, 43.9; HRMS-ESI (*m/z*): [(M+H)⁺] calcd. for C₂₁H₁₂Cl₂N₄Na 413.0337, found 413.0334.

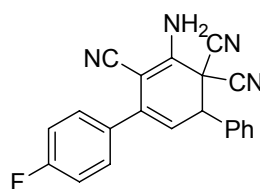
2-amino-4-(3-chlorophenyl)-6-p-tolylcyclohexa-2,4-diene-1,1,3-tricarbonitrile 3k:



obtained in 93% yield; light yellow powder; mp: 159-162 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.41-7.34 (m, 5H), 7.32-7.30 (m, 1H), 7.27-7.26 (m, 2H), 5.82 (d, *J* = 4.5 Hz, 1H), 5.60 (s, 2H), 4.24 (d, *J* = 4.0 Hz, 1H), 2.39 (s, 3H);

¹³C NMR (125 MHz, CDCl₃): δ 145.7, 140.2, 138.1, 136.1, 134.7, 130.3, 130.1, 130.0, 129.3, 129.2, 127.6, 125.7, 117.3, 115.4, 111.8, 110.4, 81.5, 48.7, 44.1, 21.2; HRMS-ESI (*m/z*): [(M+H)⁺] calcd. for C₂₁H₁₅ClN₄Na 393.0883, found 393.0878.

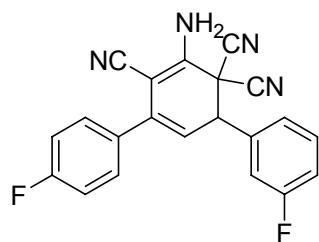
2-amino-4-(4-fluorophenyl)-6-phenylcyclohexa-2,4-diene-1,1,3-tricarbonitrile 3l:



obtained in 91% yield; white powder; mp: 160-161 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.51-7.45 (m, 5H), 7.42-7.38 (m, 2H), 7.14-7.09 (m, 2H), 5.79 (d, *J* = 3.5 Hz, 1H), 5.60 (s, 2H), 4.27 (d, *J* = 3.5 Hz, 1H), 2.39 (s, 3H); ¹³C NMR (125 MHz,

CDCl₃): δ 163.3 (d, ¹*J*_{C-F} = 247.9 Hz), 145.6, 136.4, 133.6, 132.5, 130.0, 129.4, 129.3, 116.2, 115.9, 115.7, 115.5, 111.8, 110.4, 81.9, 49.0, 44.1; HRMS-ESI (*m/z*): [(M+Na)⁺] calcd. for C₂₁H₁₃FN₄Na 359.1108, found 363.1019.

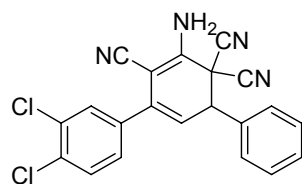
2-amino-6-(3-fluorophenyl)-4-(4-fluorophenyl)cyclohexa-2,4-diene-1,1,3-tricarbo



nitrile 3m: obtained in 89% yield; white powder; mp: 162-164 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.51-7.45 (m, 5H), 7.42-7.38 (m, 2H), 7.14-7.09 (m, 2H), 5.79 (d, *J* = 3.5 Hz, 1H), 5.60 (s, 2H), 4.27 (d, *J* = 3.5 Hz, 1H), 2.39 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 163.3 (d, ¹*J*_{C-F} = 248.0

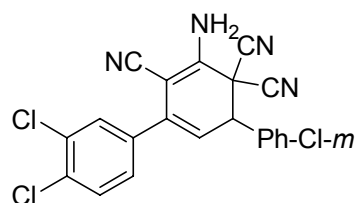
Hz), 162.9 (d, ¹*J*_{C-F} = 247.0 Hz), 145.6, 136.9, 136.0, 135.9, 132.3, 131.1, 131.0, 129.4, 129.3, 125.2, 117.2, 117.1, 116.5, 116.4, 115.9, 115.7, 115.4, 115.3, 111.6, 110.2, 81.8, 48.6, 43.8; HRMS-ESI (*m/z*): [(M+H)⁺] calcd. for C₂₁H₁₃F₂N₄ 359.1108, found 359.1106.

2-amino-4-(3,4-dichlorophenyl)-6-phenylcyclohexa-2,4-diene-1,1,3-tricarbonitrile



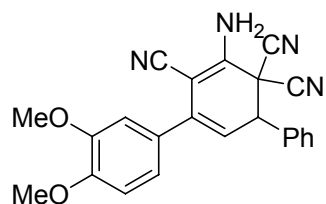
3n: obtained in 95% yield; light yellow powder; m.p.: 124-126 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.52-7.48 (m, 7H), 7.27 (dd, *J* = 1.6 Hz, 8.0 Hz, 1H), 5.84 (d, *J* = 3.5 Hz, 1H), 5.63 (s, 2H), 4.27 (d, *J* = 3.5 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 145.9, 136.2, 135.4, 133.6, 133.2, 133.1, 130.7, 130.1, 129.5, 129.4, 129.3, 126.8, 117.4, 115.2, 111.6, 110.2, 81.2, 49.0, 43.9; HRMS-ESI (*m/z*): [(M+H)⁺] calcd. for C₂₁H₁₃Cl₂N₄ 391.0517, found 391.0519.

2-amino-6-(3-chlorophenyl)-4-(3,4-dichlorophenyl)cyclohexa-2,4-diene-1,1,3-tricarbonitrile **3o**

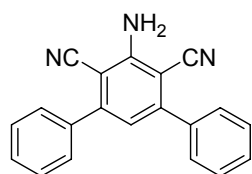


arbitrarily 3o: obtained in 95% yield; white powder; m.p. 129-130 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.53-7.51 (m, 2H), 7.49-7.47 (m, 2H), 7.44-7.35 (m, 3H), 7.28-7.26 (m, 3H), 5.79 (d, *J* = 4.0 Hz, 1H), 5.59 (s, 2H), 4.25 (d, *J* = 3.5 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 145.7, 135.9, 135.9, 135.4, 135.2, 133.7, 133.2, 130.8, 130.7, 130.5, 129.4, 129.4, 127.5, 126.8, 116.4, 114.9, 111.3, 109.9, 81.4, 48.6, 43.7, 14.2; HRMS-ESI (*m/z*): [(M+Na)⁺] calcd. for C₂₁H₁₂Cl₃N₄Na 446.9947, found 446.9942.

4-(3,4-dimethoxyphenyl)-6-phenylcyclohexa-2,4-diene-1,1,2,3-tetracarbonitrile

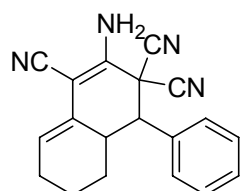


3p: obtained in 86% yield; light yellow powder; m.p. 122-124 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.53-7.48 (m, 5H), 7.01 (dd, *J* = 1.5 Hz, 8.5 Hz, 1H), 6.92-6.89 (m, 2H), 5.79 (d, *J* = 3.5 Hz, 1H), 5.53 (s, 2H), 4.27 (d, *J* = 3.0 Hz, 1H), 3.92 (s, 3H), 3.91 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 149.9, 148.9, 145.3, 136.9, 133.9, 129.9, 129.4, 129.3, 129.0, 120.2, 115.8, 115.1, 111.9, 111.1, 110.6, 110.5, 82.6, 56., 55.9, 49.1, 44.2; HRMS-ESI (*m/z*): [(M+Na)⁺] calcd. for C₂₃H₁₈N₄O₂Na 405.1327, found 405.1322.



2,6-dicyano-3,5-diphenylaniline 4a: obtained in 21% yield; white powder; ^1H NMR (500 MHz, CDCl_3): δ 7.59-7.48 (m, 10H), 6.90 (s, 1H), 5.38 (s, 2H).

2-amino-4-phenyl-4a,5,6,7-tetrahydronaphthalene-1,3,3(4H)-tricarbonitrile 7a:



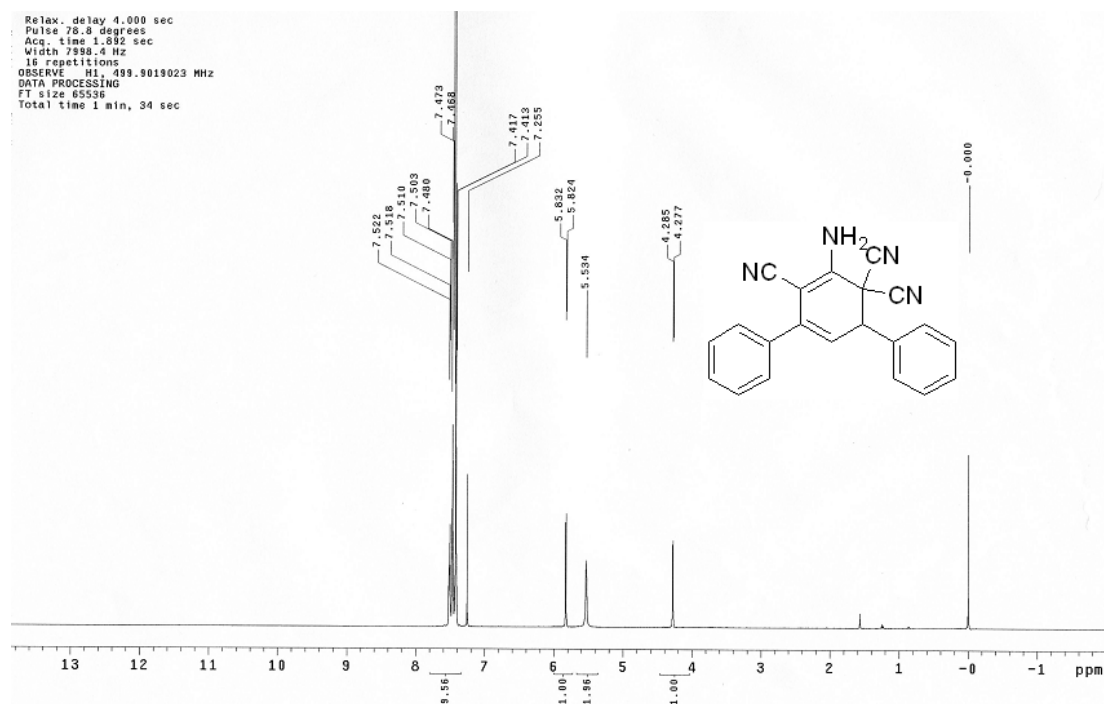
obtained in 86% yield; white powder; m.p. 254-255 °C; ^1H NMR (500 MHz, CDCl_3): δ 7.58 (m, 1H), 7.49-7.44 (m, 4H), 7.29 (m, 1H), 6.05 (d, $J = 2.5$ Hz, 1H), 4.89 (s, 2H), 3.13-3.05 (m, 1H), 2.90-2.85 (m, 1H), 2.31-2.26 (m, 1H), 2.19-2.10 (m, 1H), 1.81-1.78 (m, 1H), 1.70-1.66 (m, 1H), 1.56-1.46 (m, 1H), 0.99-0.91 (m, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 140.3, 133.6, 131.8, 129.6, 129.2, 127.5, 126.6, 125.6, 115.1, 111.9, 111.8, 88.3, 52.2, 43.2, 34.7, 27.2, 25.4, 21.4.

References:

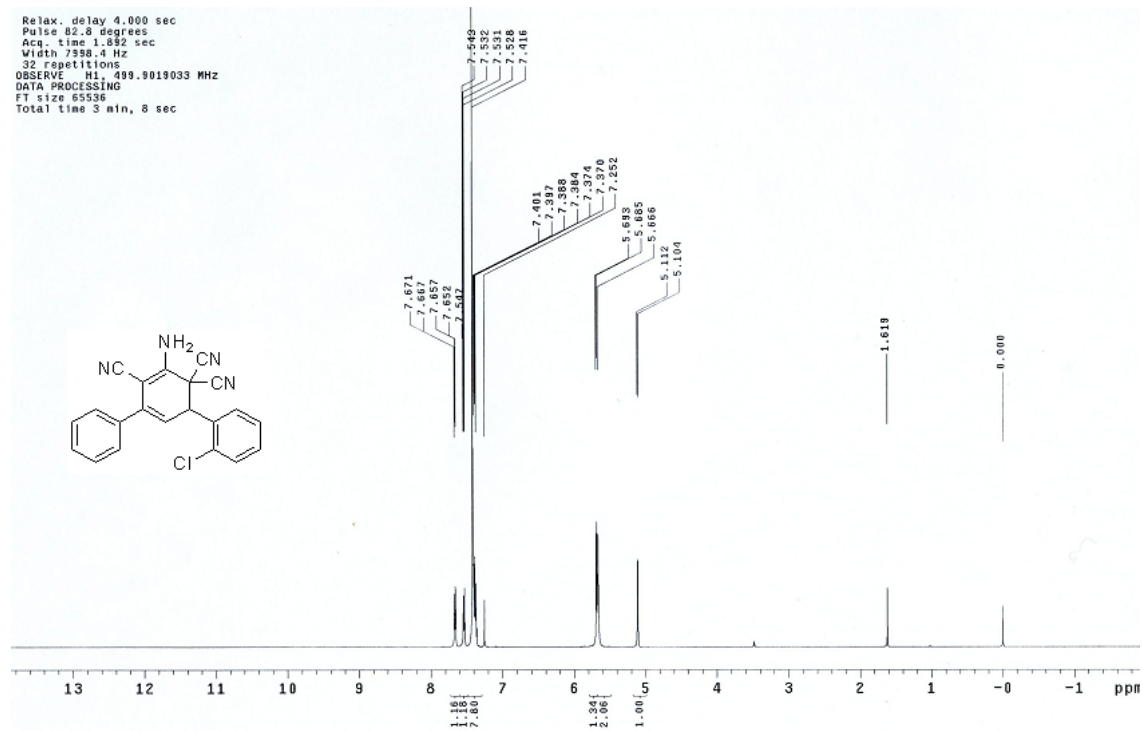
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2. Y. A. Sharanin, Y. A. Baskakov, Y. T. Abramenko, Y. G. Putsykin, A. F. Vasil'ev, E. B. Nazarova, *Zhurnal Organicheskoi Khimii*, 1980, **16**, 2192.
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4. X. S. Wang, M. M. Wang, Q. Li, C. S. Yao, S. J. Tu, *Tetrahedron*, 2007, **63**, 5265.

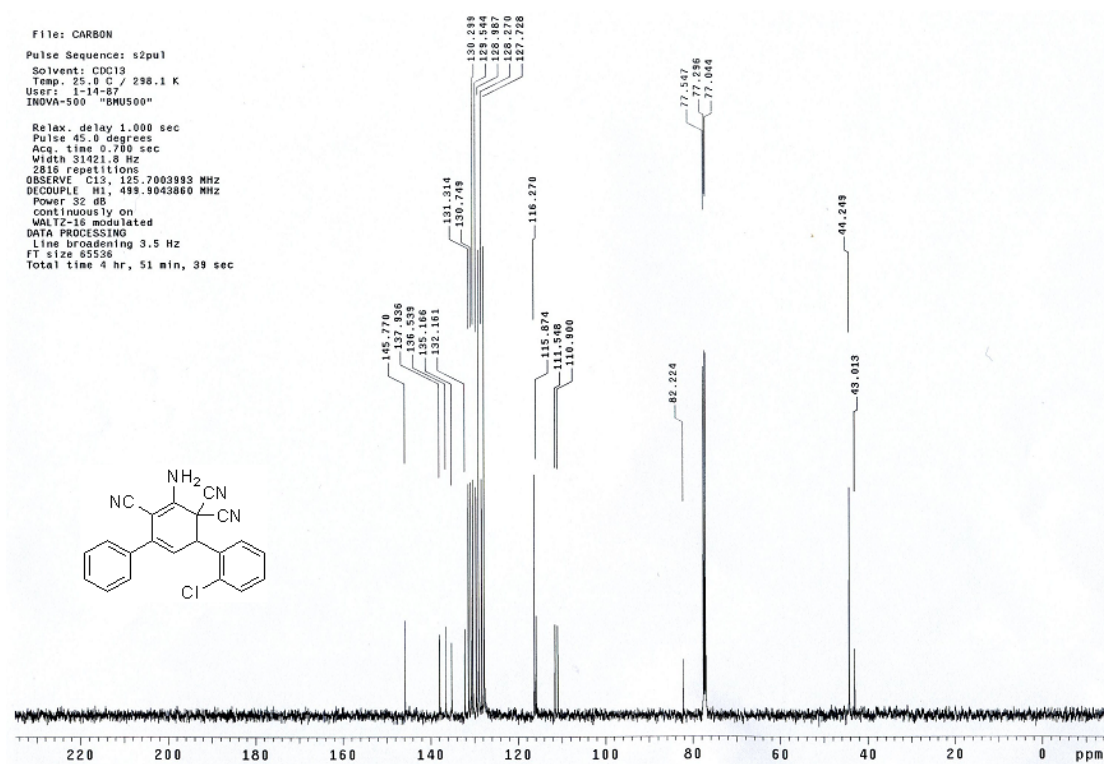
NMR spectra for product 3

3a

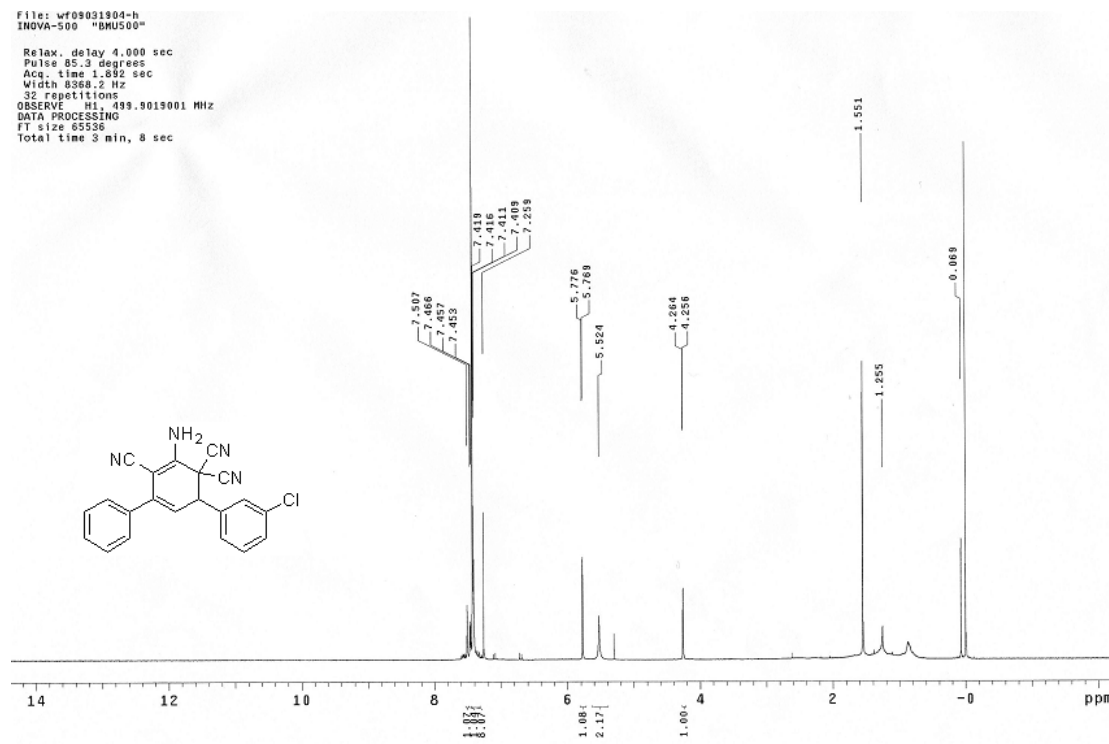


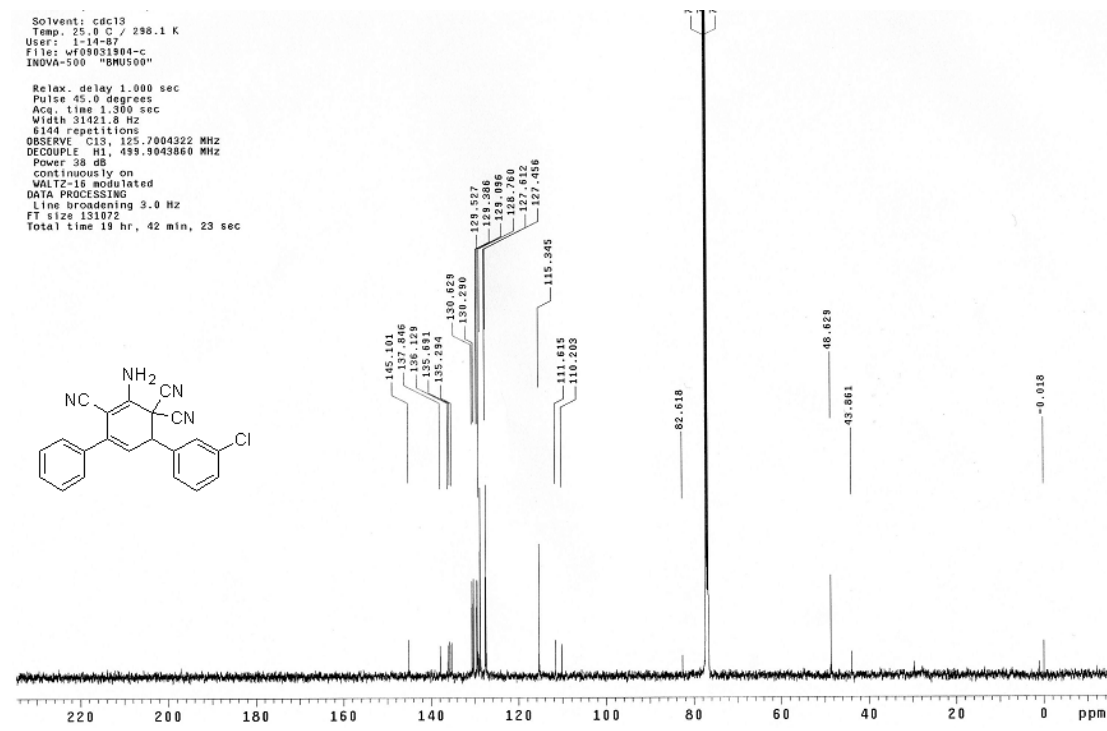
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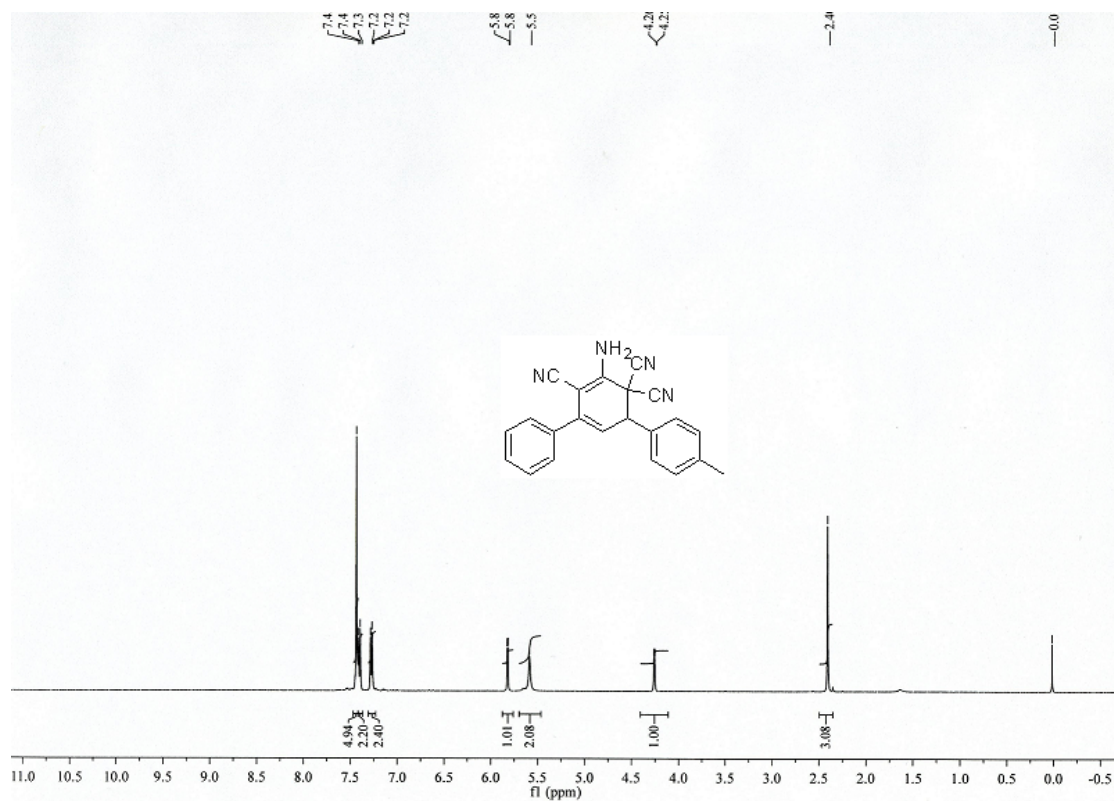


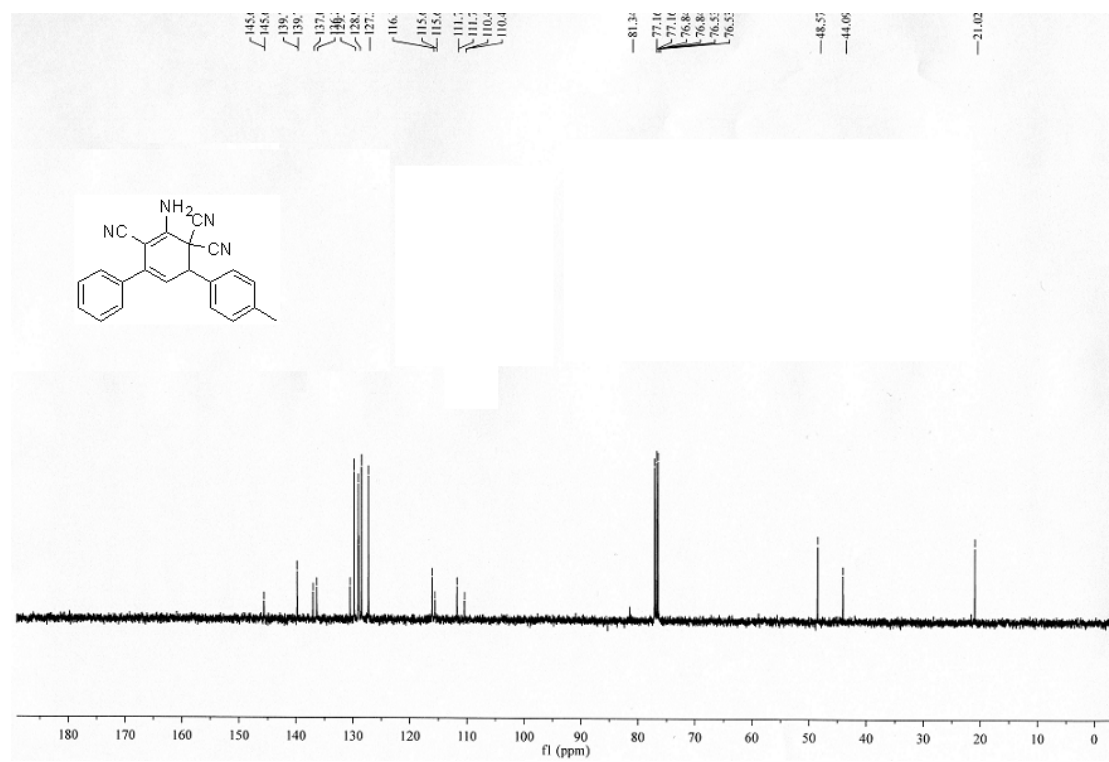
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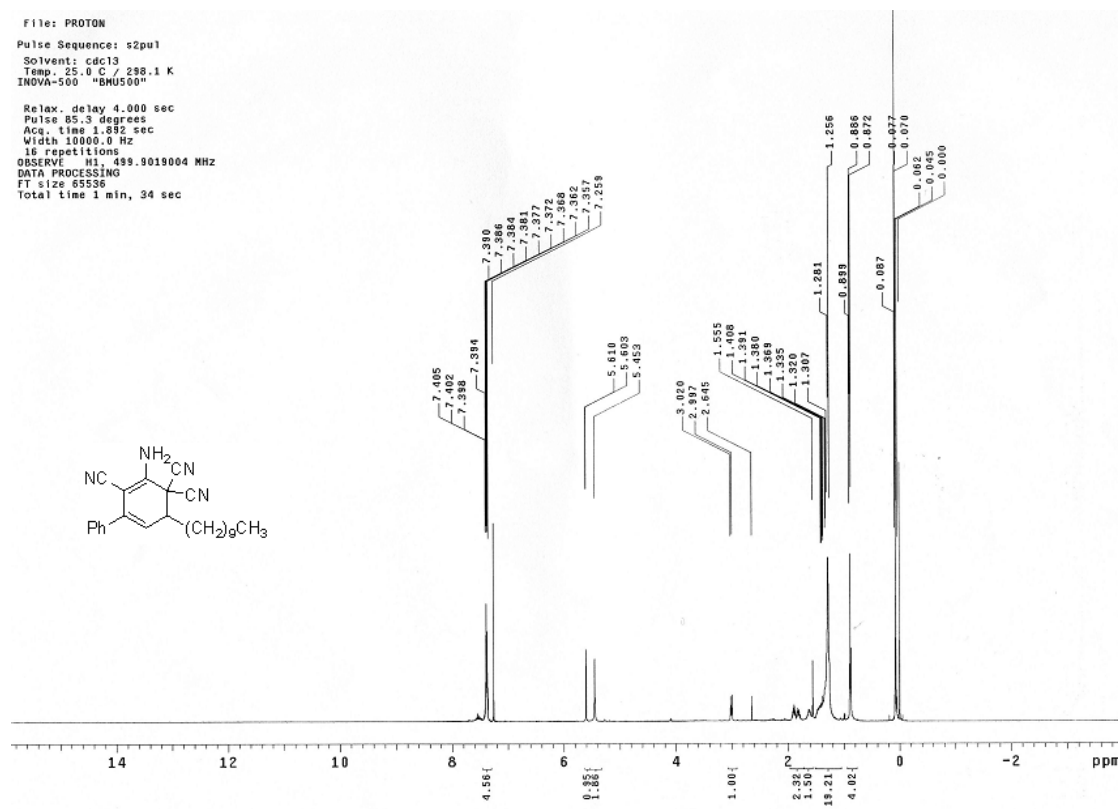


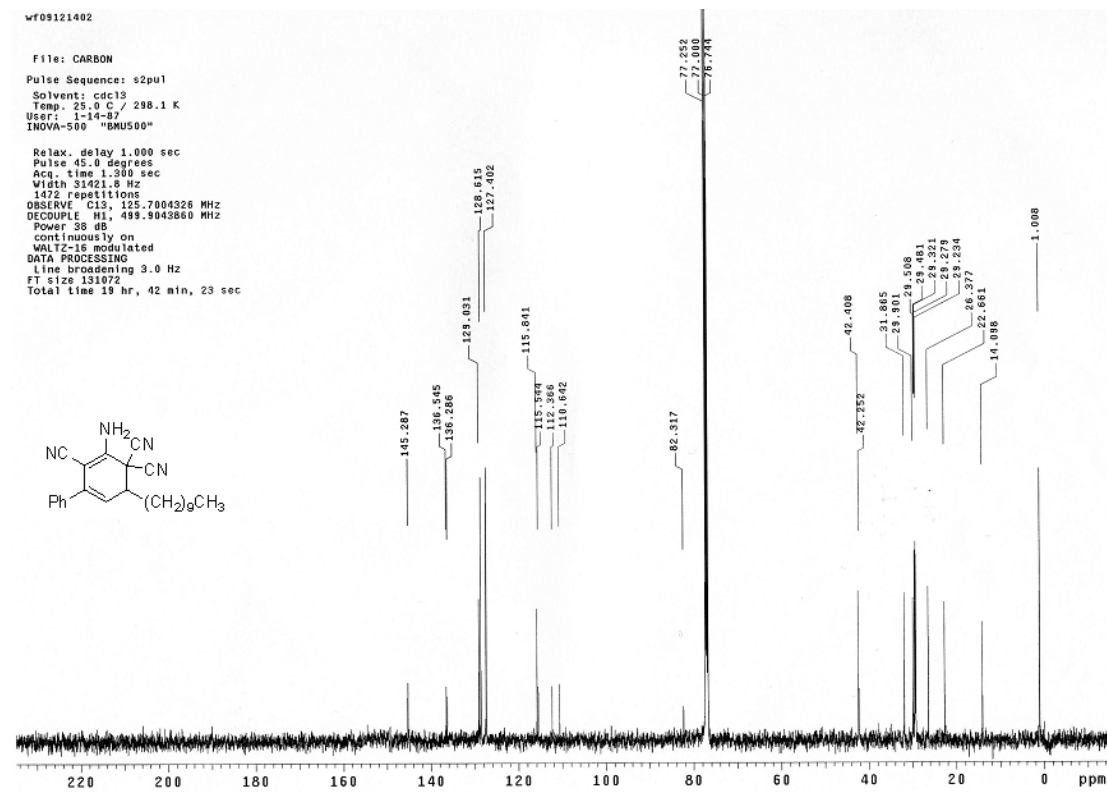
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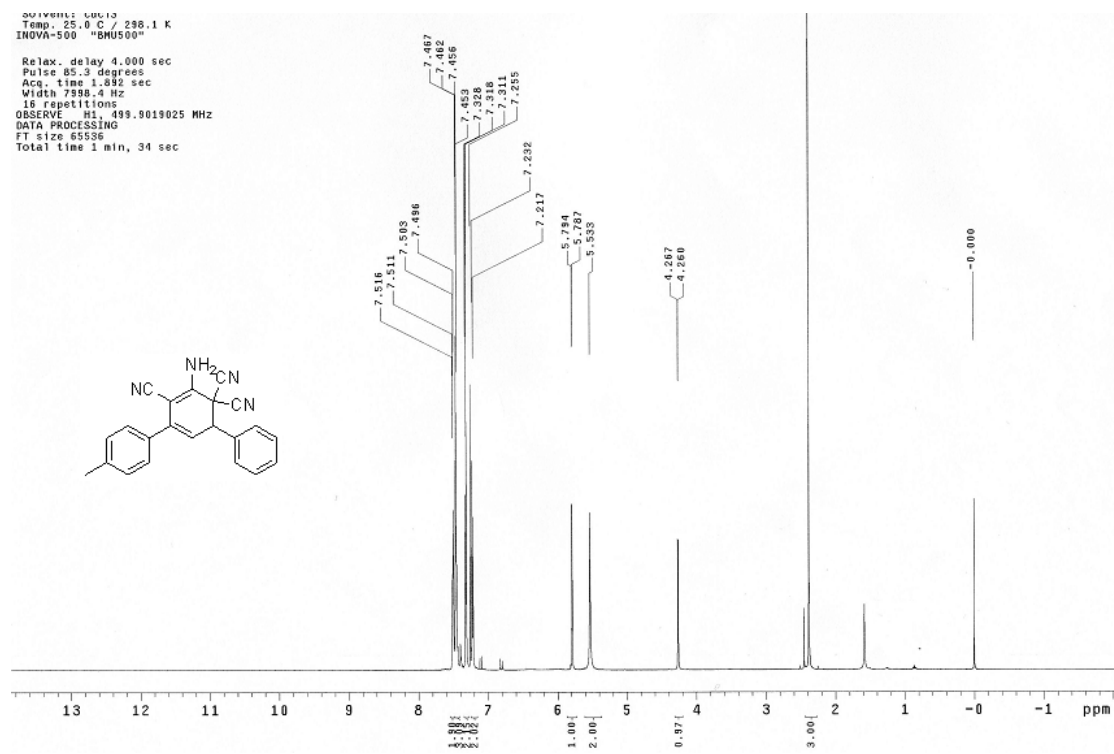


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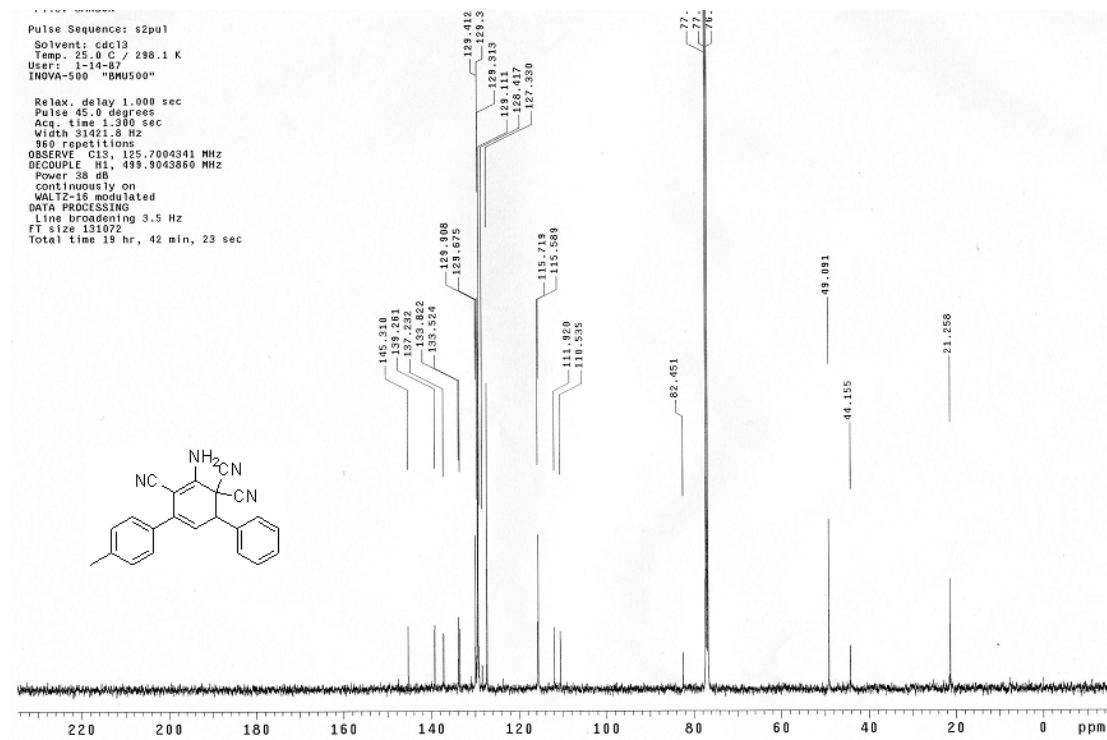




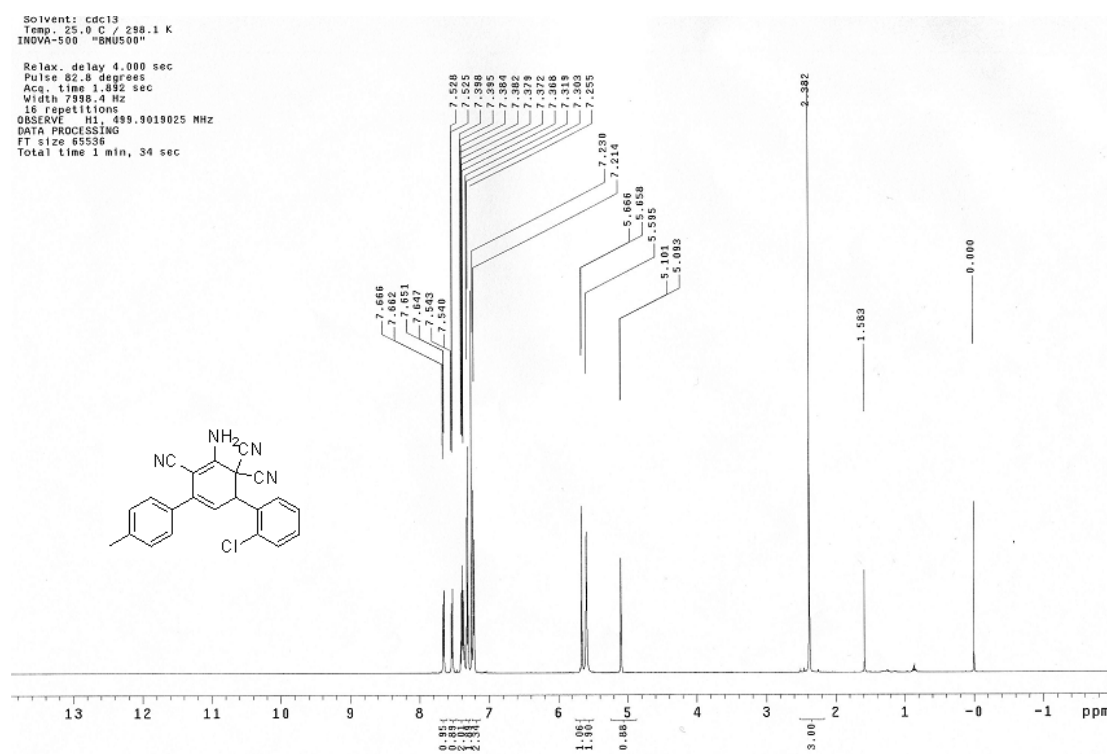
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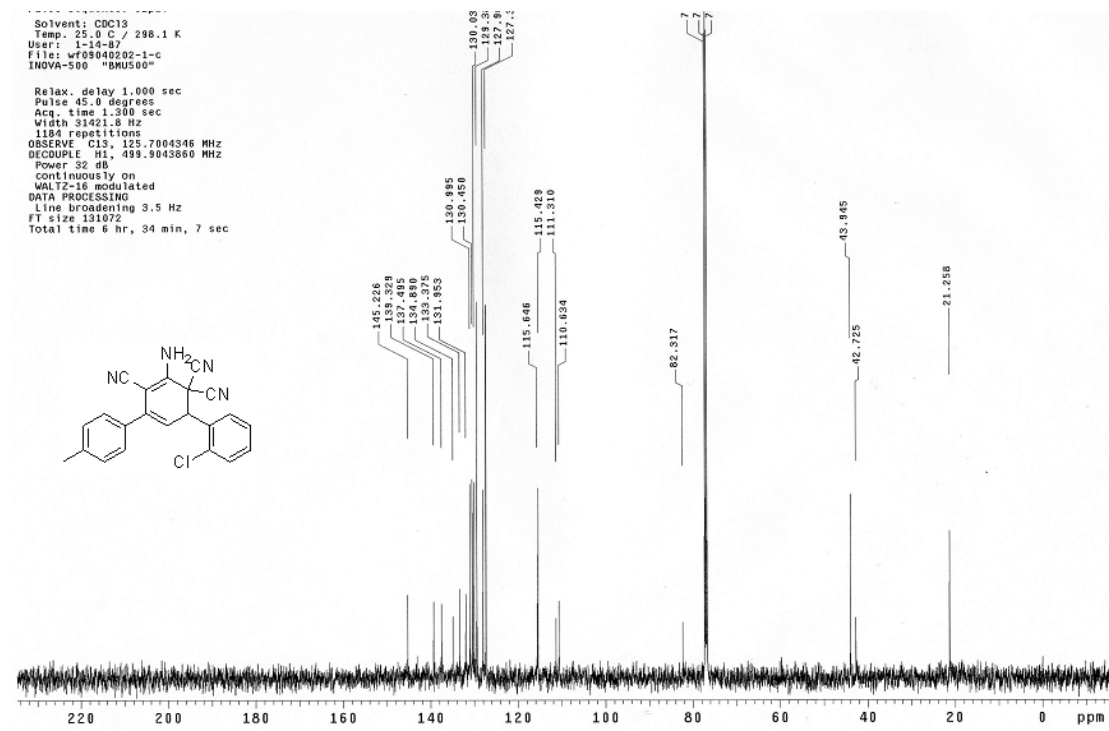


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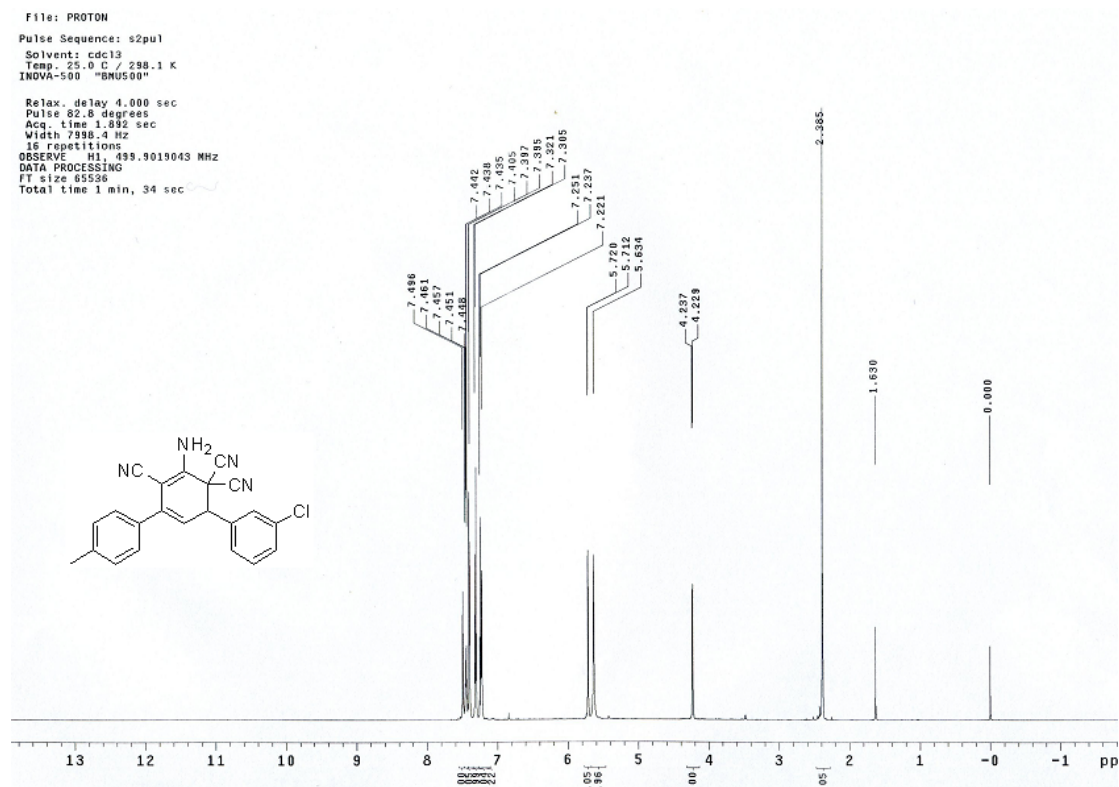


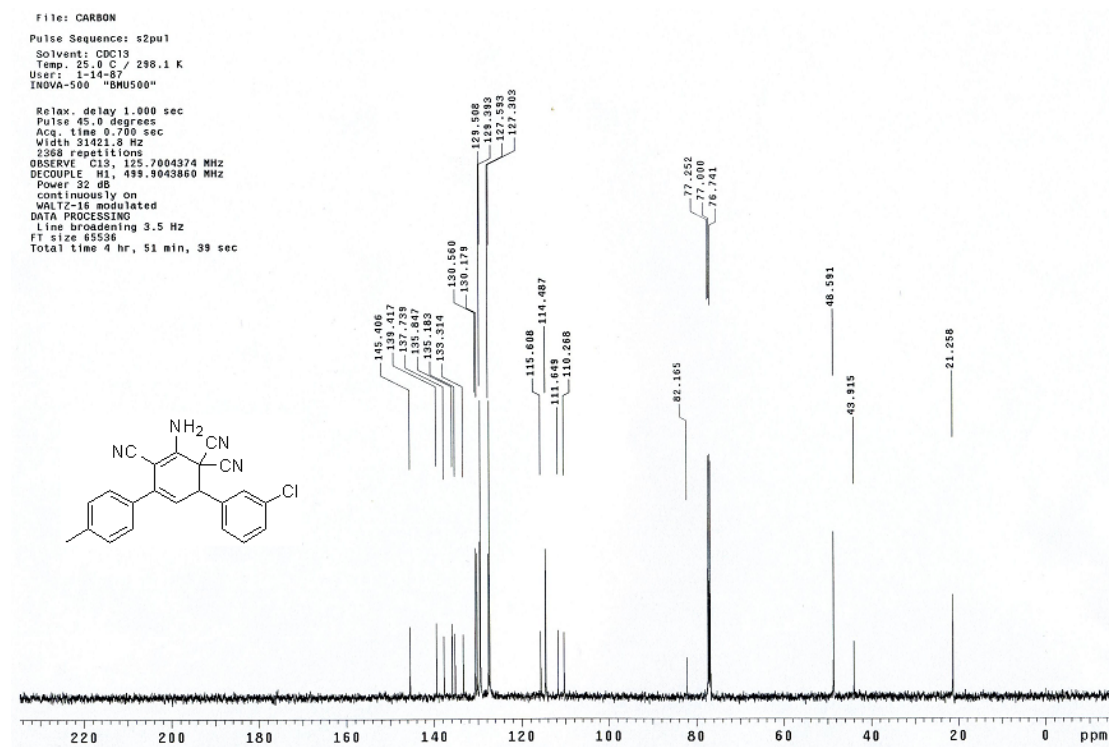
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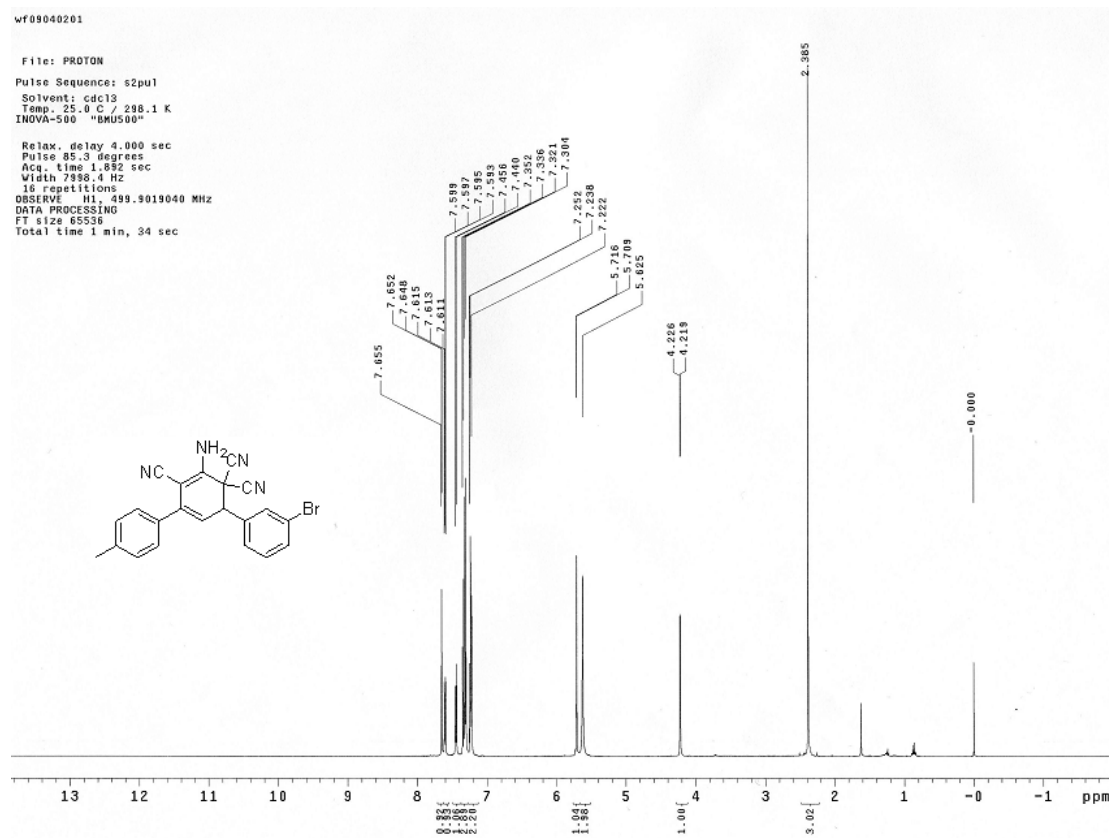


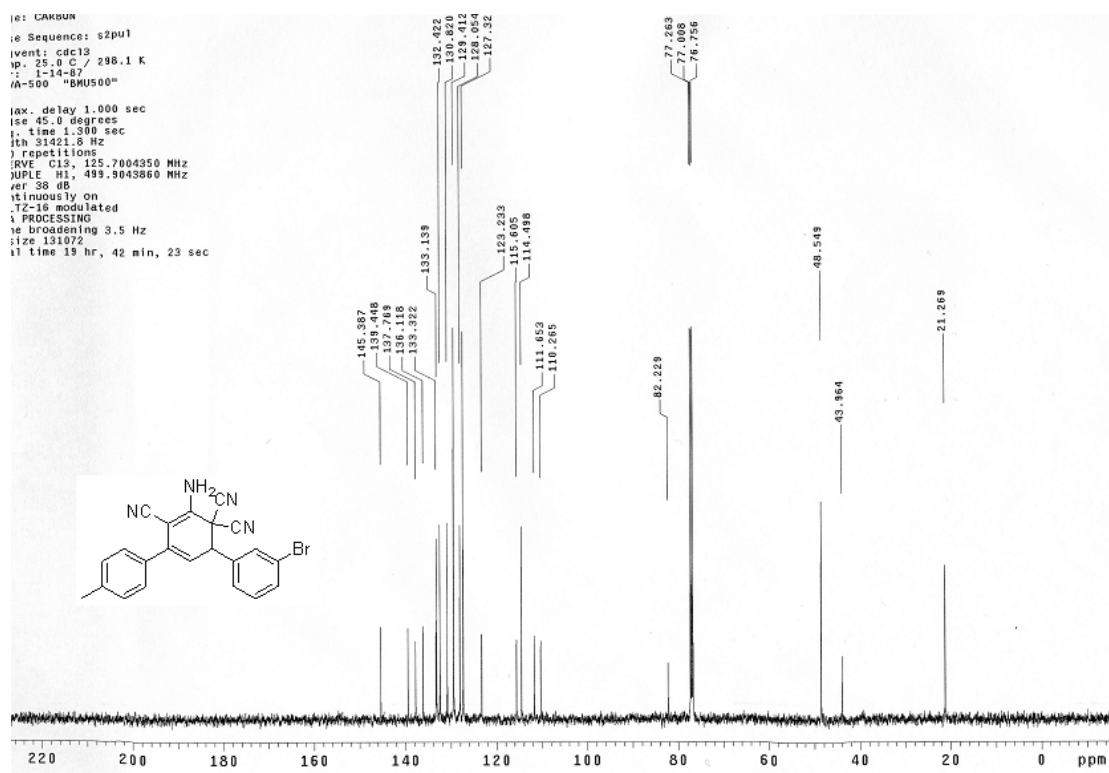
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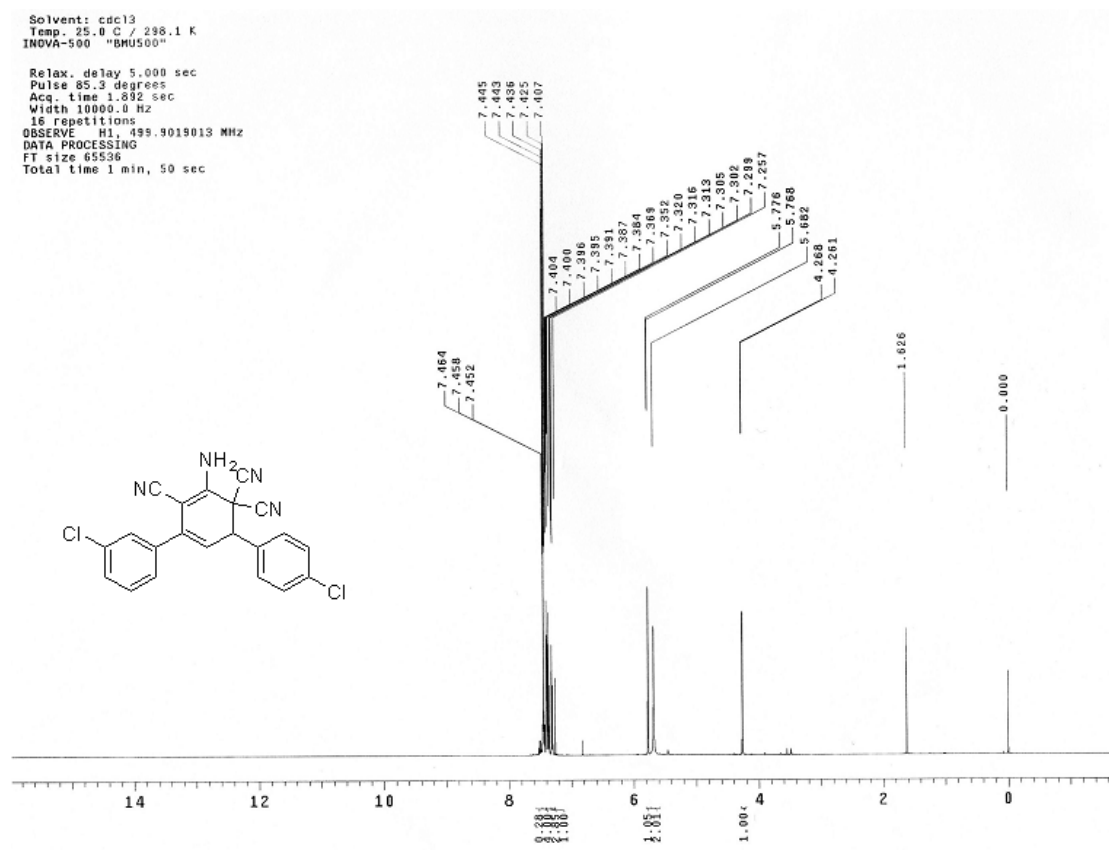


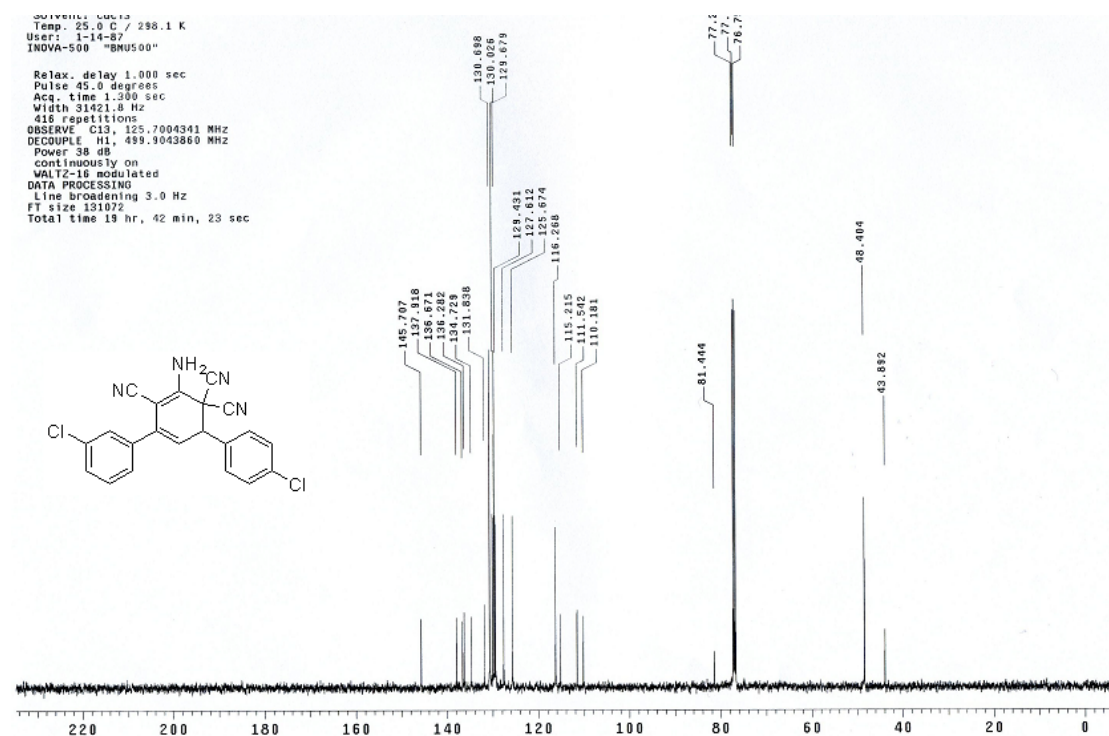
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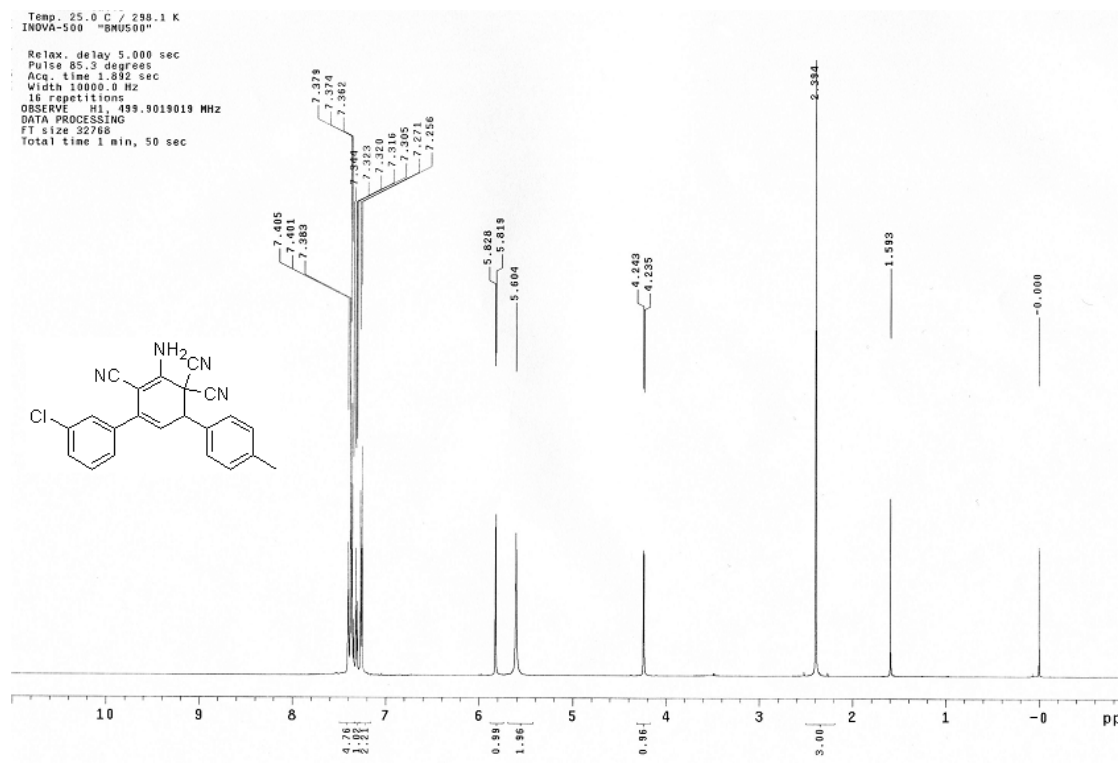


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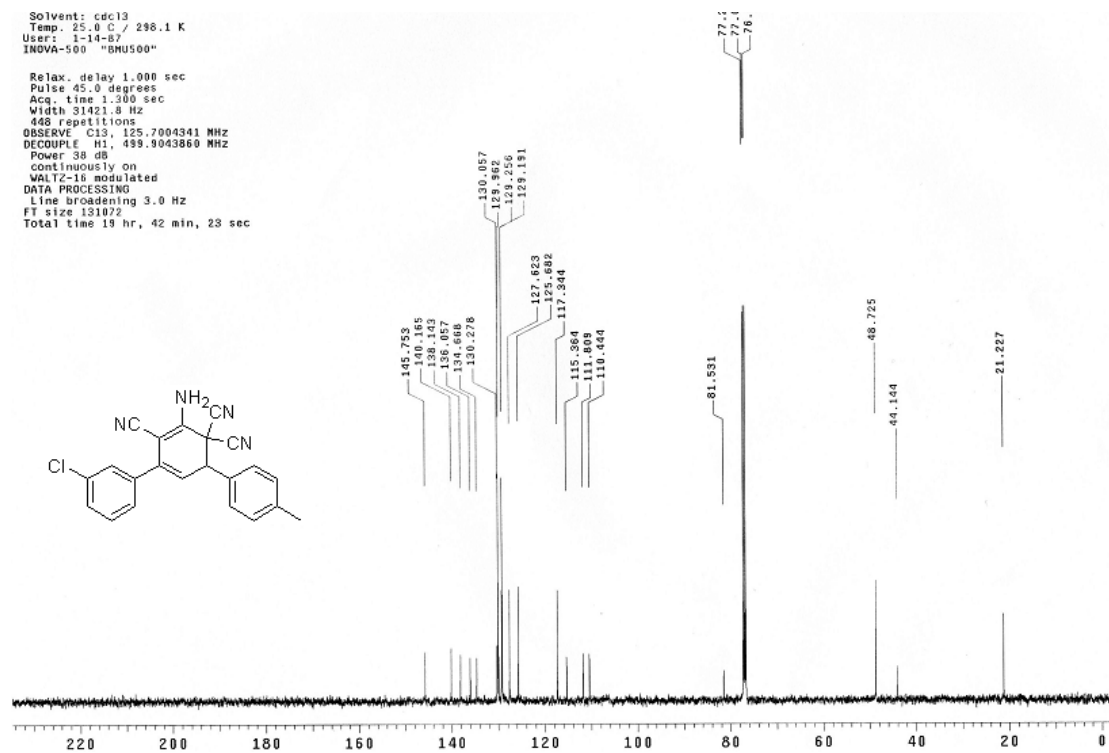




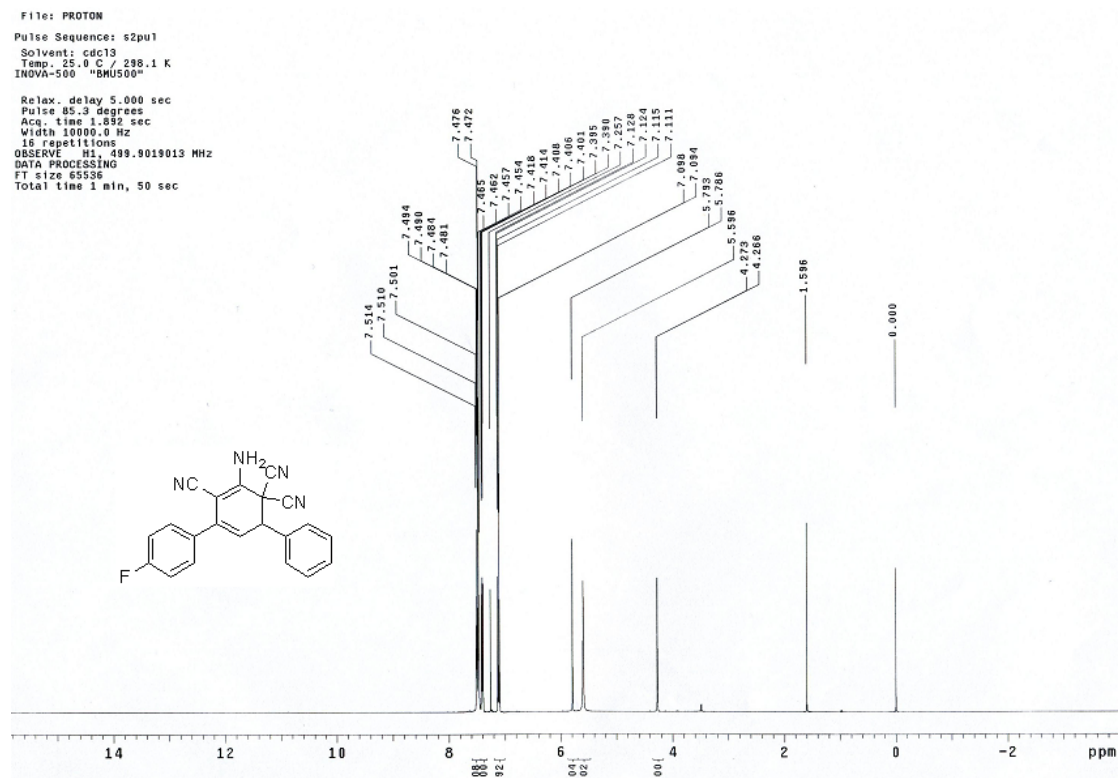
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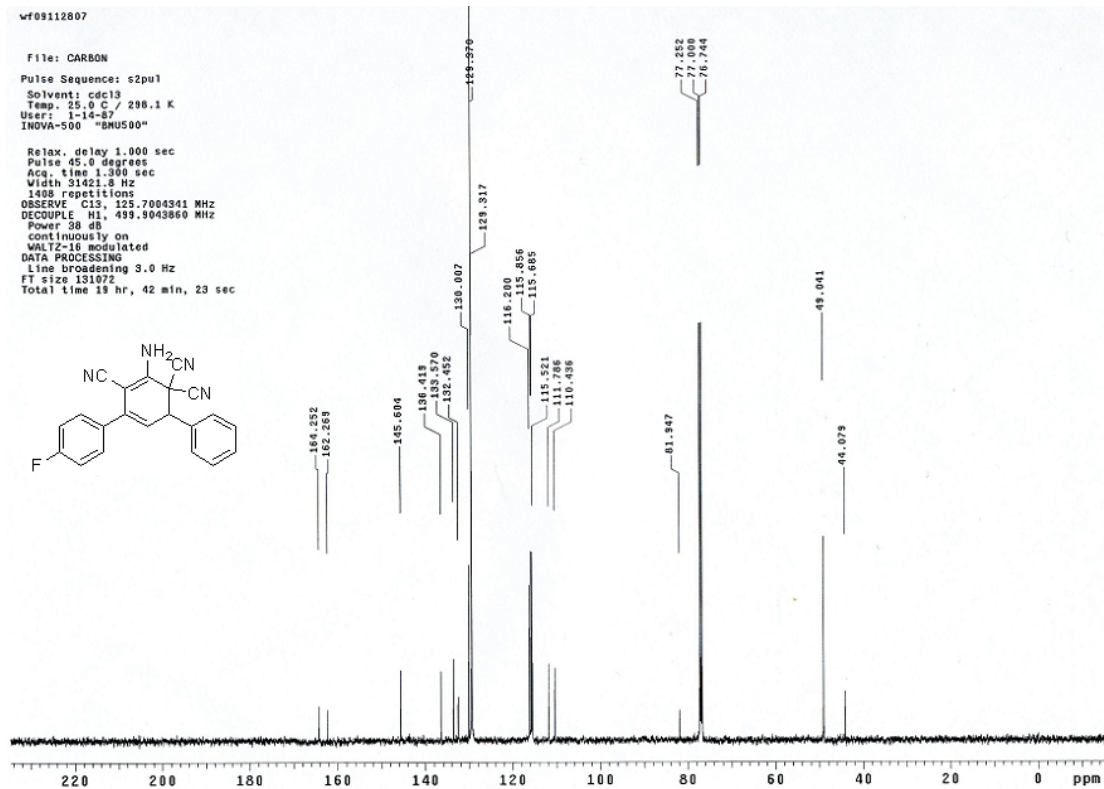


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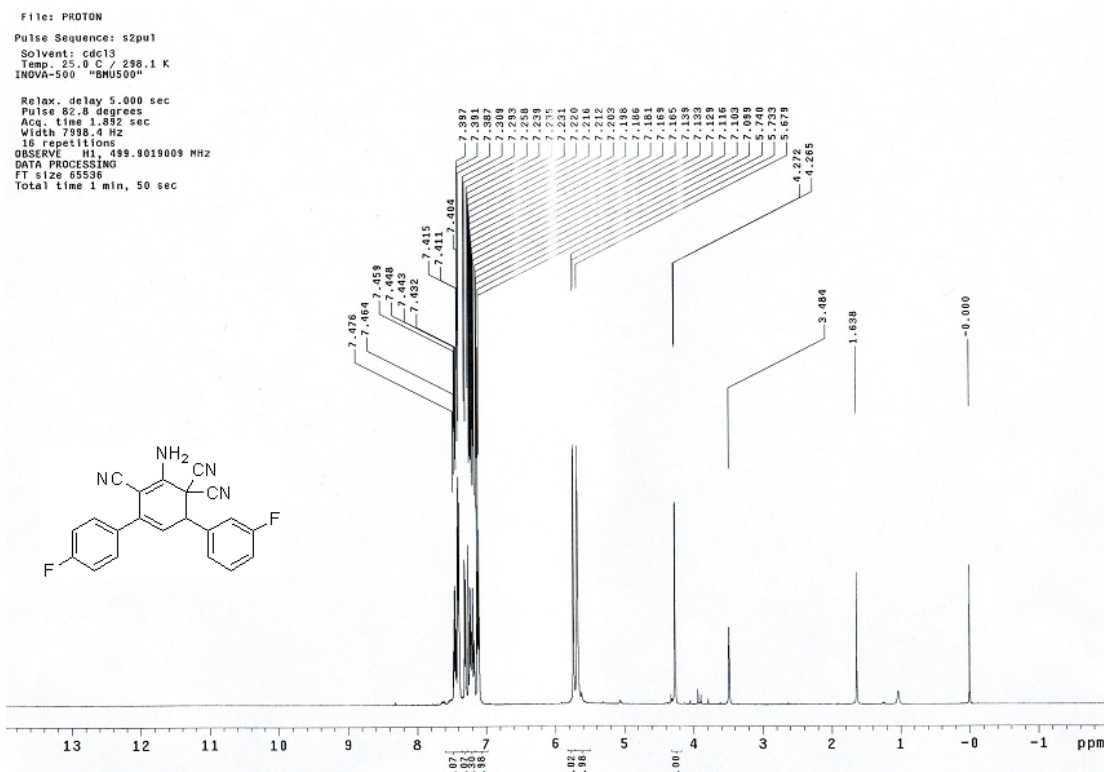


31





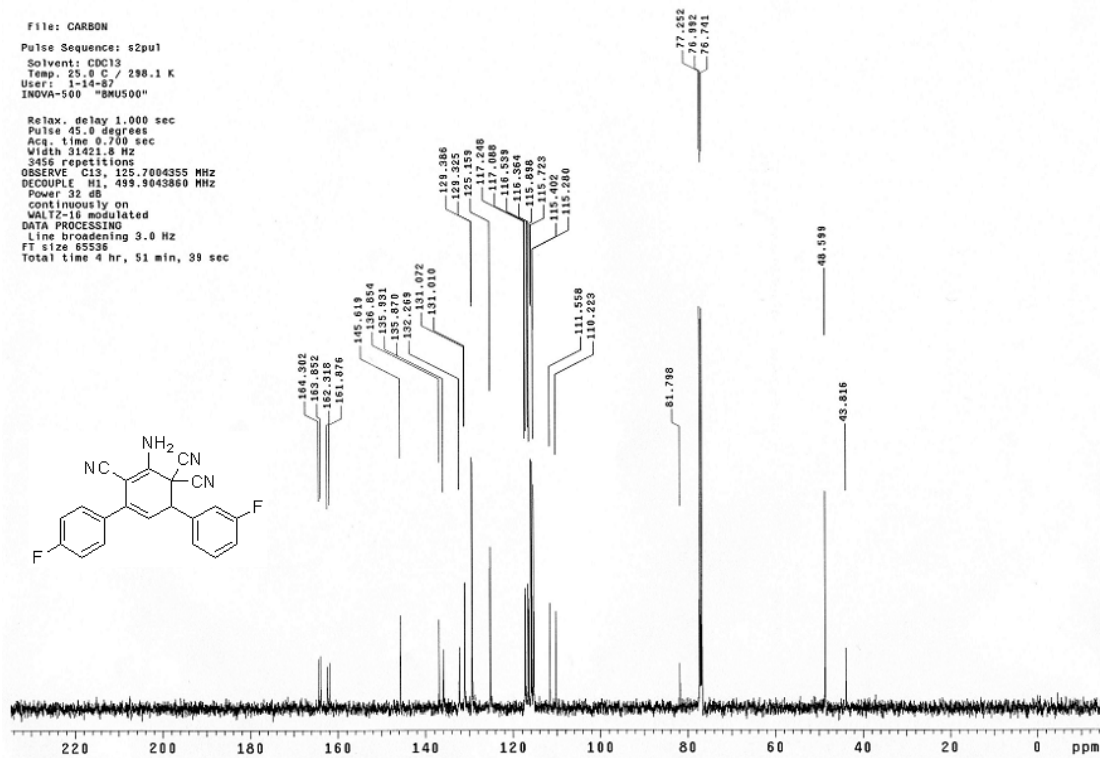
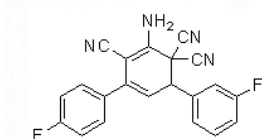
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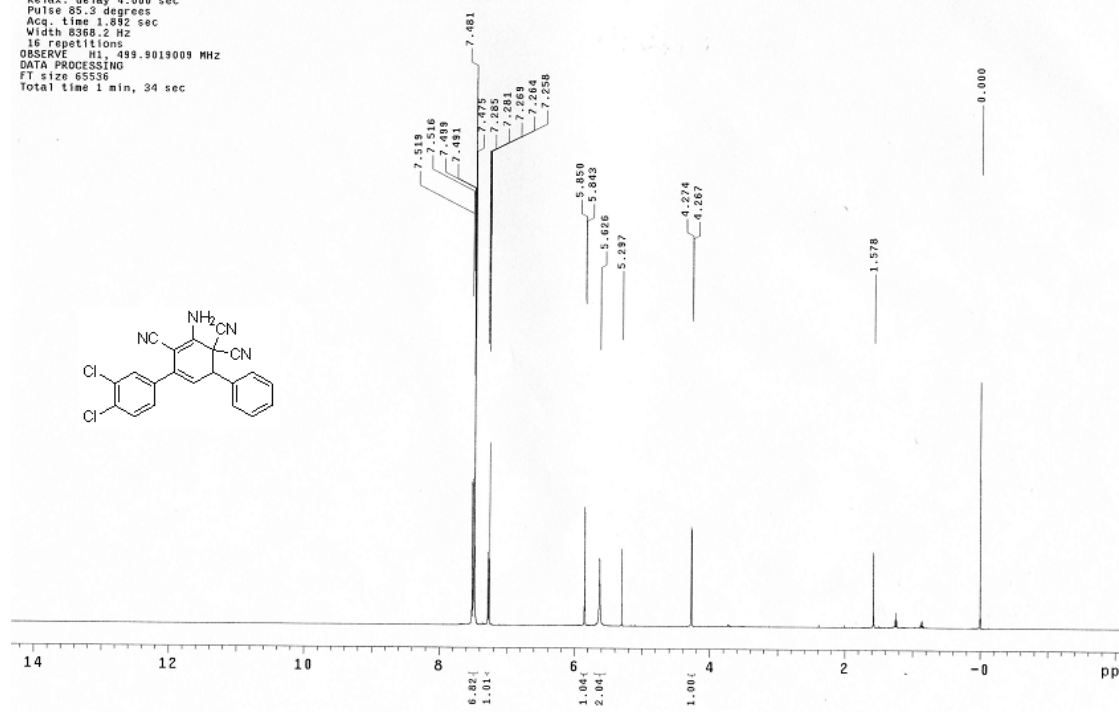
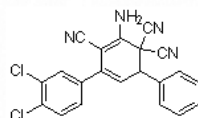
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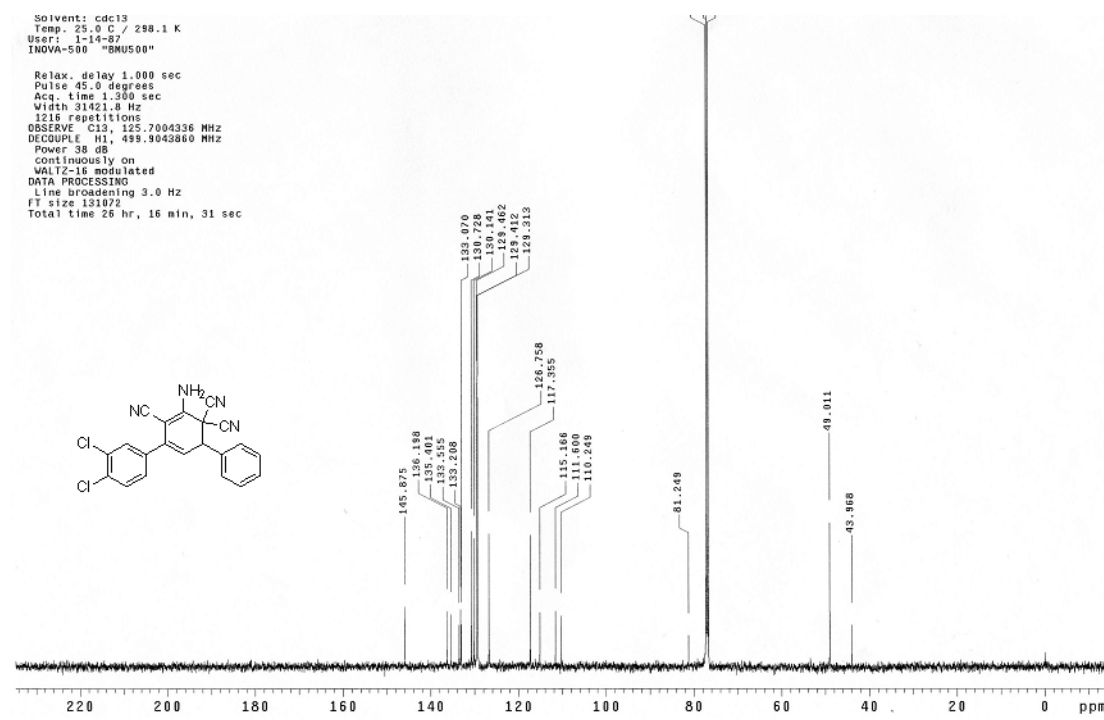
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 User: 1-14-07
 INOVA-500 "8MUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 0.700 sec
 Width 31421.8 Hz
 3456 repetitions
 OBSERVE C13, 125.7004355 MHz
 DECOUPLE H1, 499.9043869 MHz
 Power 32 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 3.0 Hz
 FT size 65536
 Total time 4 hr, 51 min, 39 sec



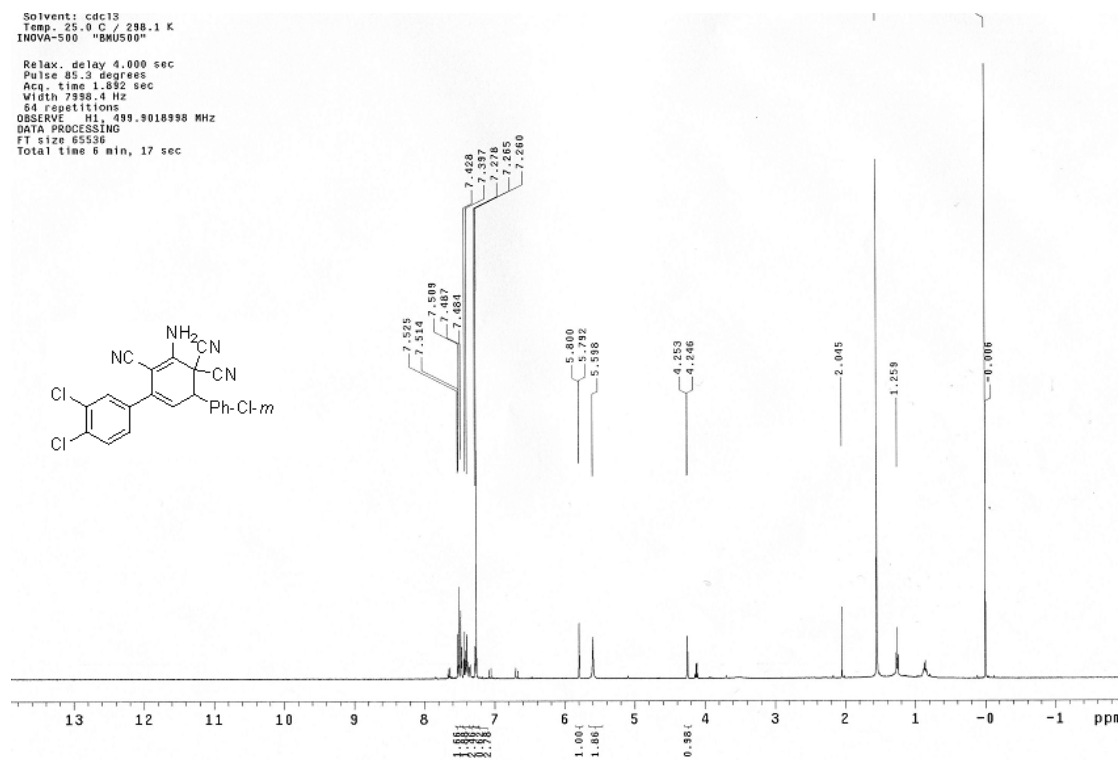
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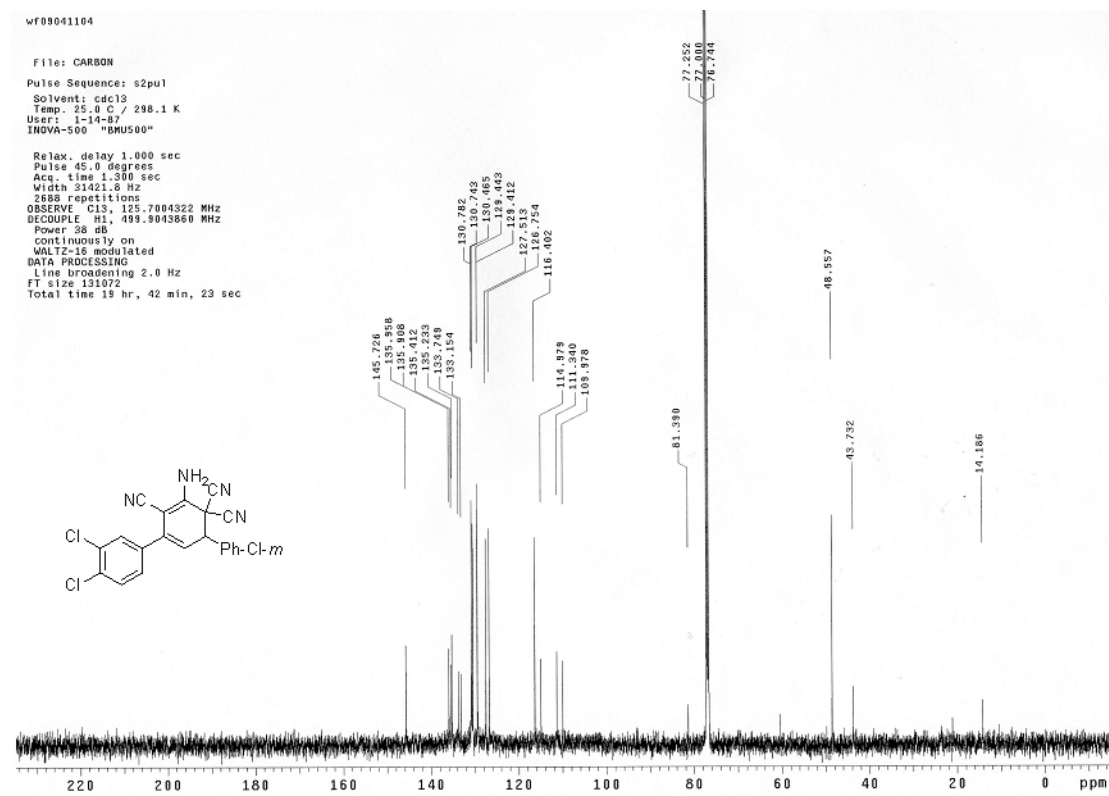
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 16 repetitions
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 DATA PROCESSING
 FT size 65536
 Total time 1 min, 34 sec



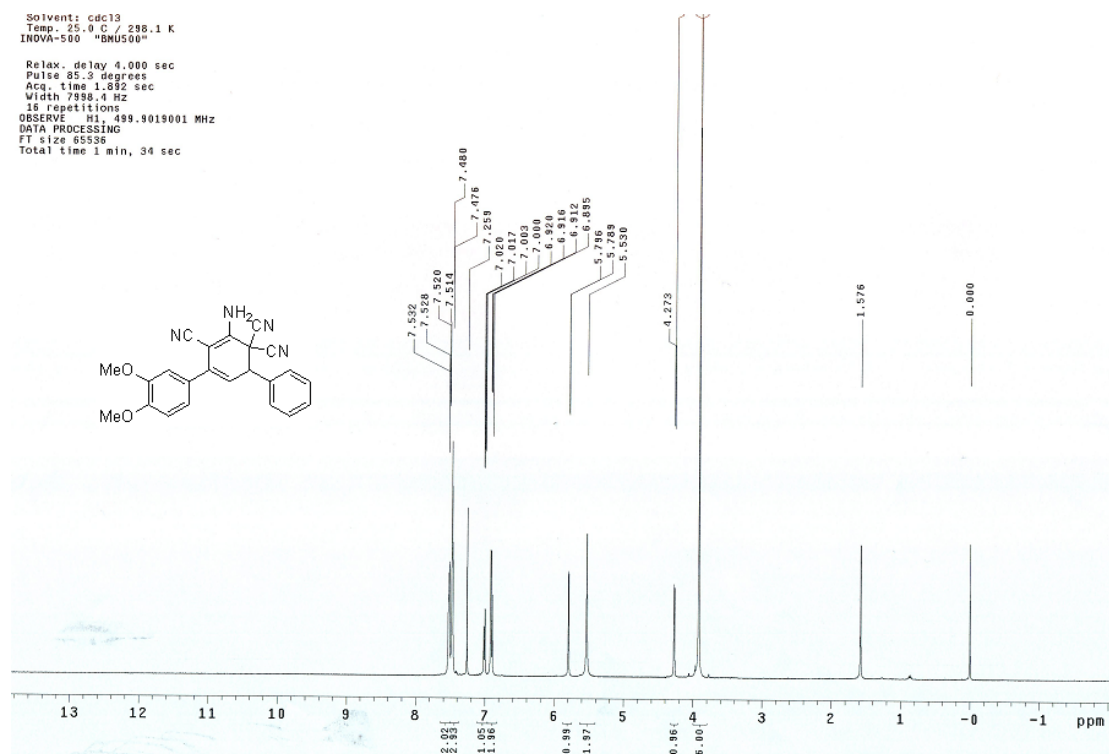


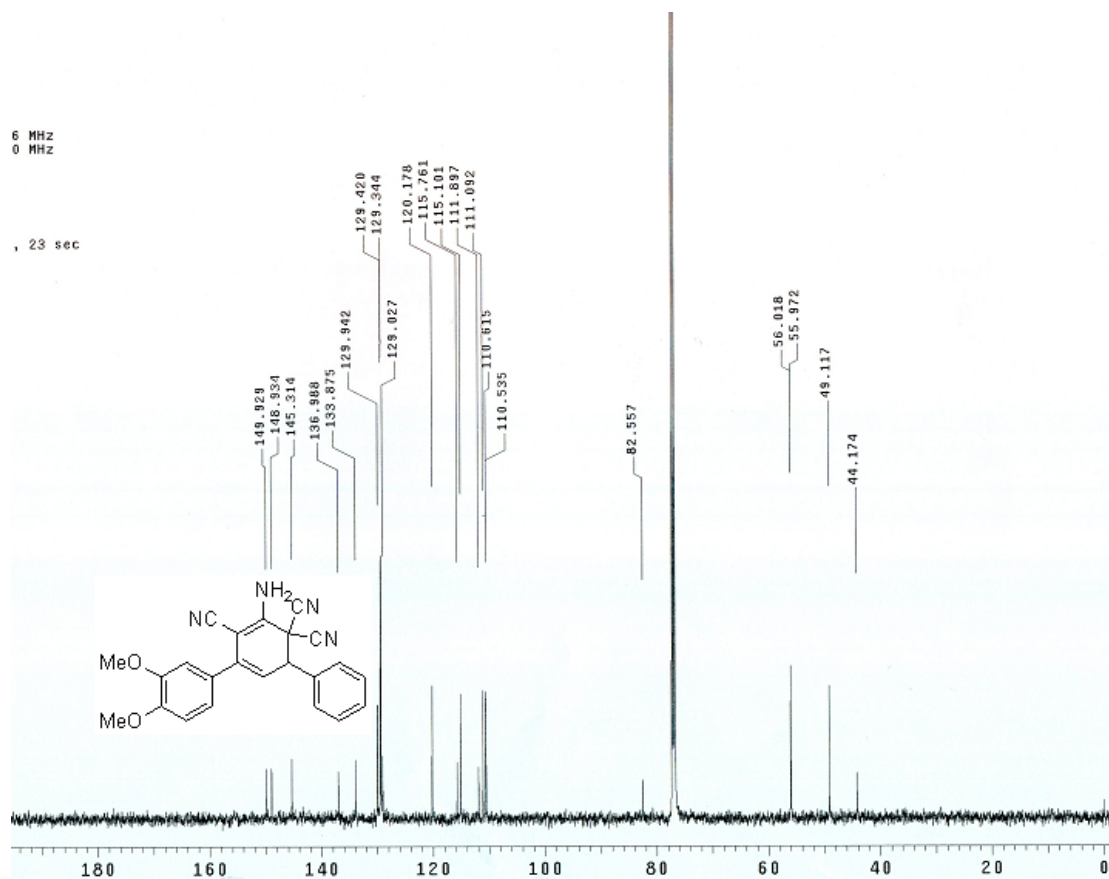
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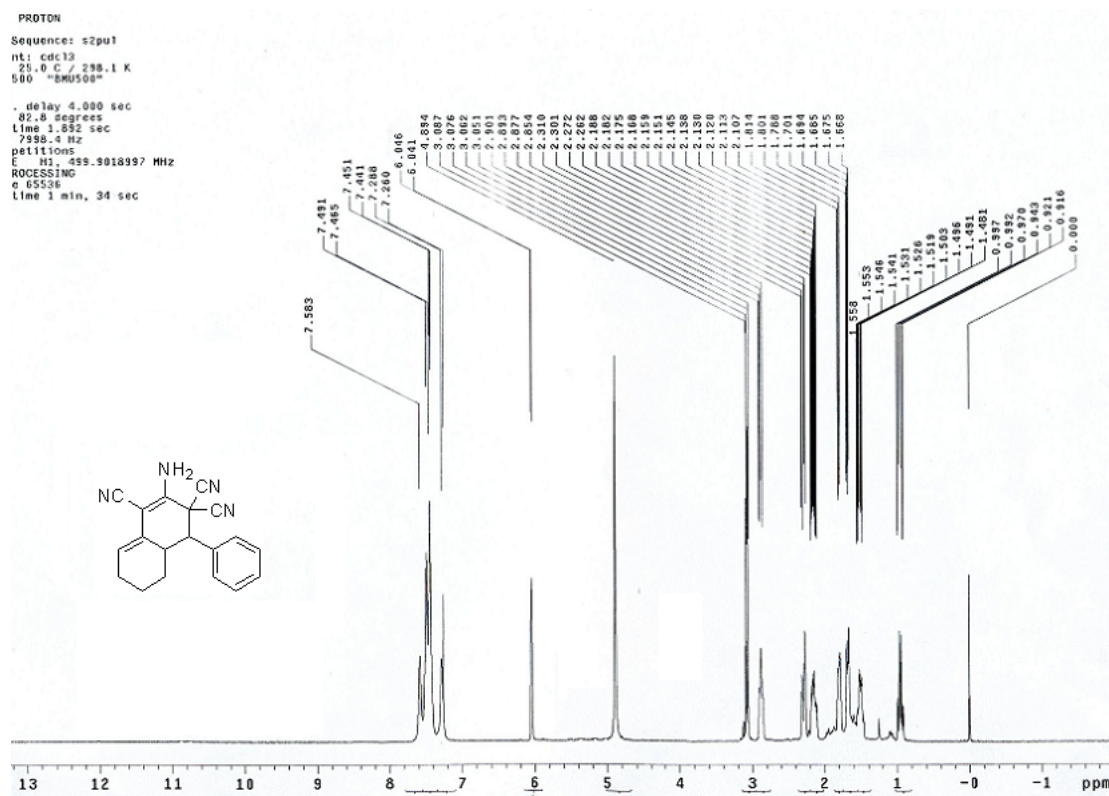


3p





7a



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wf09121309

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Solvent: CDCl3
Temp: 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMUS00"

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Pulse: 45.0 degrees
Acq. time 0.700 sec
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2888 repetitions
OBSERVE C13, 125.7004336 MHz
DECOUPLE H1, 499.9043860 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.0 Hz
FT size 65536
Total time 4 hr, 51 min, 39 sec

