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Dihydronium bromide tetrakis(di-*tert*-butylmethylsilanol)

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean *σ*(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.111
Data-to-parameter ratio = 22.5

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

The asymmetric unit of the title compound, $H_5O_2^+\cdot Br^-\cdot 4C_9H_{22}OSi$, is composed of four di-*tert*-butylmethylsilanol molecules, one Br^- anion, and two half $H_5O_2^+$ ions, each of which has one disordered H atom. The silanol molecules form hydrogen bonds to the Br anion and the $H_5O_2^+$ ions form hydrogen bonds to the hydroxyl O atoms of the silanol molecules.

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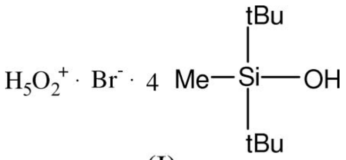
Comment

Previously, we have reported the X-ray crystal structure analyses and the syntheses of supersilanol cocrystals with 18-crown-6 and water, ${}^{t}Bu_3SiOH\cdot$(18-crown-6)$\cdot H_2O$, and with CF_3SO_3H and water, ${}^{t}Bu_3SiOH\cdot(HO_3SCF_3)\cdot 0.5H_2O$, respectively, which were accessible from the reactions of the sodium triazenide ${}^{t}Bu_3Si{-}NNa{-}N{=}N{-}Si{}^{t}Bu_3$ with H_2O in the presence of 18-crown-6 (Lerner *et al.*, 2002) or of ${}^{t}Bu_3SiO_3SCF_3$ with H_2O (Lerner *et al.*, 2005). Surprisingly, we obtained single crystals of the title compound by hydrolysis of ${}^{t}Bu_2MeSiBr$.


$H_5O_2^+\cdot Br^-\cdot 4\ Me{-}Si(tBu)_2{-}OH$

(I)

A perspective view of the title compound is shown in Fig. 1. The asymmetric unit is composed of four discrete di-*tert*-butyl-methyl-silanol molecules, one Br^- anion, and two half $H_5O_2^+$ ions, each of which has one disordered H atom. The silanol molecules form hydrogen bonds to the Br anion (Table 1) and the $H_5O_2^+$ ions form hydrogen bonds to the hydroxyl O atoms of the silanol molecules.

Experimental

A mixture of ${}^{t}Bu_2MeSiBr$ (0.23 g, 2.5 mmol), CH_2Cl_2 (10 ml) and water was stored at ambient temperature. After several weeks, slow evaporation of the solvent led to the deposition of a small quantity of colourless needles.

Crystal data

$H_5O_2^+\cdot Br^-\cdot 4C_9H_{22}OSi$
M_r = 814.36
Monoclinic, $P2_1/n$
a = 16.4814 (8) Å
b = 16.1107 (5) Å
c = 20.0624 (10) Å
β = 98.510 (4)°
V = 5268.5 (4) Å^3

Z = 4
D_x = 1.027 Mg m^{-3}
Mo *Kα* radiation
μ = 0.90 mm^{-1}
T = 173 (2) K
Needle, colourless
0.30 × 0.13 × 0.12 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
$T_{\min} = 0.773$, $T_{\max} = 0.899$

69100 measured reflections
9889 independent reflections
8181 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.065$
$\theta_{\max} = 25.7°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.111$
$S = 1.07$
9889 reflections
440 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 4.5021P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.43$ e Å^{-3}

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
O1—H1$\cdots$Br1	0.69 (3)	2.57 (4)	3.256 (2)	174 (4)
O1A—H1$A$$\cdots$Br1	0.69 (4)	2.57 (4)	3.257 (2)	170 (4)
O1B—H1$B$$\cdots$Br1	0.72 (4)	2.59 (4)	3.293 (2)	163 (4)
O1C—H1$C$$\cdots$Br1	0.73 (4)	2.54 (4)	3.263 (2)	171 (5)
O1W—H1$WA$$\cdots$O1$C$	0.84	1.85	2.637 (3)	156
O1W—H1$WB$$\cdots$O1$^{\text{i}}$	0.84	1.80	2.628 (3)	169
O1W—H1$WC$$\cdots$O1$W^{\text{i}}$	0.84	1.60	2.437 (4)	180
O2W—H2$WA$$\cdots$O1$A$	0.84	1.84	2.634 (3)	156
O2W—H2$WB$$\cdots$O1$B$	0.84	1.85	2.639 (3)	155
O2W—H2$WC$$\cdots$O2$W^{\text{ii}}$	0.84	1.60	2.442 (4)	180

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+1, -z+1$.

H atoms bonded to C were refined with fixed individual displacement parameters [$U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$] using a riding model with C—H = 0.98 Å. All H atoms bonded to O could be located in a difference map. The $H_5O_2^+$ H atoms were refined with fixed individual displacement parameters [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$] using a riding model with O—H = 0.84 Å. The disordered H atom was refined on two equally occupied sites. The hydroxyl H atoms were freely refined.

Data collection: *X-AREA* (Stoe, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON*.

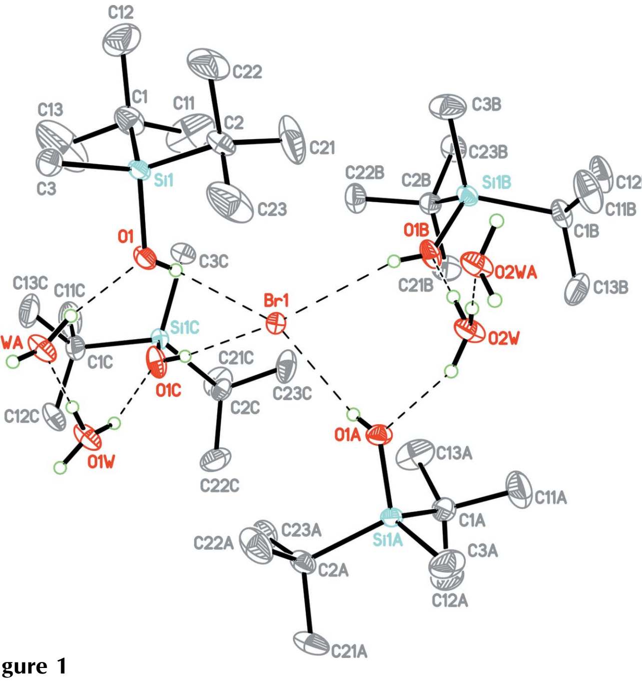


Figure 1
The asymmetric unit of the title compound, together with additional water molecules to complete the local hydrogen-bonding patterns, with displacement ellipsoids are drawn at the 30% probability level; H atoms bonded to C have been omitted for clarity. Hydrogen bonds are shown as dashed lines. Only one component of each disordered H atom of the $H_5O_2^+$ ions is shown. Symmetry operator for O1WA is $1-x, 1-y, 1-z$ and $-x, 1-y, 1-z$ for O2WA.

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