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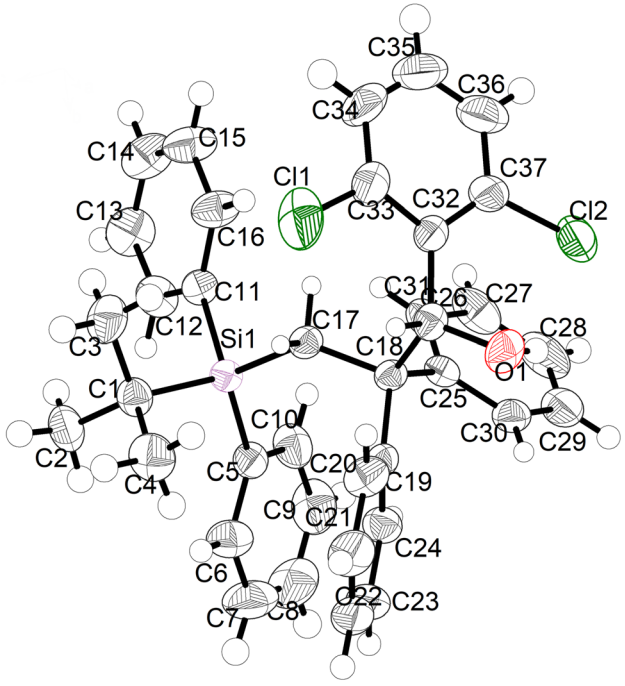
Crystal structure of 3-(*tert*-butyldiphenylsilyl)-1-(2,6-dichlorophenyl)-2,2-diphenylpropan-1-ol, $C_{37}H_{36}Cl_2OSi$



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Abstract

$C_{37}H_{36}Cl_2OSi$, monoclinic $P2_1/n$ (no. 14), a = 10.4363(14) Å, b = 17.128(2) Å, c = 17.875(3) Å, β = 90.934(2)°, V = 3194.8(8) Å^3, Z = 4, $R_{gt}(F)$ = 0.0491, $wR_{ref}(F^2)$ = 0.1449, T = 296.15 K.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.12 × 0.11 × 0.08 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.27 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.4°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	18,849, 7173, 0.030
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4927
$N(param)_{refined}$:	382
Programs:	CrysAlis^{Pro} [1], SHELX [2, 3], Diamond [4]

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

An improved synthesis was performed which was similar to the previously report [5–8]. To the solution of 2,6-dichlorobenzaldehyde (1.75 g, 10.0 mmol) in anhydrous 1,4-dioxane (100 mL) was added tert-butyldiphenylsilane-carboxylic acid (4.26 g, 15.0 mmol), 1,1-diphenylethylene (2.70 g, 15.0 mmol), $NaHCO_3$ (0.84 g, 10.0 mmol) and organic photo catalyst 4CzIPN (394 mg, 0.5 mmol) under argon atmosphere. After addition, the reaction mixture was irradiated under 440 nm Kessil light for 12 h. TLC detection showed the reaction was finished. After simple filtration by silicon gel, the solvent was removed in vacuo. The obtained residue was dissolved in dichloromethane (100 mL), washed with water (50 mL × 2), brine (50 mL × 2), and dried over anhydrous $MgSO_4$. After filtration, the solvent was evaporated in vacuo. The obtained residue was purified by column chromatography to afford white solid of 3-(tert-butyldiphenylsilyl)-1-(2,6-dichlorophenyl)-2,2-diphenylpropan-1-ol (4.38 g, 72 %), which was further purified by recrystallization from hexane and ethyl acetate. Crystals were obtained by slow evaporation from the solution at 268–270 K.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4110 (2)	0.38280 (13)	0.89461 (12)	0.0413 (5)
C2	0.4680 (3)	0.37858 (17)	0.97448 (13)	0.0568 (7)
C3	0.2924 (2)	0.32905 (17)	0.89086 (16)	0.0592 (7)
C4	0.3661 (3)	0.46710 (16)	0.87892 (17)	0.0610 (7)
C5	0.6962 (2)	0.39311 (13)	0.83799 (12)	0.0387 (5)
C6	0.7158 (3)	0.46516 (15)	0.87236 (15)	0.0544 (6)
C7	0.8369 (3)	0.49780 (19)	0.8807 (2)	0.0760 (9)
C8	0.9423 (3)	0.4586 (2)	0.85429 (19)	0.0818 (10)
C9	0.9268 (3)	0.3873 (2)	0.82068 (17)	0.0722 (9)
C10	0.8053 (2)	0.35490 (17)	0.81292 (14)	0.0529 (6)
C11	0.5509 (2)	0.23854 (12)	0.83889 (12)	0.0361 (5)
C12	0.6332 (2)	0.20960 (14)	0.89511 (14)	0.0482 (6)
C13	0.6387 (3)	0.13056 (16)	0.91247 (17)	0.0609 (7)
C14	0.5637 (3)	0.07835 (15)	0.87313 (18)	0.0643 (8)
C15	0.4824 (3)	0.10414 (15)	0.81750 (18)	0.0636 (8)
C16	0.4756 (3)	0.18340 (14)	0.80053 (15)	0.0518 (6)
C17	0.4507 (2)	0.36066 (13)	0.72768 (12)	0.0345 (5)
C18	0.49834 (19)	0.40564 (11)	0.65783 (11)	0.0315 (4)
C19	0.5053 (2)	0.49315 (12)	0.67961 (11)	0.0354 (5)
C20	0.3962 (2)	0.54074 (14)	0.67906 (15)	0.0514 (6)
C21	0.4019 (3)	0.61707 (15)	0.70533 (17)	0.0622 (7)
C22	0.5156 (3)	0.64795 (15)	0.73170 (16)	0.0615 (8)
C23	0.6237 (3)	0.60274 (14)	0.73229 (15)	0.0554 (7)
C24	0.6182 (2)	0.52604 (13)	0.70675 (13)	0.0440 (5)
C25	0.62576 (19)	0.37517 (12)	0.62846 (11)	0.0330 (4)
C26	0.6687 (2)	0.29965 (13)	0.64399 (13)	0.0440 (5)
C27	0.7802 (3)	0.27077 (17)	0.61177 (15)	0.0595 (7)
C28	0.8491 (3)	0.31643 (19)	0.56355 (17)	0.0644 (8)
C29	0.8086 (2)	0.39063 (17)	0.54715 (15)	0.0554 (7)
C30	0.6988 (2)	0.42010 (13)	0.57962 (13)	0.0416 (5)
C31	0.3906 (2)	0.39440 (13)	0.59428 (11)	0.0373 (5)
C32	0.3595 (2)	0.30944 (14)	0.57629 (12)	0.0404 (5)
C33	0.2567 (3)	0.27159 (18)	0.61096 (14)	0.0606 (8)
C34	0.2315 (4)	0.1923 (2)	0.60193 (19)	0.0885 (13)
C35	0.3063 (5)	0.1491 (2)	0.5555 (2)	0.0947 (13)
C36	0.4003 (4)	0.18283 (17)	0.51649 (18)	0.0711 (9)
C37	0.4253 (2)	0.26193 (15)	0.52568 (13)	0.0481 (6)
Cl1	0.14991 (7)	0.32299 (7)	0.66617 (5)	0.0914 (3)
Cl2	0.54013 (7)	0.29926 (5)	0.46575 (4)	0.0664 (2)
H00A	0.312120	0.415468	0.615897	0.045*
H00D	0.673162	0.470878	0.568640	0.050*
H00F	0.622512	0.268122	0.676217	0.053*
H00G	0.692384	0.495976	0.707931	0.053*
H00H	0.685547	0.244241	0.921510	0.058*
H00I	0.796519	0.306244	0.790320	0.063*
H00K	0.419719	0.200133	0.762789	0.062*
H00L	0.645447	0.492285	0.890302	0.065*
H00M	0.318714	0.520987	0.660866	0.062*
H00N	0.701018	0.623307	0.749786	0.067*
H00O	0.854773	0.421340	0.514199	0.067*
H00P	0.518915	0.699135	0.748991	0.074*
H00Q	0.328167	0.647547	0.705064	0.075*
H00E	0.256822	0.329467	0.840988	0.089*
H00J	0.229378	0.347467	0.925211	0.089*
H00R	0.317086	0.276790	0.904061	0.089*
H00T	0.807922	0.220452	0.622975	0.071*
H00U	0.923164	0.296936	0.542108	0.077*
H00S	0.498778	0.326670	0.984133	0.085*
H00V	0.403085	0.391529	1.009837	0.085*
H00W	0.537709	0.414899	0.979276	0.085*
H00X	0.693080	0.113025	0.950636	0.073*
H	0.308469	0.483325	0.917241	0.092*
HA	0.322897	0.469177	0.831140	0.092*
H00Y	0.439017	0.501258	0.878629	0.092*
H00Z	0.568105	0.025385	0.884314	0.077*
H00	0.431712	0.068681	0.791019	0.076*
H00B	0.366 (2)	0.3832 (13)	0.7388 (12)	0.040 (6)*
H00C	0.433 (2)	0.3072 (14)	0.7114 (13)	0.045 (7)*
H004	0.479602	0.427307	0.511119	0.077*
H010	0.997759	0.360589	0.803019	0.087*
H011	0.846969	0.545990	0.904040	0.091*
H012	0.448356	0.153344	0.483470	0.085*
H013	0.164485	0.168925	0.627263	0.106*
H014	1.023538	0.480501	0.859271	0.098*
H015	0.291980	0.095749	0.550824	0.114*
O1	0.41224 (17)	0.44062 (10)	0.53036 (9)	0.0514 (4)
Si1	0.53266 (5)	0.34733 (3)	0.82295 (3)	0.03081 (14)

2 Experimental details

Carbon bound hydrogen atoms were included using riding models. For the OH group additionally a rotation about the C–O bond was allowed.

3 Comment

The 3-(tert-butyldiphenylsilyl)-1-(2,6-dichlorophenyl)-2,2-diphenylpropan-1-ol is a product formed by the reductive radical polar crossover (RPC)-type reaction [9], in which the anion is generated from single-electron reduction. RPC reactions have been proven useful in the formation of multiple bonds in a single transformation, including a multicomponent reaction (MCR) manifold [10]. In this paper, a silane radical was produced through single electron oxidation and decarboxylation of tert-butyldiphenylsilanecarboxylic acid anion, which then was added to the olefin to form carbon-centered radical. Next, carbon anion was achieved after single-electron reduction with low-valence organic photo catalyst 4CzIPN. Finally, carbon anion can then be added

to aldehyde to provide the 3-silyl-propan-1-ol derivative. Different from Nozaki–Hiyama-Kishi type reaction [11], multicomponent strategy of reductive RPC reactions without alkyl metal species was rarely reported [8], especially with silane radical. Crystals of 3-(tert-butyl diphenylsilyl)-1-(2,6-dichlorophenyl)-2,2-diphenylpropan-1-ol were obtained by continuous adjustment of crystallization conditions, which revealed the specific structure of the multicomponent RPC reaction product with silanecarboxylic acid for the first time. In the meantime, this result also provides direct structural proof for the structure of the previous report [8], which is very significant for further understanding 3-silyl-propan-1-ol derivative and multicomponent RPC reaction. The title structure (see the Figure) was reported for the first time in this paper. This crystal structure is a racemic alcohol with a chiral carbon atom (C31). The propyl alcohol was linked to a 2,6-dichlorophenyl at C31, two phenyl at C18, and a silyl group at C17. The bond lengths and angles are in the expected ranges [12, 13].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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