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Rerefinement of triethylammonium iodide from new intensity data at low temperature

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The anion and the cation of the title compound, $C_6H_{16}N^+\cdot I^-$, are located on special positions of site symmetry 3*m*. As a result, there is only a sixth of an ion pair in the asymmetric unit. The anion and cation are connected *via* an N—H$\cdots$I hydrogen bond. Each methylene group is disordered over two equally occupied positions.

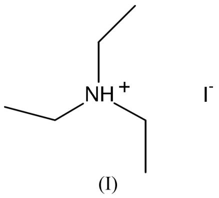
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.011 Å
Disorder in main residue
R factor = 0.025
wR factor = 0.071
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.

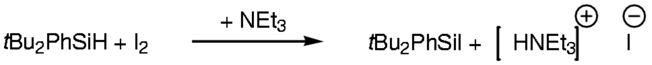
Comment

Recently, we reported on the properties and on the reactivity of di-*tert*-butylphenylsilyl halides (Lerner *et al.*, 2001; Wiberg *et al.*, 2001). We report here a new route to synthesize di-*tert*-butylphenylsilyl iodide, ^{t}Bu$_2$PhSiI, and the X-ray crystal structure analysis of triethylammonium iodide, [HNEt$_3$]I, (I).



(I)

The synthesis of di-*tert*-butylphenylsilyl iodide, ^{t}Bu$_2$PhSiI, was achieved by the reaction of di-*tert*-butylphenylsilane, ^{t}Bu$_2$PhSiH, with iodine in the presence of triethylamine as indicated in the reaction scheme below.



The cell parameters of (I) have already been published by Hendricks (1928) and the coordinates of the N, H(N) and I atoms have been determined by James *et al.* (1985). We present here the coordinates of all atoms determined from data measured at 173 K. The I and N atoms are located on special positions of site symmetry 3*m*, and the methyl C atom is located on a mirror plane. The anion and cation are connected *via* an N—H$\cdots$I hydrogen bond.

Experimental

Di-*tert*-butylphenylsilyl iodide, ^{t}Bu$_2$PhSiI, was obtained by stirring a solution of 0.329 g (1.49 mmol) di-*tert*-butylphenylsilane, ^{t}Bu$_2$PhSiH, and 0.378 g (1.49 mmol) iodine in 10 ml CH$_2$Cl$_2$ in the presence of 0.21 ml (1.49 mmol) triethylamine at 273 K. Colourless crystals of [HNEt$_3$]I were grown from the reaction solution at room temperature.

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Crystal data

$C_6H_{16}N^+ \cdot I^-$
$M_r = 229.10$
Hexagonal, $P6_3mc$
$a = 8.6555$ (11) Å
$c = 7.8351$ (9) Å
$V = 508.35$ (11) Å^3
$Z = 2$
$D_x = 1.497$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 12 855 reflections
$\theta = 3.8$–27.5°
$\mu = 3.08$ mm^{-1}
$T = 173$ (2) K
Needle, colourless
0.35 × 0.12 × 0.11 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 1990; Blessing, 1995)
$T_{\min} = 0.404$, $T_{\max} = 0.714$
5945 measured reflections

464 independent reflections
421 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\max} = 27.5°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.071$
$S = 1.09$
464 reflections
25 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 0.0681P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.00$ e Å^{-3}
$\Delta\rho_{\min} = -0.43$ e Å^{-3}
Absolute structure: Flack (1983), 232 Friedel pairs
Flack parameter = 0.50 (13)

Table 1
Selected geometric parameters (Å, °).

N1—C1$^{\rm i}$	1.452 (8)	C1—C2	1.530 (11)
N1—C1—C2	128.9 (7)		

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

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$I1	0.9 (2)	2.6 (2)	3.529 (11)	180

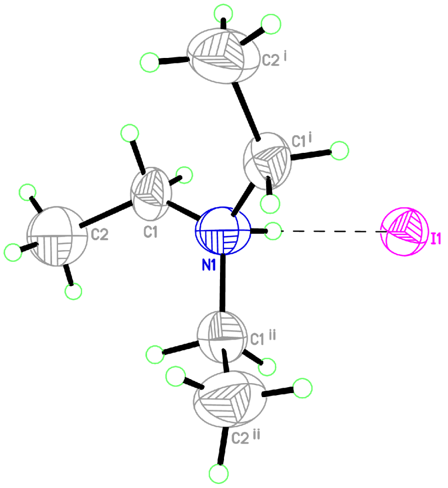

Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level. [Symmetry codes: (i) $1 - y, x - y, z$; (ii) $1 - x+y, 1 - x, z$.]

H atoms bonded to carbon were refined with fixed individual displacement parameters [$U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ or $1.5U_{\rm eq}({\rm C_{methyl}})$], using a riding model, with C—H = 0.98 (methyl) and 0.99 Å (methylene). The H atom bonded to nitrogen was refined isotropically. The methylene group and the methyl H atoms are disordered over two equally occupied positions each. The investigated crystal was an inversion twin; the ratio of the two twin components refined to 0.50 (13):0.50 (13).

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 structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 1990).

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