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

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Key indicators

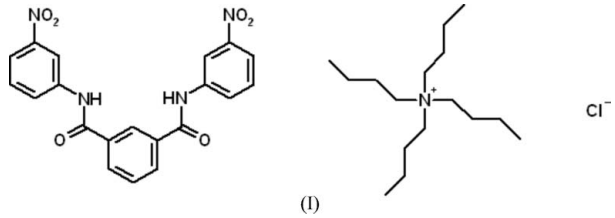
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.054
wR factor = 0.149
Data-to-parameter ratio = 18.6

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).

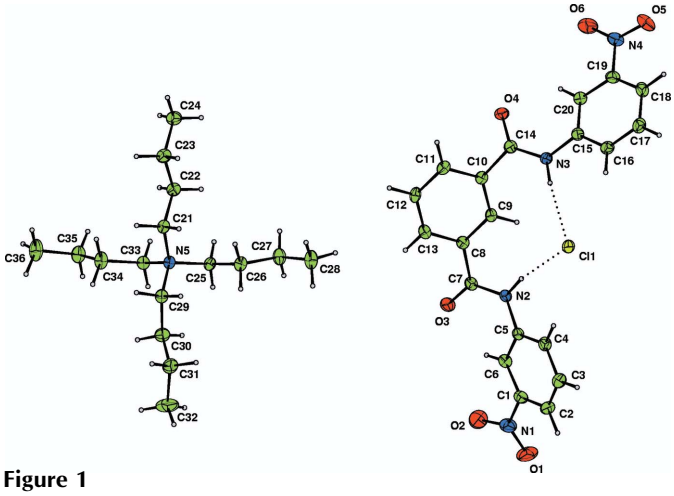


Figure 1
View of the asymmetric unit of (I), showing the atom labelling and the hydrogen-bonded chloride anion. Displacement ellipsoids are drawn at the 50% probability level and hydrogen bonds are shown as dashed lines.

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.