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4-Aminomethyl-phenylamino-bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide) tetrabutylammonium salt

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Key indicators

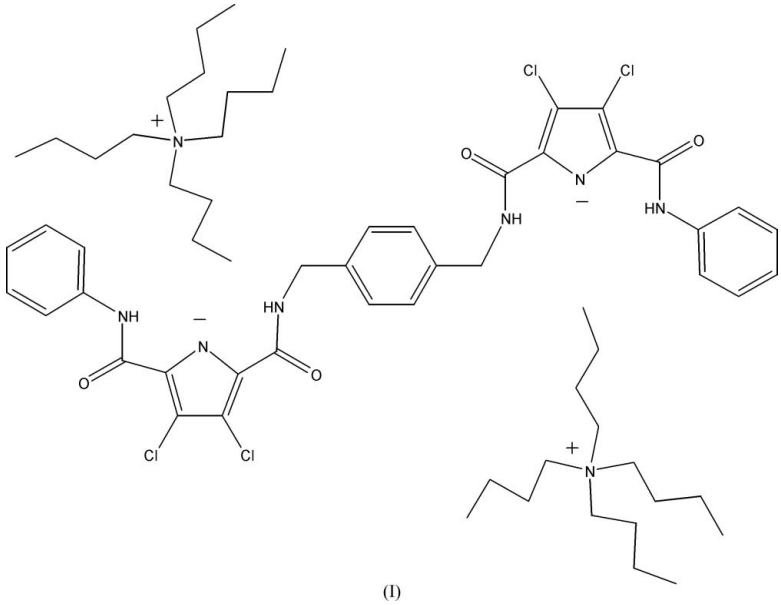
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.008 Å
R factor = 0.101
wR factor = 0.192
Data-to-parameter ratio = 15.9

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

The crystal structure of the tetrabutylammonium salt of doubly deprotonated 4-aminomethyl(phenylamino)bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide), $2C_{16}H_{36}N^{+}\cdot C_{32}H_{22}C_{l4}N_6O_4^{2-}$, has been elucidated. The anion lies on an inversion centre and adopts a twisted S shape.

Comment

4-Aminomethyl(phenylamino)bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide) crystallizes as the tetrabutylammonium salt, (I), from an acetonitrile solution of the compound in the presence of excess tetrabutylammonium fluoride. The anion adopts a twisted S shape around a centre of inversion. The pyrrole and terminal benzene ring pairs are coplanar and the angle between the central and terminal benzene ring is 73.01 (5)°.



(I)

Experimental

p-Xylenediamine (68 mg, 0.5 mmol, 1 equiv.) was added to a solution of 3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxylic acid (300 mg, 1 mmol, 2 equiv.) in DMF (30 ml) under a nitrogen atmosphere. Triethyamine (104 mg, 1 mmol, 2 equiv.), benzotriazol-1-yloxy)tripyrrolidinophosphonium hexafluorophosphate (572 mg, 1.1 mmol, 2.2 equiv.) and 5 mg (0.04 mmol, 0.04 equiv.) of *N*-hydroxybenzotriazole were added and the reaction was stirred for 72 h. The solvent was then removed and water (50 ml) was added. The product was extracted with dichloromethane (DCM, 3 50 ml). The organic phase was collected and the solvent was removed. The product was washed with diethyl ether (75 ml) and a small quantity of 10% MeOH

in DCM (v/v). The product was obtained as a white solid (132 mg, 0.19 mmol, 38%).

M.p. 590 K (decomp.). ^{1}H NMR 300 MHz in DMSO-d_6 δ (p.p.m.): 4.50 (*d*, J = 5.4, 4H, CH_2), 7.00–7.70 (*m*, 14H, ArH), 8.50 (*t*, 2H, J = 5.4, central–CONH), 10.04 (*s*, 2H, outer–CONH), 12.79 (*s*, 2H, NH-pyrrole). ^{13}C NMR 75 MHz in DMSO-d_6 δ (p.p.m.): 42.3, 112.0, 113.6, 119.8, 122.9, 123.1, 123.9, 127.4, 128.7, 137.6, 138.3, 156.5, 158.1. TOF LD$^+$ mass spectrum: m/z (%): 472 (100) $[C_{23}H_{22}Cl_2N_4O_3]^+$. Elemental analysis: Calc. for $C_{32}H_{24}Cl_4N_6O_4.H_2O$: C 53.65, H 3.66, N 11.73%; found: C 53.28, H 3.73, N 12.03%.

Crystals of the title compound were obtained by slow evaporation of an acetonitrile solution in the presence of excess tetrabutylammonium fluoride.

Crystal data

$2C_{16}H_{36}N^+\cdot C_{32}H_{22}Cl_4N_6O_4^{2-}$	Z = 1
M_r = 1181.27	D_x = 1.208 Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
a = 9.524 (2) Å	Cell parameters from 58835 reflections
b = 10.363 (3) Å	θ = 2.9–27.5°
c = 17.056 (4) Å	μ = 0.23 mm^{-1}
α = 78.293 (11)°	T = 120 (2) K
β = 88.733 (14)°	Block, colourless
γ = 80.136 (14)°	0.20 × 0.10 × 0.07 mm
V = 1623.9 (7) Å^3	

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer	5757 independent reflections
φ and ω scans	2777 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)	R_{int} = 0.111
T_{min} = 0.732, T_{max} = 0.984	θ_{max} = 26.1°
16494 measured reflections	$h = -11 \rightarrow 11$
	$k = -12 \rightarrow 12$
	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 2.4189P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.101	$(\Delta/\sigma)_{max}$ = 0.019
$wR(F^2)$ = 0.192	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
S = 1.04	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
5757 reflections	
361 parameters	
H-atom parameters constrained	

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}(C,N)].

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).

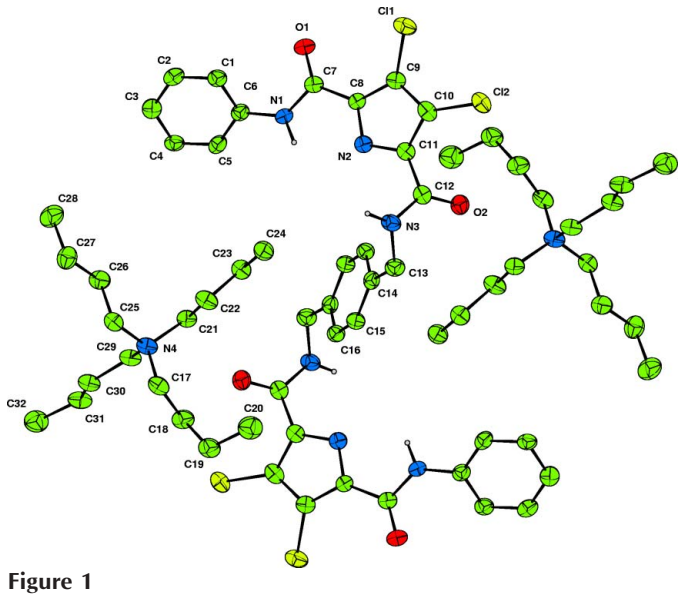


Figure 1
Structure of the title compound, with displacement ellipsoids drawn at the 50% probability level and non-acidic H atoms omitted for clarity.

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$\alpha = 78.293$ (11)°
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Cell parameters from 58835 reflections
$\theta = 2.9$–27.5°
$\mu = 0.23$ mm^{-1}
$T = 120$ K
Block, colourless
0.20 × 0.10 × 0.07 mm

Data collection

Bruker Nonius Kappa CCD 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)
$T_{min} = 0.732$, $T_{max} = 0.984$
16494 measured reflections
5757 independent reflections
2777 reflections with $I > 2\sigma(I)$
$R_{int} = 0.111$
$\theta_{max} = 26.1°$, $\theta_{min} = 3.1°$
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.101$
$wR(F^2) = 0.192$
$S = 1.04$
5757 reflections
361 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 2.4189P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Special details

Experimental. The higher angle reflections were very diffuse and weak and data above 26.09 θ theta were omitted from the refinement. As a result the _diffrn_reflns_theta_full is low (26.09) and the data are only 89% complete to this value

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}
Cl1	1.03523 (16)	0.79277 (16)	0.44484 (8)	0.0620 (5)
Cl2	0.75939 (16)	1.05446 (16)	0.41527 (8)	0.0592 (5)
O1	1.1698 (4)	0.6453 (4)	0.3030 (2)	0.0566 (11)
O2	0.5971 (4)	1.1745 (4)	0.2402 (2)	0.0538 (11)
N1	1.0803 (5)	0.7269 (5)	0.1767 (3)	0.0487 (13)
H91	1.0146	0.7881	0.1488	0.058*
N2	0.8929 (5)	0.9176 (5)	0.2225 (3)	0.0428 (12)
N3	0.7274 (5)	1.1256 (5)	0.1331 (3)	0.0509 (13)
H93	0.8143	1.0972	0.1182	0.061*
C1	1.3027 (7)	0.5813 (6)	0.1559 (3)	0.0487 (16)
H1	1.3438	0.5951	0.2031	0.058*
C2	1.3809 (6)	0.5006 (6)	0.1103 (4)	0.0499 (16)
H2	1.4759	0.4599	0.1260	0.060*
C3	1.3233 (7)	0.4776 (6)	0.0415 (4)	0.0517 (16)
H3	1.3772	0.4204	0.0107	0.062*
C4	1.1854 (7)	0.5396 (6)	0.0183 (3)	0.0500 (16)
H4	1.1449	0.5266	−0.0293	0.060*
C5	1.1074 (6)	0.6196 (6)	0.0642 (4)	0.0510 (16)
H5	1.0124	0.6601	0.0483	0.061*
C6	1.1644 (6)	0.6427 (6)	0.1337 (4)	0.0464 (15)
C7	1.0864 (6)	0.7269 (6)	0.2570 (4)	0.0448 (15)
C8	0.9809 (6)	0.8328 (6)	0.2795 (3)	0.0392 (14)
C9	0.9494 (6)	0.8649 (6)	0.3541 (3)	0.0464 (15)
C10	0.8383 (6)	0.9723 (6)	0.3426 (3)	0.0472 (16)
C11	0.8045 (6)	1.0043 (6)	0.2588 (3)	0.0433 (15)
C12	0.6989 (7)	1.1095 (6)	0.2119 (4)	0.0445 (15)
C13	0.6200 (6)	1.1883 (6)	0.0721 (3)	0.0532 (16)
H13A	0.6622	1.2501	0.0298	0.064*
H13B	0.5414	1.2421	0.0964	0.064*
C14	0.5600 (6)	1.0907 (6)	0.0344 (3)	0.0447 (15)
C15	0.5075 (6)	1.1251 (6)	−0.0432 (3)	0.0480 (15)
H15	0.5137	1.2114	−0.0738	0.058*
C16	0.5528 (6)	0.9608 (6)	0.0775 (3)	0.0457 (15)

H16	0.5903	0.9328	0.1305	0.055*
N4	0.1867 (5)	0.2167 (4)	0.3108 (3)	0.0469 (12)
C17	0.2262 (6)	0.0766 (6)	0.3622 (3)	0.0512 (16)
H17A	0.1752	0.0738	0.4135	0.061*
H17B	0.1920	0.0123	0.3346	0.061*
C18	0.3849 (6)	0.0302 (6)	0.3803 (4)	0.0601 (18)
H18A	0.4115	0.0657	0.4266	0.072*
H18B	0.4401	0.0665	0.3336	0.072*
C19	0.4236 (7)	−0.1235 (6)	0.3994 (4)	0.0638 (18)
H19A	0.5150	−0.1498	0.4297	0.077*
H19B	0.3494	−0.1605	0.4341	0.077*
C20	0.4372 (7)	−0.1837 (7)	0.3263 (4)	0.083 (2)
H20A	0.3461	−0.1609	0.2970	0.125*
H20B	0.4630	−0.2811	0.3422	0.125*
H20C	0.5113	−0.1484	0.2919	0.125*
C21	0.2295 (6)	0.2154 (5)	0.2238 (3)	0.0476 (15)
H21A	0.2134	0.3087	0.1932	0.057*
H21B	0.3329	0.1808	0.2229	0.057*
C22	0.1520 (6)	0.1335 (6)	0.1811 (3)	0.0567 (17)
H22A	0.1787	0.0376	0.2060	0.068*
H22B	0.0479	0.1595	0.1869	0.068*
C23	0.1877 (6)	0.1538 (6)	0.0943 (3)	0.0530 (16)
H23A	0.2923	0.1317	0.0891	0.064*
H23B	0.1581	0.2495	0.0696	0.064*
C24	0.1181 (6)	0.0709 (6)	0.0483 (3)	0.0577 (17)
H24A	0.1469	−0.0242	0.0720	0.087*
H24B	0.1481	0.0880	−0.0077	0.087*
H24C	0.0143	0.0952	0.0507	0.087*
C25	0.0263 (5)	0.2587 (6)	0.3169 (3)	0.0482 (16)
H25A	−0.0213	0.1900	0.3010	0.058*
H25B	0.0030	0.2599	0.3737	0.058*
C26	−0.0351 (6)	0.3940 (6)	0.2662 (3)	0.0577 (17)
H26A	0.0094	0.4640	0.2828	0.069*
H26B	−0.0122	0.3943	0.2093	0.069*
C27	−0.1949 (6)	0.4260 (7)	0.2750 (4)	0.0664 (19)
H27A	−0.2386	0.3553	0.2585	0.080*
H27B	−0.2170	0.4243	0.3321	0.080*
C28	−0.2616 (7)	0.5599 (7)	0.2263 (4)	0.082 (2)
H28A	−0.2253	0.6312	0.2454	0.123*
H28B	−0.3653	0.5718	0.2320	0.123*
H28C	−0.2374	0.5638	0.1698	0.123*
C29	0.2666 (6)	0.3149 (5)	0.3386 (3)	0.0478 (16)
H29A	0.3702	0.2818	0.3360	0.057*
H29B	0.2452	0.4019	0.3006	0.057*
C30	0.2328 (6)	0.3380 (6)	0.4224 (3)	0.0522 (16)
H30A	0.2365	0.2508	0.4598	0.063*
H30B	0.1350	0.3889	0.4230	0.063*
C31	0.3377 (7)	0.4151 (6)	0.4504 (3)	0.0605 (18)

H31A	0.3415	0.4980	0.4101	0.073*
H31B	0.4341	0.3600	0.4554	0.073*
C32	0.2938 (7)	0.4511 (7)	0.5310 (4)	0.079 (2)
H32A	0.2018	0.5115	0.5250	0.118*
H32B	0.3661	0.4953	0.5496	0.118*
H32C	0.2854	0.3694	0.5702	0.118*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0602 (10)	0.0784 (12)	0.0446 (9)	−0.0219 (9)	−0.0037 (8)	0.0032 (8)
Cl2	0.0646 (11)	0.0723 (12)	0.0455 (9)	−0.0220 (9)	0.0084 (8)	−0.0148 (8)
O1	0.056 (3)	0.051 (3)	0.055 (3)	−0.001 (2)	−0.010 (2)	0.002 (2)
O2	0.047 (3)	0.059 (3)	0.055 (3)	−0.010 (2)	0.010 (2)	−0.010 (2)
N1	0.047 (3)	0.047 (3)	0.044 (3)	0.006 (3)	−0.005 (2)	−0.003 (3)
N2	0.041 (3)	0.042 (3)	0.044 (3)	−0.012 (3)	0.004 (2)	−0.002 (3)
N3	0.045 (3)	0.063 (4)	0.040 (3)	−0.013 (3)	0.003 (2)	0.002 (3)
C1	0.051 (4)	0.045 (4)	0.046 (4)	−0.011 (4)	0.003 (3)	0.002 (3)
C2	0.042 (4)	0.039 (4)	0.062 (4)	−0.011 (3)	0.005 (3)	0.008 (3)
C3	0.062 (5)	0.035 (4)	0.057 (4)	−0.016 (4)	0.011 (3)	−0.002 (3)
C4	0.051 (4)	0.049 (4)	0.044 (4)	−0.008 (4)	−0.003 (3)	0.005 (3)
C5	0.041 (4)	0.047 (4)	0.059 (4)	0.000 (3)	0.007 (3)	−0.003 (3)
C6	0.046 (4)	0.040 (4)	0.049 (4)	−0.005 (3)	0.003 (3)	0.000 (3)
C7	0.041 (4)	0.045 (4)	0.047 (4)	−0.012 (3)	−0.001 (3)	−0.003 (3)
C8	0.036 (3)	0.037 (4)	0.045 (4)	−0.010 (3)	0.001 (3)	−0.005 (3)
C9	0.049 (4)	0.043 (4)	0.048 (4)	−0.016 (3)	−0.001 (3)	−0.004 (3)
C10	0.060 (4)	0.045 (4)	0.046 (4)	−0.030 (4)	0.006 (3)	−0.012 (3)
C11	0.043 (4)	0.044 (4)	0.046 (4)	−0.018 (3)	0.003 (3)	−0.008 (3)
C12	0.046 (4)	0.044 (4)	0.047 (4)	−0.019 (4)	0.005 (3)	−0.009 (3)
C13	0.057 (4)	0.048 (4)	0.050 (4)	−0.008 (3)	0.001 (3)	0.000 (3)
C14	0.043 (4)	0.043 (4)	0.047 (4)	−0.006 (3)	0.010 (3)	−0.007 (3)
C15	0.059 (4)	0.038 (4)	0.041 (4)	−0.010 (3)	0.007 (3)	0.005 (3)
C16	0.043 (4)	0.048 (4)	0.042 (3)	−0.005 (3)	0.004 (3)	−0.001 (3)
N4	0.049 (3)	0.047 (3)	0.044 (3)	−0.023 (3)	−0.001 (2)	0.004 (2)
C17	0.061 (4)	0.052 (4)	0.043 (3)	−0.028 (3)	0.004 (3)	0.001 (3)
C18	0.056 (4)	0.059 (5)	0.060 (4)	−0.015 (4)	0.002 (3)	0.005 (3)
C19	0.066 (4)	0.065 (5)	0.056 (4)	−0.017 (4)	0.007 (3)	0.004 (4)
C20	0.092 (6)	0.075 (5)	0.083 (5)	−0.016 (5)	−0.003 (4)	−0.012 (4)
C21	0.049 (4)	0.046 (4)	0.047 (4)	−0.015 (3)	−0.001 (3)	−0.001 (3)
C22	0.061 (4)	0.066 (4)	0.046 (4)	−0.030 (4)	0.001 (3)	−0.002 (3)
C23	0.048 (4)	0.059 (4)	0.054 (4)	−0.017 (3)	0.003 (3)	−0.011 (3)
C24	0.062 (4)	0.058 (4)	0.050 (4)	−0.013 (4)	0.008 (3)	−0.002 (3)
C25	0.042 (4)	0.063 (4)	0.045 (4)	−0.025 (3)	0.004 (3)	−0.010 (3)
C26	0.046 (4)	0.070 (5)	0.055 (4)	−0.016 (4)	0.000 (3)	−0.002 (3)
C27	0.053 (4)	0.070 (5)	0.080 (5)	−0.012 (4)	0.004 (4)	−0.023 (4)
C28	0.049 (4)	0.098 (6)	0.096 (6)	−0.001 (4)	−0.011 (4)	−0.022 (5)
C29	0.047 (4)	0.051 (4)	0.046 (4)	−0.023 (3)	−0.007 (3)	0.003 (3)
C30	0.058 (4)	0.053 (4)	0.046 (4)	−0.023 (3)	−0.007 (3)	0.001 (3)

C31	0.071 (4)	0.055 (4)	0.055 (4)	−0.022 (4)	−0.016 (3)	0.003 (3)
C32	0.082 (5)	0.079 (5)	0.078 (5)	−0.017 (4)	−0.015 (4)	−0.019 (4)

Geometric parameters (Å, º)

Cl1—C9	1.732 (6)	C2—H2	0.9500
Cl2—C10	1.730 (6)	C3—H3	0.9500
O1—C7	1.220 (6)	C4—H4	0.9500
O2—C12	1.231 (6)	C5—H5	0.9500
N1—C7	1.373 (7)	C13—H13A	0.9901
N1—C6	1.391 (7)	C13—H13B	0.9900
N2—C11	1.358 (7)	C15—H15	0.9499
N2—C8	1.362 (6)	C16—H16	0.9499
N3—C12	1.348 (7)	C17—H17A	0.9899
N3—C13	1.447 (6)	C17—H17B	0.9900
C1—C2	1.373 (8)	C18—H18A	0.9901
C1—C6	1.386 (7)	C18—H18B	0.9900
C2—C3	1.385 (8)	C19—H19A	0.9900
C3—C4	1.387 (7)	C19—H19B	0.9900
C4—C5	1.371 (7)	C20—H20A	0.9800
C5—C6	1.394 (8)	C20—H20B	0.9799
C7—C8	1.462 (8)	C20—H20C	0.9800
C8—C9	1.393 (7)	C21—H21A	0.9899
C9—C10	1.384 (7)	C21—H21B	0.9900
C10—C11	1.431 (7)	C22—H22A	0.9900
C11—C12	1.465 (8)	C22—H22B	0.9900
C13—C14	1.498 (7)	C23—H23A	0.9900
C14—C15	1.381 (7)	C23—H23B	0.9899
C14—C16	1.408 (7)	C24—H24A	0.9800
C15—C16^{i}	1.366 (7)	C24—H24B	0.9799
C16—C15^{i}	1.366 (7)	C24—H24C	0.9801
N4—C25	1.522 (7)	C25—H25A	0.9900
N4—C29	1.523 (6)	C25—H25B	0.9900
N4—C17	1.526 (6)	C26—H26A	0.9900
N4—C21	1.533 (6)	C26—H26B	0.9901
C17—C18	1.524 (7)	C27—H27A	0.9900
C18—C19	1.540 (8)	C27—H27B	0.9900
C19—C20	1.497 (8)	C28—H28A	0.9800
C21—C22	1.512 (7)	C28—H28B	0.9800
C22—C23	1.492 (7)	C28—H28C	0.9800
C23—C24	1.511 (7)	C29—H29A	0.9900
C25—C26	1.519 (7)	C29—H29B	0.9900
C26—C27	1.512 (7)	C30—H30A	0.9900
C27—C28	1.508 (8)	C30—H30B	0.9900
C29—C30	1.516 (7)	C31—H31A	0.9899
C30—C31	1.521 (7)	C31—H31B	0.9899
C31—C32	1.528 (8)	C32—H32A	0.9800
N1—H91	0.8800	C32—H32B	0.9800

N3—H93	0.8801	C32—H32C	0.9799
C1—H1	0.9500		

C7—N1—C6	128.4 (5)	C17—C18—H18A	109.3
C11—N2—C8	108.2 (5)	C19—C18—H18A	109.3
C12—N3—C13	122.2 (5)	C17—C18—H18B	109.3
C2—C1—C6	120.6 (6)	C19—C18—H18B	109.3
C1—C2—C3	121.0 (6)	H18A—C18—H18B	108.0
C2—C3—C4	119.0 (6)	C20—C19—H19A	108.9
C5—C4—C3	119.9 (6)	C18—C19—H19A	108.9
C4—C5—C6	121.5 (6)	C20—C19—H19B	108.9
C1—C6—N1	123.4 (6)	C18—C19—H19B	108.9
C1—C6—C5	118.1 (6)	H19A—C19—H19B	107.7
N1—C6—C5	118.5 (5)	C19—C20—H20A	109.5
O1—C7—N1	122.6 (6)	C19—C20—H20B	109.5
O1—C7—C8	125.4 (6)	H20A—C20—H20B	109.5
N1—C7—C8	112.0 (5)	C19—C20—H20C	109.5
N2—C8—C9	109.8 (5)	H20A—C20—H20C	109.5
N2—C8—C7	119.9 (5)	H20B—C20—H20C	109.5
C9—C8—C7	130.3 (6)	C22—C21—H21A	108.3
C10—C9—C8	107.1 (5)	N4—C21—H21A	108.4
C10—C9—Cl1	124.9 (5)	C22—C21—H21B	108.3
C8—C9—Cl1	128.0 (5)	N4—C21—H21B	108.3
C9—C10—C11	106.6 (5)	H21A—C21—H21B	107.4
C9—C10—Cl2	126.4 (5)	C23—C22—H22A	109.4
C11—C10—Cl2	126.9 (5)	C21—C22—H22A	109.4
N2—C11—C10	108.3 (5)	C23—C22—H22B	109.4
N2—C11—C12	120.5 (5)	C21—C22—H22B	109.4
C10—C11—C12	131.1 (6)	H22A—C22—H22B	108.0
O2—C12—N3	124.2 (6)	C22—C23—H23A	108.7
O2—C12—C11	124.8 (5)	C24—C23—H23A	108.7
N3—C12—C11	111.0 (5)	C22—C23—H23B	108.7
N3—C13—C14	113.6 (5)	C24—C23—H23B	108.7
C15—C14—C16	117.5 (5)	H23A—C23—H23B	107.6
C15—C14—C13	121.4 (5)	C23—C24—H24A	109.5
C16—C14—C13	121.0 (5)	C23—C24—H24B	109.5
C16^{i}—C15—C14	122.2 (5)	H24A—C24—H24B	109.5
C15^{i}—C16—C14	120.3 (5)	C23—C24—H24C	109.5
C25—N4—C29	111.0 (4)	H24A—C24—H24C	109.5
C25—N4—C17	107.3 (4)	H24B—C24—H24C	109.5
C29—N4—C17	111.2 (4)	C26—C25—H25A	108.4
C25—N4—C21	110.8 (4)	N4—C25—H25A	108.4
C29—N4—C21	106.6 (4)	C26—C25—H25B	108.6
C17—N4—C21	110.0 (4)	N4—C25—H25B	108.5
C18—C17—N4	115.1 (4)	H25A—C25—H25B	107.5
C17—C18—C19	111.6 (5)	C27—C26—H26A	109.3
C20—C19—C18	113.3 (5)	C25—C26—H26A	109.2
C22—C21—N4	115.8 (4)	C27—C26—H26B	109.4

C23—C22—C21	111.2 (4)	C25—C26—H26B	109.4
C22—C23—C24	114.1 (4)	H26A—C26—H26B	108.0
C26—C25—N4	115.2 (4)	C28—C27—H27A	108.9
C27—C26—C25	111.4 (5)	C26—C27—H27A	108.8
C28—C27—C26	113.7 (5)	C28—C27—H27B	108.8
C30—C29—N4	115.4 (4)	C26—C27—H27B	108.9
C29—C30—C31	111.1 (4)	H27A—C27—H27B	107.7
C30—C31—C32	110.9 (5)	C27—C28—H28A	109.5
C7—N1—H91	115.7	C27—C28—H28B	109.4
C6—N1—H91	115.8	H28A—C28—H28B	109.5
C12—N3—H93	118.9	C27—C28—H28C	109.4
C13—N3—H93	118.8	H28A—C28—H28C	109.5
C2—C1—H1	119.7	H28B—C28—H28C	109.5
C6—C1—H1	119.7	C30—C29—H29A	108.4
C1—C2—H2	119.5	N4—C29—H29A	108.4
C3—C2—H2	119.5	C30—C29—H29B	108.5
C2—C3—H3	120.6	N4—C29—H29B	108.4
C5—C4—H4	120.0	H29A—C29—H29B	107.5
C3—C4—H4	120.1	C29—C30—H30A	109.5
C4—C5—H5	119.2	C31—C30—H30A	109.5
C6—C5—H5	119.3	C29—C30—H30B	109.4
N3—C13—H13A	108.9	C31—C30—H30B	109.3
C14—C13—H13A	108.8	H30A—C30—H30B	108.0
N3—C13—H13B	108.8	C30—C31—H31A	109.6
C14—C13—H13B	108.9	C32—C31—H31A	109.4
H13A—C13—H13B	107.7	C30—C31—H31B	109.4
C16^{i}—C15—H15	118.9	C32—C31—H31B	109.5
C14—C15—H15	118.9	H31A—C31—H31B	108.1
C15^{i}—C16—H16	119.9	C31—C32—H32A	109.5
C14—C16—H16	119.9	C31—C32—H32B	109.5
C18—C17—H17A	108.5	H32A—C32—H32B	109.5
N4—C17—H17A	108.5	C31—C32—H32C	109.4
C18—C17—H17B	108.4	H32A—C32—H32C	109.5
N4—C17—H17B	108.5	H32B—C32—H32C	109.5
H17A—C17—H17B	107.5		

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