Synthesis and structural characterization of neutral hexacoordinate silicon(IV) complexes containing salophen and thiocyanato-\(N\) ligands

Abstract: The reaction of the \(H_2\)salophen ligand, \(2,2'-(1E,1'\text{E})-(1,2\text{-phenylenebis(azanylylidene)})\text{bis}(\text{methanylylidene})\text{diphenol}\), with one molar equivalent of Si(NCS)\(_2\), MeSi(NCS)\(_3\) (13), or HMeSi(NCS)\(_2\) afforded neutral hexacoordinate silicon complexes, which have an-NCS bi-functionality (complex 1) and mono-functionality (complex 14). The reaction of Si(NCS)\(_4\) with the \(H_2\)salophen-type ligand1,1'-(1E,1'\text{E})-(1,2-phenylenebis(azanylylidene)) \text{bis(naphthalen-2-ol)} (H\text{Noph}), afforded the hexacoordinate silicon complex 15, which has an-NCS bi-functionality. Single-crystal X-ray structural and elemental analyses were used to characterize and confirm the structure of the starting material 13 and complexes 1, 14, and 15. The complexes were characterized in solution by \(^{1}H\), \(^{13}C\), and silicon-29 nuclear magnetic resonance (\(^{29}\text{Si NMR}\)) and in the solid state by \(^{29}\text{Si cross-polarization/magic angle spinning (CP/MAS) NMR}\). Because of the poor solubility of complex 1, it was only possible to characterize it in the solid state by \(^{13}C\) and \(^{29}\text{Si CP/MAS NMR}\) and in solution by \(^{1}\text{H NMR}\).

Keywords: \(^{29}\text{Si NMR spectroscopy}\); coordination chemistry; crystal structure; hexacoordinate silicon; salophen ligand; thiocyanato-\(N\) ligands.

Introduction

Bi-functional hexacoordinate silicon compounds played an important role as a building block for the preparation of novel oligosilanes containing hexacoordinate silicon backbones (Mucha et al., 1998, 1999; González-García et al., 2009) and as an initiator for ring-opening polymerization of \(\varepsilon\)-caprolactone (Lee et al., 2003). On the other hand, Si(NCS), and HMeSi(NCS), have proved to be useful starting materials for hypercoordinate silicon containing salen (Seiler et al., 2005; González-García et al., 2008, 2009, 2015), Schiff base (Seiler et al., 2007), and \(\beta\)-diketone ligands (Seiler et al., 2003; González-García et al., 2012) without adding an auxiliary base, such as triethylamine, which acts as a proton acceptor. In this work, the synthesis, single-crystal structure, and nuclear magnetic resonance (NMR) spectroscopy characterization of hexacoordinate silicon complexes containing salophen (1 and 14) or Noph (15) ligands and thiocyanato-\(N\) functional groups are reported. Complex 1 was previously prepared (Puri et al., 2009) starting from di-ethoxy di-thiocyanato-\(N\)silane, (EtO)\(_2\)Si(NCS)\(_2\), and a \(H_2\)salophen ligand (Scheme 1). However, the \(^{29}\text{Si NMR chemical shift at} -132\text{ ppm in a CDCl}_{3}/DMSO-D_6\) solution for complex 1 appears to correspond to a pentacoordinate silicon species rather than a hexacoordinate one. Indeed, complex 1 contains two-NCS functional groups of silicon, and it is well-known that the NCS ligand causes a large displacement to higher field signals in the silicon-29 (\(^{29}\text{Si}) NMR chemical shift (Seiler et al., 2003). No further studies were performed to confirm the solution \(^{29}\text{Si NMR results}, e.g.}^{29}\text{Si solid-state NMR study or single-crystal X-ray analysis. In this context, it is well known that the}^{29}\text{Si NMR chemical shift is used as a tool to determine the silicon coordination number. Chart 1 shows selected examples of silicon compounds containing-NCS functionalities whose structure have been confirmed by a single-crystal X-ray diffraction analysis, and Table 1 shows the}^{29}\text{Si chemical shift and the coordinating framework in the silicon center.}
Results and discussion

The starting material methyltri(thiocyanato-N)silane, 13, was synthesized in 70% yield by treatment MeSiCl3 with three molar equivalents of NH4SCN by following a methodology reported (Voronkov and Dolgov, 1954), using toluene instead of benzene (Scheme 2).

The treatment of the H2salophen with one molar equivalent of Si(NCS)4, MeSi(NCS)3 or HMeSi(NCS)2 afforded complexes 1, (salophen)Si(NCS)2, and 14, (salophen)
SiMe(NCS), with 59.0%, 45.0%, and 34.5% yields, respectively (Scheme 3).

Complex 15, (Noph)Si(NCS)$_2$, was obtained analogously as complex 1 using 1,1’-((1E,1’E)-(1,2-phenylenebis(azanylylidene))bis(methanylylidene))bis(naphthalen-2-ol) (H$_2$Noph) (Abd-Elzaher, 2000) as the salophen-type ligand and Si(NCS)$_4$ as the starting material (yield 67%). The complexes 1 and 14 precipitated from the respective reaction mixture as yellow solids. A suitable crystal for the X-ray diffraction analysis of the starting material 13 was obtained by placing a neat sample at 10°C for 24 h. Figure 1 shows the asymmetric unit of 13 in the crystal structure. The starting material 13 displayed a distorted tetrahedral geometry and crystallized in a monoclinic system with the space group $P_{2}1/n$. Selected bond lengths for 13 are given in their figure caption.

Single crystals of complexes 1 and 14 were obtained by slow cooling in acetonitrile from 80°C to 20°C, while for complex 15, a single-crystal was obtained by placing a saturated solution in a Schlenk tube at 55°C in an oven for one night. Complexes 1 and 14 crystallized in a monoclinic system with the space groups $P2_1/c$ and $P2_1/n$, respectively, while complex 15 crystallized in a triclinic system with the $P\bar{1}$ space group. The Si-coordination polyhedron for all complexes can be described as a distorted octahedron that maintains the-NCS functional group in a trans position in the coordination sphere with two-NCS functional groups for complexes 1 and 15 and one for complex 14. In the asymmetric unit of the crystal structure of complex 1, two molecules of acetonitrile solvent were present after the crystallization, and one molecule of CH$_3$CN showed weak C-H⋯O intermolecular interactions in the solid-state crystal structure (Figure 2), which can be best described as a larger type of electrostatic-based attractive force (Bakar...
et al., 2017). The angle O1-Si-O2 in complex 1 (89.9°) is significantly smaller with respect to that in complexes 14 (91.0°) and 15 (93.6°), this can be due to the attractive electrostatic force of the C-H 24a · · · O1 and C-H 24a · · · O2 weak intermolecular interactions. Complex 6 showed a similar weakly electrostatic interaction with another acetonitrile molecule in the asymmetric unit of the crystal structure (González-García et al., 2015).

Table 2: Summary of the crystallographic data and structure refinement results for compounds 1, 13–15.

<table>
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<th>Formula</th>
<th>1</th>
<th>13</th>
<th>14</th>
<th>15</th>
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<td>C26H20N6O2S2Si</td>
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<td>0.29/−0.30</td>
<td>0.28/−0.23</td>
<td>0.42/−0.29</td>
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* R(F) = Σ|Fo| − |Fc|)/Σ|Fo| for the observed reflections |F| > 2σ(F)).
* wR(F) = [Σ|w(F|² − F|²|)/Σ|w(F|²)]²/².
* S = (Σ|w(F|² − F|²|)/(n − p))²/²; (n = number of reflections, p = number of parameters).
As expected, for complex 14 (Figure 3), the Si-CH$_3$ and Si-NCS distances were significantly longer than the starting material 13. In all the hexacoordinated silicon compounds, the Si-NCS distances were significantly shorter than the Si-N dative bonds.

The salophen ligand adopted a bowl conformation in the equatorial plane, which was more remarkable for complex 15 (Figure 4).

Selected bonds lengths for complexes 1, 14, and 15 are given in their figure captions. The crystal data and experimental parameters used for the crystal structure analyses of compounds 1, and 13–15 are summarized in Table 2.

The $^{29}$Si NMR chemical shift in the solid-state is at -200.4 ppm (Figure 5), and -177.1 and -202.3 ppm for complexes 1, 14, and 15, respectively, which are consistent with a hexacoordinate silicon compound with SiN$_4$O$_2$ (1 and 15) and SiN$_3$O$_2$C (14) coordinating frameworks containing a salen-type ligand (González-García et al., 2009, 2015). The $^{13}$C CP/MAS NMR spectrum of complex 1 (see online-only supporting information) showed six signals, four in the aromatic region between 110 and 140 ppm and two for the imine carbons at approximately 160 ppm. The signals of NCS and the acetonitrile solvent were not detected, probably due to the coupling of the quadrupolar nucleus $^{14}$N and the loss of acetonitrile molecule during the cross-polarization/magic angle spinning (CP/MAS) NMR rotor spinning, respectively.

In the solution, $^{29}$Si NMR analysis, complexes 14 and 15 showed signals at -167.1 and -187.9 ppm, respectively, which are consistent with hexacoordinate silicon species (Figure 6). Thus, the silicon atom maintained the hexacoordination in the DMSO-D$_6$ solution for both complexes.

Because of poor solubility, it was only possible to study complex 1 in solution by $^1$H NMR in DMSO-D$_6$ and acetone-D$_6$. Freshly prepared samples of complex 1 in DMSO-D$_6$ and acetone-D$_6$ are shown in Figure 6. In both solvents, the $^1$H NMR spectrum showed the presence of two resonance signals for the imine proton of the salophen ligand, which suggest the presence of 1 in a possible mixture of trans- and cis-isomers with a ratio of 1.00:0.75 and 1.00:0.22 in DMSO-D$_6$ and acetone-D$_6$ solution. A similar behavior was observed for complexes 14 and 15 in a DMSO-D$_6$ solution. Nevertheless, decomposition was observed for all three complexes in the DMSO-D$_6$ solution in a few days, as shown by the $^1$H NMR analysis. In this context, further experimental and computational studies are needed to understand more clearly the nature of the possible stereoisomerization and decomposition process.

Figure 5: $^{29}$Si CP/MAS NMR spectrum of complex 1.

Figure 6: $^{29}$Si spectra of complexes (A) 14 and (B) 15 in a DMSO-D$_6$ solution.

Conclusions

In this work was confirmed that complexes 1, 14, and 15 are hexacoordinate silicon compounds containing two thiocyanato-N functional or methyl groups in the trans position, and the salophen ligand is located at the equatorial plane in the crystal structure. Nevertheless, complex 1 was poorly soluble, which made studying it by $^{29}$Si NMR in the solution state impossible. The analogous complexes 13 and 15 had good solubility in DMSO-D$_6$, which allowed us to confirm that both complexes maintained the hexacoordination of the silicon atom in a DMSO-D$_6$ solution by $^{29}$Si NMR spectroscopy. The synthetic potential of MeSi(NCS)$_3$ (13) as starting material for neutral hexacoordinate silicon
complex was demonstrated. Complexes 1, 14, and 15 have the potential to be used as starting materials for the preparation of new neutral hexacoordinate silicon(IV) complexes using a substitution or condensation reaction with thiocyanato-N functionalities.

Experimental

General procedures

The reaction and manipulations were carried out under an inert atmosphere of ultra-high-purity nitrogen (99.999% Infra, León, Guanajuato, Mexico) using standard Schlenk techniques. The organic solvents were dried and purified according to standard procedures and stored under nitrogen. Elemental analysis was carried out on a PerkinElmer 2400 Series II CHNS/O Elemental Analyzer at the Chemistry Department of the Universidad de Guanajuato, Guanajuato, Mexico.

Nuclear magnetic resonance (NMR) analysis

The 1H, 13C, and 29Si NMR spectra were recorded at 25°C on a 400-MHz, 500-MHz NMR Bruker Advance III (Karlsruhe, Germany) or 600 NMR Varian/Agilent NMR spectrometer (Oxford Instruments, Oxfordshire, UK). DMSO-D6 (99.9% D, Sigma-Aldrich, St Louis, MO, USA), CDCl3 (99.8% D, Sigma-Aldrich, St Louis, MO, USA) and acetone-D6 (99.9% D, Cambridge Isotope Laboratories, Inc., Tewksbury, MA, USA) were used as the solvents, and the chemical shifts (ppm) were determined relative to the internal residual signal of the solvent at 2.50, 7.26 and 2.05 ppm for 1H NMR and 39.05 and 70.0 ppm, respectively, for 13C NMR and 70.0 and 57.83 ppm for 29Si NMR. The 1H, 13C, and 29Si NMR spectra were recorded at 25°C on a Bruker Avance III HD 400 NMR (Karlsruhe, Germany) or Varian/Agilent 600 NMR spectrometer (Palo Alto, CA, USA). Rotors of ZrO2 were used with 4.0 mm of diameter, π/2 pulse of 2.0 μs, 1.0 ms of contact time, and 5.0 s of recycling delay. Chemical shifts (ppm) were determined relative to the external adamantane (38.4 ppm) and talc (−90.0 ppm) and were spun at 9 kHz and 7 kHz for 13C and 29Si CP/MAS, respectively.

Single-crystal X-ray diffraction analysis

Suitable single crystals for X-ray single-crystal structural analysis were obtained by slow cooling from 80°C to 20°C of a clear acetonitrile solution of the complexes 1 or 14. For complex 15, a saturated solution was placed at 55°C inside an oven for one night. A single crystal for the starting material 13 was obtained from placing a neat sample at 10°C for 24 h. The crystals were mounted in an inert oil (perfluoroalkyl ether) on a SuperNova single source at the offset EosS2 diffractometer (Rigaku Oxford Diffraction, Oxfordshire, UK). The crystals were kept at 150(2) (complex 1), 100(2) (13), or 293(2) K (complexes 14 and 15) during data collection. Using Olex2 (Dolomanov et al., 2009), the structures were solved with the ShelXT (Sheldrick, 2015a) structure solution program using direct methods and refined with the ShelXL (Sheldrick, 2015b) refinement package using least squares minimization.

Synthesis of complex 1

Trichloro(methyl)silane (99%, Sigma-Aldrich, St Louis, MO, USA) 15.0 g (100 mmol) was added at 20°C to a stirred suspension of ammonium thiocyanate (97.5%, Sigma-Aldrich, St Louis, MO, USA), 23.0 g (300 mmol) in 100 mL of toluene. The reaction was refluxed for 4 h; after that, the mixture was cooled to 20°C; the resulting precipitate was filtered off and discard. The solvent was distilled at normal pressure, and the residue was distilled in vacuo to give a colorless liquid (b.p. 84°–86°C, 0.2 mbar). Yield: 15.2 g (70 mmol) 70.0%. Anal. Calc. for C26H20N6O2S2Si (540.69): C 57.76; H, 3.73; N, 15.54; S, 11.86. Found: C, 57.83; H, 3.78; N, 15.58; S, 11.77%.

A 0.9-g (2.8 mmol) sample of H2salophen was added to a clear acetone-D6 solution of 0.5 g (1.7 mmol) of HMeSi(NCS)2 in acetonitrile (20 mL). The reaction mixture was left undisturbed at room temperature for 24 h to produce a yellow crystalline solid. The yellow crystals that formed were filtered off, washed with 20 mL of diethyl ether, and dried under vacuum (0.1 mbar, 4 h). Yield: 0.7 g (1.4 mmol), 45.0%. Mp: decomposition over 190°C.

Synthesis of complex 14

A 0.5-g (1.7 mmol) sample of H2salophen was added to a stirred suspension of 0.4 g (1.6 mmol) of Si(NCS)2 in acetonitrile (20 mL). The reaction mixture was stirred for 24 h at room temperature producing a yellow solid that was dissolved by the appropriated amount of acetonitrile, and the solution was heated to ca. 80°C to obtain a clear solution then filtered. By slowly cooling it to 20°C, the yellow crystals that were formed were filtered off, washed with 20 mL of diethyl ether, and dried under vacuum (0.1 mbar, 4 h). Yield: 0.5 g (0.9 mmol), 59.0%. Mps: decomposition over 270°C. Anal. Calc. for C26H20N6O2S2Si (540.69): C, 57.76; H, 3.73; N, 15.54; S, 11.86. Found: C, 57.83; H, 3.78; N, 15.58; S, 11.77%.

Synthesis of complex 14

A 0.9-g (2.8 mmol) sample of H2salophen was added to a clear acetone-D6 solution of 0.6 g (2.7 mmol) of MeSi(NCS), in acetonitrile (20 mL). The reaction mixture was left undisturbed at room temperature for 24 h to produce a yellow crystalline solid. The yellow crystals that formed were filtered off, washed with 20 mL of diethyl ether, and dried under vacuum (0.1 mbar, 4 h). Yield: 0.7 g (1.4 mmol), 45.0%. Mps: decomposition over 190°C. Anal. Calc. C14H16N3O3Si (415.54): C, 69.68; H, 4.10; N, 18.5; S, 6.22. Found: C, 69.72; H, 4.13; N, 8.27; S, 6.27%. 1H NMR (500 MHz, DMSO-D6) δ 9.44 (s, 2H), 4.20 (dd, J = 6.1, 3.4 Hz, 2H), 7.80 (d, J = 7.7 Hz, 2H), 7.21–7.65 (m, 4H), 7.08 (dd, J = 15.2, 7.8 Hz, 4H), −0.10 (s, 3H). 13C NMR (126 MHz, DMSO-D6) δ 160.37, 159.59, 138.72, 135.30, 134.19, 130.70, 130.19, 120.44, 119.86, 117.68, 117.66, 9.54. 29Si NMR (99 MHz, DMSO-D6) δ –174.6. Synthesis of complex 14

A 1.1-g (3.4 mmol) sample of H2salophen was added to a solution of 0.5 g (3.3 mmol) of HMeSi(NCS), in acetonitrile (20 mL). The reaction mixture was stirred for 24 h at room temperature to produce a yellow solid. The solution was heated to ca. 80°C and then filtered to obtain a clear solution. By slowly cooling it to 20°C, the yellow crystals were formed.
that formed were filtered off, washed with 20 mL of diethyl ether, and dried under vacuum (0.1 mbar, 4 h). Yield: 0.5 g (1.1 mmol), 34.5%.

Synthesis of complex 15

A 0.27 g (0.65 mmol) of H₂Noph was added to a stirred suspension of 0.16 g of Si(NCS)₂ (0.61 mmol) in 20 mL of acetonitrile. The reaction mixture was stirred for 24 h at room temperature to produce a solid orange precipitate, which was placed at 55°C inside an oven for one night. The orange crystals that formed were filtered off, washed with 10 mL of diethyl ether, and dried under vacuum (0.1 mbar, 4 h). Yield 0.23 g (0.41 mmol), 67%.

Mp: decompose over 200°C. Anal. Calc. C₃₀H₁₈N₄O₂S₂Si (558.71): C, 64.49; H, 3.25; N, 10.03; S, 11.48. Found: C, 64.33; H, 3.24; N, 10.12; S, 11.31%.

¹H NMR (500 MHz, DMSO-D₆), mixture of trans- and cis- isomers. δ 9.71 (s, 1H), 9.37 (s, 1H), 8.55 (t, J = 12.2 Hz, 1H), 8.26 (t, J = 10.0 Hz, 1H), 8.02 (d, J = 27.2 Hz, 2H), 7.92 (t, J = 9.8 Hz, 1H), 7.81 – 7.80 (m, 1H), 7.67 – 7.52 (m, 2H), 7.25 – 7.23 (m, 2H), 7.09 (d, J = 9.1 Hz, 1H), 6.83 (d, J = 8.9 Hz, 1H). ¹³C NMR (126 MHz, DMSO-D₆) δ 168.19, 163.12, 157.68, 152.02, 139.83, 138.39, 136.94, 133.63, 132.98, 131.74, 129.67, 129.26, 128.99, 128.76, 128.55, 128.42, 128.22, 128.75, 127.10, 126.93, 124.22, 124.01, 123.69, 121.70, 121.23, 120.70, 120.51, 119.83, 116.79, 109.20, 108.77. ²⁹Si NMR (99 MHz, DMSO-D₆) δ – 1879. ³⁴Si CP/MAS NMR δ – 202.3.

Supplementary data

Data from the X-ray crystallographic analyses of complexes 1, 14, and 15 and the starting material 13, MeSi(NCS)₂, are available in a crystallographic information file (CIF) under CCDC No: 1534652, 1584246, 1584247, and 1862266, respectively. Online-only supplement: ¹H, ¹³C{¹H} and ²⁹Si NMR spectra of 13; H NMR in the solution state and ¹³C NMR in the solid-state CP/MAS spectra of 1.

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References


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