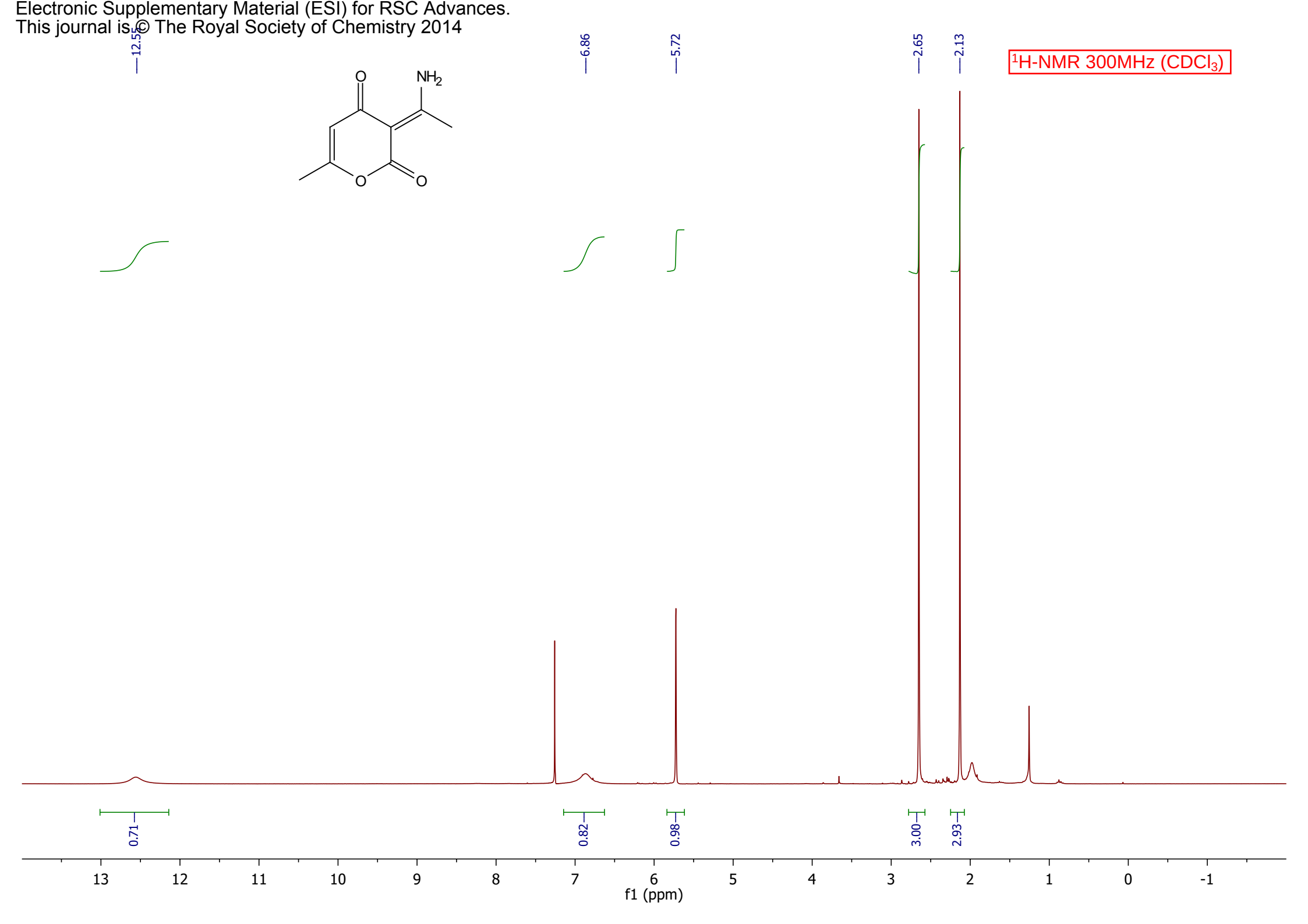
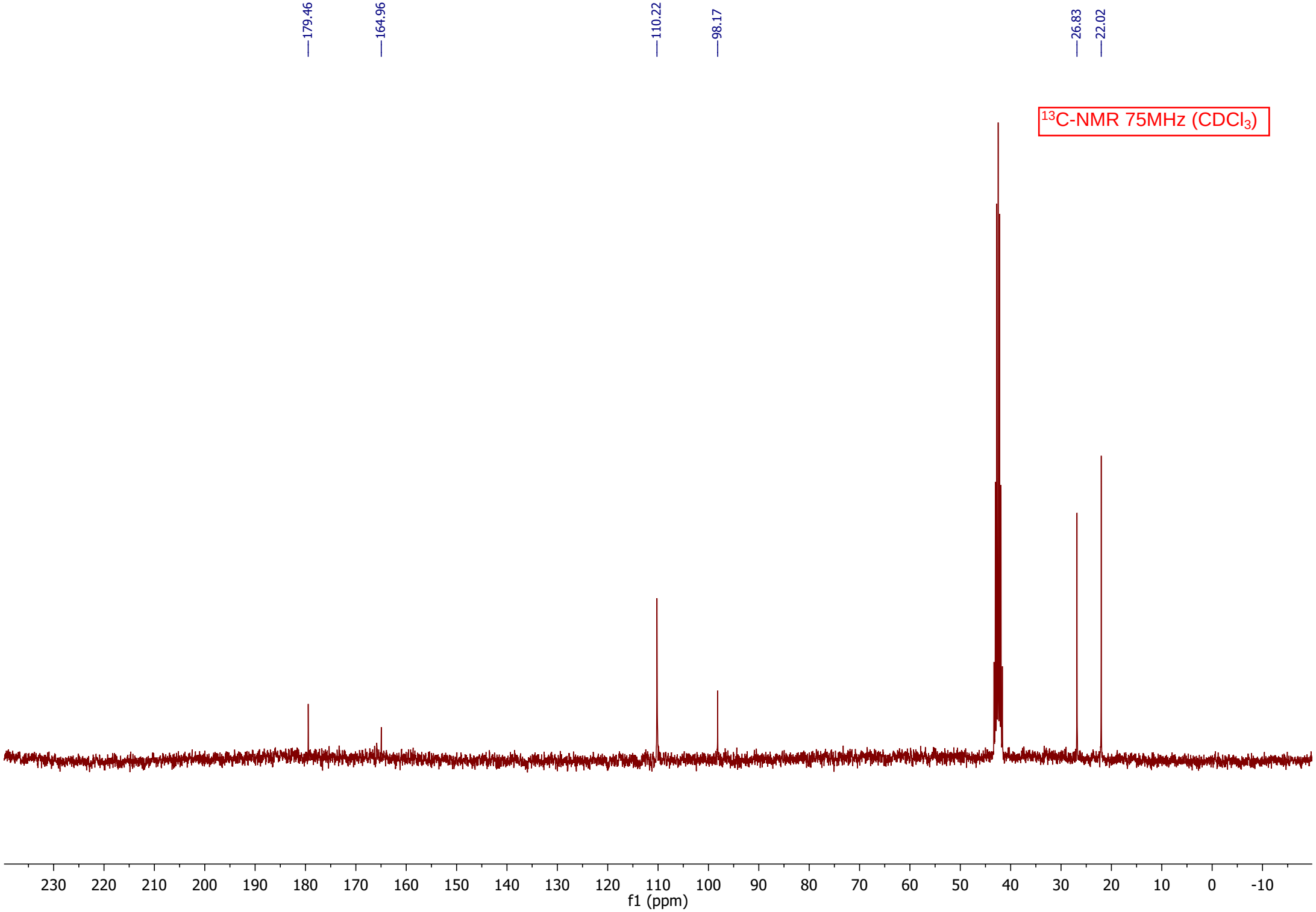
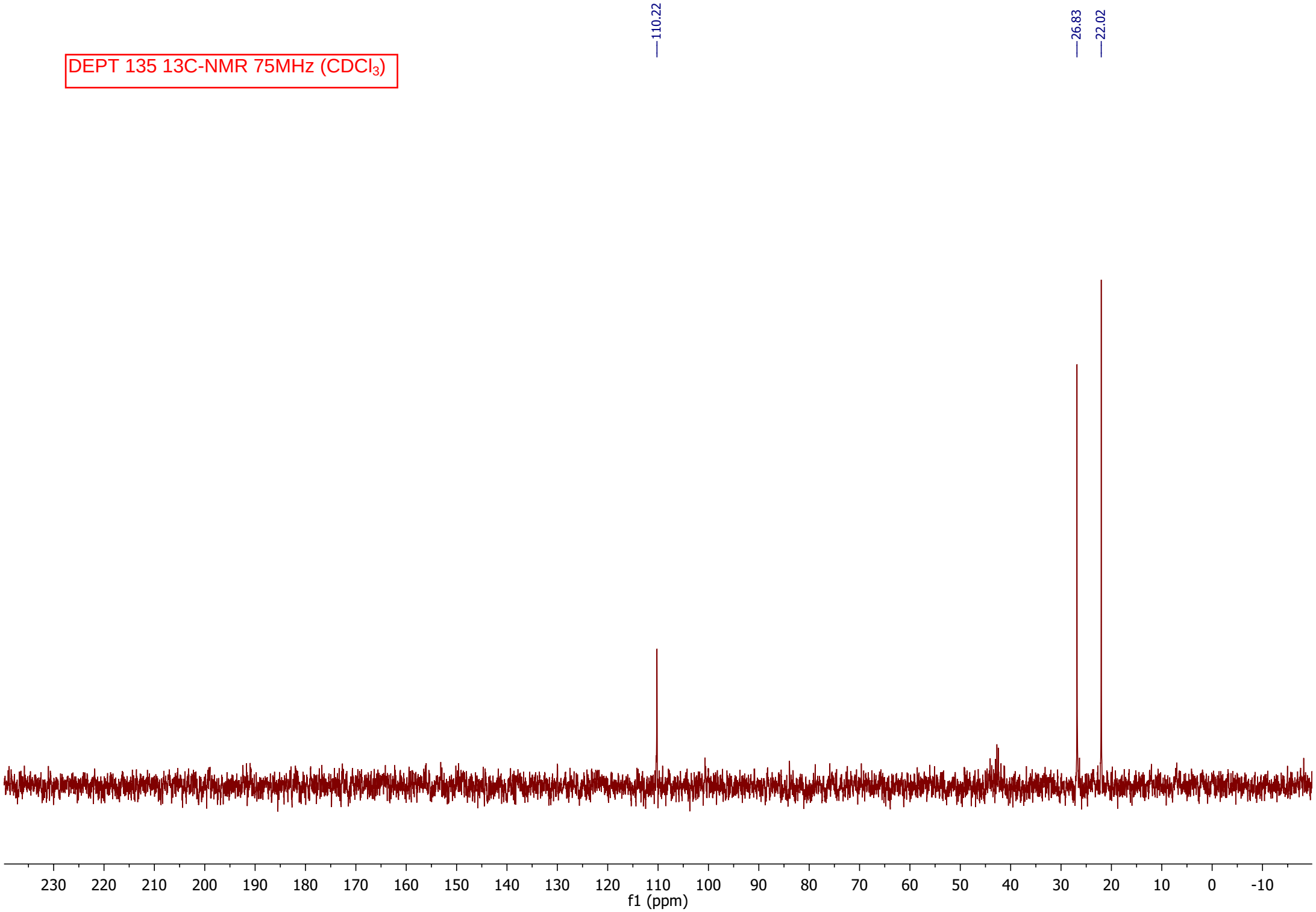
Electronic Supplementary Material (ESI) for RSC Advances.




179.46
164.96
110.22
98.17
26.83
22.02
^{13}C-NMR 75MHz ($CDCl_3$)
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

DEPT 135 13C-NMR 75MHz ($CDCl_3$)



Abstract

Experimental

Crystal data

$C_8H_9NO_3$
M_r = 167.16
Triclinic, $P\overline{1}$
a = 6.650 (3) Å
b = 8.898 (4) Å
c = 13.970 (6) Å
α = 92.24 (2)°
β = 98.98 (2)°
γ = 111.168 (19)°
V = 757.3 (6) Å^3
Z = 4
Mo $K\alpha$ radiation, λ = 0.7107 Å
μ = 0.11 mm^{-1}
T = 100 K
0.53 × 0.03 × 0.03 mm

Data collection

BRUKER APPEX-II CCD diffractometer
Absorption correction: Multi-scan BRUKER TWINABS
T_{min} = 0.721, T_{max} = 0.982
11046 measured reflections
3506 independent reflections
1756 reflections with $I > 2\sigma(I)$
R_{int} = 0.111

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.189
S = 1.00
3506 reflections
234 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N31—H31*B*···O40	0.89 (2)	1.81 (4)	2.582 (6)	143 (5)
N31—H31*A*···O40*B*^{i}	0.90 (5)	2.06 (5)	2.941 (6)	168 (6)
N31*B*—H31*C*···O40*B*	0.90 (2)	1.78 (3)	2.589 (6)	148 (5)
N31*B*—H31*D*···O20^{ii}	0.91 (2)	2.08 (4)	2.854 (6)	143 (5)

Symmetry codes: (i) $x-1, y-1, z$; (ii) $-x+1, -y+2, -z$.

Data collection: APPEX2 (BRUKER AXS, 2005); cell refinement: APPEX2 (BRUKER AXS, 2005); data reduction: APPEX2 (BRUKER AXS, 2005); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1986); program(s) used to refine structure: *SHELXL2012* (Sheldrick, 2012); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

References

NOT FOUND

supplementary materials

Experimental

(twin5)

Crystal data

$C_8H_9NO_3$
$M_r = 167.16$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.650$ (3) Å
$b = 8.898$ (4) Å
$c = 13.970$ (6) Å
$\alpha = 92.24$ (2)°
$\beta = 98.98$ (2)°
$\gamma = 111.168$ (19)°
$V = 757.3$ (6) Å^3
$Z = 4$
$F(000) = 352$
$D_x = 1.466$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å
Cell parameters from 928 reflections
$\theta = 0.3$–$18.7°$
$\mu = 0.11$ mm^{-1}
$T = 100$ K
Prism, Colourless
0.53 × 0.03 × 0.03 mm

Data collection

BRUKER APPEX-II CCD diffractometer
Radiation source: fine-focus sealed tube
ω and phi scans
Absorption correction: Multi-scan BRUKER TWINABS
$T_{min} = 0.721$, $T_{max} = 0.982$
11046 measured reflections
3506 independent reflections
1756 reflections with $I > 2\sigma(I)$
$R_{int} = 0.111$
$\theta_{max} = 25.4°$, $\theta_{min} = 1.5°$
$h = -8 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = 0 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: Full
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.189$
$S = 1.00$
3506 reflections
234 parameters
3 restraints
Hydrogen site location: Mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0842P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. The crystal was found to be a non-merohedral twin with the twin components related by a 180.0 degree rotation about the real [1 0 0] axis.TWINABS was used to apply post-collection corrections.Both twin components were used in corrections and overlaps in addition to the two components were included in the reflection file. An additional parameter was included on the refinement to properly calculate the twin ratio 0.574 (3)/0.426 (3).

Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.3141 (8)	0.9967 (4)	0.1984 (2)	0.0225 (9)
C2	0.2254 (12)	0.8295 (6)	0.1790 (4)	0.0175 (13)
C3	0.2028 (11)	0.7325 (6)	0.2587 (4)	0.0175 (12)
C4	0.2680 (10)	0.8094 (6)	0.3578 (4)	0.0142 (12)
C5	0.3651 (10)	0.9848 (6)	0.3691 (4)	0.0189 (13)
H5	0.4178	1.0403	0.4328	0.023*
C6	0.3826 (11)	1.0706 (6)	0.2925 (4)	0.0172 (13)
O20	0.1736 (8)	0.7847 (4)	0.0914 (3)	0.0251 (9)
C30	0.1133 (11)	0.5610 (6)	0.2398 (4)	0.0192 (13)
N31	0.0851 (11)	0.4716 (5)	0.3124 (3)	0.0226 (12)
H31A	0.041 (11)	0.363 (6)	0.303 (4)	0.027*
H31B	0.113 (11)	0.530 (5)	0.370 (2)	0.027*
C32	0.0470 (12)	0.4739 (6)	0.1401 (4)	0.0348 (17)
H32A	−0.0006	0.357	0.1445	0.052*
H32B	0.172	0.5089	0.1061	0.052*
H32C	−0.0741	0.4985	0.1039	0.052*
O40	0.2548 (7)	0.7329 (4)	0.4314 (3)	0.0221 (9)
C60	0.4778 (11)	1.2482 (6)	0.2949 (4)	0.0279 (15)
H60A	0.5363	1.2963	0.3625	0.042*
H60B	0.364	1.2876	0.2666	0.042*
H60C	0.5964	1.2789	0.2571	0.042*
O1B	0.6815 (7)	0.6434 (4)	0.3058 (2)	0.0222 (9)
C2B	0.6652 (13)	0.6798 (7)	0.2083 (4)	0.0222 (13)
C3B	0.7364 (11)	0.8463 (6)	0.1901 (4)	0.0149 (12)
C4B	0.8176 (12)	0.9701 (6)	0.2700 (4)	0.0162 (12)
C5B	0.8181 (10)	0.9179 (6)	0.3666 (4)	0.0160 (13)
H5B	0.8652	0.9972	0.4212	0.019*
C6B	0.7548 (11)	0.7620 (6)	0.3814 (4)	0.0161 (13)
O20B	0.5945 (9)	0.5595 (4)	0.1500 (3)	0.0351 (11)
C30B	0.7229 (12)	0.8834 (6)	0.0912 (4)	0.0184 (12)
N31B	0.7839 (12)	1.0359 (6)	0.0741 (3)	0.0281 (13)
H31C	0.845 (12)	1.100 (5)	0.131 (2)	0.034*
H31D	0.807 (10)	1.052 (6)	0.012 (2)	0.034*
C32B	0.6384 (12)	0.7616 (6)	0.0039 (4)	0.0292 (15)
H32D	0.6327	0.817	−0.0551	0.044*
H32E	0.4908	0.6859	0.0076	0.044*
H32F	0.7361	0.7018	0.0019	0.044*
O40B	0.8877 (8)	1.1189 (4)	0.2603 (2)	0.0217 (9)
C60B	0.7509 (11)	0.6911 (7)	0.4758 (4)	0.0277 (15)
H60D	0.804	0.7783	0.5294	0.042*
H60E	0.8457	0.6282	0.4817	0.042*
H60F	0.6004	0.6201	0.4788	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.026 (3)	0.018 (2)	0.020 (2)	0.005 (3)	−0.002 (3)	0.0059 (17)
C2	0.013 (4)	0.020 (3)	0.019 (3)	0.006 (4)	0.000 (4)	0.003 (3)
C3	0.015 (4)	0.018 (3)	0.018 (3)	0.003 (4)	0.006 (4)	0.003 (2)
C4	0.008 (4)	0.018 (3)	0.017 (3)	0.004 (4)	0.005 (4)	0.008 (3)
C5	0.015 (4)	0.022 (3)	0.018 (3)	0.007 (4)	0.001 (4)	0.001 (3)

C6	0.012 (4)	0.020 (3)	0.017 (3)	0.006 (4)	−0.005 (4)	−0.002 (3)
O20	0.036 (3)	0.021 (2)	0.018 (2)	0.013 (3)	0.002 (3)	0.0025 (18)
C30	0.016 (4)	0.019 (3)	0.023 (3)	0.007 (4)	0.003 (4)	0.002 (3)
N31	0.033 (4)	0.014 (3)	0.023 (3)	0.010 (4)	0.006 (4)	0.006 (2)
C32	0.057 (5)	0.014 (3)	0.023 (3)	0.001 (5)	0.008 (5)	0.000 (3)
O40	0.029 (3)	0.015 (2)	0.023 (2)	0.009 (3)	0.008 (3)	0.0059 (17)
C60	0.028 (4)	0.019 (3)	0.036 (4)	0.003 (4)	0.018 (4)	0.005 (3)
O1B	0.024 (3)	0.019 (2)	0.021 (2)	0.005 (3)	0.004 (3)	0.0039 (17)
C2B	0.030 (5)	0.023 (3)	0.016 (3)	0.014 (5)	0.001 (5)	0.005 (3)
C3B	0.015 (4)	0.015 (3)	0.019 (3)	0.010 (4)	0.006 (4)	0.005 (2)
C4B	0.013 (4)	0.016 (3)	0.019 (3)	0.005 (4)	0.006 (4)	0.007 (2)
C5B	0.016 (4)	0.010 (3)	0.017 (3)	0.000 (4)	0.000 (4)	0.000 (2)
C6B	0.012 (4)	0.018 (3)	0.016 (3)	0.006 (4)	−0.005 (4)	0.000 (3)
O20B	0.056 (3)	0.015 (2)	0.024 (2)	0.006 (3)	−0.003 (4)	0.0002 (19)
C30B	0.019 (4)	0.013 (3)	0.024 (3)	0.006 (4)	0.006 (5)	0.007 (2)
N31B	0.036 (4)	0.025 (3)	0.015 (3)	0.000 (4)	0.005 (4)	0.006 (2)
C32B	0.045 (5)	0.024 (3)	0.018 (3)	0.010 (5)	0.013 (5)	−0.003 (3)
O40B	0.027 (3)	0.015 (2)	0.023 (2)	0.006 (3)	0.009 (3)	0.0066 (17)
C60B	0.028 (4)	0.028 (3)	0.026 (4)	0.007 (4)	0.010 (4)	0.007 (3)

Geometric parameters (Å, º)

O1—C6	1.377 (6)	O1B—C6B	1.360 (6)
O1—C2	1.385 (6)	O1B—C2B	1.409 (6)
C2—O20	1.227 (6)	C2B—O20B	1.216 (6)
C2—C3	1.429 (7)	C2B—C3B	1.430 (7)
C3—C30	1.420 (7)	C3B—C4B	1.426 (7)
C3—C4	1.448 (6)	C3B—C30B	1.430 (7)
C4—O40	1.253 (5)	C4B—O40B	1.256 (5)
C4—C5	1.449 (7)	C4B—C5B	1.445 (7)
C5—C6	1.334 (7)	C5B—C6B	1.331 (7)
C5—H5	0.95	C5B—H5B	0.95
C6—C60	1.472 (6)	C6B—C60B	1.484 (7)
C30—N31	1.308 (7)	C30B—N31B	1.312 (6)
C30—C32	1.488 (7)	C30B—C32B	1.490 (7)
N31—H31A	0.90 (5)	N31B—H31C	0.90 (2)
N31—H31B	0.89 (2)	N31B—H31D	0.91 (2)
C32—H32A	0.98	C32B—H32D	0.98
C32—H32B	0.98	C32B—H32E	0.98
C32—H32C	0.98	C32B—H32F	0.98
C60—H60A	0.98	C60B—H60D	0.98
C60—H60B	0.98	C60B—H60E	0.98
C60—H60C	0.98	C60B—H60F	0.98
C6—O1—C2	121.4 (4)	C6B—O1B—C2B	121.5 (4)
O20—C2—O1	112.7 (4)	O20B—C2B—O1B	112.8 (5)
O20—C2—C3	128.3 (5)	O20B—C2B—C3B	128.8 (5)
O1—C2—C3	118.9 (4)	O1B—C2B—C3B	118.3 (5)
C30—C3—C2	119.5 (5)	C4B—C3B—C30B	121.9 (5)
C30—C3—C4	120.5 (5)	C4B—C3B—C2B	119.8 (5)
C2—C3—C4	119.9 (4)	C30B—C3B—C2B	118.3 (5)
O40—C4—C3	123.8 (5)	O40B—C4B—C3B	123.7 (5)
O40—C4—C5	120.0 (5)	O40B—C4B—C5B	119.4 (5)
C3—C4—C5	116.2 (4)	C3B—C4B—C5B	116.9 (5)

C6—C5—C4	121.8 (5)	C6B—C5B—C4B	122.0 (5)
C6—C5—H5	119.1	C6B—C5B—H5B	119
C4—C5—H5	119.1	C4B—C5B—H5B	119
C5—C6—O1	121.7 (5)	C5B—C6B—O1B	121.4 (5)
C5—C6—C60	126.7 (5)	C5B—C6B—C60B	127.9 (5)
O1—C6—C60	111.6 (4)	O1B—C6B—C60B	110.7 (4)
N31—C30—C3	119.8 (5)	N31B—C30B—C3B	118.6 (5)
N31—C30—C32	116.7 (5)	N31B—C30B—C32B	116.2 (5)
C3—C30—C32	123.4 (5)	C3B—C30B—C32B	125.2 (5)
C30—N31—H31A	122 (3)	C30B—N31B—H31C	110 (3)
C30—N31—H31B	113 (3)	C30B—N31B—H31D	113 (3)
H31A—N31—H31B	125 (5)	H31C—N31B—H31D	131 (5)
C30—C32—H32A	109.5	C30B—C32B—H32D	109.5
C30—C32—H32B	109.5	C30B—C32B—H32E	109.5
H32A—C32—H32B	109.5	H32D—C32B—H32E	109.5
C30—C32—H32C	109.5	C30B—C32B—H32F	109.5
H32A—C32—H32C	109.5	H32D—C32B—H32F	109.5
H32B—C32—H32C	109.5	H32E—C32B—H32F	109.5
C6—C60—H60A	109.5	C6B—C60B—H60D	109.5
C6—C60—H60B	109.5	C6B—C60B—H60E	109.5
H60A—C60—H60B	109.5	H60D—C60B—H60E	109.5
C6—C60—H60C	109.5	C6B—C60B—H60F	109.5
H60A—C60—H60C	109.5	H60D—C60B—H60F	109.5
H60B—C60—H60C	109.5	H60E—C60B—H60F	109.5
C6—O1—C2—O20	−179.9 (7)	C6B—O1B—C2B—O20B	−179.2 (7)
C6—O1—C2—C3	0.7 (10)	C6B—O1B—C2B—C3B	2.5 (10)
O20—C2—C3—C30	1.0 (13)	O20B—C2B—C3B—C4B	−179.1 (9)
O1—C2—C3—C30	−179.6 (7)	O1B—C2B—C3B—C4B	−1.1 (11)
O20—C2—C3—C4	−178.0 (8)	O20B—C2B—C3B—C30B	1.1 (14)
O1—C2—C3—C4	1.3 (11)	O1B—C2B—C3B—C30B	179.1 (7)
C30—C3—C4—O40	1.5 (11)	C30B—C3B—C4B—O40B	−1.9 (12)
C2—C3—C4—O40	−179.5 (7)	C2B—C3B—C4B—O40B	178.3 (8)
C30—C3—C4—C5	177.7 (7)	C30B—C3B—C4B—C5B	178.4 (7)
C2—C3—C4—C5	−3.2 (10)	C2B—C3B—C4B—C5B	−1.4 (11)
O40—C4—C5—C6	179.8 (7)	O40B—C4B—C5B—C6B	−177.0 (7)
C3—C4—C5—C6	3.4 (10)	C3B—C4B—C5B—C6B	2.7 (10)
C4—C5—C6—O1	−1.6 (12)	C4B—C5B—C6B—O1B	−1.4 (11)
C4—C5—C6—C60	−180.0 (7)	C4B—C5B—C6B—C60B	178.7 (7)
C2—O1—C6—C5	−0.6 (11)	C2B—O1B—C6B—C5B	−1.3 (10)
C2—O1—C6—C60	178.0 (6)	C2B—O1B—C6B—C60B	178.6 (6)
C2—C3—C30—N31	−177.6 (7)	C4B—C3B—C30B—N31B	−1.2 (11)
C4—C3—C30—N31	1.4 (11)	C2B—C3B—C30B—N31B	178.7 (8)
C2—C3—C30—C32	2.2 (12)	C4B—C3B—C30B—C32B	−179.4 (8)
C4—C3—C30—C32	−178.7 (7)	C2B—C3B—C30B—C32B	0.4 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N31—H31*B*···O40	0.89 (2)	1.81 (4)	2.582 (6)	143 (5)
N31—H31*A*···O40*B*^{i}	0.90 (5)	2.06 (5)	2.941 (6)	168 (6)

N31*B*—H31*C*···O40*B*	0.90 (2)	1.78 (3)	2.589 (6)	148 (5)
N31*B*—H31*D*···O20^{ii}	0.91 (2)	2.08 (4)	2.854 (6)	143 (5)

Symmetry codes: (i) *x*−1, *y*−1, *z*; (ii) −*x*+1, −*y*+2, −*z*.