

Communication

Synthesis and Crystal Structure of Dimethyl{N-[(2-oxy-1-naphthyl)methylene]leucinato}silicon

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Abstract: The title compound was prepared by reaction of the Schiff base ligand N-(2-hydroxy-1-naphthylidene)leucine with dichlorodimethylsilane in the presence of triethylamine as base. The resulting pentacoordinate silicon complex was characterized by NMR, IR, UV-Vis spectroscopy and melting point. The structure was confirmed by single-crystal X-ray diffraction data. It crystallizes in the monoclinic space group *Ic* with unit cell dimensions $a = 7.2030(6)$, $b = 22.9842(14)$, $c = 10.8946(12)$ Å, $\beta = 96.141(7)^\circ$, $V = 1793.3(3)$ Å³, $Z = 4$.

Keywords: organosilicon; silicon complex; Schiff base ligand; crystal structure

1. Introduction

The reaction of aromatic *ortho*-hydroxycarbonyl compounds with amino acids allows access to versatile Schiff base ligands. These act as tridentate O,N,O-ligands in the coordination of a wide range of metal ions; see, for instance, [1–6]. The possible applications of these complexes are manifold. They range from catalysts [7], sensors for copper [8] and non-linear optics [9] to the generation of polynuclear magnetic complexes [10–12] and different biomedical applications [13–17].

Silicon complexes with tridentate O,N,O ligands have already been investigated with regard to various possible applications. These include use in thin-film solar cells [18], as dyes [19], or as compounds with antibacterial activity [20,21]. Complexes in which the Schiff base ligand is substituted with sulfonamide residues possess, in addition to antimicrobial properties, nematocidal and insecticidal properties [22].

Herein, we wanted to expand the available pentacoordinate silicon complexes to those containing the N-[(2-oxy-1-naphthyl)methylene]leucinato ligand. Previously published related silicon complexes contain Schiff base ligands derived from *o*-hydroxyacetophenone [4,23], salicyl aldehyde [5], acetylacetone [24,25], or *o*-hydroxynaphthyl aldehyde [6].

2. Results and Discussion

N-(2-hydroxy-1-naphthylidene)leucine was synthesized from a racemic mixture of leucine and 2-hydroxy-1-naphthaldehyde as shown in Scheme 1. The reaction of the Schiff base ligand with dichlorodimethylsilane was performed in THF as the solvent in the presence of triethylamine as the base. The crude product was recrystallized from chloroform/hexane and provided the silicon complex 1. It was shown via thin-layer chromatography (1:3 mixture of chloroform and hexane) that the ligand molecule was no longer present in the product and that the batch product consisted of only one component.



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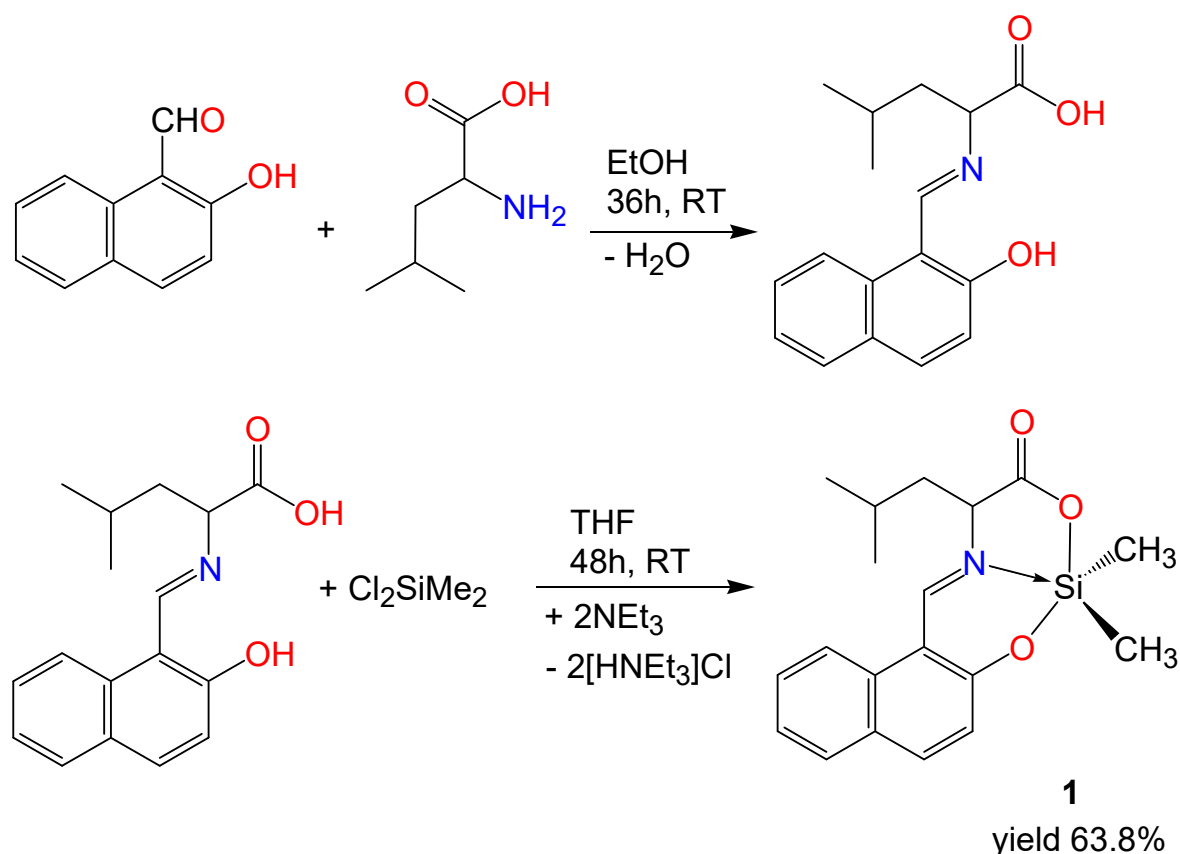
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Scheme 1. Synthesis of the Schiff base ligand N-(2-hydroxy-1-naphthylidene)leucine and the silicon complex **1**.

Compound **1** was characterized with spectroscopic methods. The ^1H NMR spectrum shows the absence of OH groups, which are present in the NMR spectrum of the ligand molecule. The ^{13}C NMR spectrum shows the presence of the ligand framework plus signals for the silicon-bound methyl groups at 1.6 and 5.2 ppm. The ^{29}Si NMR spectra in solution and in the solid state show chemical shift values of -65.6 and -68.7 ppm, respectively. This indicates the presence of a pentacoordinate silicon complex in solution and in the solid state. UV-Vis and IR data are summarized in the Experimental section. The most striking difference in the IR spectra of the ligand and product **1** is the occurrence of the C=O stretching vibrations at higher wavenumbers in **1**. The strong bands at 1692.6 and 1709.2 cm^{-1} occur due to the coordination with the silicon atom. All spectra are shown in the Supplementary Materials.

Compound **1** crystallizes in the monoclinic space group *Ic* with one molecule in the asymmetric unit (Figure 1). The space group *Ic* (containing a glide plane) shows that both enantiomers of **1** are present in the crystal [26]. The powder XRD of the batch material was compared with a powder XRD generated from the single crystal structure data in order to demonstrate the identity of both (see Supplementary Materials). The value of optical rotation of the batch product was determined to be $[\alpha]^{20}_{\text{D}} = 0^\circ$. This confirms the presence of the racemate for the batch product. This was to be expected since leucine was used as the racemate.

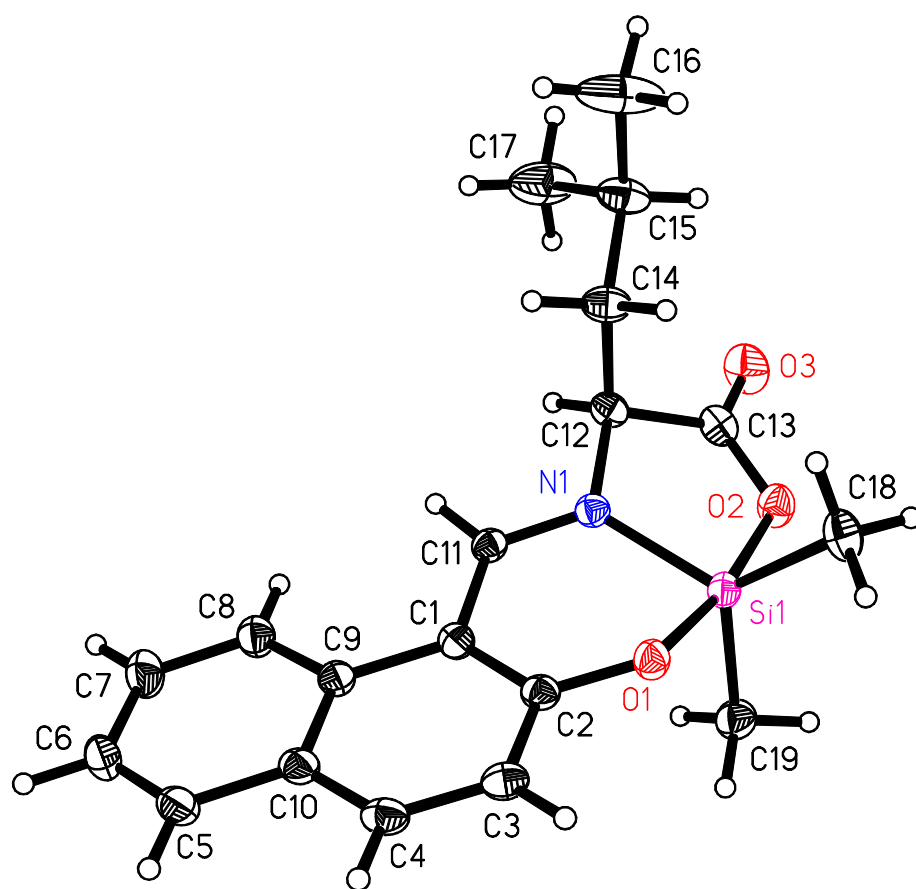


Figure 1. Molecular structure of **1**. Anisotropic displacement ellipsoids are drawn at the 50% probability level.

The silicon complex features a pentacoordinate silicon atom, coordinated by phenoxy-O1, carboxyl-O2, imine-N1 and two carbon atoms from methyl groups (C18 and C19). The parameter τ as defined by Addison et al. [27] is useful for analyzing the coordination geometry of the pentacoordinate silicon atom. The parameter is defined as $\tau = (\beta - \alpha)/60^\circ$, wherein β is the largest and α is the second-largest angle at the central atom. A perfect square pyramid has a parameter of $\tau = 0$, whereas a perfect trigonal bipyramid has $\tau = 1$. The largest angle at the silicon atom is O1–Si1–O2 with $170.47(8)^\circ$ and the second-largest is C18–Si1–C19 with $123.77(11)^\circ$ (see also Table 1). This gives a parameter of $\tau = 0.78$, which corresponds to a distorted trigonal bipyramid. The apical positions are occupied by O1 and O2 of the tridentate ligand, while the atoms N1, C18 and C19 represent the atoms in the trigonal plane.

The bond Si–O1 [$1.8039(15) \text{ \AA}$] is shorter than the bond Si1–O2 [$1.8525(17) \text{ \AA}$]. This can be explained by the electronegative character of the phenyl-bound O1 and the carboxyl-type oxygen atom O2. The bond lengths Si1–N1 and Si–C (Table 1) are similar as in similar pentacoordinate silicon complexes [4,6,23].

A comparison of **1** with silicon complexes which contain the N-[(2-oxy-1-naphthyl)methylene]valinato ligand (L') shows some striking differences in the bond angles C–Si–C (see Table 2). Very small bond angles of $78.60(6)$ and $110.8(2)^\circ$ are observed for the silacyclobutane and the silacyclohexane derivatives. The compounds L'SiEt₂ and L'SiPh₂ have larger bond angles of $118.4(4)$ to $119.7(4)^\circ$, respectively. The compound **1**, with two methyl groups at the silicon atom, features the largest bond angle in this series with a value of $123.77(11)^\circ$. This can be explained by the nature of the CH₃ groups, which carry three hydrogen atoms directly bound to the C _{α} -atom at silicon.

Table 1. Selected bond lengths [Å] and angles [°] for **1**.

Bond	Value	Angle	Value
Si1-O1	1.8039(15)	O1-Si1-O2	170.47(8)
Si1-O2	1.8525(17)	O1-Si1-N1	88.64(7)
Si1-N1	1.8574(18)	O2-Si1-N1	82.77(7)
Si1-C19	1.869(2)	O1-Si1-C19	95.38(9)
Si1-C18	1.872(2)	O2-Si1-C19	91.82(9)
O1-C2	1.312(2)	N1-Si1-C19	113.31(9)
O2-C13	1.299(3)	O1-Si1-C18	89.93(9)
O3-C13	1.228(3)	O2-Si1-C18	91.26(9)
N1-C11	1.313(3)	N1-Si1-C18	122.77(10)
N1-C12	1.484(2)	C19-Si1-C18	123.77(11)

Table 2. Comparison of bond angles C-Si-C of silicon complexes with comparable ligand environment.

Compound ^(a)	Value C-Si-C in °	Reference
L'Si(CH ₂) ₃	78.60(6)	[6]
L'Si(CH ₂) ₅	110.8(2)	[6]
L'SiEt ₂ ^(b)	118.4(4) 119.7(4)	[6]
L'SiPh ₂	119.25(10)	[6]
LSiMe ₂ (1)	123.77(11)	this work

^(a) The tridentate ligands contain different amino acids: L' = N-[(2-oxy-1-naphthyl)methylene]valinato; L = N-[(2-oxy-1-naphthyl)methylene]leucinato. ^(b) There are two crystallographic independent molecules in this crystal structure.

The potential donor atoms O1, O2 and N1 are engaged in the coordination with the silicon atom. Therefore, these atoms do not contribute to intermolecular interactions in the solid-state structure. The oxygen atom O3 from the carboxyl group is not engaged in the coordination of the central atom and forms hydrogen bonds to H8 and H11 from a neighboring molecule (Table 3 and Figure 2). These bifurcated hydrogen bonds form zig-zag chains of molecules parallel to the crystallographic bc-plane.

Table 3. Selected hydrogen bonds for **1** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C11-H11...O3#1	0.95	2.29	3.239(3)	173.2
C8-H8...O3#1	0.95	2.48	3.362(3)	153.7

Symmetry transformations used to generate equivalent atoms: #1 x, -y + 1, z + 1/2.

The leucinato group and the methyl groups at the silicon atom are orientated out of the plane of the molecule. These steric requirements hinder the formation of π - π arene stacking interactions which would normally be expected for such a compound containing a naphthyl group. Only one weak C...C contact is observed between C4 and C1A (with symmetry operation x - 0.5, 0.5 - y, z). The distance of this contact is 3.344 Å, which is only 0.056 Å less than the sum of the van der Waals radii of both atoms. The distance of the centroids of the aromatic rings involved (C1, C2, C3, C4, C9, C10) is 3.641 Å. Therefore, we can state that between the layers formed by the chains of molecules we can observe only weak C...C and H...H contacts.

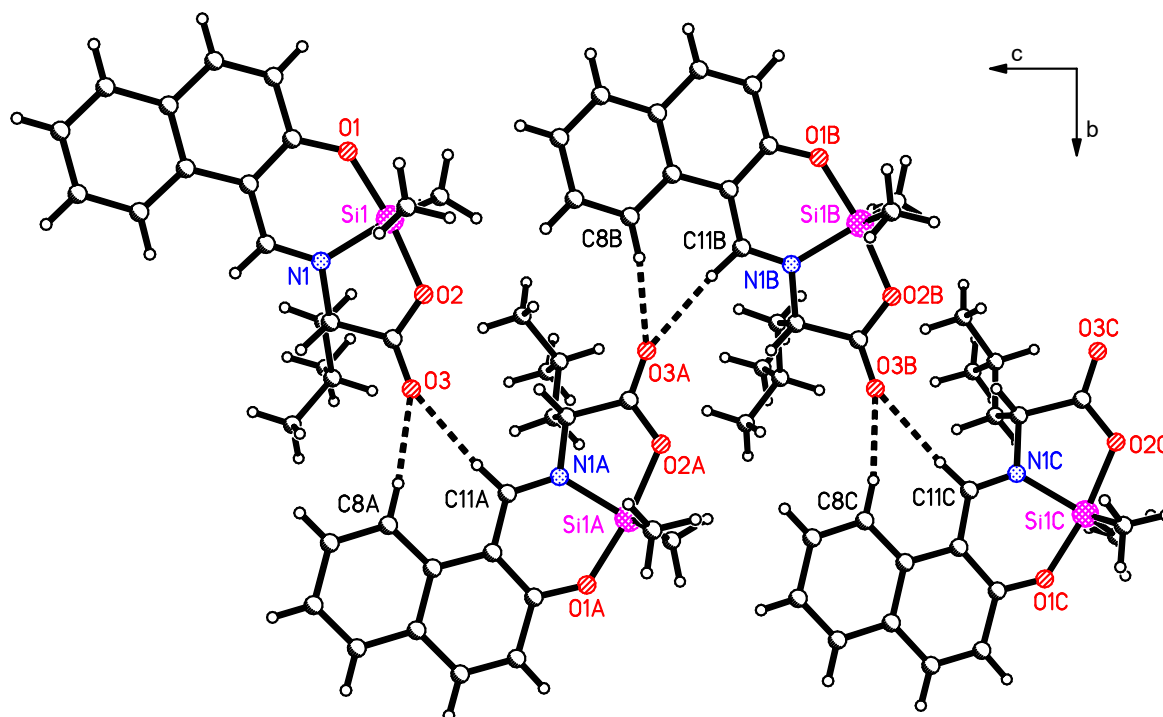


Figure 2. Intermolecular interactions in **1** between O3 and H8 and H11 from neighboring molecules (view in crystallographic *a*-direction).

There are only two structural characterized complexes with *N*-(2-oxy-1-naphthyl)methylene]leucinato (=L) in the literature. One is a pentacoordinate copper complex with the composition (L)Cu(phenanthroline) [28]. The other one is (L)(methanol)(methanolato)(oxo) vanadium(V). Therein, the vanadium atom reaches hexacoordination with the tridentate Schiff base, oxygen, methanol and methoxide as ligands [29].

In conclusion, we were able to expand the available pentacoordinate silicon complexes to those containing the *N*-(2-oxy-1-naphthyl)methylene]leucinato ligand. The dimethylsilyl derivative **1** shows a larger bond angle C-Si-C than comparable pentacoordinate silicon complexes.

3. Experimental Section

3.1. Methods

NMR spectra were measured with a BRUKER AVANCE III 500 MHz spectrometer in DMSO-*d*₆ or CDCl₃ with TMS as internal standard. UV/Vis spectra were recorded on a JASCO V-650 UV/Vis photometer in acetonitrile. IR spectra were recorded with a Nicolet 380 (Thermo Fisher) at room temperature. The products were ground together with dry potassium bromide and compressed as a tablet.

The specific rotation values were determined on the Model 241 MC digital polarimeter from Perkin Elmer at a temperature of 20 °C. The cuvette length was 10 cm. The measurements were made at a wavelength of 589 nm (sodium lamp). The powder diffractogram was recorded on the X-ray diffractometer D2 PHASER from Bruker. The melting points were determined with a “Polytherm A” hot-stage microscope from Wagner & Munz. The temperatures determined were not corrected.

3.2. Synthesis of *N*-(2-Hydroxy-1-naphthylidene)leucine

The ligand was prepared according to a method from the literature [30] and obtained as a pale yellow solid with a melting point of 172 °C [31].

¹H-NMR (DMSO-*d*₆, 500 MHz): δ [ppm] = 14.40, 13.31 (br, 2, C-OH), 9.23 (s, 1, CH=N), 6.79–8.12 (m, 6, H_{ar}), 4.52 (d, 1, CH-COO, ³J_{HH} = 7.1 Hz), 1.81 (dd, 2, CH₂, ³J_{HH} = 7.1 Hz),

1.62 (m, 1, $\text{CH}(\text{CH}_3)_2$), 0.95 (d, 3, CH_3 , $^3J_{\text{HH}} = 4.6$ Hz), 0.94 (d, 3, CH_3 , $^3J_{\text{HH}} = 4.6$ Hz); ^{13}C -NMR (DMSO-d_6 , 126 MHz): δ [ppm] = 176.0 (COO), 173.2 ($\text{C}_{\text{ar}}\text{-OH}$), 160.0 (CH=N), 137.6, 134.4, 129.4, 128.5, 126.1, 125.0, 123.0, 119.2 (8 C_{ar}), 106.8 ($\text{C}_{\text{ar}}\text{-CH=N}$), 63.0 (CH-COO), 42.2 (CH_2), 24.9 ($\text{CH}(\text{CH}_3)_2$), 23.2, 21.9 (2 CH_3).

$[\alpha]_{\text{D}}^{20} = 0^\circ$ ($c = 1$ g/100 mL in DMSO), UV/Vis (in DMSO; $c = 0.196$ mmol/L; $d = 1$ cm) λ [nm] (ϵ [$\text{Lmol}^{-1}\text{cm}^{-1}$]) = 424 (11,443), 403 (11,271), 307 (13,948).

IR (KBr): ν (cm^{-1}) = 423.9(w), 458.7(w), 489.4(w), 507.2(m), 524.5(m), 643.9(m), 669.7(m), 708(m), 746.8(s), 828.7(s), 969.9(m), 1011.4(m), 1036.9(m), 1100.4(m), 1149.3(m), 1213(m), 1270.9(m), 1340.1(m), 1366.9(s), 1425.6(m), 1456.3(m), 1469.9(m), 1506.7(m), 1576.1(m), 1632.1(s), 1642(vs), 1684.3(s), 1700.6(w), 1718.1(m), 1733.7(m), 2870.7(w), 2927.6(w), 2957.2(m), 3048.1(w).

3.3. Synthesis of Dimethyl[N-[(2-Oxy-1-Naphthyl)Methylene]Leucinato]Silicon (1)

To a solution of 1.19 g (4.17 mmol) of N-(2-hydroxy-1-naphthylidene)leucine in 30 mL of dry THF, 1.27 g (12.55 mmol, 50% excess) of triethylamine was added and the mixture was cooled in an ice bath to 0°C . Next, 0.57 g (4.42 mmol) of SiCl_2Me_2 was mixed with 20 mL of THF. This mixture was added via a dropping funnel into the solution. The reaction mixture was stirred for 48 h at room temperature. A white precipitate of triethylammonium chloride formed during that time, which was separated by filtration. The solvent was removed from the filtrate in a vacuum and the pale-yellow residue was dissolved in 25 mL of chloroform. The obtained suspension was filtered again and 5 mL of n-hexane was added. The resulting solution was stored for 3 weeks in a refrigerator at 8°C . Yellow crystals were obtained, which were suitable for crystal structure analysis. A powder XRD was recorded from the batch product and compared with a powder XRD generated from the single crystal structure data. The comparison of the data sets showed a very good agreement (see Supplementary Materials).

The purity of the product was further verified with TLC. For this purpose, chloroform and hexane (1:3) were used as eluent and the ligand was applied to the TLC plate as a reference substance. The analysis showed that the free ligand was no longer present and that the batch product consisted of only one component.

Yield: 0.91 g (63.8%), melting point: 158°C under decomposition.

^1H -NMR (CDCl_3 , 500 MHz): δ [ppm] = 8.93 (s, 1, CH=N), 7.00–8.01 (m, 6, H_{ar}), 4.26 (m, 1, CH-COO), 1.70–2.04 (m, 3, $\text{CH}_2\text{-CH}(\text{CH}_3)_2$), 1.03 (m, 6, $\text{CH}(\text{CH}_3)_2$), 0.62 (s, 3, Si-CH_3), 0.24 (s, 3, Si-CH_3); ^{13}C -NMR (CDCl_3 , 126 MHz): δ [ppm] = 171.5 (COO), 168.9 ($\text{C}_{\text{ar}}\text{-O}$), 163.2 (CH=N), 141.8, 132.3, 129.9, 129.7, 127.5, 124.9, 122.5, 118.5 (8 C_{ar}), 108.9 ($\text{C}_{\text{ar}}\text{-CH=N}$), 66.6 (CH-COO), 44.4 (CH_2), 24.0 ($\text{CH}_3\text{-CH-CH}_3$), 22.9, 22.1 ($\text{CH}_3\text{-CH-CH}_3$), 5.2 (Si-CH_3), 1.6 (Si-CH_3); ^{29}Si -NMR (CDCl_3 , 79.5 MHz): δ [ppm] = -65.6 ; ^{29}Si -CP/MAS-NMR (79.5 MHz, $\nu_{\text{rot}} = 5$ kHz): δ [ppm] = -68.7 .

$[\alpha]_{\text{D}}^{20} = 0^\circ$ ($c = 1$ g/100 mL CHCl_3), UV/Vis (CHCl_3 ; $c = 0.439$ mmol/L; $d = 1$ cm) λ [nm] (ϵ [$\text{Lmol}^{-1}\text{cm}^{-1}$]) = 411 (9152), 341 (7189), 338 (6925), 303 (2565), 260 (10,299).

IR (KBr): ν (cm^{-1}) = 422(s), 476.4(m), 492.6(s), 507.9(s), 519.8(w), 538.2(w), 576.1(vs), 658.7(s), 670.5(s), 712.4(s), 747.4(s), 761.9(s), 796.9(vs), 826.4(vs), 865.3(vs), 925.5(w), 953.3(m), 963.7(m), 981.4(s), 1012.1(s), 1033.9(w), 1046.3(w), 1088.2(m), 1100.6(m), 1117.1(w), 1133.2(s), 1143.3(s), 1170.8(s), 1206.8(vs), 1257.8(vs), 1291(s), 1343.8(s), 1370.5(s), 1402.4(vs), 1423.7(vs), 1435.9(vs), 1467(vs), 1490.3(m), 1507.1(m), 1547.7(s), 1586.8(vs), 1603.6(vvs), 1624.6(vs), 1653.1(vs), 1692.6(vs), 1709.2(vs), 1771.7(w), 1922.6(w), 1952.7(w), 2020.3(w), 2842.4(m), 2867.2(m), 2915.6(s), 2955.7(s), 3045.2(m), 3375.8(m). The last band was caused by traces of water in the air during the measurement procedure.

3.4. X-ray Data Collection and Structure Refinement

Crystallographic data of **1** were recorded on a STOE IPDS-2T diffractometer using $\text{Mo K}\alpha$ radiation ($\lambda = 0.71073$ Å) at 93 K using ω and φ scans. The crystal data and details of structure refinement are summarized in Table 4. X-Area was used as software for data collection and for cell refinement. X-RED was used for data reduction [32]. The

structure was solved by direct methods [33] and refined with full-matrix least-squares refinement on F^2 for all reflections with SHELXL [34]. The non-hydrogen atoms were refined anisotropically. All hydrogen atoms were included in the structure model in calculated positions and were refined as constrained to their bonded atoms.

Table 4. Crystal data and structure refinement for **1**.

Parameter	Value
Empirical formula	$C_{19}H_{23}NO_3Si$
Formula weight	341.47
Crystal system	Monoclinic
Space group	Ic
a	7.2030(6) Å
b	22.9842(14) Å
c	10.8946(12) Å
β	96.141(7)°
Volume	1793.3(3) Å ³
Z	4
Calculated density	1.265 g/cm ^{−3}
Absorption coefficient	0.147 mm ^{−1}
F(000)	728
Crystal size	0.550 × 0.480 × 0.300 mm
Theta range for data collection	3.257 to 28.925°
Limiting indices	−9 ≤ h ≤ 9, −30 ≤ k ≤ 30, −14 ≤ l ≤ 14
Reflections collected/unique	10,215/4380 [R(int) = 0.0330]
Completeness to theta = 25.242	99.6%
Absorption correction	Integration
Max. and min. transmission	0.9892 and 0.8775
Data/restraints/parameters	4380/2/221
Goodness-of-fit on F^2	1.036
Final R indices [I > 2sigma(I)]	$R_1 = 0.0317$, $wR_2 = 0.0819$
R indices (all data)	$R_1 = 0.0336$, $wR_2 = 0.0837$
Absolute structure parameter	0.05(7)
Largest diff. peak and hole	0.273 and −0.218 e.Å ^{−3}

CCDC 2291941 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via <https://www.ccdc.cam.ac.uk/structures> (accessed on 1 September 2023).

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/cryst13091407/s1>, Figure S1: Powder XRD, Figure S2: ¹H and ¹³C NMR spectra of the ligand N-(2-hydroxy-1-naphthylidene)leucine, Figure S3: ¹H and ¹³C NMR spectra of **1**, Figure S4: ²⁹Si NMR spectra of **1**, Figure S5: IR Spectra, Figure S6: UV-Vis spectra.

Author Contributions: U.B.: Formal Analysis, Investigation, Data Curation, Writing—Original Draft, Writing—Review and Editing, Visualization. S.F.: Formal Analysis, Investigation, Data Curation, Writing—Original Draft. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

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