Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(3-{4-[(2,4-Dinitroanilino)methyl]-phenoxy}propyl)piperidinium chloride

Holger Stark,[a] Michael Amon[a] and Michael Bolte[b]*

[a]Institut für Pharmazeutische Chemie, Johann Wolfgang Goethe-Universität Frankfurt, ZAFES/CMP, Max-von-Laue-Strasse 9, 60438 Frankfurt/Main, Germany, and [b]Institut für Anorganische Chemie, Johann Wolfgang Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany
Correspondence e-mail: bolte@chemie.uni-frankfurt.de

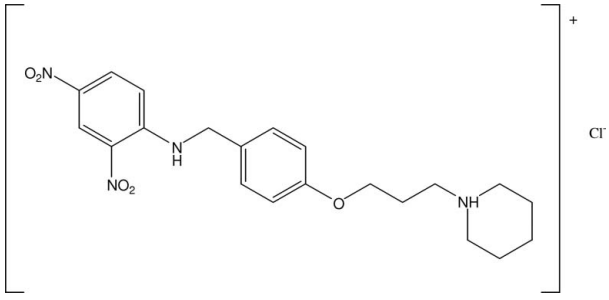
Received 10 September 2007; accepted 10 September 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; R factor = 0.072; wR factor = 0.128; data-to-parameter ratio = 14.8.

The title compound, $C_{21}H_{27}N_4O_5^+\cdot Cl^-$, is one of the parent compounds for a recently developed series of novel potent histamine H_3 receptor antagonists having additional fluorescent properties. The crystal structure is composed of discrete cations and anions connected by classical N—H···O and N—H···Cl hydrogen bonds. The crystal packing is further stabilized by weak C—H···Cl and C—H···O contacts.

Related literature

For related literature, see: Amon *et al.* (2007); Celanire *et al.* (2007).



Experimental

Crystal data

$C_{21}H_{27}N_4O_5^+\cdot Cl^-$
M_r = 450.92
Monoclinic, $P2_1/c$
a = 19.4030 (15) Å
b = 7.2300 (5) Å
c = 17.2830 (13) Å
β = 110.583 (6)°
V = 2269.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 173 (2) K
0.27 × 0.12 × 0.11 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan [*MULABS* (Spek, 2003; Blessing, 1995)]
T_{min} = 0.946, T_{max} = 0.968
27016 measured reflections
4261 independent reflections
2776 reflections with $I > 2\sigma(I)$
R_{int} = 0.074

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.128
S = 1.07
4261 reflections
288 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N41—H41···Cl1	0.97 (4)	2.14 (4)	3.109 (3)	175 (3)
N1—H1···O122	0.84 (3)	2.03 (3)	2.665 (4)	131 (3)
N1—H1···O122^{i}	0.84 (3)	2.22 (3)	2.971 (4)	149 (3)
C4—H4B···Cl1^{ii}	0.99	2.72	3.664 (3)	161
C23—H23···O141^{iii}	0.95	2.37	3.318 (4)	173
C42—H42B···Cl1^{iv}	0.99	2.73	3.514 (3)	137

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x+1, -y, -z+1$; (iii) $x, -y+\frac{5}{2}, z-\frac{1}{2}$; (iv) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA* (Stoe & Cie, 2001); data reduction: *X-AREA* (Stoe & Cie, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2536).

References

Amon, M., Ligneau, X., Camelin, J.-C., Berrebi-Bertrand, I., Schwartz, J.-C. & Stark, H. (2007). *ChemMedChem*, **2**, 708–716.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Celanire, S., Lebon, F. & Stark, H. (2007). *Drug Discovery: From Hits to Clinical Candidates*. In *The Third Histamine Receptor: Selective Ligands as Potential Therapeutic Agents in CNS Disorders*, edited by D. S. Vohora. Boca Raton: Taylor & Francis CRC Press Inc. In the press.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.

  doi:10.1107/S1600536807044157

supplementary materials

Acta Cryst. (2007). E**63**, o4074 [**doi:10.1107/S1600536807044157**]

1-(3-{4-[(2,4-Dinitroanilino)methyl]phenoxy}propyl)piperidinium chloride

H. Stark, M. Amon and M. Bolte

Comment

Histamine H_3 receptors are important targets for different central nervous system disorders (Celanire *et al.*, 2007). Within a recently developed series of novel potent histamine H_3 receptor antagonists having additional fluorescent properties the title compound can be taken as one of the parent molecules (Amon *et al.*, 2007). The compound showed subnanomolar affinity at human histamine H_3 receptors (K_i value of 0.6nM) and a good Stokes shift of 100 nm.

The structure of the title compound, $C_{21}H_{27}N_4O_5^+ Cl^-$, (I), is composed of discrete cations and anions. The bond lengths and angles in (I) are in the usual ranges. The piperidine ring adopts a chair conformation with the amino H atom in an axial position. Whereas one of the methylene C—C single bonds is in an antiperiplanar conformation [C2—C3—C4—N41 = 172.9 (3)°], the other one adopts a synclinal conformation [O1—C2—C3—C4 = 68.3 (4)°]. Both nitro groups are slightly tilted with respect to the aromatic ring to which they are attached: the dihedral angles between the planes of the nitro groups and the aromatic ring are 11.1 (5)° and 11.5 (6)° for the nitro groups N12, O121, O122 and N14, O141, O142, respectively. The dihedral angle between the two aromatic rings is 64.20 (11)°.

The amino H atom of the piperidine ring forms a classical N—H···Cl hydrogen bond (Table 1). The other amino H atom forms a bifurcated hydrogen bond. There is an intramolecular contact to one of the nitro O atoms of the aromatic ring attached to the amino group and there is an intermolecular contact to a symmetry equivalent of the same nitro O atom. As a result, a centrosymmetric dimer is formed (Fig. 2). In addition to these classical hydrogen bonds, the crystal packing is further stabilized by weak C—H···Cl and C—H···O contacts.

Experimental

The appropriate benzyl amine derivative was prepared in four steps by classical coupling reactions starting from piperidine and 3-chloropropanol. Alcohol chlorination, ether formation with 4-cyanophenol, and catalytic reduction led to the primary benzylamine which then was reacted with Sanger's reagent (1-fluoro-2,4-dinitrobenzen) in a nucleophilic aromatic substitution (Amon *et al.*, 2007). The reaction product was purified chromatographically over silica gel with dichloromethane/methanol (95:5) and NH_3 gas. Yellow blocks of (I) were obtained from a mixture of ethanol/diethyl ether (1:6 *v*/*v*) with three drops of HCl at approximately 280 K.

Refinement

The H atoms bonded to C were geometrically placed (C—H = 0.95–0.99 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$. The H atoms bonded to N were located in difference maps and their positions and U_{iso} values were freely refined.

Figures

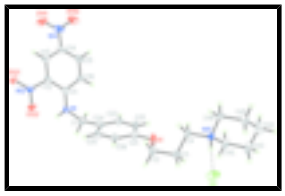


Fig. 1. Perspective view of (I) with displacement ellipsoids shown at the 50% probability level (arbitrary spheres for the hydrogen atoms).

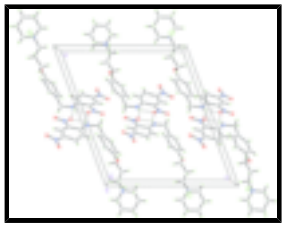


Fig. 2. Packing diagram of (I) viewed onto the *ac* plane. The classical hydrogen bonds shown as dashed lines.

1-(3-{4-[(2,4-Dinitroanilino)methyl]phenoxy}propyl)piperidinium chloride

Crystal data

$C_{21}H_{27}N_4O_5^+ \cdot Cl^-$	F_{000} = 952
M_r = 450.92	D_x = 1.320 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 9924 reflections
a = 19.4030 (15) Å	θ = 3.5–25.6º
b = 7.2300 (5) Å	μ = 0.21 mm^{-1}
c = 17.2830 (13) Å	T = 173 (2) K
β = 110.583 (6)º	Block, yellow
V = 2269.8 (3) Å^3	0.27 × 0.12 × 0.11 mm
Z = 4	

Data collection

Stoe IPDS II two-circle diffractometer	4261 independent reflections
Radiation source: fine-focus sealed tube	2776 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.074
T = 173(2) K	θ_{max} = 25.7º
ω scans	θ_{min} = 3.6º
Absorption correction: multi-scan [MULABS (Spek, 2003; Blessing, 1995)]	h = −23→23
T_{min} = 0.946, T_{max} = 0.968	k = −8→8
27016 measured reflections	l = −21→21

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.072$

$wR(F^2) = 0.128$

$S = 1.07$

4261 reflections

288 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: difmap and geom

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.26$ e Å^{-3}

$\Delta\rho_{min} = -0.24$ e Å^{-3}

Extinction correction: none

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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.06439 (14)	0.7518 (4)	0.45674 (16)	0.0220 (6)
H1	0.0424 (17)	0.661 (5)	0.4681 (19)	0.012 (8)*
C1	0.06952 (17)	0.7550 (4)	0.37402 (19)	0.0234 (7)
H1A	0.0679	0.8852	0.3557	0.028*
H1B	0.0260	0.6911	0.3351	0.028*
O1	0.32321 (13)	0.4100 (3)	0.34329 (16)	0.0323 (6)
C2	0.34155 (18)	0.2203 (5)	0.3692 (2)	0.0307 (8)
H2A	0.3002	0.1372	0.3393	0.037*
H2B	0.3519	0.2077	0.4292	0.037*
C3	0.40978 (17)	0.1709 (5)	0.3488 (2)	0.0309 (8)
H3A	0.4009	0.2000	0.2901	0.037*
H3B	0.4195	0.0366	0.3569	0.037*
C4	0.47691 (17)	0.2788 (5)	0.4039 (2)	0.0249 (7)
H4A	0.4695	0.4122	0.3906	0.030*
H4B	0.4819	0.2624	0.4624	0.030*
C11	0.09157 (16)	0.8805 (4)	0.51601 (19)	0.0193 (7)
C12	0.07709 (17)	0.8840 (4)	0.5918 (2)	0.0201 (7)
C13	0.09925 (16)	1.0306 (4)	0.64785 (19)	0.0217 (7)
H13	0.0872	1.0315	0.6966	0.026*
C14	0.13877 (19)	1.1733 (4)	0.6314 (2)	0.0282 (8)
C15	0.1599 (2)	1.1705 (5)	0.5615 (2)	0.0326 (8)
H15	0.1906	1.2654	0.5534	0.039*

C16	0.13618 (18)	1.0301 (4)	0.5051 (2)	0.0276 (7)
H16	0.1496	1.0317	0.4572	0.033*
N12	0.03606 (14)	0.7389 (3)	0.61389 (16)	0.0246 (6)
O121	0.01389 (15)	0.7619 (4)	0.67164 (15)	0.0392 (6)
O122	0.02439 (16)	0.5931 (3)	0.57364 (16)	0.0428 (7)
N14	0.15790 (19)	1.3301 (4)	0.6881 (2)	0.0417 (8)
O141	0.12909 (19)	1.3407 (4)	0.74137 (19)	0.0527 (8)
O142	0.2012 (2)	1.4472 (4)	0.6795 (2)	0.0675 (10)
C21	0.26258 (17)	0.4855 (5)	0.3546 (2)	0.0243 (7)
C22	0.24320 (19)	0.6651 (5)	0.3233 (2)	0.0277 (8)
H22	0.2719	0.7271	0.2968	0.033*
C23	0.18216 (17)	0.7521 (4)	0.3311 (2)	0.0247 (7)
H23	0.1699	0.8738	0.3100	0.030*
C24	0.13876 (17)	0.6646 (4)	0.36916 (19)	0.0201 (7)
C25	0.15833 (17)	0.4860 (5)	0.3999 (2)	0.0280 (7)
H25	0.1287	0.4233	0.4251	0.034*
C26	0.22036 (18)	0.3975 (4)	0.3944 (2)	0.0290 (8)
H26	0.2338	0.2780	0.4177	0.035*
N41	0.54659 (14)	0.2151 (4)	0.39226 (16)	0.0198 (5)
H41	0.5459 (19)	0.081 (5)	0.393 (2)	0.029 (9)*
C42	0.55035 (18)	0.2777 (5)	0.3111 (2)	0.0304 (8)
H42A	0.5507	0.4146	0.3093	0.036*
H42B	0.5063	0.2336	0.2655	0.036*
C43	0.6197 (2)	0.2028 (6)	0.2993 (2)	0.0397 (9)
H43A	0.6175	0.0660	0.2969	0.048*
H43B	0.6219	0.2485	0.2462	0.048*
C44	0.6888 (2)	0.2630 (6)	0.3697 (3)	0.0476 (10)
H44A	0.6941	0.3990	0.3686	0.057*
H44B	0.7326	0.2057	0.3629	0.057*
C45	0.68359 (18)	0.2042 (6)	0.4524 (2)	0.0371 (9)
H45A	0.7272	0.2508	0.4980	0.044*
H45B	0.6839	0.0674	0.4557	0.044*
C46	0.61356 (17)	0.2785 (5)	0.4630 (2)	0.0294 (7)
H46A	0.6107	0.2335	0.5159	0.035*
H46B	0.6151	0.4153	0.4648	0.035*
Cl1	0.55483 (6)	−0.21420 (11)	0.40105 (6)	0.0365 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0251 (14)	0.0192 (14)	0.0246 (14)	−0.0060 (11)	0.0125 (11)	−0.0030 (11)
C1	0.0246 (16)	0.0261 (17)	0.0199 (15)	0.0035 (13)	0.0085 (12)	0.0025 (13)
O1	0.0265 (13)	0.0330 (12)	0.0454 (16)	0.0112 (10)	0.0224 (11)	0.0127 (11)
C2	0.0217 (16)	0.0248 (16)	0.045 (2)	0.0040 (14)	0.0114 (15)	0.0001 (16)
C3	0.0214 (17)	0.0348 (19)	0.035 (2)	0.0043 (14)	0.0087 (15)	−0.0092 (15)
C4	0.0255 (16)	0.0261 (15)	0.0256 (17)	0.0028 (14)	0.0121 (13)	−0.0029 (14)
C11	0.0185 (16)	0.0155 (14)	0.0214 (16)	0.0041 (11)	0.0037 (13)	0.0018 (12)
C12	0.0190 (15)	0.0174 (15)	0.0234 (17)	−0.0002 (12)	0.0068 (13)	0.0035 (12)

C13	0.0228 (16)	0.0212 (15)	0.0184 (16)	0.0012 (12)	0.0040 (13)	−0.0023 (13)
C14	0.0346 (19)	0.0202 (16)	0.0232 (17)	−0.0062 (14)	0.0020 (15)	−0.0015 (13)
C15	0.038 (2)	0.0248 (17)	0.034 (2)	−0.0115 (15)	0.0110 (16)	0.0034 (15)
C16	0.0318 (18)	0.0276 (17)	0.0251 (18)	−0.0077 (14)	0.0120 (14)	0.0005 (14)
N12	0.0292 (14)	0.0220 (14)	0.0232 (14)	−0.0078 (12)	0.0100 (11)	−0.0048 (11)
O121	0.0519 (15)	0.0431 (15)	0.0337 (14)	−0.0181 (13)	0.0291 (12)	−0.0121 (12)
O122	0.073 (2)	0.0291 (13)	0.0383 (16)	−0.0257 (13)	0.0346 (14)	−0.0162 (12)
N14	0.056 (2)	0.0226 (16)	0.042 (2)	−0.0121 (14)	0.0116 (17)	−0.0057 (14)
O141	0.091 (2)	0.0304 (14)	0.0421 (17)	−0.0175 (14)	0.0300 (17)	−0.0156 (13)
O142	0.098 (3)	0.0437 (17)	0.069 (2)	−0.0476 (18)	0.041 (2)	−0.0229 (16)
C21	0.0229 (16)	0.0280 (16)	0.0247 (17)	0.0043 (14)	0.0114 (13)	0.0042 (14)
C22	0.0316 (18)	0.0292 (17)	0.0312 (19)	0.0018 (14)	0.0219 (15)	0.0107 (14)
C23	0.0299 (17)	0.0187 (16)	0.0285 (17)	0.0031 (13)	0.0139 (13)	0.0072 (13)
C24	0.0207 (16)	0.0207 (15)	0.0174 (16)	0.0012 (12)	0.0048 (13)	0.0013 (12)
C25	0.0231 (17)	0.0269 (16)	0.039 (2)	0.0003 (14)	0.0169 (15)	0.0106 (15)
C26	0.0255 (18)	0.0217 (16)	0.042 (2)	0.0035 (13)	0.0149 (16)	0.0112 (15)
N41	0.0222 (13)	0.0190 (12)	0.0202 (13)	0.0005 (11)	0.0099 (11)	0.0008 (11)
C42	0.0313 (18)	0.0391 (18)	0.0240 (17)	0.0090 (16)	0.0139 (14)	0.0092 (16)
C43	0.038 (2)	0.057 (2)	0.033 (2)	0.0114 (18)	0.0238 (17)	0.0091 (19)
C44	0.035 (2)	0.052 (3)	0.065 (3)	0.0037 (19)	0.0290 (19)	0.011 (2)
C45	0.0201 (17)	0.045 (2)	0.041 (2)	0.0007 (16)	0.0042 (15)	−0.0030 (18)
C46	0.0256 (17)	0.0300 (16)	0.0277 (18)	−0.0040 (14)	0.0034 (14)	−0.0081 (15)
Cl1	0.0588 (6)	0.0208 (4)	0.0265 (4)	0.0016 (4)	0.0107 (4)	0.0010 (4)

Geometric parameters (Å, °)

N1—C11	1.346 (4)	N14—O141	1.236 (4)
N1—C1	1.468 (4)	N14—O142	1.238 (4)
N1—H1	0.84 (3)	C21—C26	1.396 (5)
C1—C24	1.523 (4)	C21—C22	1.406 (5)
C1—H1A	0.9900	C22—C23	1.389 (5)
C1—H1B	0.9900	C22—H22	0.9500
O1—C21	1.371 (4)	C23—C24	1.390 (4)
O1—C2	1.448 (4)	C23—H23	0.9500
C2—C3	1.527 (5)	C24—C25	1.397 (4)
C2—H2A	0.9900	C25—C26	1.395 (5)
C2—H2B	0.9900	C25—H25	0.9500
C3—C4	1.530 (4)	C26—H26	0.9500
C3—H3A	0.9900	N41—C42	1.500 (4)
C3—H3B	0.9900	N41—C46	1.509 (4)
C4—N41	1.507 (4)	N41—H41	0.97 (4)
C4—H4A	0.9900	C42—C43	1.529 (5)
C4—H4B	0.9900	C42—H42A	0.9900
C11—C12	1.434 (5)	C42—H42B	0.9900
C11—C16	1.439 (4)	C43—C44	1.523 (6)
C12—C13	1.397 (4)	C43—H43A	0.9900
C12—N12	1.447 (4)	C43—H43B	0.9900
C13—C14	1.374 (5)	C44—C45	1.528 (6)
C13—H13	0.9500	C44—H44A	0.9900

C14—C15	1.406 (5)	C44—H44B	0.9900
C14—N14	1.459 (4)	C45—C46	1.531 (5)
C15—C16	1.370 (5)	C45—H45A	0.9900
C15—H15	0.9500	C45—H45B	0.9900
C16—H16	0.9500	C46—H46A	0.9900
N12—O121	1.229 (4)	C46—H46B	0.9900
N12—O122	1.239 (3)		
C11—N1—C1	126.0 (3)	O1—C21—C22	116.1 (3)
C11—N1—H1	117 (2)	C26—C21—C22	119.1 (3)
C1—N1—H1	117 (2)	C23—C22—C21	120.2 (3)
N1—C1—C24	114.2 (3)	C23—C22—H22	119.9
N1—C1—H1A	108.7	C21—C22—H22	119.9
C24—C1—H1A	108.7	C22—C23—C24	121.2 (3)
N1—C1—H1B	108.7	C22—C23—H23	119.4
C24—C1—H1B	108.7	C24—C23—H23	119.4
H1A—C1—H1B	107.6	C23—C24—C25	118.2 (3)
C21—O1—C2	117.6 (3)	C23—C24—C1	121.6 (3)
O1—C2—C3	106.8 (3)	C25—C24—C1	120.2 (3)
O1—C2—H2A	110.4	C26—C25—C24	121.6 (3)
C3—C2—H2A	110.4	C26—C25—H25	119.2
O1—C2—H2B	110.4	C24—C25—H25	119.2
C3—C2—H2B	110.4	C25—C26—C21	119.6 (3)
H2A—C2—H2B	108.6	C25—C26—H26	120.2
C2—C3—C4	110.8 (3)	C21—C26—H26	120.2
C2—C3—H3A	109.5	C42—N41—C4	111.9 (2)
C4—C3—H3A	109.5	C42—N41—C46	110.8 (3)
C2—C3—H3B	109.5	C4—N41—C46	110.9 (2)
C4—C3—H3B	109.5	C42—N41—H41	109 (2)
H3A—C3—H3B	108.1	C4—N41—H41	107 (2)
N41—C4—C3	111.6 (3)	C46—N41—H41	108 (2)
N41—C4—H4A	109.3	N41—C42—C43	110.6 (3)
C3—C4—H4A	109.3	N41—C42—H42A	109.5
N41—C4—H4B	109.3	C43—C42—H42A	109.5
C3—C4—H4B	109.3	N41—C42—H42B	109.5
H4A—C4—H4B	108.0	C43—C42—H42B	109.5
N1—C11—C12	123.9 (3)	H42A—C42—H42B	108.1
N1—C11—C16	120.8 (3)	C44—C43—C42	111.1 (3)
C12—C11—C16	115.3 (3)	C44—C43—H43A	109.4
C13—C12—C11	122.4 (3)	C42—C43—H43A	109.4
C13—C12—N12	115.5 (3)	C44—C43—H43B	109.4
C11—C12—N12	122.1 (3)	C42—C43—H43B	109.4
C14—C13—C12	119.0 (3)	H43A—C43—H43B	108.0
C14—C13—H13	120.5	C43—C44—C45	109.8 (3)
C12—C13—H13	120.5	C43—C44—H44A	109.7
C13—C14—C15	121.3 (3)	C45—C44—H44A	109.7
C13—C14—N14	118.2 (3)	C43—C44—H44B	109.7
C15—C14—N14	120.5 (3)	C45—C44—H44B	109.7
C16—C15—C14	119.7 (3)	H44A—C44—H44B	108.2
C16—C15—H15	120.2	C44—C45—C46	111.6 (3)

C14—C15—H15	120.2	C44—C45—H45A	109.3
C15—C16—C11	122.1 (3)	C46—C45—H45A	109.3
C15—C16—H16	119.0	C44—C45—H45B	109.3
C11—C16—H16	119.0	C46—C45—H45B	109.3
O121—N12—O122	121.6 (3)	H45A—C45—H45B	108.0
O121—N12—C12	119.9 (3)	N41—C46—C45	110.1 (3)
O122—N12—C12	118.5 (3)	N41—C46—H46A	109.6
O141—N14—O142	123.5 (3)	C45—C46—H46A	109.6
O141—N14—C14	118.3 (3)	N41—C46—H46B	109.6
O142—N14—C14	118.2 (3)	C45—C46—H46B	109.6
O1—C21—C26	124.8 (3)	H46A—C46—H46B	108.2
C11—N1—C1—C24	88.5 (4)	C15—C14—N14—O142	−9.5 (5)
C21—O1—C2—C3	179.2 (3)	C2—O1—C21—C26	4.8 (5)
O1—C2—C3—C4	68.3 (4)	C2—O1—C21—C22	−175.5 (3)
C2—C3—C4—N41	172.9 (3)	O1—C21—C22—C23	179.4 (3)
C1—N1—C11—C12	171.6 (3)	C26—C21—C22—C23	−0.9 (5)
C1—N1—C11—C16	−6.3 (5)	C21—C22—C23—C24	−0.4 (5)
N1—C11—C12—C13	−172.7 (3)	C22—C23—C24—C25	0.3 (5)
C16—C11—C12—C13	5.3 (4)	C22—C23—C24—C1	−176.8 (3)
N1—C11—C12—N12	5.0 (5)	N1—C1—C24—C23	−131.3 (3)
C16—C11—C12—N12	−177.0 (3)	N1—C1—C24—C25	51.7 (4)
C11—C12—C13—C14	−2.5 (5)	C23—C24—C25—C26	1.2 (5)
N12—C12—C13—C14	179.7 (3)	C1—C24—C25—C26	178.3 (3)
C12—C13—C14—C15	−2.9 (5)	C24—C25—C26—C21	−2.5 (5)
C12—C13—C14—N14	176.6 (3)	O1—C21—C26—C25	−178.0 (3)
C13—C14—C15—C16	5.1 (5)	C22—C21—C26—C25	2.4 (5)
N14—C14—C15—C16	−174.4 (3)	C3—C4—N41—C42	72.8 (3)
C14—C15—C16—C11	−1.9 (5)	C3—C4—N41—C46	−162.9 (3)
N1—C11—C16—C15	175.0 (3)	C4—N41—C42—C43	−177.2 (3)
C12—C11—C16—C15	−3.0 (5)	C46—N41—C42—C43	58.5 (4)
C13—C12—N12—O121	10.4 (4)	N41—C42—C43—C44	−57.5 (4)
C11—C12—N12—O121	−167.4 (3)	C42—C43—C44—C45	55.4 (4)
C13—C12—N12—O122	−169.4 (3)	C43—C44—C45—C46	−55.4 (4)
C11—C12—N12—O122	12.8 (4)	C42—N41—C46—C45	−58.0 (4)
C13—C14—N14—O141	−10.2 (5)	C4—N41—C46—C45	177.1 (3)
C15—C14—N14—O141	169.2 (3)	C44—C45—C46—N41	56.7 (4)
C13—C14—N14—O142	171.0 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N41—H41···Cl1	0.97 (4)	2.14 (4)	3.109 (3)	175 (3)
N1—H1···O122	0.84 (3)	2.03 (3)	2.665 (4)	131 (3)
N1—H1···O122^{i}	0.84 (3)	2.22 (3)	2.971 (4)	149 (3)
C4—H4B···Cl1^{ii}	0.99	2.72	3.664 (3)	161
C23—H23···O141^{iii}	0.95	2.37	3.318 (4)	173
C42—H42B···Cl1^{iv}	0.99	2.73	3.514 (3)	137

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*, −*z*+1; (iii) *x*, −*y*+5/2, *z*−1/2; (iv) −*x*+1, *y*+1/2, −*z*+1/2.

Fig. 1

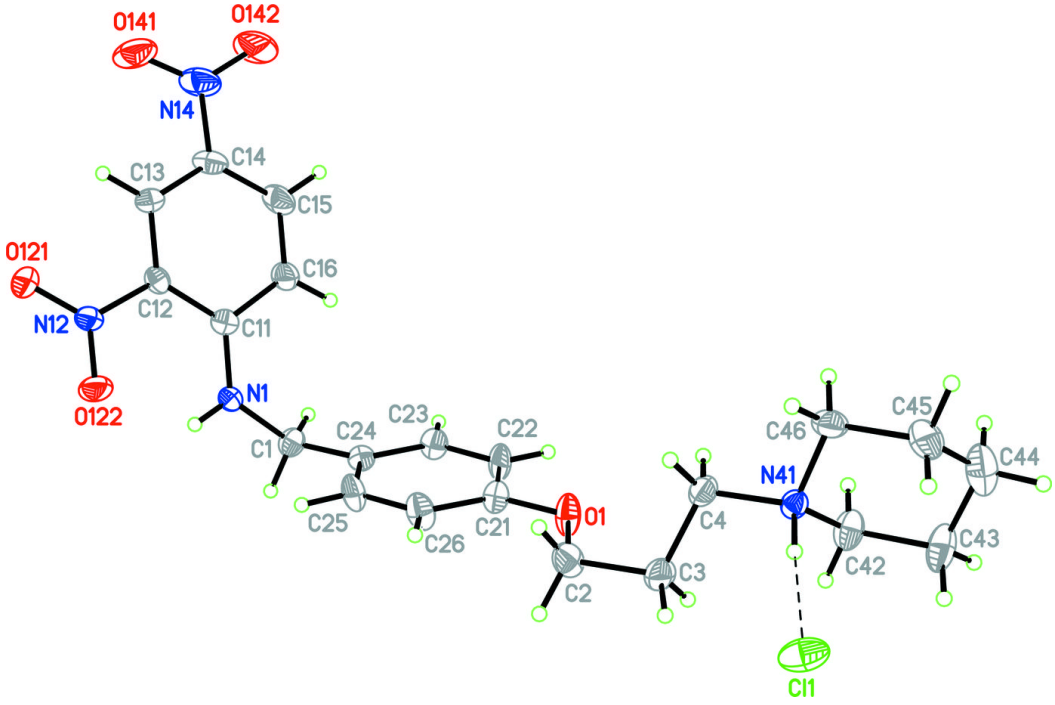

O141
O142
N14
C14
C13
C15
O121
C16
C12
N12
C11
O122
N1
C1
C24
C23
C22
C25
C26
C21
O1
C2
C3
C4
N41
C46
C45
C44
C43
C42
Cl1

Fig. 2