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1,3,5-Tris(6-chloropyrazin-2-yloxy)-benzene

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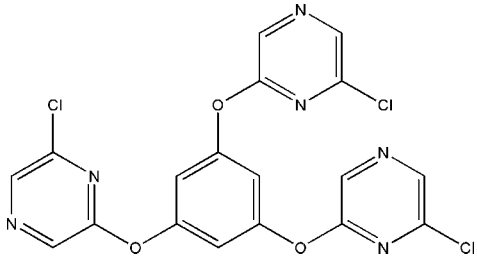
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.108; data-to-parameter ratio = 12.5.

In the title compound, $C_{18}H_9Cl_3N_6O_3$, all bond lengths and angles are normal. The dihedral angles between the benzene ring and the three pyrazine rings are 72.67 (2), 60.73 (3) and 77.74 (2)°. The crystal packing is stabilized by van der Waals forces and by a weak π–π stacking interaction between pyrazine rings, with a centroid–centroid distance of 3.487 (2) Å.

Related literature

For related literatures see: Carter & Boer (1974); Seitz *et al.* (2002); Temple *et al.* (1970).



Experimental

Crystal data

$C_{18}H_9Cl_3N_6O_3$
M_r = 463.66
Triclinic, $P\overline{1}$
a = 9.680 (2) Å
b = 10.658 (2) Å
c = 11.039 (3) Å
α = 72.768 (3)°
β = 68.308 (3)°
γ = 69.342 (3)°
V = 972.0 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.51 mm^{-1}
T = 298 (2) K
0.58 × 0.31 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.758, T_{max} = 0.863
4925 measured reflections
3375 independent reflections
2797 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.108
S = 1.04
3375 reflections
271 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2085).

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supplementary materials

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1,3,5-Tris(6-chloropyrazin-2-yloxy)benzene

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Comment

Pyrazine derivatives were shown to display antimycobacterial (Seitz *et al.*, 2002) and potential antimalarial (Temple *et al.*, 1970) activities. The title compound was prepared for the screening of these bioactivities. We report here the crystal structure of (I).

In (I) (Fig. 1), all bond lengths and angles are normal and in a good agreement with those reported previously (Carter & Boer, 1974). The dihedral angles between benzene ring (C1—C6) and three Pyrazine rings (C7—C10/N1/N2; C11—C14/N3/N4; C15—C18/N5/N6) are 72.67 (2), 60.73 (3) and 77.74 (2)°, respectively. The crystal packing is stabilized by weak π–π stacking interactions and van der Waals forces, proved by the shorter distance C*g*1···C*g*1^{ii} of 3.487 (2) Å, where C*g*1 is a centroid of Pyrazine ring (C11—C14/N3/N4) [symmetry code:(ii) –*X*,1-Y,2-*Z*].

Experimental

A flask was charged with 1.26 g (10 mmol) of 1,3,5-trihydroxybenzene, 4.47 g (30 mmol) of 2,6-dichloropyrazine and 3.18 g (30 mmol) of sodium carbonate, followed by addition of 50 ml of dried MeCN. The resultant mixture was refluxed over night. On cooling, the reaction mixture was filtered and the filtrate was portioned between 150 ml of water and 200 ml of dichloromethane. The organic phase was washed with brine, dried over sodium sulfate and evaporated on a rotary evaporator to furnish the crude product as a residue, which was chromatographed on silica gel to afford the pure product as colorless prisms. Crystals suitable for X-ray diffraction were obtained *via* slow evaporation of a solution of the title compound in ethyl acetate.

Refinement

All H atoms were placed in calculated positions, with C—H = 0.93 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2 times U_{eq}(C).

Figures

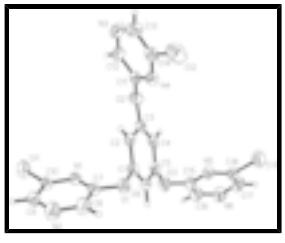


Fig. 1. View of the title compound (I), with displacement ellipsoids drawn at the 40% probability level.

1,3,5-Tris(6-chloropyrazin-2-yloxy)benzene

Crystal data

$C_{18}H_9Cl_3N_6O_3$	$Z = 2$
$M_r = 463.66$	$F_{000} = 468$
Triclinic, $P\overline{1}$	$D_x = 1.584$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.680$ (2) Å	Cell parameters from 1699 reflections
$b = 10.658$ (2) Å	$\theta = 2.8$–23.1º
$c = 11.039$ (3) Å	$\mu = 0.51$ mm^{-1}
$\alpha = 72.768$ (3)º	$T = 298$ (2) K
$\beta = 68.308$ (3)º	Prism, colorless
$\gamma = 69.342$ (3)º	0.58 × 0.31 × 0.30 mm
$V = 972.0$ (4) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	3375 independent reflections
Radiation source: fine-focus sealed tube	2797 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.017$
$T = 298$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 2.0$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$h = -9 \rightarrow 11$
$T_{min} = 0.758$, $T_{max} = 0.863$	$k = -12 \rightarrow 12$
4925 measured reflections	$l = -13 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters constrained
$wR(F^2) = 0.108$	$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.2339P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.001$
3375 reflections	$\Delta\rho_{max} = 0.22$ e Å^{-3}
271 parameters	$\Delta\rho_{min} = -0.39$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

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

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.04661 (7)	0.67966 (7)	0.44208 (6)	0.0642 (2)
Cl2	0.43548 (9)	1.27994 (7)	−0.59901 (7)	0.0772 (2)
Cl3	0.32169 (10)	1.47146 (8)	0.07383 (9)	0.0893 (3)
O1	0.43622 (16)	0.85408 (15)	0.05573 (15)	0.0530 (4)
O2	0.1867 (2)	0.93858 (16)	−0.27062 (15)	0.0616 (4)
O3	−0.01216 (16)	1.23869 (14)	0.03280 (16)	0.0513 (4)
N1	−0.1102 (3)	1.4653 (2)	0.2505 (2)	0.0743 (6)
N2	0.4938 (2)	0.6220 (2)	0.3517 (2)	0.0701 (6)
N3	0.2331 (3)	1.0283 (2)	−0.6181 (2)	0.0672 (6)
N4	0.1437 (2)	1.34503 (17)	0.05891 (17)	0.0469 (4)
N5	0.3057 (2)	1.09657 (17)	−0.42807 (17)	0.0459 (4)
N6	0.25692 (18)	0.76867 (16)	0.23874 (16)	0.0409 (4)
C1	0.0239 (4)	1.4929 (3)	0.2147 (3)	0.0718 (8)
H1B	0.0338	1.5541	0.2539	0.086*
C2	0.3070 (3)	1.1226 (3)	−0.6499 (2)	0.0600 (6)
H2B	0.3358	1.1674	−0.7377	0.072*
C3	−0.1181 (3)	1.3782 (3)	0.1916 (2)	0.0610 (6)
H3B	−0.2104	1.3560	0.2147	0.073*
C4	0.5191 (3)	0.7021 (3)	0.2337 (2)	0.0626 (7)
H4B	0.6188	0.7094	0.1867	0.075*
C5	0.1484 (3)	1.4320 (2)	0.1203 (2)	0.0542 (6)
C6	0.1940 (3)	0.9688 (3)	−0.4911 (2)	0.0602 (6)
H6B	0.1415	0.9025	−0.4643	0.072*
C7	0.3414 (2)	1.1549 (2)	−0.5553 (2)	0.0488 (5)
C8	0.2308 (3)	1.0047 (2)	−0.3975 (2)	0.0470 (5)
C9	0.3493 (3)	0.6156 (2)	0.4159 (2)	0.0540 (6)
H9A	0.3264	0.5614	0.5000	0.065*
C10	0.2025 (3)	0.9832 (2)	−0.1693 (2)	0.0467 (5)
C11	0.3125 (2)	0.8988 (2)	−0.1081 (2)	0.0467 (5)
H11A	0.3812	0.8202	−0.1386	0.056*
C12	0.2348 (2)	0.68880 (19)	0.3581 (2)	0.0407 (5)
C13	0.3169 (2)	0.9348 (2)	−0.0002 (2)	0.0421 (5)
C14	0.0097 (2)	1.3191 (2)	0.0952 (2)	0.0440 (5)

C15	0.1076 (2)	1.12977 (19)	−0.0168 (2)	0.0403 (5)
C16	0.2156 (2)	1.0492 (2)	0.0488 (2)	0.0409 (4)
H16A	0.2196	1.0713	0.1226	0.049*
C17	0.0996 (2)	1.0992 (2)	−0.1265 (2)	0.0444 (5)
H17A	0.0268	1.1553	−0.1699	0.053*
C18	0.3995 (2)	0.77556 (19)	0.1787 (2)	0.0419 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0539 (3)	0.0787 (4)	0.0503 (4)	−0.0223 (3)	−0.0150 (3)	0.0057 (3)
Cl2	0.1024 (5)	0.0756 (4)	0.0561 (4)	−0.0460 (4)	−0.0100 (4)	−0.0072 (3)
Cl3	0.0920 (5)	0.0802 (5)	0.1186 (7)	−0.0405 (4)	−0.0433 (5)	−0.0155 (4)
O1	0.0408 (8)	0.0582 (9)	0.0452 (9)	−0.0084 (7)	−0.0133 (7)	0.0055 (7)
O2	0.0966 (13)	0.0628 (10)	0.0390 (9)	−0.0401 (9)	−0.0227 (8)	−0.0036 (7)
O3	0.0463 (8)	0.0467 (8)	0.0619 (10)	−0.0073 (6)	−0.0212 (7)	−0.0118 (7)
N1	0.0842 (17)	0.0791 (15)	0.0478 (13)	−0.0113 (13)	−0.0091 (11)	−0.0225 (11)
N2	0.0501 (12)	0.0856 (15)	0.0539 (13)	−0.0002 (10)	−0.0244 (10)	0.0053 (11)
N3	0.0846 (15)	0.0855 (15)	0.0436 (12)	−0.0288 (12)	−0.0243 (11)	−0.0158 (11)
N4	0.0521 (10)	0.0416 (9)	0.0439 (10)	−0.0120 (8)	−0.0150 (8)	−0.0040 (8)
N5	0.0531 (10)	0.0477 (10)	0.0367 (10)	−0.0103 (8)	−0.0156 (8)	−0.0090 (8)
N6	0.0451 (10)	0.0397 (9)	0.0361 (9)	−0.0061 (7)	−0.0174 (8)	−0.0044 (7)
C1	0.105 (2)	0.0596 (15)	0.0553 (16)	−0.0137 (15)	−0.0331 (16)	−0.0168 (13)
C2	0.0684 (15)	0.0727 (16)	0.0363 (12)	−0.0164 (13)	−0.0167 (11)	−0.0086 (11)
C3	0.0580 (14)	0.0666 (15)	0.0427 (13)	−0.0117 (12)	−0.0069 (11)	−0.0049 (12)
C4	0.0421 (12)	0.0782 (16)	0.0521 (15)	−0.0057 (11)	−0.0173 (11)	0.0003 (12)
C5	0.0693 (15)	0.0441 (12)	0.0515 (14)	−0.0153 (11)	−0.0271 (12)	−0.0008 (10)
C6	0.0758 (16)	0.0650 (14)	0.0508 (15)	−0.0256 (13)	−0.0228 (12)	−0.0137 (12)
C7	0.0502 (12)	0.0509 (12)	0.0394 (12)	−0.0091 (10)	−0.0103 (9)	−0.0097 (10)
C8	0.0562 (12)	0.0474 (12)	0.0385 (12)	−0.0122 (10)	−0.0172 (10)	−0.0080 (9)
C9	0.0569 (14)	0.0536 (12)	0.0395 (12)	−0.0016 (10)	−0.0201 (10)	−0.0010 (10)
C10	0.0627 (13)	0.0500 (12)	0.0312 (11)	−0.0271 (10)	−0.0145 (10)	0.0016 (9)
C11	0.0499 (12)	0.0434 (11)	0.0393 (12)	−0.0156 (9)	−0.0046 (9)	−0.0049 (9)
C12	0.0454 (11)	0.0378 (10)	0.0363 (11)	−0.0049 (8)	−0.0156 (9)	−0.0067 (8)
C13	0.0395 (10)	0.0442 (11)	0.0357 (11)	−0.0132 (8)	−0.0114 (9)	0.0045 (9)
C14	0.0487 (12)	0.0385 (10)	0.0361 (11)	−0.0070 (9)	−0.0142 (9)	0.0012 (8)
C15	0.0423 (11)	0.0369 (10)	0.0390 (11)	−0.0141 (8)	−0.0114 (9)	−0.0004 (8)
C16	0.0450 (11)	0.0460 (11)	0.0325 (10)	−0.0157 (9)	−0.0140 (9)	−0.0017 (8)
C17	0.0537 (12)	0.0446 (11)	0.0379 (11)	−0.0208 (10)	−0.0214 (10)	0.0070 (9)
C18	0.0429 (11)	0.0400 (10)	0.0395 (11)	−0.0049 (8)	−0.0158 (9)	−0.0060 (9)

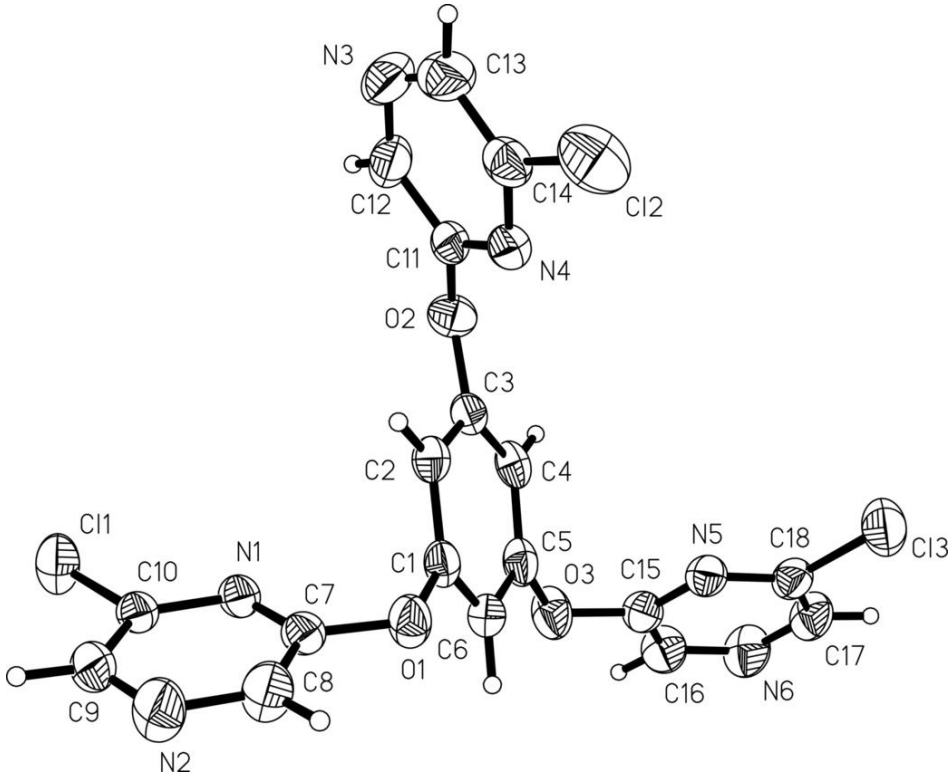
Geometric parameters (Å, °)

Cl1—C12	1.735 (2)	C1—C5	1.371 (4)
Cl2—C7	1.728 (2)	C1—H1B	0.9300
Cl3—C5	1.729 (3)	C2—C7	1.367 (3)
O1—C18	1.360 (2)	C2—H2B	0.9300
O1—C13	1.402 (2)	C3—C14	1.390 (3)
O2—C8	1.351 (3)	C3—H3B	0.9300

O2—C10	1.411 (3)	C4—C18	1.386 (3)
O3—C14	1.357 (3)	C4—H4B	0.9300
O3—C15	1.396 (2)	C6—C8	1.394 (3)
N1—C3	1.317 (3)	C6—H6B	0.9300
N1—C1	1.323 (4)	C9—C12	1.364 (3)
N2—C4	1.319 (3)	C9—H9A	0.9300
N2—C9	1.330 (3)	C10—C17	1.369 (3)
N3—C2	1.326 (3)	C10—C11	1.377 (3)
N3—C6	1.326 (3)	C11—C13	1.374 (3)
N4—C14	1.312 (3)	C11—H11A	0.9300
N4—C5	1.320 (3)	C13—C16	1.377 (3)
N5—C8	1.312 (3)	C15—C17	1.379 (3)
N5—C7	1.325 (3)	C15—C16	1.382 (3)
N6—C18	1.310 (3)	C16—H16A	0.9300
N6—C12	1.328 (2)	C17—H17A	0.9300
C18—O1—C13	118.64 (15)	O2—C8—C6	116.6 (2)
C8—O2—C10	118.93 (16)	N2—C9—C12	119.8 (2)
C14—O3—C15	121.38 (16)	N2—C9—H9A	120.1
C3—N1—C1	117.2 (2)	C12—C9—H9A	120.1
C4—N2—C9	117.46 (19)	C17—C10—C11	122.6 (2)
C2—N3—C6	116.8 (2)	C17—C10—O2	119.58 (19)
C14—N4—C5	115.09 (19)	C11—C10—O2	117.48 (19)
C8—N5—C7	114.59 (18)	C13—C11—C10	117.54 (19)
C18—N6—C12	114.85 (16)	C13—C11—H11A	121.2
N1—C1—C5	120.6 (2)	C10—C11—H11A	121.2
N1—C1—H1B	119.7	N6—C12—C9	124.2 (2)
C5—C1—H1B	119.7	N6—C12—Cl1	116.16 (14)
N3—C2—C7	120.9 (2)	C9—C12—Cl1	119.64 (17)
N3—C2—H2B	119.6	C11—C13—C16	122.78 (18)
C7—C2—H2B	119.6	C11—C13—O1	117.27 (18)
N1—C3—C14	120.9 (2)	C16—C13—O1	119.85 (19)
N1—C3—H3B	119.6	N4—C14—O3	120.43 (19)
C14—C3—H3B	119.6	N4—C14—C3	122.7 (2)
N2—C4—C18	121.0 (2)	O3—C14—C3	116.8 (2)
N2—C4—H4B	119.5	C17—C15—C16	122.55 (19)
C18—C4—H4B	119.5	C17—C15—O3	115.06 (17)
N4—C5—C1	123.6 (2)	C16—C15—O3	121.97 (19)
N4—C5—Cl3	116.79 (18)	C13—C16—C15	116.97 (19)
C1—C5—Cl3	119.6 (2)	C13—C16—H16A	121.5
N3—C6—C8	120.7 (2)	C15—C16—H16A	121.5
N3—C6—H6B	119.6	C10—C17—C15	117.57 (19)
C8—C6—H6B	119.6	C10—C17—H17A	121.2
N5—C7—C2	124.0 (2)	C15—C17—H17A	121.2
N5—C7—Cl2	116.06 (17)	N6—C18—O1	120.19 (17)
C2—C7—Cl2	119.97 (18)	N6—C18—C4	122.7 (2)
N5—C8—O2	120.29 (18)	O1—C18—C4	117.13 (19)
N5—C8—C6	123.1 (2)		
C3—N1—C1—C5	−0.2 (4)	N2—C9—C12—Cl1	179.78 (19)

C6—N3—C2—C7	0.5 (4)	C10—C11—C13—C16	1.0 (3)
C1—N1—C3—C14	−0.4 (4)	C10—C11—C13—O1	−175.44 (17)
C9—N2—C4—C18	−0.6 (4)	C18—O1—C13—C11	−111.4 (2)
C14—N4—C5—C1	−0.4 (3)	C18—O1—C13—C16	72.1 (2)
C14—N4—C5—Cl3	−178.98 (14)	C5—N4—C14—O3	175.50 (17)
N1—C1—C5—N4	0.7 (4)	C5—N4—C14—C3	−0.2 (3)
N1—C1—C5—Cl3	179.2 (2)	C15—O3—C14—N4	33.3 (3)
C2—N3—C6—C8	−0.4 (4)	C15—O3—C14—C3	−150.69 (19)
C8—N5—C7—C2	−1.0 (3)	N1—C3—C14—N4	0.7 (3)
C8—N5—C7—Cl2	178.31 (15)	N1—C3—C14—O3	−175.2 (2)
N3—C2—C7—N5	0.2 (4)	C14—O3—C15—C17	−150.15 (18)
N3—C2—C7—Cl2	−179.10 (19)	C14—O3—C15—C16	37.0 (3)
C7—N5—C8—O2	−179.47 (19)	C11—C13—C16—C15	−0.8 (3)
C7—N5—C8—C6	1.1 (3)	O1—C13—C16—C15	175.49 (16)
C10—O2—C8—N5	8.9 (3)	C17—C15—C16—C13	−0.1 (3)
C10—O2—C8—C6	−171.7 (2)	O3—C15—C16—C13	172.23 (17)
N3—C6—C8—N5	−0.5 (4)	C11—C10—C17—C15	−0.6 (3)
N3—C6—C8—O2	−179.9 (2)	O2—C10—C17—C15	172.20 (17)
C4—N2—C9—C12	1.0 (4)	C16—C15—C17—C10	0.8 (3)
C8—O2—C10—C17	76.1 (3)	O3—C15—C17—C10	−172.01 (17)
C8—O2—C10—C11	−110.8 (2)	C12—N6—C18—O1	179.91 (17)
C17—C10—C11—C13	−0.2 (3)	C12—N6—C18—C4	1.8 (3)
O2—C10—C11—C13	−173.20 (17)	C13—O1—C18—N6	4.8 (3)
C18—N6—C12—C9	−1.4 (3)	C13—O1—C18—C4	−177.0 (2)
C18—N6—C12—Cl1	178.83 (14)	N2—C4—C18—N6	−0.9 (4)
N2—C9—C12—N6	0.0 (3)	N2—C4—C18—O1	−179.0 (2)

Fig. 1


N3
C13
C12
C14
Cl2
C11
N4
O2
C3
C2
C4
Cl1
C10
N1
C1
C5
O3
C15
N5
C18
Cl3
C7
O1
C6
C8
C9
C16
N6
C17
N2