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4-Aminomethyl-phenylamino-bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide) tetrabutylammonium salt

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Key indicators

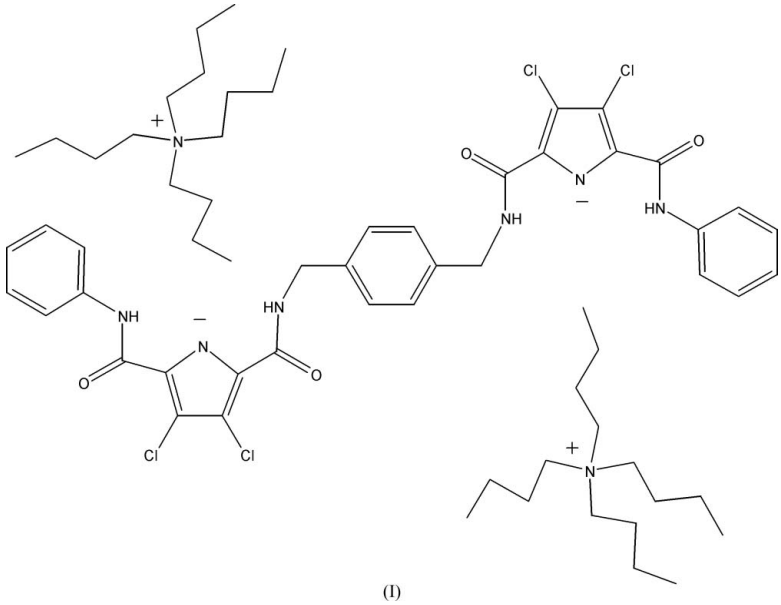
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.008 Å
R factor = 0.101
wR factor = 0.192
Data-to-parameter ratio = 15.9

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

The crystal structure of the tetrabutylammonium salt of doubly deprotonated 4-aminomethyl(phenylamino)bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide), $2C_{16}H_{36}N^{+}\cdot C_{32}H_{22}C_{l4}N_6O_4^{2-}$, has been elucidated. The anion lies on an inversion centre and adopts a twisted S shape.

Comment

4-Aminomethyl(phenylamino)bis-(3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxamide) crystallizes as the tetrabutylammonium salt, (I), from an acetonitrile solution of the compound in the presence of excess tetrabutylammonium fluoride. The anion adopts a twisted S shape around a centre of inversion. The pyrrole and terminal benzene ring pairs are coplanar and the angle between the central and terminal benzene ring is 73.01 (5)°.



(I)

Experimental

p-Xylenediamine (68 mg, 0.5 mmol, 1 equiv.) was added to a solution of 3,4-dichloro-5-phenylcarbamoyl-1*H*-pyrrole-2-carboxylic acid (300 mg, 1 mmol, 2 equiv.) in DMF (30 ml) under a nitrogen atmosphere. Triethyamine (104 mg, 1 mmol, 2 equiv.), benzotriazol-1-yloxy)tripyrrolidinophosphonium hexafluorophosphate (572 mg, 1.1 mmol, 2.2 equiv.) and 5 mg (0.04 mmol, 0.04 equiv.) of *N*-hydroxybenzotriazole were added and the reaction was stirred for 72 h. The solvent was then removed and water (50 ml) was added. The product was extracted with dichloromethane (DCM, 3 50 ml). The organic phase was collected and the solvent was removed. The product was washed with diethyl ether (75 ml) and a small quantity of 10% MeOH

in DCM (v/v). The product was obtained as a white solid (132 mg, 0.19 mmol, 38%).

M.p. 590 K (decomp.). ^{1}H NMR 300 MHz in DMSO-d_6 δ (p.p.m.): 4.50 (*d*, J = 5.4, 4H, CH_2), 7.00–7.70 (*m*, 14H, ArH), 8.50 (*t*, 2H, J = 5.4, central–CONH), 10.04 (*s*, 2H, outer–CONH), 12.79 (*s*, 2H, NH-pyrrole). ^{13}C NMR 75 MHz in DMSO-d_6 δ (p.p.m.): 42.3, 112.0, 113.6, 119.8, 122.9, 123.1, 123.9, 127.4, 128.7, 137.6, 138.3, 156.5, 158.1. TOF LD$^+$ mass spectrum: m/z (%): 472 (100) $[C_{23}H_{22}Cl_2N_4O_3]^+$. Elemental analysis: Calc. for $C_{32}H_{24}Cl_4N_6O_4.H_2O$: C 53.65, H 3.66, N 11.73%; found: C 53.28, H 3.73, N 12.03%.

Crystals of the title compound were obtained by slow evaporation of an acetonitrile solution in the presence of excess tetrabutylammonium fluoride.

Crystal data

$2C_{16}H_{36}N^+\cdot C_{32}H_{22}Cl_4N_6O_4^{2-}$	Z = 1
M_r = 1181.27	D_x = 1.208 Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
a = 9.524 (2) Å	Cell parameters from 58835 reflections
b = 10.363 (3) Å	θ = 2.9–27.5°
c = 17.056 (4) Å	μ = 0.23 mm^{-1}
α = 78.293 (11)°	T = 120 (2) K
β = 88.733 (14)°	Block, colourless
γ = 80.136 (14)°	0.20 × 0.10 × 0.07 mm
V = 1623.9 (7) Å^3	

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer	5757 independent reflections
φ and ω scans	2777 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)	R_{int} = 0.111
T_{min} = 0.732, T_{max} = 0.984	θ_{max} = 26.1°
16494 measured reflections	$h = -11 \rightarrow 11$
	$k = -12 \rightarrow 12$
	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 2.4189P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.101	$(\Delta/\sigma)_{max}$ = 0.019
$wR(F^2)$ = 0.192	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
S = 1.04	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
5757 reflections	
361 parameters	
H-atom parameters constrained	

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}(C,N)].

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).

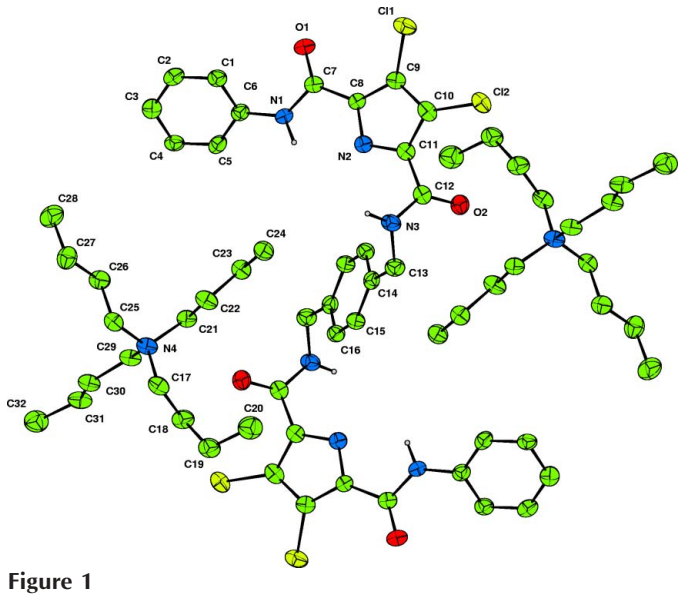


Figure 1
Structure of the title compound, with displacement ellipsoids drawn at the 50% probability level and non-acidic H atoms omitted for clarity.

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