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1-Isobutyl-8,9-dimethoxy-3-phenyl-5,6-dihidro-imidazo[5,1-*a*]isoquinolin-2-ium chloride

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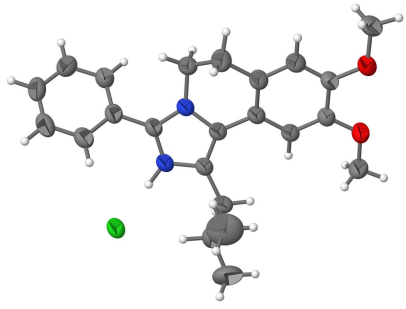
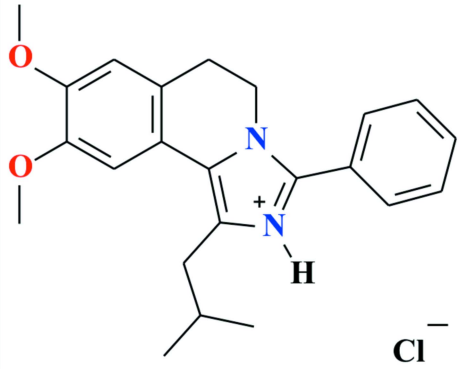
Structural data: full structural data are available from iucrdata.iucr.org

The molecular salt, $C_{23}H_{26}N_2O_2^+\cdot Cl^-$, was obtained from 1-isobutyl-8,9-dimethoxy-3-phenyl-5,6-dihydroimidazo[5,1-*a*]isoquinoline, which was synthesized by cyclocondensation of α-benzoylamino-γ-methyl-*N*-[2-(3,4-dimethoxyphenyl)ethyl]valeramide in the presence of phosphoryl chloride. The tetrahydropyridine ring adopts a twist–boat conformation. In the crystal structure, centrosymmetric dimers are formed by N—H···Cl and C—H···Cl hydrogen bonds.

3D view



Chemical scheme



Structure description

The relevance of a wide range of potent biological activities of natural and synthetic isoquinoline alkaloids is interesting for the synthesis of new isoquinoline compounds. In nature, there are compounds that contain condensed imidazole and isoquinoline rings, for example, cribrostatin 6.

Cyclization of α-benzoylamino-γ-methyl-*N*-[2-(3,4-dimethoxyphenyl)ethyl]valeramide with phosphoryl chloride based on the Bischler–Napieralski reaction results in a heterocyclic compound containing condensed imidazole and isoquinoline rings (Seganish *et al.*, 2012; Iaroshenko *et al.*, 2015; Allin *et al.*, 2005). In the reaction, phosphoryl chloride is used as a reagent and solvent (Fig. 1).

However, from the obtained 1-isobutyl-8,9-dimethoxy-3-phenyl-5,6-dihydroimidazo-[5,1-*a*]isoquinoline we could not get suitable single crystals for X-ray diffraction analysis. Good crystals of the title compound were obtained by slow evaporation of a solution of 1-isobutyl-8,9-dimethoxy-3-phenyl-5,6-dihydroimidazo[5,1-*a*]isoquinoline treated with hydrochloric acid.

The molecular structure of the title compound is shown in Fig. 2. The dihydropyridine ring occurs in a twist-boat conformation. The C6, C6*A*, C10*A* and C10*B* atoms of the dihydropyridine ring are almost coplanar (r.m.s. deviation = 0.095 Å). The C5 and N4

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1···Cl1	0.98 (3)	2.04 (3)	3.019 (2)	179
C20—H20A···Cl1^{i}	0.93	2.89	3.650 (3)	140
C5—H5B···Cl1^{ii}	0.97	2.83	3.707 (3)	152

Symmetry codes: (i) $-x+1, -y+3, -z$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

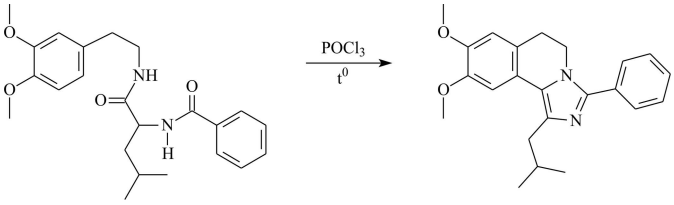


Figure 1
Reaction scheme for the preparation of the title compound.

atoms deviate from this plane by 0.806 (5) and 0.413 (5) Å, respectively. The imidazole (C1/N2/C3/N4/C10B) and benzene (C17–C22) rings are essentially planar, the dihedral angle between the planes being 41.4 (1)°. In the crystal, N2—H1···Cl1 and C20—H20A···Cl1 hydrogen bonds are observed, resulting in the formation of a centrosymmetric dimer consisting of two anions and two cations (Fig. 3 and Table 1). These dimers are linked by C5—H5B···Cl1 hydrogen bonds into a chain directed along [011].

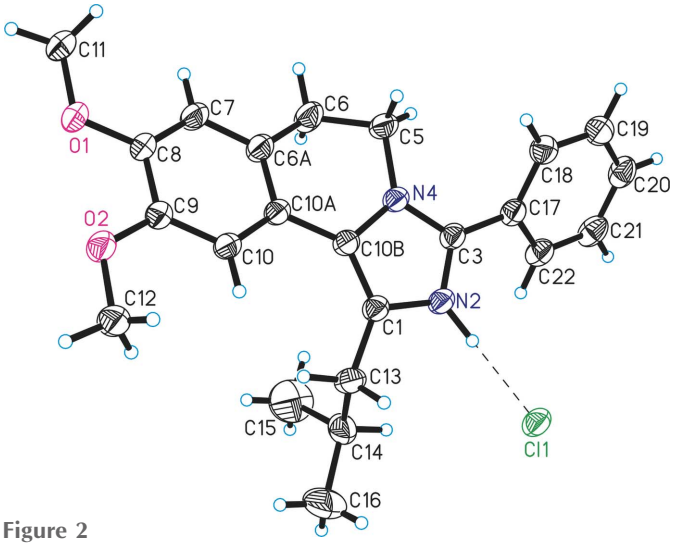


Figure 2
The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The N—H···Cl hydrogen bond is shown as a dashed line.

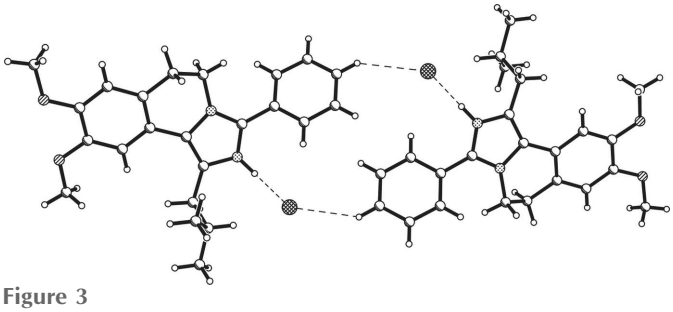


Figure 3
Formation of a centrosymmetric dimer in the crystal structure of the title compound. N—H···Cl hydrogen bonds are shown as dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{23}H_{27}N_2O_2^+\cdot Cl^-$
M_r	398.92
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	291
a, b, c (Å)	13.595 (3), 14.337 (3), 10.958 (2)
β (°)	92.23 (3)
V (Å^3)	2134.1 (7)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.74
Crystal size (mm)	0.60 × 0.53 × 0.48
Data collection	
Diffractometer	Rigaku Xcalibur Ruby
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.371, 0.434
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9283, 4347, 2990
R_{int}	0.036
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.629
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.142, 1.01
No. of reflections	4347
No. of parameters	261
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.20

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXS7* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *XP* (Bruker, 1998), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Synthesis and crystallization

To a round-bottomed flask with 0.5 g (1.25 mmol) of α-benzoylamino-γ-methyl-*N*-[2-(3,4-dimethoxyphenyl)ethyl]-valeramide was added dropwise 0.7 ml (7.64 mmol) of $POCl_3$. The reaction mixture was heated for 4 h in a boiling water bath. The course of the reaction was monitored using thin-layer chromatography (TLC). After heating, the reaction tube was filled with crushed ice, the pH of the solution was adjusted to 9 with 25% ammonium hydroxide solution. The solution was extracted with chloroform (30 ml) and the organic layer was washed with water and distilled. When acetone was added to the residue, a precipitate was formed. The precipitate was filtered off and dried and giving 0.33 g (yield 74%) of product; R_F = 0.61 (1:4 CH_3OH–$CHCl_3$ v/v); m.p. 433–436 K.

0.2 g of 1-isobutyl-8,9-dimethoxy-3-phenyl-5,6-dihydro-imidazo[5,1-*a*]isoquinoline was dissolved in 25 ml of methanol and transferred to an acidic medium with 30% HCl (pH = 3). The methanol was distilled and a precipitate was obtained

when acetone was added. The precipitate was filtered off, washed with acetone and dried in the open air. 0.18 g of 1-isobutyl-8,9-dimethoxy-3-phenyl-5,6-dihydroimidazo[5,1-*a*]-isoquinoline hydrochloride was obtained (yield 82%); R_F = 0.32 (1:4 CH_3OH–$CHCl_3$ *v*/*v*); m.p. 475–477 K.

1-Isobutyl-8,9-dimethoxy-3-phenyl-5,6-dihydroimidazo[5,1-*a*]isoquinoline hydrochloride was dissolved in a 4:1 (*v*/*v*) acetone–methanol solvent mixture and allowed to evaporation at room temperature. Colourless crystals suitable for X-ray diffraction analysis were obtained.

^{1}H NMR [400 MHz, CD_3OD, δ (p.p.m.), *J* (Hz)]: 7.69 (2*H*, *dt*, *J* = 1.9; 6.0, H18 and H22); 7.62 (2*H*, *dd*, *J* = 1.7; 6.6, H19 and H21); 7.60 (1*H*, *dt*, *J* = 1.4; 6.0, H20), 7.18 (1*H*, *s*, H7); 6.97 (1*H*, *s*, H10); 4.26 (2*H*, *t*, *J* = 6.4, CH_2-5); 3.85 (3*H*, *s*, CH_3-12); 3.83 (3*H*, *s*, CH_3-11), 3.01 (2*H*, *t*, *J* = 6.4, CH_2-6); 2.86 (2*H*, *t*, *J* = 7.3, CH_2-13); 2.09 (1*H*, *q*, *J* = 6.8, H14); 1.02 (6*H*, *d*, *J* = 6.6, CH_3-15,16).

^{13}C NMR [100 MHz, CD_3OD, δ (p.p.m.)]: 23.02 (C15, C16); 29.62 (C14); 30.61 (C6); 36.02 (C13); 44.52 (C5); 57.05 (C11); 57.27 (C12); 109.98 (C10); 113.96 (C7); 119.63 (C10*A*); 125.95 (C6*A*; C1), 128.73 (C18, C22); 131.06 (C19, C21); 131.42 (C20); 133.72 (C10*B*, C17); 144.77 (C3); 150.99 (C9); 152.16 (C8).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

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full crystallographic data

IUCrData (2019). **4**, x191390 [https://doi.org/10.1107/S2414314619013907]

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Crystal data

$C_{23}H_{27}N_2O_2^+\cdot Cl^-$
M_r = 398.92
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 13.595 (3) Å
b = 14.337 (3) Å
c = 10.958 (2) Å
β = 92.23 (3)°
V = 2134.1 (7) Å^3
Z = 4
$F(000)$ = 848
D_x = 1.242 Mg m^{-3}
Melting point: 475(2) K
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 2082 reflections
θ = 4.5–75.8°
μ = 1.74 mm^{-1}
T = 291 K
Prizmatic, colorless
0.60 × 0.53 × 0.48 mm

Data collection

Rigaku Xcalibur Ruby diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.2576 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
T_{min} = 0.371, T_{max} = 0.434
9283 measured reflections
4347 independent reflections
2990 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 76.0°, θ_{min} = 4.5°
h = −16→17
k = −18→16
l = −9→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.142
S = 1.01
4347 reflections
261 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0678P)^2 + 0.2438P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 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. 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. The H atoms bonded to C atoms were placed geometrically (with C—H distances of 0.98 Å for CH, 0.97 Å for CH_2, 0.96 Å for CH_3 and 0.93 Å for C_{ar}) and included in the refinement in a riding motion approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$ [$U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms]. The H atom of N2 was located in a difference Fourier synthesis and refined with a N2—H1 distance = 0.79 (3) Å.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.85755 (14)	0.77486 (11)	0.41517 (17)	0.0640 (5)
O2	0.89458 (14)	0.91068 (12)	0.56498 (16)	0.0623 (5)
N2	0.66229 (14)	1.27667 (13)	0.26859 (17)	0.0478 (4)
N4	0.63568 (14)	1.13705 (12)	0.20336 (17)	0.0452 (4)
C1	0.72530 (17)	1.21988 (15)	0.3370 (2)	0.0457 (5)
C3	0.60827 (16)	1.22675 (15)	0.1889 (2)	0.0459 (5)
C5	0.5983 (2)	1.05567 (16)	0.1344 (2)	0.0569 (6)
H5A	0.5742	1.0748	0.0537	0.068*
H5B	0.5443	1.0273	0.1763	0.068*
C6	0.6813 (2)	0.98620 (19)	0.1235 (3)	0.0692 (8)
H6A	0.6550	0.9283	0.0901	0.083*
H6B	0.7283	1.0103	0.0670	0.083*
C6A	0.7336 (2)	0.96661 (16)	0.2439 (2)	0.0545 (6)
C7	0.7699 (2)	0.87809 (16)	0.2724 (2)	0.0589 (6)
H7A	0.7587	0.8298	0.2169	0.071*
C8	0.82169 (18)	0.85996 (15)	0.3799 (2)	0.0503 (5)
C9	0.83978 (17)	0.93362 (15)	0.4627 (2)	0.0474 (5)
C10	0.80171 (17)	1.02087 (15)	0.4369 (2)	0.0467 (5)
H10A	0.8115	1.0688	0.4932	0.056*
C10A	0.74883 (17)	1.03854 (14)	0.3279 (2)	0.0451 (5)
C10B	0.70861 (17)	1.13078 (15)	0.29649 (19)	0.0443 (5)
C11	0.8322 (2)	0.69713 (18)	0.3379 (3)	0.0744 (8)
H11A	0.8595	0.6411	0.3732	0.112*
H11B	0.8584	0.7067	0.2588	0.112*
H11C	0.7619	0.6915	0.3301	0.112*
C12	0.9174 (2)	0.98354 (18)	0.6494 (2)	0.0648 (7)
H12A	0.9622	0.9607	0.7123	0.097*
H12B	0.8580	1.0047	0.6853	0.097*
H12C	0.9472	1.0344	0.6076	0.097*
C13	0.79478 (19)	1.26228 (17)	0.4306 (2)	0.0551 (6)
H13A	0.8276	1.2126	0.4764	0.066*
H13B	0.7571	1.2983	0.4874	0.066*

C14	0.8726 (2)	1.32535 (19)	0.3772 (3)	0.0671 (7)
H14A	0.8384	1.3731	0.3277	0.080*
C15	0.9379 (3)	1.2716 (3)	0.2951 (4)	0.1234 (15)
H15A	0.8984	1.2425	0.2313	0.185*
H15B	0.9728	1.2246	0.3417	0.185*
H15C	0.9841	1.3133	0.2598	0.185*
C16	0.9321 (3)	1.3745 (3)	0.4774 (4)	0.1160 (15)
H16A	0.9796	1.4147	0.4418	0.174*
H16B	0.9654	1.3291	0.5284	0.174*
H16C	0.8889	1.4110	0.5257	0.174*
C17	0.53556 (17)	1.26476 (16)	0.1005 (2)	0.0479 (5)
C18	0.44587 (19)	1.22054 (18)	0.0757 (2)	0.0583 (6)
H18A	0.4311	1.1652	0.1153	0.070*
C19	0.3789 (2)	1.2590 (2)	−0.0080 (3)	0.0668 (7)
H19A	0.3185	1.2299	−0.0232	0.080*
C20	0.4007 (2)	1.34047 (19)	−0.0695 (2)	0.0653 (7)
H20A	0.3561	1.3651	−0.1273	0.078*
C21	0.4889 (2)	1.38447 (18)	−0.0442 (2)	0.0648 (7)
H21A	0.5035	1.4395	−0.0847	0.078*
C22	0.5564 (2)	1.34783 (17)	0.0408 (2)	0.0558 (6)
H22A	0.6155	1.3786	0.0580	0.067*
Cl1	0.65752 (5)	1.48541 (4)	0.30437 (6)	0.0602 (2)
H1	0.661 (2)	1.345 (2)	0.280 (3)	0.076 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0725 (11)	0.0376 (8)	0.0803 (12)	0.0105 (8)	−0.0194 (9)	−0.0055 (8)
O2	0.0825 (12)	0.0436 (9)	0.0588 (10)	0.0087 (8)	−0.0214 (9)	−0.0025 (8)
N2	0.0565 (11)	0.0344 (9)	0.0520 (11)	0.0013 (8)	−0.0040 (9)	−0.0001 (8)
N4	0.0521 (10)	0.0357 (9)	0.0472 (10)	−0.0002 (8)	−0.0038 (8)	0.0003 (8)
C1	0.0519 (12)	0.0374 (11)	0.0475 (12)	−0.0005 (9)	−0.0018 (10)	0.0013 (9)
C3	0.0508 (12)	0.0376 (11)	0.0492 (12)	0.0021 (9)	0.0012 (10)	0.0027 (9)
C5	0.0723 (16)	0.0405 (12)	0.0565 (14)	−0.0001 (11)	−0.0169 (12)	−0.0052 (11)
C6	0.095 (2)	0.0496 (14)	0.0608 (16)	0.0164 (14)	−0.0204 (15)	−0.0142 (12)
C6A	0.0677 (15)	0.0418 (12)	0.0532 (14)	0.0064 (11)	−0.0093 (12)	−0.0053 (10)
C7	0.0737 (16)	0.0394 (12)	0.0624 (15)	0.0062 (11)	−0.0145 (13)	−0.0113 (11)
C8	0.0542 (13)	0.0351 (11)	0.0612 (14)	0.0045 (9)	−0.0050 (11)	−0.0011 (10)
C9	0.0511 (12)	0.0400 (11)	0.0508 (12)	0.0007 (10)	−0.0030 (10)	0.0025 (10)
C10	0.0537 (12)	0.0376 (11)	0.0484 (12)	0.0001 (9)	−0.0040 (10)	−0.0032 (10)
C10A	0.0509 (12)	0.0360 (11)	0.0481 (12)	0.0006 (9)	−0.0017 (10)	−0.0006 (9)
C10B	0.0509 (12)	0.0389 (11)	0.0428 (11)	0.0002 (9)	−0.0018 (10)	−0.0008 (9)
C11	0.0809 (19)	0.0387 (13)	0.102 (2)	0.0087 (13)	−0.0182 (17)	−0.0127 (14)
C12	0.0815 (18)	0.0543 (14)	0.0567 (15)	0.0021 (13)	−0.0198 (13)	−0.0041 (12)
C13	0.0684 (15)	0.0421 (12)	0.0540 (13)	−0.0063 (11)	−0.0065 (11)	−0.0041 (11)
C14	0.0576 (15)	0.0552 (15)	0.087 (2)	−0.0029 (12)	−0.0157 (14)	0.0146 (14)
C15	0.092 (3)	0.142 (4)	0.139 (4)	−0.012 (3)	0.043 (3)	−0.017 (3)
C16	0.093 (3)	0.093 (3)	0.159 (4)	−0.036 (2)	−0.035 (3)	−0.009 (3)

C17	0.0572 (13)	0.0414 (12)	0.0449 (12)	0.0057 (10)	−0.0011 (10)	−0.0003 (9)
C18	0.0631 (15)	0.0484 (13)	0.0627 (15)	0.0028 (11)	−0.0067 (12)	0.0042 (11)
C19	0.0687 (16)	0.0615 (16)	0.0689 (17)	0.0069 (13)	−0.0158 (13)	−0.0083 (14)
C20	0.0848 (19)	0.0565 (15)	0.0534 (15)	0.0194 (14)	−0.0134 (14)	−0.0014 (12)
C21	0.097 (2)	0.0451 (13)	0.0514 (14)	0.0094 (14)	−0.0001 (14)	0.0068 (11)
C22	0.0710 (15)	0.0434 (12)	0.0531 (14)	0.0017 (11)	0.0012 (12)	0.0017 (11)
Cl1	0.0777 (4)	0.0391 (3)	0.0627 (4)	0.0086 (3)	−0.0097 (3)	−0.0026 (3)

Geometric parameters (Å, º)

O1—C8	1.364 (3)	C11—H11B	0.9600
O1—C11	1.433 (3)	C11—H11C	0.9600
O2—C9	1.362 (3)	C12—H12A	0.9600
O2—C12	1.421 (3)	C12—H12B	0.9600
N2—C3	1.328 (3)	C12—H12C	0.9600
N2—C1	1.381 (3)	C13—C14	1.526 (4)
N2—H1	0.98 (3)	C13—H13A	0.9700
N4—C3	1.346 (3)	C13—H13B	0.9700
N4—C10B	1.398 (3)	C14—C15	1.501 (5)
N4—C5	1.470 (3)	C14—C16	1.512 (4)
C1—C10B	1.368 (3)	C14—H14A	0.9800
C1—C13	1.497 (3)	C15—H15A	0.9600
C3—C17	1.462 (3)	C15—H15B	0.9600
C5—C6	1.513 (4)	C15—H15C	0.9600
C5—H5A	0.9700	C16—H16A	0.9600
C5—H5B	0.9700	C16—H16B	0.9600
C6—C6A	1.500 (3)	C16—H16C	0.9600
C6—H6A	0.9700	C17—C18	1.392 (3)
C6—H6B	0.9700	C17—C22	1.393 (3)
C6A—C10A	1.393 (3)	C18—C19	1.381 (3)
C6A—C7	1.393 (3)	C18—H18A	0.9300
C7—C8	1.374 (3)	C19—C20	1.387 (4)
C7—H7A	0.9300	C19—H19A	0.9300
C8—C9	1.408 (3)	C20—C21	1.373 (4)
C9—C10	1.379 (3)	C20—H20A	0.9300
C10—C10A	1.393 (3)	C21—C22	1.385 (4)
C10—H10A	0.9300	C21—H21A	0.9300
C10A—C10B	1.467 (3)	C22—H22A	0.9300
C11—H11A	0.9600	Cl1—Cl1	0.0000 (19)
C8—O1—C11	116.95 (19)	H11A—C11—H11C	109.5
C9—O2—C12	117.16 (18)	H11B—C11—H11C	109.5
C3—N2—C1	110.75 (18)	O2—C12—H12A	109.5
C3—N2—H1	127.1 (16)	O2—C12—H12B	109.5
C1—N2—H1	122.1 (16)	H12A—C12—H12B	109.5
C3—N4—C10B	109.42 (18)	O2—C12—H12C	109.5
C3—N4—C5	127.51 (19)	H12A—C12—H12C	109.5
C10B—N4—C5	123.07 (18)	H12B—C12—H12C	109.5

C10B—C1—N2	106.43 (19)	C1—C13—C14	114.0 (2)
C10B—C1—C13	133.9 (2)	C1—C13—H13A	108.8
N2—C1—C13	119.61 (19)	C14—C13—H13A	108.8
N2—C3—N4	107.08 (19)	C1—C13—H13B	108.8
N2—C3—C17	125.23 (19)	C14—C13—H13B	108.8
N4—C3—C17	127.7 (2)	H13A—C13—H13B	107.7
N4—C5—C6	108.6 (2)	C15—C14—C16	111.3 (3)
N4—C5—H5A	110.0	C15—C14—C13	111.1 (3)
C6—C5—H5A	110.0	C16—C14—C13	110.9 (3)
N4—C5—H5B	110.0	C15—C14—H14A	107.8
C6—C5—H5B	110.0	C16—C14—H14A	107.8
H5A—C5—H5B	108.4	C13—C14—H14A	107.8
C6A—C6—C5	112.5 (2)	C14—C15—H15A	109.5
C6A—C6—H6A	109.1	C14—C15—H15B	109.5
C5—C6—H6A	109.1	H15A—C15—H15B	109.5
C6A—C6—H6B	109.1	C14—C15—H15C	109.5
C5—C6—H6B	109.1	H15A—C15—H15C	109.5
H6A—C6—H6B	107.8	H15B—C15—H15C	109.5
C10A—C6A—C7	118.9 (2)	C14—C16—H16A	109.5
C10A—C6A—C6	119.7 (2)	C14—C16—H16B	109.5
C7—C6A—C6	121.3 (2)	H16A—C16—H16B	109.5
C8—C7—C6A	122.0 (2)	C14—C16—H16C	109.5
C8—C7—H7A	119.0	H16A—C16—H16C	109.5
C6A—C7—H7A	119.0	H16B—C16—H16C	109.5
O1—C8—C7	125.3 (2)	C18—C17—C22	119.3 (2)
O1—C8—C9	115.9 (2)	C18—C17—C3	121.6 (2)
C7—C8—C9	118.8 (2)	C22—C17—C3	119.0 (2)
O2—C9—C10	125.2 (2)	C19—C18—C17	119.9 (2)
O2—C9—C8	115.19 (19)	C19—C18—H18A	120.1
C10—C9—C8	119.6 (2)	C17—C18—H18A	120.1
C9—C10—C10A	121.2 (2)	C18—C19—C20	120.7 (3)
C9—C10—H10A	119.4	C18—C19—H19A	119.7
C10A—C10—H10A	119.4	C20—C19—H19A	119.7
C10—C10A—C6A	119.4 (2)	C21—C20—C19	119.4 (2)
C10—C10A—C10B	122.7 (2)	C21—C20—H20A	120.3
C6A—C10A—C10B	117.9 (2)	C19—C20—H20A	120.3
C1—C10B—N4	106.30 (18)	C20—C21—C22	120.7 (3)
C1—C10B—C10A	135.2 (2)	C20—C21—H21A	119.6
N4—C10B—C10A	118.47 (19)	C22—C21—H21A	119.6
O1—C11—H11A	109.5	C21—C22—C17	119.9 (3)
O1—C11—H11B	109.5	C21—C22—H22A	120.0
H11A—C11—H11B	109.5	C17—C22—H22A	120.0
O1—C11—H11C	109.5		

C3—N2—C1—C10B	0.0 (3)	C7—C6A—C10A—C10B	−179.1 (2)
C3—N2—C1—C13	178.6 (2)	C6—C6A—C10A—C10B	2.7 (4)
C1—N2—C3—N4	−0.5 (3)	N2—C1—C10B—N4	0.4 (3)
C1—N2—C3—C17	−178.7 (2)	C13—C1—C10B—N4	−177.8 (3)

C10B—N4—C3—N2	0.7 (3)	N2—C1—C10B—C10A	179.9 (3)
C5—N4—C3—N2	−178.7 (2)	C13—C1—C10B—C10A	1.7 (5)
C10B—N4—C3—C17	178.9 (2)	C3—N4—C10B—C1	−0.7 (3)
C5—N4—C3—C17	−0.6 (4)	C5—N4—C10B—C1	178.8 (2)
C3—N4—C5—C6	146.8 (2)	C3—N4—C10B—C10A	179.7 (2)
C10B—N4—C5—C6	−32.6 (3)	C5—N4—C10B—C10A	−0.8 (3)
N4—C5—C6—C6A	49.5 (3)	C10—C10A—C10B—C1	17.4 (4)
C5—C6—C6A—C10A	−37.4 (4)	C6A—C10A—C10B—C1	−162.0 (3)
C5—C6—C6A—C7	144.4 (3)	C10—C10A—C10B—N4	−163.1 (2)
C10A—C6A—C7—C8	−1.1 (4)	C6A—C10A—C10B—N4	17.4 (3)
C6—C6A—C7—C8	177.1 (3)	C10B—C1—C13—C14	112.9 (3)
C11—O1—C8—C7	−5.2 (4)	N2—C1—C13—C14	−65.2 (3)
C11—O1—C8—C9	174.3 (2)	C1—C13—C14—C15	−62.4 (3)
C6A—C7—C8—O1	178.5 (3)	C1—C13—C14—C16	173.2 (3)
C6A—C7—C8—C9	−1.0 (4)	N2—C3—C17—C18	−139.6 (3)
C12—O2—C9—C10	−2.6 (4)	N4—C3—C17—C18	42.6 (4)
C12—O2—C9—C8	178.1 (2)	N2—C3—C17—C22	39.8 (3)
O1—C8—C9—O2	2.7 (3)	N4—C3—C17—C22	−138.0 (3)
C7—C8—C9—O2	−177.7 (2)	C22—C17—C18—C19	0.3 (4)
O1—C8—C9—C10	−176.7 (2)	C3—C17—C18—C19	179.7 (2)
C7—C8—C9—C10	2.8 (4)	C17—C18—C19—C20	1.2 (4)
O2—C9—C10—C10A	178.1 (2)	C18—C19—C20—C21	−1.7 (4)
C8—C9—C10—C10A	−2.6 (4)	C19—C20—C21—C22	0.7 (4)
C9—C10—C10A—C6A	0.4 (4)	C20—C21—C22—C17	0.9 (4)
C9—C10—C10A—C10B	−179.0 (2)	C18—C17—C22—C21	−1.4 (4)
C7—C6A—C10A—C10	1.4 (4)	C3—C17—C22—C21	179.3 (2)
C6—C6A—C10A—C10	−176.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···Cl1	0.98 (3)	2.04 (3)	3.019 (2)	179
C20—H20*A*···Cl1^{i}	0.93	2.89	3.650 (3)	140
C5—H5*B*···Cl1^{ii}	0.97	2.83	3.707 (3)	152

Symmetry codes: (i) −*x*+1, −*y*+3, −*z*; (ii) −*x*+1, *y*−1/2, −*z*+1/2.