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Bis(tetrabutylammonium) isophthalate 1-phenyl-3-[2,4,5-tris(3-phenylureido)-phenyl]urea: a synchrotron study

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Key indicators

Single-crystal synchrotron study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.149
Data-to-parameter ratio = 17.2

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

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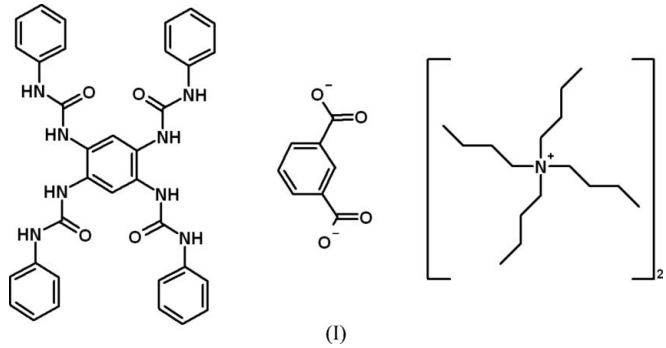
The structure of $2C_{16}H_{36}N^+\cdot C_8H_4O_4^{2-}\cdot C_{34}H_{30}N_8O_4$, comprises tapes of encapsulated hydrogen-bonded isophthalate anions which are arranged into parallel sheets interleaved with tetrabutylammonium layers; each of the two independent neutral molecules is disposed about a centre of inversion.

Comment

The title complex, (I) (Fig. 1), was prepared as part of an ongoing study into the carboxylate-binding properties of urea-based anion receptors (Brooks *et al.*, 2005*a*,*b*, 2006). The receptor of the title compound, (I), exists as two independent molecules each disposed about an inversion centre. Each of these has a similar, but not identical, conformation with the four phenyl urea arms forming a cross-like arrangement. In both cases, the least-squares planes through the pendant phenyl rings of opposing arms are parallel, from symmetry, but the arms are twisted out of the plane defined by the central aromatic ring. Constructing the least-squares planes through the rings of the symmetry-related arms and calculating the angle between them, we obtain values of 59.63 (4) (molecule 1) and 49.09 (4)° (molecule 2); it is this difference that best describes the disparity between the two independent molecules. The geometry of the isophthalate anion is unremarkable, the least-squares plane through both carboxylate groups being tilted away from the ring by 16.6 (3)°.



(I)

The guest isophthalate anions link the hosts into tapes *via* N—H···O hydrogen bonds (Fig. 2 and Table 1), each carboxylate group interacting with an adjacent receptor so that atom O5 forms three hydrogen bonds and atom O6 two; the corresponding atoms of the second independent molecule are O7 and O8, respectively. This implies that the N7—H and N2—H hydrogen bonds are bifurcated. The donor–acceptor distances range from 2.7455 (16) to 3.3616 (16) Å. Within the tapes, the two independent host molecules alternate, and these tapes interdigitate to form sheets in the $(1\bar{1}1)$ plane. The

supramolecular structure is completed by a parallel arrangement of these sheets interleaved with layers of the tetrabutylammonium counterion.

Experimental

The receptor was prepared according to a literature procedure (Mataka *et al.*, 1993). Crystals of the tetrabutylammonium isophthalate complex were obtained by slow evaporation of a solution of the receptor in acetonitrile in the presence of excess tetrabutylammonium isophthalate.

Crystal data

$2C_{16}H_{36}N^+\cdot C_8H_4O_4^-\cdot C_{34}H_{30}N_8O_4$
$M_r = 1263.69$
Triclinic, $P\overline{1}$
$a = 12.7319\ (3)$ Å
$b = 16.0019\ (4)$ Å
$c = 19.3005\ (5)$ Å
$\alpha = 96.512\ (1)^\circ$
$\beta = 108.754\ (1)^\circ$
$\gamma = 103.554\ (1)^\circ$
$V = 3542.79\ (15)$ Å^3
$Z = 2$
$D_x = 1.185$ Mg m^{-3}
Synchrotron radiation
$\lambda = 0.6727$ Å
Cell parameters from 9807 reflections
$\theta = 2.2$–28.3°
$\mu = 0.08$ mm^{-1}
$T = 120\ (2)$ K
Plate, colourless
$0.15 \times 0.10 \times 0.03$ mm

Data collection

Bruker SMART APEX2 CCD diffractometer
Fine-slice ω scans
32925 measured reflections
16104 independent reflections
12499 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 25.8^\circ$
$h = -16 \rightarrow 16$
$k = -20 \rightarrow 20$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.149$
$S = 1.02$
16104 reflections
935 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0838P)^2 + 0.6824P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O8^{i}	0.88	1.98	2.8316 (16)	164
N2—H2A···O7^{i}	0.88	2.25	3.0983 (16)	161
N2—H2A···O8^{i}	0.88	2.62	3.3616 (16)	143
N3—H3A···O7^{i}	0.88	1.98	2.7455 (16)	145
N4—H4A···O7^{i}	0.88	2.26	3.0052 (17)	142
N5—H5A···O5	0.88	2.23	3.0208 (18)	149
N6—H6···O5	0.88	1.90	2.7393 (17)	158
N7—H7···O5	0.88	2.43	3.2615 (18)	157
N8—H8···O6	0.88	1.87	2.7474 (16)	171

Symmetry code: (i) $-x+1, -y+1, -z+1$.

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; 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).

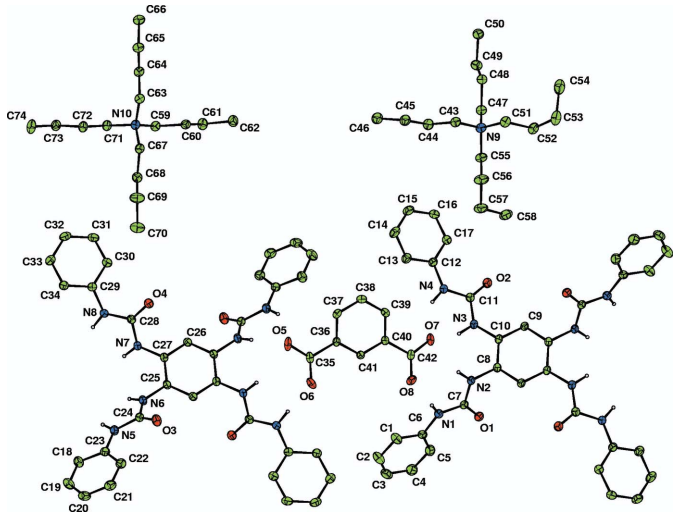


Figure 1
View of the components of (I), with the atoms of the asymmetric unit labelled; non-acidic H atoms have been omitted for clarity. Displacement ellipsoids are drawn at the 30% probability level. In the left-hand neutral molecule unlabelled atoms are related to labelled atoms by $1-x, -y, -z$. In the right-hand neutral molecule unlabelled atoms are related to labelled atoms by $-x, -y, 1-z$.

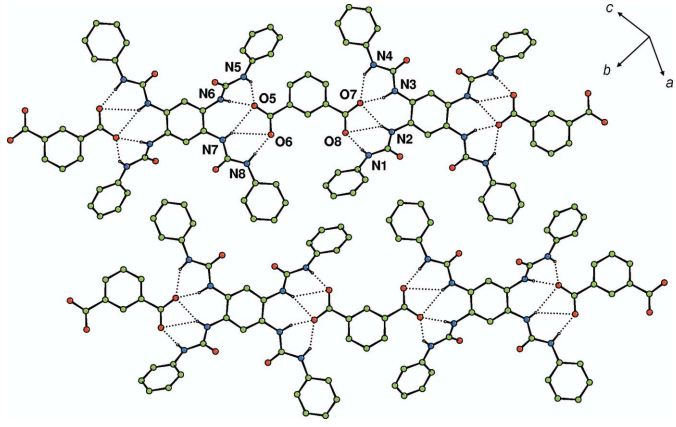


Figure 2
View of the hydrogen-bonded (dashed lines) tapes interdigitated into sheets in the $(1\overline{1}1)$ plane; non-acidic H atoms have been omitted for clarity.

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References

Brooks, S. J., Edwards, P. R., Gale, P. A. & Light, M. E. (2006). *New J. Chem.* **30**, 65–70.
Brooks, S. J., Gale, P. A. & Light, M. E. (2005*a*). *Chem. Commun.* pp. 4696–4698.
Brooks, S. J., Gale, P. A. & Light, M. E. (2005*b*). *CrystEngComm*, **7**, 586–591.

Bruker (2004). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cernik, R. J., Clegg, W., Catlow, C. R. A., Bushnell-Wye, G., Flaherty, J. V., Greaves, G. N., Hamichi, M., Burrows, I., Taylor, D. J. & Teat, S. J. (1997). *J. Synchrotron Rad.* **4**, 279–286.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Mataka, S., Irie, T., Ikezaki, Y. & Tashiro, M. (1993). *Chem. Ber.* **126**, 1819–1822.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELX97*. University of Göttingen, Germany.
Watkin, D. M., Pearce, L. & Prout, C. K. (1993). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.