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Syntheses and Reactivity of $Ph_3SiOReO_3$, $Mes_3GeOReO_3$, and $(O_3ReOPh_2SnOPh_2SnOH)_2$

Torsten Schoop, Herbert W. Roesky,* Mathias Noltemeyer, and Hans-Georg Schmidt

Institut für Anorganische Chemie der Universität Göttingen, Tammannstrasse 4, W-3400 Göttingen, Germany

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Summary: Re_2O_7 *reacts with* Ph_3SiOH, Mes_3GeOH, *and* Ph_3SnOH *to give* $Ph_3SiOReO_3$ *(1),* $Mes_3GeOReO_3$ *(2), and* $(O_3ReOPh_2SnOPh_2SnOH)_2$ *(3), respectively. In the reaction of* Mes_2BOH *with* Re_2O_7 *no rhenium-containing product was isolated. The silylperrhenate 1 reacts with 2,6-diisopropylphenyl isocyanate (ArNCO) in toluene to form* $Ph_3SiORe(NAr)_3$ *(5) in high yield. The reaction of 3 with ArNCO gives the unexpected product* $PhRe(NAr)_3$ *(6). 1 reacts with neat tert-butyl isocyanate to give* $Ph_3SiORe(N\text{-}t\text{-}Bu)_3$ *(8). The structures of* $(O_3ReOPh_2SnOPh_2SnOH)_2$ *(3),* $Ph_3SiORe(N\text{-}2,6\text{-}i\text{-}Pr_2C_6H_3)_3$ *(5),* $PhRe(N\text{-}2,6\text{-}i\text{-}Pr_2C_6H_3)_3$ *(6),* $Ph_3SiORe(N\text{-}2,6\text{-}Me_2C_6H_3)_3$ *(7), and* $Ph_3SiORe(N\text{-}t\text{-}Bu)_3$ *(8) were determined by X-ray diffraction.*

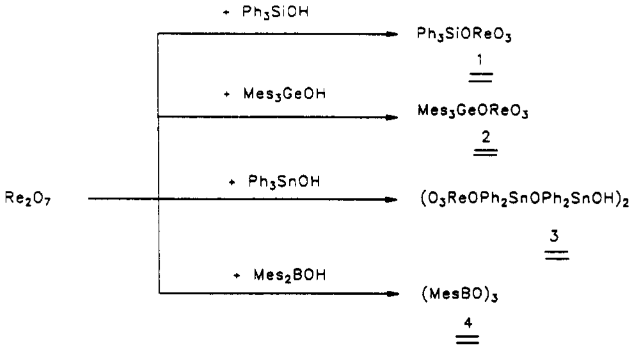
Introduction

Recently we became interested in reactions of Re_2O_7 with OH-substituted compounds[1,2] since the OH group plays an important role in the fixation of metal oxides on silica surfaces.[3] Our intention was to obtain a better understanding of the catalytic processes of such systems by investigating model reactions. In initial studies $(t\text{-}Bu)_2Si(OReO_3)_2$, prepared by reaction of $t\text{-}Bu_2Si(OH)_2$ with Re_2O_7, was found to be a model compound for reactions of metal oxides on silica surfaces by using our method for the insertion of imido groups.[4] In this paper we report our results on further studies of reactions of Re_2O_7 with group 14 and group 13 hydroxy derivatives.

Results and Discussion

1. Reactions of Re_2O_7 with Ph_3MOH (M = Si, Sn), Mes_3GeOH, and Mes_2BOH. We have carried out the reactions shown in Scheme I.

Scheme I



Ph_3SiOH reacts immediately with Re_2O_7 at room temperature in toluene, giving $Ph_3SiOReO_3$ (1) in good yield. However, the preparation of $Mes_3GeOReO_3$ (2; Mes = $2,4,6\text{-}Me_3C_6H_2$) requires longer reaction times. The expected product $Ph_3SnOReO_3$ could not be isolated from the reaction of Ph_3SnOH with Re_2O_7. The product actually isolated was $(O_3ReOPh_2SnOPh_2SnOH)_2$ (3), whose structure was determined by X-ray diffraction (Figure 1, Table I). It is remarkable that in the formation of 3 every tin atom has lost a phenyl substituent.

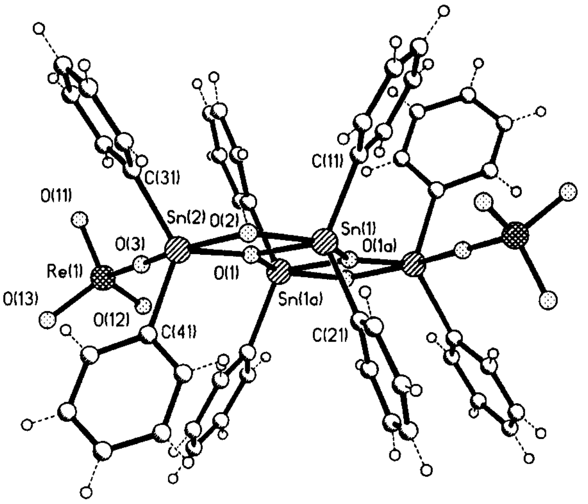


Figure 1. Molecular structure of 3.

Table I. Selected Bond Lengths (Å) and Angles (deg) of 3

Sn(1)–C(11)	2.116 (8)	Sn(1)–C(21)	2.100 (8)
Sn(1)–O(1)	2.036 (4)	Sn(1)–O(2)	2.169 (5)
Sn(1)–O(1A)	2.133 (4)	O(1)–Sn(2)	2.008 (5)
O(1)–Sn(1A)	2.133 (4)	O(2)–Sn(2)	2.114 (5)
Sn(2)–O(3)	2.238 (5)	O(3)–Re(1)	1.744 (5)
Re(1)–O(11)	1.711 (7)	Re(1)–O(12)	1.681 (7)
Re(1)–O(13)	1.695 (7)		
O(1)–Sn(1)–O(1A)	73.5 (2)	O(1)–Sn(1)–O(2)	72.4 (2)
Sn(1)–O(1)–Sn(2)	111.1 (2)	O(2)–Sn(1)–O(1A)	145.9 (2)
Sn(2)–O(1)–Sn(1A)	142.3 (2)	Sn(1)–O(1)–Sn(1A)	106.5 (2)
O(1)–Sn(2)–O(2)	74.2 (2)	Sn(1)–O(2)–Sn(2)	102.3 (2)
O(2)–Sn(2)–O(3)	155.6 (2)	O(1)–Sn(2)–O(3)	81.6 (2)
O(3)–Re–(1)–O(11)	110.8 (3)	Sn(2)–O(3)–Re(1)	169.0 (3)
O(11)–Re(1)–O(12)	107.4 (3)	O(3)–Re(1)–O(12)	109.8 (3)
O(11)–Re(1)–O(13)	108.3 (4)	O(3)–Re(1)–O(13)	109.7 (3)
		O(12)–Re(1)–O(13)	110.9 (4)

The structure of 3 involves a cluster with a framework of three four-membered rings that form a ladder-type structure.[5] The tin atoms have a distorted-trigonal-bipyramidal coordination sphere. The rhenium atoms have a distorted-tetrahedral geometry with angles ranging

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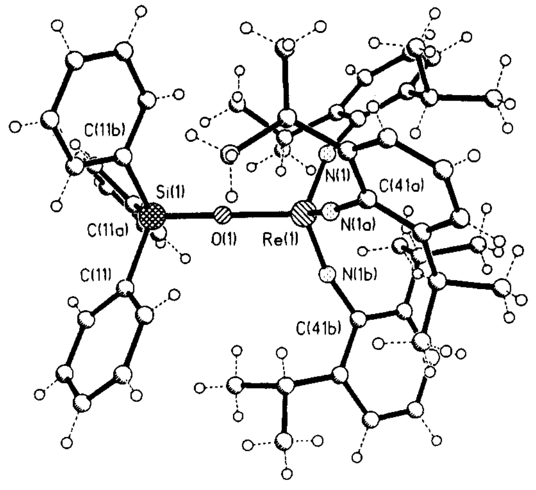


Figure 2. Molecular structure of **5**.

from 108 to 110°. A comparison with the polymeric $Me_3SnOReO_3$ shows the same coordination geometry of the rhenium atoms (108–110°).[6] The Sn(2)–O(2) bond distance (2.11 Å) is noticeably smaller than that found in $[HO(t\text{-}Bu)(Me_3SiCH_2)Sn)_2)O]_2$ (2.30 Å).[7] A possible explanation for this could be the electron-withdrawing properties of the ReO_3 group.

The reaction of Mes_2BOH with Re_2O_7 gave $(MesBO)_3$ (**4**) instead of the expected product Mes_2BOReO_3.[8]

2. Reactions of Group IV Perrhenates with Isocyanates. Compound **1** is isoelectronic with Ph_3PNReO_3, which we have reported previously[9] and which reacts with ArNCO (Ar = 2,6-i-$Pr_2C_6H_3$) in excess ArNCO, but not in toluene solution, to give $Ph_3PNRe(NAr)_3$.[10] The reaction of $Me_3SiOReO_3$ with ArNCO in toluene gave mixtures of products,[11] but **1** was found to react with ArNCO in toluene to give red crystals of $Ph_3SiORe(NAr)_3$ (**5**) in high yield.

The structure of **5** was determined by X-ray diffraction. $Ph_3SiORe(NAr)_3$ is not only isoelectronic but also isomorphous with $Ph_3PNRe(NAr)_3$. The Re=N bond lengths are comparable (1.741–1.759 Å). The molecule has a distorted-tetrahedral geometry with angles at the rhenium atom of 109–110°. The Re–O–Si angle is exactly linear (180°) (Figure 2, Table II). The Si(1)–Re(1) arrangement in the cell is in the inverted direction of that of Si(3)–Re(3). Furthermore, the substituents on Si(2) and Re(2) have an eclipsed conformation, whereas Si(1)–Re(1) and Si(3)–Re(3) have a staggered one.

Table II. Selected Bond Lengths (Å) and Angles (deg) of 5

Re(1)–O(1)	1.891 (10)	Re(1)–N(1)	1.741 (5)
Re(1)–N(1A)	1.741 (6)	Re(1)–N(1B)	1.742 (2)
Re(2)–O(2)	1.870 (8)	Re(2)–N(2)	1.759 (4)
Re(2)–N(2A)	1.758 (6)	Re(23)–N(2B)	1.760 (3)
Re(3)–O(3)	1.879 (11)	Re(3)–N(3)	1.759 (5)
Re(3)–N(3A)	1.759 (6)	Re(3)–N(3B)	1.759 (5)
Si(1)–O(1)	1.624 (11)	Si(2)–O(2)	1.626 (9)
Si(3)–O(3)	1.624 (11)		

O(1)–Re(1)–N(1A)	108.8 (2)	O(1)–Re(1)–N(1)	108.8 (2)
O(1)–Re(1)–N(1B)	108.8 (2)	N(1)–Re(1)–N(1A)	110.1 (2)
N(1A)–Re(1)–N(1B)	110.1 (2)	N(1)–Re(1)–N(1B)	110.1 (2)
O(2)–Re(2)–N(2A)	109.3 (2)	O(2)–Re(2)–N(2)	109.3 (2)
O(2)–Re(2)–N(2B)	109.3 (2)	N(2)–Re(2)–N(2A)	109.7 (2)
N(2A)–Re(2)–N(2B)	109.7 (2)	N(2)–Re(2)–N(2B)	109.6 (2)
O(3)–Re(3)–N(3A)	109.8 (2)	O(3)–Re(3)–N(3)	109.8 (2)
O(3)–Re(3)–N(3B)	109.8 (2)	N(3)–Re(3)–N(3A)	109.1 (2)
N(3A)–Re(3)–N(3B)	109.1 (2)	N(3)–Re(3)–N(3B)	109.1 (2)
Re(2)–O(2)–Si(2)	180.0 (1)	Re(1)–O(1)–Si(1)	180.0 (1)
		Re(3)–O(3)–Si(3)	180.0 (1)

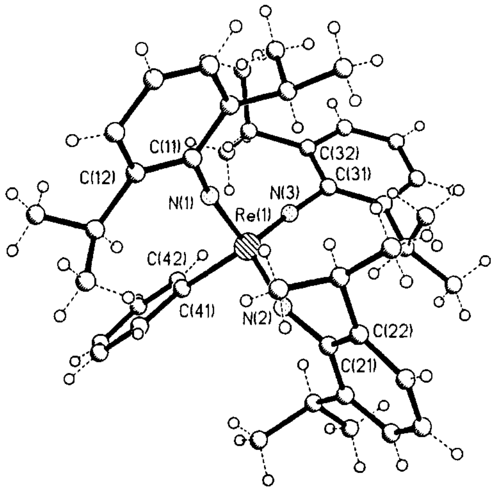


Figure 3. Molecular structure of **6**.

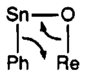
Table III. Selected Bond Lengths (Å) and Angles (deg) of 6

Re(1)–N(1)	1.761 (5)	Re(1)–N(2)	1.750 (5)
Re(1)–N(3)	1.750 (6)	Re(1)–C(41)	2.088 (8)

N(1)–Re(1)–N(2)	114.1 (2)	N(1)–Re(1)–N(3)	115.7 (3)
N(2)–Re(1)–N(3)	113.8 (2)	N(1)–Re(1)–C(41)	104.8 (3)
N(2)–Re(1)–C(41)	103.0 (3)	N(3)–Re(1)–C(41)	103.4 (3)
Re(1)–N(1)–C(11)	162.6 (4)		

In contrast to the chemistry of silylperrhenates, the chemistry of stannylperrhenates is unknown. In the reaction of **3** with ArNCO in toluene the expected stannylperrhenate imide was not formed. The isolated product was $PhRe(NAr)_3$ (**6**), whose structure was determined by X-ray diffraction (Figure 3, Table II). In **6** the coordination geometry at the rhenium atom is tetrahedrally distorted (103–115°). The Re=N bond lengths (1.750–1.761 Å) are similar to those in compound **5**. The Re–C bond length (2.088 Å) is comparable to that in $MesRe(N\text{-}t\text{-}Bu)_3$ (2.10 Å).[12]

The transfer of alkyl groups from organotin compounds to rhenium is well-known.[13] In the formation of **6** from **3** we most likely are dealing with an intramolecular Ph transformation from Sn to Re:



Recently, Herrmann et al. synthesized $PhReO_3$.[14] This compound decomposed slowly at room temperature and rapidly at higher temperatures. In contrast, the imide analogue $PhRe(NAr)_3$ (**6**) is quite stable.

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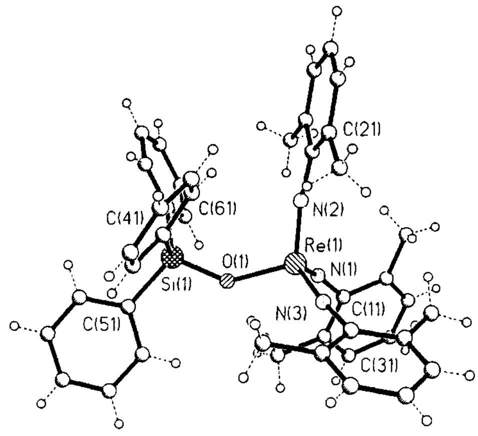

Figure 4. Molecular structure of 7.

Table IV. Selected Bond Lengths (Å) and Angles (deg) of 7

Re(1)-O(1)	1.902 (3)	Re(1)-N(1)	1.743 (3)
Re(1)-N(2)	1.750 (3)	Re(1)-N(3)	1.726 (3)
Si(1)-O(1)	1.652 (3)		
O(1)-Re(1)-N(2)	108.6 (1)	O(1)-Re(1)-N(1)	106.0 (1)
O(1)-Re(1)-N(3)	107.0 (1)	N(1)-Re(1)-N(2)	112.0 (1)
N(2)-Re(1)-N(3)	112.4 (2)	N(1)-Re(1)-N(3)	110.5 (2)
Re(1)-N(1)-C(11)	158.4 (3)	Re(1)-O(1)-Si(1)	139.7 (2)

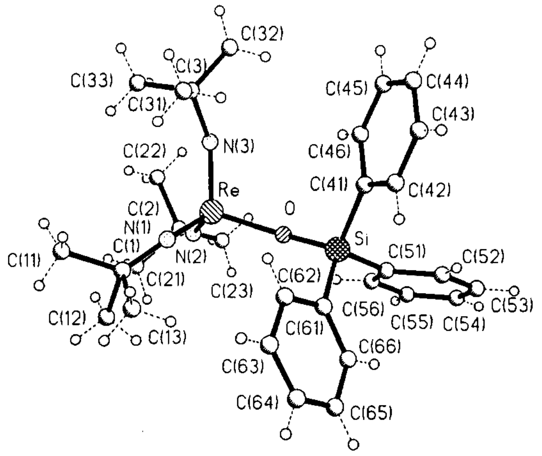

Figure 5. Molecular structure of 8.

In order to obtain information on whether 1 is a model compound for reactions of metal oxides on silica surfaces, we studied the reaction of 1 with 2,6-$Me_2C_6H_3NCO$ (Ar′NCO) is toluene. As indicated in eq 1, 7 was produced. Its structure was determined by X-ray diffraction (Figure 4, Table IV).

$$\underset{\mathbf{1}}{Ph_3SiOReO_3} + 3Ar'NCO \rightarrow \underset{\mathbf{7}}{Ph_3SiORe(NAr')_3} + 3CO_2 \quad (1)$$

Previously, conversion of Re═O by Re═N-*t*-Bu was possible only by silylamines.[15] Compound 1 also reacts with neat *t*-BuNCO at 80 °C to give $Ph_3SiORe(N\text{-}t\text{-}Bu)_3$ (8), whose structure also was determined by X-ray diffraction. The molecular structure of 8 is shown in Figure 5; selected bond lengths and angles are given in Table V.

Table V. Selected Bond Lengths (Å) and Angles (deg) of 8

Re-N(1)	1.749 (8)	Re-N(2)	1.723 (11)
Re-N(3)	1.735 (9)	Re-O	1.902 (8)
O-Si	1.620 (8)		
N(1)-Re-N(3)	108.7 (4)	N(1)-Re-N(2)	110.2 (6)
N(1)-Re-O	111.4 (5)	N(2)-Re-N(3)	111.2 (7)
N(3)-Re-O	109.0 (6)	N(2)-Re-O	106.4 (4)
Re-O-Si	152.2 (5)	Re-N(1)-C(1)	150.6 (10)

Experimental Section

All experiments were performed under a dry nitrogen atmosphere using standard Schlenk techniques or a drybox. Reagent grade toluene, hexane, and THF were distilled prior to use from benzophenone ketyl under nitrogen. Reagent grade acetonitrile was distilled from calcium hydride. Deuterated solvents were trap-to-trap distilled as follows: $CDCl_3$ and CD_3CN from CaH_2; d_8-THF from Na.

^1H and ^{29}Si NMR data are listed in ppm downfield from TMS. NMR spectra were recorded on a Bruker AM 250 spectrometer. IR spectra were recorded on a Perkin-Elmer Bio-Rad Digilab FTS-7. Microanalyses were performed by Beller Laboratory (Göttingen, Germany) or in our institute.

$Ph_3SiOReO_3$ (1). To a suspension of Re_2O_7 (3.00 g, 6.2 mmol) in 10 mL toluene was added a suspension of Ph_3SiOH (3.76 g, 13.6 mmol) in 40 mL toluene at room temperature. The resulting mixture was stirred for 1 h. The solvent was removed in vacuo, and the residue was recrystallized from hot hexane to afford 5.10 g (10.0 mmol, 81%) of a white solid: mp 110 °C; ^1H NMR ($CDCl_3$) δ 7.44–7.64 (m, 15 H, arom H); ^{29}Si NMR ($CDCl_3$) δ 3.08 (s); mass spectrum (EI) *m/e* 510 ([M^+], 68%); IR (KBr, cm^{-1}) ν 1121 vs, 1106 vs, 997 s, 964 vs, 953 vs, 904 vs. Anal. Calcd for $C_{18}H_{15}O_4ReSi$: C, 42.4; H, 3.0. Found: C, 42.3; H, 2.9.

$Mes_3GeOReO_3$ (2). To a suspension of Re_2O_7 (0.50 g, 1.0 mmol) in 5 mL toluene was added a suspension of Mes_3GeOH[16] (1.01 g, 2.3 mmol) in 15 mL toluene, and the resulting mixture was stirred for 16 h at room temperature. After the solvent was removed in vacuo, the residue was washed with hexane and then MeCN to give a white powder: dec pt 182 °C (1.28 g (1.9 mmol, 95%)); ^1H NMR (d^8-THF) δ 6.93 (s, 6 H, arom. H), 2.27 (s, 9 H, *p*-Me), 2.24 (s 18 H, *o*-Me); mass spectrum (EI) *m/e* 681 ([M^+], 4%), 560 ([M^+ – Me], 34%), 327 (MesGeO, 100%); IR (KBr, cm^{-1}) ν 950 vs, 894 vs. Anal. Calcd for $C_{27}H_{33}O_4GeRe$: C, 47.7; H, 4.9. Found: C, 47.5; H, 5.1.

$(O_3ReOPh_2SnOPh_2SnOH)_2$ (3). To a suspension of Re_2O_7 (1.00 g, 2.1 mmol) in 10 mL toluene was added a suspension of Ph_3SnOH (1.67 g, 4.5 mmol) in 20 mL toluene at room temperature. The resulting mixture was stirred for 1 day. During this time a white suspension was obtained. The solvent was evaporated in vacuo, and the white residue was dissolved in hot MeCN and left 12 h at room temperature. The solution was filtered through a glass fiber frit (G4). Concentration of the filtrate afforded 1.31 g (0.8 mmol, 38%) of octahedral colorless crystals: dec pt 332 °C; ^1H NMR (CD_3CN) δ 7.48–7.82 (m, 40 H, arom H0, 2.51 (s, 2 H, SnO*H*); mass spectrum (EI) *m/z* 77 ([Ph], 100%); IR (KBr, cm^{-1}) ν 1378 s, 997 vs, 957 vs, 908 vs, 860 vs, 727 vs, 694 vs, 664 s, 461 vs. Anal. Calcd for $C_{48}H_{42}O_{12}Re_2Sn_4$: C, 34.8; H, 2.5. Found: C, 35.8; H, 2.7.

$Ph_3SiORe(NAr)_3$ (5). A mixture consisting of 1 (0.51 g, 1.0 mmol) and 2,6-diisopropylphenyl isocyanate (0.78 mL, 3.3 mmol) in 15 mL toluene was heated at reflux for 16 h. Subsequently, the solvent was evaporated in vacuo, leaving a deep red oil which was extracted with 5 mL hot hexane, and the extract stored 2 days at room temperature to yield 0.84 g (0.9 mmol, 85%) of red crystals: mp 115 °C: ^1H ($CDCl_3$) δ 6.92–7.72 (m, 24 H, arom H), 3.25 (sept, $^3J_{HH}$ 7 Hz, 6 H, -$CH(CH_3)_2$), 0.84 (d, $^3J_{HH}$ 7 Hz, 36 H, -$CH(CH_3)_2$); ^{29}Si NMR ($CDCl_3$) δ –10.92 (s); mass spectrum (EI) *m/e* 987 ([M^+], 100%); IR (KBr, cm^{-1}) ν 1315 vs, 1261 vs, 1095 vs, 1019 vs. Anal. Calcd for $C_{54}H_{66}N_3OReSi$: C, 65.7; H, 6.7; N, 4.3. Found: C, 65.8; H, 6.9; N, 4.2.

$PhRe(NAr)_3$ (6). A mixture consisting of 3 (1.06 g, 0.7 mmol) and 2,6-diisopropylphenyl isocyanate (0.90 mL, 4.7 mmol) was heated at reflux in 20 mL toluene for 3 days. The solvent was removed in vacuo, the residue extracted with hot hexane (5 mL),

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and the extract stored for 1 day. The extract was filtered through a glass fiber filter. Concentration of this filtrate afforded dark red crystals in ca. 10% yield: mp 114 °C; ^{1}H NMR ($CDCl_3$) δ 7.18–7.62 (m, 24 H, arom H), 3.06 (sept, $^3J_{HH}$ 7 Hz, 6 H, $CH(CH_3)_2$), 1.04 (d, $^3H_{HH}$ 7 Hz, 36 H, $CH(CH_3)_2$); mass spectrum (EI) *m/z* 789 ([M$^+$], 100%); IR (KBr, cm^{-1}) ν 1378 s, 1258 m, 908 vs, 727 s. Anal. Calcd for $C_{42}H_{56}N_3Re$: C, 63.9; H, 7.1; N, 5.3. Found: C, 64.0; H, 7.6; N, 5.1.

$Ph_3SiORe(NAr')_3$ (7). A mixture of 1 (0.51 g, 1.0 mmol) and 2,6-dimethylphenyl isocyanate (0.46 mL, 3.3 mmol) was refluxed in toluene (20 mL) for 5 h. The solvent was evaporated in vacuo, the residue was extracted with hot hexane (5 mL), and the extract was stored for 12 h at room temperature to afford 0.30 g (0.4 mmol, 37%) of red-purple crystals: mp 119 °C; ^{1}H NMR ($CDCl_3$) δ 6.60–7.69 (m, 24 H, arom H), 2.13 (s, 18 H, $-CH_3$); ^{29}Si NMR ($CDCl_3$) δ −9.34 (s); mass spectrum (EI) *m/e* 819 ([M$^+$], 100%); IR (KBr, cm^{-1}) ν 1377 vs, 1315 vs, 1030 s, 907 s, 765 s, 699 s. Anal. Calcd for $C_{42}H_{42}N_3OReSi$: C, 61.6; H, 5.1; N, 5.1. Found: C, 61.6; H, 5.1; N, 4.9.

$Ph_3SiORe(N\text{-}t\text{-}Bu)_3$ (8). A suspension of 1 (0.51 g, 1.0 mmol) and 1 mL *tert*-butyl isocyanate was stirred for 10 h at 80 °C. After cooling to room temperature, and storing for 2 days, yellow crystals are obtained from the reaction mixture (0.50 g, 0.7 mmol, 74%): mp 96 °C; ^{1}H NMR ($CDCl_3$) δ 7.40–7.62 (m, 15 H, arom H), 1.21 (s, 27 H, $N-C(CH_3)_3$); ^{29}Si NMR ($CDCl_3$) δ −14.42 (s); mass spectrum (EI) *m/e* 675 ([M$^+$], 18%), 660 ([M$^+$ − Me], 100%); IR (KBr) ν 1357 vs, 1206 vs, 1116 vs, 975 vs, 803 vs, 711 vs. Anal. Calcd for $C_{30}H_{42}N_3OReSi$: C, 53.4; H, 6.2; N, 6.2. Found: C, 54.1; H, 6.4; N, 6.3.

Crystal Structure Solution and Refinement. Diffraction data were collected an a Siemens-Stoe AED2 four-circle instrument at room temperature with Mo Kα radiation, on-line profile fitting, and variable scan speeds. The structures were solved by Patterson methods (SHELX-86) and refined by full-matrix least-squares techniques (SHELX-76). Semiempirical absorption corrections were applied in all cases. Non-hydrogen atoms were refined anisotropically and hydrogen atoms inserted at calculated positions.

Crystal Data for 4: $C_{48}H_{40}O_{12}Sn_4Re_2$, M_r 1656.0, orthorhombic, space group *Pbca*, a = 17.844 (3) Å, b = 14.448 (2) Å, c = 19.837 (4) Å, V = 5114 (3) Å^3, Z = 4, ρ(calcd) = 2.15 mg/m^3, μ = 6.769 mm^{-1}, $F(000)$ = 3096, crystal size (mm) 0.7 × 0.7 × 0.8, 4493 measured reflections, 3337 unique reflections, 3083 observed reflections, goodness of fit 1.61, refinement converged with R = 0.037, R_w = 0.044, and maximum/minimum rest electron density +0.8/−1.8 e Å^{-3}.

Crystal Data for 5: $C_{54}H_{66}N_3OReSi$, M_r 987.4, trigonal, space group $P3$, a = 18.546 (3) Å, c = 12.721 (3) Å, V = 3789.6 (12) Å^3, Z = 3, ρ(calcd) = 1.30 mg/m^3, μ = 2.497 mm^{-1}, $F(000)$ = 1524, crystal size (mm) 0.5 × 0.5 × 0.5, 5205 measured reflections, 5204 unique reflections, 5082 observed reflections, goodness of fit 1.20, refinement converged with R = 0.027, R_w = 0.032, and maximum/minimum rest electron density +1.1/−1.1 e Å^{-3}.

Crystal Data for 6: $C_{42}H_{56}N_3Re$, M_r 798.1, orthorhombic, space group $P2_12_12_1$, a = 19.361 (2) Å, b = 19.417 (2) Å, c = 10.616 (1) Å, V = 3991.0 (7) Å^3, Z = 4, ρ(calcd) = 1.31 mg/m^3, μ = 3.11 mm^{-1}, $F(000)$ = 1616, crystal size (mm) 0.5 × 0.5 × 0.5, 5872 measured reflections, 5196 unique reflections, 4927 observed reflections, goodness of fit 1.70, refinement converged with R = 0.033, R_w = 0.038, and maximum/minimum rest electron density +0.7/−0.9 e Å^{-3}.

Crystal Data for 7: $C_{42}H_{42}N_3OReSi$, M_r 819.1, triclinic, space group $P1$, a = 9.7500 (10) Å, b = 11.015 (1) Å, c = 18.576 (3) Å, α = 75.87 (1)°, β = 83.66 (1)°, γ = 89.37 (1)°, V = 1922.6 (10) Å^3, Z = 2, ρ(calcd) = 1.415 mg/m^3, μ = 3.266 mm^{-1}, $F(000)$ = 824, crystal size (mm) 0.4 × 0.5 × 1.0, 5507 measured reflections, 5009 unique reflections, 4897 observed reflections, goodness of fit 2.43, refinement converged with R = 0.025, R_w = 0.033, and maximum/minimum rest electron density +0.6/−0.8 e Å^{-3}.

Crystal Data for 8: $C_{30}H_{42}N_3OReSi$, M_r 675.0, monoclinic, space group $P2_1$, a = 8.996 (3) Å, b = 21.257 (10) Å, c = 9.376 (3) Å, β = 114.04 (3)°, V = 1637.4 (8) Å^3, Z = 2, ρ(calcd) = 1.37 mg/m^3, μ = 3.771 mm^{-1}, $F(000)$ = 680, crystal size (mm) 0.5 × 0.5 × 0.6, 2819 measured reflections, 2682 unique reflections, 2632 observed reflections, goodness of fit 1.81, refinement converted with R = 0.032, R_w = 0.042, and maximum/minimum rest electron density +1.4/−1.3 e Å^{-3}.

Further details of the crystal structure determination are available upon request from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, W-7514 Eggenstein-Leopoldshafen 2, Germany, on quoting the deposition number CSD-56527, the authors, and the journal citation.

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Supplementary Material Available: For 3, 5, 6, 7, and 8 tables of atomic coordinates, displacement parameters, and bond distances and angles (43 pages). Ordering information is given on any current masthead page.

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