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# Crystal structure of 2-((*tert*-butyldimethylsilyl) oxy)-5-methylisophthalaldehyde, $C_{15}H_{22}O_3Si$

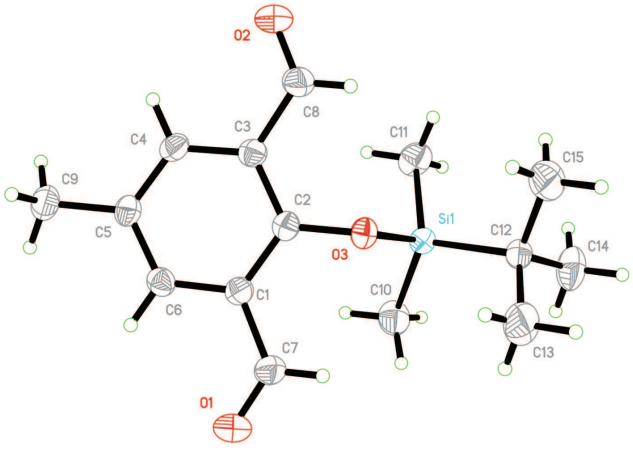


**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Block, clear light colorless |
| Size: | 0.24 × 0.21 × 0.2 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.15 $mm^{-1}$ |
| Diffractometer, scan mode: | SuperNova, $\omega$-scans |
| $\theta_{max}$, completeness: | 29.2°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 7774, 3666, 0.020 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 2834 |
| $N(param)_{refined}$: | 178 |
| Programs: | CryAlis$^{PRO}$ [1], SHELXT [2], SHELXL [3], OLEX2 [4] |

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## Abstract

$C_{15}H_{22}O_3Si$, monoclinic, $P2_1/n$ (no. 14), $a = 10.0187(5)$ Å, $b = 11.9948(5)$ Å, $c = 13.6259(7)$ Å, $\beta = 102.521(5)°$, $V = 1598.51(14)$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.0485$, $wR_{ref}(F^2) = 0.1273$, T = 293 K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

## Source of material

The title compound was synthesized from 2-hydroxy-5-methylisophthalaldehyde. *tert*-Butyldimethylsilyl chloride (181 mg, 1.2 mmol) was added to a solution of 2-hydroxy-5-methylisophthalaldehyde (164 mg, 1 mmol) and imidazole (102 mg, 1.5 mmol) in tetrahydrofuran (10 mL). The reaction mixture was stirred at room temperature for 5 h. The mixture was diluted with dichloromethane (50 mL) and washed with water (50 mL) and brine (50 mL), then dried over anhydrous $Na_2SO_4$ and evaporated to dryness. The crude product was purified by column chromatography on silica gel using petroleum ether/ethyl acetate (30/1, *v*/*v*) as eluent to afford the product as a white solid (250 mg, 91% yield). Crystals of the title compound were grown from a petroleum ether/dichloromethane (1/1, *v*/*v*) solution at room temperature.

## Experimental details

Hydrogen atoms were assigned isotropic displacement factors $U_{iso}(H) = 1.2\ U_{eq}$ (N and imidazol C), or $U_{iso}(H) = 1.5\ U_{eq}$ (methyl C) and included in the refinement using the riding model, with C—H = 0.93 Å (imidazol) or C—H = 0.96 Å (methyl), and N—H = 0.86 Å.

## Discussion

Compounds with formyl group have a wide range of applications in the synthesis of functional organic molecules such as fluorescent dyes [5–8], bioactive molecules [9–11] and photoelectric materials [12]. Formyl groups can be oxidized to carboxyl groups and reduced to alcohol for further reaction. In addition, *tert*-butyl dimethylsilyl (TBS) is used as a common protecting group for hydroxyl substituents [13–15]. This protecting group can be removed by reaction with a fluoride anion. Therefore, compounds with TBS are

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**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$).

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Si1 | 0.73184(5) | 0.72377(4) | 0.38802(4) | 0.03960(16) |
| O1 | 0.5048(2) | 0.92130(13) | 0.61619(14) | 0.0842(5) |
| O2 | 0.7438(2) | 0.36869(13) | 0.55854(14) | 0.0833(5) |
| O3 | 0.61307(12) | 0.67206(10) | 0.44568(9) | 0.0439(3) |
| C1 | 0.59476(17) | 0.73914(15) | 0.60841(14) | 0.0418(4) |
| C2 | 0.62730(16) | 0.65493(15) | 0.54673(13) | 0.0399(4) |
| C3 | 0.67308(17) | 0.55130(14) | 0.58911(14) | 0.0422(4) |
| C4 | 0.69158(18) | 0.53683(15) | 0.69269(14) | 0.0460(4) |
| H4 | 0.7227 | 0.4683 | 0.7204 | 0.055* |
| C5 | 0.66531(18) | 0.62070(16) | 0.75610(14) | 0.0446(4) |
| C6 | 0.61511(18) | 0.72083(15) | 0.71188(14) | 0.0446(4) |
| H6 | 0.5943 | 0.7777 | 0.7525 | 0.053* |
| C7 | 0.5312(2) | 0.84519(17) | 0.56626(17) | 0.0568(5) |
| H7 | 0.5105 | 0.8534 | 0.4967 | 0.068* |
| C8 | 0.6952(2) | 0.45618(17) | 0.52585(17) | 0.0551(5) |
| H8 | 0.6701 | 0.4645 | 0.4564 | 0.066* |
| C9 | 0.6875(2) | 0.60300(19) | 0.86805(16) | 0.0625(6) |
| H9A | 0.7022 | 0.6737 | 0.9018 | 0.094* |
| H9B | 0.7660 | 0.5563 | 0.8904 | 0.094* |
| H9C | 0.6083 | 0.5678 | 0.8833 | 0.094* |
| C10 | 0.7872(3) | 0.86267(18) | 0.44232(17) | 0.0661(6) |
| H10A | 0.7121 | 0.9140 | 0.4263 | 0.099* |
| H10B | 0.8616 | 0.8894 | 0.4147 | 0.099* |
| H10C | 0.8165 | 0.8564 | 0.5140 | 0.099* |
| C11 | 0.8824(2) | 0.6293(2) | 0.40960(18) | 0.0648(6) |
| H11A | 0.9154 | 0.6183 | 0.4805 | 0.097* |
| H11B | 0.9533 | 0.6621 | 0.3816 | 0.097* |
| H11C | 0.8564 | 0.5589 | 0.3778 | 0.097* |
| C12 | 0.64023(19) | 0.73192(15) | 0.25284(14) | 0.0453(4) |
| C13 | 0.5074(3) | 0.7976(3) | 0.2435(2) | 0.0997(10) |
| H13A | 0.4500 | 0.7612 | 0.2816 | 0.150* |
| H13B | 0.4609 | 0.8012 | 0.1741 | 0.150* |
| H13C | 0.5279 | 0.8718 | 0.2689 | 0.150* |
| C14 | 0.7324(3) | 0.7886(2) | 0.19187(19) | 0.0844(8) |
| H14A | 0.7531 | 0.8630 | 0.2165 | 0.127* |
| H14B | 0.6862 | 0.7915 | 0.1224 | 0.127* |
| H14C | 0.8157 | 0.7470 | 0.1984 | 0.127* |
| C15 | 0.6072(3) | 0.6148(2) | 0.2103(2) | 0.0868(9) |
| H15A | 0.6905 | 0.5735 | 0.2156 | 0.130* |
| H15B | 0.5610 | 0.6200 | 0.1409 | 0.130* |
| H15C | 0.5495 | 0.5775 | 0.2476 | 0.130* |

widely used in the design and synthesis of probes for fluoride anion detection [16–19]. Within this perspective and our continuing research efforts, we report the crystal structure of the title compound. The formyl groups, methyl group and benzene ring are coplanar. In the molecule, the Si(1)—O(3), Si(1)—C(10), Si(1)—C(11), Si(1)—C(12), and O(3)—C(2) bond lengths are found to be 1.6802(13) Å, 1.858(2) Å, 1.858(2) Å, 1.8740(19) Å, and 1.369(2) Å, respectively, which are within the range expected for similar single bonds. The bond lengths of O(1)—C(7) and O(2)—C(8) are found to be 1.202(3) and 1.201(2) Å, respectively, in accordance with a typical carbonyl double bond. The bond angles O3—Si1—C10, O3—Si1—C11, O3—Si1—C12, C10—Si1—C11, C10—Si1—C12, C11—Si1—C12, C2—O3—Si1, O3—C2—C1, O3—C2—C3, O1—C7—C1, and O2—C8—C3 are 108.99(9)°, 109.51(9)°, 103.49(7)°, 108.75(11)°, 112.64(10)°, 113.28(10)°, 126.58(10)°, 120.64(15)°, 119.99(16)°, 124.2(2)° and 124.0(2)°, respectively. One dimensional chains are formed by intermolecular C9—H9A···O2 hydrogen bonds. The chains extend through weaker C14—H14B—O2 contacts to form a two dimensional supramolecular layer.

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