Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,4-Phenylene-bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide) bis(tetrabutylammonium chloride) acetonitrile disolvate

Mark E. Light,* Philip A. Gale and Korakot Navakhun

School of Chemistry, University of Southampton, Highfield, Southampton, England SO17 1BJ

Correspondence e-mail: light@soton.ac.uk

Received 22 March 2005
Accepted 6 April 2005
Online 9 April 2005

Key indicators

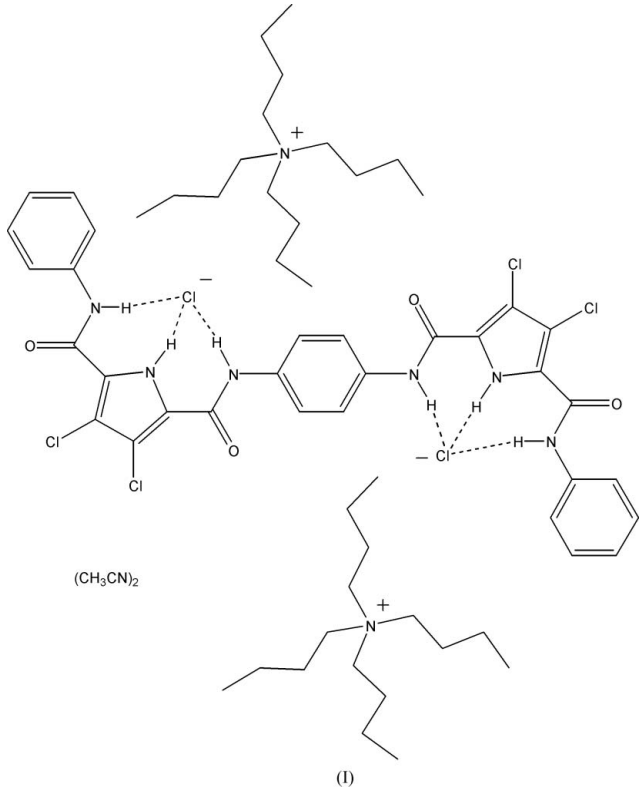
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
Disorder in solvent or counterion
R factor = 0.060
wR factor = 0.144
Data-to-parameter ratio = 18.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{30}H_{20}N_6O_4Cl_4.2C_{16}H_{36}N^+.2Cl^-.2C_2H_3N$, contains two hydrogen-bonded chloride anions bound to 1,4-phenylene-bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide) as the tetrabutylammonium salt. There is also a short pyrrolic hydrogen bond (N···Cl = 3.068 (3) Å), and two longer ones to the amino H atoms [N···Cl = 3.269 (3) Å and 3.275 (3) Å]. The neutral molecule lies on an inversion centre situated at the centre of the central benzene ring.

Comment

1,4-Phenylene-bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide) crystallizes from tetrabutylammonium chloride acetonitrile solution as a tetrabutylammonium chloride acetonitrile solvate, (I).



(I)

The receptor adopts an S-shaped conformation around a centre of inversion with one chloride bound on each side. The pyrrole and terminal benzene ring pairs are coplanar, and the angle between the central and terminal benzene rings is 32.02 (4)°. Of the three hydrogen bonds to the chloride, the pyrrolic one is the shortest, with an N···Cl distance of 3.068 (3) Å, whilst the two either side are longer, with distances of 3.269 (3) Å and 3.275 (3) Å.

Experimental

The receptor molecule, 1,4-phenylene-bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide), (1), was synthesized according to literature methods (Gale *et al.*, 2002). Crystals of the acetonitrile solvate of the tetrabutylammonium chloride complex were grown by slow evaporation of an acetonitrile solution of (1) in acetonitrile in the presence of excess tetrabutylammonium chloride.

Crystal data

$C_{30}H_{20}N_6O_4Cl_4\cdot 2C_{16}H_{36}N^+\cdot 2Cl^-\cdot 2C_2H_3N$
$M_r = 1308.24$
Monoclinic, $P2_1/c$
$a = 8.5720\ (2)$ Å
$b = 21.1088\ (5)$ Å
$c = 19.3520\ (6)$ Å
$\beta = 93.5560\ (10)^\circ$ **precision OK?**
$V = 3494.90\ (16)$ Å^3
$Z = 2$
$D_x = 1.243$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 35384 reflections
$\theta = 2.9$–26.4°
$\mu = 0.30$ mm^{-1}
$T = 120\ (2)$ K
Needle, colourless
0.15 × 0.07 × 0.05 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{min} = 0.906$, $T_{max} = 0.990$
13653 measured reflections
7107 independent reflections
4902 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 26.4^\circ$
$h = -10 \rightarrow 10$
$k = -26 \rightarrow 25$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.144$
$S = 1.11$
7107 reflections
395 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 4.3109P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.70$ e Å^{-3}
$\Delta\rho_{min} = -1.05$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0023 (4)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H91···Cl3	0.88	2.40	3.269 (3)	171.6
N2—H92···Cl3	0.88	2.20	3.068 (3)	166.8
N3—H93···Cl3	0.88	2.40	3.275 (3)	171.2

H atoms were identified in a difference map and then placed in calculated positions (N—H 0.88, aromatic C—H 0.95, methylene C—H 0.99, methyl C—H 0.98) and refined using a riding model [U_{iso}(H) = 1.2 or 1.5 times U_{eq}(parent atom)]. One arm of the tetrabutylammonium is disordered. It has been modelled as split over two possible orientations with one third and two thirds occupancy. C—C and C—N distances were restrained to standard values and the displacement parameters of split atom pairs were constrained to be equal. The deepest hole is located 1.28 Å from C9.

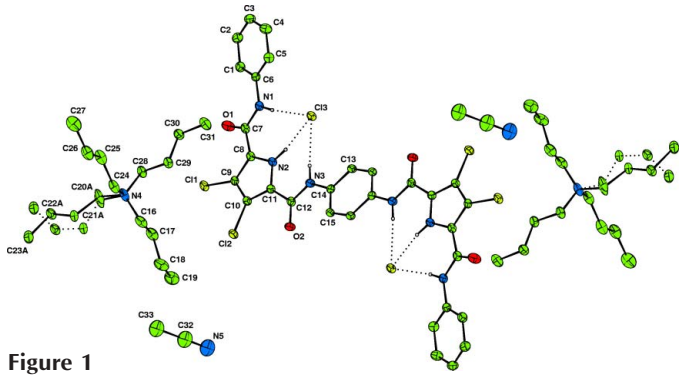


Figure 1
Structure of the title compound, with displacement ellipsoids drawn at the 35% probability level and non-acidic H atoms omitted for clarity. Both disorder components are shown.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors thank the EPSRC for funding the crystallographic facilities. PAG thanks the Royal Society for a University Research Fellowship and Universities UK for an ORS studentship to KN.

References

Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Gale, P. A., Navakhun, K., Camiolo, S., Light, M. E. & Hursthouse, M. B., (2002). *J. Am. Chem. Soc.* **124**, 11228–11229.
Hooft, R. (1998). *COLLECT*. Nonius BV, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Watkin, D. M., Pearce, L. & Prout, C. K. (1993). *CAMERON*. University of Oxford, England.

supporting information

Acta Cryst. (2005). E**61**, o1300–o1301 [https://doi.org/10.1107/S1600536805010688]

1,4-Phenylene-bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide) bis(tetrabutylammonium chloride) acetonitrile disolvate

Mark E. Light, Philip A. Gale and Korakot Navakhun

(I)

Crystal data

$C_{30}H_{20}N_6O_4Cl_4\cdot 2C_{16}H_{36}N^+\cdot 2Cl^-\cdot 2C_2H_3N$
$M_r = 1308.24$
Monoclinic, $P2_1/c$
$a = 8.5720$ (2) Å
$b = 21.1088$ (5) Å
$c = 19.3520$ (6) Å
$\beta = 93.556$ (1)°
$V = 3494.90$ (16) Å^3
$Z = 2$
$F(000) = 1396$
$D_x = 1.243$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 35384 reflections
$\theta = 2.9$–$26.4°$
$\mu = 0.30$ mm^{-1}
$T = 120$ K
Needle, colourless
0.15 × 0.07 × 0.05 mm

Data collection

Bruker-Nonius KappaCCD area-detector diffractometer
Radiation source: Rotating Anode, Bruker Nonius FR591
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans to fill the asymmetric unit
Absorption correction: multi-scan (SORTAV; Blessing, 1997)
$T_{min} = 0.906$, $T_{max} = 0.990$
13653 measured reflections
7107 independent reflections
4902 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 26.4°$, $\theta_{min} = 3.1°$
$h = -10\rightarrow10$
$k = -26\rightarrow25$
$l = -24\rightarrow24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.144$
$S = 1.11$
7107 reflections
395 parameters
71 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 4.3109P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.70$ e Å^{-3}
$\Delta\rho_{min} = -1.05$ e Å^{-3}
Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0023 (4)

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 $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl1	0.16835 (9)	0.70673 (4)	0.60764 (4)	0.0322 (2)	
Cl2	0.38459 (10)	0.57889 (4)	0.64814 (4)	0.0336 (2)	
Cl3	0.18791 (9)	0.69400 (4)	0.95662 (4)	0.0282 (2)	
N1	0.0570 (3)	0.78167 (12)	0.82639 (13)	0.0269 (6)	
H91	0.1008	0.7571	0.8588	0.032*	
N2	0.2000 (3)	0.66144 (12)	0.80256 (13)	0.0253 (6)	
H92	0.1828	0.6667	0.8465	0.030*	
N3	0.3598 (3)	0.57156 (13)	0.89035 (13)	0.0299 (6)	
H93	0.3070	0.6047	0.9037	0.036*	
O1	−0.0100 (3)	0.78668 (13)	0.71095 (13)	0.0488 (7)	
O2	0.4268 (3)	0.51780 (11)	0.79409 (12)	0.0354 (6)	
C1	−0.1002 (4)	0.87948 (16)	0.80754 (19)	0.0338 (8)	
H1	−0.1308	0.8677	0.7613	0.041*	
C2	−0.1505 (4)	0.93640 (17)	0.8346 (2)	0.0385 (9)	
H2	−0.2162	0.9635	0.8064	0.046*	
C3	−0.1069 (4)	0.95440 (17)	0.90160 (19)	0.0386 (9)	
H3	−0.1404	0.9938	0.9191	0.046*	
C4	−0.0142 (4)	0.91450 (18)	0.94291 (19)	0.0398 (9)	
H4	0.0150	0.9263	0.9893	0.048*	
C5	0.0367 (4)	0.85722 (16)	0.91719 (18)	0.0326 (8)	
H5	0.0999	0.8298	0.9460	0.039*	
C6	−0.0048 (3)	0.84006 (15)	0.84902 (17)	0.0268 (7)	
C7	0.0562 (4)	0.75944 (16)	0.76053 (17)	0.0291 (7)	
C8	0.1449 (3)	0.70049 (15)	0.75015 (16)	0.0259 (7)	
C9	0.1974 (3)	0.67619 (15)	0.68946 (16)	0.0251 (7)	
C10	0.2860 (3)	0.62214 (15)	0.70655 (16)	0.0248 (7)	
C11	0.2856 (3)	0.61305 (15)	0.77710 (16)	0.0249 (7)	
C12	0.3633 (4)	0.56283 (15)	0.82090 (16)	0.0268 (7)	
C13	0.3841 (4)	0.54326 (15)	1.01052 (17)	0.0287 (7)	
H13	0.3042	0.5733	1.0178	0.034*	
C14	0.4311 (4)	0.53353 (14)	0.94363 (16)	0.0254 (7)	
C15	0.5488 (4)	0.48980 (15)	0.93349 (17)	0.0276 (7)	
H15	0.5830	0.4827	0.8884	0.033*	
N4	0.6730 (3)	0.78866 (15)	0.53584 (13)	0.0369 (7)	
C16	0.6876 (4)	0.71763 (18)	0.53514 (19)	0.0428 (10)	

H16A	0.7130	0.7029	0.5831	0.051*	
H16B	0.7763	0.7061	0.5073	0.051*	
C17	0.5423 (4)	0.68249 (18)	0.5063 (2)	0.0448 (10)	
H17A	0.4625	0.6827	0.5411	0.054*	
H17B	0.4981	0.7047	0.4645	0.054*	
C18	0.5802 (5)	0.6145 (2)	0.4878 (2)	0.0587 (12)	
H18A	0.6625	0.6145	0.4541	0.070*	
H18B	0.6223	0.5923	0.5300	0.070*	
C19	0.4393 (5)	0.5784 (2)	0.4572 (2)	0.0608 (12)	
H19A	0.3608	0.5750	0.4918	0.091*	
H19B	0.4716	0.5359	0.4437	0.091*	
H19C	0.3945	0.6010	0.4164	0.091*	
C20A	0.630 (3)	0.8185 (17)	0.4688 (7)	0.0422 (16)	0.33
H20A	0.6117	0.8639	0.4779	0.051*	0.33
H20B	0.5279	0.8003	0.4521	0.051*	0.33
C21A	0.7378 (10)	0.8148 (5)	0.4082 (4)	0.0301 (12)	0.33
H21A	0.8400	0.8342	0.4224	0.036*	0.33
H21B	0.7561	0.7699	0.3966	0.036*	0.33
C22A	0.6656 (11)	0.8494 (5)	0.3439 (3)	0.0382 (13)	0.33
H22A	0.5558	0.8356	0.3349	0.046*	0.33
H22B	0.6654	0.8956	0.3528	0.046*	0.33
C23A	0.7577 (14)	0.8357 (6)	0.2797 (4)	0.0549 (15)	0.33
H23A	0.8626	0.8540	0.2863	0.082*	0.33
H23B	0.7031	0.8546	0.2387	0.082*	0.33
H23C	0.7659	0.7898	0.2733	0.082*	0.33
C20B	0.6322 (15)	0.8116 (7)	0.4628 (3)	0.0422 (16)	0.67
H20C	0.6492	0.8580	0.4613	0.051*	0.67
H20D	0.5196	0.8038	0.4516	0.051*	0.67
C21B	0.7262 (6)	0.7803 (2)	0.4067 (2)	0.0301 (12)	0.67
H21C	0.8386	0.7803	0.4219	0.036*	0.67
H21D	0.6922	0.7357	0.4004	0.036*	0.67
C22B	0.7025 (6)	0.8158 (2)	0.33735 (16)	0.0382 (13)	0.67
H22C	0.7477	0.7906	0.3004	0.046*	0.67
H22D	0.5893	0.8209	0.3253	0.046*	0.67
C23B	0.7807 (7)	0.8815 (2)	0.3413 (3)	0.0549 (15)	0.67
H23D	0.7279	0.9082	0.3741	0.082*	0.67
H23E	0.7729	0.9012	0.2954	0.082*	0.67
H23F	0.8910	0.8768	0.3570	0.082*	0.67
C24	0.8284 (4)	0.81522 (18)	0.56291 (18)	0.0407 (9)	
H24A	0.9060	0.8069	0.5282	0.049*	
H24B	0.8627	0.7919	0.6055	0.049*	
C25	0.8304 (4)	0.8857 (2)	0.5793 (2)	0.0481 (11)	
H25A	0.7668	0.8938	0.6194	0.058*	
H25B	0.7831	0.9094	0.5391	0.058*	
C26	0.9971 (4)	0.9094 (2)	0.5959 (2)	0.0523 (11)	
H26A	1.0439	0.8854	0.6360	0.063*	
H26B	1.0603	0.9008	0.5559	0.063*	
C27	1.0042 (5)	0.9796 (2)	0.6125 (3)	0.0670 (14)	

H27A	0.9553	1.0036	0.5736	0.101*
H27B	1.1136	0.9927	0.6203	0.101*
H27C	0.9484	0.9879	0.6542	0.101*
C28	0.5439 (4)	0.80920 (18)	0.58037 (17)	0.0355 (8)
H28A	0.4446	0.7908	0.5607	0.043*
H28B	0.5342	0.8559	0.5771	0.043*
C29	0.5623 (4)	0.79135 (17)	0.65683 (17)	0.0345 (8)
H29A	0.6707	0.8000	0.6751	0.041*
H29B	0.5414	0.7456	0.6624	0.041*
C30	0.4476 (4)	0.83005 (18)	0.69701 (18)	0.0394 (9)
H30A	0.4691	0.8757	0.6906	0.047*
H30B	0.3399	0.8216	0.6778	0.047*
C31	0.4579 (5)	0.8149 (2)	0.77423 (19)	0.0489 (10)
H31A	0.4398	0.7695	0.7809	0.073*
H31B	0.3786	0.8392	0.7971	0.073*
H31C	0.5620	0.8261	0.7943	0.073*
N5	0.1513 (5)	0.4085 (2)	0.3604 (2)	0.0760 (8)
C32	0.1877 (6)	0.4529 (3)	0.3326 (3)	0.0760 (8)
C33	0.2353 (6)	0.5087 (3)	0.2980 (3)	0.0760 (8)
H33A	0.2260	0.5454	0.3285	0.114*
H33B	0.1684	0.5149	0.2556	0.114*
H33C	0.3442	0.5042	0.2861	0.114*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0372 (4)	0.0405 (5)	0.0186 (4)	0.0071 (3)	0.0002 (3)	0.0055 (3)
Cl2	0.0420 (5)	0.0354 (5)	0.0238 (4)	0.0068 (4)	0.0060 (3)	0.0001 (4)
Cl3	0.0298 (4)	0.0346 (5)	0.0200 (4)	0.0070 (3)	−0.0008 (3)	0.0006 (3)
N1	0.0307 (14)	0.0291 (15)	0.0204 (15)	0.0052 (11)	−0.0017 (11)	0.0040 (11)
N2	0.0288 (14)	0.0290 (15)	0.0177 (14)	0.0060 (11)	−0.0010 (10)	0.0017 (11)
N3	0.0402 (16)	0.0271 (15)	0.0225 (15)	0.0138 (12)	0.0019 (11)	0.0038 (12)
O1	0.0698 (18)	0.0513 (17)	0.0244 (14)	0.0326 (14)	−0.0060 (12)	0.0028 (12)
O2	0.0508 (15)	0.0314 (14)	0.0236 (13)	0.0146 (11)	0.0002 (10)	−0.0012 (10)
C1	0.0354 (19)	0.035 (2)	0.031 (2)	0.0050 (15)	0.0032 (14)	0.0037 (16)
C2	0.040 (2)	0.033 (2)	0.043 (2)	0.0071 (15)	0.0041 (16)	0.0038 (17)
C3	0.040 (2)	0.032 (2)	0.044 (2)	0.0012 (16)	0.0071 (16)	−0.0070 (17)
C4	0.041 (2)	0.044 (2)	0.034 (2)	−0.0001 (17)	−0.0039 (16)	−0.0102 (17)
C5	0.0302 (18)	0.035 (2)	0.032 (2)	0.0011 (14)	−0.0031 (14)	−0.0003 (15)
C6	0.0255 (16)	0.0262 (17)	0.0287 (18)	−0.0005 (13)	0.0029 (13)	0.0001 (14)
C7	0.0286 (17)	0.0354 (19)	0.0233 (18)	0.0080 (14)	0.0003 (13)	0.0060 (15)
C8	0.0265 (16)	0.0316 (18)	0.0190 (17)	0.0040 (13)	−0.0029 (12)	0.0038 (13)
C9	0.0278 (16)	0.0277 (18)	0.0193 (17)	0.0001 (13)	−0.0012 (12)	0.0037 (13)
C10	0.0242 (16)	0.0280 (18)	0.0223 (17)	−0.0017 (13)	0.0008 (12)	−0.0010 (13)
C11	0.0266 (16)	0.0265 (17)	0.0211 (17)	0.0016 (13)	−0.0014 (12)	0.0009 (13)
C12	0.0292 (17)	0.0295 (18)	0.0214 (18)	0.0030 (13)	0.0003 (13)	0.0029 (14)
C13	0.0343 (18)	0.0258 (18)	0.0264 (18)	0.0104 (13)	0.0048 (13)	0.0019 (14)
C14	0.0319 (17)	0.0219 (17)	0.0222 (17)	0.0040 (13)	0.0001 (13)	0.0041 (13)

C15	0.0353 (18)	0.0259 (18)	0.0221 (17)	0.0049 (13)	0.0050 (13)	0.0006 (14)
N4	0.0233 (14)	0.065 (2)	0.0223 (15)	0.0097 (13)	0.0012 (11)	0.0114 (14)
C16	0.037 (2)	0.063 (3)	0.029 (2)	0.0218 (18)	0.0035 (15)	0.0039 (18)
C17	0.033 (2)	0.064 (3)	0.037 (2)	0.0110 (18)	0.0034 (16)	0.0024 (19)
C18	0.053 (3)	0.074 (3)	0.050 (3)	0.022 (2)	0.007 (2)	−0.009 (2)
C19	0.064 (3)	0.071 (3)	0.046 (3)	0.008 (2)	−0.007 (2)	−0.005 (2)
C20A	0.0237 (18)	0.084 (4)	0.019 (2)	0.005 (2)	0.0002 (16)	0.016 (3)
C21A	0.026 (2)	0.039 (4)	0.026 (2)	−0.004 (3)	0.0019 (16)	0.007 (3)
C22A	0.043 (3)	0.047 (4)	0.025 (2)	0.007 (3)	0.004 (2)	0.004 (3)
C23A	0.064 (4)	0.048 (3)	0.055 (4)	0.007 (3)	0.028 (3)	0.015 (3)
C20B	0.0237 (18)	0.084 (4)	0.019 (2)	0.005 (2)	0.0002 (16)	0.016 (3)
C21B	0.026 (2)	0.039 (4)	0.026 (2)	−0.004 (3)	0.0019 (16)	0.007 (3)
C22B	0.043 (3)	0.047 (4)	0.025 (2)	0.007 (3)	0.004 (2)	0.004 (3)
C23B	0.064 (4)	0.048 (3)	0.055 (4)	0.007 (3)	0.028 (3)	0.015 (3)
C24	0.0272 (18)	0.069 (3)	0.0257 (19)	0.0113 (17)	0.0005 (14)	0.0096 (18)
C25	0.0283 (19)	0.081 (3)	0.035 (2)	0.0079 (19)	−0.0012 (15)	0.010 (2)
C26	0.034 (2)	0.078 (3)	0.046 (3)	0.005 (2)	0.0065 (17)	0.005 (2)
C27	0.043 (3)	0.095 (4)	0.063 (3)	−0.002 (2)	0.003 (2)	−0.006 (3)
C28	0.0253 (17)	0.050 (2)	0.032 (2)	0.0070 (15)	0.0060 (14)	0.0088 (17)
C29	0.0330 (18)	0.041 (2)	0.030 (2)	0.0041 (15)	0.0050 (14)	0.0069 (16)
C30	0.041 (2)	0.043 (2)	0.036 (2)	−0.0016 (16)	0.0100 (16)	−0.0028 (17)
C31	0.045 (2)	0.066 (3)	0.037 (2)	−0.011 (2)	0.0103 (17)	−0.008 (2)
N5	0.0679 (17)	0.079 (2)	0.079 (2)	0.0018 (16)	−0.0066 (14)	0.0135 (16)
C32	0.0679 (17)	0.079 (2)	0.079 (2)	0.0018 (16)	−0.0066 (14)	0.0135 (16)
C33	0.0679 (17)	0.079 (2)	0.079 (2)	0.0018 (16)	−0.0066 (14)	0.0135 (16)

Geometric parameters (Å, º)

Cl1—C9	1.713 (3)	C4—H4	0.9500
Cl2—C10	1.716 (3)	C5—H5	0.9500
N1—C7	1.358 (4)	C13—H13	0.9500
N1—C6	1.421 (4)	C15—H15	0.9500
N2—C11	1.367 (4)	C16—H16A	0.9900
N2—C8	1.368 (4)	C16—H16B	0.9900
N3—C12	1.359 (4)	C17—H17A	0.9900
N3—C14	1.416 (4)	C17—H17B	0.9900
O1—C7	1.227 (4)	C18—H18A	0.9900
O2—C12	1.226 (4)	C18—H18B	0.9900
C1—C6	1.387 (4)	C19—H19A	0.9800
C1—C2	1.390 (5)	C19—H19B	0.9800
C2—C3	1.381 (5)	C19—H19C	0.9800
C3—C4	1.379 (5)	C20A—H20A	0.9900
C4—C5	1.388 (5)	C20A—H20B	0.9900
C5—C6	1.393 (5)	C21A—H21A	0.9900
C7—C8	1.478 (4)	C21A—H21B	0.9900
C8—C9	1.382 (4)	C22A—H22A	0.9900
C9—C10	1.399 (4)	C22A—H22B	0.9900
C10—C11	1.379 (4)	C23A—H23A	0.9800

C11—C12	1.489 (4)	C23A—H23B	0.9800
C13—C15^{i}	1.384 (4)	C23A—H23C	0.9800
C13—C14	1.395 (4)	C20B—H20C	0.9900
C14—C15	1.390 (4)	C20B—H20D	0.9900
C15—C13^{i}	1.384 (4)	C21B—H21C	0.9900
N4—C20A	1.469 (8)	C21B—H21D	0.9900
N4—C16	1.505 (4)	C22B—H22C	0.9900
N4—C24	1.508 (4)	C22B—H22D	0.9900
N4—C28	1.508 (4)	C23B—H23D	0.9800
N4—C20B	1.514 (5)	C23B—H23E	0.9800
C16—C17	1.525 (5)	C23B—H23F	0.9800
C17—C18	1.519 (5)	C24—H24A	0.9900
C18—C19	1.517 (5)	C24—H24B	0.9900
C20A—C21A	1.5399 (11)	C25—H25A	0.9900
C21A—C22A	1.5398 (11)	C25—H25B	0.9900
C22A—C23A	1.5396 (11)	C26—H26A	0.9900
C20B—C21B	1.5405 (11)	C26—H26B	0.9900
C21B—C22B	1.5403 (10)	C27—H27A	0.9800
C22B—C23B	1.5393 (11)	C27—H27B	0.9800
C24—C25	1.522 (5)	C27—H27C	0.9800
C25—C26	1.529 (5)	C28—H28A	0.9900
C26—C27	1.518 (5)	C28—H28B	0.9900
C28—C29	1.525 (4)	C29—H29A	0.9900
C29—C30	1.528 (5)	C29—H29B	0.9900
C30—C31	1.526 (5)	C30—H30A	0.9900
N5—C32	1.135 (6)	C30—H30B	0.9900
C32—C33	1.427 (7)	C31—H31A	0.9800
N1—H91	0.8800	C31—H31B	0.9800
N2—H92	0.8800	C31—H31C	0.9800
N3—H93	0.8800	C33—H33A	0.9800
C1—H1	0.9500	C33—H33B	0.9800
C2—H2	0.9500	C33—H33C	0.9800
C3—H3	0.9500		

C7—N1—C6	127.5 (3)	C17—C18—H18B	109.0
C11—N2—C8	110.6 (3)	H18A—C18—H18B	107.8
C12—N3—C14	127.5 (3)	C18—C19—H19A	109.5
C6—C1—C2	119.1 (3)	C18—C19—H19B	109.5
C3—C2—C1	121.3 (3)	H19A—C19—H19B	109.5
C2—C3—C4	119.3 (3)	C18—C19—H19C	109.5
C3—C4—C5	120.5 (3)	H19A—C19—H19C	109.5
C4—C5—C6	119.9 (3)	H19B—C19—H19C	109.5
C1—C6—C5	119.9 (3)	N4—C20A—H20A	107.0
C1—C6—N1	124.0 (3)	C21A—C20A—H20A	107.0
C5—C6—N1	116.0 (3)	N4—C20A—H20B	107.0
O1—C7—N1	123.2 (3)	C21A—C20A—H20B	107.0
O1—C7—C8	120.3 (3)	H20A—C20A—H20B	106.7
N1—C7—C8	116.5 (3)	C22A—C21A—H21A	109.4

N2—C8—C9	106.9 (3)	C20A—C21A—H21A	109.4
N2—C8—C7	124.3 (3)	C22A—C21A—H21B	109.4
C9—C8—C7	128.7 (3)	C20A—C21A—H21B	109.4
C8—C9—C10	107.6 (3)	H21A—C21A—H21B	108.0
C8—C9—Cl1	127.5 (2)	C23A—C22A—H22A	109.4
C10—C9—Cl1	124.8 (2)	C21A—C22A—H22A	109.4
C11—C10—C9	108.2 (3)	C23A—C22A—H22B	109.4
C11—C10—Cl2	127.6 (2)	C21A—C22A—H22B	109.4
C9—C10—Cl2	124.1 (2)	H22A—C22A—H22B	108.0
N2—C11—C10	106.7 (3)	N4—C20B—H20C	108.6
N2—C11—C12	123.9 (3)	C21B—C20B—H20C	108.6
C10—C11—C12	129.4 (3)	N4—C20B—H20D	108.6
O2—C12—N3	124.2 (3)	C21B—C20B—H20D	108.6
O2—C12—C11	120.4 (3)	H20C—C20B—H20D	107.5
N3—C12—C11	115.4 (3)	C22B—C21B—H21C	109.4
C15^{i}—C13—C14	121.5 (3)	C20B—C21B—H21C	109.4
C15—C14—C13	118.9 (3)	C22B—C21B—H21D	109.4
C15—C14—N3	123.9 (3)	C20B—C21B—H21D	109.4
C13—C14—N3	117.1 (3)	H21C—C21B—H21D	108.0
C13^{i}—C15—C14	119.6 (3)	C23B—C22B—H22C	109.4
C20A—N4—C16	115.8 (15)	C21B—C22B—H22C	109.4
C20A—N4—C24	108.5 (15)	C23B—C22B—H22D	109.4
C16—N4—C24	107.6 (3)	C21B—C22B—H22D	109.4
C20A—N4—C28	103.2 (5)	H22C—C22B—H22D	108.0
C16—N4—C28	110.8 (3)	C22B—C23B—H23D	109.5
C24—N4—C28	111.0 (3)	C22B—C23B—H23E	109.5
C16—N4—C20B	109.0 (6)	H23D—C23B—H23E	109.5
C24—N4—C20B	110.7 (7)	C22B—C23B—H23F	109.5
C28—N4—C20B	107.8 (3)	H23D—C23B—H23F	109.5
N4—C16—C17	115.0 (3)	H23E—C23B—H23F	109.5
C18—C17—C16	111.4 (3)	N4—C24—H24A	108.3
C19—C18—C17	112.9 (3)	C25—C24—H24A	108.3
N4—C20A—C21A	121.4 (7)	N4—C24—H24B	108.3
C22A—C21A—C20A	111.18 (11)	C25—C24—H24B	108.3
C23A—C22A—C21A	111.22 (11)	H24A—C24—H24B	107.4
N4—C20B—C21B	114.8 (4)	C24—C25—H25A	109.4
C22B—C21B—C20B	111.06 (10)	C26—C25—H25A	109.4
C23B—C22B—C21B	111.19 (10)	C24—C25—H25B	109.4
N4—C24—C25	115.8 (3)	C26—C25—H25B	109.4
C24—C25—C26	111.2 (3)	H25A—C25—H25B	108.0
C27—C26—C25	112.9 (3)	C27—C26—H26A	109.0
N4—C28—C29	116.6 (3)	C25—C26—H26A	109.0
C28—C29—C30	109.3 (3)	C27—C26—H26B	109.0
C31—C30—C29	112.7 (3)	C25—C26—H26B	109.0
N5—C32—C33	179.3 (6)	H26A—C26—H26B	107.8
C7—N1—H91	116.3	C26—C27—H27A	109.5
C6—N1—H91	116.3	C26—C27—H27B	109.5
C11—N2—H92	124.7	H27A—C27—H27B	109.5

C8—N2—H92	124.7	C26—C27—H27C	109.5
C12—N3—H93	116.3	H27A—C27—H27C	109.5
C14—N3—H93	116.3	H27B—C27—H27C	109.5
C6—C1—H1	120.4	N4—C28—H28A	108.1
C2—C1—H1	120.4	C29—C28—H28A	108.1
C3—C2—H2	119.4	N4—C28—H28B	108.1
C1—C2—H2	119.4	C29—C28—H28B	108.1
C4—C3—H3	120.4	H28A—C28—H28B	107.3
C2—C3—H3	120.4	C28—C29—H29A	109.8
C3—C4—H4	119.8	C30—C29—H29A	109.8
C5—C4—H4	119.8	C28—C29—H29B	109.8
C4—C5—H5	120.0	C30—C29—H29B	109.8
C6—C5—H5	120.0	H29A—C29—H29B	108.3
C15^{i}—C13—H13	119.2	C31—C30—H30A	109.0
C14—C13—H13	119.2	C29—C30—H30A	109.0
C13^{i}—C15—H15	120.2	C31—C30—H30B	109.0
C14—C15—H15	120.2	C29—C30—H30B	109.0
N4—C16—H16A	108.5	H30A—C30—H30B	107.8
C17—C16—H16A	108.5	C30—C31—H31A	109.5
N4—C16—H16B	108.5	C30—C31—H31B	109.5
C17—C16—H16B	108.5	H31A—C31—H31B	109.5
H16A—C16—H16B	107.5	C30—C31—H31C	109.5
C18—C17—H17A	109.4	H31A—C31—H31C	109.5
C16—C17—H17A	109.4	H31B—C31—H31C	109.5
C18—C17—H17B	109.4	C32—C33—H33A	109.5
C16—C17—H17B	109.4	C32—C33—H33B	109.5
H17A—C17—H17B	108.0	H33A—C33—H33B	109.5
C19—C18—H18A	109.0	C32—C33—H33C	109.5
C17—C18—H18A	109.0	H33A—C33—H33C	109.5
C19—C18—H18B	109.0	H33B—C33—H33C	109.5

Symmetry code: (i) $-x+1, -y+1, -z+2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H91···Cl3	0.88	2.40	3.269 (3)	172
N2—H92···Cl3	0.88	2.20	3.068 (3)	167
N3—H93···Cl3	0.88	2.40	3.275 (3)	171