

Supporting Information

for

Diastereoselective Protonation on Radical Anions of Electron-Deficient Alkenes via Photoinduced Electron Transfer

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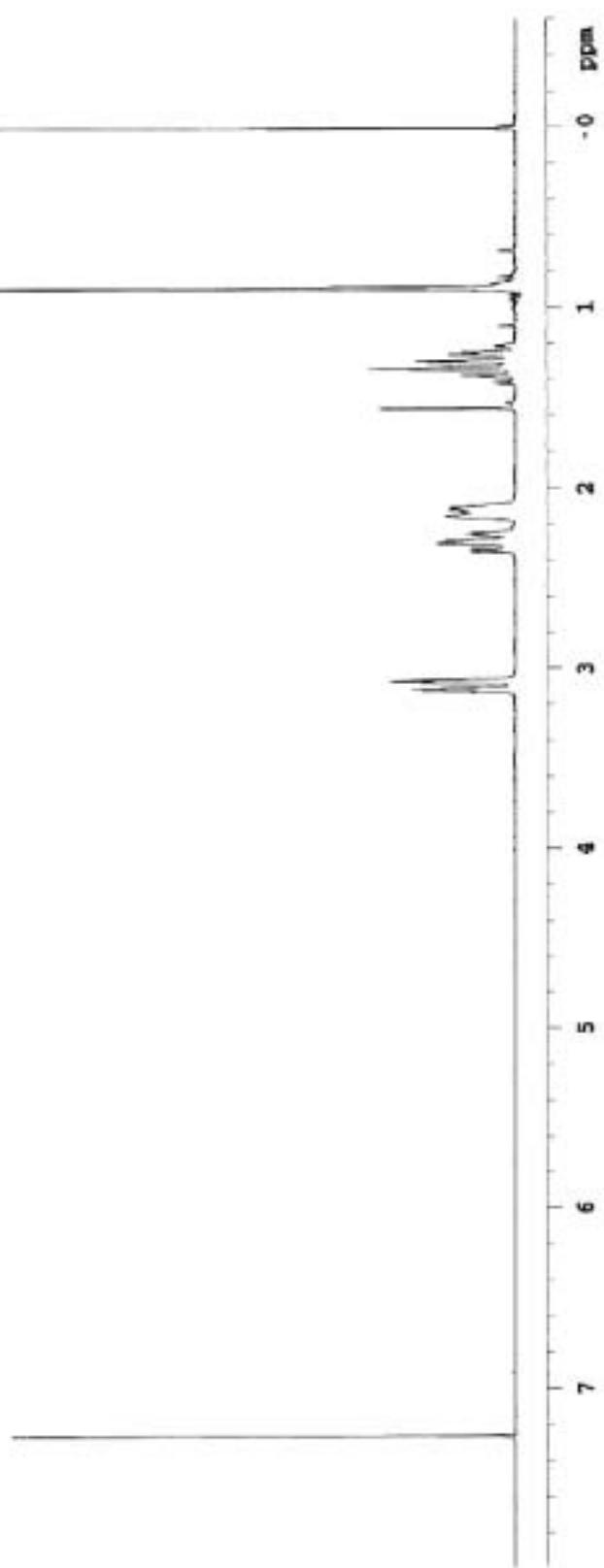
mizuno@chem.osakafu-u.ac.jp

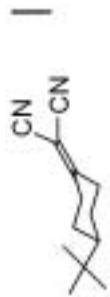
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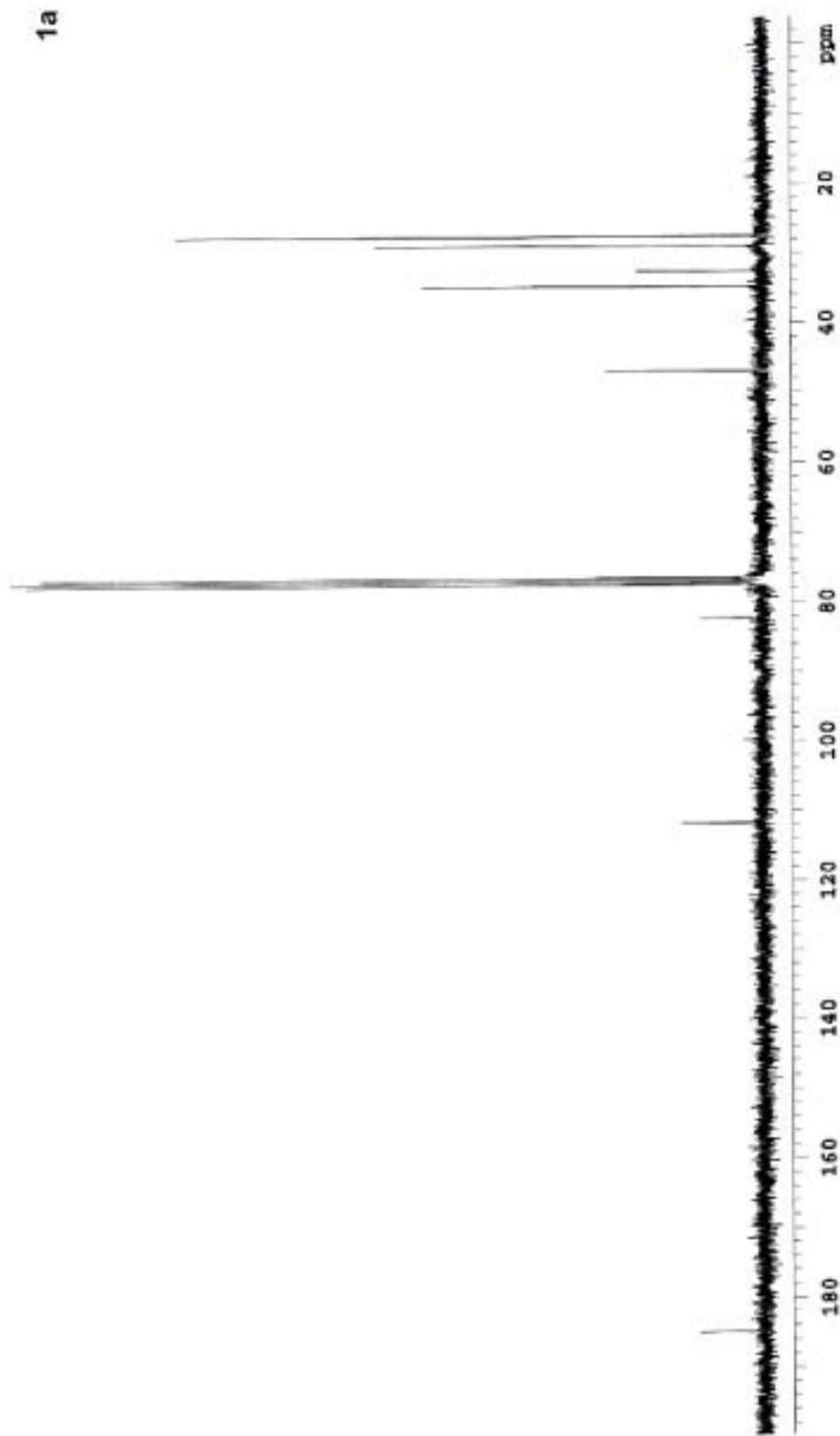


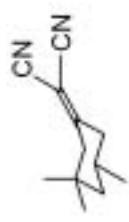
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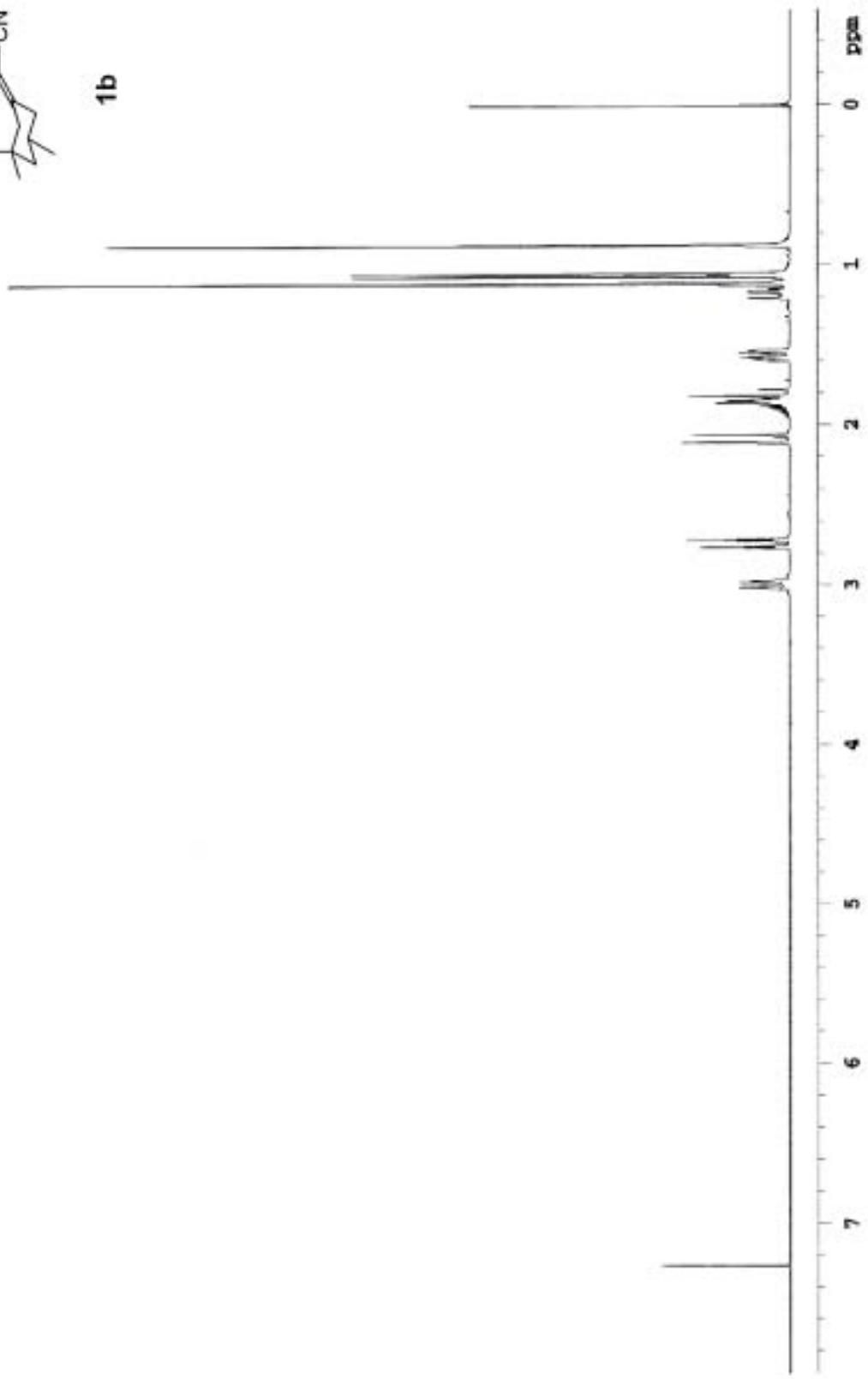


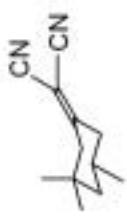
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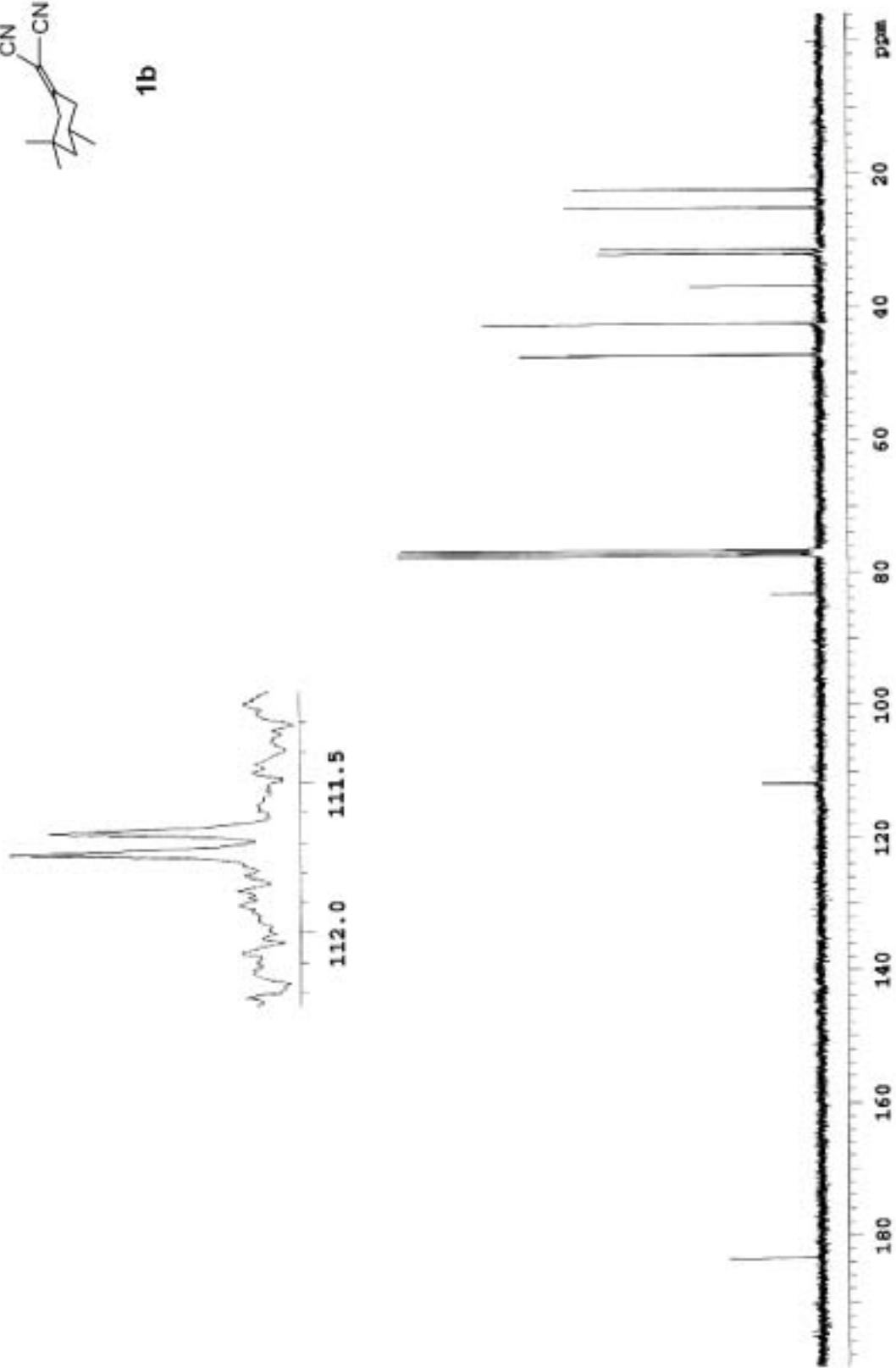


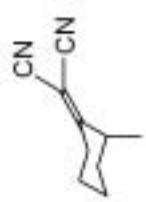
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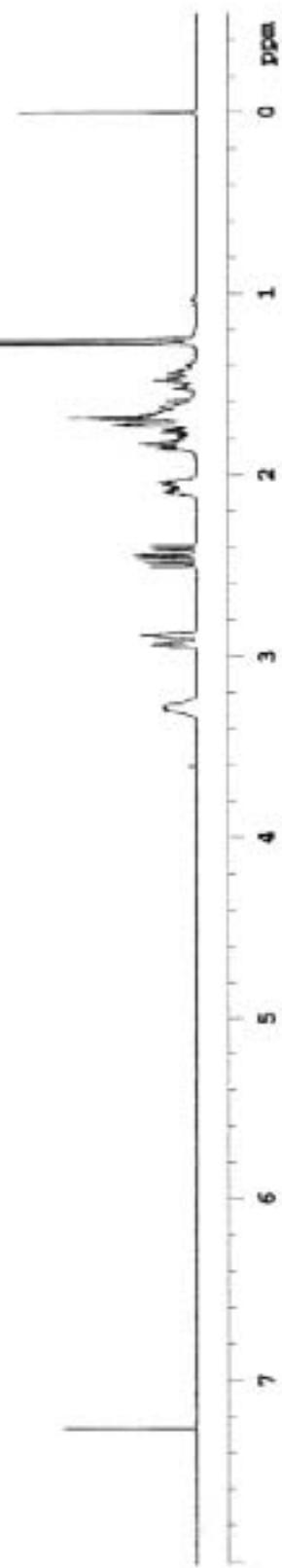


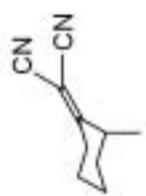
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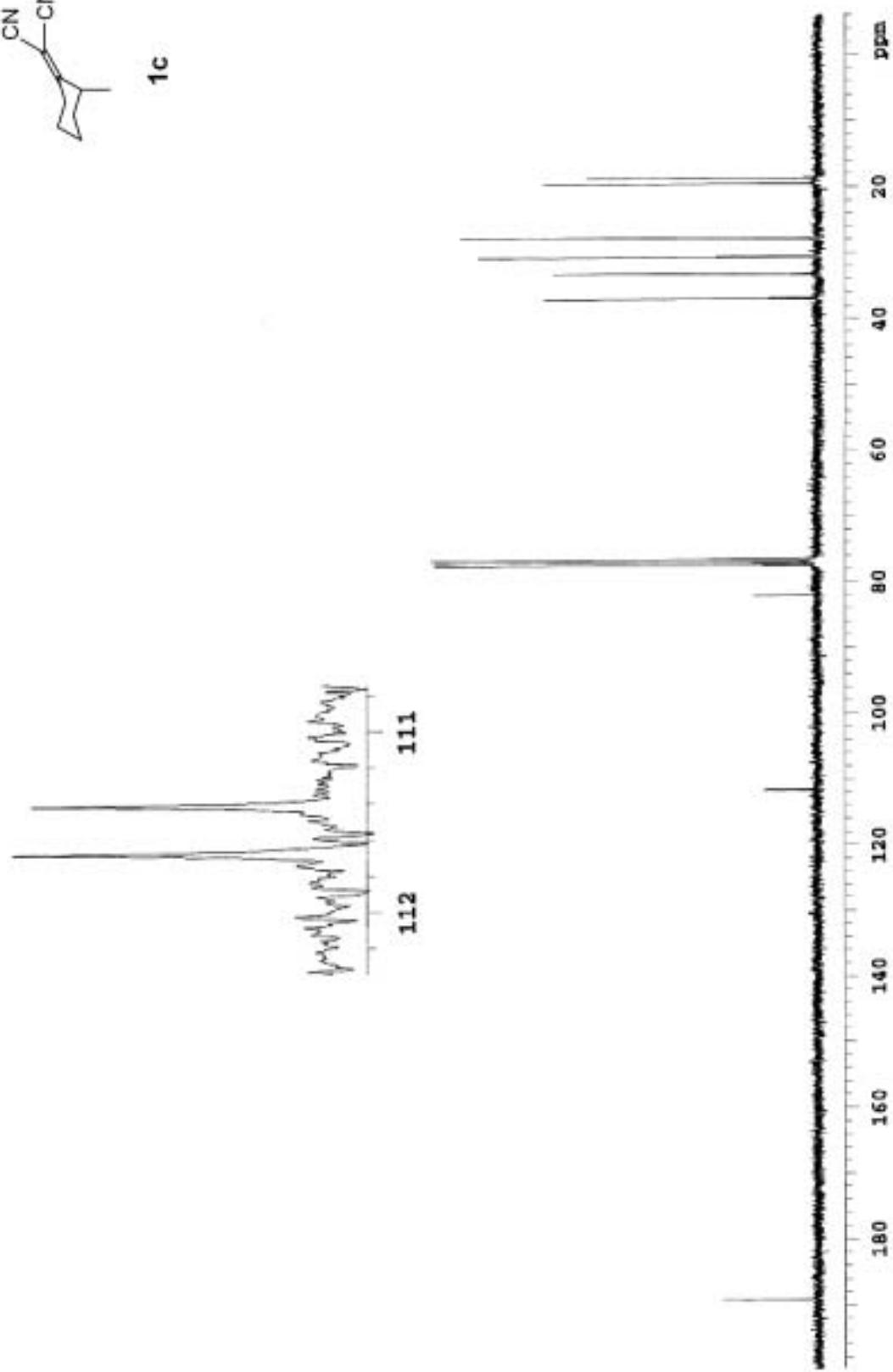


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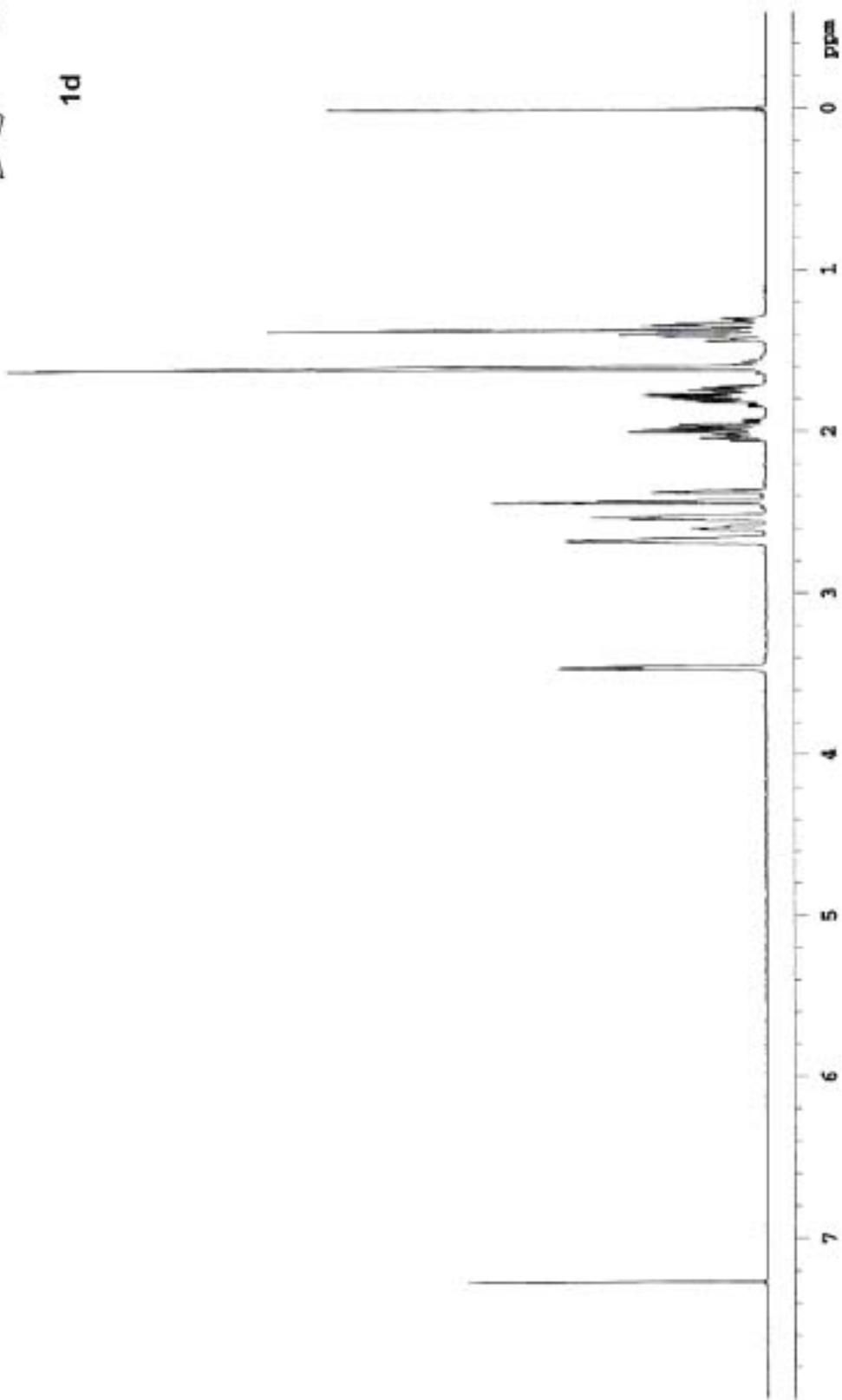


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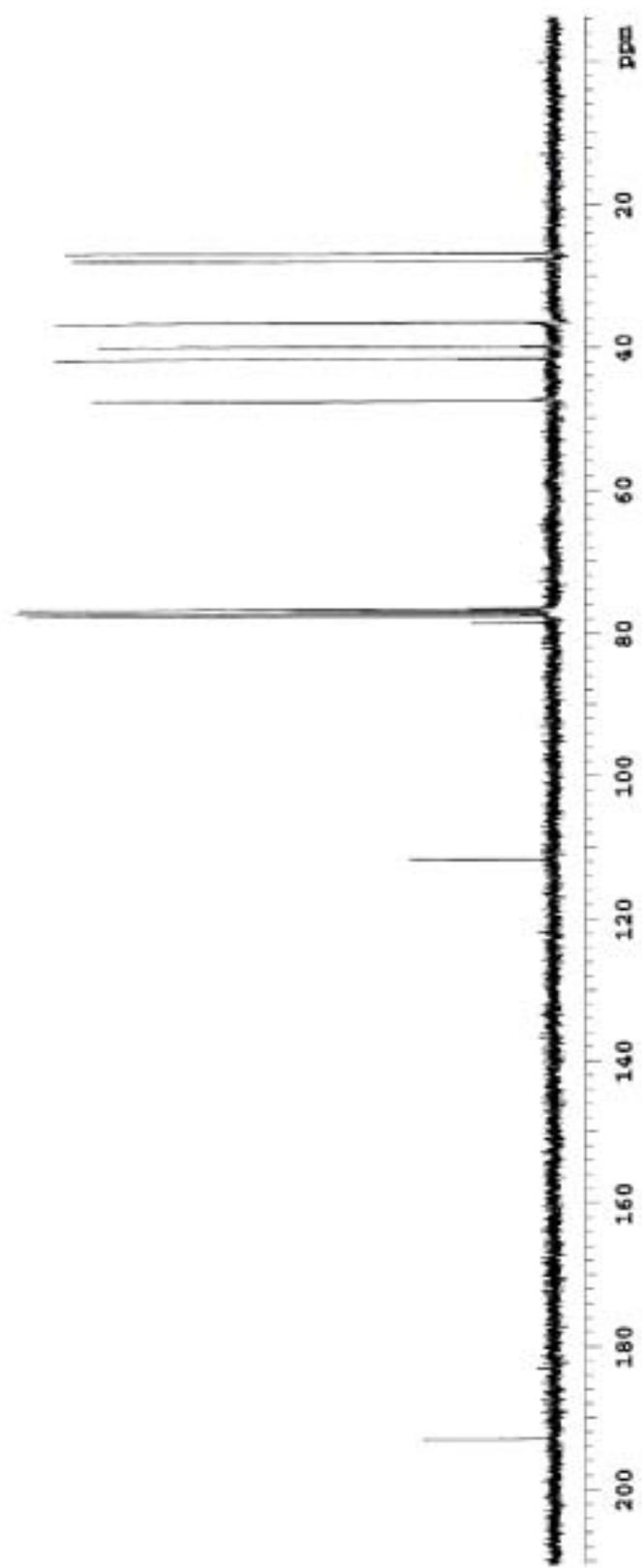


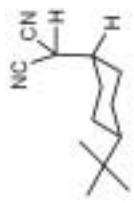
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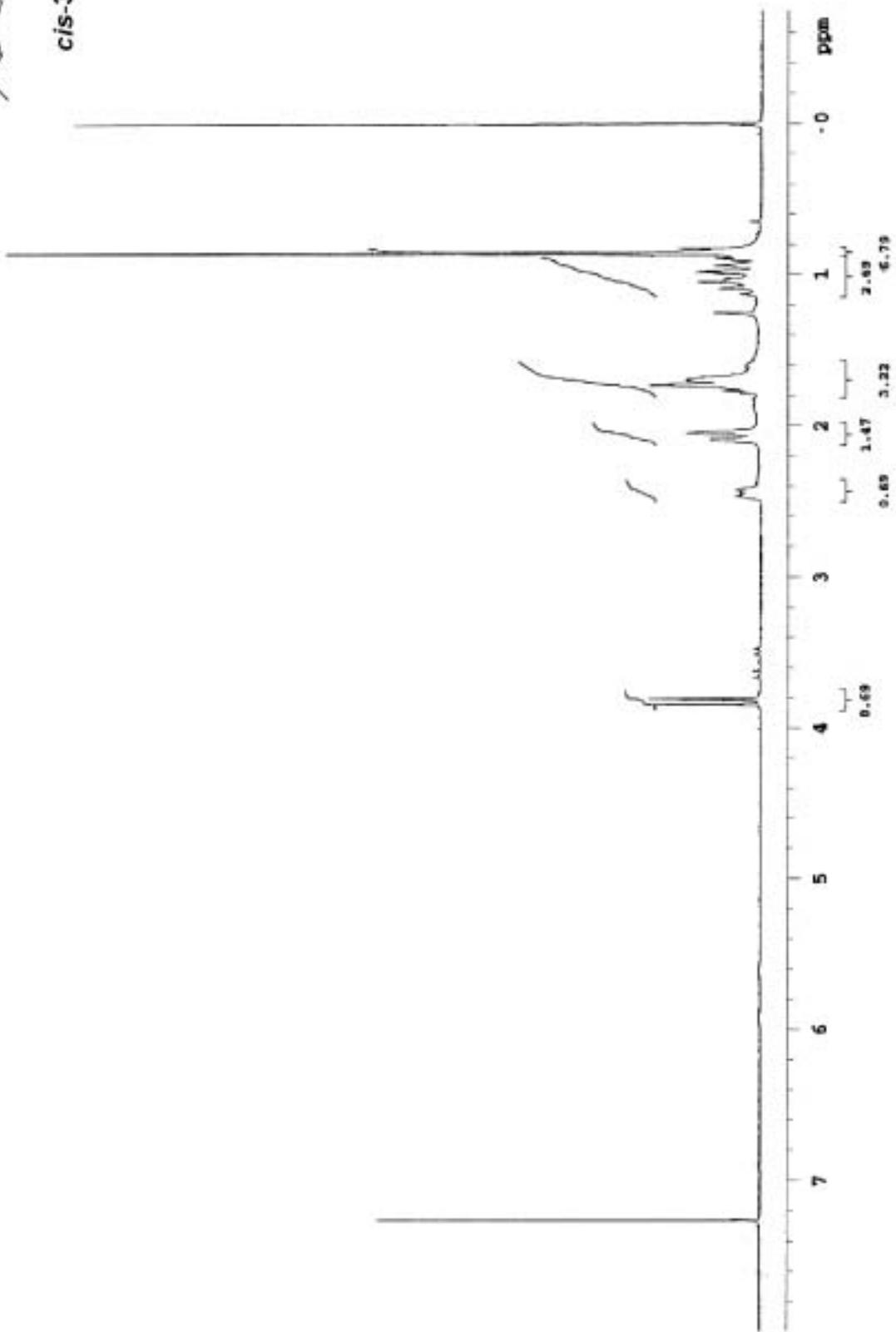


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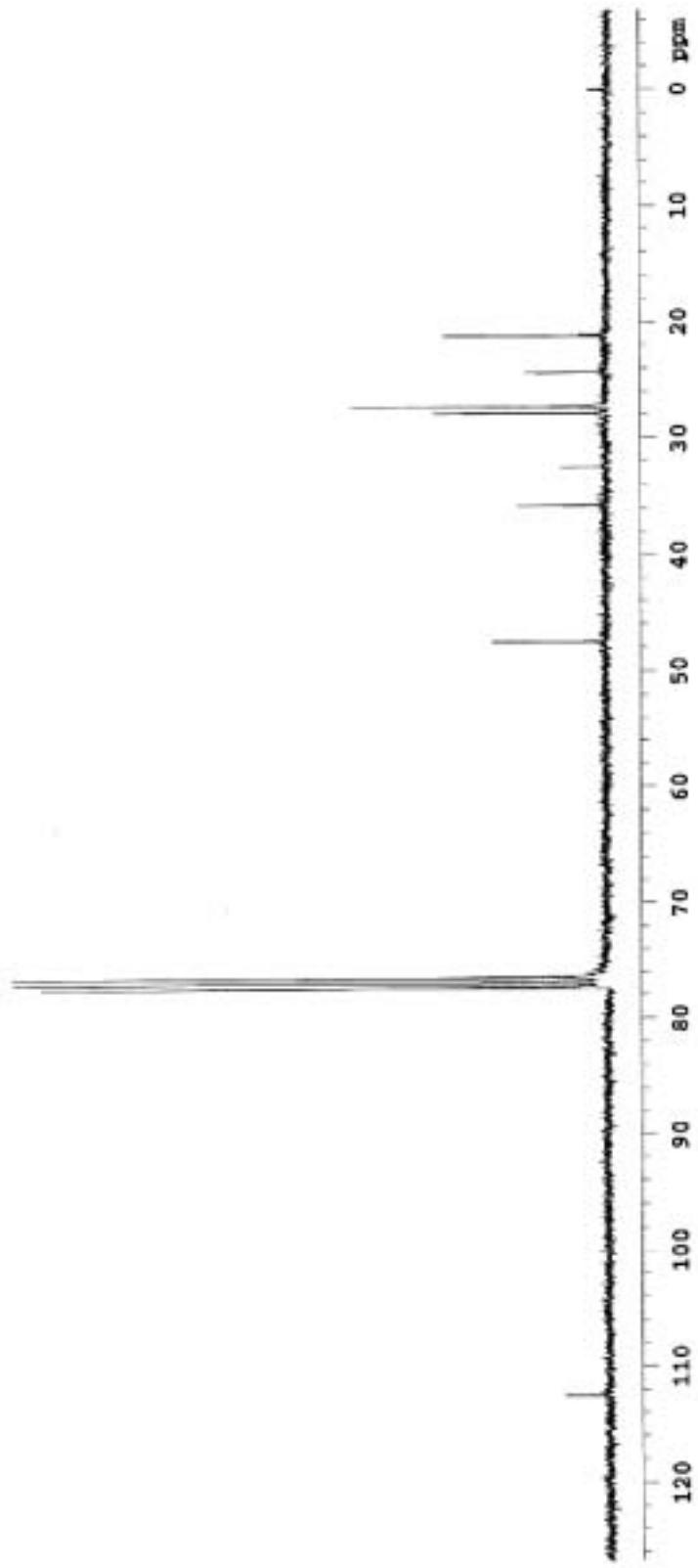


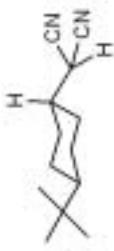
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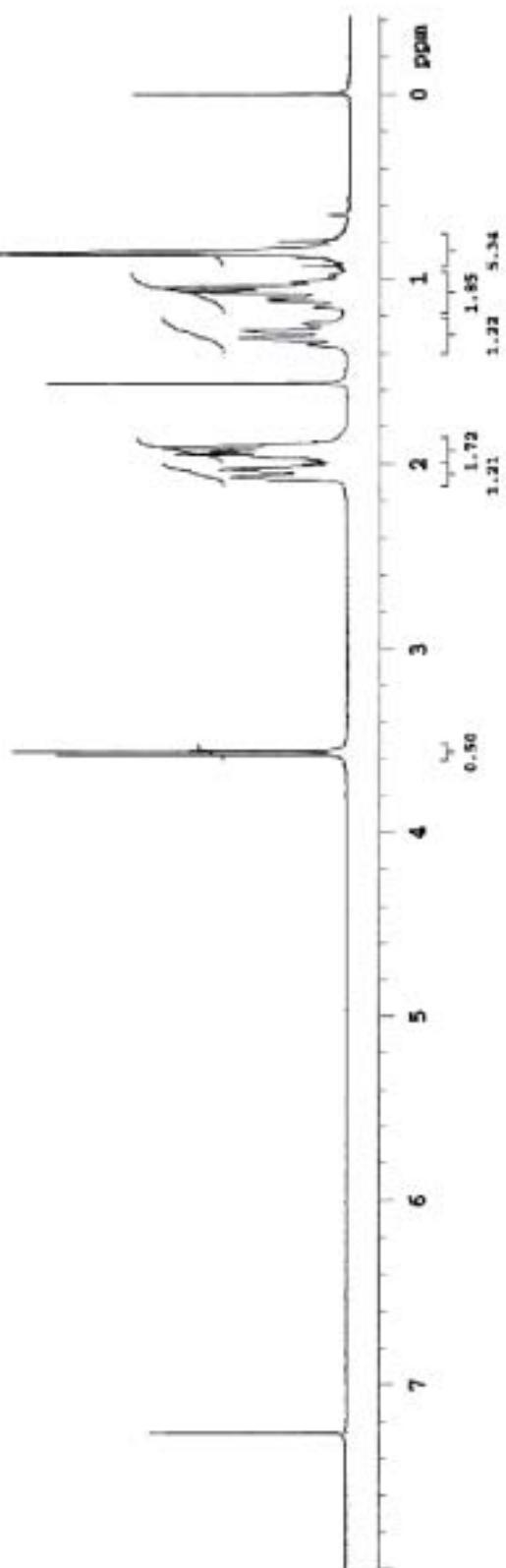


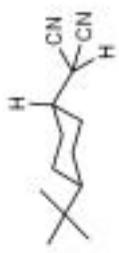
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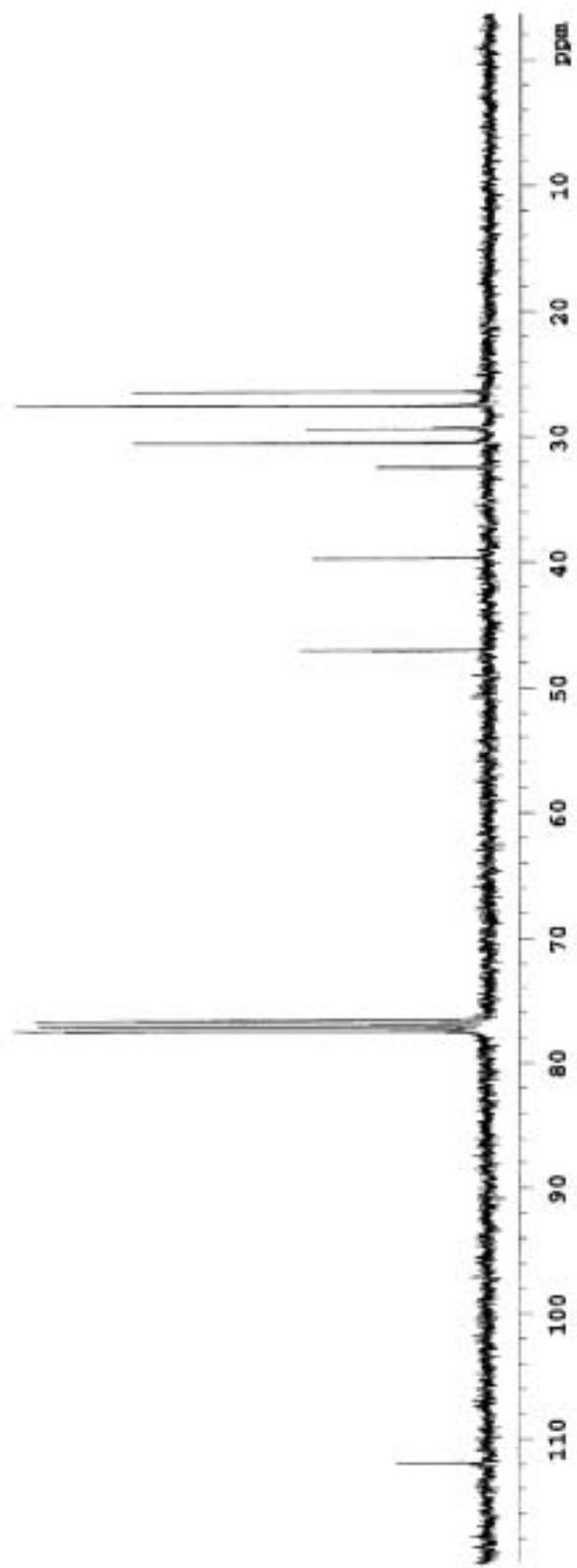


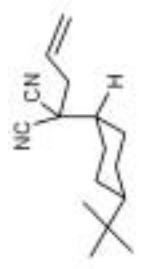
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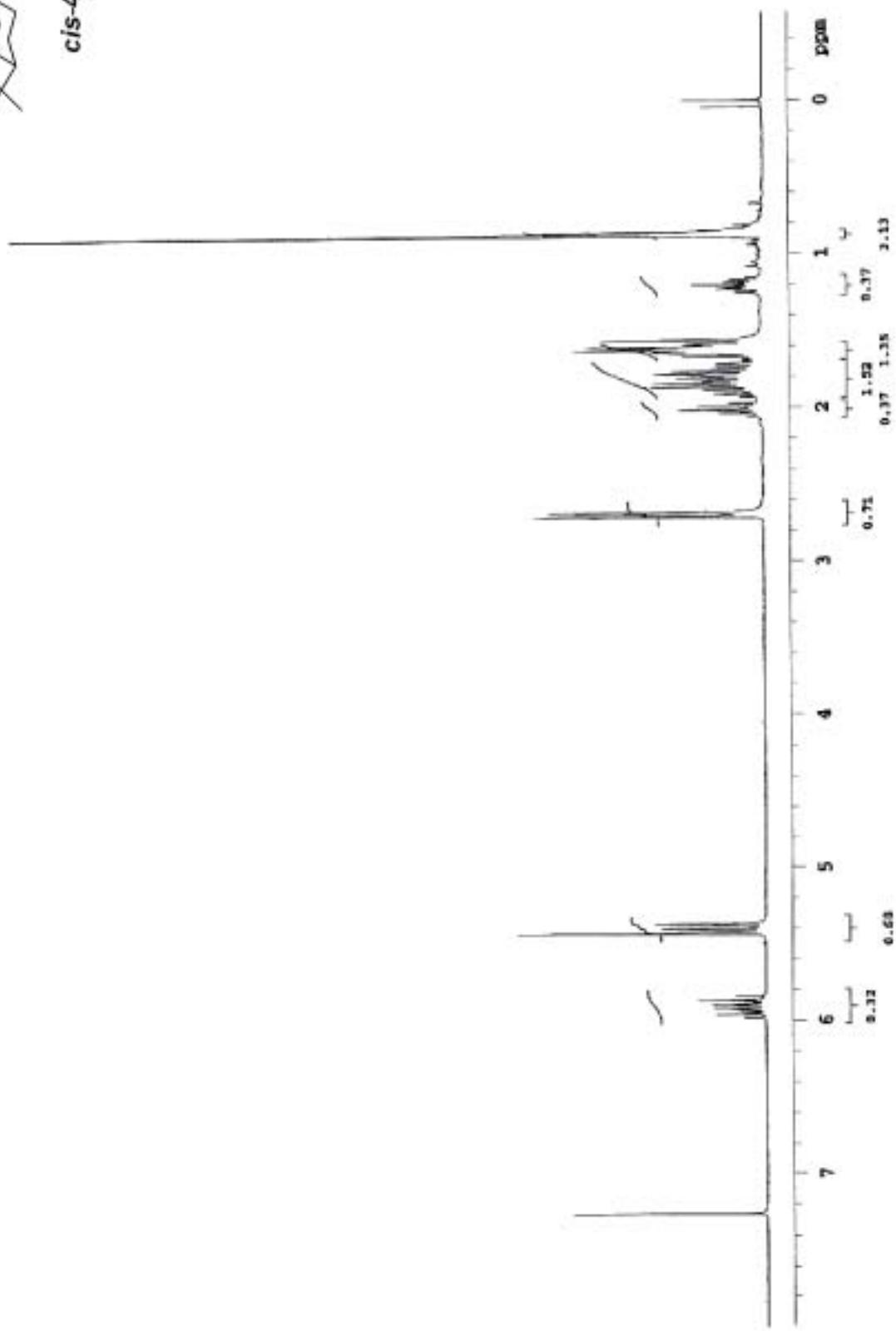


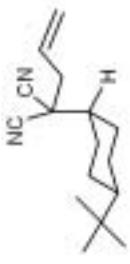
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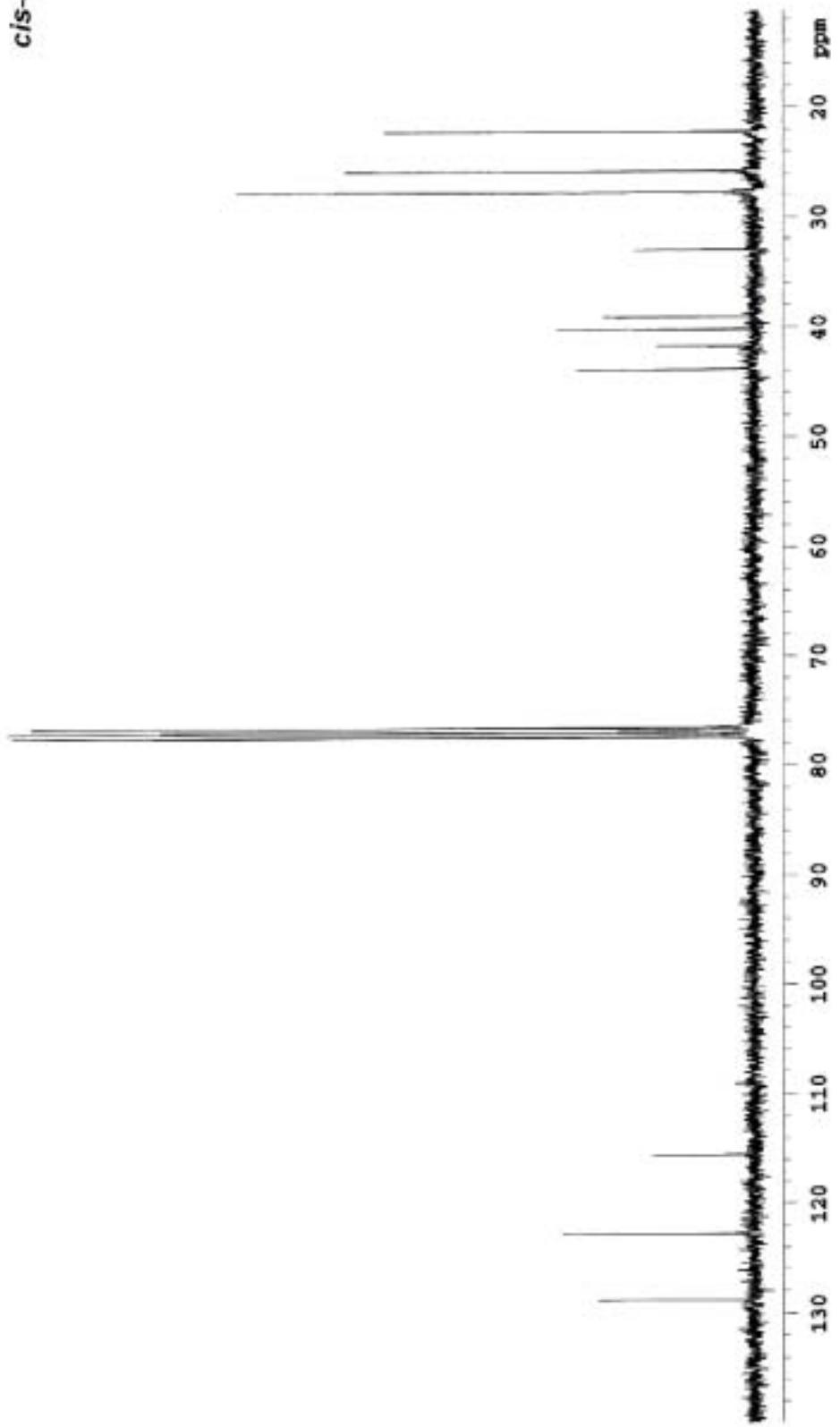


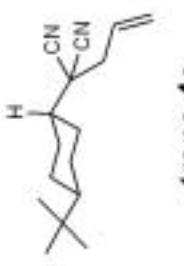
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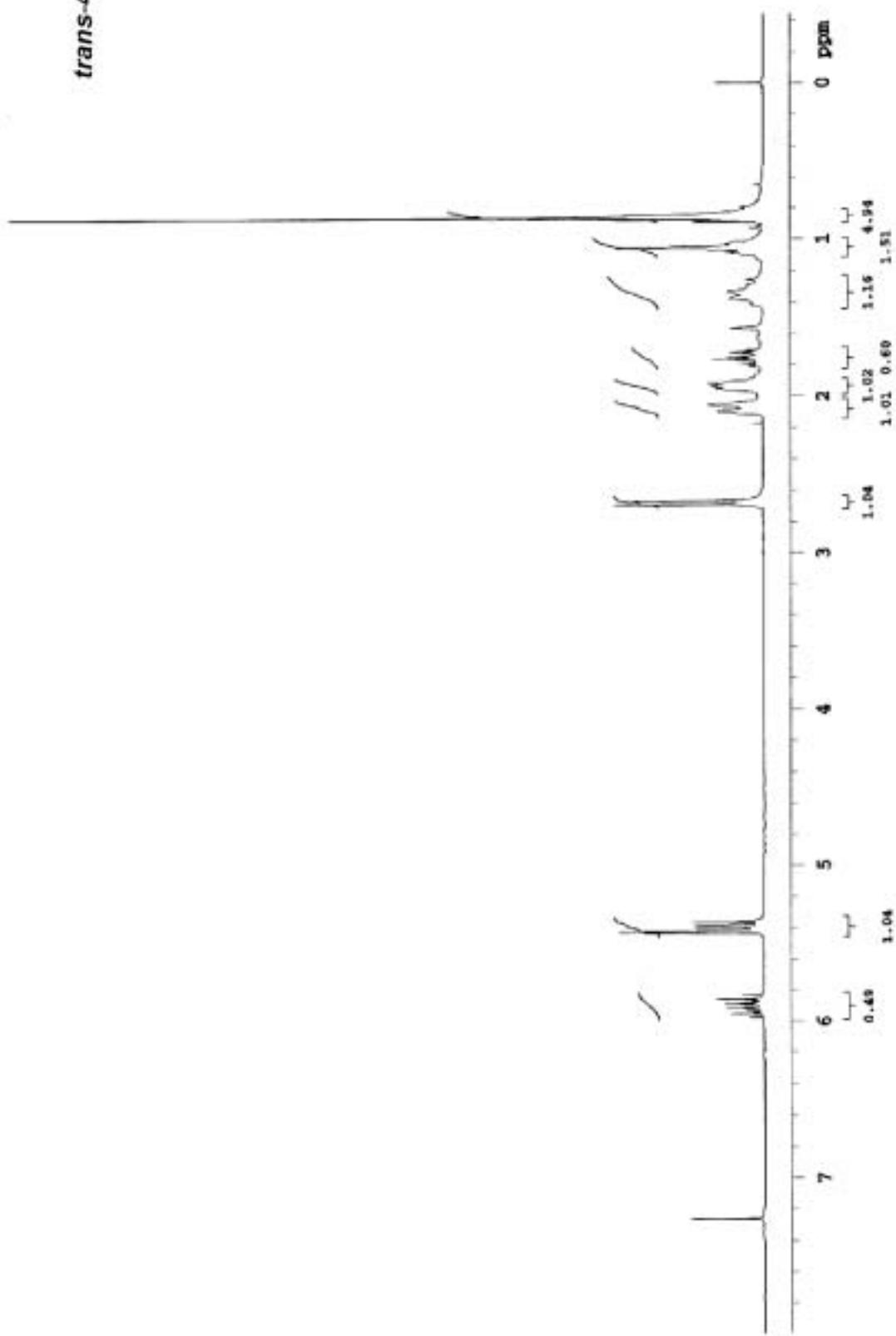


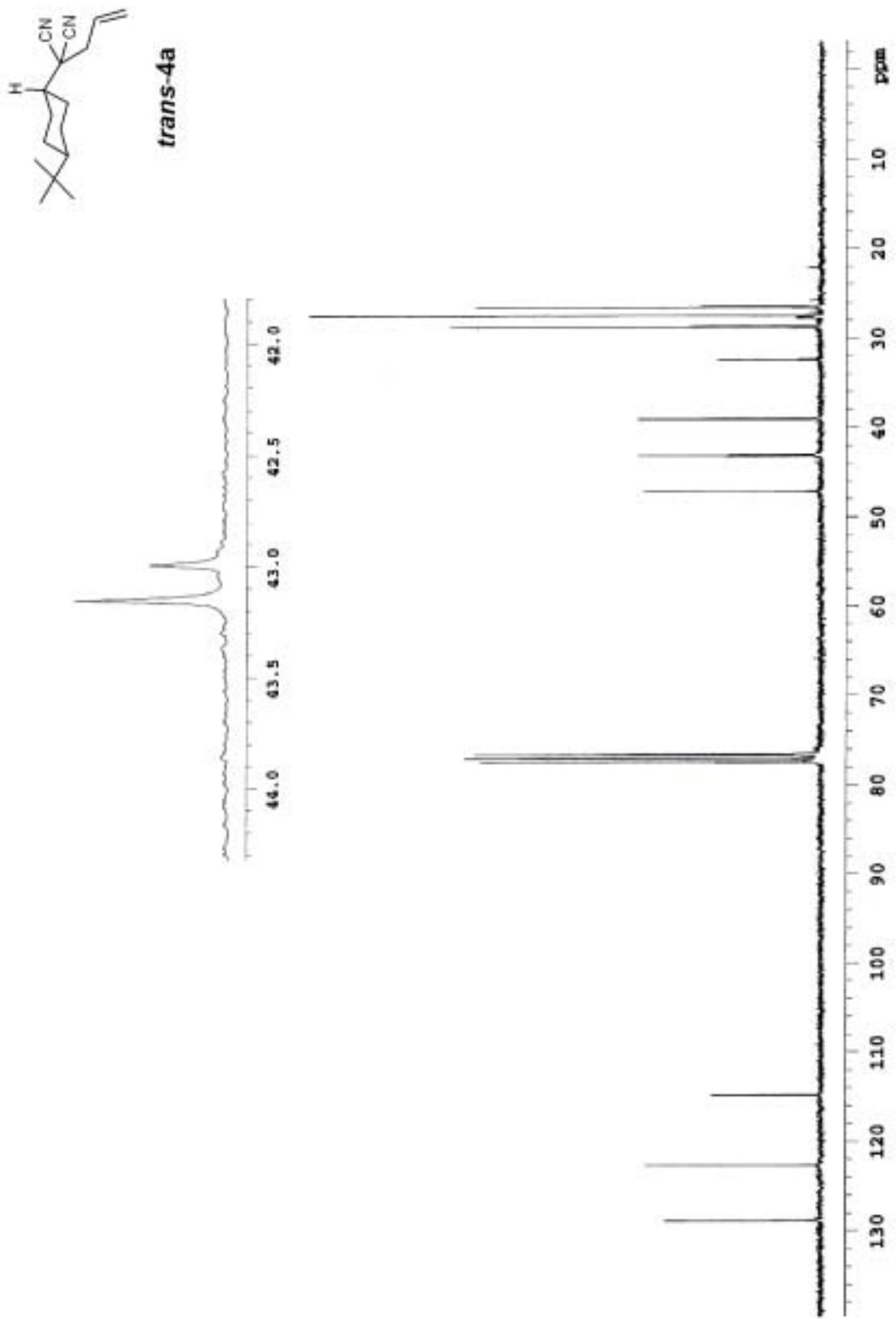
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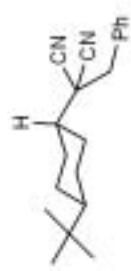




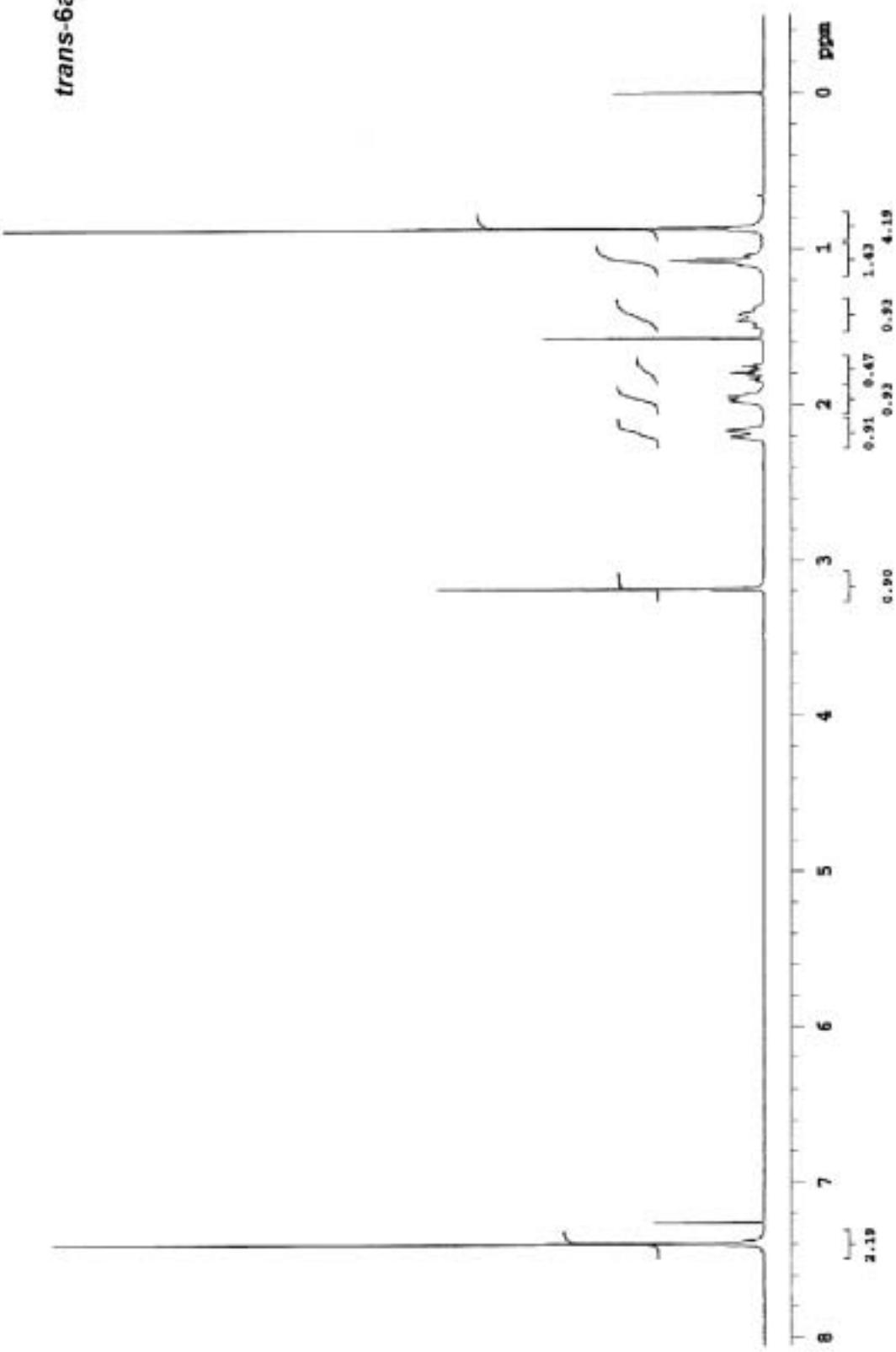
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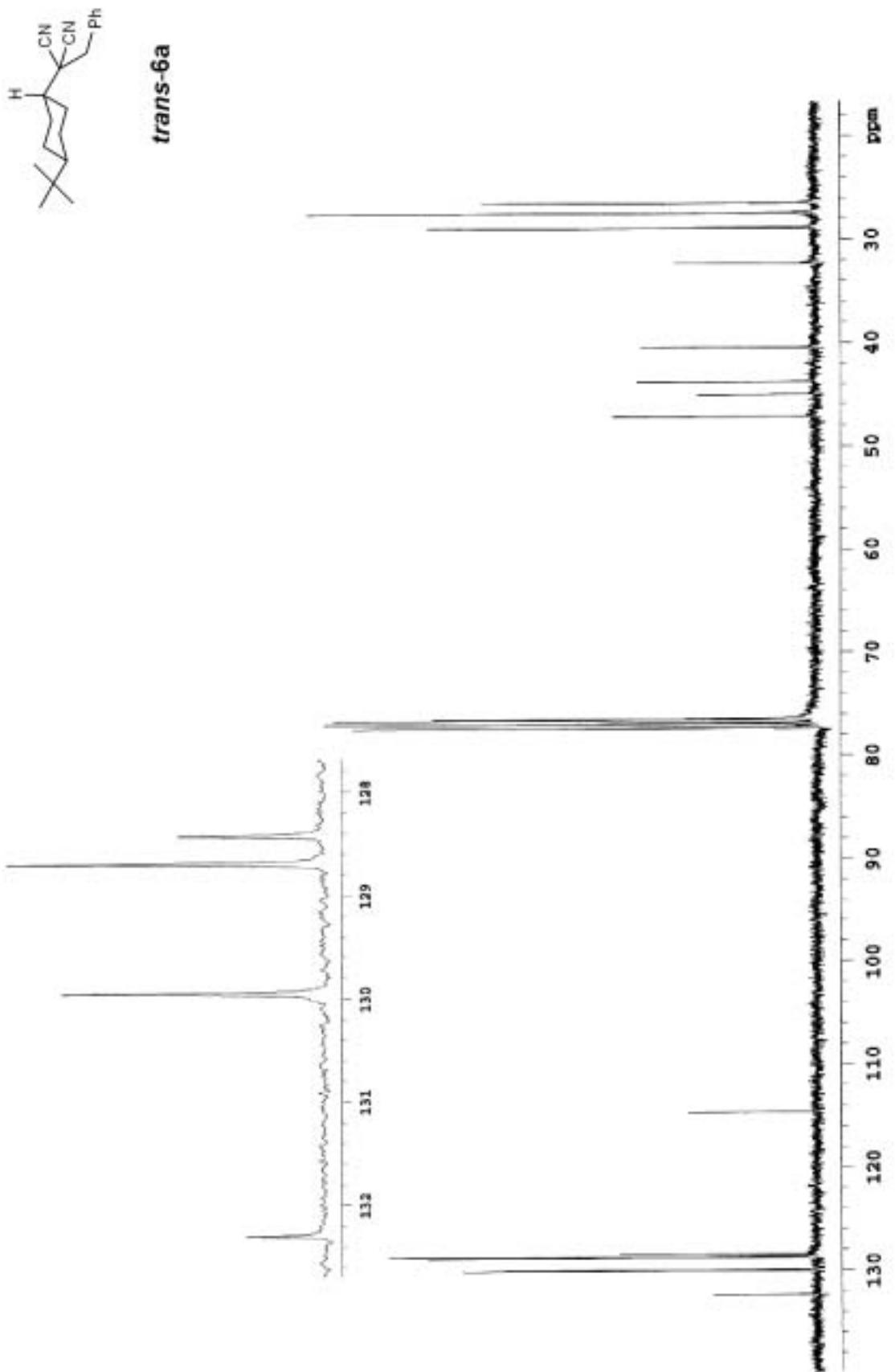


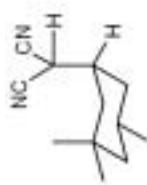




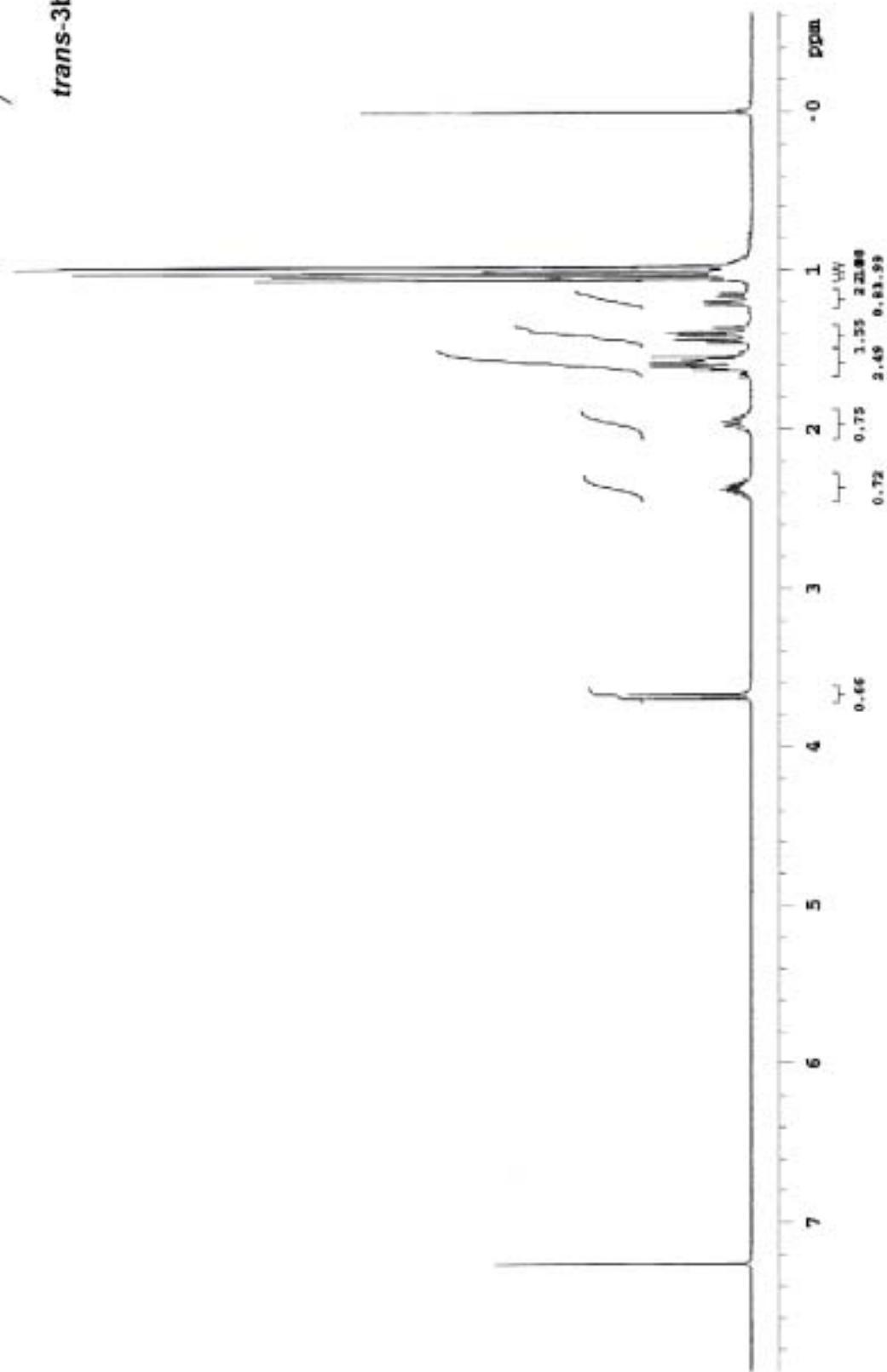
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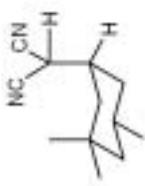




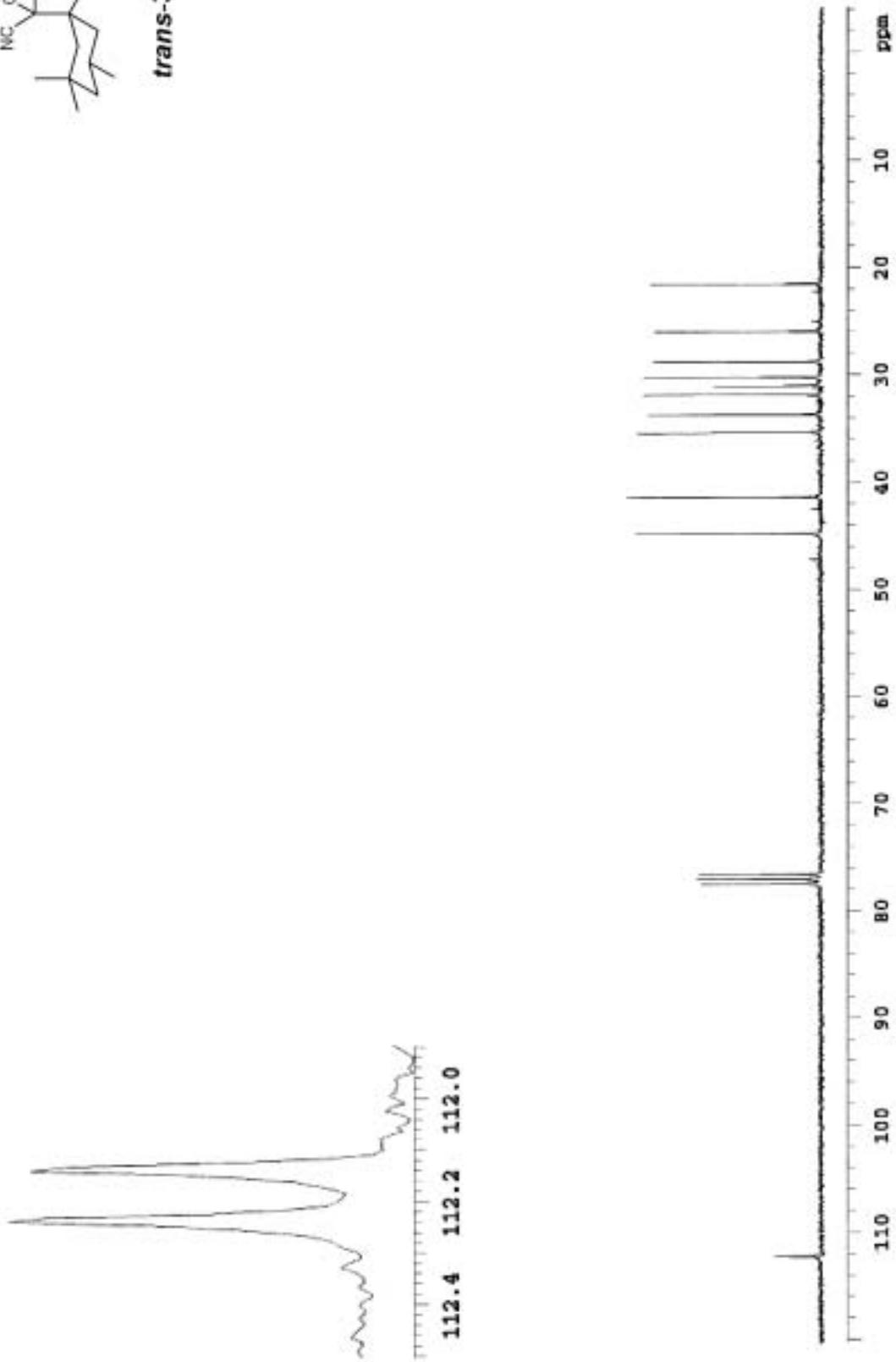


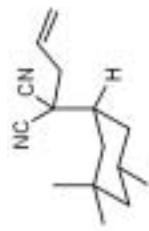
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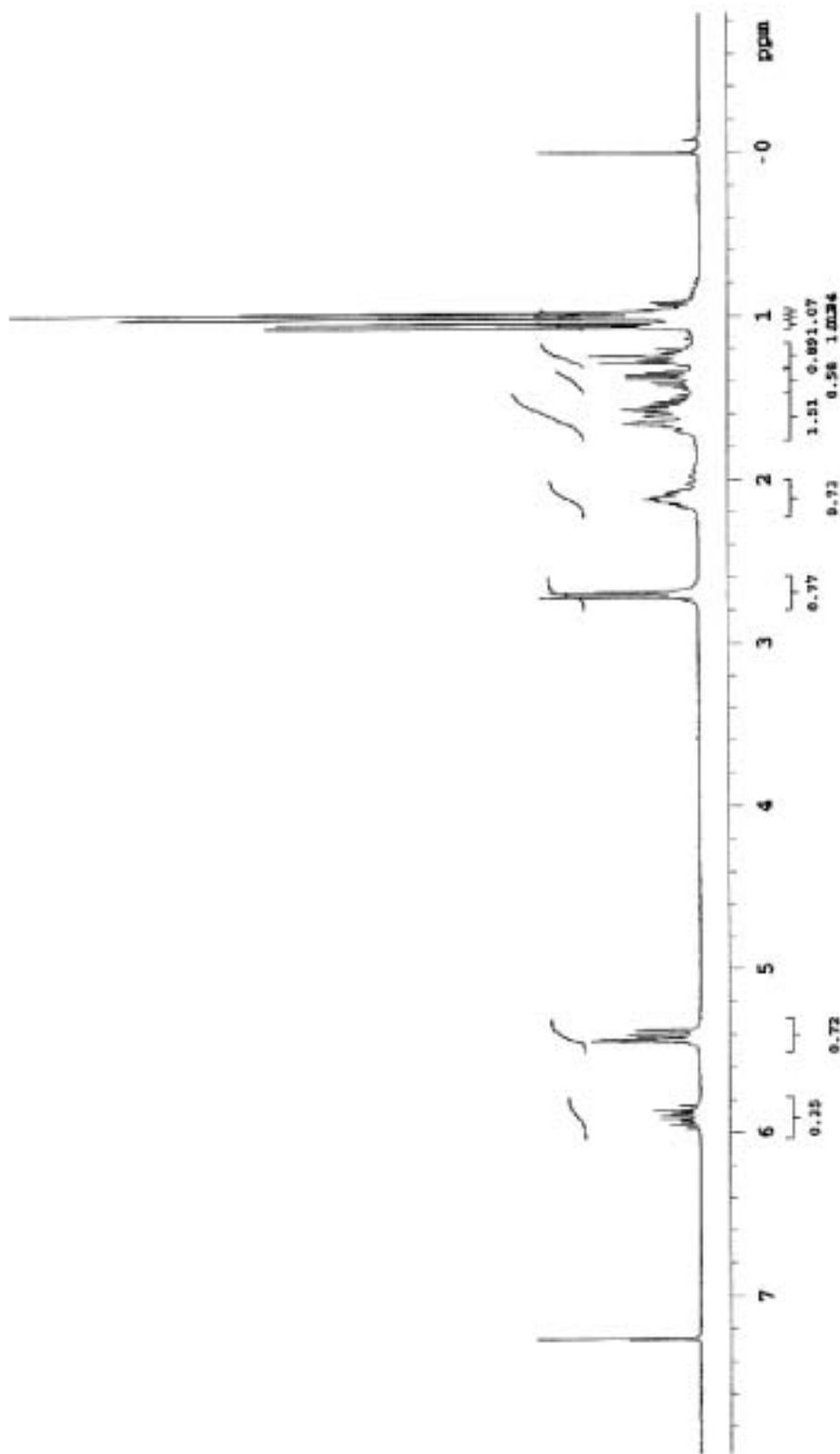


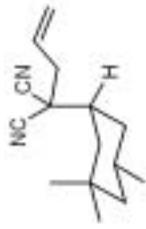
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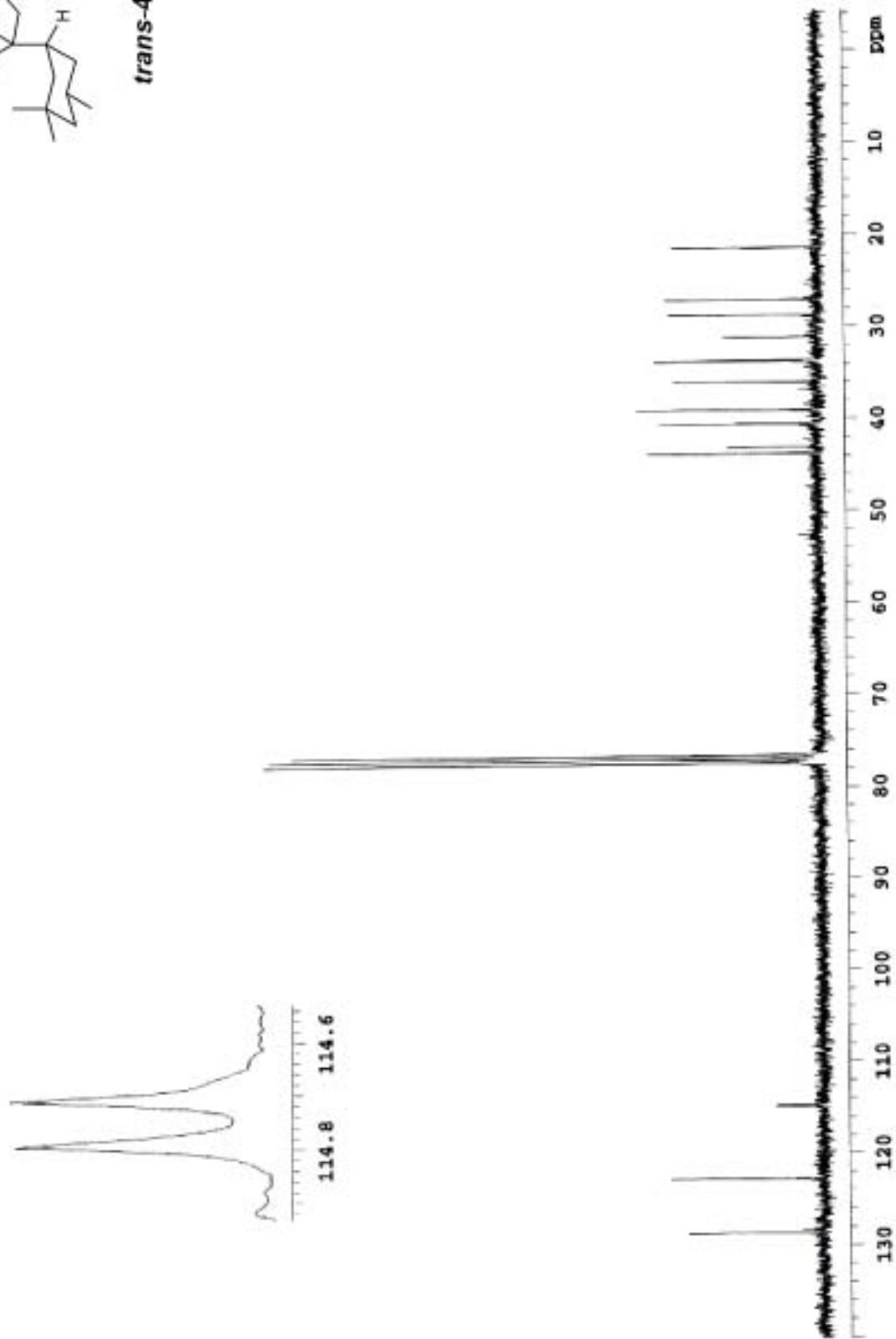


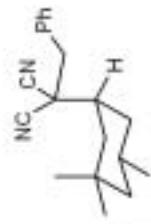
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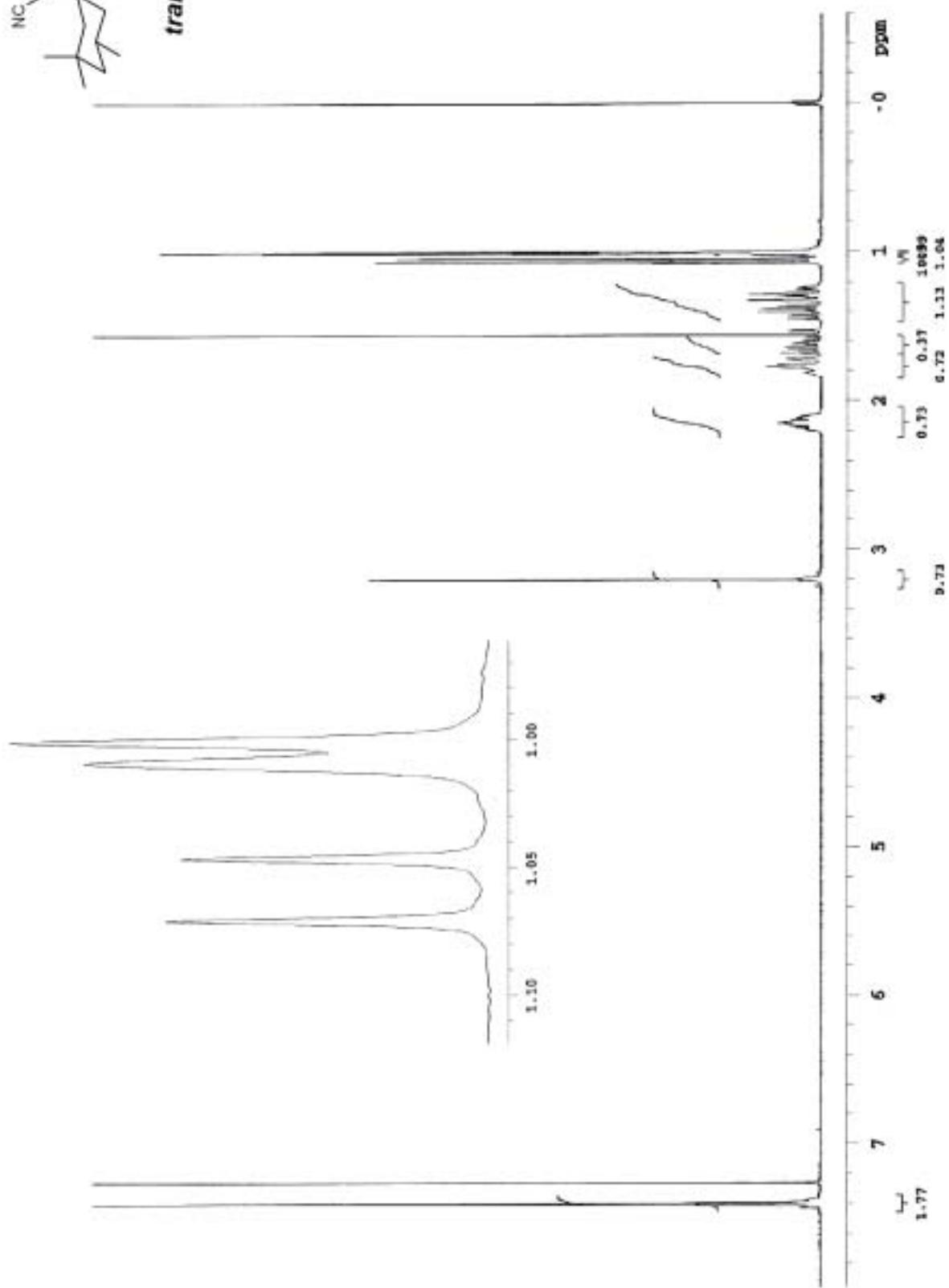


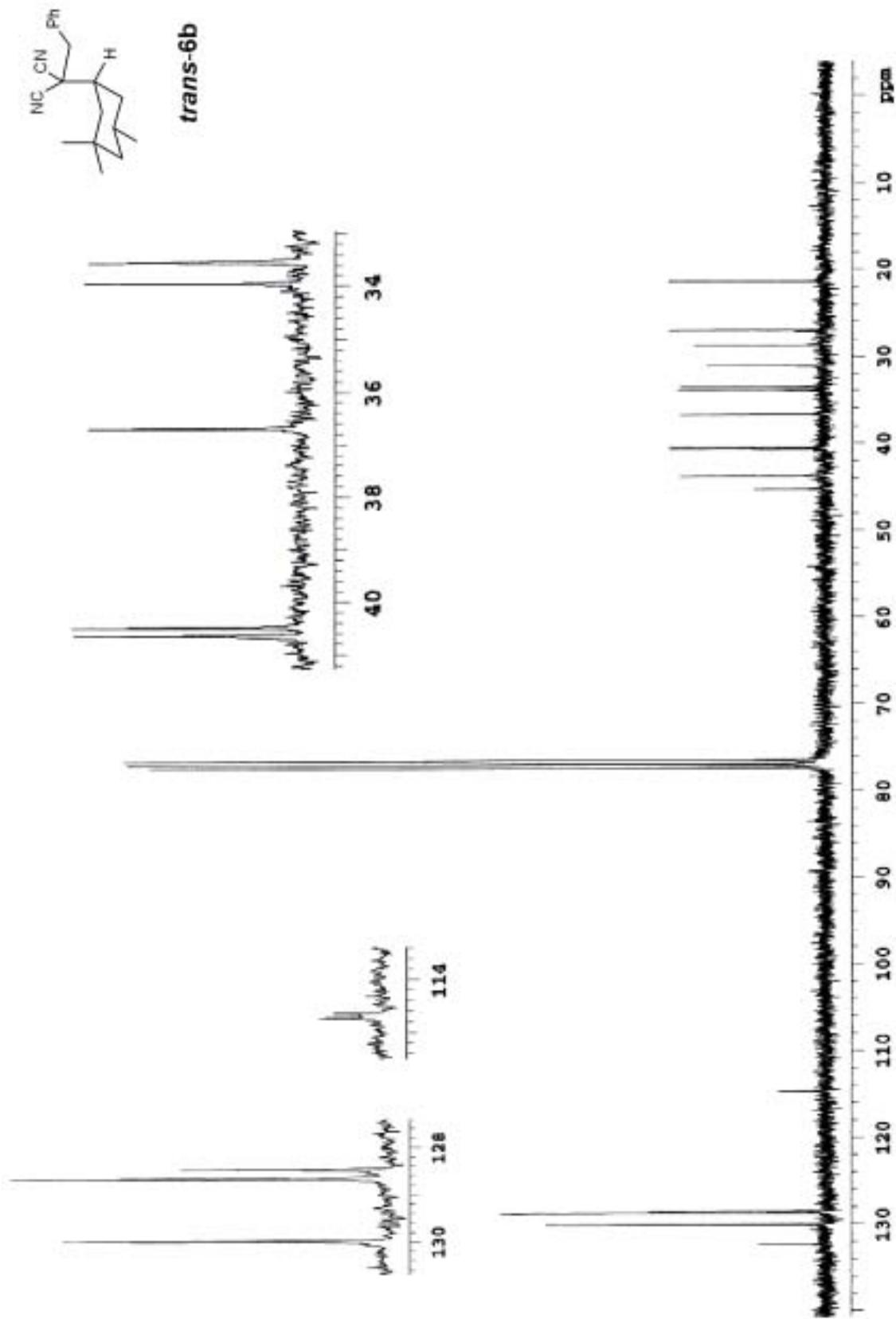
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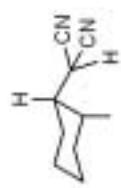




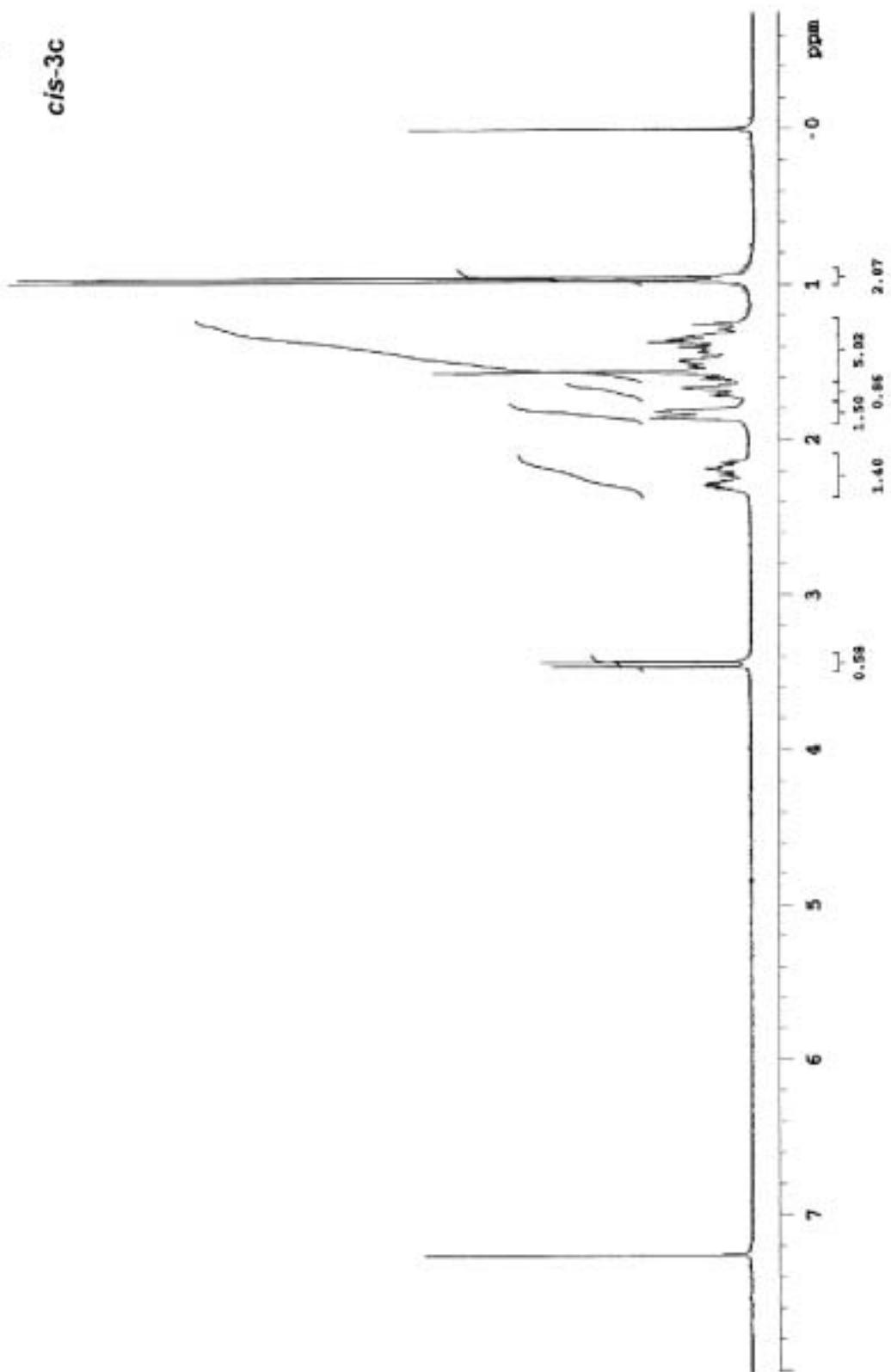
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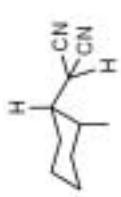




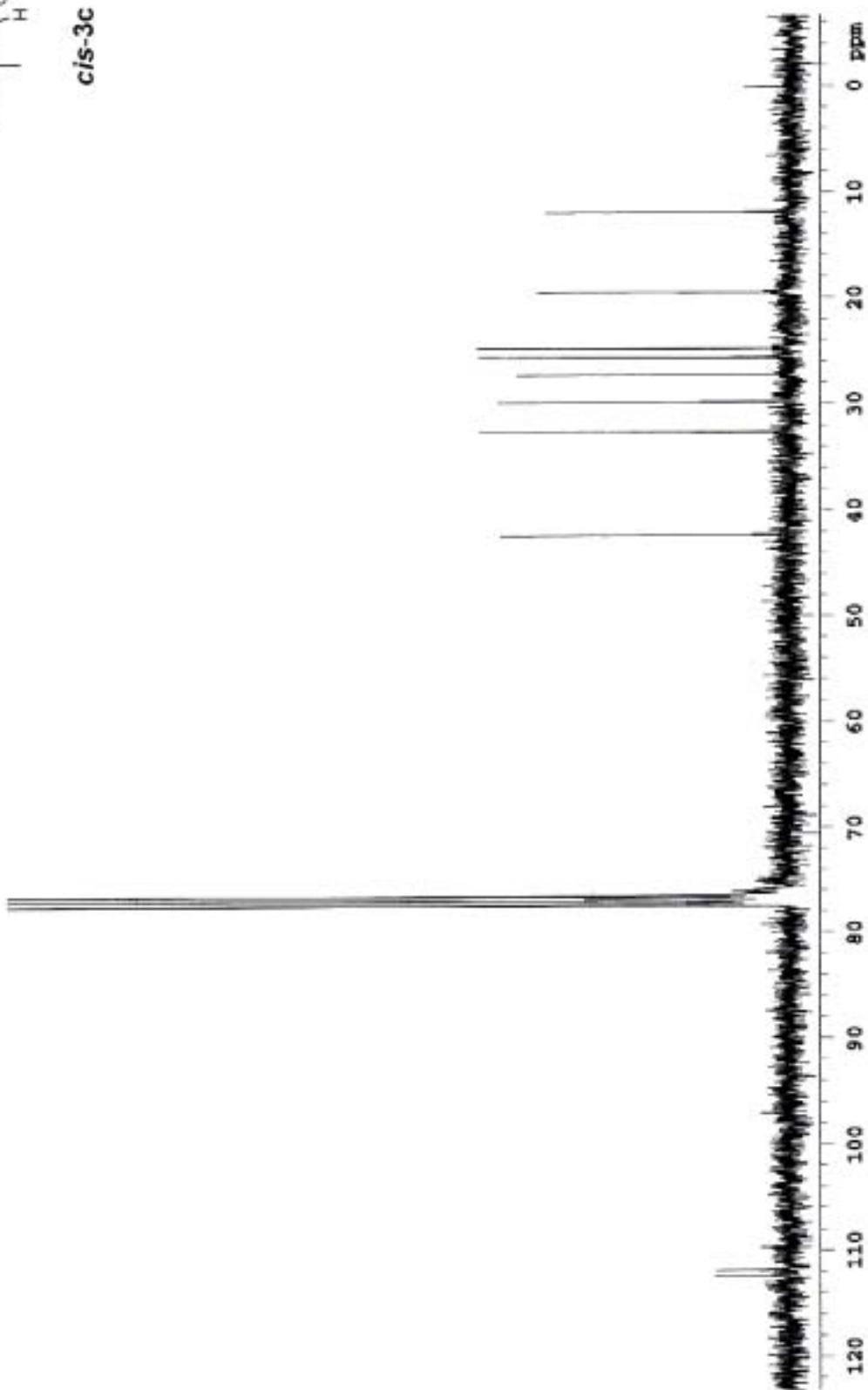


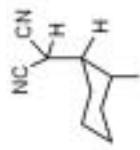
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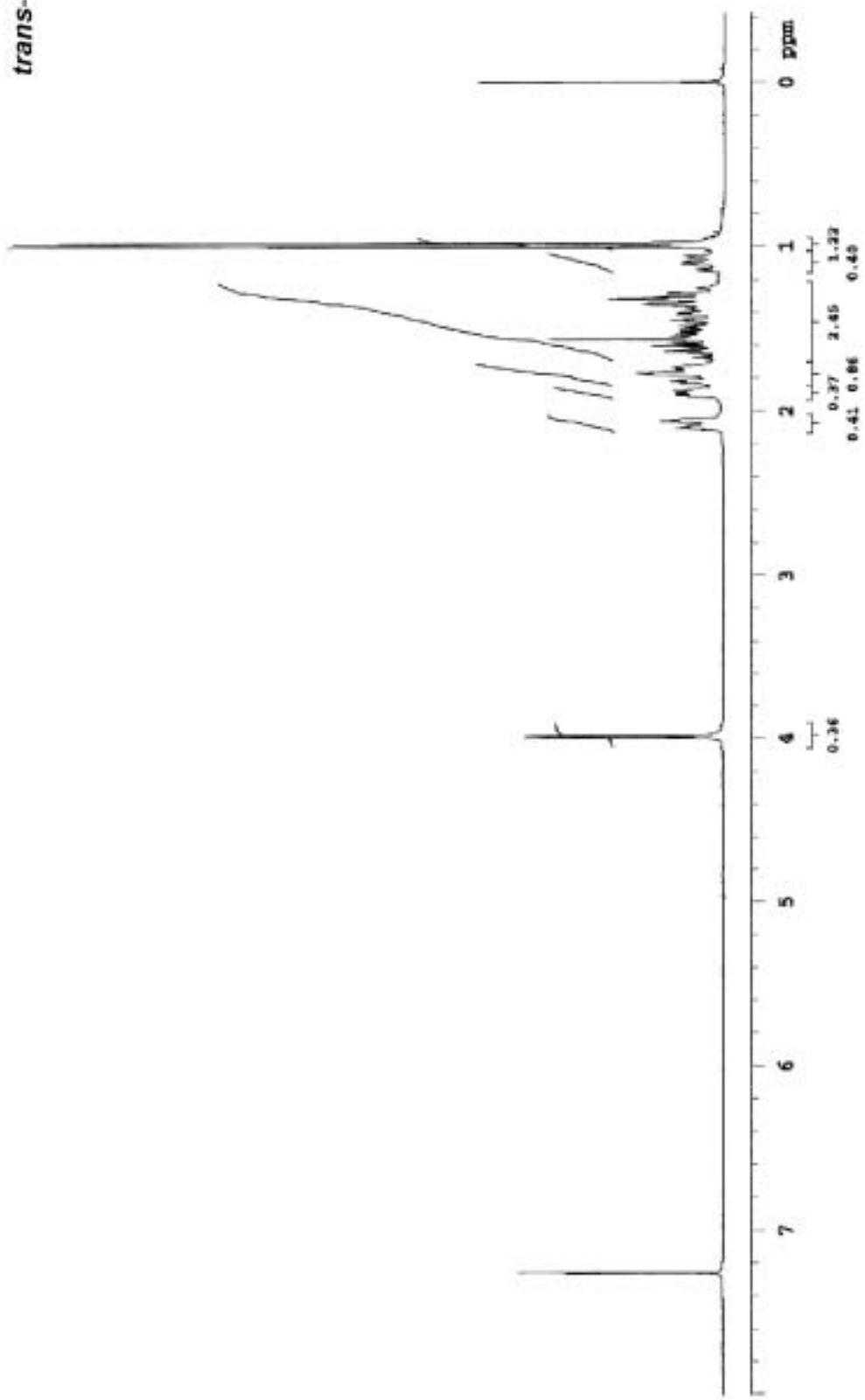


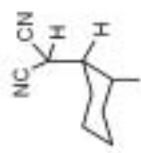
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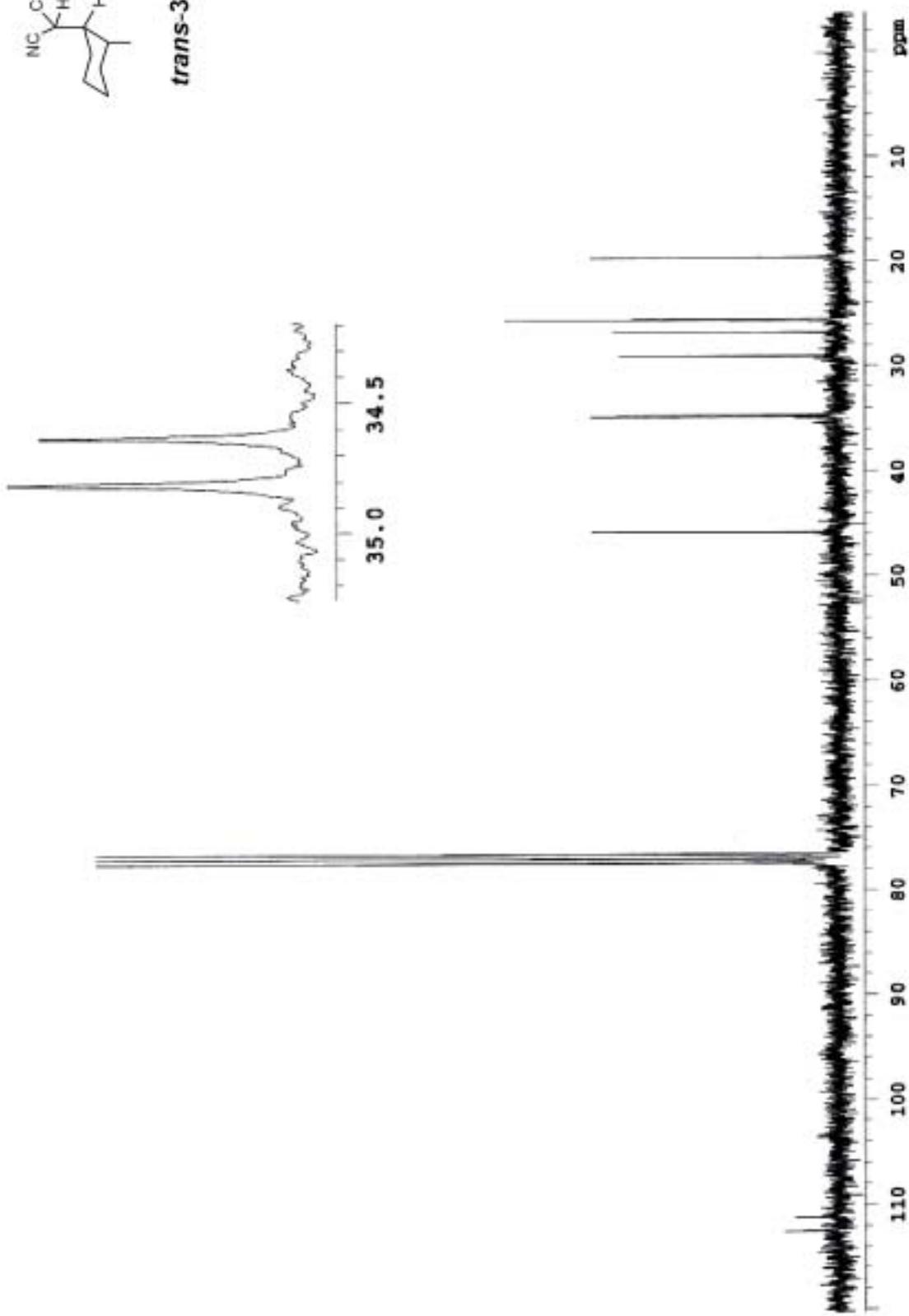


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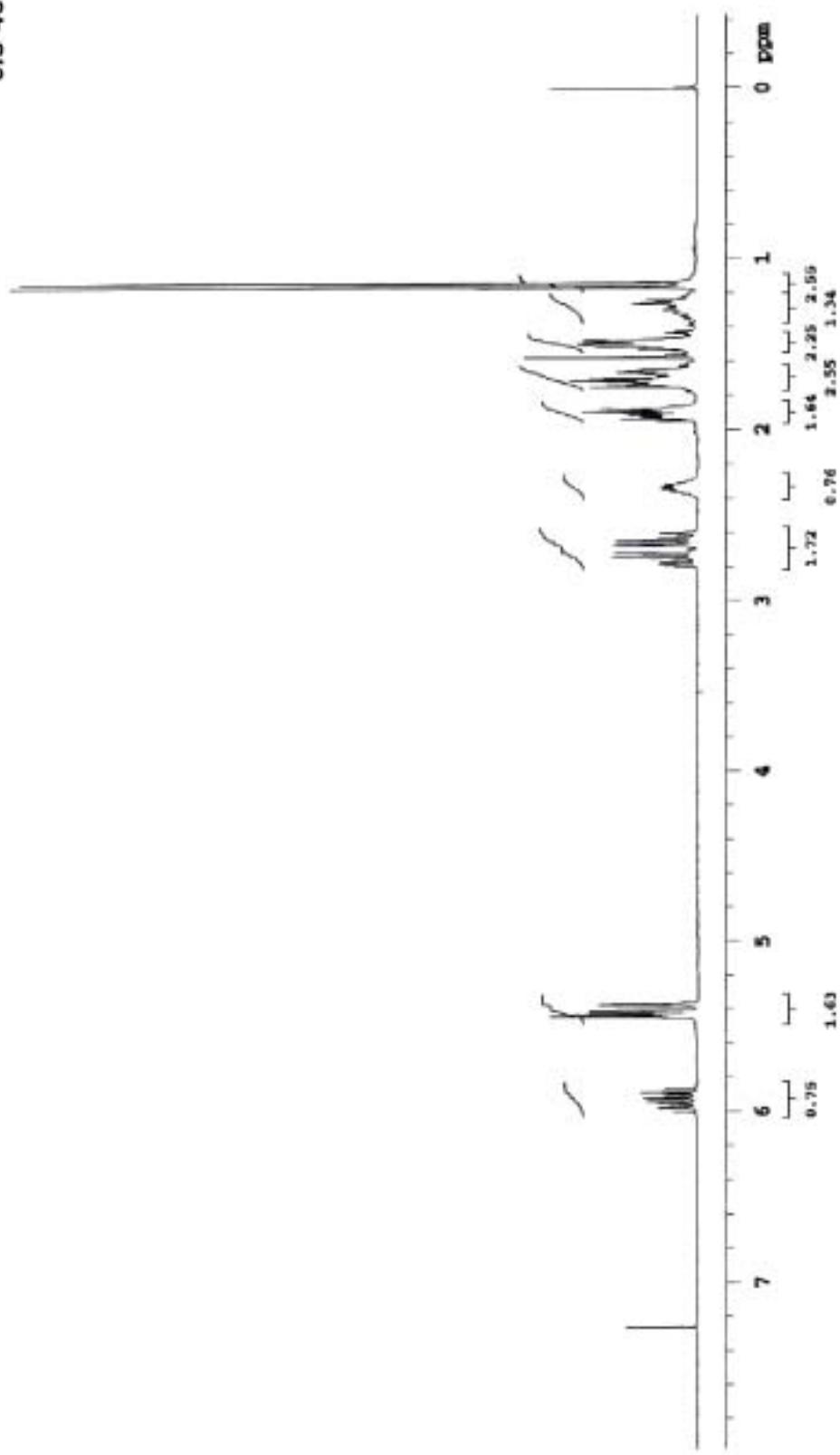


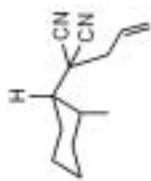
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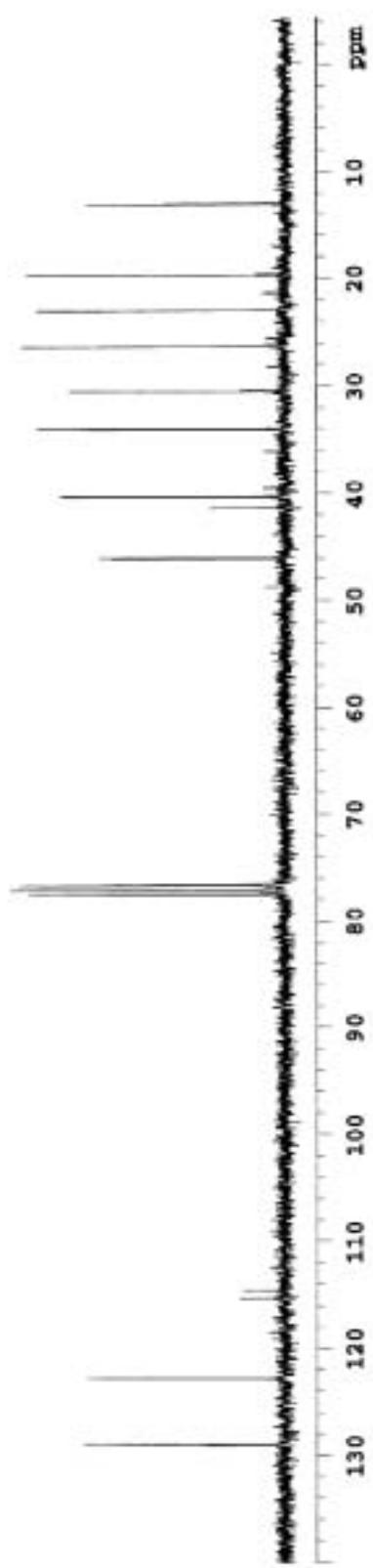


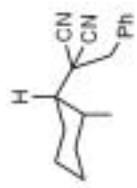
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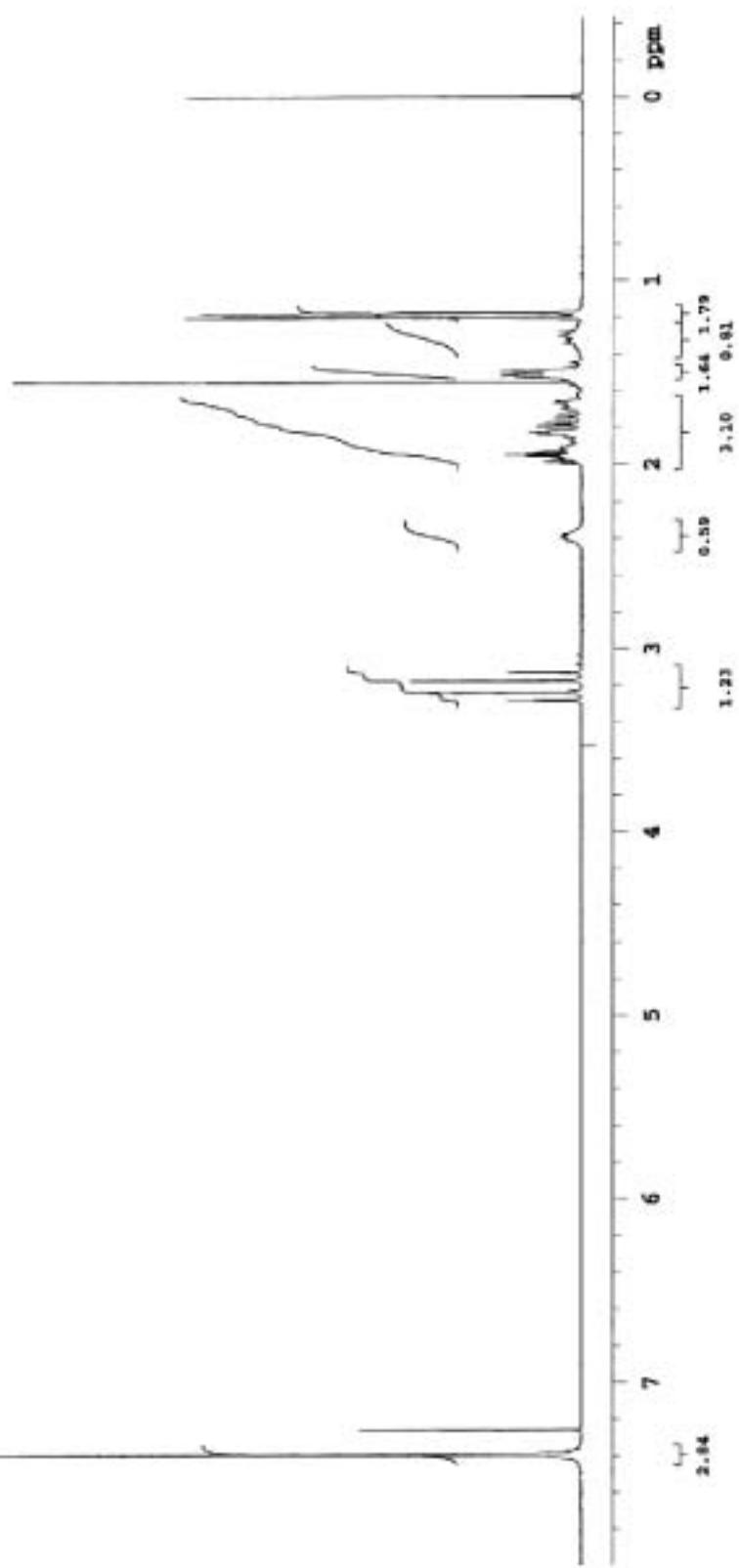


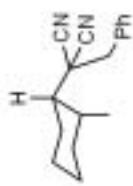
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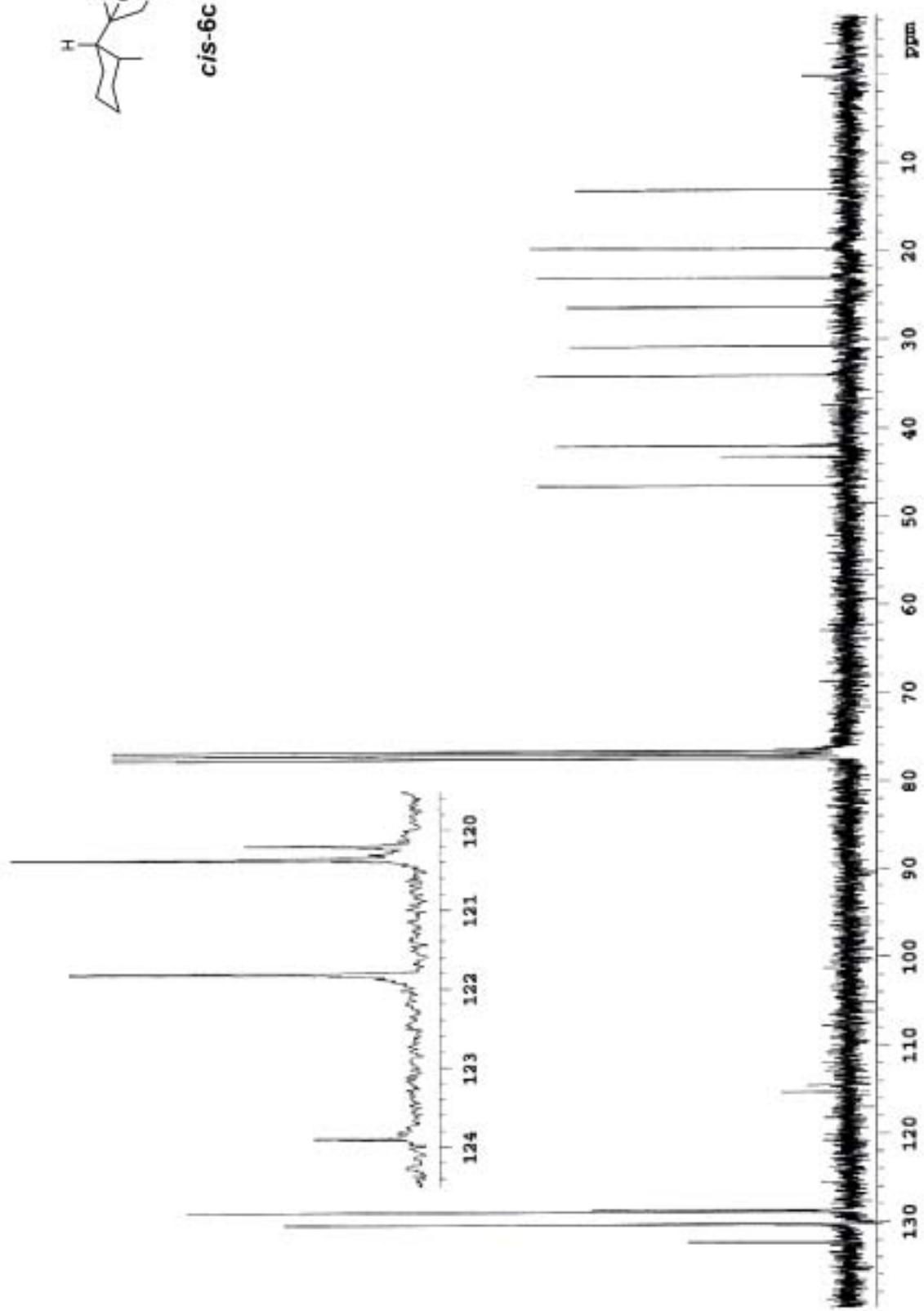


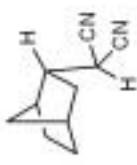
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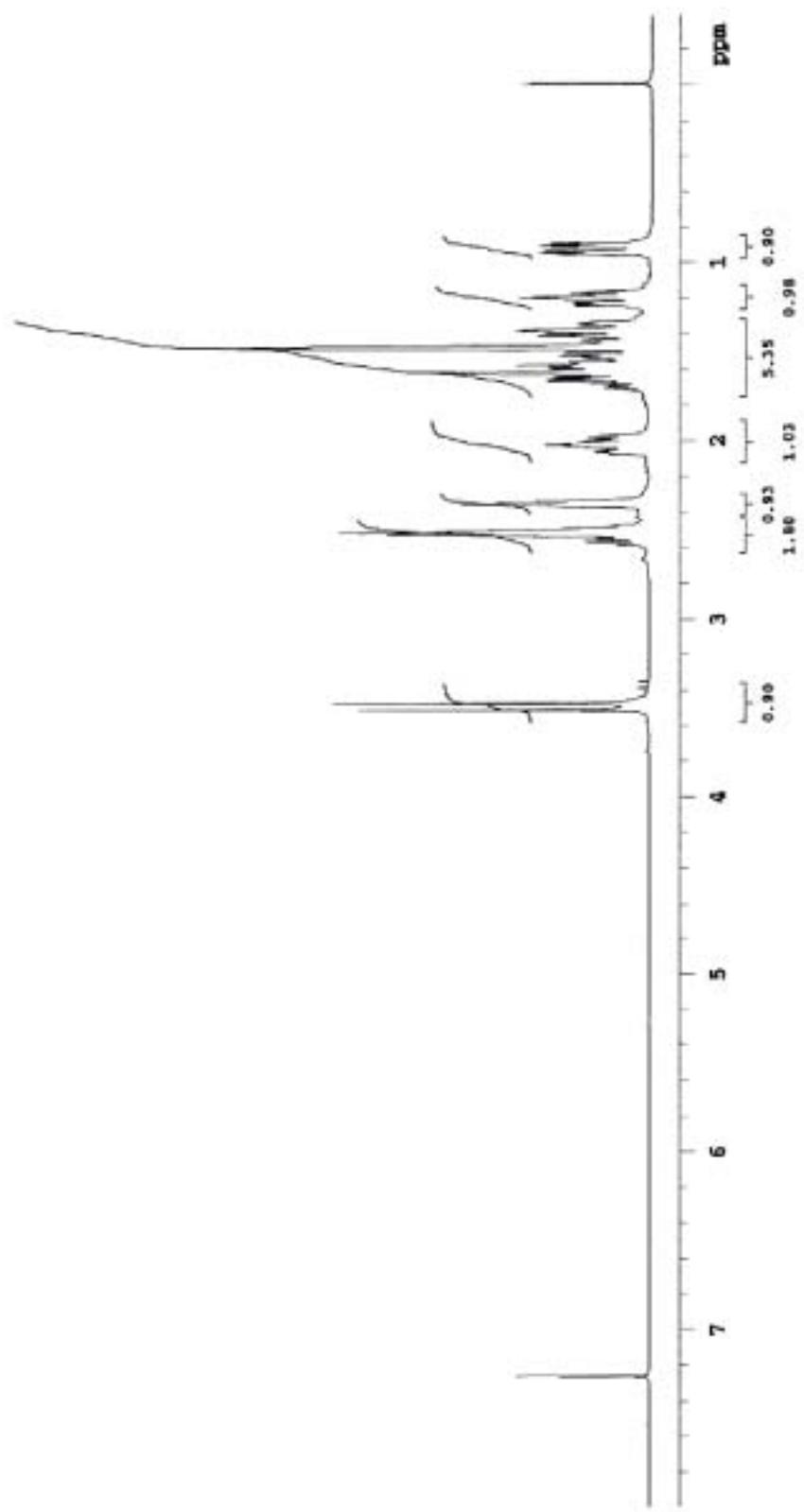


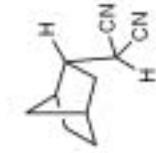
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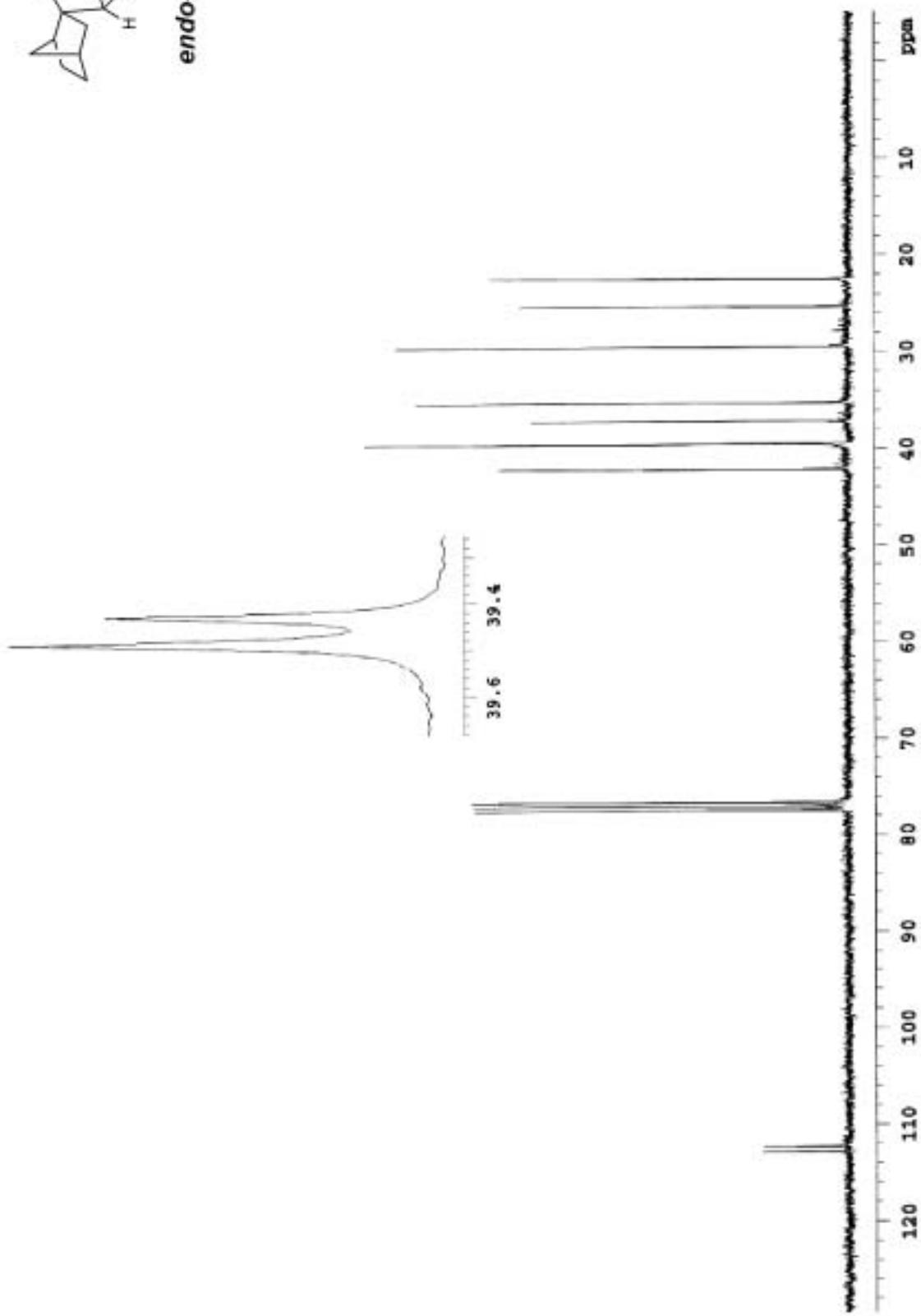


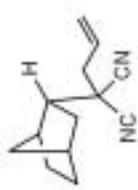
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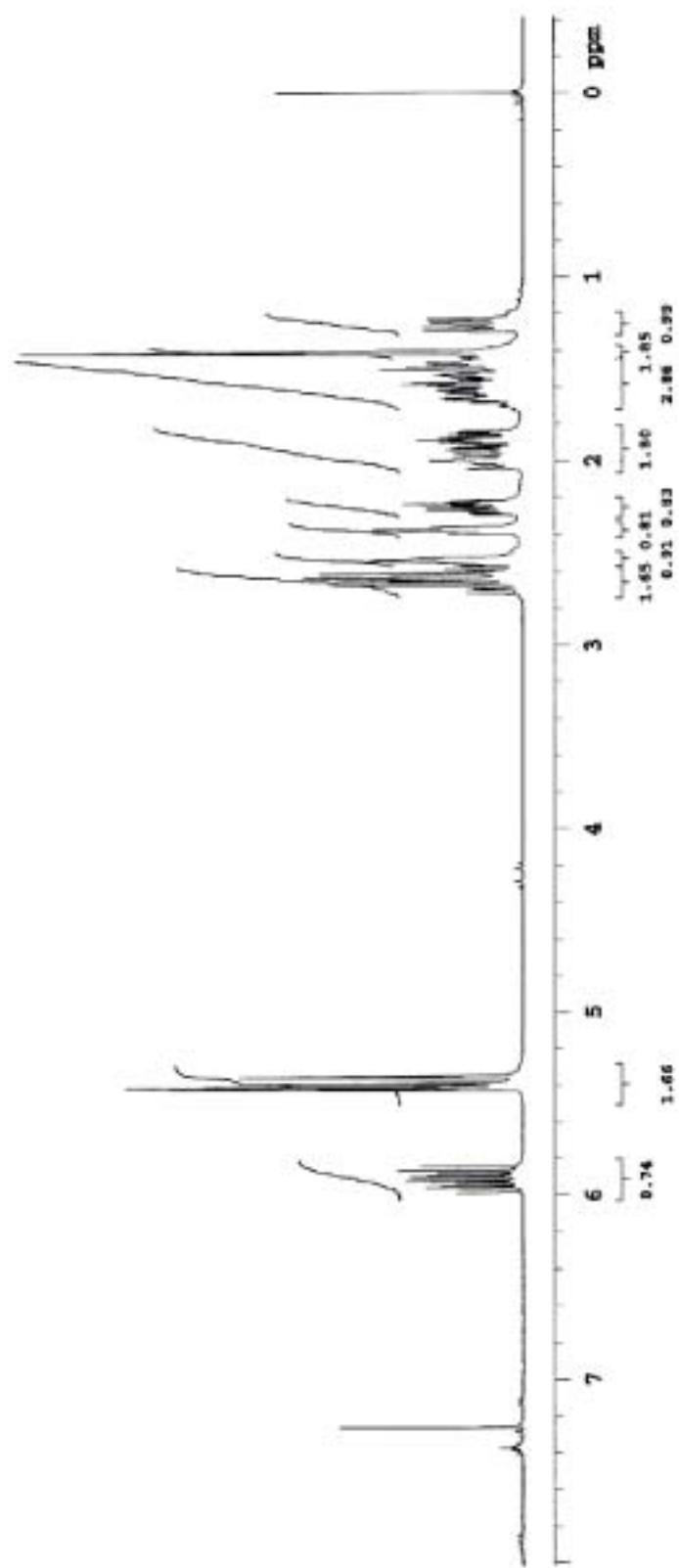


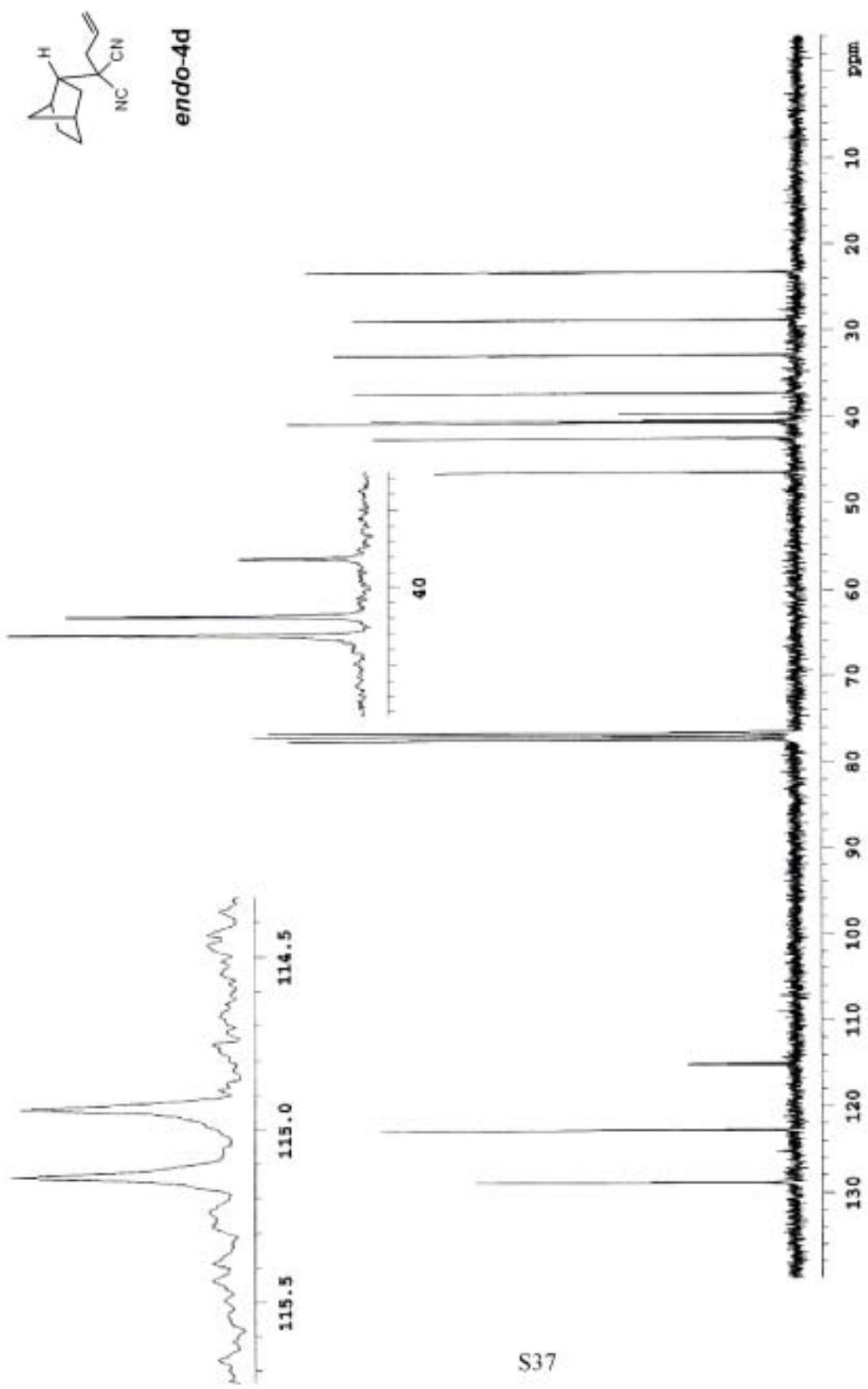
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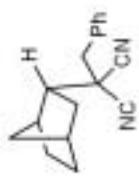




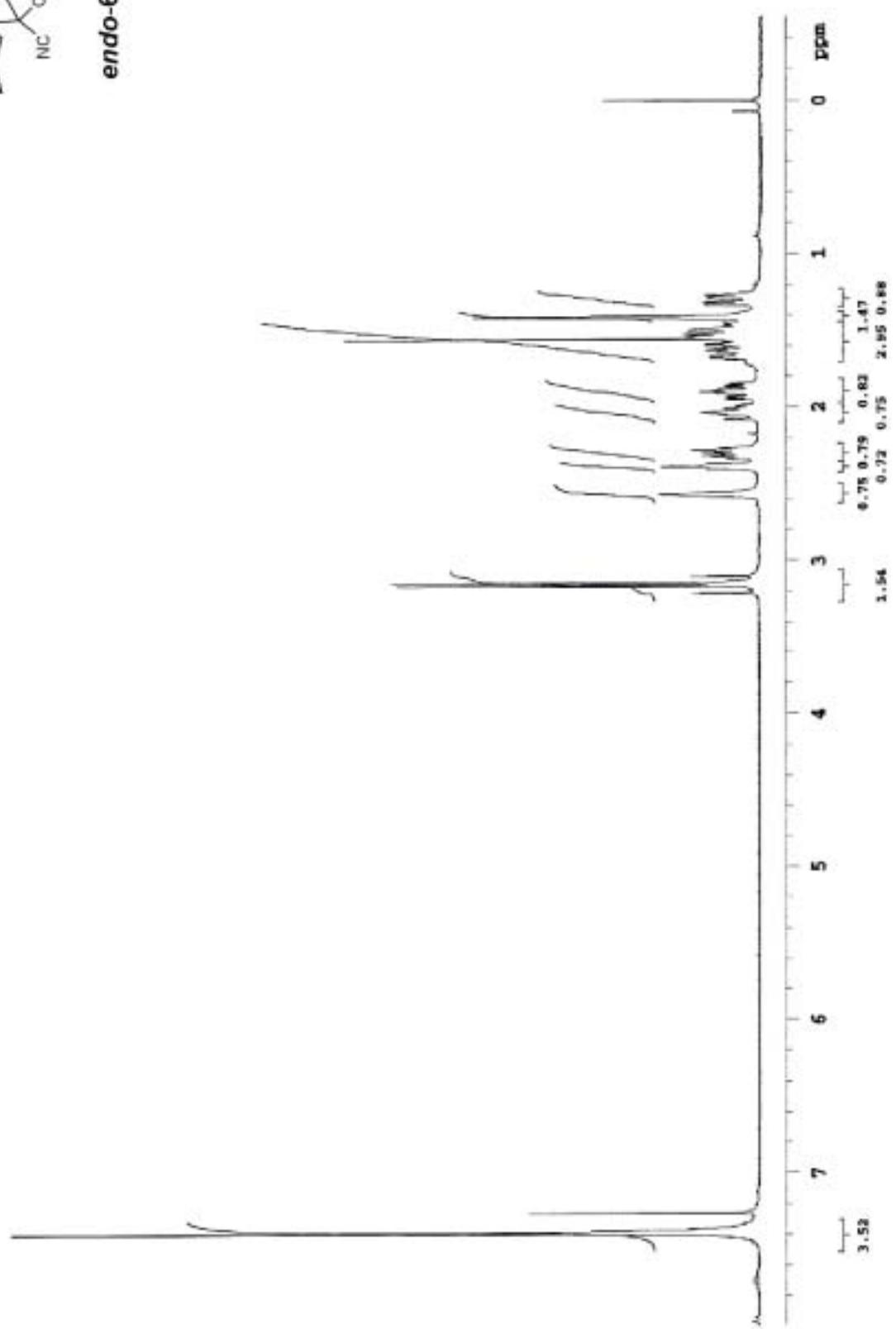
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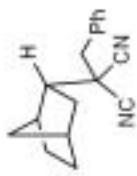
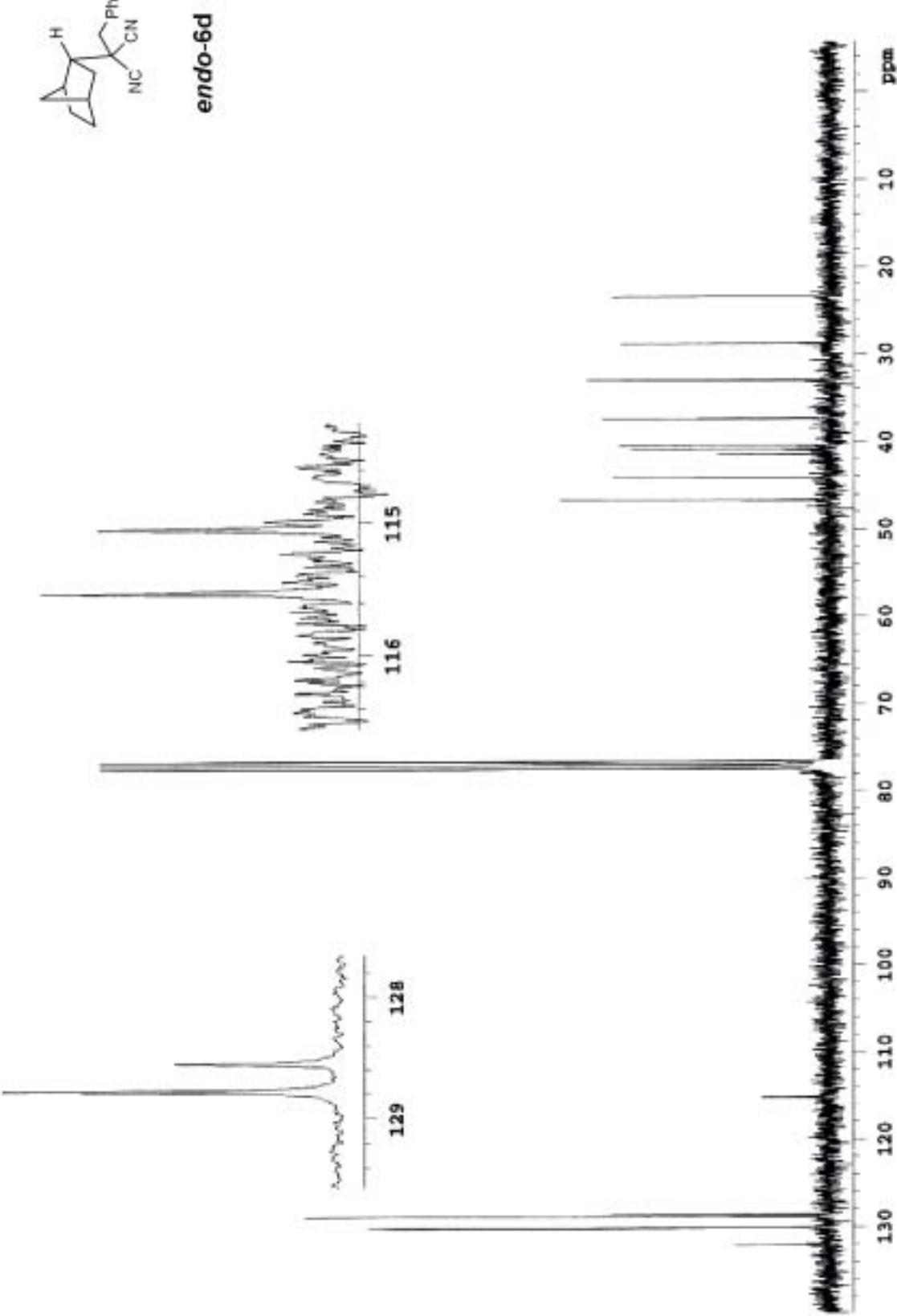




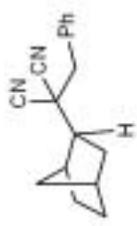


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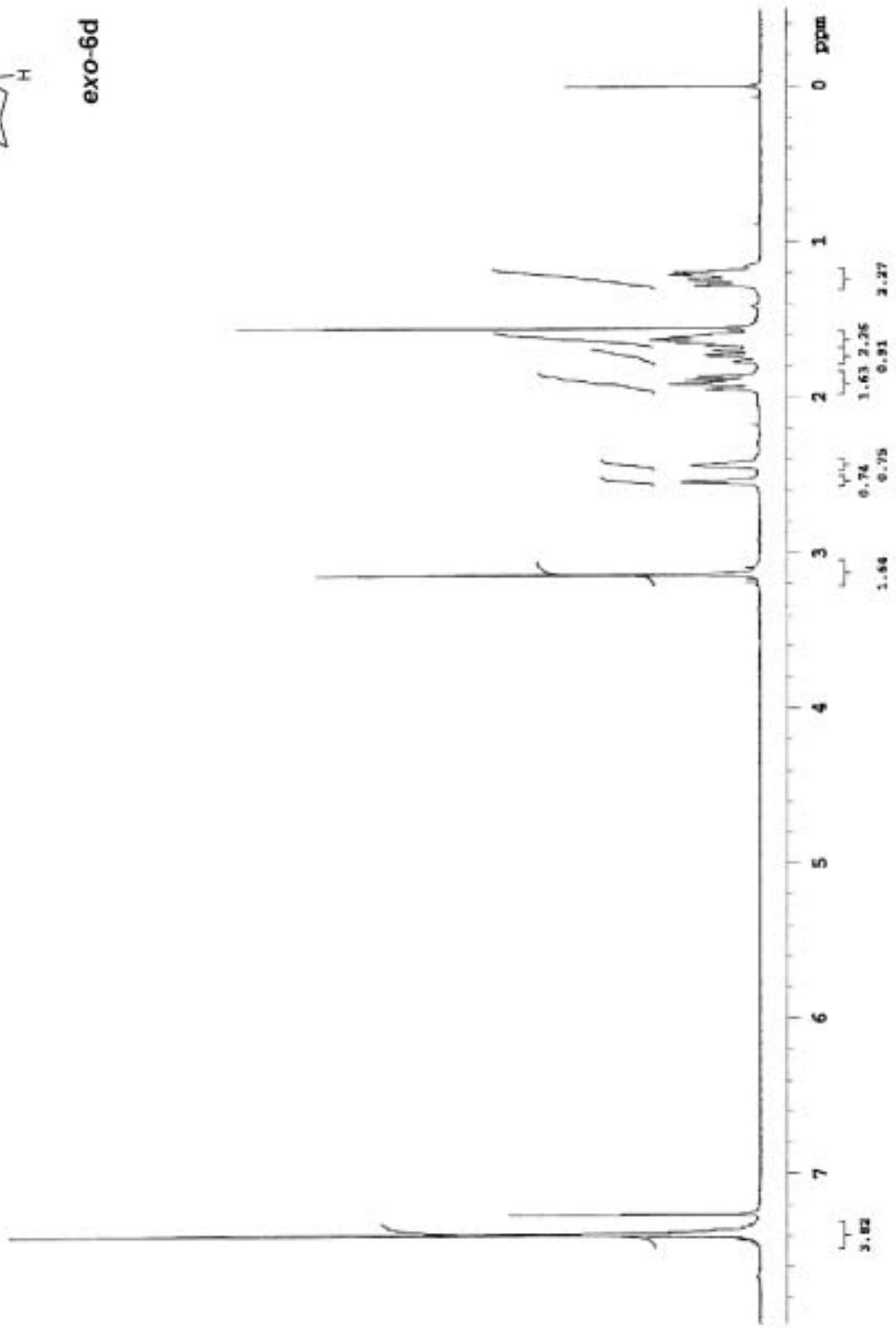


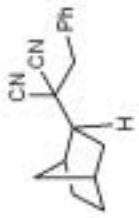


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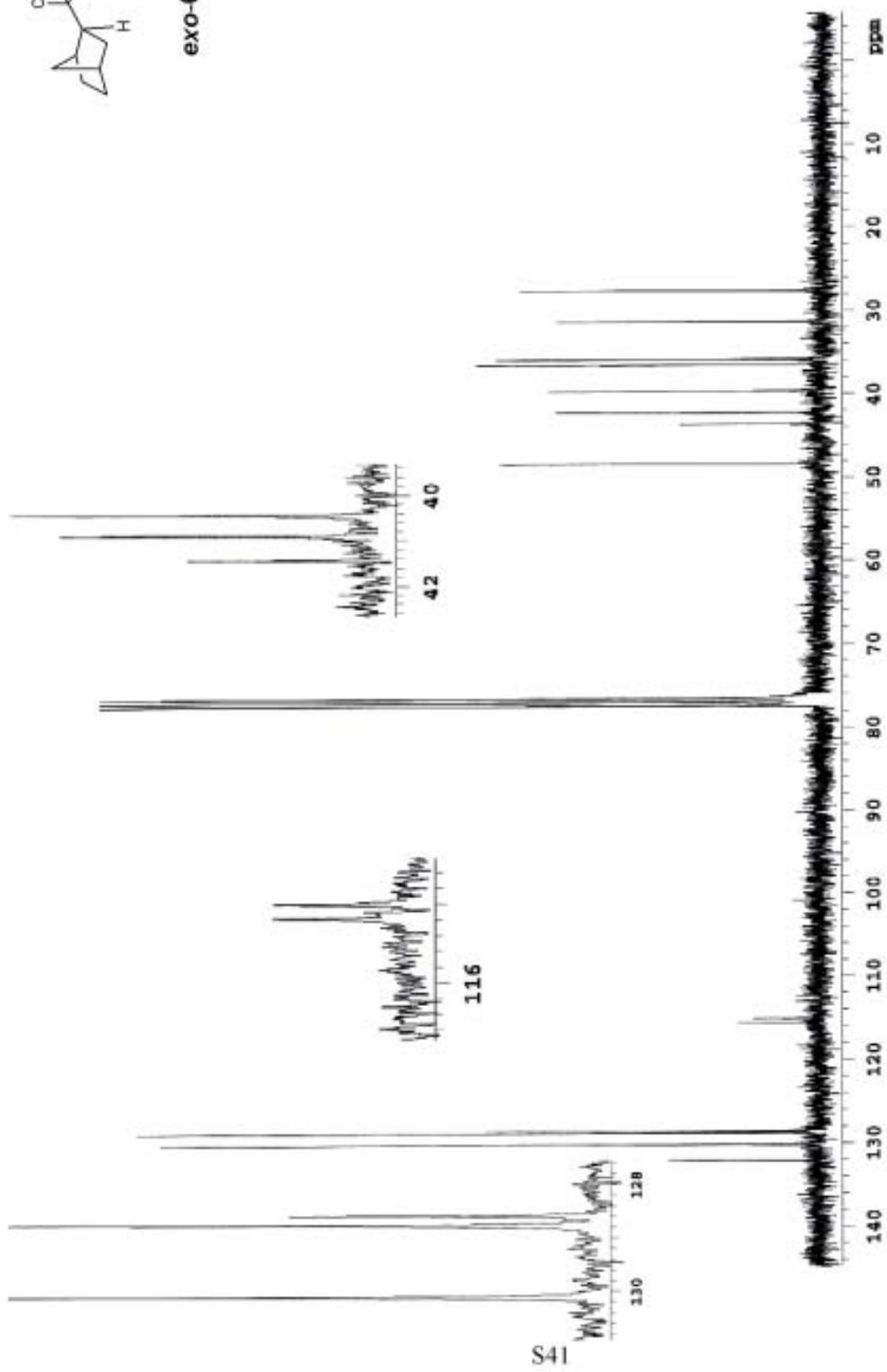


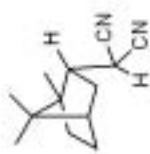
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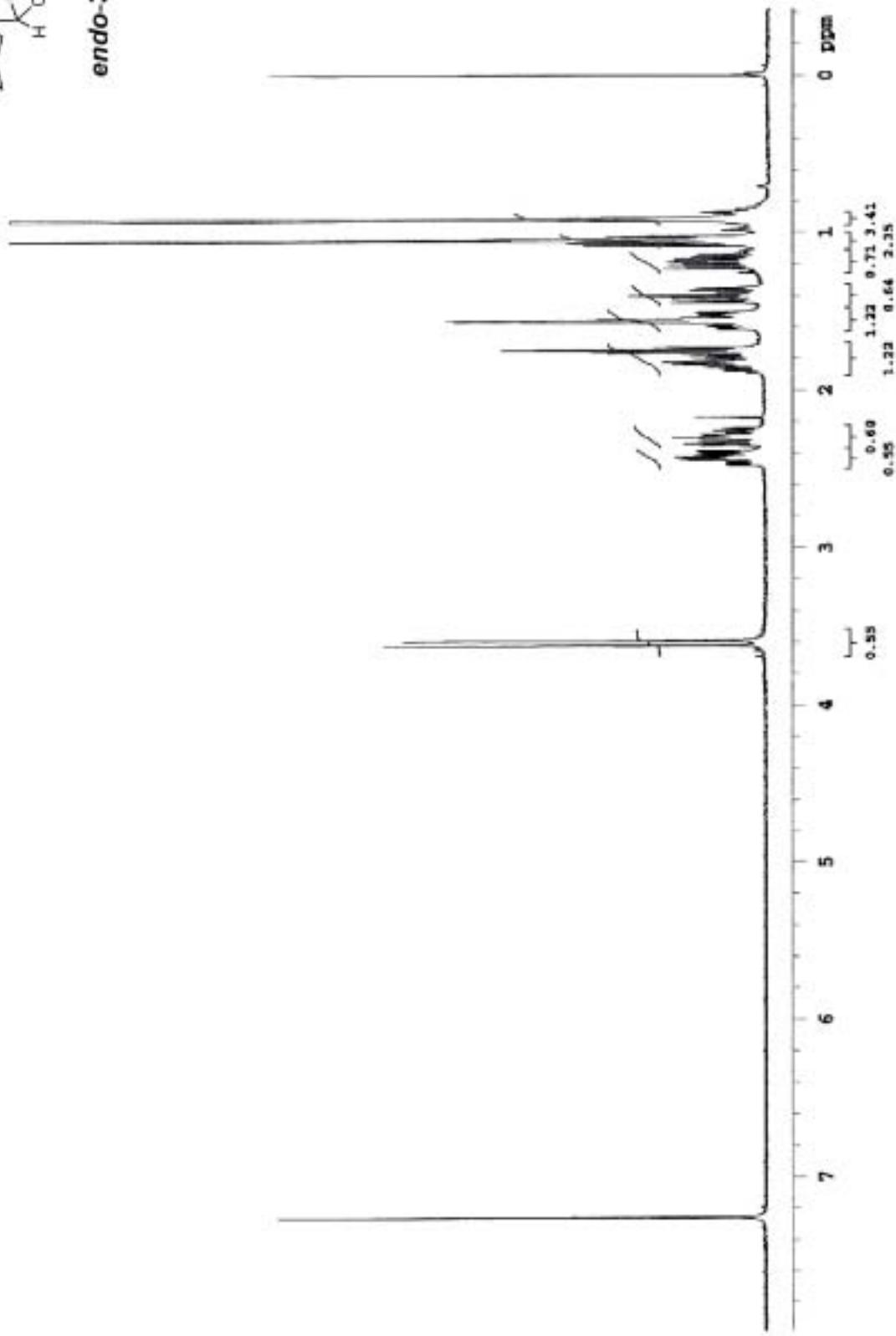


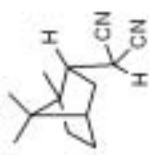
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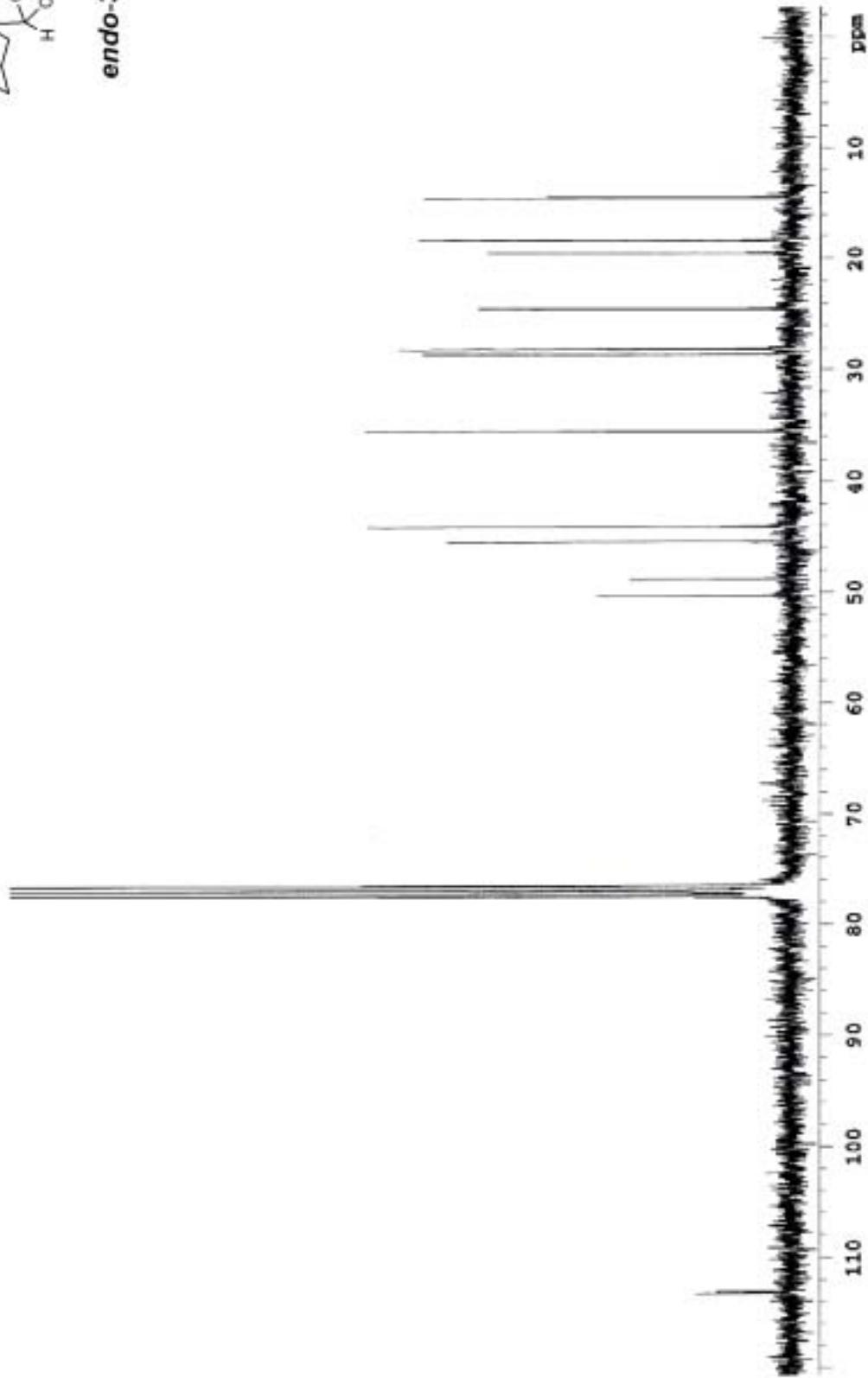


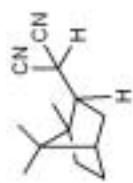
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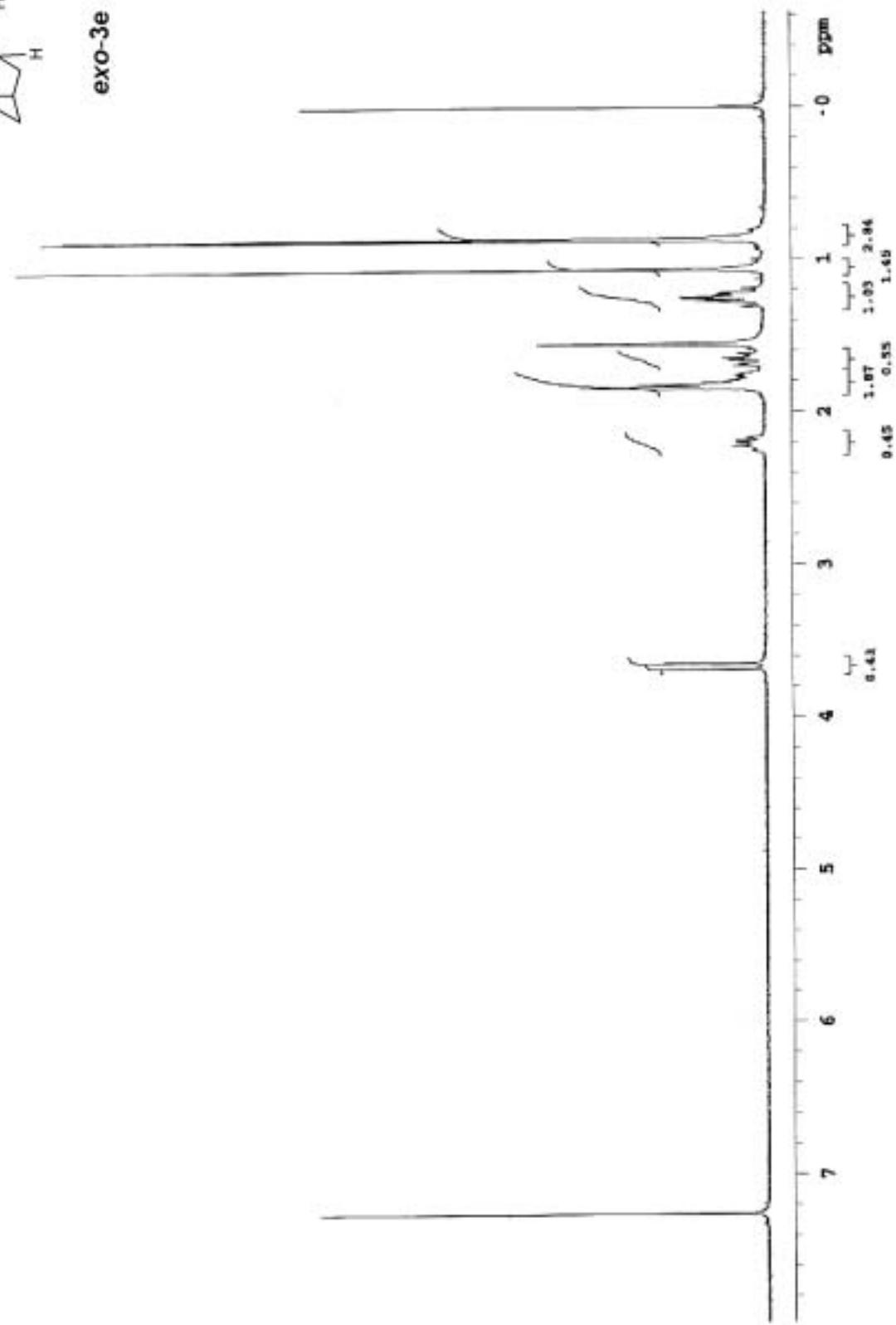


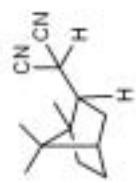
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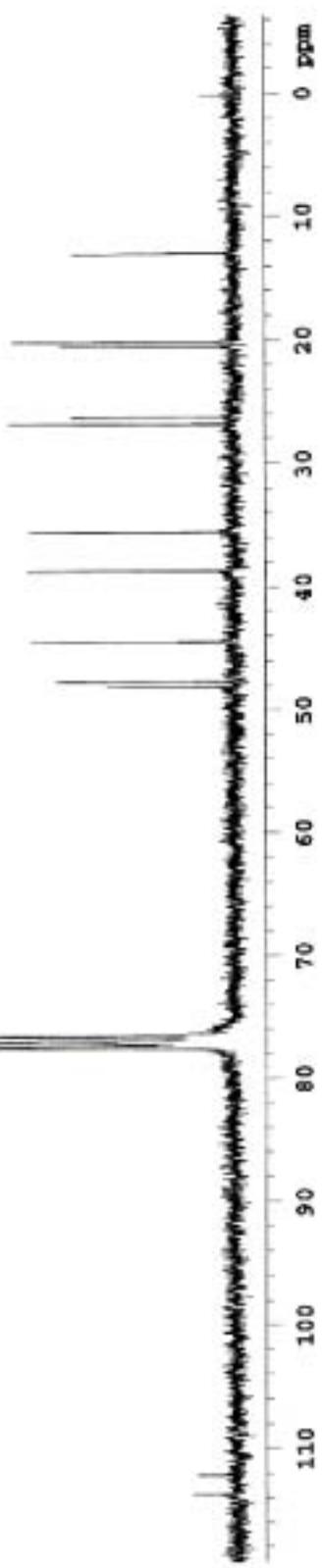
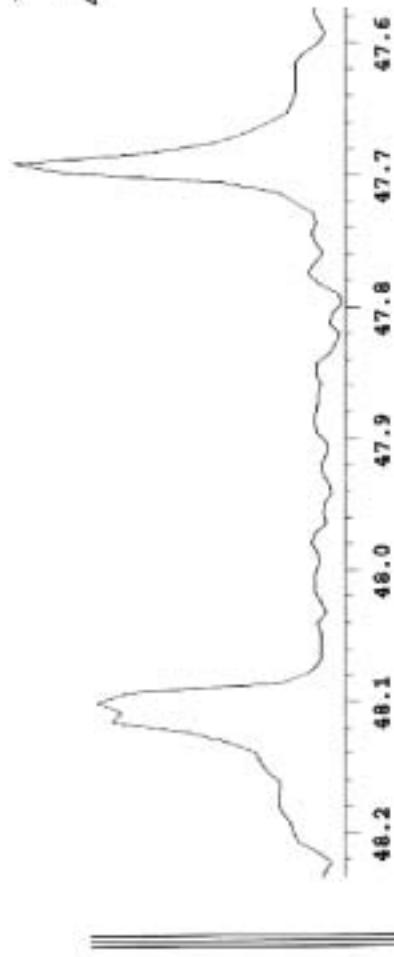


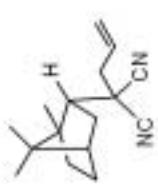
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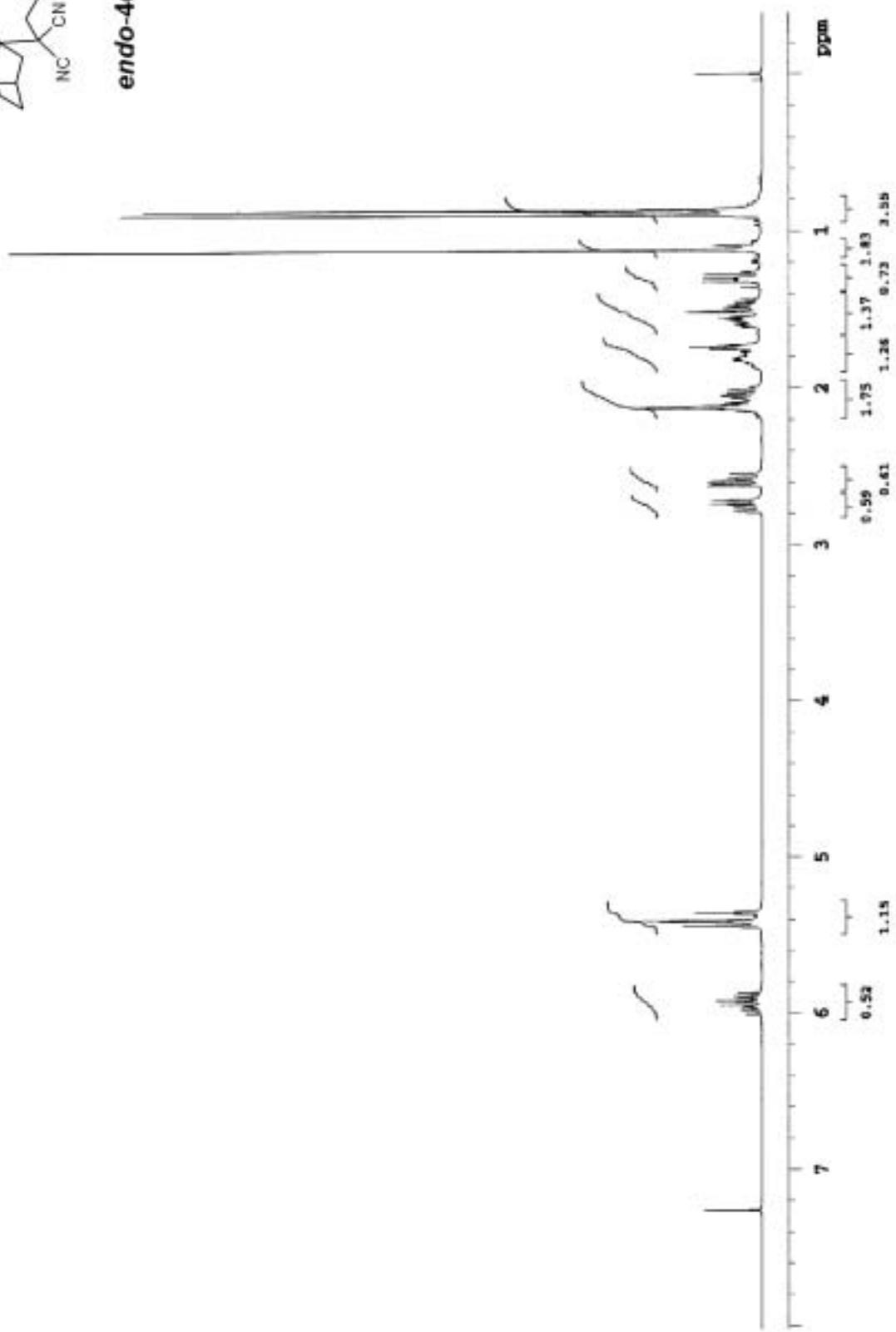


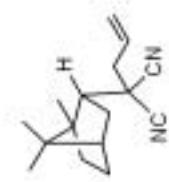
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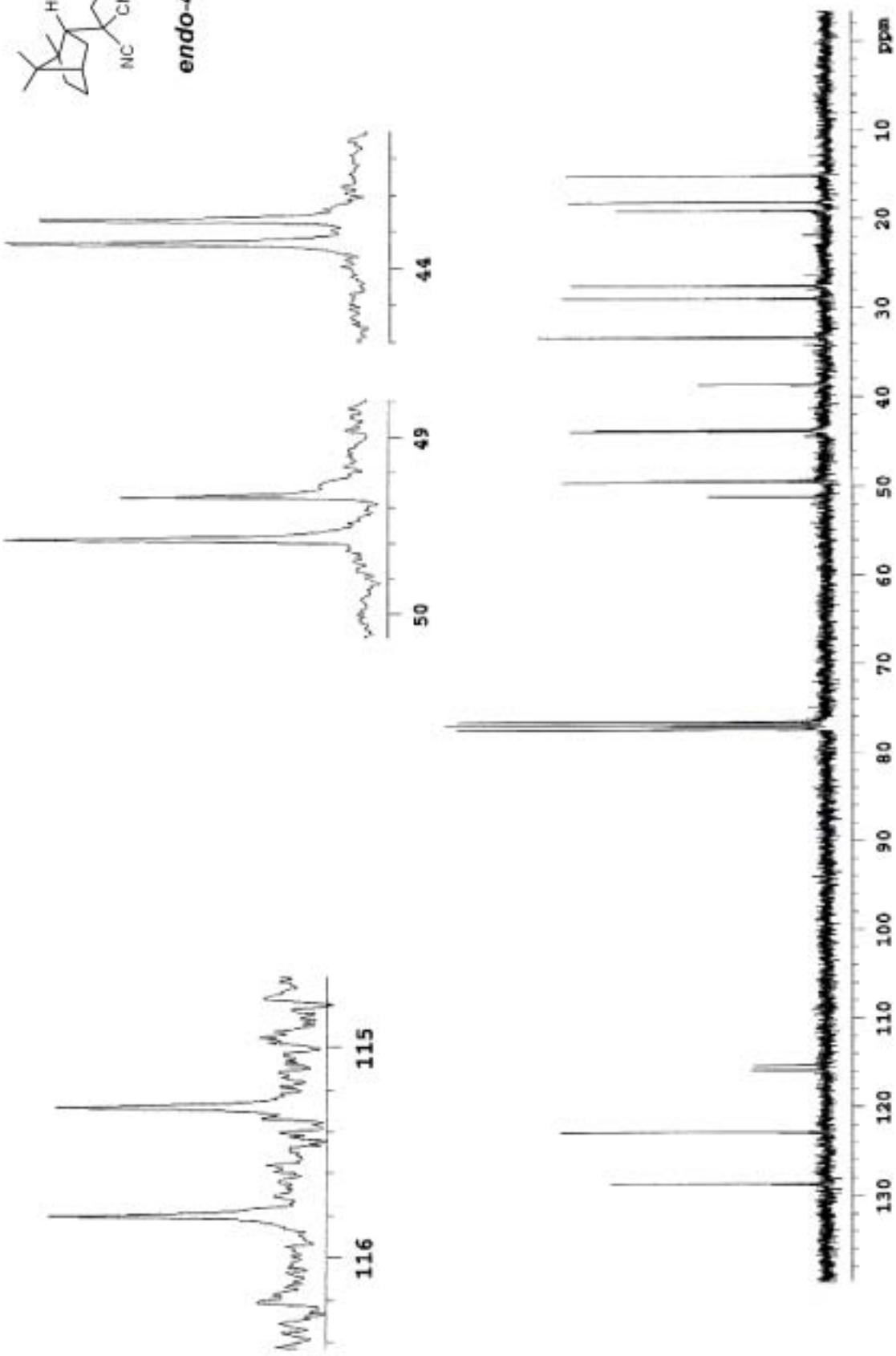


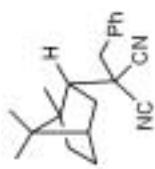
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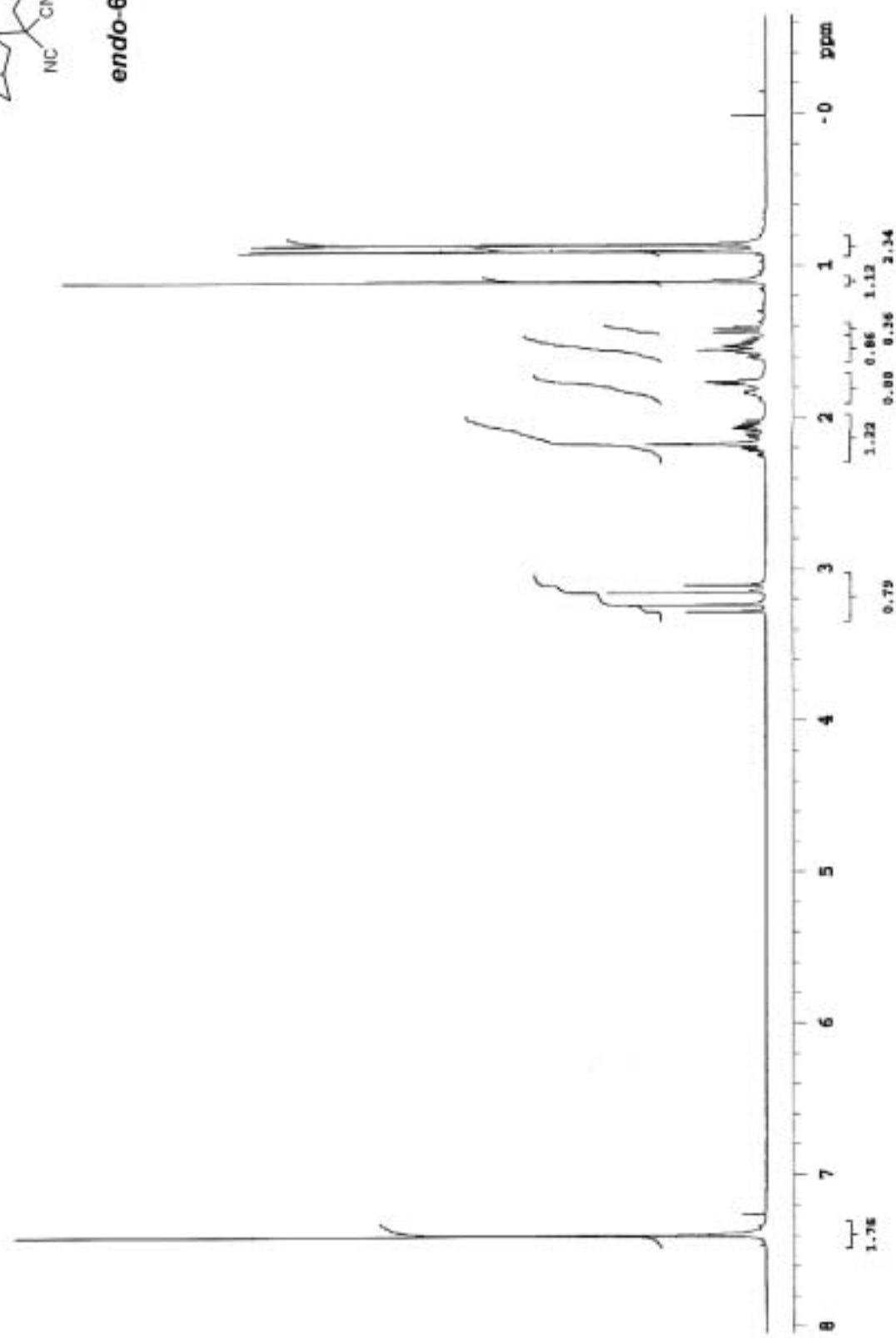


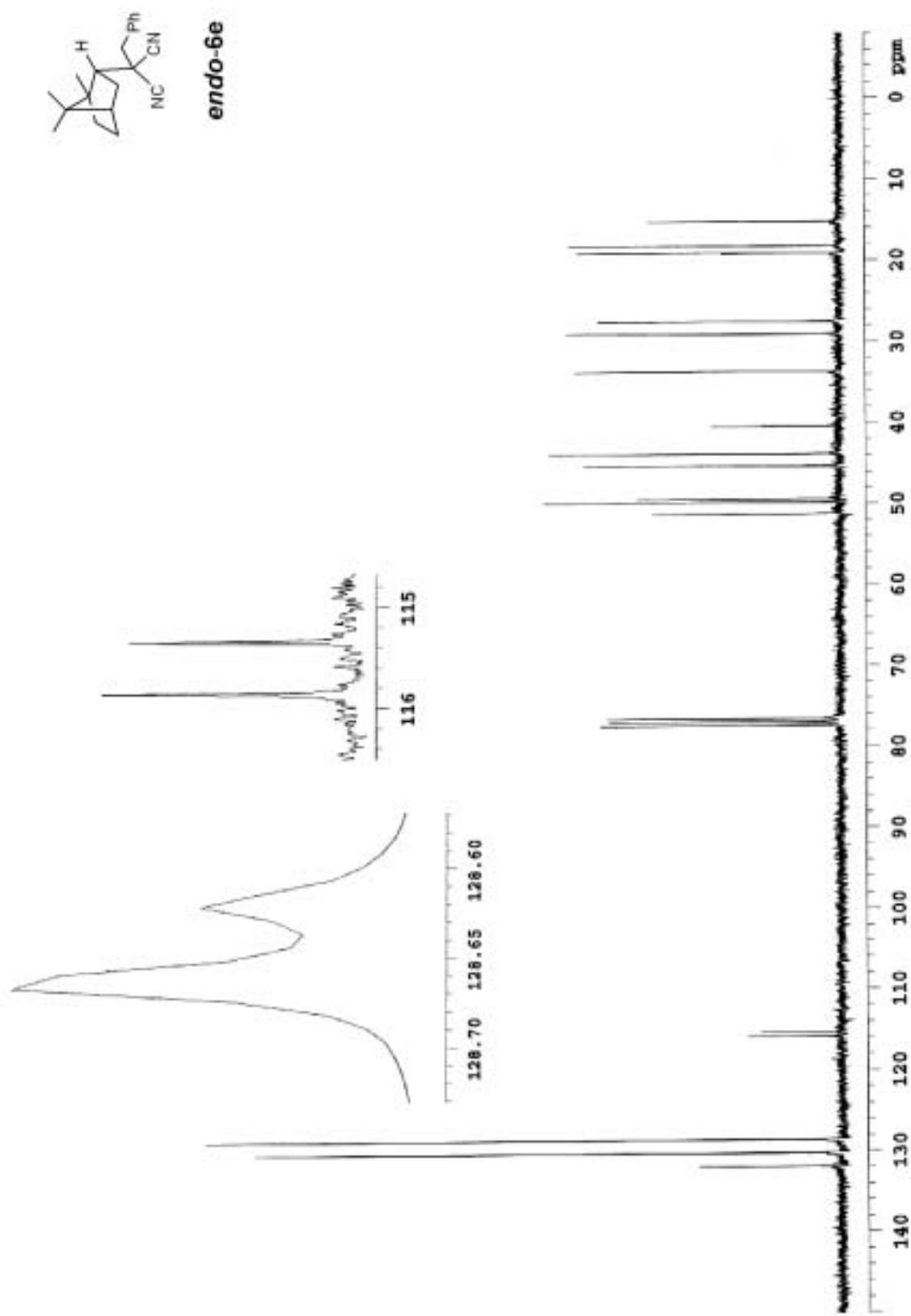
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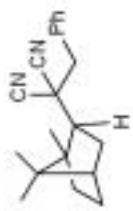




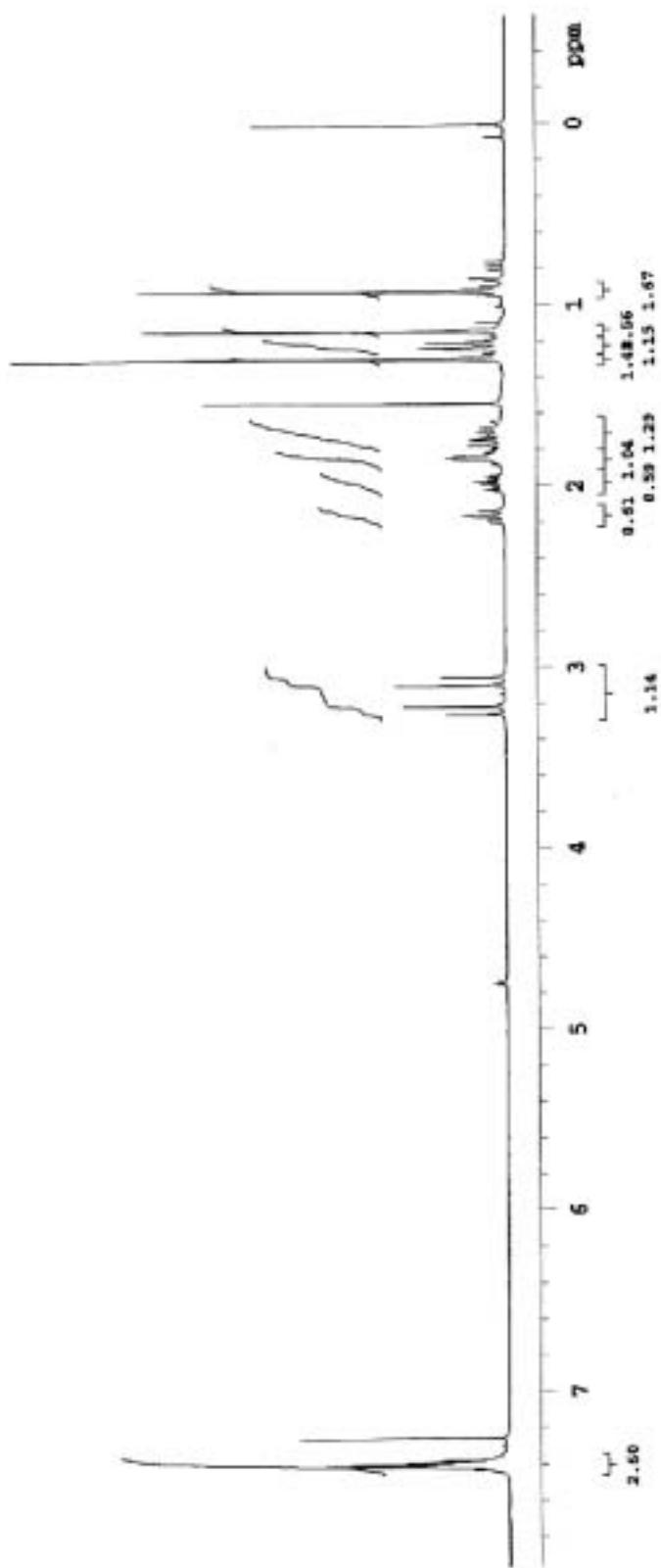
endo-*oppo*

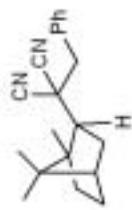
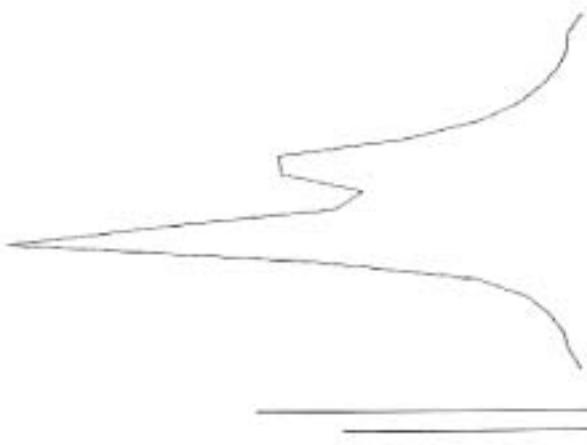




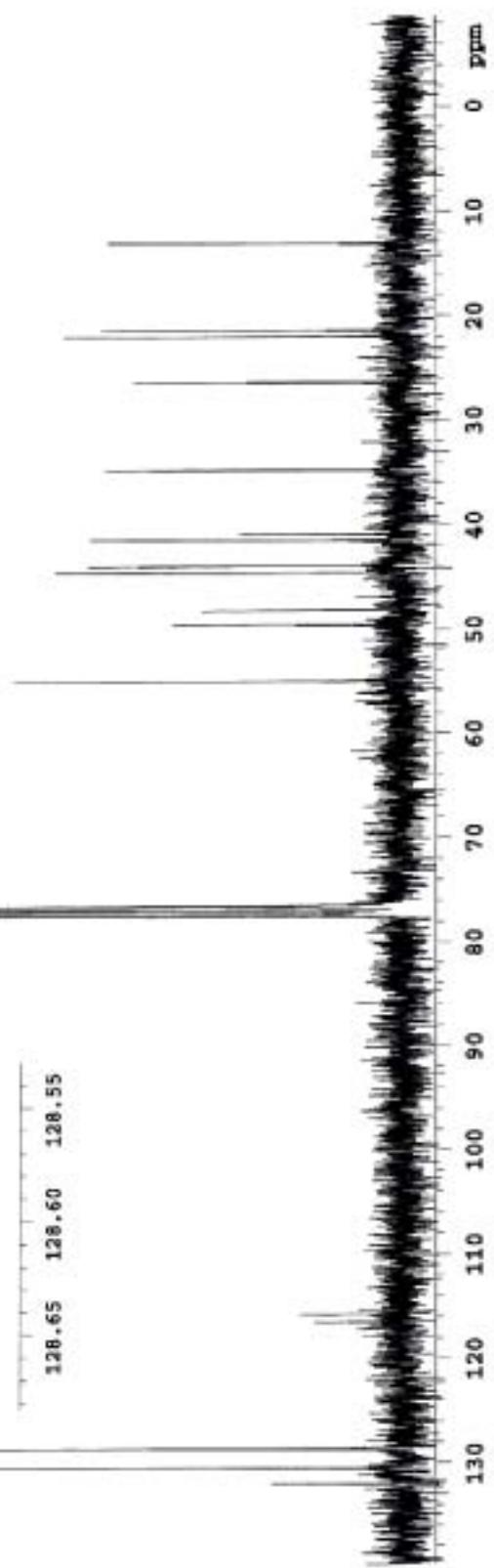


exo-6e





exo-6e



X-ray crystallographic Data of *cis*-3a

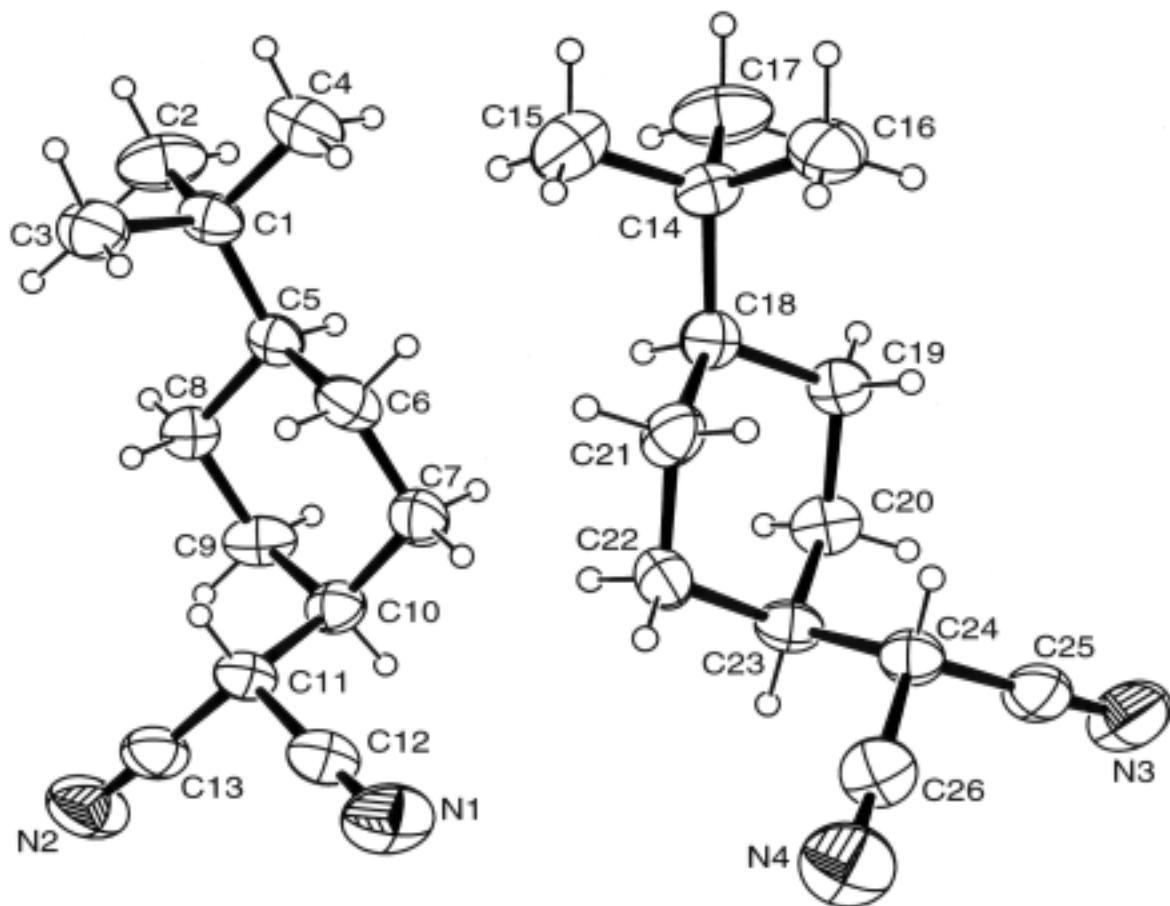


Figure S1. ORTEP drawing of *cis*-3a (ellipsoids at 30% probability).

Experimental

Data Collection

A colorless prism crystal of C₁₃H₂₀N₂ having approximate dimensions of 0.80 x 0.35 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-K α radiation.

Indexing was performed from 3° oscillations that were exposed for 180 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions:

$$\begin{array}{ll} a = & 8.3392(3) \text{ \AA} \\ b = & 11.6677(4) \text{ \AA} \\ c = & 15.175(2) \text{ \AA} \\ V = & 1353.5(2) \text{ \AA}^3 \end{array} \quad \begin{array}{ll} \alpha = & 103.712(6)^\circ \\ \beta = & 99.04(1)^\circ \\ \gamma = & 104.228(5)^\circ \end{array}$$

For Z = 4 and F.W. = 204.31, the calculated density is 1.00 g/cm³. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P-1 (#2)

The data were collected at a temperature of 23 ± 1°C to a maximum 2θ value of 55.0°. A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0° in 5.0° step, at χ=45.0° and φ = 0.0°. The exposure rate was 300.0 [sec./°]. A second sweep was performed using ω scans from 0.0 to 160.0° in 5.0° step, at χ=45.0° and φ = 180.0°. The exposure rate was 300.0 [sec./°]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

A total of 5113 reflections was collected.

The linear absorption coefficient, μ , for Mo-K α radiation is 0.6 cm^{-1} . An empirical absorption correction was applied which resulted in transmission factors ranging from 0.65 to 1.46. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement³ on F was based on 2515 observed reflections ($I > 3.60\sigma(I)$) and 311 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \sum ||F_O| - |F_C|| / \sum |F_O| = 0.100$$

$$R_w = [\sum w (|F_O| - |F_C|)^2 / \sum w F_O^2]^{1/2} = 0.120$$

The standard deviation of an observation of unit weight⁴ was 1.03. A Sheldrick weighting scheme was used. Plots of $\sum w (|F_O| - |F_C|)^2$ versus $|F_O|$, reflection order in data collection, $\sin \theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.62 and -0.55 e $^-$ /Å 3 , respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in F_{calc} ⁶; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbell⁸. All calculations were performed using the CrystalStructure^{9,10} crystallographic software package.

References

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Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$$\sum w(|F_o| - |F_c|)^2 \quad \text{where } w = \text{Least Squares weights.}$$

(4) Standard deviation of an observation of unit weight:

$$[\sum w(|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$$

where: N_o = number of observations
 N_v = number of variables

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(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

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EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₁₃ H ₂₀ N ₂
Formula Weight	204.31
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.80 X 0.35 X 0.10 mm
Crystal System	triclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 180.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 8.3392(3) Å b = 11.6677(4) Å c = 15.175(2) Å α = 103.712(6) ° β = 99.04(1) ° γ = 104.228(5) ° V = 1353.5(2) Å ³
Space Group	P-1 (#2)
Z value	4
D _{calc}	1.003 g/cm ³

F_{000}

448.00

$\mu(\text{MoK}\alpha)$

0.59 cm⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71069 \text{ \AA}$) graphite monochromated
Detector Aperture	270 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0, \phi=0.0$)	130.0 - 190.0°
Exposure Rate	300.0 sec./°
ω oscillation Range ($\chi=45.0, \phi=180.0$)	0.0 - 160.0°
Exposure Rate	300.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	55.0°
No. of Reflections Measured	Total: 5113
Corrections	Lorentz-polarization Absorption (trans. factors: 0.6519 - 1.4612)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F
Function Minimized	$\Sigma w (F_o - F_c)^2$
Least Squares Weights	$1/[0.0007F_o^2 + 1.0000\sigma(F_o^2) + 0.0900]$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 3.60\sigma(I)$)	2515
No. Variables	311
Reflection/Parameter Ratio	8.09
Residuals: R ($I > 3.60\sigma(I)$)	0.100
Residuals: R _w ($I > 3.60\sigma(I)$)	0.120
Goodness of Fit Indicator	1.033
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.62 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.55 e ⁻ /Å ³

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$

atom	x	y	z	B_{eq}
N(1)	0.5616	0.3098	0.0153	8.757
N(2)	0.3237	0.0026	0.1103	9.590
N(3)	0.8173	1.0047	0.1119	9.538
N(4)	0.8335	0.7048	-0.1154	8.912
C(23)	0.8777	0.7175	0.1201	4.974
C(5)	0.9161	0.2992	0.3804	4.527
C(8)	0.7535	0.1883	0.3496	4.717
C(26)	0.8785	0.7540	-0.0372	6.075
C(19)	1.1147	0.8193	0.2710	4.630
C(21)	1.1518	0.6517	0.1445	5.535
C(24)	0.9337	0.8132	0.0662	4.749
C(14)	1.3785	0.7378	0.3026	4.903
C(7)	0.7606	0.3980	0.2721	6.172
C(1)	1.0796	0.2683	0.4178	5.190
C(20)	0.9218	0.7795	0.2267	5.616
C(25)	0.8671	0.9208	0.0899	6.135
C(18)	1.1894	0.7090	0.2509	4.586
C(11)	0.5832	0.1980	0.1485	4.835
C(10)	0.5982	0.2930	0.2452	5.198
C(22)	0.9613	0.6139	0.0997	5.797
C(6)	0.9207	0.3596	0.3002	5.672
C(12)	0.5702	0.2599	0.0708	5.927
C(13)	0.4352	0.0870	0.1251	6.356
C(9)	0.5933	0.2269	0.3237	5.794
C(4)	1.2366	0.3840	0.4451	7.554
C(17)	1.3916	0.7738	0.4088	8.764
C(16)	1.4948	0.8433	0.2765	7.627
C(3)	1.1143	0.1627	0.3454	7.438
C(2)	1.0668	0.2246	0.5073	8.642
C(15)	1.4437	0.6239	0.2782	7.757
H(1)	1.0500	0.2886	0.5535	10.718
H(2)	0.9728	0.1525	0.4924	10.718
H(3)	1.1680	0.2070	0.5304	10.720
H(4)	1.0228	0.0893	0.3313	8.440
H(5)	1.1241	0.1857	0.2900	8.436
H(6)	1.2169	0.1487	0.3708	8.434
H(7)	1.3363	0.3653	0.4690	8.178

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$ (continued)

atom	x	y	z	B_{eq}
H(8)	1.2497	0.4114	0.3918	8.176
H(9)	1.2183	0.4471	0.4914	8.181
H(10)	0.8987	0.3573	0.4307	5.261
H(11)	0.7638	0.1317	0.2959	5.822
H(12)	0.7441	0.1497	0.3978	5.834
H(13)	0.4977	0.1553	0.3023	7.037
H(14)	0.5818	0.2806	0.3786	7.041
H(15)	1.0188	0.4293	0.3180	6.262
H(16)	0.9284	0.3004	0.2472	6.260
H(17)	0.7707	0.4325	0.2218	7.000
H(18)	0.7567	0.4592	0.3249	7.006
H(19)	0.5043	0.3254	0.2388	6.323
H(20)	0.6840	0.1735	0.1530	5.526
H(21)	1.5060	0.7934	0.4419	10.516
H(22)	1.3514	0.8435	0.4254	10.515
H(23)	1.3231	0.7066	0.4240	10.517
H(24)	1.3745	0.5582	0.2947	9.757
H(25)	1.4389	0.5993	0.2133	9.756
H(26)	1.5578	0.6442	0.3120	9.751
H(27)	1.6079	0.8620	0.3112	8.977
H(28)	1.4918	0.8192	0.2118	8.973
H(29)	1.4563	0.9142	0.2911	8.976
H(30)	1.1239	0.6483	0.2735	5.641
H(31)	1.1731	0.8792	0.2450	5.517
H(32)	1.1337	0.8538	0.3364	5.520
H(33)	0.8845	0.8512	0.2362	6.963
H(34)	0.8638	0.7249	0.2567	6.967
H(35)	1.2102	0.7124	0.1193	6.848
H(36)	1.1935	0.5821	0.1319	6.838
H(37)	0.9068	0.5491	0.1225	6.497
H(38)	0.9458	0.5843	0.0340	6.496
H(39)	0.7581	0.6823	0.0992	5.785
H(40)	1.0543	0.8431	0.0831	5.547

$$B_{\text{eq}} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
N(1)	0.1155	0.1482	0.0733	0.0210	0.0211	0.0586
N(2)	0.1176	0.1083	0.1031	-0.0211	-0.0135	0.0467
N(3)	0.1712	0.1132	0.0738	0.0864	-0.0102	-0.0021
N(4)	0.1458	0.1341	0.0578	0.0445	0.0313	0.0176
C(23)	0.0433	0.0806	0.0595	0.0125	0.0094	0.0173
C(5)	0.0585	0.0639	0.0440	0.0178	0.0141	0.0039
C(8)	0.0600	0.0727	0.0518	0.0165	0.0197	0.0263
C(26)	0.0841	0.0897	0.0621	0.0303	0.0251	0.0206
C(19)	0.0677	0.0693	0.0378	0.0227	0.0156	0.0085
C(21)	0.0883	0.0707	0.0577	0.0414	0.0139	0.0135
C(24)	0.0641	0.0668	0.0451	0.0179	0.0085	0.0117
C(14)	0.0568	0.0775	0.0568	0.0235	0.0111	0.0256
C(7)	0.0954	0.0671	0.0593	0.0169	-0.0002	0.0136
C(1)	0.0615	0.0802	0.0452	0.0150	0.0116	0.0056
C(20)	0.0646	0.0945	0.0616	0.0284	0.0241	0.0245
C(25)	0.0961	0.0768	0.0494	0.0331	-0.0033	0.0031
C(18)	0.0601	0.0630	0.0555	0.0142	0.0185	0.0256
C(11)	0.0556	0.0717	0.0480	0.0101	0.0053	0.0150
C(10)	0.0585	0.0775	0.0643	0.0292	0.0145	0.0158
C(22)	0.0860	0.0528	0.0668	0.0097	-0.0006	0.0131
C(6)	0.0656	0.0724	0.0601	-0.0045	0.0040	0.0195
C(12)	0.0658	0.0904	0.0564	0.0107	0.0071	0.0152
C(13)	0.0825	0.0858	0.0610	0.0084	-0.0028	0.0280
C(9)	0.0587	0.1162	0.0480	0.0270	0.0177	0.0239
C(4)	0.0718	0.1019	0.0853	0.0060	0.0047	0.0040
C(17)	0.0906	0.1851	0.0572	0.0513	0.0066	0.0305
C(16)	0.0570	0.1040	0.1231	0.0097	0.0108	0.0433
C(3)	0.0705	0.1034	0.0932	0.0405	0.0077	-0.0085
C(2)	0.0871	0.1742	0.0780	0.0399	0.0112	0.0603
C(15)	0.0922	0.1091	0.1075	0.0475	0.0156	0.0437

The general temperature factor expression: $\exp(-2\pi^2(a^*2U_{11}h^2 + b^*2U_{22}k^2 + c^*2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
N(1)	C(12)	1.135612	N(2)	C(13)	1.124032
N(3)	C(25)	1.153742	N(4)	C(26)	1.145458
C(23)	C(24)	1.559565	C(23)	C(20)	1.549631
C(23)	C(22)	1.531615	C(5)	C(8)	1.543382
C(5)	C(1)	1.551496	C(5)	C(6)	1.544621
C(8)	C(9)	1.535208	C(26)	C(24)	1.499940
C(19)	C(20)	1.549188	C(19)	C(18)	1.551109
C(21)	C(18)	1.542192	C(21)	C(22)	1.534093
C(24)	C(25)	1.485354	C(14)	C(18)	1.562033
C(14)	C(17)	1.543313	C(14)	C(16)	1.542067
C(14)	C(15)	1.543671	C(7)	C(10)	1.508343
C(7)	C(6)	1.539687	C(1)	C(4)	1.550106
C(1)	C(3)	1.562668	C(1)	C(2)	1.569718
C(11)	C(10)	1.581781	C(11)	C(12)	1.524333
C(11)	C(13)	1.478797	C(10)	C(9)	1.564732

Table 4. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(23)	H(39)	0.949986	C(5)	H(10)	0.949955
C(8)	H(11)	0.950041	C(8)	H(12)	0.949964
C(19)	H(31)	0.949994	C(19)	H(32)	0.950029
C(21)	H(35)	0.950001	C(21)	H(36)	0.950016
C(24)	H(40)	0.949968	C(7)	H(17)	0.950016
C(7)	H(18)	0.950033	C(20)	H(33)	0.950023
C(20)	H(34)	0.950025	C(18)	H(30)	0.950031
C(11)	H(20)	0.949970	C(10)	H(19)	0.949968
C(22)	H(37)	0.949939	C(22)	H(38)	0.950010
C(6)	H(15)	0.949975	C(6)	H(16)	0.950101
C(9)	H(13)	0.950040	C(9)	H(14)	0.949989
C(4)	H(7)	0.949951	C(4)	H(8)	0.949950
C(4)	H(9)	0.950048	C(17)	H(21)	0.949982
C(17)	H(22)	0.950032	C(17)	H(23)	0.949985
C(16)	H(27)	0.949967	C(16)	H(28)	0.950045
C(16)	H(29)	0.949960	C(3)	H(4)	0.949953
C(3)	H(5)	0.950089	C(3)	H(6)	0.949978
C(2)	H(1)	0.950106	C(2)	H(2)	0.950012
C(2)	H(3)	0.950034	C(15)	H(24)	0.950060
C(15)	H(25)	0.950099	C(15)	H(26)	0.949922

Table 5. Bond angles ($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(24)	C(23)	C(20)	111.548612	C(24)	C(23)	C(22)	111.176156
C(20)	C(23)	C(22)	109.100356	C(8)	C(5)	C(1)	114.127378
C(8)	C(5)	C(6)	107.054541	C(1)	C(5)	C(6)	116.443739
C(9)	C(8)	C(5)	111.938187	C(24)	C(26)	N(4)	177.523881
C(20)	C(19)	C(18)	111.666563	C(18)	C(21)	C(22)	111.920304
C(25)	C(24)	C(23)	113.055946	C(25)	C(24)	C(26)	108.663507
C(23)	C(24)	C(26)	111.011499	C(18)	C(14)	C(17)	109.079223
C(18)	C(14)	C(16)	110.766880	C(18)	C(14)	C(15)	111.724846
C(17)	C(14)	C(16)	110.065845	C(17)	C(14)	C(15)	107.152143
C(16)	C(14)	C(15)	107.980804	C(10)	C(7)	C(6)	113.458017
C(4)	C(1)	C(3)	108.284773	C(4)	C(1)	C(2)	107.279338
C(4)	C(1)	C(5)	110.552452	C(3)	C(1)	C(2)	106.787828
C(3)	C(1)	C(5)	112.673881	C(2)	C(1)	C(5)	111.031369
C(23)	C(20)	C(19)	113.178138	N(3)	C(25)	C(24)	177.316983
C(19)	C(18)	C(21)	108.592116	C(19)	C(18)	C(14)	114.942192
C(21)	C(18)	C(14)	114.809396	C(10)	C(11)	C(12)	110.099502
C(10)	C(11)	C(13)	112.598201	C(12)	C(11)	C(13)	109.036989
C(9)	C(10)	C(7)	108.937648	C(9)	C(10)	C(11)	110.423015
C(7)	C(10)	C(11)	111.246069	C(23)	C(22)	C(21)	114.930665
C(5)	C(6)	C(7)	113.743270	N(1)	C(12)	C(11)	177.648182
N(2)	C(13)	C(11)	177.742555	C(8)	C(9)	C(10)	113.601510

Table 6. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(24)	C(23)	H(39)	107.480101	C(20)	C(23)	H(39)	108.949926
C(22)	C(23)	H(39)	108.504083	C(8)	C(5)	H(10)	105.652663
C(1)	C(5)	H(10)	106.793824	C(6)	C(5)	H(10)	105.946211
C(9)	C(8)	H(11)	108.346337	C(9)	C(8)	H(12)	109.894289
H(11)	C(8)	H(12)	109.466153	H(11)	C(8)	C(5)	107.296632
H(12)	C(8)	C(5)	109.828857	C(20)	C(19)	H(31)	109.541376
C(20)	C(19)	H(32)	109.493892	C(18)	C(19)	H(31)	107.194047
C(18)	C(19)	H(32)	109.441698	H(31)	C(19)	H(32)	109.457883
C(18)	C(21)	H(35)	107.494533	C(18)	C(21)	H(36)	108.919472
C(22)	C(21)	H(35)	108.653687	C(22)	C(21)	H(36)	110.323259
H(35)	C(21)	H(36)	109.464471	C(25)	C(24)	H(40)	107.757305
H(40)	C(24)	C(23)	107.426258	H(40)	C(24)	C(26)	108.794035
C(10)	C(7)	H(17)	109.405302	C(10)	C(7)	H(18)	108.699926
C(6)	C(7)	H(17)	109.291641	C(6)	C(7)	H(18)	106.435045
H(17)	C(7)	H(18)	109.460171	H(33)	C(20)	H(34)	109.456406
H(33)	C(20)	C(23)	106.964347	H(33)	C(20)	C(19)	108.302592
H(34)	C(20)	C(23)	110.090024	H(34)	C(20)	C(19)	108.776343
H(30)	C(18)	C(19)	105.579067	H(30)	C(18)	C(21)	105.503047
H(30)	C(18)	C(14)	106.550932	C(10)	C(11)	H(20)	107.278131
C(12)	C(11)	H(20)	108.954832	C(13)	C(11)	H(20)	108.795183
C(9)	C(10)	H(19)	109.597178	H(19)	C(10)	C(7)	108.935665
H(19)	C(10)	C(11)	107.670924	H(37)	C(22)	H(38)	109.460601
H(37)	C(22)	C(23)	108.753678	H(37)	C(22)	C(21)	107.065957
H(38)	C(22)	C(23)	108.013475	H(38)	C(22)	C(21)	108.534618
H(15)	C(6)	H(16)	109.451989	H(15)	C(6)	C(5)	108.675033
H(15)	C(6)	C(7)	110.041960	H(16)	C(6)	C(5)	107.551803
H(16)	C(6)	C(7)	107.281773	H(13)	C(9)	H(14)	109.458462
H(13)	C(9)	C(8)	108.805980	H(13)	C(9)	C(10)	107.799081
H(14)	C(9)	C(8)	107.461020	H(14)	C(9)	C(10)	109.665788
H(7)	C(4)	H(8)	109.477038	H(7)	C(4)	H(9)	109.474254
H(7)	C(4)	C(1)	110.504738	H(8)	C(4)	H(9)	109.469241
H(8)	C(4)	C(1)	109.367591	H(9)	C(4)	C(1)	108.528302
H(21)	C(17)	H(22)	109.473603	H(21)	C(17)	H(23)	109.470752
H(21)	C(17)	C(14)	110.673986	H(22)	C(17)	H(23)	109.472665
H(22)	C(17)	C(14)	108.698913	H(23)	C(17)	C(14)	109.030605
H(27)	C(16)	H(28)	109.466272	H(27)	C(16)	H(29)	109.469312
H(27)	C(16)	C(14)	109.309677	H(28)	C(16)	H(29)	109.480982

Table 6. Bond angles involving hydrogens ($^{\circ}$) -- continued

atom	atom	atom	angle	atom	atom	atom	angle
H(28)	C(16)	C(14)	110.037860	H(29)	C(16)	C(14)	109.061617
H(4)	C(3)	H(5)	109.460316	H(4)	C(3)	H(6)	109.479690
H(4)	C(3)	C(1)	109.332672	H(5)	C(3)	H(6)	109.463013
H(5)	C(3)	C(1)	109.613678	H(6)	C(3)	C(1)	109.477842
H(1)	C(2)	H(2)	109.467390	H(1)	C(2)	H(3)	109.477355
H(1)	C(2)	C(1)	108.525560	H(2)	C(2)	H(3)	109.467126
H(2)	C(2)	C(1)	109.278921	H(3)	C(2)	C(1)	110.604154
H(24)	C(15)	H(25)	109.477978	H(24)	C(15)	H(26)	109.478897
H(24)	C(15)	C(14)	109.234313	H(25)	C(15)	H(26)	109.460148
H(25)	C(15)	C(14)	109.786938	H(26)	C(15)	C(14)	109.388587

Table 7. Torsion Angles($^{\circ}$)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(20)	C(23)	C(24)	C(26)	-175.6(6)	C(20)	C(23)	C(24)	C(25)	-53.2(7)
C(20)	C(23)	C(24)	H(40)	65.6(8)	C(22)	C(23)	C(24)	C(26)	62.4(7)
C(22)	C(23)	C(24)	C(25)	-175.2(5)	C(22)	C(23)	C(24)	H(40)	-56.5(7)
H(39)	C(23)	C(24)	C(26)	-56.2(9)	H(39)	C(23)	C(24)	C(25)	66.2(8)
H(39)	C(23)	C(24)	H(40)	-175.1(8)	C(24)	C(23)	C(20)	C(19)	-71.8(8)
C(24)	C(23)	C(20)	H(33)	47.4(9)	C(24)	C(23)	C(20)	H(34)	166.2(8)
C(22)	C(23)	C(20)	C(19)	51.4(8)	C(22)	C(23)	C(20)	H(33)	170.6(7)
C(22)	C(23)	C(20)	H(34)	-70.6(9)	H(39)	C(23)	C(20)	C(19)	169.7(8)
H(39)	C(23)	C(20)	H(33)	-71(1)	H(39)	C(23)	C(20)	H(34)	47(1)
C(24)	C(23)	C(22)	C(21)	72.3(7)	C(24)	C(23)	C(22)	H(37)	-167.7(6)
C(24)	C(23)	C(22)	H(38)	-49.0(8)	C(20)	C(23)	C(22)	C(21)	-51.1(8)
C(20)	C(23)	C(22)	H(37)	68.9(8)	C(20)	C(23)	C(22)	H(38)	-172.4(7)
H(39)	C(23)	C(22)	C(21)	-169.7(8)	H(39)	C(23)	C(22)	H(37)	-49(1)
H(39)	C(23)	C(22)	H(38)	68(1)	C(1)	C(5)	C(8)	C(9)	173.6(5)
C(1)	C(5)	C(8)	H(11)	-67.7(9)	C(1)	C(5)	C(8)	H(12)	51.2(9)
C(6)	C(5)	C(8)	C(9)	-56.1(7)	C(6)	C(5)	C(8)	H(11)	62.7(9)
C(6)	C(5)	C(8)	H(12)	-178.4(8)	H(10)	C(5)	C(8)	C(9)	56.5(9)
H(10)	C(5)	C(8)	H(11)	175.3(9)	H(10)	C(5)	C(8)	H(12)	-65(1)
C(8)	C(5)	C(1)	C(4)	178.9(6)	C(8)	C(5)	C(1)	C(3)	57.5(8)
C(8)	C(5)	C(1)	C(2)	-62.2(7)	C(6)	C(5)	C(1)	C(4)	53.3(7)
C(6)	C(5)	C(1)	C(3)	-68.0(7)	C(6)	C(5)	C(1)	C(2)	172.2(5)
H(10)	C(5)	C(1)	C(4)	-64.8(9)	H(10)	C(5)	C(1)	C(3)	173.9(7)
H(10)	C(5)	C(1)	C(2)	54.2(9)	C(8)	C(5)	C(6)	C(7)	55.9(7)
C(8)	C(5)	C(6)	H(15)	178.9(8)	C(8)	C(5)	C(6)	H(16)	-62.7(8)
C(1)	C(5)	C(6)	C(7)	-175.0(5)	C(1)	C(5)	C(6)	H(15)	-52(1)
C(1)	C(5)	C(6)	H(16)	66.3(8)	H(10)	C(5)	C(6)	C(7)	-56.5(7)
H(10)	C(5)	C(6)	H(15)	66(1)	H(10)	C(5)	C(6)	H(16)	-175.1(8)
C(5)	C(8)	C(9)	C(10)	57.0(7)	C(5)	C(8)	C(9)	H(13)	177.1(7)
C(5)	C(8)	C(9)	H(14)	-64(1)	H(11)	C(8)	C(9)	C(10)	-61.1(9)
H(11)	C(8)	C(9)	H(13)	58(1)	H(11)	C(8)	C(9)	H(14)	177.4(9)
H(12)	C(8)	C(9)	C(10)	179.3(6)	H(12)	C(8)	C(9)	H(13)	-60.6(9)
H(12)	C(8)	C(9)	H(14)	57(1)	C(18)	C(19)	C(20)	C(23)	-56.5(8)
C(18)	C(19)	C(20)	H(33)	-174.9(8)	C(18)	C(19)	C(20)	H(34)	66.2(9)
H(31)	C(19)	C(20)	C(23)	62.1(9)	H(31)	C(19)	C(20)	H(33)	-56(1)
H(31)	C(19)	C(20)	H(34)	-175.2(9)	H(32)	C(19)	C(20)	C(23)	-177.9(8)
H(32)	C(19)	C(20)	H(33)	63(1)	H(32)	C(19)	C(20)	H(34)	-55(1)
C(20)	C(19)	C(18)	C(21)	56.7(7)	C(20)	C(19)	C(18)	C(14)	-173.2(5)

Table 7. Torsion angles ($^{\circ}$) -- continued

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(20)	C(19)	C(18)	H(30)	-56.1(7)	H(31)	C(19)	C(18)	C(21)	-63.3(7)
H(31)	C(19)	C(18)	C(14)	66.8(8)	H(31)	C(19)	C(18)	H(30)	-176.1(7)
H(32)	C(19)	C(18)	C(21)	178.1(7)	H(32)	C(19)	C(18)	C(14)	-51.8(9)
H(32)	C(19)	C(18)	H(30)	65.3(8)	C(22)	C(21)	C(18)	C(19)	-55.6(7)
C(22)	C(21)	C(18)	C(14)	174.2(6)	C(22)	C(21)	C(18)	H(30)	57(1)
H(35)	C(21)	C(18)	C(19)	63(1)	H(35)	C(21)	C(18)	C(14)	-66(1)
H(35)	C(21)	C(18)	H(30)	176(1)	H(36)	C(21)	C(18)	C(19)	-177.8(7)
H(36)	C(21)	C(18)	C(14)	52.0(9)	H(36)	C(21)	C(18)	H(30)	-65(1)
C(18)	C(21)	C(22)	C(23)	55.2(9)	C(18)	C(21)	C(22)	H(37)	-65.7(9)
C(18)	C(21)	C(22)	H(38)	176.3(8)	H(35)	C(21)	C(22)	C(23)	-63(1)
H(35)	C(21)	C(22)	H(37)	175.8(9)	H(35)	C(21)	C(22)	H(38)	57(1)
H(36)	C(21)	C(22)	C(23)	176.7(8)	H(36)	C(21)	C(22)	H(37)	55(1)
H(36)	C(21)	C(22)	H(38)	-62(1)	C(17)	C(14)	C(18)	C(19)	62.3(8)
C(17)	C(14)	C(18)	C(21)	-170.7(6)	C(17)	C(14)	C(18)	H(30)	-54.3(8)
C(16)	C(14)	C(18)	C(19)	-59.0(7)	C(16)	C(14)	C(18)	C(21)	68.1(7)
C(16)	C(14)	C(18)	H(30)	-175.6(7)	C(15)	C(14)	C(18)	C(19)	-179.4(6)
C(15)	C(14)	C(18)	C(21)	-52.4(8)	C(15)	C(14)	C(18)	H(30)	64.0(8)
C(18)	C(14)	C(17)	H(21)	-178(1)	C(18)	C(14)	C(17)	H(22)	-58(1)
C(18)	C(14)	C(17)	H(23)	60(1)	C(16)	C(14)	C(17)	H(21)	-57(1)
C(16)	C(14)	C(17)	H(22)	63(1)	C(16)	C(14)	C(17)	H(23)	-177(1)
C(15)	C(14)	C(17)	H(21)	60(1)	C(15)	C(14)	C(17)	H(22)	-179(359)
C(15)	C(14)	C(17)	H(23)	-60(1)	C(18)	C(14)	C(16)	H(27)	177.0(9)
C(18)	C(14)	C(16)	H(28)	-62(1)	C(18)	C(14)	C(16)	H(29)	57(1)
C(17)	C(14)	C(16)	H(27)	56(1)	C(17)	C(14)	C(16)	H(28)	176.5(9)
C(17)	C(14)	C(16)	H(29)	-63(1)	C(15)	C(14)	C(16)	H(27)	-60(1)
C(15)	C(14)	C(16)	H(28)	59(1)	C(15)	C(14)	C(16)	H(29)	179(881)
C(18)	C(14)	C(15)	H(24)	-58(1)	C(18)	C(14)	C(15)	H(25)	61(1)
C(18)	C(14)	C(15)	H(26)	-178(1)	C(17)	C(14)	C(15)	H(24)	60.6(9)
C(17)	C(14)	C(15)	H(25)	-179.3(8)	C(17)	C(14)	C(15)	H(26)	-59(1)
C(16)	C(14)	C(15)	H(24)	179.2(8)	C(16)	C(14)	C(15)	H(25)	-60.8(9)
C(16)	C(14)	C(15)	H(26)	59(1)	C(6)	C(7)	C(10)	C(11)	-71.0(8)
C(6)	C(7)	C(10)	C(9)	51.0(8)	C(6)	C(7)	C(10)	H(19)	170.5(8)
H(17)	C(7)	C(10)	C(11)	51(1)	H(17)	C(7)	C(10)	C(9)	173.3(9)
H(17)	C(7)	C(10)	H(19)	-67(1)	H(18)	C(7)	C(10)	C(11)	170.8(9)
H(18)	C(7)	C(10)	C(9)	-67(1)	H(18)	C(7)	C(10)	H(19)	52(1)
C(10)	C(7)	C(6)	C(5)	-56.2(7)	C(10)	C(7)	C(6)	H(15)	-178.4(8)
C(10)	C(7)	C(6)	H(16)	62(1)	H(17)	C(7)	C(6)	C(5)	-178.6(6)

Table 7. Torsion angles ($^{\circ}$) -- continued

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
H(17)	C(7)	C(6)	H(15)	59(1)	H(17)	C(7)	C(6)	H(16)	-59(1)
H(18)	C(7)	C(6)	C(5)	63.2(9)	H(18)	C(7)	C(6)	H(15)	-58(1)
H(18)	C(7)	C(6)	H(16)	-177(1)	C(5)	C(1)	C(4)	H(7)	179.0(9)
C(5)	C(1)	C(4)	H(8)	-60(1)	C(5)	C(1)	C(4)	H(9)	58(1)
C(3)	C(1)	C(4)	H(7)	-57(1)	C(3)	C(1)	C(4)	H(8)	63(1)
C(3)	C(1)	C(4)	H(9)	-177.2(9)	C(2)	C(1)	C(4)	H(7)	57(1)
C(2)	C(1)	C(4)	H(8)	178.4(9)	C(2)	C(1)	C(4)	H(9)	-62(1)
C(5)	C(1)	C(3)	H(4)	-61(1)	C(5)	C(1)	C(3)	H(5)	58.4(9)
C(5)	C(1)	C(3)	H(6)	178.5(9)	C(4)	C(1)	C(3)	H(4)	175.8(9)
C(4)	C(1)	C(3)	H(5)	-64.2(9)	C(4)	C(1)	C(3)	H(6)	55(1)
C(2)	C(1)	C(3)	H(4)	60(1)	C(2)	C(1)	C(3)	H(5)	-179.4(7)
C(2)	C(1)	C(3)	H(6)	-59(1)	C(5)	C(1)	C(2)	H(1)	-59.0(9)
C(5)	C(1)	C(2)	H(2)	60(1)	C(5)	C(1)	C(2)	H(3)	-179.1(9)
C(4)	C(1)	C(2)	H(1)	61(1)	C(4)	C(1)	C(2)	H(2)	-178(1)
C(4)	C(1)	C(2)	H(3)	-58(1)	C(3)	C(1)	C(2)	H(1)	177.8(8)
C(3)	C(1)	C(2)	H(2)	-62(1)	C(3)	C(1)	C(2)	H(3)	57(1)
C(12)	C(11)	C(10)	C(7)	-64.5(8)	C(12)	C(11)	C(10)	C(9)	174.4(5)
C(12)	C(11)	C(10)	H(19)	54.7(9)	C(13)	C(11)	C(10)	C(7)	173.5(6)
C(13)	C(11)	C(10)	C(9)	52.5(8)	C(13)	C(11)	C(10)	H(19)	-67(1)
H(20)	C(11)	C(10)	C(7)	53(1)	H(20)	C(11)	C(10)	C(9)	-67.2(8)
H(20)	C(11)	C(10)	H(19)	173.2(9)	C(7)	C(10)	C(9)	C(8)	-52.3(7)
C(7)	C(10)	C(9)	H(13)	-172.9(8)	C(7)	C(10)	C(9)	H(14)	68.0(9)
C(11)	C(10)	C(9)	C(8)	70.2(6)	C(11)	C(10)	C(9)	H(13)	-50(1)
C(11)	C(10)	C(9)	H(14)	-169.6(7)	H(19)	C(10)	C(9)	C(8)	-171.4(6)
H(19)	C(10)	C(9)	H(13)	67(1)	H(19)	C(10)	C(9)	H(14)	-51.1(9)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

X-ray crystallographic Data of *trans*-6a

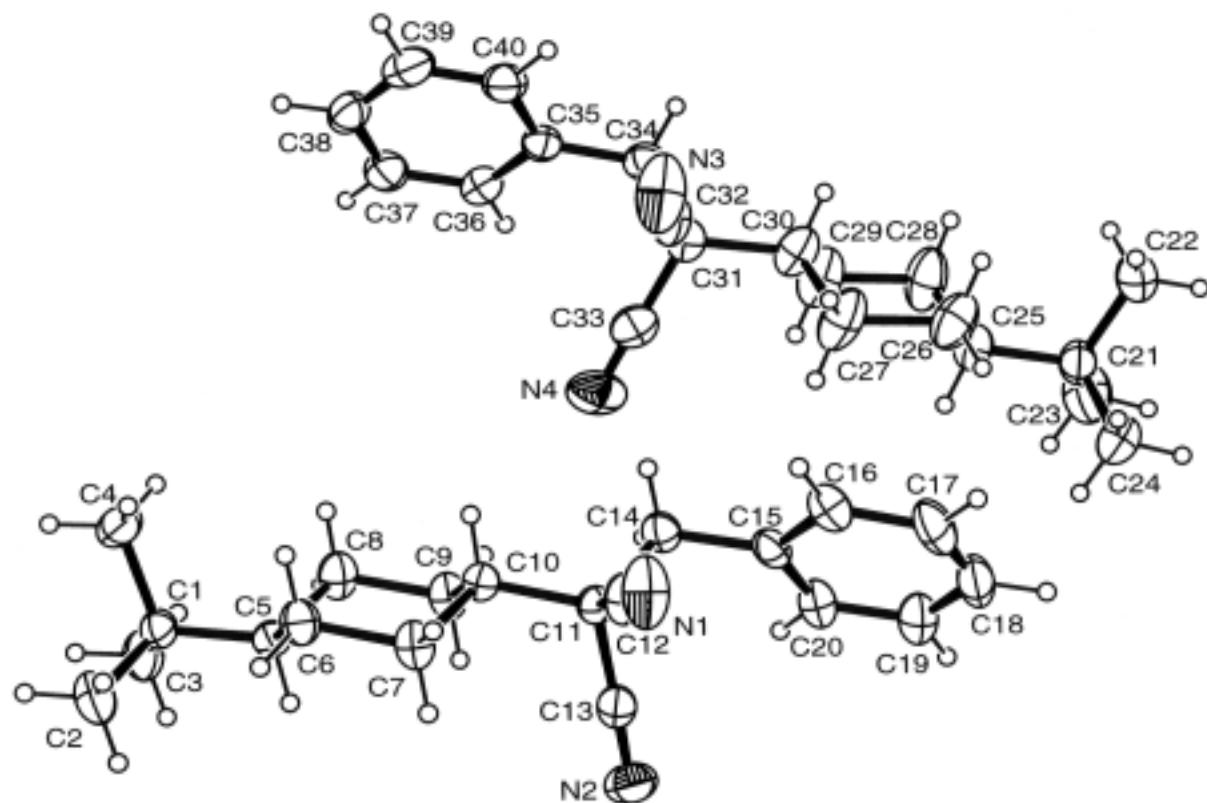


Figure S2. ORTEP drawing of *trans*-6a (ellipsoids at 30% probability).

Experimental

Data Collection

A colorless prism crystal of C₂₀H₂₆N₂ having approximate dimensions of 0.80 x 0.30 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-K α radiation.

Indexing was performed from 3° oscillations that were exposed for 180 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

$$\begin{aligned}a &= 16.7104(3) \text{ \AA} \\b &= 6.4424(1) \text{ \AA} & \beta &= 110.457(2)^\circ \\c &= 17.8094(4) \text{ \AA} \\V &= 1796.36(6) \text{ \AA}^3\end{aligned}$$

For Z = 4 and F.W. = 294.44, the calculated density is 1.09 g/cm³. Based on the systematic absences of:

$$0k0: k \pm 2n$$

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$$P2_1 (\#4)$$

The data were collected at a temperature of 23 ± 1°C to a maximum 2θ value of 55.0°. A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0° in 5.0° step, at χ=45.0° and φ = 0.0°. The exposure rate was 300.0 [sec./°]. A second sweep was performed using ω scans from 0.0 to 160.0° in 5.0° step, at χ=45.0° and φ = 180.0°. The exposure rate was 300.0 [sec./°]. The

crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 4342 reflections that were collected, 4338 were unique ($R_{\text{int}} = 0.049$); equivalent reflections were merged.

The linear absorption coefficient, μ , for Mo-K α radiation is 0.6 cm $^{-1}$. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement³ on F was based on 3823 observed reflections ($I > 0.50\sigma(I)$) and 449 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \sum ||F_O| - |F_C|| / \sum |F_O| = 0.079$$

$$R_w = [\sum w (|F_O| - |F_C|)^2 / \sum w F_O^2]^{1/2} = 0.136$$

The standard deviation of an observation of unit weight⁴ was 1.03. A Sheldrick weighting scheme was used. Plots of $\sum w (|F_O| - |F_C|)^2$ versus $|F_O|$, reflection order in data collection, $\sin \theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.95 and -0.47 e $^-/\text{\AA}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in F_{calc} ⁶; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbell⁸. All calculations were performed using the CrystalStructure^{9,10} crystallographic software package.

References

- (1) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.
- (2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
- (3) Least Squares function minimized:
- $$\sum w(|F_o| - |F_c|)^2 \quad \text{where } w = \text{Least Squares weights.}$$
- (4) Standard deviation of an observation of unit weight:
- $$[\sum w(|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$$
- where: N_o = number of observations
 N_v = number of variables
- (5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).
- (6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).
- (7) Creagh, D. C. & McAuley, W.J.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).
- (8) Creagh, D. C. & Hubbell, J.H.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).
- (9) CrystalStructure 3.5.1: Crystal Structure Analysis Package, Rigaku and Rigaku/MSC (2000-2003). 9009 New Trails Dr. The Woodlands TX 77381 USA.
- (10) CRYSTALS Issue 10: Watkin, D.J., Prout, C.K. Carruthers, J.R. & Betteridge, P.W. Chemical Crystallography Laboratory, Oxford, UK. (1996)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₂₀ H ₂₆ N ₂
Formula Weight	294.44
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.80 X 0.30 X 0.20 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 180.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 16.7104(3) Å b = 6.4424(1) Å c = 17.8094(4) Å β = 110.457(2) ° V = 1796.36(6) Å ³
Space Group	P2 ₁ (#4)
Z value	4
D _{calc}	1.089 g/cm ³
F ₀₀₀	640.00

$\mu(\text{MoK}\alpha)$

0.64 cm⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71069 \text{ \AA}$) graphite monochromated
Detector Aperture	270 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0, \phi=0.0$)	130.0 - 190.0°
Exposure Rate	300.0 sec./°
ω oscillation Range ($\chi=45.0, \phi=180.0$)	0.0 - 160.0°
Exposure Rate	300.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	55.0°
No. of Reflections Measured	Total: 4342
Corrections	Unique: 4338 ($R_{\text{int}} = 0.049$) Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F
Function Minimized	$\Sigma w (F_o - F_c)^2$
Least Squares Weights	$1/[0.0010F_o^2 + 1.0000\sigma(F_o)^2 + 0.1000]$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 0.50\sigma(I)$)	3823
No. Variables	449
Reflection/Parameter Ratio	8.51
Residuals: R ($I > 0.50\sigma(I)$)	0.094
Residuals: R ($I > 2.00\sigma(I)$)	0.079
Residuals: $R_w (I > 0.50\sigma(I))$	0.136
Goodness of Fit Indicator	1.030
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	$0.95 \text{ e}^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-0.47 \text{ e}^-/\text{\AA}^3$

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$

atom	x	y	z	B_{eq}
N(1)	1.4016	-0.7492	0.2798	6.819
N(2)	1.5370	-0.1892	0.3052	6.056
N(3)	1.0348	-0.4397	0.2213	8.985
N(4)	1.1613	0.1164	0.2049	7.658
C(5)	1.3708	-0.2095	-0.0016	3.509
C(7)	1.4056	-0.4574	0.1147	4.069
C(11)	1.3850	-0.3541	0.2449	3.349
C(14)	1.3228	-0.2602	0.2834	3.966
C(10)	1.3511	-0.3315	0.1521	3.368
C(13)	1.4714	-0.2603	0.2799	4.049
C(9)	1.3466	-0.1066	0.1258	4.330
C(8)	1.3150	-0.0890	0.0346	4.661
C(33)	1.1089	0.0485	0.2237	5.224
C(34)	0.9529	0.0520	0.2001	4.455
C(3)	1.3566	0.0543	-0.1091	5.236
C(15)	1.3507	-0.2812	0.3732	3.914
C(1)	1.3479	-0.1759	-0.0923	4.108
C(40)	0.8848	-0.1479	0.0724	4.949
C(31)	1.0415	-0.0414	0.2496	4.339
C(38)	0.8804	-0.0152	-0.0524	5.621
C(20)	1.3965	-0.1278	0.4222	4.952
C(35)	0.9267	0.0252	0.1115	4.215
C(12)	1.3954	-0.5804	0.2653	4.569
C(19)	1.4228	-0.1495	0.5048	5.765
C(16)	1.3323	-0.4586	0.4079	5.083
C(37)	0.9210	0.1616	-0.0158	5.342
C(36)	0.9441	0.1811	0.0669	4.613
C(39)	0.8619	-0.1682	-0.0087	5.877
C(4)	1.2569	-0.2481	-0.1406	6.113
C(6)	1.3724	-0.4338	0.0237	4.246
C(29)	1.0703	0.1959	0.3704	6.945
C(2)	1.4080	-0.2986	-0.1225	5.462
C(18)	1.4047	-0.3206	0.5388	6.111
C(21)	1.1892	0.1091	0.6010	5.060
C(25)	1.1648	0.0842	0.5104	5.213
C(26)	1.1558	-0.1356	0.4830	6.945
C(24)	1.2748	-0.0026	0.6440	6.742

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$ (continued)

atom	x	y	z	B_{eq}
C(22)	1.1228	0.0182	0.6320	6.667
C(27)	1.1422	-0.1441	0.3906	7.501
C(30)	1.0627	-0.0211	0.3428	5.494
C(17)	1.3616	-0.4744	0.4921	6.274
C(32)	1.0409	-0.2670	0.2330	5.620
C(28)	1.0844	0.2018	0.4611	7.814
C(23)	1.2030	0.3379	0.6246	8.400
H(1)	1.4031	-0.4423	-0.1130	6.947
H(2)	1.3949	-0.2759	-0.1782	6.946
H(3)	1.4647	-0.2538	-0.0940	6.947
H(4)	1.4131	0.0978	-0.0793	6.377
H(5)	1.3455	0.0750	-0.1646	6.377
H(6)	1.3172	0.1332	-0.0931	6.379
H(7)	1.2177	-0.1694	-0.1244	7.005
H(8)	1.2459	-0.2261	-0.1960	7.003
H(9)	1.2505	-0.3913	-0.1313	7.007
H(10)	1.4267	-0.1556	0.0235	4.177
H(11)	1.3168	-0.4912	0.0013	5.197
H(12)	1.4104	-0.5073	0.0042	5.198
H(13)	1.4630	-0.4102	0.1364	4.997
H(14)	1.4031	-0.5997	0.1277	5.000
H(15)	1.3125	0.0528	0.0193	5.571
H(16)	1.2594	-0.1475	0.0141	5.573
H(17)	1.3094	-0.0316	0.1459	5.318
H(18)	1.4023	-0.0485	0.1466	5.320
H(19)	1.2946	-0.3852	0.1323	4.031
H(20)	1.3162	-0.1166	0.2706	4.857
H(21)	1.2692	-0.3280	0.2609	4.858
H(22)	1.3012	-0.5687	0.3751	6.433
H(23)	1.3490	-0.5958	0.5161	8.118
H(24)	1.4226	-0.3332	0.5954	7.618
H(25)	1.4553	-0.0429	0.5387	6.981
H(26)	1.4104	-0.0063	0.3991	6.165
H(27)	1.1384	0.0413	0.6880	8.174
H(28)	1.0698	0.0841	0.6046	8.174
H(29)	1.1177	-0.1267	0.6215	8.174
H(30)	1.2246	0.3520	0.6812	10.257

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(31)	1.2453	0.3810	0.6035	10.257
H(32)	1.1535	0.4219	0.6028	10.253
H(33)	1.2654	-0.1466	0.6328	7.720
H(34)	1.3162	0.0465	0.6228	7.718
H(35)	1.2949	0.0187	0.7003	7.718
H(36)	1.2113	0.1407	0.4982	6.063
H(37)	1.2072	-0.2064	0.5117	7.625
H(38)	1.1094	-0.2005	0.4929	7.623
H(39)	1.1905	-0.0891	0.3808	8.309
H(40)	1.1347	-0.2854	0.3745	8.313
H(41)	1.0941	0.3419	0.4786	9.171
H(42)	1.0350	0.1509	0.4697	9.168
H(43)	1.1176	0.2575	0.3610	7.814
H(44)	1.0197	0.2700	0.3417	7.816
H(45)	1.0157	-0.0791	0.3539	6.188
H(46)	0.9110	-0.0173	0.2157	5.334
H(47)	0.9531	0.1955	0.2124	5.331
H(48)	0.9720	0.3041	0.0924	5.357
H(49)	0.9334	0.2694	-0.0464	6.379
H(50)	0.8660	-0.0292	-0.1087	6.548
H(51)	0.8328	-0.2888	-0.0353	6.775
H(52)	0.8714	-0.2550	0.1027	5.938

$$B_{eq} = \frac{8}{3} \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
N(1)	0.1557	0.0422	0.0650	0.0154	0.0433	0.0076
N(2)	0.0515	0.0907	0.0816	-0.0034	0.0154	-0.0219
N(3)	0.2048	0.0530	0.0762	0.0144	0.0398	0.0027
N(4)	0.0612	0.1002	0.1267	0.0226	0.0292	0.0396
C(5)	0.0434	0.0452	0.0439	-0.0027	0.0141	-0.0018
C(7)	0.0724	0.0386	0.0473	0.0128	0.0255	0.0030
C(11)	0.0455	0.0403	0.0411	0.0044	0.0147	0.0036
C(14)	0.0511	0.0519	0.0508	0.0113	0.0217	0.0046
C(10)	0.0461	0.0393	0.0423	0.0031	0.0151	-0.0007
C(13)	0.0555	0.0512	0.0474	0.0159	0.0184	0.0019
C(9)	0.0719	0.0454	0.0511	0.0133	0.0264	0.0051
C(8)	0.0716	0.0559	0.0490	0.0210	0.0202	0.0078
C(33)	0.0556	0.0611	0.0732	0.0188	0.0117	0.0101
C(34)	0.0480	0.0581	0.0628	0.0042	0.0187	-0.0057
C(3)	0.0925	0.0549	0.0545	-0.0027	0.0295	0.0096
C(15)	0.0497	0.0550	0.0518	0.0137	0.0275	0.0127
C(1)	0.0549	0.0590	0.0426	-0.0118	0.0175	0.0001
C(40)	0.0545	0.0655	0.0679	-0.0080	0.0211	-0.0040
C(31)	0.0628	0.0484	0.0530	0.0097	0.0193	-0.0002
C(38)	0.0502	0.1022	0.0549	0.0100	0.0106	-0.0123
C(20)	0.0807	0.0621	0.0524	0.0073	0.0320	0.0068
C(35)	0.0430	0.0629	0.0539	0.0050	0.0164	-0.0018
C(12)	0.0777	0.0528	0.0437	0.0118	0.0221	0.0006
C(19)	0.0887	0.0801	0.0521	0.0053	0.0270	-0.0006
C(16)	0.0758	0.0612	0.0667	-0.0004	0.0382	0.0062
C(37)	0.0572	0.0801	0.0646	0.0044	0.0199	0.0109
C(36)	0.0523	0.0578	0.0596	0.0010	0.0126	-0.0046
C(39)	0.0519	0.0794	0.0832	-0.0025	0.0126	-0.0236
C(4)	0.0630	0.1076	0.0512	-0.0280	0.0070	0.0074
C(6)	0.0666	0.0479	0.0500	0.0026	0.0244	-0.0042
C(29)	0.1245	0.0510	0.0718	0.0239	0.0135	0.0031
C(2)	0.0933	0.0683	0.0584	-0.0062	0.0421	-0.0016
C(18)	0.0881	0.1010	0.0521	0.0211	0.0359	0.0182
C(21)	0.0750	0.0692	0.0471	-0.0101	0.0200	-0.0001
C(25)	0.0729	0.0659	0.0531	0.0066	0.0144	0.0052
C(26)	0.1026	0.0713	0.0675	0.0075	0.0014	0.0124
C(24)	0.0788	0.1070	0.0587	-0.0032	0.0093	-0.0029

Table 2. Anisotropic Displacement Parameters (continued)

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(22)	0.0936	0.1045	0.0608	-0.0077	0.0340	-0.0038
C(27)	0.1052	0.0950	0.0631	0.0530	0.0020	-0.0099
C(30)	0.0744	0.0631	0.0584	0.0204	0.0071	-0.0082
C(17)	0.1010	0.0796	0.0765	0.0162	0.0544	0.0319
C(32)	0.1032	0.0541	0.0547	0.0130	0.0257	0.0046
C(28)	0.1310	0.0993	0.0600	0.0469	0.0251	-0.0070
C(23)	0.1545	0.0811	0.0892	-0.0183	0.0496	-0.0173

The general temperature factor expression: $\exp(-2\pi^2(a^*2U_{11}h^2 + b^*2U_{22}k^2 + c^*2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
N(1)	C(12)	1.11(1)	N(2)	C(13)	1.13(1)
N(3)	C(32)	1.13(1)	N(4)	C(33)	1.13(1)
C(5)	C(8)	1.52(1)	C(5)	C(1)	1.54(1)
C(5)	C(6)	1.51(1)	C(7)	C(10)	1.54(1)
C(7)	C(6)	1.53(1)	C(11)	C(14)	1.55(1)
C(11)	C(10)	1.555(9)	C(11)	C(13)	1.49(1)
C(11)	C(12)	1.50(1)	C(14)	C(15)	1.51(1)
C(10)	C(9)	1.52(1)	C(9)	C(8)	1.53(1)
C(33)	C(31)	1.48(1)	C(34)	C(31)	1.56(1)
C(34)	C(35)	1.49(1)	C(3)	C(1)	1.53(1)
C(15)	C(20)	1.36(1)	C(15)	C(16)	1.38(1)
C(1)	C(4)	1.53(1)	C(1)	C(2)	1.52(1)
C(40)	C(35)	1.37(1)	C(40)	C(39)	1.36(1)
C(31)	C(30)	1.58(1)	C(31)	C(32)	1.48(1)
C(38)	C(37)	1.37(1)	C(38)	C(39)	1.36(2)
C(20)	C(19)	1.39(1)	C(35)	C(36)	1.37(1)
C(19)	C(18)	1.34(2)	C(16)	C(17)	1.41(1)
C(37)	C(36)	1.39(1)	C(29)	C(30)	1.47(1)
C(29)	C(28)	1.55(1)	C(18)	C(17)	1.33(2)
C(21)	C(25)	1.53(1)	C(21)	C(24)	1.54(1)
C(21)	C(22)	1.52(2)	C(21)	C(23)	1.53(2)
C(25)	C(26)	1.49(2)	C(25)	C(28)	1.53(1)
C(26)	C(27)	1.58(1)	C(27)	C(30)	1.53(1)

Table 4. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(5)	H(10)	0.950(9)	C(7)	H(13)	0.95(1)
C(7)	H(14)	0.95(1)	C(14)	H(20)	0.95(1)
C(14)	H(21)	0.95(1)	C(10)	H(19)	0.95(1)
C(9)	H(17)	0.95(1)	C(9)	H(18)	0.95(1)
C(8)	H(15)	0.95(1)	C(8)	H(16)	0.95(1)
C(34)	H(46)	0.95(1)	C(34)	H(47)	0.95(1)
C(3)	H(4)	0.95(1)	C(3)	H(5)	0.95(1)
C(3)	H(6)	0.95(2)	C(40)	H(52)	0.95(1)
C(38)	H(50)	0.95(1)	C(20)	H(26)	0.95(1)
C(19)	H(25)	0.95(1)	C(16)	H(22)	0.95(1)
C(37)	H(49)	0.95(1)	C(36)	H(48)	0.95(1)
C(39)	H(51)	0.95(2)	C(4)	H(7)	0.95(1)
C(4)	H(8)	0.95(1)	C(4)	H(9)	0.95(2)
C(6)	H(11)	0.95(1)	C(6)	H(12)	0.95(1)
C(29)	H(43)	0.95(2)	C(29)	H(44)	0.95(2)
C(2)	H(1)	0.95(1)	C(2)	H(2)	0.95(1)
C(2)	H(3)	0.95(1)	C(18)	H(24)	0.95(1)
C(25)	H(36)	0.95(1)	C(26)	H(37)	0.95(2)
C(26)	H(38)	0.95(2)	C(24)	H(33)	0.95(2)
C(24)	H(34)	0.95(2)	C(24)	H(35)	0.95(1)
C(22)	H(27)	0.95(1)	C(22)	H(28)	0.95(1)
C(22)	H(29)	0.95(2)	C(27)	H(39)	0.95(2)
C(27)	H(40)	0.95(2)	C(30)	H(45)	0.95(1)
C(17)	H(23)	0.95(2)	C(28)	H(41)	0.95(2)
C(28)	H(42)	0.95(2)	C(23)	H(30)	0.95(2)
C(23)	H(31)	0.95(2)	C(23)	H(32)	0.95(2)

Table 5. Bond angles ($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(8)	C(5)	C(1)	114.2(6)	C(8)	C(5)	C(6)	108.1(7)
C(1)	C(5)	C(6)	114.8(6)	C(10)	C(7)	C(6)	111.0(6)
C(14)	C(11)	C(10)	112.3(5)	C(14)	C(11)	C(13)	111.0(6)
C(14)	C(11)	C(12)	108.1(7)	C(10)	C(11)	C(13)	110.3(6)
C(10)	C(11)	C(12)	108.5(6)	C(13)	C(11)	C(12)	106.3(6)
C(15)	C(14)	C(11)	115.2(6)	C(9)	C(10)	C(7)	110.1(7)
C(9)	C(10)	C(11)	112.2(6)	C(7)	C(10)	C(11)	111.1(5)
N(2)	C(13)	C(11)	178.8(9)	C(8)	C(9)	C(10)	111.2(6)
C(5)	C(8)	C(9)	112.2(6)	C(31)	C(33)	N(4)	179.0(9)
C(31)	C(34)	C(35)	115.1(7)	C(20)	C(15)	C(16)	118.4(7)
C(20)	C(15)	C(14)	120.7(8)	C(16)	C(15)	C(14)	120.9(7)
C(4)	C(1)	C(2)	107.1(7)	C(4)	C(1)	C(5)	112.3(7)
C(4)	C(1)	C(3)	109.1(7)	C(2)	C(1)	C(5)	110.2(6)
C(2)	C(1)	C(3)	108.5(8)	C(5)	C(1)	C(3)	109.6(6)
C(35)	C(40)	C(39)	121.4(9)	C(30)	C(31)	C(32)	106.0(7)
C(30)	C(31)	C(33)	112.5(7)	C(30)	C(31)	C(34)	112.6(8)
C(32)	C(31)	C(33)	106.0(9)	C(32)	C(31)	C(34)	109.1(7)
C(33)	C(31)	C(34)	110.4(7)	C(37)	C(38)	C(39)	120.4(8)
C(19)	C(20)	C(15)	120.3(9)	C(36)	C(35)	C(34)	118.9(7)
C(36)	C(35)	C(40)	118.3(7)	C(34)	C(35)	C(40)	122.7(8)
N(1)	C(12)	C(11)	178(1)	C(18)	C(19)	C(20)	121.6(9)
C(17)	C(16)	C(15)	119.1(8)	C(36)	C(37)	C(38)	119.2(9)
C(35)	C(36)	C(37)	120.6(8)	C(40)	C(39)	C(38)	120(1)
C(5)	C(6)	C(7)	112.0(6)	C(30)	C(29)	C(28)	109.6(9)
C(17)	C(18)	C(19)	119.2(9)	C(25)	C(21)	C(24)	109.4(8)
C(25)	C(21)	C(22)	112.6(7)	C(25)	C(21)	C(23)	110.5(8)
C(24)	C(21)	C(22)	108.0(8)	C(24)	C(21)	C(23)	106.8(8)
C(22)	C(21)	C(23)	109(1)	C(26)	C(25)	C(28)	108.4(8)
C(26)	C(25)	C(21)	113.9(8)	C(28)	C(25)	C(21)	114.3(9)
C(27)	C(26)	C(25)	109.6(9)	C(30)	C(27)	C(26)	110.0(9)
C(31)	C(30)	C(29)	113.0(8)	C(31)	C(30)	C(27)	112.0(8)
C(29)	C(30)	C(27)	110.5(8)	C(16)	C(17)	C(18)	121(1)
N(3)	C(32)	C(31)	174(1)	C(29)	C(28)	C(25)	111(1)

Table 6. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(8)	C(5)	H(10)	105.2(8)	C(1)	C(5)	H(10)	106.6(8)
C(6)	C(5)	H(10)	107.3(7)	C(10)	C(7)	H(13)	108.7(8)
C(10)	C(7)	H(14)	108.7(9)	C(6)	C(7)	H(13)	110.0(9)
C(6)	C(7)	H(14)	109.0(8)	H(13)	C(7)	H(14)	109.5(9)
C(15)	C(14)	H(20)	108.0(8)	C(15)	C(14)	H(21)	107.7(8)
H(20)	C(14)	H(21)	109.5(9)	H(20)	C(14)	C(11)	108.2(9)
H(21)	C(14)	C(11)	108.2(8)	C(9)	C(10)	H(19)	107.3(7)
H(19)	C(10)	C(7)	108.1(8)	H(19)	C(10)	C(11)	107.8(8)
C(8)	C(9)	H(17)	109.5(8)	C(8)	C(9)	H(18)	108.2(9)
H(17)	C(9)	H(18)	109(1)	H(17)	C(9)	C(10)	109(1)
H(18)	C(9)	C(10)	108.7(8)	H(15)	C(8)	H(16)	109(1)
H(15)	C(8)	C(5)	109(1)	H(15)	C(8)	C(9)	109.9(8)
H(16)	C(8)	C(5)	107.3(9)	H(16)	C(8)	C(9)	107.9(9)
C(31)	C(34)	H(46)	107.7(9)	C(31)	C(34)	H(47)	108.7(7)
C(35)	C(34)	H(46)	106.3(7)	C(35)	C(34)	H(47)	109.4(9)
H(46)	C(34)	H(47)	109(1)	C(1)	C(3)	H(4)	108.7(9)
C(1)	C(3)	H(5)	109(1)	C(1)	C(3)	H(6)	109(1)
H(4)	C(3)	H(5)	109(1)	H(4)	C(3)	H(6)	109(1)
H(5)	C(3)	H(6)	109(1)	C(35)	C(40)	H(52)	118(1)
C(39)	C(40)	H(52)	119(1)	C(37)	C(38)	H(50)	118(1)
C(39)	C(38)	H(50)	120(1)	C(19)	C(20)	H(26)	120(1)
H(26)	C(20)	C(15)	119.2(8)	C(18)	C(19)	H(25)	118(1)
H(25)	C(19)	C(20)	119(1)	C(17)	C(16)	H(22)	120(1)
H(22)	C(16)	C(15)	120(1)	C(36)	C(37)	H(49)	120(1)
H(49)	C(37)	C(38)	120(1)	H(48)	C(36)	C(35)	120.1(9)
H(48)	C(36)	C(37)	119(1)	H(51)	C(39)	C(40)	120(1)
H(51)	C(39)	C(38)	119(1)	H(7)	C(4)	H(8)	109(1)
H(7)	C(4)	H(9)	109(1)	H(7)	C(4)	C(1)	108(1)
H(8)	C(4)	H(9)	109(1)	H(8)	C(4)	C(1)	109(1)
H(9)	C(4)	C(1)	110.3(8)	H(11)	C(6)	H(12)	109(1)
H(11)	C(6)	C(5)	109.5(8)	H(11)	C(6)	C(7)	110.3(9)
H(12)	C(6)	C(5)	108(1)	H(12)	C(6)	C(7)	107.3(8)
C(30)	C(29)	H(43)	108(1)	C(30)	C(29)	H(44)	109(1)
C(28)	C(29)	H(43)	109(1)	C(28)	C(29)	H(44)	109(1)
H(43)	C(29)	H(44)	109(1)	H(1)	C(2)	H(2)	109(1)
H(1)	C(2)	H(3)	109(1)	H(1)	C(2)	C(1)	109(1)
H(2)	C(2)	H(3)	109(1)	H(2)	C(2)	C(1)	110(1)

Table 6. Bond angles involving hydrogens ($^{\circ}$) -- continued

atom	atom	atom	angle	atom	atom	atom	angle
H(3)	C(2)	C(1)	108(1)	C(17)	C(18)	H(24)	119(1)
H(24)	C(18)	C(19)	120(1)	C(26)	C(25)	H(36)	106(1)
C(28)	C(25)	H(36)	107(1)	H(36)	C(25)	C(21)	105.3(9)
C(27)	C(26)	H(37)	108(1)	C(27)	C(26)	H(38)	110(1)
H(37)	C(26)	H(38)	109(1)	H(37)	C(26)	C(25)	108(1)
H(38)	C(26)	C(25)	110(1)	H(33)	C(24)	H(34)	109(1)
H(33)	C(24)	H(35)	109(1)	H(33)	C(24)	C(21)	107.1(9)
H(34)	C(24)	H(35)	109(1)	H(34)	C(24)	C(21)	108(1)
H(35)	C(24)	C(21)	112(1)	H(27)	C(22)	H(28)	109(1)
H(27)	C(22)	H(29)	109(1)	H(27)	C(22)	C(21)	110(1)
H(28)	C(22)	H(29)	109(1)	H(28)	C(22)	C(21)	108(1)
H(29)	C(22)	C(21)	109(1)	C(30)	C(27)	H(39)	109(1)
C(30)	C(27)	H(40)	109(1)	H(39)	C(27)	H(40)	109(1)
H(39)	C(27)	C(26)	110(1)	H(40)	C(27)	C(26)	107(1)
H(45)	C(30)	C(31)	106.8(8)	H(45)	C(30)	C(29)	106(1)
H(45)	C(30)	C(27)	107(1)	H(23)	C(17)	C(16)	119(1)
H(23)	C(17)	C(18)	119(1)	H(41)	C(28)	H(42)	109(1)
H(41)	C(28)	C(29)	108(1)	H(41)	C(28)	C(25)	105(1)
H(42)	C(28)	C(29)	109(1)	H(42)	C(28)	C(25)	112(1)
H(30)	C(23)	H(31)	109(1)	H(30)	C(23)	H(32)	109(1)
H(30)	C(23)	C(21)	110(1)	H(31)	C(23)	H(32)	109(1)
H(31)	C(23)	C(21)	103(1)	H(32)	C(23)	C(21)	114(1)

Table 7. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(1)	C(5)	C(8)	C(9)	-173.2(7)	C(1)	C(5)	C(8)	H(15)	-50.5(9)
C(1)	C(5)	C(8)	H(16)	68(1)	C(6)	C(5)	C(8)	C(9)	57.7(8)
C(6)	C(5)	C(8)	H(15)	-179.7(7)	C(6)	C(5)	C(8)	H(16)	-60.7(9)
H(10)	C(5)	C(8)	C(9)	-56(1)	H(10)	C(5)	C(8)	H(15)	65(1)
H(10)	C(5)	C(8)	H(16)	-175.1(9)	C(8)	C(5)	C(1)	C(3)	59.5(9)
C(8)	C(5)	C(1)	C(4)	-61(1)	C(8)	C(5)	C(1)	C(2)	178.8(7)
C(6)	C(5)	C(1)	C(3)	-174.8(7)	C(6)	C(5)	C(1)	C(4)	63.8(9)
C(6)	C(5)	C(1)	C(2)	-55.5(9)	H(10)	C(5)	C(1)	C(3)	-56(1)
H(10)	C(5)	C(1)	C(4)	-177.6(9)	H(10)	C(5)	C(1)	C(2)	63(1)
C(8)	C(5)	C(6)	C(7)	-58.0(8)	C(8)	C(5)	C(6)	H(11)	64(1)
C(8)	C(5)	C(6)	H(12)	-176.0(7)	C(1)	C(5)	C(6)	C(7)	173.3(7)
C(1)	C(5)	C(6)	H(11)	-64(1)	C(1)	C(5)	C(6)	H(12)	55.2(9)
H(10)	C(5)	C(6)	C(7)	55(1)	H(10)	C(5)	C(6)	H(11)	177(1)
H(10)	C(5)	C(6)	H(12)	-63(1)	C(6)	C(7)	C(10)	C(11)	-179.6(6)
C(6)	C(7)	C(10)	C(9)	-54.7(8)	C(6)	C(7)	C(10)	H(19)	62.2(9)
H(13)	C(7)	C(10)	C(11)	-58(1)	H(13)	C(7)	C(10)	C(9)	66.4(9)
H(13)	C(7)	C(10)	H(19)	-176.7(9)	H(14)	C(7)	C(10)	C(11)	60.5(8)
H(14)	C(7)	C(10)	C(9)	-174.5(7)	H(14)	C(7)	C(10)	H(19)	-57.6(9)
C(10)	C(7)	C(6)	C(5)	57.8(9)	C(10)	C(7)	C(6)	H(11)	-64(1)
C(10)	C(7)	C(6)	H(12)	176.4(9)	H(13)	C(7)	C(6)	C(5)	-62(1)
H(13)	C(7)	C(6)	H(11)	175(1)	H(13)	C(7)	C(6)	H(12)	56(1)
H(14)	C(7)	C(6)	C(5)	177.5(9)	H(14)	C(7)	C(6)	H(11)	55(1)
H(14)	C(7)	C(6)	H(12)	-63(1)	C(10)	C(11)	C(14)	C(15)	178.6(6)
C(10)	C(11)	C(14)	H(20)	-60.4(8)	C(10)	C(11)	C(14)	H(21)	58(1)
C(13)	C(11)	C(14)	C(15)	-57.3(9)	C(13)	C(11)	C(14)	H(20)	63.7(8)
C(13)	C(11)	C(14)	H(21)	-177.8(8)	C(12)	C(11)	C(14)	C(15)	58.9(8)
C(12)	C(11)	C(14)	H(20)	179(719)	C(12)	C(11)	C(14)	H(21)	-61.6(8)
C(14)	C(11)	C(10)	C(7)	-170.3(6)	C(14)	C(11)	C(10)	C(9)	66.0(8)
C(14)	C(11)	C(10)	H(19)	-51(1)	C(13)	C(11)	C(10)	C(7)	65.3(8)
C(13)	C(11)	C(10)	C(9)	-58.4(8)	C(13)	C(11)	C(10)	H(19)	-176.4(8)
C(12)	C(11)	C(10)	C(7)	-50.7(8)	C(12)	C(11)	C(10)	C(9)	-174.5(7)
C(12)	C(11)	C(10)	H(19)	67.5(9)	C(11)	C(14)	C(15)	C(20)	92(1)
C(11)	C(14)	C(15)	C(16)	-85(1)	H(20)	C(14)	C(15)	C(20)	-28(1)
H(20)	C(14)	C(15)	C(16)	153.0(9)	H(21)	C(14)	C(15)	C(20)	-146(1)
H(21)	C(14)	C(15)	C(16)	34(1)	C(7)	C(10)	C(9)	C(8)	54.4(8)
C(7)	C(10)	C(9)	H(17)	175.7(7)	C(7)	C(10)	C(9)	H(18)	-64(1)
C(11)	C(10)	C(9)	C(8)	178.7(7)	C(11)	C(10)	C(9)	H(17)	-59.9(9)

Table 7. Torsion angles ($^{\circ}$) -- continued

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(11)	C(10)	C(9)	H(18)	59(1)	H(19)	C(10)	C(9)	C(8)	-62(1)
H(19)	C(10)	C(9)	H(17)	58(1)	H(19)	C(10)	C(9)	H(18)	178(1)
C(10)	C(9)	C(8)	C(5)	-57(1)	C(10)	C(9)	C(8)	H(15)	179(719)
C(10)	C(9)	C(8)	H(16)	60(1)	H(17)	C(9)	C(8)	C(5)	-178.8(9)
H(17)	C(9)	C(8)	H(15)	58(1)	H(17)	C(9)	C(8)	H(16)	-60(1)
H(18)	C(9)	C(8)	C(5)	61(1)	H(18)	C(9)	C(8)	H(15)	-60(1)
H(18)	C(9)	C(8)	H(16)	179(881)	C(35)	C(34)	C(31)	C(33)	57(1)
C(35)	C(34)	C(31)	C(30)	-176.2(8)	C(35)	C(34)	C(31)	C(32)	-58(1)
H(46)	C(34)	C(31)	C(33)	175.6(9)	H(46)	C(34)	C(31)	C(30)	-57(1)
H(46)	C(34)	C(31)	C(32)	59(1)	H(47)	C(34)	C(31)	C(33)	-65(1)
H(47)	C(34)	C(31)	C(30)	60(1)	H(47)	C(34)	C(31)	C(32)	178(1)
C(31)	C(34)	C(35)	C(40)	87(1)	C(31)	C(34)	C(35)	C(36)	-93(1)
H(46)	C(34)	C(35)	C(40)	-32(1)	H(46)	C(34)	C(35)	C(36)	146(1)
H(47)	C(34)	C(35)	C(40)	-150.3(9)	H(47)	C(34)	C(35)	C(36)	28(1)
H(4)	C(3)	C(1)	C(5)	57(1)	H(4)	C(3)	C(1)	C(4)	-179(508)
H(4)	C(3)	C(1)	C(2)	-63(1)	H(5)	C(3)	C(1)	C(5)	176(1)
H(5)	C(3)	C(1)	C(4)	-59(1)	H(5)	C(3)	C(1)	C(2)	56(1)
H(6)	C(3)	C(1)	C(5)	-62(1)	H(6)	C(3)	C(1)	C(4)	60(1)
H(6)	C(3)	C(1)	C(2)	177.0(8)	C(14)	C(15)	C(20)	C(19)	-179.3(8)
C(14)	C(15)	C(20)	H(26)	0(1)	C(16)	C(15)	C(20)	C(19)	0(1)
C(16)	C(15)	C(20)	H(26)	178(1)	C(14)	C(15)	C(16)	C(17)	178.3(9)
C(14)	C(15)	C(16)	H(22)	0(1)	C(20)	C(15)	C(16)	C(17)	0(1)
C(20)	C(15)	C(16)	H(22)	-179(1)	C(5)	C(1)	C(4)	H(7)	61(1)
C(5)	C(1)	C(4)	H(8)	-179(1)	C(5)	C(1)	C(4)	H(9)	-58(1)
C(3)	C(1)	C(4)	H(7)	-60(1)	C(3)	C(1)	C(4)	H(8)	59(1)
C(3)	C(1)	C(4)	H(9)	179(1)	C(2)	C(1)	C(4)	H(7)	-177(1)
C(2)	C(1)	C(4)	H(8)	-58(1)	C(2)	C(1)	C(4)	H(9)	62(1)
C(5)	C(1)	C(2)	H(1)	60(1)	C(5)	C(1)	C(2)	H(2)	-178(1)
C(5)	C(1)	C(2)	H(3)	-58(1)	C(3)	C(1)	C(2)	H(1)	-179.5(7)
C(3)	C(1)	C(2)	H(2)	-58(1)	C(3)	C(1)	C(2)	H(3)	61(1)
C(4)	C(1)	C(2)	H(1)	-61.9(9)	C(4)	C(1)	C(2)	H(2)	58(1)
C(4)	C(1)	C(2)	H(3)	178(1)	C(39)	C(40)	C(35)	C(34)	-179(719)
C(39)	C(40)	C(35)	C(36)	1(1)	H(52)	C(40)	C(35)	C(34)	0(1)
H(52)	C(40)	C(35)	C(36)	-178(1)	C(35)	C(40)	C(39)	C(38)	0(1)
C(35)	C(40)	C(39)	H(51)	-179(881)	H(52)	C(40)	C(39)	C(38)	179(359)
H(52)	C(40)	C(39)	H(51)	0(1)	C(33)	C(31)	C(30)	C(29)	61(1)
C(33)	C(31)	C(30)	C(27)	-63(1)	C(33)	C(31)	C(30)	H(45)	178(1)

Table 7. Torsion angles ($^{\circ}$) -- continued

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(34)	C(31)	C(30)	C(29)	-63(1)	C(34)	C(31)	C(30)	C(27)	170.5(8)
C(34)	C(31)	C(30)	H(45)	52(1)	C(32)	C(31)	C(30)	C(29)	177.0(9)
C(32)	C(31)	C(30)	C(27)	51(1)	C(32)	C(31)	C(30)	H(45)	-66(1)
C(39)	C(38)	C(37)	C(36)	0(1)	C(39)	C(38)	C(37)	H(49)	-179(1)
H(50)	C(38)	C(37)	C(36)	-178(1)	H(50)	C(38)	C(37)	H(49)	1(1)
C(37)	C(38)	C(39)	C(40)	0(1)	C(37)	C(38)	C(39)	H(51)	178(1)
H(50)	C(38)	C(39)	C(40)	178(1)	H(50)	C(38)	C(39)	H(51)	-1(1)
C(15)	C(20)	C(19)	C(18)	0(1)	C(15)	C(20)	C(19)	H(25)	179(1)
H(26)	C(20)	C(19)	C(18)	-179(1)	H(26)	C(20)	C(19)	H(25)	0(2)
C(34)	C(35)	C(36)	C(37)	179(623)	C(34)	C(35)	C(36)	H(48)	0(1)
C(40)	C(35)	C(36)	C(37)	-1(1)	C(40)	C(35)	C(36)	H(48)	178(1)
C(20)	C(19)	C(18)	C(17)	0(1)	C(20)	C(19)	C(18)	H(24)	-179(359)
H(25)	C(19)	C(18)	C(17)	-177(1)	H(25)	C(19)	C(18)	H(24)	1(2)
C(15)	C(16)	C(17)	C(18)	1(1)	C(15)	C(16)	C(17)	H(23)	179(881)
H(22)	C(16)	C(17)	C(18)	-179(359)	H(22)	C(16)	C(17)	H(23)	-1(2)
C(38)	C(37)	C(36)	C(35)	0(1)	C(38)	C(37)	C(36)	H(48)	-179(359)
H(49)	C(37)	C(36)	C(35)	-179(359)	H(49)	C(37)	C(36)	H(48)	0(1)
C(28)	C(29)	C(30)	C(31)	175.9(9)	C(28)	C(29)	C(30)	C(27)	-57(1)
C(28)	C(29)	C(30)	H(45)	58(1)	H(43)	C(29)	C(30)	C(31)	-64(1)
H(43)	C(29)	C(30)	C(27)	62(1)	H(43)	C(29)	C(30)	H(45)	178(1)
H(44)	C(29)	C(30)	C(31)	55(1)	H(44)	C(29)	C(30)	C(27)	-177(1)
H(44)	C(29)	C(30)	H(45)	-61(1)	C(30)	C(29)	C(28)	C(25)	59(1)
C(30)	C(29)	C(28)	H(41)	175(1)	C(30)	C(29)	C(28)	H(42)	-65(1)
H(43)	C(29)	C(28)	C(25)	-59(1)	H(43)	C(29)	C(28)	H(41)	55(1)
H(43)	C(29)	C(28)	H(42)	175(1)	H(44)	C(29)	C(28)	C(25)	179(622)
H(44)	C(29)	C(28)	H(41)	-64(1)	H(44)	C(29)	C(28)	H(42)	54(1)
C(19)	C(18)	C(17)	C(16)	-1(1)	C(19)	C(18)	C(17)	H(23)	179(622)
H(24)	C(18)	C(17)	C(16)	178(1)	H(24)	C(18)	C(17)	H(23)	0(2)
C(24)	C(21)	C(25)	C(26)	58(1)	C(24)	C(21)	C(25)	C(28)	-175.7(9)
C(24)	C(21)	C(25)	H(36)	-57(1)	C(22)	C(21)	C(25)	C(26)	-61(1)
C(22)	C(21)	C(25)	C(28)	64(1)	C(22)	C(21)	C(25)	H(36)	-177(1)
C(23)	C(21)	C(25)	C(26)	176.2(9)	C(23)	C(21)	C(25)	C(28)	-58(1)
C(23)	C(21)	C(25)	H(36)	59(1)	C(25)	C(21)	C(24)	H(33)	-64(1)
C(25)	C(21)	C(24)	H(34)	53(1)	C(25)	C(21)	C(24)	H(35)	175(1)
C(22)	C(21)	C(24)	H(33)	58(1)	C(22)	C(21)	C(24)	H(34)	176(1)
C(22)	C(21)	C(24)	H(35)	-62(1)	C(23)	C(21)	C(24)	H(33)	175(1)
C(23)	C(21)	C(24)	H(34)	-66(1)	C(23)	C(21)	C(24)	H(35)	55(1)

Table 7. Torsion angles ($^{\circ}$) -- continued

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(25)	C(21)	C(22)	H(27)	-177(1)	C(25)	C(21)	C(22)	H(28)	-57(1)
C(25)	C(21)	C(22)	H(29)	61(1)	C(24)	C(21)	C(22)	H(27)	61(1)
C(24)	C(21)	C(22)	H(28)	-178(1)	C(24)	C(21)	C(22)	H(29)	-59(1)
C(23)	C(21)	C(22)	H(27)	-54(1)	C(23)	C(21)	C(22)	H(28)	65(1)
C(23)	C(21)	C(22)	H(29)	-175.0(8)	C(25)	C(21)	C(23)	H(30)	-173(1)
C(25)	C(21)	C(23)	H(31)	-56(1)	C(25)	C(21)	C(23)	H(32)	62(1)
C(24)	C(21)	C(23)	H(30)	-54(1)	C(24)	C(21)	C(23)	H(31)	62(1)
C(24)	C(21)	C(23)	H(32)	-178(1)	C(22)	C(21)	C(23)	H(30)	62(1)
C(22)	C(21)	C(23)	H(31)	179(1)	C(22)	C(21)	C(23)	H(32)	-61(1)
C(21)	C(25)	C(26)	C(27)	-172.8(9)	C(21)	C(25)	C(26)	H(37)	-54(1)
C(21)	C(25)	C(26)	H(38)	65(1)	C(28)	C(25)	C(26)	C(27)	58(1)
C(28)	C(25)	C(26)	H(37)	176(1)	C(28)	C(25)	C(26)	H(38)	-63(1)
H(36)	C(25)	C(26)	C(27)	-57(1)	H(36)	C(25)	C(26)	H(37)	60(1)
H(36)	C(25)	C(26)	H(38)	-179(1)	C(21)	C(25)	C(28)	C(29)	172.0(9)
C(21)	C(25)	C(28)	H(41)	54(1)	C(21)	C(25)	C(28)	H(42)	-64(1)
C(26)	C(25)	C(28)	C(29)	-59(1)	C(26)	C(25)	C(28)	H(41)	-177(1)
C(26)	C(25)	C(28)	H(42)	63(1)	H(36)	C(25)	C(28)	C(29)	55(1)
H(36)	C(25)	C(28)	H(41)	-62(1)	H(36)	C(25)	C(28)	H(42)	178(1)
C(25)	C(26)	C(27)	C(30)	-58(1)	C(25)	C(26)	C(27)	H(39)	62(1)
C(25)	C(26)	C(27)	H(40)	-178(1)	H(37)	C(26)	C(27)	C(30)	-176(1)
H(37)	C(26)	C(27)	H(39)	-55(1)	H(37)	C(26)	C(27)	H(40)	64(1)
H(38)	C(26)	C(27)	C(30)	63(1)	H(38)	C(26)	C(27)	H(39)	-175(1)
H(38)	C(26)	C(27)	H(40)	-55(1)	C(26)	C(27)	C(30)	C(31)	-174.8(9)
C(26)	C(27)	C(30)	C(29)	58(1)	C(26)	C(27)	C(30)	H(45)	-57(1)
H(39)	C(27)	C(30)	C(31)	63(1)	H(39)	C(27)	C(30)	C(29)	-63(1)
H(39)	C(27)	C(30)	H(45)	-179(719)	H(40)	C(27)	C(30)	C(31)	-56(1)
H(40)	C(27)	C(30)	C(29)	176(1)	H(40)	C(27)	C(30)	H(45)	60(1)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

X-ray crystallographic Data of *trans*-6b

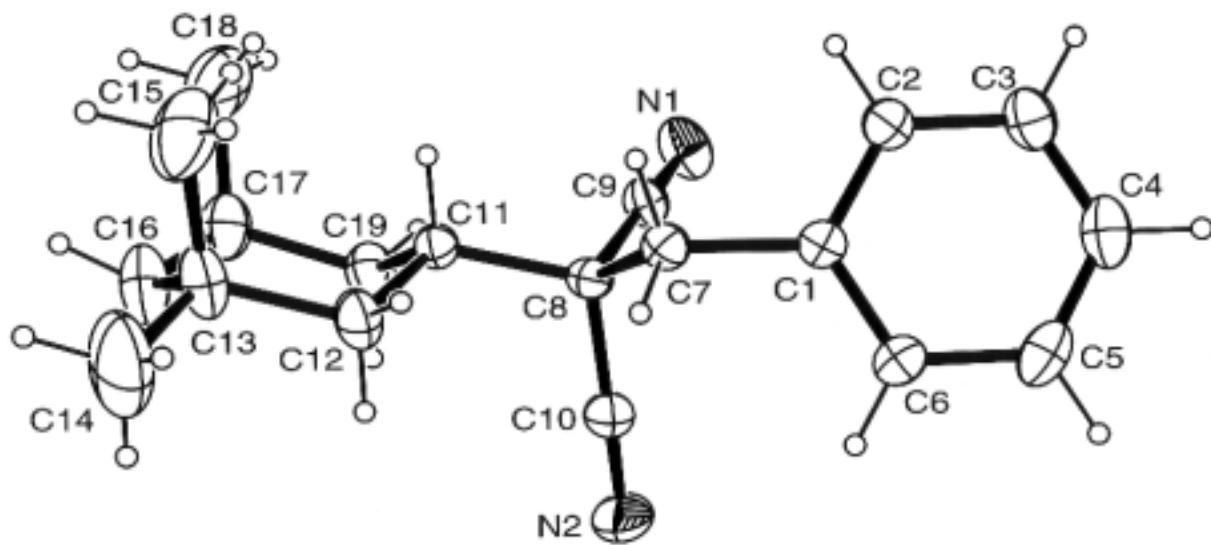


Figure S3. ORTEP drawing of *trans*-6b (ellipsoids at 20% probability).

Experimental

Data Collection

A colorless prism crystal of C₁₉H₂₄N₂ having approximate dimensions of 0.60 x 0.20 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-K α radiation.

Indexing was performed from 3° oscillations that were exposed for 300 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a C-centered monoclinic cell with dimensions:

$$\begin{aligned}a &= 35.436(3) \text{ \AA} \\b &= 6.3489(5) \text{ \AA} \quad \beta = 97.655(3)^\circ \\c &= 15.747(1) \text{ \AA} \\V &= 3511.3(5) \text{ \AA}^3\end{aligned}$$

For Z = 8 and F.W. = 280.41, the calculated density is 1.06 g/cm³. Based on the systematic absences of:

$$\begin{aligned}\text{hkl: } h+k &\pm 2n \\h0l: \quad l &\pm 2n\end{aligned}$$

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$$C2/c (\#15)$$

The data were collected at a temperature of 23 ± 10°C to a maximum 2θ value of 55.0°. A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0° in 5.0° step, at χ=45.0° and φ = 0.0°. The exposure rate was 600.0 [sec./0]. A second sweep was performed using ω scans from 0.0 to 160.0° in

5.0° step, at $\chi=45.0^\circ$ and $\phi = 180.0^\circ$. The exposure rate was 600.0 [sec./0]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 8316 reflections that were collected, 4158 were unique ($R_{\text{int}} = 0.000$); equivalent reflections were merged.

The linear absorption coefficient, μ , for Mo-K α radiation is 0.6 cm $^{-1}$. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement³ on F^2 was based on 2596 observed reflections and 214 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R_1 = \sum ||F_O| - |F_C|| / \sum |F_O| = 0.091$$

$$wR_2 = [\sum (w(F_O^2 - F_C^2)^2) / \sum w(F_O^2)^2]^{1/2} = 0.214$$

The standard deviation of an observation of unit weight⁴ was 0.89. A Sheldrick weighting scheme was used. Plots of $\sum w(|F_O| - |F_C|)^2$ versus $|F_O|$, reflection order in data collection, $\sin \theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.84 and -0.43 e $^-/\text{\AA}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in F_{calc} ⁶; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbell⁸. All calculations were performed using the CrystalStructure^{9,10} crystallographic software package.

References

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- (2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
- (3) Least Squares function minimized:
- $$\sum w(F_o^2 - F_c^2)^2 \quad \text{where } w = \text{Least Squares weights.}$$
- (4) Standard deviation of an observation of unit weight:
- $$[\sum w(F_o^2 - F_c^2)^2 / (N_o - N_v)]^{1/2}$$
- where: N_o = number of observations
 N_v = number of variables
- (5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).
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- (9) CrystalStructure 3.5.1: Crystal Structure Analysis Package, Rigaku and Rigaku/MSC (2000-2003). 9009 New Trails Dr. The Woodlands TX 77381 USA.
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EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₁₉ H ₂₄ N ₂
Formula Weight	280.41
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.60 X 0.20 X 0.10 mm
Crystal System	monoclinic
Lattice Type	C-centered
Indexing Images	3 oscillations @ 300.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 35.436(3) Å b = 6.3489(5) Å c = 15.747(1) Å β = 97.655(3) ° V = 3511.3(5) Å ³
Space Group	C2/c (#15)
Z value	8
D _{calc}	1.061 g/cm ³
F ₀₀₀	1216.00

$\mu(\text{MoK}\alpha)$

0.62 cm⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71069 \text{ \AA}$) graphite monochromated
Detector Aperture	270 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0, \phi=0.0$)	130.0 - 190.0°
Exposure Rate	600.0 sec./°
ω oscillation Range ($\chi=45.0, \phi=180.0$)	0.0 - 160.0°
Exposure Rate	600.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	55.0°
No. of Reflections Measured	Total: 8316
Corrections	Unique: 4158 ($R_{\text{int}} = 0.000$) Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma w (Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0010Fo^2 + 3.0000\sigma(Fo^2) + 0.5000]/(4Fo^2)$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 1.00\sigma(I)$)	2596
No. Variables	214
Reflection/Parameter Ratio	12.13
Residuals: R ($I > 1.00\sigma(I)$)	0.102
Residuals: R1 ($I > 2.00\sigma(I)$)	0.091
Residuals: wR2 ($I > 1.00\sigma(I)$)	0.214
Goodness of Fit Indicator	0.885
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	$0.84 \text{ e}^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-0.43 \text{ e}^-/\text{\AA}^3$

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$

atom	x	y	z	B_{eq}
N(1)	0.3498(1)	0.2230(8)	0.2732(3)	6.7(1)
N(2)	0.3470(1)	-0.2994(7)	0.1107(4)	8.2(1)
C(1)	0.2739(1)	0.1677(6)	0.0985(2)	3.85(8)
C(7)	0.3103(1)	0.1985(7)	0.0599(2)	4.14(8)
C(11)	0.3838(1)	0.1964(7)	0.0800(3)	4.12(8)
C(8)	0.3473(1)	0.1115(6)	0.1134(2)	3.93(8)
C(15)	0.4148(2)	0.484(1)	-0.0593(5)	11.9(3)
C(2)	0.2599(1)	0.3296(7)	0.1429(3)	4.8(1)
C(9)	0.3483(1)	0.1755(7)	0.2040(3)	4.5(1)
C(6)	0.2538(1)	-0.0185(7)	0.0888(3)	4.9(1)
C(5)	0.2196(1)	-0.0387(9)	0.1236(3)	6.2(1)
C(4)	0.2066(1)	0.125(1)	0.1676(3)	6.3(1)
C(10)	0.3470(1)	-0.1221(8)	0.1125(3)	5.2(1)
C(14)	0.4217(2)	0.175(2)	-0.1373(5)	15.4(4)
C(3)	0.2264(1)	0.3071(9)	0.1777(3)	5.7(1)
C(19)	0.4201(1)	0.1205(9)	0.1335(3)	5.9(1)
C(12)	0.3852(1)	0.1458(9)	-0.0139(3)	5.8(1)
C(17)	0.4562(1)	0.223(1)	0.1065(4)	7.1(2)
C(13)	0.4203(1)	0.238(1)	-0.0474(3)	7.2(2)
C(16)	0.4567(1)	0.194(1)	0.0122(4)	8.9(2)
C(18)	0.4610(2)	0.450(1)	0.1403(5)	11.0(2)

$$B_{\text{eq}} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/ B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.2739(1)	0.4576(7)	0.1502(3)	5.6(1)
H(2)	0.2166(1)	0.4203(9)	0.2077(3)	6.8(1)
H(3)	0.1834(1)	0.1111(1)	0.1913(3)	7.6(2)
H(4)	0.2057(1)	-0.1670(9)	0.1177(3)	7.3(2)
H(5)	0.2631(1)	-0.1310(7)	0.0577(3)	5.8(1)
H(6)	0.3073(1)	0.1315(7)	0.0055(2)	5.0(1)
H(7)	0.3136(1)	0.3456(7)	0.0527(2)	5.0(1)
H(8)	0.3836(1)	0.3455(7)	0.0848(3)	5.0(1)
H(9)	0.3863(1)	-0.0030(9)	-0.0193(3)	7.1(2)
H(10)	0.3627(1)	0.1969(9)	-0.0473(3)	7.1(2)
H(11)	0.4138(2)	0.548(1)	-0.0050(5)	14.5(3)
H(12)	0.4355(2)	0.541(1)	-0.0843(5)	14.5(3)
H(13)	0.3917(2)	0.511(1)	-0.0957(5)	14.5(3)
H(14)	0.3985(2)	0.211(2)	-0.1716(5)	19.0(5)
H(15)	0.4424(2)	0.240(2)	-0.1601(5)	19.0(5)
H(16)	0.4249(2)	0.026(2)	-0.1373(5)	19.0(5)
H(17)	0.4765(1)	0.279(1)	-0.0048(4)	10.9(2)
H(18)	0.4620(1)	0.050(1)	0.0029(4)	10.9(2)
H(19)	0.4775(1)	0.148(1)	0.1344(4)	8.5(2)
H(20)	0.4615(2)	0.453(1)	0.2008(5)	13.1(3)
H(21)	0.4840(2)	0.507(1)	0.1256(5)	13.1(3)
H(22)	0.4400(2)	0.530(1)	0.1140(5)	13.1(3)
H(23)	0.4188(1)	0.1495(9)	0.1923(3)	7.1(1)
H(24)	0.4215(1)	-0.0274(9)	0.1254(3)	7.1(1)

$$B_{eq} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 3. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
N(1)	0.069(2)	0.122(4)	0.062(2)	0.013(2)	0.004(2)	-0.015(3)
N(2)	0.102(3)	0.050(3)	0.163(5)	0.001(3)	0.032(3)	0.001(3)
C(1)	0.050(2)	0.054(2)	0.040(2)	-0.000(2)	0.000(2)	0.002(2)
C(7)	0.054(2)	0.054(2)	0.050(2)	-0.000(2)	0.007(2)	0.000(2)
C(11)	0.048(2)	0.051(2)	0.060(2)	0.002(2)	0.012(2)	0.003(2)
C(8)	0.057(2)	0.044(2)	0.049(2)	0.003(2)	0.009(2)	-0.002(2)
C(15)	0.121(5)	0.161(7)	0.175(8)	0.001(5)	0.040(5)	0.083(6)
C(2)	0.057(2)	0.065(3)	0.058(2)	0.001(2)	0.001(2)	-0.003(2)
C(9)	0.051(2)	0.068(3)	0.055(2)	0.005(2)	0.007(2)	-0.000(2)
C(6)	0.064(2)	0.059(3)	0.060(2)	-0.008(2)	0.003(2)	0.000(2)
C(5)	0.065(3)	0.088(4)	0.078(3)	-0.022(3)	-0.002(2)	0.027(3)
C(4)	0.060(3)	0.122(5)	0.059(3)	0.009(3)	0.012(2)	0.028(3)
C(10)	0.071(3)	0.052(3)	0.079(3)	0.002(2)	0.018(2)	-0.000(2)
C(14)	0.166(7)	0.32(1)	0.119(6)	-0.064(9)	0.076(6)	-0.058(8)
C(3)	0.066(3)	0.099(4)	0.052(2)	0.008(3)	0.011(2)	0.001(3)
C(19)	0.061(3)	0.083(3)	0.080(3)	0.009(3)	0.012(2)	0.010(3)
C(12)	0.065(2)	0.097(4)	0.063(3)	-0.002(3)	0.021(2)	-0.006(3)
C(17)	0.056(3)	0.117(5)	0.097(4)	0.004(3)	0.012(2)	0.013(4)
C(13)	0.068(3)	0.140(6)	0.071(3)	0.005(3)	0.028(2)	0.004(3)
C(16)	0.071(3)	0.158(7)	0.116(5)	0.009(4)	0.037(3)	0.023(5)
C(18)	0.096(4)	0.162(8)	0.156(7)	-0.038(5)	0.007(4)	-0.012(6)

The general temperature factor expression: $\exp(-2\pi^2(a^*2U_{11}h^2 + b^*2U_{22}k^2 + c^*2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
N(1)	C(9)	1.125(6)	N(2)	C(10)	1.126(6)
C(1)	C(7)	1.509(5)	C(1)	C(2)	1.373(6)
C(1)	C(6)	1.379(6)	C(7)	C(8)	1.561(5)
C(11)	C(8)	1.557(6)	C(11)	C(19)	1.519(5)
C(11)	C(12)	1.519(6)	C(8)	C(9)	1.479(6)
C(8)	C(10)	1.483(6)	C(15)	C(13)	1.58(1)
C(2)	C(3)	1.380(6)	C(6)	C(5)	1.399(6)
C(5)	C(4)	1.366(8)	C(4)	C(3)	1.349(8)
C(14)	C(13)	1.48(1)	C(19)	C(17)	1.547(7)
C(12)	C(13)	1.533(7)	C(17)	C(16)	1.499(9)
C(17)	C(18)	1.53(1)	C(13)	C(16)	1.519(7)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(7)	H(6)	0.950(5)	C(7)	H(7)	0.950(6)
C(11)	H(8)	0.950(6)	C(15)	H(11)	0.95(1)
C(15)	H(12)	0.95(1)	C(15)	H(13)	0.95(1)
C(2)	H(1)	0.950(6)	C(6)	H(5)	0.950(6)
C(5)	H(4)	0.950(8)	C(4)	H(3)	0.950(7)
C(14)	H(14)	0.95(1)	C(14)	H(15)	0.95(1)
C(14)	H(16)	0.95(2)	C(3)	H(2)	0.950(7)
C(19)	H(23)	0.950(7)	C(19)	H(24)	0.950(8)
C(12)	H(9)	0.950(8)	C(12)	H(10)	0.950(6)
C(17)	H(19)	0.950(7)	C(16)	H(17)	0.950(9)
C(16)	H(18)	0.95(1)	C(18)	H(20)	0.95(1)
C(18)	H(21)	0.95(1)	C(18)	H(22)	0.95(1)

Table 6. Bond angles ($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(7)	C(1)	C(2)	119.6(4)	C(7)	C(1)	C(6)	121.6(4)
C(2)	C(1)	C(6)	118.8(4)	C(8)	C(7)	C(1)	116.0(3)
C(8)	C(11)	C(19)	112.5(3)	C(8)	C(11)	C(12)	113.0(3)
C(19)	C(11)	C(12)	109.8(3)	C(9)	C(8)	C(10)	106.4(3)
C(9)	C(8)	C(7)	109.5(3)	C(9)	C(8)	C(11)	108.4(3)
C(10)	C(8)	C(7)	110.2(3)	C(10)	C(8)	C(11)	110.3(3)
C(7)	C(8)	C(11)	111.8(3)	C(3)	C(2)	C(1)	120.9(4)
N(1)	C(9)	C(8)	178.8(4)	C(5)	C(6)	C(1)	119.8(4)
C(4)	C(5)	C(6)	119.9(5)	C(3)	C(4)	C(5)	120.4(5)
N(2)	C(10)	C(8)	179.0(5)	C(2)	C(3)	C(4)	120.3(5)
C(17)	C(19)	C(11)	112.5(4)	C(13)	C(12)	C(11)	112.7(4)
C(16)	C(17)	C(18)	116.5(6)	C(16)	C(17)	C(19)	109.9(4)
C(18)	C(17)	C(19)	110.8(5)	C(16)	C(13)	C(15)	109.7(5)
C(16)	C(13)	C(14)	113.9(5)	C(16)	C(13)	C(12)	112.2(5)
C(15)	C(13)	C(14)	100.1(6)	C(15)	C(13)	C(12)	109.1(5)
C(14)	C(13)	C(12)	111.0(5)	C(17)	C(16)	C(13)	117.9(5)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(8)	C(7)	H(6)	108.1(4)	C(8)	C(7)	H(7)	107.7(4)
H(6)	C(7)	H(7)	109.5(5)	H(6)	C(7)	C(1)	107.9(4)
H(7)	C(7)	C(1)	107.5(4)	C(8)	C(11)	H(8)	107.6(4)
C(19)	C(11)	H(8)	106.7(4)	C(12)	C(11)	H(8)	106.8(5)
C(13)	C(15)	H(11)	109.4(9)	C(13)	C(15)	H(12)	109.7(8)
C(13)	C(15)	H(13)	109.4(8)	H(11)	C(15)	H(12)	109(1)
H(11)	C(15)	H(13)	109(1)	H(12)	C(15)	H(13)	109(1)
C(3)	C(2)	H(1)	120.1(5)	H(1)	C(2)	C(1)	118.9(5)
C(5)	C(6)	H(5)	120.8(5)	H(5)	C(6)	C(1)	119.4(5)
C(4)	C(5)	H(4)	119.8(6)	H(4)	C(5)	C(6)	120.3(6)
C(3)	C(4)	H(3)	120.1(7)	H(3)	C(4)	C(5)	119.5(7)
C(13)	C(14)	H(14)	109.8(9)	C(13)	C(14)	H(15)	111.7(9)
C(13)	C(14)	H(16)	106.9(9)	H(14)	C(14)	H(15)	109(1)
H(14)	C(14)	H(16)	109(1)	H(15)	C(14)	H(16)	109(1)
H(2)	C(3)	C(2)	120.5(6)	H(2)	C(3)	C(4)	119.2(5)
C(17)	C(19)	H(23)	109.6(5)	C(17)	C(19)	H(24)	108.6(5)
H(23)	C(19)	H(24)	109.5(7)	H(23)	C(19)	C(11)	109.4(5)
H(24)	C(19)	C(11)	107.1(4)	C(13)	C(12)	H(9)	107.7(5)
C(13)	C(12)	H(10)	109.6(5)	H(9)	C(12)	H(10)	109.4(6)
H(9)	C(12)	C(11)	107.8(5)	H(10)	C(12)	C(11)	109.5(5)
C(16)	C(17)	H(19)	106.4(6)	C(18)	C(17)	H(19)	105.6(6)
H(19)	C(17)	C(19)	107.1(6)	H(17)	C(16)	H(18)	109.5(8)
H(17)	C(16)	C(17)	108.4(7)	H(17)	C(16)	C(13)	108.7(7)
H(18)	C(16)	C(17)	107.5(7)	H(18)	C(16)	C(13)	104.6(6)
H(20)	C(18)	H(21)	109.5(9)	H(20)	C(18)	H(22)	109(1)
H(20)	C(18)	C(17)	111.0(9)	H(21)	C(18)	H(22)	109(1)
H(21)	C(18)	C(17)	109.3(8)	H(22)	C(18)	C(17)	108.1(8)

Table 8. Torsion Angles($^{\circ}$)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(2)	C(1)	C(7)	C(8)	98.0(4)	C(6)	C(1)	C(7)	C(8)	-83.4(4)
C(7)	C(1)	C(2)	C(3)	179.0(3)	C(6)	C(1)	C(2)	C(3)	0.3(6)
C(7)	C(1)	C(6)	C(5)	-178.3(4)	C(2)	C(1)	C(6)	C(5)	0.2(6)
C(1)	C(7)	C(8)	C(11)	-166.4(3)	C(1)	C(7)	C(8)	C(9)	-46.3(5)
C(1)	C(7)	C(8)	C(10)	70.5(5)	C(19)	C(11)	C(8)	C(7)	177.6(3)
C(19)	C(11)	C(8)	C(9)	56.8(4)	C(19)	C(11)	C(8)	C(10)	-59.4(4)
C(12)	C(11)	C(8)	C(7)	-57.3(4)	C(12)	C(11)	C(8)	C(9)	-178.1(3)
C(12)	C(11)	C(8)	C(10)	65.7(4)	C(8)	C(11)	C(19)	C(17)	-173.9(4)
C(12)	C(11)	C(19)	C(17)	59.3(5)	C(8)	C(11)	C(12)	C(13)	177.4(4)
C(19)	C(11)	C(12)	C(13)	-56.1(5)	C(1)	C(2)	C(3)	C(4)	-0.8(6)
C(1)	C(6)	C(5)	C(4)	-0.4(6)	C(6)	C(5)	C(4)	C(3)	0.0(7)
C(5)	C(4)	C(3)	C(2)	0.6(7)	C(11)	C(19)	C(17)	C(16)	-53.2(6)
C(11)	C(19)	C(17)	C(18)	76.9(6)	C(11)	C(12)	C(13)	C(15)	-74.1(6)
C(11)	C(12)	C(13)	C(14)	176.6(6)	C(11)	C(12)	C(13)	C(16)	47.7(7)
C(19)	C(17)	C(16)	C(13)	46.0(8)	C(18)	C(17)	C(16)	C(13)	-81.1(7)
C(15)	C(13)	C(16)	C(17)	77.5(7)	C(14)	C(13)	C(16)	C(17)	-171.3(6)
C(12)	C(13)	C(16)	C(17)	-44.0(8)					

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Torsion Angles involving hydrogens($^{\circ}$)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(2)	C(1)	C(7)	H(6)	-140.6(4)	C(2)	C(1)	C(7)	H(7)	-22.6(5)
C(6)	C(1)	C(7)	H(6)	38.0(6)	C(6)	C(1)	C(7)	H(7)	156.0(4)
C(7)	C(1)	C(2)	H(1)	-2.0(6)	C(6)	C(1)	C(2)	H(1)	179.4(4)
C(7)	C(1)	C(6)	H(5)	0.9(6)	C(2)	C(1)	C(6)	H(5)	179.4(4)
H(6)	C(7)	C(8)	C(11)	72.3(5)	H(6)	C(7)	C(8)	C(9)	-167.6(4)
H(6)	C(7)	C(8)	C(10)	-50.8(5)	H(7)	C(7)	C(8)	C(11)	-45.9(5)
H(7)	C(7)	C(8)	C(9)	74.2(5)	H(7)	C(7)	C(8)	C(10)	-169.0(4)
H(8)	C(11)	C(8)	C(7)	60.4(5)	H(8)	C(11)	C(8)	C(9)	-60.4(5)
H(8)	C(11)	C(8)	C(10)	-176.6(4)	C(8)	C(11)	C(19)	H(23)	-51.8(6)
C(8)	C(11)	C(19)	H(24)	66.8(6)	C(12)	C(11)	C(19)	H(23)	-178.6(5)
C(12)	C(11)	C(19)	H(24)	-60.0(6)	H(8)	C(11)	C(19)	C(17)	-56.1(6)
H(8)	C(11)	C(19)	H(23)	66.0(7)	H(8)	C(11)	C(19)	H(24)	-175.4(6)
C(8)	C(11)	C(12)	H(9)	-63.9(5)	C(8)	C(11)	C(12)	H(10)	55.0(7)
C(19)	C(11)	C(12)	H(9)	62.6(5)	C(19)	C(11)	C(12)	H(10)	-178.5(5)
H(8)	C(11)	C(12)	C(13)	59.2(5)	H(8)	C(11)	C(12)	H(9)	177.9(5)
H(8)	C(11)	C(12)	H(10)	-63.1(7)	H(11)	C(15)	C(13)	C(14)	-179.0(7)
H(11)	C(15)	C(13)	C(12)	64.4(8)	H(11)	C(15)	C(13)	C(16)	-58.9(8)
H(12)	C(15)	C(13)	C(14)	-59.0(9)	H(12)	C(15)	C(13)	C(12)	-175.5(7)
H(12)	C(15)	C(13)	C(16)	61.1(9)	H(13)	C(15)	C(13)	C(14)	61.1(9)
H(13)	C(15)	C(13)	C(12)	-55.5(9)	H(13)	C(15)	C(13)	C(16)	-178.8(8)
C(1)	C(2)	C(3)	H(2)	-179.2(5)	H(1)	C(2)	C(3)	C(4)	-179.8(5)
H(1)	C(2)	C(3)	H(2)	1.8(8)	C(1)	C(6)	C(5)	H(4)	-179.3(5)
H(5)	C(6)	C(5)	C(4)	-179.6(5)	H(5)	C(6)	C(5)	H(4)	1.5(8)
C(6)	C(5)	C(4)	H(3)	-179(359)	H(4)	C(5)	C(4)	C(3)	178.9(5)
H(4)	C(5)	C(4)	H(3)	-0.9(9)	C(5)	C(4)	C(3)	H(2)	179.0(5)
H(3)	C(4)	C(3)	C(2)	-179.6(5)	H(3)	C(4)	C(3)	H(2)	-1.1(9)
H(14)	C(14)	C(13)	C(15)	-61(1)	H(14)	C(14)	C(13)	C(12)	54(1)
H(14)	C(14)	C(13)	C(16)	-178(1)	H(15)	C(14)	C(13)	C(15)	60(1)
H(15)	C(14)	C(13)	C(12)	175(1)	H(15)	C(14)	C(13)	C(16)	-56(1)
H(16)	C(14)	C(13)	C(15)	-179(359)	H(16)	C(14)	C(13)	C(12)	-64.6(9)
H(16)	C(14)	C(13)	C(16)	63(1)	C(11)	C(19)	C(17)	H(19)	-168.4(6)
H(23)	C(19)	C(17)	C(16)	-175.2(6)	H(23)	C(19)	C(17)	C(18)	-45.0(7)
H(23)	C(19)	C(17)	H(19)	69.6(8)	H(24)	C(19)	C(17)	C(16)	65.3(6)
H(24)	C(19)	C(17)	C(18)	-164.6(5)	H(24)	C(19)	C(17)	H(19)	-49.9(8)
H(9)	C(12)	C(13)	C(15)	167.2(5)	H(9)	C(12)	C(13)	C(14)	57.8(7)
H(9)	C(12)	C(13)	C(16)	-71.0(6)	H(10)	C(12)	C(13)	C(15)	48.2(7)
H(10)	C(12)	C(13)	C(14)	-61.2(8)	H(10)	C(12)	C(13)	C(16)	170.0(6)

Table 9. Torsion angles involving hydrogens (°) -- continued

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(19)	C(17)	C(16)	H(17)	170.0(7)	C(19)	C(17)	C(16)	H(18)	-71.8(7)
C(18)	C(17)	C(16)	H(17)	42.9(8)	C(18)	C(17)	C(16)	H(18)	161.2(6)
H(19)	C(17)	C(16)	C(13)	161.6(7)	H(19)	C(17)	C(16)	H(17)	-74.4(9)
H(19)	C(17)	C(16)	H(18)	43.8(8)	C(19)	C(17)	C(18)	H(20)	56.1(8)
C(19)	C(17)	C(18)	H(21)	176.9(7)	C(19)	C(17)	C(18)	H(22)	-64.0(9)
C(16)	C(17)	C(18)	H(20)	-177.3(6)	C(16)	C(17)	C(18)	H(21)	-56.5(8)
C(16)	C(17)	C(18)	H(22)	62.6(9)	H(19)	C(17)	C(18)	H(20)	-59.5(9)
H(19)	C(17)	C(18)	H(21)	61(1)	H(19)	C(17)	C(18)	H(22)	-179(359)
C(15)	C(13)	C(16)	H(17)	-46.4(9)	C(15)	C(13)	C(16)	H(18)	-163.2(6)
C(14)	C(13)	C(16)	H(17)	64.9(9)	C(14)	C(13)	C(16)	H(18)	-52.0(8)
C(12)	C(13)	C(16)	H(17)	-167.8(7)	C(12)	C(13)	C(16)	H(18)	75.3(8)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

X-ray crystallographic Data of *cis*-6c

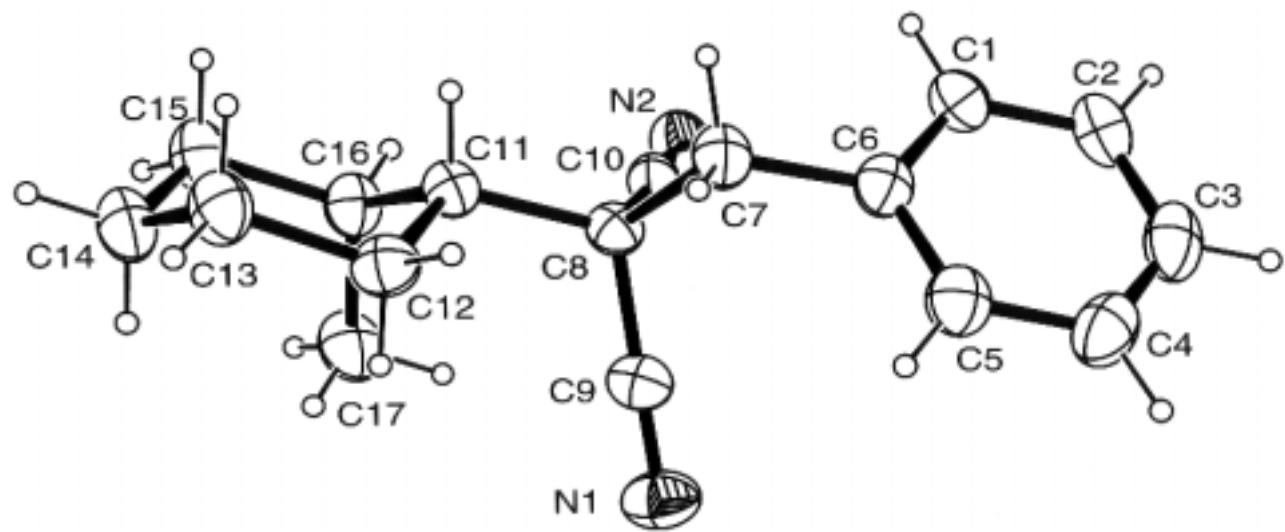


Figure S4. ORTEP drawing of *cis*-6c (ellipsoids at 20% probability).

Experimental

Data Collection

A colorless block crystal of C₁₇H₂₀N₂ having approximate dimensions of 0.50 x 0.25 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-K α radiation.

Indexing was performed from 3° oscillations that were exposed for 36 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions:

$$\begin{aligned}a &= 10.6730(5) \text{ \AA} \\b &= 10.9928(7) \text{ \AA} \\c &= 12.8104(9) \text{ \AA} \\V &= 1503.0(2) \text{ \AA}^3\end{aligned}$$

For Z = 4 and F.W. = 252.36, the calculated density is 1.12 g/cm³. The systematic absences of:

$$\begin{aligned}h00: \quad h &\pm 2n \\0k0: \quad k &\pm 2n \\00l: \quad l &\pm 2n\end{aligned}$$

uniquely determine the space group to be:

P2₁2₁2₁ (#19)

The data were collected at a temperature of 23 ± 1°C to a maximum 2θ value of 54.0°. A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0° in 5.0° step, at χ=45.0° and φ = 0.0°. The exposure rate was 100.2 [sec./°]. A second sweep was performed using ω scans from 0.0 to 160.0° in

5.0° step, at $\chi=45.0^\circ$ and $\phi = 180.0^\circ$. The exposure rate was 100.2 [sec./0]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 3792 reflections that were collected, 1896 were unique ($R_{\text{int}} = 0.000$); equivalent reflections were merged.

The linear absorption coefficient, μ , for Mo-K α radiation is 0.7 cm $^{-1}$. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement³ on F^2 was based on 1878 observed reflections and 192 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R_1 = \sum ||F_O| - |F_C|| / \sum |F_O| = 0.061$$

$$wR_2 = [\sum (w(F_O^2 - F_C^2)^2) / \sum w(F_O^2)^2]^{1/2} = 0.185$$

The standard deviation of an observation of unit weight⁴ was 0.91. A Sheldrick weighting scheme was used. Plots of $\sum w(|F_O| - |F_C|)^2$ versus $|F_O|$, reflection order in data collection, $\sin \theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.36 and -0.32 e $^-/\text{\AA}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in F_{calc} ⁶; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbell⁸. All calculations were performed using the CrystalStructure^{9,10} crystallographic software package.

References

- (1) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.
- (2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
- (3) Least Squares function minimized:
- $$\sum w(F_o^2 - F_c^2)^2 \quad \text{where } w = \text{Least Squares weights.}$$
- (4) Standard deviation of an observation of unit weight:
- $$[\sum w(F_o^2 - F_c^2)^2 / (N_o - N_v)]^{1/2}$$
- where: N_o = number of observations
 N_v = number of variables
- (5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).
- (6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).
- (7) Creagh, D. C. & McAuley, W.J.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).
- (8) Creagh, D. C. & Hubbell, J.H.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).
- (9) CrystalStructure 3.5.1: Crystal Structure Analysis Package, Rigaku and Rigaku/MSC (2000-2003). 9009 New Trails Dr. The Woodlands TX 77381 USA.
- (10) CRYSTALS Issue 10: Watkin, D.J., Prout, C.K. Carruthers, J.R. & Betteridge, P.W. Chemical Crystallography Laboratory, Oxford, UK. (1996)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₁₇ H ₂₀ N ₂
Formula Weight	252.36
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.50 X 0.25 X 0.20 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 36.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 10.6730(5) Å b = 10.9928(7) Å c = 12.8104(9) Å V = 1503.0(2) Å ³
Space Group	P2 ₁ 2 ₁ 2 ₁ (#19)
Z value	4
D _{calc}	1.115 g/cm ³
F ₀₀₀	544.00
μ(MoKα)	0.66 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71069 \text{ \AA}$) graphite monochromated
Detector Aperture	270 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0, \phi=0.0$)	130.0 - 190.0°
Exposure Rate	100.2 sec./°
ω oscillation Range ($\chi=45.0, \phi=180.0$)	0.0 - 160.0°
Exposure Rate	100.2 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	54.0°
No. of Reflections Measured	Total: 3792
Corrections	Unique: 1896 ($R_{\text{int}} = 0.000$) Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma w (Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0010Fo^2 + 0.4000\sigma(Fo^2) + 0.0400]/(4Fo^2)$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	1878
No. Variables	192
Reflection/Parameter Ratio	9.78
Residuals: R (All reflections)	0.111
Residuals: R1 ($I > 2.00\sigma(I)$)	0.061
Residuals: wR2 (All reflections)	0.185
Goodness of Fit Indicator	0.908
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	$0.36 \text{ e}^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-0.32 \text{ e}^-/\text{\AA}^3$

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$

atom	x	y	z	B_{eq}
C(10)	0.1153(5)	0.1380(5)	0.2666(3)	5.5(1)
N(2)	0.0404(4)	0.0792(4)	0.2273(3)	6.9(1)
N(1)	0.4345(4)	0.1334(5)	0.2754(4)	8.0(1)
C(8)	0.2092(4)	0.2159(4)	0.3192(3)	5.1(1)
C(11)	0.1822(5)	0.2183(4)	0.4413(3)	5.4(1)
C(6)	0.2205(5)	0.3563(4)	0.1540(4)	5.9(1)
C(9)	0.3354(5)	0.1657(4)	0.2943(4)	5.9(1)
C(12)	0.2910(5)	0.2744(5)	0.5024(4)	6.6(1)
C(13)	0.2548(6)	0.2903(5)	0.6193(4)	7.5(2)
C(7)	0.1994(6)	0.3480(4)	0.2714(3)	6.3(1)
C(1)	0.1220(5)	0.3433(5)	0.0851(4)	6.8(1)
C(2)	0.1427(7)	0.3481(5)	-0.0236(4)	7.5(2)
C(17)	0.2348(7)	-0.0090(5)	0.4734(4)	8.4(2)
C(14)	0.2162(6)	0.1708(5)	0.6674(4)	7.7(2)
C(3)	0.2612(8)	0.3685(5)	-0.0606(5)	8.3(2)
C(15)	0.1118(6)	0.1133(5)	0.6057(4)	7.5(1)
C(16)	0.1413(5)	0.0944(5)	0.4891(4)	6.2(1)
C(4)	0.3599(7)	0.3832(5)	0.0062(5)	8.3(2)
C(5)	0.3383(6)	0.3767(5)	0.1134(5)	7.2(1)

$$B_{\text{eq}} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/ B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.0403(5)	0.3318(5)	0.1129(4)	8.1(2)
H(2)	0.0760(7)	0.3390(5)	-0.0721(4)	9.0(2)
H(3)	0.2739(8)	0.3711(5)	-0.1340(5)	9.9(2)
H(4)	0.4404(7)	0.3975(5)	-0.0231(5)	9.9(2)
H(5)	0.4070(6)	0.3838(5)	0.1601(5)	8.6(2)
H(6)	0.1179(6)	0.3784(4)	0.2857(3)	7.5(2)
H(7)	0.2599(6)	0.3979(4)	0.3049(3)	7.5(2)
H(8)	0.1144(5)	0.2731(4)	0.4509(3)	6.4(1)
H(9)	0.3594(5)	0.2194(5)	0.4976(4)	7.9(2)
H(10)	0.3136(5)	0.3498(5)	0.4716(4)	7.9(2)
H(11)	0.3221(6)	0.3250(5)	0.6575(4)	9.0(2)
H(12)	0.1842(6)	0.3428(5)	0.6227(4)	9.0(2)
H(13)	0.2857(6)	0.1175(5)	0.6600(4)	9.3(2)
H(14)	0.1970(6)	0.1802(5)	0.7394(4)	9.3(2)
H(15)	0.0913(6)	0.0376(5)	0.6371(4)	9.1(2)
H(16)	0.0428(6)	0.1674(5)	0.6120(4)	9.1(2)
H(17)	0.0669(5)	0.0691(5)	0.4547(4)	7.5(2)
H(18)	0.2049(7)	-0.0824(5)	0.5037(4)	10.1(2)
H(19)	0.2518(7)	-0.0219(5)	0.4015(4)	10.1(2)
H(20)	0.3094(7)	0.0156(5)	0.5079(4)	10.1(2)

$$B_{eq} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 3. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(10)	0.083(3)	0.066(3)	0.061(2)	0.010(3)	0.005(3)	0.004(2)
N(2)	0.088(3)	0.090(3)	0.085(3)	-0.011(3)	-0.009(2)	0.002(3)
N(1)	0.085(3)	0.102(3)	0.118(4)	0.014(3)	0.001(3)	-0.030(3)
C(8)	0.071(3)	0.053(2)	0.069(3)	0.005(2)	-0.003(2)	-0.005(2)
C(11)	0.077(3)	0.059(2)	0.068(3)	0.001(2)	-0.004(2)	-0.002(2)
C(6)	0.096(3)	0.052(2)	0.075(3)	0.001(3)	0.009(3)	0.003(2)
C(9)	0.073(3)	0.064(3)	0.086(3)	0.003(3)	-0.009(3)	-0.013(3)
C(12)	0.080(3)	0.081(3)	0.089(3)	0.000(3)	0.001(3)	-0.008(3)
C(13)	0.114(4)	0.096(4)	0.075(3)	-0.004(4)	-0.015(3)	-0.015(3)
C(7)	0.110(4)	0.056(2)	0.072(3)	0.010(3)	0.003(3)	-0.002(2)
C(1)	0.095(4)	0.075(3)	0.088(3)	0.008(3)	-0.002(3)	0.015(3)
C(2)	0.124(5)	0.079(4)	0.082(4)	-0.005(4)	-0.019(4)	0.016(3)
C(17)	0.157(5)	0.063(3)	0.099(4)	0.028(4)	-0.009(4)	0.001(3)
C(14)	0.130(5)	0.092(4)	0.071(3)	0.006(4)	-0.016(3)	0.001(3)
C(3)	0.171(7)	0.061(3)	0.081(4)	0.009(4)	0.021(4)	0.009(3)
C(15)	0.128(4)	0.079(3)	0.080(3)	-0.007(4)	-0.007(4)	0.010(3)
C(16)	0.098(4)	0.066(3)	0.072(3)	-0.006(3)	-0.002(3)	0.002(2)
C(4)	0.125(5)	0.086(4)	0.105(4)	-0.004(4)	0.034(4)	0.009(4)
C(5)	0.108(4)	0.075(3)	0.090(4)	-0.005(3)	0.002(3)	0.005(3)

The general temperature factor expression: $\exp(-2\pi^2(a^*2U_{11}h^2 + b^*2U_{22}k^2 + c^*2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
C(10)	N(2)	1.145(7)	C(10)	C(8)	1.481(7)
N(1)	C(9)	1.141(7)	C(8)	C(11)	1.590(6)
C(8)	C(9)	1.491(7)	C(8)	C(7)	1.579(6)
C(11)	C(12)	1.530(7)	C(11)	C(16)	1.557(7)
C(6)	C(7)	1.523(6)	C(6)	C(1)	1.380(8)
C(6)	C(5)	1.379(8)	C(12)	C(13)	1.557(7)
C(13)	C(14)	1.508(8)	C(1)	C(2)	1.411(7)
C(2)	C(3)	1.37(1)	C(17)	C(16)	1.526(8)
C(14)	C(15)	1.505(8)	C(3)	C(4)	1.37(1)
C(15)	C(16)	1.540(7)	C(4)	C(5)	1.394(9)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(11)	H(8)	0.950(7)	C(12)	H(9)	0.950(7)
C(12)	H(10)	0.950(8)	C(13)	H(11)	0.950(8)
C(13)	H(12)	0.950(8)	C(7)	H(6)	0.950(8)
C(7)	H(7)	0.950(7)	C(1)	H(1)	0.950(8)
C(2)	H(2)	0.950(9)	C(17)	H(18)	0.950(8)
C(17)	H(19)	0.950(8)	C(17)	H(20)	0.95(1)
C(14)	H(13)	0.950(9)	C(14)	H(14)	0.950(8)
C(3)	H(3)	0.950(9)	C(15)	H(15)	0.950(8)
C(15)	H(16)	0.950(8)	C(16)	H(17)	0.950(8)
C(4)	H(4)	0.95(1)	C(5)	H(5)	0.950(9)

Table 6. Bond angles ($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
N(2)	C(10)	C(8)	178.3(5)	C(11)	C(8)	C(9)	112.4(4)
C(11)	C(8)	C(7)	110.8(3)	C(11)	C(8)	C(10)	109.5(4)
C(9)	C(8)	C(7)	108.5(4)	C(9)	C(8)	C(10)	107.5(4)
C(7)	C(8)	C(10)	108.1(4)	C(12)	C(11)	C(16)	111.4(4)
C(12)	C(11)	C(8)	111.9(4)	C(16)	C(11)	C(8)	115.1(4)
C(7)	C(6)	C(1)	120.9(5)	C(7)	C(6)	C(5)	121.1(5)
C(1)	C(6)	C(5)	118.0(5)	N(1)	C(9)	C(8)	176.4(5)
C(13)	C(12)	C(11)	110.4(4)	C(14)	C(13)	C(12)	111.3(5)
C(8)	C(7)	C(6)	115.3(4)	C(2)	C(1)	C(6)	120.5(5)
C(3)	C(2)	C(1)	119.5(6)	C(15)	C(14)	C(13)	110.7(4)
C(4)	C(3)	C(2)	121.0(6)	C(16)	C(15)	C(14)	114.5(5)
C(11)	C(16)	C(17)	114.6(4)	C(11)	C(16)	C(15)	108.7(4)
C(17)	C(16)	C(15)	111.2(4)	C(5)	C(4)	C(3)	118.9(7)
C(6)	C(5)	C(4)	122.1(6)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(12)	C(11)	H(8)	104.9(5)	C(16)	C(11)	H(8)	106.8(5)
H(8)	C(11)	C(8)	106.0(5)	C(13)	C(12)	H(9)	109.0(6)
C(13)	C(12)	H(10)	111.4(6)	H(9)	C(12)	H(10)	109.5(7)
H(9)	C(12)	C(11)	107.1(6)	H(10)	C(12)	C(11)	109.4(5)
C(14)	C(13)	H(11)	110.2(6)	C(14)	C(13)	H(12)	107.1(6)
H(11)	C(13)	H(12)	109.5(7)	H(11)	C(13)	C(12)	110.7(6)
H(12)	C(13)	C(12)	108.0(5)	H(6)	C(7)	H(7)	109.5(6)
H(6)	C(7)	C(8)	108.1(5)	H(6)	C(7)	C(6)	107.7(5)
H(7)	C(7)	C(8)	108.1(5)	H(7)	C(7)	C(6)	108.1(5)
C(2)	C(1)	H(1)	121.3(6)	H(1)	C(1)	C(6)	118.2(6)
C(3)	C(2)	H(2)	118.9(7)	H(2)	C(2)	C(1)	121.5(7)
C(16)	C(17)	H(18)	111.0(7)	C(16)	C(17)	H(19)	111.4(6)
C(16)	C(17)	H(20)	105.9(6)	H(18)	C(17)	H(19)	109.5(7)
H(18)	C(17)	H(20)	109.5(8)	H(19)	C(17)	H(20)	109.5(9)
C(15)	C(14)	H(13)	105.4(6)	C(15)	C(14)	H(14)	113.3(7)
H(13)	C(14)	H(14)	109.5(7)	H(13)	C(14)	C(13)	106.5(6)
H(14)	C(14)	C(13)	111.1(6)	C(4)	C(3)	H(3)	120(1)
H(3)	C(3)	C(2)	118.6(9)	C(16)	C(15)	H(15)	109.8(5)
C(16)	C(15)	H(16)	109.0(5)	H(15)	C(15)	H(16)	109.5(7)
H(15)	C(15)	C(14)	108.5(6)	H(16)	C(15)	C(14)	105.5(6)
H(17)	C(16)	C(11)	107.9(5)	H(17)	C(16)	C(17)	105.6(5)
H(17)	C(16)	C(15)	108.6(6)	C(5)	C(4)	H(4)	123.2(8)
H(4)	C(4)	C(3)	117.9(8)	H(5)	C(5)	C(6)	118.6(7)
H(5)	C(5)	C(4)	119.3(7)				

Table 8. Torsion Angles($^{\circ}$)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(10)	C(8)	C(11)	C(12)	167.9(4)	C(10)	C(8)	C(11)	C(16)	39.5(5)
C(9)	C(8)	C(11)	C(12)	48.5(5)	C(9)	C(8)	C(11)	C(16)	-79.9(5)
C(7)	C(8)	C(11)	C(12)	-73.0(5)	C(7)	C(8)	C(11)	C(16)	158.6(4)
C(10)	C(8)	C(7)	C(6)	-58.6(6)	C(11)	C(8)	C(7)	C(6)	-178.6(4)
C(9)	C(8)	C(7)	C(6)	57.6(6)	C(8)	C(11)	C(12)	C(13)	172.8(4)
C(16)	C(11)	C(12)	C(13)	-56.8(5)	C(8)	C(11)	C(16)	C(17)	58.0(6)
C(8)	C(11)	C(16)	C(15)	-176.9(4)	C(12)	C(11)	C(16)	C(17)	-70.7(5)
C(12)	C(11)	C(16)	C(15)	54.5(5)	C(1)	C(6)	C(7)	C(8)	88.9(6)
C(5)	C(6)	C(7)	C(8)	-91.0(6)	C(7)	C(6)	C(1)	C(2)	-178.4(5)
C(5)	C(6)	C(1)	C(2)	1.4(7)	C(7)	C(6)	C(5)	C(4)	179.3(5)
C(1)	C(6)	C(5)	C(4)	-0.6(8)	C(11)	C(12)	C(13)	C(14)	57.0(6)
C(12)	C(13)	C(14)	C(15)	-55.3(6)	C(6)	C(1)	C(2)	C(3)	-1.4(8)
C(1)	C(2)	C(3)	C(4)	0.5(8)	C(13)	C(14)	C(15)	C(16)	55.7(6)
C(2)	C(3)	C(4)	C(5)	0.3(9)	C(14)	C(15)	C(16)	C(11)	-54.4(6)
C(14)	C(15)	C(16)	C(17)	72.7(6)	C(3)	C(4)	C(5)	C(6)	-0.3(9)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Torsion Angles involving hydrogens($^{\circ}$)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(10)	C(8)	C(11)	H(8)	-78.4(5)	C(9)	C(8)	C(11)	H(8)	162.3(5)
C(7)	C(8)	C(11)	H(8)	40.8(6)	C(10)	C(8)	C(7)	H(6)	61.9(6)
C(10)	C(8)	C(7)	H(7)	-179.7(5)	C(11)	C(8)	C(7)	H(6)	-58.1(6)
C(11)	C(8)	C(7)	H(7)	60.3(6)	C(9)	C(8)	C(7)	H(6)	178.2(5)
C(9)	C(8)	C(7)	H(7)	-63.4(6)	C(8)	C(11)	C(12)	H(9)	-68.6(6)
C(8)	C(11)	C(12)	H(10)	49.9(7)	C(16)	C(11)	C(12)	H(9)	61.7(6)
C(16)	C(11)	C(12)	H(10)	-179.8(5)	H(8)	C(11)	C(12)	C(13)	58.3(6)
H(8)	C(11)	C(12)	H(9)	176.9(6)	H(8)	C(11)	C(12)	H(10)	-64.6(7)
C(8)	C(11)	C(16)	H(17)	-59.3(6)	C(12)	C(11)	C(16)	H(17)	172.1(5)
H(8)	C(11)	C(16)	C(17)	175.3(5)	H(8)	C(11)	C(16)	C(15)	-59.5(6)
H(8)	C(11)	C(16)	H(17)	58.1(7)	C(1)	C(6)	C(7)	H(6)	-31.9(7)
C(1)	C(6)	C(7)	H(7)	-150.1(6)	C(5)	C(6)	C(7)	H(6)	148.2(5)
C(5)	C(6)	C(7)	H(7)	30.0(7)	C(7)	C(6)	C(1)	H(1)	2.0(8)
C(5)	C(6)	C(1)	H(1)	-178.1(6)	C(7)	C(6)	C(5)	H(5)	1.3(8)
C(1)	C(6)	C(5)	H(5)	-178.6(6)	C(11)	C(12)	C(13)	H(11)	179(804)
C(11)	C(12)	C(13)	H(12)	-60.3(7)	H(9)	C(12)	C(13)	C(14)	-60.4(7)
H(9)	C(12)	C(13)	H(11)	62.6(8)	H(9)	C(12)	C(13)	H(12)	-177.6(7)
H(10)	C(12)	C(13)	C(14)	178.8(6)	H(10)	C(12)	C(13)	H(11)	-58.3(8)
H(10)	C(12)	C(13)	H(12)	61.5(8)	C(12)	C(13)	C(14)	H(13)	58.8(7)
C(12)	C(13)	C(14)	H(14)	177.9(7)	H(11)	C(13)	C(14)	C(15)	-178.5(6)
H(11)	C(13)	C(14)	H(13)	-64.4(8)	H(11)	C(13)	C(14)	H(14)	54.7(9)
H(12)	C(13)	C(14)	C(15)	62.5(7)	H(12)	C(13)	C(14)	H(13)	176.6(7)
H(12)	C(13)	C(14)	H(14)	-64.3(9)	C(6)	C(1)	C(2)	H(2)	179(359)
H(1)	C(1)	C(2)	C(3)	178.1(6)	H(1)	C(1)	C(2)	H(2)	0(1)
C(1)	C(2)	C(3)	H(3)	179(359)	H(2)	C(2)	C(3)	C(4)	179.4(7)
H(2)	C(2)	C(3)	H(3)	-1(1)	H(18)	C(17)	C(16)	C(11)	-179.6(6)
H(18)	C(17)	C(16)	C(15)	56.6(8)	H(18)	C(17)	C(16)	H(17)	-61.1(8)
H(19)	C(17)	C(16)	C(11)	-57.4(8)	H(19)	C(17)	C(16)	C(15)	178.8(7)
H(19)	C(17)	C(16)	H(17)	61.2(9)	H(20)	C(17)	C(16)	C(11)	61.6(7)
H(20)	C(17)	C(16)	C(15)	-62.2(7)	H(20)	C(17)	C(16)	H(17)	-179(719)
C(13)	C(14)	C(15)	H(15)	178.7(6)	C(13)	C(14)	C(15)	H(16)	-64.1(7)
H(13)	C(14)	C(15)	C(16)	-59.1(7)	H(13)	C(14)	C(15)	H(15)	63.9(8)
H(13)	C(14)	C(15)	H(16)	-178.9(7)	H(14)	C(14)	C(15)	C(16)	-178.8(6)
H(14)	C(14)	C(15)	H(15)	-55.8(9)	H(14)	C(14)	C(15)	H(16)	61.4(8)
C(2)	C(3)	C(4)	H(4)	-179(508)	H(3)	C(3)	C(4)	C(5)	-178.9(7)
H(3)	C(3)	C(4)	H(4)	1(1)	C(14)	C(15)	C(16)	H(17)	-171.6(6)
H(15)	C(15)	C(16)	C(11)	-176.7(6)	H(15)	C(15)	C(16)	C(17)	-49.6(8)

Table 9. Torsion angles involving hydrogens (°) -- continued

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
H(15)	C(15)	C(16)	H(17)	66.2(8)	H(16)	C(15)	C(16)	C(11)	63.4(7)
H(16)	C(15)	C(16)	C(17)	-169.5(6)	H(16)	C(15)	C(16)	H(17)	-53.7(8)
C(3)	C(4)	C(5)	H(5)	177.7(6)	H(4)	C(4)	C(5)	C(6)	179(508)
H(4)	C(4)	C(5)	H(5)	-2(1)					

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

X-ray crystallographic Data of *endo*-6d

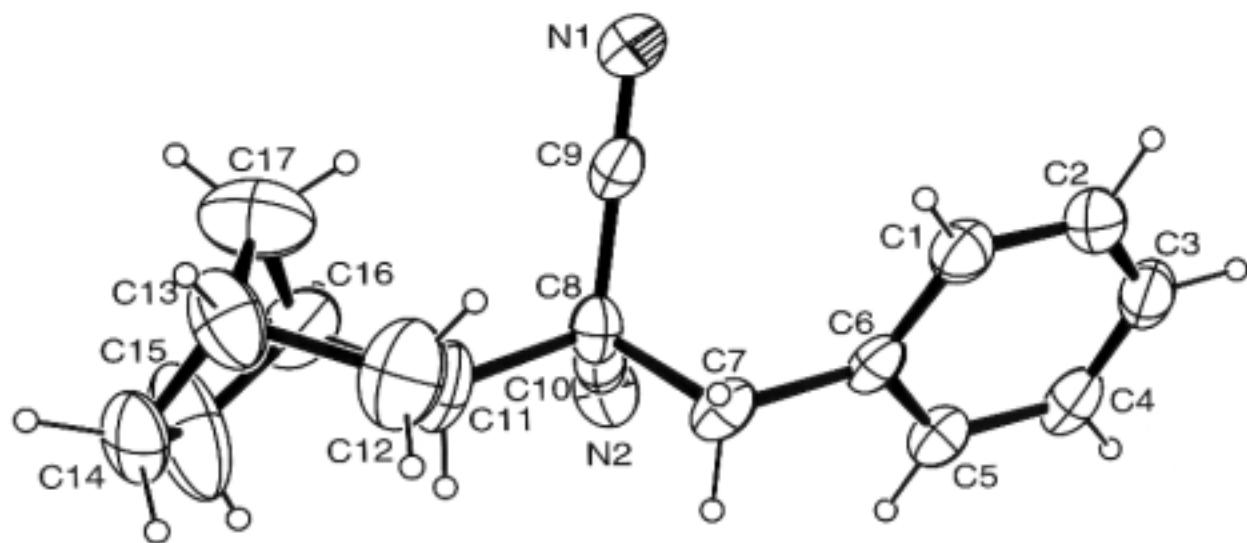


Figure S5. ORTEP drawing of *endo*-6d (ellipsoids at 20% probability).

Experimental

Data Collection

A colorless platelet crystal of C₁₇H₁₈N₂ having approximate dimensions of 0.60 x 0.50 x 0.05 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-K α radiation.

Indexing was performed from 3 ° oscillations that were exposed for 36 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions:

$$\begin{aligned}a &= 17.61(2) \text{ \AA} \\b &= 9.93(1) \text{ \AA} \\c &= 8.376(9) \text{ \AA} \\V &= 1463(2) \text{ \AA}^3\end{aligned}$$

For Z = 4 and F.W. = 250.34, the calculated density is 1.14 g/cm³. Based on the systematic absences of:

$$\begin{aligned}0kl: \quad k+l &\pm 2n \\h0l: \quad h &\pm 2n\end{aligned}$$

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

Pna2₁ (#33)

The data were collected at a temperature of 23 ± 10C to a maximum 2θ value of 54.9°. A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0° in 5.0° step, at χ=45.0° and φ = 0.0°. The exposure rate was 150.0 [sec./0]. A second sweep was performed using ω scans from 0.0 to 160.0° in

5.0° step, at $\chi=45.0^\circ$ and $\phi = 180.0^\circ$. The exposure rate was 150.0 [sec./0]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 28490 reflections that were collected, 1946 were unique ($R_{\text{int}} = 0.056$); equivalent reflections were merged.

The linear absorption coefficient, μ , for Mo-K α radiation is 0.7 cm $^{-1}$. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement³ on F^2 was based on 1788 observed reflections and 190 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R_1 = \sum ||F_O| - |F_C|| / \sum |F_O| = 0.098$$

$$wR_2 = [\sum (w(F_O^2 - F_C^2)^2) / \sum w(F_O^2)^2]^{1/2} = 0.223$$

The standard deviation of an observation of unit weight⁴ was 1.23. A Sheldrick weighting scheme was used. Plots of $\sum w(|F_O| - |F_C|)^2$ versus $|F_O|$, reflection order in data collection, $\sin \theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.43 and -0.46 e $^-/\text{\AA}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in F_{calc} ⁶; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbell⁸. All calculations were performed using the CrystalStructure^{9,10} crystallographic software package.

References

- (1) SHELX97: Sheldrick, G.M. (1997).
- (2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
- (3) Least Squares function minimized:
- $$\sum w(F_o^2 - F_c^2)^2 \quad \text{where } w = \text{Least Squares weights.}$$
- (4) Standard deviation of an observation of unit weight:
- $$[\sum w(F_o^2 - F_c^2)^2 / (N_o - N_v)]^{1/2}$$
- where: N_o = number of observations
 N_v = number of variables
- (5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).
- (6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).
- (7) Creagh, D. C. & McAuley, W.J.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).
- (8) Creagh, D. C. & Hubbell, J.H.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).
- (9) CrystalStructure 3.5.1: Crystal Structure Analysis Package, Rigaku and Rigaku/MSC (2000-2003). 9009 New Trails Dr. The Woodlands TX 77381 USA.
- (10) CRYSTALS Issue 10: Watkin, D.J., Prout, C.K. Carruthers, J.R. & Betteridge, P.W. Chemical Crystallography Laboratory, Oxford, UK. (1996)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₁₇ H ₁₈ N ₂
Formula Weight	250.34
Crystal Color, Habit	colorless, platelet
Crystal Dimensions	0.60 X 0.50 X 0.05 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 36.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 17.61(2) Å b = 9.93(1) Å c = 8.376(9) Å V = 1463(2) Å ³
Space Group	Pna2 ₁ (#33)
Z value	4
D _{calc}	1.136 g/cm ³
F ₀₀₀	536.00
μ(MoKα)	0.67 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71075 \text{ \AA}$) graphite monochromated
Detector Aperture	270 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0, \phi=0.0$)	130.0 - 190.0°
Exposure Rate	150.0 sec./°
ω oscillation Range ($\chi=45.0, \phi=180.0$)	0.0 - 160.0°
Exposure Rate	150.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	54.9°
No. of Reflections Measured	Total: 28490
Corrections	Unique: 1946 ($R_{\text{int}} = 0.056$) Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma w (Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0020Fo^2+3.0000\sigma(Fo^2)+0.5000]/(4Fo^2)$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	1788
No. Variables	190
Reflection/Parameter Ratio	9.41
Residuals: R (All reflections)	0.142
Residuals: R1 ($I > 2.00\sigma(I)$)	0.098
Residuals: wR2 (All reflections)	0.223
Goodness of Fit Indicator	1.228
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.43 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.46 e ⁻ /Å ³

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$

atom	x	y	z	B_{eq}
N(1)	0.1745(3)	-0.0585(6)	0.6965(9)	8.5(2)
N(2)	0.3244(3)	-0.0924(7)	0.3006(9)	8.6(2)
C(1)	0.0940(4)	0.1569(6)	0.4046(8)	6.9(2)
C(2)	0.0328(4)	0.1291(7)	0.311(1)	7.7(2)
C(3)	0.0407(5)	0.1040(6)	0.154(1)	8.0(2)
C(4)	0.1116(5)	0.1045(6)	0.0854(9)	7.7(2)
C(5)	0.1736(4)	0.1341(6)	0.1792(8)	6.8(2)
C(6)	0.1680(3)	0.1603(5)	0.3421(7)	5.6(1)
C(7)	0.2349(3)	0.1954(5)	0.4430(7)	6.5(1)
C(8)	0.2747(3)	0.0719(6)	0.5232(8)	6.2(1)
C(9)	0.2179(4)	-0.0032(6)	0.6204(8)	6.4(1)
C(10)	0.3032(3)	-0.0232(6)	0.3980(9)	6.5(2)
C(11)	0.3482(5)	0.1205(7)	0.619(1)	9.9(2)
C(12)	0.3382(8)	0.220(1)	0.743(1)	15.0(4)
C(13)	0.3878(5)	0.170(1)	0.885(1)	10.9(3)
C(14)	0.4646(4)	0.1831(8)	0.836(1)	9.8(2)
C(15)	0.4706(5)	0.073(2)	0.708(2)	16.6(5)
C(16)	0.4014(7)	0.0080(7)	0.697(1)	11.0(3)
C(17)	0.3668(7)	0.030(1)	0.860(2)	13.6(4)

$$B_{\text{eq}} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/ B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.0871(4)	0.1810(6)	0.5134(8)	8.3(2)
H(2)	-0.0166(4)	0.1250(7)	0.356(1)	9.3(2)
H(3)	-0.0027(5)	0.0841(6)	0.091(1)	9.6(2)
H(4)	0.1158(5)	0.0872(6)	-0.0258(9)	9.2(2)
H(5)	0.2230(4)	0.1366(6)	0.1333(8)	8.1(2)
H(6)	0.2712(3)	0.2354(5)	0.3739(7)	7.8(2)
H(7)	0.2204(3)	0.2586(5)	0.5226(7)	7.8(2)
H(8)	0.3794(5)	0.1626(7)	0.541(1)	11.9(3)
H(9)	0.3519(8)	0.309(1)	0.715(1)	18.1(5)
H(10)	0.2851(8)	0.216(1)	0.764(1)	18.0(5)
H(11)	0.3766(5)	0.209(1)	0.986(1)	13.1(3)
H(12)	0.4741(4)	0.2698(8)	0.792(1)	11.8(3)
H(13)	0.5002(4)	0.1653(8)	0.918(1)	11.8(3)
H(14)	0.4803(5)	0.114(2)	0.608(2)	19.9(6)
H(15)	0.5102(5)	0.012(2)	0.733(2)	19.9(6)
H(16)	0.3981(7)	-0.0802(7)	0.653(1)	13.2(3)
H(17)	0.3833(7)	-0.031(1)	0.940(2)	16.3(4)
H(18)	0.3132(7)	0.025(1)	0.849(2)	16.3(4)

$$B_{eq} = \frac{8}{3} \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 3. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
N(1)	0.110(4)	0.109(4)	0.102(4)	-0.001(3)	-0.009(4)	0.035(4)
N(2)	0.101(4)	0.111(4)	0.114(5)	0.005(3)	-0.013(4)	-0.023(4)
C(1)	0.101(4)	0.085(4)	0.078(4)	0.017(3)	0.006(4)	0.006(3)
C(2)	0.087(4)	0.106(5)	0.101(6)	0.006(3)	0.000(4)	0.018(4)
C(3)	0.112(6)	0.084(4)	0.108(6)	0.005(4)	-0.041(5)	0.017(4)
C(4)	0.129(6)	0.091(4)	0.071(4)	0.018(4)	-0.020(5)	0.016(3)
C(5)	0.099(4)	0.077(3)	0.080(4)	0.012(3)	-0.006(4)	0.014(3)
C(6)	0.092(4)	0.055(3)	0.065(3)	0.005(3)	-0.010(3)	0.010(3)
C(7)	0.108(4)	0.060(3)	0.078(4)	0.007(3)	-0.005(4)	0.010(3)
C(8)	0.080(3)	0.083(4)	0.071(4)	0.001(3)	-0.021(3)	-0.009(3)
C(9)	0.098(4)	0.071(3)	0.073(3)	0.007(3)	-0.020(4)	0.014(3)
C(10)	0.071(3)	0.079(4)	0.096(5)	0.002(3)	-0.018(3)	0.004(4)
C(11)	0.169(7)	0.099(5)	0.109(6)	0.002(5)	-0.073(6)	-0.002(5)
C(12)	0.27(1)	0.162(9)	0.142(8)	0.075(9)	-0.044(9)	-0.040(8)
C(13)	0.121(6)	0.170(9)	0.124(7)	0.006(6)	-0.024(6)	-0.072(6)
C(14)	0.118(6)	0.108(5)	0.148(7)	-0.007(4)	-0.057(6)	-0.036(5)
C(15)	0.116(6)	0.29(1)	0.22(1)	-0.006(8)	-0.028(8)	-0.14(1)
C(16)	0.23(1)	0.065(4)	0.117(6)	-0.026(5)	-0.039(7)	-0.007(4)
C(17)	0.21(1)	0.124(7)	0.18(1)	-0.009(7)	0.052(9)	-0.014(8)

The general temperature factor expression: $\exp(-2\pi^2(a^*2U_{11}h^2 + b^*2U_{22}k^2 + c^*2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
N(1)	C(9)	1.135(9)	N(2)	C(10)	1.13(1)
C(1)	C(2)	1.36(1)	C(1)	C(6)	1.404(9)
C(2)	C(3)	1.34(1)	C(3)	C(4)	1.38(1)
C(4)	C(5)	1.38(1)	C(5)	C(6)	1.393(9)
C(6)	C(7)	1.491(8)	C(7)	C(8)	1.564(8)
C(8)	C(9)	1.490(9)	C(8)	C(10)	1.498(9)
C(8)	C(11)	1.60(1)	C(11)	C(12)	1.44(1)
C(11)	C(16)	1.60(1)	C(12)	C(13)	1.55(2)
C(13)	C(14)	1.42(1)	C(13)	C(17)	1.45(1)
C(14)	C(15)	1.53(2)	C(15)	C(16)	1.38(2)
C(16)	C(17)	1.51(2)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.95(1)	C(2)	H(2)	0.95(1)
C(3)	H(3)	0.95(1)	C(4)	H(4)	0.95(1)
C(5)	H(5)	0.950(9)	C(7)	H(6)	0.950(8)
C(7)	H(7)	0.950(8)	C(11)	H(8)	0.95(1)
C(12)	H(9)	0.95(2)	C(12)	H(10)	0.95(2)
C(13)	H(11)	0.95(1)	C(14)	H(12)	0.95(1)
C(14)	H(13)	0.95(1)	C(15)	H(14)	0.95(2)
C(15)	H(15)	0.95(2)	C(16)	H(16)	0.95(1)
C(17)	H(17)	0.95(2)	C(17)	H(18)	0.95(2)

Table 6. Bond angles ($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(2)	C(1)	C(6)	121.5(6)	C(3)	C(2)	C(1)	121.3(7)
C(4)	C(3)	C(2)	120.3(7)	C(5)	C(4)	C(3)	118.7(7)
C(6)	C(5)	C(4)	122.8(6)	C(7)	C(6)	C(1)	121.8(5)
C(7)	C(6)	C(5)	122.9(5)	C(1)	C(6)	C(5)	115.3(6)
C(8)	C(7)	C(6)	114.5(4)	C(9)	C(8)	C(10)	107.0(5)
C(9)	C(8)	C(11)	114.8(6)	C(9)	C(8)	C(7)	109.0(5)
C(10)	C(8)	C(11)	105.8(5)	C(10)	C(8)	C(7)	110.1(5)
C(11)	C(8)	C(7)	110.1(5)	N(1)	C(9)	C(8)	178.7(7)
N(2)	C(10)	C(8)	178.2(7)	C(12)	C(11)	C(16)	105.0(8)
C(12)	C(11)	C(8)	118.0(8)	C(16)	C(11)	C(8)	117.9(6)
C(13)	C(12)	C(11)	105.4(9)	C(14)	C(13)	C(17)	106.7(9)
C(14)	C(13)	C(12)	106.6(8)	C(17)	C(13)	C(12)	92.8(9)
C(15)	C(14)	C(13)	101.8(7)	C(16)	C(15)	C(14)	108.8(8)
C(17)	C(16)	C(11)	91.6(8)	C(17)	C(16)	C(15)	102.8(9)
C(11)	C(16)	C(15)	102.5(8)	C(13)	C(17)	C(16)	99.6(9)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(2)	C(1)	H(1)	120.3(8)	C(6)	C(1)	H(1)	118.1(7)
C(3)	C(2)	H(2)	118.6(9)	H(2)	C(2)	C(1)	120.0(9)
C(4)	C(3)	H(3)	119.9(9)	H(3)	C(3)	C(2)	119.8(9)
C(5)	C(4)	H(4)	122.4(9)	H(4)	C(4)	C(3)	118.8(9)
C(6)	C(5)	H(5)	117.2(7)	H(5)	C(5)	C(4)	120.0(8)
C(8)	C(7)	H(6)	106.7(6)	C(8)	C(7)	H(7)	109.6(6)
H(6)	C(7)	H(7)	109.5(7)	H(6)	C(7)	C(6)	106.5(6)
H(7)	C(7)	C(6)	109.9(6)	C(12)	C(11)	H(8)	105.5(9)
C(16)	C(11)	H(8)	104(1)	H(8)	C(11)	C(8)	104.7(9)
C(13)	C(12)	H(9)	109(1)	C(13)	C(12)	H(10)	113(1)
H(9)	C(12)	H(10)	109(1)	H(9)	C(12)	C(11)	115(1)
H(10)	C(12)	C(11)	102(1)	C(14)	C(13)	H(11)	114(1)
C(17)	C(13)	H(11)	117(1)	H(11)	C(13)	C(12)	116(1)
C(15)	C(14)	H(12)	111(1)	C(15)	C(14)	H(13)	109.3(9)
H(12)	C(14)	H(13)	109(1)	H(12)	C(14)	C(13)	111.2(9)
H(13)	C(14)	C(13)	113.6(9)	C(16)	C(15)	H(14)	107(1)
C(16)	C(15)	H(15)	111(1)	H(14)	C(15)	H(15)	109(1)
H(14)	C(15)	C(14)	109(1)	H(15)	C(15)	C(14)	111(1)
C(17)	C(16)	H(16)	117(1)	H(16)	C(16)	C(11)	116(1)
H(16)	C(16)	C(15)	120(1)	H(17)	C(17)	H(18)	109(1)
H(17)	C(17)	C(13)	115(1)	H(17)	C(17)	C(16)	115(1)
H(18)	C(17)	C(13)	108(1)	H(18)	C(17)	C(16)	107(1)

Table 8. Torsion Angles($^{\circ}$)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(6)	C(1)	C(2)	C(3)	0(1)	C(2)	C(1)	C(6)	C(5)	0.2(8)
C(2)	C(1)	C(6)	C(7)	177.8(5)	C(1)	C(2)	C(3)	C(4)	0(1)
C(2)	C(3)	C(4)	C(5)	-1.4(9)	C(3)	C(4)	C(5)	C(6)	1.7(9)
C(4)	C(5)	C(6)	C(1)	-1.1(8)	C(4)	C(5)	C(6)	C(7)	-178.6(5)
C(1)	C(6)	C(7)	C(8)	91.4(6)	C(5)	C(6)	C(7)	C(8)	-91.3(6)
C(6)	C(7)	C(8)	C(9)	-56.6(7)	C(6)	C(7)	C(8)	C(10)	60.4(6)
C(6)	C(7)	C(8)	C(11)	176.6(5)	C(7)	C(8)	C(11)	C(12)	57.8(9)
C(7)	C(8)	C(11)	C(16)	-174.4(6)	C(9)	C(8)	C(11)	C(12)	-65.6(9)
C(9)	C(8)	C(11)	C(16)	62.2(9)	C(10)	C(8)	C(11)	C(12)	176.7(8)
C(10)	C(8)	C(11)	C(16)	-55.5(8)	C(8)	C(11)	C(12)	C(13)	136.8(8)
C(16)	C(11)	C(12)	C(13)	3(1)	C(8)	C(11)	C(16)	C(15)	156.0(9)
C(8)	C(11)	C(16)	C(17)	-100.5(8)	C(12)	C(11)	C(16)	C(15)	-70(1)
C(12)	C(11)	C(16)	C(17)	33.3(9)	C(11)	C(12)	C(13)	C(14)	68(1)
C(11)	C(12)	C(13)	C(17)	-39(1)	C(12)	C(13)	C(14)	C(15)	-69(1)
C(17)	C(13)	C(14)	C(15)	28(1)	C(12)	C(13)	C(17)	C(16)	64.3(9)
C(14)	C(13)	C(17)	C(16)	-43(1)	C(13)	C(14)	C(15)	C(16)	-1(1)
C(14)	C(15)	C(16)	C(11)	69(1)	C(14)	C(15)	C(16)	C(17)	-25(1)
C(11)	C(16)	C(17)	C(13)	-61.7(9)	C(15)	C(16)	C(17)	C(13)	41(1)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Torsion Angles involving hydrogens($^{\circ}$)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(6)	C(1)	C(2)	H(2)	178.3(8)	H(1)	C(1)	C(2)	C(3)	175.3(7)
H(1)	C(1)	C(2)	H(2)	-6(1)	H(1)	C(1)	C(6)	C(5)	-175.2(7)
H(1)	C(1)	C(6)	C(7)	2.3(9)	C(1)	C(2)	C(3)	H(3)	179.4(8)
H(2)	C(2)	C(3)	C(4)	-177.7(8)	H(2)	C(2)	C(3)	H(3)	1(1)
C(2)	C(3)	C(4)	H(4)	-179.4(7)	H(3)	C(3)	C(4)	C(5)	179(508)
H(3)	C(3)	C(4)	H(4)	1(1)	C(3)	C(4)	C(5)	H(5)	-178.8(7)
H(4)	C(4)	C(5)	C(6)	179(359)	H(4)	C(4)	C(5)	H(5)	0(1)
H(5)	C(5)	C(6)	C(1)	179.4(6)	H(5)	C(5)	C(6)	C(7)	1.8(9)
C(1)	C(6)	C(7)	H(6)	-151.0(6)	C(1)	C(6)	C(7)	H(7)	-32.5(8)
C(5)	C(6)	C(7)	H(6)	26.4(8)	C(5)	C(6)	C(7)	H(7)	144.8(6)
H(6)	C(7)	C(8)	C(9)	-174.2(6)	H(6)	C(7)	C(8)	C(10)	-57.2(7)
H(6)	C(7)	C(8)	C(11)	59.0(8)	H(7)	C(7)	C(8)	C(9)	67.3(7)
H(7)	C(7)	C(8)	C(10)	-175.6(6)	H(7)	C(7)	C(8)	C(11)	-59.4(8)
C(7)	C(8)	C(11)	H(8)	-59.0(9)	C(9)	C(8)	C(11)	H(8)	177.6(8)
C(10)	C(8)	C(11)	H(8)	59.9(9)	C(8)	C(11)	C(12)	H(9)	-101(1)
C(8)	C(11)	C(12)	H(10)	17(1)	C(16)	C(11)	C(12)	H(9)	124(1)
C(16)	C(11)	C(12)	H(10)	-116(1)	H(8)	C(11)	C(12)	C(13)	-106(1)
H(8)	C(11)	C(12)	H(9)	14(1)	H(8)	C(11)	C(12)	H(10)	133(1)
C(8)	C(11)	C(16)	H(16)	21(1)	C(12)	C(11)	C(16)	H(16)	155(1)
H(8)	C(11)	C(16)	C(15)	40(1)	H(8)	C(11)	C(16)	C(17)	144.0(9)
H(8)	C(11)	C(16)	H(16)	-94(1)	C(11)	C(12)	C(13)	H(11)	-162(1)
H(9)	C(12)	C(13)	C(14)	-56(1)	H(9)	C(12)	C(13)	C(17)	-164(1)
H(9)	C(12)	C(13)	H(11)	72(1)	H(10)	C(12)	C(13)	C(14)	-179(359)
H(10)	C(12)	C(13)	C(17)	72(1)	H(10)	C(12)	C(13)	H(11)	-50(1)
C(12)	C(13)	C(14)	H(12)	49(1)	C(12)	C(13)	C(14)	H(13)	173.1(9)
C(17)	C(13)	C(14)	H(12)	147(1)	C(17)	C(13)	C(14)	H(13)	-88(1)
H(11)	C(13)	C(14)	C(15)	160(1)	H(11)	C(13)	C(14)	H(12)	-80(1)
H(11)	C(13)	C(14)	H(13)	43(1)	C(12)	C(13)	C(17)	H(17)	-171(1)
C(12)	C(13)	C(17)	H(18)	-48(1)	C(14)	C(13)	C(17)	H(17)	79(1)
C(14)	C(13)	C(17)	H(18)	-156(1)	H(11)	C(13)	C(17)	C(16)	-174(1)
H(11)	C(13)	C(17)	H(17)	-50(1)	H(11)	C(13)	C(17)	H(18)	72(1)
C(13)	C(14)	C(15)	H(14)	115(1)	C(13)	C(14)	C(15)	H(15)	-123(1)
H(12)	C(14)	C(15)	C(16)	-119(1)	H(12)	C(14)	C(15)	H(14)	-2(1)
H(12)	C(14)	C(15)	H(15)	117(1)	H(13)	C(14)	C(15)	C(16)	119(1)
H(13)	C(14)	C(15)	H(14)	-123(1)	H(13)	C(14)	C(15)	H(15)	-3(1)
C(14)	C(15)	C(16)	H(16)	-158(1)	H(14)	C(15)	C(16)	C(11)	-48(1)
H(14)	C(15)	C(16)	C(17)	-143(1)	H(14)	C(15)	C(16)	H(16)	83(1)

Table 9. Torsion angles involving hydrogens (°) -- continued

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
H(15)	C(15)	C(16)	C(11)	-168(1)	H(15)	C(15)	C(16)	C(17)	97(1)
H(15)	C(15)	C(16)	H(16)	-35(2)	C(11)	C(16)	C(17)	H(17)	174(1)
C(11)	C(16)	C(17)	H(18)	51(1)	C(15)	C(16)	C(17)	H(17)	-82(1)
C(15)	C(16)	C(17)	H(18)	154(1)	H(16)	C(16)	C(17)	C(13)	176(1)
H(16)	C(16)	C(17)	H(17)	52(2)	H(16)	C(16)	C(17)	H(18)	-69(1)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

X-ray crystallographic Data of *endo*-6e

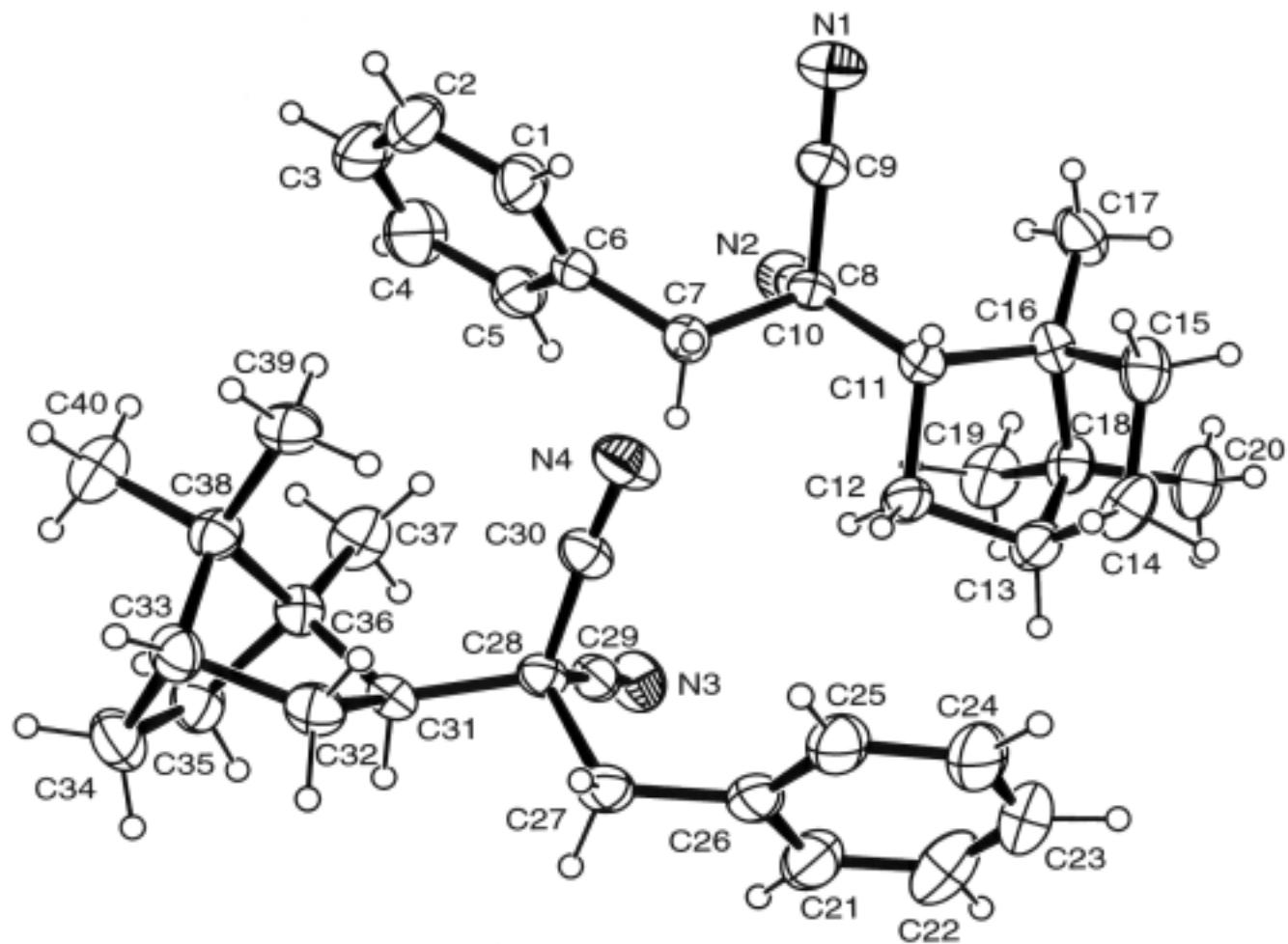


Figure S6. ORTEP drawing of *endo*-6e (ellipsoids at 30% probability).

Experimental

Data Collection

A colorless prism crystal of $C_{20}H_{24}N_2$ having approximate dimensions of $0.75 \times 0.50 \times 0.25$ mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-K α radiation.

Indexing was performed from 3° oscillations that were exposed for 180 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a C-centered triclinic cell with dimensions:

$$\begin{array}{ll} a = 11.7618(7) \text{ \AA} & \alpha = 92.734(3)^\circ \\ b = 13.1850(8) \text{ \AA} & \beta = 112.610(2)^\circ \\ c = 13.7331(8) \text{ \AA} & \gamma = 114.056(3)^\circ \\ V = 1741.7(2) \text{ \AA}^3 & \end{array}$$

For $Z = 4$ and F.W. = 292.42, the calculated density is 1.12 g/cm^3 . Based on the systematic absences of:

$$hkl: h+k \pm 2n$$

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$$C1 (\#1)$$

The data were collected at a temperature of $23 \pm 1^\circ\text{C}$ to a maximum 2θ value of 54.9° . A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0° in 5.0° step, at $\chi=45.0^\circ$ and $\phi = 0.0^\circ$. The exposure rate was $289.8 \text{ [sec./}^\circ]$. A second sweep was performed using ω scans from 0.0 to 160.0° in 5.0° step, at $\chi=45.0^\circ$ and $\phi = 180.0^\circ$. The exposure rate was $289.8 \text{ [sec./}^\circ]$. The

crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

A total of 7730 reflections was collected.

The linear absorption coefficient, μ , for Mo-K α radiation is 0.7 cm $^{-1}$. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.91 to 1.13. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement³ on F^2 was based on 3717 observed reflections and 445 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R_1 = \sum ||F_O| - |F_C|| / \sum |F_O| = 0.061$$

$$wR_2 = [\sum (w (|F_O|^2 - |F_C|^2)^2) / \sum w(|F_O|^2)^2]^{1/2} = 0.132$$

The standard deviation of an observation of unit weight⁴ was 1.06. A Sheldrick weighting scheme was used. Plots of $\sum w (|F_O| - |F_C|)^2$ versus $|F_O|$, reflection order in data collection, $\sin \theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.56 and -0.44 e $^-/\text{\AA}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in F_{calc} ⁶; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbell⁸. All calculations were performed using the CrystalStructure^{9,10} crystallographic software package.

References

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(3) Least Squares function minimized:

$$\sum w(F_o^2 - F_c^2)^2 \quad \text{where } w = \text{Least Squares weights.}$$

(4) Standard deviation of an observation of unit weight:

$$[\sum w(F_o^2 - F_c^2)^2 / (N_o - N_v)]^{1/2}$$

where: N_o = number of observations
 N_v = number of variables

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EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₂₀ H ₂₄ N ₂
Formula Weight	292.42
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.75 X 0.50 X 0.25 mm
Crystal System	triclinic
Lattice Type	C-centered
Indexing Images	3 oscillations @ 180.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 11.7618(7) Å b = 13.1850(8) Å c = 13.7331(8) Å α = 92.734(3) ° β = 112.610(2) ° γ = 114.056(3) ° V = 1741.7(2) Å ³
Space Group	C1 (#1)
Z value	4
D _{calc}	1.115 g/cm ³

F_{000} 632.00

$\mu(\text{MoK}\alpha)$ 0.65 cm⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71069 \text{ \AA}$) graphite monochromated
Detector Aperture	270 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0, \phi=0.0$)	130.0 - 190.0°
Exposure Rate	289.8 sec./°
ω oscillation Range ($\chi=45.0, \phi=180.0$)	0.0 - 160.0°
Exposure Rate	289.8 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	54.9°
No. of Reflections Measured	Total: 7730
Corrections	Lorentz-polarization Absorption (trans. factors: 0.9064 - 1.1311)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\sum w (Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0010Fo^2 + 1.0000\sigma(Fo^2) + 0.2000]/(4Fo^2)$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 1.00\sigma(I)$)	3717
No. Variables	445
Reflection/Parameter Ratio	8.35
Residuals: R ($I > 1.00\sigma(I)$)	0.063
Residuals: R1 ($I > 2.00\sigma(I)$)	0.061
Residuals: wR2 ($I > 1.00\sigma(I)$)	0.132
Goodness of Fit Indicator	1.059
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	$0.56 \text{ e}^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-0.44 \text{ e}^-/\text{\AA}^3$

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$

atom	x	y	z	B_{eq}
N(3)	0.5260	0.1798	1.0270	5.318
N(4)	0.3400	-0.1733	0.9992	5.292
N(1)	-0.1059	-0.1227	1.0410	5.912
N(2)	0.0613	0.2169	1.0246	5.862
C(10)	0.0636	0.1326	1.0072	3.943
C(8)	0.0706	0.0257	0.9843	3.250
C(30)	0.4429	-0.0958	1.0201	3.820
C(29)	0.5496	0.1052	1.0376	3.802
C(28)	0.5763	0.0045	1.0434	3.290
C(11)	0.0483	0.0020	0.8628	3.292
C(37)	0.6860	0.0241	1.2645	3.603
C(7)	0.2198	0.0452	1.0634	4.064
C(26)	0.5070	-0.0426	0.8351	4.492
C(38)	0.7964	0.1499	1.3293	3.892
C(6)	0.2645	0.0838	1.1831	4.002
C(31)	0.6934	0.0132	1.1521	3.245
C(9)	-0.0313	-0.0613	1.0132	4.065
C(12)	0.1002	-0.0798	0.8327	4.365
C(32)	0.8414	0.1090	1.1806	4.223
C(33)	0.9103	0.1427	1.3053	4.342
C(18)	-0.1427	-0.1701	0.7199	4.410
C(17)	-0.0993	-0.0418	0.7648	3.844
C(27)	0.6185	-0.0114	0.9497	4.252
C(13)	-0.0067	-0.1429	0.7151	4.640
C(1)	0.3465	0.1994	1.2365	5.635
C(39)	0.7582	0.2431	1.2906	4.898
C(25)	0.4108	-0.1552	0.7825	6.032
C(35)	0.7647	-0.0406	1.3246	5.263
C(36)	0.5455	-0.0156	1.2623	5.343
C(14)	0.0050	-0.0545	0.6458	5.695
C(19)	-0.1749	-0.2493	0.7928	6.049
C(5)	0.2306	0.0047	1.2431	5.556
C(16)	-0.2041	-0.0155	0.7824	5.598
C(15)	-0.0653	0.0102	0.6748	5.130
C(21)	0.4987	0.0394	0.7796	5.958
C(40)	0.8336	0.1715	1.4506	6.063
C(34)	0.9160	0.0370	1.3449	5.911

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$ (continued)

atom	x	y	z	B_{eq}
C(20)	-0.2702	-0.2248	0.6086	6.739
C(4)	0.2786	0.0425	1.3542	7.584
C(23)	0.3038	-0.1019	0.6245	8.118
C(2)	0.3907	0.2341	1.3464	7.757
C(3)	0.3570	0.1554	1.4039	7.747
C(22)	0.3965	0.0082	0.6731	8.113
C(24)	0.3089	-0.1828	0.6769	8.272
H(1)	0.3696	0.2533	1.1951	6.573
H(2)	0.4477	0.3130	1.3824	8.738
H(3)	0.3901	0.1791	1.4801	9.967
H(4)	0.2496	-0.0112	1.3941	10.114
H(5)	0.1796	-0.0745	1.2076	7.154
H(6)	0.2227	-0.0252	1.0522	5.053
H(7)	0.2847	0.1013	1.0447	5.050
H(8)	0.1038	0.0741	0.8547	3.931
H(9)	0.1901	-0.0385	0.8365	5.150
H(10)	0.1020	-0.1306	0.8794	5.147
H(11)	-0.0020	-0.2082	0.6883	5.297
H(12)	0.1000	-0.0047	0.6661	6.377
H(13)	-0.0411	-0.0897	0.5703	6.383
H(14)	-0.1466	-0.0011	0.6132	5.795
H(15)	-0.0034	0.0898	0.7027	5.795
H(16)	-0.1671	0.0650	0.8053	7.112
H(17)	-0.2878	-0.0445	0.7171	7.113
H(18)	-0.2225	-0.0504	0.8373	7.114
H(19)	-0.2563	-0.2568	0.7975	6.204
H(20)	-0.1884	-0.3224	0.7639	6.205
H(21)	-0.0987	-0.2177	0.8632	6.198
H(22)	-0.3504	-0.2322	0.6156	6.444
H(23)	-0.2554	-0.1783	0.5596	6.441
H(24)	-0.2834	-0.2984	0.5816	6.444
H(29)	0.4169	-0.2136	0.8190	7.347
H(28)	0.2425	-0.2600	0.6399	8.756
H(27)	0.2305	-0.1199	0.5542	9.644
H(26)	0.3958	0.0648	0.6329	10.792
H(25)	0.5629	0.1175	0.8149	7.872
H(30)	0.6956	0.0580	0.9588	5.728

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$ (continued)

atom	x	y	z	B_{eq}
H(31)	0.6449	-0.0709	0.9573	5.729
H(32)	0.6977	-0.0566	1.1409	4.058
H(33)	0.8898	0.0814	1.1543	5.279
H(34)	0.8361	0.1715	1.1510	5.274
H(35)	0.9966	0.2106	1.3375	4.716
H(36)	0.9427	0.0001	1.3030	6.880
H(37)	0.9785	0.0563	1.4197	6.880
H(38)	0.7592	-0.0483	1.3913	5.932
H(39)	0.7281	-0.1143	1.2801	5.932
H(40)	0.5547	-0.0058	1.3344	6.225
H(41)	0.5032	0.0278	1.2248	6.223
H(42)	0.4895	-0.0943	1.2255	6.223
H(43)	0.8351	0.3164	1.3289	6.065
H(44)	0.7334	0.2332	1.2150	6.062
H(45)	0.6817	0.2371	1.3027	6.065
H(46)	0.8584	0.1156	1.4797	6.648
H(47)	0.9100	0.2456	1.4866	6.650
H(48)	0.7565	0.1671	1.4610	6.650

$$B_{\text{eq}} = \frac{8}{3} \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
N(3)	0.0799	0.0753	0.0805	0.0545	0.0454	0.0382
N(4)	0.0494	0.0659	0.0755	0.0199	0.0249	0.0189
N(1)	0.0830	0.0822	0.0891	0.0435	0.0580	0.0430
N(2)	0.0839	0.0561	0.0975	0.0403	0.0460	0.0163
C(10)	0.0503	0.0511	0.0578	0.0272	0.0284	0.0174
C(8)	0.0410	0.0431	0.0474	0.0232	0.0231	0.0113
C(30)	0.0479	0.0555	0.0489	0.0300	0.0214	0.0155
C(29)	0.0515	0.0572	0.0551	0.0332	0.0318	0.0266
C(28)	0.0457	0.0467	0.0465	0.0271	0.0268	0.0175
C(11)	0.0367	0.0421	0.0457	0.0163	0.0196	0.0113
C(37)	0.0479	0.0393	0.0478	0.0177	0.0214	0.0161
C(7)	0.0461	0.0682	0.0456	0.0331	0.0188	0.0105
C(26)	0.0713	0.0717	0.0497	0.0447	0.0348	0.0184
C(38)	0.0522	0.0414	0.0501	0.0189	0.0217	0.0098
C(6)	0.0607	0.0626	0.0434	0.0423	0.0221	0.0141
C(31)	0.0381	0.0416	0.0488	0.0223	0.0196	0.0149
C(9)	0.0565	0.0560	0.0617	0.0355	0.0337	0.0241
C(12)	0.0462	0.0664	0.0504	0.0274	0.0189	-0.0004
C(32)	0.0444	0.0564	0.0662	0.0223	0.0317	0.0163
C(33)	0.0401	0.0497	0.0595	0.0150	0.0139	0.0076
C(18)	0.0448	0.0463	0.0500	0.0066	0.0118	0.0075
C(17)	0.0429	0.0543	0.0478	0.0232	0.0175	0.0198
C(27)	0.0599	0.0658	0.0558	0.0378	0.0347	0.0189
C(13)	0.0570	0.0617	0.0490	0.0242	0.0200	0.0021
C(1)	0.0921	0.0616	0.0544	0.0384	0.0229	0.0205
C(39)	0.0706	0.0458	0.0756	0.0307	0.0342	0.0130
C(25)	0.1035	0.0686	0.0606	0.0433	0.0360	0.0141
C(35)	0.0793	0.0524	0.0561	0.0328	0.0149	0.0214
C(36)	0.0621	0.0742	0.0608	0.0152	0.0391	0.0162
C(14)	0.0746	0.0786	0.0488	0.0170	0.0341	0.0086
C(19)	0.0759	0.0430	0.0775	0.0023	0.0293	0.0112
C(5)	0.0948	0.0688	0.0630	0.0510	0.0344	0.0259
C(16)	0.0572	0.0934	0.0747	0.0479	0.0259	0.0306
C(15)	0.0665	0.0636	0.0533	0.0207	0.0242	0.0270
C(21)	0.1094	0.0800	0.0599	0.0576	0.0433	0.0271
C(40)	0.0829	0.0732	0.0544	0.0251	0.0244	0.0051
C(34)	0.0685	0.0754	0.0740	0.0456	0.0119	0.0187

Table 2. Anisotropic Displacement Parameters (continued)

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(20)	0.0570	0.0856	0.0614	0.0065	0.0062	0.0068
C(4)	0.1418	0.1129	0.0656	0.0773	0.0533	0.0518
C(23)	0.1229	0.1367	0.0457	0.0749	0.0195	0.0122
C(2)	0.1407	0.0794	0.0566	0.0645	0.0137	-0.0001
C(3)	0.1590	0.1096	0.0470	0.0870	0.0389	0.0208
C(22)	0.1630	0.1241	0.0547	0.0995	0.0430	0.0354
C(24)	0.1149	0.0988	0.0635	0.0296	0.0293	-0.0144

The general temperature factor expression: $\exp(-2\pi^2(a^*{}^2U_{11}h^2 + b^*{}^2U_{22}k^2 + c^*{}^2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
N(3)	C(29)	1.13(1)	N(4)	C(30)	1.13(1)
N(1)	C(9)	1.13(1)	N(2)	C(10)	1.14(1)
C(10)	C(8)	1.47(1)	C(8)	C(11)	1.58(1)
C(8)	C(7)	1.57(1)	C(8)	C(9)	1.48(1)
C(30)	C(28)	1.48(1)	C(29)	C(28)	1.48(1)
C(28)	C(31)	1.55(1)	C(28)	C(27)	1.58(1)
C(11)	C(12)	1.55(2)	C(11)	C(17)	1.572(9)
C(37)	C(38)	1.563(9)	C(37)	C(31)	1.58(1)
C(37)	C(35)	1.55(1)	C(37)	C(36)	1.50(1)
C(7)	C(6)	1.51(1)	C(26)	C(27)	1.51(1)
C(26)	C(25)	1.38(1)	C(26)	C(21)	1.37(2)
C(38)	C(33)	1.53(2)	C(38)	C(39)	1.52(1)
C(38)	C(40)	1.53(1)	C(6)	C(1)	1.39(1)
C(6)	C(5)	1.39(1)	C(31)	C(32)	1.55(1)
C(12)	C(13)	1.53(1)	C(32)	C(33)	1.53(1)
C(33)	C(34)	1.54(2)	C(18)	C(17)	1.57(1)
C(18)	C(13)	1.52(2)	C(18)	C(19)	1.52(1)
C(18)	C(20)	1.54(1)	C(17)	C(16)	1.51(2)
C(17)	C(15)	1.55(1)	C(13)	C(14)	1.54(1)
C(1)	C(2)	1.38(2)	C(25)	C(24)	1.39(1)
C(35)	C(34)	1.55(1)	C(14)	C(15)	1.54(2)
C(5)	C(4)	1.39(2)	C(21)	C(22)	1.39(1)
C(4)	C(3)	1.35(2)	C(23)	C(22)	1.34(2)
C(23)	C(24)	1.32(2)	C(2)	C(3)	1.36(2)

Table 4. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(11)	H(8)	0.95(1)	C(7)	H(6)	0.95(2)
C(7)	H(7)	0.95(1)	C(31)	H(32)	0.95(1)
C(12)	H(9)	0.95(1)	C(12)	H(10)	0.95(1)
C(32)	H(33)	0.95(2)	C(32)	H(34)	0.95(1)
C(33)	H(35)	0.950(9)	C(27)	H(30)	0.95(1)
C(27)	H(31)	0.95(2)	C(13)	H(11)	0.95(2)
C(1)	H(1)	0.95(2)	C(39)	H(43)	0.95(1)
C(39)	H(44)	0.95(1)	C(39)	H(45)	0.95(2)
C(25)	H(29)	0.95(2)	C(35)	H(38)	0.95(2)
C(35)	H(39)	0.95(1)	C(36)	H(40)	0.95(2)
C(36)	H(41)	0.95(2)	C(36)	H(42)	0.95(1)
C(14)	H(12)	0.95(1)	C(14)	H(13)	0.95(1)
C(19)	H(19)	0.95(2)	C(19)	H(20)	0.95(2)
C(19)	H(21)	0.95(1)	C(5)	H(5)	0.95(1)
C(16)	H(16)	0.95(2)	C(16)	H(17)	0.95(1)
C(16)	H(18)	0.95(2)	C(15)	H(14)	0.95(1)
C(15)	H(15)	0.95(1)	C(21)	H(25)	0.95(1)
C(40)	H(46)	0.95(2)	C(40)	H(47)	0.95(1)
C(40)	H(48)	0.95(2)	C(34)	H(36)	0.95(2)
C(34)	H(37)	0.95(1)	C(20)	H(22)	0.95(2)
C(20)	H(23)	0.95(2)	C(20)	H(24)	0.95(2)
C(4)	H(4)	0.95(2)	C(23)	H(27)	0.95(2)
C(2)	H(2)	0.95(2)	C(3)	H(3)	0.95(2)
C(22)	H(26)	0.95(2)	C(24)	H(28)	0.95(2)

Table 5. Bond angles ($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(8)	C(10)	N(2)	178(1)	C(11)	C(8)	C(7)	109.6(8)
C(11)	C(8)	C(9)	117.4(5)	C(11)	C(8)	C(10)	107.7(7)
C(7)	C(8)	C(9)	108.4(7)	C(7)	C(8)	C(10)	107.5(6)
C(9)	C(8)	C(10)	105.8(8)	C(28)	C(30)	N(4)	178(1)
C(28)	C(29)	N(3)	176.0(9)	C(31)	C(28)	C(27)	107.7(7)
C(31)	C(28)	C(30)	110.6(7)	C(31)	C(28)	C(29)	115.7(6)
C(27)	C(28)	C(30)	108.8(6)	C(27)	C(28)	C(29)	107.1(7)
C(30)	C(28)	C(29)	106.6(8)	C(12)	C(11)	C(17)	102.8(6)
C(12)	C(11)	C(8)	116.4(7)	C(17)	C(11)	C(8)	121.1(7)
C(38)	C(37)	C(31)	104.4(6)	C(38)	C(37)	C(35)	99.8(6)
C(38)	C(37)	C(36)	116.2(8)	C(31)	C(37)	C(35)	102.4(8)
C(31)	C(37)	C(36)	118.1(6)	C(35)	C(37)	C(36)	113.6(8)
C(6)	C(7)	C(8)	116.1(9)	C(27)	C(26)	C(25)	121.0(9)
C(27)	C(26)	C(21)	121.2(7)	C(25)	C(26)	C(21)	117.8(8)
C(33)	C(38)	C(39)	115.2(8)	C(33)	C(38)	C(40)	113.1(8)
C(33)	C(38)	C(37)	94.0(7)	C(39)	C(38)	C(40)	105.2(9)
C(39)	C(38)	C(37)	116.0(6)	C(40)	C(38)	C(37)	113.5(7)
C(1)	C(6)	C(5)	118.8(8)	C(1)	C(6)	C(7)	120.5(8)
C(5)	C(6)	C(7)	120.6(7)	C(32)	C(31)	C(28)	115.5(7)
C(32)	C(31)	C(37)	102.6(5)	C(28)	C(31)	C(37)	123.0(8)
N(1)	C(9)	C(8)	175.8(9)	C(13)	C(12)	C(11)	103.0(8)
C(33)	C(32)	C(31)	102.9(7)	C(34)	C(33)	C(38)	102.6(8)
C(34)	C(33)	C(32)	107.3(7)	C(38)	C(33)	C(32)	102.2(7)
C(17)	C(18)	C(13)	94.1(6)	C(17)	C(18)	C(19)	115.4(8)
C(17)	C(18)	C(20)	113.1(8)	C(13)	C(18)	C(19)	114.5(9)
C(13)	C(18)	C(20)	113.8(9)	C(19)	C(18)	C(20)	106.1(7)
C(16)	C(17)	C(15)	112.1(9)	C(16)	C(17)	C(11)	118.3(7)
C(16)	C(17)	C(18)	117.8(7)	C(15)	C(17)	C(11)	102.9(6)
C(15)	C(17)	C(18)	99.8(7)	C(11)	C(17)	C(18)	103.5(7)
C(28)	C(27)	C(26)	115.9(9)	C(14)	C(13)	C(12)	107.7(7)
C(14)	C(13)	C(18)	103.1(9)	C(12)	C(13)	C(18)	102.1(8)
C(2)	C(1)	C(6)	120(1)	C(24)	C(25)	C(26)	120(1)
C(34)	C(35)	C(37)	103.1(8)	C(15)	C(14)	C(13)	103(1)
C(4)	C(5)	C(6)	119.3(9)	C(17)	C(15)	C(14)	103.7(9)
C(22)	C(21)	C(26)	120(1)	C(33)	C(34)	C(35)	103.6(9)
C(3)	C(4)	C(5)	120(1)	C(22)	C(23)	C(24)	120(1)
C(3)	C(2)	C(1)	119(1)	C(4)	C(3)	C(2)	120(1)

Table 5. Bond angles ($^{\circ}$) -- continued

atom C(21)	atom C(22)	atom C(23)	angle 120(1)	atom C(25)	atom C(24)	atom C(23)	angle 120(1)
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Table 6. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(12)	C(11)	H(8)	105(1)	C(17)	C(11)	H(8)	104.9(8)
H(8)	C(11)	C(8)	104.8(7)	C(6)	C(7)	H(6)	107.4(9)
C(6)	C(7)	H(7)	108.0(7)	H(6)	C(7)	H(7)	109(1)
H(6)	C(7)	C(8)	107.7(7)	H(7)	C(7)	C(8)	108.0(9)
C(32)	C(31)	H(32)	105.2(9)	H(32)	C(31)	C(28)	104.4(7)
H(32)	C(31)	C(37)	104.5(9)	C(13)	C(12)	H(9)	110(1)
C(13)	C(12)	H(10)	111.9(8)	H(9)	C(12)	H(10)	109(1)
H(9)	C(12)	C(11)	111(1)	H(10)	C(12)	C(11)	110(1)
C(33)	C(32)	H(33)	111.2(9)	C(33)	C(32)	H(34)	111.5(9)
H(33)	C(32)	H(34)	109(1)	H(33)	C(32)	C(31)	111.4(8)
H(34)	C(32)	C(31)	110.3(9)	C(34)	C(33)	H(35)	114(1)
H(35)	C(33)	C(38)	114(1)	H(35)	C(33)	C(32)	114(1)
H(30)	C(27)	H(31)	109(1)	H(30)	C(27)	C(28)	108(1)
H(30)	C(27)	C(26)	107(1)	H(31)	C(27)	C(28)	107(1)
H(31)	C(27)	C(26)	107.6(9)	C(14)	C(13)	H(11)	114(1)
H(11)	C(13)	C(12)	114(1)	H(11)	C(13)	C(18)	114.1(9)
C(2)	C(1)	H(1)	121(1)	H(1)	C(1)	C(6)	118(1)
H(43)	C(39)	H(44)	109(1)	H(43)	C(39)	H(45)	109(1)
H(43)	C(39)	C(38)	110(1)	H(44)	C(39)	H(45)	109(1)
H(44)	C(39)	C(38)	108(1)	H(45)	C(39)	C(38)	109(1)
C(24)	C(25)	H(29)	120(1)	H(29)	C(25)	C(26)	118(1)
C(34)	C(35)	H(38)	111.0(9)	C(34)	C(35)	H(39)	111(1)
H(38)	C(35)	H(39)	109(1)	H(38)	C(35)	C(37)	111(1)
H(39)	C(35)	C(37)	110.3(9)	H(40)	C(36)	H(41)	109(1)
H(40)	C(36)	H(42)	109(1)	H(40)	C(36)	C(37)	110.0(9)
H(41)	C(36)	H(42)	109(1)	H(41)	C(36)	C(37)	109(1)
H(42)	C(36)	C(37)	109(1)	C(15)	C(14)	H(12)	111(1)
C(15)	C(14)	H(13)	111(1)	H(12)	C(14)	H(13)	109(1)
H(12)	C(14)	C(13)	109(1)	H(13)	C(14)	C(13)	112(1)
H(19)	C(19)	H(20)	109(1)	H(19)	C(19)	H(21)	109(1)
H(19)	C(19)	C(18)	110(1)	H(20)	C(19)	H(21)	109(1)
H(20)	C(19)	C(18)	109(1)	H(21)	C(19)	C(18)	108(1)
C(4)	C(5)	H(5)	121(1)	H(5)	C(5)	C(6)	118(1)
H(16)	C(16)	H(17)	109(1)	H(16)	C(16)	H(18)	109(1)
H(16)	C(16)	C(17)	109(1)	H(17)	C(16)	H(18)	109(1)
H(17)	C(16)	C(17)	110(1)	H(18)	C(16)	C(17)	108(1)
H(14)	C(15)	H(15)	109(1)	H(14)	C(15)	C(17)	111(1)

Table 6. Bond angles involving hydrogens ($^{\circ}$) -- continued

atom	atom	atom	angle	atom	atom	atom	angle
H(14)	C(15)	C(14)	110(1)	H(15)	C(15)	C(17)	109(1)
H(15)	C(15)	C(14)	110(1)	C(22)	C(21)	H(25)	120(1)
H(25)	C(21)	C(26)	119(1)	H(46)	C(40)	H(47)	109(1)
H(46)	C(40)	H(48)	109(1)	H(46)	C(40)	C(38)	109(1)
H(47)	C(40)	H(48)	109(1)	H(47)	C(40)	C(38)	109(1)
H(48)	C(40)	C(38)	110(1)	H(36)	C(34)	H(37)	109(1)
H(36)	C(34)	C(33)	110(1)	H(36)	C(34)	C(35)	110.6(9)
H(37)	C(34)	C(33)	111(1)	H(37)	C(34)	C(35)	111(1)
H(22)	C(20)	H(23)	109(1)	H(22)	C(20)	H(24)	109(1)
H(22)	C(20)	C(18)	110(1)	H(23)	C(20)	H(24)	109(1)
H(23)	C(20)	C(18)	109.5(9)	H(24)	C(20)	C(18)	108(1)
C(3)	C(4)	H(4)	119(1)	H(4)	C(4)	C(5)	119(1)
C(22)	C(23)	H(27)	118(2)	C(24)	C(23)	H(27)	120(1)
C(3)	C(2)	H(2)	120(1)	H(2)	C(2)	C(1)	119(1)
H(3)	C(3)	C(4)	119(1)	H(3)	C(3)	C(2)	120(1)
H(26)	C(22)	C(21)	119(1)	H(26)	C(22)	C(23)	119(1)
H(28)	C(24)	C(25)	121(1)	H(28)	C(24)	C(23)	118(1)

Table 7. Torsion Angles($^{\circ}$)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(10)	C(8)	C(11)	C(12)	159.7(6)	C(10)	C(8)	C(11)	C(17)	-74.2(8)
C(10)	C(8)	C(11)	H(8)	43(1)	C(7)	C(8)	C(11)	C(12)	43.1(8)
C(7)	C(8)	C(11)	C(17)	169.2(7)	C(7)	C(8)	C(11)	H(8)	-72(1)
C(9)	C(8)	C(11)	C(12)	-81.1(9)	C(9)	C(8)	C(11)	C(17)	44(1)
C(9)	C(8)	C(11)	H(8)	163(1)	C(10)	C(8)	C(7)	C(6)	55(1)
C(10)	C(8)	C(7)	H(6)	176(1)	C(10)	C(8)	C(7)	H(7)	-65(1)
C(11)	C(8)	C(7)	C(6)	172.5(7)	C(11)	C(8)	C(7)	H(6)	-67(1)
C(11)	C(8)	C(7)	H(7)	51(1)	C(9)	C(8)	C(7)	C(6)	-58(1)
C(9)	C(8)	C(7)	H(6)	62(1)	C(9)	C(8)	C(7)	H(7)	-179(508)
C(30)	C(28)	C(31)	C(37)	-58(1)	C(30)	C(28)	C(31)	C(32)	174.9(8)
C(30)	C(28)	C(31)	H(32)	59(1)	C(29)	C(28)	C(31)	C(37)	63.0(9)
C(29)	C(28)	C(31)	C(32)	-63(1)	C(29)	C(28)	C(31)	H(32)	-178.6(9)
C(27)	C(28)	C(31)	C(37)	-177.2(6)	C(27)	C(28)	C(31)	C(32)	56.1(9)
C(27)	C(28)	C(31)	H(32)	-58(1)	C(30)	C(28)	C(27)	C(26)	49(1)
C(30)	C(28)	C(27)	H(30)	170(1)	C(30)	C(28)	C(27)	H(31)	-70.9(9)
C(29)	C(28)	C(27)	C(26)	-65.4(8)	C(29)	C(28)	C(27)	H(30)	55(1)
C(29)	C(28)	C(27)	H(31)	174.2(7)	C(31)	C(28)	C(27)	C(26)	169.5(7)
C(31)	C(28)	C(27)	H(30)	-69(1)	C(31)	C(28)	C(27)	H(31)	49.1(8)
C(8)	C(11)	C(12)	C(13)	145.3(6)	C(8)	C(11)	C(12)	H(9)	-96(1)
C(8)	C(11)	C(12)	H(10)	25.5(9)	C(17)	C(11)	C(12)	C(13)	10.5(8)
C(17)	C(11)	C(12)	H(9)	128.7(9)	C(17)	C(11)	C(12)	H(10)	-109.3(8)
H(8)	C(11)	C(12)	C(13)	-99.1(8)	H(8)	C(11)	C(12)	H(9)	19(1)
H(8)	C(11)	C(12)	H(10)	141.1(8)	C(8)	C(11)	C(17)	C(18)	-107.3(9)
C(8)	C(11)	C(17)	C(16)	25(1)	C(8)	C(11)	C(17)	C(15)	149.2(8)
C(12)	C(11)	C(17)	C(18)	24.7(8)	C(12)	C(11)	C(17)	C(16)	157.1(8)
C(12)	C(11)	C(17)	C(15)	-78.8(8)	H(8)	C(11)	C(17)	C(18)	134(1)
H(8)	C(11)	C(17)	C(16)	-93(1)	H(8)	C(11)	C(17)	C(15)	31(1)
C(31)	C(37)	C(38)	C(33)	-48.3(8)	C(31)	C(37)	C(38)	C(39)	72(1)
C(31)	C(37)	C(38)	C(40)	-165.6(9)	C(35)	C(37)	C(38)	C(33)	57.3(8)
C(35)	C(37)	C(38)	C(39)	177.9(9)	C(35)	C(37)	C(38)	C(40)	-60(1)
C(36)	C(37)	C(38)	C(33)	179(623)	C(36)	C(37)	C(38)	C(39)	-59(1)
C(36)	C(37)	C(38)	C(40)	62(1)	C(38)	C(37)	C(31)	C(28)	-109.2(7)
C(38)	C(37)	C(31)	C(32)	23.0(9)	C(38)	C(37)	C(31)	H(32)	132.5(8)
C(35)	C(37)	C(31)	C(28)	147.2(6)	C(35)	C(37)	C(31)	C(32)	-80.7(7)
C(35)	C(37)	C(31)	H(32)	28.9(7)	C(36)	C(37)	C(31)	C(28)	21(1)
C(36)	C(37)	C(31)	C(32)	153.8(7)	C(36)	C(37)	C(31)	H(32)	-96.6(9)
C(38)	C(37)	C(35)	C(34)	-39(1)	C(38)	C(37)	C(35)	H(38)	79(1)

Table 7. Torsion angles ($^{\circ}$) -- continued

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(38)	C(37)	C(35)	H(39)	-158(1)	C(31)	C(37)	C(35)	C(34)	67.6(8)
C(31)	C(37)	C(35)	H(38)	-173.3(7)	C(31)	C(37)	C(35)	H(39)	-51(1)
C(36)	C(37)	C(35)	C(34)	-164.0(7)	C(36)	C(37)	C(35)	H(38)	-44(1)
C(36)	C(37)	C(35)	H(39)	76(1)	C(38)	C(37)	C(36)	H(40)	-55(1)
C(38)	C(37)	C(36)	H(41)	64(1)	C(38)	C(37)	C(36)	H(42)	-175(1)
C(31)	C(37)	C(36)	H(40)	179(1)	C(31)	C(37)	C(36)	H(41)	-60(1)
C(31)	C(37)	C(36)	H(42)	59(1)	C(35)	C(37)	C(36)	H(40)	59(1)
C(35)	C(37)	C(36)	H(41)	179.5(9)	C(35)	C(37)	C(36)	H(42)	-60(1)
C(8)	C(7)	C(6)	C(1)	-97(1)	C(8)	C(7)	C(6)	C(5)	85(1)
H(6)	C(7)	C(6)	C(1)	142(1)	H(6)	C(7)	C(6)	C(5)	-34(1)
H(7)	C(7)	C(6)	C(1)	24(1)	H(7)	C(7)	C(6)	C(5)	-152(1)
C(25)	C(26)	C(27)	C(28)	-85(1)	C(25)	C(26)	C(27)	H(30)	152(1)
C(25)	C(26)	C(27)	H(31)	34(1)	C(21)	C(26)	C(27)	C(28)	95(1)
C(21)	C(26)	C(27)	H(30)	-26(1)	C(21)	C(26)	C(27)	H(31)	-144(1)
C(27)	C(26)	C(25)	C(24)	-179(880)	C(27)	C(26)	C(25)	H(29)	-1(2)
C(21)	C(26)	C(25)	C(24)	0(2)	C(21)	C(26)	C(25)	H(29)	177(1)
C(27)	C(26)	C(21)	C(22)	178(1)	C(27)	C(26)	C(21)	H(25)	-1(2)
C(25)	C(26)	C(21)	C(22)	0(2)	C(25)	C(26)	C(21)	H(25)	178(1)
C(37)	C(38)	C(33)	C(32)	56.8(7)	C(37)	C(38)	C(33)	C(34)	-54.3(7)
C(37)	C(38)	C(33)	H(35)	-178(1)	C(39)	C(38)	C(33)	C(32)	-64.5(7)
C(39)	C(38)	C(33)	C(34)	-175.6(6)	C(39)	C(38)	C(33)	H(35)	60(1)
C(40)	C(38)	C(33)	C(32)	174.4(6)	C(40)	C(38)	C(33)	C(34)	63.3(7)
C(40)	C(38)	C(33)	H(35)	-61(1)	C(37)	C(38)	C(39)	H(43)	-173(1)
C(37)	C(38)	C(39)	H(44)	-53(1)	C(37)	C(38)	C(39)	H(45)	66(1)
C(33)	C(38)	C(39)	H(43)	-64(1)	C(33)	C(38)	C(39)	H(44)	55(1)
C(33)	C(38)	C(39)	H(45)	174.8(8)	C(40)	C(38)	C(39)	H(43)	60(1)
C(40)	C(38)	C(39)	H(44)	-179(359)	C(40)	C(38)	C(39)	H(45)	-60.0(9)
C(37)	C(38)	C(40)	H(46)	52(1)	C(37)	C(38)	C(40)	H(47)	171(1)
C(37)	C(38)	C(40)	H(48)	-68(1)	C(33)	C(38)	C(40)	H(46)	-53(1)
C(33)	C(38)	C(40)	H(47)	66(1)	C(33)	C(38)	C(40)	H(48)	-173(1)
C(39)	C(38)	C(40)	H(46)	179(719)	C(39)	C(38)	C(40)	H(47)	-60(1)
C(39)	C(38)	C(40)	H(48)	59(1)	C(7)	C(6)	C(1)	C(2)	-177(1)
C(7)	C(6)	C(1)	H(1)	3(2)	C(5)	C(6)	C(1)	C(2)	-1(2)
C(5)	C(6)	C(1)	H(1)	179(719)	C(7)	C(6)	C(5)	C(4)	177(1)
C(7)	C(6)	C(5)	H(5)	0(2)	C(1)	C(6)	C(5)	C(4)	0(2)
C(1)	C(6)	C(5)	H(5)	-176(1)	C(28)	C(31)	C(32)	C(33)	148.9(8)
C(28)	C(31)	C(32)	H(33)	-91(1)	C(28)	C(31)	C(32)	H(34)	29(1)

Table 7. Torsion angles ($^{\circ}$) -- continued

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(37)	C(31)	C(32)	C(33)	12.4(9)	C(37)	C(31)	C(32)	H(33)	131(1)
C(37)	C(31)	C(32)	H(34)	-106(1)	H(32)	C(31)	C(32)	C(33)	-96(1)
H(32)	C(31)	C(32)	H(33)	22(1)	H(32)	C(31)	C(32)	H(34)	144(1)
C(11)	C(12)	C(13)	C(18)	-43.8(9)	C(11)	C(12)	C(13)	C(14)	64(1)
C(11)	C(12)	C(13)	H(11)	-167(1)	H(9)	C(12)	C(13)	C(18)	-162(1)
H(9)	C(12)	C(13)	C(14)	-54(1)	H(9)	C(12)	C(13)	H(11)	73(1)
H(10)	C(12)	C(13)	C(18)	75(1)	H(10)	C(12)	C(13)	C(14)	-176(1)
H(10)	C(12)	C(13)	H(11)	-48(1)	C(31)	C(32)	C(33)	C(38)	-44.8(9)
C(31)	C(32)	C(33)	C(34)	62(1)	C(31)	C(32)	C(33)	H(35)	-168(1)
H(33)	C(32)	C(33)	C(38)	-164.1(9)	H(33)	C(32)	C(33)	C(34)	-56(1)
H(33)	C(32)	C(33)	H(35)	71(1)	H(34)	C(32)	C(33)	C(38)	73(1)
H(34)	C(32)	C(33)	C(34)	-179(1)	H(34)	C(32)	C(33)	H(35)	-50(1)
C(38)	C(33)	C(34)	C(35)	31.2(9)	C(38)	C(33)	C(34)	H(36)	149.5(9)
C(38)	C(33)	C(34)	H(37)	-88(1)	C(32)	C(33)	C(34)	C(35)	-76(1)
C(32)	C(33)	C(34)	H(36)	42(1)	C(32)	C(33)	C(34)	H(37)	164(1)
H(35)	C(33)	C(34)	C(35)	155(1)	H(35)	C(33)	C(34)	H(36)	-86(1)
H(35)	C(33)	C(34)	H(37)	35(2)	C(13)	C(18)	C(17)	C(11)	-49.7(8)
C(13)	C(18)	C(17)	C(16)	177.7(8)	C(13)	C(18)	C(17)	C(15)	56.3(7)
C(19)	C(18)	C(17)	C(11)	69.9(9)	C(19)	C(18)	C(17)	C(16)	-62(1)
C(19)	C(18)	C(17)	C(15)	175.8(7)	C(20)	C(18)	C(17)	C(11)	-167.7(8)
C(20)	C(18)	C(17)	C(16)	59(1)	C(20)	C(18)	C(17)	C(15)	-61(1)
C(17)	C(18)	C(13)	C(12)	57.0(8)	C(17)	C(18)	C(13)	C(14)	-54.6(7)
C(17)	C(18)	C(13)	H(11)	-179(1)	C(19)	C(18)	C(13)	C(12)	-63.3(9)
C(19)	C(18)	C(13)	C(14)	-174.9(6)	C(19)	C(18)	C(13)	H(11)	60(1)
C(20)	C(18)	C(13)	C(12)	174.5(8)	C(20)	C(18)	C(13)	C(14)	62.8(9)
C(20)	C(18)	C(13)	H(11)	-61(1)	C(17)	C(18)	C(19)	H(19)	66(1)
C(17)	C(18)	C(19)	H(20)	-172(1)	C(17)	C(18)	C(19)	H(21)	-52(1)
C(13)	C(18)	C(19)	H(19)	174.5(9)	C(13)	C(18)	C(19)	H(20)	-65(1)
C(13)	C(18)	C(19)	H(21)	54(1)	C(20)	C(18)	C(19)	H(19)	-59(1)
C(20)	C(18)	C(19)	H(20)	61(1)	C(20)	C(18)	C(19)	H(21)	-179(1)
C(17)	C(18)	C(20)	H(22)	-67(1)	C(17)	C(18)	C(20)	H(23)	52(1)
C(17)	C(18)	C(20)	H(24)	172(1)	C(13)	C(18)	C(20)	H(22)	-173(1)
C(13)	C(18)	C(20)	H(23)	-53(1)	C(13)	C(18)	C(20)	H(24)	66(1)
C(19)	C(18)	C(20)	H(22)	59(1)	C(19)	C(18)	C(20)	H(23)	179(880)
C(19)	C(18)	C(20)	H(24)	-60(1)	C(11)	C(17)	C(16)	H(16)	57(1)
C(11)	C(17)	C(16)	H(17)	178(1)	C(11)	C(17)	C(16)	H(18)	-61(1)
C(18)	C(17)	C(16)	H(16)	-176(1)	C(18)	C(17)	C(16)	H(17)	-55(1)

Table 7. Torsion angles ($^{\circ}$) -- continued

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(18)	C(17)	C(16)	H(18)	64(1)	C(15)	C(17)	C(16)	H(16)	-61(1)
C(15)	C(17)	C(16)	H(17)	58(1)	C(15)	C(17)	C(16)	H(18)	179.0(8)
C(11)	C(17)	C(15)	C(14)	68.3(8)	C(11)	C(17)	C(15)	H(14)	-172(1)
C(11)	C(17)	C(15)	H(15)	-50(1)	C(18)	C(17)	C(15)	C(14)	-38.1(7)
C(18)	C(17)	C(15)	H(14)	81(1)	C(18)	C(17)	C(15)	H(15)	-156(1)
C(16)	C(17)	C(15)	C(14)	-163.6(5)	C(16)	C(17)	C(15)	H(14)	-43(1)
C(16)	C(17)	C(15)	H(15)	77(1)	C(12)	C(13)	C(14)	C(15)	-75(1)
C(12)	C(13)	C(14)	H(12)	43(1)	C(12)	C(13)	C(14)	H(13)	165(1)
C(18)	C(13)	C(14)	C(15)	32.4(7)	C(18)	C(13)	C(14)	H(12)	150(1)
C(18)	C(13)	C(14)	H(13)	-87(1)	H(11)	C(13)	C(14)	C(15)	156.8(9)
H(11)	C(13)	C(14)	H(12)	-84(1)	H(11)	C(13)	C(14)	H(13)	37(1)
C(6)	C(1)	C(2)	C(3)	1(2)	C(6)	C(1)	C(2)	H(2)	179(358)
H(1)	C(1)	C(2)	C(3)	-179(718)	H(1)	C(1)	C(2)	H(2)	-2(3)
C(26)	C(25)	C(24)	C(23)	1(2)	C(26)	C(25)	C(24)	H(28)	179(803)
H(29)	C(25)	C(24)	C(23)	-177(2)	H(29)	C(25)	C(24)	H(28)	1(3)
C(37)	C(35)	C(34)	C(33)	5(1)	C(37)	C(35)	C(34)	H(36)	-112(1)
C(37)	C(35)	C(34)	H(37)	125(1)	H(38)	C(35)	C(34)	C(33)	-113(1)
H(38)	C(35)	C(34)	H(36)	128(1)	H(38)	C(35)	C(34)	H(37)	6(2)
H(39)	C(35)	C(34)	C(33)	124(1)	H(39)	C(35)	C(34)	H(36)	6(1)
H(39)	C(35)	C(34)	H(37)	-115(1)	C(13)	C(14)	C(15)	C(17)	4.4(7)
C(13)	C(14)	C(15)	H(14)	-115(1)	C(13)	C(14)	C(15)	H(15)	122(1)
H(12)	C(14)	C(15)	C(17)	-113(1)	H(12)	C(14)	C(15)	H(14)	126(1)
H(12)	C(14)	C(15)	H(15)	4(1)	H(13)	C(14)	C(15)	C(17)	124(1)
H(13)	C(14)	C(15)	H(14)	4(1)	H(13)	C(14)	C(15)	H(15)	-117(1)
C(6)	C(5)	C(4)	C(3)	0(2)	C(6)	C(5)	C(4)	H(4)	175(2)
H(5)	C(5)	C(4)	C(3)	176(2)	H(5)	C(5)	C(4)	H(4)	-8(3)
C(26)	C(21)	C(22)	C(23)	1(3)	C(26)	C(21)	C(22)	H(26)	-174(2)
H(25)	C(21)	C(22)	C(23)	-178(2)	H(25)	C(21)	C(22)	H(26)	5(3)
C(5)	C(4)	C(3)	C(2)	0(3)	C(5)	C(4)	C(3)	H(3)	-178(358)
H(4)	C(4)	C(3)	C(2)	-175(2)	H(4)	C(4)	C(3)	H(3)	6(3)
C(24)	C(23)	C(22)	C(21)	0(3)	C(24)	C(23)	C(22)	H(26)	175(2)
H(27)	C(23)	C(22)	C(21)	174(2)	H(27)	C(23)	C(22)	H(26)	-9(4)
C(22)	C(23)	C(24)	C(25)	0(3)	C(22)	C(23)	C(24)	H(28)	-179(507)
H(27)	C(23)	C(24)	C(25)	-175(2)	H(27)	C(23)	C(24)	H(28)	5(3)
C(1)	C(2)	C(3)	C(4)	0(3)	C(1)	C(2)	C(3)	H(3)	177(2)
H(2)	C(2)	C(3)	C(4)	-178(2)	H(2)	C(2)	C(3)	H(3)	0(3)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table. Summary of Crystallographic Data

	<i>trans</i> -6a	<i>trans</i> -6b	<i>cis</i> -6c	<i>exo</i> -6d	<i>exo</i> -6e
formula	C ₂₀ H ₂₆ N ₂	C ₁₉ H ₂₄ N ₂	C ₁₇ H ₂₀ N ₂	C ₁₇ H ₁₈ N ₂	C ₂₀ H ₂₄ N ₂
mol wt	294.44	280.41	252.36	250.34	292.42
cryst dimens / mm	0.80 x 0.30 x 0.20	0.60 x 0.20 x 0.10	0.50 x 0.25 x 0.20	0.60 x 0.50 x 0.05	0.75 x 0.50 x 0.25
cryst system	monoclinic	monoclinic	orthorhombic	orthorhombic	triclinic
space group	P2 ₁	C2/c	P2 ₁ 2 ₁ 2 ₁	Pna2 ₁	C1
a / Å	16.7104(3)	35.436(3)	10.6730(5)	17.61(2)	11.7618(7)
b / Å	6.4424(1)	6.3489(5)	10.9928(7)	9.93(1)	13.1850(8)
c / Å	17.8094(4)	15.747(1)	12.8104(9)	8.376(9)	13.7331(8)
α / deg	90	90	90	90	92.734(3)
β / deg	110.457(2)	97.655(3)	90	90	112.610(2)
γ / deg	90	90	90	90	114.056(3)
V / Å ³	1796.36(6)	3511.3(5)	1503.0(2)	1463(2)	1741.7(2)
Z	4	8	4	4	4
D _{calcd} / g cm ⁻³	1.09	1.061	1.115	1.136	1.115
F ₀₀₀	640.00	1216.00	544.00	536.00	632.00
μ(Mo Ka) / cm ⁻¹	0.64	0.62	0.66	0.67	0.65
temp / K	296	296	296	296	296
2θmax / deg	55.0	55.0	54.0	54.9	54.9
no. of reflections	4342 (Total)	8316 (Total)	3792 (Total)	28490 (Total)	7730 (Total)
measured	4338 (Unique)	4158 (Unique)	1896 (Unique)	1946 (Unique)	
no. observations	3823(I > 0.50s(I))	2596(I > 1.00s(I))	1878 (All reflections)	1788 (All reflections)	3717 (I > 1.00s(I))
no. variables	449	214	192	190	445
rfln/parameter ratio	8.51	12.13	9.78	9.41	8.35
R ^a	0.094 (I>0.50 (I)) ^b	0.102 (I>1.00 (I))	0.111 (All reflections)	0.142 (All reflections)	0.063 (I>1.00 (I))
R1 ^a	0.079 (I>2.00 (I)) ^b	0.091 (I>2.00 (I))	0.061 (I>2.00 (I))	0.098 (I>2.00 (I))	0.061 (I>2.00 (I))
Rw2 ^a	0.136 (I>0.50 (I)) ^b	0.214 (I>1.00 (I))	0.185 (All reflections)	0.223 (All reflections)	0.132 (I>1.00 (I))
goodness of fit ^a	1.030 ^b	0.885	0.908	1.228	1.059

^a Refinement was by Full-matrix least-squares on F². ^b Refinement was by Full-matrix least-squares on F.

General Experimental Method. Photoirradiation was carried out with 300-W high-pressure mercury arc lamp. TLC was performed on silica gel plates; visualization was accomplished by UV light (254 nm) or by exposing to I₂ vapor. Column chromatography was performed on silica gel. ¹H and ¹³C NMR spectra were obtained with 300 MHz and 75 MHz NMR spectrometers, respectively, using CDCl₃ as a solvent and chemical shifts were reported in δ (parts per million) relative to Me₄Si. Other spectral and analytical data and HPLC separation were performed on standard laboratory instruments. All commercially available chemicals for syntheses of substrates **1a-e** were used without further purification. Acetonitrile was distilled two times over P₂O₅ and once over CaH₂ before use. Phenanthrene was recrystallized from methanol. Acetic acid and **7** were used as purchased. All reactions were carried out under argon atmosphere.