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Synthesis and crystal structure of 4-benzyl-4-pentylmorpholin-4-ium chloride

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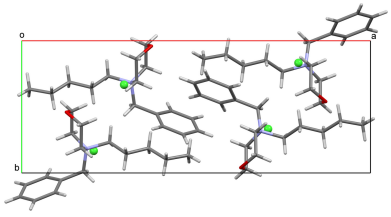
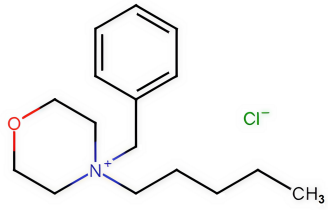
The reaction of *N*-pentylmorpholine with benzyl chloride resulted in the title compound, $C_{16}H_{26}ClNO$, which crystallizes in the orthorhombic space group $Pna2_1$ with $Z = 4$. In the crystal, the chloride ions are surrounded by four cations, forming layers.

1. Chemical context

Morpholine is a multipurpose chemical that is used as a solvent for resins, dyes and waxes. One of its most important uses is as a chemical intermediate in the preparation of pesticides (Muruganandam *et al.*, 2009). A number of morpholine derivatives have been described as analgesics and local anesthetics. The morpholinomethyl derivative of pyrizinamide (morphozinamide) has been found to be more effective in the treatment of tuberculosis than pyrizinamide (Sedavkina *et al.*, 1984). Quaternary morpholine halides were found to achieve total disinfection against *Staphylococcus aureus* ATCC 25923 and *Escherichia coli* ATCC 25922. (Morandini *et al.*, 2021). Additionally, most drugs containing a morpholine moiety in their structure have been found to exhibit significant biological properties (Basavaraja *et al.*, 2010). Quaternary morpholine halides are valuable precursors for the preparation of ionic liquids (ILs) by ion metathesis (Kim *et al.*, 2005). The excellent conductivity, broad electrochemical window, thermal stability, and low volatility of ILs have made them promising media for electrochemical processes (Zein El Abedin *et al.*, 2004, 2005). In particular, ILs based on the morpholinium cation are favored because of their low cost, easy synthesis, and electrochemical stability (Kim *et al.*, 2006). We report here a new example structure of this class.





2. Structural commentary

The title compound crystallizes in the orthorhombic space group $Pna2_1$ with $Z = 4$. The asymmetric unit consists of a

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

*Cg*1 is the centroid of the benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*B*···Cl1^{i}	0.97	2.78	3.708 (2)	160
C3—H3*B*···Cl1^{ii}	0.97	2.78	3.645 (2)	149
C6—H6*B*···Cl1	0.97	2.77	3.477 (2)	130
C12—H12*B*···Cl1^{ii}	0.97	2.75	3.639 (2)	153
C15—H15···*Cg*1^{iii}	0.93	3.28	4.104 (3)	149

Symmetry codes: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, z+\frac{1}{2}$; (ii) $x, y, z+1$; (iii) $-x+1, -y+1, z+\frac{1}{2}$.

4-benzyl-4-pentylmorpholin-4-ium cation with a quaternary nitrogen atom and the chloride counter-anion, which ensures neutrality (Fig. 1). The average C—N bond length of 1.521 Å and C—N—C angle of 109° are consistent with the geometry of a charged quaternary nitrogen atom found in different structures (Rousselin & Clavel, 2024). In the cation, the morpholinium ring adopts a chair conformation with puckering parameters (Cremer & Pople, 1975) of the ring Q = 0.5711 (18) Å, θ = 4.26 (18)°, φ = 29 (2)°. Weak intramolecular C—H ···Cl hydrogen bonds help to consolidate the conformation of the molecule (Table 1). The pentyl group carbon atoms lie in a plane with an r.m.s. deviation of 0.0252 Å.

3. Supramolecular features

The crystal packing is shown in Fig. 2, where four cations, accompanied by counter-ions, are arranged head-to-tail in the unit cell. An examination of the distribution of the positively charged nitrogen atoms in the morpholinium cations and the chloride counter-ions shows that the crystal forms ion layers parallel to the *bc* plane, which corresponds to the planar surface of the monocrystal (Fig. 3). Within these layers, each nitrogen atom forms short contacts with four chloride ions at distances of 3.938 (2), 4.657 (2), 4.892 (2), and 4.988 (2) Å. The chloride ions are separated by a distance of 6.3470 (4) Å, forming a two-dimensional structure typical of salts with a cyclobutane-like puckering conformation. Each chloride ion is surrounded by methylene groups, which form weak C—H···Cl hydrogen bonds (Table 1). The arrangement and geometry of the nitrogen atoms are similar, with a nitrogen–nitrogen distance of 6.673 (1) Å (Fig. 4). These layers are packed through the partial intercalation of alkyl and phenyl groups along the *a* axis, forming C_{ar}—H···π interactions (Table 1).

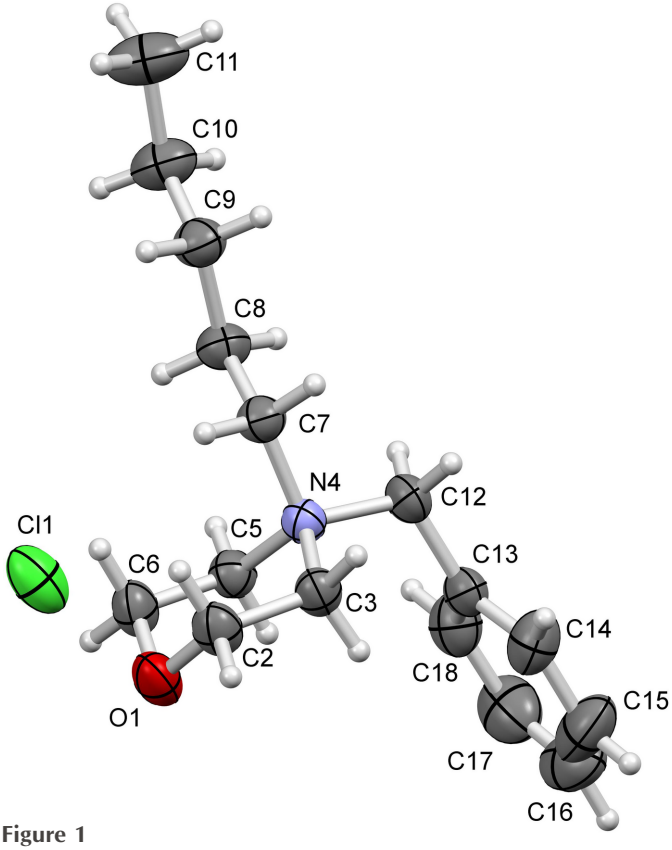


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

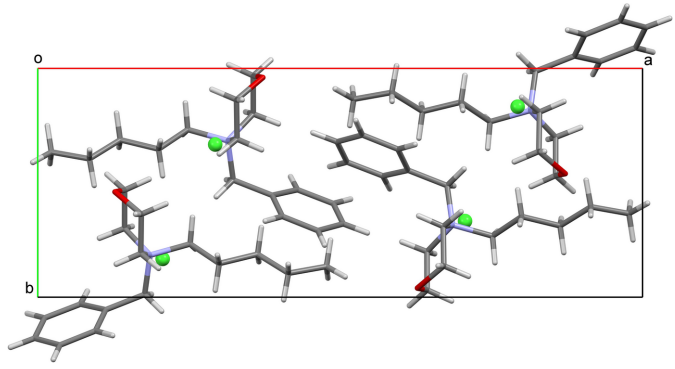


Figure 2
The packing of the title compound.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.46 of November 2024; Groom *et al.*, 2016) for

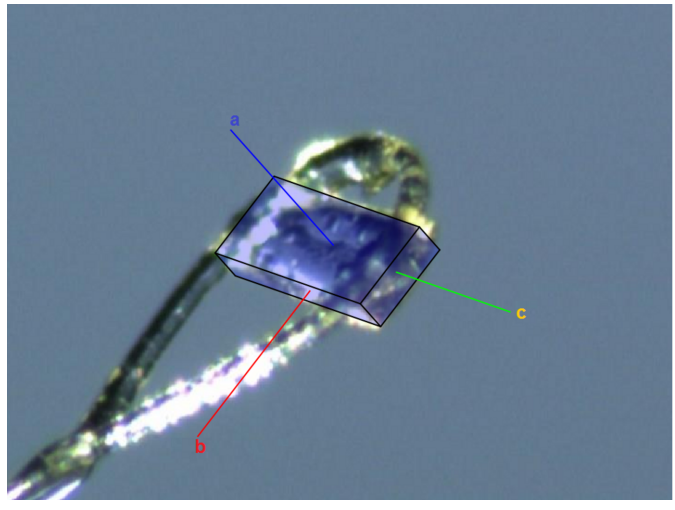


Figure 3
Screenshot from the face-indexing procedure (showing the unit-cell axes).

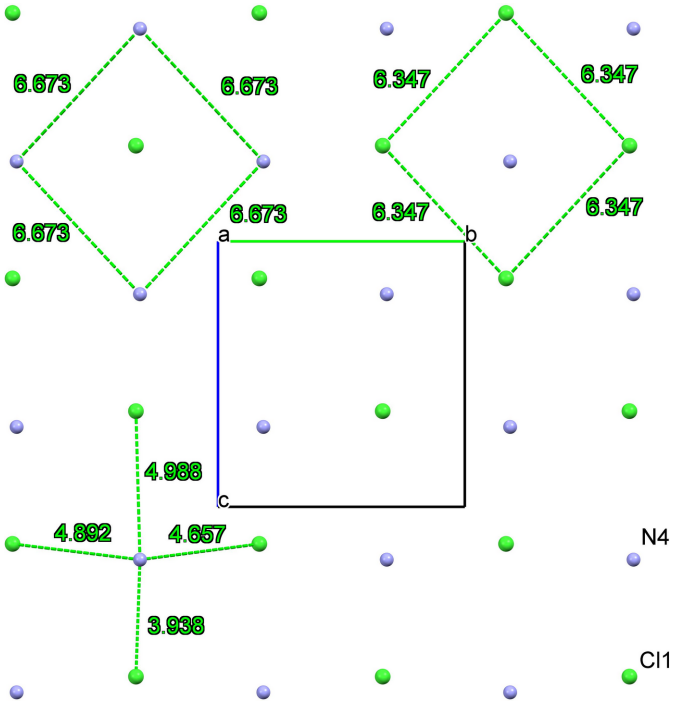


Figure 4
Distribution of positively charged nitrogen atoms and chloride counter-ions in a layer. Interatomic distances are given in Å.

structures containing a morpholine fragment with three bonded nitrogen atom returned 2745 hits. A search for structures containing a morpholin-4-ium fragment returned 188 hits, while a search for the morpholin-4-ium fragment with a benzyl substituent produced 10 matches. Homologous structures with methyl and ethyl substituents are MOKJOM (Bian, 2009*a*) and DOKYAE (Bian, 2009*b*). The C—N bond length in a neutral morpholine fragment is approximately 1.46–1.48 Å (Groom *et al.*, 2016; Mutalliev *et al.*, 2022) while the C—N bond in a morpholin-4-ium structure, as mentioned above, measures around 1.52 Å.

5. Synthesis and crystallization

N-pentyl morpholine, $C_9H_{19}NO$. To a 50 ml round-bottom flask, 4.95 g (0.06 mol) of morpholine were added. After adding 0.6 mol of ethanol as the solvent, 0.06 mol of potassium carbonate (K_2CO_3) and then 7.50 ml (0.06 mol) of pentyl bromide were added. The reaction mixture was heated under reflux with magnetic stirring for 1–9 h (monitored by TLC). Afterward, the solvent was evaporated. The remaining potassium carbonate in the solution was dissolved in water, and the reaction product was extracted with chloroform ($CHCl_3$). After the chloroform had evaporated, the residue was dried under vacuum. Yield 6.5 g (72.0%).

^{1}H-NMR (600 MHz, $CDCl_3$, δ, ppm *J*/Hz): 0.85 (3H, *t*, *J* = 7.2, H-11), 1.26 (4H, *m*, H-9,10), 1.44 (2H, *kd*, *J* = 7.4, 2.1, H-8), 2.27 (2H, *dt*, *J* = 7.9, 2.4, H-7), 2.39 (4H, *s*, H-2,6), 3.68 (4H, *s*, H-3,5).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{26}NO^+\cdot Cl^-$
M_r	283.83
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	293
a, *b*, *c* (Å)	21.8109 (4), 8.2459 (2), 8.8751 (2)
V (Å^3)	1596.19 (6)
Z	4
Radiation type	Cu *Kα*
μ (mm^{-1})	2.05
Crystal size (mm)	0.30 × 0.10 × 0.05
Data collection	
Diffractometer	Bruker D8 VENTURE dual wavelength Mo/Cu
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.620, 0.754
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22843, 3168, 3064
R_{int}	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.029, 0.074, 1.06
No. of reflections	3168
No. of parameters	173
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.21
Absolute structure	Flack *x* determined using 1329 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.014 (6)

Computer programs: *APEX5* (Bruker, 2023), *SAINT* (Bruker, 2019), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2014/7* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 14.09 (C-11), 22.41 (C-10), 26.31 (C-8), 29.89 (C-9), 53.62 (C-2,6), 59.29 (C-7), 66,93 (C-3,5).

IR spectrum (KBr, ν_{max}, cm^{-1}): 2957, 2931, 2856, 2807, 1708, 1454, 1358, 1271, 1118, 1071-1757, 1034, 1004, 914, 864, 796, 628.

4-Benzyl-4-pentylmorpholin-4-ium chloride, $C_{16}H_{26}ClNO$. To a 50 ml round-bottom flask, 2 g (0.013 mol) of *N*-pentyl morpholine were added. After adding 5.4 ml (0.104 mol) of acetonitrile as the solvent, 0.013 mol of potassium carbonate (K_2CO_3) were added, followed by benzyl chloride in a 1:1 ratio, *i.e.*, 0.013 mol. The reaction mixture was heated under reflux with magnetic stirring for 5 h (monitored by TLC). Then the solvent was evaporated, the remaining potassium carbonate was dissolved in water, and the reaction product was extracted with chloroform ($CHCl_3$). After the chloroform had evaporated, the product was dried under vacuum. The obtained product was purified using column chromatography. Yield 3.16 g (94.0%), m.p. 467–469 K. Single crystals were obtained by slow evaporation of an acetone solution.

^{1}H-NMR (600 MHz, $CDCl_3$, δ, ppm *J*/Hz): 0.87 (3H, *t*, *J* = 6.19, H-11), 1.33 (4H, *m*, H-9,10), 1.80 (2H, *m*, H-8), 3.40 (2H, *dd*, *J* = 9.63, 2.38, H-7), 3.58 (4H, *m*, H-1,5), 3.76 (2H, *t*, *J* = 10.41 H-12), 3.95 (2H, *m*, H-2), 4.07 (2H, *d*, *J* = 13.94 H-4), 7.40 (3H, *m*, H-16,17,15), 7.58 (2H, *d*, *J* = 4.95 H-18,14).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 13.94 (C-18), 21.84 (C-17), 22.31 (C-15), 28.42 (C-16), 56.33 (C-2,4), 56.98 (C-7), 60,62 (C-1,5), 64,85 (C-8), 126.77 (C-9), 129.42 (C-11,13), 130.86 (C-12), 133.40 (C-10,14).

IR spectrum (KBr, ν_{max}, cm^{-1}): 2976, 2951, 2873, 1495, 1458, 1393, 1216, 1121, 1050, 1019, 991, 946, 932, 912, 886, 860, 764.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were placed in calculated positions and refined as riding on their parent atoms [C—H = 0.93–0.97 Å with U_{iso}(H) = 1.2U_{eq}(C)].

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Synthesis and crystal structure of 4-benzyl-4-pentylmorpholin-4-ium chloride

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Computing details

4-Benzyl-4-pentylmorpholin-4-ium chloride

Crystal data

$C_{16}H_{26}NO^+\cdot Cl^-$
M_r = 283.83
Orthorhombic, $Pna2_1$
a = 21.8109 (4) Å
b = 8.2459 (2) Å
c = 8.8751 (2) Å
V = 1596.19 (6) Å^3
Z = 4
$F(000)$ = 616
D_x = 1.181 Mg m^{-3}
Melting point: 468(2) K
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9944 reflections
θ = 4.1–74.5°
μ = 2.05 mm^{-1}
T = 293 K
Plate, colourless
0.30 × 0.10 × 0.05 mm

Data collection

Bruker D8 VENTURE dual wavelength Mo/Cu diffractometer
Radiation source: microfocus X-ray source, Incoatec IμS 3.0 Microfocus Source
Mirror monochromator
ω–φ scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.620, T_{max} = 0.754
22843 measured reflections
3168 independent reflections
3064 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 74.6°, θ_{min} = 4.1°
h = −27→25
k = −9→10
l = −11→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.074
S = 1.06
3168 reflections
173 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 0.1423P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack x determined using 1329 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.014 (6)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.29346 (3)	0.33262 (7)	0.13961 (7)	0.06703 (18)
O1	0.37213 (7)	0.03995 (18)	0.55365 (16)	0.0541 (4)
N4	0.31419 (6)	0.31617 (17)	0.69908 (18)	0.0378 (3)
C2	0.35629 (9)	0.0320 (2)	0.7090 (2)	0.0489 (4)
H2A	0.3854	−0.0368	0.7614	0.059*
H2B	0.3159	−0.0160	0.7195	0.059*
C3	0.35639 (9)	0.1990 (2)	0.7799 (2)	0.0431 (4)
H3A	0.3978	0.2418	0.7786	0.052*
H3B	0.3437	0.1900	0.8843	0.052*
C5	0.32960 (9)	0.3091 (2)	0.5339 (2)	0.0421 (4)
H5A	0.3700	0.3553	0.5177	0.050*
H5B	0.3002	0.3736	0.4778	0.050*
C6	0.32864 (10)	0.1372 (2)	0.4759 (2)	0.0492 (4)
H6A	0.2880	0.0919	0.4892	0.059*
H6B	0.3380	0.1369	0.3690	0.059*
C7	0.24799 (8)	0.2672 (2)	0.7317 (2)	0.0428 (4)
H7A	0.2441	0.1512	0.7163	0.051*
H7B	0.2394	0.2890	0.8370	0.051*
C8	0.19981 (8)	0.3526 (2)	0.6362 (3)	0.0468 (4)
H8A	0.2029	0.3170	0.5323	0.056*
H8B	0.2064	0.4689	0.6391	0.056*
C9	0.13639 (8)	0.3120 (2)	0.6983 (3)	0.0502 (5)
H9A	0.1303	0.1956	0.6940	0.060*
H9B	0.1343	0.3447	0.8032	0.060*
C10	0.08545 (9)	0.3951 (3)	0.6118 (3)	0.0613 (6)
H10A	0.0858	0.3565	0.5085	0.074*
H10B	0.0933	0.5109	0.6098	0.074*
C11	0.02260 (11)	0.3655 (4)	0.6789 (4)	0.0841 (10)
H11A	−0.0082	0.4107	0.6139	0.126*
H11B	0.0202	0.4160	0.7762	0.126*
H11C	0.0159	0.2509	0.6892	0.126*
C12	0.32319 (8)	0.4877 (2)	0.7628 (2)	0.0453 (4)
H12A	0.2952	0.5606	0.7114	0.054*
H12B	0.3120	0.4868	0.8685	0.054*
C13	0.38755 (8)	0.5547 (2)	0.7483 (2)	0.0437 (4)
C14	0.42946 (11)	0.5302 (3)	0.8635 (3)	0.0612 (6)
H14	0.4180	0.4735	0.9496	0.073*
C15	0.48864 (12)	0.5907 (3)	0.8500 (4)	0.0801 (8)
H15	0.5172	0.5711	0.9256	0.096*

C16	0.50513 (13)	0.6793 (3)	0.7253 (5)	0.0831 (9)
H16	0.5449	0.7188	0.7161	0.100*
C17	0.46324 (14)	0.7091 (3)	0.6154 (4)	0.0769 (8)
H17	0.4742	0.7719	0.5326	0.092*
C18	0.40444 (11)	0.6468 (2)	0.6257 (3)	0.0584 (5)
H18	0.3762	0.6673	0.5495	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0981 (4)	0.0586 (3)	0.0444 (2)	0.0226 (3)	0.0031 (3)	0.0032 (3)
O1	0.0621 (9)	0.0502 (8)	0.0500 (8)	0.0156 (6)	−0.0040 (7)	−0.0115 (6)
N4	0.0375 (7)	0.0369 (7)	0.0389 (8)	0.0034 (5)	−0.0024 (6)	−0.0031 (6)
C2	0.0556 (11)	0.0426 (10)	0.0483 (11)	0.0106 (8)	−0.0070 (9)	−0.0021 (9)
C3	0.0452 (9)	0.0433 (9)	0.0409 (9)	0.0078 (7)	−0.0066 (8)	−0.0025 (8)
C5	0.0435 (9)	0.0452 (10)	0.0376 (9)	0.0017 (7)	−0.0001 (7)	−0.0011 (7)
C6	0.0596 (12)	0.0470 (10)	0.0409 (10)	0.0041 (8)	−0.0070 (9)	−0.0069 (8)
C7	0.0385 (8)	0.0427 (9)	0.0473 (9)	0.0002 (7)	0.0003 (7)	0.0010 (8)
C8	0.0396 (9)	0.0508 (9)	0.0501 (9)	0.0027 (7)	−0.0008 (9)	0.0026 (10)
C9	0.0419 (9)	0.0503 (10)	0.0583 (12)	−0.0014 (8)	0.0010 (9)	−0.0007 (9)
C10	0.0434 (10)	0.0626 (12)	0.0780 (17)	0.0017 (9)	0.0005 (10)	0.0120 (11)
C11	0.0460 (12)	0.0957 (19)	0.110 (3)	0.0070 (11)	0.0087 (13)	0.0243 (17)
C12	0.0468 (9)	0.0386 (9)	0.0505 (11)	0.0034 (7)	−0.0001 (8)	−0.0088 (8)
C13	0.0460 (9)	0.0341 (8)	0.0510 (10)	0.0011 (7)	0.0000 (8)	−0.0075 (7)
C14	0.0660 (13)	0.0534 (12)	0.0642 (13)	−0.0100 (10)	−0.0163 (12)	0.0010 (10)
C15	0.0621 (14)	0.0643 (15)	0.114 (2)	−0.0117 (12)	−0.0296 (15)	−0.0005 (16)
C16	0.0594 (13)	0.0580 (14)	0.132 (3)	−0.0143 (11)	0.0110 (17)	−0.0100 (16)
C17	0.0891 (18)	0.0552 (13)	0.086 (2)	−0.0174 (12)	0.0222 (16)	0.0003 (14)
C18	0.0734 (14)	0.0413 (9)	0.0604 (13)	−0.0024 (9)	−0.0029 (12)	0.0001 (11)

Geometric parameters (Å, º)

O1—C6	1.421 (3)	C9—H9A	0.9700
O1—C2	1.423 (2)	C9—H9B	0.9700
N4—C5	1.506 (3)	C10—C11	1.515 (3)
N4—C3	1.515 (2)	C10—H10A	0.9700
N4—C7	1.527 (2)	C10—H10B	0.9700
N4—C12	1.536 (2)	C11—H11A	0.9600
C2—C3	1.514 (3)	C11—H11B	0.9600
C2—H2A	0.9700	C11—H11C	0.9600
C2—H2B	0.9700	C12—C13	1.514 (3)
C3—H3A	0.9700	C12—H12A	0.9700
C3—H3B	0.9700	C12—H12B	0.9700
C5—C6	1.508 (3)	C13—C18	1.378 (3)
C5—H5A	0.9700	C13—C14	1.386 (3)
C5—H5B	0.9700	C14—C15	1.389 (3)
C6—H6A	0.9700	C14—H14	0.9300
C6—H6B	0.9700	C15—C16	1.374 (5)

C7—C8	1.522 (3)	C15—H15	0.9300
C7—H7A	0.9700	C16—C17	1.359 (5)
C7—H7B	0.9700	C16—H16	0.9300
C8—C9	1.526 (3)	C17—C18	1.384 (4)
C8—H8A	0.9700	C17—H17	0.9300
C8—H8B	0.9700	C18—H18	0.9300
C9—C10	1.514 (3)		

C6—O1—C2	109.55 (15)	C10—C9—C8	112.49 (18)
C5—N4—C3	107.53 (14)	C10—C9—H9A	109.1
C5—N4—C7	112.67 (14)	C8—C9—H9A	109.1
C3—N4—C7	108.43 (14)	C10—C9—H9B	109.1
C5—N4—C12	111.44 (14)	C8—C9—H9B	109.1
C3—N4—C12	109.60 (13)	H9A—C9—H9B	107.8
C7—N4—C12	107.12 (13)	C9—C10—C11	113.1 (2)
O1—C2—C3	111.15 (17)	C9—C10—H10A	109.0
O1—C2—H2A	109.4	C11—C10—H10A	109.0
C3—C2—H2A	109.4	C9—C10—H10B	109.0
O1—C2—H2B	109.4	C11—C10—H10B	109.0
C3—C2—H2B	109.4	H10A—C10—H10B	107.8
H2A—C2—H2B	108.0	C10—C11—H11A	109.5
C2—C3—N4	112.47 (14)	C10—C11—H11B	109.5
C2—C3—H3A	109.1	H11A—C11—H11B	109.5
N4—C3—H3A	109.1	C10—C11—H11C	109.5
C2—C3—H3B	109.1	H11A—C11—H11C	109.5
N4—C3—H3B	109.1	H11B—C11—H11C	109.5
H3A—C3—H3B	107.8	C13—C12—N4	115.05 (14)
N4—C5—C6	111.45 (16)	C13—C12—H12A	108.5
N4—C5—H5A	109.3	N4—C12—H12A	108.5
C6—C5—H5A	109.3	C13—C12—H12B	108.5
N4—C5—H5B	109.3	N4—C12—H12B	108.5
C6—C5—H5B	109.3	H12A—C12—H12B	107.5
H5A—C5—H5B	108.0	C18—C13—C14	119.1 (2)
O1—C6—C5	110.81 (16)	C18—C13—C12	121.06 (19)
O1—C6—H6A	109.5	C14—C13—C12	119.7 (2)
C5—C6—H6A	109.5	C13—C14—C15	119.8 (3)
O1—C6—H6B	109.5	C13—C14—H14	120.1
C5—C6—H6B	109.5	C15—C14—H14	120.1
H6A—C6—H6B	108.1	C16—C15—C14	120.2 (3)
C8—C7—N4	115.16 (16)	C16—C15—H15	119.9
C8—C7—H7A	108.5	C14—C15—H15	119.9
N4—C7—H7A	108.5	C17—C16—C15	119.9 (2)
C8—C7—H7B	108.5	C17—C16—H16	120.1
N4—C7—H7B	108.5	C15—C16—H16	120.1
H7A—C7—H7B	107.5	C16—C17—C18	120.6 (3)
C7—C8—C9	108.86 (19)	C16—C17—H17	119.7
C7—C8—H8A	109.9	C18—C17—H17	119.7
C9—C8—H8A	109.9	C13—C18—C17	120.3 (2)

C7—C8—H8B	109.9	C13—C18—H18	119.9
C9—C8—H8B	109.9	C17—C18—H18	119.9
H8A—C8—H8B	108.3		

C6—O1—C2—C3	−60.6 (2)	C8—C9—C10—C11	−175.9 (2)
O1—C2—C3—N4	56.3 (2)	C5—N4—C12—C13	−59.3 (2)
C5—N4—C3—C2	−51.0 (2)	C3—N4—C12—C13	59.6 (2)
C7—N4—C3—C2	71.1 (2)	C7—N4—C12—C13	176.99 (16)
C12—N4—C3—C2	−172.25 (16)	N4—C12—C13—C18	92.4 (2)
C3—N4—C5—C6	52.57 (19)	N4—C12—C13—C14	−90.8 (2)
C7—N4—C5—C6	−66.85 (19)	C18—C13—C14—C15	−3.6 (3)
C12—N4—C5—C6	172.70 (15)	C12—C13—C14—C15	179.6 (2)
C2—O1—C6—C5	62.8 (2)	C13—C14—C15—C16	2.2 (4)
N4—C5—C6—O1	−60.2 (2)	C14—C15—C16—C17	0.6 (4)
C5—N4—C7—C8	−50.2 (2)	C15—C16—C17—C18	−2.0 (4)
C3—N4—C7—C8	−169.13 (16)	C14—C13—C18—C17	2.3 (3)
C12—N4—C7—C8	72.7 (2)	C12—C13—C18—C17	179.0 (2)
N4—C7—C8—C9	−171.03 (16)	C16—C17—C18—C13	0.5 (4)
C7—C8—C9—C10	178.75 (19)		

Hydrogen-bond geometry (Å, º)

*Cg*1 is the centroid of the benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*B*···Cl1^{i}	0.97	2.78	3.708 (2)	160
C3—H3*B*···Cl1^{ii}	0.97	2.78	3.645 (2)	149
C6—H6*B*···Cl1	0.97	2.77	3.477 (2)	130
C12—H12*B*···Cl1^{ii}	0.97	2.75	3.639 (2)	153
C15—H15···*Cg*1^{iii}	0.93	3.28	4.104 (3)	149

Symmetry codes: (i) −*x*+1/2, *y*−1/2, *z*+1/2; (ii) *x*, *y*, *z*+1; (iii) −*x*+1, −*y*+1, *z*+1/2.