A cocrystal of diethylammonium chloride and diphenylamine

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Key indicators

Single-crystal X-ray study
$T = 100$ K
Mean σ(C–C) = 0.002 Å
R factor = 0.027
wR factor = 0.070
Data-to-parameter ratio = 20.8

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

The title compound, diethylammonium chloride–diphenylamine (1/1), $C_4H_{12}N^+\cdot Cl^-\cdot C_{12}H_{11}N$, consists of discrete diethylammonium cations, chloride anions and diphenylamine molecules linked together by several N—H···Cl hydrogen bonds.

Comment

The silylation of diphenylamine with 2-chloro-2,6-dimethyl-2,6-disilaheptane in pentane in the presence of diethylamine was attempted. Light yellow crystals of the title compound, (I), were obtained from the crude product mixture.

(I)
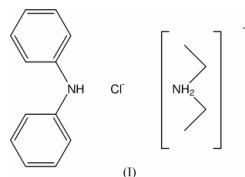
Compound (I) consists of discrete diethylammonium cations, chloride anions and diphenylamine molecules. Each chloride anion forms three hydrogen bonds (Table 2). The dihedral angle between the two aromatic rings of the diphenylamine is 37.91 (4)°.

Experimental

An equimolar mixture (9 mmol) of 2-chloro-2,6-dimethyl-2,6-disilaheptane, diphenylamine and diethylamine in 150 ml of pentane was heated to 309 K for 2 h. After filtration and removal of the solvent a yellow oil was obtained. Light yellow crystals of $(H_5C_2)_2NH_2^+\cdot Cl^-\cdot (H_5C_6)_2NH$ were obtained directly from the crude reaction product upon standing for 21 d.

Crystal data

$C_4H_{12}N^+\cdot Cl^-\cdot C_{12}H_{11}N$	$D_x = 1.148$ Mg m^{-3}
$M_r = 278.81$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 19430
$a = 9.0679$ (6) Å	reflections
$b = 10.0077$ (6) Å	$\theta = 2.4$–27.5°
$c = 17.8386$ (13) Å	$\mu = 0.23$ mm^{-1}
$\beta = 94.726$ (6)°	$T = 100$ (2) K
$V = 1613.33$ (19) Å^3	Block, light yellow
$Z = 4$	$0.26 \times 0.24 \times 0.15$ mm

Data collection

Stoe IPDS II two-circle diffractometer	3833 independent reflections
	3087 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.042$
Absorption correction: multi-scan (*MULABS*; Spek, 1990; Blessing, 1995)	$\theta_{max} = 27.9°$
	$h = -11 \rightarrow 11$
	$k = -13 \rightarrow 13$
$T_{min} = 0.955$, $T_{max} = 0.968$	$l = -23 \rightarrow 23$
27910 measured reflections	

Refinement

Refinement on F^2	H atoms treated by a mixture of
$R[F^2 > 2\sigma(F^2)] = 0.027$	independent and constrained
$wR(F^2) = 0.070$	refinement
$S = 0.97$	$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2]$
3833 reflections	where $P = (F_o^2 + 2F_c^2)/3$
184 parameters	$(\Delta/\sigma)_{max} = 0.001$
	$\Delta\rho_{max} = 0.21$ e Å^{-3}
	$\Delta\rho_{min} = -0.17$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—N3	1.4924 (13)	N1—C21	1.3944 (12)
N3—C4	1.4919 (14)	N1—C11	1.3989 (13)
C4—N3—C2	113.79 (8)	C21—N1—C11	129.96 (8)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N3—H3$A\cdots$Cl1	0.908 (15)	2.260 (15)	3.1581 (9)	170.0 (12)
N3—H3$B\cdots$Cl1^{ii}	0.913 (15)	2.218 (15)	3.0946 (9)	160.7 (12)
N1—H1$\cdots$Cl1	0.851 (14)	2.467 (14)	3.2909 (9)	162.9 (12)

Symmetry code: (ii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z$.

H atoms bonded to C atoms were refined with fixed individual displacement parameters [U_{iso}(H) = $1.2U_{eq}$(C) or $1.5U_{eq}$(C$_{methyl}$)], using a riding model, with aromatic C—H = 0.95 Å, methylene C—H = 0.99 Å and methyl C—H = 0.98 Å. H atoms bonded to N atoms were refined independently with isotropic displacement parameters.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine
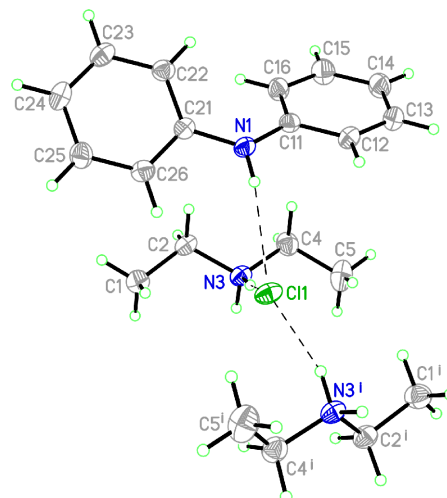

Figure 1
Perspective view of the asymmetric unit, with the atom numbering; displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines. [Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$.]

structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97.

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