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N,*N*′-Bis(3-nitrophenyl)isophthalamide tetrabutylammonium chloride

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Key indicators

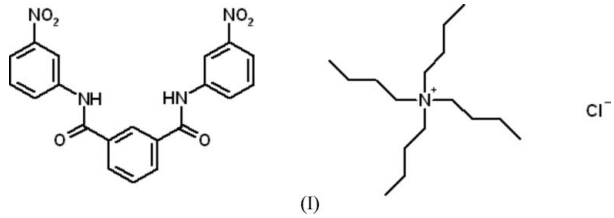
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.054
wR factor = 0.149
Data-to-parameter ratio = 18.6

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

The receptor of the title compound, $C_{14}H_{36}N^+\cdot Cl^-\cdot C_{20}H_{14}N_4O_6$, binds a chloride anion *via* two N—H···Cl hydrogen bonds [N···Cl = 3.2367 (14) Å and 3.3239 (15)°].

Comment

This work forms part of an ongoing study on the conformational properties of the anion complexes of isophthalamides and their derivatives.



(I)

The receptor in the title chloride complex, (I), adopts a similar conformation to that of a bromide–isophthalamide complex reported by Kavallieratos *et al.* (1997). In both cases, the anion lies above the least-squares plane through the central aromatic ring. In the case of the chloride complex, the angle between the plane through the central aromatic ring and a plane defined by the anion and the amide H atoms is 45.54 (4)°, whilst for the larger bromide anion the angle was found to be 63.63 (6)°. The larger size of the bromide anion is also evident in the hydrogen-bond donor–acceptor distances, which were found to be 3.634 (4) and 3.436 (4) Å for the two H···Br interactions, and are 3.3239 (15) and 3.2367 (14) Å for the H···Cl interactions in the structure reported here (Table 1).

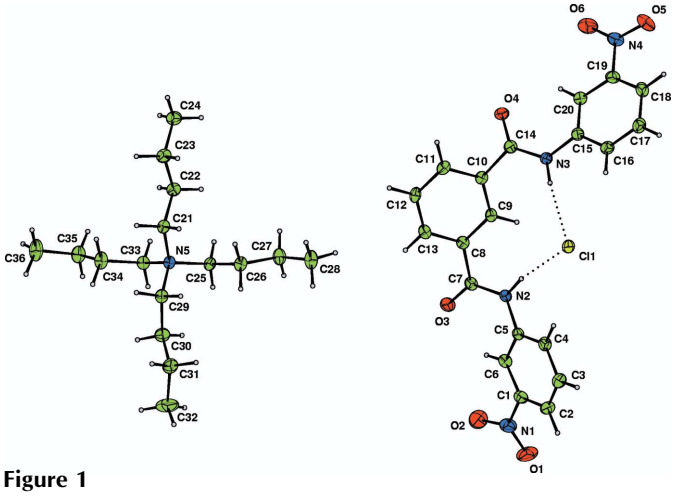


Figure 1
View of the asymmetric unit of (I), showing the atom labelling and the hydrogen-bonded chloride anion. Displacement ellipsoids are drawn at the 50% probability level and hydrogen bonds are shown as dashed lines.

It is interesting to note that the current chloride structure and the previous bromide structure form discrete 1:1 receptor–anion units, whilst the fluoride complex of a similar compound reported by Coles *et al.* (2003) forms a double helix with a 2:2 receptor-to-anion stoichiometry. This double unit is also present in the fluoride complex of a 1,3-diamidoanthraquinone (a 'twisted' isophthalamide analogue) reported by Brooks *et al.* (2005)

Experimental

The title compound was prepared as reported previously by Moore *et al.* (1997) and Coles *et al.* (2003) Crystals were obtained by slow evaporation of a solution of the receptor in the presence of excess tetrabutylammonium chloride.

Crystal data

$C_{16}H_{36}N^+\cdot Cl^-\cdot C_{20}H_{14}N_4O_6$
M_r = 684.26
Monoclinic, $P2_1/n$
a = 11.6508 (2) Å
b = 26.0390 (4) Å
c = 12.0569 (2) Å
β = 96.753 (1)°
V = 3632.39 (10) Å^3
Z = 4
D_x = 1.251 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24972 reflections
θ = 2.9–27.5°
μ = 0.16 mm^{-1}
T = 120 (2) K
Slab, colourless
0.60 × 0.60 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.902, T_{max} = 0.985
20923 measured reflections
8136 independent reflections
6851 reflections with $I > 2\sigma(I)$
R_{int} = 0.078
θ_{max} = 27.5°
$h = -15 \rightarrow 14$
$k = -31 \rightarrow 33$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.149
S = 0.99
8136 reflections
438 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0795P)^2 + 1.9526P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0057 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···Cl1	0.88	2.38	3.2367 (14)	163
N3—H3A···Cl1	0.88	2.46	3.3239 (15)	166

All H atoms were positioned with ideal geometry and allowed to ride on their parent atoms, with C—H = 0.95 (aromatic), 0.96 (methylene), 0.98 (methyl) and 0.88 Å (N—H), and with U_{iso}(H) = 1.2U_{eq}(aromatic, methylene and NH H atoms) or 1.5U_{eq}(methyl C).

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); 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).

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References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Brooks, S. J., Evans, L. S., Gale, P. A., Hursthouse, M. B. & Light, M. E. (2005). *Chem. Commun.* pp. 734–736.
Coles, S. J., Frey, G. J., Gale, P. A., Hursthouse, M. B., Light, M. E., Navakhun, K. & Thomas, G. L. (2003). *Chem. Commun.* pp. 568–569.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Kavallieratos, K., de Gale, S. R., Austin, D. J. & Crabtree, R. H. (1997). *J. Am. Chem. Soc.* **119**, 2325–2326.
Moore, J. A. & Kaur, S. (1997). *Macromolecules*, **30**, 3427–3438.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Watkin, D. M., Pearce, L. & Prout, C. K. (1993). *CAMERON*. University of Oxford, England.

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$\beta = 96.753\ (1)°$
$V = 3632.39\ (10)$ Å^3
$Z = 4$
$F(000) = 1464$
$D_x = 1.251$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 24972 reflections
$\theta = 2.9–27.5°$
$\mu = 0.16$ mm^{-1}
$T = 120$ K
Rod, colourless
0.60 × 0.60 × 0.10 mm

Data collection

Bruker–Nonius CCD camera on κ-goniostat diffractometer
Radiation source: Bruker Nonius FR591 Rotating Anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SORTAV; Blessing, 1995)
$T_{min} = 0.902$, $T_{max} = 0.985$
20923 measured reflections
8136 independent reflections
6851 reflections with $I > 2\sigma(I)$
$R_{int} = 0.078$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.9°$
$h = -15\rightarrow14$
$k = -31\rightarrow33$
$l = -14\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.149$
$S = 0.99$
8136 reflections
438 parameters
288 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
$w = 1/[\sigma^2(F_o^2) + (0.0795P)^2 + 1.9526P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.004$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0057 (16)

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.
All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.95 Å (Caromatic), 0.96 (Cmethylene), 0.98 (methyl) and 0.88 (N—H) with U_{iso}(H) = 1.2Ueq(Caromatic, Cmethylene and N—H) or 1.5Ueq(Cmethyl).
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	0.84106 (3)	0.239970 (16)	0.68422 (3)	0.02238 (13)
C1	0.66025 (16)	0.45752 (7)	0.71558 (14)	0.0243 (4)
C2	0.77063 (17)	0.46541 (7)	0.68776 (15)	0.0281 (4)
H2	0.7949	0.4982	0.6649	0.034*
C3	0.84463 (17)	0.42344 (8)	0.69460 (16)	0.0291 (4)
H3	0.9217	0.4276	0.6778	0.035*
C4	0.80711 (15)	0.37543 (7)	0.72582 (14)	0.0240 (4)
H4	0.8584	0.3470	0.7289	0.029*
C5	0.69425 (14)	0.36863 (6)	0.75282 (13)	0.0196 (3)
C6	0.61969 (15)	0.41058 (7)	0.74947 (14)	0.0220 (3)
H6	0.5437	0.4072	0.7697	0.026*
C7	0.57944 (14)	0.30687 (7)	0.84893 (14)	0.0212 (3)
C8	0.57061 (14)	0.25100 (6)	0.87738 (14)	0.0189 (3)
C9	0.60390 (13)	0.21178 (6)	0.80876 (13)	0.0188 (3)
H9	0.6298	0.2199	0.7390	0.023*
C10	0.59913 (13)	0.16064 (6)	0.84253 (14)	0.0192 (3)
C11	0.56073 (14)	0.14881 (7)	0.94520 (14)	0.0217 (3)
H11	0.5588	0.1141	0.9691	0.026*
C12	0.52536 (14)	0.18790 (7)	1.01235 (14)	0.0222 (3)
H12	0.4987	0.1798	1.0817	0.027*
C13	0.52900 (14)	0.23845 (7)	0.97825 (14)	0.0212 (3)
H13	0.5031	0.2649	1.0236	0.025*
C14	0.63103 (15)	0.11653 (7)	0.77184 (14)	0.0217 (3)
C15	0.75027 (15)	0.09243 (6)	0.62296 (14)	0.0211 (3)
C16	0.85457 (15)	0.10464 (7)	0.58215 (15)	0.0240 (4)
H16	0.8934	0.1357	0.6045	0.029*
C17	0.90159 (17)	0.07147 (7)	0.50890 (16)	0.0297 (4)
H17	0.9725	0.0800	0.4818	0.036*
C18	0.84591 (19)	0.02603 (7)	0.47510 (16)	0.0313 (4)
H18	0.8779	0.0031	0.4258	0.038*
C19	0.74235 (18)	0.01530 (7)	0.51560 (15)	0.0281 (4)
C20	0.69181 (16)	0.04725 (7)	0.58902 (15)	0.0245 (4)
H20	0.6204	0.0386	0.6149	0.029*

N1	0.57972 (15)	0.50147 (6)	0.70908 (14)	0.0312 (4)
N2	0.66191 (12)	0.31854 (5)	0.78099 (12)	0.0202 (3)
H2A	0.6976	0.2927	0.7526	0.024*
N3	0.70663 (12)	0.12787 (5)	0.69629 (12)	0.0208 (3)
H3A	0.7300	0.1599	0.6932	0.025*
N4	0.68118 (18)	−0.03238 (6)	0.47957 (15)	0.0387 (4)
O1	0.60268 (15)	0.53933 (6)	0.65573 (15)	0.0474 (4)
O2	0.49396 (14)	0.49842 (6)	0.75905 (15)	0.0458 (4)
O3	0.51966 (12)	0.33909 (5)	0.88865 (12)	0.0320 (3)
O4	0.59087 (13)	0.07377 (5)	0.78344 (12)	0.0328 (3)
O5	0.7175 (2)	−0.05704 (7)	0.40400 (17)	0.0684 (6)
O6	0.59797 (16)	−0.04552 (6)	0.52535 (15)	0.0501 (4)
C21	0.22245 (14)	0.21926 (6)	0.88104 (13)	0.0190 (3)
H21A	0.2108	0.2205	0.9610	0.023*
H21B	0.3066	0.2217	0.8766	0.023*
C22	0.16391 (15)	0.26590 (6)	0.82291 (14)	0.0221 (3)
H22A	0.0791	0.2631	0.8226	0.027*
H22B	0.1810	0.2671	0.7444	0.027*
C23	0.20702 (16)	0.31501 (7)	0.88303 (15)	0.0252 (4)
H23A	0.1891	0.3137	0.9612	0.030*
H23B	0.2920	0.3172	0.8845	0.030*
C24	0.15184 (17)	0.36281 (7)	0.82650 (16)	0.0289 (4)
H24A	0.1696	0.3643	0.7491	0.043*
H24B	0.1826	0.3935	0.8668	0.043*
H24C	0.0679	0.3614	0.8273	0.043*
C25	0.18847 (14)	0.16516 (7)	0.70861 (13)	0.0192 (3)
H25A	0.1375	0.1920	0.6714	0.023*
H25B	0.1589	0.1315	0.6798	0.023*
C26	0.31027 (14)	0.17269 (7)	0.67593 (14)	0.0219 (3)
H26A	0.3472	0.2025	0.7168	0.026*
H26B	0.3576	0.1419	0.6972	0.026*
C27	0.30582 (16)	0.18169 (8)	0.55045 (14)	0.0263 (4)
H27A	0.2627	0.1532	0.5101	0.032*
H27B	0.2634	0.2140	0.5306	0.032*
C28	0.42588 (17)	0.18511 (9)	0.51296 (16)	0.0343 (5)
H28A	0.4707	0.2117	0.5564	0.051*
H28B	0.4191	0.1939	0.4334	0.051*
H28C	0.4651	0.1519	0.5249	0.051*
C29	0.25648 (14)	0.12629 (7)	0.89477 (14)	0.0205 (3)
H29A	0.3365	0.1314	0.8769	0.025*
H29B	0.2571	0.1317	0.9761	0.025*
C30	0.22193 (16)	0.07086 (7)	0.86828 (16)	0.0262 (4)
H30A	0.1911	0.0679	0.7884	0.031*
H30B	0.1601	0.0605	0.9135	0.031*
C31	0.32561 (17)	0.03500 (8)	0.89341 (17)	0.0316 (4)
H31A	0.3840	0.0434	0.8429	0.038*
H31B	0.3612	0.0408	0.9711	0.038*
C32	0.2923 (2)	−0.02112 (9)	0.8791 (2)	0.0516 (6)

H32A	0.2386	−0.0303	0.9327	0.077*
H32B	0.3618	−0.0425	0.8923	0.077*
H32C	0.2549	−0.0269	0.8030	0.077*
C33	0.05375 (13)	0.15878 (7)	0.84952 (13)	0.0186 (3)
H33A	0.0296	0.1248	0.8179	0.022*
H33B	0.0067	0.1852	0.8059	0.022*
C34	0.02629 (15)	0.16085 (8)	0.97008 (15)	0.0266 (4)
H34A	0.0601	0.1923	1.0066	0.032*
H34B	0.0610	0.1308	1.0117	0.032*
C35	−0.10470 (15)	0.16081 (8)	0.97318 (15)	0.0275 (4)
H35A	−0.1392	0.1900	0.9283	0.033*
H35B	−0.1376	0.1287	0.9388	0.033*
C36	−0.13702 (17)	0.16509 (10)	1.09185 (17)	0.0383 (5)
H36A	−0.1110	0.1343	1.1342	0.058*
H36B	−0.2211	0.1683	1.0893	0.058*
H36C	−0.0998	0.1954	1.1282	0.058*
N5	0.17984 (11)	0.16721 (5)	0.83367 (11)	0.0175 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0200 (2)	0.0217 (2)	0.0268 (2)	0.00065 (15)	0.00836 (15)	0.00045 (15)
C1	0.0297 (9)	0.0193 (8)	0.0231 (8)	0.0020 (7)	0.0001 (7)	−0.0013 (6)
C2	0.0354 (10)	0.0213 (8)	0.0280 (9)	−0.0039 (7)	0.0056 (7)	0.0019 (7)
C3	0.0276 (9)	0.0286 (9)	0.0325 (9)	−0.0043 (7)	0.0095 (7)	0.0003 (7)
C4	0.0240 (8)	0.0235 (9)	0.0252 (8)	0.0028 (7)	0.0062 (7)	0.0011 (7)
C5	0.0224 (8)	0.0189 (8)	0.0173 (7)	−0.0004 (6)	0.0017 (6)	−0.0014 (6)
C6	0.0213 (8)	0.0212 (8)	0.0230 (8)	0.0011 (7)	0.0008 (6)	−0.0018 (6)
C7	0.0172 (7)	0.0211 (8)	0.0257 (8)	0.0004 (6)	0.0039 (6)	−0.0031 (6)
C8	0.0123 (7)	0.0211 (8)	0.0232 (8)	−0.0006 (6)	0.0022 (6)	−0.0023 (6)
C9	0.0143 (7)	0.0217 (8)	0.0210 (7)	−0.0003 (6)	0.0045 (6)	−0.0013 (6)
C10	0.0126 (7)	0.0215 (8)	0.0239 (8)	−0.0005 (6)	0.0037 (6)	−0.0004 (6)
C11	0.0162 (7)	0.0238 (8)	0.0251 (8)	0.0003 (6)	0.0029 (6)	0.0037 (6)
C12	0.0166 (7)	0.0302 (9)	0.0202 (7)	−0.0019 (7)	0.0038 (6)	0.0006 (7)
C13	0.0135 (7)	0.0269 (9)	0.0237 (8)	−0.0022 (6)	0.0034 (6)	−0.0058 (6)
C14	0.0201 (8)	0.0200 (8)	0.0257 (8)	0.0024 (6)	0.0052 (6)	0.0004 (6)
C15	0.0244 (8)	0.0183 (8)	0.0211 (7)	0.0039 (6)	0.0046 (6)	0.0024 (6)
C16	0.0249 (8)	0.0207 (8)	0.0272 (8)	0.0028 (7)	0.0058 (7)	0.0034 (7)
C17	0.0320 (9)	0.0271 (9)	0.0324 (9)	0.0074 (8)	0.0144 (8)	0.0049 (7)
C18	0.0437 (11)	0.0244 (9)	0.0280 (9)	0.0099 (8)	0.0143 (8)	0.0018 (7)
C19	0.0412 (10)	0.0184 (8)	0.0253 (8)	0.0016 (7)	0.0054 (7)	0.0003 (7)
C20	0.0291 (9)	0.0198 (8)	0.0254 (8)	−0.0004 (7)	0.0058 (7)	−0.0002 (7)
N1	0.0369 (9)	0.0227 (8)	0.0331 (8)	0.0051 (7)	0.0003 (7)	0.0010 (6)
N2	0.0197 (7)	0.0172 (7)	0.0247 (7)	0.0017 (5)	0.0069 (5)	−0.0016 (5)
N3	0.0211 (7)	0.0174 (7)	0.0251 (7)	−0.0011 (5)	0.0075 (6)	−0.0010 (5)
N4	0.0596 (12)	0.0230 (8)	0.0346 (9)	−0.0039 (8)	0.0098 (8)	−0.0068 (7)
O1	0.0552 (10)	0.0256 (8)	0.0617 (10)	0.0064 (7)	0.0088 (8)	0.0160 (7)
O2	0.0430 (9)	0.0374 (9)	0.0597 (10)	0.0161 (7)	0.0179 (8)	0.0103 (7)

O3	0.0300 (7)	0.0228 (7)	0.0466 (8)	0.0043 (6)	0.0191 (6)	−0.0027 (6)
O4	0.0383 (8)	0.0203 (7)	0.0438 (8)	−0.0041 (6)	0.0213 (6)	−0.0020 (6)
O5	0.1113 (17)	0.0391 (10)	0.0619 (12)	−0.0173 (11)	0.0402 (12)	−0.0280 (9)
O6	0.0586 (11)	0.0344 (9)	0.0589 (10)	−0.0178 (8)	0.0135 (9)	−0.0135 (7)
C21	0.0178 (7)	0.0209 (8)	0.0178 (7)	−0.0026 (6)	−0.0007 (6)	−0.0032 (6)
C22	0.0205 (8)	0.0202 (8)	0.0250 (8)	−0.0010 (6)	0.0002 (6)	−0.0019 (6)
C23	0.0249 (8)	0.0226 (9)	0.0277 (8)	−0.0035 (7)	0.0009 (7)	−0.0048 (7)
C24	0.0327 (10)	0.0228 (9)	0.0315 (9)	−0.0032 (8)	0.0046 (8)	−0.0034 (7)
C25	0.0185 (7)	0.0230 (8)	0.0156 (7)	−0.0006 (6)	0.0007 (6)	−0.0027 (6)
C26	0.0175 (8)	0.0280 (9)	0.0201 (8)	0.0005 (7)	0.0016 (6)	−0.0021 (6)
C27	0.0230 (8)	0.0356 (10)	0.0204 (8)	−0.0018 (7)	0.0031 (7)	−0.0003 (7)
C28	0.0285 (10)	0.0484 (12)	0.0277 (9)	−0.0010 (9)	0.0101 (8)	0.0010 (9)
C29	0.0163 (7)	0.0236 (8)	0.0211 (7)	0.0030 (6)	−0.0004 (6)	0.0019 (6)
C30	0.0232 (8)	0.0241 (9)	0.0310 (9)	0.0027 (7)	0.0018 (7)	0.0001 (7)
C31	0.0309 (10)	0.0312 (10)	0.0330 (10)	0.0114 (8)	0.0050 (8)	0.0034 (8)
C32	0.0627 (16)	0.0269 (11)	0.0639 (15)	0.0154 (11)	0.0012 (13)	0.0013 (10)
C33	0.0126 (7)	0.0233 (8)	0.0195 (7)	−0.0004 (6)	−0.0003 (6)	0.0000 (6)
C34	0.0171 (8)	0.0409 (10)	0.0218 (8)	0.0019 (7)	0.0014 (6)	0.0013 (7)
C35	0.0169 (8)	0.0383 (10)	0.0276 (9)	0.0001 (7)	0.0034 (7)	−0.0006 (7)
C36	0.0220 (9)	0.0644 (15)	0.0298 (10)	0.0037 (9)	0.0076 (8)	0.0037 (9)
N5	0.0146 (6)	0.0204 (7)	0.0172 (6)	0.0002 (5)	0.0004 (5)	−0.0006 (5)

Geometric parameters (Å, º)

C1—C2	1.382 (3)	C21—H21B	0.9900
C1—C6	1.389 (2)	C22—C23	1.525 (2)
C1—N1	1.476 (2)	C22—H22A	0.9900
C2—C3	1.389 (3)	C22—H22B	0.9900
C2—H2	0.9500	C23—C24	1.524 (3)
C3—C4	1.391 (3)	C23—H23A	0.9900
C3—H3	0.9500	C23—H23B	0.9900
C4—C5	1.402 (2)	C24—H24A	0.9800
C4—H4	0.9500	C24—H24B	0.9800
C5—C6	1.393 (2)	C24—H24C	0.9800
C5—N2	1.410 (2)	C25—N5	1.5239 (19)
C6—H6	0.9500	C25—C26	1.529 (2)
C7—O3	1.223 (2)	C25—H25A	0.9900
C7—N2	1.368 (2)	C25—H25B	0.9900
C7—C8	1.501 (2)	C26—C27	1.526 (2)
C8—C9	1.398 (2)	C26—H26A	0.9900
C8—C13	1.400 (2)	C26—H26B	0.9900
C9—C10	1.395 (2)	C27—C28	1.522 (2)
C9—H9	0.9500	C27—H27A	0.9900
C10—C11	1.399 (2)	C27—H27B	0.9900
C10—C14	1.503 (2)	C28—H28A	0.9800
C11—C12	1.393 (2)	C28—H28B	0.9800
C11—H11	0.9500	C28—H28C	0.9800
C12—C13	1.381 (3)	C29—C30	1.522 (2)

C12—H12	0.9500	C29—N5	1.523 (2)
C13—H13	0.9500	C29—H29A	0.9900
C14—O4	1.222 (2)	C29—H29B	0.9900
C14—N3	1.372 (2)	C30—C31	1.529 (3)
C15—C20	1.396 (2)	C30—H30A	0.9900
C15—C16	1.400 (2)	C30—H30B	0.9900
C15—N3	1.413 (2)	C31—C32	1.517 (3)
C16—C17	1.393 (2)	C31—H31A	0.9900
C16—H16	0.9500	C31—H31B	0.9900
C17—C18	1.387 (3)	C32—H32A	0.9800
C17—H17	0.9500	C32—H32B	0.9800
C18—C19	1.382 (3)	C32—H32C	0.9800
C18—H18	0.9500	C33—N5	1.520 (2)
C19—C20	1.395 (2)	C33—C34	1.526 (2)
C19—N4	1.472 (3)	C33—H33A	0.9900
C20—H20	0.9500	C33—H33B	0.9900
N1—O1	1.224 (2)	C34—C35	1.531 (2)
N1—O2	1.228 (2)	C34—H34A	0.9900
N2—H2A	0.8800	C34—H34B	0.9900
N3—H3A	0.8800	C35—C36	1.526 (3)
N4—O6	1.220 (2)	C35—H35A	0.9900
N4—O5	1.230 (2)	C35—H35B	0.9900
C21—C22	1.523 (2)	C36—H36A	0.9800
C21—N5	1.531 (2)	C36—H36B	0.9800
C21—H21A	0.9900	C36—H36C	0.9800

C2—C1—C6	124.10 (17)	C24—C23—H23B	109.2
C2—C1—N1	118.40 (16)	C22—C23—H23B	109.2
C6—C1—N1	117.50 (16)	H23A—C23—H23B	107.9
C1—C2—C3	117.22 (17)	C23—C24—H24A	109.5
C1—C2—H2	121.4	C23—C24—H24B	109.5
C3—C2—H2	121.4	H24A—C24—H24B	109.5
C2—C3—C4	120.73 (17)	C23—C24—H24C	109.5
C2—C3—H3	119.6	H24A—C24—H24C	109.5
C4—C3—H3	119.6	H24B—C24—H24C	109.5
C3—C4—C5	120.62 (17)	N5—C25—C26	114.93 (13)
C3—C4—H4	119.7	N5—C25—H25A	108.5
C5—C4—H4	119.7	C26—C25—H25A	108.5
C6—C5—C4	119.52 (16)	N5—C25—H25B	108.5
C6—C5—N2	123.28 (15)	C26—C25—H25B	108.5
C4—C5—N2	117.19 (15)	H25A—C25—H25B	107.5
C1—C6—C5	117.76 (16)	C27—C26—C25	110.53 (14)
C1—C6—H6	121.1	C27—C26—H26A	109.5
C5—C6—H6	121.1	C25—C26—H26A	109.5
O3—C7—N2	123.66 (16)	C27—C26—H26B	109.5
O3—C7—C8	121.18 (15)	C25—C26—H26B	109.5
N2—C7—C8	115.08 (14)	H26A—C26—H26B	108.1
C9—C8—C13	119.49 (16)	C28—C27—C26	112.22 (15)

C9—C8—C7	122.80 (15)	C28—C27—H27A	109.2
C13—C8—C7	117.70 (15)	C26—C27—H27A	109.2
C10—C9—C8	120.08 (15)	C28—C27—H27B	109.2
C10—C9—H9	120.0	C26—C27—H27B	109.2
C8—C9—H9	120.0	H27A—C27—H27B	107.9
C9—C10—C11	119.75 (15)	C27—C28—H28A	109.5
C9—C10—C14	122.87 (15)	C27—C28—H28B	109.5
C11—C10—C14	117.37 (15)	H28A—C28—H28B	109.5
C12—C11—C10	120.04 (16)	C27—C28—H28C	109.5
C12—C11—H11	120.0	H28A—C28—H28C	109.5
C10—C11—H11	120.0	H28B—C28—H28C	109.5
C13—C12—C11	120.13 (15)	C30—C29—N5	115.87 (13)
C13—C12—H12	119.9	C30—C29—H29A	108.3
C11—C12—H12	119.9	N5—C29—H29A	108.3
C12—C13—C8	120.46 (16)	C30—C29—H29B	108.3
C12—C13—H13	119.8	N5—C29—H29B	108.3
C8—C13—H13	119.8	H29A—C29—H29B	107.4
O4—C14—N3	123.81 (16)	C29—C30—C31	110.76 (15)
O4—C14—C10	120.52 (15)	C29—C30—H30A	109.5
N3—C14—C10	115.67 (15)	C31—C30—H30A	109.5
C20—C15—C16	120.12 (16)	C29—C30—H30B	109.5
C20—C15—N3	122.31 (15)	C31—C30—H30B	109.5
C16—C15—N3	117.55 (15)	H30A—C30—H30B	108.1
C17—C16—C15	120.29 (17)	C32—C31—C30	112.35 (18)
C17—C16—H16	119.9	C32—C31—H31A	109.1
C15—C16—H16	119.9	C30—C31—H31A	109.1
C18—C17—C16	120.66 (17)	C32—C31—H31B	109.1
C18—C17—H17	119.7	C30—C31—H31B	109.1
C16—C17—H17	119.7	H31A—C31—H31B	107.9
C19—C18—C17	117.77 (17)	C31—C32—H32A	109.5
C19—C18—H18	121.1	C31—C32—H32B	109.5
C17—C18—H18	121.1	H32A—C32—H32B	109.5
C18—C19—C20	123.70 (18)	C31—C32—H32C	109.5
C18—C19—N4	118.60 (17)	H32A—C32—H32C	109.5
C20—C19—N4	117.69 (17)	H32B—C32—H32C	109.5
C19—C20—C15	117.44 (17)	N5—C33—C34	115.40 (13)
C19—C20—H20	121.3	N5—C33—H33A	108.4
C15—C20—H20	121.3	C34—C33—H33A	108.4
O1—N1—O2	123.41 (17)	N5—C33—H33B	108.4
O1—N1—C1	118.40 (17)	C34—C33—H33B	108.4
O2—N1—C1	118.18 (16)	H33A—C33—H33B	107.5
C7—N2—C5	125.17 (14)	C33—C34—C35	110.15 (14)
C7—N2—H2A	117.4	C33—C34—H34A	109.6
C5—N2—H2A	117.4	C35—C34—H34A	109.6
C14—N3—C15	125.54 (14)	C33—C34—H34B	109.6
C14—N3—H3A	117.2	C35—C34—H34B	109.6
C15—N3—H3A	117.2	H34A—C34—H34B	108.1
O6—N4—O5	123.38 (19)	C36—C35—C34	112.30 (15)

O6—N4—C19	119.10 (16)	C36—C35—H35A	109.1
O5—N4—C19	117.52 (19)	C34—C35—H35A	109.1
C22—C21—N5	115.24 (13)	C36—C35—H35B	109.1
C22—C21—H21A	108.5	C34—C35—H35B	109.1
N5—C21—H21A	108.5	H35A—C35—H35B	107.9
C22—C21—H21B	108.5	C35—C36—H36A	109.5
N5—C21—H21B	108.5	C35—C36—H36B	109.5
H21A—C21—H21B	107.5	H36A—C36—H36B	109.5
C21—C22—C23	110.19 (14)	C35—C36—H36C	109.5
C21—C22—H22A	109.6	H36A—C36—H36C	109.5
C23—C22—H22A	109.6	H36B—C36—H36C	109.5
C21—C22—H22B	109.6	C33—N5—C29	111.01 (12)
C23—C22—H22B	109.6	C33—N5—C25	107.26 (12)
H22A—C22—H22B	108.1	C29—N5—C25	110.54 (12)
C24—C23—C22	111.96 (15)	C33—N5—C21	110.91 (12)
C24—C23—H23A	109.2	C29—N5—C21	107.03 (12)
C22—C23—H23A	109.2	C25—N5—C21	110.12 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···Cl1	0.88	2.38	3.2367 (14)	163
N3—H3*A*···Cl1	0.88	2.46	3.3239 (15)	166