Morpholine-Based Immonium and Halogenoamidinium Salts as Coupling Reagents in Peptide Synthesis

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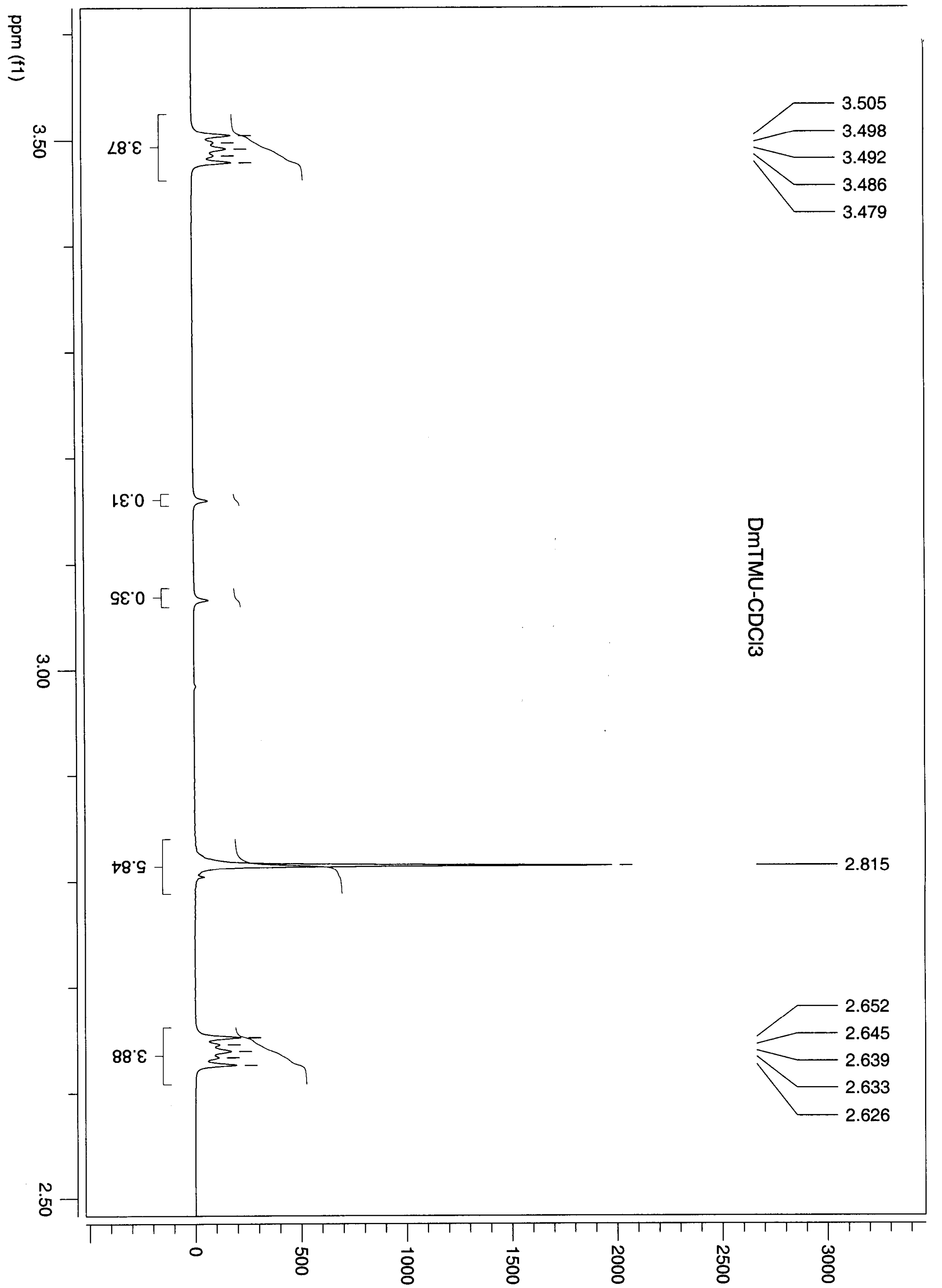
Supporting Information

Content:

General Procedures

TLC was performed on silica plates (8×4 cm) using suitable solvent systems and visualization by a Spectroline UV Lamp Model CM-10 (254 nm). Melting points were obtained in open capillary tubes and were uncorrected. Infrared spectra (IR) were recorded on a FT-IR instrument as KBr pellets. The absorption bands (ν_{max}) were given in wave numbers (cm^{-1}). NMR spectra were recorded on a 400 MHz spectrometer at room temperature. Tetramethylsilane (TMS) was used as reference for all NMR spectra with chemical shifts reported as ppm relative to TMS. HPLC analyses were carried out using Column C18, 5μ, 4.6x150mm with dual λ absorbance detector.

HPLC-MS analyses were carried out using Column C18, 5μ, 4.6x150mm with dual λ absorbance detector. All solvents used for recrystallization, extraction, column chromatography and TLC were of commercial grade, distilled before use and stored under dry conditions.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2.50
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0.35
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3.505
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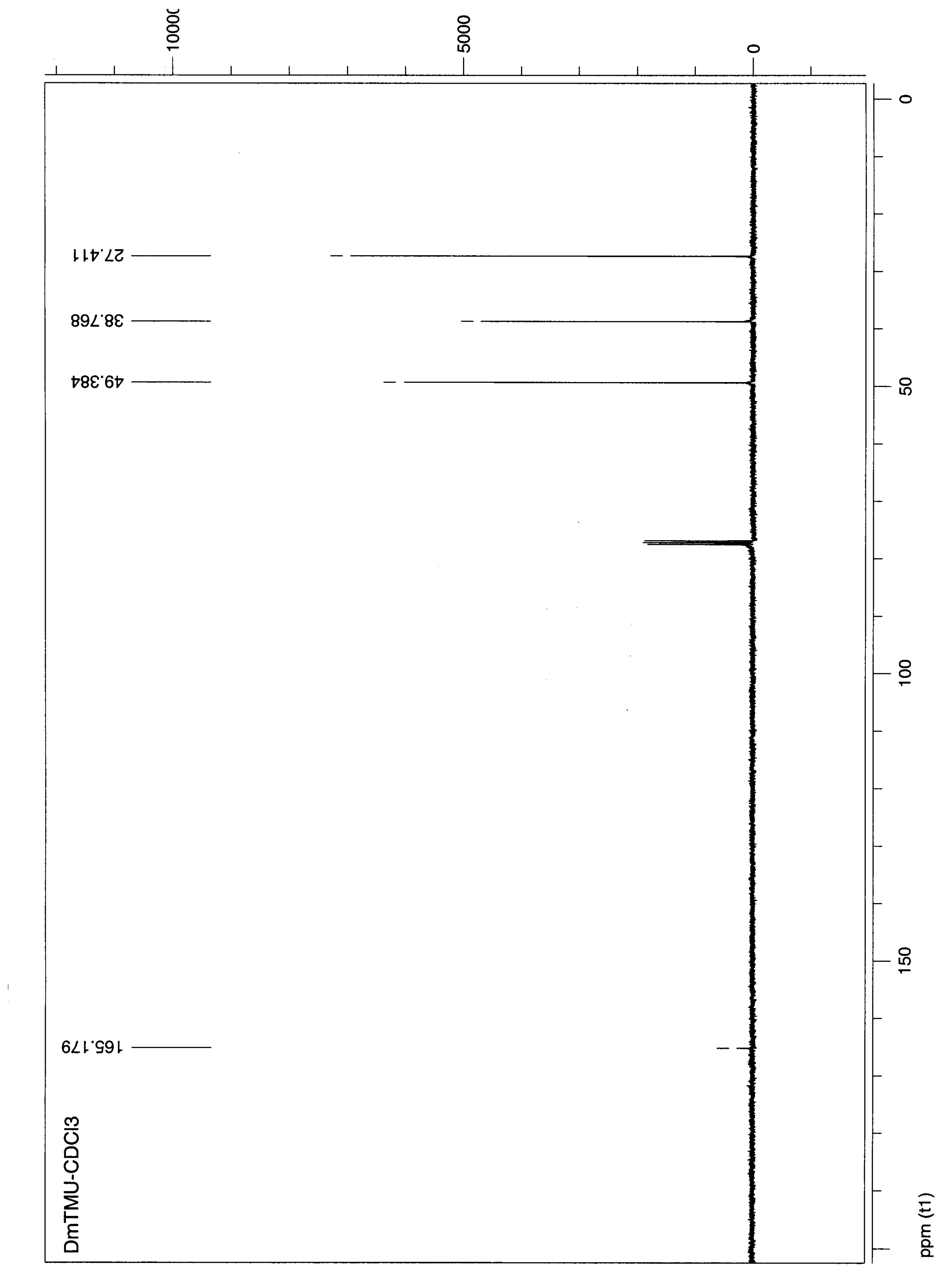
3.492
3.486
3.479
DmTMU-CDCl3
2.815
2.652
2.645
2.639
2.633
2.626
0
500
1000
1500
2000
2500
3000

3 ii, H-NMR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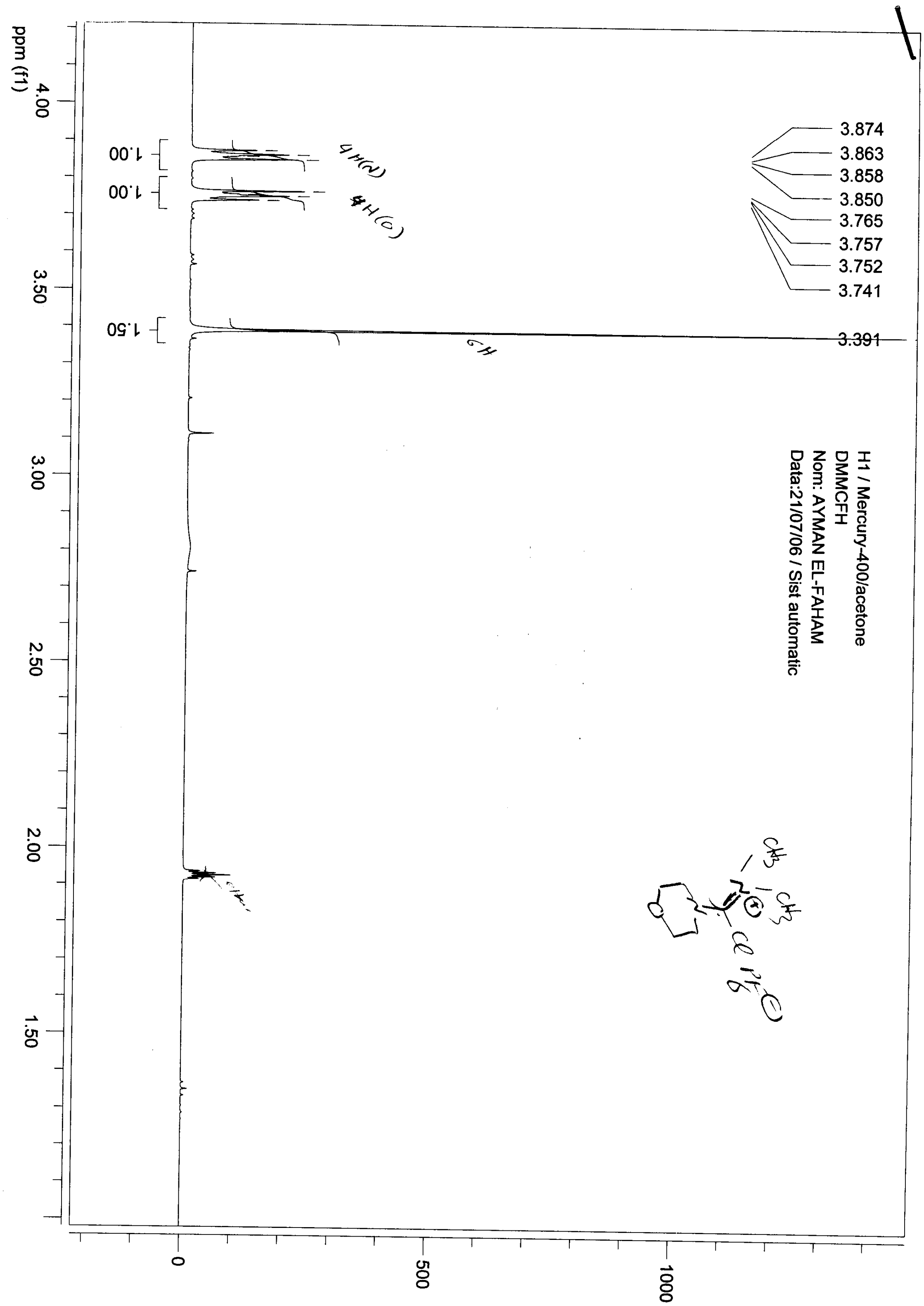
3ii, C-NMR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DMMCFH
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3.757
3.752
3.741
3.391
1.00
1.00
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3.00
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2.00
1.50
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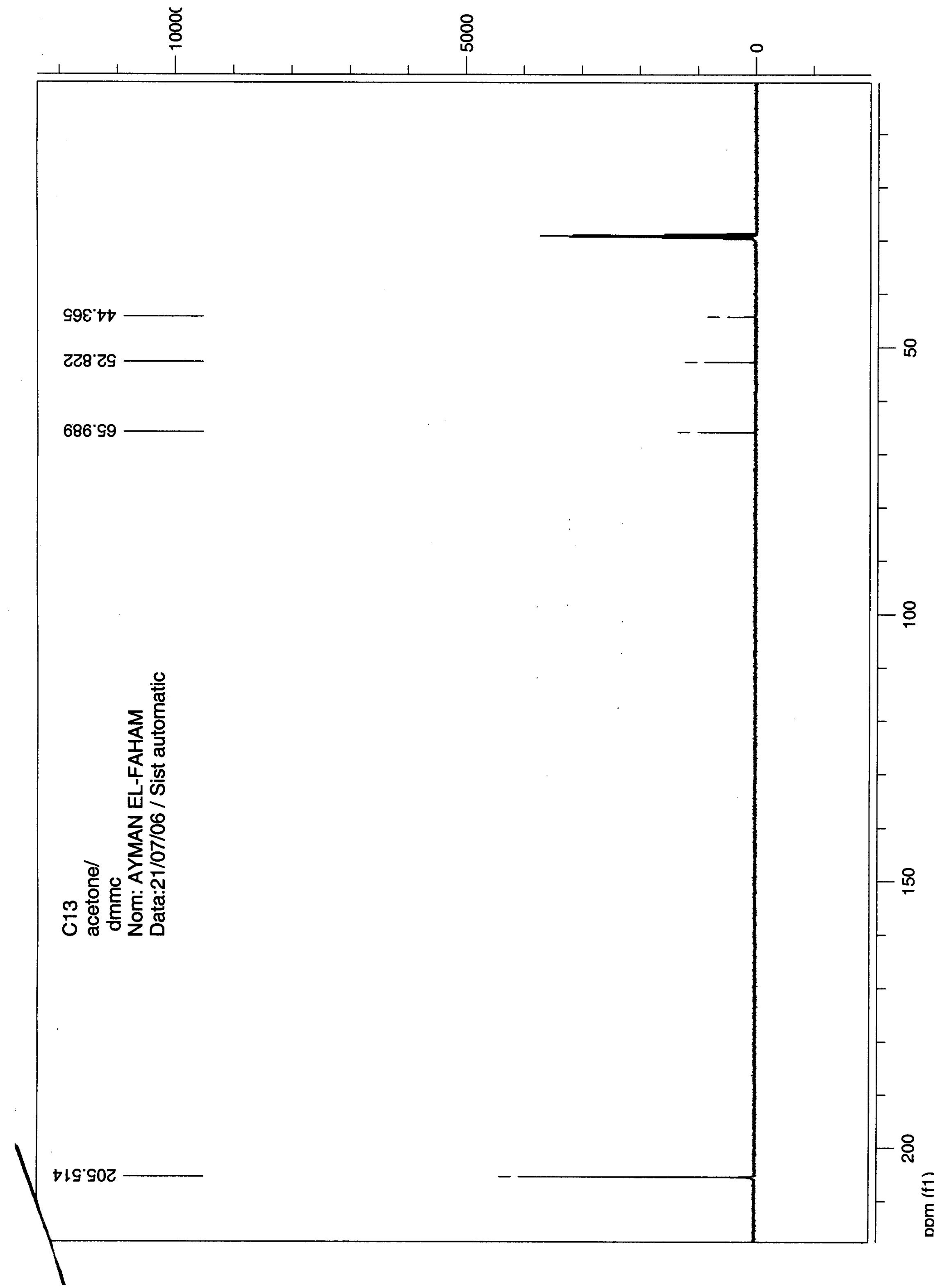
500
1000

4i, H-NMR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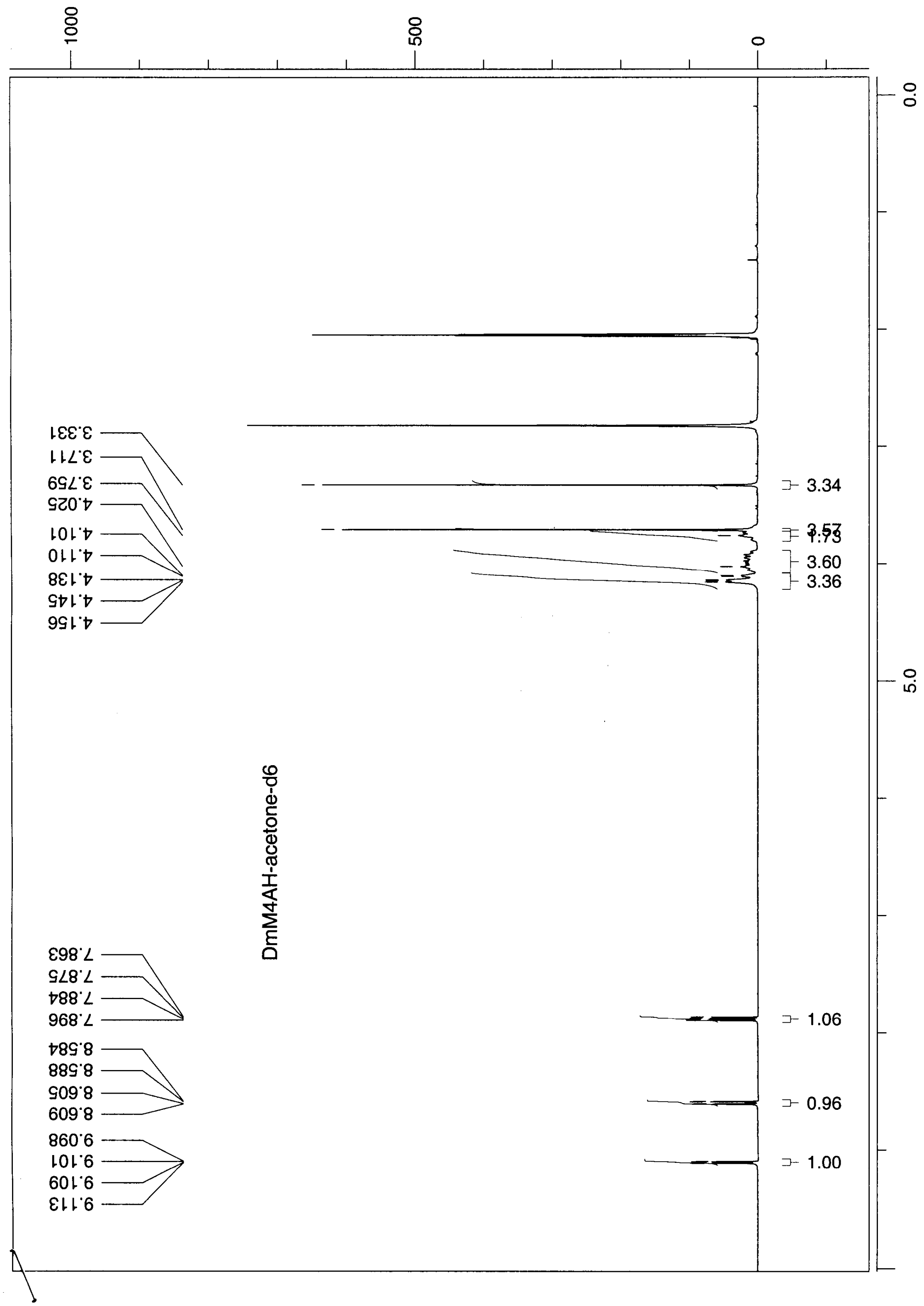
4i, C-NMR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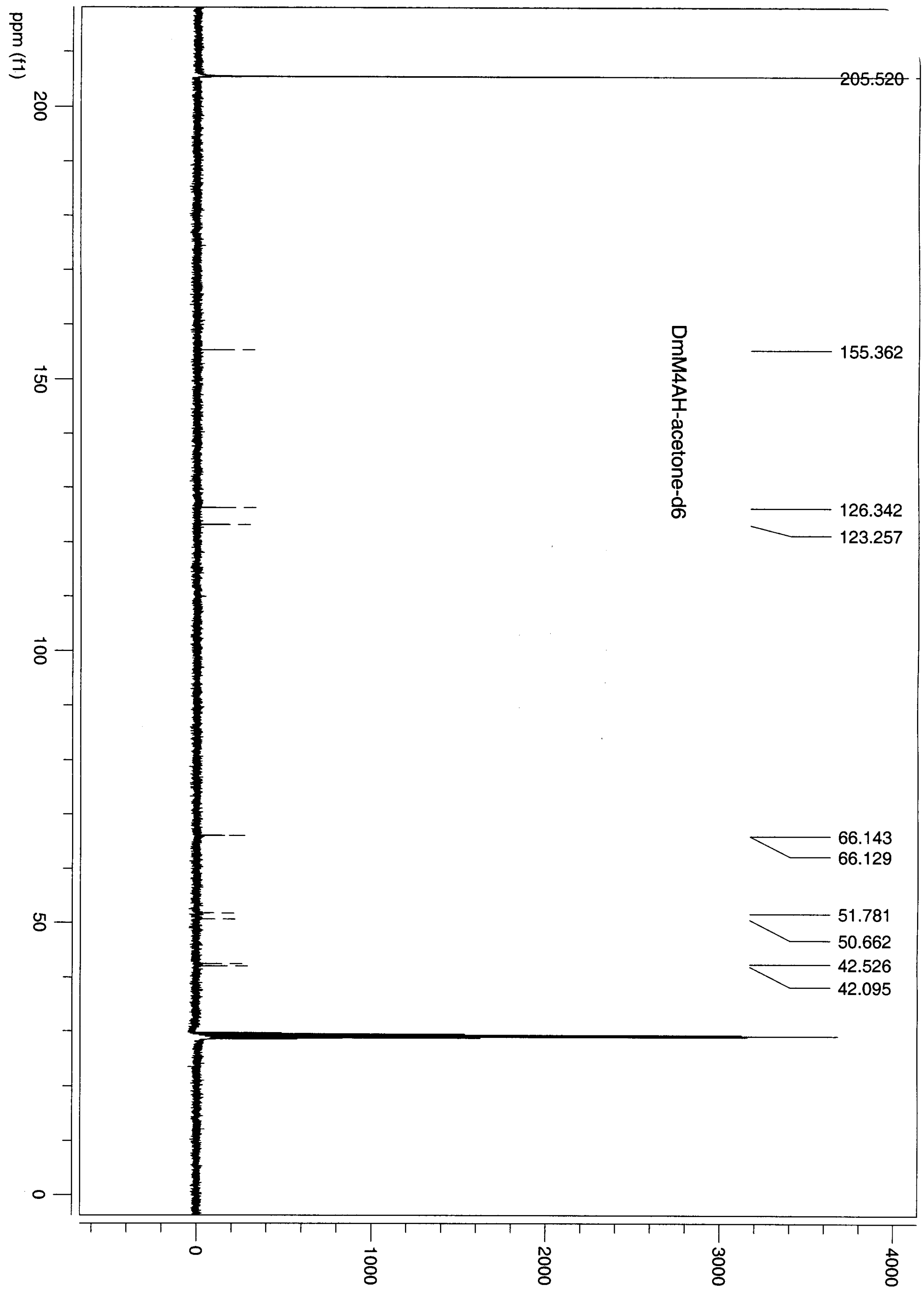
7.875
7.884
7.896
8.584
8.588
8.605
8.609
9.098
9.101
9.109
9.113
3.34
3.57
1.73
3.60
3.36
1.06
0.96
1.00
1000
500
0
0.0
5.0

5c, H-NMR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155.362
126.342
123.257
66.143
66.129
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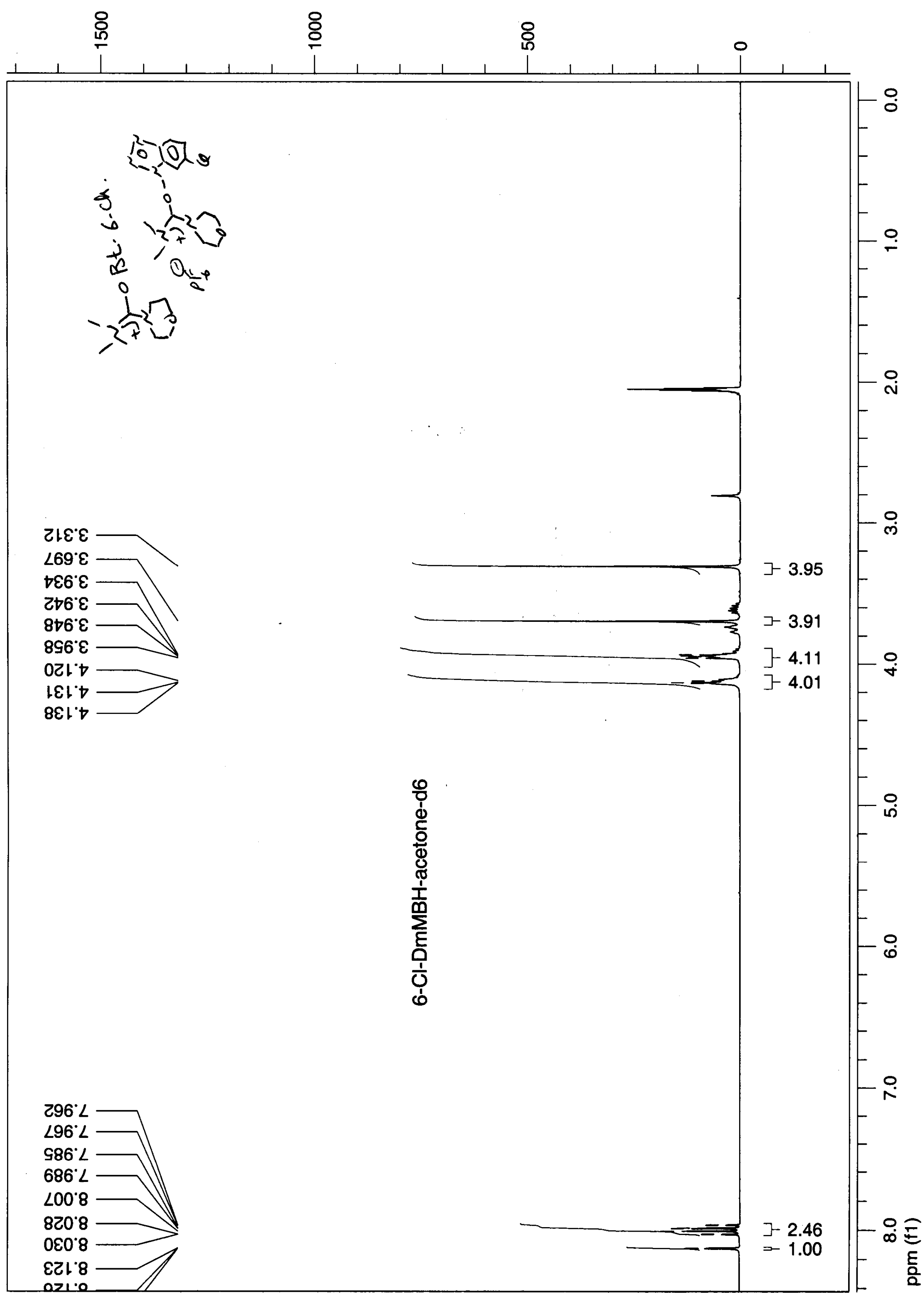
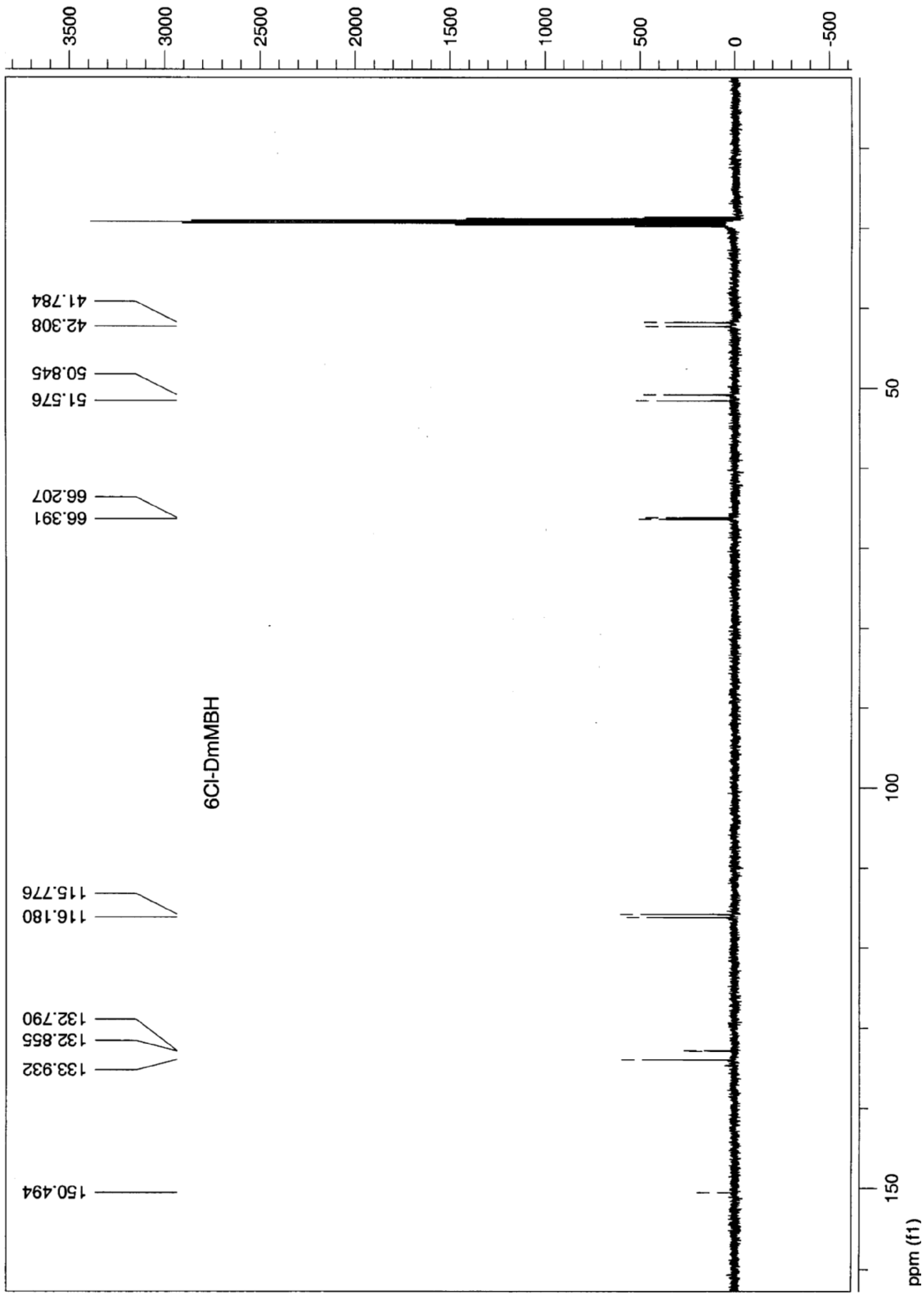
42.095
ppm (f1)
200
150
100
50
0
0
1000
2000
3000
4000

5c, C-NMR



5d. H-NMR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42.308
50.845
51.576
66.207
66.391
115.776
116.180
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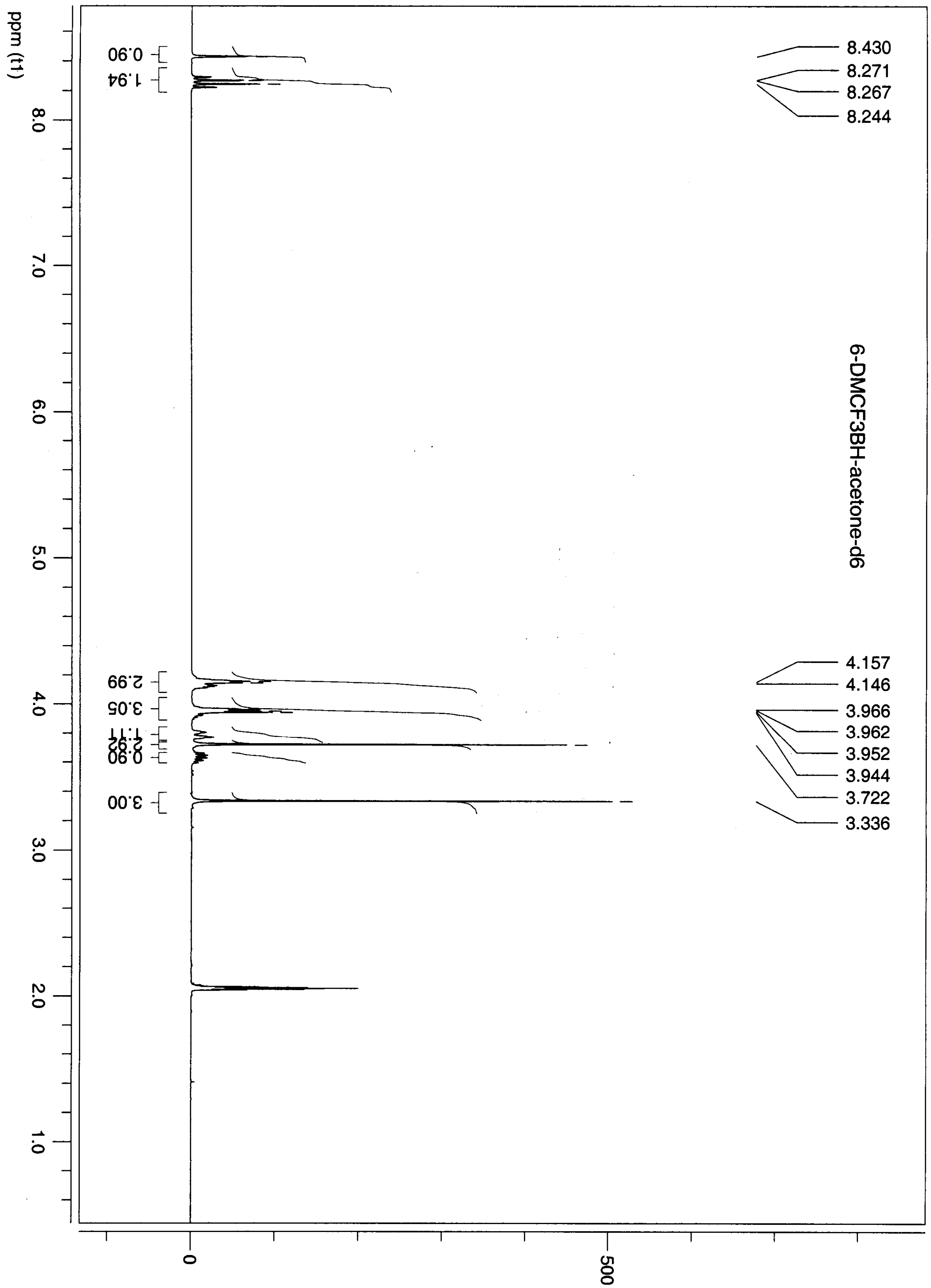
132.855
133.932
150.494
ppm (f1)

5d, C-NMR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8.267
8.244
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4.146
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3.962
3.952
3.944
3.722
3.336
0.90
1.94
2.99
3.05
1.11
2.92
0.90
3.00
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7.0
6.0
5.0
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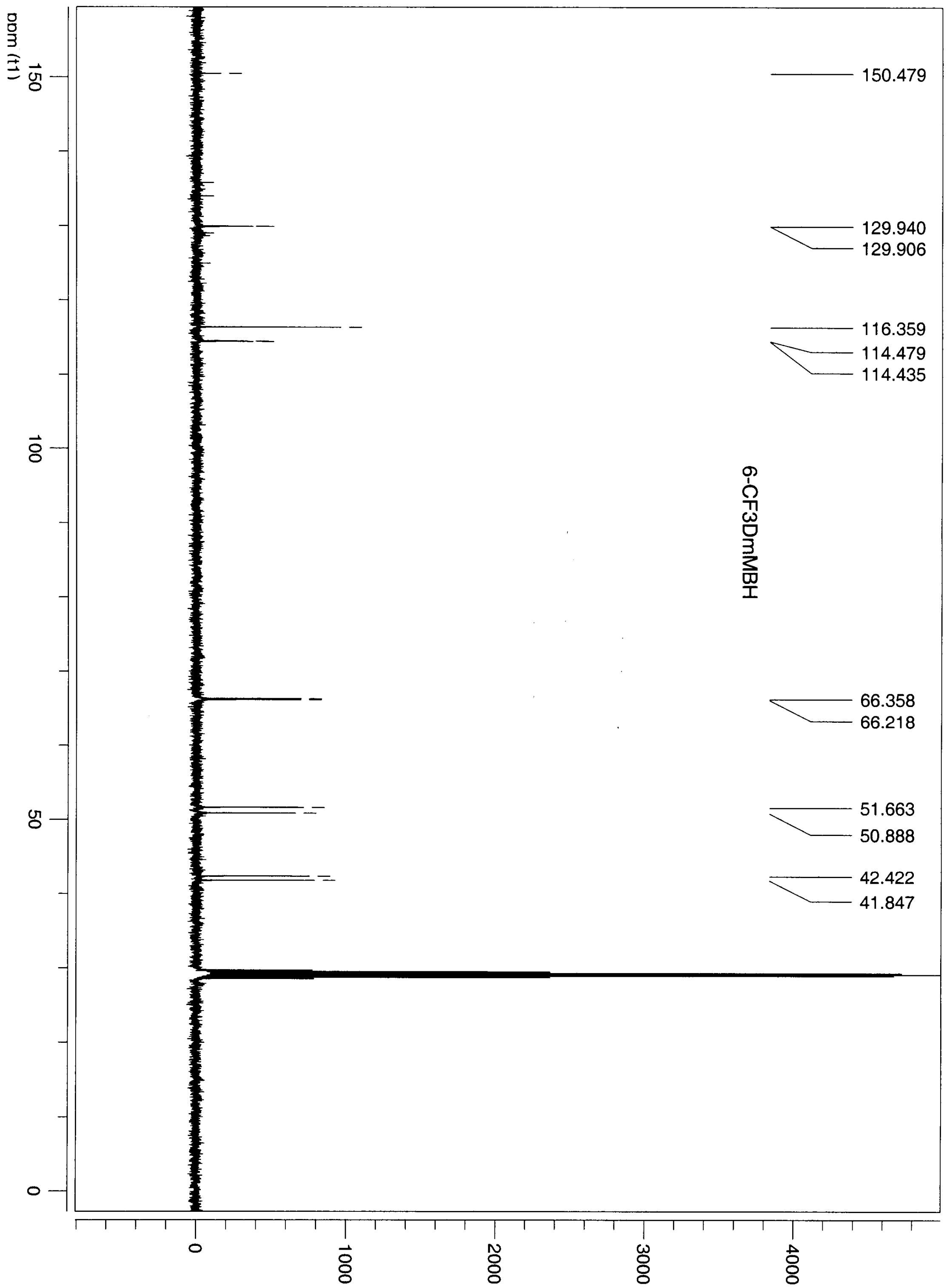
3.0
2.0
1.0
0
500

5e, H-NMR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100
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129.906
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114.479
114.435
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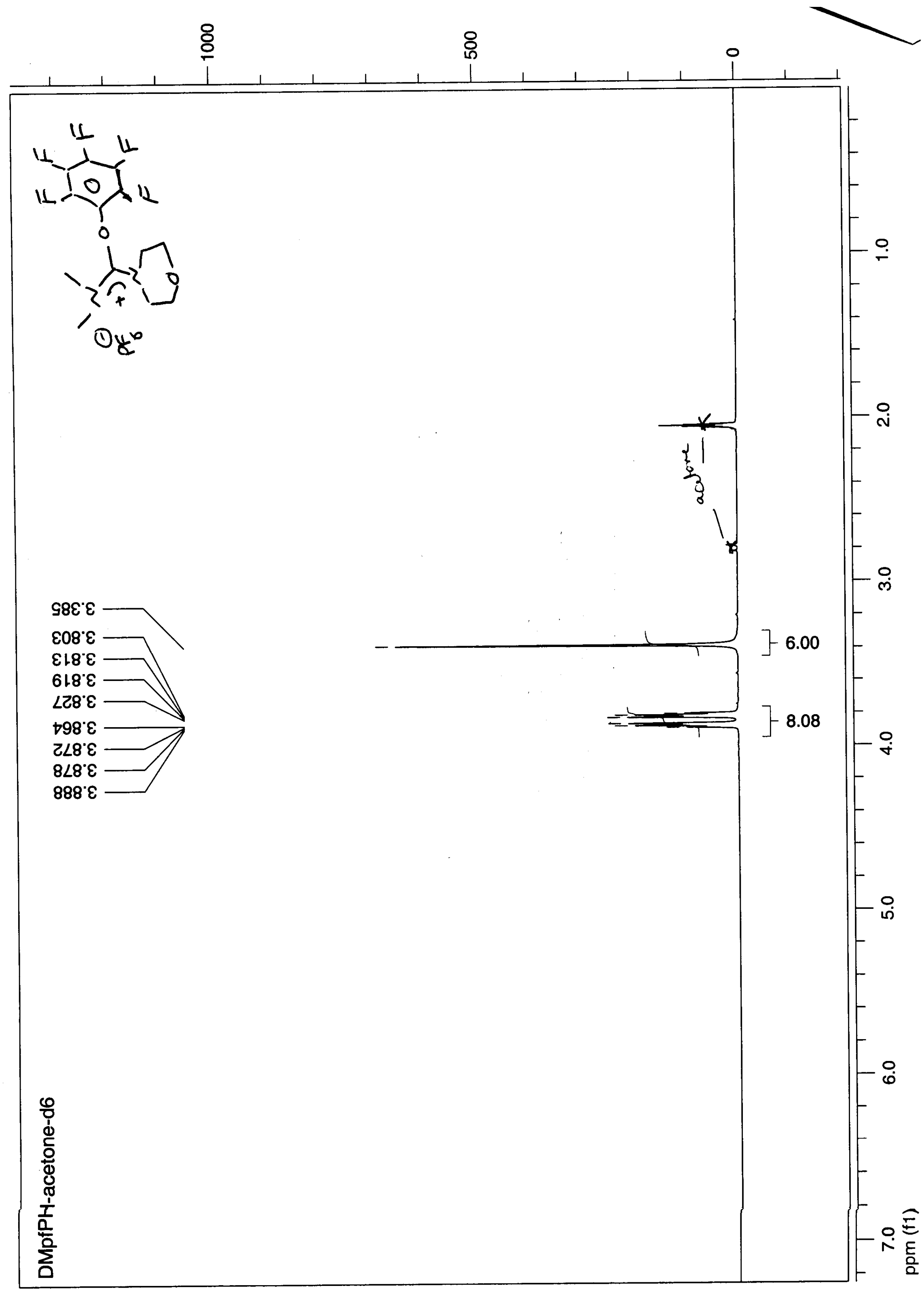
66.218
51.663
50.888
42.422
41.847
1000
2000
3000
4000

5e, C-NMR


DMpfPH-acetone-d6
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3.803
3.813
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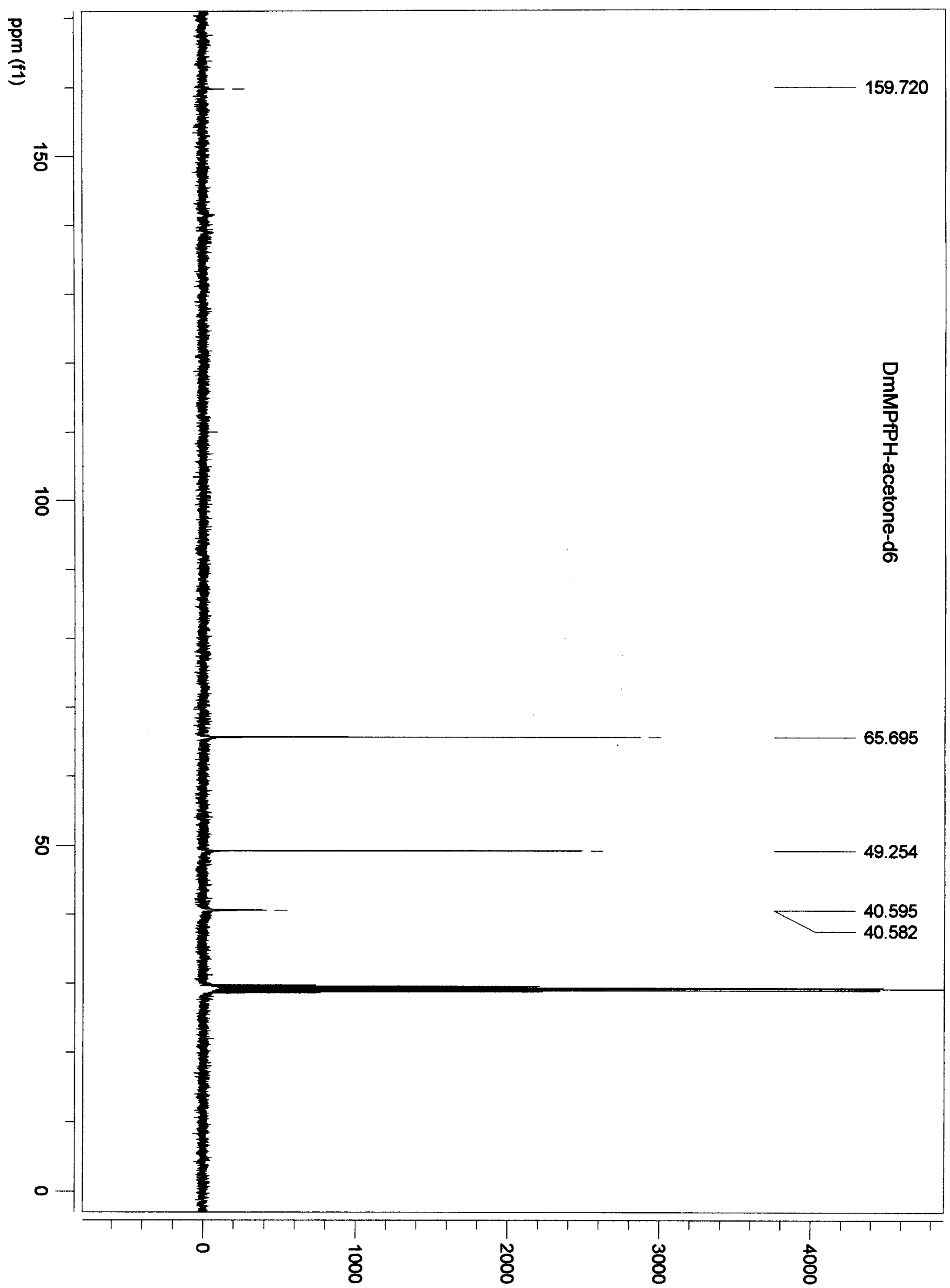
3.888
6.00
8.08
ppm (f1)
acetone

5f, H-NMR


ppm (f1)
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DmMPfPH-acetone-d6
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49.254
40.595
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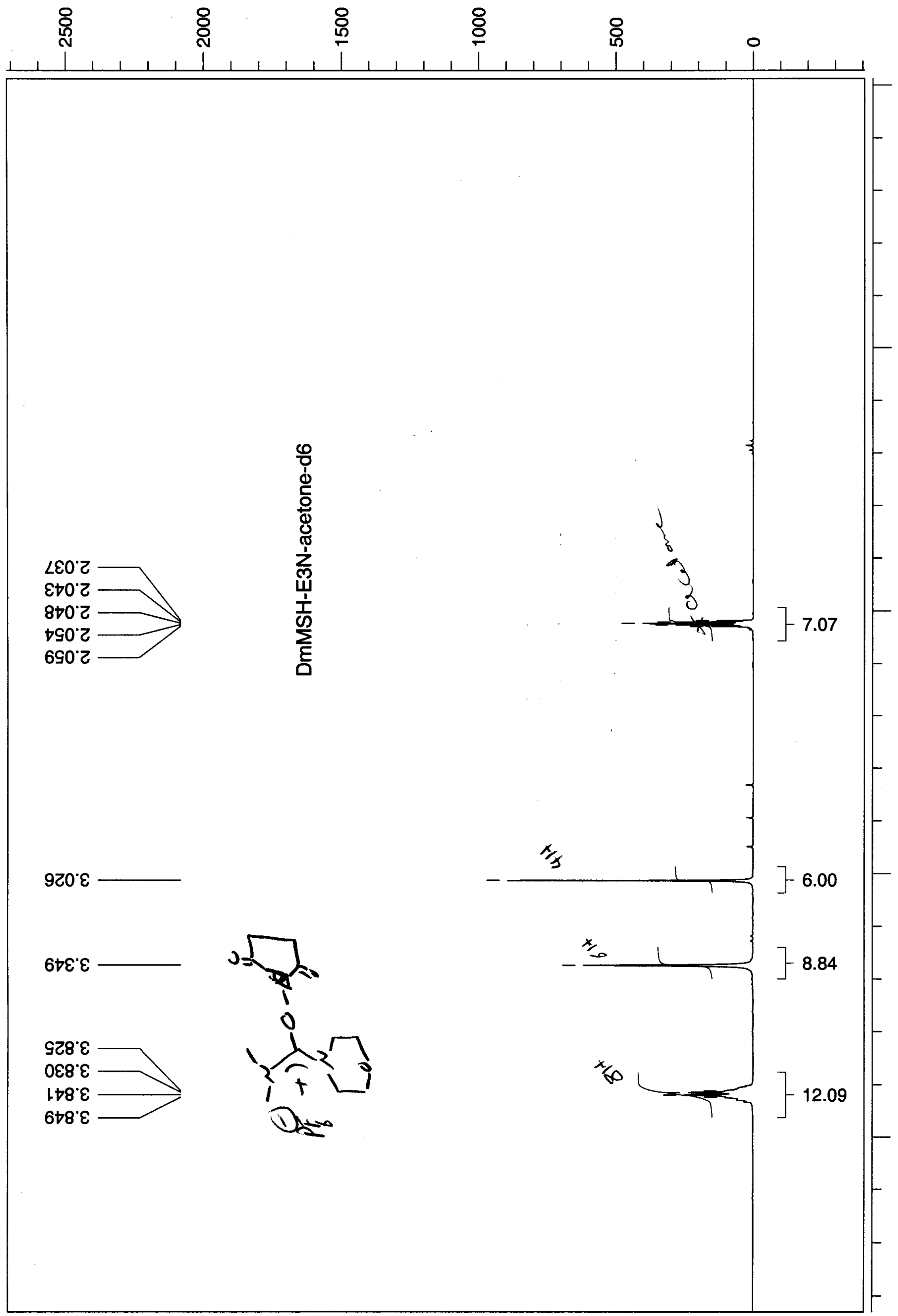
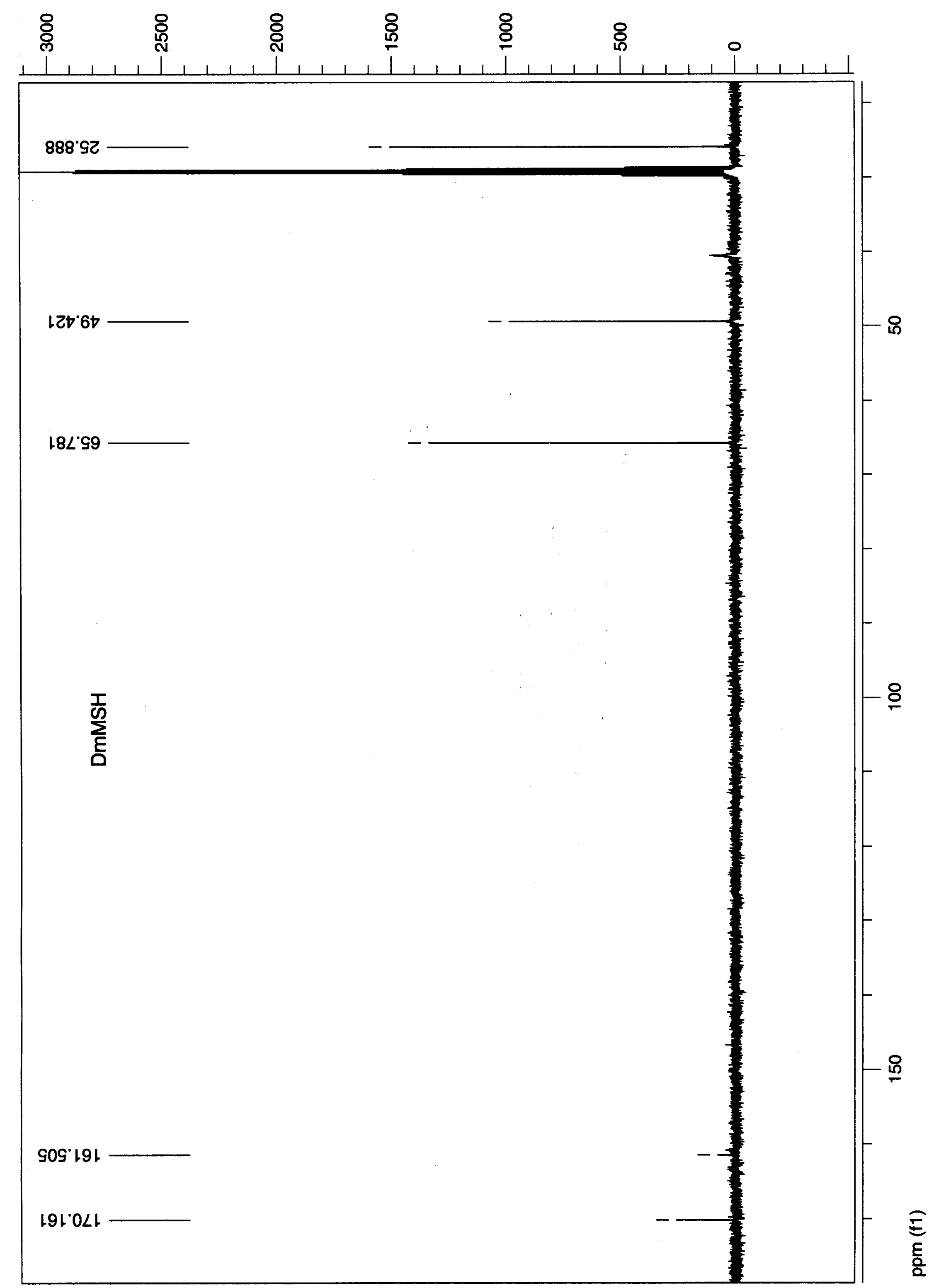
150
100
50
0
1000
2000
3000
4000

5f, C-NMR

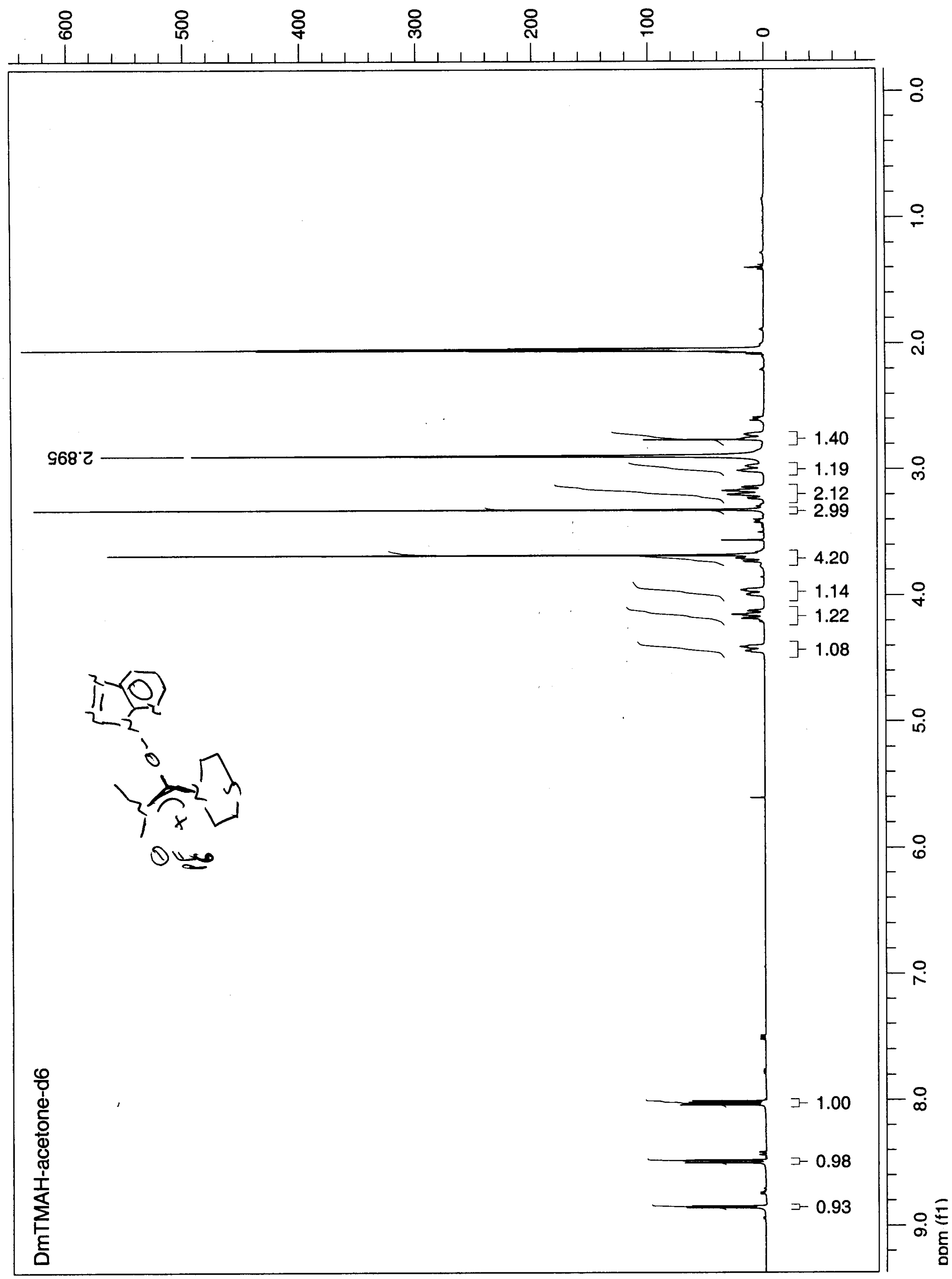


DmMSH-E3N-acetone-d6
2.037
2.043
2.048
2.054
2.059
3.026
3.349
3.825
3.830
3.841
3.849
7.07
6.00
8.84
12.09
2500
2000
1500
1000
500
0

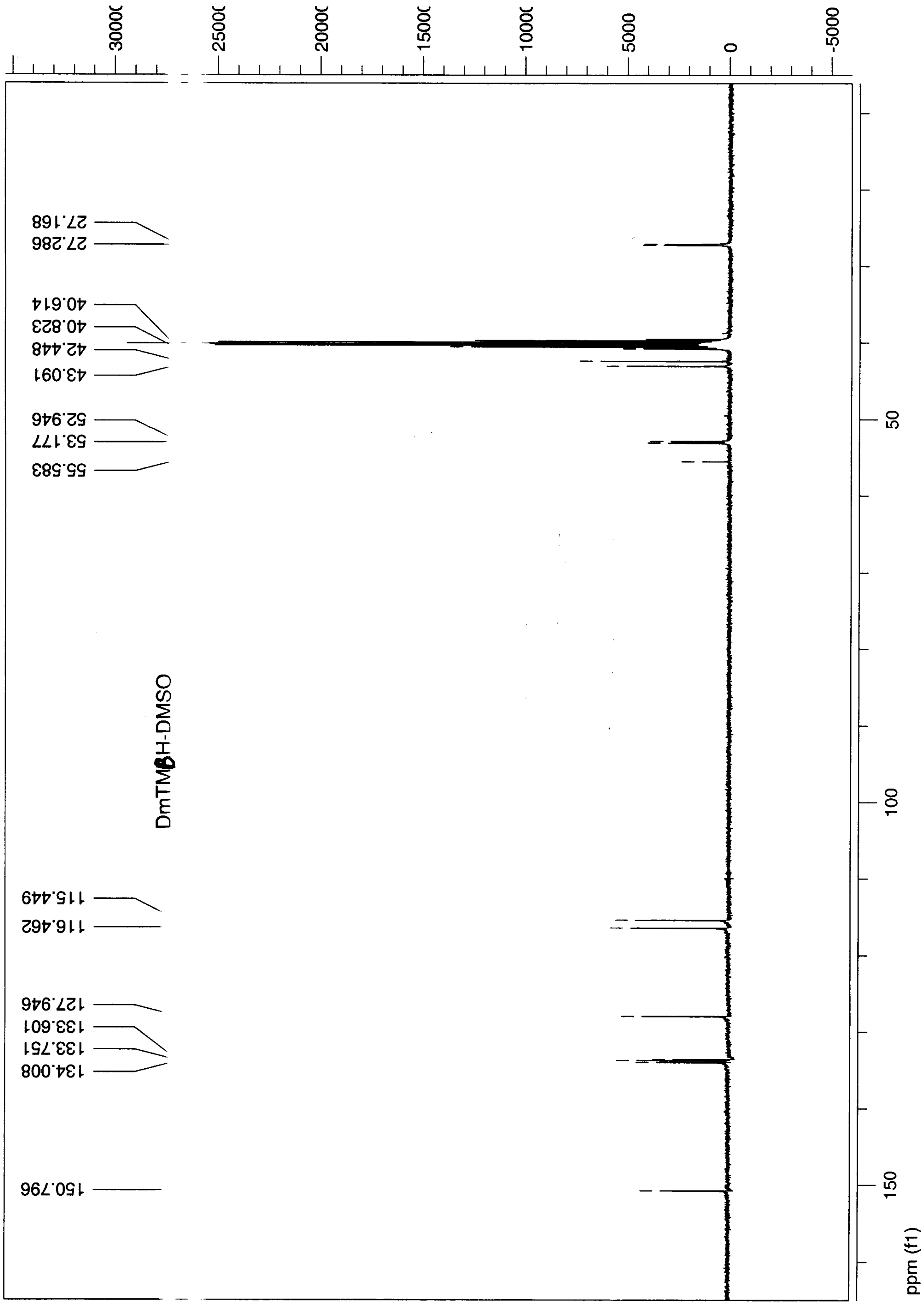
5g, H-NMR



5g, C-NMR



6a, H-NMR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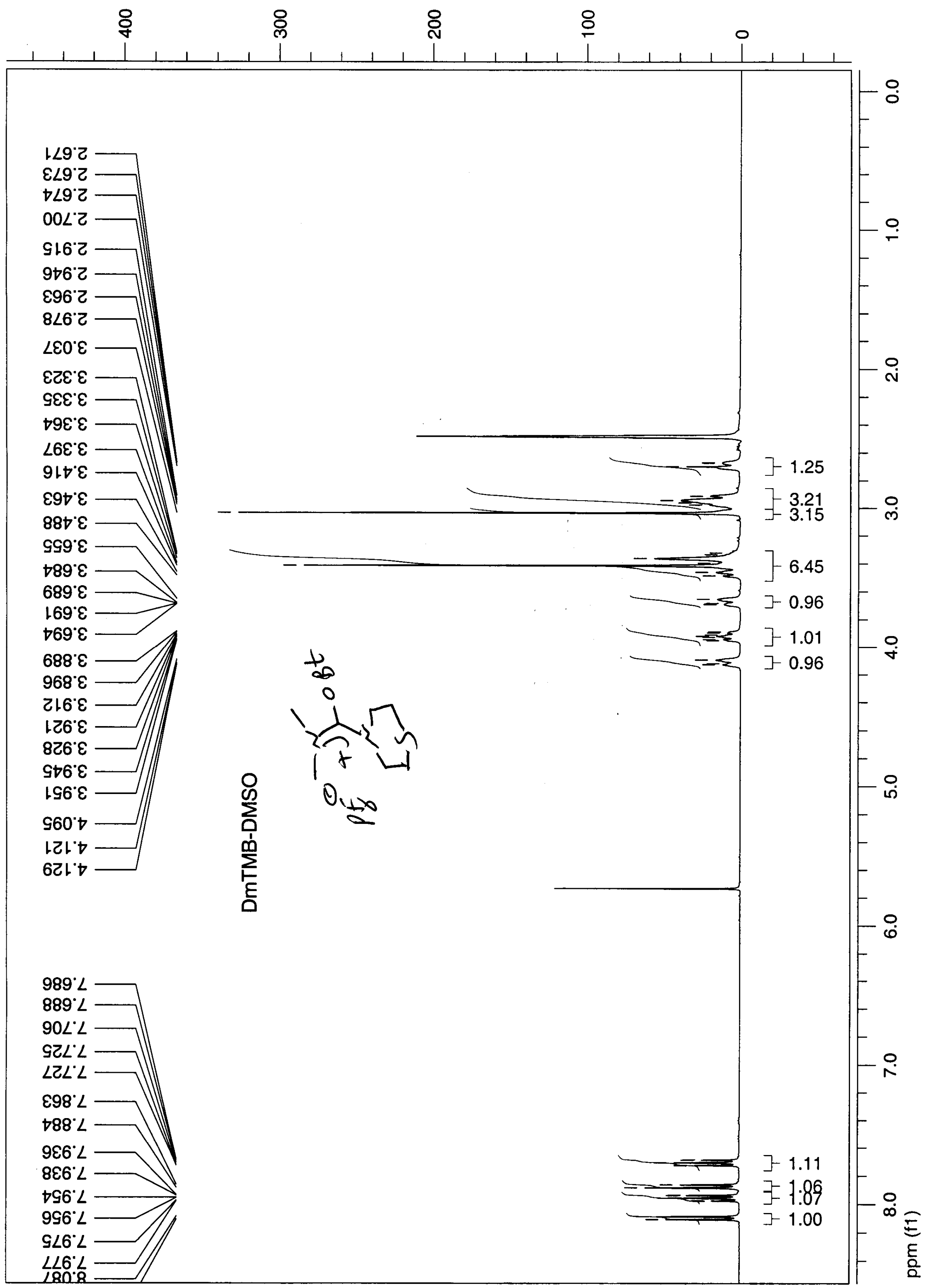
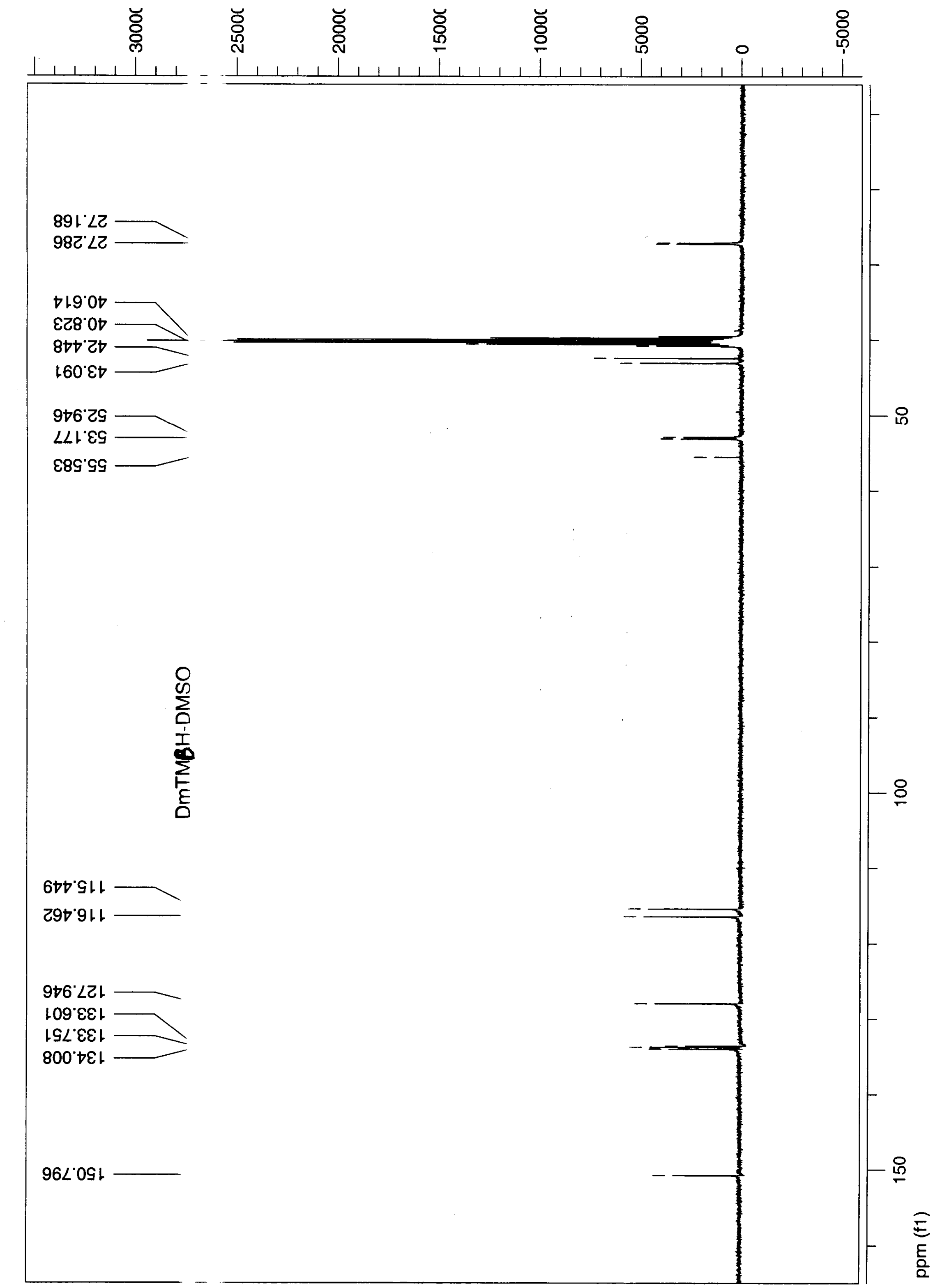
27.286
40.614
40.823
42.448
43.091
52.946
53.177
55.583
DmTMBH-DMSO
115.449
116.462
127.946
133.601
133.751
134.008
150.796
ppm (f1)

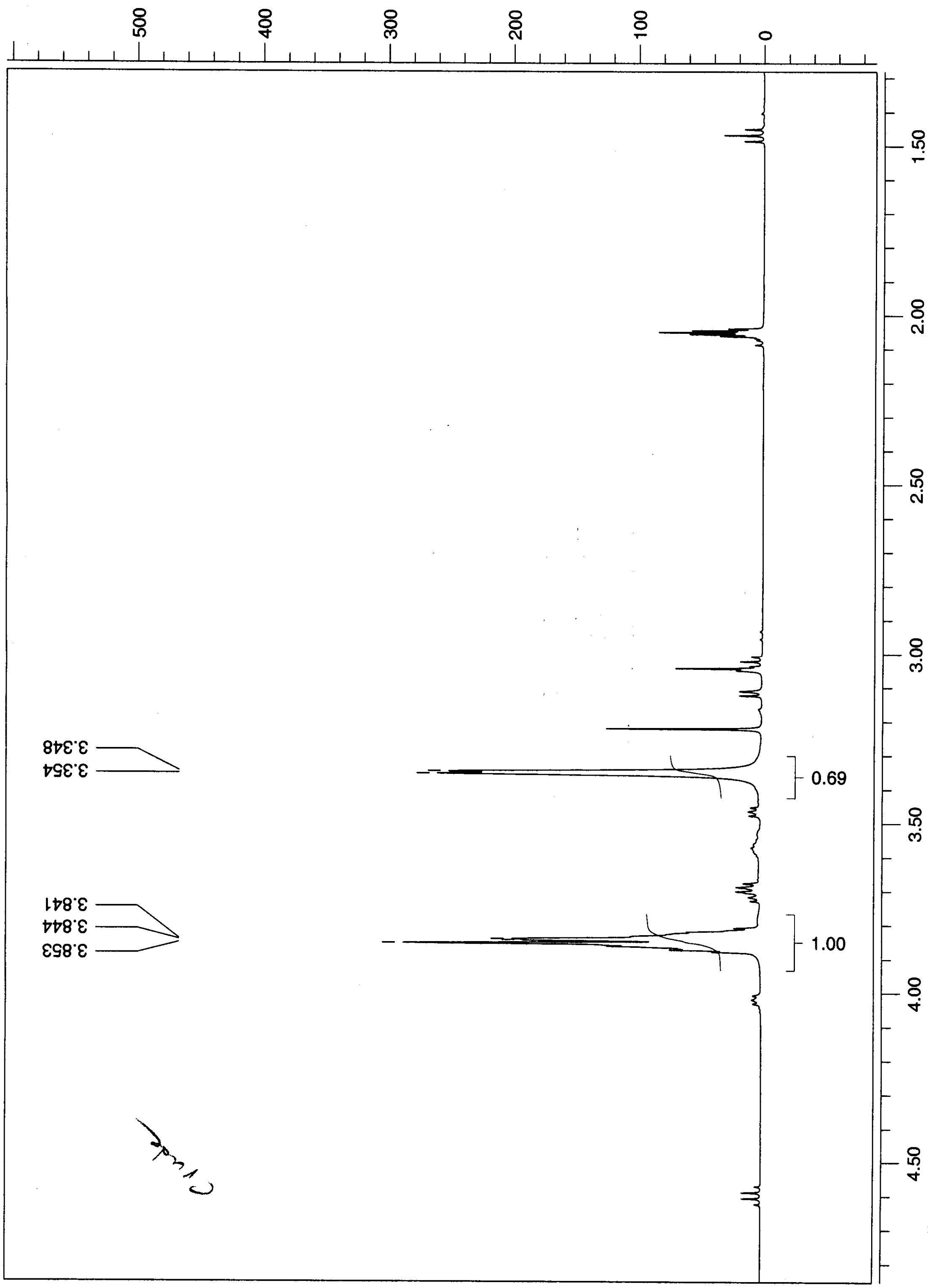
6a, C-NMr



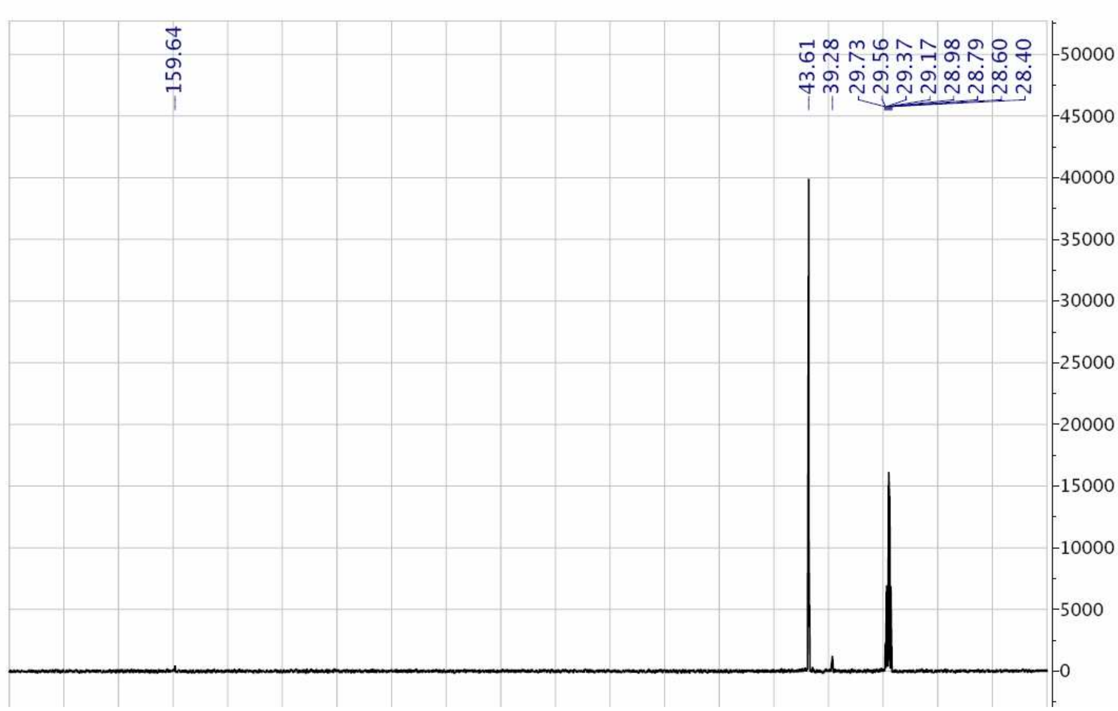
6b, H-NMR


27.168
27.286
40.614
40.823
42.448
43.091
52.946
53.177
55.583
DmTMBH-DMSO
115.449
116.462
127.946
133.601
133.751
134.008
150.796
ppm (f1)

6b, C-NMR



7, H-NMR



159.64
43.61
39.28
29.73
29.56
29.37
29.17
28.98
28.79
28.60
28.40
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0

7, C-NMR

5a, X-ray table

24 Aug 2006

Louis J. Farrugia

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Experimental

Crystal data

$C_{12}H_{18}N_6O_2.F_6P$	$\lambda = 0.71073$ Å
$M_r = 423.29$	Mo $K\alpha$ radiation
Monoclinic	$\lambda = 0.7107$ Å
$P2_1/c$	Cell parameters from 4304 reflections
$a = 6.1372$ (10) Å	$\theta = 3.39$–$22.95°$
$b = 22.607$ (4) Å	$\mu = 0.256$ mm^{-1}
$c = 12.0984$ (19) Å	$T = 100$ (2) K
$\beta = 102.067$ (9)°	Prism
$V = 1641.4$ (5) Å^3	Colourless
$Z = 4$	$0.46 \times 0.08 \times 0.06$ mm
$D_x = 1.713$ Mg m^{-3}	Crystal source: synthesis as described
D_m not measured	

Data collection

BRUKER APPEX–II CCD diffractometer	1699 reflections with
Four-circle diffractometer	>2sigma(I)
ω and phi scans	$R_{\text{int}} = 0.1171$
Absorption correction:	$\theta_{\text{max}} = 23.25°$
multi-scan BRUKER *SADABS*	$h = -6 \rightarrow 6$
Please give reference	$k = 0 \rightarrow 25$
$T_{\text{min}} = 0.8913$, $T_{\text{max}} = 0.9848$	$l = 0 \rightarrow 13$
23665 measured reflections	**? standard reflections**
2350 independent reflections	**every ? reflections**
	intensity decay: ?%

Refinement

Refinement on F^2

$R(F) = 0.0686$

$wR(F^2) = 0.1857$

$S = 0.988$

2350 reflections

304 parameters

H-atom parameters constrained

$w=1/[\sigma^2(F_o^2) + (0.1317P)^2 + 0.0000P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0$

$\Delta\rho_{\max} = 0.432$ e Å^{-3}

$\Delta\rho_{\min} = -0.449$ e Å^{-3}

Extinction correction: *SHELXL*

Extinction coefficient: 0.019 (5)

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a^i a^j \mathbf{a}_i.\mathbf{a}_j$.

	Occupancy	x	y	z	U_{eq}
N1	1	0.2637 (7)	0.09246 (17)	0.6003 (3)	0.0232 (11)
N2	1	0.4708 (7)	0.06605 (19)	0.6289 (4)	0.0282 (11)
N3	1	0.5511 (7)	0.06601 (17)	0.5371 (3)	0.0224 (10)
C4	1	0.4019 (8)	0.09336 (19)	0.4461 (4)	0.0205 (12)
N5	1	0.4406 (7)	0.10146 (17)	0.3431 (4)	0.0286 (11)
C6	1	0.2699 (9)	0.1294 (2)	0.2757 (5)	0.0328 (14)
C7	1	0.0756 (9)	0.1481 (2)	0.3091 (5)	0.0321 (14)
C8	1	0.0414 (9)	0.1387 (2)	0.4153 (5)	0.0271 (13)
C9	1	0.2200 (8)	0.10994 (19)	0.4881 (4)	0.0219 (12)
O10	1	0.7403 (6)	0.04504 (15)	0.5343 (3)	0.0315 (10)
C11	1	0.1456 (8)	0.1058 (2)	0.6834 (4)	0.0244 (13)
N12	1	0.0663 (7)	0.16010 (19)	0.6844 (4)	0.0327 (12)
C13	1	−0.1297 (10)	0.1751 (3)	0.7294 (6)	0.0523 (18)
C14	1	0.1813 (11)	0.2119 (2)	0.6475 (6)	0.0447 (17)
N15	1	0.1193 (7)	0.06379 (18)	0.7556 (4)	0.0282 (11)
C16	1	0.1229 (9)	−0.0001 (2)	0.7291 (4)	0.0302 (13)
C17	1	0.2786 (10)	−0.0314 (3)	0.8245 (5)	0.0411 (16)
O18	1	0.2159 (6)	−0.0212 (2)	0.9299 (3)	0.0466 (12)
C19	1	0.2307 (10)	0.0406 (3)	0.9557 (5)	0.0449 (16)
C20	1	0.0726 (9)	0.0759 (2)	0.8688 (4)	0.0338 (14)
P31A	0.67	0.5454 (5)	0.21756 (18)	0.9486 (3)	0.0360 (8)
F32A	0.67	0.8061 (9)	0.2055 (2)	0.9883 (5)	0.0447 (14)
F33A	0.67	0.5377 (16)	0.1552 (7)	0.8792 (10)	0.029 (2)
F34A	0.67	0.5868 (9)	0.2502 (2)	0.8375 (5)	0.0470 (14)
F35A	0.67	0.2816 (14)	0.2297 (4)	0.9103 (7)	0.047 (2)
F36A	0.67	0.5003 (10)	0.1859 (2)	1.0592 (6)	0.0475 (15)
F37A	0.67	0.5736 (9)	0.28012 (19)	1.0141 (4)	0.0484 (14)
P31B	0.33	0.5704 (8)	0.1862 (3)	0.9923 (6)	0.0242 (11)
F36B	0.33	0.4696 (15)	0.1319 (4)	1.0483 (7)	0.033 (2)
F32B	0.33	0.8133 (14)	0.1587 (4)	1.0239 (8)	0.035 (2)
F37B	0.33	0.6170 (17)	0.2199 (4)	1.1105 (8)	0.037 (2)
F34B	0.33	0.665 (2)	0.2425 (5)	0.9330 (11)	0.050 (3)
F35B	0.33	0.329 (2)	0.2135 (7)	0.9550 (14)	0.040 (4)
F33B	0.33	0.479 (5)	0.1547 (15)	0.875 (2)	0.029 (2)

Table 2. *Anisotropic displacement parameters* (Å²)

U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}

N1	0.022 (3)	0.034 (2)	0.010 (2)	−0.0003 (18)	−0.0040 (19)	0.0006 (18)
N2	0.025 (3)	0.045 (3)	0.011 (3)	0.001 (2)	−0.004 (2)	−0.001 (2)
N3	0.019 (3)	0.033 (2)	0.011 (3)	−0.0004 (19)	−0.0061 (19)	−0.0003 (18)
C4	0.027 (3)	0.021 (2)	0.010 (3)	−0.001 (2)	−0.006 (2)	0.003 (2)
N5	0.037 (3)	0.030 (2)	0.017 (3)	−0.004 (2)	0.000 (2)	0.0034 (19)
C6	0.044 (4)	0.037 (3)	0.015 (3)	−0.001 (3)	−0.002 (3)	0.008 (2)
C7	0.031 (3)	0.036 (3)	0.024 (3)	0.004 (2)	−0.008 (3)	0.018 (2)
C8	0.026 (3)	0.031 (3)	0.022 (3)	0.003 (2)	−0.001 (2)	0.009 (2)
C9	0.026 (3)	0.021 (2)	0.015 (3)	−0.002 (2)	−0.006 (2)	0.001 (2)
O10	0.026 (2)	0.046 (2)	0.020 (2)	0.0049 (17)	−0.0008 (17)	0.0051 (17)
C11	0.021 (3)	0.038 (3)	0.009 (3)	−0.008 (2)	−0.007 (2)	0.000 (2)
N12	0.038 (3)	0.039 (3)	0.021 (3)	−0.003 (2)	0.005 (2)	−0.004 (2)
C13	0.053 (4)	0.064 (4)	0.045 (4)	0.018 (3)	0.022 (3)	0.011 (3)
C14	0.065 (5)	0.034 (3)	0.040 (4)	−0.009 (3)	0.022 (3)	−0.011 (3)
N15	0.028 (3)	0.042 (2)	0.011 (2)	−0.006 (2)	−0.0046 (19)	0.000 (2)
C16	0.034 (3)	0.037 (3)	0.016 (3)	−0.004 (2)	−0.006 (2)	0.005 (2)
C17	0.038 (4)	0.061 (4)	0.021 (4)	0.006 (3)	−0.001 (3)	0.020 (3)
O18	0.037 (3)	0.082 (3)	0.018 (2)	0.009 (2)	−0.0003 (19)	0.020 (2)
C19	0.032 (4)	0.080 (5)	0.017 (3)	−0.002 (3)	−0.008 (3)	0.006 (3)
C20	0.028 (3)	0.055 (3)	0.016 (3)	−0.008 (3)	−0.002 (2)	0.000 (3)
P31A	0.051 (2)	0.0350 (17)	0.0236 (17)	−0.0011 (15)	0.0123 (14)	−0.0039 (14)
F32A	0.045 (4)	0.052 (3)	0.038 (3)	−0.005 (3)	0.013 (3)	−0.014 (3)
F33A	0.017 (7)	0.0403 (17)	0.028 (2)	−0.003 (4)	0.001 (4)	−0.0025 (15)
F34A	0.078 (4)	0.040 (3)	0.027 (3)	0.008 (3)	0.023 (3)	0.009 (2)
F35A	0.045 (5)	0.052 (4)	0.041 (6)	0.018 (3)	0.008 (4)	−0.006 (4)
F36A	0.054 (4)	0.063 (4)	0.030 (4)	0.009 (3)	0.018 (3)	0.013 (3)
F37A	0.070 (4)	0.042 (3)	0.035 (3)	0.002 (2)	0.016 (3)	−0.013 (2)
P31B	0.027 (3)	0.025 (3)	0.019 (3)	−0.007 (2)	0.000 (2)	−0.005 (3)
F36B	0.046 (6)	0.037 (5)	0.014 (5)	−0.013 (4)	0.001 (4)	−0.001 (4)
F32B	0.036 (6)	0.045 (6)	0.022 (6)	0.001 (4)	−0.003 (4)	−0.005 (4)
F37B	0.041 (6)	0.035 (5)	0.030 (6)	0.000 (5)	−0.007 (5)	−0.018 (5)
F34B	0.066 (9)	0.039 (6)	0.041 (9)	−0.021 (7)	0.002 (7)	0.020 (6)
F35B	0.019 (8)	0.047 (9)	0.044 (11)	0.011 (6)	−0.015 (7)	−0.009 (7)
F33B	0.017 (7)	0.0403 (17)	0.028 (2)	−0.003 (4)	0.001 (4)	−0.0025 (15)

Table 3. *Selected geometric parameters (Å, °)*

N1—N2	1.381 (6)	N15—C16	1.482 (7)
N1—C9	1.385 (6)	N15—C20	1.483 (7)
N1—C11	1.390 (6)	C16—C17	1.511 (7)
N2—N3	1.306 (6)	C16—H16A	0.99
N3—O10	1.261 (5)	C16—H16B	0.99
N3—C4	1.418 (6)	C17—O18	1.425 (7)
C4—N5	1.329 (6)	C17—H17A	0.99
C4—C9	1.372 (7)	C17—H17B	0.99
N5—C6	1.344 (7)	O18—C19	1.431 (7)
C6—C7	1.402 (8)	C19—C20	1.502 (8)
C6—H6	0.95	C19—H19A	0.99
C7—C8	1.361 (7)	C19—H19B	0.99
C7—H7	0.95	C20—H20A	0.99
C8—C9	1.413 (7)	C20—H20B	0.99
C8—H8	0.95	P31A—F36A	1.593 (6)
O10—H10	0.84	P31A—F32A	1.595 (6)
C11—N12	1.321 (7)	P31A—F34A	1.600 (6)
C11—N15	1.324 (6)	P31A—F35A	1.612 (8)
N12—C13	1.460 (7)	P31A—F37A	1.613 (5)
N12—C14	1.484 (7)	P31A—F33A	1.637 (12)
C13—H13A	0.98	P31B—F35B	1.581 (13)
C13—H13B	0.98	P31B—F33B	1.59 (2)
C13—H13C	0.98	P31B—F32B	1.587 (10)
C14—H14A	0.98	P31B—F36B	1.588 (10)
C14—H14B	0.98	P31B—F37B	1.593 (10)
C14—H14C	0.98	P31B—F34B	1.628 (11)
N2—N1—C9	110.4 (4)	C7—C6—H6	117.7
N2—N1—C11	120.3 (4)	C8—C7—C6	122.2 (5)
C9—N1—C11	128.5 (4)	C8—C7—H7	118.9
N3—N2—N1	106.1 (4)	C6—C7—H7	118.9
O10—N3—N2	122.4 (4)	C7—C8—C9	114.1 (5)
O10—N3—C4	126.3 (4)	C7—C8—H8	123
N2—N3—C4	111.3 (4)	C9—C8—H8	123
N5—C4—C9	128.9 (5)	C4—C9—N1	106.2 (4)
N5—C4—N3	125.1 (4)	C4—C9—C8	118.7 (5)
C9—C4—N3	106.0 (4)	N1—C9—C8	135.1 (5)
C4—N5—C6	111.4 (4)	N3—O10—H10	109.5
N5—C6—C7	124.7 (5)	N12—C11—N15	124.4 (5)
N5—C6—H6	117.7	N12—C11—N1	117.2 (4)

N15—C11—N1	118.4 (4)	H19A—C19—H19B	108
C11—N12—C13	123.6 (5)	N15—C20—C19	109.1 (5)
C11—N12—C14	122.1 (5)	N15—C20—H20A	109.9
C13—N12—C14	114.1 (5)	C19—C20—H20A	109.9
N12—C13—H13A	109.5	N15—C20—H20B	109.9
N12—C13—H13B	109.5	C19—C20—H20B	109.9
H13A—C13—H13B	109.5	H20A—C20—H20B	108.3
N12—C13—H13C	109.5	F36A—P31A—F32A	90.5 (4)
H13A—C13—H13C	109.5	F36A—P31A—F34A	178.9 (4)
H13B—C13—H13C	109.5	F32A—P31A—F34A	90.4 (4)
N12—C14—H14A	109.5	F36A—P31A—F35A	88.7 (4)
N12—C14—H14B	109.5	F32A—P31A—F35A	179.2 (5)
H14A—C14—H14B	109.5	F34A—P31A—F35A	90.3 (4)
N12—C14—H14C	109.5	F36A—P31A—F37A	90.1 (3)
H14A—C14—H14C	109.5	F32A—P31A—F37A	90.3 (3)
H14B—C14—H14C	109.5	F34A—P31A—F37A	89.3 (4)
C11—N15—C16	123.3 (4)	F35A—P31A—F37A	89.4 (4)
C11—N15—C20	123.4 (4)	F36A—P31A—F33A	93.0 (7)
C16—N15—C20	113.3 (4)	F32A—P31A—F33A	85.8 (4)
N15—C16—C17	109.1 (4)	F34A—P31A—F33A	87.7 (6)
N15—C16—H16A	109.9	F35A—P31A—F33A	94.6 (5)
C17—C16—H16A	109.9	F37A—P31A—F33A	175.0 (5)
N15—C16—H16B	109.9	F35B—P31B—F33B	77.7 (12)
C17—C16—H16B	109.9	F35B—P31B—F32B	177.4 (9)
H16A—C16—H16B	108.3	F33B—P31B—F32B	100.2 (12)
O18—C17—C16	111.2 (5)	F35B—P31B—F36B	89.9 (7)
O18—C17—H17A	109.4	F33B—P31B—F36B	86.6 (13)
C16—C17—H17A	109.4	F32B—P31B—F36B	91.4 (6)
O18—C17—H17B	109.4	F35B—P31B—F37B	92.9 (8)
C16—C17—H17B	109.4	F33B—P31B—F37B	170.0 (12)
H17A—C17—H17B	108	F32B—P31B—F37B	89.3 (5)
C17—O18—C19	109.7 (4)	F36B—P31B—F37B	90.0 (7)
O18—C19—C20	111.4 (5)	F35B—P31B—F34B	88.2 (8)
O18—C19—H19A	109.4	F33B—P31B—F34B	92.6 (14)
C20—C19—H19A	109.4	F32B—P31B—F34B	90.4 (6)
O18—C19—H19B	109.4	F36B—P31B—F34B	178.1 (7)
C20—C19—H19B	109.4	F37B—P31B—F34B	90.5 (6)

C9—N1—N2—N3	−1.2 (5)	C7—C8—C9—N1	178.5 (5)
C11—N1—N2—N3	−172.1 (4)	N2—N1—C11—N12	129.7 (5)
N1—N2—N3—O10	179.7 (4)	C9—N1—C11—N12	−39.4 (7)
N1—N2—N3—C4	1.2 (5)	N2—N1—C11—N15	−50.6 (6)
O10—N3—C4—N5	−0.5 (7)	C9—N1—C11—N15	140.4 (5)
N2—N3—C4—N5	177.9 (4)	N15—C11—N12—C13	−26.5 (8)
O10—N3—C4—C9	−179.1 (4)	N1—C11—N12—C13	153.3 (5)
N2—N3—C4—C9	−0.7 (5)	N15—C11—N12—C14	148.6 (5)
C9—C4—N5—C6	−0.8 (7)	N1—C11—N12—C14	−31.6 (7)
N3—C4—N5—C6	−179.1 (4)	N12—C11—N15—C16	154.0 (5)
C4—N5—C6—C7	0.4 (7)	N1—C11—N15—C16	−25.7 (7)
N5—C6—C7—C8	−0.9 (8)	N12—C11—N15—C20	−22.9 (8)
C6—C7—C8—C9	1.4 (7)	N1—C11—N15—C20	157.3 (4)
N5—C4—C9—N1	−178.6 (5)	C11—N15—C16—C17	130.8 (5)
N3—C4—C9—N1	−0.1 (5)	C20—N15—C16—C17	−52.0 (6)
N5—C4—C9—C8	1.5 (7)	N15—C16—C17—O18	56.3 (6)
N3—C4—C9—C8	−180.0 (4)	C16—C17—O18—C19	−62.1 (6)
N2—N1—C9—C4	0.8 (5)	C17—O18—C19—C20	62.4 (6)
C11—N1—C9—C4	170.7 (4)	C11—N15—C20—C19	−130.6 (5)
N2—N1—C9—C8	−179.3 (5)	C16—N15—C20—C19	52.1 (6)
C11—N1—C9—C8	−9.4 (8)	O18—C19—C20—N15	−56.5 (6)
C7—C8—C9—C4	−1.6 (6)		

5b, X-ray Table

21 Aug 2006

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Experimental

Crystal data

$C_{13}H_{18}N_5O_2.F_6P$	$\lambda = 0.71073$ Å
$M_r = 421.29$	Mo $K\alpha$ radiation
Monoclinic	$\lambda = 0.7107$ Å
$P2_1/c$	Cell parameters from 4248 reflections
$a = 6.12000$ (10) Å	$\theta = 3.11–28.93°$
$b = 23.9910$ (5) Å	$\mu = 0.249$ mm^{-1}
$c = 11.7260$ (3) Å	$T = 100$ (2) K
$\beta = 103.4190$ (10)°	Prism
$V = 1674.67$ (6) Å^3	Colourless
$Z = 4$	$0.42 \times 0.14 \times 0.04$ mm
$D_x = 1.671$ Mg m^{-3}	Crystal source: synthesis as described
D_m not measured	

Data collection

BRUKER APPEX–II CCD diffractometer	3363 reflections with
Four-circle diffractometer	>2sigma(I)
ω and phi scans	$R_{\text{int}} = 0.0438$
Absorption correction:	$\theta_{\max} = 30.13°$
multi-scan BRUKER *SADABS*	$h = -8 \rightarrow 8$
Please give reference	$k = 0 \rightarrow 29$
$T_{\min} = 0.9026$, $T_{\max} = 0.9901$	$l = 0 \rightarrow 16$
4945 measured reflections	**? standard reflections**
4660 independent reflections	**every ? reflections**
	intensity decay: ?%

Refinement

Refinement on F^2

$R(F) = 0.0412$

$wR(F^2) = 0.0978$

$S = 1.065$

4945 reflections

247 parameters

H-atom parameters constrained

$w=1/[\sigma^2(F_o^2) + (0.0472P)^2 + 0.2196P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.349$ e Å^{-3}

$\Delta\rho_{min} = -0.406$ e Å^{-3}

Extinction correction: *SHELXL*

Extinction coefficient: 0.0005 (5)

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
N1	0.7273 (2)	0.09800 (6)	0.60240 (11)	0.0121 (3)
N2	0.9301 (2)	0.07005 (6)	0.62712 (12)	0.0139 (3)
N3	1.0005 (2)	0.07268 (6)	0.52963 (12)	0.0133 (3)
C4	0.8498 (2)	0.10157 (7)	0.44029 (14)	0.0130 (3)
C5	0.8600 (3)	0.11409 (7)	0.32590 (14)	0.0163 (4)
C6	0.6809 (3)	0.14395 (8)	0.26113 (14)	0.0171 (4)
C7	0.5003 (3)	0.16043 (8)	0.30954 (14)	0.0167 (4)
C8	0.4917 (2)	0.14787 (7)	0.42301 (14)	0.0149 (3)
C9	0.6735 (2)	0.11791 (7)	0.48843 (13)	0.0127 (3)
O10	1.18668 (18)	0.05196 (5)	0.52057 (11)	0.0195 (3)
C11	0.6132 (2)	0.10739 (7)	0.69103 (13)	0.0113 (3)
N12	0.5357 (2)	0.15831 (6)	0.69835 (11)	0.0119 (3)
C13	0.6497 (3)	0.20863 (7)	0.67022 (15)	0.0168 (4)
C14	0.3376 (3)	0.16904 (8)	0.74555 (15)	0.0171 (4)
N15	0.5886 (2)	0.06529 (6)	0.75927 (11)	0.0115 (3)
C16	0.5805 (3)	0.00642 (7)	0.72264 (14)	0.0152 (3)
C17	0.7455 (3)	−0.02729 (8)	0.81346 (15)	0.0165 (3)
O18	0.70399 (19)	−0.02066 (5)	0.92685 (10)	0.0176 (3)
C19	0.7322 (3)	0.03647 (7)	0.96123 (14)	0.0162 (4)
C20	0.5621 (3)	0.07326 (7)	0.88057 (13)	0.0132 (3)
P30	1.02931 (7)	0.19145 (2)	0.99909 (4)	0.01580 (11)
F31	1.06681 (18)	0.22767 (5)	1.11713 (10)	0.0319 (3)
F32	0.98752 (17)	0.13784 (5)	1.07223 (9)	0.0247 (2)
F33	1.29154 (15)	0.17737 (5)	1.02706 (9)	0.0280 (3)
F34	0.76599 (16)	0.20526 (5)	0.97114 (10)	0.0270 (3)
F35	1.0722 (2)	0.24478 (5)	0.92649 (12)	0.0425 (3)
F36	0.98941 (15)	0.15416 (5)	0.88218 (8)	0.0229 (2)

Table 2. *Anisotropic displacement parameters* (Å²)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N1	0.0113 (5)	0.0147 (8)	0.0106 (6)	0.0030 (5)	0.0036 (5)	0.0032 (5)
N2	0.0119 (6)	0.0149 (8)	0.0150 (6)	0.0028 (5)	0.0033 (5)	−0.0001 (5)
N3	0.0120 (6)	0.0132 (8)	0.0152 (6)	0.0010 (5)	0.0044 (5)	−0.0010 (5)
C4	0.0122 (6)	0.0123 (9)	0.0145 (7)	−0.0015 (6)	0.0032 (6)	−0.0010 (6)
C5	0.0170 (7)	0.0189 (11)	0.0150 (8)	−0.0036 (6)	0.0079 (6)	−0.0022 (6)
C6	0.0212 (8)	0.0190 (11)	0.0120 (7)	−0.0042 (7)	0.0058 (6)	0.0002 (6)
C7	0.0172 (7)	0.0173 (10)	0.0143 (7)	−0.0013 (6)	0.0011 (6)	0.0021 (6)

C8	0.0126 (7)	0.0171 (10)	0.0151 (7)	0.0001 (6)	0.0034 (6)	0.0008 (6)
C9	0.0138 (7)	0.0137 (10)	0.0110 (7)	−0.0021 (6)	0.0036 (6)	0.0006 (6)
O10	0.0128 (5)	0.0241 (8)	0.0233 (6)	0.0056 (5)	0.0074 (5)	−0.0005 (5)
C11	0.0089 (6)	0.0151 (10)	0.0098 (7)	−0.0008 (6)	0.0020 (5)	0.0001 (6)
N12	0.0125 (6)	0.0114 (8)	0.0127 (6)	0.0007 (5)	0.0044 (5)	0.0010 (5)
C13	0.0205 (8)	0.0110 (10)	0.0194 (8)	−0.0015 (6)	0.0057 (6)	0.0019 (7)
C14	0.0137 (7)	0.0178 (10)	0.0215 (8)	0.0043 (6)	0.0079 (6)	0.0024 (7)
N15	0.0146 (6)	0.0088 (8)	0.0111 (6)	−0.0004 (5)	0.0032 (5)	0.0002 (5)
C16	0.0193 (8)	0.0105 (10)	0.0158 (7)	−0.0017 (6)	0.0038 (6)	−0.0005 (6)
C17	0.0183 (7)	0.0123 (10)	0.0197 (8)	0.0015 (6)	0.0063 (6)	0.0013 (7)
O18	0.0210 (6)	0.0138 (7)	0.0190 (6)	0.0008 (5)	0.0067 (5)	0.0042 (5)
C19	0.0174 (7)	0.0179 (11)	0.0134 (7)	0.0016 (6)	0.0037 (6)	0.0034 (6)
C20	0.0170 (7)	0.0132 (10)	0.0108 (7)	0.0004 (6)	0.0057 (6)	0.0010 (6)
P30	0.01194 (18)	0.0182 (3)	0.0166 (2)	−0.00174 (16)	0.00211 (15)	−0.00217 (18)
F31	0.0260 (5)	0.0313 (7)	0.0343 (6)	0.0011 (5)	−0.0010 (5)	−0.0207 (5)
F32	0.0333 (6)	0.0235 (7)	0.0194 (5)	−0.0021 (5)	0.0105 (4)	0.0006 (4)
F33	0.0121 (4)	0.0479 (8)	0.0225 (5)	0.0015 (4)	0.0013 (4)	−0.0048 (5)
F34	0.0149 (4)	0.0332 (7)	0.0309 (6)	0.0047 (4)	0.0013 (4)	−0.0093 (5)
F35	0.0412 (7)	0.0313 (8)	0.0510 (8)	−0.0121 (6)	0.0028 (6)	0.0176 (6)
F36	0.0175 (4)	0.0377 (7)	0.0140 (5)	−0.0014 (4)	0.0047 (4)	−0.0057 (4)

Table 3. *Selected geometric parameters (Å, °)*

N1—N2	1.3811 (17)	C14—H14A	0.98
N1—C9	1.3850 (19)	C14—H14B	0.98
N1—C11	1.3982 (19)	C14—H14C	0.98
N2—N3	1.3137 (18)	N15—C16	1.474 (2)
N3—O10	1.2697 (16)	N15—C20	1.4809 (19)
N3—C4	1.407 (2)	C16—C17	1.519 (2)
C4—C9	1.385 (2)	C16—H16A	0.99
C4—C5	1.390 (2)	C16—H16B	0.99
C5—C6	1.380 (2)	C17—O18	1.419 (2)
C5—H5	0.95	C17—H17A	0.99
C6—C7	1.411 (2)	C17—H17B	0.99
C6—H6	0.95	O18—C19	1.428 (2)
C7—C8	1.377 (2)	C19—C20	1.517 (2)
C7—H7	0.95	C19—H19A	0.99
C8—C9	1.395 (2)	C19—H19B	0.99
C8—H8	0.95	C20—H20A	0.99
C11—N15	1.319 (2)	C20—H20B	0.99
C11—N12	1.320 (2)	P30—F35	1.5919 (13)
N12—C14	1.4677 (19)	P30—F33	1.5978 (10)
N12—C13	1.469 (2)	P30—F32	1.5990 (12)
C13—H13A	0.98	P30—F34	1.6028 (10)
C13—H13B	0.98	P30—F31	1.6049 (12)
C13—H13C	0.98	P30—F36	1.6075 (11)
N2—N1—C9	111.76 (12)	C8—C7—C6	122.46 (15)
N2—N1—C11	120.11 (12)	C8—C7—H7	118.8
C9—N1—C11	127.87 (13)	C6—C7—H7	118.8
N3—N2—N1	104.74 (12)	C7—C8—C9	116.06 (15)
O10—N3—N2	122.42 (13)	C7—C8—H8	122
O10—N3—C4	125.19 (13)	C9—C8—H8	122
N2—N3—C4	112.38 (12)	N1—C9—C4	105.22 (13)
C9—C4—C5	123.06 (15)	N1—C9—C8	133.52 (15)
C9—C4—N3	105.90 (14)	C4—C9—C8	121.26 (15)
C5—C4—N3	131.03 (14)	N15—C11—N12	125.16 (14)
C6—C5—C4	115.81 (15)	N15—C11—N1	118.33 (14)
C6—C5—H5	122.1	N12—C11—N1	116.51 (14)
C4—C5—H5	122.1	C11—N12—C14	121.82 (14)
C5—C6—C7	121.34 (15)	C11—N12—C13	123.31 (13)
C5—C6—H6	119.3	C14—N12—C13	114.66 (13)
C7—C6—H6	119.3	N12—C13—H13A	109.5

N12—C13—H13B	109.5	O18—C19—C20	111.22 (13)
H13A—C13—H13B	109.5	O18—C19—H19A	109.4
N12—C13—H13C	109.5	C20—C19—H19A	109.4
H13A—C13—H13C	109.5	O18—C19—H19B	109.4
H13B—C13—H13C	109.5	C20—C19—H19B	109.4
N12—C14—H14A	109.5	H19A—C19—H19B	108
N12—C14—H14B	109.5	N15—C20—C19	108.14 (13)
H14A—C14—H14B	109.5	N15—C20—H20A	110.1
N12—C14—H14C	109.5	C19—C20—H20A	110.1
H14A—C14—H14C	109.5	N15—C20—H20B	110.1
H14B—C14—H14C	109.5	C19—C20—H20B	110.1
C11—N15—C16	123.92 (13)	H20A—C20—H20B	108.4
C11—N15—C20	122.50 (14)	F35—P30—F33	89.84 (7)
C16—N15—C20	113.58 (13)	F35—P30—F32	179.72 (7)
N15—C16—C17	109.52 (13)	F33—P30—F32	89.88 (6)
N15—C16—H16A	109.8	F35—P30—F34	90.42 (7)
C17—C16—H16A	109.8	F33—P30—F34	179.72 (7)
N15—C16—H16B	109.8	F32—P30—F34	89.86 (6)
C17—C16—H16B	109.8	F35—P30—F31	91.02 (7)
H16A—C16—H16B	108.2	F33—P30—F31	90.09 (6)
O18—C17—C16	111.27 (13)	F32—P30—F31	88.94 (6)
O18—C17—H17A	109.4	F34—P30—F31	90.01 (6)
C16—C17—H17A	109.4	F35—P30—F36	90.14 (7)
O18—C17—H17B	109.4	F33—P30—F36	90.22 (6)
C16—C17—H17B	109.4	F32—P30—F36	89.90 (6)
H17A—C17—H17B	108	F34—P30—F36	89.67 (6)
C17—O18—C19	109.62 (13)	F31—P30—F36	178.80 (7)

C9—N1—N2—N3	−0.01 (17)	C7—C8—C9—C4	−0.7 (2)
C11—N1—N2—N3	−174.54 (14)	N2—N1—C11—N15	−47.6 (2)
N1—N2—N3—O10	178.63 (14)	C9—N1—C11—N15	138.86 (16)
N1—N2—N3—C4	−0.12 (17)	N2—N1—C11—N12	133.26 (15)
O10—N3—C4—C9	−178.51 (15)	C9—N1—C11—N12	−40.3 (2)
N2—N3—C4—C9	0.20 (19)	N15—C11—N12—C14	−27.0 (2)
O10—N3—C4—C5	0.7 (3)	N1—C11—N12—C14	152.08 (14)
N2—N3—C4—C5	179.42 (17)	N15—C11—N12—C13	147.49 (15)
C9—C4—C5—C6	−0.4 (3)	N1—C11—N12—C13	−33.4 (2)
N3—C4—C5—C6	−179.52 (17)	N12—C11—N15—C16	151.47 (15)
C4—C5—C6—C7	0.1 (3)	N1—C11—N15—C16	−27.6 (2)
C5—C6—C7—C8	0.0 (3)	N12—C11—N15—C20	−27.4 (2)
C6—C7—C8—C9	0.3 (3)	N1—C11—N15—C20	153.55 (13)
N2—N1—C9—C4	0.13 (18)	C11—N15—C16—C17	129.33 (15)
C11—N1—C9—C4	174.13 (15)	C20—N15—C16—C17	−51.73 (17)
N2—N1—C9—C8	179.85 (18)	N15—C16—C17—O18	55.26 (18)
C11—N1—C9—C8	−6.1 (3)	C16—C17—O18—C19	−61.57 (16)
C5—C4—C9—N1	−179.49 (15)	C17—O18—C19—C20	63.31 (16)
N3—C4—C9—N1	−0.19 (18)	C11—N15—C20—C19	−128.42 (15)
C5—C4—C9—C8	0.7 (3)	C16—N15—C20—C19	52.62 (16)
N3—C4—C9—C8	−179.95 (15)	O18—C19—C20—N15	−57.59 (16)
C7—C8—C9—N1	179.65 (18)		