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1,4-Phenylene-bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide) bis(tetrabutylammonium chloride) acetonitrile disolvate

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Key indicators

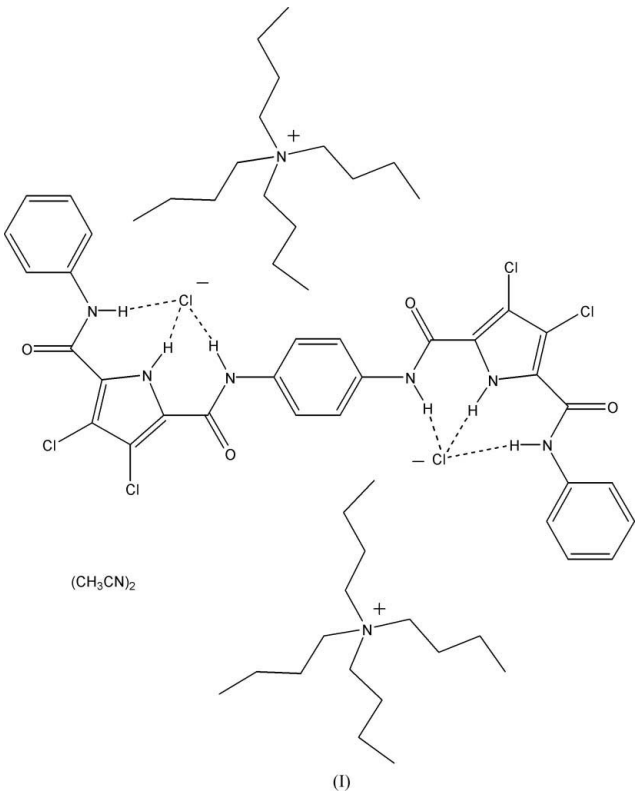
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
Disorder in solvent or counterion
R factor = 0.060
wR factor = 0.144
Data-to-parameter ratio = 18.0

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

The title compound, $C_{30}H_{20}N_6O_4Cl_4.2C_{16}H_{36}N^+.2Cl^-.2C_2H_3N$, contains two hydrogen-bonded chloride anions bound to 1,4-phenylene-bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide) as the tetrabutylammonium salt. There is also a short pyrrolic hydrogen bond (N···Cl = 3.068 (3) Å), and two longer ones to the amino H atoms [N···Cl = 3.269 (3) Å and 3.275 (3) Å]. The neutral molecule lies on an inversion centre situated at the centre of the central benzene ring.

Comment

1,4-Phenylene-bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide) crystallizes from tetrabutylammonium chloride acetonitrile solution as a tetrabutylammonium chloride acetonitrile solvate, (I).



(I)

The receptor adopts an S-shaped conformation around a centre of inversion with one chloride bound on each side. The pyrrole and terminal benzene ring pairs are coplanar, and the angle between the central and terminal benzene rings is 32.02 (4)°. Of the three hydrogen bonds to the chloride, the pyrrolic one is the shortest, with an N···Cl distance of 3.068 (3) Å, whilst the two either side are longer, with distances of 3.269 (3) Å and 3.275 (3) Å.

Experimental

The receptor molecule, 1,4-phenylene-bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide), (1), was synthesized according to literature methods (Gale *et al.*, 2002). Crystals of the acetonitrile solvate of the tetrabutylammonium chloride complex were grown by slow evaporation of an acetonitrile solution of (1) in acetonitrile in the presence of excess tetrabutylammonium chloride.

Crystal data

$C_{30}H_{20}N_6O_4Cl_4\cdot 2C_{16}H_{36}N^+\cdot 2Cl^-\cdot 2C_2H_3N$
M_r = 1308.24
Monoclinic, $P2_1/c$
a = 8.5720 (2) Å
b = 21.1088 (5) Å
c = 19.3520 (6) Å
β = 93.5560 (10)° **precision OK?**
V = 3494.90 (16) Å^3
Z = 2
D_x = 1.243 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 35384 reflections
θ = 2.9–26.4°
μ = 0.30 mm^{-1}
T = 120 (2) K
Needle, colourless
0.15 × 0.07 × 0.05 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.906, T_{max} = 0.990
13653 measured reflections
7107 independent reflections
4902 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 26.4°
$h = -10 \rightarrow 10$
$k = -26 \rightarrow 25$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.144
S = 1.11
7107 reflections
395 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 4.3109P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −1.05 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0023 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H91···Cl3	0.88	2.40	3.269 (3)	171.6
N2—H92···Cl3	0.88	2.20	3.068 (3)	166.8
N3—H93···Cl3	0.88	2.40	3.275 (3)	171.2

H atoms were identified in a difference map and then placed in calculated positions (N—H 0.88, aromatic C—H 0.95, methylene C—H 0.99, methyl C—H 0.98) and refined using a riding model [U_{iso}(H) = 1.2 or 1.5 times U_{eq}(parent atom)]. One arm of the tetrabutylammonium is disordered. It has been modelled as split over two possible orientations with one third and two thirds occupancy. C—C and C—N distances were restrained to standard values and the displacement parameters of split atom pairs were constrained to be equal. The deepest hole is located 1.28 Å from C9.

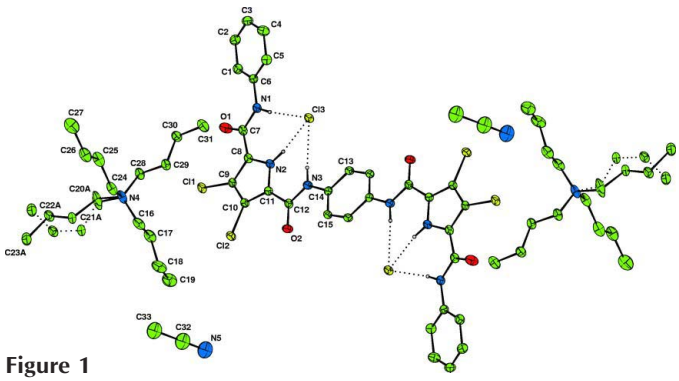


Figure 1
Structure of the title compound, with displacement ellipsoids drawn at the 35% probability level and non-acidic H atoms omitted for clarity. Both disorder components are shown.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); 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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