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6-Chloro-3-[4-(hexyloxy)phenyl]-[1,2,4]triazolo-[4,3-*b*]pyridazine

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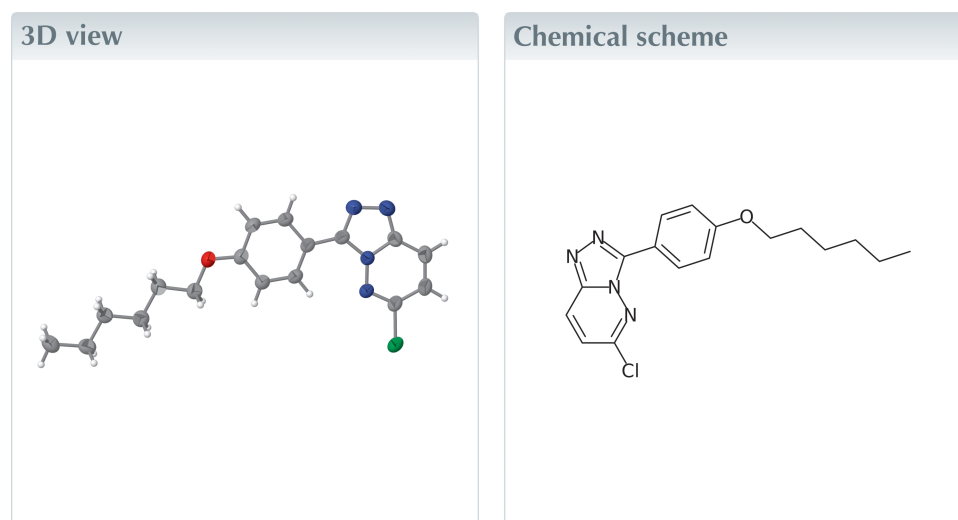
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Keywords: crystal structure; heterocycles.**CCDC reference:** 2466356**Structural data:** full structural data are available from iucrdata.iucr.org

Molecules of the title compound, C₁₇H₁₉ClN₄O, are essentially planar, the dihedral angles between the planes of the tetraazaindene ring system and the benzene ring and between the benzene ring and the hexyloxy chain being 1.56 (10)° and 5.02 (17)°, respectively. In the crystal, pairs of molecules are connected *via* π - π interactions between the phenyl ring and the triazolopyridazine moiety.



Structure description

The title compound (Fig. 1) was prepared in preparation for a project on discotic liquid crystals (Jochem *et al.*, 2022; Rieth *et al.*, 2020; Tober *et al.*, 2019). Huisgen and co-workers reported the triazoloannulation to 1,3,5-triazine (Huisgen *et al.*, 1960), but other chloroazines are also suitable substrates (Preis *et al.*; 2011, Schollmeyer & Detert, 2014). Molecules of the title compound, C₁₇H₁₉N₄OCl, are almost completely planar. The tetraazaindene framework (C1–N9) is planar (r.m.s. deviation 0.0098 Å). Further minor deviations from planarity are the small dihedral angle of 1.56 (3)° between the planes of tetraazaindene and the phenyl ring and of 5.02 (3)° between phenyl ring and the planar hexyloxy chain in a perfect *all-anti* conformation. A small distance of 3.5056 (13) Å indicates π - π interactions between the centroids of the bicyclic unit and of the phenyl ring. These neighbouring molecules are connected *via* a centre of inversion. In the monoclinic unit cell, eight of the planar molecules are arranged in planes parallel to (302) (Fig. 2).

Synthesis and crystallization

A solution of 5-*p*-hexyloxyphenyltetrazole (200 mg) and 3,6-dichloropyridazine (60 mg) in 10 ml xylenes/pyridine (5/1) was slowly heated to reflux and stirred at this temperature for 8 d. The solution was extracted with 1 *N* hydrochloric acid and brine, solvents were evaporated, and the residue was purified by chromatography on silica using toluene/ethyl

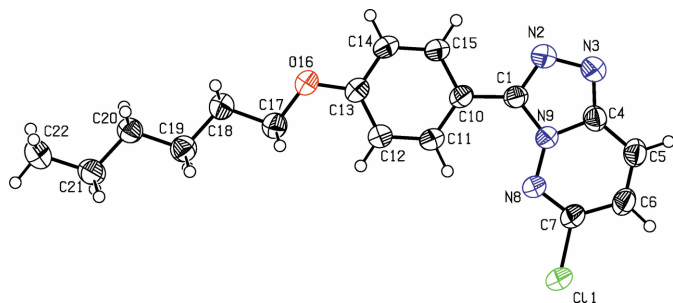


Figure 1
View (Spek, 2009) of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

acetate 2/1 with 1% triethylamine as eluent. Yield: 39 mg (29%) of a brownish, crystalline solid with m.p. = 375 K. The annotation of NMR signals follows IUPAC nomenclature. ¹H-NMR (400 MHz, CDCl₃): 8.36 (*d*, *J* = 9 Hz, 2 H, 2-H, 6-H ph); 8.09 (*d*, *J* = 9.6 Hz, 1H, 4-H pyridazin), 7.09 (*d*, *J* = 9.6 Hz, 1H, 5-H pyridazin), 7.03 (*d*, *J* = 9 Hz, 2 H, 3-H, 5-H ph), 4.01 (*t*, *J* = 6.6 Hz, 2 H, OCH₂), 1.83–1.76 (*m*, 2 H, CH₂), 1.50–1.43 (*m*, 2 H, CH₂), 1.36–1.31 (*m*, 4 H, CH₂), 0.90 (*t*, 3 H, CH₃); ¹³C-NMR (100 MHz, CDCl₃): 161.0 (C4 ph), 149.0 (C6 pyridazin), 148.0 (C-1 ph), 143.3 (C-3 pyridazin), 129.2 (C-2,6 ph), 126.5 (C4 pyridazin), 121.4 (C-5 pyridazin), 117.6 (C-1 ph), 114.7 (C-3,5 ph), 68.1 (OCH₂), 31.5, 25.7, 22.6, 14.0 (CH₂), 14.0 (CH₃). IR: (ATR): 3045, 2956, 2938, 2918, 2865, 1608, 1538, 1458, 1253, 1057, 830 cm⁻¹. FD–MS: 330.1 (*M*⁺); HR-ESI-MS: found 353.1160, calculated 3351.1145 for *M*+Na⁺.

Refinement

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

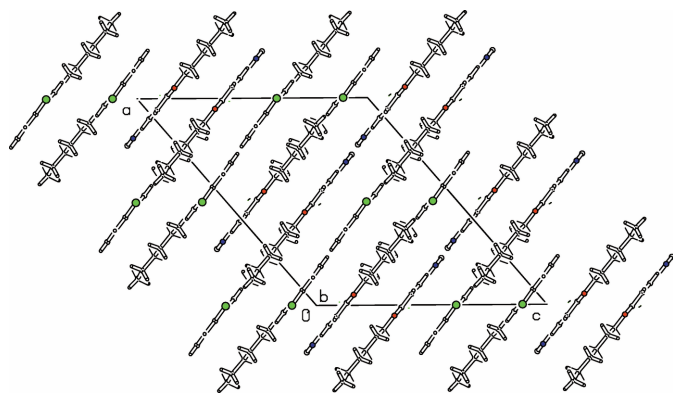


Figure 2
Part of the packing diagram. View along the *b*-axis direction (Spek, 2009).

Table 1
Experimental details.

Crystal data	
Chemical formula	C ₁₇ H ₁₉ ClN ₄ O
<i>M</i> _r	330.81
Crystal system, space group	Monoclinic, <i>C2/c</i>
Temperature (K)	193
<i>a</i> , <i>b</i> , <i>c</i> (Å)	21.4463 (13), 11.3646 (3), 18.0798 (11)
β (°)	130.696 (2)
<i>V</i> (Å ³)	3341.0 (3)
<i>Z</i>	8
Radiation type	Cu <i>K</i> α
μ (mm ⁻¹)	2.10
Crystal size (mm)	0.55 × 0.45 × 0.30
Data collection	
Diffractometer	Enraf–Nonius CAD-4
Absorption correction	ψ scan (<i>Corinc</i> ; Dräger & Gattow, 1971)
<i>T</i> _{min} , <i>T</i> _{max}	0.842, 0.996
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	3271, 3163, 2806
<i>R</i> _{int}	0.059
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.609
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.042, 0.124, 1.04
No. of reflections	3163
No. of parameters	209
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.24, −0.28

Computer programs: *CAD-4 Software V5* (Enraf–Nonius, 1989), *Corinc* (Dräger & Gattow, 1971), *SIR97* (Altomare *et al.*, 1999), *SHELXL2019/2* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

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full crystallographic data

IUCrData (2025). **10**, x250566 [<https://doi.org/10.1107/S2414314625005668>]

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6-Chloro-3-[4-(hexyloxy)phenyl]-[1,2,4]triazolo[4,3-*b*]pyridazine*Crystal data*

$C_{17}H_{19}ClN_4O$

$M_r = 330.81$

Monoclinic, $C2/c$

$a = 21.4463$ (13) Å

$b = 11.3646$ (3) Å

$c = 18.0798$ (11) Å

$\beta = 130.696$ (2)°

$V = 3341.0$ (3) Å³

$Z = 8$

$F(000) = 1392$

$D_x = 1.315$ Mg m⁻³

Melting point: 375 K

Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å

Cell parameters from 25 reflections

$\theta = 65$ – 70°

$\mu = 2.10$ mm⁻¹

$T = 193$ K

Block, light brown

$0.55 \times 0.45 \times 0.30$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: rotating anode

Graphite monochromator

$\omega/2\theta$ scans

Absorption correction: ψ scan

(*Corinc*; Dräger & Gattow, 1971)

$T_{\min} = 0.842$, $T_{\max} = 0.996$

3271 measured reflections

3163 independent reflections

2806 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.059$

$\theta_{\max} = 70.0^\circ$, $\theta_{\min} = 4.6^\circ$

$h = -19 \rightarrow 26$

$k = 0 \rightarrow 13$

$l = -22 \rightarrow 0$

3 standard reflections every 60 min

intensity decay: 2%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.124$

$S = 1.04$

3163 reflections

209 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0751P)^2 + 1.3637P]$

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

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

$\Delta\rho_{\max} = 0.24$ e Å⁻³

$\Delta\rho_{\min} = -0.28$ e Å⁻³

Special details

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

Refinement. Hydrogen atoms were placed at calculated positions and were refined in the riding-model approximation with $C_{\text{aromatic}}\text{-H} = 0.95 \text{ \AA}$ and with $C\text{-H} = 0.99 \text{ \AA}$ for the remaining H atoms, and with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C}_{\text{methyl}})$ and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ for the other H atoms.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.49915 (3)	0.11106 (4)	0.10533 (3)	0.06749 (19)
C1	0.37217 (9)	0.49223 (14)	-0.07286 (11)	0.0422 (3)
N2	0.30929 (9)	0.51615 (13)	-0.16533 (10)	0.0541 (4)
N3	0.27439 (9)	0.41338 (14)	-0.21728 (11)	0.0574 (4)
C4	0.31738 (10)	0.32689 (15)	-0.15461 (12)	0.0468 (4)
C5	0.31084 (11)	0.20313 (16)	-0.16525 (13)	0.0510 (4)
H5	0.268361	0.167534	-0.226405	0.061*
C6	0.36682 (11)	0.13741 (16)	-0.08582 (13)	0.0522 (4)
H6	0.365474	0.053973	-0.089820	0.063*
C7	0.42824 (11)	0.19581 (14)	0.00451 (12)	0.0481 (4)
N8	0.43661 (8)	0.30881 (11)	0.01962 (9)	0.0428 (3)
N9	0.37955 (8)	0.37244 (11)	-0.06273 (9)	0.0398 (3)
C10	0.42205 (9)	0.58130 (13)	0.00276 (11)	0.0408 (3)
C11	0.48876 (10)	0.55610 (14)	0.09998 (12)	0.0462 (4)
H11	0.503990	0.476445	0.119914	0.055*
C12	0.53343 (10)	0.64531 (15)	0.16833 (12)	0.0476 (4)
H12	0.578976	0.626419	0.234247	0.057*
C13	0.51163 (10)	0.76159 (14)	0.14048 (12)	0.0441 (4)
C14	0.44518 (11)	0.78813 (14)	0.04373 (13)	0.0494 (4)
H14	0.430003	0.867913	0.024251	0.059*
C15	0.40134 (10)	0.70008 (14)	-0.02385 (12)	0.0476 (4)
H15	0.356206	0.719750	-0.089757	0.057*
O16	0.55092 (8)	0.85588 (10)	0.20141 (9)	0.0520 (3)
C17	0.61958 (11)	0.83485 (15)	0.30264 (12)	0.0496 (4)
H17A	0.664066	0.793793	0.309993	0.060*
H17B	0.602617	0.785342	0.331914	0.060*
C18	0.64901 (11)	0.95325 (16)	0.35216 (12)	0.0517 (4)
H18A	0.603087	0.994333	0.341648	0.062*
H18B	0.665797	1.001365	0.322062	0.062*
C19	0.72129 (11)	0.94299 (16)	0.46149 (13)	0.0513 (4)
H19A	0.702989	0.900642	0.492264	0.062*
H19B	0.765200	0.895645	0.471925	0.062*
C20	0.75647 (10)	1.06144 (16)	0.51114 (12)	0.0489 (4)
H20A	0.775972	1.102823	0.481346	0.059*
H20B	0.712101	1.109480	0.499045	0.059*
C21	0.82697 (11)	1.05264 (17)	0.62029 (13)	0.0558 (4)
H21A	0.869662	1.000154	0.632441	0.067*
H21B	0.806465	1.015937	0.650538	0.067*
C22	0.86625 (13)	1.1704 (2)	0.66969 (15)	0.0689 (6)
H22A	0.890830	1.204507	0.644107	0.103*
H22B	0.909034	1.158522	0.740232	0.103*

H22C 0.824192 1.223861 0.656558 0.103*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0834 (4)	0.0346 (2)	0.0562 (3)	0.00329 (19)	0.0331 (3)	0.00725 (17)
C1	0.0409 (8)	0.0376 (8)	0.0442 (8)	0.0017 (6)	0.0259 (7)	0.0044 (6)
N2	0.0495 (8)	0.0452 (8)	0.0473 (8)	0.0014 (6)	0.0226 (7)	0.0035 (6)
N3	0.0511 (8)	0.0510 (8)	0.0463 (8)	-0.0028 (7)	0.0213 (7)	0.0013 (6)
C4	0.0442 (8)	0.0476 (9)	0.0421 (8)	-0.0065 (7)	0.0254 (7)	-0.0027 (7)
C5	0.0532 (9)	0.0470 (9)	0.0500 (9)	-0.0121 (7)	0.0324 (8)	-0.0089 (7)
C6	0.0608 (10)	0.0381 (8)	0.0559 (10)	-0.0097 (7)	0.0373 (9)	-0.0061 (7)
C7	0.0570 (9)	0.0352 (8)	0.0504 (9)	-0.0023 (7)	0.0342 (8)	0.0021 (7)
N8	0.0469 (7)	0.0346 (7)	0.0421 (7)	0.0004 (5)	0.0270 (6)	0.0035 (5)
N9	0.0404 (6)	0.0351 (6)	0.0398 (7)	-0.0013 (5)	0.0244 (6)	0.0013 (5)
C10	0.0416 (8)	0.0338 (7)	0.0471 (8)	0.0009 (6)	0.0290 (7)	0.0023 (6)
C11	0.0490 (8)	0.0346 (8)	0.0477 (9)	0.0035 (6)	0.0283 (8)	0.0053 (6)
C12	0.0501 (9)	0.0395 (8)	0.0440 (8)	0.0023 (7)	0.0266 (8)	0.0017 (7)
C13	0.0481 (8)	0.0370 (8)	0.0504 (9)	0.0008 (6)	0.0334 (8)	-0.0008 (6)
C14	0.0531 (9)	0.0336 (8)	0.0541 (9)	0.0063 (7)	0.0317 (8)	0.0052 (7)
C15	0.0458 (8)	0.0379 (8)	0.0472 (9)	0.0047 (7)	0.0252 (7)	0.0061 (7)
O16	0.0586 (7)	0.0372 (6)	0.0490 (7)	0.0005 (5)	0.0302 (6)	-0.0034 (5)
C17	0.0540 (9)	0.0458 (9)	0.0483 (9)	0.0007 (7)	0.0331 (8)	-0.0015 (7)
C18	0.0531 (9)	0.0462 (9)	0.0514 (10)	-0.0002 (7)	0.0321 (8)	-0.0034 (7)
C19	0.0529 (9)	0.0499 (10)	0.0521 (9)	-0.0004 (8)	0.0346 (8)	0.0001 (8)
C20	0.0504 (9)	0.0496 (10)	0.0451 (9)	0.0020 (7)	0.0304 (8)	0.0015 (7)
C21	0.0532 (9)	0.0597 (11)	0.0466 (9)	0.0050 (8)	0.0290 (8)	0.0039 (8)
C22	0.0638 (12)	0.0718 (14)	0.0470 (10)	-0.0048 (10)	0.0255 (9)	-0.0034 (9)

Geometric parameters (Å, °)

C11—C7	1.7169 (17)	C14—C15	1.371 (2)
C1—N2	1.322 (2)	C14—H14	0.9500
C1—N9	1.369 (2)	C15—H15	0.9500
C1—C10	1.460 (2)	O16—C17	1.435 (2)
N2—N3	1.375 (2)	C17—C18	1.508 (2)
N3—C4	1.318 (2)	C17—H17A	0.9900
C4—N9	1.384 (2)	C17—H17B	0.9900
C4—C5	1.414 (2)	C18—C19	1.526 (2)
C5—C6	1.346 (3)	C18—H18A	0.9900
C5—H5	0.9500	C18—H18B	0.9900
C6—C7	1.427 (2)	C19—C20	1.518 (2)
C6—H6	0.9500	C19—H19A	0.9900
C7—N8	1.301 (2)	C19—H19B	0.9900
N8—N9	1.3642 (18)	C20—C21	1.516 (2)
C10—C11	1.393 (2)	C20—H20A	0.9900
C10—C15	1.404 (2)	C20—H20B	0.9900
C11—C12	1.389 (2)	C21—C22	1.522 (3)

C11—H11	0.9500	C21—H21A	0.9900
C12—C13	1.383 (2)	C21—H21B	0.9900
C12—H12	0.9500	C22—H22A	0.9800
C13—O16	1.364 (2)	C22—H22B	0.9800
C13—C14	1.390 (2)	C22—H22C	0.9800
N2—C1—N9	107.90 (14)	C10—C15—H15	119.5
N2—C1—C10	124.21 (14)	C13—O16—C17	118.61 (13)
N9—C1—C10	127.88 (14)	O16—C17—C18	107.02 (14)
C1—N2—N3	109.95 (14)	O16—C17—H17A	110.3
C4—N3—N2	106.39 (13)	C18—C17—H17A	110.3
N3—C4—N9	109.83 (15)	O16—C17—H17B	110.3
N3—C4—C5	132.29 (16)	C18—C17—H17B	110.3
N9—C4—C5	117.88 (15)	H17A—C17—H17B	108.6
C6—C5—C4	117.79 (16)	C17—C18—C19	112.29 (15)
C6—C5—H5	121.1	C17—C18—H18A	109.1
C4—C5—H5	121.1	C19—C18—H18A	109.1
C5—C6—C7	118.57 (16)	C17—C18—H18B	109.1
C5—C6—H6	120.7	C19—C18—H18B	109.1
C7—C6—H6	120.7	H18A—C18—H18B	107.9
N8—C7—C6	126.68 (16)	C20—C19—C18	113.06 (15)
N8—C7—C11	115.18 (13)	C20—C19—H19A	109.0
C6—C7—C11	118.14 (13)	C18—C19—H19A	109.0
C7—N8—N9	113.04 (13)	C20—C19—H19B	109.0
N8—N9—C1	128.05 (13)	C18—C19—H19B	109.0
N8—N9—C4	126.02 (13)	H19A—C19—H19B	107.8
C1—N9—C4	105.92 (13)	C21—C20—C19	113.61 (15)
C11—C10—C15	117.73 (15)	C21—C20—H20A	108.8
C11—C10—C1	124.19 (14)	C19—C20—H20A	108.8
C15—C10—C1	118.08 (14)	C21—C20—H20B	108.8
C12—C11—C10	121.19 (15)	C19—C20—H20B	108.8
C12—C11—H11	119.4	H20A—C20—H20B	107.7
C10—C11—H11	119.4	C20—C21—C22	113.99 (16)
C13—C12—C11	120.03 (15)	C20—C21—H21A	108.8
C13—C12—H12	120.0	C22—C21—H21A	108.8
C11—C12—H12	120.0	C20—C21—H21B	108.8
O16—C13—C12	124.93 (15)	C22—C21—H21B	108.8
O16—C13—C14	115.63 (14)	H21A—C21—H21B	107.7
C12—C13—C14	119.44 (16)	C21—C22—H22A	109.5
C15—C14—C13	120.52 (15)	C21—C22—H22B	109.5
C15—C14—H14	119.7	H22A—C22—H22B	109.5
C13—C14—H14	119.7	C21—C22—H22C	109.5
C14—C15—C10	121.08 (15)	H22A—C22—H22C	109.5
C14—C15—H15	119.5	H22B—C22—H22C	109.5
N9—C1—N2—N3	0.05 (19)	N2—C1—C10—C11	-179.38 (16)
C10—C1—N2—N3	-178.86 (14)	N9—C1—C10—C11	1.9 (2)
C1—N2—N3—C4	-0.1 (2)	N2—C1—C10—C15	1.0 (2)

N2—N3—C4—N9	0.12 (19)	N9—C1—C10—C15	-177.68 (15)
N2—N3—C4—C5	-179.79 (18)	C15—C10—C11—C12	-0.1 (2)
N3—C4—C5—C6	178.23 (18)	C1—C10—C11—C12	-179.74 (15)
N9—C4—C5—C6	-1.7 (2)	C10—C11—C12—C13	0.4 (3)
C4—C5—C6—C7	1.2 (2)	C11—C12—C13—O16	179.81 (15)
C5—C6—C7—N8	0.2 (3)	C11—C12—C13—C14	-0.4 (3)
C5—C6—C7—C11	179.87 (14)	O16—C13—C14—C15	179.88 (15)
C6—C7—N8—N9	-1.0 (2)	C12—C13—C14—C15	0.0 (3)
C11—C7—N8—N9	179.28 (10)	C13—C14—C15—C10	0.3 (3)
C7—N8—N9—C1	-178.30 (15)	C11—C10—C15—C14	-0.2 (2)
C7—N8—N9—C4	0.5 (2)	C1—C10—C15—C14	179.43 (15)
N2—C1—N9—N8	179.00 (14)	C12—C13—O16—C17	-0.9 (2)
C10—C1—N9—N8	-2.1 (3)	C14—C13—O16—C17	179.32 (14)
N2—C1—N9—C4	0.02 (17)	C13—O16—C17—C18	-178.57 (13)
C10—C1—N9—C4	178.88 (15)	O16—C17—C18—C19	178.74 (13)
N3—C4—N9—N8	-179.09 (14)	C17—C18—C19—C20	175.09 (14)
C5—C4—N9—N8	0.8 (2)	C18—C19—C20—C21	178.53 (15)
N3—C4—N9—C1	-0.09 (18)	C19—C20—C21—C22	176.26 (17)
C5—C4—N9—C1	179.84 (15)		
