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Structural and computational studies on the nature of Au (iii) ...Q interactions

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2018
https://hdl.handle.net/10539/27310

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Appendix A: data for chapter 3.

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1.2 UV VIS data for **1**

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1.4 ^{13}C NMR data for **1**

1.5 HMRS data for **1**

1.6 X-ray crystallography data for **1**

1.7 CIF check data for **1**

2. Characterisation data of ***N*,*N*'-Bis(cyclohexyl)-2,6-pyridinedicarboxamide**, compound **2.**

2.1 FTIR data for **2**

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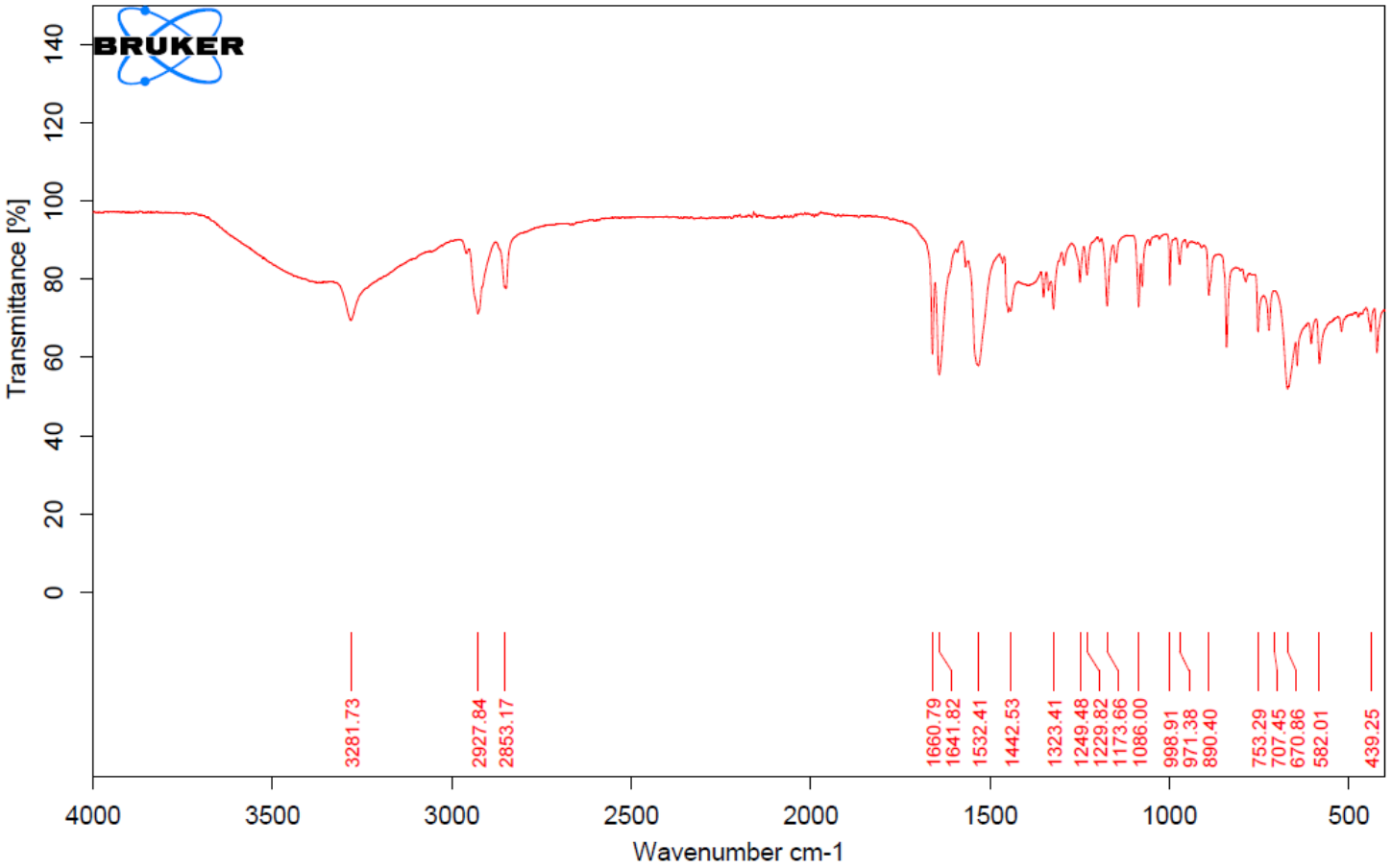
3. DNMR data

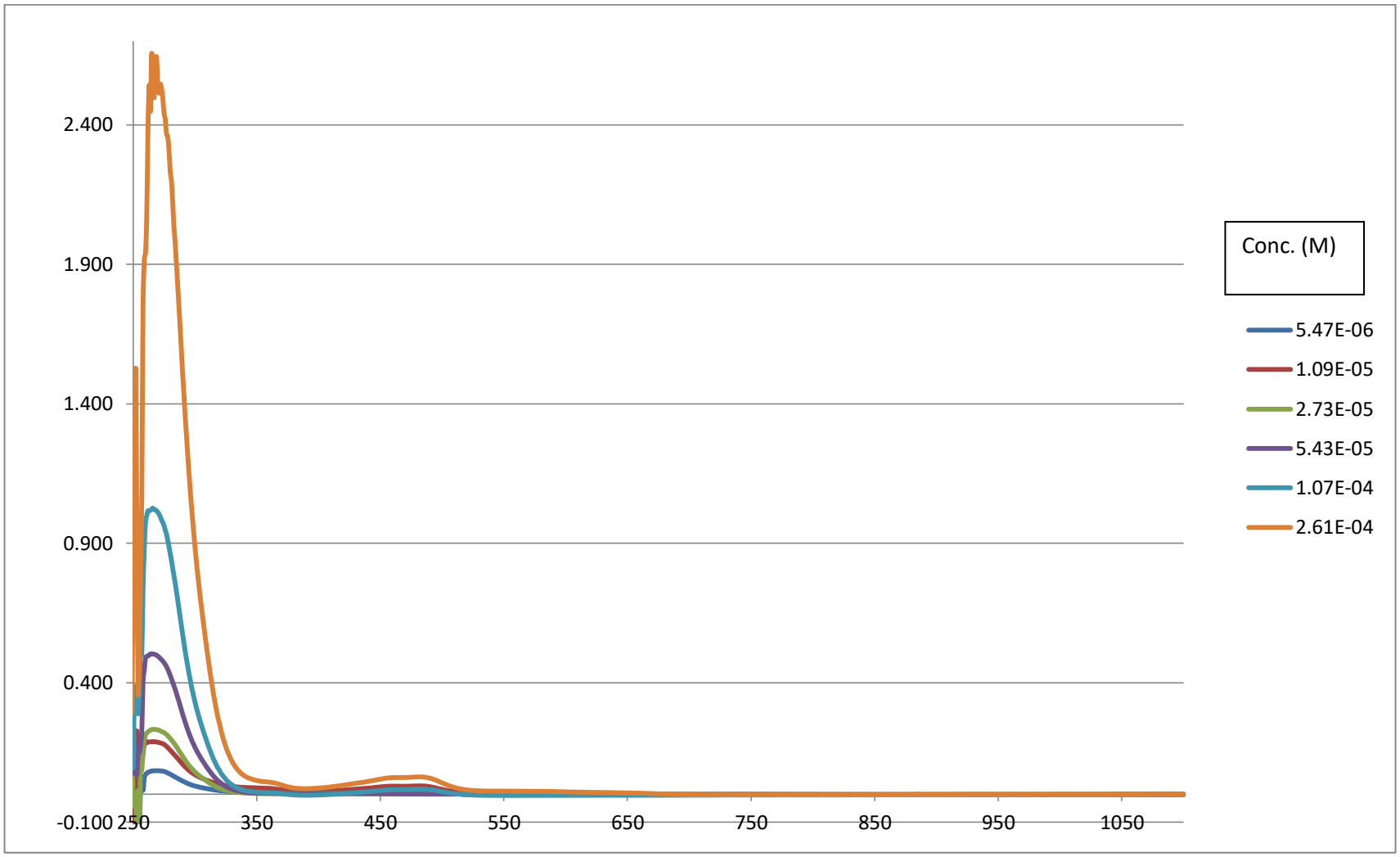
3.1 log(conc) vs shift (ppm) for **2**

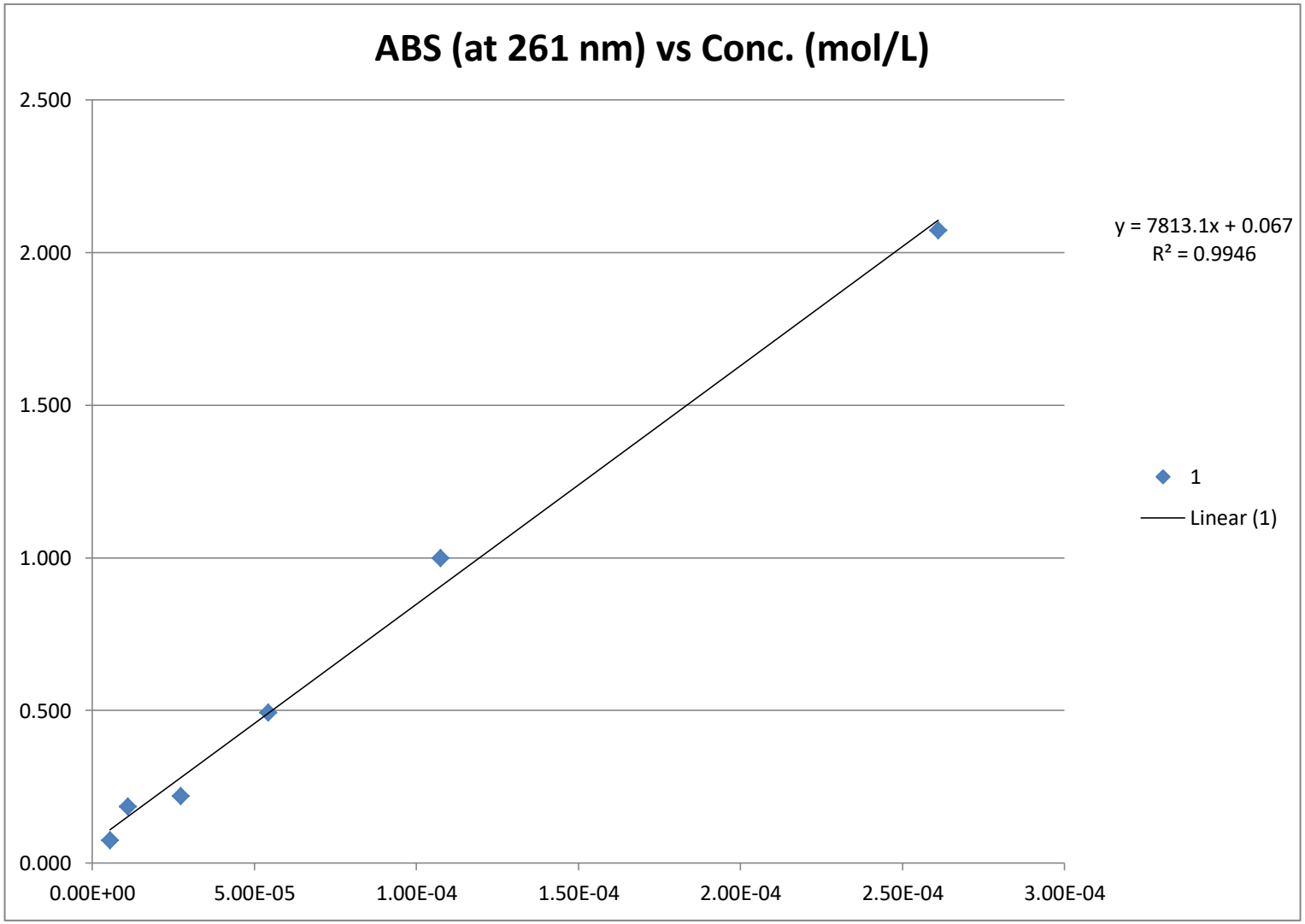
1.1 FTIR data for 1



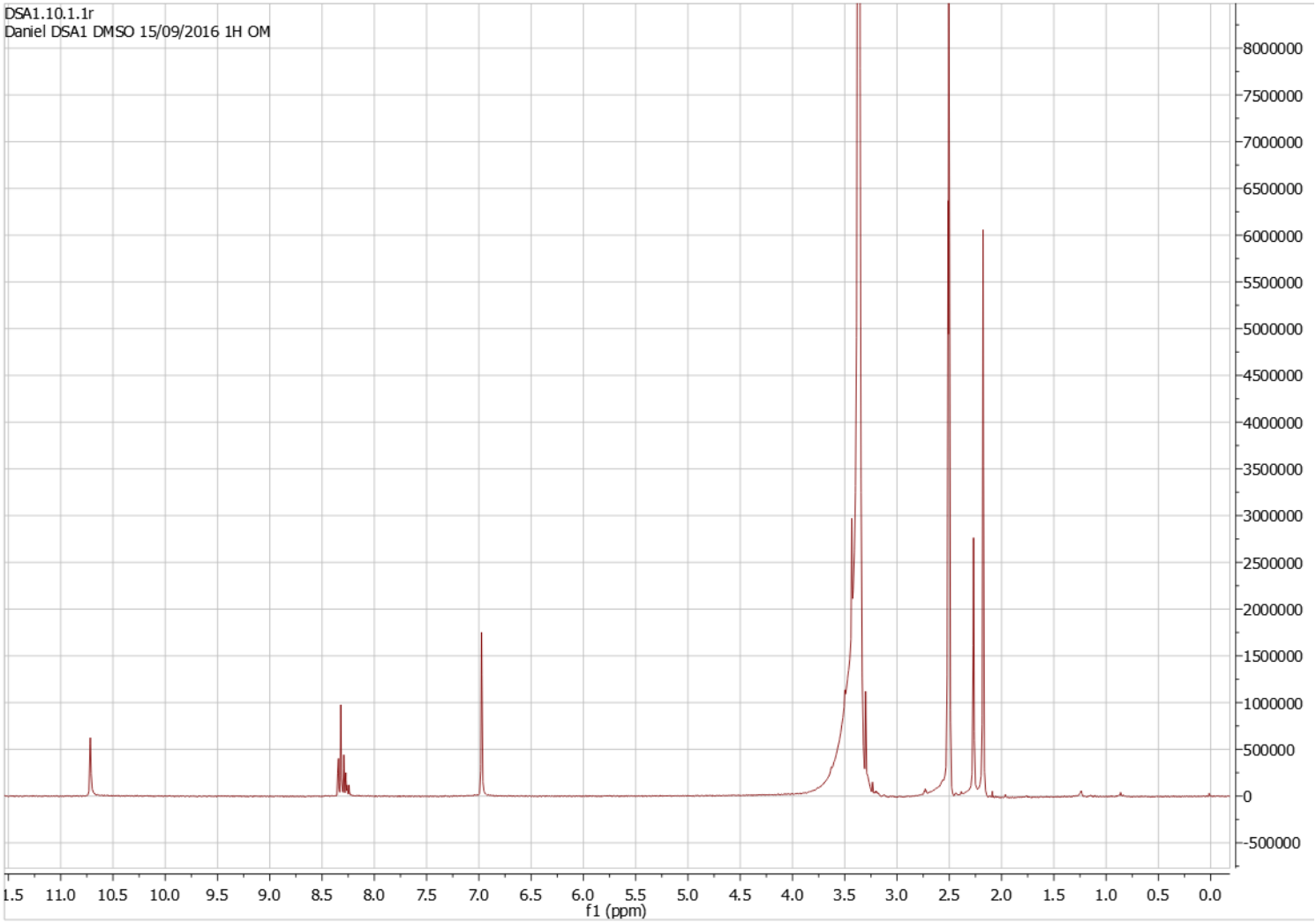
C:\WITS\Measurements\Daniel\2017\sample.0	sample	Instrument type and / or accessory	14/03/2017

1.2 UV VIS data for 1

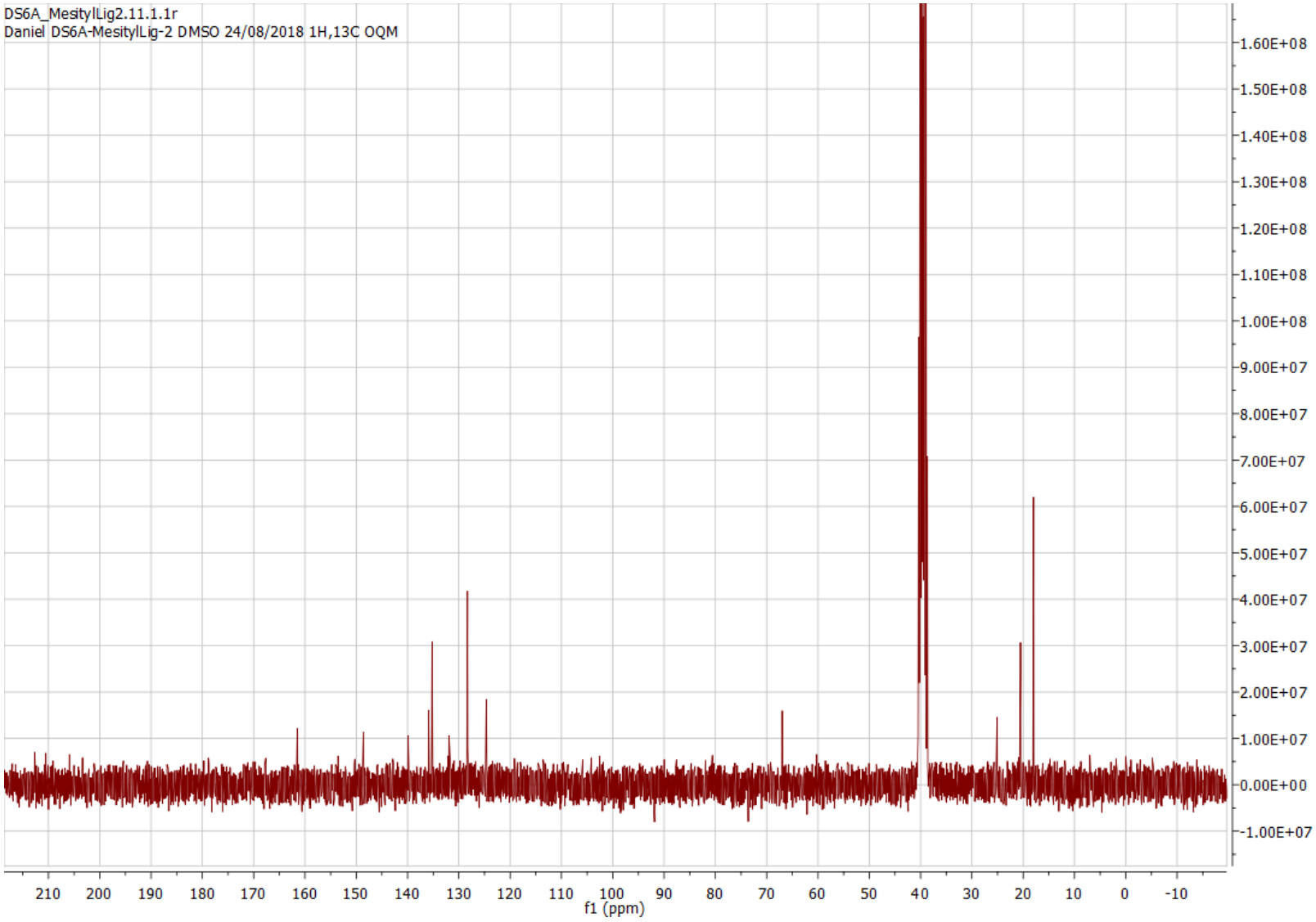



ABS (at 261 nm) vs Conc. (mol/L)
2.500
2.000
1.500
1.000
0.500
0.000
0.00E+00
5.00E-05
1.00E-04
1.50E-04
2.00E-04
2.50E-04
3.00E-04
y = 7813.1x + 0.067
R² = 0.9946
1
Linear (1)

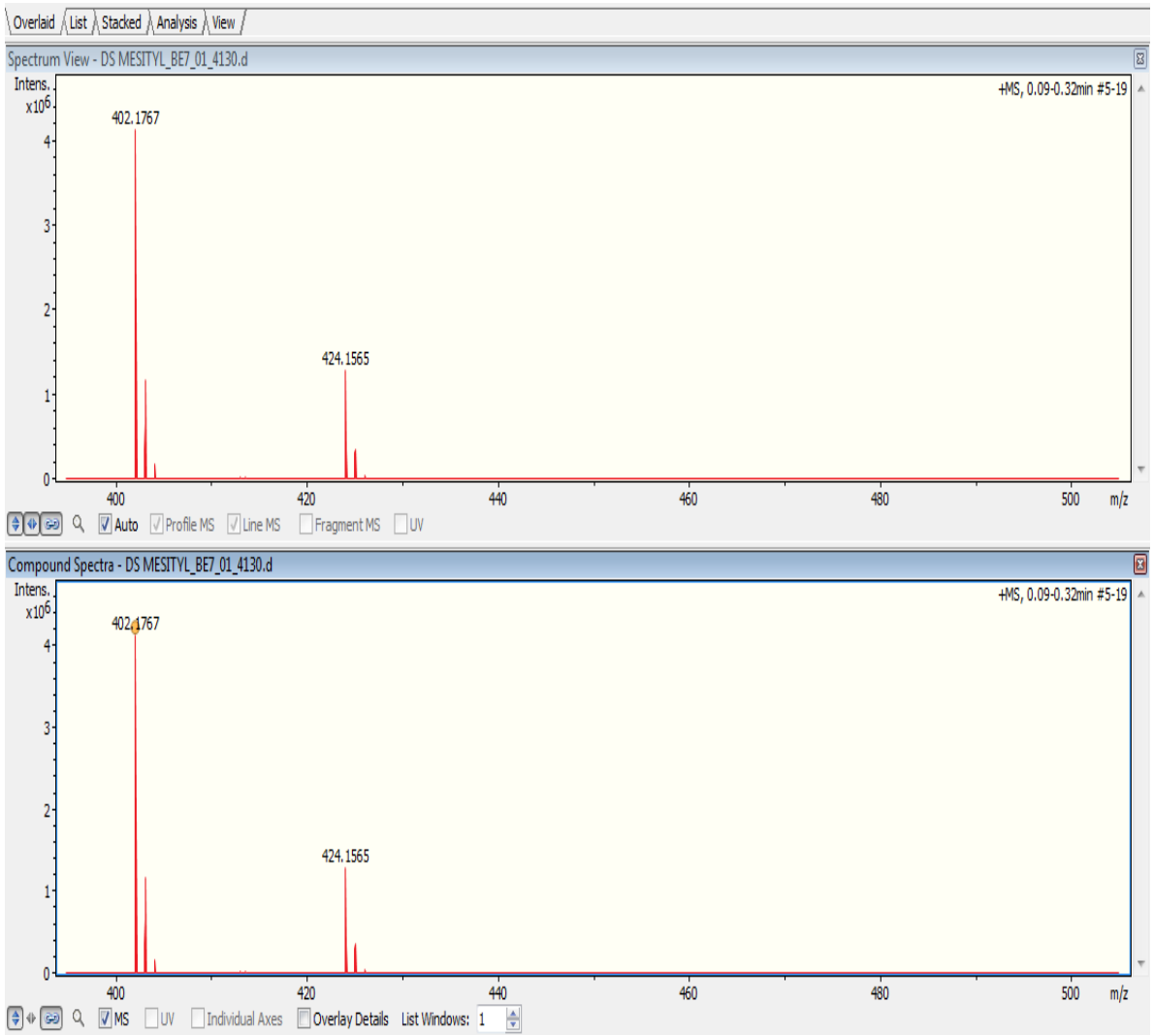
1.3 ^{1}H NMR data for 1



1.4 ^{13}C NMR data for 1

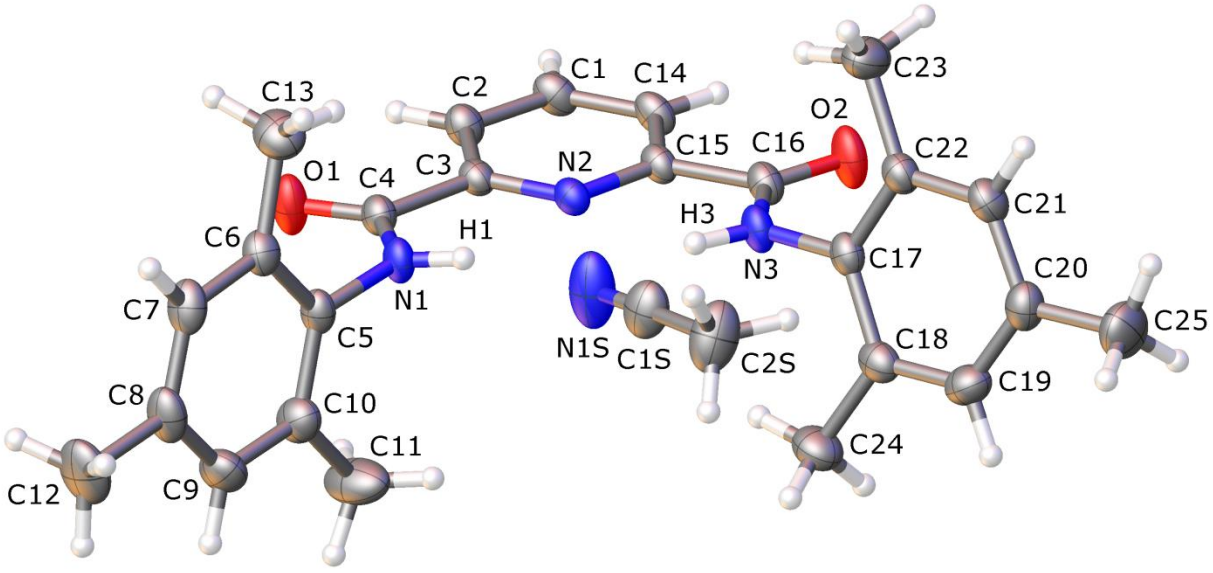


1.5 HMRS data for 1



1.6 X-ray crystallography data for 1

mo_17ov_ds7c_0s



Computing details

Program(s) used to solve structure: Superflip (Palatinus & Chapuis, 2007;Palatinus & van der Lee, 2008; Palatinus *et al.*, 2012); program(s) used to refine structure: olex2.refine (Bourhis *et al.*, 2015); molecular graphics: Olex2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: Olex2 (Dolomanov *et al.*, 2009).

References

Bourhis, L. J., Dolomanov, O. V., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2015). *Acta Cryst.* A**71**, 59–75.

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Palatinus, L. & Chapuis, G. (2007). *J. Appl. Cryst.* **40**, 786–790 Palatinus, L. & van der Lee, A. (2008). J. Appl. Cryst. 41, 975-984; Palatinus, L., Prathapa, S. J. & van Smaalen, S. (2012). J. Appl. Cryst. 45, 575-580.

(mo_17ov_ds7c_0s)

Crystal data

$C_{25}H_{27}N_3O_2 \cdot C_2H_3N$	D_x = 1.146 Mg m^{-3}
M_r = 442.56	Mo $K\alpha$ radiation, λ = 0.71073 Å
Orthorhombic, $Pca2_1$	Cell parameters from 9951 reflections
a = 14.4964 (6) Å	θ = 3.2–27.1°
b = 13.6049 (5) Å	μ = 0.07 mm^{-1}
c = 13.0108 (6) Å	T = 173 K
V = 2566.02 (18) Å^3	Block, clear yellow
Z = 4	0.66 × 0.41 × 0.17 mm
F(000) = 944.3884	

Data collection

Bruker SMART APEX2 area detector diffractometer	5316 reflections with $I \geq 2u(I)$
Radiation source: microfocus sealed X-ray tube, Incoatec Iμs	R_{int} = 0.091
Mirror optics monochromator	θ_{max} = 27.1°, θ_{min} = 3.0°
Detector resolution: 7.9 pixels mm^{-1}	h = -18→18
ω and ϕ scans	k = -17→17
39142 measured reflections	l = -16→16
5640 independent reflections	

Refinement

Refinement on F^2	Primary atom site location: iterative
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$w = 1/[\sigma^2(F_o^2) + (0.0992P)^2 + 0.4227P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.144	$(\Delta/\sigma)_{max} < 0.001$
S = 1.05	$\Delta\rangle_{max}$ = 0.30 e Å^{-3}
5640 reflections	$\Delta\rangle_{min}$ = -0.26 e Å^{-3}
298 parameters	Absolute structure: Flack, H. D. (1983). Acta Cryst. A39, 876-881.
0 restraints	Absolute structure parameter: -0.1 (9)
46 constraints	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for (mo_17ov_ds7c_0s)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.58301 (9)	0.02476 (11)	0.32311 (12)	0.0413 (3)
O2	0.97273 (8)	0.29105 (11)	0.48514 (13)	0.0421 (4)
N2	0.75882 (9)	0.18058 (10)	0.41919 (10)	0.0225 (3)
N1	0.57382 (9)	0.16704 (11)	0.41328 (11)	0.0270 (3)
H1	0.60519 (9)	0.21813 (11)	0.43563 (11)	0.0324 (4)*
N3	0.82470 (9)	0.33758 (11)	0.51630 (12)	0.0285 (3)
H3	0.76645 (9)	0.32217 (11)	0.50613 (12)	0.0342 (4)*
C2	0.77713 (11)	0.03590 (13)	0.31585 (14)	0.0277 (3)
H2	0.74931 (11)	-0.01614 (13)	0.27830 (14)	0.0333 (4)*
C18	0.83235 (11)	0.42551 (14)	0.67945 (14)	0.0307 (4)
C3	0.72327 (11)	0.10515 (12)	0.36712 (12)	0.0235 (3)
C4	0.61961 (11)	0.09468 (12)	0.36532 (13)	0.0255 (3)
C15	0.85100 (11)	0.18925 (12)	0.42156 (13)	0.0241 (3)
C6	0.41703 (12)	0.20686 (12)	0.35732 (14)	0.0282 (3)
C5	0.47610 (11)	0.16485 (12)	0.42973 (14)	0.0260 (3)
C16	0.88957 (11)	0.27727 (13)	0.47813 (14)	0.0272 (3)
N1S	0.61724 (12)	0.37092 (16)	0.5113 (2)	0.0586 (6)
C21	0.89314 (12)	0.59358 (13)	0.57665 (15)	0.0312 (4)
H21	0.91182 (12)	0.65156 (13)	0.54169 (15)	0.0374 (4)*
C17	0.84457 (11)	0.42475 (12)	0.57211 (13)	0.0261 (3)
C19	0.85420 (13)	0.51058 (15)	0.73283 (14)	0.0356 (4)
H19	0.84752 (13)	0.51124 (15)	0.80546 (14)	0.0427 (5)*
C22	0.87390 (12)	0.50895 (13)	0.51974 (14)	0.0282 (3)
C24	0.79648 (15)	0.33559 (17)	0.73337 (17)	0.0423 (5)
H24a	0.78518 (15)	0.28352 (17)	0.68288 (17)	0.0635 (7)*
H24b	0.84214 (15)	0.31298 (17)	0.78364 (17)	0.0635 (7)*
H24c	0.73874 (15)	0.35166 (17)	0.76876 (17)	0.0635 (7)*
C1	0.87232 (12)	0.04477 (14)	0.32098 (14)	0.0300 (4)
H1a	0.91092 (12)	-0.00212 (14)	0.28816 (14)	0.0360 (4)*
C7	0.32294 (13)	0.20726 (14)	0.37877 (16)	0.0344 (4)
H7	0.28206 (13)	0.23643 (14)	0.33054 (16)	0.0413 (5)*
C14	0.91049 (12)	0.12295 (13)	0.37460 (14)	0.0276 (3)
H14	0.97547 (12)	0.13093 (13)	0.37909 (14)	0.0331 (4)*
C9	0.34733 (14)	0.12366 (16)	0.53704 (15)	0.0391 (4)
H9	0.32356 (14)	0.09481 (16)	0.59804 (15)	0.0469 (5)*
C20	0.88558 (12)	0.59507 (14)	0.68363 (15)	0.0339 (4)
C10	0.44230 (13)	0.12178 (15)	0.51918 (14)	0.0349 (4)
C23	0.88600 (17)	0.50944 (17)	0.40452 (15)	0.0420 (5)

H23a	0.86966 (17)	0.44476 (17)	0.37682 (15)	0.0630 (7)*
H23b	0.84579 (17)	0.55957 (17)	0.37424 (15)	0.0630 (7)*
H23c	0.95040 (17)	0.52430 (17)	0.38771 (15)	0.0630 (7)*
C1S	0.61341 (13)	0.44552 (17)	0.54872 (18)	0.0418 (5)
C13	0.45419 (16)	0.24890 (17)	0.25845 (17)	0.0440 (5)
H13a	0.52147 (16)	0.24204 (17)	0.25723 (17)	0.0661 (7)*
H13b	0.42749 (16)	0.21338 (17)	0.20006 (17)	0.0661 (7)*
H13c	0.43775 (16)	0.31863 (17)	0.25378 (17)	0.0661 (7)*
C8	0.28670 (13)	0.16689 (15)	0.46743 (17)	0.0371 (4)
C25	0.91241 (19)	0.68669 (18)	0.74146 (19)	0.0526 (6)
H25a	0.93244 (19)	0.73724 (18)	0.69265 (19)	0.0788 (9)*
H25b	0.85924 (19)	0.71087 (18)	0.78049 (19)	0.0788 (9)*
H25c	0.96301 (19)	0.67155 (18)	0.78883 (19)	0.0788 (9)*
C12	0.18398 (15)	0.1670 (2)	0.4882 (2)	0.0554 (6)
H12a	0.17182 (15)	0.1354 (2)	0.5544 (2)	0.0830 (10)*
H12b	0.16147 (15)	0.2349 (2)	0.4900 (2)	0.0830 (10)*
H12c	0.15213 (15)	0.1309 (2)	0.4335 (2)	0.0830 (10)*
C11	0.50606 (17)	0.0733 (2)	0.59451 (18)	0.0596 (7)
H11a	0.47022 (17)	0.0473 (2)	0.65231 (18)	0.0894 (11)*
H11b	0.53885 (17)	0.0193 (2)	0.56052 (18)	0.0894 (11)*
H11c	0.55077 (17)	0.1216 (2)	0.61992 (18)	0.0894 (11)*
C2S	0.60709 (18)	0.54038 (18)	0.5983 (2)	0.0555 (7)
H2Sa	0.5731 (14)	0.5859 (5)	0.5539 (8)	0.0832 (10)*
H2Sb	0.5746 (14)	0.5334 (3)	0.6640 (9)	0.0832 (10)*
H2Sc	0.66926 (18)	0.5661 (8)	0.6107 (16)	0.0832 (10)*

Atomic displacement parameters (Å^2) for (mo_17ov_ds7c_0s)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0221 (6)	0.0422 (8)	0.0596 (8)	-0.0043 (5)	0.0004 (6)	-0.0209 (7)
O2	0.0182 (6)	0.0406 (8)	0.0676 (9)	-0.0031 (5)	-0.0031 (6)	-0.0118 (7)
N2	0.0163 (6)	0.0242 (6)	0.0272 (6)	0.0012 (5)	0.0006 (5)	0.0030 (5)
N1	0.0157 (6)	0.0300 (7)	0.0353 (7)	-0.0019 (5)	-0.0020 (5)	-0.0049 (6)
N3	0.0161 (6)	0.0284 (7)	0.0410 (8)	-0.0027 (5)	-0.0012 (5)	-0.0026 (6)
C2	0.0224 (8)	0.0264 (7)	0.0344 (8)	0.0008 (6)	0.0019 (7)	-0.0009 (6)
C18	0.0216 (7)	0.0364 (9)	0.0341 (9)	0.0002 (7)	-0.0008 (6)	0.0072 (7)
C3	0.0197 (7)	0.0250 (7)	0.0259 (7)	-0.0008 (6)	0.0000 (6)	0.0040 (6)
C4	0.0188 (7)	0.0283 (7)	0.0294 (8)	-0.0002 (6)	-0.0014 (6)	0.0000 (7)
C15	0.0162 (7)	0.0251 (7)	0.0311 (8)	0.0002 (6)	-0.0001 (6)	0.0049 (6)
C6	0.0239 (8)	0.0240 (7)	0.0367 (8)	0.0011 (6)	-0.0022 (7)	-0.0011 (7)
C5	0.0173 (7)	0.0272 (7)	0.0336 (8)	0.0018 (6)	-0.0004 (6)	-0.0059 (6)
C16	0.0195 (7)	0.0269 (8)	0.0352 (8)	-0.0013 (6)	-0.0006 (6)	0.0027 (7)

N1S	0.0277 (8)	0.0542 (12)	0.0938 (17)	0.0073 (8)	-0.0010 (9)	-0.0335 (12)
C21	0.0275 (8)	0.0272 (8)	0.0388 (9)	-0.0030 (7)	0.0025 (7)	0.0006 (7)
C17	0.0178 (7)	0.0261 (8)	0.0344 (8)	-0.0005 (6)	-0.0019 (6)	-0.0004 (6)
C19	0.0326 (9)	0.0465 (11)	0.0277 (8)	0.0025 (8)	-0.0001 (7)	0.0014 (8)
C22	0.0228 (7)	0.0294 (8)	0.0322 (8)	-0.0026 (6)	0.0002 (6)	0.0019 (7)
C24	0.0409 (11)	0.0438 (11)	0.0423 (10)	-0.0046 (9)	0.0018 (9)	0.0137 (8)
C1	0.0234 (8)	0.0283 (8)	0.0382 (8)	0.0036 (6)	0.0065 (7)	0.0018 (7)
C7	0.0222 (8)	0.0311 (8)	0.0501 (11)	0.0063 (7)	-0.0080 (7)	-0.0053 (8)
C14	0.0171 (7)	0.0270 (8)	0.0387 (9)	0.0025 (6)	0.0044 (6)	0.0035 (7)
C9	0.0288 (9)	0.0513 (11)	0.0372 (10)	-0.0015 (8)	0.0078 (7)	0.0006 (8)
C20	0.0273 (8)	0.0347 (10)	0.0396 (10)	-0.0009 (7)	-0.0027 (7)	-0.0062 (8)
C10	0.0241 (8)	0.0444 (10)	0.0363 (9)	0.0037 (8)	-0.0005 (7)	-0.0001 (8)
C23	0.0512 (12)	0.0410 (11)	0.0337 (10)	-0.0087 (9)	0.0030 (9)	0.0027 (8)
C1S	0.0261 (9)	0.0461 (11)	0.0531 (12)	0.0083 (8)	-0.0044 (8)	-0.0102 (10)
C13	0.0407 (10)	0.0441 (11)	0.0473 (11)	0.0026 (9)	-0.0036 (9)	0.0123 (9)
C8	0.0184 (8)	0.0388 (10)	0.0541 (11)	0.0019 (7)	0.0028 (7)	-0.0110 (9)
C25	0.0598 (14)	0.0476 (12)	0.0503 (12)	-0.0095 (11)	-0.0005 (11)	-0.0165 (10)
C12	0.0219 (9)	0.0644 (15)	0.0798 (17)	0.0014 (9)	0.0110 (10)	-0.0103 (13)
C11	0.0404 (12)	0.095 (2)	0.0435 (12)	0.0108 (13)	-0.0020 (10)	0.0253 (13)
C2S	0.0494 (13)	0.0431 (12)	0.0740 (17)	0.0132 (10)	-0.0186 (12)	-0.0175 (11)

Geometric parameters (Å, º) for (mo_17ov_ds7c_0s)

O1—C4	1.220 (2)	C24—H24b	0.9800
O2—C16	1.223 (2)	C24—H24c	0.9800
N2—C3	1.333 (2)	C1—H1a	0.9500
N2—C15	1.3418 (19)	C1—C14	1.387 (3)
N1—H1	0.8800	C7—H7	0.9500
N1—C4	1.341 (2)	C7—C8	1.381 (3)
N1—C5	1.433 (2)	C14—H14	0.9500
N3—H3	0.8800	C9—H9	0.9500
N3—C16	1.343 (2)	C9—C10	1.396 (3)
N3—C17	1.420 (2)	C9—C8	1.392 (3)
C2—H2	0.9500	C20—C25	1.507 (3)
C2—C3	1.394 (2)	C10—C11	1.500 (3)
C2—C1	1.387 (2)	C23—H23a	0.9800
C18—C17	1.408 (2)	C23—H23b	0.9800
C18—C19	1.386 (3)	C23—H23c	0.9800
C18—C24	1.503 (3)	C1S—C2S	1.446 (3)
C3—C4	1.510 (2)	C13—H13a	0.9800
C15—C16	1.513 (2)	C13—H13b	0.9800
C15—C14	1.390 (2)	C13—H13c	0.9800

C6—C5	1.395 (2)	C8—C12	1.513 (3)
C6—C7	1.392 (2)	C25—H25a	0.9800
C6—C13	1.507 (3)	C25—H25b	0.9800
C5—C10	1.392 (3)	C25—H25c	0.9800
N1S—C1S	1.127 (3)	C12—H12a	0.9800
C21—H21	0.9500	C12—H12b	0.9800
C21—C22	1.397 (2)	C12—H12c	0.9800
C21—C20	1.396 (3)	C11—H11a	0.9800
C17—C22	1.399 (2)	C11—H11b	0.9800
C19—H19	0.9500	C11—H11c	0.9800
C19—C20	1.392 (3)	C2S—H2Sa	0.9800
C22—C23	1.509 (3)	C2S—H2Sb	0.9800
C24—H24a	0.9800	C2S—H2Sc	0.9800
C15—N2—C3	117.66 (13)	C8—C7—H7	118.71 (11)
C4—N1—H1	118.54 (9)	C1—C14—C15	118.12 (15)
C5—N1—H1	118.54 (9)	H14—C14—C15	120.94 (10)
C5—N1—C4	122.92 (14)	H14—C14—C1	120.94 (10)
C16—N3—H3	118.07 (9)	C10—C9—H9	119.27 (11)
C17—N3—H3	118.07 (8)	C8—C9—H9	119.27 (12)
C17—N3—C16	123.87 (14)	C8—C9—C10	121.46 (18)
C3—C2—H2	120.80 (10)	C19—C20—C21	118.17 (17)
C1—C2—H2	120.80 (10)	C25—C20—C21	119.30 (19)
C1—C2—C3	118.40 (16)	C25—C20—C19	122.53 (19)
C19—C18—C17	118.31 (16)	C9—C10—C5	118.58 (17)
C24—C18—C17	120.04 (18)	C11—C10—C5	120.97 (17)
C24—C18—C19	121.65 (17)	C11—C10—C9	120.45 (18)
C2—C3—N2	123.16 (15)	H23a—C23—C22	109.5
C4—C3—N2	117.73 (14)	H23b—C23—C22	109.5
C4—C3—C2	119.11 (14)	H23b—C23—H23a	109.5
N1—C4—O1	124.51 (15)	H23c—C23—C22	109.5
C3—C4—O1	120.90 (15)	H23c—C23—H23a	109.5
C3—C4—N1	114.59 (14)	H23c—C23—H23b	109.5
C16—C15—N2	116.67 (14)	C2S—C1S—N1S	178.8 (3)
C14—C15—N2	123.43 (15)	H13a—C13—C6	109.5
C14—C15—C16	119.90 (14)	H13b—C13—C6	109.5
C7—C6—C5	117.87 (17)	H13b—C13—H13a	109.5
C13—C6—C5	120.82 (16)	H13c—C13—C6	109.5
C13—C6—C7	121.31 (17)	H13c—C13—H13a	109.5
C6—C5—N1	119.82 (16)	H13c—C13—H13b	109.5
C10—C5—N1	118.78 (15)	C9—C8—C7	118.10 (17)
C10—C5—C6	121.39 (15)	C12—C8—C7	121.5 (2)

N3—C16—O2	124.65 (16)	C12—C8—C9	120.4 (2)
C15—C16—O2	121.46 (16)	H25a—C25—C20	109.5
C15—C16—N3	113.88 (14)	H25b—C25—C20	109.5
C22—C21—H21	119.18 (10)	H25b—C25—H25a	109.5
C20—C21—H21	119.18 (11)	H25c—C25—C20	109.5
C20—C21—C22	121.64 (17)	H25c—C25—H25a	109.5
C18—C17—N3	119.20 (16)	H25c—C25—H25b	109.5
C22—C17—N3	119.76 (15)	H12a—C12—C8	109.5
C22—C17—C18	121.03 (16)	H12b—C12—C8	109.5
H19—C19—C18	118.87 (10)	H12b—C12—H12a	109.5
C20—C19—C18	122.25 (17)	H12c—C12—C8	109.5
C20—C19—H19	118.87 (11)	H12c—C12—H12a	109.5
C17—C22—C21	118.51 (16)	H12c—C12—H12b	109.5
C23—C22—C21	119.97 (17)	H11a—C11—C10	109.5
C23—C22—C17	121.51 (17)	H11b—C11—C10	109.5
H24a—C24—C18	109.5	H11b—C11—H11a	109.5
H24b—C24—C18	109.5	H11c—C11—C10	109.5
H24b—C24—H24a	109.5	H11c—C11—H11a	109.5
H24c—C24—C18	109.5	H11c—C11—H11b	109.5
H24c—C24—H24a	109.5	H2Sa—C2S—C1S	109.5
H24c—C24—H24b	109.5	H2Sb—C2S—C1S	109.5
H1a—C1—C2	120.40 (10)	H2Sb—C2S—H2Sa	109.5
C14—C1—C2	119.21 (16)	H2Sc—C2S—C1S	109.5
C14—C1—H1a	120.40 (10)	H2Sc—C2S—H2Sa	109.5
H7—C7—C6	118.71 (11)	H2Sc—C2S—H2Sb	109.5
C8—C7—C6	122.58 (17)		

Document origin: *publCIF* [Westrip, S. P. (2010). *J. Apply. Cryst.*, **43**, 920-925].

1.7 CIF check:

checkCIF/PLATON (full publication check)

Structure factors have been supplied for datablock(s) mo_17ov_ds7c_0s

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this report
Structure factor report

Datablock: mo_17ov_ds7c_0s

Bond precision: C-C = 0.0038 A Wavelength=0.71073

Cell: a=14.4964(6) b=13.6049(5) c=13.0108(6)
alpha=90 beta=90 gamma=90

Temperature: 173 K

	Calculated	Reported
Volume	2566.02(18)	2566.02(18)
Space group	P c a 21	P c a 21
Hall group	P 2c -2ac	P 2c -2ac
Moiety formula	C25 H27 N3 O2, C2 H3 N	C25 H27 N3 O2, C2 H3 N
Sum formula	C27 H30 N4 O2	C27 H30 N4 O2
Mr	442.55	442.55
Dx,g cm-3	1.146	1.146
Z	4	4
Mu (mm-1)	0.074	0.074
F000	944.0	944.0
F000'	944.36	
h,k,lmax	18,17,16	18,17,16
Nref	5687[2976]	5641
Tmin,Tmax	0.964,0.987	
Tmin'	0.952	

Correction method= Not given

Data completeness= 1.90/0.99 Theta(max)= 27.137

R(reflections)= 0.0505(5317) wR2(reflections)= 0.1440(5641)

S = 1.045 Npar= 299

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

STRVA01_ALERT_4_C Flack test results are ambiguous.
From the CIF: _refine_ls_abs_structure_Flack 0.500
From the CIF: _refine_ls_abs_structure_Flack_su 0.600

PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 17 Report
PLAT913_ALERT_3_C Missing # of Very Strong Reflections in FCF .... 10 Note
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 4 Check

Alert level G

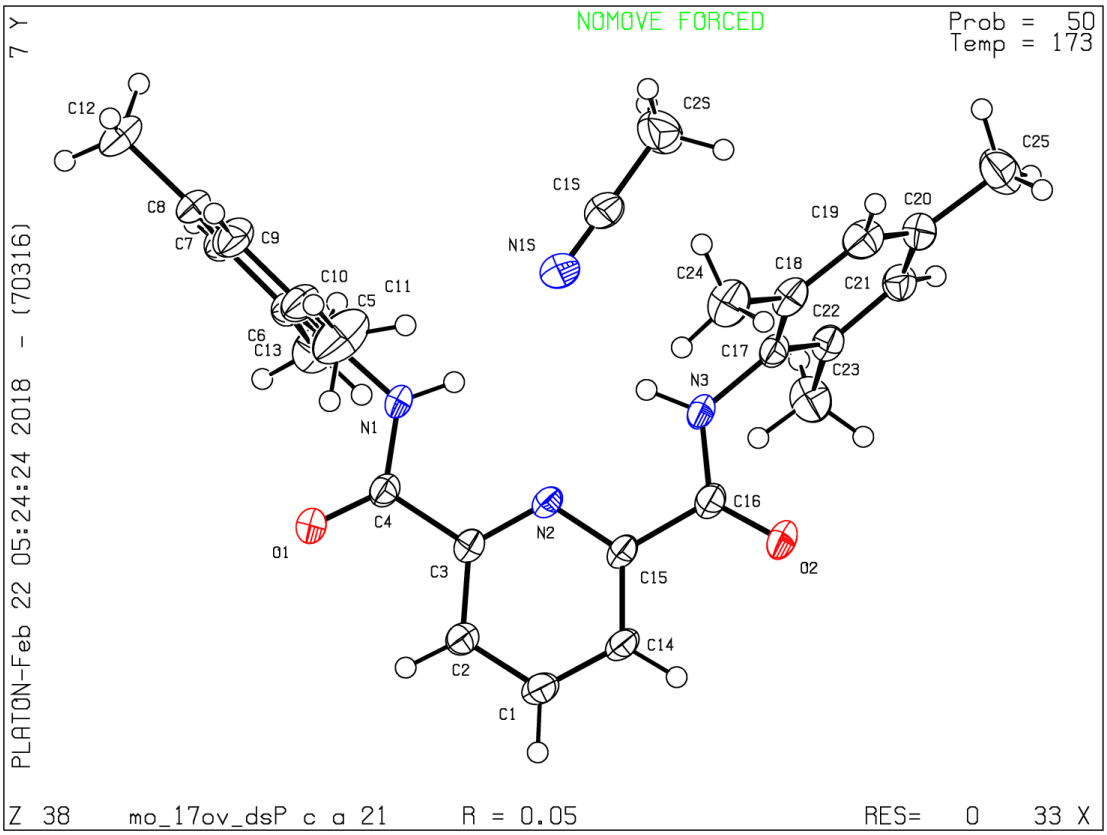
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..... 2 Report
PLAT012_ALERT_1_G No _shelx_res_checksum Found in CIF ..... Please Check
PLAT032_ALERT_4_G Std. Uncertainty on Flack Parameter Value High . 0.600 Report
PLAT063_ALERT_4_G Crystal Size Likely too Large for Beam Size .... 0.66 mm
PLAT380_ALERT_4_G Incorrectly? Oriented X(sp2)-Methyl Moiety ..... C13 Check
And 5 other PLAT380 Alerts
More ...
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..... 3 Note
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min). 4 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 2 Note
PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ... 5 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 6 Info

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
4 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
15 **ALERT level G** = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
2 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 ALERT type 3 Indicator that the structure quality may be low
11 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

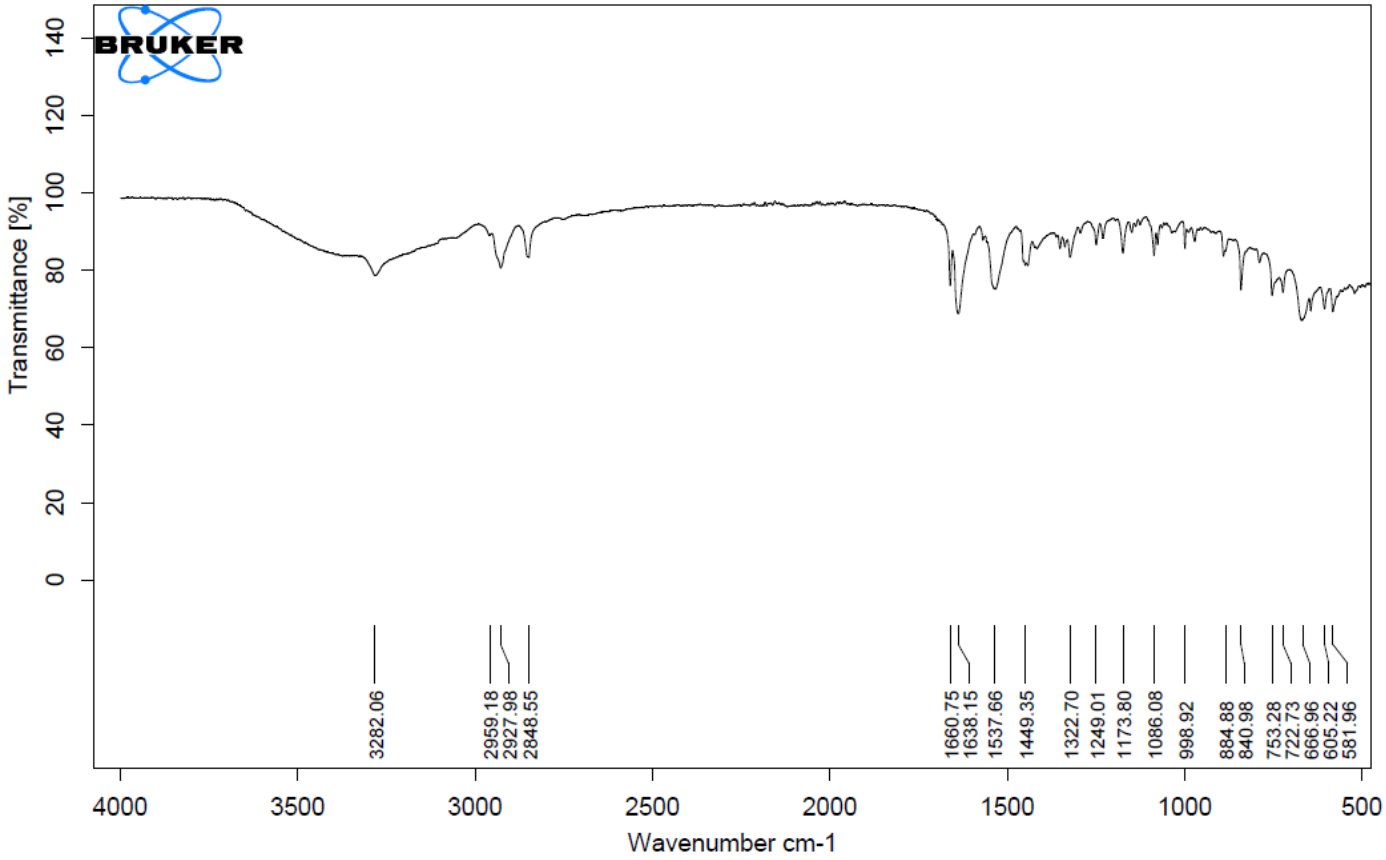
PLATON version of 30/01/2018; check.def file version of 30/01/2018

Datablock mo_17ov_ds7c_0s - ellipsoid plot



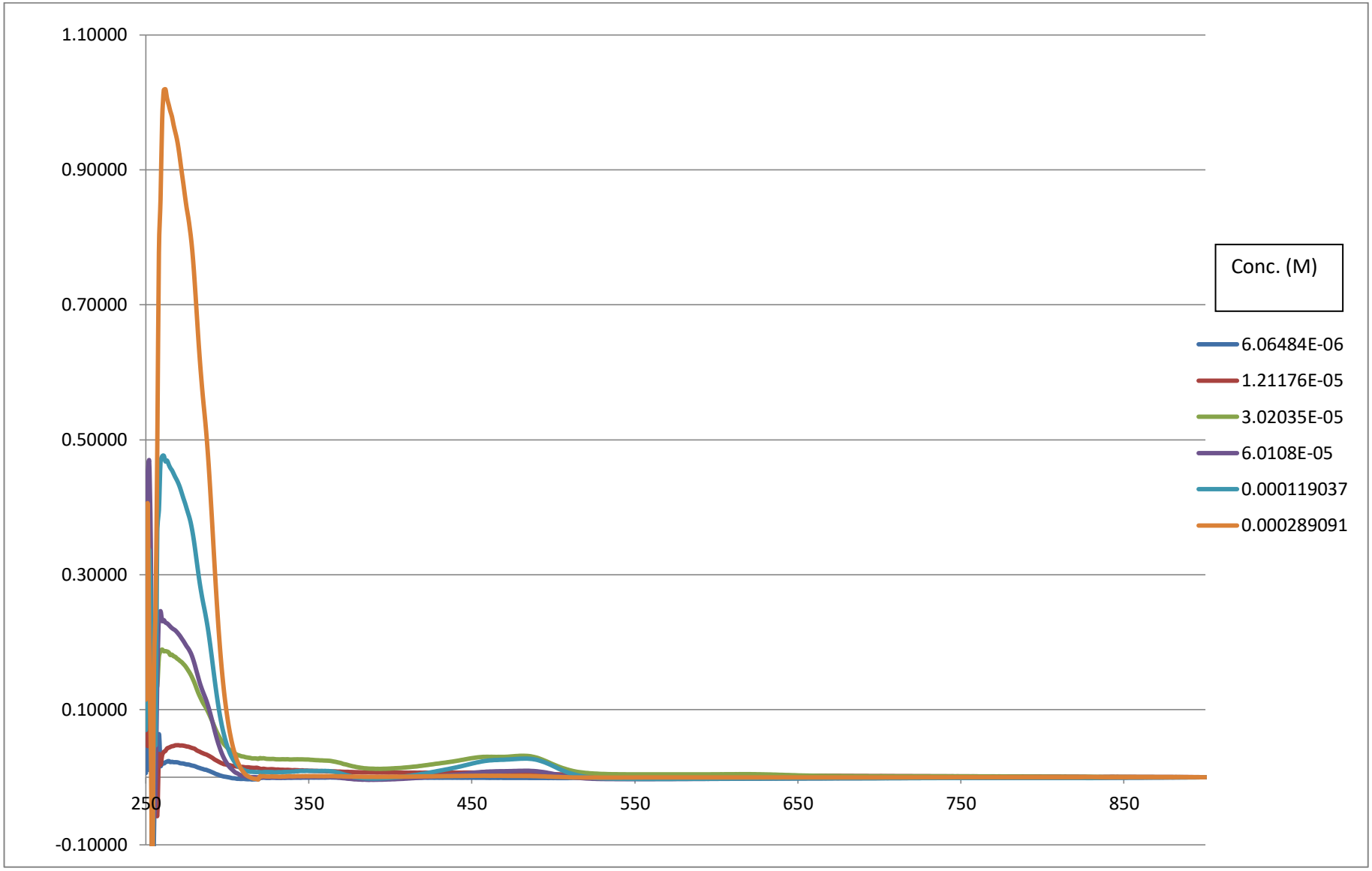
Download CIF editor (publCIF) from the IUCr
Download CIF editor (enCIFer) from the CCDC
Test a new CIF entry

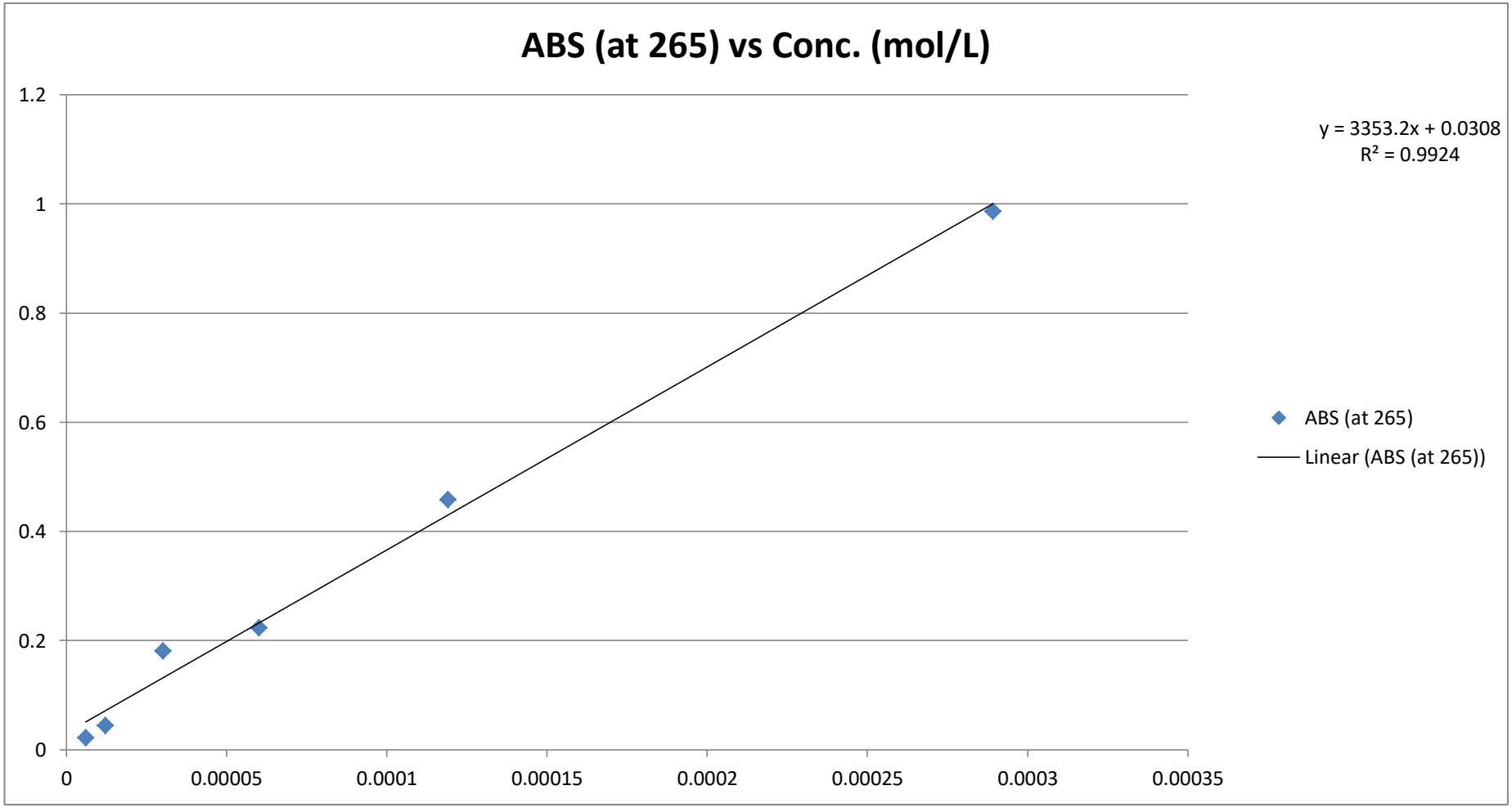
2.1 FTIR data for 2



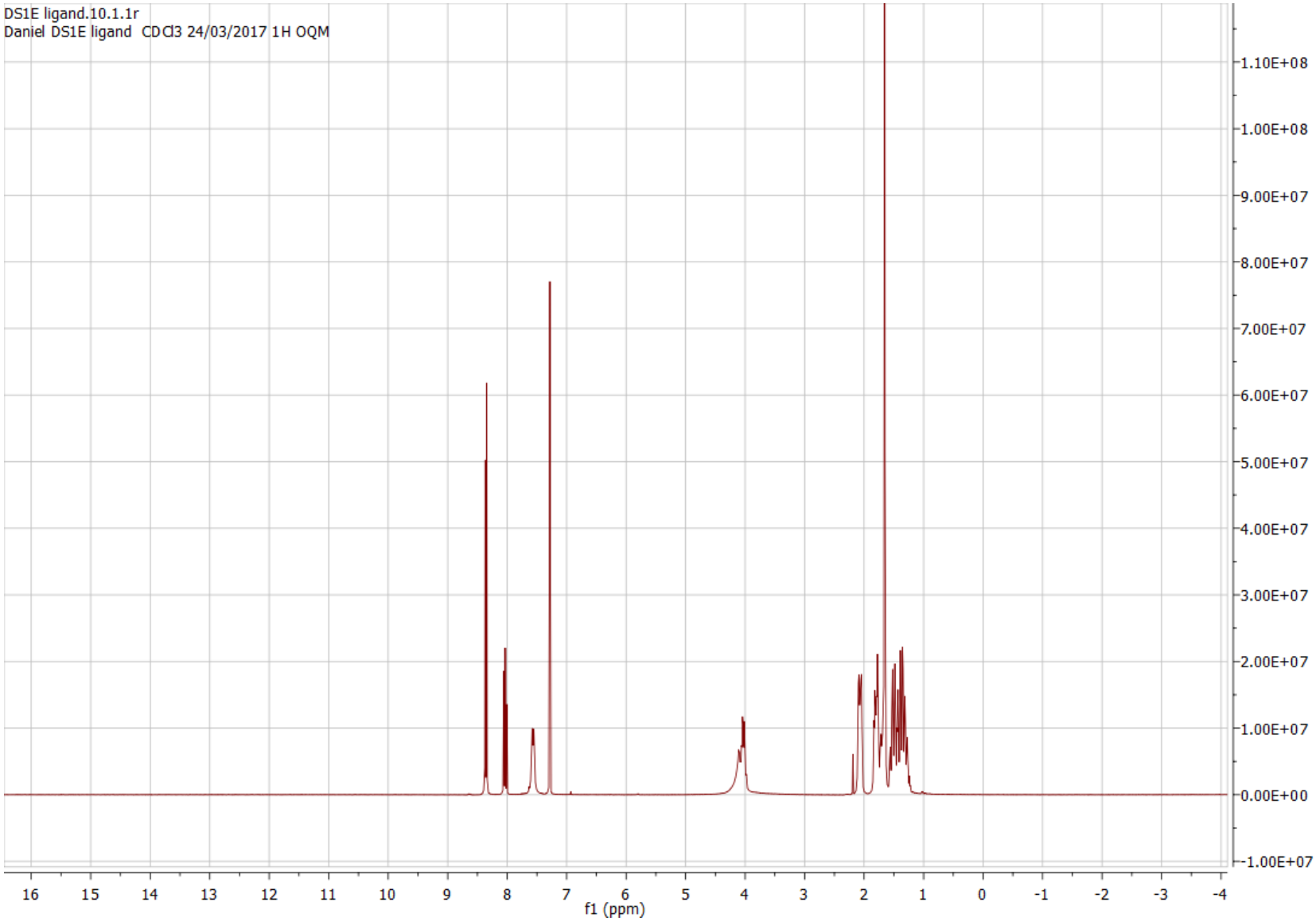
C:\WITS\Measurements\Daniel\Au Complex\DS1E ligand.0	DS1E ligand	Instrument type and / or accessory	24/02/2017

2.2 UV VIS data for 2

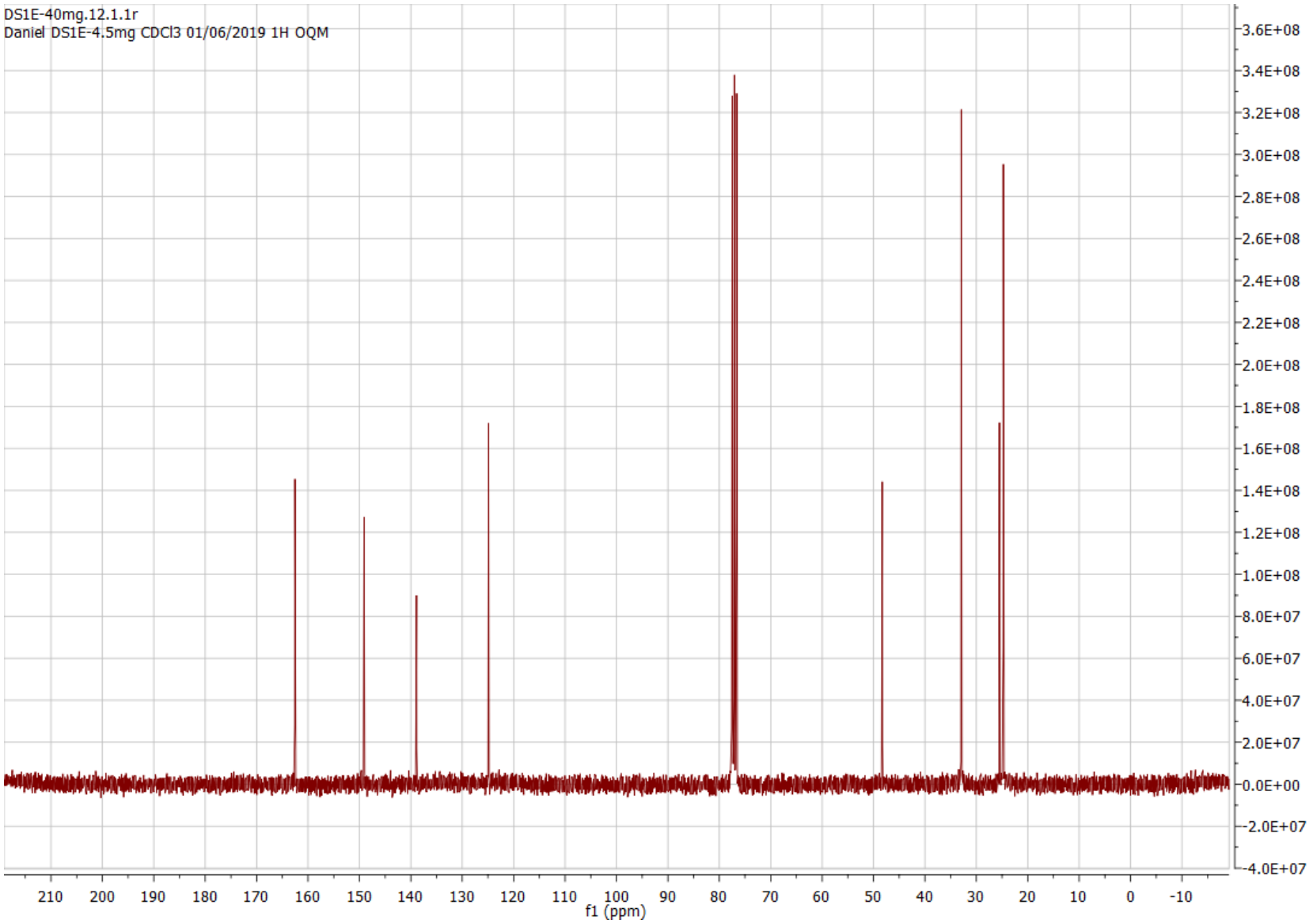



ABS (at 265) vs Conc. (mol/L)
y = 3353.2x + 0.0308
R² = 0.9924
ABS (at 265)
Linear (ABS (at 265))
1.2
1
0.8
0.6
0.4
0.2
0
0
0.00005
0.0001
0.00015
0.0002
0.00025
0.0003
0.00035

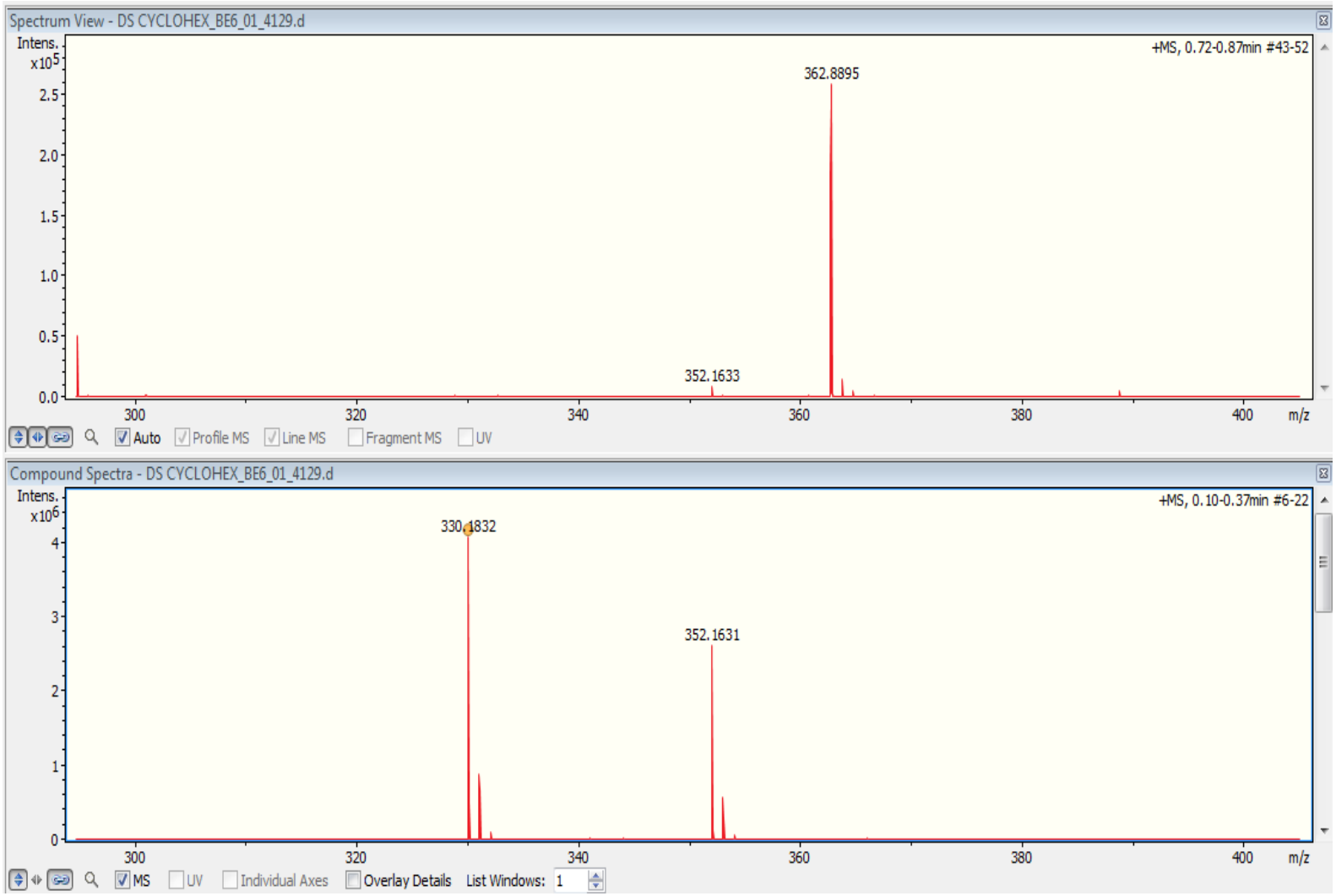
2.3 ^{1}H NMR data for 2



2.4 ^{13}C NMR data for 2

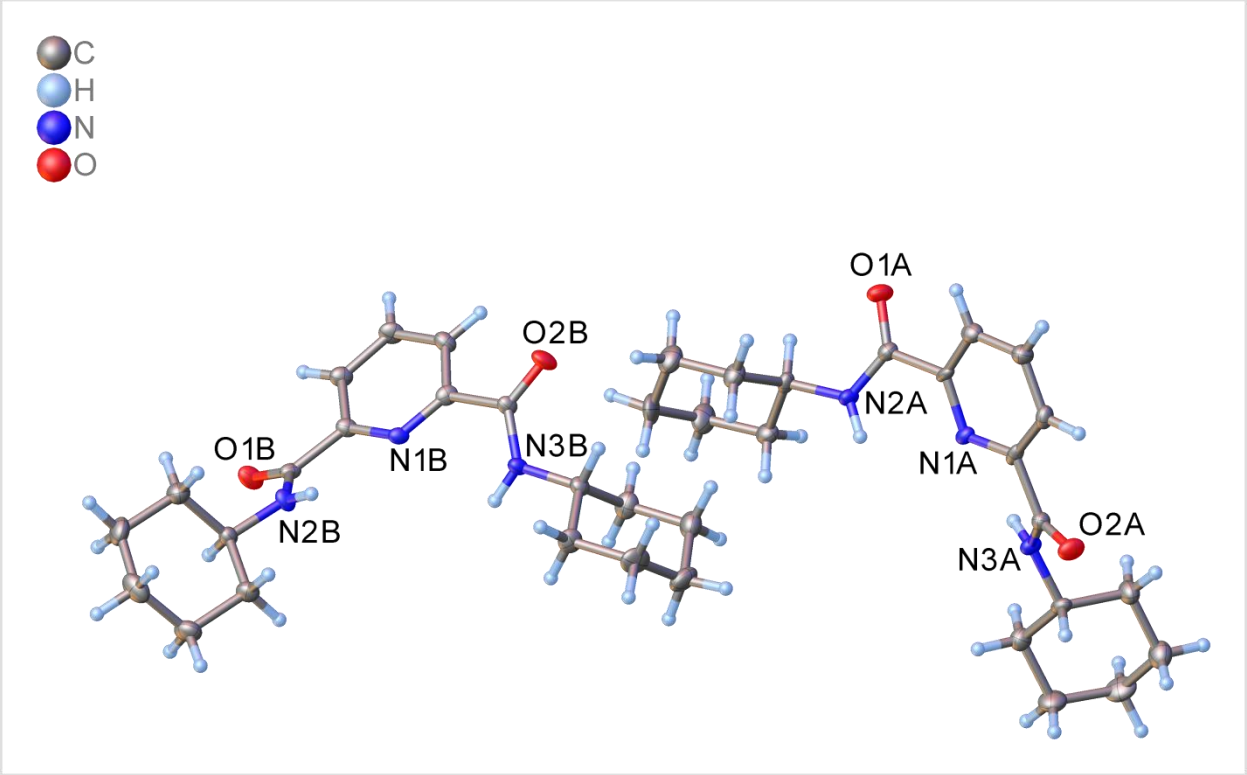


2.5 HMRS data for 2



2.6 X-ray crystallography data for 2

17o_ds_1e2_sadabs



Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Results and discussion

Computing details

Program(s) used to solve structure: *SHELXS* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015); molecular graphics: Olex2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: Olex2 (Dolomanov *et al.*, 2009).

References

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

(17o_ds_1e2_sadabs)

Crystal data

$C_{19}H_{27}N_3O_2$	$Z = 4$
$M_r = 329.43$	$F(000) = 712$
Triclinic, $P\bar{1}$	$D_x = 1.235$ Mg m^{-3}
$a = 8.3574$ (2) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 8.3564$ (2) Å	Cell parameters from 6426 reflections
$c = 25.9552$ (7) Å	$\theta = 2.5$–$28.1°$
$\alpha = 81.439$ (2)°	$\mu = 0.08$ mm^{-1}
$\beta = 81.446$ (1)°	$T = 173$ K
$\gamma = 89.965$ (1)°	Blocky, colourless
$V = 1772.09$ (8) Å^3	0.39 × 0.17 × 0.16 mm

Data collection

Bruker SMART APEX2 area detector diffractometer	8413 independent reflections
Radiation source: microfocus sealed X-ray tube, Incoatec Iμs	6608 reflections with $I > 2\sigma(I)$
Mirror optics monochromator	$R_{int} = 0.052$
Detector resolution: 7.9 pixels mm^{-1}	$\theta_{max} = 28.0°$, $\theta_{min} = 1.6°$
ω and ϕ scans	$h = -10 \rightarrow 11$
Absorption correction: multi-scan *SADABS2012*/1 (Bruker,2012) was used for absorption correction. wR2(int) was 0.0741 before and 0.0622 after correction. The Ratio of minimum to maximum transmission is 0.8399. The λ/2 correction factor is 0.0015.	$k = -10 \rightarrow 11$
$T_{min} = 0.626$, $T_{max} = 0.746$	$l = -34 \rightarrow 34$
25657 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)] = 0.094$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.240$	$w = 1/[\sigma^2(F_o^2) + (0.1836P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
8413 reflections	$\Delta\rangle_{max} = 1.41$ e Å^{-3}
450 parameters	$\Delta\rangle_{min} = -0.37$ e Å^{-3}
0 restraints	

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 twinned and refined using the twin law [1 -1 0] to account for the intensity data. Residual electron density peaks in the plane of each pyridine ring indicate possible further (yet unresolvable) disorder or twinning.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for (17o_ds_1e2_sadabs)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1A	0.3583 (3)	0.6290 (2)	0.42000 (9)	0.0276 (5)
O2A	0.2857 (3)	-0.1288 (2)	0.57931 (9)	0.0275 (5)
N1A	0.3817 (3)	0.2505 (3)	0.49945 (8)	0.0172 (6)
N2A	0.5667 (3)	0.4543 (3)	0.42024 (10)	0.0191 (5)
N3A	0.4951 (3)	0.0458 (3)	0.57880 (9)	0.0195 (5)
C1A	0.3079 (4)	0.3682 (3)	0.47007 (10)	0.0174 (6)
C2A	0.1440 (4)	0.3725 (4)	0.46901 (11)	0.0221 (7)
H2AA	0.0990	0.4578	0.4476	0.027*
C3A	0.0461 (4)	0.2509 (4)	0.49954 (12)	0.0240 (7)
H3AA	-0.0676	0.2518	0.4996	0.029*
C4A	0.1155 (4)	0.1271 (4)	0.53018 (11)	0.0216 (7)
H4A	0.0514	0.0405	0.5511	0.026*
C5A	0.2808 (4)	0.1345 (3)	0.52912 (10)	0.0191 (7)
C6A	0.4158 (4)	0.4967 (3)	0.43448 (11)	0.0189 (6)
C7A	0.3556 (4)	0.0045 (3)	0.56435 (10)	0.0189 (6)
C8A	0.6794 (4)	0.5569 (3)	0.37979 (11)	0.0192 (6)
H8A	0.6507	0.6724	0.3814	0.023*
C9A	0.8530 (4)	0.5354 (4)	0.39011 (12)	0.0246 (7)
H9AA	0.8622	0.5635	0.4253	0.030*
H9AB	0.8824	0.4206	0.3901	0.030*
C10A	0.9685 (4)	0.6408 (5)	0.34886 (14)	0.0353 (8)
H10A	1.0810	0.6179	0.3549	0.042*
H10B	0.9482	0.7560	0.3519	0.042*
C11A	0.9490 (5)	0.6109 (5)	0.29329 (14)	0.0378 (9)
H11A	1.0189	0.6890	0.2671	0.045*
H11B	0.9853	0.5005	0.2886	0.045*
C12A	0.7756 (5)	0.6282 (5)	0.28330 (13)	0.0353 (9)
H12A	0.7439	0.7424	0.2832	0.042*

H12B	0.7669	0.5994	0.2482	0.042*
C13A	0.6613 (4)	0.5207 (4)	0.32482 (11)	0.0264 (7)
H13A	0.6854	0.4058	0.3226	0.032*
H13B	0.5484	0.5395	0.3185	0.032*
C14A	0.5696 (4)	-0.0576 (3)	0.61928 (11)	0.0188 (6)
H14A	0.5464	-0.1731	0.6165	0.023*
C15A	0.7512 (4)	-0.0311 (4)	0.61023 (12)	0.0252 (7)
H15A	0.7770	0.0839	0.6112	0.030*
H15B	0.7965	-0.0566	0.5750	0.030*
C16A	0.8278 (5)	-0.1378 (5)	0.65218 (14)	0.0347 (8)
H16A	0.9457	-0.1141	0.6468	0.042*
H16B	0.8120	-0.2529	0.6486	0.042*
C17A	0.7543 (5)	-0.1104 (5)	0.70760 (14)	0.0362 (9)
H17A	0.8005	-0.1874	0.7340	0.043*
H17B	0.7818	0.0008	0.7128	0.043*
C18A	0.5733 (5)	-0.1335 (5)	0.71569 (13)	0.0339 (8)
H18A	0.5279	-0.1084	0.7510	0.041*
H18B	0.5466	-0.2483	0.7147	0.041*
C19A	0.4948 (4)	-0.0265 (4)	0.67410 (11)	0.0291 (7)
H19A	0.5112	0.0887	0.6773	0.035*
H19B	0.3770	-0.0503	0.6796	0.035*
O1B	1.1272 (2)	0.2175 (3)	-0.08123 (9)	0.0258 (5)
O2B	0.3735 (2)	0.1421 (3)	0.07984 (9)	0.0262 (5)
N1B	0.7499 (3)	0.1238 (3)	-0.00096 (9)	0.0153 (5)
N2B	0.9534 (3)	0.0079 (3)	-0.08108 (10)	0.0190 (5)
N3B	0.5481 (3)	-0.0677 (3)	0.07857 (10)	0.0187 (5)
C1B	0.8672 (3)	0.2185 (4)	-0.03026 (11)	0.0175 (6)
C2B	0.8716 (4)	0.3868 (4)	-0.03107 (12)	0.0208 (6)
H2BA	0.9570	0.4510	-0.0526	0.025*
C3B	0.7506 (4)	0.4593 (4)	-0.00029 (12)	0.0231 (7)
H3BA	0.7516	0.5728	0.0001	0.028*
C4B	0.6281 (4)	0.3592 (4)	0.02989 (12)	0.0208 (6)
H4B	0.5409	0.4031	0.0508	0.025*
C5B	0.6356 (3)	0.1907 (4)	0.02888 (11)	0.0184 (6)
C6B	0.9949 (3)	0.1467 (3)	-0.06604 (11)	0.0171 (6)
C7B	0.5057 (3)	0.0854 (3)	0.06434 (11)	0.0163 (6)
C8B	1.0554 (3)	-0.0639 (4)	-0.12186 (11)	0.0184 (6)
H8B	1.1706	-0.0335	-0.1210	0.022*
C9B	1.0378 (4)	-0.2479 (4)	-0.10985 (12)	0.0241 (7)
H9BA	0.9240	-0.2811	-0.1096	0.029*
H9BB	1.0673	-0.2864	-0.0746	0.029*
C10B	1.1487 (4)	-0.3238 (4)	-0.15186 (13)	0.0310 (7)

H10C	1.1330	-0.4429	-0.1450	0.037*
H10D	1.2630	-0.2986	-0.1497	0.037*
C11B	1.1121 (5)	-0.2591 (5)	-0.20779 (14)	0.0350 (9)
H11C	1.1910	-0.3030	-0.2342	0.042*
H11D	1.0025	-0.2965	-0.2114	0.042*
C12B	1.1215 (5)	-0.0754 (5)	-0.21843 (12)	0.0331 (8)
H12C	1.2350	-0.0388	-0.2200	0.040*
H12D	1.0878	-0.0376	-0.2531	0.040*
C13B	1.0153 (4)	-0.0006 (4)	-0.17655 (12)	0.0279 (7)
H13C	0.9004	-0.0260	-0.1778	0.033*
H13D	1.0308	0.1185	-0.1837	0.033*
C14B	0.4480 (3)	-0.1763 (4)	0.12019 (11)	0.0191 (6)
H14B	0.3324	-0.1458	0.1198	0.023*
C15B	0.4651 (4)	-0.3493 (4)	0.10969 (12)	0.0245 (7)
H15C	0.5793	-0.3815	0.1090	0.029*
H15D	0.4339	-0.3581	0.0749	0.029*
C16B	0.3563 (4)	-0.4630 (4)	0.15294 (14)	0.0335 (8)
H16C	0.2415	-0.4361	0.1514	0.040*
H16D	0.3713	-0.5761	0.1464	0.040*
C17B	0.3953 (5)	-0.4485 (4)	0.20765 (14)	0.0365 (9)
H17C	0.5056	-0.4881	0.2108	0.044*
H17D	0.3179	-0.5164	0.2347	0.044*
C18B	0.3848 (5)	-0.2719 (5)	0.21706 (13)	0.0327 (8)
H18C	0.2711	-0.2374	0.2184	0.039*
H18D	0.4185	-0.2632	0.2515	0.039*
C19B	0.4908 (4)	-0.1605 (4)	0.17423 (11)	0.0287 (7)
H19C	0.6058	-0.1877	0.1751	0.034*
H19D	0.4766	-0.0472	0.1807	0.034*
H3A	0.523 (4)	0.141 (4)	0.5687 (13)	0.018 (8)*
H3B	0.648 (4)	-0.107 (4)	0.0715 (13)	0.019 (8)*
H2A	0.583 (5)	0.349 (5)	0.4319 (16)	0.040 (11)*
H2B	0.866 (4)	-0.032 (4)	-0.0712 (13)	0.020 (9)*

Atomic displacement parameters (Å^2) for (17o_ds_1e2_sadabs)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0244 (12)	0.0177 (11)	0.0377 (13)	0.0031 (9)	-0.0039 (10)	0.0049 (9)
O2A	0.0276 (13)	0.0168 (11)	0.0360 (12)	-0.0041 (9)	-0.0054 (10)	0.0035 (9)
N1A	0.0236 (14)	0.0138 (12)	0.0143 (11)	0.0017 (10)	-0.0041 (10)	-0.0009 (9)
N2A	0.0186 (13)	0.0134 (11)	0.0227 (12)	0.0009 (10)	-0.0007 (10)	0.0031 (9)
N3A	0.0204 (13)	0.0144 (12)	0.0223 (12)	-0.0004 (10)	-0.0035 (10)	0.0024 (9)
C1A	0.0304 (18)	0.0106 (13)	0.0109 (11)	0.0057 (12)	-0.0037 (12)	-0.0001 (9)

C2A	0.0243 (17)	0.0238 (15)	0.0191 (13)	0.0044 (13)	-0.0062 (13)	-0.0031 (11)
C3A	0.0225 (17)	0.0226 (15)	0.0256 (15)	0.0039 (13)	-0.0015 (12)	-0.0011 (12)
C4A	0.0254 (17)	0.0195 (14)	0.0178 (13)	-0.0047 (13)	0.0021 (12)	-0.0013 (11)
C5A	0.036 (2)	0.0084 (12)	0.0121 (12)	-0.0021 (13)	-0.0009 (13)	-0.0017 (9)
C6A	0.0209 (15)	0.0172 (13)	0.0180 (13)	0.0006 (12)	-0.0040 (11)	0.0002 (10)
C7A	0.0185 (14)	0.0180 (13)	0.0187 (13)	0.0013 (11)	-0.0002 (11)	-0.0008 (10)
C8A	0.0250 (16)	0.0142 (13)	0.0161 (13)	-0.0031 (12)	0.0023 (12)	0.0001 (10)
C9A	0.0270 (18)	0.0250 (16)	0.0219 (14)	0.0037 (13)	-0.0059 (13)	-0.0018 (12)
C10A	0.0222 (17)	0.045 (2)	0.0351 (18)	-0.0092 (15)	0.0002 (14)	0.0020 (16)
C11A	0.032 (2)	0.045 (2)	0.0271 (17)	-0.0041 (16)	0.0143 (15)	0.0049 (16)
C12A	0.034 (2)	0.045 (2)	0.0252 (16)	-0.0005 (17)	-0.0075 (15)	0.0027 (15)
C13A	0.0271 (17)	0.0302 (17)	0.0209 (14)	-0.0032 (13)	-0.0055 (12)	0.0007 (12)
C14A	0.0250 (16)	0.0127 (13)	0.0189 (13)	0.0044 (12)	-0.0053 (12)	-0.0012 (10)
C15A	0.0254 (18)	0.0245 (16)	0.0243 (15)	-0.0008 (13)	-0.0017 (13)	-0.0012 (12)
C16A	0.0293 (19)	0.039 (2)	0.0357 (18)	0.0097 (15)	-0.0124 (15)	0.0004 (15)
C17A	0.041 (2)	0.039 (2)	0.0339 (18)	0.0045 (17)	-0.0229 (17)	-0.0041 (16)
C18A	0.034 (2)	0.043 (2)	0.0245 (16)	0.0019 (16)	-0.0038 (14)	-0.0048 (15)
C19A	0.0287 (18)	0.0367 (18)	0.0213 (15)	0.0074 (14)	-0.0013 (13)	-0.0051 (13)
O1B	0.0169 (11)	0.0183 (11)	0.0409 (13)	-0.0049 (9)	0.0025 (9)	-0.0073 (9)
O2B	0.0145 (11)	0.0206 (11)	0.0416 (14)	0.0033 (9)	0.0024 (9)	-0.0055 (10)
N1B	0.0134 (12)	0.0114 (11)	0.0218 (11)	-0.0010 (9)	-0.0047 (9)	-0.0026 (9)
N2B	0.0119 (12)	0.0191 (12)	0.0257 (13)	-0.0040 (10)	0.0018 (10)	-0.0073 (10)
N3B	0.0157 (12)	0.0141 (12)	0.0247 (12)	0.0020 (10)	0.0008 (9)	-0.0017 (10)
C1B	0.0144 (14)	0.0158 (15)	0.0233 (13)	-0.0011 (11)	-0.0044 (11)	-0.0047 (12)
C2B	0.0190 (15)	0.0160 (15)	0.0278 (15)	-0.0018 (12)	-0.0045 (12)	-0.0031 (12)
C3B	0.0256 (16)	0.0135 (14)	0.0316 (15)	-0.0023 (12)	-0.0051 (13)	-0.0068 (12)
C4B	0.0172 (14)	0.0211 (16)	0.0252 (14)	0.0019 (12)	-0.0024 (11)	-0.0083 (12)
C5B	0.0172 (14)	0.0159 (15)	0.0229 (13)	-0.0045 (11)	-0.0067 (11)	-0.0020 (12)
C6B	0.0139 (13)	0.0128 (13)	0.0239 (13)	-0.0001 (10)	-0.0044 (11)	0.0007 (11)
C7B	0.0147 (13)	0.0127 (13)	0.0218 (13)	-0.0022 (10)	-0.0015 (10)	-0.0046 (10)
C8B	0.0185 (14)	0.0157 (14)	0.0199 (14)	-0.0001 (11)	0.0007 (11)	-0.0022 (11)
C9B	0.0270 (17)	0.0190 (16)	0.0267 (15)	0.0036 (13)	-0.0029 (12)	-0.0056 (12)
C10B	0.0363 (19)	0.0228 (16)	0.0353 (17)	0.0086 (14)	-0.0054 (15)	-0.0087 (14)
C11B	0.0303 (19)	0.044 (2)	0.0352 (18)	0.0040 (16)	-0.0041 (15)	-0.0221 (17)
C12B	0.036 (2)	0.042 (2)	0.0180 (15)	0.0000 (16)	-0.0004 (14)	0.0010 (14)
C13B	0.0294 (17)	0.0263 (16)	0.0267 (16)	0.0051 (13)	-0.0030 (13)	-0.0013 (13)
C14B	0.0150 (14)	0.0189 (14)	0.0223 (13)	0.0004 (11)	-0.0004 (11)	-0.0027 (11)
C15B	0.0250 (17)	0.0178 (16)	0.0302 (16)	-0.0074 (12)	-0.0029 (13)	-0.0033 (13)
C16B	0.0338 (18)	0.0155 (15)	0.048 (2)	-0.0093 (13)	-0.0020 (16)	0.0017 (14)
C17B	0.038 (2)	0.0274 (19)	0.0380 (19)	-0.0023 (15)	-0.0058 (16)	0.0130 (16)
C18B	0.032 (2)	0.040 (2)	0.0237 (15)	0.0007 (15)	-0.0007 (14)	0.0000 (15)
C19B	0.0345 (18)	0.0270 (16)	0.0253 (15)	-0.0054 (14)	-0.0045 (13)	-0.0062 (13)

Geometric parameters (Å, º) for (17o_ds_1e2_sadabs)

O1A—C6A	1.234 (4)	O1B—C6B	1.237 (3)
O2A—C7A	1.239 (3)	O2B—C7B	1.237 (3)
N1A—C1A	1.358 (4)	N1B—C1B	1.329 (3)
N1A—C5A	1.353 (3)	N1B—C5B	1.316 (4)
N2A—C6A	1.324 (4)	N2B—C6B	1.339 (4)
N2A—C8A	1.471 (4)	N2B—C8B	1.458 (4)
N2A—H2A	0.90 (4)	N2B—H2B	0.80 (3)
N3A—C7A	1.338 (4)	N3B—C7B	1.341 (4)
N3A—C14A	1.470 (4)	N3B—C14B	1.460 (4)
N3A—H3A	0.82 (3)	N3B—H3B	0.90 (4)
C1A—C2A	1.374 (5)	C1B—C2B	1.404 (4)
C1A—C6A	1.509 (4)	C1B—C6B	1.494 (4)
C2A—H2AA	0.9500	C2B—H2BA	0.9500
C2A—C3A	1.377 (4)	C2B—C3B	1.389 (4)
C3A—H3AA	0.9500	C3B—H3BA	0.9500
C3A—C4A	1.390 (4)	C3B—C4B	1.389 (4)
C4A—H4A	0.9500	C4B—H4B	0.9500
C4A—C5A	1.379 (5)	C4B—C5B	1.413 (4)
C5A—C7A	1.510 (4)	C5B—C7B	1.504 (4)
C8A—H8A	1.0000	C8B—H8B	1.0000
C8A—C9A	1.520 (5)	C8B—C9B	1.526 (4)
C8A—C13A	1.530 (4)	C8B—C13B	1.525 (4)
C9A—H9AA	0.9900	C9B—H9BA	0.9900
C9A—H9AB	0.9900	C9B—H9BB	0.9900
C9A—C10A	1.505 (4)	C9B—C10B	1.534 (4)
C10A—H10A	0.9900	C10B—H10C	0.9900
C10A—H10B	0.9900	C10B—H10D	0.9900
C10A—C11A	1.532 (5)	C10B—C11B	1.547 (5)
C11A—H11A	0.9900	C11B—H11C	0.9900
C11A—H11B	0.9900	C11B—H11D	0.9900
C11A—C12A	1.513 (6)	C11B—C12B	1.518 (5)
C12A—H12A	0.9900	C12B—H12C	0.9900
C12A—H12B	0.9900	C12B—H12D	0.9900
C12A—C13A	1.512 (4)	C12B—C13B	1.508 (5)
C13A—H13A	0.9900	C13B—H13C	0.9900
C13A—H13B	0.9900	C13B—H13D	0.9900
C14A—H14A	1.0000	C14B—H14B	1.0000
C14A—C15A	1.512 (5)	C14B—C15B	1.513 (4)
C14A—C19A	1.526 (4)	C14B—C19B	1.522 (4)

C15A—H15A	0.9900	C15B—H15C	0.9900
C15A—H15B	0.9900	C15B—H15D	0.9900
C15A—C16A	1.519 (5)	C15B—C16B	1.539 (4)
C16A—H16A	0.9900	C16B—H16C	0.9900
C16A—H16B	0.9900	C16B—H16D	0.9900
C16A—C17A	1.528 (5)	C16B—C17B	1.525 (5)
C17A—H17A	0.9900	C17B—H17C	0.9900
C17A—H17B	0.9900	C17B—H17D	0.9900
C17A—C18A	1.505 (6)	C17B—C18B	1.532 (6)
C18A—H18A	0.9900	C18B—H18C	0.9900
C18A—H18B	0.9900	C18B—H18D	0.9900
C18A—C19A	1.522 (5)	C18B—C19B	1.512 (4)
C19A—H19A	0.9900	C19B—H19C	0.9900
C19A—H19B	0.9900	C19B—H19D	0.9900
C5A—N1A—C1A	114.9 (3)	C5B—N1B—C1B	118.5 (2)
C6A—N2A—C8A	122.0 (2)	C6B—N2B—C8B	122.0 (2)
C6A—N2A—H2A	112 (3)	C6B—N2B—H2B	121 (3)
C8A—N2A—H2A	124 (3)	C8B—N2B—H2B	116 (2)
C7A—N3A—C14A	122.1 (2)	C7B—N3B—C14B	121.4 (2)
C7A—N3A—H3A	114 (2)	C7B—N3B—H3B	125 (2)
C14A—N3A—H3A	122 (2)	C14B—N3B—H3B	111 (2)
N1A—C1A—C2A	124.2 (3)	N1B—C1B—C2B	122.3 (3)
N1A—C1A—C6A	117.0 (3)	N1B—C1B—C6B	119.6 (3)
C2A—C1A—C6A	118.7 (3)	C2B—C1B—C6B	118.1 (2)
C1A—C2A—H2AA	120.6	C1B—C2B—H2BA	120.1
C1A—C2A—C3A	118.8 (3)	C3B—C2B—C1B	119.8 (3)
C3A—C2A—H2AA	120.6	C3B—C2B—H2BA	120.1
C2A—C3A—H3AA	120.4	C2B—C3B—H3BA	121.3
C2A—C3A—C4A	119.3 (3)	C4B—C3B—C2B	117.3 (3)
C4A—C3A—H3AA	120.4	C4B—C3B—H3BA	121.3
C3A—C4A—H4A	121.2	C3B—C4B—H4B	120.6
C5A—C4A—C3A	117.6 (3)	C3B—C4B—C5B	118.8 (3)
C5A—C4A—H4A	121.2	C5B—C4B—H4B	120.6
N1A—C5A—C4A	125.2 (3)	N1B—C5B—C4B	123.2 (3)
N1A—C5A—C7A	117.1 (3)	N1B—C5B—C7B	119.6 (3)
C4A—C5A—C7A	117.7 (3)	C4B—C5B—C7B	117.2 (3)
O1A—C6A—N2A	125.2 (3)	O1B—C6B—N2B	124.0 (3)
O1A—C6A—C1A	119.1 (3)	O1B—C6B—C1B	120.2 (2)
N2A—C6A—C1A	115.7 (2)	N2B—C6B—C1B	115.8 (2)
O2A—C7A—N3A	124.1 (3)	O2B—C7B—N3B	124.4 (2)
O2A—C7A—C5A	120.2 (3)	O2B—C7B—C5B	120.7 (3)

N3A—C7A—C5A	115.7 (2)	N3B—C7B—C5B	114.8 (2)
N2A—C8A—H8A	108.0	N2B—C8B—H8B	108.2
N2A—C8A—C9A	111.1 (2)	N2B—C8B—C9B	109.5 (2)
N2A—C8A—C13A	110.4 (2)	N2B—C8B—C13B	111.5 (2)
C9A—C8A—H8A	108.0	C9B—C8B—H8B	108.2
C9A—C8A—C13A	111.1 (3)	C13B—C8B—H8B	108.2
C13A—C8A—H8A	108.0	C13B—C8B—C9B	111.2 (2)
C8A—C9A—H9AA	109.4	C8B—C9B—H9BA	109.7
C8A—C9A—H9AB	109.4	C8B—C9B—H9BB	109.7
H9AA—C9A—H9AB	108.0	C8B—C9B—C10B	109.6 (2)
C10A—C9A—C8A	111.2 (3)	H9BA—C9B—H9BB	108.2
C10A—C9A—H9AA	109.4	C10B—C9B—H9BA	109.7
C10A—C9A—H9AB	109.4	C10B—C9B—H9BB	109.7
C9A—C10A—H10A	109.4	C9B—C10B—H10C	109.4
C9A—C10A—H10B	109.4	C9B—C10B—H10D	109.4
C9A—C10A—C11A	111.3 (3)	C9B—C10B—C11B	111.2 (3)
H10A—C10A—H10B	108.0	H10C—C10B—H10D	108.0
C11A—C10A—H10A	109.4	C11B—C10B—H10C	109.4
C11A—C10A—H10B	109.4	C11B—C10B—H10D	109.4
C10A—C11A—H11A	109.2	C10B—C11B—H11C	109.4
C10A—C11A—H11B	109.2	C10B—C11B—H11D	109.4
H11A—C11A—H11B	107.9	H11C—C11B—H11D	108.0
C12A—C11A—C10A	112.1 (3)	C12B—C11B—C10B	111.2 (3)
C12A—C11A—H11A	109.2	C12B—C11B—H11C	109.4
C12A—C11A—H11B	109.2	C12B—C11B—H11D	109.4
C11A—C12A—H12A	109.3	C11B—C12B—H12C	109.2
C11A—C12A—H12B	109.3	C11B—C12B—H12D	109.2
H12A—C12A—H12B	108.0	H12C—C12B—H12D	107.9
C13A—C12A—C11A	111.5 (3)	C13B—C12B—C11B	112.1 (3)
C13A—C12A—H12A	109.3	C13B—C12B—H12C	109.2
C13A—C12A—H12B	109.3	C13B—C12B—H12D	109.2
C8A—C13A—H13A	109.6	C8B—C13B—H13C	109.4
C8A—C13A—H13B	109.6	C8B—C13B—H13D	109.4
C12A—C13A—C8A	110.2 (3)	C12B—C13B—C8B	111.1 (3)
C12A—C13A—H13A	109.6	C12B—C13B—H13C	109.4
C12A—C13A—H13B	109.6	C12B—C13B—H13D	109.4
H13A—C13A—H13B	108.1	H13C—C13B—H13D	108.0
N3A—C14A—H14A	108.1	N3B—C14B—H14B	108.2
N3A—C14A—C15A	110.7 (2)	N3B—C14B—C15B	110.1 (2)
N3A—C14A—C19A	110.2 (2)	N3B—C14B—C19B	111.4 (2)
C15A—C14A—H14A	108.1	C15B—C14B—H14B	108.2
C15A—C14A—C19A	111.5 (2)	C15B—C14B—C19B	110.7 (3)

C19A—C14A—H14A	108.1	C19B—C14B—H14B	108.2
C14A—C15A—H15A	109.5	C14B—C15B—H15C	109.7
C14A—C15A—H15B	109.5	C14B—C15B—H15D	109.7
C14A—C15A—C16A	110.6 (3)	C14B—C15B—C16B	110.0 (3)
H15A—C15A—H15B	108.1	H15C—C15B—H15D	108.2
C16A—C15A—H15A	109.5	C16B—C15B—H15C	109.7
C16A—C15A—H15B	109.5	C16B—C15B—H15D	109.7
C15A—C16A—H16A	109.3	C15B—C16B—H16C	109.3
C15A—C16A—H16B	109.3	C15B—C16B—H16D	109.3
C15A—C16A—C17A	111.5 (3)	H16C—C16B—H16D	108.0
H16A—C16A—H16B	108.0	C17B—C16B—C15B	111.6 (3)
C17A—C16A—H16A	109.3	C17B—C16B—H16C	109.3
C17A—C16A—H16B	109.3	C17B—C16B—H16D	109.3
C16A—C17A—H17A	109.5	C16B—C17B—H17C	109.6
C16A—C17A—H17B	109.5	C16B—C17B—H17D	109.6
H17A—C17A—H17B	108.1	C16B—C17B—C18B	110.2 (3)
C18A—C17A—C16A	110.6 (3)	H17C—C17B—H17D	108.1
C18A—C17A—H17A	109.5	C18B—C17B—H17C	109.6
C18A—C17A—H17B	109.5	C18B—C17B—H17D	109.6
C17A—C18A—H18A	109.1	C17B—C18B—H18C	109.2
C17A—C18A—H18B	109.1	C17B—C18B—H18D	109.2
C17A—C18A—C19A	112.4 (3)	H18C—C18B—H18D	107.9
H18A—C18A—H18B	107.9	C19B—C18B—C17B	111.8 (3)
C19A—C18A—H18A	109.1	C19B—C18B—H18C	109.2
C19A—C18A—H18B	109.1	C19B—C18B—H18D	109.2
C14A—C19A—H19A	109.7	C14B—C19B—H19C	109.5
C14A—C19A—H19B	109.7	C14B—C19B—H19D	109.5
C18A—C19A—C14A	109.8 (3)	C18B—C19B—C14B	110.9 (3)
C18A—C19A—H19A	109.7	C18B—C19B—H19C	109.5
C18A—C19A—H19B	109.7	C18B—C19B—H19D	109.5
H19A—C19A—H19B	108.2	H19C—C19B—H19D	108.1
N1A—C1A—C2A—C3A	0.2 (4)	N1B—C1B—C2B—C3B	0.3 (4)
N1A—C1A—C6A—O1A	-157.7 (3)	N1B—C1B—C6B—O1B	-158.4 (3)
N1A—C1A—C6A—N2A	24.0 (3)	N1B—C1B—C6B—N2B	23.7 (4)
N1A—C5A—C7A—O2A	-157.1 (3)	N1B—C5B—C7B—O2B	-157.4 (3)
N1A—C5A—C7A—N3A	24.9 (3)	N1B—C5B—C7B—N3B	25.1 (4)
N2A—C8A—C9A—C10A	-179.8 (3)	N2B—C8B—C9B—C10B	-178.4 (2)

N2A—C8A—C13A—C12A	179.2 (3)	N2B—C8B—C13B—C12B	180.0 (3)
N3A—C14A—C15A—C16A	179.9 (2)	N3B—C14B—C15B—C16B	-178.7 (3)
N3A—C14A—C19A—C18A	-179.7 (3)	N3B—C14B—C19B—C18B	179.2 (3)
C1A—N1A—C5A—C4A	1.6 (4)	C1B—N1B—C5B—C4B	2.4 (4)
C1A—N1A—C5A—C7A	-177.5 (2)	C1B—N1B—C5B—C7B	-177.1 (2)
C1A—C2A—C3A—C4A	-0.5 (4)	C1B—C2B—C3B—C4B	-0.7 (4)
C2A—C1A—C6A—O1A	25.3 (4)	C2B—C1B—C6B—O1B	24.9 (4)
C2A—C1A—C6A—N2A	-153.0 (3)	C2B—C1B—C6B—N2B	-152.9 (3)
C2A—C3A—C4A—C5A	1.3 (4)	C2B—C3B—C4B—C5B	1.8 (4)
C3A—C4A—C5A—N1A	-2.0 (4)	C3B—C4B—C5B—N1B	-2.8 (4)
C3A—C4A—C5A—C7A	177.1 (2)	C3B—C4B—C5B—C7B	176.7 (3)
C4A—C5A—C7A—O2A	23.7 (4)	C4B—C5B—C7B—O2B	23.1 (4)
C4A—C5A—C7A—N3A	-154.2 (3)	C4B—C5B—C7B—N3B	-154.5 (3)
C5A—N1A—C1A—C2A	-0.7 (4)	C5B—N1B—C1B—C2B	-1.1 (4)
C5A—N1A—C1A—C6A	-177.5 (2)	C5B—N1B—C1B—C6B	-177.6 (2)
C6A—N2A—C8A—C9A	149.9 (3)	C6B—N2B—C8B—C9B	147.7 (3)
C6A—N2A—C8A—C13A	-86.5 (3)	C6B—N2B—C8B—C13B	-88.8 (3)
C6A—C1A—C2A—C3A	176.9 (3)	C6B—C1B—C2B—C3B	176.9 (3)
C7A—N3A—C14A—C15A	152.6 (3)	C7B—N3B—C14B—C15B	149.6 (3)
C7A—N3A—C14A—C19A	-83.6 (3)	C7B—N3B—C14B—C19B	-87.3 (3)
C8A—N2A—C6A—O1A	-7.3 (5)	C8B—N2B—C6B—O1B	-7.1 (4)
C8A—N2A—C6A—C1A	170.9 (2)	C8B—N2B—C6B—C1B	170.7 (3)
C8A—C9A—C10A—C11A	-54.6 (4)	C8B—C9B—C10B—C11B	-56.3 (4)
C9A—C8A—C13A—C12A	-57.1 (4)	C9B—C8B—C13B—C12B	-57.5 (3)
C9A—C10A—C11A—C12A	53.6 (4)	C9B—C10B—C11B—C12B	54.5 (4)

C10A—C11A—C12A—C13A	-54.5 (4)	C10B—C11B—C12B—C13B	-53.7 (4)
C11A—C12A—C13A—C8A	55.9 (4)	C11B—C12B—C13B—C8B	55.2 (4)
C13A—C8A—C9A—C10A	56.9 (3)	C13B—C8B—C9B—C10B	57.9 (3)
C14A—N3A—C7A—O2A	-7.5 (4)	C14B—N3B—C7B—O2B	-8.9 (4)
C14A—N3A—C7A—C5A	170.4 (2)	C14B—N3B—C7B—C5B	168.5 (2)
C14A—C15A—C16A—C17A	-55.7 (4)	C14B—C15B—C16B—C17B	-57.0 (4)
C15A—C14A—C19A—C18A	-56.4 (4)	C15B—C14B—C19B—C18B	-57.9 (4)
C15A—C16A—C17A—C18A	54.8 (4)	C15B—C16B—C17B—C18B	54.9 (4)
C16A—C17A—C18A—C19A	-55.3 (4)	C16B—C17B—C18B—C19B	-54.6 (4)
C17A—C18A—C19A—C14A	56.0 (4)	C17B—C18B—C19B—C14B	56.3 (4)
C19A—C14A—C15A—C16A	56.8 (3)	C19B—C14B—C15B—C16B	57.7 (3)

Document origin: *publCIF* [Westrip, S. P. (2010). *J. Apply. Cryst.*, **43**, 920-925].

2.7 CIF Check

checkCIF (basic structural check) running

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) 17o_ds_1e2_sadabs

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this report

Structure factor report

Datablock: 17o_ds_1e2_sadabs

Bond precision: C-C = 0.0048 A Wavelength=0.71073

Cell: a=8.3574(2) b=8.3564(2) c=25.9552(7)
alpha=81.439(2) beta=81.446(1) gamma=89.965(1)

Temperature: 173 K

	Calculated	Reported
Volume	1772.09(8)	1772.09(8)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C19 H27 N3 O2	C19 H27 N3 O2
Sum formula	C19 H27 N3 O2	C19 H27 N3 O2
Mr	329.44	329.43
Dx,g cm-3	1.235	1.235
Z	4	4
Mu (mm-1)	0.081	0.081
F000	712.0	712.0
F000'	712.27	
h,k,lmax	11,11,34	11,11,34
Nref	8530	8413
Tmin,Tmax	0.984,0.987	0.626,0.746
Tmin'	0.969	

Correction method= # Reported T Limits: Tmin=0.626 Tmax=0.746
AbsCorr = MULTI-SCAN

Data completeness= 0.986 Theta(max)= 27.997

R(reflections)= 0.0940(6608) wR2(reflections)= 0.2427(8413)

S = 1.035 Npar= 434

The following ALERTS were generated. Each ALERT has the format **test-name_ALERT_alert-type_alert-level**.

Click on the hyperlinks for more details of the test.

Alert level B

PLAT097_ALERT_2_B Large Reported Max. (Positive) Residual Density 1.42 eA-3

Alert level C

DIFMX02_ALERT_1_C The maximum difference density is > 0.1*ZMAX*0.75
The relevant atom site should be identified.
PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual Density .... 3.85 Report
PLAT230_ALERT_2_C Hirshfeld Test Diff for C11A --C12A . 5.3 s.u.
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00483 Ang.
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 55 Report

Alert level G

PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..... 4 Report
PLAT012_ALERT_1_G No _shelx_res_checksum Found in CIF ..... Please Check
PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large 0.18 Report
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..... 8 Note
PLAT870_ALERT_4_G ALERTS Related to Twinning Effects Suppressed .. ! Info
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min). 1 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 61 Note
PLAT931_ALERT_5_G Found Twin Law (1-1 0)[ ] Est. BASF 0.46 Check
PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File ... 2 Note

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0 **ALERT level A** = Most likely a serious problem - resolve or explain 1 **ALERT level B** = A potentially serious problem, consider carefully

5 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
9 **ALERT level G** = General information/check it is not something unexpected

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
5 ALERT type 2 Indicator that the structure model may be wrong or deficient
3 ALERT type 3 Indicator that the structure quality may be low
3 ALERT type 4 Improvement, methodology, query or suggestion
2 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

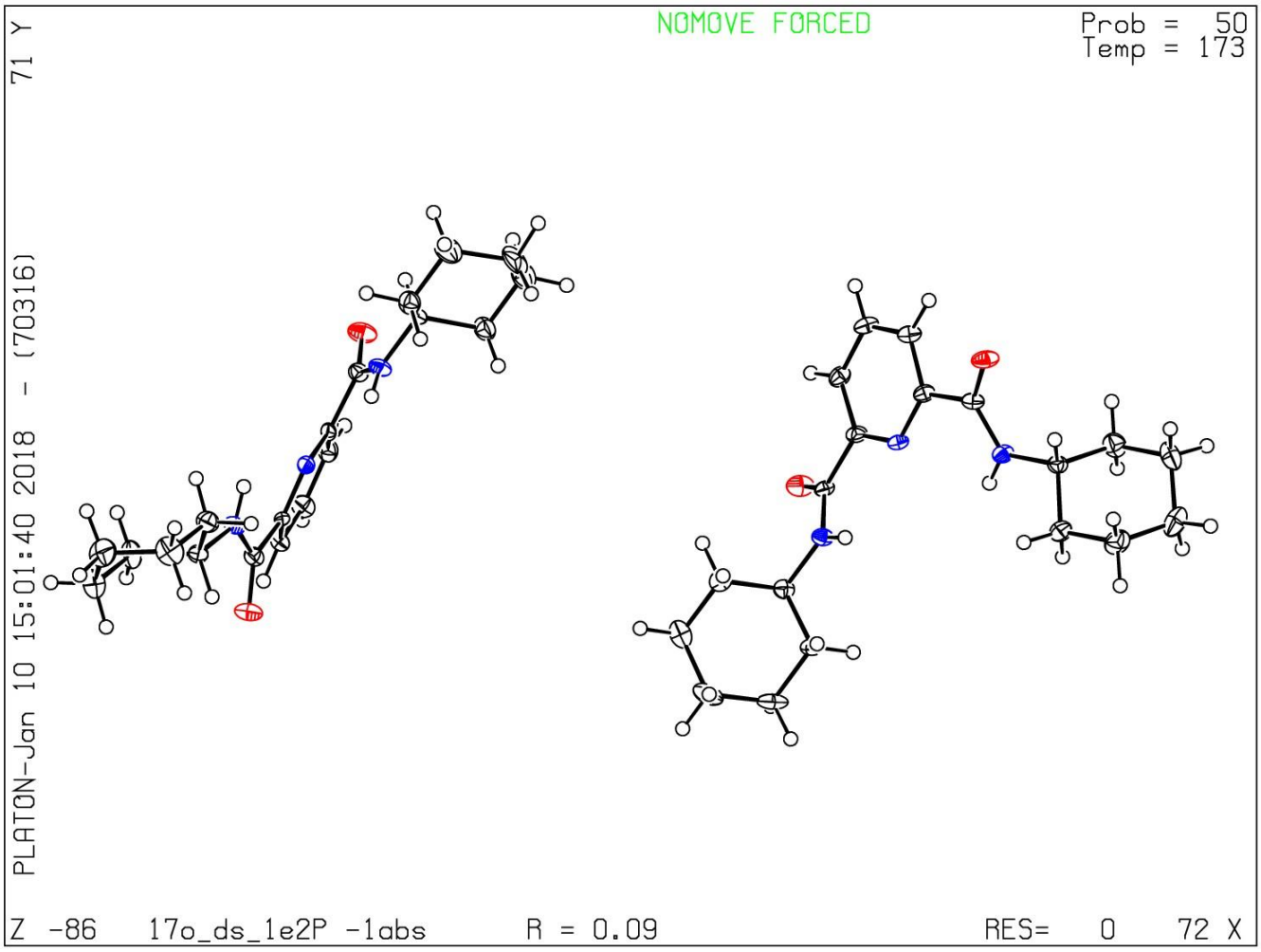
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 13/12/2017; check.def file version of 12/12/2017

Datablock 17o_ds_1e2_sadabs - ellipsoid plot



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Download CIF editor
(enCIFer) from the CCDC
Test a new CIF entry

3.1 log(conc) vs shift (ppm) for 2

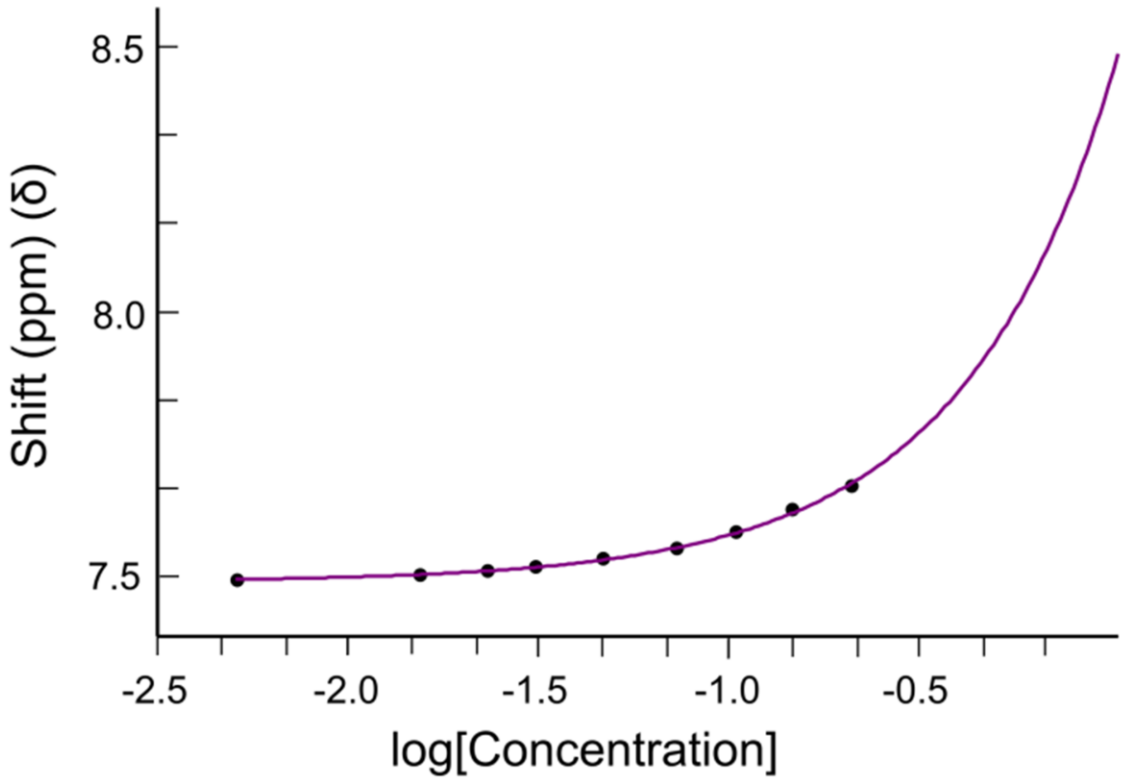


Figure B1. Plot of log[concentration] vs shift (ppm) (δ) for **2**. $A_2 = 8.5$, $A_1 / (0.5 + 0.5 \times (1 + 8 \times 10K_D) \times 0.5) + A_2 \times (1-(0.5 + 0.5 \times (1+8K_D \times 10x) \times 0.5))$ A scaled Levenberg-Marquardt algorithm was used to fit the curve to the data. $A_1 = 7.50$ +/- 0.0018 $K_D = -0.029$ +/- 0.00060 $R^2 = 0.997$

Supplementary data for chapter 4 (appendix B).

Contents:

1. Characterisation data of $[Au(L1)(8\text{-}H\text{-}Quin)]^+$

1.1 FTIR $[Au(L1)(8\text{-}H\text{-}Quin)]^+$

1.2 UV VIS $[Au(L1)(8\text{-}H\text{-}Quin)]^+$

1.3 ^1H NMR $[Au(L1)(8\text{-}H\text{-}Quin)]^+$

1.4 HMRS $[Au(L1)(8\text{-}H\text{-}Quin)]^+$

1.5 X-ray crystallography data $[Au(L1)(8\text{-}H\text{-}Quin)]^+$

1.6 CIF check $[Au(L1)(8\text{-}H\text{-}Quin)]^+$

1.7 ^{13}C NMR $[Au(L1)(8\text{-}H\text{-}Quin)]^+$

2. Characterisation data of [Pd(L1)(8-H-Quin)]

2.1 FTIR [Pd(L1)(8-H-Quin)]

2.2 UV VIS [Pd(L1)(8-H-Quin)]

2.3 ^1H NMR [Pd(L1)(8-H-Quin)]

2.4 HMRS [Pd(L1)(8-H-Quin)]

2.5 X-ray crystallography data [Pd(L1)(8-H-Quin)]

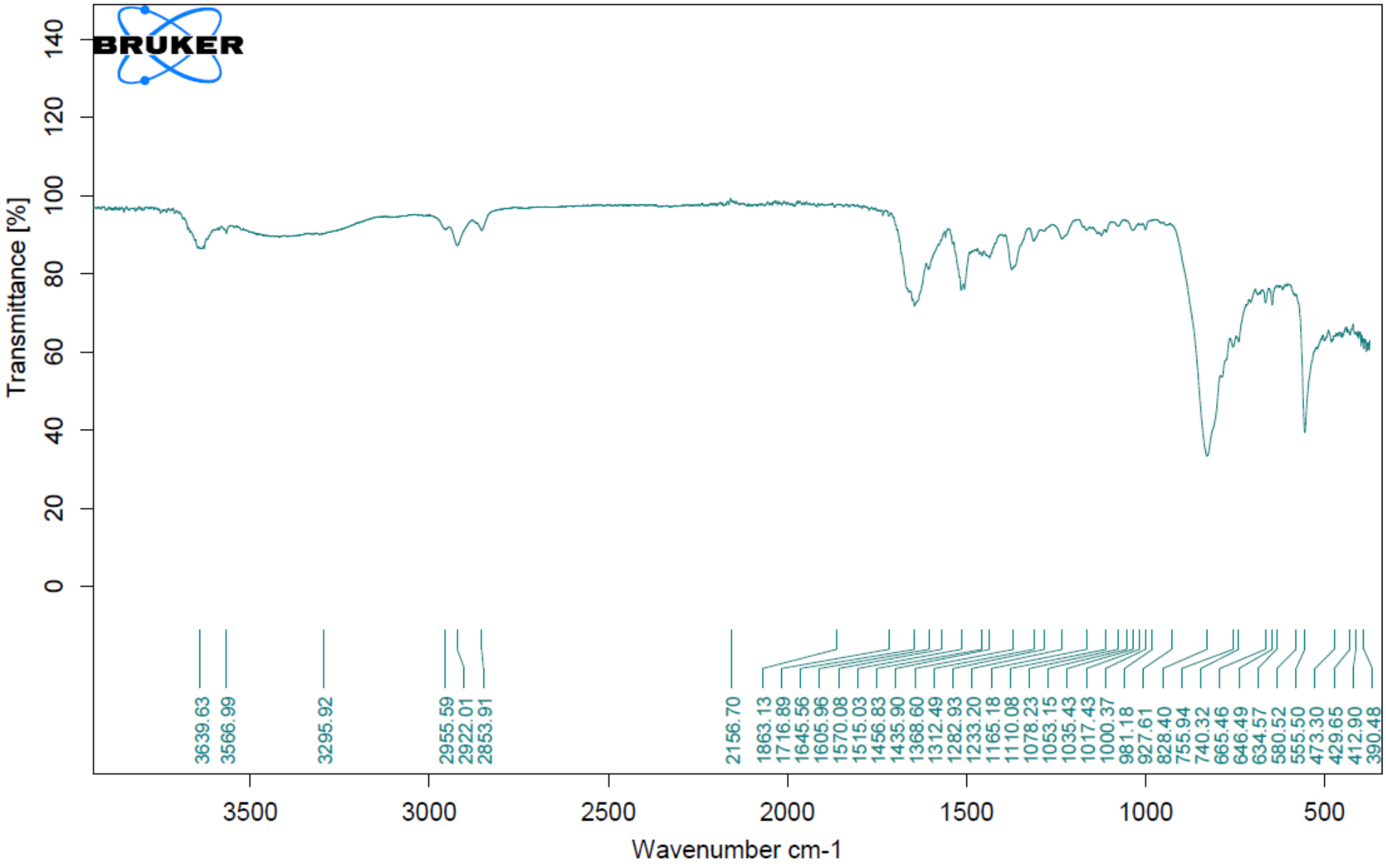
2.6 CIF check [Pd(L1)(8-H-Quin)]

2.6 ^{13}C NMR NMR [Pd(L1)(8-H-Quin)]

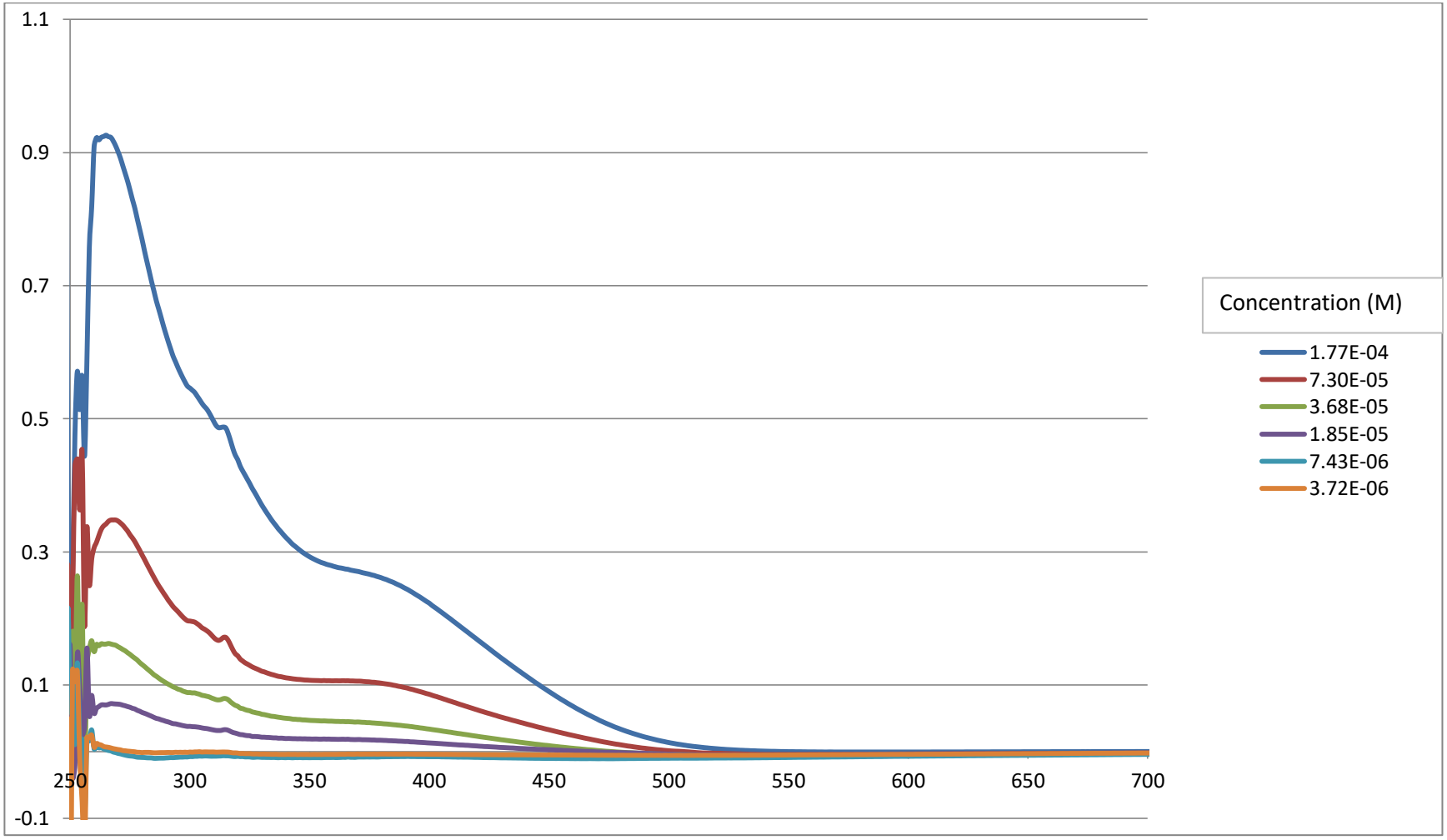
3. Comparative DFT study

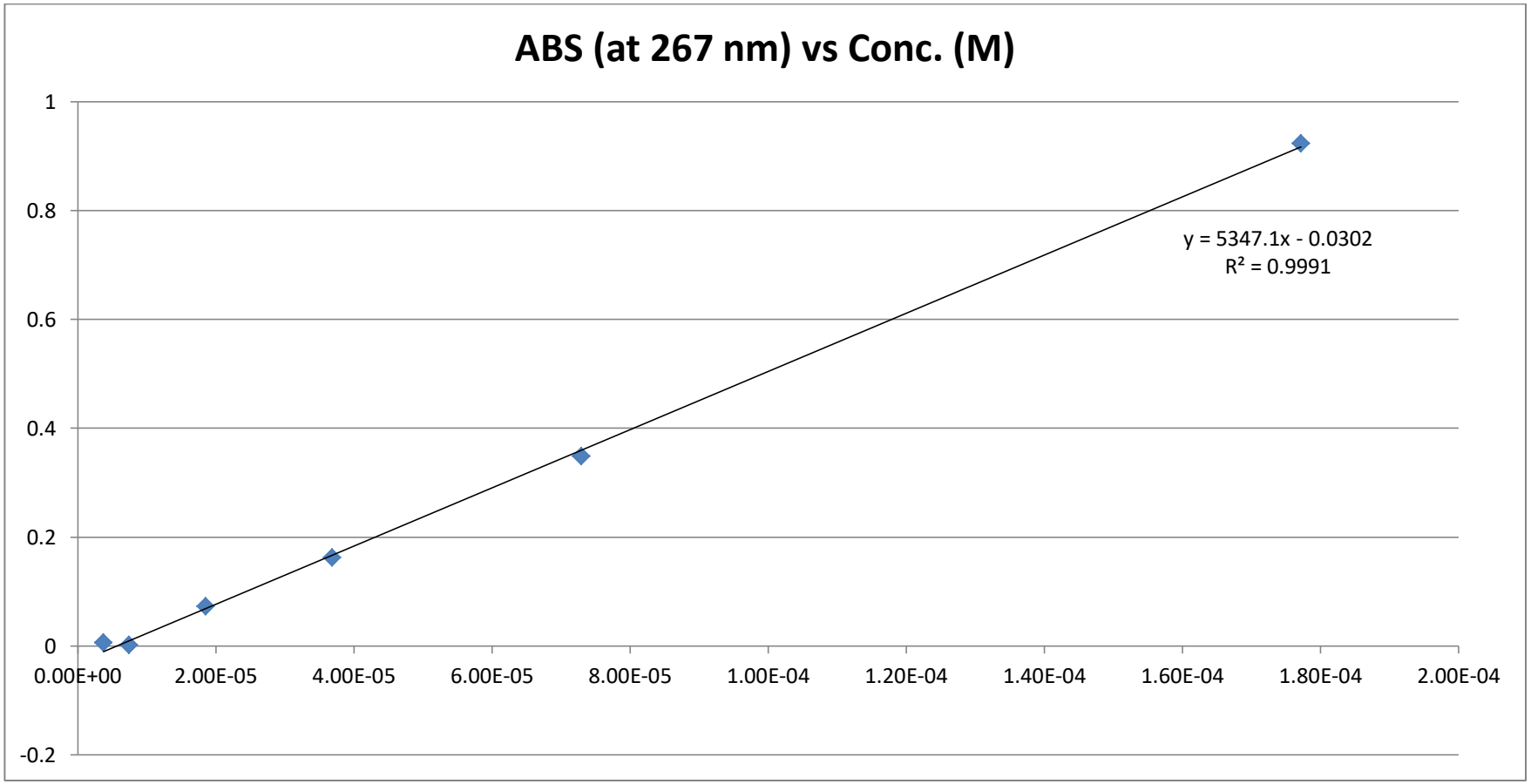
3.1 HSEh1PBE statistical analysis

1.1 FTIR [Au(L1)(8-H-Quin)]$^{+}$

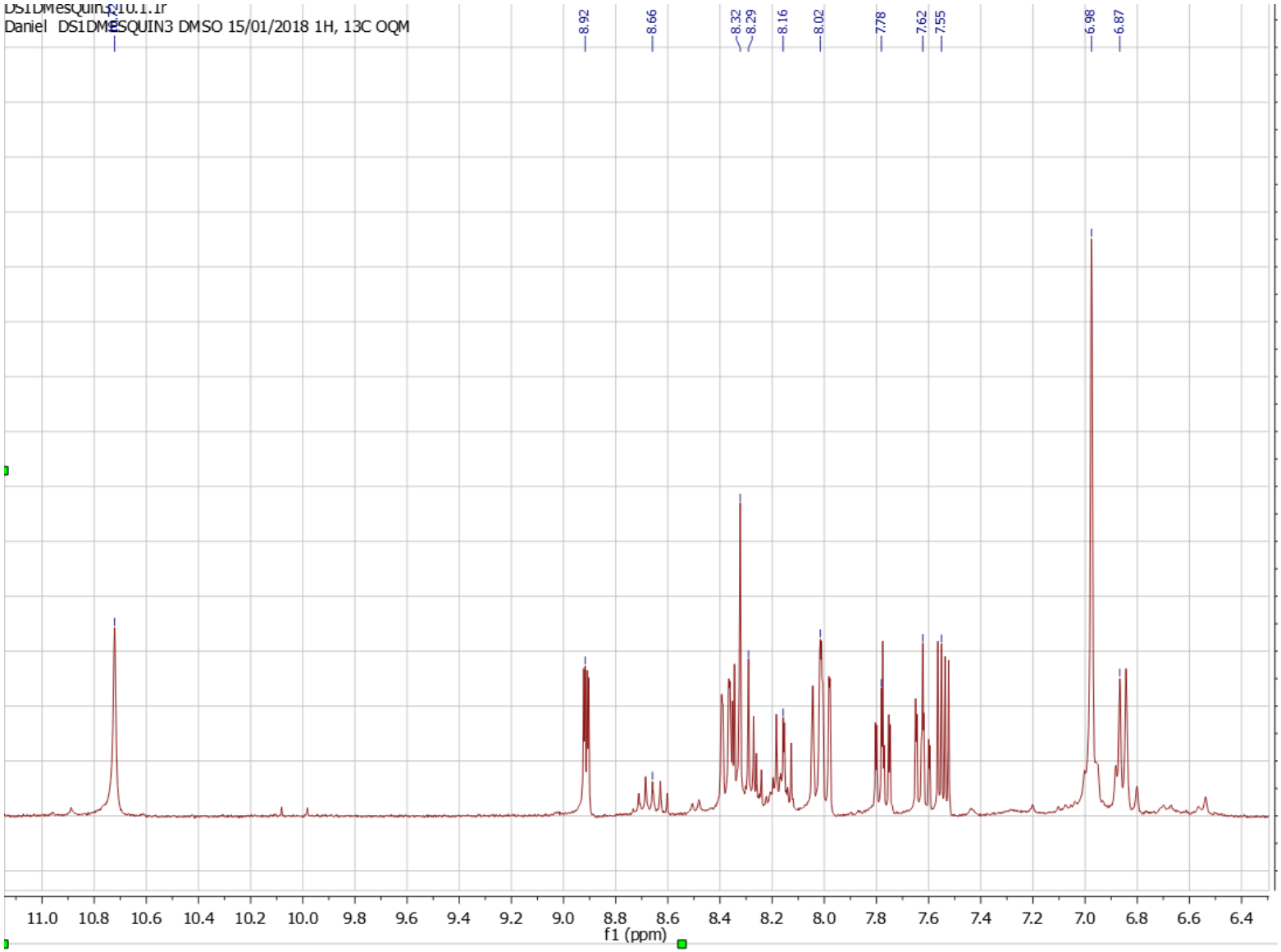


1.2 UV VIS [Au(L1)(8-H-Quin)]$^{+}$

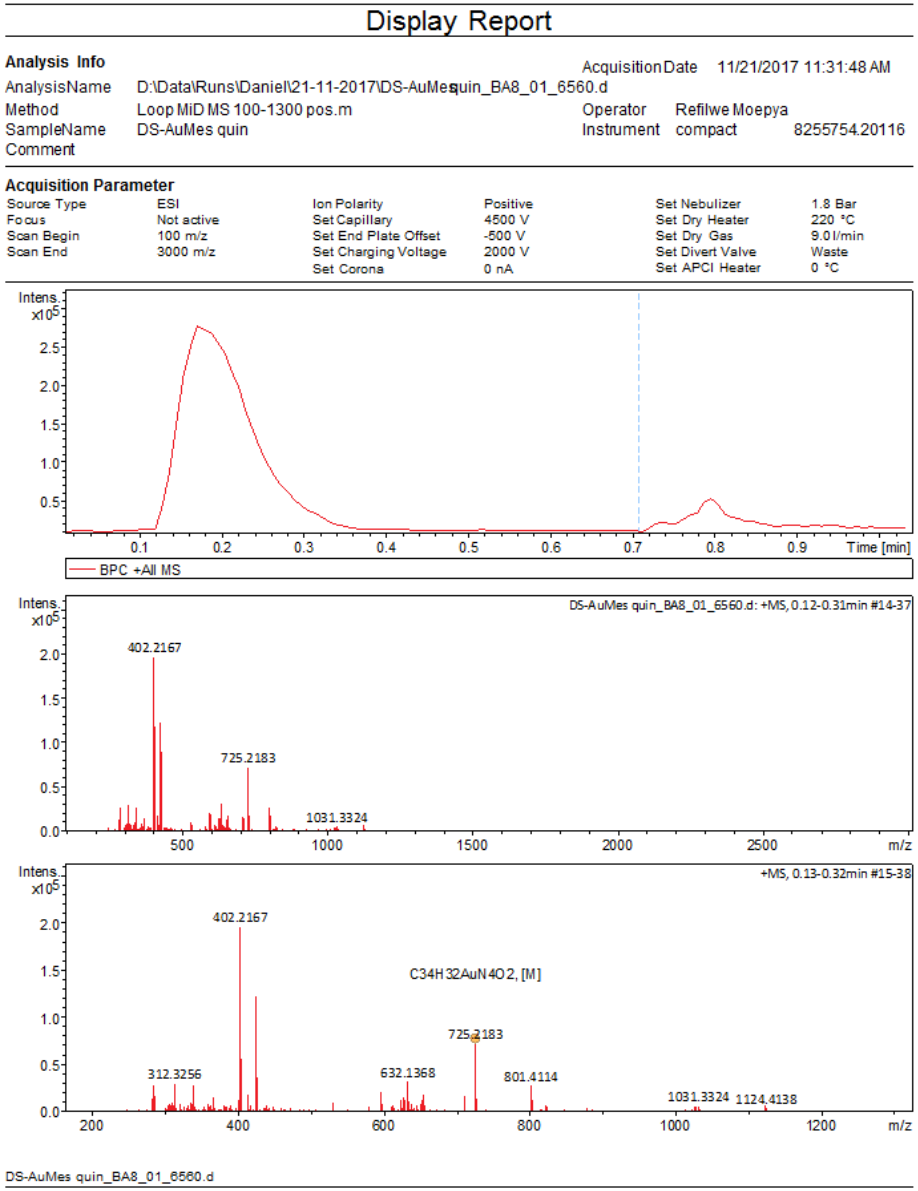



ABS (at 267 nm) vs Conc. (M)
y = 5347.1x - 0.0302
R² = 0.9991
1
0.8
0.6
0.4
0.2
0
-0.2
0.00E+00
2.00E-05
4.00E-05
6.00E-05
8.00E-05
1.00E-04
1.20E-04
1.40E-04
1.60E-04
1.80E-04
2.00E-04

1.4 ^{1}H NMR [Au(L1)(8-H-Quin)]

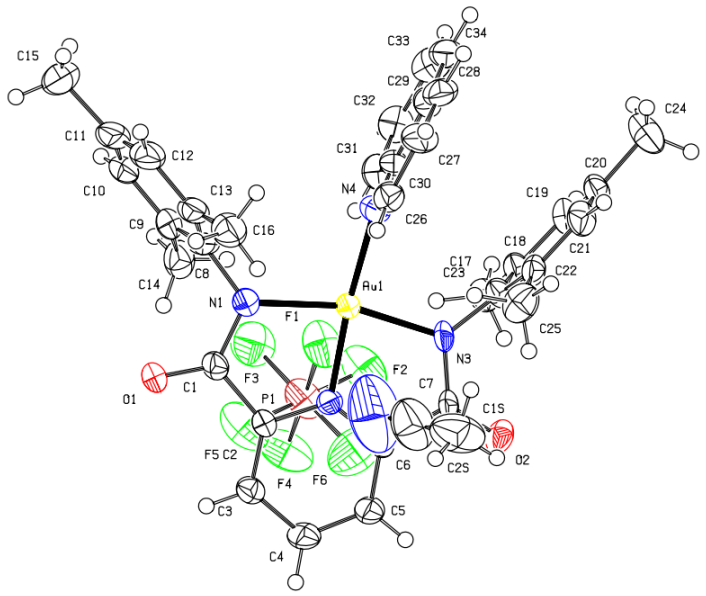


1.5 HMRS [Au(L1)(8-H-Quin)]$^{+}$



1.6 X-ray crystallography data $[Au(L1)(8\text{-H-Quin})]^+$

mo_17O_DS_V5_AuMesQ_CR2_0m



(mo_17o_ds_v5_aumesq_cr2_0m)

Crystal data

$C_{34}H_{32}AuN_4O_2 \cdot F_6P \cdot C_2H_3N$	D_x = 1.704 Mg m^{-3}
M_r = 911.62	Mo $K\alpha$ radiation, λ = 0.71073 Å
Orthorhombic, $Pna2_1$	Cell parameters from 9783 reflections
a = 14.4091 (11) Å	θ = 3.1–27.2°
b = 15.3724 (12) Å	μ = 4.26 mm^{-1}
c = 16.0382 (13) Å	T = 296 K
V = 3552.5 (5) Å^3	Shard, orange
Z = 4	0.20 × 0.19 × 0.18 mm
F(000) = 1800	

Data collection

Bruker D8 Venture Photon I area detector diffractometer	7887 independent reflections
Radiation source: microfocus sealed X-ray tube, Incoatec Iμs	6562 reflections with $I > 2\sigma(I)$
Mirror optics monochromator	R_{int} = 0.066
Detector resolution: 7.9 pixels mm^{-1}	θ_{max} = 27.3°, θ_{min} = 2.9°
ω and ϕ scans	h = -18→18
Absorption correction: multi-scan *SADABS2016*/2 (Bruker,2016/2) was used for absorption correction. wR2(int) was 0.1050 before and 0.0909 after correction. The Ratio of	k = -19→19

minimum to maximum transmission is 0.8381. The λ/2 correction factor is Not present.	
T_{min} = 0.625, T_{max} = 0.746	l = -20→20
61859 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.049	$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 25.7909P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.130	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.05	$\Delta\rangle_{max}$ = 6.52 e Å^{-3}
7887 reflections	$\Delta\rangle_{min}$ = -2.44 e Å^{-3}
462 parameters	Absolute structure: Flack x determined using 2842 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
25 restraints	Absolute structure parameter: -0.003 (5)
Primary atom site location: structure-invariant direct methods	

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. Refined as a 2-component twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Au1	0.70885 (2)	0.01699 (2)	0.14077 (5)	0.02873 (14)
P1	0.7631 (4)	-0.1915 (3)	-0.0638 (3)	0.0521 (10)
N4	0.6426 (7)	0.1335 (7)	0.1311 (9)	0.036 (3)
N2	0.7828 (6)	-0.0867 (7)	0.1620 (6)	0.030 (3)
F1	0.7259 (8)	-0.1048 (7)	-0.0187 (6)	0.064 (3)
F2	0.7964 (9)	-0.1345 (8)	-0.1406 (7)	0.076 (4)
F5	0.7289 (10)	-0.2455 (8)	0.0140 (6)	0.080 (4)
F4	0.7973 (9)	-0.2759 (8)	-0.1088 (7)	0.078 (4)
F6	0.8609 (9)	-0.1784 (11)	-0.0200 (8)	0.098 (4)
C30	0.6185 (10)	0.1691 (10)	0.0586 (10)	0.033 (3)
C28	0.5936 (9)	0.3080 (9)	0.1279 (13)	0.048 (5)
H28	0.5788	0.3669	0.1263	0.058*
C26	0.6417 (11)	0.1834 (11)	0.1982 (11)	0.039 (4)

H26	0.6605	0.1582	0.2481	0.047*
C27	0.6149 (13)	0.2707 (11)	0.2005 (12)	0.045 (4)
H27	0.6120	0.3014	0.2505	0.053*
C29	0.5934 (12)	0.2574 (11)	0.0508 (10)	0.040 (4)
F3	0.6623 (9)	-0.2017 (8)	-0.1050 (8)	0.078 (3)
C6	0.8735 (7)	-0.0845 (6)	0.1461 (14)	0.032 (3)
C2	0.7384 (9)	-0.1570 (8)	0.1950 (7)	0.028 (2)
C3	0.7887 (9)	-0.2281 (9)	0.2142 (9)	0.034 (3)
H3	0.7602	-0.2767	0.2374	0.041*
C5	0.9257 (9)	-0.1539 (8)	0.1642 (8)	0.036 (3)
H5	0.9891	-0.1526	0.1538	0.043*
C4	0.8854 (10)	-0.2281 (9)	0.1986 (9)	0.038 (3)
H4	0.9213	-0.2766	0.2111	0.046*
O1	0.5862 (7)	-0.2020 (6)	0.2329 (6)	0.039 (2)
O2	0.9840 (7)	0.0123 (6)	0.0864 (8)	0.046 (3)
N3	0.8328 (7)	0.0626 (7)	0.1000 (7)	0.0352 (19)
N1	0.6046 (8)	-0.0600 (7)	0.1841 (7)	0.036 (2)
C1	0.6351 (9)	-0.1424 (9)	0.2072 (8)	0.031 (3)
C7	0.9035 (9)	0.0029 (8)	0.1089 (9)	0.0352 (19)
C22	0.8560 (9)	0.2174 (9)	0.1144 (9)	0.034 (3)
C17	0.8465 (10)	0.1444 (9)	0.0646 (8)	0.034 (3)
C23	0.8391 (11)	0.0743 (10)	-0.0790 (9)	0.040 (3)
H23A	0.9001	0.0495	-0.0828	0.059*
H23B	0.8187	0.0915	-0.1336	0.059*
H23C	0.7970	0.0320	-0.0566	0.059*
C18	0.8417 (9)	0.1523 (9)	-0.0230 (9)	0.035 (3)
C19	0.8407 (10)	0.2363 (9)	-0.0571 (9)	0.039 (3)
H19	0.8372	0.2425	-0.1147	0.047*
C21	0.8540 (10)	0.2994 (9)	0.0771 (10)	0.042 (3)
H21	0.8589	0.3486	0.1106	0.050*
C20	0.8450 (10)	0.3096 (9)	-0.0084 (10)	0.040 (3)
C25	0.8715 (13)	0.2078 (14)	0.2076 (10)	0.054 (5)
H25A	0.8181	0.1810	0.2325	0.081*
H25B	0.8811	0.2641	0.2319	0.081*
H25C	0.9251	0.1721	0.2173	0.081*
C31	0.6148 (11)	0.1173 (10)	-0.0174 (9)	0.043 (3)
H31	0.6303	0.0586	-0.0158	0.052*
C34	0.5685 (11)	0.2953 (11)	-0.0247 (10)	0.047 (4)
H34	0.5535	0.3540	-0.0276	0.057*
C32	0.5893 (11)	0.1541 (11)	-0.0891 (10)	0.047 (4)
H32	0.5860	0.1200	-0.1370	0.056*
C33	0.5666 (12)	0.2448 (13)	-0.0944 (12)	0.059 (4)
H33	0.5507	0.2691	-0.1455	0.070*

C13	0.4631 (11)	0.0017 (10)	0.2426 (10)	0.043 (4)
C8	0.5071 (10)	-0.0450 (10)	0.1794 (9)	0.038 (3)
C9	0.4571 (10)	-0.0824 (10)	0.1138 (9)	0.043 (4)
C12	0.3655 (12)	0.0108 (11)	0.2387 (12)	0.052 (4)
H12	0.3346	0.0421	0.2798	0.063*
C10	0.3612 (11)	-0.0731 (10)	0.1111 (11)	0.053 (4)
H10	0.3276	-0.0977	0.0676	0.064*
C14	0.5038 (11)	-0.1325 (11)	0.0461 (10)	0.051 (4)
H14A	0.5508	-0.0969	0.0207	0.076*
H14B	0.4589	-0.1489	0.0048	0.076*
H14C	0.5319	-0.1837	0.0691	0.076*
C11	0.3154 (13)	-0.0267 (12)	0.1738 (13)	0.060 (5)
C16	0.5137 (12)	0.0425 (11)	0.3133 (11)	0.052 (4)
H16A	0.5788	0.0298	0.3087	0.078*
H16B	0.4905	0.0198	0.3650	0.078*
H16C	0.5047	0.1044	0.3120	0.078*
C15	0.2095 (11)	-0.0210 (14)	0.1722 (15)	0.072 (7)
H15A	0.1890	0.0190	0.2143	0.107*
H15B	0.1836	-0.0774	0.1829	0.107*
H15C	0.1895	-0.0009	0.1185	0.107*
C24	0.8400 (15)	0.3978 (11)	-0.0467 (12)	0.062 (5)
H24A	0.9012	0.4166	-0.0617	0.093*
H24B	0.8137	0.4379	-0.0074	0.093*
H24C	0.8017	0.3956	-0.0957	0.093*
C2S	0.877 (2)	0.047 (2)	0.3694 (18)	0.090 (8)
N1S	0.831 (3)	0.005 (2)	0.326 (2)	0.152 (12)
C1S	0.9271 (19)	0.1008 (15)	0.4235 (15)	0.090 (7)
H1SA	0.9896	0.1067	0.4035	0.136*
H1SB	0.9280	0.0757	0.4783	0.136*
H1SC	0.8982	0.1570	0.4258	0.136*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Au1	0.0258 (2)	0.0244 (2)	0.0359 (2)	-0.00007 (15)	0.0031 (4)	0.0038 (4)
P1	0.079 (3)	0.042 (2)	0.0352 (19)	-0.005 (2)	-0.003 (2)	-0.0068 (17)
N4	0.039 (5)	0.031 (5)	0.036 (8)	0.006 (4)	0.005 (6)	0.005 (6)
N2	0.026 (5)	0.021 (4)	0.043 (8)	0.000 (4)	-0.005 (4)	-0.005 (4)
F1	0.086 (7)	0.051 (6)	0.056 (6)	-0.010 (5)	0.019 (5)	-0.018 (5)
F2	0.117 (11)	0.058 (7)	0.054 (6)	0.002 (6)	0.026 (6)	-0.001 (5)
F5	0.133 (11)	0.068 (7)	0.039 (5)	0.002 (7)	0.007 (6)	0.010 (5)
F4	0.131 (11)	0.054 (7)	0.051 (6)	0.020 (7)	-0.009 (7)	-0.013 (5)
F6	0.076 (8)	0.135 (13)	0.081 (9)	0.005 (8)	-0.016 (7)	-0.025 (8)

C30	0.029 (7)	0.023 (7)	0.046 (9)	0.006 (6)	0.008 (6)	-0.004 (6)
C28	0.034 (6)	0.037 (7)	0.073 (14)	0.008 (5)	-0.008 (8)	-0.009 (9)
C26	0.035 (8)	0.039 (9)	0.044 (9)	0.001 (7)	0.003 (6)	-0.001 (7)
C27	0.050 (10)	0.031 (8)	0.052 (10)	0.008 (7)	-0.009 (8)	-0.012 (7)
C29	0.037 (8)	0.034 (8)	0.048 (9)	0.000 (6)	0.007 (7)	0.007 (7)
F3	0.087 (9)	0.066 (7)	0.082 (8)	-0.008 (7)	-0.022 (7)	-0.012 (6)
C6	0.032 (5)	0.015 (4)	0.047 (7)	0.001 (4)	0.008 (8)	-0.012 (8)
C2	0.031 (6)	0.024 (6)	0.028 (6)	-0.003 (5)	0.001 (5)	0.002 (5)
C3	0.036 (7)	0.026 (6)	0.039 (7)	0.002 (5)	0.002 (6)	0.000 (5)
C5	0.033 (6)	0.028 (6)	0.046 (9)	0.004 (5)	0.001 (5)	-0.006 (5)
C4	0.038 (7)	0.030 (7)	0.046 (8)	0.011 (6)	0.004 (6)	-0.007 (6)
O1	0.036 (5)	0.029 (5)	0.052 (6)	-0.005 (4)	0.001 (4)	0.010 (4)
O2	0.029 (5)	0.034 (5)	0.076 (7)	-0.001 (4)	0.016 (5)	-0.015 (5)
N3	0.030 (4)	0.027 (4)	0.049 (5)	-0.014 (3)	-0.005 (4)	0.007 (4)
N1	0.032 (6)	0.030 (6)	0.045 (6)	0.000 (5)	0.004 (5)	0.004 (5)
C1	0.035 (6)	0.033 (7)	0.025 (6)	-0.004 (6)	-0.004 (5)	0.008 (5)
C7	0.030 (4)	0.027 (4)	0.049 (5)	-0.014 (3)	-0.005 (4)	0.007 (4)
C22	0.027 (6)	0.027 (6)	0.047 (9)	-0.006 (5)	0.006 (5)	-0.007 (5)
C17	0.040 (7)	0.025 (6)	0.037 (7)	-0.004 (6)	-0.002 (6)	0.000 (5)
C23	0.046 (8)	0.033 (7)	0.040 (8)	-0.001 (7)	0.005 (6)	-0.007 (6)
C18	0.035 (7)	0.027 (6)	0.042 (7)	-0.005 (5)	0.002 (6)	0.001 (6)
C19	0.042 (8)	0.035 (7)	0.041 (7)	-0.013 (6)	0.005 (6)	0.001 (6)
C21	0.044 (8)	0.029 (7)	0.051 (8)	-0.006 (6)	0.008 (7)	-0.013 (6)
C20	0.042 (7)	0.024 (6)	0.055 (9)	-0.005 (6)	0.008 (6)	0.001 (6)
C25	0.047 (10)	0.074 (13)	0.040 (9)	-0.002 (9)	0.009 (8)	0.000 (9)
C31	0.048 (8)	0.040 (8)	0.041 (8)	-0.006 (7)	-0.002 (6)	-0.008 (6)
C34	0.043 (8)	0.047 (9)	0.053 (9)	0.011 (7)	0.001 (7)	0.012 (7)
C32	0.051 (9)	0.050 (9)	0.039 (8)	-0.002 (7)	0.002 (7)	-0.008 (7)
C33	0.048 (9)	0.072 (12)	0.056 (10)	0.007 (9)	-0.001 (8)	0.002 (9)
C13	0.040 (8)	0.038 (8)	0.052 (8)	0.011 (6)	0.013 (7)	0.020 (6)
C8	0.032 (7)	0.039 (7)	0.044 (7)	0.014 (6)	0.007 (6)	0.017 (6)
C9	0.038 (7)	0.039 (7)	0.054 (9)	-0.005 (6)	0.003 (6)	0.020 (6)
C12	0.047 (9)	0.046 (9)	0.064 (10)	0.017 (7)	0.021 (8)	0.018 (8)
C10	0.043 (8)	0.038 (7)	0.078 (12)	-0.003 (7)	-0.015 (7)	0.029 (7)
C14	0.052 (9)	0.051 (10)	0.048 (8)	-0.016 (8)	-0.011 (7)	0.005 (7)
C11	0.045 (9)	0.056 (10)	0.079 (12)	0.012 (8)	0.007 (8)	0.041 (9)
C16	0.053 (9)	0.045 (9)	0.057 (9)	0.006 (8)	0.013 (8)	0.003 (8)
C15	0.038 (9)	0.085 (15)	0.092 (16)	0.008 (8)	0.002 (8)	0.034 (11)
C24	0.083 (13)	0.037 (9)	0.067 (12)	-0.004 (9)	0.010 (10)	0.011 (8)
C2S	0.114 (13)	0.082 (12)	0.075 (11)	-0.021 (11)	-0.009 (10)	0.008 (10)
N1S	0.21 (2)	0.15 (2)	0.094 (16)	-0.077 (18)	-0.006 (18)	-0.009 (15)
C1S	0.113 (17)	0.077 (13)	0.081 (14)	0.017 (13)	-0.044 (13)	0.002 (11)

Geometric parameters (Å, º) for (mo_17o_ds_v5_aumesq_cr2_0m)

Au1—N4	2.035 (10)	N3—C7	1.379 (18)
Au1—N2	1.948 (10)	N3—C17	1.395 (17)
Au1—N3	2.027 (10)	N1—C1	1.391 (17)
Au1—N1	2.035 (11)	N1—C8	1.426 (17)
P1—F1	1.609 (11)	C22—C17	1.384 (18)
P1—F2	1.585 (12)	C22—C21	1.40 (2)
P1—F5	1.579 (12)	C22—C25	1.52 (2)
P1—F4	1.564 (12)	C17—C18	1.412 (19)
P1—F6	1.587 (13)	C23—C18	1.498 (19)
P1—F3	1.603 (13)	C18—C19	1.402 (19)
N4—C30	1.33 (2)	C19—C20	1.37 (2)
N4—C26	1.32 (2)	C21—C20	1.39 (2)
N2—C6	1.332 (14)	C20—C24	1.49 (2)
N2—C2	1.363 (16)	C31—C32	1.33 (2)
C30—C29	1.41 (2)	C34—C33	1.36 (2)
C30—C31	1.46 (2)	C32—C33	1.43 (2)
C28—C27	1.33 (3)	C13—C8	1.39 (2)
C28—C29	1.46 (2)	C13—C12	1.42 (2)
C26—C27	1.40 (2)	C13—C16	1.49 (2)
C29—C34	1.39 (2)	C8—C9	1.40 (2)
C6—C5	1.337 (17)	C9—C10	1.39 (2)
C6—C7	1.532 (18)	C9—C14	1.49 (2)
C2—C3	1.347 (18)	C12—C11	1.39 (3)
C2—C1	1.517 (18)	C10—C11	1.40 (3)
C3—C4	1.415 (19)	C11—C15	1.53 (2)
C5—C4	1.39 (2)	C2S—N1S	1.15 (4)
O1—C1	1.228 (16)	C2S—C1S	1.41 (4)
O2—C7	1.224 (17)		
N2—Au1—N4	172.0 (5)	C7—N3—Au1	112.8 (8)
N2—Au1—N3	81.8 (4)	C7—N3—C17	122.6 (11)
N2—Au1—N1	82.4 (4)	C17—N3—Au1	124.6 (9)
N3—Au1—N4	94.8 (4)	C1—N1—Au1	112.8 (8)
N3—Au1—N1	164.1 (5)	C1—N1—C8	118.2 (11)
N1—Au1—N4	101.1 (4)	C8—N1—Au1	128.0 (9)
F2—P1—F1	89.6 (6)	O1—C1—C2	119.7 (12)
F2—P1—F6	90.3 (8)	O1—C1—N1	126.0 (12)
F2—P1—F3	90.5 (7)	N1—C1—C2	114.3 (11)
F5—P1—F1	88.6 (6)	O2—C7—C6	119.1 (12)
F5—P1—F2	178.2 (7)	O2—C7—N3	126.2 (12)
F5—P1—F6	89.6 (8)	N3—C7—C6	114.6 (11)
F5—P1—F3	89.5 (7)	C17—C22—C21	118.8 (13)
F4—P1—F1	178.7 (7)	C17—C22—C25	120.3 (15)

F4—P1—F2	90.3 (7)	C21—C22—C25	120.8 (14)
F4—P1—F5	91.5 (7)	N3—C17—C18	118.4 (12)
F4—P1—F6	91.8 (8)	C22—C17—N3	120.6 (12)
F4—P1—F3	90.8 (7)	C22—C17—C18	120.7 (13)
F6—P1—F1	89.5 (7)	C17—C18—C23	122.0 (12)
F6—P1—F3	177.3 (8)	C19—C18—C17	117.9 (13)
F3—P1—F1	88.0 (7)	C19—C18—C23	120.2 (13)
C30—N4—Au1	123.4 (10)	C20—C19—C18	122.3 (14)
C26—N4—Au1	117.0 (11)	C20—C21—C22	121.9 (13)
C26—N4—C30	118.1 (12)	C19—C20—C21	118.3 (13)
C6—N2—Au1	118.8 (9)	C19—C20—C24	120.7 (15)
C6—N2—C2	123.8 (11)	C21—C20—C24	121.0 (14)
C2—N2—Au1	117.4 (8)	C32—C31—C30	120.0 (15)
N4—C30—C29	122.6 (14)	C33—C34—C29	118.8 (16)
N4—C30—C31	121.1 (13)	C31—C32—C33	121.7 (15)
C29—C30—C31	116.3 (14)	C34—C33—C32	120.1 (17)
C27—C28—C29	120.7 (13)	C8—C13—C12	118.0 (17)
N4—C26—C27	125.7 (16)	C8—C13—C16	123.3 (14)
C28—C27—C26	116.9 (16)	C12—C13—C16	118.7 (15)
C30—C29—C28	116.0 (15)	C13—C8—N1	119.5 (14)
C34—C29—C30	123.0 (15)	C13—C8—C9	121.6 (13)
C34—C29—C28	121.0 (15)	C9—C8—N1	118.8 (13)
N2—C6—C5	119.3 (13)	C8—C9—C14	121.8 (13)
N2—C6—C7	112.0 (10)	C10—C9—C8	119.6 (15)
C5—C6—C7	128.7 (11)	C10—C9—C14	118.6 (15)
N2—C2—C1	113.1 (10)	C11—C12—C13	120.5 (17)
C3—C2—N2	118.6 (12)	C9—C10—C11	120.0 (17)
C3—C2—C1	128.3 (12)	C12—C11—C10	120.3 (16)
C2—C3—C4	119.3 (13)	C12—C11—C15	120.4 (19)
C6—C5—C4	120.3 (12)	C10—C11—C15	119 (2)
C5—C4—C3	118.7 (13)	N1S—C2S—C1S	176 (4)

Document origin: *publCIF* [Westrip, S. P. (2010). *J. Apply. Cryst.*, **43**, 920-925].

1.6 CIF check [Au(L1)(8-H-Quin)]$^{+}$

checkCIF (basic structural check) running

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) mo_17o_ds_v5_aumesq_cr2_0m

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this

report Structure factor report

Datablock: mo_17o_ds_v5_aumesq_cr2_0m

```
Bond precision:     C-C = 0.0219 A
           Wavelength=0.71073 Cell:       a=14.4091(11)
           b=15.3724(12)   c=16.0382(13)
           alpha=90        beta=90        gamma=90
Temperature: 296 K
                  Calculated                    Reported
Volume            3552.5(5)                     3552.5(5)
Space group       P n a 21                      P n a 21
Hall group        P 2c -2n                      P 2c -2n
Moiety formula    C34 H32 Au N4 O2, F6 P, C2  H3 N C34 H32 Au N4 O2, F6 P,
C2  H3 N Sum formula                            C36 H35 Au F6 N5 O2 P    C36
H35 Au F6 N5 O2  P
Mr                911.63                       911.62
Dx,g cm-3         1.704                        1.704
Z                 4                            4
Mu  (mm-1)        4.258                        4.258
F000              1800.0                       1800.0
F000'            1793.39
h,k,lmax          18,19,20                     18,19,20
Nref              7991[ 4140]                  7887
Tmin,Tmax         0.448,0.463                  0.625,0.746
Tmin'             0.414
Correction method= # Reported T Limits: Tmin=0.625
Tmax=0.746 AbsCorr = MULTI-SCAN
Data completeness= 1.91/0.99   Theta(max)= 27.278
R(reflections)= 0.0492( 6562)     wR2(reflections)= 0.1296( 7887)
S = 1.049            Npar= 462
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The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level. Click on the

hyperlinks for more details of the test.

Alert level B

PLAT342_ALERT_3_B Low Bond Precision on C-C Bonds ..... 0.02194 Ang.

Alert level C

DIFMX02_ALERT_1_C The maximum difference density is > 0.1*ZMAX*0.75
The relevant atom site should be identified.
PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual Density ....
2.67 Report PLAT097_ALERT_2_C Large Reported
Max. (Positive) Residual Density 6.52 eA-3
PLAT244_ALERT_4_C Low 'Solvent' Ueq as Compared to Neighbors of
C2S Check PLAT369_ALERT_2_C Long C(sp2)-
C(sp2) Bond C6 - C7 .. 1.53 Ang.
PLAT910_ALERT_3_C Missing # of FCF Reflection(s) Below Theta(Min) 6 Note

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Alert level G

PLAT003_ALERT_2_G Number of Uiso or Uij Restrained non-H Atoms ... 3 Report
PLAT012_ALERT_1_G No _shelx_res_checksum found in CIF ..... Please
Check PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT Unusually
Large 25.79 Why ? PLAT171_ALERT_4_G The CIF-Embedded
.res File Contains EADP Records 1 Report
PLAT178_ALERT_4_G The CIF-Embedded .res File Contains SIMU Records
1 Report PLAT186_ALERT_4_G The CIF-Embedded .res
File Contains ISOR Records 1 Report
PLAT244_ALERT_4_G Low 'Solvent'
Ueq as Compared to Neighbors of P1 Check
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels 3
Note

PLAT790_ALERT_4_G Centre of Gravity not Within Unit Cell: Resd. #
2
Note F6 P
PLAT860_ALERT_3_G Number of Least-Squares Restraints 25
Note PLAT870_ALERT_4_G ALERTS Related to Twinning Effects Suppressed
.. ! Info

PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600
31
Note PLAT933_ALERT_2_G Number of OMIT Records in Embedded .res File
... 3
Note

0 **ALERT level A** = Most likely a serious problem - resolve or explain 1 **ALERT level B** = A potentially serious problem, consider carefully

6 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight 13 **ALERT level G** = General information/check it is not something unexpected

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
6 ALERT type 2 Indicator that the structure model may be wrong or deficient 3 ALERT type 3 Indicator that the structure quality may be low
9 ALERT type 4 Improvement, methodology, query or suggestion 0 ALERT type 5 Informative message, check

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It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

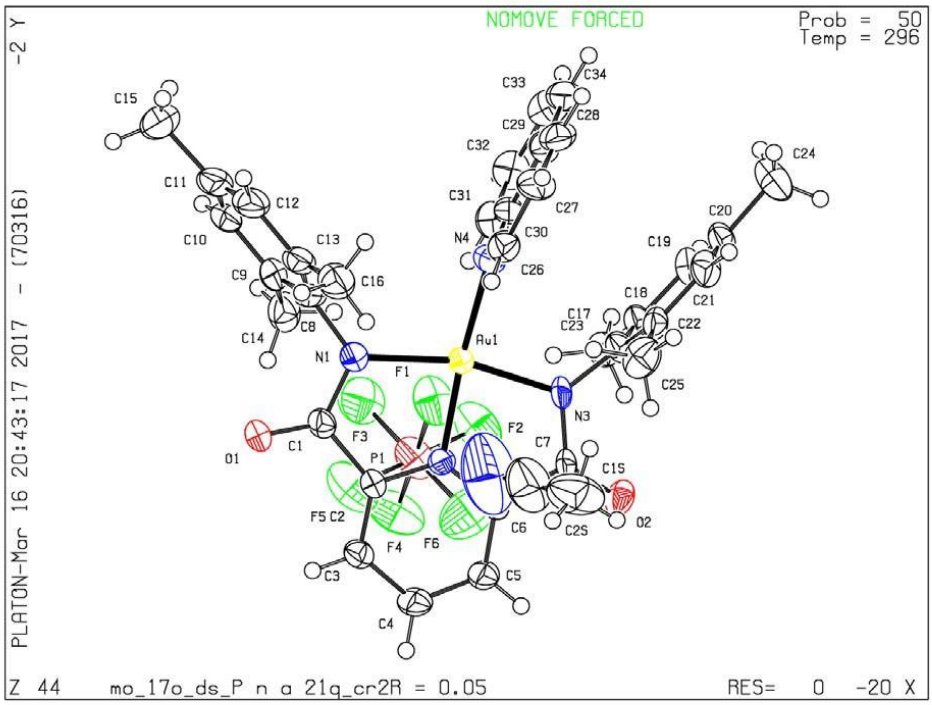
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 26/02/2017; check.def file version of 21/02/2017

Datablock mo_17o_ds_v5_aumesq_cr2_0m - ellipsoid plot



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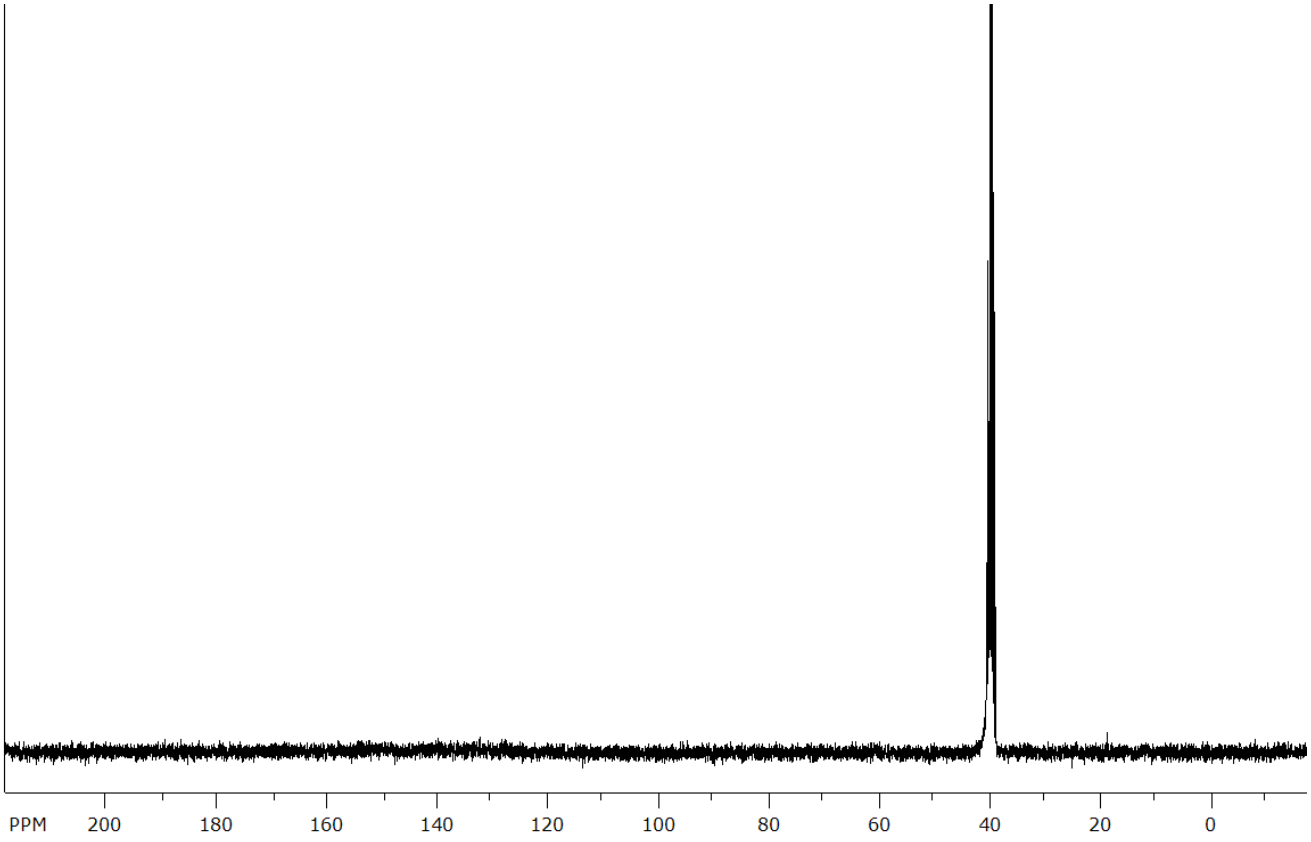
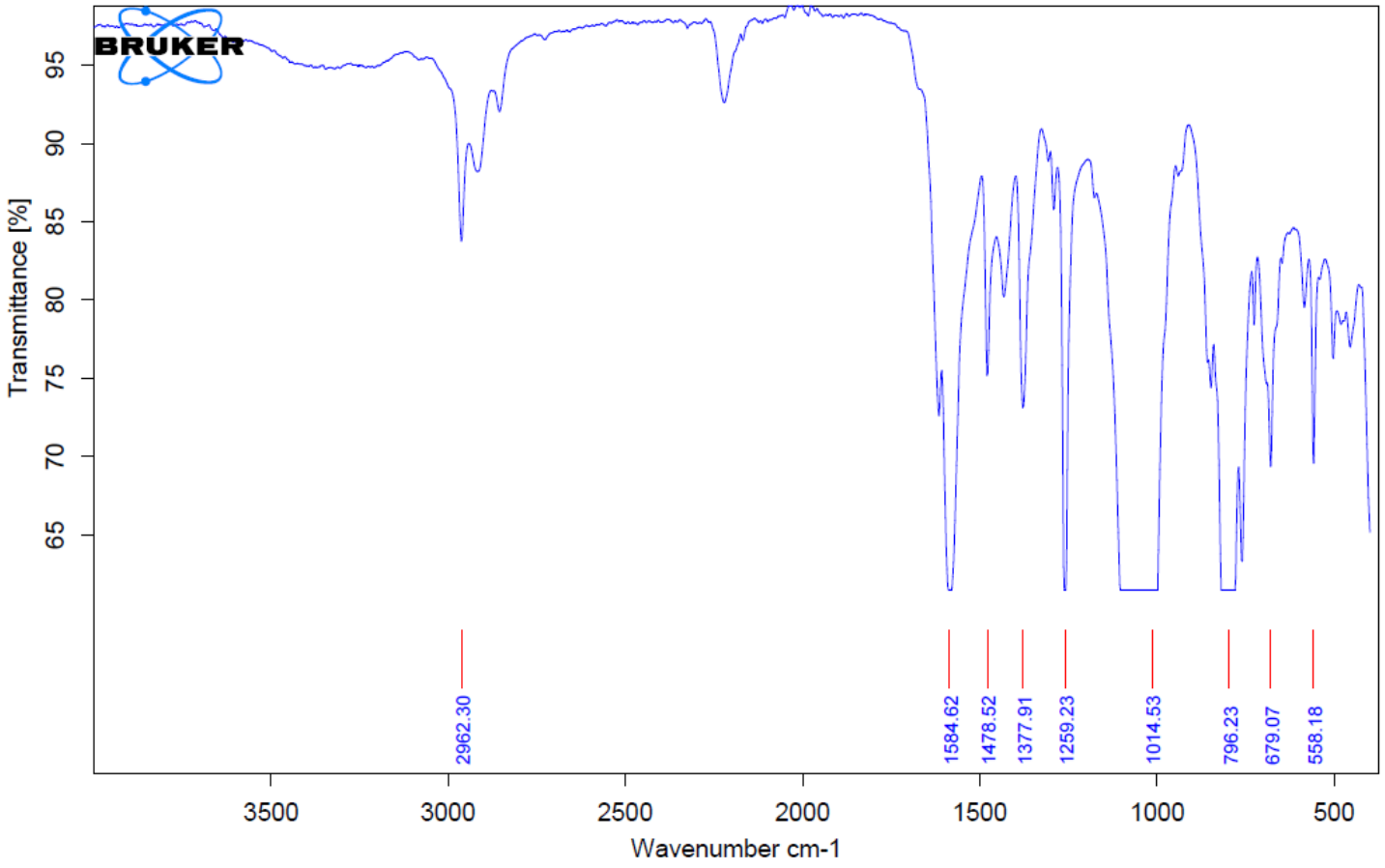
1.7 ^{13}C NMR $[Au(L1)(8\text{-H-Quin})]^{+}$

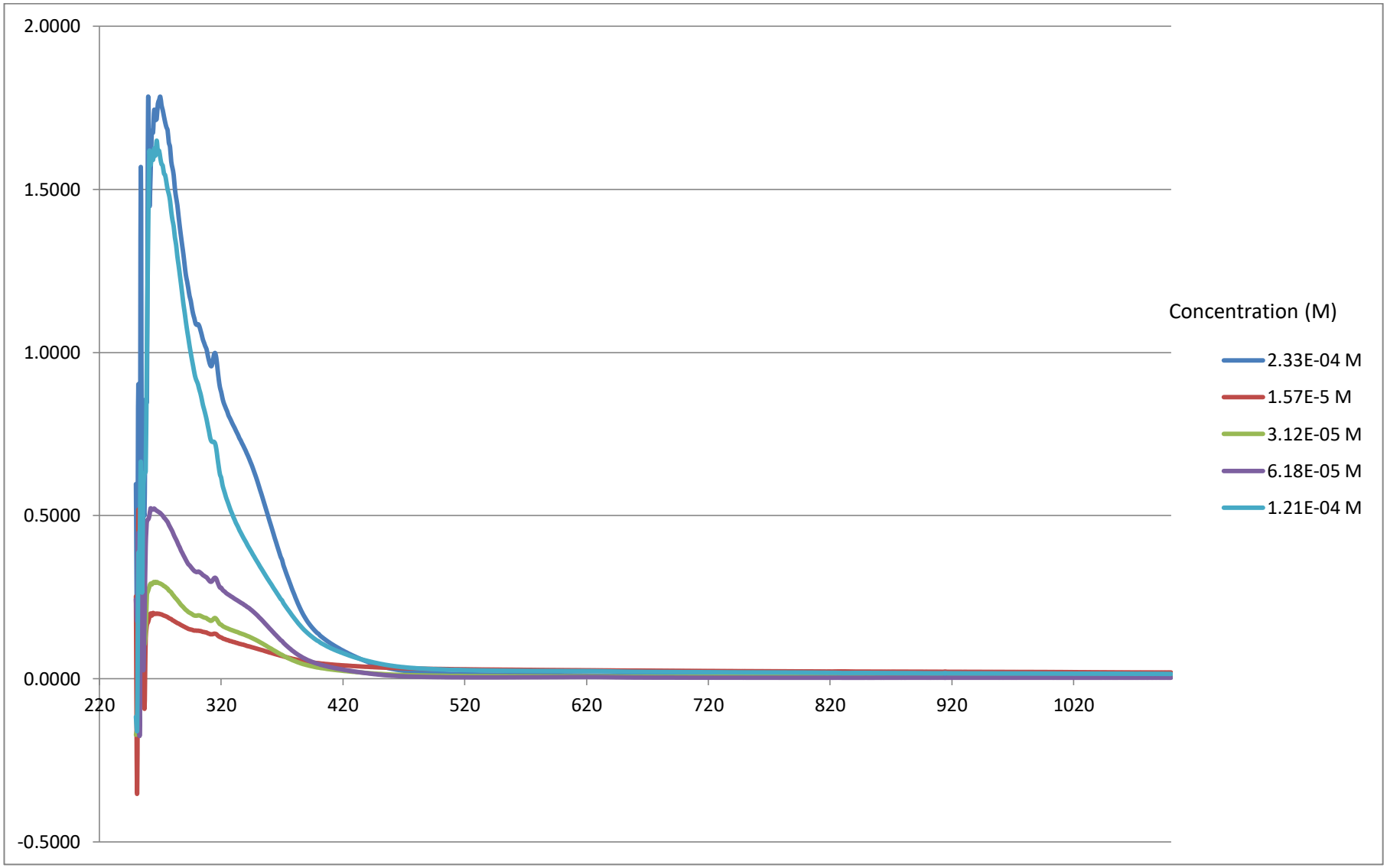


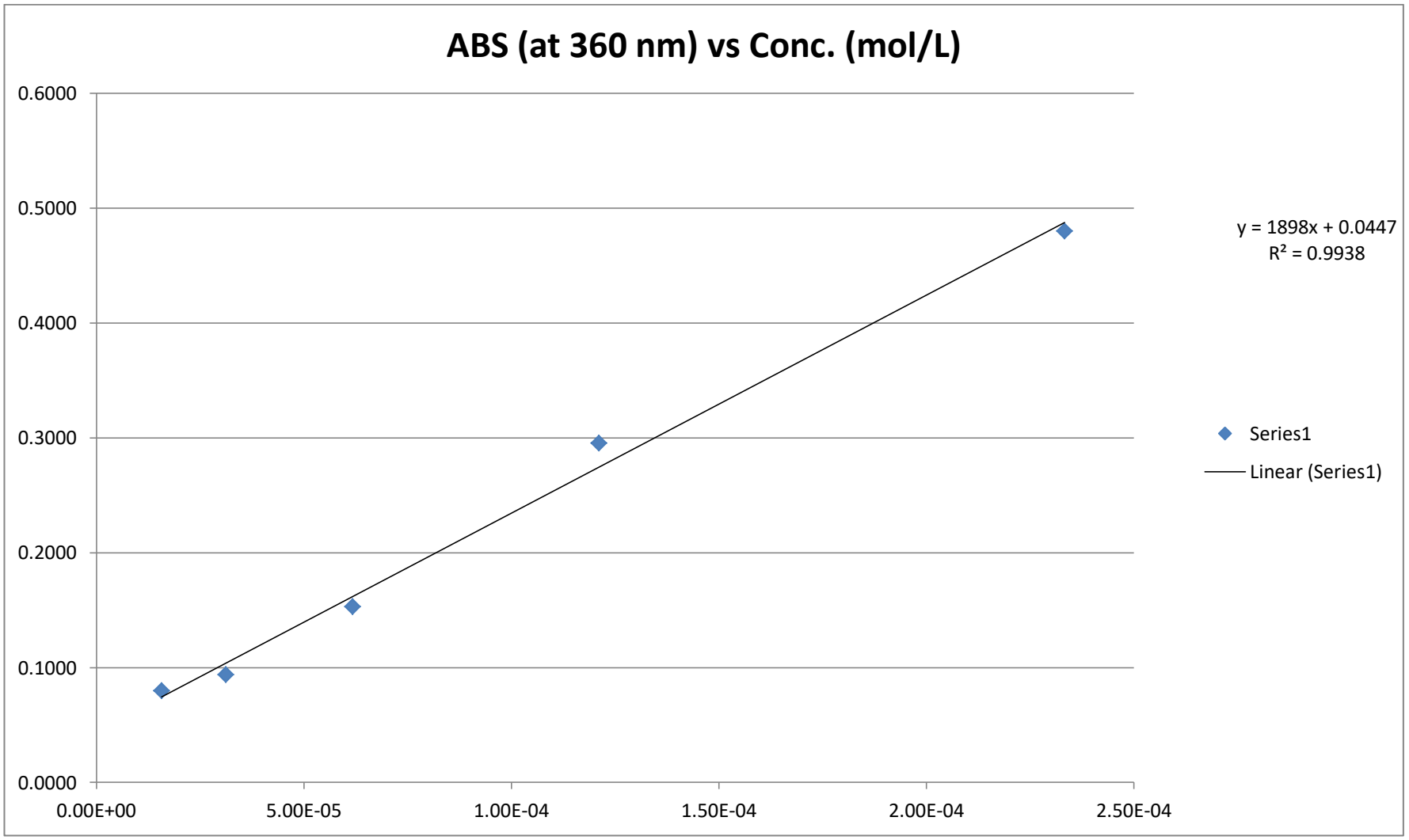
2.1 FTIR [Pd(L1)(8-H-Quin)]



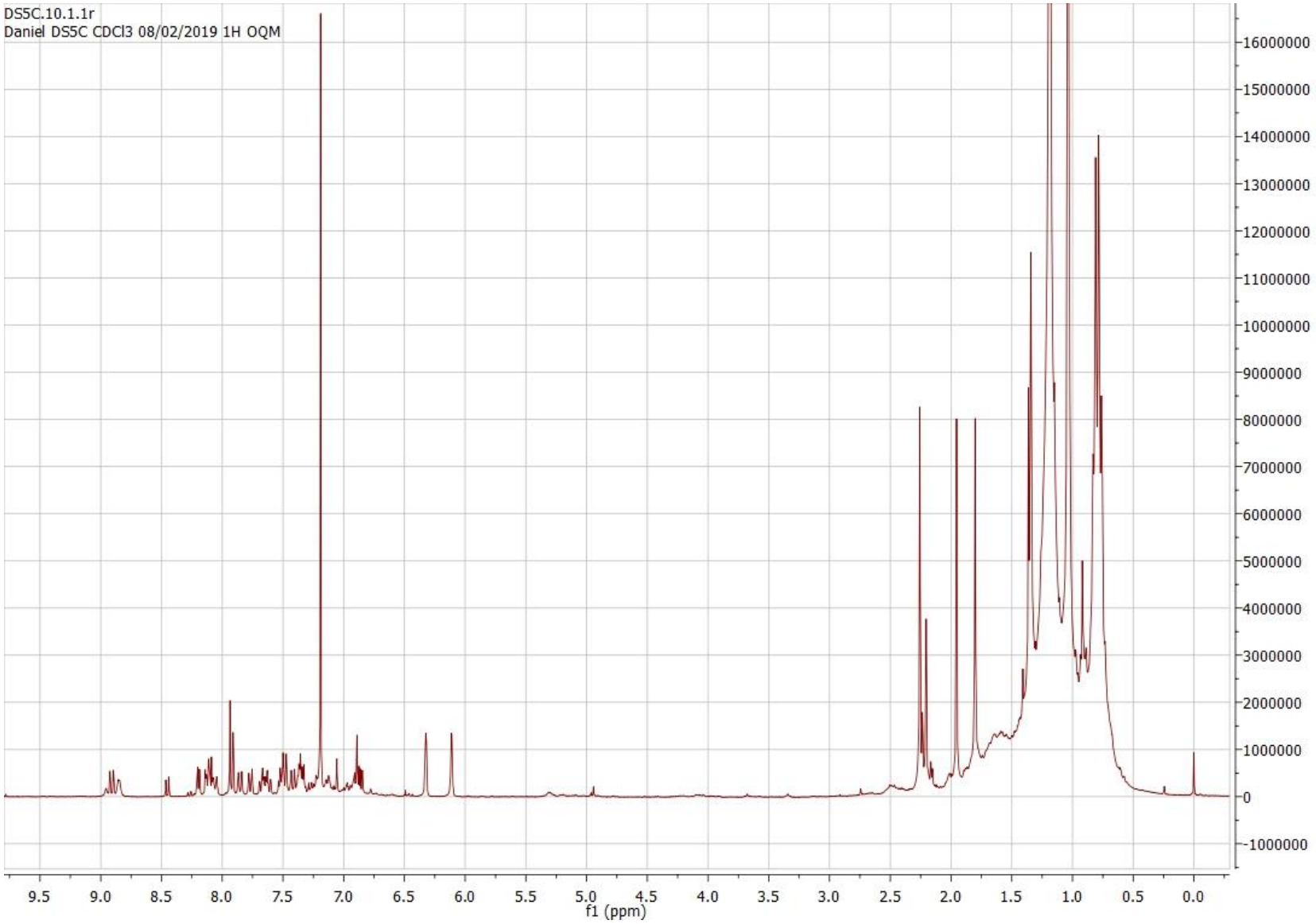
C:\WITS\Measurements\Daniel\Pd Complexes\DS3C reflux.1	DS3C reflux	Instrument type and / or accessory	31/08/2016

2.2 UV VIS [Pd(L1)(8-H-Quin)]

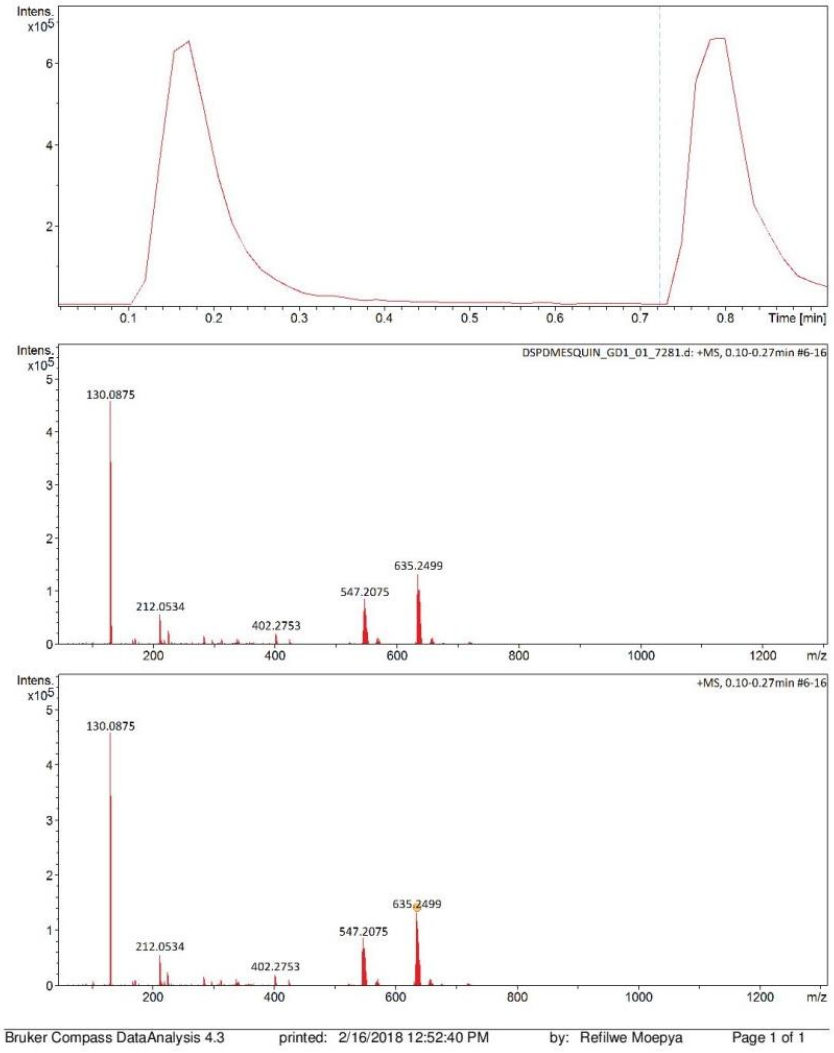



ABS (at 360 nm) vs Conc. (mol/L)
0.6000
0.5000
0.4000
0.3000
0.2000
0.1000
0.0000
0.00E+00
5.00E-05
1.00E-04
1.50E-04
2.00E-04
2.50E-04
y = 1898x + 0.0447
R² = 0.9938
Series1
Linear (Series1)

2.3 ^{1}H NMR [Pd(L1)(8-H-Quin)]

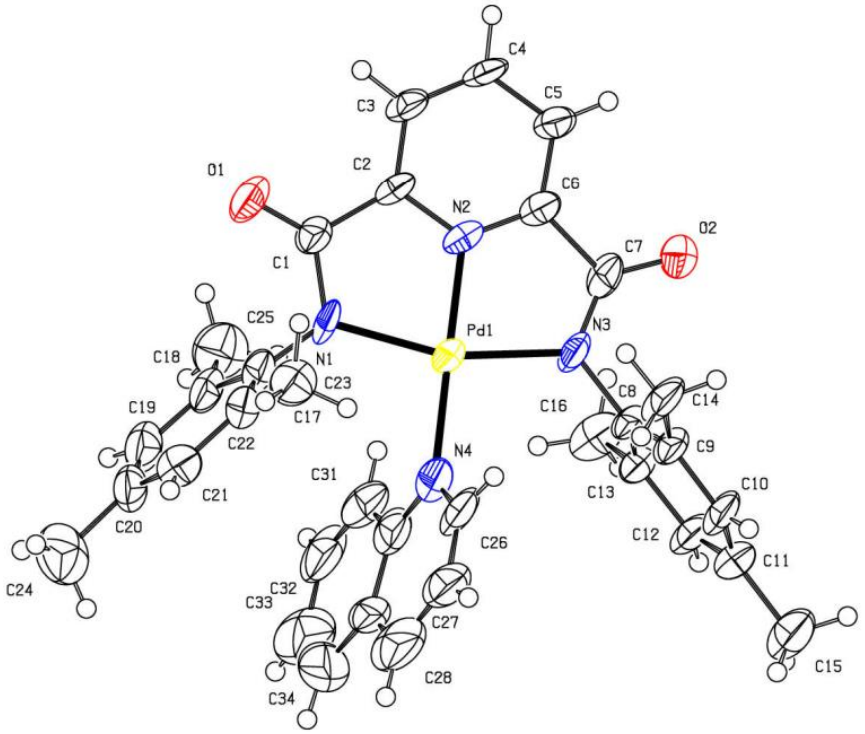


2.4 HMRS [Pd(L1)(8-H-Quin)]



2.5 X-ray crystallography data [Pd(L1)(8-H-Quin)]

DS5C_red2



(ds5c_red2)

Crystal data

$C_{34}H_{32}N_4O_2Pd$	D_x = 1.179 Mg m^{-3}
M_r = 635.03	Mo $K\alpha$ radiation, λ = 0.71073 Å
Orthorhombic, *Pbcn*	Cell parameters from 8141 reflections
a = 33.3299 (15) Å	θ = 2.3–25.4°
b = 13.9723 (6) Å	μ = 0.55 mm^{-1}
c = 15.3608 (6) Å	*T* = 296 K
V = 7153.5 (5) Å^3	Plate, yellow
Z = 8	0.31 × 0.25 × 0.11 mm
F(000) = 2608	

Data collection

Bruker SMART APEX2 area detector diffractometer	8818 independent reflections
Radiation source: microfocus sealed X-ray tube, Incoatec Iμs	5323 reflections with $I > 2\sigma(I)$
Mirror optics monochromator	R_{int} = 0.061
Detector resolution: 7.9 pixels mm^{-1}	θ_{max} = 28.4°, θ_{min} = 1.2°
ω and ϕ scans	*h* = -44→29
Absorption correction: multi-scan *SADABS2012*/1 (Bruker,2012) was used for absorption correction. wR2(int) was 0.0620 before and 0.0505 after correction. The Ratio of	*k* = -18→16

minimum to maximum transmission is 0.8856. The λ/2 correction factor is 0.0015.	
T_{min} = 0.660, T_{max} = 0.746	l = -20→16
60135 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.141	H-atom parameters constrained
$wR(F^2)$ = 0.450	$w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.66	$(\Delta/\sigma)_{max}$ = 0.001
8818 reflections	$\Delta\rangle_{max}$ = 5.82 e Å^{-3}
376 parameters	$\Delta\rangle_{min}$ = -5.40 e Å^{-3}
0 restraints	

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for (ds5c_red2)

	x	y	z	U_{iso}*/U_{eq}
Pd1	0.16133 (2)	0.75442 (5)	0.02063 (5)	0.0411 (3)
N2	0.1784 (2)	0.8665 (5)	0.0843 (5)	0.0380 (16)
N1	0.1387 (3)	0.7182 (7)	0.1398 (5)	0.050 (2)
O1	0.1384 (2)	0.7813 (6)	0.2810 (5)	0.0576 (18)
N4	0.1489 (3)	0.6298 (7)	-0.0442 (6)	0.057 (2)
C6	0.1995 (3)	0.9320 (6)	0.0417 (6)	0.040 (2)
N3	0.1865 (3)	0.8300 (6)	-0.0791 (5)	0.0446 (18)
C12	0.1565 (4)	0.7877 (9)	-0.3078 (7)	0.054 (3)
H12	0.1343	0.7993	-0.3429	0.065*
O2	0.2222 (2)	0.9679 (5)	-0.1011 (5)	0.0554 (18)
C5	0.2162 (3)	1.0109 (6)	0.0869 (6)	0.042 (2)
H5	0.2315	1.0570	0.0587	0.050*
C13	0.1557 (3)	0.8142 (7)	-0.2190 (6)	0.048 (2)
C18	0.0777 (3)	0.6353 (8)	0.1772 (6)	0.048 (2)
C30	0.1124 (4)	0.5954 (9)	-0.0553 (7)	0.060 (3)
C11	0.1913 (4)	0.7434 (7)	-0.3436 (8)	0.057 (3)
C17	0.1199 (3)	0.6358 (8)	0.1640 (6)	0.051 (2)
C7	0.2036 (3)	0.9115 (7)	-0.0543 (6)	0.043 (2)
C22	0.1402 (3)	0.5510 (7)	0.1687 (6)	0.044 (2)

C21	0.1196 (4)	0.4651 (9)	0.1823 (8)	0.061 (3)
H21	0.1340	0.4082	0.1851	0.073*
C3	0.1872 (3)	0.9444 (7)	0.2169 (6)	0.043 (2)
H3	0.1835	0.9474	0.2769	0.052*
C1	0.1468 (3)	0.7863 (7)	0.2057 (6)	0.045 (2)
C2	0.1716 (3)	0.8692 (6)	0.1713 (5)	0.0370 (19)
C8	0.1889 (3)	0.8000 (7)	-0.1683 (5)	0.046 (2)
C23	0.1862 (3)	0.5496 (9)	0.1618 (8)	0.058 (3)
H23A	0.1940	0.5580	0.1021	0.087*
H23B	0.1961	0.4893	0.1827	0.087*
H23C	0.1972	0.6005	0.1963	0.087*
C4	0.2082 (3)	1.0156 (7)	0.1753 (6)	0.046 (2)
H4	0.2173	1.0679	0.2072	0.055*
C9	0.2235 (4)	0.7578 (6)	-0.2016 (6)	0.045 (2)
C29	0.1033 (5)	0.5009 (9)	-0.0871 (7)	0.073 (4)
C20	0.0786 (3)	0.4623 (8)	0.1918 (7)	0.056 (3)
C19	0.0589 (3)	0.5492 (9)	0.1925 (7)	0.061 (3)
H19	0.0315	0.5496	0.2038	0.073*
C27	0.1769 (5)	0.4848 (9)	-0.1006 (7)	0.070 (3)
H27	0.2007	0.4533	-0.1143	0.084*
C14	0.2609 (4)	0.7411 (8)	-0.1470 (7)	0.060 (3)
H14A	0.2831	0.7744	-0.1725	0.090*
H14B	0.2666	0.6738	-0.1448	0.090*
H14C	0.2564	0.7645	-0.0890	0.090*
C31	0.0776 (5)	0.6544 (11)	-0.0339 (7)	0.079 (4)
H31	0.0820	0.7155	-0.0119	0.095*
C16	0.1186 (4)	0.8671 (10)	-0.1855 (8)	0.074 (3)
H16A	0.1012	0.8818	-0.2335	0.111*
H16B	0.1266	0.9253	-0.1574	0.111*
H16C	0.1046	0.8273	-0.1445	0.111*
C28	0.1386 (8)	0.4394 (14)	-0.1111 (9)	0.123 (8)
H28	0.1358	0.3769	-0.1310	0.147*
C26	0.1768 (4)	0.5746 (9)	-0.0703 (6)	0.062 (3)
H26	0.2022	0.6022	-0.0678	0.075*
C15	0.1919 (5)	0.7139 (10)	-0.4372 (8)	0.073 (3)
H15A	0.2001	0.6482	-0.4415	0.109*
H15B	0.2105	0.7534	-0.4686	0.109*
H15C	0.1656	0.7213	-0.4616	0.109*
C25	0.0538 (4)	0.7272 (11)	0.1741 (12)	0.081 (4)
H25A	0.0598	0.7652	0.2246	0.122*
H25B	0.0257	0.7125	0.1733	0.122*
H25C	0.0607	0.7624	0.1226	0.122*
C10	0.2240 (4)	0.7304 (8)	-0.2860 (6)	0.051 (3)
H10	0.2471	0.7014	-0.3074	0.061*
C24	0.0581 (4)	0.3643 (11)	0.2058 (11)	0.089 (4)
H24A	0.0554	0.3323	0.1508	0.134*

H24B	0.0321	0.3738	0.2309	0.134*
H24C	0.0742	0.3260	0.2442	0.134*
C32	0.0398 (4)	0.6247 (14)	-0.0447 (8)	0.089 (5)
H32	0.0175	0.6628	-0.0334	0.107*
C34	0.0657 (8)	0.4640 (17)	-0.0954 (12)	0.122 (8)
H34	0.0607	0.4015	-0.1130	0.146*
C33	0.0371 (7)	0.525 (2)	-0.0762 (13)	0.126 (8)
H33	0.0111	0.5024	-0.0836	0.151*

Atomic displacement parameters (Å^2) for (ds5c_red2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.0593 (5)	0.0427 (5)	0.0214 (5)	-0.0090 (3)	0.0025 (3)	-0.0016 (3)
N2	0.043 (4)	0.047 (4)	0.024 (4)	0.005 (3)	-0.002 (3)	-0.011 (3)
N1	0.065 (5)	0.069 (5)	0.016 (4)	-0.009 (4)	0.012 (3)	0.006 (4)
O1	0.061 (4)	0.083 (5)	0.029 (4)	0.000 (4)	0.011 (3)	-0.005 (4)
N4	0.065 (6)	0.073 (6)	0.033 (5)	-0.015 (5)	0.007 (4)	0.000 (4)
C6	0.053 (5)	0.038 (4)	0.029 (5)	0.007 (4)	-0.004 (4)	-0.006 (4)
N3	0.069 (5)	0.046 (4)	0.018 (4)	0.002 (4)	0.010 (3)	0.001 (3)
C12	0.084 (8)	0.057 (6)	0.022 (5)	0.006 (6)	0.001 (4)	-0.006 (5)
O2	0.077 (5)	0.051 (4)	0.038 (4)	-0.011 (4)	0.005 (3)	0.006 (3)
C5	0.064 (6)	0.032 (4)	0.031 (5)	0.003 (4)	-0.006 (4)	-0.006 (4)
C13	0.075 (7)	0.044 (5)	0.026 (5)	-0.009 (5)	0.006 (4)	-0.004 (4)
C18	0.051 (5)	0.062 (6)	0.031 (5)	0.001 (5)	0.015 (4)	-0.001 (4)
C30	0.068 (7)	0.084 (8)	0.028 (5)	-0.014 (6)	-0.001 (5)	0.007 (5)
C11	0.074 (7)	0.060 (7)	0.036 (6)	-0.011 (5)	0.013 (5)	-0.016 (4)
C17	0.063 (6)	0.065 (6)	0.026 (5)	-0.014 (5)	0.003 (4)	0.007 (4)
C7	0.058 (6)	0.046 (5)	0.024 (4)	0.014 (4)	-0.003 (4)	0.013 (4)
C22	0.050 (5)	0.050 (5)	0.034 (5)	-0.004 (4)	0.006 (4)	0.005 (4)
C21	0.067 (7)	0.066 (7)	0.049 (7)	-0.010 (5)	0.006 (5)	-0.010 (5)
C3	0.057 (5)	0.049 (5)	0.023 (5)	0.001 (4)	-0.010 (4)	-0.008 (4)
C1	0.065 (6)	0.041 (5)	0.027 (5)	-0.003 (5)	-0.001 (4)	0.002 (4)
C2	0.048 (5)	0.043 (5)	0.020 (4)	0.008 (4)	-0.004 (3)	-0.008 (4)
C8	0.081 (7)	0.044 (5)	0.015 (4)	-0.012 (5)	-0.003 (4)	0.001 (4)
C23	0.044 (5)	0.077 (8)	0.053 (7)	-0.003 (5)	-0.003 (5)	-0.004 (6)
C4	0.066 (6)	0.047 (5)	0.024 (5)	0.001 (4)	-0.009 (4)	-0.017 (4)
C9	0.074 (7)	0.042 (5)	0.019 (4)	-0.004 (4)	-0.001 (4)	0.003 (3)
C29	0.135 (12)	0.057 (7)	0.027 (6)	-0.035 (8)	-0.007 (6)	0.001 (5)
C20	0.049 (6)	0.076 (8)	0.041 (6)	-0.021 (5)	0.007 (4)	0.008 (5)
C19	0.048 (6)	0.093 (9)	0.042 (6)	-0.015 (6)	0.007 (5)	0.006 (6)
C27	0.109 (10)	0.064 (8)	0.036 (6)	0.001 (7)	-0.002 (6)	-0.004 (5)
C14	0.090 (9)	0.070 (8)	0.019 (5)	0.008 (6)	0.000 (5)	-0.001 (4)
C31	0.103 (11)	0.107 (11)	0.028 (6)	-0.022 (9)	-0.009 (6)	-0.003 (6)
C16	0.092 (9)	0.080 (8)	0.050 (7)	0.012 (7)	-0.002 (6)	-0.012 (6)
C28	0.24 (3)	0.090 (12)	0.036 (8)	-0.026 (15)	-0.007 (11)	0.010 (7)
C26	0.096 (9)	0.070 (7)	0.021 (5)	0.009 (6)	0.015 (5)	-0.001 (5)

C15	0.100 (10)	0.080 (8)	0.038 (7)	0.010 (8)	0.006 (6)	-0.002 (6)
C25	0.071 (8)	0.085 (9)	0.087 (11)	0.011 (7)	0.012 (8)	-0.009 (8)
C10	0.075 (7)	0.060 (6)	0.018 (5)	0.007 (5)	0.006 (4)	0.003 (4)
C24	0.059 (8)	0.104 (11)	0.104 (12)	-0.012 (7)	0.014 (7)	0.003 (9)
C32	0.071 (8)	0.167 (16)	0.030 (6)	-0.026 (9)	0.000 (5)	-0.003 (8)
C34	0.17 (2)	0.135 (17)	0.064 (11)	-0.091 (16)	-0.013 (12)	0.003 (11)
C33	0.119 (15)	0.19 (2)	0.068 (12)	-0.054 (15)	-0.016 (11)	-0.019 (14)

Geometric parameters (Å, º) for (ds5c_red2)

Pd1—N2	1.932 (7)	C23—H23A	0.9600
Pd1—N1	2.045 (7)	C23—H23B	0.9600
Pd1—N4	2.048 (10)	C23—H23C	0.9600
Pd1—N3	2.041 (7)	C4—H4	0.9300
N2—C6	1.326 (12)	C9—C14	1.519 (17)
N2—C2	1.356 (11)	C9—C10	1.352 (14)
N1—C17	1.362 (13)	C29—C28	1.50 (3)
N1—C1	1.415 (13)	C29—C34	1.36 (2)
O1—C1	1.192 (11)	C20—C19	1.380 (16)
N4—C30	1.320 (14)	C20—C24	1.545 (17)
N4—C26	1.274 (15)	C19—H19	0.9300
C6—C5	1.416 (12)	C27—H27	0.9300
C6—C7	1.509 (13)	C27—C28	1.44 (3)
N3—C7	1.329 (13)	C27—C26	1.338 (17)
N3—C8	1.436 (11)	C14—H14A	0.9600
C12—H12	0.9300	C14—H14B	0.9600
C12—C13	1.413 (14)	C14—H14C	0.9600
C12—C11	1.425 (17)	C31—H31	0.9300
O2—C7	1.234 (11)	C31—C32	1.337 (18)
C5—H5	0.9300	C16—H16A	0.9600
C5—C4	1.386 (13)	C16—H16B	0.9600
C13—C8	1.366 (15)	C16—H16C	0.9600
C13—C16	1.532 (16)	C28—H28	0.9300
C18—C17	1.419 (14)	C26—H26	0.9300
C18—C19	1.377 (15)	C15—H15A	0.9600
C18—C25	1.512 (17)	C15—H15B	0.9600
C30—C29	1.440 (17)	C15—H15C	0.9600
C30—C31	1.458 (19)	C25—H25A	0.9600
C11—C15	1.496 (16)	C25—H25B	0.9600
C11—C10	1.415 (18)	C25—H25C	0.9600
C17—C22	1.367 (15)	C10—H10	0.9300
C22—C21	1.399 (14)	C24—H24A	0.9600
C22—C23	1.536 (14)	C24—H24B	0.9600
C21—H21	0.9300	C24—H24C	0.9600
C21—C20	1.375 (16)	C32—H32	0.9300
C3—H3	0.9300	C32—C33	1.47 (3)

C3—C2	1.365 (12)	C34—H34	0.9300
C3—C4	1.375 (14)	C34—C33	1.32 (3)
C1—C2	1.519 (14)	C33—H33	0.9300
C8—C9	1.394 (15)		
N2—Pd1—N1	81.7 (3)	C5—C4—H4	119.4
N2—Pd1—N4	174.2 (4)	C3—C4—C5	121.2 (8)
N2—Pd1—N3	80.7 (3)	C3—C4—H4	119.4
N1—Pd1—N4	98.6 (4)	C8—C9—C14	122.8 (9)
N3—Pd1—N1	162.2 (3)	C10—C9—C8	118.7 (10)
N3—Pd1—N4	99.1 (3)	C10—C9—C14	118.5 (11)
C6—N2—Pd1	117.8 (6)	C30—C29—C28	116.3 (14)
C6—N2—C2	123.7 (8)	C34—C29—C30	125.0 (18)
C2—N2—Pd1	118.2 (6)	C34—C29—C28	118.8 (17)
C17—N1—Pd1	128.6 (7)	C21—C20—C19	116.7 (10)
C17—N1—C1	117.5 (8)	C21—C20—C24	118.6 (11)
C1—N1—Pd1	113.8 (6)	C19—C20—C24	124.6 (10)
C30—N4—Pd1	124.0 (9)	C18—C19—C20	123.4 (10)
C26—N4—Pd1	121.3 (9)	C18—C19—H19	118.3
C26—N4—C30	114.4 (12)	C20—C19—H19	118.3
N2—C6—C5	120.3 (8)	C28—C27—H27	121.6
N2—C6—C7	113.5 (8)	C26—C27—H27	121.6
C5—C6—C7	126.2 (9)	C26—C27—C28	116.8 (16)
C7—N3—Pd1	113.9 (6)	C9—C14—H14A	109.5
C7—N3—C8	120.0 (8)	C9—C14—H14B	109.5
C8—N3—Pd1	126.0 (6)	C9—C14—H14C	109.5
C13—C12—H12	119.9	H14A—C14—H14B	109.5
C13—C12—C11	120.1 (11)	H14A—C14—H14C	109.5
C11—C12—H12	119.9	H14B—C14—H14C	109.5
C6—C5—H5	121.9	C30—C31—H31	118.5
C4—C5—C6	116.3 (9)	C32—C31—C30	123.0 (15)
C4—C5—H5	121.9	C32—C31—H31	118.5
C12—C13—C16	117.8 (10)	C13—C16—H16A	109.5
C8—C13—C12	119.8 (10)	C13—C16—H16B	109.5
C8—C13—C16	122.2 (9)	C13—C16—H16C	109.5
C17—C18—C25	120.9 (10)	H16A—C16—H16B	109.5
C19—C18—C17	118.7 (10)	H16A—C16—H16C	109.5
C19—C18—C25	120.5 (10)	H16B—C16—H16C	109.5
N4—C30—C29	124.9 (13)	C29—C28—H28	122.7
N4—C30—C31	119.8 (12)	C27—C28—C29	114.6 (15)
C29—C30—C31	115.3 (12)	C27—C28—H28	122.7
C12—C11—C15	120.2 (12)	N4—C26—C27	132.7 (14)
C10—C11—C12	116.1 (10)	N4—C26—H26	113.6
C10—C11—C15	123.7 (11)	C27—C26—H26	113.6
N1—C17—C18	119.9 (10)	C11—C15—H15A	109.5
N1—C17—C22	121.3 (10)	C11—C15—H15B	109.5

C22—C17—C18	118.6 (9)	C11—C15—H15C	109.5
N3—C7—C6	113.8 (8)	H15A—C15—H15B	109.5
O2—C7—C6	119.6 (9)	H15A—C15—H15C	109.5
O2—C7—N3	126.6 (9)	H15B—C15—H15C	109.5
C17—C22—C21	120.5 (9)	C18—C25—H25A	109.5
C17—C22—C23	120.1 (9)	C18—C25—H25B	109.5
C21—C22—C23	119.3 (9)	C18—C25—H25C	109.5
C22—C21—H21	119.1	H25A—C25—H25B	109.5
C20—C21—C22	121.9 (11)	H25A—C25—H25C	109.5
C20—C21—H21	119.1	H25B—C25—H25C	109.5
C2—C3—H3	119.6	C11—C10—H10	118.1
C2—C3—C4	120.8 (9)	C9—C10—C11	123.7 (11)
C4—C3—H3	119.6	C9—C10—H10	118.1
N1—C1—C2	111.6 (8)	C20—C24—H24A	109.5
O1—C1—N1	127.5 (10)	C20—C24—H24B	109.5
O1—C1—C2	120.7 (9)	C20—C24—H24C	109.5
N2—C2—C3	117.7 (9)	H24A—C24—H24B	109.5
N2—C2—C1	114.4 (8)	H24A—C24—H24C	109.5
C3—C2—C1	127.9 (8)	H24B—C24—H24C	109.5
C13—C8—N3	117.2 (9)	C31—C32—H32	123.5
C13—C8—C9	121.5 (9)	C31—C32—C33	113.0 (17)
C9—C8—N3	121.2 (9)	C33—C32—H32	123.5
C22—C23—H23A	109.5	C29—C34—H34	123.2
C22—C23—H23B	109.5	C33—C34—C29	114 (2)
C22—C23—H23C	109.5	C33—C34—H34	123.2
H23A—C23—H23B	109.5	C32—C33—H33	115.0
H23A—C23—H23C	109.5	C34—C33—C32	130.0 (19)
H23B—C23—H23C	109.5	C34—C33—H33	115.0
Pd1—N2—C6—C5	-173.8 (7)	C30—C31—C32—C33	2.6 (19)
Pd1—N2—C6—C7	5.4 (10)	C11—C12—C13—C8	2.2 (16)
Pd1—N2—C2—C3	173.8 (7)	C11—C12—C13—C16	176.4 (11)
Pd1—N2—C2—C1	-6.7 (10)	C17—N1—C1—O1	-1.6 (17)
Pd1—N1—C17—C18	-105.8 (11)	C17—N1—C1—C2	173.9 (9)
Pd1—N1—C17—C22	68.5 (13)	C17—C18—C19—C20	-3.0 (16)
Pd1—N1—C1—O1	-177.0 (9)	C17—C22—C21—C20	-0.2 (16)
Pd1—N1—C1—C2	-1.6 (11)	C7—C6—C5—C4	179.5 (9)
Pd1—N4—C30—C29	-168.9 (8)	C7—N3—C8—C13	-101.7 (11)
Pd1—N4—C30—C31	10.6 (14)	C7—N3—C8—C9	78.6 (12)
Pd1—N4—C26—C27	167.7 (11)	C22—C21—C20—C19	-3.8 (16)
Pd1—N3—C7—C6	-1.8 (10)	C22—C21—C20—C24	179.4 (11)
Pd1—N3—C7—O2	176.8 (8)	C21—C20—C19—	5.4 (17)

		C18	
Pd1—N3—C8—C13	82.1 (10)	C1—N1—C17—C18	79.6 (12)
Pd1—N3—C8—C9	-97.7 (10)	C1—N1—C17—C22	-106.2 (12)
N2—C6—C5—C4	-1.4 (13)	C2—N2—C6—C5	-0.7 (13)
N2—C6—C7—N3	-2.2 (11)	C2—N2—C6—C7	178.5 (8)
N2—C6—C7—O2	179.2 (9)	C2—C3—C4—C5	-3.5 (15)
N1—C17—C22—C21	-171.5 (10)	C8—N3—C7—C6	-178.5 (8)
N1—C17—C22—C23	10.4 (14)	C8—N3—C7—O2	0.1 (15)
N1—C1—C2—N2	5.2 (12)	C8—C9—C10—C11	1.0 (16)
N1—C1—C2—C3	-175.4 (9)	C23—C22—C21—C20	177.8 (10)
O1—C1—C2—N2	-179.0 (9)	C4—C3—C2—N2	1.3 (14)
O1—C1—C2—C3	0.4 (16)	C4—C3—C2—C1	-178.1 (9)
N4—C30—C29—C28	-1.5 (16)	C29—C30—C31—C32	-1.4 (17)
N4—C30—C29—C34	177.3 (13)	C29—C34—C33—C32	-2 (3)
N4—C30—C31—C32	179.0 (11)	C19—C18—C17—N1	173.1 (9)
C6—N2—C2—C3	0.8 (13)	C19—C18—C17—C22	-1.2 (14)
C6—N2—C2—C1	-179.8 (8)	C14—C9—C10—C11	-179.1 (10)
C6—C5—C4—C3	3.4 (14)	C31—C30—C29—C28	178.9 (10)
N3—C8—C9—C14	-0.3 (14)	C31—C30—C29—C34	-2.2 (18)
N3—C8—C9—C10	179.6 (9)	C31—C32—C33—C34	-1 (3)
C12—C13—C8—N3	178.8 (9)	C16—C13—C8—N3	4.9 (14)
C12—C13—C8—C9	-1.5 (15)	C16—C13—C8—C9	-175.3 (10)
C12—C11—C10—C9	-0.3 (17)	C28—C29—C34—C33	-177.3 (16)
C5—C6—C7—N3	177.0 (9)	C28—C27—C26—N4	3 (2)
C5—C6—C7—O2	-1.7 (14)	C26—N4—C30—C29	4.9 (15)
C13—C12—C11—C15	179.0 (11)	C26—N4—C30—C31	-175.6 (10)
C13—C12—C11—C10	-1.4 (16)	C26—C27—C28—C29	0.8 (17)
C13—C8—C9—C14	-180.0 (9)	C15—C11—C10—C9	179.3 (11)
C13—C8—C9—C10	-0.1 (15)	C25—C18—C17—N1	-6.5 (16)
C18—C17—C22—C21	2.7 (15)	C25—C18—C17—C22	179.1 (11)
C18—C17—C22—C23	-175.3 (9)	C25—C18—C19—C20	176.7 (12)
C30—N4—C26—C27	-6.3 (18)	C24—C20—C19—C18	-177.9 (12)
C30—C29—C28—C27	-1.4 (16)	C34—C29—C28—C27	179.6 (13)
C30—C29—C34—C33	4 (2)		

Document origin: *publCIF* [Westrip, S. P. (2010). *J. Apply. Cryst.*, **43**, 920-925].

2.6 CIF check [Pd(L1)(8-H-Quin)]

checkCIF/PLATON (basic structural check)

Structure factors have been supplied for datablock(s) ds5c_red2

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this

report Structure factor report

Datablock: ds5c_red2

Bond precision: C-C = 0.0182 A
Wavelength=0.71073 Cell: a=33.3299(15)
b=13.9723(6) c=15.3608(6)
alpha=90 beta=90
gamma=90 Temperature:
296 K

	Calculated	Reported
Volume	7153.5(5)	7153.5(5)
Space group	P b c n	P b c n
Hall group	-P 2n 2ab	-P 2n 2ab
Moiety formula	C34 H32 N4 O2 Pd	C34 H32 N4 O2 Pd
Sum formula	C34 H32 N4 O2 Pd	C34 H32 N4 O2 Pd
Mr	635.04	635.03
Dx,g cm-3	1.179	1.179
Z	8	8
Mu (mm-1)	0.550	0.550
F000	2608.0	2608.0
F000'	2600.88	
h,k,lmax	44,18,20	44,18,20
Nref	8991	8818
Tmin,Tmax	0.848,0.940	0.660,0.746
Tmin'	0.841	

Correction method= # Reported T Limits: Tmin=0.660 Tmax=0.746
AbsCorr = MULTI-SCAN

Data completeness= 0.981 Theta(max)= 28.391

R(reflections)= 0.1412(5323) wR2(reflections)= 0.4496(8818)

S = 1.660 Npar= 376

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level. Click on the hyperlinks for more details of the test.

Alert level B

DIFMN02_ALERT_2_B The minimum difference density is < -0.1*ZMAX*1.00
_refine_diff_density_min given = -5.398 Test value = -4.600
PLAT084_ALERT_3_B High wR2 Value (i.e. > 0.25) ..... 0.45 Report
PLAT097_ALERT_2_B Large Reported Max. (Positive) Residual Density 5.82 eA-3
PLAT098_ALERT_2_B Large Reported Min. (Negative) Residual Density -5.40 eA-

3 PLAT234_ALERT_4_B Large Hirshfeld Difference C27 -- C28 .. 0.29 Ang.
PLAT234_ALERT_4_B Large Hirshfeld Difference C28 -- C29 .. 0.26 Ang.

Alert level C

DIFMN03_ALERT_1_C The minimum difference density is < -
0.1*ZMAX*0.75 The relevant atom site should be identified.
DIFMX02_ALERT_1_C The maximum difference density is >
0.1*ZMAX*0.75 The relevant atom site should be identified.
PLAT082_ALERT_2_C High R1 Value ..... 0.14 Report

PLAT220_ALERT_2_C Non-Solvent Resd 1 C Ueq(max)/Ueq(min) Range 3.4 Ratio
PLAT234_ALERT_4_C Large Hirshfeld Difference Pd1 -- N4 .. 0.16 Ang.

And 3 other PLAT234 Alerts

More ...
PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of C28 Check

And 2 other PLAT241 Alerts

More ...
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C18 Check

And 2 other PLAT242 Alerts

More ...
PLAT250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor .... 2.4 Note PLAT342_ALERT_3_C Low
Bond Precision on C-C Bonds ..... 0.01818 Ang. PLAT905_ALERT_3_C
Negative K value in the Analysis of Variance ... -6.684 Report PLAT911_ALERT_3_C
Missing # FCF Refl Between THmin & STh/L= 0.600 92 Report PLAT918_ALERT_3_C

Reflection(s) with I(obs) much Smaller I(calc) . 2 Check

Alert level G

PLAT072_ALERT_2_G SHELXL First Parameter in WGHT Unusually Large 0.20 Report
PLAT335_ALERT_2_G Check Large C6 Ring C-C Range C29 -C34 0.17 Ang.
PLAT606_ALERT_4_G VERY LARGE Solvent Accessible VOID(S) in Structure ! Info PLAT802_ALERT_4_G
CIF Input Record(s) with more than 80 Characters 1 Info PLAT869_ALERT_4_G
ALERTS Related to the use of SQUEEZE Suppressed ! Info
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 82 Note

---

PLAT933_ALERT_2_G Number of OMIT records in Embedded RES ..... 90 Note

0 **ALERT level A** = Most likely a serious problem - resolve or explain 6 **ALERT level B** = A potentially serious problem, consider carefully

19 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight 7 **ALERT level G** = General information/check it is not something unexpected

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data 15 ALERT type 2 Indicator that the structure model may be wrong or deficient 5 ALERT type 3 Indicator that the structure quality may be low

10 ALERT type 4 Improvement, methodology, query or suggestion 0 ALERT type 5 Informative message, check

---

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

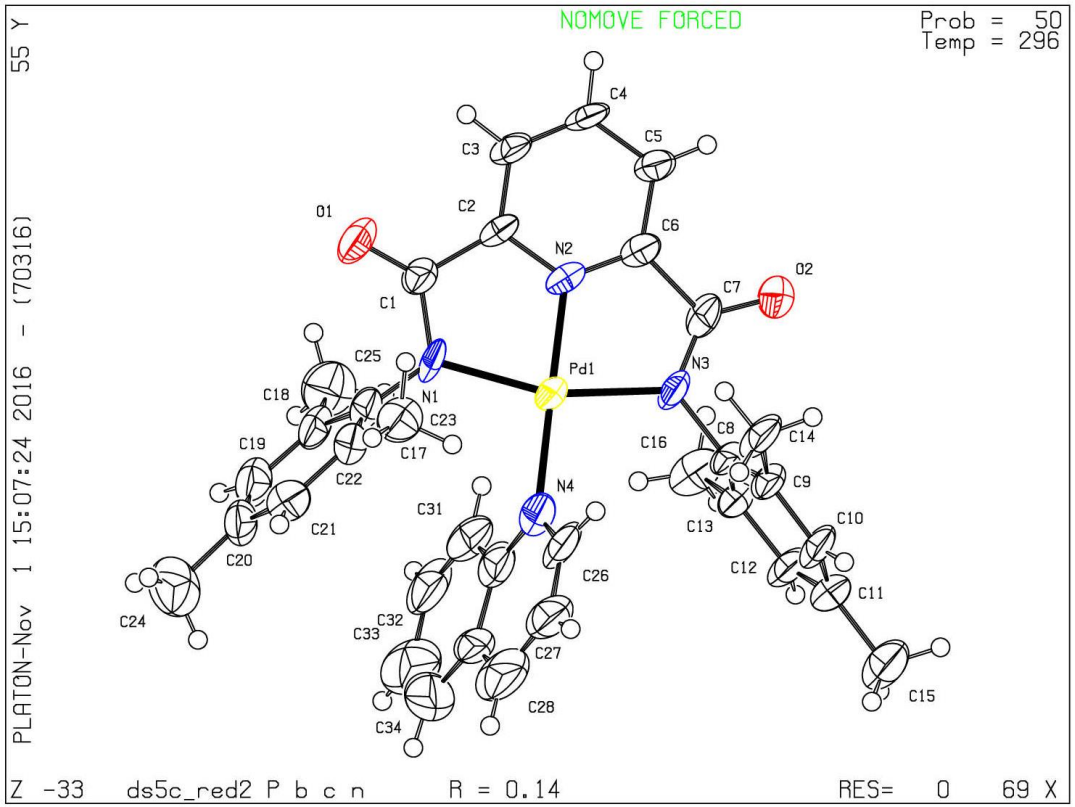
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 11/08/2016; check.def file version of 04/08/2016

Datablock ds5c_red2 - ellipsoid plot



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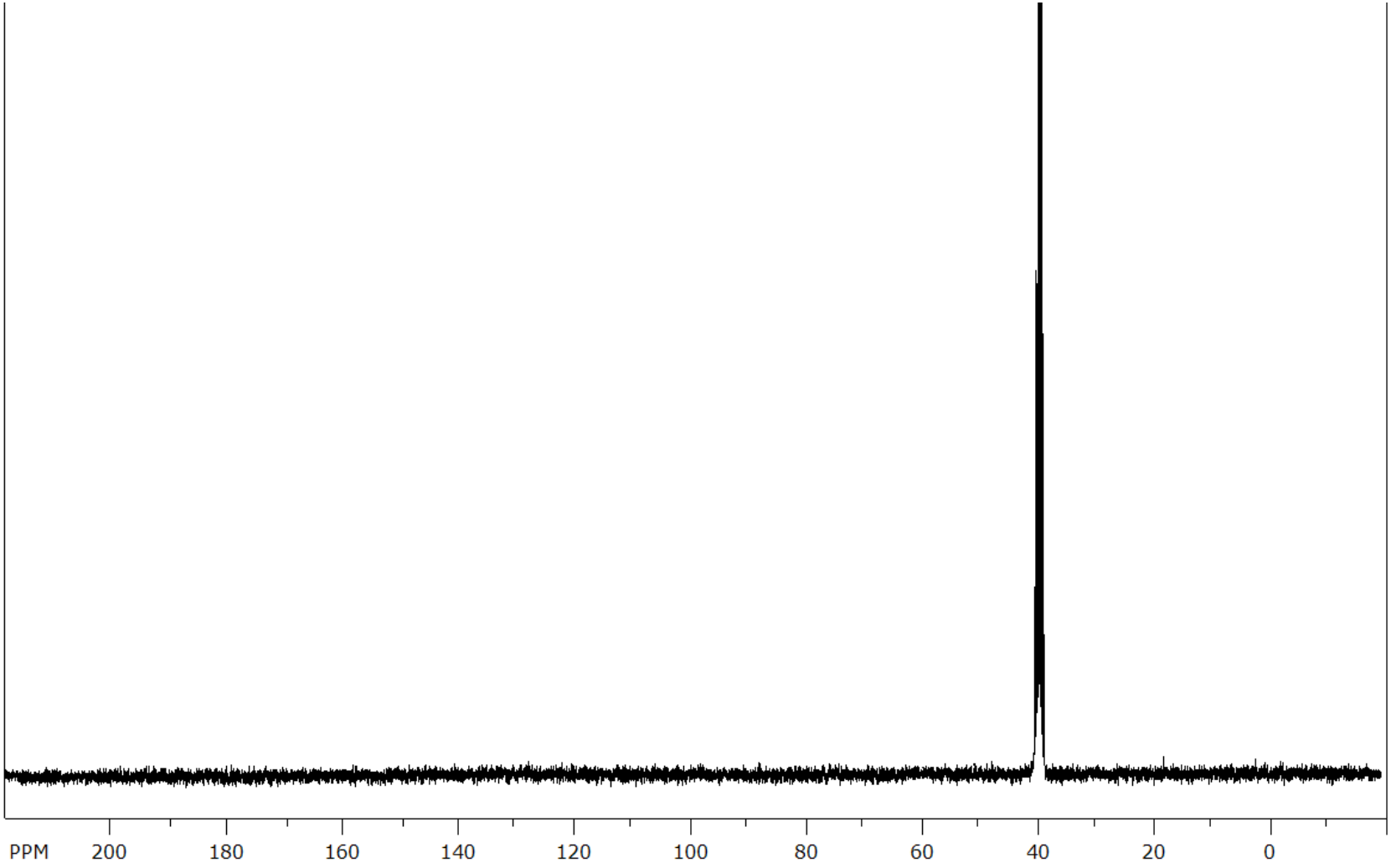
2.6 ^{13}C NMR NMR [Pd(L1)(8-H-Quin)]



3.1 HSEh1PBE statistical analysis

Table 3.1. Selected geometrical parameters comparing the structural accuracy of various DFT functionals relative to the X-ray data for [Au(Mes)(Quin)]+

Functional	Basis set	Au-N(amide)	Au-N(amide)'	Au-Npy	Au-Nquin	Au---H	Phi Ar1*	Phi Ar2**	Phi Quin
HSEh1PBE	DGDZVP/GEN	2.268	2.254	2.113	2.243	2.797	-120.49	62.59	-125.22
diff.		*0.241*	*0.219*	*0.166*	*0.208*	*-0.031*	*-19.98*	*-41.44*	*-33.185*
M06	DGDZVP/GEN	2.278	2.259	2.131	2.251	2.871	-125.73	59.6	-123.73
diff.		*0.251*	*0.224*	*0.184*	*0.216*	*0.043*	*-25.22*	*-44.43*	*-31.695*
wB97XD	DGDZVP/GEN	2.170	2.154	2.062	2.202	2.815	-120.92	66.49	-121.35
diff.		*0.143*	*0.119*	*0.115*	*0.167*	*-0.013*	*-20.41*	*-37.54*	*-29.315*
X-ray	**n/a**	**2.027**	**2.035**	**1.947**	**2.035**	**2.828**	**-100.51**	**104.03**	**-92.035**

*Left side mesityl ring in pi-contact with quinoline ring (measured from the carbonyl carbon through the amide nitrogen to the upper ortho-carbon of the ring.

**Right side mesityl ring measured from the carbonyl carbon through the amide nitrogen to the upper ortho-carbon of the ring.

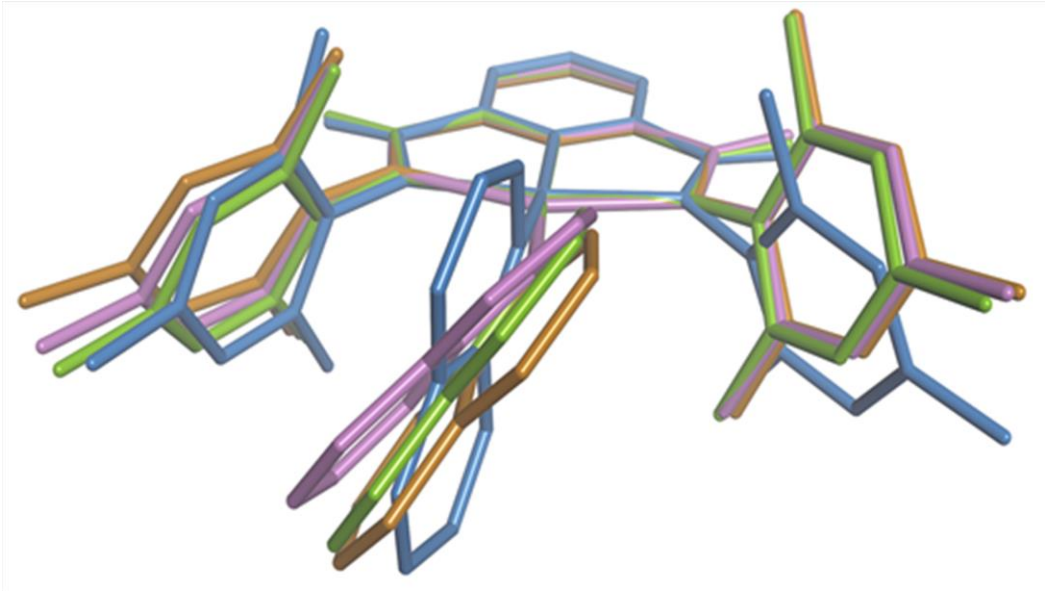


Figure 3.1: Diagram showing the overlaid DFT output structures. X-ray (blue), wB97XD (green), HSEh1PBE (orange), M06 (pink).

Table 3.1: Table of RMSD values. SI (all): similarity index for all atoms

Color	**Structure**	**RMSD 1**	**SI (all)**
blue	X-ray	0	1
green	wB97XD	0.0906	0.913
orange	HSEh1PBE	0.155	0.891
pink	M06	0.161	0.897

RMSD 1: metal, 3 x N atoms, 2 x carbonyl carbon (6 atoms total)

M06: hybrid functional parametrized for transition metals, including dispersion

wB97XD: Long range corrected functionals. The non-Coulomb part of exchange functionals typically dies off too rapidly and becomes very inaccurate at large distances, making them unsuitable for modeling processes such as electron excitations to high orbitals. Various schemes have been devised to handle such cases. wB97XD: The latest functional from Head-Gordon and coworkers, which includes empirical dispersion.

Electronic appendix content:

See attached DVD:

DVD contents:

Files:

- **AIMAll .mgpvis files:** Contains output files from the AIM analysis that was concluded on all compounds discussed.
- **Gaussian 09 .gif and .out files:** Contains input and output files used in the DFT studies of the compounds.
- **Structure images:** Contains .png structural images of the compounds studied.
- **Tabulated DFT results:** Contains Microsoft Excel spreadsheets that contain raw structural and electronic parameters and data.