

## 4-(Trimethylsilyl)benzoic acid

Monika C. Haberecht,<sup>a</sup> Hannes Vitze,<sup>a</sup> Hans-Wolfram Lerner<sup>a</sup> and Michael Bolte<sup>b\*</sup>

<sup>a</sup>Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany, and <sup>b</sup>Institut für Organische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany

Correspondence e-mail: bolte@chemie.uni-frankfurt.de

## Key indicators

Single-crystal X-ray study

$T = 100\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

$R$  factor = 0.034

$wR$  factor = 0.063

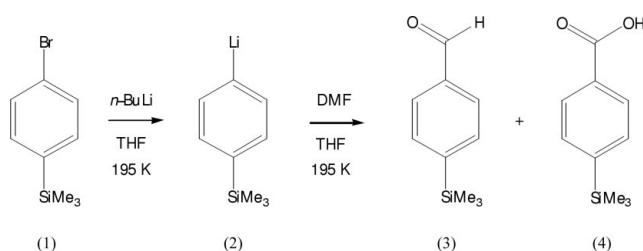
Data-to-parameter ratio = 17.5

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

The title compound,  $\text{C}_{10}\text{H}_{14}\text{O}_2\text{Si}$ , crystallizes as a centrosymmetric  $\text{O}-\text{H}\cdots\text{O}$  hydrogen-bonded dimer with two almost identical molecules in the asymmetric unit.

## Comment

Dibromoboryl compounds with an aromatic ring system are conveniently accessible from (trimethylsilyl)arenes and  $\text{BBr}_3$ . We became interested in using this type of reaction in the synthesis of dendrimeric borylarenes. The starting material for the synthesis of one of our dendrimeric building blocks is 4-(trimethylsilyl)benzaldehyde, (3) (see scheme), which can be obtained on reacting the *para*-lithium salt of (trimethylsilyl)benzene, (2), with *N,N*-dimethylformamide in tetrahydrofuran at low temperatures. 4-(Trimethylsilyl)benzoic acid, (4), occurs as a side product in this reaction.



The title compound, (4), crystallizes with two independent molecules in the asymmetric unit. These two molecules are almost identical. A least-squares fit of all non-H atoms gives an r.m.s. deviation of 0.098 Å. Both molecules form  $\text{O}-\text{H}\cdots\text{O}$

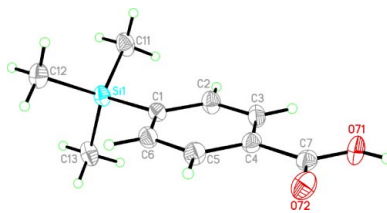


Figure 1

Perspective view of molecule 1 of the title compound, with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level.

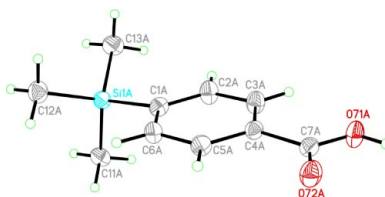


Figure 2

Perspective view of molecule 2 of the title compound, with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level.

hydrogen-bonded centrosymmetric dimers. This structural motif is typical for benzoic acid derivatives.

### Experimental

A solution of 1-bromo-4-(trimethylsilyl)benzene (28.04 mmol) in dry tetrahydrofuran (100 ml) was cooled to 195 K and *n*-butyllithium 2.5 M in *n*-hexane (33.65 mmol) was added dropwise. After stirring for 90 min at low temperature, dry *N,N*-dimethylformamide (33.65 mmol) was added using a dropping funnel. The reaction mixture was slowly warmed to room temperature, quenched with water and then extracted with diethyl ether. The organic layers were combined, washed with brine and dried over MgSO<sub>4</sub>. Evaporation of the solvent yielded a yellow oil which consisted of the two products (3) and (4). 4-(Trimethylsilyl)benzaldehyde (3.135 g, 62.7%) was obtained by distillation *in vacuo* (391 K, 0.01 mbar). 4-(Trimethylsilyl)benzoic acid (0.562 g, 10.3%) crystallized from the residue of the distillation.

#### Crystal data

C <sub>10</sub> H <sub>14</sub> O <sub>2</sub> Si	Z = 4
<i>M<sub>r</sub></i> = 194.30	<i>D<sub>x</sub></i> = 1.185 Mg m <sup>-3</sup>
Triclinic, <i>P</i> $\bar{1}$	Mo <i>K</i> α radiation
<i>a</i> = 6.2858 (10) Å	Cell parameters from 7303 reflections
<i>b</i> = 12.2693 (17) Å	$\theta$ = 3.7–25.7°
<i>c</i> = 14.258 (2) Å	$\mu$ = 0.18 mm <sup>-1</sup>
$\alpha$ = 96.528 (12)°	<i>T</i> = 100 (2) K
$\beta$ = 91.642 (13)°	Plate, colourless
$\gamma$ = 93.932 (12)°	0.36 × 0.24 × 0.09 mm
<i>V</i> = 1089.2 (3) Å <sup>3</sup>	

#### Data collection

Stoe IPDS II two-circle diffractometer	4142 independent reflections
$\omega$ scans	2297 reflections with <i>I</i> > 2σ( <i>I</i> )
Absorption correction: multi-scan ( <i>MULABS</i> ; Spek, 1990; Blessing, 1995)	<i>R</i> <sub>int</sub> = 0.056
<i>T</i> <sub>min</sub> = 0.947, <i>T</i> <sub>max</sub> = 0.992	$\theta$ <sub>max</sub> = 25.7°
12520 measured reflections	<i>h</i> = -7 → 7
	<i>k</i> = -14 → 14
	<i>l</i> = -17 → 17

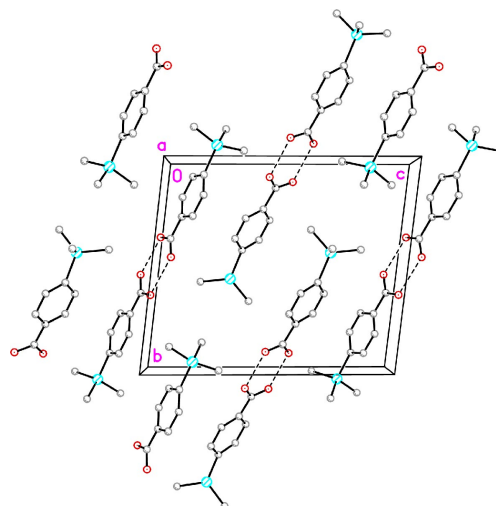
#### Refinement

Refinement on <i>F</i> <sup>2</sup>	H-atom parameters constrained
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )] = 0.034	$w = 1/[\sigma^2(F_o^2) + (0.0184P)^2]$
<i>wR</i> ( <i>F</i> <sup>2</sup> ) = 0.063	where $P = (F_o^2 + 2F_c^2)/3$
<i>S</i> = 0.90	( $\Delta/\sigma$ ) <sub>max</sub> = 0.001
4142 reflections	$\Delta\rho$ <sub>max</sub> = 0.21 e Å <sup>-3</sup>
237 parameters	$\Delta\rho$ <sub>min</sub> = -0.24 e Å <sup>-3</sup>

**Table 1**

Selected geometric parameters (Å).

Si1—C11	1.859 (2)	Si1A—C12A	1.856 (2)
Si1—C12	1.863 (2)	Si1A—C13A	1.861 (2)
Si1—C13	1.867 (2)	Si1A—C11A	1.868 (2)
Si1—C1	1.889 (2)	Si1A—C1A	1.894 (2)
C4—C7	1.490 (3)	C4A—C7A	1.492 (3)
C7—O72	1.258 (3)	C7A—O72A	1.240 (3)
C7—O71	1.287 (3)	C7A—O71A	1.304 (3)



**Figure 3**

Packing diagram of the title compound, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines but the H atoms have been omitted.

**Table 2**

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O71—H71...O72 <sup>i</sup>	0.84	1.82	2.643 (2)	166
O71A—H71A...O72A <sup>ii</sup>	0.84	1.80	2.643 (2)	175

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

H atoms were refined with fixed individual displacement parameters [*U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C,O) or 1.5*U*<sub>eq</sub>(C<sub>methyl</sub>)] using a riding model, with O—H = 0.84 Å, C—H = 0.95 Å or methyl C—H = 0.98 Å. The dihedral angles about the hydroxyl groups were allowed to refine

Data collection: *X-AREA* (Stoe & Cie, 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*.

### References

- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.  
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.  
 Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.  
 Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.  
 Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.