UNIVERSITÄT DUISBURG ESSEN

Offen im Denken

# Synthese und Reaktivität elektronenreicher Metallasilylene

ERGÄNZENDE INFORMATIONEN


vorgelegt von

**Juliane Schoening M. Sc.**

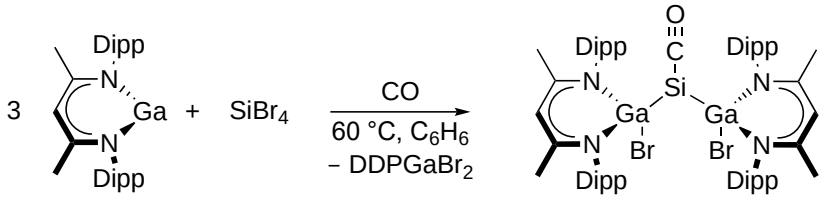
geboren in Essen

FAKULTÄT FÜR CHEMIE DER UNIVERSITÄT DUISBURG-ESSEN


ESSEN 2022

## 1. [DDP(Br)Ga]$_2$Si–CO 1

### 1.1. Synthese [DDP(Br)Ga]$_2$Si–CO **1**




Dipp = 2,6-$^i$Pr$_2$C$_6$H$_3$ **1**

Zunächst wurden DDPGa (1 g, 2.05 mmol) und $SiBr_4$ (0.238 g, 84.9 µL, 0.684 mmol) in 3 mL Benzol vorgelegt, für eine halbe Stunde bei Raumtemperatur gerührt und daraufhin auf −30 °C gekühlt. Die Lösung wurde entgast und mit CO gefüllt. Das Gemisch wurde daraufhin für zwei Tage unter Rühren auf 60 °C erhitzt. Nachdem das Gemisch auf Raumtemperatur gebracht wurde, wurde etwa ein Drittel des Lösungsmittels im Vakuum entfernt und zur Kristallisation bei 8 °C für einen Tag gelagert. Die entstandenen orangeroten Kristalle wurden von der Mutterlauge separiert und im Vakuum getrocknet.

**Ausbeute:** 610 mg (0.512 mmol, 75%)

**Smp.** 176-177 °C (Zersetzung)

**Elementaranalyse** von $C_{59}H_{82}N_4Br_2Ga_2OSi$ gefunden (berechnet): C 59.58 (59.52), H 6.92 (6.94), N 4.73 (4.71).

**IR**: ν 2961, 2926, 2865, 1945, 1520, 1457, 1430, 1379, 1319, 1256, 1173, 1106, 1023, 934, 869, 795, 761, 643, 533, 497, 441 cm$^{-1}$.

**UV-Vis** ($C_6H_6$): $\lambda_{max}$ 358, 444 nm.

**$^1$H NMR** ($C_6D_6$, 300 MHz): δ 7.15-6.97 (m, 12 H, $C_6H_3(^iPr)_2$), 4.94 (s, 2 H, γ-C*H*-), 3.77 (sept, $^3J_{HH}$ = 6.9 Hz, 4 H, -C*H*$(CH_3)_2$), 3.17 (sept, $^3J_{HH}$ = 6.9 Hz, 4 H, -C*H*$(CH_3)_2$), 1.49 (s, 12 H, ArNCC*H*$_3$), 1.33 (d, $^3J_{HH}$ = 6.6 Hz, 12 H, -CH(C*H*$_3)_2$), 1.25 (d, $^3J_{HH}$ = 6.9 Hz, 12 H, -CH(C*H*$_3)_2$), 1.21 (d, $^3J_{HH}$ = 6.6 Hz, 12 H, -CH(C*H*$_3)_2$), 0.97 (d, $^3J_{HH}$ = 6.6 Hz, 12 H, -CH(C*H*$_3)_2$) ppm.

**$^{13}$C NMR** ($C_6D_6$, 75 MHz): δ 206.5 (Si*C*O), 169.1 (ArN*C*CH$_3$), 146.5, 142.7, 141.5, 127.7, 125.7, 123.7 (*C*$_6$H$_3$), 98.7 (γ-*C*H-), 29.7, 29.0 (-*C*H$(CH_3)_2$), 28.2, 25.1, 24.4, 24.2 (-CH(*C*H$_3)_2$), 24.0 (ArNC*C*H$_3$) ppm.

**$^{29}$Si NMR** ($C_6D_6$, 59 MHz): δ –256.5 ppm.

## 1.2. Spektren [DDP(Br)Ga]$_2$Si–CO **1**

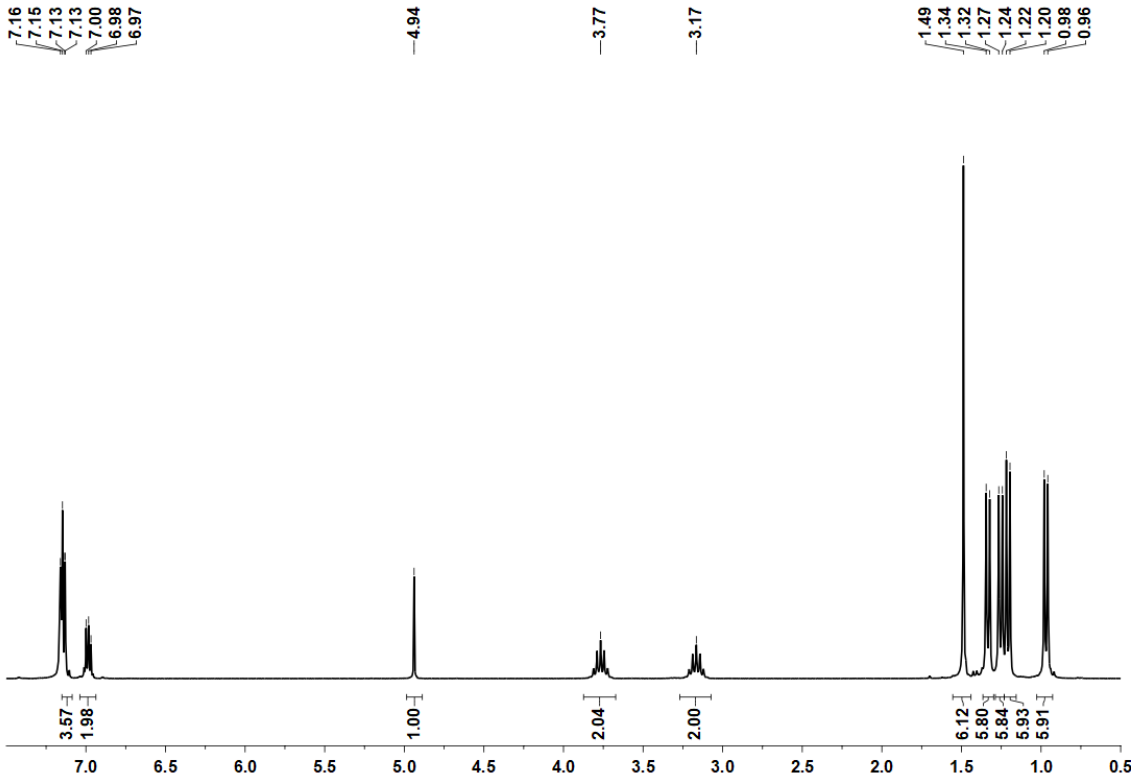



Abbildung 1: $^{1}$H-NMR-Spektrum von [DDP(Br)Ga]$_2$Si–CO **1** in $C_6D_6$.

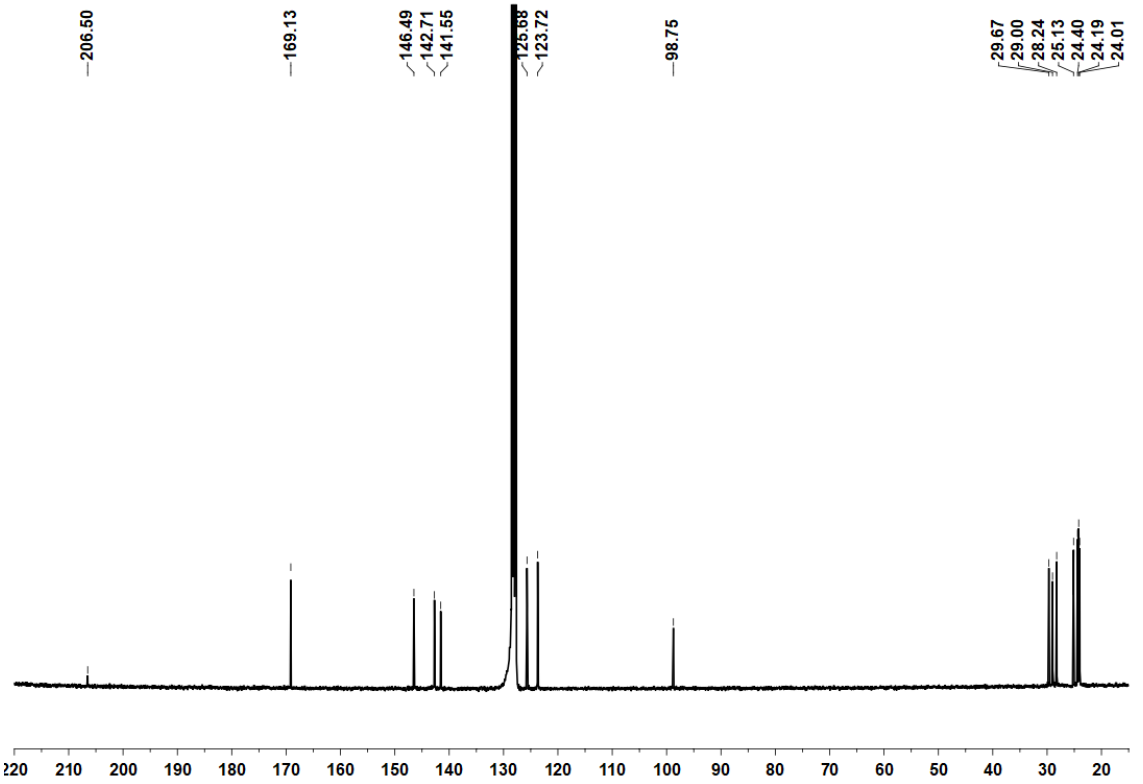



Abbildung 2: $^{13}$C-NMR-Spektrum von [DDP(Br)Ga]$_2$Si–CO **1** in $C_6D_6$.

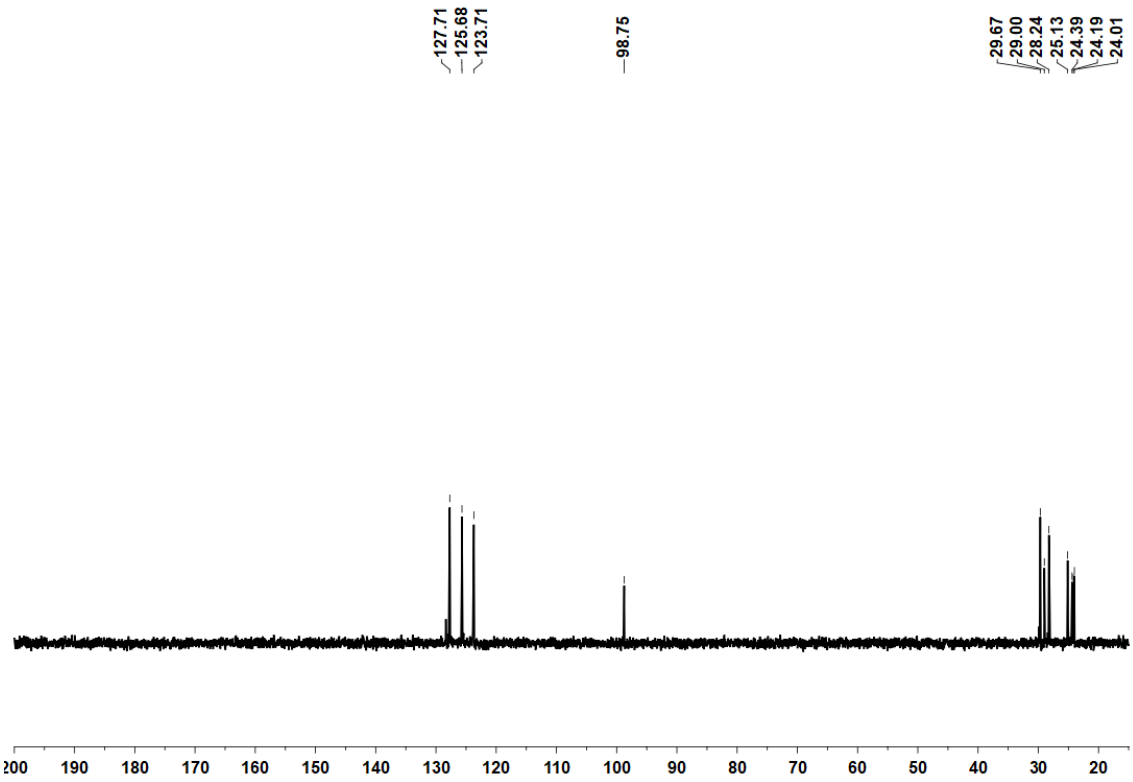



Abbildung 3: $^{13}$C-NMR-Spektrum (DEPT135) von [DDP(Br)Ga]$_2$Si–CO **1** in $C_6D_6$.

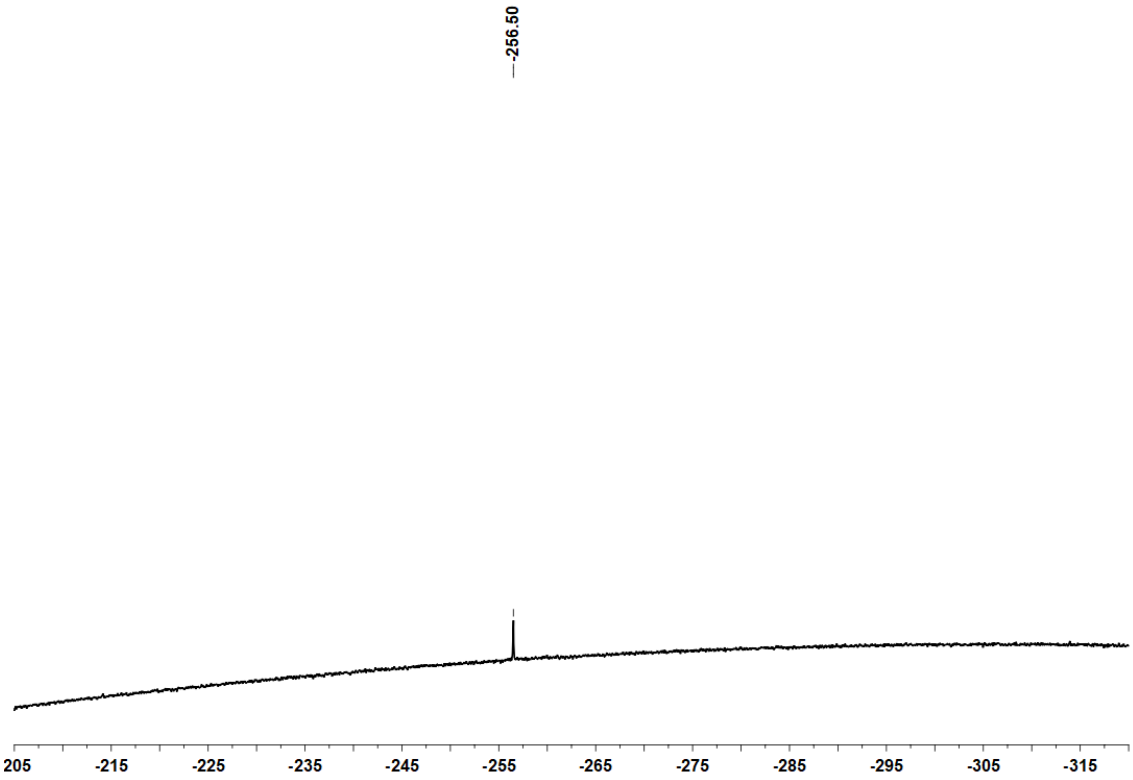



Abbildung 4: $^{29}$Si-NMR-Spektrum von [DDP(Br)Ga]$_2$Si–CO **1** in $C_6D_6$.

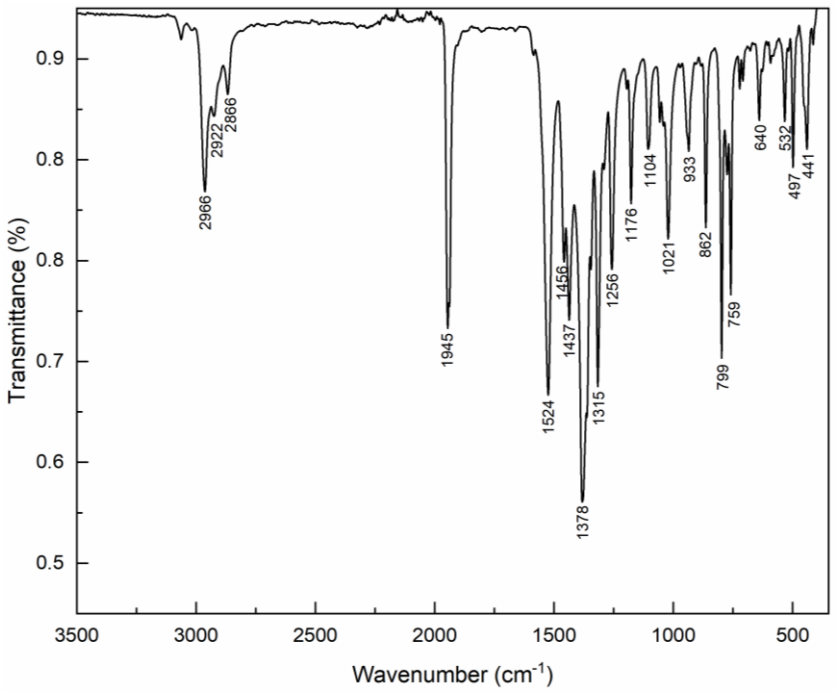



Abbildung 5: ATR-IR Spektrum von $[DDP(Br)Ga]_2Si–CO$ **1.**

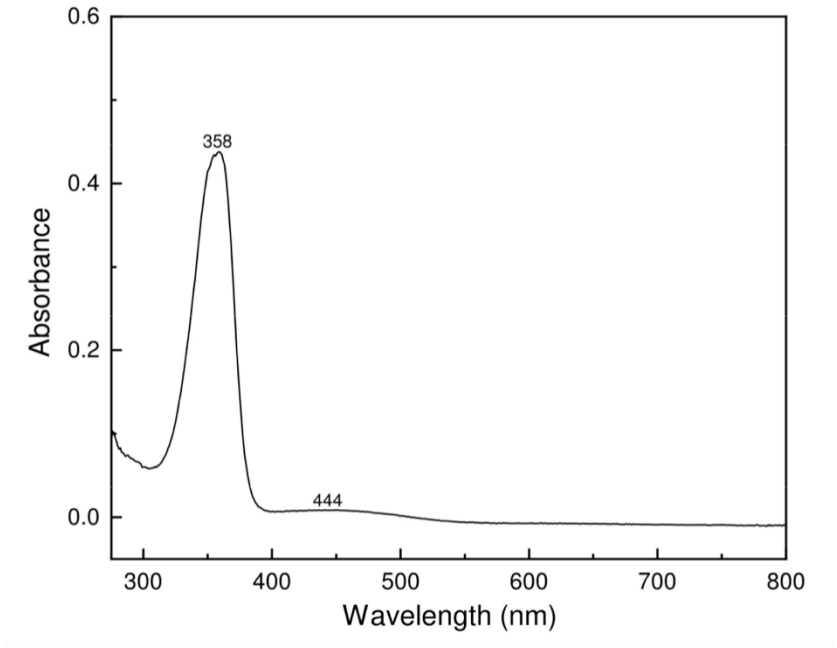



Abbildung 6: UV-Vis Spektrum einer 0.01194 mM Lösung von $[DDP(Br)Ga]_2Si–CO$ **1** in $C_6H_6$.

## 1.3. Kristallografische Daten $[DDP(Br)Ga]_2Si–CO$ **1**

Tabelle 1: Crystal structure data

| **Identification code** | **jus_132_a2_tw5** |
|---|---|
| Empirical formula | C59 H82 Br2 Ga2 N4 O Si |
| Formula weight | 1190.63 |
| Density (calculated) | 1.319 g·cm$^{-3}$ |
| *F*(000) | 2472 |
| Temperature | 100(2) K |
| Crystal size | 0.167 × 0.118 × 0.074 mm |
| Crystal colour | pale orange |
| Crystal description | tablet |
| Wavelength | 0.71073 Å |
| Crystal system | monoclinic |
| Space group | *C 2/c* |
| Unit cell dimensions | |
| *a* [Å] | 23.9753(15) |
| *b* [Å] | 10.8256(7) |
| *c* [Å] | 23.4438(16) |
| *α* [°] | 90 |
| *β* [°] | 99.840(4) |
| *γ* [°] | 90 |
| Volume | 5995.3(7) Å$^3$ |
| *Z* | 4 |
| Cell measurement reflections used | 4523 |
| Cell measurement *θ* min/max | 2.81°/28.32° |
| Diffractometer control software | BRUKER APEX2(v2009.5-1) |
| Diffractometer measurement device | Bruker D8 KAPPA II (APEX II detector) |
| Diffractometer measurement method | Data collection strategy APEX 2/COSMO |
| *θ* range for data collection | 1.724°- 33.344° |
| Completeness to *θ* = 25.242° | 99.4% |
| Completeness to $\theta_{max}$ = 33.344° | 91.1% |
| Index ranges | -36 ≤ *h* ≤ 36 |
| | 0 ≤ *k* ≤ 16 |
| | 0 ≤ *l* ≤ 36 |
| Computing data reduction | BRUKER APEX2(v2009.5-1) |
| Absorption coefficient | 2.292 mm$^{-1}$ |
| Absorption correction | Semi-empirical from equivalents |
| Computation absorption correction | TWINABS |
| Max./min. Transmission | 0.75/0.64 |
| $R_{merg}$ before/after correction | 0.0693/0.0616 |
| Computing structure solution | BRUKER APEX2(v2009.5-1) |
| Computing structure refinement | SHELXL-2017/1 (Sheldrick, 2017) |

| | |
|---|---|
| Refinement method | Full-matrix least-squares on $F^2$ |
| Reflections collected | 75013 |
| Independent reflections | 10218 |
| $R_{int}$ | 0.0542 |
| Reflections with $I > 2\sigma(I)$ | 7680 |
| Restraints | 156 |
| Parameter | 444 |
| GooF | 1.022 |
| Weighting details | $w = 1/[\sigma^2(F_{obs}^2) + (0.0418P)^2]$ |
| | where $P = (F_{obs}^2 + 2F_{calc}^2)/3$ |
| $R_1$ [$I > 2\sigma(I)$] | 0.0373 |
| $wR_2$ [$I > 2\sigma(I)$] | 0.0726 |
| $R_1$ [all data] | 0.0656 |
| $wR_2$ [all data] | 0.0789 |
| Absolute structure parameter | |
| Largest diff. peak and hole | 0.516/-0.669 |

Tabelle 2: Atomic coordinates ( × $10^4$) and equivalent isotropic displacement parameters (Å$^2$ × $10^3$) for jus_132_a2_tw5. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | *x* | *y* | *z* | $U_{eq}$ | | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|---|---|---|---|---|
| Br(1) | -312(1) | 4304(1) | 1452(1) | 27(1) | C(16) | -277(12) | 10817(17) | 1765(6) | 49(3) |
| Ga(1) | -15(1) | 6394(1) | 1591(1) | 21(1) | H(16A) | -542 | 11504 | 1664 | 74 |
| Si(1) | 0 | 7465(1) | 2500 | 23(1) | H(16B) | 112 | 11128 | 1821 | 74 |
| O(1) | 1257(2) | 7822(5) | 2715(2) | 56(1) | H(16C) | -348 | 10428 | 2124 | 74 |
| C(30) | 787(2) | 7604(5) | 2637(2) | 32(1) | C(17) | -263(7) | 10417(11) | 704(4) | 79(5) |
| N(1) | -499(1) | 7240(2) | 950(1) | 23(1) | H(17A) | -235 | 9758 | 424 | 118 |
| N(2) | 668(1) | 6418(2) | 1242(1) | 27(1) | H(17B) | 91 | 10892 | 770 | 118 |
| C(1) | -398(1) | 7048(2) | 415(1) | 28(1) | H(17C) | -579 | 10965 | 552 | 118 |
| C(2) | 91(1) | 6513(2) | 291(1) | 30(1) | C(15') | -382(7) | 9920(13) | 1083(4) | 42(3) |
| H(2) | 77 | 6245 | -97 | 36 | H(15') | -72 | 9304 | 1075 | 51 |
| C(3) | 606(1) | 6317(2) | 666(1) | 33(1) | C(16') | -238(10) | 10704(17) | 1616(7) | 61(5) |
| C(4) | -829(1) | 7474(3) | -99(1) | 42(1) | H(16D) | 115 | 11151 | 1605 | 92 |
| H(4A) | -886 | 8367 | -73 | 64 | H(16E) | -192 | 10175 | 1960 | 92 |
| H(4B) | -1189 | 7046 | -97 | 64 | H(16F) | -544 | 11297 | 1631 | 92 |
| H(4C) | -690 | 7283 | -459 | 64 | C(17') | -415(6) | 10787(10) | 550(5) | 59(2) |
| C(5) | 1107(2) | 6009(3) | 390(2) | 58(1) | H(17D) | -551 | 10319 | 195 | 88 |
| H(5A) | 1002 | 6071 | -31 | 88 | H(17E) | -38 | 11120 | 533 | 88 |
| H(5B) | 1233 | 5165 | 496 | 88 | H(17F) | -677 | 11467 | 584 | 88 |
| H(5C) | 1415 | 6589 | 526 | 88 | C(18') | 1227(5) | 6405(8) | 1662(3) | 21(2) |
| C(6) | -998(1) | 7953(2) | 1024(1) | 30(1) | C(19') | 1492(6) | 5298(13) | 1856(6) | 21(2) |
| C(7) | -1526(1) | 7371(3) | 974(1) | 33(1) | C(20') | 2058(7) | 5339(12) | 2145(9) | 38(3) |
| C(8) | -1992(1) | 8101(3) | 1038(2) | 46(1) | H(20') | 2256 | 4599 | 2266 | 45 |
| H(8) | -2354 | 7725 | 1010 | 56 | C(21') | 2318(5) | 6474(9) | 2248(5) | 41(2) |
| C(9) | -1941(2) | 9342(3) | 1138(2) | 56(1) | H(21') | 2704 | 6505 | 2428 | 49 |
| H(9) | -2266 | 9822 | 1170 | 68 | C(22') | 2030(3) | 7574(6) | 2096(3) | 40(2) |
| C(10) | -1419(2) | 9898(3) | 1195(2) | 54(1) | H(22') | 2211 | 8341 | 2201 | 47 |
| H(10) | -1387 | 10759 | 1272 | 65 | C(23') | 1477(2) | 7557(5) | 1790(3) | 30(1) |
| C(11) | -936(1) | 9222(3) | 1140(1) | 42(1) | C(27') | 1179(3) | 8771(5) | 1632(3) | 39(1) |
| C(12) | -1618(1) | 6004(3) | 859(1) | 33(1) | H(27') | 763 | 8594 | 1570 | 47 |
| H(12) | -1257 | 5644 | 773 | 39 | C(28') | 1305(5) | 9288(9) | 1069(4) | 60(3) |
| C(13) | -1763(1) | 5356(3) | 1395(2) | 43(1) | H(28A) | 1183 | 8695 | 757 | 89 |
| H(13A) | -2123 | 5675 | 1479 | 65 | H(28B) | 1712 | 9436 | 1104 | 89 |
| H(13B) | -1462 | 5512 | 1727 | 65 | H(28C) | 1100 | 10067 | 982 | 89 |
| H(13C) | -1796 | 4465 | 1324 | 65 | C(29') | 1295(4) | 9738(6) | 2123(4) | 58(2) |
| C(14) | -2090(1) | 5751(3) | 334(1) | 43(1) | H(29A) | 1700 | 9936 | 2201 | 87 |
| H(14A) | -2014 | 6225 | -1 | 64 | H(29B) | 1182 | 9403 | 2474 | 87 |
| H(14B) | -2456 | 6002 | 428 | 64 | H(29C) | 1078 | 10490 | 2006 | 87 |
| H(14C) | -2099 | 4868 | 242 | 64 | C(18) | 1256(5) | 6530(8) | 1481(3) | 23(2) |
| C(15) | -363(8) | 9855(13) | 1272(4) | 39(3) | C(19) | 1533(6) | 5468(12) | 1734(6) | 20(2) |
| H(15) | -71 | 9195 | 1378 | 47 | C(20) | 2080(6) | 5605(10) | 2056(8) | 26(2) |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| H(20) | 2263 | 4922 | 2264 | 32 | H(29E) | 1583 | 10067 | 1818 | 113 |
| C(21) | 2356(5) | 6738(8) | 2071(4) | 37(2) | H(29F) | 976 | 9549 | 1896 | 113 |
| H(21) | 2729 | 6818 | 2283 | 44 | C(24) | 1242(1) | 4118(2) | 1713(1) | 30(1) |
| C(22) | 2095(2) | 7741(6) | 1783(3) | 36(1) | H(24') | 886 | 4233 | 1425 | 36 |
| H(22) | 2294 | 8501 | 1792 | 44 | H(24) | 881 | 4169 | 1427 | 36 |
| C(23) | 1538(2) | 7667(5) | 1474(3) | 32(1) | C(25) | 1627(1) | 3196(3) | 1465(1) | 37(1) |
| C(27) | 1262(3) | 8820(7) | 1201(5) | 45(2) | H(25A) | 1425 | 2412 | 1379 | 55 |
| H(27) | 871 | 8593 | 1006 | 54 | H(25B) | 1971 | 3056 | 1749 | 55 |
| C(28) | 1585(4) | 9283(9) | 729(3) | 66(2) | H(25C) | 1729 | 3533 | 1109 | 55 |
| H(28D) | 1610 | 8618 | 451 | 99 | C(26) | 1088(1) | 3624(3) | 2274(1) | 44(1) |
| H(28E) | 1968 | 9536 | 908 | 99 | H(26A) | 882 | 2842 | 2198 | 67 |
| H(28F) | 1385 | 9989 | 528 | 99 | H(26B) | 849 | 4225 | 2431 | 67 |
| C(29) | 1207(5) | 9832(6) | 1615(5) | 75(3) | H(26C) | 1435 | 3485 | 2556 | 67 |
| H(29D) | 1025 | 10546 | 1403 | 113 | | | | | |

Tabelle 3: Anisotropic displacement parameters ($Å^2 \times 10^3$) for jus_132_a2_tw5. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Br(1) | 28(1) | 19(1) | 33(1) | 0(1) | 4(1) | 0(1) | C(15') | 46(4) | 25(3) | 48(7) | 3(5) | -13(6) | -5(3) |
| Ga(1) | 28(1) | 17(1) | 17(1) | -2(1) | 1(1) | 3(1) | C(16') | 90(12) | 38(7) | 44(8) | 12(5) | -25(7) | -29(8) |
| Si(1) | 34(1) | 19(1) | 15(1) | 0 | 2(1) | 0 | C(17') | 77(6) | 43(5) | 54(6) | 22(4) | 5(5) | -6(4) |
| O(1) | 51(3) | 87(4) | 30(2) | -14(2) | 9(2) | -35(3) | C(18') | 24(3) | 21(3) | 19(4) | -4(3) | 6(3) | -6(2) |
| C(30) | 35(3) | 50(3) | 13(2) | -1(2) | 4(2) | -8(3) | C(19') | 27(4) | 28(4) | 9(5) | -1(3) | 6(3) | -4(3) |
| N(1) | 28(1) | 23(1) | 17(1) | 2(1) | 2(1) | 1(1) | C(20') | 41(4) | 37(5) | 32(7) | 1(4) | -5(4) | -2(4) |
| N(2) | 24(1) | 21(1) | 36(1) | -9(1) | 2(1) | -1(1) | C(21') | 32(4) | 46(4) | 40(6) | -2(4) | -9(4) | -3(3) |
| C(1) | 37(1) | 26(1) | 20(1) | 1(1) | 0(1) | -11(1) | C(22') | 36(3) | 40(3) | 40(4) | -2(3) | -1(3) | -9(3) |
| C(2) | 46(2) | 28(1) | 20(1) | -8(1) | 14(1) | -10(1) | C(23') | 28(3) | 26(2) | 36(3) | -5(2) | 6(3) | -4(2) |
| C(3) | 39(1) | 20(1) | 44(2) | -7(1) | 21(1) | -4(1) | C(27') | 31(3) | 25(3) | 57(4) | -3(3) | -4(3) | -11(2) |
| C(4) | 53(2) | 48(2) | 23(2) | 7(1) | -5(1) | -13(2) | C(28') | 106(8) | 46(5) | 24(4) | 12(4) | 4(4) | 5(6) |
| C(5) | 56(2) | 43(2) | 88(3) | -6(2) | 46(2) | -1(2) | C(29') | 73(5) | 29(3) | 77(6) | -6(3) | 29(4) | 10(3) |
| C(6) | 34(1) | 28(1) | 28(1) | 6(1) | 5(1) | 9(1) | C(18) | 25(3) | 23(3) | 23(4) | -5(3) | 8(4) | -4(2) |
| C(7) | 30(1) | 36(1) | 33(2) | 6(1) | 5(1) | 9(1) | C(19) | 25(3) | 27(4) | 11(5) | -3(3) | 10(3) | -4(3) |
| C(8) | 40(2) | 49(2) | 51(2) | 4(2) | 11(2) | 15(1) | C(20) | 24(3) | 31(4) | 23(5) | -4(4) | -1(3) | 2(3) |
| C(9) | 51(2) | 51(2) | 67(3) | 4(2) | 9(2) | 29(2) | C(21) | 27(3) | 43(4) | 38(6) | -15(3) | 1(3) | -14(3) |
| C(10) | 61(2) | 32(2) | 66(2) | 0(2) | 2(2) | 16(2) | C(22) | 31(3) | 36(3) | 44(4) | -9(3) | 11(3) | -12(2) |
| C(11) | 49(2) | 26(1) | 49(2) | 4(1) | 3(2) | 8(1) | C(23) | 25(3) | 32(3) | 41(3) | -9(3) | 12(3) | -11(2) |
| C(12) | 25(1) | 35(1) | 38(2) | 7(1) | 6(1) | 3(1) | C(27) | 32(3) | 32(4) | 72(7) | 11(4) | 11(4) | -10(3) |
| C(13) | 34(2) | 50(2) | 48(2) | 16(2) | 14(2) | 4(1) | C(28) | 63(5) | 85(6) | 48(4) | 17(4) | 4(4) | -3(5) |
| C(14) | 29(1) | 47(2) | 51(2) | 5(2) | 6(1) | -1(1) | C(29) | 122(8) | 26(3) | 83(7) | 6(4) | 31(6) | 0(4) |
| C(15) | 75(7) | 22(3) | 22(4) | 6(3) | 14(6) | 6(4) | C(24) | 29(1) | 32(1) | 28(1) | -7(1) | -1(1) | 7(1) |
| C(16) | 92(8) | 26(4) | 30(6) | -8(4) | 9(7) | -1(4) | C(25) | 31(1) | 40(1) | 34(2) | -12(1) | -7(1) | 12(1) |
| C(17) | 136(13) | 68(7) | 31(5) | 11(4) | 14(6) | -60(8) | C(26) | 45(2) | 50(2) | 35(2) | 0(1) | 0(1) | 10(1) |

Tabelle 4: Bond lengths [Å] for jus_132_a2_tw5.

| | | | | | |
|---|---|---|---|---|---|
| Br(1)-Ga(1) | 2.3777(3) | C(7)-C(8) | 1.398(4) | C(21')-C(22') | 1.392(9) |
| Ga(1)-N(2) | 1.952(2) | C(7)-C(12) | 1.513(4) | C(22')-C(23') | 1.395(7) |
| Ga(1)-N(1) | 1.9623(19) | C(8)-C(9) | 1.366(5) | C(23')-C(27') | 1.511(8) |
| Ga(1)-Si(1) | 2.4203(5) | C(9)-C(10) | 1.373(5) | C(27')-C(28') | 1.511(10) |
| Si(1)-C(30)#1 | 1.865(6) | C(10)-C(11) | 1.395(5) | C(27')-C(29') | 1.546(10) |
| Si(1)-C(30) | 1.865(6) | C(11)-C(15) | 1.519(18) | C(18)-C(23) | 1.407(9) |
| O(1)-C(30) | 1.136(7) | C(11)-C(15') | 1.553(16) | C(18)-C(19) | 1.407(9) |
| N(1)-C(1) | 1.336(3) | C(12)-C(13) | 1.531(4) | C(19)-C(20) | 1.407(10) |
| N(1)-C(6) | 1.458(3) | C(12)-C(14) | 1.548(4) | C(19)-C(24) | 1.617(14) |
| N(2)-C(3) | 1.338(3) | C(15)-C(17) | 1.519(11) | C(20)-C(21) | 1.391(9) |
| N(2)-C(18) | 1.428(11) | C(15)-C(16) | 1.544(13) | C(21)-C(22) | 1.372(9) |
| N(2)-C(18') | 1.520(11) | C(15')-C(16') | 1.502(15) | C(22)-C(23) | 1.408(7) |
| C(1)-C(2) | 1.381(4) | C(15')-C(17') | 1.554(12) | C(23)-C(27) | 1.504(11) |
| C(1)-C(4) | 1.519(4) | C(18')-C(23') | 1.395(9) | C(27)-C(29) | 1.484(13) |
| C(2)-C(3) | 1.404(4) | C(18')-C(19') | 1.396(9) | C(27)-C(28) | 1.539(12) |
| C(3)-C(5) | 1.497(4) | C(19')-C(20') | 1.409(10) | C(24)-C(26) | 1.524(4) |
| C(6)-C(7) | 1.400(4) | C(19')-C(24) | 1.427(16) | C(24)-C(25) | 1.539(4) |
| C(6)-C(11) | 1.404(4) | C(20')-C(21') | 1.381(10) | | |

#1 -x,y,-z+1/2

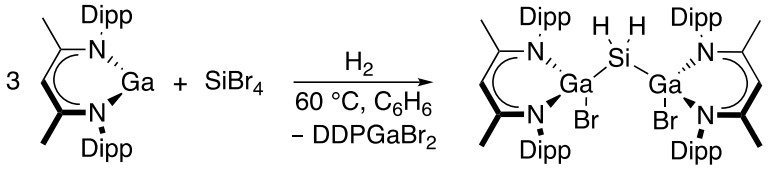
Tabelle 5: Bond angles [°] for jus_132_a2_tw5.

| | | | | | |
|---|---|---|---|---|---|
| N(2)-Ga(1)-N(1) | 95.86(9) | C(7)-C(6)-N(1) | 120.0(2) | C(20')-C(21')-C(22') | 121.8(10) |
| N(2)-Ga(1)-Br(1) | 102.09(6) | C(11)-C(6)-N(1) | 118.6(3) | C(21')-C(22')-C(23') | 120.5(7) |
| N(1)-Ga(1)-Br(1) | 102.37(6) | C(8)-C(7)-C(6) | 117.7(3) | C(18')-C(23')-C(22') | 117.3(7) |
| N(2)-Ga(1)-Si(1) | 118.39(6) | C(8)-C(7)-C(12) | 118.5(3) | C(18')-C(23')-C(27') | 123.8(6) |
| N(1)-Ga(1)-Si(1) | 111.58(6) | C(6)-C(7)-C(12) | 123.8(2) | C(22')-C(23')-C(27') | 118.9(5) |
| Br(1)-Ga(1)-Si(1) | 122.37(2) | C(9)-C(8)-C(7) | 121.6(3) | C(28')-C(27')-C(23') | 112.2(7) |
| C(30)#1-Si(1)-C(30) | 170.7(4) | C(8)-C(9)-C(10) | 120.1(3) | C(28')-C(27')-C(29') | 111.2(6) |
| C(30)#1-Si(1)-Ga(1) | 91.47(15) | C(9)-C(10)-C(11) | 121.2(3) | C(23')-C(27')-C(29') | 112.9(6) |
| C(30)-Si(1)-Ga(1) | 92.97(15) | C(10)-C(11)-C(6) | 117.9(3) | C(23)-C(18)-C(19) | 121.8(10) |
| C(30)#1-Si(1)-Ga(1)#1 | 92.97(15) | C(10)-C(11)-C(15) | 118.8(6) | C(23)-C(18)-N(2) | 120.9(8) |
| C(30)-Si(1)-Ga(1)#1 | 91.47(15) | C(6)-C(11)-C(15) | 122.9(6) | C(19)-C(18)-N(2) | 117.3(8) |
| Ga(1)-Si(1)-Ga(1)#1 | 122.73(4) | C(10)-C(11)-C(15') | 119.2(6) | C(20)-C(19)-C(18) | 118.1(12) |
| O(1)-C(30)-Si(1) | 172.7(6) | C(6)-C(11)-C(15') | 121.6(6) | C(20)-C(19)-C(24) | 118.0(8) |
| C(1)-N(1)-C(6) | 118.6(2) | C(7)-C(12)-C(13) | 110.4(3) | C(18)-C(19)-C(24) | 123.8(10) |
| C(1)-N(1)-Ga(1) | 117.91(17) | C(7)-C(12)-C(14) | 112.3(2) | C(21)-C(20)-C(19) | 120.1(11) |
| C(6)-N(1)-Ga(1) | 123.16(16) | C(13)-C(12)-C(14) | 109.6(2) | C(22)-C(21)-C(20) | 120.7(9) |
| C(3)-N(2)-C(18) | 109.6(4) | C(11)-C(15)-C(17) | 106.0(10) | C(21)-C(22)-C(23) | 121.4(7) |
| C(3)-N(2)-C(18') | 125.9(4) | C(11)-C(15)-C(16) | 117.1(15) | C(18)-C(23)-C(22) | 117.3(7) |
| C(3)-N(2)-Ga(1) | 117.67(17) | C(17)-C(15)-C(16) | 111.4(11) | C(18)-C(23)-C(27) | 123.6(7) |
| C(18)-N(2)-Ga(1) | 132.7(3) | C(16')-C(15')-C(11) | 106.3(12) | C(22)-C(23)-C(27) | 118.8(5) |
| C(18')-N(2)-Ga(1) | 116.0(3) | C(16')-C(15')-C(17') | 107.5(11) | C(29)-C(27)-C(23) | 114.6(9) |
| N(1)-C(1)-C(2) | 124.1(2) | C(11)-C(15')-C(17') | 115.9(10) | C(29)-C(27)-C(28) | 110.0(7) |
| N(1)-C(1)-C(4) | 119.3(2) | C(23')-C(18')-C(19') | 122.8(10) | C(23)-C(27)-C(28) | 109.7(7) |
| C(2)-C(1)-C(4) | 116.6(2) | C(23')-C(18')-N(2) | 115.5(7) | C(19')-C(24)-C(26) | 105.4(5) |
| C(1)-C(2)-C(3) | 128.2(2) | C(19')-C(18')-N(2) | 121.4(9) | C(19')-C(24)-C(25) | 114.4(6) |
| N(2)-C(3)-C(2) | 123.7(2) | C(18')-C(19')-C(20') | 118.5(12) | C(26)-C(24)-C(25) | 110.3(2) |
| N(2)-C(3)-C(5) | 119.6(3) | C(18')-C(19')-C(24) | 122.8(11) | C(26)-C(24)-C(19) | 117.2(5) |
| C(2)-C(3)-C(5) | 116.6(3) | C(20')-C(19')-C(24) | 118.2(10) | C(25)-C(24)-C(19) | 108.3(5) |
| C(7)-C(6)-C(11) | 121.4(3) | C(21')-C(20')-C(19') | 118.7(12) | | |

#1 -x,y,-z+1/2

## 2. [DDP(Br)Ga]$_2$SiH$_2$ 2

### 2.1. Synthese [DDP(Br)Ga]$_2$SiH$_2$ **2**




Dipp = 2,6-$^i$Pr$_2$C$_6$H$_3$ **2**

Variante A: Zunächst wurden DDPGa (0.118 g, 0.242 mmol) und SiBr$_4$ (0.028 g, 10 μL, 0.0805 mmol) in 0.7 mL Benzol-*d*$_6$ vorgelegt und auf –30 °C gekühlt. Die Lösung wurde entgast und mit H$_2$ gefüllt. Das Gemisch wurde daraufhin für zwei Tage auf 60 °C erhitzt. Nachdem das Gemisch auf Raumtemperatur gebracht wurde, wurde das Lösungsmittel im Vakuum entfernt und zur Kristallisation 0.5 mL Hexan hinzugefügt und bei 4 °C für sieben Tage gelagert. Die entstandenen farblosen Kristalle wurden von der Mutterlauge separiert und im Vakuum getrocknet.

Variante B: Es wurde [DDP(Br)Ga]$_2$Si–CO **1** (0.03 g, 0.025 mmol) in einem Young-NMR vorgelegt und mit einer Atmosphäre H$_2$ gefüllt. Die Reaktionsmischung wurde für zwei Tage auf 60 °C erhitzt, wobei [DDP(Br)Ga]$_2$SiH$_2$ **2** entsteht. Das Lösungsmittel wurde im Vakuum entfernt und 0.3 mL Hexan zur Kristallisation hinzugefügt und bei 4 °C gelagert. Nach sieben Tagen konnten farblose Kristalle isoliert werden.

**Ausbeute:** 67 mg (0.058 mmol, 69%)

**Smp.** 240-241 °C

**Elementaranalyse** von $C_{58}H_{84}N_4Br_2Ga_2Si$ gefunden (berechnet): C 59.88 (59.81), H 7.22 (7.27), N 4.79 (4.81).

**IR**: ν 2961, 2922, 2866, 2112, 1525, 1457, 1437, 1378, 1314, 1259, 1176, 1101, 1021, 937, 902, 862, 794, 755, 683, 636, 532, 497, 441, 406 cm$^{-1}$.

**$^1$H NMR** (C$_6$D$_6$, 300 MHz): δ 7.15-6.98 (m, 12 H, C$_6$*H*$_3$($^i$Pr)$_2$), 4.89 (s, 2 H, γ-C*H*-), 3.71 (sept, $^3J_{HH}$ = 6.6 Hz, 4 H, -C*H*(CH$_3$)$_2$), 3.05 (sept, $^3J_{HH}$ = 6.6 Hz, 4 H, -C*H*(CH$_3$)$_2$), 2.12 (s, 1 H, Si*H*$_2$), 1.52 (s, 12 H, ArNCC*H*$_3$), 1.24 (d, $^3J_{HH}$ = 6.6 Hz, 12 H, -CH(C*H*$_3$)$_2$), 1.19 (d, $^3J_{HH}$ = 6.9 Hz, 12 H, -CH(C*H*$_3$)$_2$), 1.14 (d, $^3J_{HH}$ = 6.6 Hz, 12 H, -CH(C*H*$_3$)$_2$), 0.97 (d, $^3$J$_{HH}$ = 6.6 Hz, 12 H, -CH(C*H*$_3$)$_2$) ppm.

**$^{13}$C NMR** (C$_6$D$_6$, 75 MHz): δ 168.6 (ArNCCH$_3$), 146.4, 142.5, 141.2, 127.4, 125.6, 123.5 (*C*$_6$H$_3$), 98.1 (γ-*C*H-), 29.5, 29.0 (-*C*H(CH$_3$)$_2$), 28.1, 25.1, 24.4, 23.8 (-CH(*C*H$_3$)$_2$), 23.6 (ArNC*C*H$_3$) ppm.

**$^{29}$Si NMR** (C$_6$D$_6$, 59 MHz): δ –130.6 ppm.

## 2.2. Spektren $[DDP(Br)Ga]_2SiH_2$ **2**

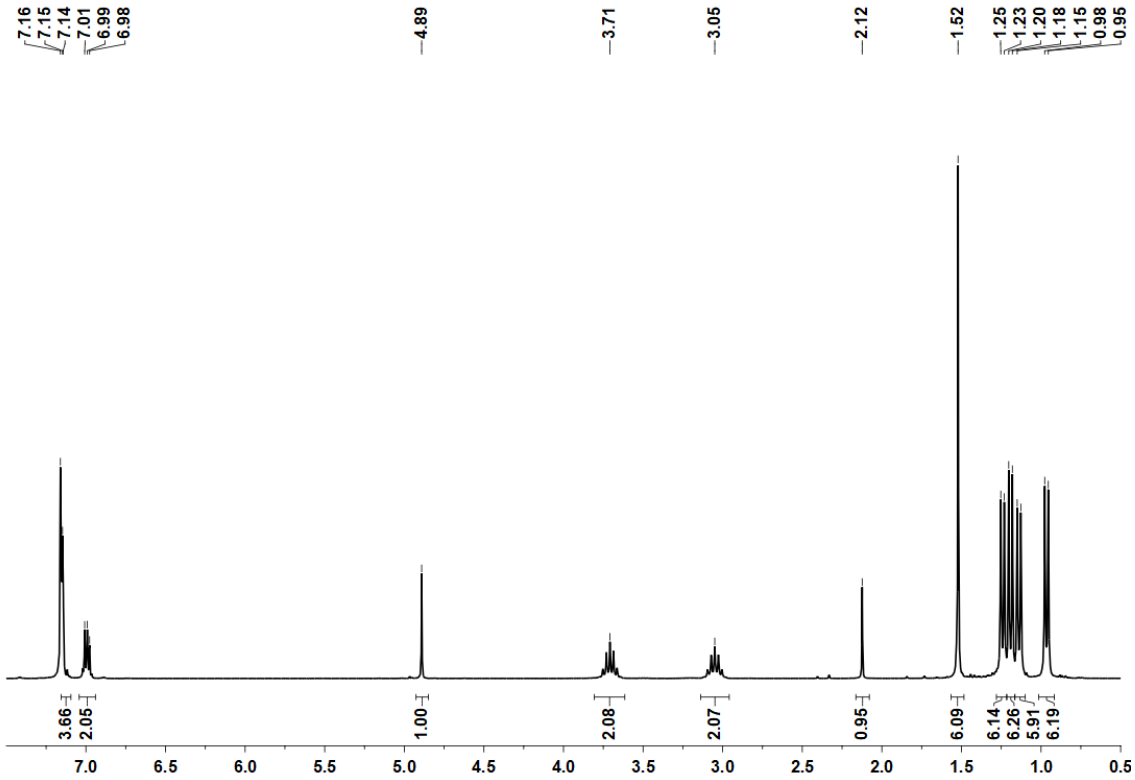



Abbildung 7: $^{1}$H-NMR-Spektrum von $[DDP(Br)Ga]_2SiH_2$ **2** in $C_6D_6$.

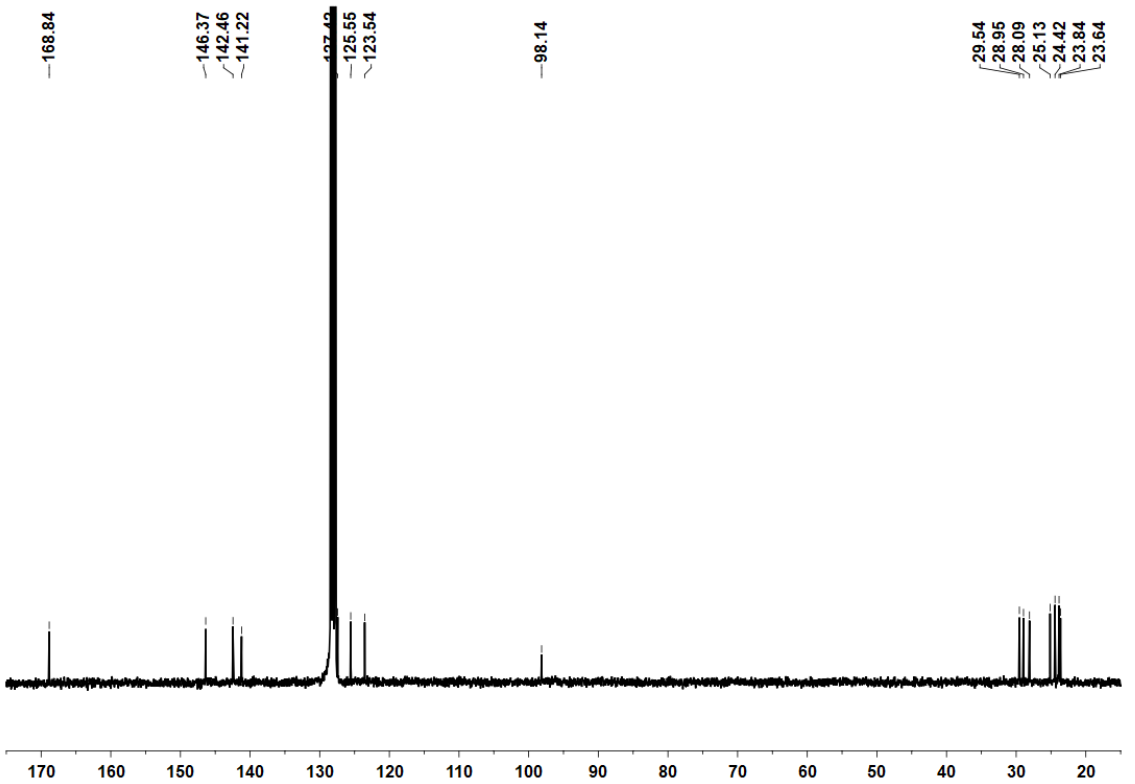



Abbildung 8: $^{13}$C-NMR-Spektrum von $[DDP(Br)Ga]_2SiH_2$ **2** in $C_6D_6$.

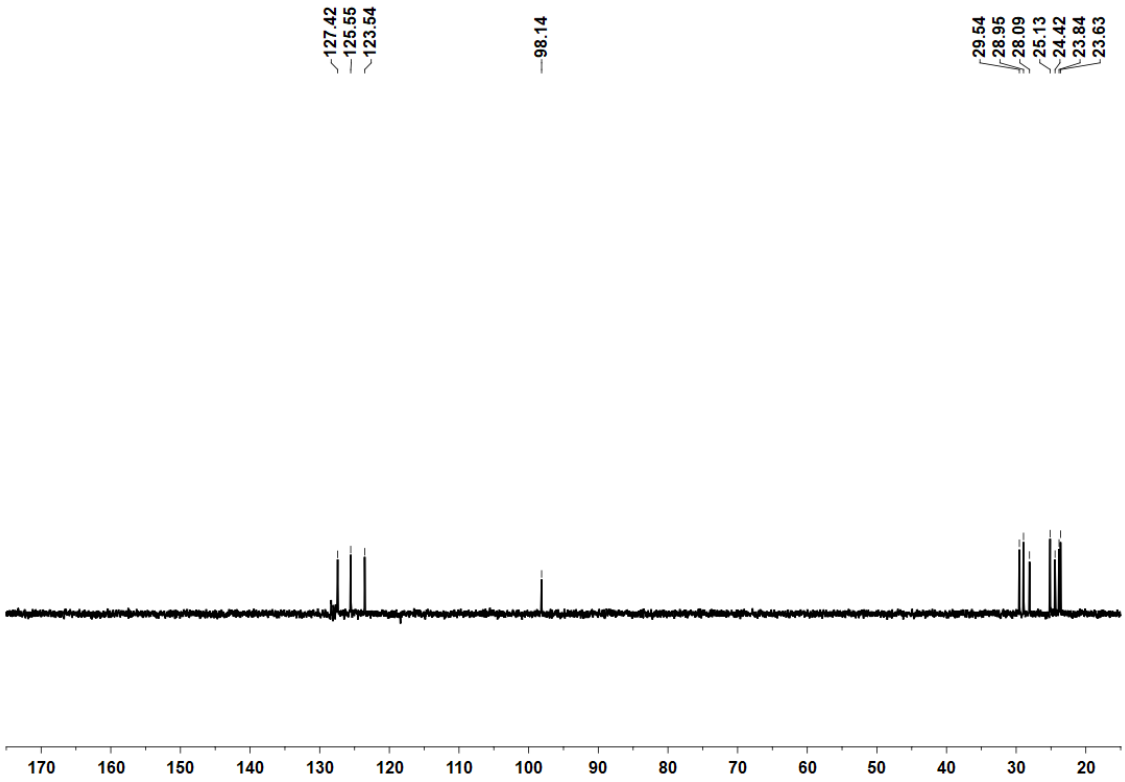



Abbildung 9: $^{13}$C-NMR-Spektrum (DEPT135) von $[DDP(Br)Ga]_2SiH_2$ **2** in $C_6D_6$.

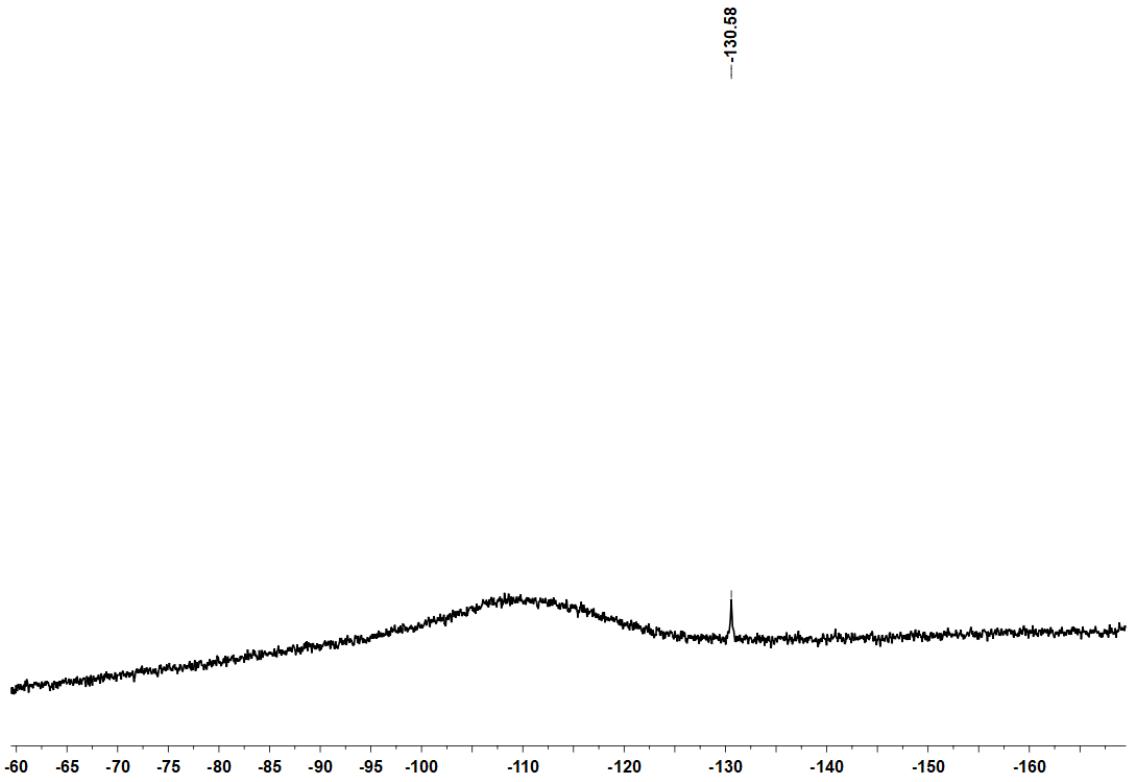



Abbildung 10: $^{29}$Si-NMR-Spektrum von $[DDP(Br)Ga]_2SiH_2$ **2** in $C_6D_6$.

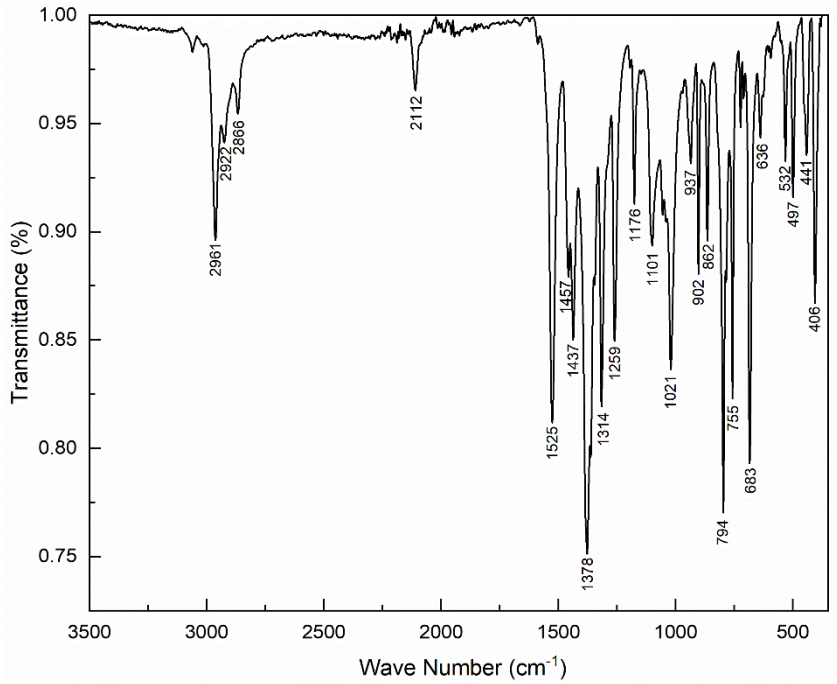
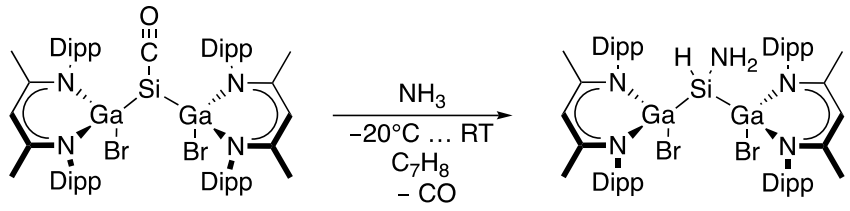



Abbildung 11: ATR-IR Spektrum von $[DDP(Br)Ga]_2SiH_2$ **2**.

2.3. Kristallografische Daten $[DDP(Br)Ga]_2SiH_2$ **2**

## 3. [DDP(Br)Ga]$_2$(H)NH$_2$ 3

### 3.1. Synthese [DDP(Br)Ga]$_2$(H)NH$_2$ **3**




**1** **3**

Dipp = 2,6-$^i$Pr$_2$C$_6$H$_3$

Es wurden 100 mg [DDP(Br)Ga]$_2$Si–CO **1** (0.084 mmol) in einem Kolben vorgelegt und in 2 mL Toluol gelöst. Die Lösung wurde eingefroren und die Atmosphäre entfernt. Daraufhin wurde eine Atmosphäre Ammoniak bei –35 °C hinzugegeben. Dabei entfärbte sich langsam die orangene Lösung. Nach etwa 15 Minuten Rühren bei –35 °C und 15 Minuten bei Raumtemperatur wurde das Lösungsmittel im Vakuum entfernt und 0.8 mL Hexan hinzugegeben. Dabei löste sich nur ein kleiner Anteil, der von der Lösung separiert wurde. Bei –30 °C kristallisierte der gelöste Anteil in Form von farblosen Kristallen aus.

**Ausbeute:** 42.4 mg (0.036 mmol, 43 %)

**Smp.** 189 °C

**Elementaranalyse** von $C_{58}H_{85}Br_2Ga_2N_5Si$: gefunden (berechnet) C 59.58 (59.05), H 6.92 (7.24), N 4.73 (5.94) %.

**IR**: $\nu$ 3428, 3345, 3127, 3020, 2953, 2859, 2060, 1520, 1453, 1430, 1377, 1311, 1254, 1172, 1100, 1016, 937, 856, 794, 756, 668, 625, 534, 442 cm$^{-1}$.

**$^1$H NMR** ($C_6D_6$, 400 MHz): $\delta$ 7.15-6.97 (m, 12 H, $C_6H_3(^iPr)_2$), 4.96 (s, 2 H, $\gamma$-C*H*), 4.67 (t, $^3J_{HH}$ = 4.6 Hz, 1H, Si*H*), 3.94 (sept, $^3J_{HH}$ = 6.7 Hz, 2 H, -C*H*$(CH_3)_2$), 3.67 (sept, $^3J_{HH}$ = 6.7 Hz, 2 H, -C*H*$(CH_3)_2$), 3.23 (sept, $^3J_{HH}$ = 6.7 Hz, 4 H, -C*H*$(CH_3)_2$), 1.54 (s, 6 H, ArNCC*H$_3$*), 1.51 (s, 6 H, ArNCC*H$_3$*), 1.42 (d, $^3J_{HH}$ = 6.6 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.30 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.23 (d, $^3J_{HH}$ = 6.6 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.21 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.20 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.13 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H$_3$*)$_2$), 0.98 (d, $^3J_{HH}$ = 6.8 Hz, 12 H, -CH(C*H$_3$*)$_2$), –1.67 (d, $^3J_{HH}$ = 4.4 Hz, 2 H, SiN*H*$_2$) ppm.

**$^{13}$C NMR** ($C_6D_6$, 101 MHz): $\delta$ 168.8, 168.6 (ArN*C*CH$_3$), 147.0, 146.2 (N*C*C(CH(CH$_3$)$_2$)), 143.2, 143.0, 142.3, 141.5 (NC*C*(CH(CH$_3$)$_2$)), 127.5, 127.3, 125.6, 125.4, 123.4, 123.4 (*C*$_6$H$_3$), 98.6 ($\gamma$-*C*H-), 29.7 (-*C*H(CH$_3$)$_2$, 29.4, 29.2, 28.7 (-CH(*C*H$_3$)$_2$), 28.4, 28.0 (-*C*H(CH$_3$)$_2$, 25.2, 24.9, 24.6, 24.2, 24.0 (-CH(*C*H$_3$)$_2$), 23.8, 23.7(ArNC*C*H$_3$), 23.3 (-CH(*C*H$_3$)$_2$) ppm.

**$^{29}$Si NMR** ($C_6D_6$, 119 MHz, DEPT90): –39.6 ppm ($^1J_{HSi}$ = 175.6 Hz)

## 3.2. Spektren $[DDP(Br)Ga]_2(H)NH_2$ **3**

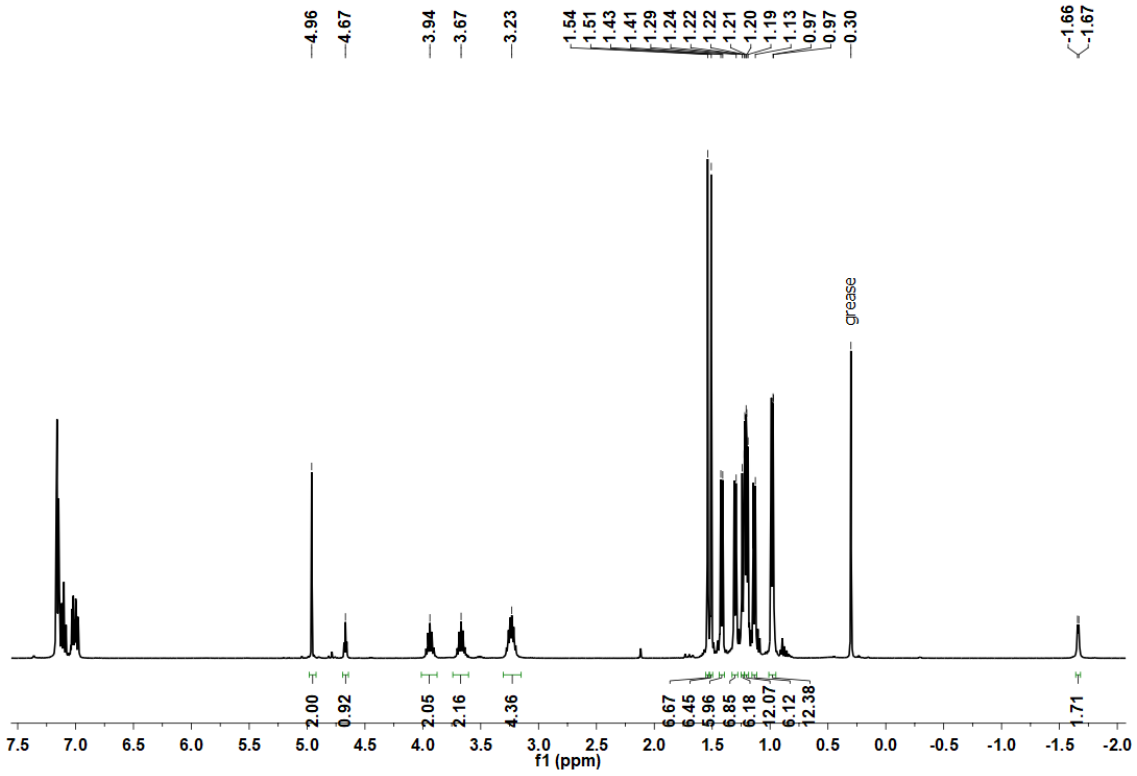



Abbildung 12: $^{1}$H-NMR-Spektrum von $[DDP(Br)Ga]_2Si(H)NH_2$ **3** in $C_6D_6$.

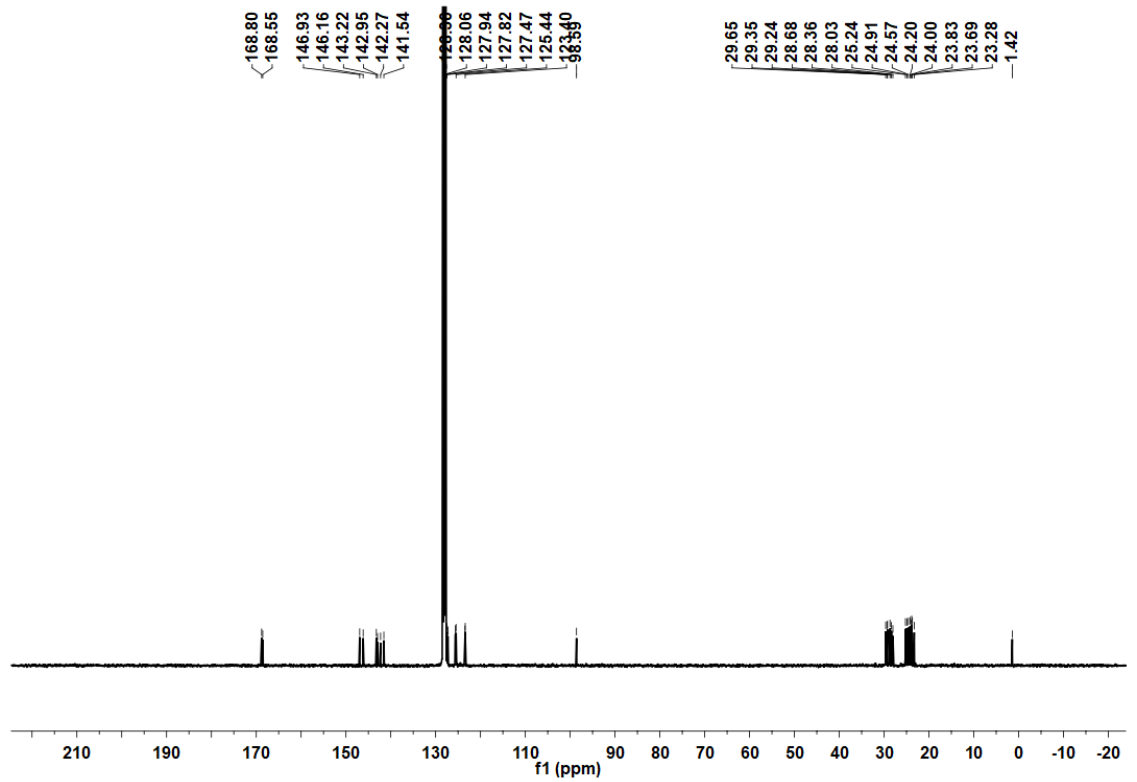



Abbildung 13: $^{13}$C-NMR-Spektrum von $[DDP(Br)Ga]_2Si(H)NH_2$ **3** in $C_6D_6$.

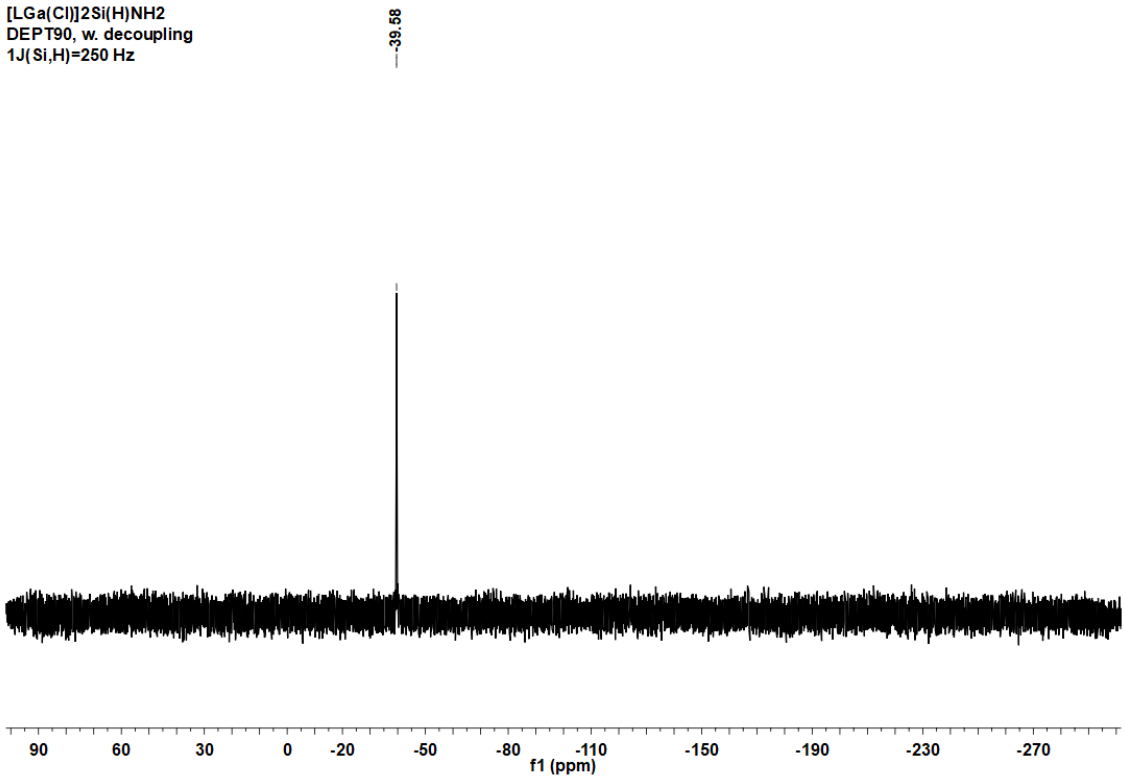



Abbildung 14: $^{29}$Si-NMR-Spektrum (DEPT90) von [DDP(Br)Ga]$_2$Si(H)NH$_2$ **3** in $C_6D_6$.

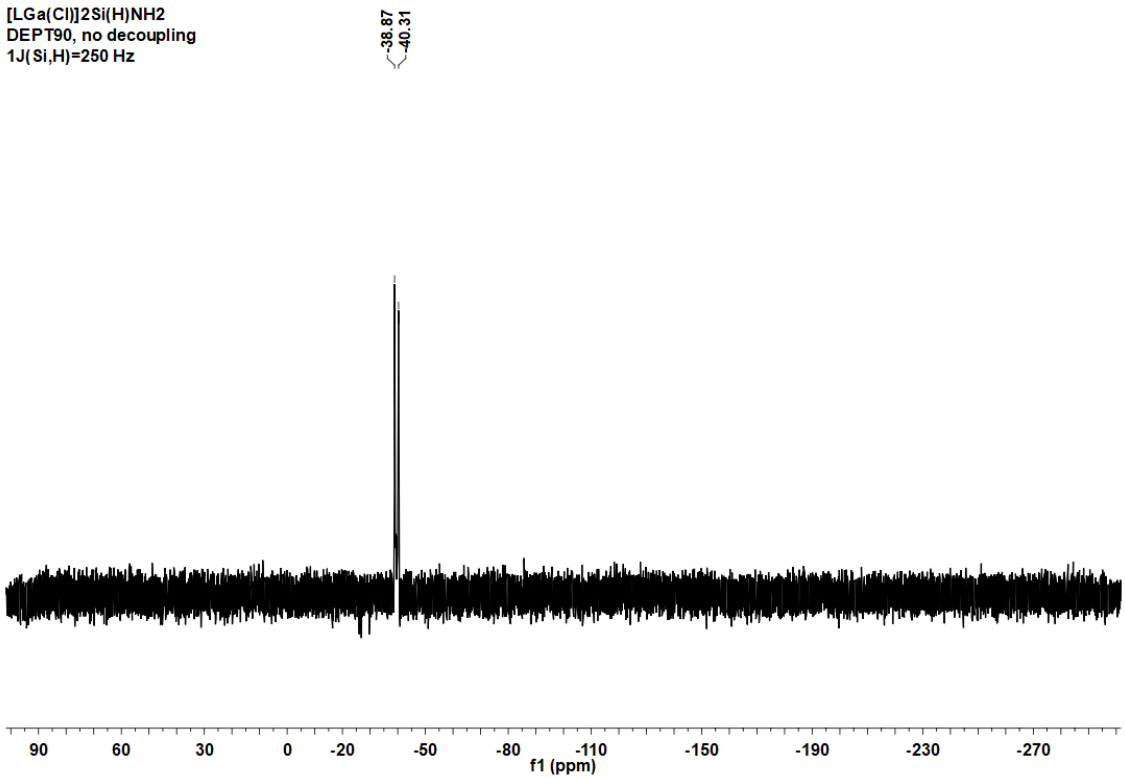



Abbildung 15: $^{29}$Si{$^1$H}-NMR-Spektrum (DEPT90) von [DDP(Br)Ga]$_2$Si(H)NH$_2$ **3** in $C_6D_6$.

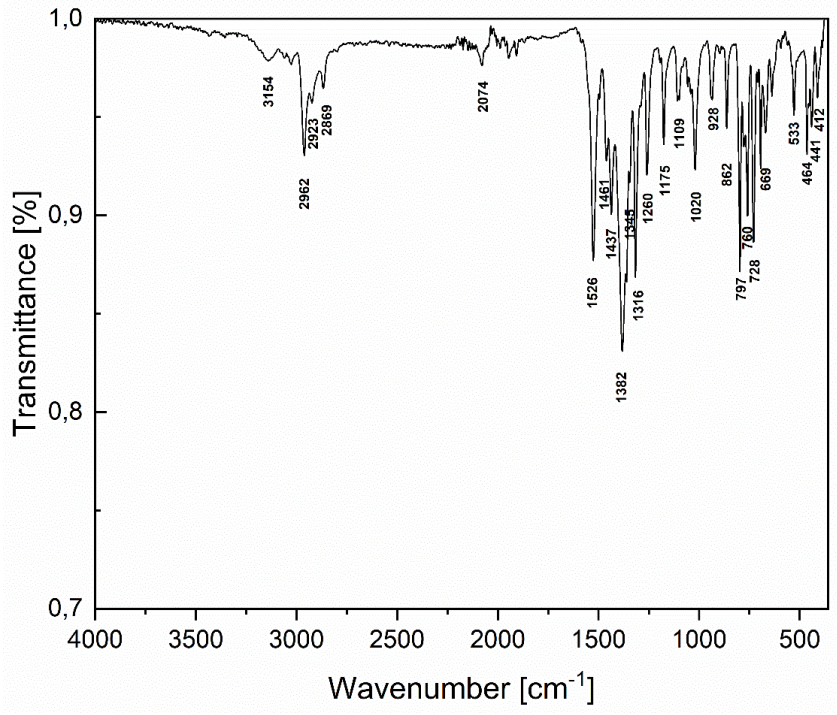



Abbildung 16: ATR-IR Spektrum von $[DDP(Br)Ga]_2Si(H)NH_2$ **3**.

**Figure S19**: ATR-IR spectrum of $[L(Br)Ga]_2Si(H)NH_2$ **4**.

## 3.1. Kristallografische Daten $[DDP(Br)Ga]_2(H)NH_2$ **3**

Tabelle 6: Crystal structure data

| **Identification code** | **jus_165m** |
|---|---|
| Empirical formula | C58 H85 Br2 Ga2 N5 Si |
| Formula weight | 1179.65 |
| Density (calculated) | 1.297 g·cm$^{-3}$ |
| *F*(000) | 2456 |
| Temperature | 100(2) K |
| Crystal size | 0.309 × 0.148 × 0.111 mm |
| Crystal colour | colourless |
| Crystal description | block |
| Wavelength | 1.54178 Å |
| Crystal system | monoclinic |
| Space group | *C 2/c* |
| Unit cell dimensions | |
| *a* [Å] | 23.796(3) |
| *b* [Å] | 10.9213(12) |
| *c* [Å] | 23.597(3) |
| *α* [°] | 90 |
| *β* [°] | 99.872(3) |
| *γ* [°] | 90 |
| Volume | 6041.8(11) Å$^3$ |
| *Z* | 4 |
| Cell measurement reflections used | 9984 |
| Cell measurement *θ* min/max | 3.80°/80.27° |
| Diffractometer control software | Bruker APEX3(v2017.3-0) |
| Diffractometer measurement device | Bruker D8 Venture (Photon II detector) |
| Diffractometer measurement method | Data collection strategy APEX 3/Queen |
| *θ* range for data collection | 3.771°- 80.635° |
| Completeness to *θ* = 67.679° | 100.0% |
| Completeness to $\theta_{max}$ = 80.635° | 99.5% |
| Index ranges | -26 ≤ *h* ≤ 30 |
| | -13 ≤ *k* ≤ 13 |
| | -30 ≤ *l* ≤ 30 |
| Computing data reduction | Bruker APEX3(v2017.3-0) |
| Absorption coefficient | 3.117 mm$^{-1}$ |
| Absorption correction | Semi-empirical from equivalents |
| Computation absorption correction | SADABS |
| Max./min. Transmission | 0.75/0.61 |
| $R_{merg}$ before/after correction | 0.1300/0.0815 |
| Computing structure solution | Bruker APEX3(v2017.3-0) |
| Computing structure refinement | SHELXL-2017/1 (Sheldrick, 2017) |

| | |
|---|---|
| Refinement method | Full-matrix least-squares on $F^2$ |
| Reflections collected | 89420 |
| Independent reflections | 6604 |
| $R_{int}$ | 0.0540 |
| Reflections with $I > 2\sigma(I)$ | 6567 |
| Restraints | 140 |
| Parameter | 450 |
| GooF | 1.370 |
| Weighting details | $w = 1/[\sigma^2(F_{obs}^2) + 25.8204P]$ |
| | where $P = (F_{obs}^2 + 2F_{calc}^2)/3$ |
| $R_1$ [$I > 2\sigma(I)$] | 0.0393 |
| $wR_2$ [$I > 2\sigma(I)$] | 0.0977 |
| $R_1$ [all data] | 0.0395 |
| $wR_2$ [all data] | 0.0978 |
| Absolute structure parameter | |
| Largest diff. peak and hole | 0.334/-0.526 |

Tabelle 7: Atomic coordinates ( × $10^4$) and equivalent isotropic displacement parameters (Å$^2$ × $10^3$) for jus_165m. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | *x* | *y* | *z* | $U_{eq}$ | | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|---|---|---|---|---|
| Br(1) | 4665(1) | 5692(1) | 1418(1) | 24(1) | C(6') | 6262(6) | 3555(13) | 1486(4) | 18(2) |
| Ga(1) | 4982(1) | 3625(1) | 1582(1) | 19(1) | C(7') | 6555(3) | 2423(7) | 1498(3) | 31(1) |
| Si(1) | 4853(1) | 2671(1) | 2489(1) | 20(1) | C(8') | 7106(4) | 2363(9) | 1807(4) | 38(2) |
| H(1) | 5270(30) | 1680(60) | 2530(30) | 24(17) | H(8') | 7311 | 1616 | 1815 | 45 |
| N(3) | 4197(3) | 1984(7) | 2427(3) | 38(2) | C(9') | 7355(8) | 3331(17) | 2094(7) | 38(3) |
| H(4) | 3970(50) | 2550(100) | 2490(50) | 70(30) | H(9') | 7727 | 3249 | 2313 | 45 |
| H(3) | 4120(50) | 1500(110) | 2160(50) | 80(40) | C(10') | 7078(14) | 4450(20) | 2077(14) | 35(4) |
| N(1) | 5671(1) | 3610(2) | 1233(1) | 24(1) | H(10') | 7290 | 5107 | 2272 | 42 |
| N(2) | 4502(1) | 2761(2) | 942(1) | 20(1) | C(11') | 6517(12) | 4720(20) | 1802(11) | 24(3) |
| C(1) | 5609(1) | 3716(3) | 662(1) | 28(1) | C(12') | 6298(4) | 1283(9) | 1198(6) | 51(2) |
| C(2) | 5096(1) | 3497(3) | 291(1) | 28(1) | H(12') | 5886 | 1450 | 1048 | 61 |
| H(2) | 5080 | 3763 | -95 | 34 | C(13') | 6594(5) | 996(10) | 677(5) | 70(3) |
| C(3) | 4603(1) | 2941(3) | 411(1) | 24(1) | H(13D) | 6429 | 252 | 485 | 105 |
| C(4) | 6119(2) | 4064(4) | 393(2) | 51(1) | H(13E) | 7003 | 873 | 811 | 105 |
| H(4A) | 6010 | 4063 | -27 | 76 | H(13F) | 6538 | 1683 | 406 | 105 |
| H(4B) | 6426 | 3472 | 508 | 76 | C(14') | 6333(6) | 179(8) | 1572(6) | 78(4) |
| H(4C) | 6250 | 4884 | 524 | 76 | H(14D) | 6119 | -493 | 1361 | 118 |
| C(5) | 4175(2) | 2501(4) | -96(1) | 37(1) | H(14E) | 6170 | 366 | 1916 | 118 |
| H(5A) | 4306 | 2721 | -454 | 55 | H(14F) | 6733 | -63 | 1685 | 118 |
| H(5B) | 3805 | 2888 | -87 | 55 | C(15) | 6226(1) | 5895(3) | 1716(1) | 27(1) |
| H(5C) | 4136 | 1610 | -75 | 55 | H(15) | 5864 | 5832 | 1431 | 32 |
| C(6) | 6223(6) | 3593(14) | 1647(4) | 21(2) | H(15') | 5868 | 5785 | 1429 | 32 |
| C(7) | 6490(3) | 2470(7) | 1778(3) | 29(1) | C(16) | 6608(1) | 6824(3) | 1474(2) | 34(1) |
| C(8) | 7055(4) | 2477(9) | 2075(4) | 40(2) | H(16A) | 6715 | 6499 | 1121 | 52 |
| H(8) | 7246 | 1721 | 2166 | 48 | H(16B) | 6953 | 6971 | 1759 | 52 |
| C(9) | 7340(7) | 3562(16) | 2238(7) | 35(3) | H(16C) | 6400 | 7595 | 1390 | 52 |
| H(9) | 7727 | 3547 | 2425 | 42 | C(17) | 6068(2) | 6368(4) | 2278(2) | 38(1) |
| C(10) | 7059(12) | 4660(20) | 2126(12) | 28(3) | H(17A) | 5863 | 7146 | 2207 | 58 |
| H(10) | 7223 | 5423 | 2261 | 34 | H(17B) | 6416 | 6495 | 2561 | 58 |
| C(11) | 6495(12) | 4560(20) | 1787(13) | 27(4) | H(17C) | 5824 | 5767 | 2427 | 58 |
| C(12) | 6186(3) | 1261(6) | 1630(4) | 37(2) | C(18) | 4013(1) | 2028(3) | 1019(1) | 27(1) |
| H(12) | 5768 | 1423 | 1586 | 44 | C(19) | 4087(2) | 769(3) | 1137(2) | 44(1) |
| C(13) | 6300(6) | 779(11) | 1062(5) | 60(3) | C(20) | 3610(2) | 77(4) | 1203(2) | 53(1) |
| H(13A) | 6152 | 1358 | 756 | 90 | H(20) | 3653 | -777 | 1275 | 63 |
| H(13B) | 6110 | -15 | 982 | 90 | C(21) | 3084(2) | 596(4) | 1166(2) | 52(1) |
| H(13C) | 6712 | 678 | 1079 | 90 | H(21) | 2764 | 109 | 1212 | 62 |
| C(14) | 6334(4) | 309(7) | 2104(4) | 49(2) | C(22) | 3020(2) | 1835(4) | 1061(2) | 41(1) |
| H(14A) | 6051 | -351 | 2046 | 73 | H(22) | 2654 | 2194 | 1043 | 49 |
| H(14B) | 6333 | 695 | 2479 | 73 | C(23) | 3473(1) | 2574(3) | 981(1) | 30(1) |
| H(14C) | 6714 | -28 | 2092 | 73 | C(24) | 4635(4) | 98(8) | 1053(5) | 32(2) |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| H(24) | 4931 | 727 | 1012 | 38 | C(26') | 4765(11) | -840(17) | 1761(7) | 56(4) |
| C(25) | 4587(5) | -783(9) | 540(5) | 52(2) | H(26D) | 5163 | -1116 | 1835 | 84 |
| H(25A) | 4471 | -327 | 182 | 78 | H(26E) | 4516 | -1529 | 1620 | 84 |
| H(25B) | 4303 | -1415 | 576 | 78 | H(26F) | 4661 | -526 | 2118 | 84 |
| H(25C) | 4958 | -1169 | 536 | 78 | C(27) | 3367(1) | 3929(3) | 868(1) | 29(1) |
| C(26) | 4832(7) | -605(12) | 1606(6) | 44(3) | H(27) | 3726 | 4301 | 781 | 35 |
| H(26A) | 5189 | -1034 | 1581 | 66 | C(28) | 3225(2) | 4555(4) | 1408(2) | 41(1) |
| H(26B) | 4540 | -1203 | 1663 | 66 | H(28A) | 3203 | 5443 | 1348 | 62 |
| H(26C) | 4895 | -34 | 1931 | 66 | H(28B) | 3523 | 4369 | 1737 | 62 |
| C(24') | 4695(5) | 189(10) | 1303(4) | 35(2) | H(28C) | 2857 | 4254 | 1484 | 62 |
| H(24') | 4970 | 859 | 1445 | 42 | C(29) | 2889(1) | 4165(4) | 352(2) | 40(1) |
| C(25') | 4836(6) | -281(12) | 731(5) | 70(3) | H(29A) | 2977 | 3731 | 14 | 60 |
| H(25D) | 4529 | -822 | 548 | 105 | H(29B) | 2862 | 5045 | 271 | 60 |
| H(25E) | 5196 | -736 | 803 | 105 | H(29C) | 2526 | 3869 | 441 | 60 |
| H(25F) | 4872 | 414 | 477 | 105 | | | | | |

Tabelle 8: Anisotropic displacement parameters ($Å^2 \times 10^3$) for jus_165m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Br(1) | 23(1) | 20(1) | 30(1) | -1(1) | 5(1) | 1(1) | C(10') | 29(6) | 43(7) | 32(7) | 0(5) | -3(5) | -3(5) |
| Ga(1) | 23(1) | 18(1) | 14(1) | 1(1) | 1(1) | -2(1) | C(11') | 23(5) | 31(7) | 17(6) | 7(4) | 2(4) | -12(4) |
| Si(1) | 27(1) | 19(1) | 14(1) | 0(1) | 3(1) | -1(1) | C(12') | 29(4) | 32(5) | 90(8) | -4(4) | 8(4) | 15(3) |
| N(3) | 45(4) | 46(4) | 24(3) | -10(3) | 8(3) | -20(3) | C(13') | 81(7) | 66(7) | 59(6) | -13(5) | 1(5) | 9(6) |
| N(1) | 18(1) | 21(1) | 33(1) | 3(1) | 3(1) | 2(1) | C(14') | 117(10) | 26(4) | 105(9) | -1(5) | 54(8) | 7(5) |
| N(2) | 21(1) | 22(1) | 17(1) | -3(1) | 3(1) | -2(1) | C(15) | 23(1) | 33(2) | 23(1) | 5(1) | -2(1) | -4(1) |
| C(1) | 30(2) | 22(2) | 37(2) | 2(1) | 18(1) | 4(1) | C(16) | 28(2) | 40(2) | 31(2) | 7(1) | -5(1) | -11(1) |
| C(2) | 40(2) | 27(2) | 19(1) | 4(1) | 12(1) | 8(1) | C(17) | 40(2) | 43(2) | 32(2) | -3(2) | 6(1) | -5(2) |
| C(3) | 30(2) | 24(1) | 16(1) | -1(1) | 1(1) | 7(1) | C(18) | 28(2) | 28(2) | 26(2) | -7(1) | 6(1) | -9(1) |
| C(4) | 46(2) | 47(2) | 70(3) | -8(2) | 39(2) | -8(2) | C(19) | 44(2) | 27(2) | 63(3) | -4(2) | 14(2) | -7(2) |
| C(5) | 42(2) | 44(2) | 21(2) | -10(1) | -4(1) | 8(2) | C(20) | 60(3) | 33(2) | 67(3) | 3(2) | 16(2) | -17(2) |
| C(6) | 17(3) | 34(3) | 15(5) | 7(4) | 8(4) | 3(2) | C(21) | 45(2) | 58(3) | 54(2) | 1(2) | 11(2) | -28(2) |
| C(7) | 24(3) | 33(3) | 29(4) | 8(3) | 4(3) | 4(2) | C(22) | 30(2) | 49(2) | 43(2) | -5(2) | 8(2) | -14(2) |
| C(8) | 29(4) | 41(4) | 45(5) | 12(4) | -6(4) | 6(3) | C(23) | 26(2) | 38(2) | 25(2) | -6(1) | 3(1) | -8(1) |
| C(9) | 21(4) | 46(6) | 34(8) | 4(4) | -8(5) | 6(4) | C(24) | 35(4) | 20(4) | 37(6) | -7(5) | -4(5) | 1(3) |
| C(10) | 21(5) | 41(7) | 23(6) | 9(6) | 0(4) | 6(5) | C(25) | 62(6) | 40(5) | 56(6) | -9(4) | 17(5) | 4(4) |
| C(11) | 20(6) | 30(5) | 31(8) | 15(5) | 0(5) | 5(4) | C(26) | 64(8) | 19(6) | 44(8) | -6(6) | -7(6) | 15(5) |
| C(12) | 32(3) | 28(3) | 49(4) | 4(3) | 1(3) | 9(3) | C(24') | 62(6) | 29(4) | 14(4) | -11(4) | 6(4) | -3(4) |
| C(13) | 92(8) | 48(6) | 38(4) | -3(4) | 7(5) | -3(6) | C(25') | 117(11) | 61(7) | 33(5) | -7(5) | 13(6) | 23(7) |
| C(14) | 60(5) | 31(4) | 54(5) | 8(3) | 5(4) | 2(3) | C(26') | 97(9) | 32(7) | 43(8) | 13(5) | 20(6) | -2(6) |
| C(6') | 16(3) | 24(3) | 15(5) | 10(4) | 9(4) | 2(2) | C(27) | 18(1) | 39(2) | 32(2) | -7(1) | 4(1) | -1(1) |
| C(7') | 27(3) | 32(3) | 37(4) | 11(3) | 12(3) | 10(2) | C(28) | 32(2) | 52(2) | 41(2) | -14(2) | 11(2) | -1(2) |
| C(8') | 28(3) | 40(4) | 47(5) | 11(4) | 10(4) | 14(3) | C(29) | 23(2) | 53(2) | 41(2) | -4(2) | -1(1) | 3(2) |
| C(9') | 29(4) | 51(7) | 31(8) | 14(4) | 2(4) | 12(4) | | | | | | | |

Tabelle 9: Bond lengths [Å] for jus_165m.

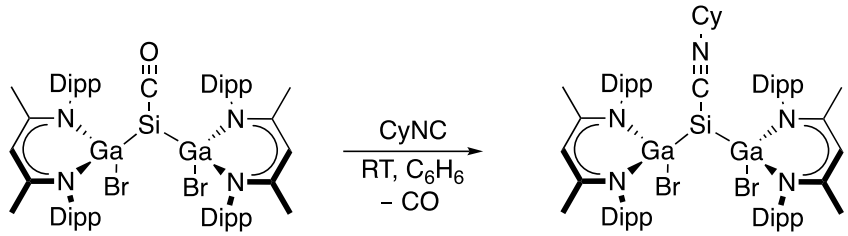
| | | | | | |
|---|---|---|---|---|---|
| Br(1)-Ga(1) | 2.3909(5) | C(7)-C(12) | 1.516(10) | C(15)-C(17) | 1.531(5) |
| Ga(1)-N(1) | 1.954(2) | C(8)-C(9) | 1.387(17) | C(15)-C(16) | 1.535(4) |
| Ga(1)-N(2) | 1.971(2) | C(9)-C(10) | 1.38(2) | C(18)-C(23) | 1.406(5) |
| Ga(1)-Si(1) | 2.4471(19) | C(10)-C(11) | 1.44(4) | C(18)-C(19) | 1.408(5) |
| Si(1)-N(3) | 1.716(6) | C(11)-C(15) | 1.59(3) | C(19)-C(20) | 1.394(5) |
| N(1)-C(1) | 1.337(4) | C(12)-C(13) | 1.507(13) | C(19)-C(24) | 1.537(10) |
| N(1)-C(6') | 1.433(15) | C(12)-C(14) | 1.523(11) | C(19)-C(24') | 1.567(12) |
| N(1)-C(6) | 1.497(15) | C(6')-C(7') | 1.417(15) | C(20)-C(21) | 1.363(7) |
| N(2)-C(3) | 1.331(4) | C(6')-C(11') | 1.55(3) | C(21)-C(22) | 1.380(6) |
| N(2)-C(18) | 1.447(4) | C(7')-C(8') | 1.387(12) | C(22)-C(23) | 1.385(5) |
| C(1)-C(2) | 1.396(5) | C(7')-C(12') | 1.509(14) | C(23)-C(27) | 1.517(5) |
| C(1)-C(4) | 1.509(4) | C(8')-C(9') | 1.338(17) | C(24)-C(26) | 1.517(16) |
| C(2)-C(3) | 1.391(5) | C(9')-C(10') | 1.39(2) | C(24)-C(25) | 1.535(13) |
| C(3)-C(5) | 1.511(4) | C(10')-C(11') | 1.41(5) | C(24')-C(25') | 1.533(11) |
| C(6)-C(11) | 1.26(3) | C(11')-C(15) | 1.45(3) | C(24')-C(26') | 1.549(15) |
| C(6)-C(7) | 1.391(16) | C(12')-C(14') | 1.489(15) | C(27)-C(28) | 1.533(5) |
| C(7)-C(8) | 1.405(11) | C(12')-C(13') | 1.549(16) | C(27)-C(29) | 1.538(5) |

Tabelle 10: Bond angles [°] for jus_165m.

| | | | | | |
|---|---|---|---|---|---|
| N(1)-Ga(1)-N(2) | 95.30(10) | C(8)-C(7)-C(12) | 119.8(7) | C(17)-C(15)-C(16) | 110.4(3) |
| N(1)-Ga(1)-Br(1) | 101.76(7) | C(9)-C(8)-C(7) | 121.6(11) | C(17)-C(15)-C(11) | 111.9(11) |
| N(2)-Ga(1)-Br(1) | 101.69(7) | C(10)-C(9)-C(8) | 119.5(17) | C(16)-C(15)-C(11) | 113.0(11) |
| N(1)-Ga(1)-Si(1) | 127.17(8) | C(9)-C(10)-C(11) | 115(2) | C(23)-C(18)-C(19) | 120.6(3) |
| N(2)-Ga(1)-Si(1) | 108.83(8) | C(6)-C(11)-C(10) | 127(2) | C(23)-C(18)-N(2) | 120.1(3) |
| Br(1)-Ga(1)-Si(1) | 117.39(4) | C(6)-C(11)-C(15) | 124(2) | C(19)-C(18)-N(2) | 119.3(3) |
| N(3)-Si(1)-Ga(1) | 111.1(2) | C(10)-C(11)-C(15) | 108.0(18) | C(20)-C(19)-C(18) | 118.4(4) |
| C(1)-N(1)-C(6') | 110.7(4) | C(13)-C(12)-C(7) | 111.0(8) | C(20)-C(19)-C(24) | 118.7(5) |
| C(1)-N(1)-C(6) | 126.2(4) | C(13)-C(12)-C(14) | 111.1(7) | C(18)-C(19)-C(24) | 121.3(5) |
| C(1)-N(1)-Ga(1) | 118.0(2) | C(7)-C(12)-C(14) | 112.6(6) | C(20)-C(19)-C(24') | 119.2(5) |
| C(6')-N(1)-Ga(1) | 131.3(4) | C(7')-C(6')-N(1) | 119.3(10) | C(18)-C(19)-C(24') | 121.5(5) |
| C(6)-N(1)-Ga(1) | 115.6(4) | C(7')-C(6')-C(11') | 124.0(15) | C(21)-C(20)-C(19) | 121.6(4) |
| C(3)-N(2)-C(18) | 118.6(2) | N(1)-C(6')-C(11') | 116.2(15) | C(20)-C(21)-C(22) | 119.4(4) |
| C(3)-N(2)-Ga(1) | 118.3(2) | C(8')-C(7')-C(6') | 117.9(9) | C(21)-C(22)-C(23) | 122.1(4) |
| C(18)-N(2)-Ga(1) | 122.78(18) | C(8')-C(7')-C(12') | 118.6(7) | C(22)-C(23)-C(18) | 117.9(3) |
| N(1)-C(1)-C(2) | 123.3(3) | C(6')-C(7')-C(12') | 123.5(8) | C(22)-C(23)-C(27) | 118.7(3) |
| N(1)-C(1)-C(4) | 119.3(3) | C(9')-C(8')-C(7') | 121.7(11) | C(18)-C(23)-C(27) | 123.3(3) |
| C(2)-C(1)-C(4) | 117.4(3) | C(8')-C(9')-C(10') | 121.0(19) | C(26)-C(24)-C(25) | 109.3(9) |
| C(3)-C(2)-C(1) | 128.7(3) | C(9')-C(10')-C(11') | 127(2) | C(26)-C(24)-C(19) | 106.0(9) |
| N(2)-C(3)-C(2) | 123.5(3) | C(10')-C(11')-C(15) | 129.7(19) | C(25)-C(24)-C(19) | 116.8(7) |
| N(2)-C(3)-C(5) | 119.4(3) | C(10')-C(11')-C(6') | 108(2) | C(25')-C(24')-C(26') | 110.9(11) |
| C(2)-C(3)-C(5) | 117.1(3) | C(15)-C(11')-C(6') | 121(2) | C(25')-C(24')-C(19) | 104.2(8) |
| C(11)-C(6)-C(7) | 119.3(17) | C(14')-C(12')-C(7') | 114.4(10) | C(26')-C(24')-C(19) | 116.9(12) |
| C(11)-C(6)-N(1) | 121.1(17) | C(14')-C(12')-C(13') | 108.7(9) | C(23)-C(27)-C(28) | 110.1(3) |
| C(7)-C(6)-N(1) | 118.1(11) | C(7')-C(12')-C(13') | 109.6(8) | C(23)-C(27)-C(29) | 112.4(3) |
| C(6)-C(7)-C(8) | 117.8(9) | C(11')-C(15)-C(17) | 111.0(11) | C(28)-C(27)-C(29) | 110.2(3) |
| C(6)-C(7)-C(12) | 122.4(8) | C(11')-C(15)-C(16) | 109.7(10) | | |

## 4. [DDP(Br)Ga]$_2$Si–CNCy 4

### 4.1. Synthese [DDP(Br)Ga]$_2$–CNCy **4**




**1** **4**

Dipp = 2,6-$^{i}$Pr$_2$C$_6$H$_3$
Cy = Cyclohexyl

Zu einer Lösung von [DDP(Br)Ga]$_2$Si–CO **1** (0.060 g, 0.05 mmol) in 0.8 mL Benzol wurde 6.26 µL Cyclohexylisocyanid (0.0055 g, 0.05 mmol) gegeben und für einen Tag bei Raumtemperatur gerührt. Anschließend wurde das Lösungsmittel im Vakuum entfernt. Zur Kristallisation wurden 0.8 mL *n*-Hexan zum Rückstand gegeben und nach einem Tag konnten orangene Kristalle von [DDP(Br)Ga]$_2$Si–CNCy **4** isoliert werden.

**Ausbeute:** 37 mg (0.029 mmol, 58%)

**Smp.** 180 °C (Zersetzung)

**Elementaranalyse** von $C_{65}H_{93}N_5Br_2Ga_2Si$: gefunden (berechnet) C 60.5 (61.39), H 7.65 (7.37), N 4.98 (5.51) %.

**IR**: ν 2962, 2923, 2862, 2093, 1526, 1460, 1435, 1383, 1317, 1258, 1177, 1095, 1016, 939, 859, 794, 757, 705, 637, 527 cm$^{-1}$.

**UV-Vis** ($C_6H_6$): $\lambda_{max}$ 357 nm.

**$^{1}$H NMR** ($C_6D_6$, 300 MHz): δ 7.15-6.97 (m, 12 H, C$_6$*H*$_3$($^{i}$Pr)$_2$), 5.06 (s, 2 H, γ-C*H*-), 3.90 (sept, $^{3}J_{HH}$ = 6.7 Hz, 4 H, -C*H*(CH$_3$)$_2$), 3.17 (sept, $^{3}J_{HH}$ = 6.8 Hz, 4 H, -C*H*(CH$_3$)$_2$), 2.85 (m, 1 H, Cy-C*H*), 1.80-0.80 (m, 10 H, Cy-C*H*$_2$), 1.50 (s, 12 H, ArNCC*H*$_3$), 1.38 (d, $^{3}J_{HH}$ = 6.7 Hz, 12 H, -CH(C*H*$_3$)$_2$), 1.25 (d, $^{3}J_{HH}$ = 6.7 Hz, 12 H, -CH(C*H*$_3$)$_2$), 1.18 (d, $^{3}J_{HH}$ = 6.8 Hz, 12 H, -CH(C*H*$_3$)$_2$), 1.03 (d, $^{3}J_{HH}$ = 6.8 Hz, 12 H, -CH(C*H*$_3$)$_2$).

**$^{13}$C NMR** ($C_6D_6$, 75 MHz): δ 169.1 (ArN*C*CH$_3$), 146.4 (Cy-N*C*), 143.4 (N*C*C(CH(CH$_3$)$_2$)), 143.3 (NC*C*(CH(CH$_3$)$_2$)), 128.9, 127.4, 125.5, 124.6 (*C*$_6$H$_3$), 100.1 (γ-*C*H-), 58.2 (Cy-*C*H), 32.1 (Cy-*C*H$_2$), 29.5, 28.9, 28.7 (-*C*H(CH$_3$)$_2$), 25.7(Cy-*C*H$_2$), 25.4 (-CH(*C*H$_3$)$_2$), 25.3 (Cy-*C*H$_2$), 25.2, 24.9 (-CH(*C*H$_3$)$_2$), 24.8 (ArNC*C*H$_3$).

**$^{29}$Si NMR** ($C_6D_6$, 59 MHz): δ –175.4 ppm.

## 4.2. Spektren [DDP(Br)Ga]$_2$–CNCy **4**

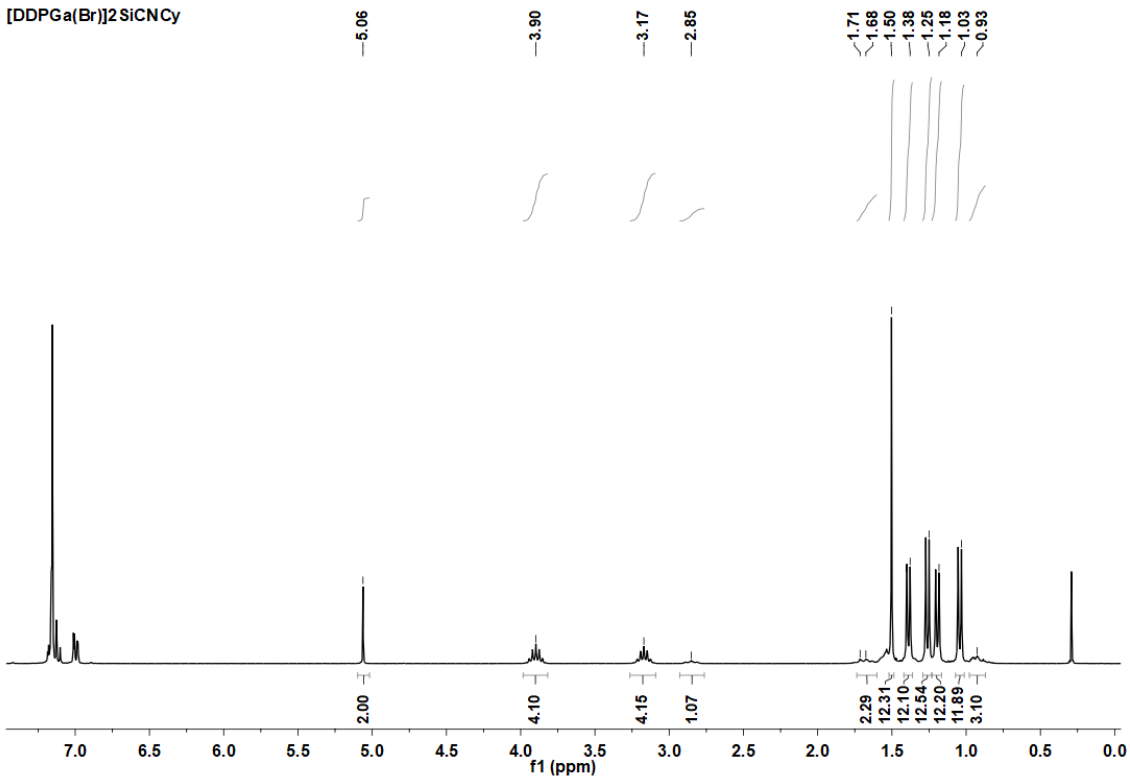



Abbildung 17: $^{1}$H-NMR-Spektrum von [DDP(Br)Ga]$_2$Si–CNCy **4** in $C_6D_6$.

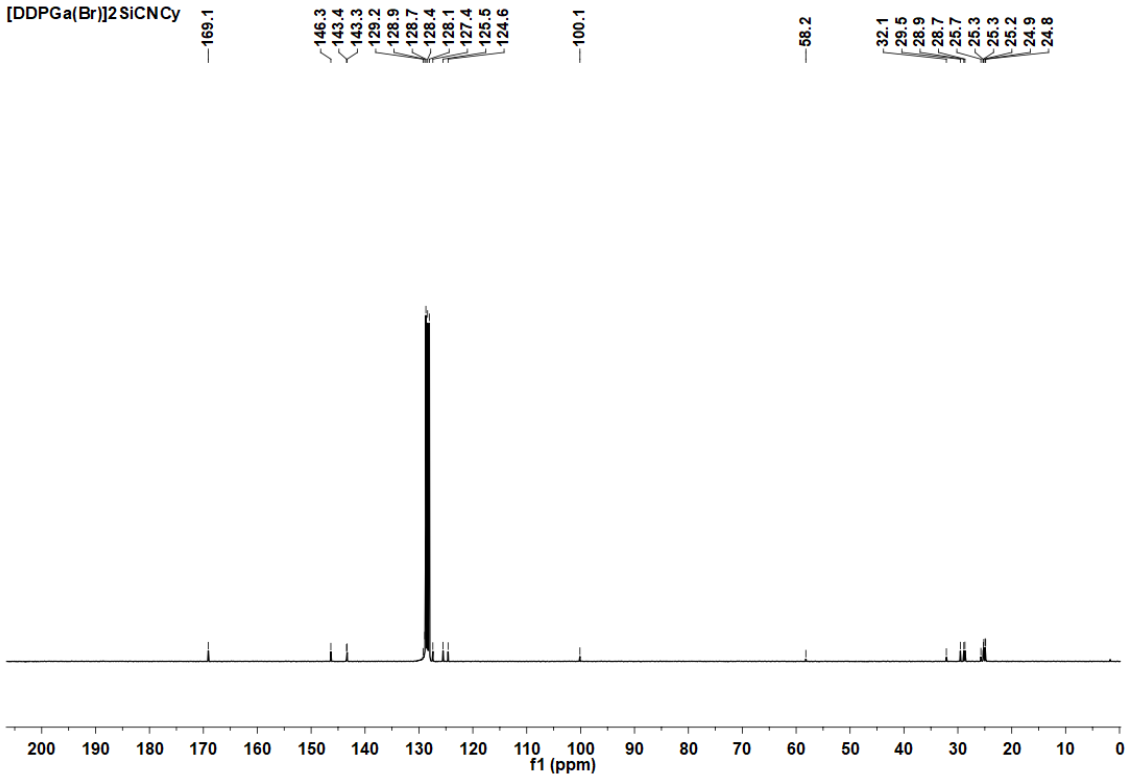



Abbildung 18: $^{13}$C-NMR-Spektrum von [DDP(Br)Ga]$_2$Si–CNCy **4** in $C_6D_6$.

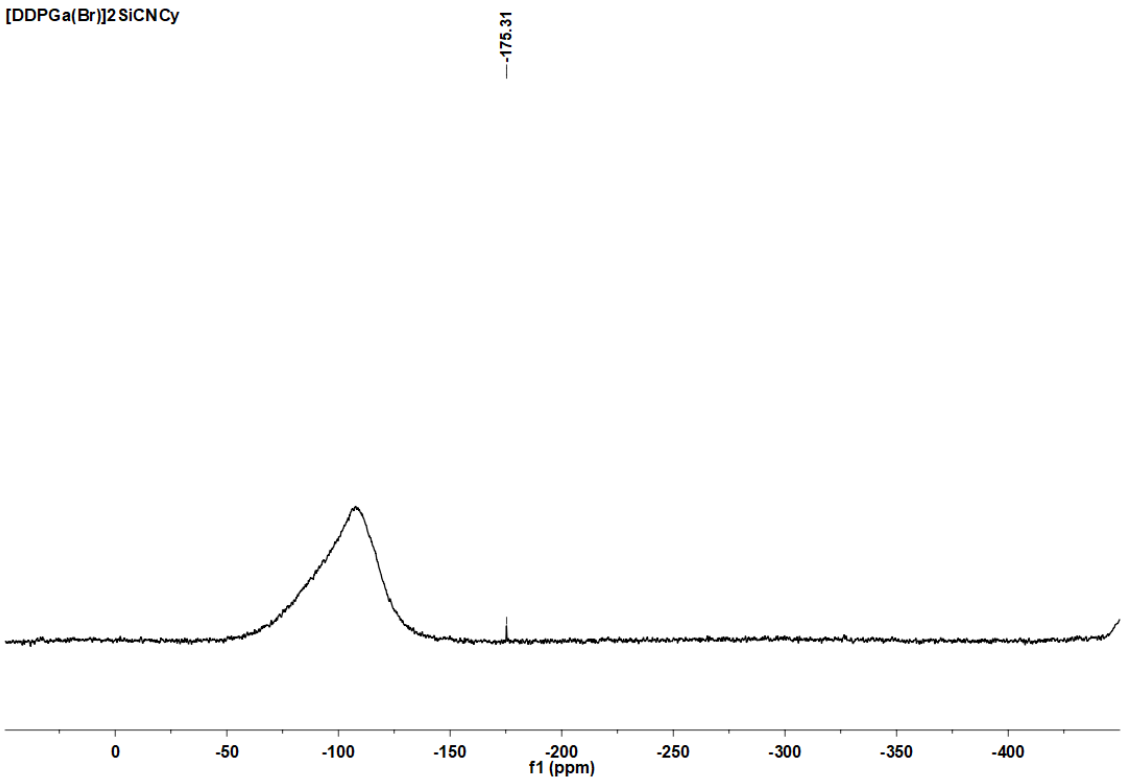



Abbildung 19: $^{29}$Si-NMR-Spektrum von [DDP(Br)Ga]$_2$Si–CNCy **4** in $C_6D_6$.

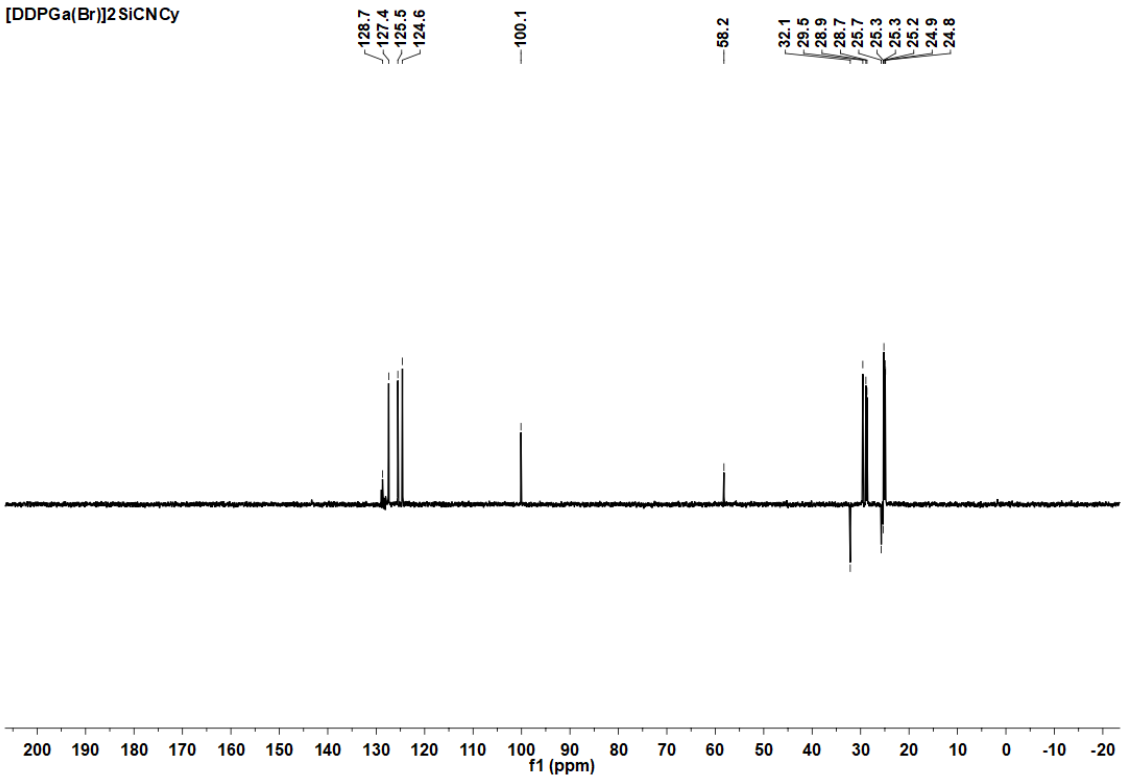



Abbildung 20: $^{13}$C-NMR-Spektrum (DEPT135) von [DDP(Br)Ga]$_2$Si–CNCy **4** in $C_6D_6$.

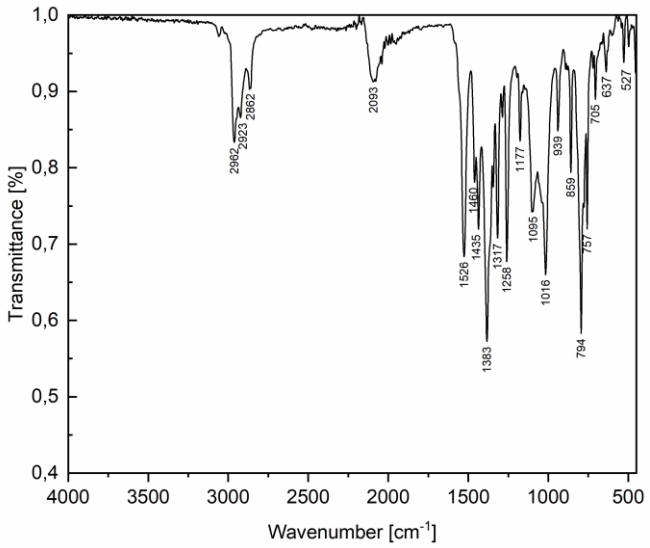



Abbildung 21: ATR-IR Spektrum von $[DDP(Br)Ga]_2Si–CNCy$ **4**.

## 4.3. Kristallografische Daten $[DDP(Br)Ga]_2$–CNCy **4**

Tabelle 11: Crystal structure data

| **Identification code** | **jus_159bm** |
|---|---|
| Empirical formula | C71 H99 Br2 Ga2 N5 Si |
| Formula weight | 1349.90 |
| Density (calculated) | 1.295 g·cm$^{-3}$ |
| *F*(000) | 2824 |
| Temperature | 100(2) K |
| Crystal size | 0.290 × 0.280 × 0.210 mm |
| Crystal colour | orange |
| Crystal description | block |
| Wavelength | 0.71073 Å |
| Crystal system | monoclinic |
| Space group | *P 21/c* |
| Unit cell dimensions | |
| *a* [Å] | 14.4078(19) |
| *b* [Å] | 12.8192(17) |
| *c* [Å] | 37.515(5) |
| *α* [°] | 90 |
| *β* [°] | 92.373(7) |
| *γ* [°] | 90 |
| Volume | 6922.9(16) Å$^3$ |
| *Z* | 4 |
| Cell measurement reflections used | 9372 |
| Cell measurement *θ* min/max | 2.21°/31.63° |
| Diffractometer control software | BRUKER APEX2(v2009.5-1) |
| Diffractometer measurement device | Bruker D8 KAPPA II (APEX II detector) |
| Diffractometer measurement method | Data collection strategy APEX 2/COSMO |
| *θ* range for data collection | 1.679°- 33.345° |
| Completeness to *θ* = 25.242° | 100.0% |
| Completeness to $\theta_{max}$ = 33.345° | 96.4% |
| Index ranges | -21 ≤ *h* ≤ 22 |
| | -19 ≤ *k* ≤ 18 |
| | -56 ≤ *l* ≤ 54 |
| Computing data reduction | BRUKER APEX2(v2009.5-1) |
| Absorption coefficient | 1.993 mm$^{-1}$ |
| Absorption correction | Semi-empirical from equivalents |
| Computation absorption correction | SADABS |
| Max./min. Transmission | 0.75/0.65 |
| $R_{merg}$ before/after correction | 0.0650/0.0467 |
| Computing structure solution | BRUKER APEX2(v2009.5-1) |
| Computing structure refinement | SHELXL-2017/1 (Sheldrick, 2017) |

| | |
|---|---|
| Refinement method | Full-matrix least-squares on $F^2$ |
| Reflections collected | 285674 |
| Independent reflections | 25865 |
| $R_{int}$ | 0.0408 |
| Reflections with $I > 2\sigma(I)$ | 19586 |
| Restraints | 78 |
| Parameter | 826 |
| GooF | 1.044 |
| Weighting details | $w = 1/[\sigma^2(F_{obs}^2) + (0.0334P)^2 + .4181P]$ |
| | where $P = (F_{obs}^2 + 2F_{calc}^2)/3$ |
| $R_1$ [$I > 2\sigma(I)$] | 0.0344 |
| $wR_2$ [$I > 2\sigma(I)$] | 0.0735 |
| $R_1$ [all data] | 0.0594 |
| $wR_2$ [all data] | 0.0818 |
| Absolute structure parameter | |
| Largest diff. peak and hole | 0.910/-0.795 |

Tabelle 12: Atomic coordinates ( × $10^4$) and equivalent isotropic displacement parameters ($Å^2$ × $10^3$) for jus_159bm. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | *x* | *y* | *z* | $U_{eq}$ | | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|---|---|---|---|---|
| Br(1) | 3357(1) | 1646(1) | 4665(1) | 22(1) | H(14C) | 3383 | 4758 | 3360 | 53 |
| Br(2) | 1182(1) | -359(1) | 3006(1) | 26(1) | C(15) | 2791(1) | 250(1) | 2283(1) | 23(1) |
| Ga(1) | 1600(1) | 1390(1) | 3171(1) | 15(1) | H(15) | 2124 | 361 | 2336 | 27 |
| Ga(2) | 3448(1) | 702(1) | 4102(1) | 16(1) | C(16) | 2863(2) | 171(2) | 1877(1) | 36(1) |
| Si(1) | 2739(1) | 1778(1) | 3642(1) | 21(1) | H(16A) | 2426 | -358 | 1783 | 53 |
| N(1) | 1985(1) | 1958(1) | 2704(1) | 16(1) | H(16B) | 3497 | -28 | 1820 | 53 |
| N(2) | 392(1) | 2087(1) | 3149(1) | 17(1) | H(16C) | 2711 | 847 | 1767 | 53 |
| N(3) | 4816(1) | 684(1) | 4099(1) | 16(1) | C(17) | 3112(1) | -772(1) | 2458(1) | 24(1) |
| N(4) | 3323(1) | -729(1) | 4282(1) | 19(1) | H(17A) | 2734 | -1350 | 2361 | 37 |
| N(5) | 1559(1) | 2848(2) | 4175(1) | 40(1) | H(17B) | 3040 | -727 | 2717 | 37 |
| C(1) | 1349(1) | 2220(1) | 2454(1) | 19(1) | H(17C) | 3766 | -895 | 2410 | 37 |
| C(2) | 390(1) | 2228(1) | 2509(1) | 22(1) | C(18) | -81(1) | 2464(1) | 3457(1) | 18(1) |
| H(2) | -5 | 2236 | 2300 | 26 | C(19) | -59(1) | 3542(1) | 3525(1) | 22(1) |
| C(3) | -49(1) | 2226(1) | 2829(1) | 22(1) | C(20) | -547(1) | 3916(1) | 3811(1) | 28(1) |
| C(4) | 1620(1) | 2547(1) | 2086(1) | 26(1) | H(20) | -549 | 4643 | 3861 | 34 |
| H(4A) | 1530 | 1960 | 1920 | 39 | C(21) | -1030(1) | 3245(2) | 4024(1) | 34(1) |
| H(4B) | 2274 | 2758 | 2094 | 39 | H(21) | -1356 | 3511 | 4220 | 40 |
| H(4C) | 1232 | 3135 | 2004 | 39 | C(22) | -1040(1) | 2187(2) | 3954(1) | 30(1) |
| C(5) | -1085(1) | 2425(2) | 2814(1) | 43(1) | H(22) | -1370 | 1733 | 4104 | 36 |
| H(5A) | -1311 | 2460 | 2564 | 65 | C(23) | -575(1) | 1772(1) | 3667(1) | 21(1) |
| H(5B) | -1213 | 3088 | 2932 | 65 | C(24) | 468(1) | 4297(1) | 3296(1) | 27(1) |
| H(5C) | -1401 | 1857 | 2935 | 65 | H(24) | 681 | 3912 | 3082 | 33 |
| C(6) | 2959(1) | 1994(1) | 2624(1) | 18(1) | H(24') | 873 | 3869 | 3142 | 33 |
| C(7) | 3494(1) | 2853(1) | 2746(1) | 23(1) | C(25) | 1358(5) | 4735(4) | 3513(2) | 29(1) |
| C(8) | 4436(1) | 2864(2) | 2673(1) | 30(1) | H(25A) | 1687 | 5229 | 3364 | 44 |
| H(8) | 4811 | 3431 | 2755 | 36 | H(25B) | 1773 | 4155 | 3580 | 44 |
| C(9) | 4832(1) | 2070(2) | 2484(1) | 32(1) | H(25C) | 1162 | 5089 | 3729 | 44 |
| H(9) | 5474 | 2093 | 2437 | 39 | C(26) | -87(4) | 5255(6) | 3168(2) | 49(2) |
| C(10) | 4294(1) | 1238(2) | 2362(1) | 27(1) | H(26A) | 276 | 5651 | 2999 | 73 |
| H(10) | 4572 | 697 | 2230 | 33 | H(26B) | -218 | 5698 | 3373 | 73 |
| C(11) | 3347(1) | 1178(1) | 2430(1) | 21(1) | H(26C) | -673 | 5030 | 3050 | 73 |
| C(12) | 3068(1) | 3781(1) | 2932(1) | 26(1) | C(25') | 1041(11) | 4971(13) | 3488(3) | 44(3) |
| H(12) | 2516 | 3521 | 3059 | 31 | H(25D) | 1360 | 5430 | 3324 | 66 |
| C(13) | 2724(2) | 4600(2) | 2660(1) | 44(1) | H(25E) | 1502 | 4569 | 3630 | 66 |
| H(13A) | 3241 | 4824 | 2517 | 67 | H(25F) | 670 | 5392 | 3647 | 66 |
| H(13B) | 2475 | 5202 | 2786 | 67 | C(26') | -292(7) | 4850(11) | 3037(4) | 51(3) |
| H(13C) | 2234 | 4296 | 2504 | 67 | H(26D) | 16 | 5343 | 2881 | 77 |
| C(14) | 3731(2) | 4280(2) | 3212(1) | 36(1) | H(26E) | -737 | 5225 | 3180 | 77 |
| H(14A) | 4214 | 4669 | 3092 | 53 | H(26F) | -620 | 4321 | 2892 | 77 |
| H(14B) | 4020 | 3735 | 3362 | 53 | C(27) | -621(1) | 616(1) | 3589(1) | 23(1) |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| H(27) | -375 | 505 | 3346 | 27 | H(45A) | 6731 | 2815 | 4465 | 46 |
| C(28) | -5(1) | 0(2) | 3854(1) | 33(1) | H(45B) | 6112 | 3675 | 4653 | 46 |
| H(28A) | -9 | -739 | 3786 | 49 | H(45C) | 6514 | 3902 | 4269 | 46 |
| H(28B) | -243 | 73 | 4094 | 49 | C(46) | 4660(1) | 3749(2) | 4170(1) | 33(1) |
| H(28C) | 631 | 268 | 3853 | 49 | H(46A) | 4908 | 4262 | 4005 | 50 |
| C(29) | -1614(1) | 189(2) | 3583(1) | 38(1) | H(46B) | 4517 | 4095 | 4394 | 50 |
| H(29A) | -2006 | 593 | 3414 | 58 | H(46C) | 4093 | 3439 | 4063 | 50 |
| H(29B) | -1859 | 245 | 3822 | 58 | C(47) | 2495(1) | -1359(1) | 4222(1) | 21(1) |
| H(29C) | -1612 | -544 | 3509 | 58 | C(48) | 2429(1) | -2005(1) | 3921(1) | 24(1) |
| C(30) | 5274(1) | 144(1) | 4353(1) | 18(1) | C(49) | 1630(1) | -2612(1) | 3868(1) | 32(1) |
| C(31) | 4857(1) | -602(1) | 4567(1) | 20(1) | H(49) | 1566 | -3043 | 3662 | 38 |
| H(31) | 5211 | -818 | 4773 | 24 | C(50) | 928(1) | -2597(1) | 4109(1) | 34(1) |
| C(32) | 3989(1) | -1070(1) | 4515(1) | 20(1) | H(50) | 397 | -3027 | 4072 | 40 |
| C(33) | 6306(1) | 295(1) | 4412(1) | 24(1) | C(51) | 1003(1) | -1960(1) | 4403(1) | 29(1) |
| H(33A) | 6630 | -24 | 4215 | 36 | H(51) | 517 | -1951 | 4567 | 35 |
| H(33B) | 6513 | -36 | 4637 | 36 | C(52) | 1781(1) | -1322(1) | 4466(1) | 24(1) |
| H(33C) | 6448 | 1042 | 4422 | 36 | C(53) | 3207(1) | -2077(1) | 3658(1) | 27(1) |
| C(34) | 3826(1) | -2046(1) | 4731(1) | 29(1) | H(53) | 3438 | -1353 | 3616 | 32 |
| H(34A) | 3615 | -2611 | 4571 | 43 | C(54) | 4031(1) | -2727(2) | 3807(1) | 37(1) |
| H(34B) | 3350 | -1907 | 4904 | 43 | H(54A) | 4497 | -2793 | 3626 | 56 |
| H(34C) | 4405 | -2252 | 4857 | 43 | H(54B) | 3812 | -3422 | 3873 | 56 |
| C(35) | 5347(1) | 1258(1) | 3844(1) | 17(1) | H(54C) | 4308 | -2380 | 4019 | 56 |
| C(36) | 5608(1) | 728(1) | 3537(1) | 19(1) | C(55) | 2886(1) | -2525(1) | 3296(1) | 33(1) |
| C(37) | 6101(1) | 1278(1) | 3286(1) | 24(1) | H(55A) | 2339 | -2141 | 3204 | 49 |
| H(37) | 6275 | 935 | 3074 | 28 | H(55B) | 2727 | -3263 | 3323 | 49 |
| C(38) | 6342(1) | 2310(1) | 3339(1) | 28(1) | H(55C) | 3387 | -2458 | 3128 | 49 |
| H(38) | 6676 | 2672 | 3164 | 33 | C(56) | 1811(1) | -618(2) | 4792(1) | 28(1) |
| C(39) | 6097(1) | 2815(1) | 3646(1) | 26(1) | H(56) | 2456 | -335 | 4824 | 34 |
| H(39) | 6270 | 3524 | 3681 | 32 | C(57) | 1576(1) | -1200(2) | 5134(1) | 42(1) |
| C(40) | 5599(1) | 2306(1) | 3907(1) | 21(1) | H(57A) | 920 | -1406 | 5120 | 63 |
| C(41) | 5355(1) | -407(1) | 3465(1) | 21(1) | H(57B) | 1691 | -742 | 5340 | 63 |
| H(41) | 5014 | -672 | 3674 | 25 | H(57C) | 1966 | -1824 | 5160 | 63 |
| C(42) | 6211(1) | -1101(2) | 3423(1) | 32(1) | C(58) | 1147(1) | 305(2) | 4735(1) | 33(1) |
| H(42A) | 6011 | -1819 | 3373 | 48 | H(58A) | 1322 | 704 | 4525 | 49 |
| H(42B) | 6599 | -1085 | 3644 | 48 | H(58B) | 1183 | 757 | 4946 | 49 |
| H(42C) | 6569 | -842 | 3225 | 48 | H(58C) | 510 | 46 | 4698 | 49 |
| C(43) | 4706(1) | -480(2) | 3135(1) | 31(1) | C(59) | 1965(1) | 2390(1) | 3960(1) | 28(1) |
| H(43A) | 5023 | -213 | 2928 | 47 | C(60) | 1330(4) | 3588(4) | 4422(2) | 31(1) |
| H(43B) | 4147 | -64 | 3172 | 47 | H(60) | 788 | 3997 | 4322 | 37 |
| H(43C) | 4530 | -1210 | 3094 | 47 | C(61) | 1038(2) | 3042(3) | 4756(1) | 35(1) |
| C(44) | 5385(1) | 2891(1) | 4244(1) | 24(1) | H(61A) | 1557 | 2609 | 4855 | 42 |
| H(44) | 5119 | 2382 | 4415 | 29 | H(61B) | 505 | 2576 | 4698 | 42 |
| C(45) | 6265(1) | 3364(1) | 4424(1) | 31(1) | C(62) | 764(3) | 3842(3) | 5032(1) | 44(1) |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| H(62A) | 201 | 4218 | 4943 | 53 | H(63C) | 2099 | 5787 | 5158 | 64 |
| H(62B) | 612 | 3478 | 5255 | 53 | H(63D) | 2630 | 4691 | 5141 | 64 |
| C(63) | 1531(3) | 4615(3) | 5111(1) | 40(1) | C(64') | 2447(6) | 5389(5) | 4646(2) | 46(2) |
| H(63A) | 1311 | 5148 | 5279 | 49 | H(64C) | 3079 | 5694 | 4659 | 56 |
| H(63B) | 2066 | 4250 | 5229 | 49 | H(64D) | 2018 | 5912 | 4537 | 56 |
| C(64) | 1842(2) | 5144(2) | 4781(1) | 36(1) | C(65') | 2444(6) | 4406(6) | 4412(2) | 32(2) |
| H(64A) | 2371 | 5611 | 4843 | 44 | H(65C) | 2597 | 4600 | 4165 | 38 |
| H(64B) | 1329 | 5577 | 4678 | 44 | H(65D) | 2927 | 3916 | 4505 | 38 |
| C(65) | 2137(3) | 4350(3) | 4503(1) | 33(1) | C11 | 2747(2) | 1102(2) | 1000(1) | 56(1) |
| H(65A) | 2691 | 3961 | 4595 | 40 | H11 | 2856 | 474 | 1129 | 67 |
| H(65B) | 2301 | 4715 | 4281 | 40 | C21 | 1925(2) | 1635(2) | 1035(1) | 54(1) |
| C(60') | 1519(8) | 3879(9) | 4405(4) | 27(2) | H21 | 1474 | 1384 | 1191 | 65 |
| H(60') | 1051 | 4359 | 4288 | 32 | C31 | 1760(2) | 2544(2) | 840(1) | 49(1) |
| C(61') | 1201(5) | 3624(8) | 4784(2) | 41(2) | H31 | 1198 | 2921 | 863 | 59 |
| H(61C) | 566 | 3329 | 4768 | 49 | C41 | 2421(2) | 2889(2) | 612(1) | 47(1) |
| H(61D) | 1621 | 3094 | 4895 | 49 | H41 | 2306 | 3498 | 473 | 56 |
| C(62') | 1207(7) | 4557(10) | 5007(3) | 56(3) | C51 | 3242(2) | 2361(2) | 586(1) | 47(1) |
| H(62C) | 1055 | 4356 | 5253 | 67 | H51 | 3698 | 2613 | 431 | 56 |
| H(62D) | 717 | 5039 | 4915 | 67 | C61 | 3407(2) | 1473(2) | 780(1) | 50(1) |
| C(63') | 2152(6) | 5134(7) | 5019(2) | 53(2) | H61 | 3980 | 1114 | 763 | 61 |

Tabelle 13: Anisotropic displacement parameters ($Å^2 \times 10^3$) for jus_159bm. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Br(1) | 21(1) | 28(1) | 16(1) | 2(1) | 3(1) | 7(1) | C(10) | 22(1) | 35(1) | 26(1) | 1(1) | 8(1) | 2(1) |
| Br(2) | 28(1) | 18(1) | 34(1) | -2(1) | 11(1) | 0(1) | C(11) | 20(1) | 27(1) | 17(1) | 0(1) | 3(1) | 0(1) |
| Ga(1) | 13(1) | 17(1) | 14(1) | 1(1) | 2(1) | 2(1) | C(12) | 36(1) | 20(1) | 21(1) | 0(1) | 3(1) | -6(1) |
| Ga(2) | 13(1) | 19(1) | 16(1) | 5(1) | 3(1) | 2(1) | C(13) | 80(2) | 25(1) | 27(1) | 2(1) | -1(1) | 4(1) |
| Si(1) | 20(1) | 20(1) | 22(1) | 5(1) | -5(1) | -1(1) | C(14) | 44(1) | 31(1) | 31(1) | -4(1) | 4(1) | -15(1) |
| N(1) | 17(1) | 18(1) | 14(1) | 0(1) | 2(1) | 1(1) | C(15) | 20(1) | 29(1) | 20(1) | -6(1) | 1(1) | 3(1) |
| N(2) | 16(1) | 22(1) | 14(1) | 0(1) | 2(1) | 4(1) | C(16) | 41(1) | 45(1) | 20(1) | -9(1) | 2(1) | 8(1) |
| N(3) | 15(1) | 19(1) | 15(1) | 3(1) | 2(1) | 0(1) | C(17) | 19(1) | 26(1) | 29(1) | -8(1) | 2(1) | 1(1) |
| N(4) | 15(1) | 22(1) | 20(1) | 6(1) | 2(1) | 0(1) | C(18) | 15(1) | 25(1) | 16(1) | 0(1) | 1(1) | 6(1) |
| N(5) | 41(1) | 45(1) | 34(1) | -9(1) | -13(1) | 17(1) | C(19) | 21(1) | 25(1) | 19(1) | 0(1) | 0(1) | 6(1) |
| C(1) | 25(1) | 17(1) | 14(1) | -1(1) | 1(1) | 2(1) | C(20) | 28(1) | 29(1) | 28(1) | -6(1) | 4(1) | 9(1) |
| C(2) | 23(1) | 27(1) | 15(1) | -1(1) | -3(1) | 7(1) | C(21) | 32(1) | 41(1) | 28(1) | -8(1) | 13(1) | 6(1) |
| C(3) | 19(1) | 28(1) | 19(1) | 0(1) | -1(1) | 7(1) | C(22) | 26(1) | 40(1) | 23(1) | 1(1) | 10(1) | 2(1) |
| C(4) | 33(1) | 31(1) | 14(1) | 3(1) | 3(1) | 0(1) | C(23) | 16(1) | 28(1) | 17(1) | 0(1) | 2(1) | 2(1) |
| C(5) | 23(1) | 85(2) | 22(1) | -1(1) | -2(1) | 24(1) | C(24) | 34(1) | 24(1) | 25(1) | 3(1) | 5(1) | 6(1) |
| C(6) | 18(1) | 24(1) | 14(1) | 1(1) | 4(1) | -2(1) | C(25) | 37(3) | 21(2) | 30(2) | 0(1) | 2(2) | -5(2) |
| C(7) | 27(1) | 24(1) | 18(1) | 3(1) | 4(1) | -6(1) | C(26) | 49(3) | 42(3) | 54(3) | 25(2) | -3(2) | 9(2) |
| C(8) | 27(1) | 34(1) | 30(1) | 4(1) | 4(1) | -13(1) | C(25') | 49(7) | 49(7) | 35(4) | -14(4) | 16(5) | -20(5) |
| C(9) | 23(1) | 41(1) | 34(1) | 6(1) | 9(1) | -5(1) | C(26') | 47(5) | 39(5) | 68(7) | 30(5) | -3(4) | 6(4) |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| C(27) | 17(1) | 28(1) | 22(1) | 2(1) | 2(1) | -1(1) | C(53) | 26(1) | 20(1) | 35(1) | -1(1) | 8(1) | -1(1) |
| C(28) | 32(1) | 34(1) | 31(1) | 8(1) | -1(1) | 2(1) | C(54) | 29(1) | 34(1) | 48(1) | -3(1) | 8(1) | 8(1) |
| C(29) | 21(1) | 43(1) | 52(1) | -6(1) | 4(1) | -6(1) | C(55) | 36(1) | 22(1) | 41(1) | -4(1) | 7(1) | -3(1) |
| C(30) | 15(1) | 21(1) | 17(1) | 1(1) | 2(1) | 3(1) | C(56) | 18(1) | 47(1) | 20(1) | 9(1) | 4(1) | -3(1) |
| C(31) | 18(1) | 25(1) | 18(1) | 6(1) | 2(1) | 3(1) | C(57) | 24(1) | 76(2) | 25(1) | 18(1) | 5(1) | -7(1) |
| C(32) | 18(1) | 24(1) | 20(1) | 7(1) | 4(1) | 2(1) | C(58) | 21(1) | 50(1) | 27(1) | -5(1) | 4(1) | 2(1) |
| C(33) | 16(1) | 29(1) | 26(1) | 7(1) | -1(1) | 1(1) | C(59) | 28(1) | 28(1) | 28(1) | -2(1) | -11(1) | 6(1) |
| C(34) | 23(1) | 32(1) | 31(1) | 18(1) | 3(1) | 1(1) | C(60) | 27(2) | 35(3) | 31(2) | -18(2) | -8(2) | 14(2) |
| C(35) | 14(1) | 22(1) | 16(1) | 4(1) | 2(1) | 0(1) | C(61) | 29(1) | 26(2) | 51(2) | -6(1) | 4(1) | 9(1) |
| C(36) | 16(1) | 25(1) | 17(1) | 2(1) | 2(1) | 0(1) | C(62) | 49(2) | 38(2) | 45(2) | 2(1) | 19(2) | 22(2) |
| C(37) | 21(1) | 31(1) | 19(1) | 2(1) | 5(1) | -2(1) | C(63) | 62(3) | 35(2) | 25(2) | -11(1) | -4(2) | 20(2) |
| C(38) | 28(1) | 32(1) | 24(1) | 8(1) | 8(1) | -5(1) | C(64) | 47(2) | 25(1) | 36(2) | -6(1) | -11(1) | 3(1) |
| C(39) | 31(1) | 24(1) | 24(1) | 5(1) | 4(1) | -5(1) | C(65) | 30(2) | 40(2) | 29(2) | 2(1) | 2(1) | 7(2) |
| C(40) | 22(1) | 22(1) | 19(1) | 4(1) | 2(1) | -1(1) | C(60') | 22(4) | 25(5) | 34(3) | -19(3) | -4(3) | 8(3) |
| C(41) | 19(1) | 25(1) | 20(1) | -2(1) | 3(1) | -3(1) | C(61') | 36(3) | 55(5) | 32(3) | -11(3) | -3(2) | 1(3) |
| C(42) | 26(1) | 27(1) | 43(1) | -3(1) | 6(1) | 1(1) | C(62') | 38(4) | 97(7) | 32(5) | -36(5) | -9(3) | 19(4) |
| C(43) | 32(1) | 36(1) | 26(1) | -2(1) | -4(1) | -6(1) | C(63') | 59(5) | 51(4) | 49(4) | -28(3) | -17(4) | 17(4) |
| C(44) | 31(1) | 22(1) | 21(1) | 2(1) | 2(1) | -2(1) | C(64') | 47(4) | 31(3) | 59(4) | -10(3) | -23(3) | 7(3) |
| C(45) | 40(1) | 26(1) | 25(1) | 3(1) | -2(1) | -8(1) | C(65') | 28(4) | 24(3) | 43(4) | -1(3) | 0(3) | 3(3) |
| C(46) | 40(1) | 28(1) | 32(1) | -5(1) | 1(1) | 7(1) | C11 | 106(2) | 36(1) | 26(1) | 6(1) | 8(1) | 11(1) |
| C(47) | 17(1) | 21(1) | 26(1) | 9(1) | 3(1) | 0(1) | C21 | 80(2) | 44(1) | 39(1) | -3(1) | 24(1) | -14(1) |
| C(48) | 22(1) | 18(1) | 35(1) | 6(1) | 5(1) | 1(1) | C31 | 45(1) | 40(1) | 63(2) | -9(1) | 1(1) | -7(1) |
| C(49) | 26(1) | 21(1) | 49(1) | -1(1) | 5(1) | -3(1) | C41 | 55(1) | 35(1) | 49(1) | 13(1) | -18(1) | -16(1) |
| C(50) | 22(1) | 26(1) | 53(1) | 6(1) | 4(1) | -5(1) | C51 | 49(1) | 55(1) | 37(1) | 8(1) | -2(1) | -16(1) |
| C(51) | 18(1) | 33(1) | 37(1) | 12(1) | 5(1) | -2(1) | C61 | 62(2) | 55(1) | 34(1) | -3(1) | -3(1) | 10(1) |
| C(52) | 18(1) | 31(1) | 24(1) | 11(1) | 3(1) | 0(1) | | | | | | | |

T

abelle 14: Bond lengths [Å] for jus_159bm.

| | | | | | |
|---|---|---|---|---|---|
| Br(1)-Ga(2) | 2.4418(4) | C(12)-C(14) | 1.528(2) | C(44)-C(46) | 1.535(2) |
| Br(2)-Ga(1) | 2.3962(4) | C(12)-C(13) | 1.532(3) | C(44)-C(45) | 1.536(2) |
| Ga(1)-N(2) | 1.9562(12) | C(15)-C(17) | 1.530(2) | C(47)-C(48) | 1.403(2) |
| Ga(1)-N(1) | 1.9975(12) | C(15)-C(16) | 1.534(2) | C(47)-C(52) | 1.406(2) |
| Ga(1)-Si(1) | 2.4107(5) | C(18)-C(23) | 1.400(2) | C(48)-C(49) | 1.396(2) |
| Ga(2)-N(4) | 1.9651(13) | C(18)-C(19) | 1.406(2) | C(48)-C(53) | 1.527(2) |
| Ga(2)-N(3) | 1.9712(12) | C(19)-C(20) | 1.393(2) | C(49)-C(50) | 1.386(3) |
| Ga(2)-Si(1) | 2.4058(5) | C(19)-C(24) | 1.520(2) | C(50)-C(51) | 1.372(3) |
| Si(1)-C(59) | 1.8419(19) | C(20)-C(21) | 1.382(3) | C(51)-C(52) | 1.399(2) |
| N(1)-C(1) | 1.3254(19) | C(21)-C(22) | 1.381(3) | C(52)-C(56) | 1.518(3) |
| N(1)-C(6) | 1.4479(19) | C(22)-C(23) | 1.398(2) | C(53)-C(55) | 1.526(3) |
| N(2)-C(3) | 1.3472(19) | C(23)-C(27) | 1.511(2) | C(53)-C(54) | 1.537(3) |
| N(2)-C(18) | 1.4455(18) | C(24)-C(25') | 1.379(12) | C(56)-C(58) | 1.531(3) |
| N(3)-C(30) | 1.3315(19) | C(24)-C(26) | 1.532(5) | C(56)-C(57) | 1.536(2) |
| N(3)-C(35) | 1.4483(18) | C(24)-C(25) | 1.593(6) | C(60)-C(61) | 1.511(7) |
| N(4)-C(32) | 1.3432(19) | C(24)-C(26') | 1.598(10) | C(60)-C(65) | 1.539(6) |
| N(4)-C(47) | 1.451(2) | C(27)-C(28) | 1.527(2) | C(61)-C(62) | 1.521(4) |
| N(5)-C(59) | 1.173(2) | C(27)-C(29) | 1.531(2) | C(62)-C(63) | 1.504(6) |
| N(5)-C(60) | 1.377(6) | C(30)-C(31) | 1.399(2) | C(63)-C(64) | 1.498(5) |
| N(5)-C(60') | 1.582(11) | C(30)-C(33) | 1.507(2) | C(64)-C(65) | 1.530(5) |
| C(1)-C(2) | 1.406(2) | C(31)-C(32) | 1.394(2) | C(60')-C(65') | 1.494(11) |
| C(1)-C(4) | 1.511(2) | C(32)-C(34) | 1.515(2) | C(60')-C(61') | 1.547(15) |
| C(2)-C(3) | 1.381(2) | C(35)-C(36) | 1.403(2) | C(61')-C(62') | 1.459(12) |
| C(3)-C(5) | 1.512(2) | C(35)-C(40) | 1.409(2) | C(62')-C(63') | 1.549(13) |
| C(6)-C(11) | 1.405(2) | C(36)-C(37) | 1.395(2) | C(63')-C(64') | 1.516(13) |
| C(6)-C(7) | 1.409(2) | C(36)-C(41) | 1.521(2) | C(64')-C(65') | 1.537(10) |
| C(7)-C(8) | 1.395(2) | C(37)-C(38) | 1.380(2) | C11-C61 | 1.369(4) |
| C(7)-C(12) | 1.523(2) | C(38)-C(39) | 1.380(2) | C11-C21 | 1.378(4) |
| C(8)-C(9) | 1.378(3) | C(39)-C(40) | 1.399(2) | C21-C31 | 1.391(4) |
| C(9)-C(10) | 1.385(3) | C(40)-C(44) | 1.514(2) | C31-C41 | 1.377(3) |
| C(10)-C(11) | 1.400(2) | C(41)-C(43) | 1.523(2) | C41-C51 | 1.370(4) |
| C(11)-C(15) | 1.525(2) | C(41)-C(42) | 1.534(2) | C51-C61 | 1.368(3) |

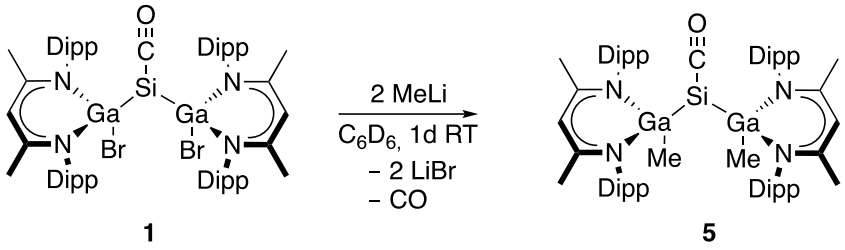
Tabelle 15: Bond angles [°] for jus_159bm.

| | | | | | |
|---|---|---|---|---|---|
| N(2)-Ga(1)-N(1) | 94.32(5) | C(9)-C(8)-C(7) | 121.33(17) | C(40)-C(35)-N(3) | 120.97(13) |
| N(2)-Ga(1)-Br(2) | 101.73(4) | C(8)-C(9)-C(10) | 120.06(16) | C(37)-C(36)-C(35) | 118.12(15) |
| N(1)-Ga(1)-Br(2) | 100.95(4) | C(9)-C(10)-C(11) | 121.24(16) | C(37)-C(36)-C(41) | 119.28(14) |
| N(2)-Ga(1)-Si(1) | 120.88(4) | C(10)-C(11)-C(6) | 117.70(15) | C(35)-C(36)-C(41) | 122.58(13) |
| N(1)-Ga(1)-Si(1) | 111.24(4) | C(10)-C(11)-C(15) | 118.56(14) | C(38)-C(37)-C(36) | 121.33(15) |
| Br(2)-Ga(1)-Si(1) | 122.592(13) | C(6)-C(11)-C(15) | 123.72(13) | C(39)-C(38)-C(37) | 119.88(15) |
| N(4)-Ga(2)-N(3) | 95.56(5) | C(7)-C(12)-C(14) | 113.00(16) | C(38)-C(39)-C(40) | 121.47(16) |
| N(4)-Ga(2)-Si(1) | 137.65(4) | C(7)-C(12)-C(13) | 110.77(14) | C(39)-C(40)-C(35) | 117.68(14) |
| N(3)-Ga(2)-Si(1) | 113.43(4) | C(14)-C(12)-C(13) | 110.21(15) | C(39)-C(40)-C(44) | 118.87(14) |
| N(4)-Ga(2)-Br(1) | 99.02(4) | C(11)-C(15)-C(17) | 111.57(13) | C(35)-C(40)-C(44) | 123.42(13) |
| N(3)-Ga(2)-Br(1) | 95.80(4) | C(11)-C(15)-C(16) | 110.78(15) | C(36)-C(41)-C(43) | 109.70(14) |
| Si(1)-Ga(2)-Br(1) | 107.522(17) | C(17)-C(15)-C(16) | 109.88(14) | C(36)-C(41)-C(42) | 112.66(13) |
| C(59)-Si(1)-Ga(2) | 91.68(6) | C(23)-C(18)-C(19) | 121.85(14) | C(43)-C(41)-C(42) | 110.45(14) |
| C(59)-Si(1)-Ga(1) | 98.65(6) | C(23)-C(18)-N(2) | 120.30(14) | C(40)-C(44)-C(46) | 111.27(14) |
| Ga(2)-Si(1)-Ga(1) | 131.59(2) | C(19)-C(18)-N(2) | 117.79(13) | C(40)-C(44)-C(45) | 111.66(14) |
| C(1)-N(1)-C(6) | 119.55(12) | C(20)-C(19)-C(18) | 118.12(15) | C(46)-C(44)-C(45) | 109.89(15) |
| C(1)-N(1)-Ga(1) | 120.28(10) | C(20)-C(19)-C(24) | 119.84(15) | C(48)-C(47)-C(52) | 121.20(15) |
| C(6)-N(1)-Ga(1) | 119.91(9) | C(18)-C(19)-C(24) | 122.04(14) | C(48)-C(47)-N(4) | 118.79(14) |
| C(3)-N(2)-C(18) | 116.51(12) | C(21)-C(20)-C(19) | 120.94(17) | C(52)-C(47)-N(4) | 120.01(15) |
| C(3)-N(2)-Ga(1) | 118.92(10) | C(22)-C(21)-C(20) | 120.12(16) | C(49)-C(48)-C(47) | 118.18(15) |
| C(18)-N(2)-Ga(1) | 124.56(9) | C(21)-C(22)-C(23) | 121.31(17) | C(49)-C(48)-C(53) | 119.82(16) |
| C(30)-N(3)-C(35) | 118.47(12) | C(22)-C(23)-C(18) | 117.65(15) | C(47)-C(48)-C(53) | 121.99(14) |
| C(30)-N(3)-Ga(2) | 117.88(10) | C(22)-C(23)-C(27) | 120.26(15) | C(50)-C(49)-C(48) | 121.20(18) |
| C(35)-N(3)-Ga(2) | 123.61(9) | C(18)-C(23)-C(27) | 122.08(14) | C(51)-C(50)-C(49) | 119.90(17) |
| C(32)-N(4)-C(47) | 118.66(13) | C(25')-C(24)-C(19) | 113.9(6) | C(50)-C(51)-C(52) | 121.32(16) |
| C(32)-N(4)-Ga(2) | 117.00(10) | C(19)-C(24)-C(26) | 115.0(2) | C(51)-C(52)-C(47) | 118.19(17) |
| C(47)-N(4)-Ga(2) | 123.59(10) | C(19)-C(24)-C(25) | 110.1(2) | C(51)-C(52)-C(56) | 118.68(15) |
| C(59)-N(5)-C(60) | 161.5(3) | C(26)-C(24)-C(25) | 106.0(3) | C(47)-C(52)-C(56) | 123.13(15) |
| C(59)-N(5)-C(60') | 145.6(5) | C(25')-C(24)-C(26') | 114.9(6) | C(55)-C(53)-C(48) | 113.16(15) |
| N(1)-C(1)-C(2) | 123.54(13) | C(19)-C(24)-C(26') | 106.3(4) | C(55)-C(53)-C(54) | 108.81(15) |
| N(1)-C(1)-C(4) | 121.33(14) | C(23)-C(27)-C(28) | 111.20(14) | C(48)-C(53)-C(54) | 111.88(15) |
| C(2)-C(1)-C(4) | 115.13(14) | C(23)-C(27)-C(29) | 112.77(14) | C(52)-C(56)-C(58) | 110.61(14) |
| C(3)-C(2)-C(1) | 128.02(14) | C(28)-C(27)-C(29) | 109.94(15) | C(52)-C(56)-C(57) | 112.55(17) |
| N(2)-C(3)-C(2) | 124.03(14) | N(3)-C(30)-C(31) | 123.64(13) | C(58)-C(56)-C(57) | 109.44(15) |
| N(2)-C(3)-C(5) | 119.02(14) | N(3)-C(30)-C(33) | 119.66(13) | N(5)-C(59)-Si(1) | 172.24(17) |
| C(2)-C(3)-C(5) | 116.92(14) | C(31)-C(30)-C(33) | 116.65(13) | N(5)-C(60)-C(61) | 108.8(4) |
| C(11)-C(6)-C(7) | 121.72(14) | C(32)-C(31)-C(30) | 128.17(14) | N(5)-C(60)-C(65) | 111.7(4) |
| C(11)-C(6)-N(1) | 119.55(13) | N(4)-C(32)-C(31) | 124.31(14) | C(61)-C(60)-C(65) | 111.5(4) |
| C(7)-C(6)-N(1) | 118.73(13) | N(4)-C(32)-C(34) | 119.62(14) | C(60)-C(61)-C(62) | 109.9(3) |
| C(8)-C(7)-C(6) | 117.95(15) | C(31)-C(32)-C(34) | 116.04(14) | C(63)-C(62)-C(61) | 111.7(3) |
| C(8)-C(7)-C(12) | 119.72(15) | C(36)-C(35)-C(40) | 121.49(13) | C(64)-C(63)-C(62) | 112.3(3) |
| C(6)-C(7)-C(12) | 122.24(14) | C(36)-C(35)-N(3) | 117.54(13) | C(63)-C(64)-C(65) | 111.4(3) |

| | | | | | |
|---|---|---|---|---|---|
| C(64)-C(65)-C(60) | 109.1(3) | C(61')-C(62')-C(63') | 113.1(7) | C11-C21-C31 | 119.6(2) |
| C(65')-C(60')-C(61') | 112.2(9) | C(64')-C(63')-C(62') | 110.8(7) | C41-C31-C21 | 119.2(2) |
| C(65')-C(60')-N(5) | 109.5(9) | C(63')-C(64')-C(65') | 111.0(7) | C51-C41-C31 | 120.5(2) |
| C(61')-C(60')-N(5) | 110.2(7) | C(60')-C(65')-C(64') | 111.2(8) | C61-C51-C41 | 120.3(2) |
| C(62')-C(61')-C(60') | 111.1(8) | C61-C11-C21 | 120.4(2) | C51-C61-C11 | 120.0(2) |

## 5. [DDP(Me)Ga]$_2$Si–CO 5

### 5.1. Synthese [DDP(Me)Ga]$_2$–CO **5**




**1** **5**

Dipp = 2,6-$^i$Pr$_2$C$_6$H$_3$

Es wurden 100 mg [DDP(Br)Ga]$_2$Si–CO **1** (0.084 mmol) und 1.85 mg Methyllithium (0.168 mmol) in einem Schlenkrohr vorgelegt und 1 mL Toluol dem Gemisch beigefügt. Die Lösung wurde für zwei Tage bei Raumtemperatur gerührt, wobei ein farbloser Niederschlag entstand. Die Mutterlauge wurde vom Niederschlag entfernt und eingeengt. Es konnten orangene Kristalle von [DDP(Me)Ga]$_2$Si–CO **5** nach Lagerung bei 4.5 °C erhalten werden.

**Ausbeute**: 56.2 mg (0.053 mmol, 63 %)

**Smp**. 249°C (Zersetzung)

**Elementaranalyse** von $C_{61}H_{88}N_4Ga_2OSi$: gefunden (berechnet) C 68.7 (69.06), H 8.48 (8.36), N 5.33 (5.28) %.

**IR**: ν 3060, 2958, 2925, 2868, 1942, 1906, 1549, 1517, 1435, 1383, 1314, 1254, 1177, 1100, 1017, 935, 855, 794, 757, 697, 636, 536, 444 cm$^{-1}$.

**$^1$H NMR** ($C_6D_6$, 400 MHz): δ 7.15-6.97 (m, 12 H, $C_6H_3(^iPr)_2$), 4.76 (s, 2 H, γ-C*H*), 3.44 (sept, $^3J_{HH}$ = 6.8 Hz, 4 H, -C*H*(CH$_3$)$_2$), 3.17 (sept, $^3J_{HH}$ = 6.8 Hz, 4 H, -C*H*(CH$_3$)$_2$), 1.47 (s, 12 H, ArNCC*H$_3$*), 1.34 (d, $^3J_{HH}$ = 6.8 Hz, 12 H, -CH(C*H$_3$*)$_2$), 1.18 (d, $^3J_{HH}$ = 6.8 Hz, 12 H, -CH(C*H$_3$*)$_2$), 1.10 (d, $^3J_{HH}$ = 6.8 Hz, 12 H, -CH(C*H$_3$*)$_2$), 1.07 (d, $^3J_{HH}$ = 6.8 Hz, 12 H, -CH(C*H$_3$*)$_2$), 0.32 (s, 6 H, GaC*H$_3$*) ppm.

**$^{13}$C NMR** ($C_6D_6$, 101 MHz): δ 207.5 (Si*C*O), 168.0 (ArN*C*CH$_3$), 144.6 (N*C*C(CH(CH$_3$)$_2$)), 143.2, 143.0 (NC*C*(CH(CH$_3$)$_2$)), 126.8, 124.4, 124.1 (*C*$_6$H$_3$), 96.7 (γ-*C*H-), 29.4 (-*C*H(CH$_3$)$_2$), 27.7 (-CH(*C*H$_3$)$_2$), 27.6 (-*C*H(CH$_3$)$_2$), 25.2, 24.3, 24.2, (-CH(*C*H$_3$)$_2$), 24.2 (ArNC*C*H$_3$), –0.26 (Ga*C*H$_3$) ppm.

**$^{29}$Si NMR** ($C_6D_6$, 79 MHz): δ –285.2 ppm.

## 5.2. Spektren [DDP(Me)Ga]$_2$–CO **5**

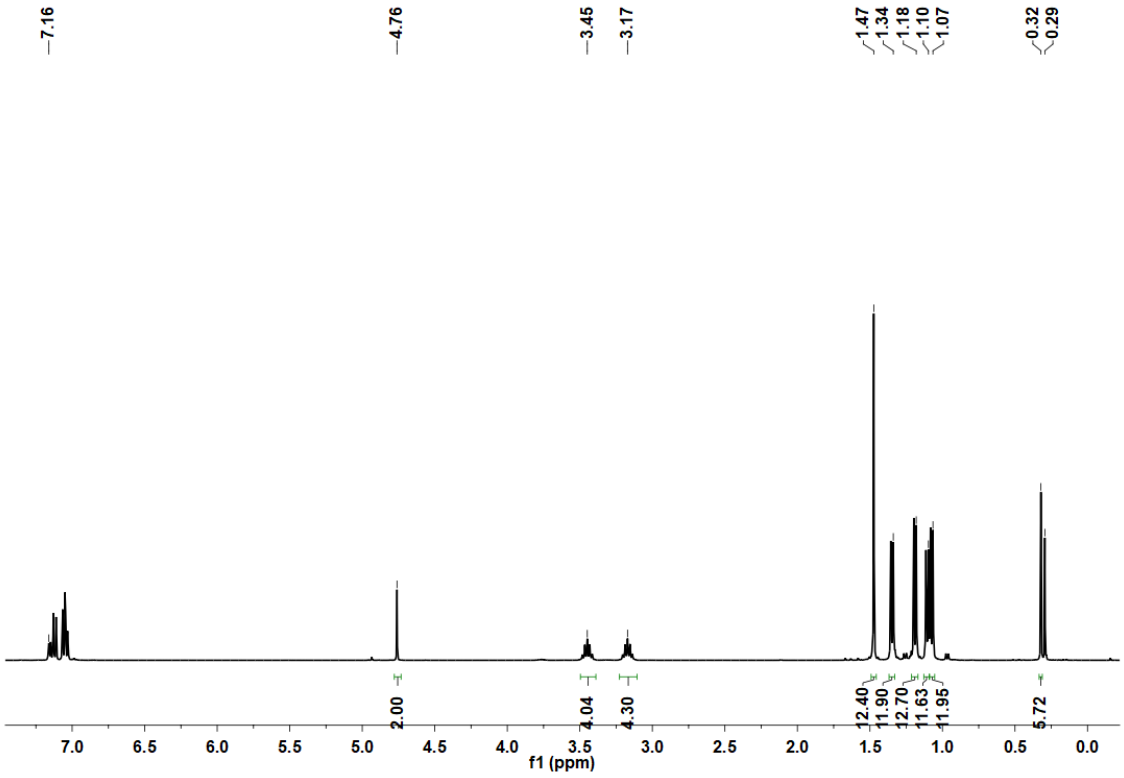



Abbildung 22: $^{1}$H-NMR-Spektrum von [DDP(Me)Ga]$_2$Si–CO **5** in $C_6D_6$.

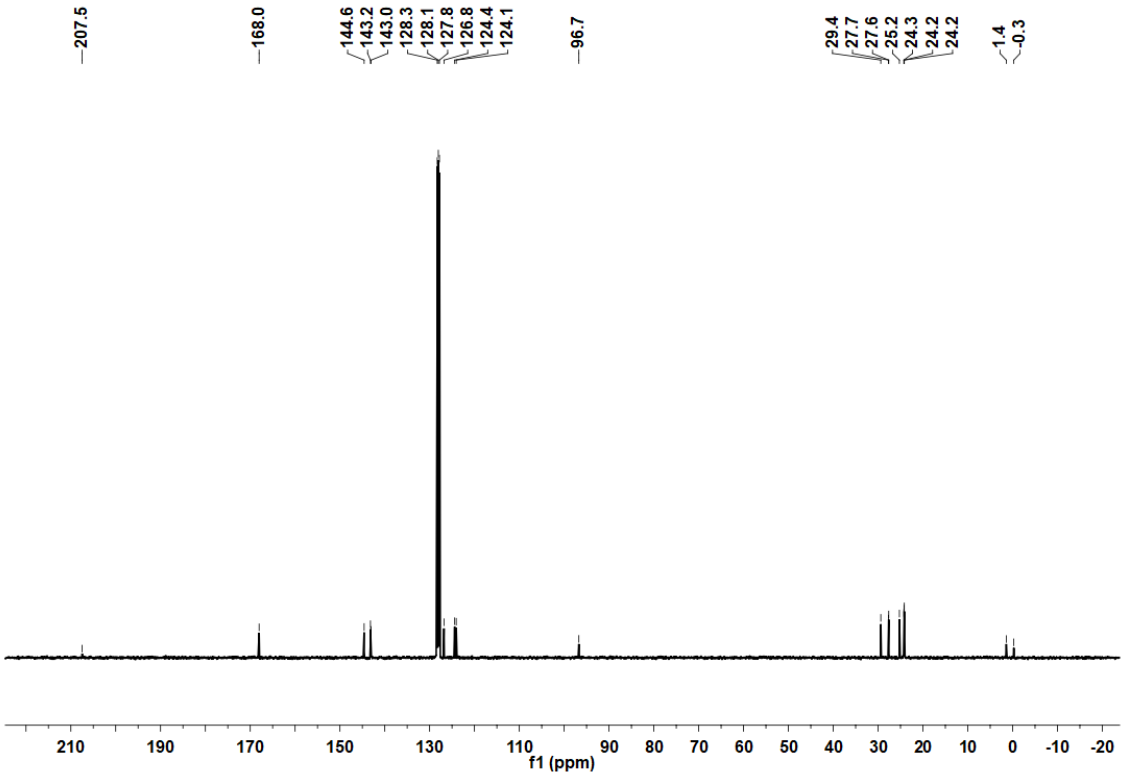



Abbildung 23: $^{13}$C-NMR-Spektrum von [DDP(Me)Ga]$_2$Si–CO **5** in $C_6D_6$.

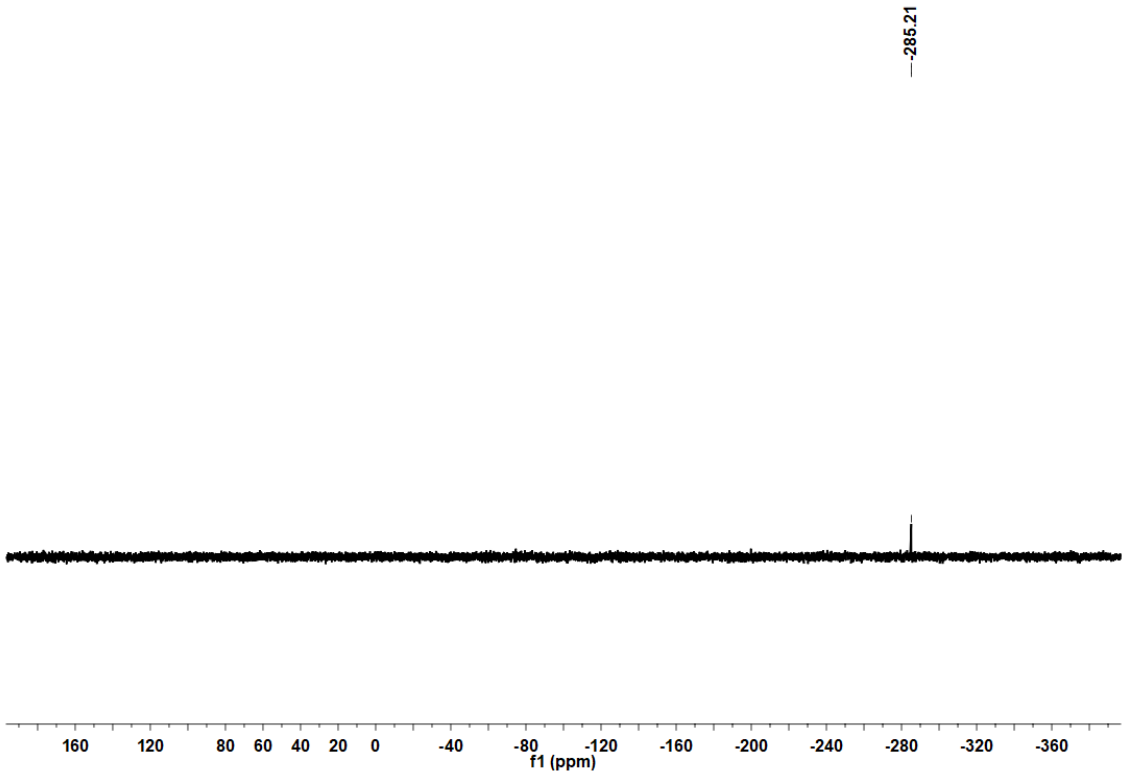



Abbildung 24: $^{29}$Si-NMR-Spektrum von [DDP(Me)Ga]$_2$Si–CO **5** in $C_6D_6$.

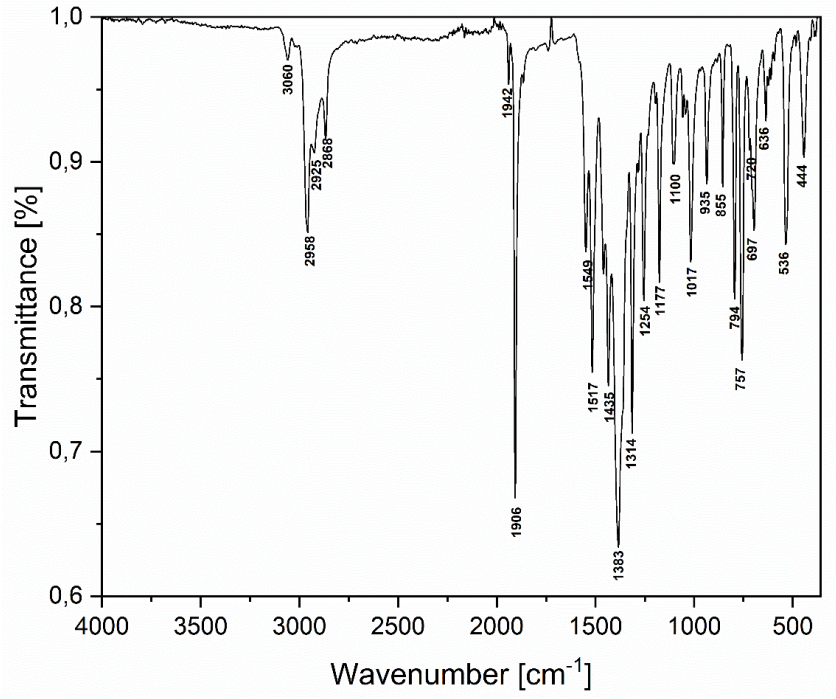



Abbildung 25: ATR-IR Spektrum von [DDP(Me)Ga]$_2$Si–CO **5**.

## 5.3. Kristallografische Daten $[DDP(Me)Ga]_2$–CO **5**

Tabelle 16: Crystal structure data

| **Identification code** | **jus_237m** |
|---|---|
| Empirical formula | C61 H88 Ga2 N4 O Si |
| Formula weight | 1060.88 |
| Density (calculated) | 1.202 g·cm$^{-3}$ |
| *F*(000) | 1132 |
| Temperature | 100(2) K |
| Crystal size | 0.253 × 0.235 × 0.154 mm |
| Crystal colour | orange |
| Crystal description | block |
| Wavelength | 1.54178 Å |
| Crystal system | triclinic |
| Space group | *P -1* |
| Unit cell dimensions | |
| *a* [Å] | 11.8953(3) |
| *b* [Å] | 13.8148(3) |
| *c* [Å] | 19.8705(6) |
| *α* [°] | 79.3889(14) |
| *β* [°] | 84.2512(15) |
| *γ* [°] | 66.0199(13) |
| Volume | 2931.32(14) Å$^3$ |
| *Z* | 2 |
| Cell measurement reflections used | 9840 |
| Cell measurement *θ* min/max | 3.55°/80.30° |
| Diffractometer control software | Bruker APEX3(v2017.3-0) |
| Diffractometer measurement device | Bruker D8 Venture (Photon II detector) |
| Diffractometer measurement method | Data collection strategy APEX 3/Queen |
| *θ* range for data collection | 2.263°- 81.691° |
| Completeness to *θ* = 67.679° | 99.9% |
| Completeness to $\theta_{max}$ = 81.691° | 98.7% |
| Index ranges | -14 ≤ *h* ≤ 12 |
| | -17 ≤ *k* ≤ 17 |
| | -25 ≤ *l* ≤ 25 |
| Computing data reduction | Bruker APEX3(v2017.3-0) |
| Absorption coefficient | 1.630 mm$^{-1}$ |
| Absorption correction | Semi-empirical from equivalents |
| Computation absorption correction | SADABS |
| Max./min. Transmission | 0.75/0.64 |
| $R_{merg}$ before/after correction | 0.0919/0.0683 |
| Computing structure solution | Bruker APEX3(v2017.3-0) |
| Computing structure refinement | SHELXL-2017/1 (Sheldrick, 2017) |

| | |
|---|---|
| Refinement method | Full-matrix least-squares on $F^2$ |
| Reflections collected | 280503 |
| Independent reflections | 12804 |
| $R_{int}$ | 0.0420 |
| Reflections with $I > 2\sigma(I)$ | 12396 |
| Restraints | 47 |
| Parameter | 674 |
| GooF | 1.025 |
| Weighting details | $w = 1/[\sigma^2(F_{obs}^2) + (0.0396P)^2 + 1.6627P]$ |
| | where $P = (F_{obs}^2 + 2F_{calc}^2)/3$ |
| $R_1$ [$I > 2\sigma(I)$] | 0.0291 |
| $wR_2$ [$I > 2\sigma(I)$] | 0.0768 |
| $R_1$ [all data] | 0.0298 |
| $wR_2$ [all data] | 0.0775 |
| Absolute structure parameter | |
| Largest diff. peak and hole | 0.749/-0.577 |

Tabelle 17: Atomic coordinates ( × $10^4$) and equivalent isotropic displacement parameters (Å$^2$ × $10^3$) for jus_237m. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | *x* | *y* | *z* | $U_{eq}$ | | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|---|---|---|---|---|
| Ga(1) | 7577(1) | 1934(1) | 7387(1) | 18(1) | H(15) | 7590 | -458 | 8151 | 47 |
| Ga(2) | 4044(1) | 4157(1) | 7006(1) | 19(1) | C(16) | 8394(2) | -1838(2) | 8842(1) | 61(1) |
| Si(1) | 5396(1) | 2323(1) | 7441(1) | 27(1) | H(16A) | 8400 | -2325 | 8538 | 92 |
| O(1) | 4939(1) | 2579(1) | 8875(1) | 66(1) | H(16B) | 8233 | -2117 | 9315 | 92 |
| N(1) | 8428(1) | 865(1) | 8188(1) | 22(1) | H(16C) | 9195 | -1785 | 8808 | 92 |
| N(2) | 8414(1) | 912(1) | 6716(1) | 19(1) | C(17) | 6161(2) | -828(2) | 8618(2) | 62(1) |
| N(3) | 2479(1) | 4068(1) | 6786(1) | 19(1) | H(17A) | 5523 | -123 | 8459 | 93 |
| N(4) | 3144(1) | 5313(1) | 7580(1) | 23(1) | H(17B) | 5940 | -1100 | 9081 | 93 |
| C(1) | 9609(1) | 237(1) | 8098(1) | 22(1) | H(17C) | 6232 | -1327 | 8308 | 93 |
| C(2) | 10159(1) | 36(1) | 7454(1) | 21(1) | C(18) | 7904(1) | 981(1) | 6070(1) | 22(1) |
| H(2) | 11033 | -284 | 7437 | 25 | C(19) | 8109(1) | 1641(1) | 5483(1) | 24(1) |
| C(3) | 9578(1) | 247(1) | 6829(1) | 20(1) | C(20) | 7626(1) | 1662(1) | 4864(1) | 30(1) |
| C(4) | 10420(1) | -305(1) | 8711(1) | 29(1) | H(20) | 7736 | 2118 | 4467 | 36 |
| H(4A) | 11218 | -818 | 8564 | 44 | C(21) | 6992(1) | 1030(1) | 4820(1) | 35(1) |
| H(4B) | 10024 | -686 | 9050 | 44 | H(21) | 6698 | 1034 | 4392 | 42 |
| H(4C) | 10545 | 236 | 8915 | 44 | C(22) | 6789(1) | 395(1) | 5398(1) | 34(1) |
| C(5) | 10352(1) | -346(1) | 6271(1) | 26(1) | H(22) | 6351 | -35 | 5365 | 40 |
| H(5A) | 9933 | -739 | 6109 | 39 | C(23) | 7215(1) | 371(1) | 6035(1) | 27(1) |
| H(5B) | 11153 | -854 | 6451 | 39 | C(24) | 8833(1) | 2327(1) | 5489(1) | 27(1) |
| H(5C) | 10472 | 167 | 5890 | 39 | H(24) | 9187 | 2158 | 5953 | 32 |
| C(6) | 7877(1) | 823(1) | 8868(1) | 26(1) | C(25) | 9897(2) | 2102(1) | 4955(1) | 34(1) |
| C(7) | 7858(1) | 1529(1) | 9294(1) | 30(1) | H(25A) | 9564 | 2316 | 4494 | 51 |
| C(8) | 7288(2) | 1474(2) | 9945(1) | 39(1) | H(25B) | 10424 | 1334 | 5025 | 51 |
| H(8) | 7259 | 1950 | 10242 | 46 | H(25C) | 10381 | 2512 | 5005 | 51 |
| C(9) | 6769(2) | 734(2) | 10158(1) | 45(1) | C(26) | 7992(2) | 3521(1) | 5360(1) | 42(1) |
| H(9) | 6388 | 705 | 10601 | 55 | H(26A) | 7703 | 3719 | 4890 | 63 |
| C(10) | 6798(2) | 39(2) | 9735(1) | 43(1) | H(26B) | 8451 | 3945 | 5426 | 63 |
| H(10) | 6438 | -466 | 9891 | 52 | H(26C) | 7284 | 3663 | 5682 | 63 |
| C(11) | 7347(1) | 63(1) | 9080(1) | 34(1) | C(27) | 6940(1) | -314(1) | 6664(1) | 33(1) |
| C(12) | 8399(2) | 2363(1) | 9078(1) | 34(1) | H(27) | 7064 | -47 | 7074 | 39 |
| H(12) | 8905 | 2194 | 8649 | 41 | C(28) | 7830(2) | -1489(1) | 6710(1) | 52(1) |
| C(13) | 7363(2) | 3483(1) | 8912(1) | 44(1) | H(28A) | 7751 | -1770 | 6307 | 78 |
| H(13A) | 7721 | 4019 | 8784 | 67 | H(28B) | 7636 | -1906 | 7125 | 78 |
| H(13B) | 6817 | 3647 | 9316 | 67 | H(28C) | 8676 | -1546 | 6728 | 78 |
| H(13C) | 6892 | 3496 | 8531 | 67 | C(29) | 5598(2) | -190(2) | 6691(1) | 52(1) |
| C(14) | 9245(2) | 2342(2) | 9619(1) | 47(1) | H(29A) | 5054 | 573 | 6634 | 78 |
| H(14A) | 9903 | 1624 | 9709 | 71 | H(29B) | 5414 | -550 | 7135 | 78 |
| H(14B) | 8766 | 2520 | 10043 | 71 | H(29C) | 5468 | -514 | 6323 | 78 |
| H(14C) | 9606 | 2868 | 9450 | 71 | C(30) | 1481(1) | 4966(1) | 6680(1) | 23(1) |
| C(15) | 7387(2) | -729(1) | 8629(1) | 39(1) | C(31) | 1374(1) | 5936(1) | 6867(1) | 27(1) |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| H(31) | 745 | 6571 | 6653 | 32 | C(50) | 4466(2) | 6045(1) | 9238(1) | 40(1) |
| C(32) | 2086(1) | 6073(1) | 7332(1) | 27(1) | H(50) | 4739 | 6229 | 9611 | 48 |
| C(33) | 375(1) | 4983(1) | 6360(1) | 31(1) | C(51) | 3676(2) | 5522(1) | 9354(1) | 38(1) |
| H(33A) | 555 | 4929 | 5873 | 47 | H(51) | 3417 | 5345 | 9811 | 45 |
| H(33B) | -332 | 5655 | 6409 | 47 | C(52) | 3247(1) | 5246(1) | 8819(1) | 30(1) |
| H(33C) | 182 | 4375 | 6590 | 47 | C(53) | 4834(2) | 6406(2) | 7305(1) | 36(1) |
| C(34) | 1601(2) | 7169(1) | 7552(1) | 39(1) | H(53) | 4346 | 6280 | 6975 | 43 |
| H(34A) | 2119 | 7551 | 7344 | 58 | C(54) | 6190(2) | 5712(2) | 7189(1) | 53(1) |
| H(34B) | 1617 | 7084 | 8052 | 58 | H(54A) | 6446 | 5914 | 6719 | 80 |
| H(34C) | 753 | 7582 | 7402 | 58 | H(54B) | 6312 | 4955 | 7264 | 80 |
| C(35) | 2451(1) | 3075(1) | 6674(1) | 21(1) | H(54C) | 6685 | 5822 | 7511 | 80 |
| C(36) | 2635(1) | 2798(1) | 6016(1) | 22(1) | C(55) | 4611(3) | 7602(2) | 7166(1) | 66(1) |
| C(37) | 2569(1) | 1833(1) | 5939(1) | 28(1) | H(55A) | 4816 | 7800 | 6684 | 98 |
| H(37) | 2689 | 1632 | 5496 | 33 | H(55B) | 5131 | 7732 | 7460 | 98 |
| C(38) | 2334(1) | 1171(1) | 6494(1) | 32(1) | H(55C) | 3744 | 8035 | 7263 | 98 |
| H(38) | 2270 | 530 | 6430 | 39 | C(53') | 5438(15) | 6019(13) | 7338(6) | 23(4) |
| C(39) | 2192(1) | 1439(1) | 7144(1) | 31(1) | H(53') | 5545 | 5372 | 7139 | 28 |
| H(39) | 2048 | 972 | 7524 | 37 | C(54') | 6730(14) | 5957(15) | 7403(9) | 34(4) |
| C(40) | 2256(1) | 2386(1) | 7249(1) | 25(1) | H(54D) | 7260 | 5228 | 7608 | 51 |
| C(41) | 2941(1) | 3483(1) | 5395(1) | 24(1) | H(54E) | 6700 | 6466 | 7696 | 51 |
| H(41) | 2832 | 4178 | 5531 | 29 | H(54F) | 7063 | 6136 | 6949 | 51 |
| C(42) | 4279(1) | 2925(1) | 5164(1) | 33(1) | C(55') | 4713(16) | 6981(14) | 6821(10) | 41(5) |
| H(42A) | 4389 | 2267 | 4995 | 50 | H(55D) | 5178 | 6958 | 6384 | 61 |
| H(42B) | 4816 | 2743 | 5551 | 50 | H(55E) | 4577 | 7643 | 6991 | 61 |
| H(42C) | 4493 | 3405 | 4796 | 50 | H(55F) | 3918 | 6961 | 6753 | 61 |
| C(43) | 2098(2) | 3726(1) | 4797(1) | 31(1) | C(56) | 2338(2) | 4706(1) | 8977(1) | 34(1) |
| H(43A) | 2291 | 4207 | 4420 | 47 | H(56) | 2446 | 4265 | 8610 | 41 |
| H(43B) | 1237 | 4072 | 4948 | 47 | C(57) | 1001(2) | 5531(2) | 8968(1) | 45(1) |
| H(43C) | 2227 | 3056 | 4640 | 47 | H(57A) | 799 | 5916 | 8501 | 68 |
| C(44) | 2110(1) | 2681(1) | 7962(1) | 28(1) | H(57B) | 901 | 6043 | 9275 | 68 |
| H(44) | 2653 | 3071 | 7975 | 33 | H(57C) | 449 | 5162 | 9123 | 68 |
| C(45) | 791(1) | 3450(1) | 8112(1) | 35(1) | C(58) | 2545(2) | 3959(2) | 9665(1) | 45(1) |
| H(45A) | 579 | 4117 | 7784 | 52 | H(58A) | 2027 | 3551 | 9704 | 68 |
| H(45B) | 728 | 3610 | 8578 | 52 | H(58B) | 2328 | 4385 | 10038 | 68 |
| H(45C) | 221 | 3115 | 8070 | 52 | H(58C) | 3412 | 3462 | 9695 | 68 |
| C(46) | 2526(2) | 1704(1) | 8528(1) | 40(1) | C(59) | 8388(1) | 2968(1) | 7188(1) | 24(1) |
| H(46A) | 1930 | 1369 | 8585 | 60 | H(59A) | 9278 | 2575 | 7130 | 35 |
| H(46B) | 2577 | 1931 | 8959 | 60 | H(59B) | 8222 | 3372 | 7568 | 35 |
| H(46C) | 3336 | 1185 | 8402 | 60 | H(59C) | 8064 | 3465 | 6766 | 35 |
| C(47) | 3641(1) | 5525(1) | 8148(1) | 26(1) | C(60) | 4568(1) | 4865(1) | 6140(1) | 28(1) |
| C(48) | 4439(2) | 6056(1) | 8023(1) | 33(1) | H(60A) | 4722 | 4421 | 5781 | 42 |
| C(49) | 4855(2) | 6299(1) | 8581(1) | 38(1) | H(60B) | 5323 | 4949 | 6214 | 42 |
| H(49) | 5414 | 6644 | 8503 | 45 | H(60C) | 3916 | 5571 | 5998 | 42 |

| | | | | |
|---|---|---|---|---|
| C(61) | 5178(1) | 2524(1) | 8307(1) | 39(1) |

Tabelle 18: Anisotropic displacement parameters (Å$^2$ × 10$^3$) for jus_237m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ga(1) | 18(1) | 18(1) | 17(1) | 1(1) | -3(1) | -6(1) | C(29) | 35(1) | 51(1) | 75(1) | 4(1) | -6(1) | -27(1) |
| Ga(2) | 21(1) | 16(1) | 19(1) | 1(1) | -6(1) | -8(1) | C(30) | 22(1) | 25(1) | 18(1) | 1(1) | -7(1) | -5(1) |
| Si(1) | 18(1) | 20(1) | 38(1) | 4(1) | -6(1) | -6(1) | C(31) | 29(1) | 19(1) | 24(1) | 2(1) | -10(1) | -1(1) |
| O(1) | 36(1) | 87(1) | 33(1) | 8(1) | 0(1) | 12(1) | C(32) | 35(1) | 16(1) | 24(1) | 1(1) | -7(1) | -5(1) |
| N(1) | 18(1) | 24(1) | 19(1) | 4(1) | -1(1) | -6(1) | C(33) | 23(1) | 34(1) | 31(1) | -5(1) | -11(1) | -4(1) |
| N(2) | 19(1) | 18(1) | 21(1) | 0(1) | -3(1) | -8(1) | C(34) | 50(1) | 18(1) | 40(1) | -5(1) | -16(1) | -2(1) |
| N(3) | 20(1) | 19(1) | 19(1) | 0(1) | -6(1) | -7(1) | C(35) | 17(1) | 20(1) | 26(1) | -1(1) | -6(1) | -9(1) |
| N(4) | 31(1) | 16(1) | 20(1) | 0(1) | -9(1) | -7(1) | C(36) | 19(1) | 22(1) | 26(1) | -2(1) | -7(1) | -8(1) |
| C(1) | 19(1) | 21(1) | 23(1) | 5(1) | -3(1) | -7(1) | C(37) | 28(1) | 27(1) | 32(1) | -7(1) | -6(1) | -12(1) |
| C(2) | 17(1) | 19(1) | 25(1) | 1(1) | -1(1) | -7(1) | C(38) | 34(1) | 27(1) | 44(1) | -5(1) | -6(1) | -19(1) |
| C(3) | 21(1) | 15(1) | 24(1) | 0(1) | 0(1) | -9(1) | C(39) | 32(1) | 30(1) | 36(1) | 2(1) | -4(1) | -20(1) |
| C(4) | 20(1) | 35(1) | 24(1) | 6(1) | -4(1) | -5(1) | C(40) | 21(1) | 27(1) | 28(1) | 1(1) | -5(1) | -12(1) |
| C(5) | 24(1) | 23(1) | 27(1) | -5(1) | -2(1) | -6(1) | C(41) | 26(1) | 22(1) | 23(1) | -3(1) | -4(1) | -8(1) |
| C(6) | 17(1) | 30(1) | 20(1) | 10(1) | -1(1) | -4(1) | C(42) | 30(1) | 31(1) | 36(1) | -6(1) | 3(1) | -11(1) |
| C(7) | 25(1) | 35(1) | 18(1) | 6(1) | 0(1) | -4(1) | C(43) | 39(1) | 30(1) | 25(1) | -3(1) | -10(1) | -12(1) |
| C(8) | 31(1) | 48(1) | 20(1) | 6(1) | 2(1) | -4(1) | C(44) | 28(1) | 33(1) | 25(1) | 2(1) | -4(1) | -17(1) |
| C(9) | 29(1) | 60(1) | 26(1) | 16(1) | 6(1) | -7(1) | C(45) | 30(1) | 48(1) | 28(1) | -4(1) | 0(1) | -18(1) |
| C(10) | 28(1) | 54(1) | 35(1) | 21(1) | 1(1) | -16(1) | C(46) | 48(1) | 42(1) | 30(1) | 8(1) | -7(1) | -23(1) |
| C(11) | 21(1) | 37(1) | 32(1) | 15(1) | -3(1) | -8(1) | C(47) | 36(1) | 17(1) | 23(1) | -1(1) | -11(1) | -7(1) |
| C(12) | 46(1) | 38(1) | 15(1) | -2(1) | 3(1) | -16(1) | C(48) | 49(1) | 24(1) | 29(1) | -1(1) | -12(1) | -17(1) |
| C(13) | 64(1) | 36(1) | 24(1) | -4(1) | 6(1) | -13(1) | C(49) | 49(1) | 29(1) | 39(1) | -7(1) | -15(1) | -17(1) |
| C(14) | 66(1) | 66(1) | 19(1) | -4(1) | 0(1) | -38(1) | C(50) | 49(1) | 37(1) | 33(1) | -12(1) | -17(1) | -11(1) |
| C(15) | 35(1) | 38(1) | 43(1) | 14(1) | -4(1) | -20(1) | C(51) | 46(1) | 40(1) | 25(1) | -9(1) | -9(1) | -11(1) |
| C(16) | 41(1) | 49(1) | 84(2) | -6(1) | -9(1) | -9(1) | C(52) | 35(1) | 25(1) | 24(1) | -5(1) | -8(1) | -6(1) |
| C(17) | 36(1) | 51(1) | 101(2) | 1(1) | -11(1) | -21(1) | C(53) | 50(1) | 34(1) | 34(1) | 3(1) | -13(1) | -28(1) |
| C(18) | 21(1) | 20(1) | 25(1) | -6(1) | -5(1) | -5(1) | C(54) | 49(1) | 82(2) | 38(1) | -1(1) | -4(1) | -38(1) |
| C(19) | 23(1) | 23(1) | 23(1) | -6(1) | -4(1) | -4(1) | C(55) | 119(2) | 45(1) | 55(1) | 13(1) | -31(1) | -58(2) |
| C(20) | 26(1) | 36(1) | 24(1) | -10(1) | -4(1) | -4(1) | C(53') | 24(7) | 30(7) | 26(6) | -8(5) | -7(5) | -17(5) |
| C(21) | 23(1) | 47(1) | 33(1) | -22(1) | -5(1) | -5(1) | C(54') | 34(7) | 48(9) | 32(8) | -3(7) | -11(6) | -28(7) |
| C(22) | 22(1) | 38(1) | 46(1) | -22(1) | -3(1) | -9(1) | C(55') | 48(9) | 37(9) | 40(9) | 15(7) | -11(7) | -26(7) |
| C(23) | 20(1) | 24(1) | 37(1) | -10(1) | -3(1) | -6(1) | C(56) | 40(1) | 36(1) | 25(1) | -7(1) | -1(1) | -14(1) |
| C(24) | 38(1) | 25(1) | 18(1) | 0(1) | -7(1) | -13(1) | C(57) | 39(1) | 49(1) | 44(1) | -10(1) | -2(1) | -13(1) |
| C(25) | 45(1) | 42(1) | 22(1) | 0(1) | -3(1) | -26(1) | C(58) | 51(1) | 49(1) | 32(1) | -2(1) | 4(1) | -17(1) |
| C(26) | 64(1) | 26(1) | 31(1) | -1(1) | -16(1) | -11(1) | C(59) | 32(1) | 26(1) | 18(1) | -2(1) | 0(1) | -18(1) |
| C(27) | 28(1) | 28(1) | 47(1) | -6(1) | -3(1) | -15(1) | C(60) | 34(1) | 30(1) | 24(1) | 4(1) | -6(1) | -19(1) |
| C(28) | 51(1) | 28(1) | 67(1) | -2(1) | -1(1) | -9(1) | C(61) | 22(1) | 43(1) | 34(1) | 9(1) | -2(1) | 2(1) |

Tabelle 19: Bond lengths [Å] for jus_237m.

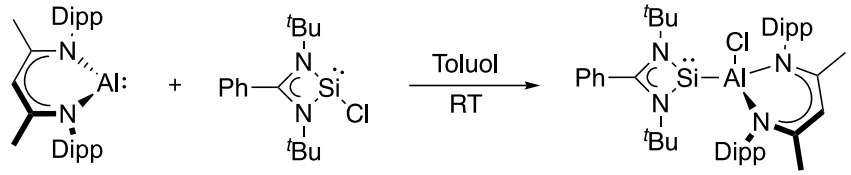
| | | | | | |
|---|---|---|---|---|---|
| Ga(1)-C(59) | 1.9898(13) | C(8)-C(9) | 1.381(3) | C(36)-C(37) | 1.4032(19) |
| Ga(1)-N(1) | 1.9963(11) | C(9)-C(10) | 1.377(3) | C(36)-C(41) | 1.5185(19) |
| Ga(1)-N(2) | 2.0130(11) | C(10)-C(11) | 1.398(2) | C(37)-C(38) | 1.381(2) |
| Ga(1)-Si(1) | 2.4213(4) | C(11)-C(15) | 1.519(3) | C(38)-C(39) | 1.384(2) |
| Ga(2)-C(60) | 1.9923(14) | C(12)-C(14) | 1.533(2) | C(39)-C(40) | 1.395(2) |
| Ga(2)-N(4) | 2.0082(11) | C(12)-C(13) | 1.538(2) | C(40)-C(44) | 1.520(2) |
| Ga(2)-N(3) | 2.0105(11) | C(15)-C(17) | 1.521(2) | C(41)-C(42) | 1.525(2) |
| Ga(2)-Si(1) | 2.4314(4) | C(15)-C(16) | 1.527(3) | C(41)-C(43) | 1.5333(19) |
| Si(1)-C(61) | 1.7703(18) | C(18)-C(19) | 1.4077(19) | C(44)-C(46) | 1.530(2) |
| O(1)-C(61) | 1.144(2) | C(18)-C(23) | 1.4090(19) | C(44)-C(45) | 1.532(2) |
| N(1)-C(1) | 1.3320(17) | C(19)-C(20) | 1.3992(19) | C(47)-C(48) | 1.397(2) |
| N(1)-C(6) | 1.4422(16) | C(19)-C(24) | 1.519(2) | C(47)-C(52) | 1.408(2) |
| N(2)-C(3) | 1.3279(16) | C(20)-C(21) | 1.384(2) | C(48)-C(49) | 1.404(2) |
| N(2)-C(18) | 1.4441(16) | C(21)-C(22) | 1.376(3) | C(48)-C(53) | 1.511(2) |
| N(3)-C(30) | 1.3228(17) | C(22)-C(23) | 1.398(2) | C(48)-C(53') | 1.708(15) |
| N(3)-C(35) | 1.4439(16) | C(23)-C(27) | 1.516(2) | C(49)-C(50) | 1.373(3) |
| N(4)-C(32) | 1.3374(18) | C(24)-C(26) | 1.530(2) | C(50)-C(51) | 1.380(3) |
| N(4)-C(47) | 1.4510(16) | C(24)-C(25) | 1.535(2) | C(51)-C(52) | 1.398(2) |
| C(1)-C(2) | 1.3994(18) | C(27)-C(28) | 1.527(2) | C(52)-C(56) | 1.524(2) |
| C(1)-C(4) | 1.5115(18) | C(27)-C(29) | 1.532(2) | C(53)-C(54) | 1.525(3) |
| C(2)-C(3) | 1.4075(18) | C(30)-C(31) | 1.410(2) | C(53)-C(55) | 1.538(3) |
| C(3)-C(5) | 1.5059(18) | C(30)-C(33) | 1.5082(18) | C(53')-C(55') | 1.520(13) |
| C(6)-C(7) | 1.397(2) | C(31)-C(32) | 1.399(2) | C(53')-C(54') | 1.521(13) |
| C(6)-C(11) | 1.414(2) | C(32)-C(34) | 1.5171(19) | C(56)-C(58) | 1.530(2) |
| C(7)-C(8) | 1.4027(19) | C(35)-C(36) | 1.4006(19) | C(56)-C(57) | 1.534(2) |
| C(7)-C(12) | 1.514(2) | C(35)-C(40) | 1.4100(18) | | |

Tabelle 20: Bond angles [°] for jus_237m.

| | | | | | |
|---|---|---|---|---|---|
| C(59)-Ga(1)-N(1) | 106.34(5) | C(9)-C(8)-C(7) | 120.64(18) | C(35)-C(36)-C(37) | 118.03(12) |
| C(59)-Ga(1)-N(2) | 103.28(5) | C(10)-C(9)-C(8) | 120.57(15) | C(35)-C(36)-C(41) | 122.50(12) |
| N(1)-Ga(1)-N(2) | 93.37(4) | C(9)-C(10)-C(11) | 121.20(16) | C(37)-C(36)-C(41) | 119.43(12) |
| C(59)-Ga(1)-Si(1) | 128.06(4) | C(10)-C(11)-C(6) | 117.67(17) | C(38)-C(37)-C(36) | 121.21(14) |
| N(1)-Ga(1)-Si(1) | 111.15(3) | C(10)-C(11)-C(15) | 120.00(15) | C(37)-C(38)-C(39) | 120.02(13) |
| N(2)-Ga(1)-Si(1) | 108.90(3) | C(6)-C(11)-C(15) | 122.31(14) | C(38)-C(39)-C(40) | 121.01(14) |
| C(60)-Ga(2)-N(4) | 106.47(5) | C(7)-C(12)-C(14) | 112.09(13) | C(39)-C(40)-C(35) | 118.35(13) |
| C(60)-Ga(2)-N(3) | 105.09(5) | C(7)-C(12)-C(13) | 110.06(14) | C(39)-C(40)-C(44) | 121.22(13) |
| N(4)-Ga(2)-N(3) | 92.52(5) | C(14)-C(12)-C(13) | 111.11(15) | C(35)-C(40)-C(44) | 120.43(12) |
| C(60)-Ga(2)-Si(1) | 117.41(5) | C(11)-C(15)-C(17) | 113.02(16) | C(36)-C(41)-C(42) | 110.14(11) |
| N(4)-Ga(2)-Si(1) | 125.32(3) | C(11)-C(15)-C(16) | 111.62(16) | C(36)-C(41)-C(43) | 112.24(11) |
| N(3)-Ga(2)-Si(1) | 105.09(3) | C(17)-C(15)-C(16) | 109.40(16) | C(42)-C(41)-C(43) | 109.45(12) |
| C(61)-Si(1)-Ga(1) | 95.16(5) | C(19)-C(18)-C(23) | 120.64(12) | C(40)-C(44)-C(46) | 113.22(13) |
| C(61)-Si(1)-Ga(2) | 94.13(5) | C(19)-C(18)-N(2) | 120.28(12) | C(40)-C(44)-C(45) | 111.78(12) |
| Ga(1)-Si(1)-Ga(2) | 116.105(16) | C(23)-C(18)-N(2) | 119.07(12) | C(46)-C(44)-C(45) | 110.38(13) |
| C(1)-N(1)-C(6) | 118.84(11) | C(20)-C(19)-C(18) | 118.32(13) | C(48)-C(47)-C(52) | 120.93(13) |
| C(1)-N(1)-Ga(1) | 117.27(8) | C(20)-C(19)-C(24) | 118.53(13) | C(48)-C(47)-N(4) | 120.09(13) |
| C(6)-N(1)-Ga(1) | 123.50(8) | C(18)-C(19)-C(24) | 123.15(12) | C(52)-C(47)-N(4) | 118.86(13) |
| C(3)-N(2)-C(18) | 118.23(11) | C(21)-C(20)-C(19) | 121.35(15) | C(47)-C(48)-C(49) | 118.81(15) |
| C(3)-N(2)-Ga(1) | 116.96(9) | C(22)-C(21)-C(20) | 119.75(14) | C(47)-C(48)-C(53) | 122.04(14) |
| C(18)-N(2)-Ga(1) | 123.95(8) | C(21)-C(22)-C(23) | 121.33(14) | C(49)-C(48)-C(53) | 119.12(15) |
| C(30)-N(3)-C(35) | 119.50(11) | C(22)-C(23)-C(18) | 118.52(14) | C(47)-C(48)-C(53') | 126.6(5) |
| C(30)-N(3)-Ga(2) | 118.45(9) | C(22)-C(23)-C(27) | 119.76(13) | C(49)-C(48)-C(53') | 109.9(5) |
| C(35)-N(3)-Ga(2) | 121.76(8) | C(18)-C(23)-C(27) | 121.72(13) | C(50)-C(49)-C(48) | 120.95(16) |
| C(32)-N(4)-C(47) | 116.64(11) | C(19)-C(24)-C(26) | 110.80(13) | C(49)-C(50)-C(51) | 119.61(14) |
| C(32)-N(4)-Ga(2) | 115.92(9) | C(19)-C(24)-C(25) | 112.32(12) | C(50)-C(51)-C(52) | 121.90(16) |
| C(47)-N(4)-Ga(2) | 126.10(9) | C(26)-C(24)-C(25) | 109.00(13) | C(51)-C(52)-C(47) | 117.78(15) |
| N(1)-C(1)-C(2) | 123.21(12) | C(23)-C(27)-C(28) | 111.59(14) | C(51)-C(52)-C(56) | 119.53(14) |
| N(1)-C(1)-C(4) | 119.90(12) | C(23)-C(27)-C(29) | 111.37(14) | C(47)-C(52)-C(56) | 122.64(13) |
| C(2)-C(1)-C(4) | 116.88(12) | C(28)-C(27)-C(29) | 111.52(15) | C(48)-C(53)-C(54) | 108.91(16) |
| C(1)-C(2)-C(3) | 128.07(12) | N(3)-C(30)-C(31) | 123.04(12) | C(48)-C(53)-C(55) | 112.80(19) |
| N(2)-C(3)-C(2) | 124.15(12) | N(3)-C(30)-C(33) | 120.32(12) | C(54)-C(53)-C(55) | 110.5(2) |
| N(2)-C(3)-C(5) | 119.75(12) | C(31)-C(30)-C(33) | 116.63(12) | C(55')-C(53')-C(54') | 111.1(12) |
| C(2)-C(3)-C(5) | 116.09(11) | C(32)-C(31)-C(30) | 127.53(12) | C(55')-C(53')-C(48) | 104.2(12) |
| C(7)-C(6)-C(11) | 121.63(13) | N(4)-C(32)-C(31) | 124.31(12) | C(54')-C(53')-C(48) | 122.8(10) |
| C(7)-C(6)-N(1) | 120.32(13) | N(4)-C(32)-C(34) | 119.55(13) | C(52)-C(56)-C(58) | 113.32(14) |
| C(11)-C(6)-N(1) | 118.04(14) | C(31)-C(32)-C(34) | 116.13(13) | C(52)-C(56)-C(57) | 111.70(14) |
| C(6)-C(7)-C(8) | 118.29(15) | C(36)-C(35)-C(40) | 121.30(12) | C(58)-C(56)-C(57) | 108.33(14) |
| C(6)-C(7)-C(12) | 122.88(12) | C(36)-C(35)-N(3) | 120.79(11) | O(1)-C(61)-Si(1) | 170.82(19) |
| C(8)-C(7)-C(12) | 118.82(15) | C(40)-C(35)-N(3) | 117.89(12) | | |

## 6. PhC(N$^t$Bu)$_2$SiAl(Cl)DDP 6

### 6.1. Synthese PhC(N$^t$Bu)$_2$SiAl(Cl)DDP **6**




Dipp = 2,6-$^i$Pr$_2$C$_6$H$_3$ **6**

Es wurden 198.2 mg PhC(N$^t$Bu)$_2$SiCl (0.678 mmol) und 300 mg DDPAl (0.67 mmol) in 5 mL Toluol gelöst und für einen Tag bei Raumtemperatur gerührt. Danach wurde die Lösung auf ein Viertel eingeengt und bei –18 °C gelagert, wobei PhC(N$^t$Bu)$_2$SiAl(Cl)DDP **6** in Form von roten Kristallen erhalten werden konnte.

**Ausbeute:** 339 mg (0.46 mmol, 68 %)

**Smp.** 184 °C

**Elementaranalyse** von $C_{44}H_{64}ClAlN_4Si$: gefunden (berechnet) C 70.4 (71.46), H 8.31 (8.72), N 7.58 (7.58) %.

**IR**: ν 3041, 2951, 2856, 1549, 1514, 1457, 1433, 1384, 1355, 1315, 1245, 1201, 1172, 1098, 1015, 933, 864, 794, 757, 701, 645, 526, 478 cm$^{-1}$.

**$^1$H NMR** ($C_6D_6$, 400 MHz): δ 7.5-6.80 (m, 11 H, C$_6$*H*$_3$($^i$Pr)$_2$ & C$_6$*H*$_5$), 5.20 (s, 1 H, γ-C*H*), 3.86 (sept, $^3J_{HH}$ = 6.8 Hz, 2 H, -C*H*(CH$_3$)$_2$), 3.77 (sept, $^3J_{HH}$ = 6.8 Hz, 2 H, -C*H*(CH$_3$)$_2$), 1.71 (d, $^3J_{HH}$ = 6.9 Hz, 6 H, -CH(C*H*$_3$)$_2$), 1.63 (s, 6 H, ArNCC*H*$_3$), 1.58 (d, $^3J_{HH}$ = 6.9 Hz, 6 H, -CH(C*H*$_3$)$_2$), 1.24 (d, $^3J_{HH}$ = 6.9 Hz, 6 H, -CH(C*H*$_3$)$_2$), 1.15 (d, $^3J_{HH}$ = 6.9 Hz, 6 H, -CH(C*H*$_3$)$_2$), 0.87 (s, 18 H, C(C*H*$_3$)$_3$) ppm.

**$^{13}$C NMR** ($C_6D_6$, 101 MHz): δ 169.7 (ArN*C*CH$_3$), 146.3, 144.2, 143.1 (*C*$_6$H$_3$), 136.5, 133.1, 130.2, 129.2, 128.6, 127.2, 127.9 (*C*$_6$H$_5$), 125.7, 124.8 (*C*$_6$H$_3$), 100.1 (γ-*C*H-), 53.1 (*C*(CH$_3$)$_3$), 31.0 (C(*C*H$_3$)$_3$), 30.3, 28.4 (-*C*H(CH$_3$)$_2$), 27.5, 25.2, 25.0, 24.7 (C(*C*H$_3$)$_3$), 24.5 (ArNC*C*H$_3$) ppm.

**$^{29}$Si NMR** ($C_6D_6$, 79 MHz): δ 93.0 ppm

## 6.2. Spektren PhC(N$^t$Bu)$_2$SiAl(Cl)DDP **6**

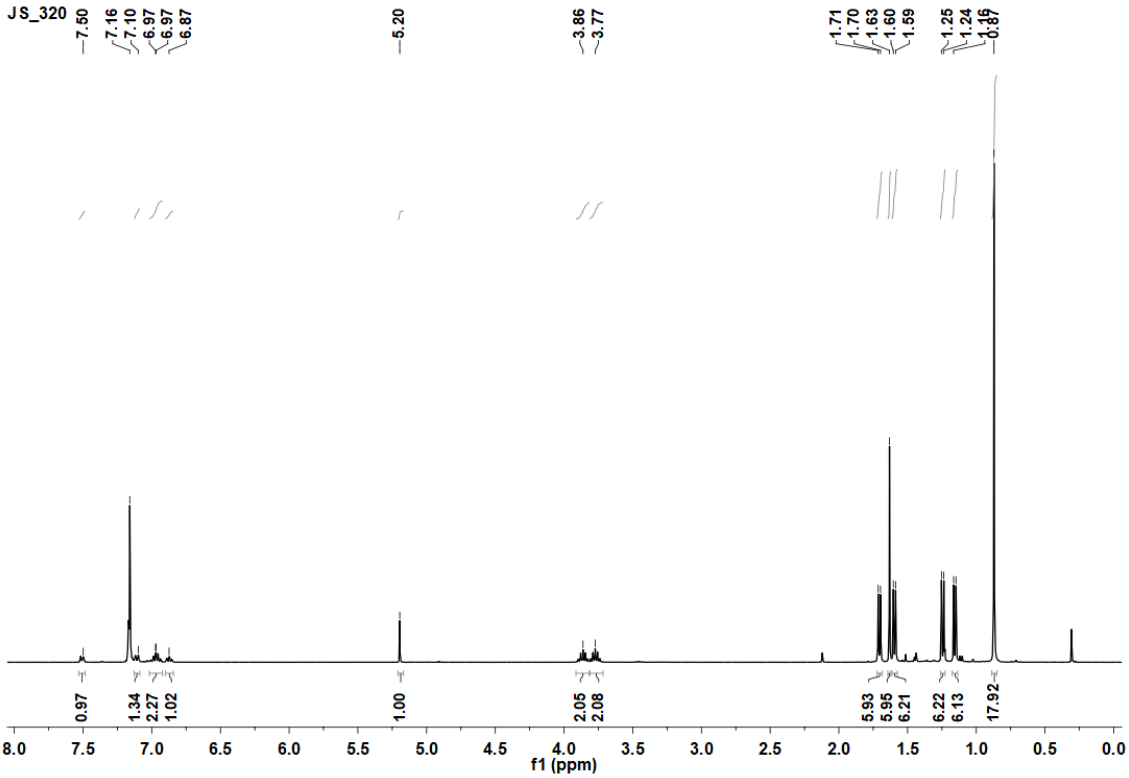



Abbildung 26: $^1$H-NMR-Spektrum von PhC(N$^t$Bu)$_2$SiAl(Cl)DDP **6** in $C_6D_6$.

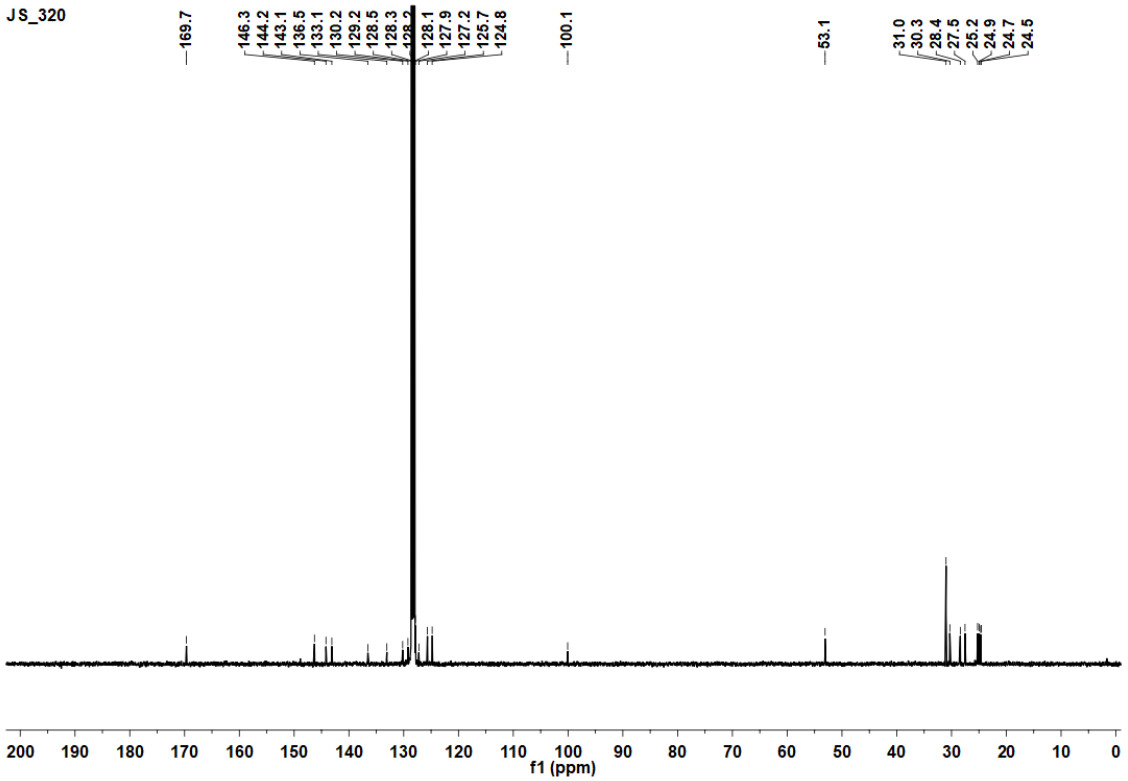



Abbildung 27: $^{13}$C-NMR-Spektrum von PhC(N$^t$Bu)$_2$SiAl(Cl)DDP **6** in $C_6D_6$.

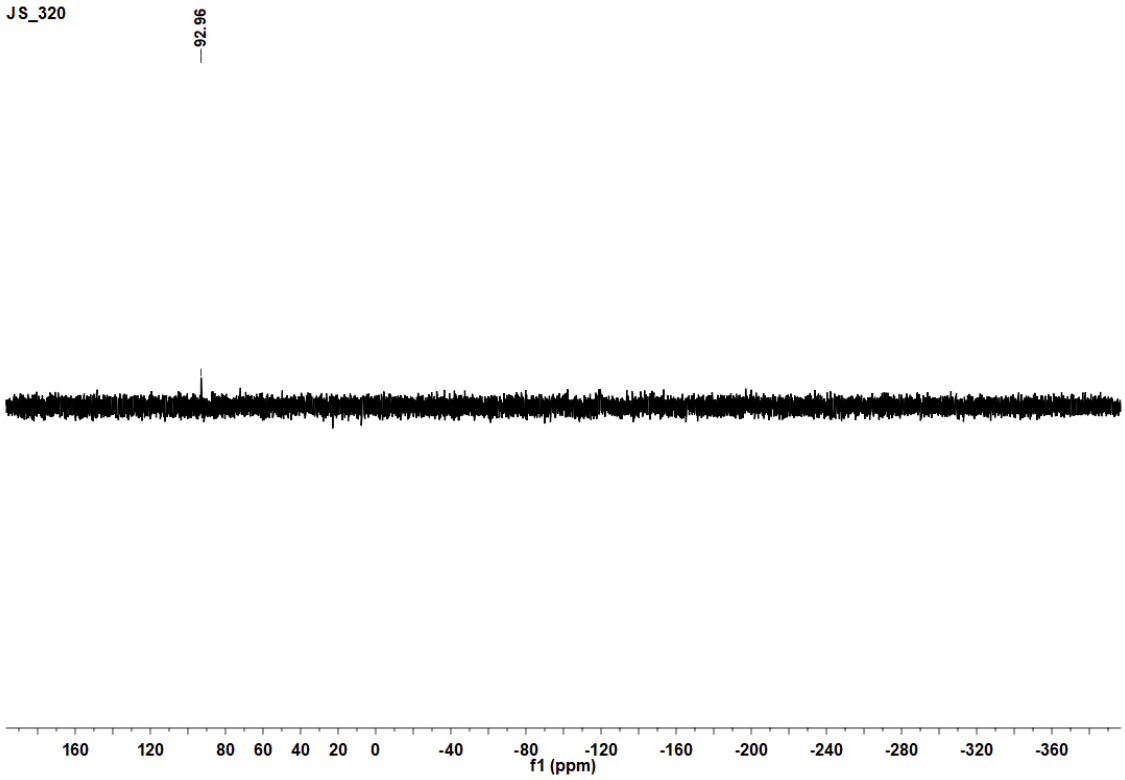



Abbildung 28: $^{29}$Si-NMR-Spektrum von PhC(N$^t$Bu)$_2$SiAl(Cl)DDP **6** in $C_6D_6$.

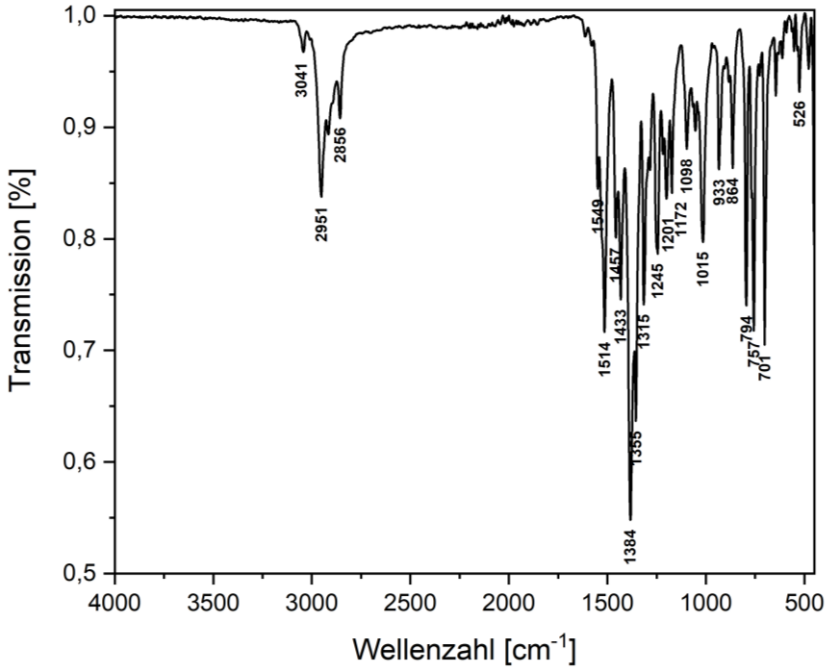



Abbildung 29: ATR-IR Spektrum von PhC(N$^t$Bu)$_2$SiAl(Cl)DDP **6**.

## 6.3. Kristallografische Daten PhC(N$^t$Bu)$_2$SiAl(Cl)DDP **6**

Tabelle 21: Crystal structure data

| **Identification code** | **jus_320m** |
|---|---|
| Empirical formula | C44 H64 Al Cl N4 Si |
| Formula weight | 739.51 |
| Density (calculated) | 1.145 g·cm$^{-3}$ |
| *F*(000) | 800 |
| Temperature | 100(2) K |
| Crystal size | 0.244 × 0.177 × 0.064 mm |
| Crystal colour | orange |
| Crystal description | tablet |
| Wavelength | 1.54178 Å |
| Crystal system | triclinic |
| Space group | *P -1* |
| Unit cell dimensions | |
| *a* [Å] | 10.3109(14) |
| *b* [Å] | 12.2797(17) |
| *c* [Å] | 18.139(3) |
| *α* [°] | 83.213(5) |
| *β* [°] | 75.928(4) |
| *γ* [°] | 74.666(4) |
| Volume | 2144.9(5) Å$^3$ |
| *Z* | 2 |
| Cell measurement reflections used | 9754 |
| Cell measurement *θ* min/max | 2.52°/78.95° |
| Diffractometer control software | Bruker APEX3(v2017.3-0) |
| Diffractometer measurement device | Bruker D8 Venture (Photon II detector) |
| Diffractometer measurement method | Data collection strategy APEX 3/Queen |
| *θ* range for data collection | 2.515°- 79.340° |
| Completeness to *θ* = 67.679° | 99.3% |
| Completeness to $\theta_{max}$ = 79.340° | 94.3% |
| Index ranges | -11 ≤ *h* ≤ 13 |
| | -15 ≤ *k* ≤ 13 |
| | -22 ≤ *l* ≤ 22 |
| Computing data reduction | Bruker APEX3(v2017.3-0) |
| Absorption coefficient | 1.503 mm$^{-1}$ |
| Absorption correction | Semi-empirical from equivalents |
| Computation absorption correction | SADABS |
| Max./min. Transmission | 0.75/0.58 |
| $R_{merg}$ before/after correction | 0.1350/0.0740 |
| Computing structure solution | Bruker APEX3(v2017.3-0) |
| Computing structure refinement | SHELXL-2017/1 (Sheldrick, 2017) |

| | |
|---|---|
| Refinement method | Full-matrix least-squares on $F^2$ |
| Reflections collected | 50962 |
| Independent reflections | 8778 |
| $R_{int}$ | 0.0661 |
| Reflections with $I > 2\sigma(I)$ | 7692 |
| Restraints | 0 |
| Parameter | 476 |
| GooF | 1.059 |
| Weighting details | $w = 1/[\sigma^2(F_{obs}^2) + (0.0878P)^2 + 0.4475P]$ |
| | where $P = (F_{obs}^2 + 2F_{calc}^2)/3$ |
| $R_1$ [$I > 2\sigma(I)$] | 0.0438 |
| $wR_2$ [$I > 2\sigma(I)$] | 0.1237 |
| $R_1$ [all data] | 0.0533 |
| $wR_2$ [all data] | 0.1332 |
| Absolute structure parameter | |
| Largest diff. peak and hole | 0.827/-0.371 |

Tabelle 22: Atomic coordinates ( × $10^4$) and equivalent isotropic displacement parameters (Å$^2$ × $10^3$) for jus_320m. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | *x* | *y* | *z* | $U_{eq}$ | | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|---|---|---|---|---|
| Cl(1) | 5985(1) | 8804(1) | 3345(1) | 26(1) | C(16) | 13039(2) | 6821(2) | 3959(1) | 31(1) |
| Si(1) | 9334(1) | 8815(1) | 1595(1) | 21(1) | H(16A) | 13676 | 6092 | 3816 | 46 |
| Al(1) | 8198(1) | 8771(1) | 3018(1) | 18(1) | H(16B) | 12484 | 6740 | 4475 | 46 |
| N(1) | 9056(1) | 7678(1) | 3710(1) | 20(1) | H(16C) | 13566 | 7384 | 3944 | 46 |
| N(2) | 8172(1) | 10070(1) | 3548(1) | 19(1) | C(17) | 12914(2) | 7512(2) | 2616(1) | 33(1) |
| N(3) | 7781(1) | 9080(1) | 1173(1) | 21(1) | H(17A) | 13583 | 6831 | 2408 | 49 |
| N(4) | 9168(1) | 7462(1) | 1269(1) | 22(1) | H(17B) | 13402 | 8075 | 2664 | 49 |
| C(1) | 8835(1) | 7888(1) | 4451(1) | 22(1) | H(17C) | 12287 | 7824 | 2273 | 49 |
| C(2) | 8316(2) | 8967(1) | 4721(1) | 24(1) | C(18) | 8062(1) | 11151(1) | 3126(1) | 20(1) |
| H(2) | 8060 | 9009 | 5259 | 28 | C(19) | 6787(2) | 11951(1) | 3181(1) | 22(1) |
| C(3) | 8130(1) | 10003(1) | 4291(1) | 22(1) | C(20) | 6745(2) | 12934(1) | 2702(1) | 26(1) |
| C(4) | 9191(2) | 6925(1) | 5021(1) | 28(1) | H(20) | 5896 | 13488 | 2730 | 32 |
| H(4A) | 8717 | 6342 | 4995 | 42 | C(21) | 7910(2) | 13118(1) | 2191(1) | 28(1) |
| H(4B) | 8900 | 7206 | 5534 | 42 | H(21) | 7851 | 13778 | 1858 | 33 |
| H(4C) | 10190 | 6602 | 4906 | 42 | C(22) | 9165(2) | 12337(1) | 2164(1) | 26(1) |
| C(5) | 7909(2) | 11038(2) | 4725(1) | 29(1) | H(22) | 9966 | 12479 | 1820 | 31 |
| H(5A) | 8005 | 11688 | 4365 | 43 | C(23) | 9273(2) | 11349(1) | 2632(1) | 22(1) |
| H(5B) | 8596 | 10901 | 5036 | 43 | C(24) | 5459(2) | 11802(1) | 3723(1) | 25(1) |
| H(5C) | 6982 | 11195 | 5055 | 43 | H(24) | 5673 | 11070 | 4028 | 30 |
| C(6) | 9865(1) | 6556(1) | 3498(1) | 21(1) | C(25) | 4855(2) | 12759(2) | 4280(1) | 33(1) |
| C(7) | 9190(2) | 5716(1) | 3457(1) | 23(1) | H(25A) | 4519 | 13470 | 4001 | 50 |
| C(8) | 9993(2) | 4647(1) | 3235(1) | 27(1) | H(25B) | 5574 | 12841 | 4523 | 50 |
| H(8) | 9554 | 4076 | 3189 | 32 | H(25C) | 4090 | 12575 | 4671 | 50 |
| C(9) | 11422(2) | 4411(1) | 3081(1) | 29(1) | C(26) | 4374(2) | 11752(2) | 3288(1) | 30(1) |
| H(9) | 11955 | 3683 | 2928 | 35 | H(26A) | 4174 | 12451 | 2970 | 45 |
| C(10) | 12070(2) | 5234(1) | 3149(1) | 27(1) | H(26B) | 3527 | 11666 | 3651 | 45 |
| H(10) | 13048 | 5055 | 3059 | 33 | H(26C) | 4727 | 11105 | 2965 | 45 |
| C(11) | 11315(2) | 6328(1) | 3350(1) | 23(1) | C(27) | 10662(2) | 10524(1) | 2638(1) | 24(1) |
| C(12) | 7627(2) | 5932(1) | 3655(1) | 24(1) | H(27) | 10514 | 9742 | 2713 | 29 |
| H(12) | 7246 | 6680 | 3889 | 29 | C(28) | 11209(2) | 10722(2) | 3314(1) | 34(1) |
| C(13) | 7129(2) | 5040(2) | 4235(1) | 35(1) | H(28A) | 12115 | 10203 | 3306 | 51 |
| H(13A) | 6119 | 5234 | 4368 | 52 | H(28B) | 10567 | 10586 | 3791 | 51 |
| H(13B) | 7480 | 5017 | 4694 | 52 | H(28C) | 11293 | 11504 | 3276 | 51 |
| H(13C) | 7470 | 4298 | 4016 | 52 | C(29) | 11734(2) | 10580(2) | 1904(1) | 33(1) |
| C(14) | 7076(2) | 6007(2) | 2936(1) | 29(1) | H(29A) | 11348 | 10520 | 1471 | 49 |
| H(14A) | 7430 | 6568 | 2567 | 44 | H(29B) | 12549 | 9954 | 1914 | 49 |
| H(14B) | 6066 | 6235 | 3066 | 44 | H(29C) | 11996 | 11300 | 1852 | 49 |
| H(14C) | 7380 | 5267 | 2714 | 44 | C(30) | 8075(2) | 8017(1) | 976(1) | 22(1) |
| C(15) | 12086(2) | 7210(1) | 3398(1) | 25(1) | C(31) | 7291(2) | 7508(1) | 571(1) | 24(1) |
| H(15) | 11387 | 7911 | 3587 | 30 | C(32) | 7523(2) | 7530(1) | -219(1) | 27(1) |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| H(32) | 8191 | 7895 | -525 | 32 | C(40) | 5276(2) | 9916(2) | 1324(1) | 32(1) |
| C(33) | 6783(2) | 7020(2) | -564(1) | 33(1) | H(40A) | 4593 | 10626 | 1257 | 49 |
| H(33) | 6957 | 7031 | -1103 | 39 | H(40B) | 5176 | 9691 | 1867 | 49 |
| C(34) | 5797(2) | 6497(2) | -124(1) | 37(1) | H(40C) | 5128 | 9324 | 1058 | 49 |
| H(34) | 5294 | 6150 | -359 | 44 | C(41) | 10253(2) | 6465(1) | 948(1) | 24(1) |
| C(35) | 5547(2) | 6482(2) | 661(1) | 38(1) | C(42) | 10888(2) | 6731(2) | 112(1) | 30(1) |
| H(35) | 4864 | 6129 | 964 | 45 | H(42A) | 11691 | 6115 | -70 | 46 |
| C(36) | 6292(2) | 6981(2) | 1010(1) | 32(1) | H(42B) | 11177 | 7440 | 65 | 46 |
| H(36) | 6118 | 6961 | 1550 | 38 | H(42C) | 10203 | 6807 | -195 | 46 |
| C(37) | 6721(2) | 10085(1) | 995(1) | 24(1) | C(43) | 9702(2) | 5409(2) | 1038(1) | 32(1) |
| C(38) | 6918(2) | 11065(2) | 1362(1) | 32(1) | H(43A) | 8993 | 5528 | 742 | 48 |
| H(38A) | 7849 | 11171 | 1148 | 48 | H(43B) | 9301 | 5259 | 1577 | 48 |
| H(38B) | 6796 | 10899 | 1912 | 48 | H(43C) | 10459 | 4762 | 853 | 48 |
| H(38C) | 6237 | 11758 | 1260 | 48 | C(44) | 11370(2) | 6270(2) | 1400(1) | 32(1) |
| C(39) | 6942(2) | 10345(2) | 134(1) | 32(1) | H(44A) | 12090 | 5592 | 1234 | 47 |
| H(39A) | 7902 | 10378 | -76 | 47 | H(44B) | 10964 | 6167 | 1944 | 47 |
| H(39B) | 6326 | 11075 | 28 | 47 | H(44C) | 11772 | 6925 | 1315 | 47 |
| H(39C) | 6740 | 9749 | -103 | 47 | | | | | |

Tabelle 23: Anisotropic displacement parameters ($Å^2 \times 10^3$) for jus_320m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Cl(1) | 22(1) | 19(1) | 39(1) | -2(1) | -9(1) | -6(1) | C(16) | 30(1) | 32(1) | 33(1) | -2(1) | -12(1) | -7(1) |
| Si(1) | 22(1) | 19(1) | 23(1) | -5(1) | -6(1) | -6(1) | C(17) | 34(1) | 36(1) | 32(1) | -1(1) | -8(1) | -16(1) |
| Al(1) | 20(1) | 14(1) | 23(1) | -3(1) | -6(1) | -4(1) | C(18) | 25(1) | 15(1) | 22(1) | -5(1) | -7(1) | -7(1) |
| N(1) | 20(1) | 16(1) | 23(1) | -2(1) | -5(1) | -4(1) | C(19) | 26(1) | 17(1) | 26(1) | -6(1) | -7(1) | -6(1) |
| N(2) | 19(1) | 15(1) | 24(1) | -4(1) | -5(1) | -5(1) | C(20) | 32(1) | 16(1) | 33(1) | -5(1) | -10(1) | -4(1) |
| N(3) | 23(1) | 19(1) | 24(1) | -4(1) | -7(1) | -6(1) | C(21) | 41(1) | 15(1) | 30(1) | -1(1) | -9(1) | -10(1) |
| N(4) | 24(1) | 19(1) | 24(1) | -6(1) | -6(1) | -5(1) | C(22) | 32(1) | 22(1) | 26(1) | -4(1) | -5(1) | -13(1) |
| C(1) | 21(1) | 22(1) | 23(1) | 0(1) | -5(1) | -6(1) | C(23) | 26(1) | 19(1) | 24(1) | -5(1) | -7(1) | -9(1) |
| C(2) | 27(1) | 24(1) | 21(1) | -3(1) | -5(1) | -6(1) | C(24) | 24(1) | 18(1) | 31(1) | -6(1) | -4(1) | -4(1) |
| C(3) | 22(1) | 21(1) | 25(1) | -6(1) | -6(1) | -6(1) | C(25) | 32(1) | 27(1) | 37(1) | -12(1) | -2(1) | -3(1) |
| C(4) | 31(1) | 26(1) | 25(1) | 1(1) | -6(1) | -3(1) | C(26) | 23(1) | 26(1) | 41(1) | -7(1) | -8(1) | -3(1) |
| C(5) | 39(1) | 24(1) | 26(1) | -6(1) | -11(1) | -9(1) | C(27) | 23(1) | 24(1) | 27(1) | -4(1) | -6(1) | -10(1) |
| C(6) | 23(1) | 18(1) | 22(1) | -1(1) | -6(1) | -2(1) | C(28) | 30(1) | 41(1) | 38(1) | -5(1) | -14(1) | -13(1) |
| C(7) | 26(1) | 19(1) | 24(1) | 1(1) | -6(1) | -5(1) | C(29) | 27(1) | 34(1) | 36(1) | -1(1) | -2(1) | -10(1) |
| C(8) | 31(1) | 17(1) | 33(1) | -1(1) | -7(1) | -6(1) | C(30) | 22(1) | 22(1) | 22(1) | -4(1) | -4(1) | -6(1) |
| C(9) | 31(1) | 18(1) | 34(1) | -2(1) | -8(1) | 2(1) | C(31) | 27(1) | 18(1) | 28(1) | -5(1) | -9(1) | -5(1) |
| C(10) | 22(1) | 25(1) | 31(1) | -1(1) | -7(1) | 1(1) | C(32) | 32(1) | 23(1) | 28(1) | -5(1) | -10(1) | -6(1) |
| C(11) | 23(1) | 21(1) | 24(1) | 0(1) | -7(1) | -4(1) | C(33) | 43(1) | 27(1) | 32(1) | -8(1) | -17(1) | -6(1) |
| C(12) | 25(1) | 16(1) | 31(1) | -2(1) | -4(1) | -6(1) | C(34) | 42(1) | 30(1) | 50(1) | -10(1) | -23(1) | -12(1) |
| C(13) | 35(1) | 25(1) | 42(1) | 3(1) | -3(1) | -11(1) | C(35) | 36(1) | 40(1) | 45(1) | -2(1) | -13(1) | -20(1) |
| C(14) | 28(1) | 25(1) | 37(1) | -8(1) | -8(1) | -7(1) | C(36) | 33(1) | 36(1) | 32(1) | -3(1) | -9(1) | -14(1) |
| C(15) | 21(1) | 23(1) | 30(1) | -3(1) | -7(1) | -4(1) | C(37) | 25(1) | 20(1) | 27(1) | -6(1) | -10(1) | -2(1) |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| C(38) | 37(1) | 22(1) | 39(1) | -10(1) | -17(1) | 0(1) | C(42) | 30(1) | 30(1) | 30(1) | -7(1) | -3(1) | -4(1) |
| C(39) | 41(1) | 24(1) | 29(1) | -3(1) | -12(1) | -2(1) | C(43) | 35(1) | 21(1) | 38(1) | -8(1) | -4(1) | -6(1) |
| C(40) | 25(1) | 32(1) | 40(1) | -4(1) | -10(1) | -3(1) | C(44) | 30(1) | 28(1) | 35(1) | -10(1) | -10(1) | 2(1) |
| C(41) | 25(1) | 18(1) | 27(1) | -7(1) | -5(1) | -2(1) | | | | | | | |

Tabelle 24: Bond lengths [Å] for jus_320m.

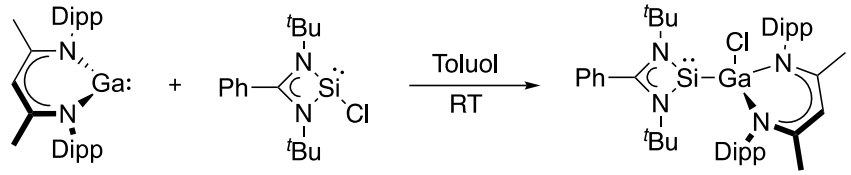
| | | | | | |
|---|---|---|---|---|---|
| Cl(1)-Al(1) | 2.2043(6) | C(6)-C(7) | 1.406(2) | C(23)-C(27) | 1.522(2) |
| Si(1)-N(3) | 1.8760(12) | C(6)-C(11) | 1.410(2) | C(24)-C(26) | 1.535(2) |
| Si(1)-N(4) | 1.8888(13) | C(7)-C(8) | 1.400(2) | C(24)-C(25) | 1.542(2) |
| Si(1)-C(30) | 2.3552(15) | C(7)-C(12) | 1.520(2) | C(27)-C(29) | 1.517(2) |
| Si(1)-Al(1) | 2.5620(6) | C(8)-C(9) | 1.388(2) | C(27)-C(28) | 1.537(2) |
| Al(1)-N(1) | 1.9176(13) | C(9)-C(10) | 1.382(2) | C(30)-C(31) | 1.4996(19) |
| Al(1)-N(2) | 1.9505(13) | C(10)-C(11) | 1.402(2) | C(31)-C(32) | 1.392(2) |
| N(1)-C(1) | 1.3500(18) | C(11)-C(15) | 1.524(2) | C(31)-C(36) | 1.396(2) |
| N(1)-C(6) | 1.4514(19) | C(12)-C(13) | 1.526(2) | C(32)-C(33) | 1.394(2) |
| N(2)-C(3) | 1.3296(19) | C(12)-C(14) | 1.529(2) | C(33)-C(34) | 1.383(3) |
| N(2)-C(18) | 1.4438(19) | C(15)-C(17) | 1.528(2) | C(34)-C(35) | 1.382(3) |
| N(3)-C(30) | 1.331(2) | C(15)-C(16) | 1.532(2) | C(35)-C(36) | 1.393(2) |
| N(3)-C(37) | 1.4788(19) | C(18)-C(19) | 1.407(2) | C(37)-C(38) | 1.523(2) |
| N(4)-C(30) | 1.3477(19) | C(18)-C(23) | 1.409(2) | C(37)-C(40) | 1.523(2) |
| N(4)-C(41) | 1.4892(19) | C(19)-C(20) | 1.399(2) | C(37)-C(39) | 1.530(2) |
| C(1)-C(2) | 1.392(2) | C(19)-C(24) | 1.520(2) | C(41)-C(44) | 1.524(2) |
| C(1)-C(4) | 1.506(2) | C(20)-C(21) | 1.381(2) | C(41)-C(43) | 1.525(2) |
| C(2)-C(3) | 1.405(2) | C(21)-C(22) | 1.386(2) | C(41)-C(42) | 1.533(2) |
| C(3)-C(5) | 1.510(2) | C(22)-C(23) | 1.392(2) | | |

Tabelle 25: Bond angles [°] for jus_320m.

| | | | | | |
|---|---|---|---|---|---|
| N(3)-Si(1)-N(4) | 69.10(6) | N(2)-C(3)-C(2) | 122.48(14) | C(20)-C(21)-C(22) | 119.91(15) |
| N(3)-Si(1)-C(30) | 34.36(6) | N(2)-C(3)-C(5) | 122.10(14) | C(21)-C(22)-C(23) | 121.20(14) |
| N(4)-Si(1)-C(30) | 34.88(5) | C(2)-C(3)-C(5) | 115.40(13) | C(22)-C(23)-C(18) | 118.10(14) |
| N(3)-Si(1)-Al(1) | 100.49(4) | C(7)-C(6)-C(11) | 121.49(14) | C(22)-C(23)-C(27) | 121.34(13) |
| N(4)-Si(1)-Al(1) | 103.85(4) | C(7)-C(6)-N(1) | 119.48(12) | C(18)-C(23)-C(27) | 120.52(14) |
| C(30)-Si(1)-Al(1) | 107.22(4) | C(11)-C(6)-N(1) | 119.02(13) | C(19)-C(24)-C(26) | 111.33(13) |
| N(1)-Al(1)-N(2) | 94.39(5) | C(8)-C(7)-C(6) | 118.43(14) | C(19)-C(24)-C(25) | 111.65(13) |
| N(1)-Al(1)-Cl(1) | 106.00(4) | C(8)-C(7)-C(12) | 119.48(14) | C(26)-C(24)-C(25) | 109.37(13) |
| N(2)-Al(1)-Cl(1) | 99.18(4) | C(6)-C(7)-C(12) | 122.09(13) | C(29)-C(27)-C(23) | 113.71(13) |
| N(1)-Al(1)-Si(1) | 120.08(4) | C(9)-C(8)-C(7) | 120.76(15) | C(29)-C(27)-C(28) | 109.88(13) |
| N(2)-Al(1)-Si(1) | 115.17(4) | C(10)-C(9)-C(8) | 120.09(15) | C(23)-C(27)-C(28) | 109.88(13) |
| Cl(1)-Al(1)-Si(1) | 117.81(2) | C(9)-C(10)-C(11) | 121.45(14) | N(3)-C(30)-N(4) | 105.69(12) |
| C(1)-N(1)-C(6) | 116.02(12) | C(10)-C(11)-C(6) | 117.72(14) | N(3)-C(30)-C(31) | 127.58(13) |
| C(1)-N(1)-Al(1) | 120.84(10) | C(10)-C(11)-C(15) | 119.17(13) | N(4)-C(30)-C(31) | 126.54(14) |
| C(6)-N(1)-Al(1) | 122.91(9) | C(6)-C(11)-C(15) | 123.11(14) | N(3)-C(30)-Si(1) | 52.70(7) |
| C(3)-N(2)-C(18) | 119.96(12) | C(7)-C(12)-C(13) | 112.17(13) | N(4)-C(30)-Si(1) | 53.28(7) |
| C(3)-N(2)-Al(1) | 122.01(11) | C(7)-C(12)-C(14) | 110.52(12) | C(31)-C(30)-Si(1) | 179.13(11) |
| C(18)-N(2)-Al(1) | 117.76(9) | C(13)-C(12)-C(14) | 110.92(13) | C(32)-C(31)-C(36) | 118.76(14) |
| C(30)-N(3)-C(37) | 130.61(12) | C(11)-C(15)-C(17) | 111.25(13) | C(32)-C(31)-C(30) | 123.10(14) |
| C(30)-N(3)-Si(1) | 92.94(9) | C(11)-C(15)-C(16) | 112.06(13) | C(36)-C(31)-C(30) | 118.14(13) |
| C(37)-N(3)-Si(1) | 135.83(10) | C(17)-C(15)-C(16) | 109.90(13) | C(31)-C(32)-C(33) | 120.52(16) |
| C(30)-N(4)-C(41) | 125.78(12) | C(19)-C(18)-C(23) | 121.55(14) | C(34)-C(33)-C(32) | 120.28(16) |
| C(30)-N(4)-Si(1) | 91.84(10) | C(19)-C(18)-N(2) | 121.25(13) | C(35)-C(34)-C(33) | 119.69(15) |
| C(41)-N(4)-Si(1) | 130.13(10) | C(23)-C(18)-N(2) | 117.14(13) | C(34)-C(35)-C(36) | 120.38(17) |
| N(1)-C(1)-C(2) | 123.22(14) | C(20)-C(19)-C(18) | 117.71(14) | C(35)-C(36)-C(31) | 120.37(16) |
| N(1)-C(1)-C(4) | 119.62(14) | C(20)-C(19)-C(24) | 118.42(14) | N(3)-C(37)-C(38) | 106.07(12) |
| C(2)-C(1)-C(4) | 117.15(13) | C(18)-C(19)-C(24) | 123.87(14) | N(3)-C(37)-C(40) | 111.00(13) |
| C(1)-C(2)-C(3) | 127.44(13) | C(21)-C(20)-C(19) | 121.43(15) | C(38)-C(37)-C(40) | 109.75(13) |

## 7. PhC(N$^t$Bu)$_2$SiGa(Cl)DDP 7

### 7.1. Synthese PhC(N$^t$Bu)$_2$SiGa(Cl)DDP **7**




Dipp = 2,6-$^i$Pr$_2$C$_6$H$_3$ **7**

Es wurden 300 mg PhC(N$^t$Bu)$_2$SiCl (1.01 mmol) und 491 mg DDPGa (1.01 mmol) in 5 mL Toluol gelöst und für einen Tag bei Raumtemperatur gerührt. Danach wurde die Lösung auf die Hälfte eingeengt und bei –18 °C gelagert, wobei PhC(N$^t$Bu)$_2$SiGa(Cl)DDP **7** in Form von gelben Kristallen erhalten werden konnte.

**Ausbeute**: 548 mg (0.70 mmol, 69 %).

**Smp.** 174 °C.

**Elementaranalyse** von $C_{44}H_{64}ClGaN_4Si$: gefunden (berechnet) C 67.8 (67.56), H 8.67 (8.25), N 7.35 (7.16) %.

**IR**: ν 3039 ,2951, 2913, 2850, 1553, 1516, 1457, 1433, 1385, 1355, 1315, 1255, 1199, 1172, 1096, 1016, 935, 854, 791, 756, 703, 612, 522, 475, 473 cm$^{-1}$.

**$^1$H NMR** ($C_6D_6$, 400 MHz): δ 7.6-6.80 (m, 11 H, $C_6H_3$($^i$Pr)$_2$ & $C_6H_5$), 5.14 (s, 1 H, γ-C*H*), 3.93 (sept, $^3J_{HH}$ = 6.8 Hz, 2 H, -C*H*(CH$_3$)$_2$), 3.85 (sept, $^3J_{HH}$ = 6.8 Hz, 2 H, -C*H*(CH$_3$)$_2$), 1.70 (s, 6 H, ArNCC*H$_3$*), 1.67 (d, $^3J_{HH}$ = 6.9 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.62 (d, $^3J_{HH}$ = 6.9 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.27 (d, $^3J_{HH}$ = 6.9 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.21 (d, $^3J_{HH}$ = 6.9 Hz, 6 H, -CH(C*H$_3$*)$_2$), 0.83 (s, 18 H, C(C*H$_3$*)$_3$) ppm.

**$^{13}$C NMR** ($C_6D_6$, 101 MHz): δ 167.5 (ArN*C*CH$_3$), 156.3 (*C*$_6$H$_5$), 146.3, 144.7 (N*C*C(CH(CH$_3$)$_2$)), 143.7 (NC*C*(CH(CH$_3$)$_2$)), 135.4, 128.4 (*C*$_6$H$_3$), 132.4, 129.7, 129.2, 128.2, 127.3 (*C*$_6$H$_5$), 127.4, 125.6, 124.5 (*C*$_6$H$_3$) 98.4 (γ-*C*H-), 53.4 (*C*(CH$_3$)$_3$), 30.8 (C(*C*H$_3$)$_3$), 30.1, 28.3 (-CH(*C*H$_3$)$_2$), 27.6, 25.3, 25.0 (-*C*H(CH$_3$)$_2$), 24.6 (ArNC*C*H$_3$), 24.4 (-*C*H(CH$_3$)$_2$) ppm.

**$^{29}$Si NMR** ($C_6D_6$, 79 MHz): δ 65.1 ppm

## 7.2. Spektren PhC(N$^t$Bu)$_2$SiGa(Cl)DDP **7**

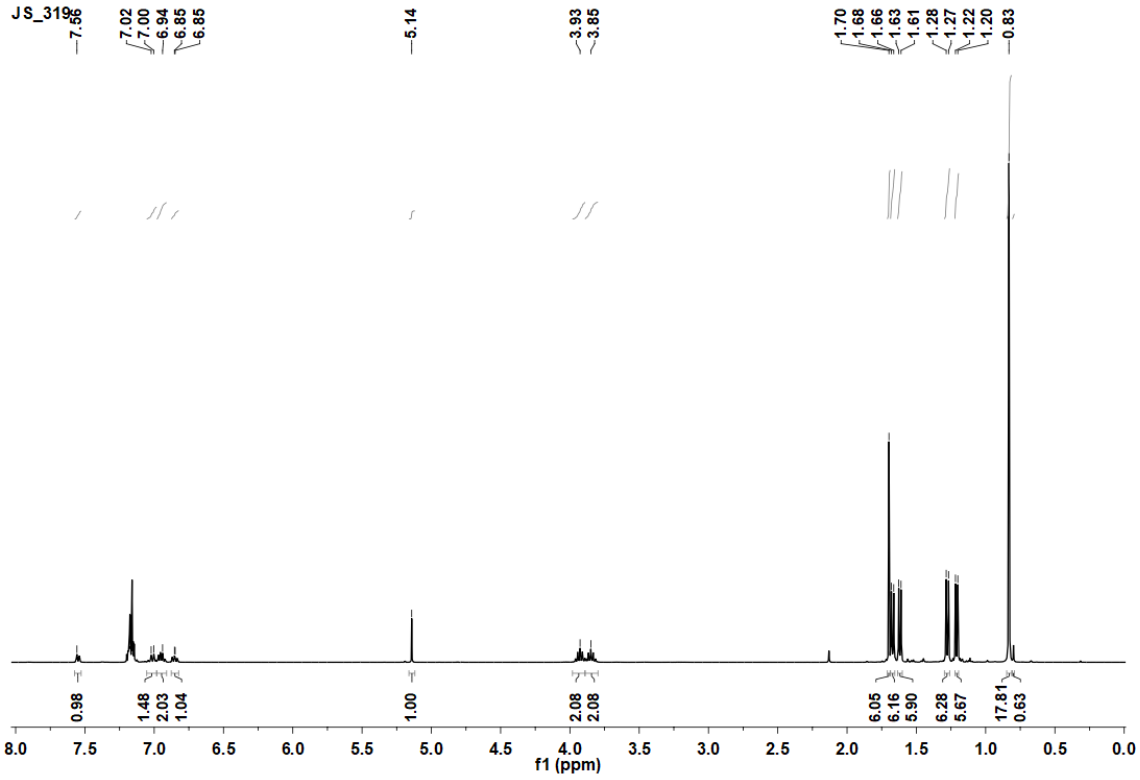



Abbildung 30: $^1$H-NMR-Spektrum von PhC(N$^t$Bu)$_2$SiGa(Cl)DDP **7** in $C_6D_6$.

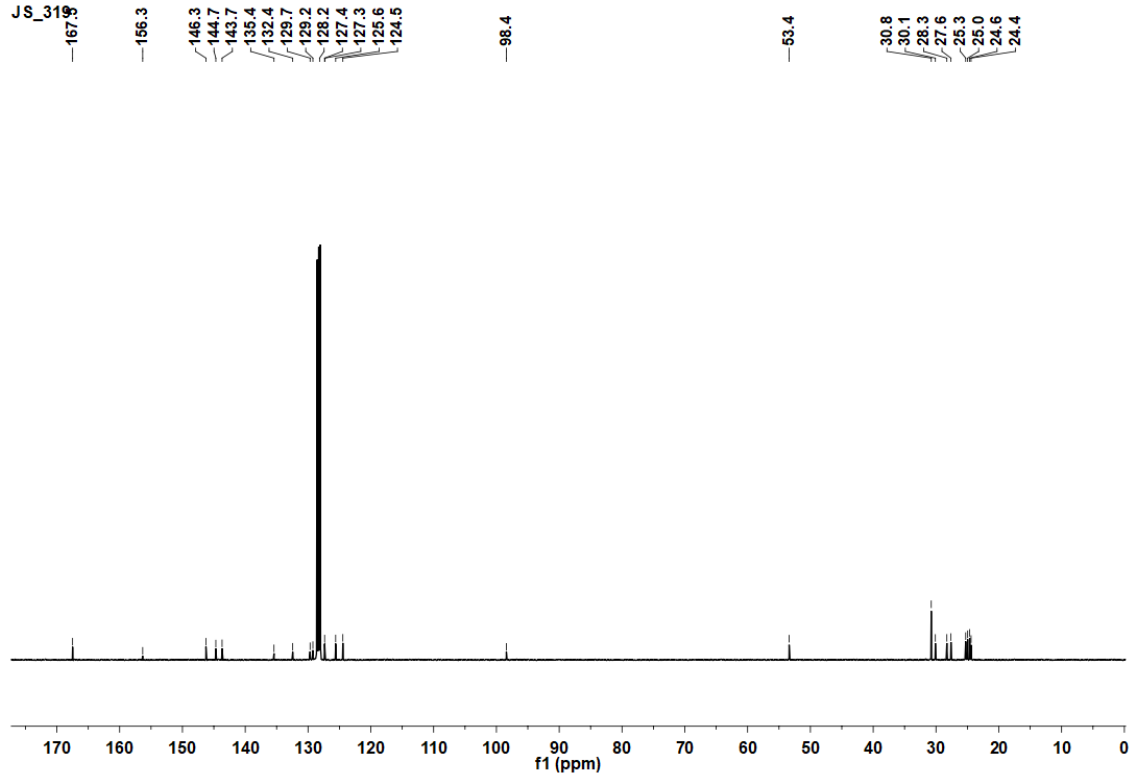



Abbildung 31: $^{13}$C-NMR-Spektrum von PhC(N$^t$Bu)$_2$SiGa(Cl)DDP **7** in $C_6D_6$.

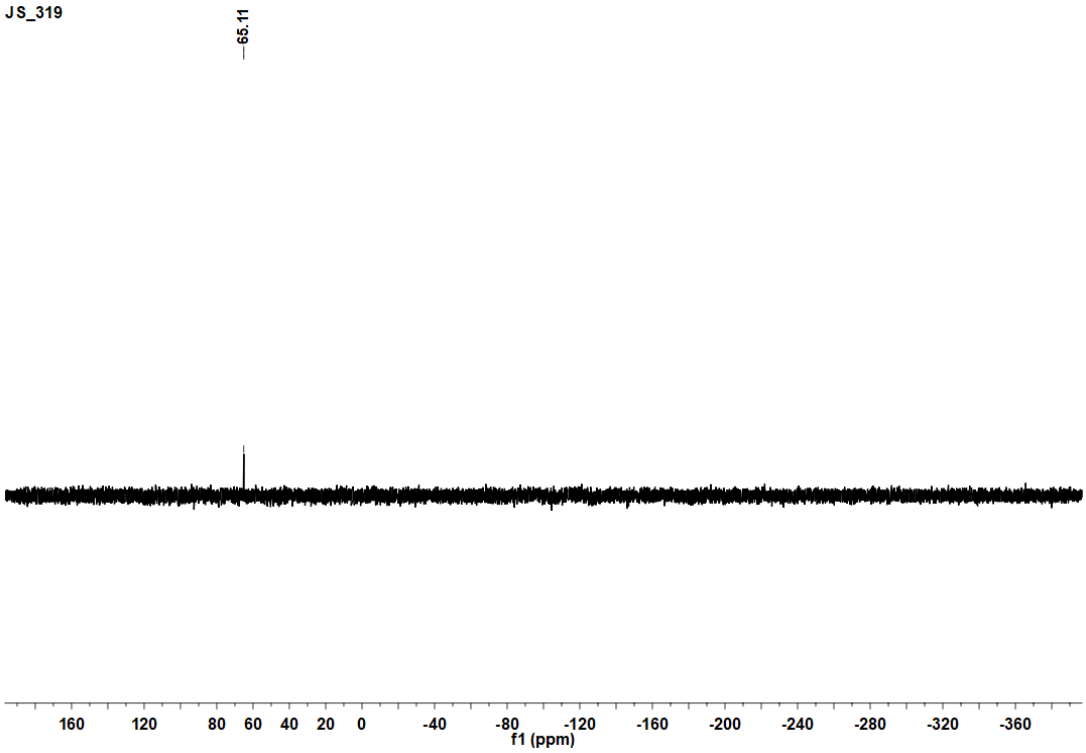



Abbildung 32: $^{29}$Si-NMR-Spektrum von PhC(N$^t$Bu)$_2$SiGa(Cl)DDP **7** in $C_6D_6$.

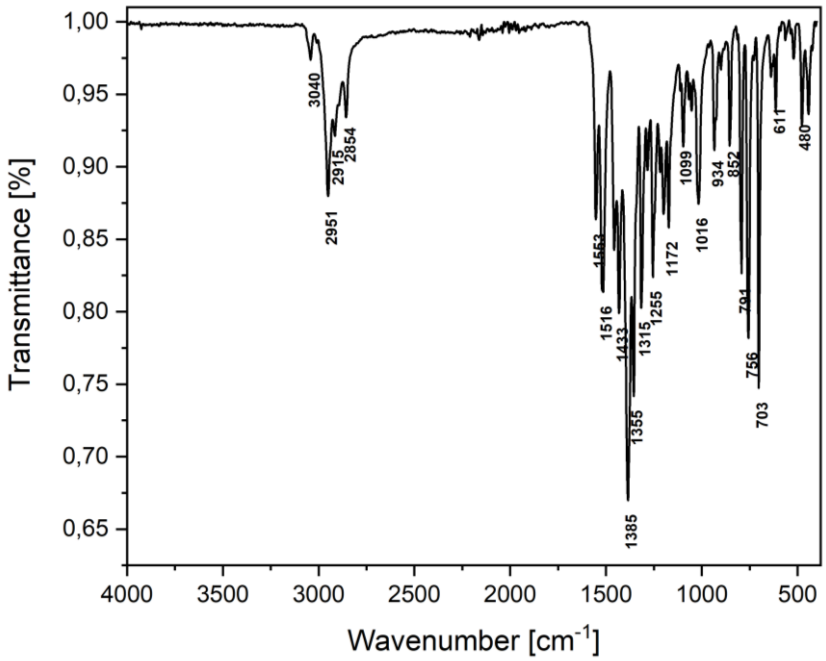



Abbildung 33: ATR-IR Spektrum von PhC(N$^t$Bu)$_2$SiGa(Cl)DDP **7**.

## 7.3. Kristallografische Daten PhC(N$^t$Bu)$_2$SiGa(Cl)DDP **7**

Tabelle 26: Crystal structure data

| **Identification code** | **jus_319am** |
|---|---|
| Empirical formula | C44 H64 Cl Ga N4 Si |
| Formula weight | 782.25 |
| Density (calculated) | 1.211 g·cm$^{-3}$ |
| *F*(000) | 836 |
| Temperature | 100(2) K |
| Crystal size | 0.276 × 0.153 × 0.060 mm |
| Crystal colour | yellow |
| Crystal description | tablet |
| Wavelength | 1.54178 Å |
| Crystal system | triclinic |
| Space group | *P -1* |
| Unit cell dimensions | |
| *a* [Å] | 10.3006(12) |
| *b* [Å] | 12.3066(14) |
| *c* [Å] | 18.091(2) |
| *α* [°] | 83.414(3) |
| *β* [°] | 76.176(3) |
| *γ* [°] | 74.690(3) |
| Volume | 2144.6(4) Å$^3$ |
| *Z* | 2 |
| Cell measurement reflections used | 9321 |
| Cell measurement *θ* min/max | 2.52°/80.52° |
| Diffractometer control software | Bruker APEX3(v2017.3-0) |
| Diffractometer measurement device | Bruker D8 Venture (Photon II detector) |
| Diffractometer measurement method | Data collection strategy APEX 3/Queen |
| *θ* range for data collection | 2.519°- 80.762° |
| Completeness to *θ* = 67.679° | 100.0% |
| Completeness to $\theta_{max}$ = 80.762° | 99.0% |
| Index ranges | -13 ≤ *h* ≤ 12 |
| | -15 ≤ *k* ≤ 15 |
| | -23 ≤ *l* ≤ 23 |
| Computing data reduction | Bruker APEX3(v2017.3-0) |
| Absorption coefficient | 1.967 mm$^{-1}$ |
| Absorption correction | Semi-empirical from equivalents |
| Computation absorption correction | SADABS |
| Max./min. Transmission | 0.75/0.62 |
| $R_{merg}$ before/after correction | 0.1284/0.0476 |
| Computing structure solution | Bruker APEX3(v2017.3-0) |
| Computing structure refinement | SHELXL-2017/1 (Sheldrick, 2017) |

| | |
|---|---|
| Refinement method | Full-matrix least-squares on $F^2$ |
| Reflections collected | 128800 |
| Independent reflections | 9329 |
| $R_{int}$ | 0.0299 |
| Reflections with $I > 2\sigma(I)$ | 9170 |
| Restraints | 0 |
| Parameter | 476 |
| GooF | 1.030 |
| Weighting details | $w = 1/[\sigma^2(F_{obs}^2) + (0.0270P)^2 + 1.1138P]$ |
| | where $P = (F_{obs}^2 + 2F_{calc}^2)/3$ |
| $R_1$ [$I > 2\sigma(I)$] | 0.0234 |
| $wR_2$ [$I > 2\sigma(I)$] | 0.0604 |
| $R_1$ [all data] | 0.0237 |
| $wR_2$ [all data] | 0.0606 |
| Absolute structure parameter | |
| Largest diff. peak and hole | 0.325/-0.370 |

Tabelle 27: Atomic coordinates ( × $10^4$) and equivalent isotropic displacement parameters (Å$^2$ × $10^3$) for jus_319am. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | *x* | *y* | *z* | $U_{eq}$ | | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|---|---|---|---|---|
| Ga(1) | 3261(1) | 3764(1) | 7960(1) | 12(1) | C(16) | 8075(1) | 1802(1) | 8962(1) | 25(1) |
| Cl(1) | 973(1) | 3791(1) | 8328(1) | 20(1) | H(16A) | 8721 | 1084 | 8807 | 37 |
| Si(1) | 4364(1) | 3833(1) | 6560(1) | 14(1) | H(16B) | 7531 | 1701 | 9478 | 37 |
| N(1) | 4089(1) | 2662(1) | 8709(1) | 14(1) | H(16C) | 8591 | 2369 | 8956 | 37 |
| N(2) | 3163(1) | 5092(1) | 8547(1) | 13(1) | C(17) | 7928(1) | 2514(1) | 7622(1) | 27(1) |
| N(3) | 2784(1) | 4084(1) | 6166(1) | 15(1) | H(17A) | 8581 | 1835 | 7402 | 40 |
| N(4) | 4185(1) | 2474(1) | 6256(1) | 15(1) | H(17B) | 8435 | 3060 | 7677 | 40 |
| C(1) | 3837(1) | 2890(1) | 9446(1) | 16(1) | H(17C) | 7292 | 2848 | 7285 | 40 |
| C(2) | 3297(1) | 3967(1) | 9716(1) | 17(1) | C(18) | 3054(1) | 6169(1) | 8128(1) | 14(1) |
| H(2) | 3023 | 4007 | 10254 | 21 | C(19) | 1777(1) | 6962(1) | 8180(1) | 16(1) |
| C(3) | 3105(1) | 5004(1) | 9290(1) | 15(1) | C(20) | 1736(1) | 7941(1) | 7699(1) | 20(1) |
| C(4) | 4196(1) | 1939(1) | 10026(1) | 21(1) | H(20) | 888 | 8494 | 7725 | 24 |
| H(4A) | 3753 | 1340 | 9989 | 32 | C(21) | 2903(1) | 8123(1) | 7185(1) | 22(1) |
| H(4B) | 3869 | 2224 | 10539 | 32 | H(21) | 2841 | 8778 | 6849 | 26 |
| H(4C) | 5198 | 1638 | 9926 | 32 | C(22) | 4159(1) | 7349(1) | 7162(1) | 19(1) |
| C(5) | 2866(1) | 6029(1) | 9739(1) | 22(1) | H(22) | 4961 | 7490 | 6820 | 23 |
| H(5A) | 2938 | 6688 | 9387 | 33 | C(23) | 4265(1) | 6365(1) | 7633(1) | 15(1) |
| H(5B) | 3562 | 5897 | 10048 | 33 | C(24) | 449(1) | 6807(1) | 8722(1) | 19(1) |
| H(5C) | 1945 | 6166 | 10074 | 33 | H(24) | 668 | 6077 | 9027 | 22 |
| C(6) | 4892(1) | 1547(1) | 8498(1) | 15(1) | C(25) | -627(1) | 6749(1) | 8281(1) | 24(1) |
| C(7) | 4212(1) | 711(1) | 8453(1) | 17(1) | H(25A) | -808 | 7436 | 7950 | 36 |
| C(8) | 5013(1) | -353(1) | 8226(1) | 21(1) | H(25B) | -1484 | 6684 | 8641 | 36 |
| H(8) | 4573 | -921 | 8176 | 25 | H(25C) | -278 | 6090 | 7968 | 36 |
| C(9) | 6438(1) | -591(1) | 8074(1) | 22(1) | C(26) | -160(1) | 7763(1) | 9280(1) | 26(1) |
| H(9) | 6969 | -1317 | 7919 | 26 | H(26A) | -502 | 8470 | 8999 | 39 |
| C(10) | 7093(1) | 230(1) | 8146(1) | 21(1) | H(26B) | 557 | 7849 | 9524 | 39 |
| H(10) | 8071 | 51 | 8053 | 25 | H(26C) | -923 | 7577 | 9670 | 39 |
| C(11) | 6340(1) | 1316(1) | 8353(1) | 17(1) | C(27) | 5649(1) | 5548(1) | 7648(1) | 18(1) |
| C(12) | 2650(1) | 934(1) | 8652(1) | 18(1) | H(27) | 5502 | 4768 | 7721 | 21 |
| H(12) | 2274 | 1685 | 8882 | 21 | C(28) | 6186(1) | 5744(1) | 8330(1) | 28(1) |
| C(13) | 2148(1) | 49(1) | 9238(1) | 28(1) | H(28A) | 7078 | 5210 | 8335 | 42 |
| H(13A) | 1138 | 253 | 9378 | 42 | H(28B) | 5525 | 5632 | 8804 | 42 |
| H(13B) | 2516 | 19 | 9694 | 42 | H(28C) | 6297 | 6517 | 8287 | 42 |
| H(13C) | 2469 | -692 | 9018 | 42 | C(29) | 6733(1) | 5600(1) | 6914(1) | 26(1) |
| C(14) | 2096(1) | 999(1) | 7930(1) | 22(1) | H(29A) | 6357 | 5528 | 6477 | 39 |
| H(14A) | 2448 | 1556 | 7559 | 33 | H(29B) | 7552 | 4981 | 6931 | 39 |
| H(14B) | 1086 | 1226 | 8059 | 33 | H(29C) | 6987 | 6323 | 6860 | 39 |
| H(14C) | 2400 | 258 | 7710 | 33 | C(30) | 3077(1) | 3021(1) | 5971(1) | 15(1) |
| C(15) | 7109(1) | 2198(1) | 8407(1) | 19(1) | C(31) | 2292(1) | 2507(1) | 5568(1) | 18(1) |
| H(15) | 6409 | 2894 | 8603 | 22 | C(32) | 2523(1) | 2525(1) | 4776(1) | 21(1) |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| H(32) | 3192 | 2885 | 4469 | 25 | C(40) | 1912(1) | 6055(1) | 6367(1) | 25(1) |
| C(33) | 1776(1) | 2015(1) | 4434(1) | 26(1) | H(40A) | 2842 | 6162 | 6157 | 37 |
| H(33) | 1941 | 2024 | 3894 | 31 | H(40B) | 1790 | 5885 | 6918 | 37 |
| C(34) | 793(1) | 1493(1) | 4879(1) | 30(1) | H(40C) | 1229 | 6746 | 6267 | 37 |
| H(34) | 289 | 1143 | 4644 | 36 | C(41) | 5265(1) | 1477(1) | 5939(1) | 17(1) |
| C(35) | 546(1) | 1484(1) | 5668(1) | 31(1) | C(42) | 5901(1) | 1726(1) | 5099(1) | 23(1) |
| H(35) | -135 | 1134 | 5973 | 37 | H(42A) | 6698 | 1106 | 4920 | 35 |
| C(36) | 1295(1) | 1986(1) | 6012(1) | 25(1) | H(42B) | 6198 | 2430 | 5047 | 35 |
| H(36) | 1127 | 1975 | 6553 | 30 | H(42C) | 5213 | 1802 | 4792 | 35 |
| C(37) | 1717(1) | 5080(1) | 5991(1) | 17(1) | C(43) | 4707(1) | 423(1) | 6035(1) | 25(1) |
| C(38) | 1936(1) | 5347(1) | 5128(1) | 25(1) | H(43A) | 3979 | 545 | 5749 | 37 |
| H(38A) | 2891 | 5393 | 4922 | 37 | H(43B) | 4327 | 272 | 6577 | 37 |
| H(38B) | 1308 | 6070 | 5025 | 37 | H(43C) | 5455 | -223 | 5843 | 37 |
| H(38C) | 1748 | 4748 | 4887 | 37 | C(44) | 6380(1) | 1286(1) | 6394(1) | 25(1) |
| C(39) | 277(1) | 4904(1) | 6321(1) | 26(1) | H(44A) | 7104 | 612 | 6227 | 37 |
| H(39A) | -412 | 5606 | 6250 | 38 | H(44B) | 5975 | 1183 | 6938 | 37 |
| H(39B) | 179 | 4689 | 6867 | 38 | H(44C) | 6778 | 1942 | 6311 | 37 |
| H(39C) | 136 | 4304 | 6059 | 38 | | | | | |

Tabelle 28: Anisotropic displacement parameters ($Å^2 \times 10^3$) for jus_319am. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ga(1) | 12(1) | 11(1) | 11(1) | -1(1) | -4(1) | -1(1) | C(16) | 23(1) | 29(1) | 23(1) | 0(1) | -9(1) | -5(1) |
| Cl(1) | 15(1) | 17(1) | 28(1) | 0(1) | -7(1) | -4(1) | C(17) | 28(1) | 33(1) | 22(1) | 3(1) | -6(1) | -14(1) |
| Si(1) | 13(1) | 16(1) | 13(1) | -2(1) | -3(1) | -3(1) | C(18) | 17(1) | 12(1) | 12(1) | -2(1) | -5(1) | -5(1) |
| N(1) | 13(1) | 13(1) | 13(1) | 1(1) | -4(1) | -1(1) | C(19) | 18(1) | 14(1) | 16(1) | -4(1) | -4(1) | -3(1) |
| N(2) | 12(1) | 13(1) | 12(1) | -1(1) | -3(1) | -2(1) | C(20) | 24(1) | 13(1) | 23(1) | -3(1) | -7(1) | -2(1) |
| N(3) | 14(1) | 16(1) | 14(1) | -2(1) | -5(1) | -2(1) | C(21) | 33(1) | 14(1) | 19(1) | 1(1) | -6(1) | -7(1) |
| N(4) | 15(1) | 15(1) | 14(1) | -4(1) | -3(1) | -1(1) | C(22) | 24(1) | 19(1) | 17(1) | -2(1) | -2(1) | -10(1) |
| C(1) | 13(1) | 19(1) | 14(1) | 2(1) | -3(1) | -3(1) | C(23) | 18(1) | 16(1) | 14(1) | -3(1) | -4(1) | -6(1) |
| C(2) | 19(1) | 21(1) | 11(1) | -1(1) | -3(1) | -3(1) | C(24) | 16(1) | 16(1) | 21(1) | -3(1) | -2(1) | -1(1) |
| C(3) | 14(1) | 18(1) | 14(1) | -3(1) | -2(1) | -3(1) | C(25) | 16(1) | 23(1) | 31(1) | -4(1) | -6(1) | -1(1) |
| C(4) | 24(1) | 22(1) | 15(1) | 3(1) | -4(1) | 0(1) | C(26) | 23(1) | 24(1) | 27(1) | -9(1) | 0(1) | 0(1) |
| C(5) | 31(1) | 20(1) | 16(1) | -4(1) | -7(1) | -5(1) | C(27) | 14(1) | 22(1) | 18(1) | -1(1) | -3(1) | -7(1) |
| C(6) | 17(1) | 13(1) | 12(1) | 2(1) | -4(1) | 1(1) | C(28) | 22(1) | 38(1) | 28(1) | -3(1) | -11(1) | -9(1) |
| C(7) | 18(1) | 15(1) | 15(1) | 2(1) | -4(1) | -2(1) | C(29) | 19(1) | 30(1) | 26(1) | 1(1) | 2(1) | -8(1) |
| C(8) | 24(1) | 14(1) | 22(1) | 1(1) | -6(1) | -2(1) | C(30) | 15(1) | 18(1) | 11(1) | -1(1) | -2(1) | -4(1) |
| C(9) | 24(1) | 14(1) | 24(1) | 0(1) | -6(1) | 4(1) | C(31) | 18(1) | 17(1) | 19(1) | -3(1) | -6(1) | -3(1) |
| C(10) | 16(1) | 20(1) | 21(1) | 1(1) | -5(1) | 3(1) | C(32) | 25(1) | 19(1) | 19(1) | -2(1) | -7(1) | -3(1) |
| C(11) | 16(1) | 17(1) | 15(1) | 2(1) | -5(1) | -1(1) | C(33) | 34(1) | 24(1) | 23(1) | -6(1) | -14(1) | -3(1) |
| C(12) | 18(1) | 14(1) | 20(1) | 0(1) | -2(1) | -4(1) | C(34) | 34(1) | 27(1) | 38(1) | -6(1) | -19(1) | -9(1) |
| C(13) | 26(1) | 23(1) | 31(1) | 5(1) | 0(1) | -9(1) | C(35) | 29(1) | 35(1) | 36(1) | -1(1) | -10(1) | -17(1) |
| C(14) | 19(1) | 23(1) | 25(1) | -5(1) | -5(1) | -4(1) | C(36) | 25(1) | 32(1) | 22(1) | -1(1) | -7(1) | -12(1) |
| C(15) | 14(1) | 20(1) | 20(1) | -1(1) | -4(1) | -2(1) | C(37) | 17(1) | 17(1) | 16(1) | -2(1) | -6(1) | 1(1) |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| C(38) | 33(1) | 21(1) | 18(1) | 0(1) | -9(1) | 1(1) | C(42) | 23(1) | 25(1) | 18(1) | -4(1) | 0(1) | -2(1) |
| C(39) | 16(1) | 29(1) | 30(1) | -1(1) | -6(1) | -1(1) | C(43) | 28(1) | 17(1) | 27(1) | -6(1) | -1(1) | -3(1) |
| C(40) | 28(1) | 19(1) | 28(1) | -7(1) | -14(1) | 2(1) | C(44) | 22(1) | 24(1) | 25(1) | -7(1) | -8(1) | 5(1) |
| C(41) | 17(1) | 16(1) | 16(1) | -4(1) | -2(1) | 0(1) | | | | | | | |

Tabelle 29: Bond lengths [Å] for jus_319am.

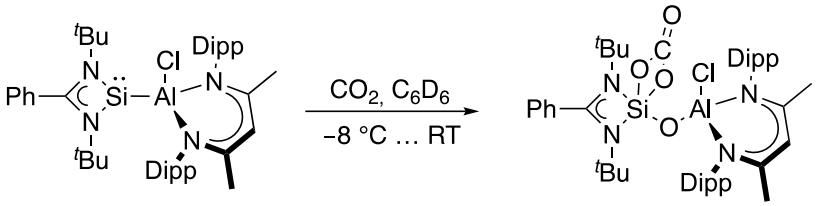
| | | | | | |
|---|---|---|---|---|---|
| Ga(1)-N(1) | 1.9810(9) | C(6)-C(11) | 1.4077(15) | C(23)-C(27) | 1.5167(15) |
| Ga(1)-N(2) | 2.0183(9) | C(6)-C(7) | 1.4086(16) | C(24)-C(25) | 1.5330(16) |
| Ga(1)-Cl(1) | 2.2837(4) | C(7)-C(8) | 1.3979(15) | C(24)-C(26) | 1.5416(16) |
| Ga(1)-Si(1) | 2.5170(4) | C(7)-C(12) | 1.5191(15) | C(27)-C(29) | 1.5250(15) |
| Si(1)-N(3) | 1.8686(9) | C(8)-C(9) | 1.3835(17) | C(27)-C(28) | 1.5333(16) |
| Si(1)-N(4) | 1.8859(10) | C(9)-C(10) | 1.3879(17) | C(30)-C(31) | 1.4972(15) |
| Si(1)-C(30) | 2.3467(11) | C(10)-C(11) | 1.3992(15) | C(31)-C(32) | 1.3935(16) |
| N(1)-C(1) | 1.3434(14) | C(11)-C(15) | 1.5260(16) | C(31)-C(36) | 1.3953(16) |
| N(1)-C(6) | 1.4433(13) | C(12)-C(14) | 1.5313(16) | C(32)-C(33) | 1.3932(17) |
| N(2)-C(3) | 1.3234(14) | C(12)-C(13) | 1.5331(15) | C(33)-C(34) | 1.385(2) |
| N(2)-C(18) | 1.4405(13) | C(15)-C(16) | 1.5312(16) | C(34)-C(35) | 1.388(2) |
| N(3)-C(30) | 1.3313(14) | C(15)-C(17) | 1.5338(16) | C(35)-C(36) | 1.3915(17) |
| N(3)-C(37) | 1.4748(13) | C(18)-C(19) | 1.4056(15) | C(37)-C(39) | 1.5234(16) |
| N(4)-C(30) | 1.3481(14) | C(18)-C(23) | 1.4113(15) | C(37)-C(40) | 1.5240(16) |
| N(4)-C(41) | 1.4856(13) | C(19)-C(20) | 1.3997(15) | C(37)-C(38) | 1.5326(15) |
| C(1)-C(2) | 1.3911(15) | C(19)-C(24) | 1.5246(15) | C(41)-C(44) | 1.5228(16) |
| C(1)-C(4) | 1.5104(14) | C(20)-C(21) | 1.3846(17) | C(41)-C(43) | 1.5287(16) |
| C(2)-C(3) | 1.4058(15) | C(21)-C(22) | 1.3841(17) | C(41)-C(42) | 1.5348(15) |
| C(3)-C(5) | 1.5121(15) | C(22)-C(23) | 1.3937(15) | | |

Tabelle 30: Bond angles [°] for jus_319am.

| | | | | | |
|---|---|---|---|---|---|
| N(1)-Ga(1)-N(2) | 92.65(4) | C(11)-C(6)-C(7) | 121.49(10) | C(19)-C(24)-C(26) | 111.53(9) |
| N(1)-Ga(1)-Cl(1) | 103.13(3) | C(11)-C(6)-N(1) | 119.08(10) | C(25)-C(24)-C(26) | 109.52(9) |
| N(2)-Ga(1)-Cl(1) | 96.27(3) | C(7)-C(6)-N(1) | 119.42(9) | C(23)-C(27)-C(29) | 113.65(10) |
| N(1)-Ga(1)-Si(1) | 122.91(3) | C(8)-C(7)-C(6) | 118.33(10) | C(23)-C(27)-C(28) | 110.23(9) |
| N(2)-Ga(1)-Si(1) | 116.85(3) | C(8)-C(7)-C(12) | 119.86(10) | C(29)-C(27)-C(28) | 110.02(10) |
| Cl(1)-Ga(1)-Si(1) | 119.136(11) | C(6)-C(7)-C(12) | 121.81(9) | N(3)-C(30)-N(4) | 105.78(9) |
| N(3)-Si(1)-N(4) | 69.38(4) | C(9)-C(8)-C(7) | 120.90(11) | N(3)-C(30)-C(31) | 127.69(10) |
| N(3)-Si(1)-C(30) | 34.51(4) | C(8)-C(9)-C(10) | 120.11(10) | N(4)-C(30)-C(31) | 126.40(10) |
| N(4)-Si(1)-C(30) | 35.05(4) | C(9)-C(10)-C(11) | 121.26(11) | N(3)-C(30)-Si(1) | 52.69(5) |
| N(3)-Si(1)-Ga(1) | 98.97(3) | C(10)-C(11)-C(6) | 117.85(10) | N(4)-C(30)-Si(1) | 53.45(5) |
| N(4)-Si(1)-Ga(1) | 101.91(3) | C(10)-C(11)-C(15) | 119.31(10) | C(31)-C(30)-Si(1) | 177.94(8) |
| C(30)-Si(1)-Ga(1) | 105.44(3) | C(6)-C(11)-C(15) | 122.84(10) | C(32)-C(31)-C(36) | 119.26(11) |
| C(1)-N(1)-C(6) | 117.28(9) | C(7)-C(12)-C(14) | 110.37(9) | C(32)-C(31)-C(30) | 122.82(10) |
| C(1)-N(1)-Ga(1) | 121.40(7) | C(7)-C(12)-C(13) | 112.09(9) | C(36)-C(31)-C(30) | 117.92(10) |
| C(6)-N(1)-Ga(1) | 121.12(7) | C(14)-C(12)-C(13) | 110.86(10) | C(33)-C(32)-C(31) | 120.19(11) |
| C(3)-N(2)-C(18) | 121.10(9) | C(11)-C(15)-C(16) | 111.94(9) | C(34)-C(33)-C(32) | 120.24(12) |
| C(3)-N(2)-Ga(1) | 122.25(7) | C(11)-C(15)-C(17) | 111.36(10) | C(33)-C(34)-C(35) | 119.91(12) |
| C(18)-N(2)-Ga(1) | 116.50(6) | C(16)-C(15)-C(17) | 109.73(10) | C(34)-C(35)-C(36) | 120.07(12) |
| C(30)-N(3)-C(37) | 130.64(9) | C(19)-C(18)-C(23) | 121.60(10) | C(35)-C(36)-C(31) | 120.32(12) |
| C(30)-N(3)-Si(1) | 92.80(7) | C(19)-C(18)-N(2) | 121.13(9) | N(3)-C(37)-C(39) | 111.02(9) |
| C(37)-N(3)-Si(1) | 135.78(7) | C(23)-C(18)-N(2) | 117.18(9) | N(3)-C(37)-C(40) | 105.77(9) |
| C(30)-N(4)-C(41) | 126.28(9) | C(20)-C(19)-C(18) | 117.50(10) | C(39)-C(37)-C(40) | 109.66(10) |
| C(30)-N(4)-Si(1) | 91.50(7) | C(20)-C(19)-C(24) | 118.89(10) | N(3)-C(37)-C(38) | 110.39(9) |
| C(41)-N(4)-Si(1) | 129.90(7) | C(18)-C(19)-C(24) | 123.61(10) | C(39)-C(37)-C(38) | 110.88(10) |
| N(1)-C(1)-C(2) | 124.02(10) | C(21)-C(20)-C(19) | 121.60(11) | C(40)-C(37)-C(38) | 108.98(10) |
| N(1)-C(1)-C(4) | 119.19(10) | C(22)-C(21)-C(20) | 119.94(10) | N(4)-C(41)-C(44) | 105.70(9) |
| C(2)-C(1)-C(4) | 116.76(10) | C(21)-C(22)-C(23) | 120.95(11) | N(4)-C(41)-C(43) | 111.74(9) |
| C(1)-C(2)-C(3) | 127.96(10) | C(22)-C(23)-C(18) | 118.30(10) | C(44)-C(41)-C(43) | 109.12(10) |
| N(2)-C(3)-C(2) | 123.34(10) | C(22)-C(23)-C(27) | 121.37(10) | N(4)-C(41)-C(42) | 110.60(9) |
| N(2)-C(3)-C(5) | 121.68(10) | C(18)-C(23)-C(27) | 120.26(9) | C(44)-C(41)-C(42) | 108.42(10) |
| C(2)-C(3)-C(5) | 114.95(9) | C(19)-C(24)-C(25) | 111.04(9) | C(43)-C(41)-C(42) | 111.05(9) |

## 8. PhC(N$^t$Bu)$_2$Si(O$_2$CO)OAl(Cl)DDP 8

### 8.1. Synthese PhC(N$^t$Bu)$_2$Si(O$_2$CO)OAl(Cl)DDP **8**




**6** **8**

Dipp = 2,6-$^i$Pr$_2$C$_6$H$_3$

Es wurden 25 mg PhC(N$^t$Bu)$_2$SiAl(Cl)DDP **6** (0.033 mmol) in 2 mL Toluol gelöst. Die Lösung wurde gefroren und die Atmosphäre im Vakuum entfernt. Danach wurde das Kältebad entfernt und die Atmosphäre mit Kohlenstoffdioxid versetzt. Dabei entfärbte sich die zunächst rote Lösung unter Gasentwicklung. Die Lösung wurde auf etwa ein Viertel eingeengt und bei 4 °C gelagert, wobei PhC(N$^t$Bu)$_2$Si(O$_2$CO)OAl(Cl)DDP **8** in Form von farblosen Kristallen erhalten werden konnte.

**Ausbeute** 11.4 mg (0.014 mmol, 42 %).

**Smp.** 215 °C.

**Elementaranalyse** von $C_{45}H_{64}ClAlN_4O_4Si$: gefunden (berechnet) C 64.3 (66.27), H 8.67 (7.91), N 6.87 (6.87)%.

**IR**: ν 2956, 2920, 2857, 1779, 1611, 1524, 1458, 1360, 1311, 1248, 1212, 1175, 1092, 1056, 1018, 1000, 935, 887, 844, 813, 797, 771, 757, 708, 664, 641, 534, 479, 451 cm$^{-1}$.

**$^1$H NMR** ($C_6D_6$, 400 MHz): δ 7.6-6.80 (m, 11 H, $C_6H_3$($^i$Pr)$_2$ & $C_6H_5$), 4.92 (s, 1 H, γ-C*H*), 3.56 (sept, $^3J_{HH}$ = 6.8 Hz, 2 H, -C*H*(CH$_3$)$_2$), 3.50 (sept, $^3J_{HH}$ = 6.8 Hz, 2 H, -C*H*(CH$_3$)$_2$), 1.82 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.53 (d, $^3J_{HH}$ = 6.6 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.49 (s, 6 H, ArNCC*H$_3$*), 1.19 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.02 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H$_3$*)$_2$), 0.68 (s, 18 H, C(C*H$_3$*)$_3$) ppm.

**$^{13}$C NMR** ($C_6D_6$, 101 MHz): δ 176.4 (NCN), 172.5 (ArN*C*CH$_3$), 154.3 (*C*O$_3$), 145.2, 144.8, 140.0 (*C*$_6$H$_3$), 132.1, 130.1, 129.6, 127.5, 125.4, 125.4, 125.0, 98.9(γ-*C*H-), 53.9 (*C*(CH$_3$)$_3$), 30.8 (C(*C*H$_3$)$_3$), 30.1, 28.2 (-*C*H(CH$_3$)$_2$), 27.2, 25.2, 25.0 (CH(*C*H$_3$)$_2$), 24.1(ArNC*C*H$_3$), 23.6 (CH(*C*H$_3$)$_2$) ppm.

**$^{29}$Si NMR** ($C_6D_6$, 119 MHz): δ Es konnte bislang kein $^{29}$Si-NMR-Signal für **8** erhalten werden.

## 8.2. Spektren PhC(N$^t$Bu)$_2$Si(O$_2$CO)OAl(Cl)DDP **8**

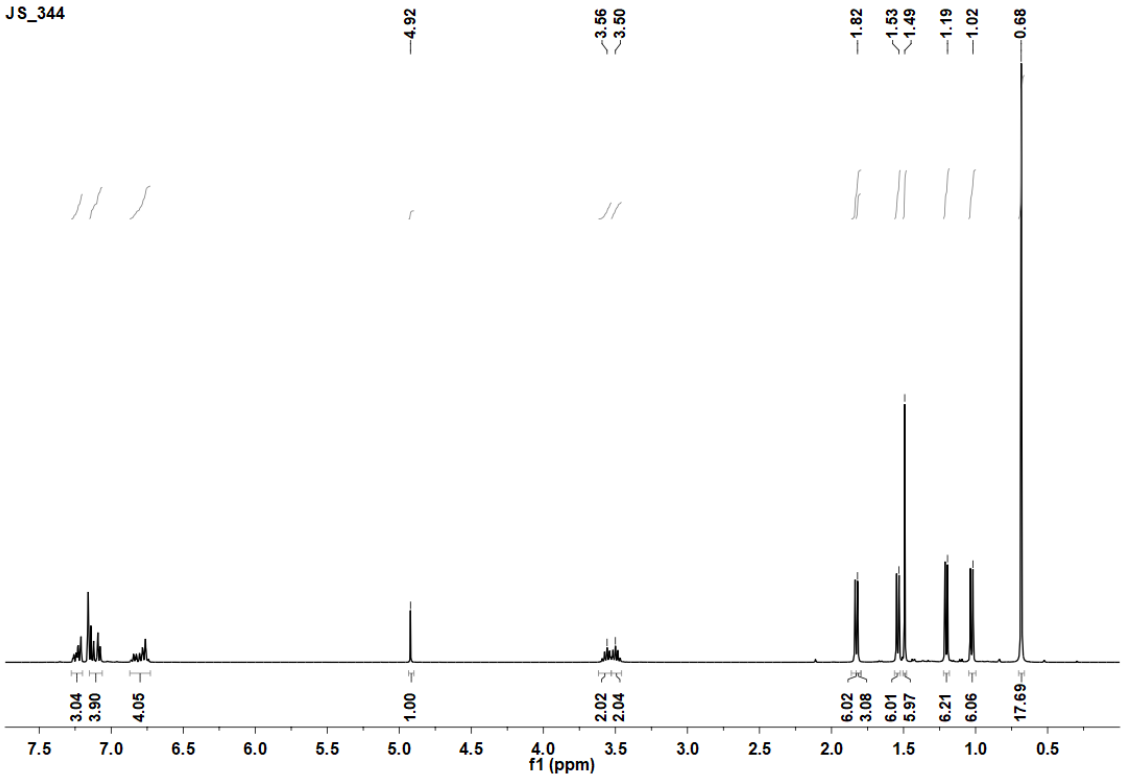



Abbildung 34: $^1$H-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(O$_2$CO)OAl(Cl)DDP **8** in $C_6D_6$.

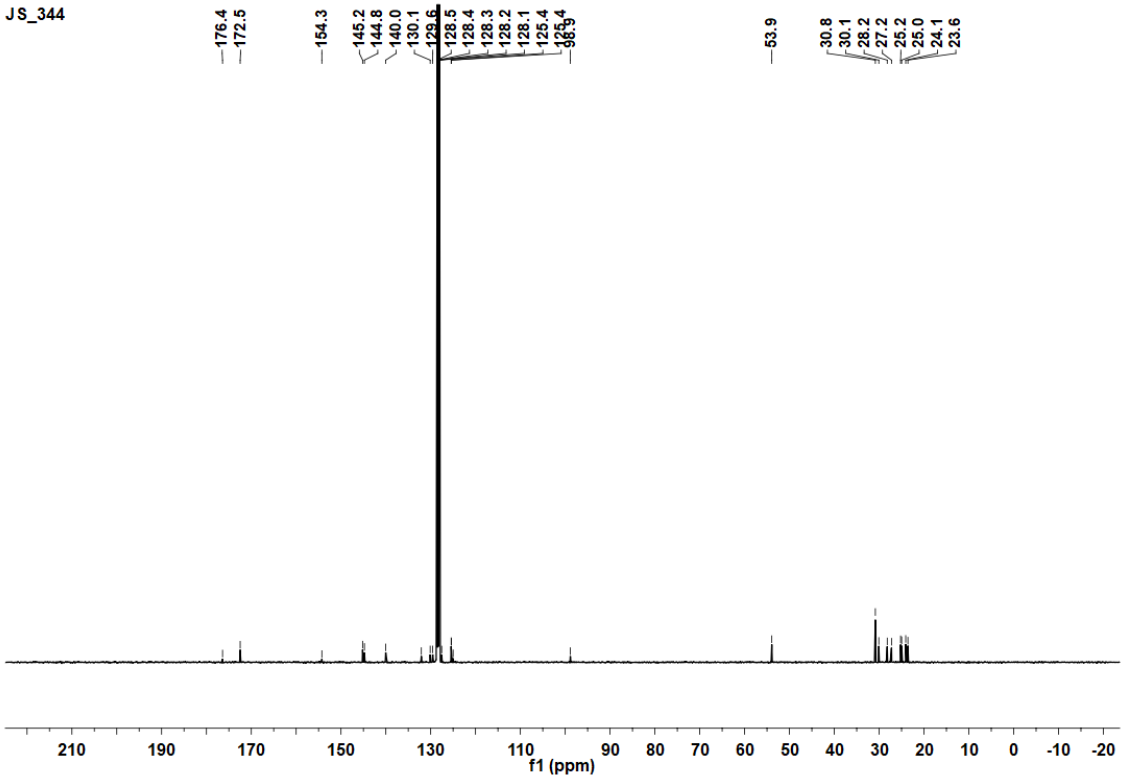



Abbildung 35: $^{13}$C-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(O$_2$CO)OAl(Cl)DDP **8** in $C_6D_6$.

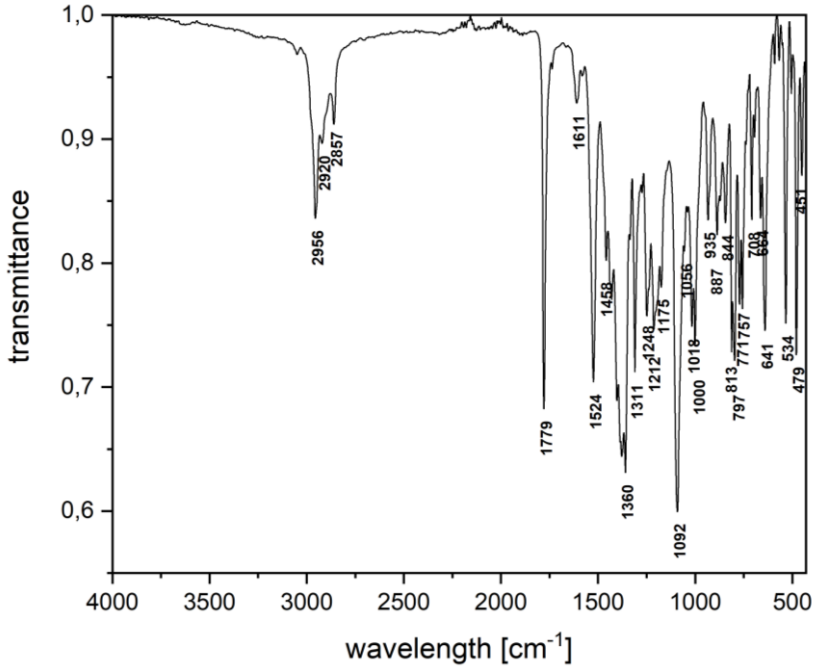



Abbildung 36: ATR-IR Spektrum von PhC(N$^t$Bu)$_2$Si(O$_2$CO)OAl(Cl)DDP **8**.

## 8.3. Kristallografische Daten PhC(N$^t$Bu)$_2$Si(O$_2$CO)OAl(Cl)DDP **8**

Tabelle 31: Crystal structure

| **Identification code** | **jus_378m** |
|---|---|
| Empirical formula | C45 H64 Al Cl N4 O4 Si |
| Formula weight | 815.52 |
| Density (calculated) | 1.209 g·cm$^{-3}$ |
| *F*(000) | 1752 |
| Temperature | 100(2) K |
| Crystal size | 0.199 × 0.180 × 0.114 mm |
| Crystal colour | colourless |
| Crystal description | tablet |
| Wavelength | 0.71073 Å |
| Crystal system | monoclinic |
| Space group | *P 21/c* |
| Unit cell dimensions | |
| *a* [Å] | 10.4793(15) |
| *b* [Å] | 19.161(3) |
| *c* [Å] | 22.356(3) |
| *α* [°] | 90 |
| *β* [°] | 93.896(3) |
| *γ* [°] | 90 |
| Volume | 4478.7(11) Å$^3$ |
| *Z* | 4 |
| Cell measurement reflections used | 9701 |
| Cell measurement *θ* min/max | 2.31°/32.78° |
| Diffractometer control software | BRUKER APEX3(v2019.1-0) |
| Diffractometer measurement device | Bruker D8 KAPPA II (APEX II detector) |
| Diffractometer measurement method | Data collection strategy APEX 3/QUEEN |
| *θ* range for data collection | 2.113°- 33.310° |
| Completeness to *θ* = 25.242° | 100.0% |
| Completeness to $\theta_{max}$ = 33.310° | 99.8% |
| Index ranges | -16 ≤ *h* ≤ 16 |
| | -29 ≤ *k* ≤ 29 |
| | -34 ≤ *l* ≤ 34 |
| Computing data reduction | BRUKER APEX3(v2019.1-0) |
| Absorption coefficient | 0.177 mm$^{-1}$ |
| Absorption correction | Semi-empirical from equivalents |
| Computation absorption correction | SADABS |
| Max./min. Transmission | 0.75/0.67 |

| | |
|---|---|
| $R_{merg}$ before/after correction | 0.0587/0.0533 |
| Computing structure solution | BRUKER APEX3(v2019.1-0) |
| Computing structure refinement | SHELXL-2017/1 (Sheldrick, 2017) |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Reflections collected | 180752 |
| Independent reflections | 17269 |
| $R_{int}$ | 0.0577 |
| Reflections with $I > 2\sigma(I)$ | 13184 |
| Restraints | 0 |
| Parameter | 521 |
| GooF | 1.054 |
| Weighting details | $w = 1/[\sigma^2(F_{obs}^2) + (0.0555P)^2 + 1.2332P]$ |
| | where $P = (F_{obs}^2 + 2F_{calc}^2)/3$ |
| $R_1$ [$I > 2\sigma(I)$] | 0.0407 |
| $wR_2$ [$I > 2\sigma(I)$] | 0.1016 |
| $R_1$ [all data] | 0.0612 |
| $wR_2$ [all data] | 0.1127 |
| Absolute structure parameter | |
| Largest diff. peak and hole | 0.587/-0.315 |

Tabelle 32: Atomic coordinates ( × $10^4$) and equivalent isotropic displacement parameters (Å$^2$ × $10^3$) for jus_378m. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | *x* | *y* | *z* | $U_{eq}$ | | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|---|---|---|---|---|
| Cl(1) | 1196(1) | 7533(1) | 4042(1) | 25(1) | H(14B) | -126 | 5850 | 4308 | 33 |
| Si(1) | 3052(1) | 7394(1) | 2383(1) | 12(1) | H(14C) | 352 | 5271 | 3859 | 33 |
| Al(1) | 3164(1) | 7472(1) | 3843(1) | 11(1) | C(15) | 6008(1) | 6363(1) | 3479(1) | 15(1) |
| O(1) | 3288(1) | 7493(1) | 3089(1) | 15(1) | H(15) | 5609 | 6828 | 3385 | 18 |
| O(2) | 4152(1) | 6699(1) | 2256(1) | 17(1) | C(16) | 6911(1) | 6451(1) | 4044(1) | 22(1) |
| O(3) | 4154(1) | 7743(1) | 1927(1) | 18(1) | H(16A) | 7253 | 5995 | 4170 | 34 |
| O(4) | 5709(1) | 6997(1) | 1638(1) | 27(1) | H(16B) | 6439 | 6653 | 4365 | 34 |
| N(1) | 3914(1) | 6688(1) | 4230(1) | 11(1) | H(16C) | 7617 | 6762 | 3956 | 34 |
| N(2) | 4078(1) | 8173(1) | 4276(1) | 12(1) | C(17) | 6819(1) | 6170(1) | 2959(1) | 21(1) |
| N(3) | 1810(1) | 8077(1) | 2144(1) | 15(1) | H(17A) | 6258 | 6086 | 2598 | 31 |
| N(4) | 1655(1) | 6970(1) | 2036(1) | 14(1) | H(17B) | 7310 | 5746 | 3061 | 31 |
| C(1) | 4327(1) | 6741(1) | 4811(1) | 12(1) | H(17C) | 7407 | 6553 | 2886 | 31 |
| C(2) | 4482(1) | 7383(1) | 5109(1) | 14(1) | C(18) | 4315(1) | 8856(1) | 4024(1) | 13(1) |
| H(2) | 4600 | 7364 | 5534 | 17 | C(19) | 3459(1) | 9411(1) | 4099(1) | 15(1) |
| C(3) | 4481(1) | 8051(1) | 4850(1) | 13(1) | C(20) | 3722(1) | 10053(1) | 3833(1) | 18(1) |
| C(4) | 4718(1) | 6100(1) | 5169(1) | 18(1) | H(20) | 3152 | 10433 | 3875 | 21 |
| H(4A) | 5169 | 5776 | 4917 | 27 | C(21) | 4793(1) | 10147(1) | 3510(1) | 19(1) |
| H(4B) | 3955 | 5871 | 5307 | 27 | H(21) | 4952 | 10586 | 3333 | 23 |
| H(4C) | 5285 | 6235 | 5517 | 27 | C(22) | 5629(1) | 9595(1) | 3448(1) | 18(1) |
| C(5) | 4993(1) | 8639(1) | 5243(1) | 20(1) | H(22) | 6365 | 9663 | 3229 | 22 |
| H(5A) | 5577 | 8925 | 5022 | 30 | C(23) | 5412(1) | 8941(1) | 3701(1) | 15(1) |
| H(5B) | 5454 | 8447 | 5602 | 30 | C(24) | 2273(1) | 9346(1) | 4452(1) | 18(1) |
| H(5C) | 4281 | 8929 | 5361 | 30 | H(24) | 2300 | 8881 | 4656 | 22 |
| C(6) | 3923(1) | 6008(1) | 3942(1) | 12(1) | C(25) | 2233(1) | 9915(1) | 4936(1) | 27(1) |
| C(7) | 2932(1) | 5529(1) | 4026(1) | 14(1) | H(25A) | 2167 | 10374 | 4745 | 41 |
| C(8) | 2969(1) | 4881(1) | 3738(1) | 18(1) | H(25B) | 3017 | 9893 | 5201 | 41 |
| H(8) | 2311 | 4550 | 3791 | 21 | H(25C) | 1490 | 9838 | 5172 | 41 |
| C(9) | 3945(1) | 4713(1) | 3378(1) | 20(1) | C(26) | 1050(1) | 9381(1) | 4038(1) | 26(1) |
| H(9) | 3958 | 4271 | 3187 | 24 | H(26A) | 999 | 9835 | 3837 | 39 |
| C(10) | 4910(1) | 5196(1) | 3296(1) | 18(1) | H(26B) | 305 | 9321 | 4276 | 39 |
| H(10) | 5575 | 5078 | 3046 | 22 | H(26C) | 1061 | 9009 | 3738 | 39 |
| C(11) | 4927(1) | 5848(1) | 3573(1) | 14(1) | C(27) | 6384(1) | 8361(1) | 3640(1) | 17(1) |
| C(12) | 1834(1) | 5677(1) | 4418(1) | 16(1) | H(27) | 5983 | 7915 | 3763 | 20 |
| H(12) | 1999 | 6137 | 4620 | 20 | C(28) | 7572(1) | 8486(1) | 4062(1) | 33(1) |
| C(13) | 1754(1) | 5116(1) | 4907(1) | 24(1) | H(28A) | 8199 | 8116 | 4007 | 49 |
| H(13A) | 1513 | 4669 | 4720 | 36 | H(28B) | 7331 | 8482 | 4478 | 49 |
| H(13B) | 1109 | 5253 | 5183 | 36 | H(28C) | 7947 | 8939 | 3973 | 49 |
| H(13C) | 2588 | 5071 | 5130 | 36 | C(29) | 6760(1) | 8272(1) | 2996(1) | 27(1) |
| C(14) | 551(1) | 5724(1) | 4047(1) | 22(1) | H(29A) | 5988 | 8216 | 2728 | 40 |
| H(14A) | 607 | 6080 | 3735 | 33 | H(29B) | 7303 | 7858 | 2970 | 40 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| H(29C) | 7231 | 8685 | 2876 | 40 | H(39B) | 236 | 8969 | 2586 | 44 |
| C(30) | 1099(1) | 7588(1) | 1873(1) | 14(1) | H(39C) | -167 | 9009 | 1884 | 44 |
| C(31) | 5(1) | 7688(1) | 1425(1) | 16(1) | C(40) | 2792(1) | 9127(1) | 2520(1) | 20(1) |
| C(32) | -1227(1) | 7825(1) | 1594(1) | 22(1) | H(40A) | 3638 | 8966 | 2418 | 30 |
| H(32) | -1387 | 7851 | 2007 | 26 | H(40B) | 2620 | 8958 | 2920 | 30 |
| C(33) | -2215(1) | 7922(1) | 1158(1) | 29(1) | H(40C) | 2770 | 9639 | 2515 | 30 |
| H(33) | -3055 | 8018 | 1272 | 35 | C(41) | 1196(1) | 6246(1) | 1924(1) | 16(1) |
| C(34) | -1983(1) | 7881(1) | 558(1) | 35(1) | C(42) | -254(1) | 6195(1) | 1954(1) | 31(1) |
| H(34) | -2668 | 7943 | 262 | 42 | H(42A) | -676 | 6422 | 1601 | 47 |
| C(35) | -761(1) | 7749(1) | 385(1) | 32(1) | H(42B) | -506 | 6428 | 2318 | 47 |
| H(35) | -607 | 7726 | -28 | 39 | H(42C) | -507 | 5703 | 1962 | 47 |
| C(36) | 239(1) | 7650(1) | 819(1) | 22(1) | C(43) | 1600(1) | 5991(1) | 1316(1) | 27(1) |
| H(36) | 1078 | 7557 | 703 | 27 | H(43A) | 1173 | 6273 | 996 | 41 |
| C(37) | 1778(1) | 8845(1) | 2062(1) | 18(1) | H(43B) | 1357 | 5500 | 1260 | 41 |
| C(38) | 2111(1) | 9043(1) | 1428(1) | 28(1) | H(43C) | 2529 | 6037 | 1303 | 41 |
| H(38A) | 1446 | 8868 | 1137 | 43 | C(44) | 1828(1) | 5787(1) | 2420(1) | 24(1) |
| H(38B) | 2936 | 8836 | 1345 | 43 | H(44A) | 1536 | 5304 | 2363 | 36 |
| H(38C) | 2165 | 9552 | 1395 | 43 | H(44B) | 1593 | 5956 | 2811 | 36 |
| C(39) | 479(1) | 9147(1) | 2199(1) | 29(1) | H(44C) | 2759 | 5806 | 2403 | 36 |
| H(39A) | 535 | 9658 | 2215 | 44 | C(45) | 4790(1) | 7121(1) | 1912(1) | 19(1) |

Tabelle 33: Anisotropic displacement parameters ($Å^2 \times 10^3$) for jus_378m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Cl(1) | 14(1) | 20(1) | 43(1) | 1(1) | 6(1) | 1(1) | C(10) | 21(1) | 14(1) | 21(1) | -2(1) | 4(1) | 2(1) |
| Si(1) | 16(1) | 11(1) | 10(1) | 0(1) | -2(1) | -1(1) | C(11) | 15(1) | 12(1) | 14(1) | 0(1) | 0(1) | 1(1) |
| Al(1) | 12(1) | 9(1) | 10(1) | 0(1) | 0(1) | 0(1) | C(12) | 18(1) | 14(1) | 17(1) | 1(1) | 3(1) | -4(1) |
| O(1) | 22(1) | 14(1) | 10(1) | 0(1) | -3(1) | 0(1) | C(13) | 30(1) | 22(1) | 21(1) | 5(1) | 5(1) | -8(1) |
| O(2) | 18(1) | 15(1) | 18(1) | 0(1) | 1(1) | 0(1) | C(14) | 17(1) | 23(1) | 26(1) | 0(1) | 2(1) | -4(1) |
| O(3) | 23(1) | 16(1) | 16(1) | 2(1) | 2(1) | -2(1) | C(15) | 14(1) | 13(1) | 17(1) | 1(1) | 2(1) | 1(1) |
| O(4) | 27(1) | 31(1) | 25(1) | -2(1) | 10(1) | 0(1) | C(16) | 16(1) | 30(1) | 21(1) | 1(1) | -1(1) | -3(1) |
| N(1) | 13(1) | 9(1) | 11(1) | 1(1) | 0(1) | 0(1) | C(17) | 20(1) | 19(1) | 23(1) | -1(1) | 7(1) | 1(1) |
| N(2) | 15(1) | 10(1) | 10(1) | 0(1) | 0(1) | -1(1) | C(18) | 18(1) | 10(1) | 11(1) | 0(1) | -1(1) | -2(1) |
| N(3) | 19(1) | 12(1) | 14(1) | 1(1) | -4(1) | -1(1) | C(19) | 20(1) | 11(1) | 12(1) | -1(1) | -1(1) | -1(1) |
| N(4) | 17(1) | 11(1) | 14(1) | -1(1) | -2(1) | -1(1) | C(20) | 25(1) | 10(1) | 17(1) | -1(1) | -3(1) | -1(1) |
| C(1) | 14(1) | 12(1) | 11(1) | 2(1) | 0(1) | -1(1) | C(21) | 27(1) | 12(1) | 18(1) | 2(1) | -2(1) | -5(1) |
| C(2) | 19(1) | 13(1) | 10(1) | 1(1) | -1(1) | -2(1) | C(22) | 23(1) | 15(1) | 17(1) | 1(1) | 2(1) | -5(1) |
| C(3) | 17(1) | 12(1) | 10(1) | -1(1) | 0(1) | -1(1) | C(23) | 18(1) | 12(1) | 13(1) | -1(1) | 0(1) | -4(1) |
| C(4) | 24(1) | 14(1) | 14(1) | 5(1) | -4(1) | 0(1) | C(24) | 23(1) | 13(1) | 19(1) | 0(1) | 4(1) | 2(1) |
| C(5) | 32(1) | 15(1) | 13(1) | -2(1) | -4(1) | -4(1) | C(25) | 41(1) | 18(1) | 24(1) | -4(1) | 11(1) | 3(1) |
| C(6) | 15(1) | 9(1) | 12(1) | 1(1) | -1(1) | 0(1) | C(26) | 21(1) | 24(1) | 33(1) | 4(1) | 2(1) | |
| C(7) | 17(1) | 11(1) | 14(1) | 1(1) | 0(1) | -1(1) | C(27) | 18(1) | 15(1) | 18(1) | 0(1) | 3(1) | |
| C(8) | 22(1) | 10(1) | 20(1) | 0(1) | 0(1) | -3(1) | C(28) | 27(1) | 33(1) | 36(1) | -9(1) | -10(1) | 8(1) |
| C(9) | 27(1) | 11(1) | 22(1) | -3(1) | 1(1) | 0(1) | C(29) | 32(1) | 27(1) | 23(1) | -3(1) | 9(1) | 2(1) |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| C(30) | 16(1) | 14(1) | 11(1) | 0(1) | 0(1) | -1(1) | C(38) | 50(1) | 18(1) | 16(1) | 6(1) | -4(1) | -6(1) |
| C(31) | 16(1) | 16(1) | 14(1) | 1(1) | -2(1) | -1(1) | C(39) | 29(1) | 18(1) | 40(1) | -3(1) | -6(1) | 5(1) |
| C(32) | 19(1) | 22(1) | 24(1) | 4(1) | 2(1) | -1(1) | C(40) | 29(1) | 12(1) | 18(1) | 0(1) | -5(1) | -3(1) |
| C(33) | 17(1) | 28(1) | 41(1) | 9(1) | -2(1) | -1(1) | C(41) | 19(1) | 12(1) | 18(1) | -3(1) | 0(1) | -3(1) |
| C(34) | 25(1) | 44(1) | 35(1) | 10(1) | -14(1) | -3(1) | C(42) | 21(1) | 18(1) | 56(1) | -2(1) | 3(1) | -6(1) |
| C(35) | 32(1) | 45(1) | 18(1) | 4(1) | -8(1) | -2(1) | C(43) | 42(1) | 20(1) | 19(1) | -7(1) | 2(1) | 1(1) |
| C(36) | 22(1) | 29(1) | 16(1) | 1(1) | -2(1) | -1(1) | C(44) | 33(1) | 15(1) | 23(1) | 4(1) | -2(1) | -5(1) |
| C(37) | 26(1) | 11(1) | 16(1) | 2(1) | -5(1) | 0(1) | C(45) | 22(1) | 20(1) | 15(1) | -2(1) | 1(1) | -2(1) |

Tabelle 34: Bond lengths [Å] for jus_378m.

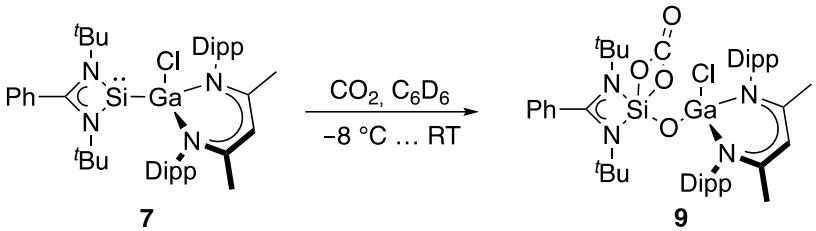
| | | | | | |
|---|---|---|---|---|---|
| Cl(1)-Al(1) | 2.1419(5) | N(4)-C(41) | 1.4852(13) | C(20)-C(21) | 1.3865(16) |
| Si(1)-O(1) | 1.5921(8) | C(1)-C(2) | 1.4033(13) | C(21)-C(22) | 1.3857(16) |
| Si(1)-O(3) | 1.7260(8) | C(1)-C(4) | 1.5087(13) | C(22)-C(23) | 1.4001(14) |
| Si(1)-O(2) | 1.7963(8) | C(2)-C(3) | 1.4052(13) | C(23)-C(27) | 1.5196(15) |
| Si(1)-N(4) | 1.8026(9) | C(3)-C(5) | 1.5057(14) | C(24)-C(26) | 1.5300(17) |
| Si(1)-N(3) | 1.8968(9) | C(6)-C(7) | 1.4083(13) | C(24)-C(25) | 1.5393(16) |
| Si(1)-C(45) | 2.2264(12) | C(6)-C(11) | 1.4136(14) | C(27)-C(29) | 1.5280(16) |
| Si(1)-C(30) | 2.3041(11) | C(7)-C(8) | 1.4018(14) | C(27)-C(28) | 1.5290(17) |
| Al(1)-O(1) | 1.6999(8) | C(7)-C(12) | 1.5203(14) | C(30)-C(31) | 1.4830(14) |
| Al(1)-N(2) | 1.8799(9) | C(8)-C(9) | 1.3815(16) | C(31)-C(36) | 1.3943(15) |
| Al(1)-N(1) | 1.8803(8) | C(9)-C(10) | 1.3911(15) | C(31)-C(32) | 1.3945(16) |
| O(2)-C(45) | 1.3267(13) | C(10)-C(11) | 1.3945(14) | C(32)-C(33) | 1.3861(17) |
| O(3)-C(45) | 1.3682(14) | C(11)-C(15) | 1.5271(14) | C(33)-C(34) | 1.381(2) |
| O(4)-C(45) | 1.2001(14) | C(12)-C(14) | 1.5351(15) | C(34)-C(35) | 1.386(2) |
| N(1)-C(1) | 1.3438(12) | C(12)-C(13) | 1.5386(15) | C(35)-C(36) | 1.3916(17) |
| N(1)-C(6) | 1.4532(12) | C(15)-C(17) | 1.5311(15) | C(37)-C(40) | 1.5239(15) |
| N(2)-C(3) | 1.3424(12) | C(15)-C(16) | 1.5341(15) | C(37)-C(39) | 1.5295(17) |
| N(2)-C(18) | 1.4527(12) | C(18)-C(23) | 1.4081(14) | C(37)-C(38) | 1.5302(16) |
| N(3)-C(30) | 1.3191(13) | C(18)-C(19) | 1.4082(14) | C(41)-C(42) | 1.5282(17) |
| N(3)-C(37) | 1.4822(13) | C(19)-C(20) | 1.4025(14) | C(41)-C(44) | 1.5299(16) |
| N(4)-C(30) | 1.3580(13) | C(19)-C(24) | 1.5210(15) | C(41)-C(43) | 1.5302(16) |

Tabelle 35: Bond angles [°] for jus_378m.

| | | | | | |
|---|---|---|---|---|---|
| O(1)-Si(1)-O(3) | 118.34(4) | C(30)-N(4)-C(41) | 129.89(9) | C(18)-C(23)-C(27) | 122.49(9) |
| O(1)-Si(1)-O(2) | 100.74(4) | C(30)-N(4)-Si(1) | 92.52(6) | C(19)-C(24)-C(26) | 111.36(9) |
| O(3)-Si(1)-O(2) | 74.48(4) | C(41)-N(4)-Si(1) | 137.56(7) | C(19)-C(24)-C(25) | 111.68(9) |
| O(1)-Si(1)-N(4) | 123.09(4) | N(1)-C(1)-C(2) | 122.98(9) | C(26)-C(24)-C(25) | 109.39(10) |
| O(3)-Si(1)-N(4) | 118.50(4) | N(1)-C(1)-C(4) | 120.64(9) | C(23)-C(27)-C(29) | 112.56(9) |
| O(2)-Si(1)-N(4) | 96.29(4) | C(2)-C(1)-C(4) | 116.30(8) | C(23)-C(27)-C(28) | 110.70(9) |
| O(1)-Si(1)-N(3) | 104.74(4) | C(1)-C(2)-C(3) | 127.27(9) | C(29)-C(27)-C(28) | 110.38(10) |
| O(3)-Si(1)-N(3) | 92.27(4) | N(2)-C(3)-C(2) | 122.97(9) | N(3)-C(30)-N(4) | 106.11(9) |
| O(2)-Si(1)-N(3) | 154.50(4) | N(2)-C(3)-C(5) | 120.24(9) | N(3)-C(30)-C(31) | 127.21(9) |
| N(4)-Si(1)-N(3) | 70.62(4) | C(2)-C(3)-C(5) | 116.78(9) | N(4)-C(30)-C(31) | 126.32(9) |
| O(1)-Si(1)-C(45) | 114.80(4) | C(7)-C(6)-C(11) | 121.44(9) | N(3)-C(30)-Si(1) | 55.41(5) |
| O(3)-Si(1)-C(45) | 37.91(4) | C(7)-C(6)-N(1) | 119.81(8) | N(4)-C(30)-Si(1) | 51.41(5) |
| O(2)-Si(1)-C(45) | 36.58(4) | C(11)-C(6)-N(1) | 118.75(8) | C(31)-C(30)-Si(1) | 167.15(7) |
| N(4)-Si(1)-C(45) | 110.96(4) | C(8)-C(7)-C(6) | 118.21(9) | C(36)-C(31)-C(32) | 120.08(10) |
| N(3)-Si(1)-C(45) | 126.65(4) | C(8)-C(7)-C(12) | 118.41(9) | C(36)-C(31)-C(30) | 118.07(10) |
| O(1)-Si(1)-C(30) | 122.93(4) | C(6)-C(7)-C(12) | 123.38(9) | C(32)-C(31)-C(30) | 121.85(10) |
| O(3)-Si(1)-C(30) | 104.64(4) | C(9)-C(8)-C(7) | 121.22(10) | C(33)-C(32)-C(31) | 119.69(11) |
| O(2)-Si(1)-C(30) | 126.84(4) | C(8)-C(9)-C(10) | 119.69(10) | C(34)-C(33)-C(32) | 120.18(12) |
| N(4)-Si(1)-C(30) | 36.07(4) | C(9)-C(10)-C(11) | 121.71(10) | C(33)-C(34)-C(35) | 120.55(12) |
| N(3)-Si(1)-C(30) | 34.92(4) | C(10)-C(11)-C(6) | 117.73(9) | C(34)-C(35)-C(36) | 119.78(12) |
| C(45)-Si(1)-C(30) | 122.15(4) | C(10)-C(11)-C(15) | 120.39(9) | C(35)-C(36)-C(31) | 119.71(12) |
| O(1)-Al(1)-N(2) | 115.09(4) | C(6)-C(11)-C(15) | 121.88(8) | N(3)-C(37)-C(40) | 105.09(8) |
| O(1)-Al(1)-N(1) | 114.68(4) | C(7)-C(12)-C(14) | 111.56(9) | N(3)-C(37)-C(39) | 111.38(9) |
| N(2)-Al(1)-N(1) | 98.68(4) | C(7)-C(12)-C(13) | 111.13(9) | C(40)-C(37)-C(39) | 108.67(9) |
| O(1)-Al(1)-Cl(1) | 110.14(3) | C(14)-C(12)-C(13) | 109.71(9) | N(3)-C(37)-C(38) | 110.83(9) |
| N(2)-Al(1)-Cl(1) | 108.57(3) | C(11)-C(15)-C(17) | 113.74(8) | C(40)-C(37)-C(38) | 109.98(10) |
| N(1)-Al(1)-Cl(1) | 109.01(3) | C(11)-C(15)-C(16) | 112.31(8) | C(39)-C(37)-C(38) | 110.73(10) |
| Si(1)-O(1)-Al(1) | 164.39(5) | C(17)-C(15)-C(16) | 107.94(9) | N(4)-C(41)-C(42) | 111.28(9) |
| C(45)-O(2)-Si(1) | 89.64(6) | C(23)-C(18)-C(19) | 121.52(9) | N(4)-C(41)-C(44) | 107.19(8) |
| C(45)-O(3)-Si(1) | 91.29(6) | C(23)-C(18)-N(2) | 118.03(8) | C(42)-C(41)-C(44) | 108.33(10) |
| C(1)-N(1)-C(6) | 119.00(8) | C(19)-C(18)-N(2) | 120.45(9) | N(4)-C(41)-C(43) | 110.02(9) |
| C(1)-N(1)-Al(1) | 118.80(6) | C(20)-C(19)-C(18) | 117.83(9) | C(42)-C(41)-C(43) | 110.76(10) |
| C(6)-N(1)-Al(1) | 121.81(6) | C(20)-C(19)-C(24) | 118.83(9) | C(44)-C(41)-C(43) | 109.15(10) |
| C(3)-N(2)-C(18) | 118.47(8) | C(18)-C(19)-C(24) | 123.34(9) | O(4)-C(45)-O(2) | 129.03(11) |
| C(3)-N(2)-Al(1) | 118.93(6) | C(21)-C(20)-C(19) | 121.57(10) | O(4)-C(45)-O(3) | 126.38(10) |
| C(18)-N(2)-Al(1) | 122.53(6) | C(22)-C(21)-C(20) | 119.55(9) | O(2)-C(45)-O(3) | 104.59(9) |
| C(30)-N(3)-C(37) | 129.89(9) | C(21)-C(22)-C(23) | 121.41(10) | O(4)-C(45)-Si(1) | 176.80(10) |
| C(30)-N(3)-Si(1) | 89.67(6) | C(22)-C(23)-C(18) | 118.11(9) | O(2)-C(45)-Si(1) | 53.79(5) |
| C(37)-N(3)-Si(1) | 136.81(7) | C(22)-C(23)-C(27) | 119.34(9) | O(3)-C(45)-Si(1) | 50.81(5) |

## 9. PhC(N$^t$Bu)$_2$Si(O$_2$CO)OGa(Cl)DDP 9

### 9.1. Synthese PhC(N$^t$Bu)$_2$Si(O$_2$CO)OGa(Cl)DDP **9**




**7** **9**

Dipp = 2,6-$^i$Pr$_2$C$_6$H$_3$

Es wurden 15 mg PhC(N$^t$Bu)$_2$SiGa(Cl)DDP **7** (0.019 mmol) in 0.5 mL $C_6D_6$ gelöst, die Lösung eingefroren und die Atmosphäre im Vakuum entfernt. Danach wurde das Kältebad entfernt und die Atmosphäre mit Kohlenstoffdioxid versetzt. Dabei entfärbte sich die zunächst gelbe Lösung unter Gasentwicklung. Die Lösung wurde auf etwa die Hälfte eingeengt und bei −18 °C gelagert, wobei PhC(N$^t$Bu)$_2$Si(O$_2$CO)OGa(Cl)DDP **9** in Form von farblosen Kristallen erhalten werden konnte.

**Ausbeute** 8.3 mg (0.010 mmol, 52 %).

**Smp.** 182 °C.

**Elementaranalyse** von $C_{45}H_{64}ClGaN_4O_4Si$: gefunden (berechnet) C 67.8 (62.68), H 8.67 (7.95), N 7.35 (6.50)%.

**IR**: ν 2954, 2862, 1785, 1523, 1462, 1431, 1388, 1362, 1308, 1258, 1204, 1173, 1092, 996, 927, 873, 842, 815, 796, 754, 715, 654, 604, 523, 481 cm$^{-1}$.

**$^1$H NMR** ($C_6D_6$, 400 MHz): δ 7.6-6.80 (m, 11 H, $C_6H_3$($^i$Pr)$_2$ & $C_6H_5$), 4.77 (s, 1 H, γ-C*H*), 3.49 (d sept, $^3J_{HH}$ = 6.7 Hz, 4 H, -C*H*(CH$_3$)$_2$), 1.80 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H*$_3$)$_2$), 1.54 (d, $^3J_{HH}$ = 6.7 Hz, 6 H, -CH(C*H*$_3$)$_2$), 1.48 (s, 6 H, ArNCC*H*$_3$), 1.18 (d, $^3J_{HH}$ = 6.9 Hz, 6 H, -CH(C*H*$_3$)$_2$), 1.04 (d, $^3J_{HH}$ = 6.9 Hz, 6 H, -CH(C*H*$_3$)$_2$), 0.68 (s, 18 H, C(C*H*$_3$)$_3$) ppm.

**$^{13}$C NMR** ($C_6D_6$, 101 MHz): δ 175.6 (NCN), 171.4 (ArN*C*CH$_3$), 154.0 (*C*O$_3$), 144.6, 144.3, 139.6 (*C*$_6$H$_3$), 131.7, 129.6, 129.0, 127.0, 124.9, 124.5, 96.7(γ-*C*H-), 53.4 (*C*(CH$_3$)$_3$), 30.4 (C(*C*H$_3$)$_3$), 29.4, 27.8 (-*C*H(CH$_3$)$_2$), 26.4, 24.7, 24.6 (CH(*C*H$_3$)$_2$), 23.7(ArNC*C*H$_3$), 23.2 (CH(*C*H$_3$)$_2$) ppm.

**$^{29}$Si NMR** ($C_6D_6$, 79 MHz): δ –116.4 ppm

## 9.2. Spektren PhC(N$^t$Bu)$_2$Si(O$_2$CO)OGa(Cl)DDP **9**

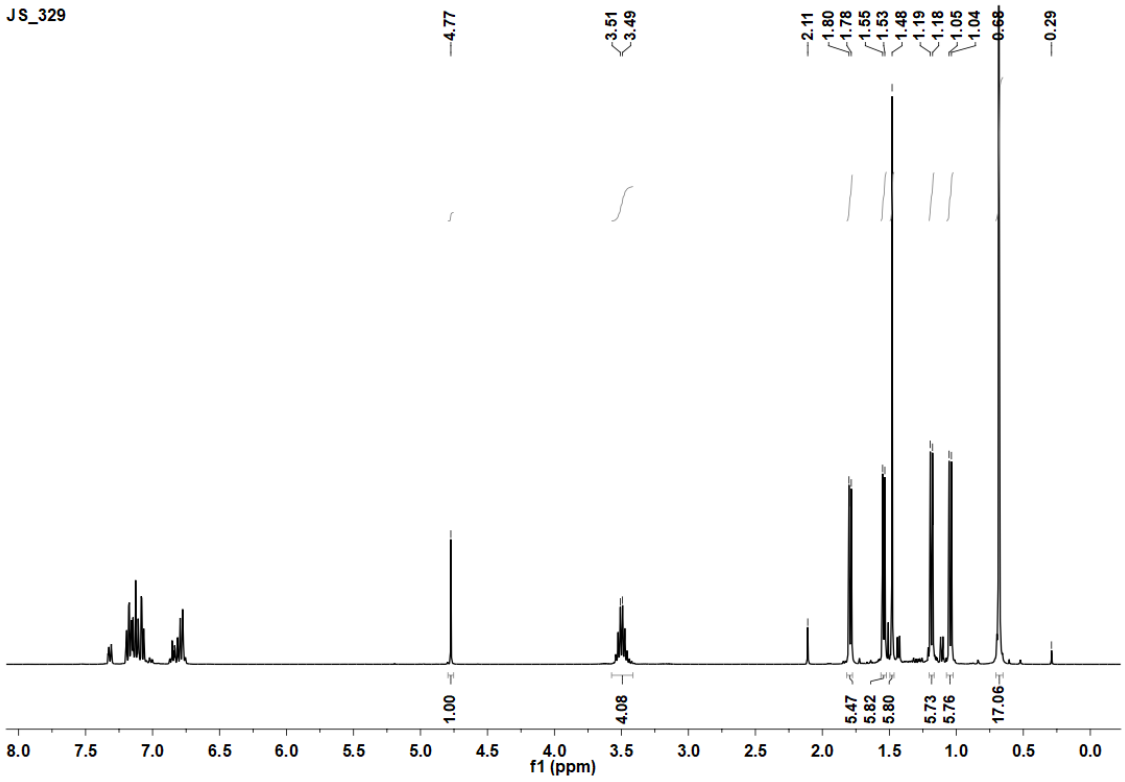



Abbildung 37: $^1$H-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(O$_2$CO)OGa (Cl)DDP **9** in $C_6D_6$.

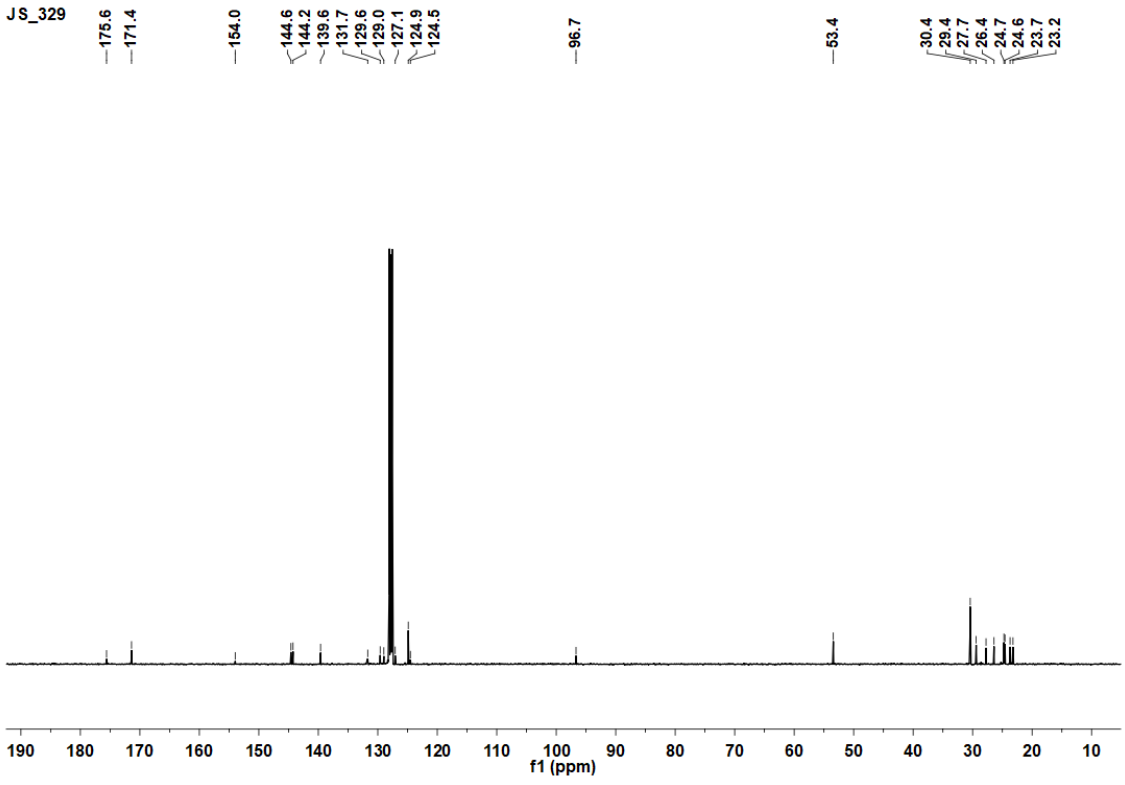



Abbildung 38: $^{13}$C-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(O$_2$CO)OGa (Cl)DDP **9** in $C_6D_6$.

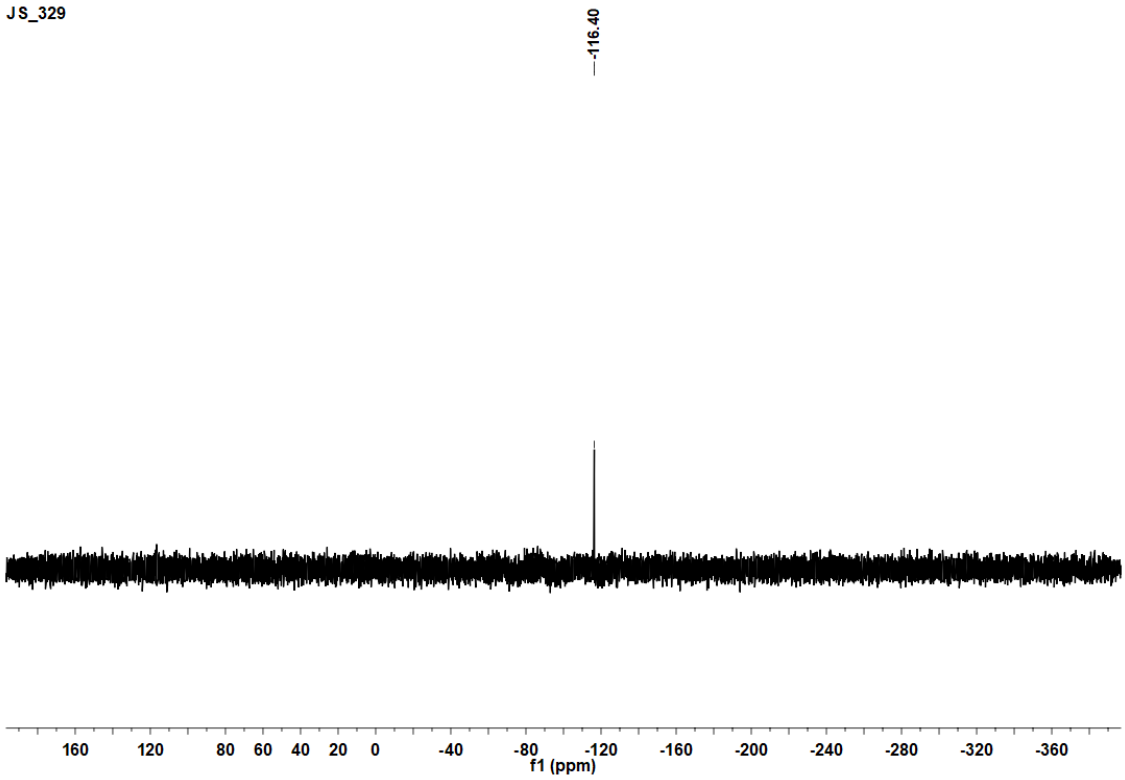



Abbildung 39: $^{29}$Si-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(O$_2$CO)OGa (Cl)DDP **9** in $C_6D_6$.

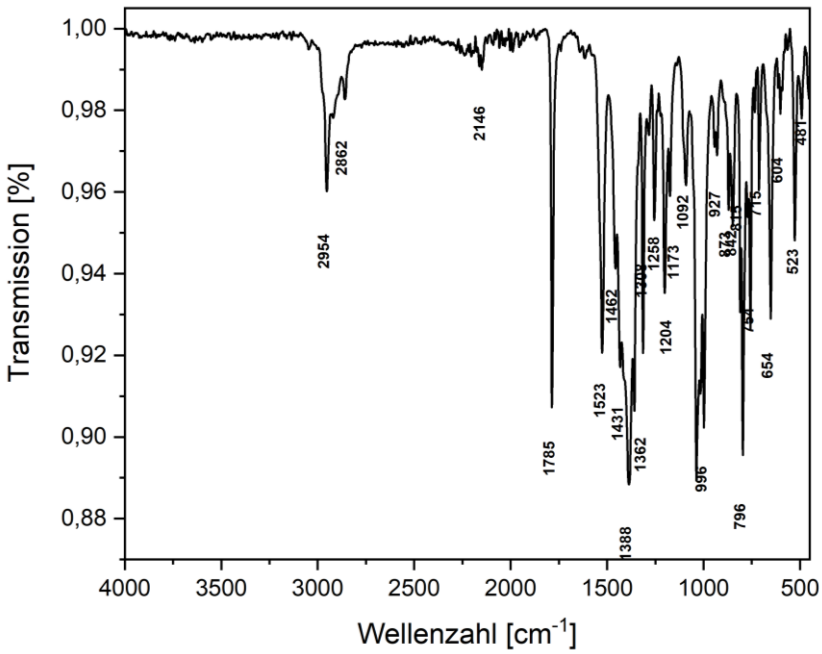



Abbildung 40: ATR-IR Spektrum von PhC(N$^t$Bu)$_2$Si(O$_2$CO)OGa (Cl)DDP **9**.

## 9.3. Kristallografische Daten PhC(N$^t$Bu)$_2$Si(O$_2$CO)OGa(Cl)DDP **9**

Tabelle 36: Crystal structure data

| **Identification code** | **jus_329_tw5** |
|---|---|
| Empirical formula | C45 H64 Cl Ga N4 O4 Si |
| Formula weight | 858.26 |
| Density (calculated) | 1.271 g·cm$^{-3}$ |
| *F*(000) | 1824 |
| Temperature | 100(2) K |
| Crystal size | 0.228 × 0.179 × 0.108 mm |
| Crystal colour | colourless |
| Crystal description | block |
| Wavelength | 1.54178 Å |
| Crystal system | monoclinic |
| Space group | *P 21/n* |
| Unit cell dimensions | |
| *a* [Å] | 15.6883(16) |
| *b* [Å] | 14.5790(15) |
| *c* [Å] | 19.616(2) |
| *α* [°] | 90 |
| *β* [°] | 92.105(4) |
| *γ* [°] | 90 |
| Volume | 4483.6(8) Å$^3$ |
| *Z* | 4 |
| Cell measurement reflections used | 9327 |
| Cell measurement *θ* min/max | 3.67°/79.10° |
| Diffractometer control software | Bruker APEX3(v2017.3-0) |
| Diffractometer measurement device | Bruker D8 Venture (Photon II detector) |
| Diffractometer measurement method | Data collection strategy APEX 3/Queen |
| *θ* range for data collection | 3.544°- 79.685° |
| Completeness to *θ* = 67.679° | 99.9% |
| Completeness to $\theta_{max}$ = 79.685° | 98.9% |
| Index ranges | -19 ≤ *h* ≤ 19 |
| | 0 ≤ *k* ≤ 18 |
| | 0 ≤ *l* ≤ 25 |
| Computing data reduction | Bruker APEX3(v2017.3-0) |
| Absorption coefficient | 1.998 mm$^{-1}$ |
| Absorption correction | Semi-empirical from equivalents |
| Computation absorption correction | TWINABS |
| Max./min. Transmission | 0.75/0.62 |
| $R_{merg}$ before/after correction | 0.1219/0.0830 and 0.1285/0.0830 |
| Computing structure solution | Bruker APEX3(v2017.3-0) |
| Computing structure refinement | SHELXL-2017/1 (Sheldrick, 2017) |

| | |
|---|---|
| Refinement method | Full-matrix least-squares on $F^2$ |
| Reflections collected | 232158 |
| Independent reflections | 9801 |
| $R_{int}$ | 0.0707 |
| Reflections with $I > 2\sigma(I)$ | 8909 |
| Restraints | 0 |
| Parameter | 590 |
| GooF | 1.079 |
| Weighting details | $w = 1/[\sigma^2(F_{obs}^2) + (0.0769P)^2 + 4.3464P]$ |
| | where $P = (F_{obs}^2 + 2F_{calc}^2)/3$ |
| $R_1$ [$I > 2\sigma(I)$] | 0.0448 |
| $wR_2$ [$I > 2\sigma(I)$] | 0.1257 |
| $R_1$ [all data] | 0.0568 |
| $wR_2$ [all data] | 0.1431 |
| Absolute structure parameter | |
| Largest diff. peak and hole | 1.059/-0.977 |

Tabelle 37: Atomic coordinates ( × $10^4$) and equivalent isotropic displacement parameters (Å$^2$ × $10^3$) for jus_329_tw5. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | *x* | *y* | *z* | $U_{eq}$ | | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|---|---|---|---|---|
| Ga(1) | 4321(1) | 2152(1) | 7412(1) | 21(1) | H(14C) | 2197 | 3322 | 5656 | 49 |
| Cl(1) | 3343(1) | 3235(1) | 7414(1) | 25(1) | C(15) | 5924(2) | 961(2) | 6370(2) | 32(1) |
| Si(1) | 5905(1) | 3585(1) | 7548(1) | 23(1) | H(15) | 5731 | 1143 | 6831 | 38 |
| O(1) | 5345(1) | 2685(1) | 7464(1) | 30(1) | C(16) | 5831(5) | -74(4) | 6315(4) | 108(3) |
| N(1) | 4148(2) | 1368(2) | 6622(1) | 22(1) | H(16A) | 5226 | -238 | 6316 | 162 |
| N(2) | 4123(2) | 1266(2) | 8118(1) | 26(1) | H(16B) | 6133 | -366 | 6703 | 162 |
| N(3) | 5337(2) | 4474(2) | 8047(1) | 26(1) | H(16C) | 6075 | -285 | 5889 | 162 |
| N(4) | 5536(2) | 4518(2) | 6975(1) | 26(1) | C(17) | 6848(3) | 1240(4) | 6334(2) | 59(1) |
| C(1) | 3700(2) | 598(2) | 6682(2) | 27(1) | H(17A) | 7084 | 994 | 5915 | 88 |
| C(2) | 3415(2) | 256(2) | 7299(2) | 27(1) | H(17B) | 7171 | 995 | 6731 | 88 |
| H(2) | 2991 | -208 | 7261 | 32 | H(17C) | 6890 | 1910 | 6332 | 88 |
| C(3) | 3670(2) | 504(2) | 7964(2) | 26(1) | C(18) | 4462(2) | 1402(2) | 8812(2) | 26(1) |
| C(4) | 3493(3) | 9(2) | 6066(2) | 36(1) | C(19) | 3936(2) | 1737(2) | 9313(2) | 28(1) |
| H(4A) | 3774 | 261 | 5669 | 54 | C(20) | 4294(2) | 1881(2) | 9971(2) | 32(1) |
| H(4B) | 2875 | 1 | 5976 | 54 | H(20) | 3954 | 2124 | 10319 | 38 |
| H(4C) | 3696 | -617 | 6152 | 54 | C(21) | 5144(2) | 1670(3) | 10114(2) | 36(1) |
| C(5) | 3426(2) | -144(2) | 8521(2) | 32(1) | H(21) | 5383 | 1768 | 10560 | 43 |
| H(5A) | 3359 | -764 | 8333 | 49 | C(22) | 5643(2) | 1320(3) | 9614(2) | 34(1) |
| H(5B) | 2887 | 55 | 8709 | 49 | H(22) | 6221 | 1170 | 9725 | 41 |
| H(5C) | 3874 | -146 | 8883 | 49 | C(23) | 5325(2) | 1178(2) | 8949(2) | 27(1) |
| C(6) | 4475(2) | 1615(2) | 5965(2) | 24(1) | C(24) | 2992(2) | 1956(3) | 9188(2) | 35(1) |
| C(7) | 3919(2) | 2003(2) | 5462(2) | 24(1) | H(24) | 2823 | 1748 | 8716 | 42 |
| C(8) | 4252(2) | 2211(2) | 4832(2) | 30(1) | C(25) | 2430(3) | 1449(3) | 9686(2) | 46(1) |
| H(8) | 3890 | 2465 | 4482 | 36 | H(25A) | 1830 | 1504 | 9533 | 68 |
| C(9) | 5102(2) | 2054(3) | 4707(2) | 36(1) | H(25B) | 2508 | 1718 | 10142 | 68 |
| H(9) | 5321 | 2214 | 4278 | 43 | H(25C) | 2591 | 799 | 9703 | 68 |
| C(10) | 5636(2) | 1662(3) | 5208(2) | 35(1) | C(26) | 2819(3) | 2987(3) | 9231(2) | 41(1) |
| H(10) | 6216 | 1547 | 5113 | 42 | H(26A) | 3154 | 3309 | 8893 | 62 |
| C(11) | 5336(2) | 1436(2) | 5844(2) | 27(1) | H(26B) | 2981 | 3209 | 9689 | 62 |
| C(12) | 2983(2) | 2195(2) | 5572(2) | 29(1) | H(26C) | 2210 | 3103 | 9137 | 62 |
| H(12) | 2841 | 1948 | 6029 | 35 | C(27) | 5884(2) | 762(2) | 8418(2) | 32(1) |
| C(13) | 2401(3) | 1718(3) | 5029(2) | 52(1) | H(27) | 5658 | 968 | 7961 | 38 |
| H(13A) | 2473 | 2009 | 4585 | 79 | C(28) | 5849(4) | -271(3) | 8430(4) | 75(2) |
| H(13B) | 1805 | 1774 | 5156 | 79 | H(28A) | 5270 | -475 | 8301 | 113 |
| H(13C) | 2554 | 1068 | 5002 | 79 | H(28B) | 6003 | -491 | 8890 | 113 |
| C(14) | 2801(2) | 3219(3) | 5568(2) | 32(1) | H(28C) | 6251 | -519 | 8107 | 113 |
| H(14A) | 2931 | 3475 | 5122 | 49 | C(29) | 6799(4) | 1068(6) | 8493(3) | 90(3) |
| H(14B) | 3155 | 3519 | 5924 | 49 | H(29A) | 7112 | 854 | 8100 | 135 |
| H(29B) | 7058 | 812 | 8913 | 135 | H(42B) | 6796 | 4903 | 6202 | 48 |
| H(29C) | 6821 | 1740 | 8514 | 135 | H(42C) | 6290 | 5830 | 6351 | 48 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| C(30) | 5221(2) | 4993(2) | 7494(2) | 22(1) | C(43) | 5455(4) | 3835(4) | 5844(3) | 28(1) |
| C(31) | 4843(2) | 5925(2) | 7456(1) | 21(1) | H(43A) | 5492 | 3969 | 5357 | 43 |
| C(32) | 5369(2) | 6689(2) | 7460(2) | 26(1) | H(43B) | 4915 | 3522 | 5925 | 43 |
| H(32) | 5970 | 6617 | 7495 | 31 | H(43C) | 5932 | 3439 | 5992 | 43 |
| C(33) | 5016(2) | 7556(2) | 7415(2) | 34(1) | C(44) | 4678(4) | 5295(5) | 5991(3) | 32(1) |
| H(33) | 5376 | 8080 | 7417 | 41 | H(44A) | 4646 | 5320 | 5492 | 48 |
| C(34) | 4138(2) | 7664(2) | 7366(2) | 40(1) | H(44B) | 4729 | 5919 | 6175 | 48 |
| H(34) | 3898 | 8261 | 7332 | 49 | H(44C) | 4161 | 5007 | 6156 | 48 |
| C(35) | 3610(2) | 6899(3) | 7366(2) | 39(1) | C(42') | 5719(4) | 5669(5) | 6041(3) | 22(2) |
| H(35) | 3008 | 6974 | 7339 | 47 | H(42D) | 5715 | 5720 | 5542 | 33 |
| C(36) | 3959(2) | 6031(2) | 7407(2) | 31(1) | H(42E) | 6288 | 5821 | 6230 | 33 |
| H(36) | 3598 | 5507 | 7401 | 37 | H(42F) | 5300 | 6096 | 6221 | 33 |
| C(37) | 5244(2) | 4704(2) | 8776(2) | 26(1) | C(43') | 6175(5) | 4034(6) | 5929(3) | 28(2) |
| C(38) | 6010(3) | 5287(3) | 9020(2) | 42(1) | H(43D) | 6147 | 4079 | 5430 | 42 |
| H(38A) | 6539 | 4958 | 8934 | 63 | H(43E) | 6051 | 3403 | 6065 | 42 |
| H(38B) | 5976 | 5405 | 9510 | 63 | H(43F) | 6748 | 4205 | 6101 | 42 |
| H(38C) | 6003 | 5871 | 8773 | 63 | C(44') | 4629(4) | 4391(5) | 6017(3) | 24(2) |
| C(39) | 4400(3) | 5199(3) | 8895(2) | 40(1) | H(44D) | 4572 | 4406 | 5518 | 36 |
| H(39A) | 4436 | 5833 | 8731 | 61 | H(44E) | 4201 | 4797 | 6210 | 36 |
| H(39B) | 4290 | 5201 | 9384 | 61 | H(44F) | 4541 | 3763 | 6179 | 36 |
| H(39C) | 3935 | 4881 | 8646 | 61 | O(2) | 6730(3) | 3223(4) | 7015(3) | 35(1) |
| C(40) | 5239(2) | 3794(2) | 9154(2) | 27(1) | O(3) | 6747(2) | 3612(4) | 8052(3) | 33(1) |
| H(40A) | 4773 | 3411 | 8968 | 41 | O(4) | 7996(3) | 3121(4) | 7621(5) | 52(2) |
| H(40B) | 5157 | 3905 | 9640 | 41 | C(45) | 7252(6) | 3284(6) | 7547(7) | 42(3) |
| H(40C) | 5784 | 3480 | 9098 | 41 | O(2') | 6999(5) | 3583(6) | 7325(5) | 30(2) |
| C(41) | 5494(2) | 4707(2) | 6237(2) | 23(1) | O(3') | 6614(4) | 3129(5) | 8291(3) | 25(2) |
| C(42) | 6285(4) | 5263(4) | 6083(3) | 32(1) | O(4') | 8021(4) | 2966(5) | 8063(5) | 36(2) |
| H(42A) | 6277 | 5413 | 5596 | 48 | C(45') | 7295(7) | 3188(7) | 7911(7) | 27(2) |

Tabelle 38: Anisotropic displacement parameters (Å$^2$ × 10$^3$) for jus_329_tw5. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ga(1) | 26(1) | 14(1) | 22(1) | 2(1) | -8(1) | -2(1) | C(25) | 34(2) | 41(2) | 62(3) | 7(2) | -4(2) | -2(2) |
| Cl(1) | 27(1) | 20(1) | 27(1) | 2(1) | -1(1) | 1(1) | C(26) | 48(2) | 34(2) | 43(2) | 4(2) | -1(2) | 8(2) |
| Si(1) | 26(1) | 16(1) | 28(1) | -1(1) | 9(1) | -2(1) | C(27) | 34(2) | 34(2) | 27(2) | 11(1) | -6(1) | -1(1) |
| O(1) | 27(1) | 19(1) | 42(1) | -1(1) | -15(1) | 1(1) | C(28) | 56(3) | 34(2) | 139(5) | -1(3) | 46(3) | 2(2) |
| N(1) | 26(1) | 16(1) | 22(1) | -1(1) | -7(1) | -1(1) | C(29) | 65(3) | 134(6) | 74(4) | -45(4) | 41(3) | -60(4) |
| N(2) | 36(1) | 18(1) | 24(1) | 4(1) | -11(1) | -3(1) | C(30) | 26(1) | 18(1) | 20(1) | -4(1) | 1(1) | -4(1) |
| N(3) | 40(1) | 22(1) | 17(1) | 0(1) | 6(1) | -1(1) | C(31) | 24(1) | 20(1) | 18(1) | -1(1) | -1(1) | 0(1) |
| N(4) | 42(2) | 18(1) | 17(1) | -1(1) | 6(1) | 4(1) | C(32) | 27(1) | 22(1) | 30(2) | 0(1) | 0(1) | -1(1) |
| C(1) | 31(2) | 17(1) | 32(2) | -3(1) | -11(1) | 1(1) | C(33) | 47(2) | 19(1) | 37(2) | -4(1) | -1(2) | 1(1) |
| C(2) | 31(1) | 17(1) | 32(2) | 2(1) | -10(1) | -4(1) | C(34) | 51(2) | 30(2) | 41(2) | 1(2) | -1(2) | 16(2) |
| C(3) | 28(1) | 16(1) | 35(2) | 7(1) | -6(1) | -2(1) | C(35) | 29(2) | 51(2) | 38(2) | 3(2) | -5(1) | 14(1) |
| C(4) | 50(2) | 22(2) | 34(2) | -3(1) | -12(2) | -10(2) | C(36) | 25(1) | 38(2) | 28(2) | 3(1) | -4(1) | -2(1) |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| C(5) | 38(2) | 22(2) | 37(2) | 9(1) | -4(1) | -4(1) | C(37) | 37(2) | 24(2) | 16(1) | -2(1) | 3(1) | -2(1) |
| C(6) | 30(2) | 19(1) | 21(1) | -4(1) | -5(1) | 2(1) | C(38) | 63(2) | 38(2) | 26(2) | -7(2) | 8(2) | -24(2) |
| C(7) | 29(2) | 17(1) | 26(1) | -4(1) | -8(1) | 1(1) | C(39) | 57(2) | 42(2) | 22(2) | -1(2) | 7(2) | 16(2) |
| C(8) | 38(2) | 31(2) | 20(1) | -4(1) | -6(1) | 10(1) | C(40) | 32(2) | 28(2) | 21(1) | 2(1) | 1(1) | -4(1) |
| C(9) | 45(2) | 42(2) | 20(1) | -4(1) | 4(1) | 11(2) | C(41) | 27(1) | 23(2) | 18(1) | 1(1) | 4(1) | -2(1) |
| C(10) | 34(2) | 41(2) | 29(2) | -10(2) | 0(1) | 12(2) | C(42) | 36(3) | 30(3) | 31(3) | 0(2) | 7(2) | -9(3) |
| C(11) | 34(2) | 23(2) | 24(1) | -9(1) | -5(1) | 11(1) | C(43) | 34(3) | 29(3) | 23(3) | -3(2) | 3(2) | -3(2) |
| C(12) | 24(2) | 33(2) | 31(2) | 10(1) | -10(1) | -6(1) | C(44) | 40(3) | 37(3) | 18(3) | 0(2) | -5(2) | 12(3) |
| C(13) | 47(2) | 48(2) | 60(3) | 8(2) | -32(2) | -14(2) | C(42') | 29(4) | 24(3) | 14(3) | 3(2) | 2(2) | 0(3) |
| C(14) | 30(2) | 36(2) | 30(2) | 4(1) | -1(1) | 12(1) | C(43') | 29(4) | 40(4) | 15(3) | 4(3) | 2(2) | 6(3) |
| C(15) | 32(2) | 31(2) | 32(2) | -6(1) | -7(1) | 12(1) | C(44') | 27(3) | 31(4) | 13(3) | 4(3) | 2(2) | -6(3) |
| C(16) | 119(6) | 34(3) | 164(7) | -5(3) | -98(5) | 22(3) | O(2) | 31(2) | 30(3) | 47(3) | 5(2) | 14(2) | 2(2) |
| C(17) | 30(2) | 88(4) | 58(3) | 19(3) | 1(2) | 17(2) | O(3) | 21(2) | 31(3) | 46(3) | -3(2) | -6(2) | -2(2) |
| C(18) | 34(2) | 19(1) | 24(2) | 7(1) | -9(1) | -7(1) | O(4) | 15(2) | 42(3) | 100(6) | -3(3) | 4(3) | 0(2) |
| C(19) | 35(2) | 22(1) | 26(2) | 7(1) | -7(1) | -2(1) | C(45) | 27(4) | 19(4) | 81(8) | 13(5) | 24(5) | 0(3) |
| C(20) | 38(2) | 34(2) | 24(2) | 7(1) | -3(1) | -5(2) | O(2') | 22(3) | 22(4) | 45(5) | -1(4) | 9(3) | -6(3) |
| C(21) | 40(2) | 41(2) | 26(2) | 10(2) | -10(1) | -8(2) | O(3') | 24(3) | 28(4) | 22(3) | -2(3) | -8(2) | 6(2) |
| C(22) | 32(2) | 43(2) | 26(2) | 8(2) | -7(1) | -7(2) | O(4') | 24(3) | 30(3) | 52(5) | -1(3) | -12(3) | 5(2) |
| C(23) | 32(2) | 22(1) | 25(2) | 10(1) | -6(1) | -5(1) | C(45') | 22(4) | 14(4) | 46(6) | -3(5) | 2(5) | -9(3) |
| C(24) | 34(2) | 33(2) | 36(2) | 1(1) | -8(1) | 3(1) | | | | | | | |

Tabelle 39: Bond lengths [Å] for jus_329_tw5.

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Ga(1)-O(1) | 1.785(2) | N(3)-C(30) | 1.329(4) | C(15)-C(17) | 1.509(5) | C(33)-C(34) | 1.386(5) |
| Ga(1)-N(2) | 1.926(3) | N(3)-C(37) | 1.482(4) | C(15)-C(16) | 1.519(6) | C(34)-C(35) | 1.390(6) |
| Ga(1)-N(1) | 1.936(2) | N(4)-C(30) | 1.342(4) | C(18)-C(19) | 1.394(5) | C(35)-C(36) | 1.380(5) |
| Ga(1)-Cl(1) | 2.2015(7) | N(4)-C(41) | 1.471(4) | C(18)-C(23) | 1.409(4) | C(37)-C(40) | 1.519(4) |
| Si(1)-O(1) | 1.584(2) | C(1)-C(2) | 1.398(5) | C(19)-C(20) | 1.406(4) | C(37)-C(38) | 1.534(5) |
| Si(1)-O(3) | 1.622(4) | C(1)-C(4) | 1.508(4) | C(19)-C(24) | 1.527(5) | C(37)-C(39) | 1.534(5) |
| Si(1)-O(2) | 1.774(4) | C(2)-C(3) | 1.399(4) | C(20)-C(21) | 1.386(5) | C(41)-C(44') | 1.483(7) |
| Si(1)-O(2') | 1.787(7) | C(3)-C(5) | 1.505(4) | C(21)-C(22) | 1.376(6) | C(41)-C(43) | 1.487(6) |
| Si(1)-N(4) | 1.844(3) | C(6)-C(11) | 1.403(4) | C(22)-C(23) | 1.394(4) | C(41)-C(42') | 1.501(7) |
| Si(1)-N(3) | 1.870(3) | C(6)-C(7) | 1.411(4) | C(23)-C(27) | 1.513(5) | C(41)-C(42) | 1.522(6) |
| Si(1)-O(3') | 1.921(6) | C(7)-C(8) | 1.393(5) | C(24)-C(25) | 1.531(6) | C(41)-C(43') | 1.587(8) |
| Si(1)-C(45) | 2.159(9) | C(7)-C(12) | 1.517(4) | C(24)-C(26) | 1.531(5) | C(41)-C(44) | 1.601(6) |
| Si(1)-C(30) | 2.317(3) | C(8)-C(9) | 1.384(5) | C(27)-C(29) | 1.505(6) | O(2)-C(45) | 1.306(13) |
| Si(1)-C(45') | 2.343(11) | C(9)-C(10) | 1.390(5) | C(27)-C(28) | 1.507(6) | O(3)-C(45) | 1.377(11) |
| N(1)-C(1) | 1.332(4) | C(10)-C(11) | 1.390(5) | C(30)-C(31) | 1.483(4) | O(4)-C(45) | 1.196(11) |
| N(1)-C(6) | 1.451(4) | C(11)-C(15) | 1.526(4) | C(31)-C(32) | 1.387(4) | O(2')-C(45') | 1.353(13) |
| N(2)-C(3) | 1.346(4) | C(12)-C(14) | 1.520(5) | C(31)-C(36) | 1.395(4) | O(3')-C(45') | 1.328(13) |
| N(2)-C(18) | 1.457(4) | C(12)-C(13) | 1.543(5) | C(32)-C(33) | 1.381(4) | O(4')-C(45') | 1.210(13) |

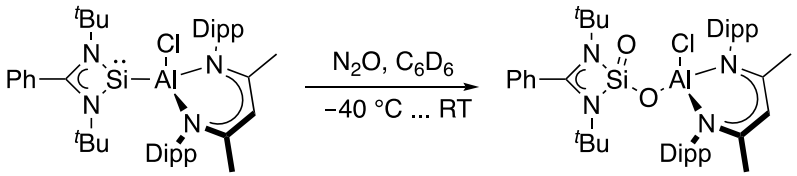
Tabelle 40: Bond angles [°] for jus_329_tw5.

| | | | | | |
|---|---|---|---|---|---|
| O(1)-Ga(1)-N(2) | 114.77(11) | Si(1)-O(1)-Ga(1) | 149.43(14) | C(18)-C(19)-C(24) | 123.8(3) |
| O(1)-Ga(1)-N(1) | 113.70(11) | C(1)-N(1)-C(6) | 119.5(3) | C(20)-C(19)-C(24) | 118.0(3) |
| N(2)-Ga(1)-N(1) | 99.10(11) | C(1)-N(1)-Ga(1) | 119.0(2) | C(21)-C(20)-C(19) | 120.1(3) |
| O(1)-Ga(1)-Cl(1) | 108.26(7) | C(6)-N(1)-Ga(1) | 121.43(19) | C(22)-C(21)-C(20) | 120.5(3) |
| N(2)-Ga(1)-Cl(1) | 110.44(9) | C(3)-N(2)-C(18) | 119.5(3) | C(21)-C(22)-C(23) | 121.9(3) |
| N(1)-Ga(1)-Cl(1) | 110.34(8) | C(3)-N(2)-Ga(1) | 119.3(2) | C(22)-C(23)-C(18) | 116.9(3) |
| O(1)-Si(1)-O(3) | 121.2(2) | C(18)-N(2)-Ga(1) | 121.2(2) | C(22)-C(23)-C(27) | 120.5(3) |
| O(1)-Si(1)-O(2) | 96.1(2) | C(30)-N(3)-C(37) | 130.0(3) | C(18)-C(23)-C(27) | 122.5(3) |
| O(3)-Si(1)-O(2) | 76.8(2) | C(30)-N(3)-Si(1) | 91.22(18) | C(19)-C(24)-C(25) | 112.0(3) |
| O(1)-Si(1)-O(2') | 120.5(3) | C(37)-N(3)-Si(1) | 136.7(2) | C(19)-C(24)-C(26) | 111.7(3) |
| O(1)-Si(1)-N(4) | 112.84(12) | C(30)-N(4)-C(41) | 130.3(3) | C(25)-C(24)-C(26) | 109.4(3) |
| O(3)-Si(1)-N(4) | 125.8(2) | C(30)-N(4)-Si(1) | 91.94(18) | C(29)-C(27)-C(28) | 109.3(5) |
| O(2)-Si(1)-N(4) | 94.60(17) | C(41)-N(4)-Si(1) | 137.6(2) | C(29)-C(27)-C(23) | 112.9(3) |
| O(2')-Si(1)-N(4) | 97.8(3) | N(1)-C(1)-C(2) | 124.3(3) | C(28)-C(27)-C(23) | 111.6(3) |
| O(1)-Si(1)-N(3) | 110.83(13) | N(1)-C(1)-C(4) | 120.5(3) | N(3)-C(30)-N(4) | 106.4(2) |
| O(3)-Si(1)-N(3) | 93.26(18) | C(2)-C(1)-C(4) | 115.1(3) | N(3)-C(30)-C(31) | 127.3(3) |
| O(2)-Si(1)-N(3) | 152.5(2) | C(1)-C(2)-C(3) | 128.8(3) | N(4)-C(30)-C(31) | 126.3(3) |
| O(2')-Si(1)-N(3) | 127.5(3) | N(2)-C(3)-C(2) | 123.6(3) | N(3)-C(30)-Si(1) | 53.78(15) |
| N(4)-Si(1)-N(3) | 70.31(11) | N(2)-C(3)-C(5) | 120.2(3) | N(4)-C(30)-Si(1) | 52.70(15) |
| O(1)-Si(1)-O(3') | 95.4(3) | C(2)-C(3)-C(5) | 116.3(3) | C(31)-C(30)-Si(1) | 176.0(2) |
| O(2')-Si(1)-O(3') | 69.5(4) | C(11)-C(6)-C(7) | 122.1(3) | C(32)-C(31)-C(36) | 120.1(3) |
| N(4)-Si(1)-O(3') | 151.4(3) | C(11)-C(6)-N(1) | 118.5(3) | C(32)-C(31)-C(30) | 119.9(2) |
| N(3)-Si(1)-O(3') | 96.7(2) | C(7)-C(6)-N(1) | 119.4(3) | C(36)-C(31)-C(30) | 120.0(3) |
| O(1)-Si(1)-C(45) | 111.7(2) | C(8)-C(7)-C(6) | 117.7(3) | C(33)-C(32)-C(31) | 119.9(3) |
| O(3)-Si(1)-C(45) | 39.6(4) | C(8)-C(7)-C(12) | 118.9(3) | C(32)-C(33)-C(34) | 120.2(3) |
| O(2)-Si(1)-C(45) | 37.2(4) | C(6)-C(7)-C(12) | 123.5(3) | C(33)-C(34)-C(35) | 120.0(3) |
| N(4)-Si(1)-C(45) | 115.8(3) | C(9)-C(8)-C(7) | 121.1(3) | C(36)-C(35)-C(34) | 120.1(3) |
| N(3)-Si(1)-C(45) | 128.8(3) | C(8)-C(9)-C(10) | 120.2(3) | C(35)-C(36)-C(31) | 119.8(3) |
| O(1)-Si(1)-C(30) | 118.41(10) | C(9)-C(10)-C(11) | 121.1(3) | N(3)-C(37)-C(40) | 106.0(3) |
| O(3)-Si(1)-C(30) | 111.8(2) | C(10)-C(11)-C(6) | 117.9(3) | N(3)-C(37)-C(38) | 108.9(3) |
| O(2)-Si(1)-C(30) | 125.73(16) | C(10)-C(11)-C(15) | 119.9(3) | C(40)-C(37)-C(38) | 110.5(3) |
| O(2')-Si(1)-C(30) | 116.0(3) | C(6)-C(11)-C(15) | 122.1(3) | N(3)-C(37)-C(39) | 111.6(3) |
| N(4)-Si(1)-C(30) | 35.36(10) | C(7)-C(12)-C(14) | 111.3(3) | C(40)-C(37)-C(39) | 108.5(3) |
| N(3)-Si(1)-C(30) | 34.99(11) | C(7)-C(12)-C(13) | 111.7(3) | C(38)-C(37)-C(39) | 111.2(3) |
| O(3')-Si(1)-C(30) | 126.5(2) | C(14)-C(12)-C(13) | 109.4(3) | N(4)-C(41)-C(44') | 103.6(3) |
| C(45)-Si(1)-C(30) | 129.2(2) | C(17)-C(15)-C(16) | 110.7(4) | N(4)-C(41)-C(43) | 110.5(3) |
| O(1)-Si(1)-C(45') | 109.4(3) | C(17)-C(15)-C(11) | 113.9(3) | N(4)-C(41)-C(42') | 115.2(3) |
| O(2')-Si(1)-C(45') | 35.1(4) | C(16)-C(15)-C(11) | 110.4(3) | C(44')-C(41)-C(42') | 115.9(4) |
| N(4)-Si(1)-C(45') | 129.5(3) | C(19)-C(18)-C(23) | 122.4(3) | N(4)-C(41)-C(42) | 106.8(3) |
| N(3)-Si(1)-C(45') | 117.9(3) | C(19)-C(18)-N(2) | 120.1(3) | C(43)-C(41)-C(42) | 111.8(4) |
| O(3')-Si(1)-C(45') | 34.5(4) | C(23)-C(18)-N(2) | 117.5(3) | N(4)-C(41)-C(43') | 104.7(3) |
| C(30)-Si(1)-C(45') | 131.1(3) | C(18)-C(19)-C(20) | 118.3(3) | C(44')-C(41)-C(43') | 108.6(4) |

| | | | | | |
|---|---|---|---|---|---|
| C(42')-C(41)-C(43') | 108.2(4) | O(4)-C(45)-O(3) | 124.6(12) | O(4')-C(45')-O(3') | 127.8(11) |
| N(4)-C(41)-C(44) | 113.7(3) | O(2)-C(45)-O(3) | 103.8(7) | O(4')-C(45')-O(2') | 127.8(11) |
| C(43)-C(41)-C(44) | 106.5(4) | O(4)-C(45)-Si(1) | 173.0(10) | O(3')-C(45')-O(2') | 104.3(8) |
| C(42)-C(41)-C(44) | 107.6(4) | O(2)-C(45)-Si(1) | 55.2(4) | O(4')-C(45')-Si(1) | 176.4(9) |
| C(45)-O(2)-Si(1) | 87.6(5) | O(3)-C(45)-Si(1) | 48.7(3) | O(3')-C(45')-Si(1) | 55.1(5) |
| C(45)-O(3)-Si(1) | 91.7(6) | C(45')-O(2')-Si(1) | 95.5(6) | O(2')-C(45')-Si(1) | 49.4(4) |
| O(4)-C(45)-O(2) | 131.7(10) | C(45')-O(3')-Si(1) | 90.4(6) | | |

## 10. PhC(N$^t$Bu)$_2$Si(O)OAl(Cl)DDP 10

### 10.1. Synthese PhC(N$^t$Bu)$_2$Si(O)OAl(Cl)DDP **10**




**6** **10**

Dipp = 2,6-$^i$Pr$_2$C$_6$H$_3$

Es wurden 20 mg PhC(N$^t$Bu)$_2$SiAl(Cl)DDP **6** (0.026 mmol) in 0.5 mL $C_6D_6$ gelöst, die Lösung eingefroren, die Atmosphäre im Vakuum entfernt und anschließend mit Distickstoffoxid versetzt. Dabei entfärbte sich die zunächst rote Lösung unter Gasentwicklung. Die Bildung von nur einem Produkt konnte im $^1$H-NMR-Spektrum beobachtet werden, die analog zu denen von **11** sind.

**$^1$H NMR** ($C_6D_6$, 400 MHz): $\delta$ 7.30-6.77 (m, 11 H, $C_6H_3$($^i$Pr)$_2$ & $C_6H_5$), 4.97 (s, 1 H, $\gamma$-C*H*), 3.58 (d sept, $^3J_{HH}$ = 6.7 Hz, 4 H, -C*H*(CH$_3$)$_2$), 2.01 (d, $^3J_{HH}$ = 6.7 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.55 (d, $^3J_{HH}$ = 6.5 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.55 (s, 6 H, ArNCC*H$_3$*), 1.21 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.10 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H$_3$*)$_2$), 0.81 (s, 18 H, C(C*H$_3$*)$_3$) ppm.

**$^{13}$C NMR** ($C_6D_6$, 101 MHz): $\delta$ 175.0 (NCN), 171.8 (ArNCCH$_3$), 145.2, 145.1, 140.0 (*C*$_6$H$_3$), 131.4, 129.9, 128.5, 128.3, 128.1, 125.3, 125.0, 98.3 ($\gamma$-*C*H-), 53.3 (*C*(CH$_3$)$_3$), 31.0 (C(*C*H$_3$)$_3$), 29.8, 28.0 (*C*H(CH$_3$)$_2$), 26.9, 25.5, 24.9, 24.6 (CH(*C*H$_3$)$_2$), 23.7 (ArNC*C*H$_3$) ppm. **$^{29}$Si NMR** ($C_6D_6$, 79 MHz): $\delta$ –76.8 ppm

## 10.2. Spektren PhC(N$^t$Bu)$_2$Si(O)OAl(Cl)DDP **10**

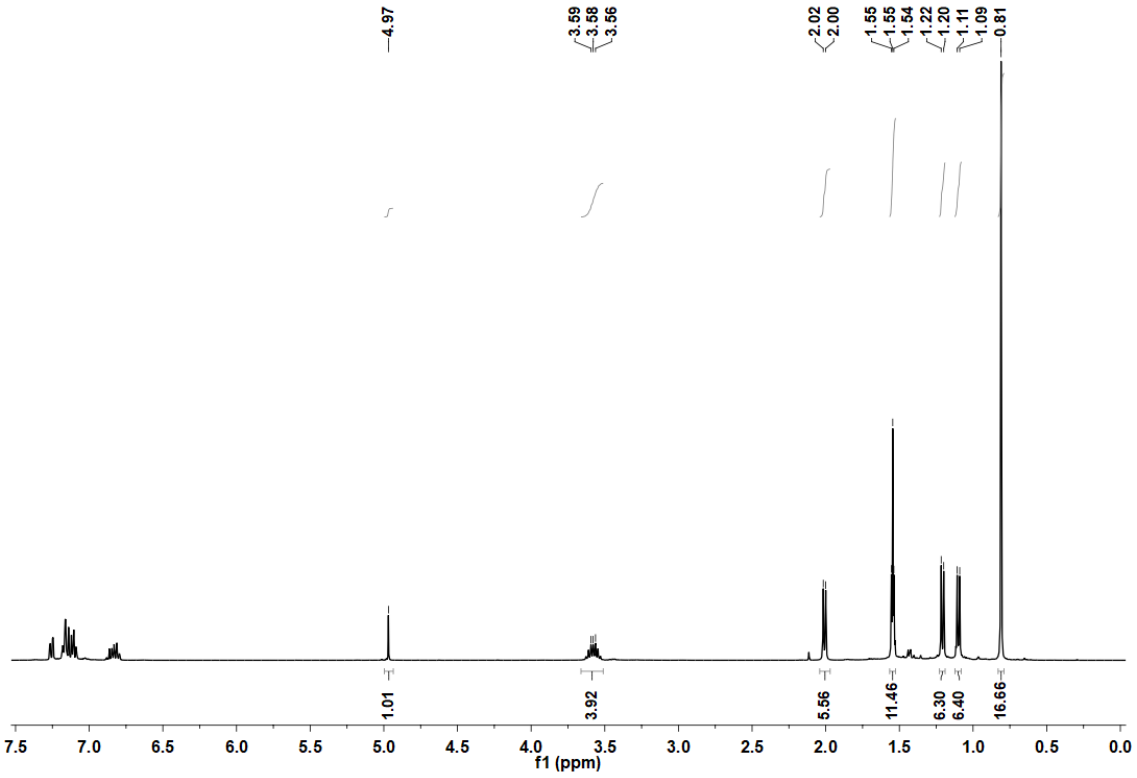



Abbildung 41: $^1$H-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(O)OAl(Cl)DDP **10** in $C_6D_6$.

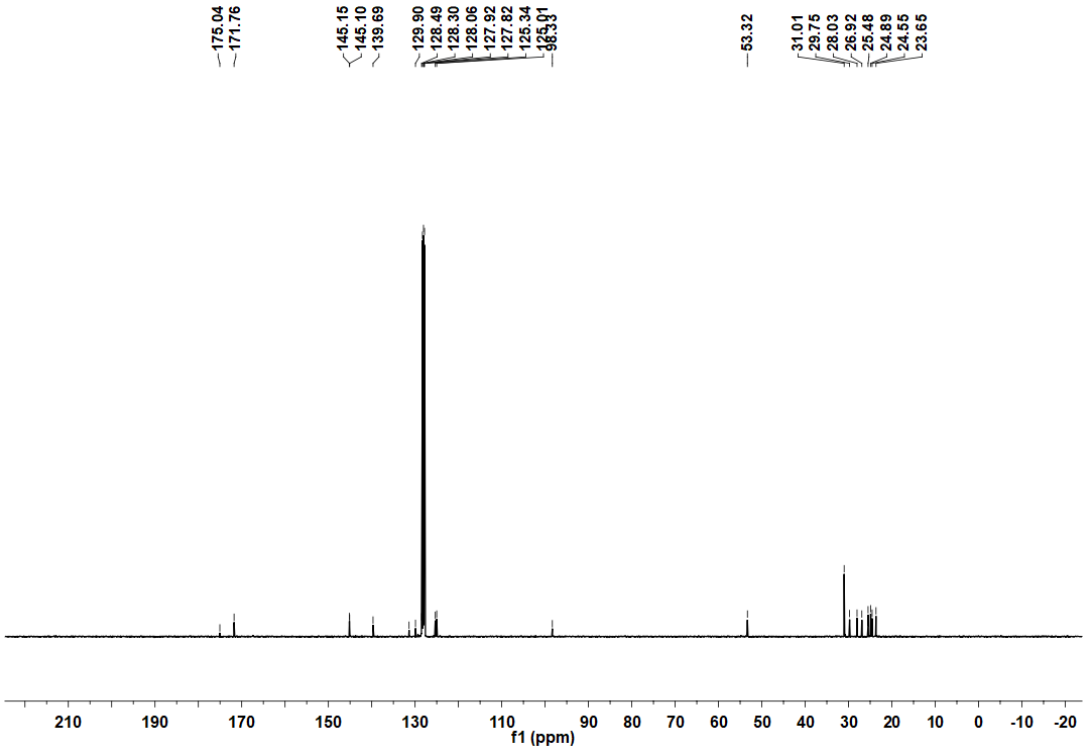



Abbildung 42: $^{13}$C-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(O)OAl(Cl)DDP **10** in $C_6D_6$.

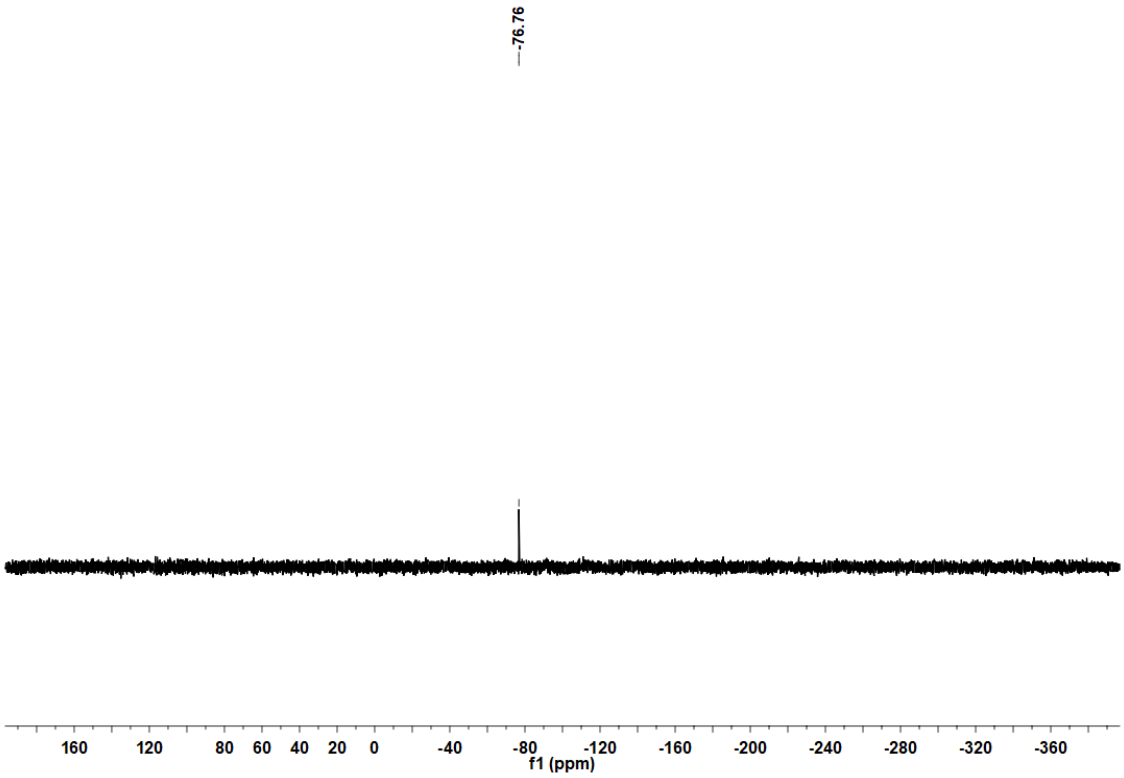
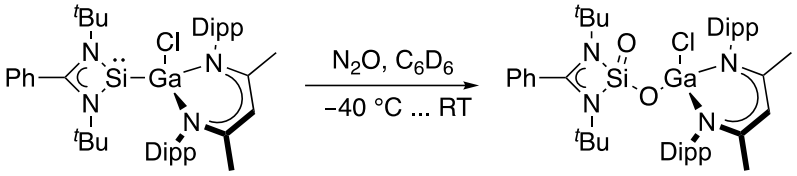



Abbildung 43: $^{29}$Si-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(O)OAl(Cl)DDP **10** in $C_6D_6$.

## 11. PhC(N$^t$Bu)$_2$Si(O)OGa(Cl)DDP 11

### 11.1. Synthese PhC(N$^t$Bu)$_2$Si(O)OGa(Cl)DDP **11**




**7** **11**

Dipp = 2,6-$^i$Pr$_2$C$_6$H$_3$

Es wurden 20 mg PhC(N$^t$Bu)$_2$SiGa(Cl)DDP **7** (0.026 mmol) in 0.5 mL $C_6D_6$ gelöst, die Lösung eingefroren, die Atmosphäre im Vakuum entfernt und anschließend mit Distickstoffoxid versetzt. Dabei entfärbte sich die zunächst gelbe Lösung unter Gasentwicklung. Die Lösung wurde auf etwa ein Fünftel eingeengt und bei −18 °C gelagert, wobei PhC(N$^t$Bu)$_2$Si(O)OGa(Cl)DDP **11** in Form von farblosen Kristallen erhalten werden konnte.

**Ausbeute** 9.5 mg (0.0117 mmol, 45 %).

**Smp.** 198 °C.

**Elementaranalyse** von $C_{44}H_{68}ClGaN_4O_2Si$: gefunden (berechnet) C 64.2 (64.58), H 8.13 (8.38), N 7.78 (6.85)%.

**IR**: ν 3636, 3051, 2956, 2915, 2854, 1613, 1528, 1436,1379, 1357, 1306, 1261, 1199, 1143, 1085, 993, 978, 938, 871, 831, 795, 755, 706, 635, 526, 490 cm$^{-1}$.

**$^1$H NMR** ($C_6D_6$, 400 MHz): δ 7.26-6.79 (m, 11 H, $C_6H_3$($^i$Pr)$_2$ & $C_6H_5$), 4.83 (s, 1 H, γ-C*H*), 3.53 (d sept, $^3J_{HH}$ = 6.7 Hz, 4 H, -C*H*(CH$_3$)$_2$), 1.97 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.56 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.54 (s, 6 H, ArNCC*H$_3$*), 1.18 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H$_3$*)$_2$), 1.09 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H$_3$*)$_2$), 0.82 (s, 18 H, C(C*H$_3$*)$_3$) ppm.

**$^{13}$C NMR** ($C_6D_6$, 101 MHz): δ 174.8 (NCN), 171.4 (ArNCCH$_3$), 145.3, 145.1, 140.0 (*C*$_6$H$_3$), 131.8, 130.1, 128.7, 128.4, 128.2, 125.5, 125.2, 96.8 (γ-*C*H-), 53.6 (*C*(CH$_3$)$_3$), 31.3 (C(*C*H$_3$)$_3$), 29.9, 28.3 (*C*H(CH$_3$)$_2$), 26.7, 25.8, 25.2, 24.8 (CH(*C*H$_3$)$_2$), 24.0 (ArNC*C*H$_3$) ppm.

**$^{29}$Si NMR** ($C_6D_6$, 59 MHz): δ –74.7 ppm

## 11.2. Spektren PhC(N$^t$Bu)$_2$Si(O)OGa(Cl)DDP **11**

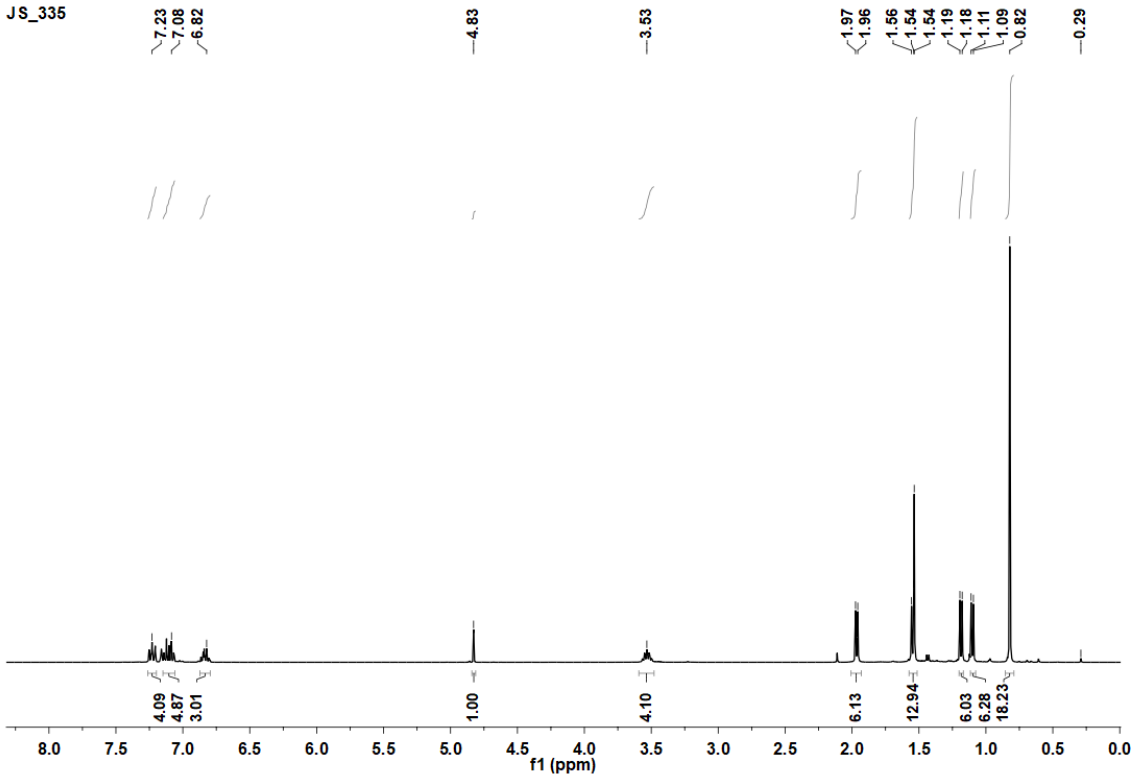



Abbildung 44: $^1$H-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(O)OGa(Cl)DDP **11** in $C_6D_6$.

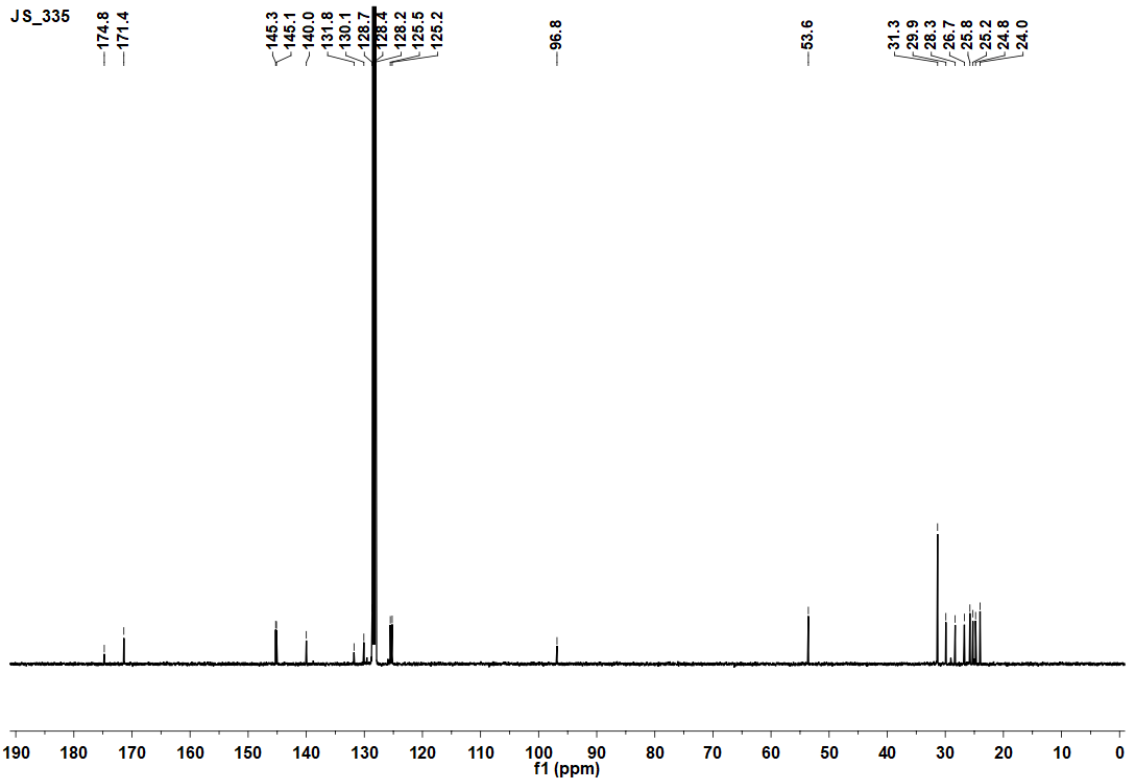



Abbildung 45: $^{13}$C-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(O)OGa(Cl)DDP **11** in $C_6D_6$.

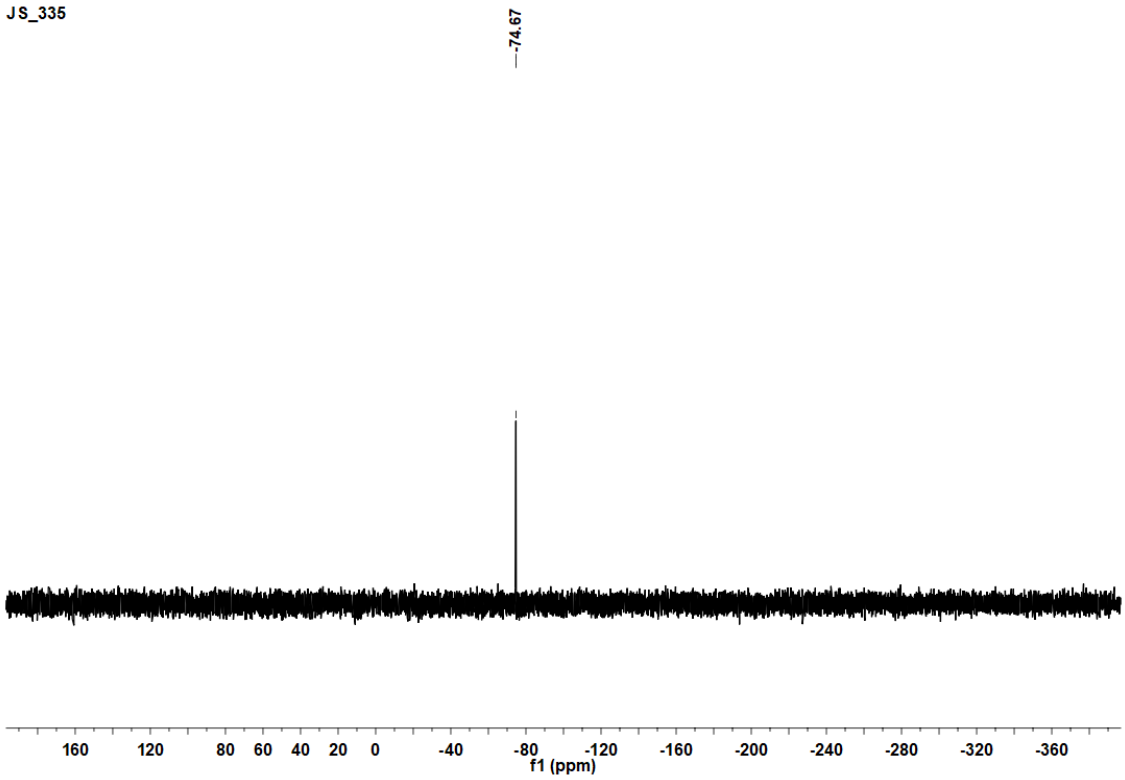



Abbildung 46: $^{29}$Si-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(O)OGa(Cl)DDP **11** in $C_6D_6$.

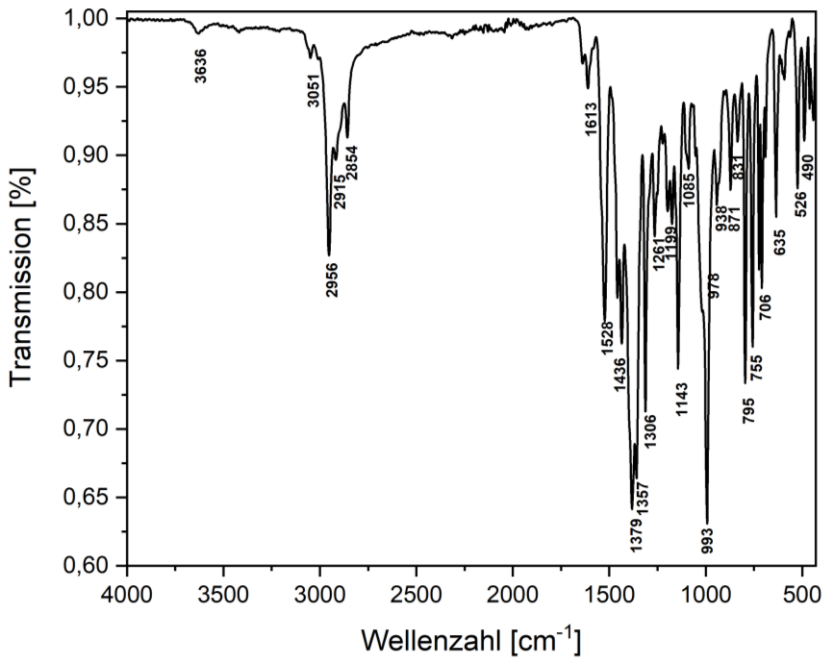



Abbildung 47: ATR-IR Spektrum von PhC(N$^t$Bu)$_2$Si(O)OGa(Cl)DDP **11**.

## 11.3. Kristallografische Daten PhC($N^tBu$)$_2$Si(O)OGa(Cl)DDP **11**

Tabelle 41: Crystal structure data

| **Identification code** | **jus_335m** |
|---|---|
| Empirical formula | C47.50 H68 Cl Ga N4 O2 Si |
| Formula weight | 860.31 |
| Density (calculated) | 1.218 g·cm$^{-3}$ |
| *F*(000) | 1836 |
| Temperature | 100(2) K |
| Crystal size | 0.113 × 0.095 × 0.068 mm |
| Crystal colour | colourless |
| Crystal description | tablet |
| Wavelength | 0.71073 Å |
| Crystal system | monoclinic |
| Space group | *P 21/c* |
| Unit cell dimensions | |
| *a* [Å] | 10.4441(15) |
| *b* [Å] | 23.436(3) |
| *c* [Å] | 19.593(3) |
| *α* [°] | 90 |
| *β* [°] | 102.062(3) |
| *γ* [°] | 90 |
| Volume | 4689.8(11) Å$^3$ |
| *Z* | 4 |
| Cell measurement reflections used | 9397 |
| Cell measurement *θ* min/max | 2.23°/26.50° |
| Diffractometer control software | BRUKER APEX3(v2019.1-0) |
| Diffractometer measurement device | Bruker D8 KAPPA II (APEX II detector) |
| Diffractometer measurement method | Data collection strategy APEX 3/QUEEN |
| *θ* range for data collection | 1.738°- 30.653° |
| Completeness to *θ* = 25.242° | 100.0% |
| Completeness to $\theta_{max}$ = 30.653° | 99.4% |
| Index ranges | -14 ≤ *h* ≤ 14 |
| | -33 ≤ *k* ≤ 33 |
| | -28 ≤ *l* ≤ 25 |
| Computing data reduction | BRUKER APEX3(v2019.1-0) |
| Absorption coefficient | 0.710 mm$^{-1}$ |
| Absorption correction | Semi-empirical from equivalents |
| Computation absorption correction | SADABS |
| Max./min. Transmission | 0.75/0.69 |
| $R_{merg}$ before/after correction | 0.0707/0.0648 |
| Computing structure solution | BRUKER APEX3(v2019.1-0) |
| Computing structure refinement | SHELXL-2017/1 (Sheldrick, 2017) |

| | |
|---|---|
| Refinement method | Full-matrix least-squares on $F^2$ |
| Reflections collected | 102299 |
| Independent reflections | 14406 |
| $R_{int}$ | 0.1029 |
| Reflections with $I > 2\sigma(I)$ | 10314 |
| Restraints | 76 |
| Parameter | 558 |
| GooF | 1.016 |
| Weighting details | $w = 1/[\sigma^2(F_{obs}^2) + (0.0313P)^2 + 2.8680P]$ |
| | where $P = (F_{obs}^2 + 2F_{calc}^2)/3$ |
| $R_1$ [$I > 2\sigma(I)$] | 0.0463 |
| $wR_2$ [$I > 2\sigma(I)$] | 0.0890 |
| $R_1$ [all data] | 0.0800 |
| $wR_2$ [all data] | 0.1006 |
| Absolute structure parameter | |
| Largest diff. peak and hole | 0.478/-0.521 |

Tabelle 42: Atomic coordinates ( × $10^4$) and equivalent isotropic displacement parameters (Å$^2$ × $10^3$) for jus_335m. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | *x* | *y* | *z* | $U_{eq}$ | | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|---|---|---|---|---|
| Ga(1) | 946(1) | 7089(1) | 3242(1) | 12(1) | H(14A) | 1897 | 5036 | 2302 | 51 |
| Cl(1) | 2895(1) | 6757(1) | 3236(1) | 20(1) | H(14B) | 2329 | 5574 | 2800 | 51 |
| Si(1) | 958(1) | 6767(1) | 4863(1) | 13(1) | H(14C) | 2870 | 5503 | 2098 | 51 |
| O(1) | 513(1) | 6965(1) | 4066(1) | 16(1) | C(15) | -2559(2) | 6653(1) | 3082(1) | 24(1) |
| O(2) | -74(1) | 6724(1) | 5314(1) | 20(1) | H(15) | -1952 | 6986 | 3112 | 29 |
| N(1) | -287(2) | 6773(1) | 2461(1) | 14(1) | C(16) | -3810(3) | 6805(2) | 2554(1) | 51(1) |
| N(2) | 820(2) | 7874(1) | 2971(1) | 12(1) | H(16A) | -3593 | 6893 | 2102 | 76 |
| N(3) | 2193(2) | 6200(1) | 4947(1) | 16(1) | H(16B) | -4223 | 7138 | 2720 | 76 |
| N(4) | 2562(2) | 7075(1) | 5260(1) | 15(1) | H(16C) | -4416 | 6481 | 2500 | 76 |
| C(1) | -659(2) | 7076(1) | 1878(1) | 16(1) | C(17) | -2862(3) | 6566(1) | 3803(1) | 33(1) |
| C(2) | -270(2) | 7638(1) | 1793(1) | 15(1) | H(17A) | -3234 | 6917 | 3951 | 49 |
| H(2) | -459 | 7780 | 1328 | 18 | H(17B) | -2053 | 6471 | 4137 | 49 |
| C(3) | 360(2) | 8018(1) | 2301(1) | 14(1) | H(17C) | -3492 | 6254 | 3784 | 49 |
| C(4) | -1563(2) | 6815(1) | 1256(1) | 25(1) | C(18) | 1169(2) | 8326(1) | 3480(1) | 13(1) |
| H(4A) | -2116 | 7114 | 995 | 37 | C(19) | 2442(2) | 8550(1) | 3626(1) | 16(1) |
| H(4B) | -2117 | 6528 | 1415 | 37 | C(20) | 2706(2) | 9003(1) | 4096(1) | 21(1) |
| H(4C) | -1044 | 6634 | 953 | 37 | H(20) | 3562 | 9161 | 4203 | 25 |
| C(5) | 506(2) | 8622(1) | 2070(1) | 18(1) | C(21) | 1751(2) | 9226(1) | 4409(1) | 21(1) |
| H(5A) | 287 | 8641 | 1559 | 27 | H(21) | 1947 | 9539 | 4721 | 26 |
| H(5B) | 1411 | 8748 | 2238 | 27 | C(22) | 510(2) | 8993(1) | 4268(1) | 19(1) |
| H(5C) | -86 | 8871 | 2261 | 27 | H(22) | -142 | 9148 | 4488 | 23 |
| C(6) | -810(2) | 6206(1) | 2514(1) | 18(1) | C(23) | 197(2) | 8535(1) | 3810(1) | 15(1) |
| C(7) | -225(2) | 5736(1) | 2258(1) | 21(1) | C(24) | 3518(2) | 8321(1) | 3288(1) | 17(1) |
| C(8) | -759(3) | 5196(1) | 2325(1) | 30(1) | H(24) | 3119 | 8033 | 2930 | 20 |
| H(8) | -393 | 4871 | 2148 | 36 | C(25) | 4143(2) | 8787(1) | 2919(1) | 24(1) |
| C(9) | -1805(3) | 5129(1) | 2643(1) | 39(1) | H(25A) | 4785 | 8615 | 2681 | 37 |
| H(9) | -2144 | 4759 | 2691 | 46 | H(25B) | 4581 | 9065 | 3263 | 37 |
| C(10) | -2368(2) | 5598(1) | 2894(1) | 33(1) | H(25C) | 3462 | 8979 | 2576 | 37 |
| H(10) | -3091 | 5545 | 3113 | 39 | C(26) | 4576(2) | 8021(1) | 3824(1) | 23(1) |
| C(11) | -1888(2) | 6146(1) | 2830(1) | 21(1) | H(26A) | 5223 | 7852 | 3587 | 34 |
| C(12) | 942(2) | 5780(1) | 1912(1) | 25(1) | H(26B) | 4176 | 7721 | 4058 | 34 |
| H(12) | 1201 | 6191 | 1911 | 30 | H(26C) | 5009 | 8299 | 4170 | 34 |
| C(13) | 609(3) | 5573(1) | 1154(1) | 39(1) | C(27) | -1171(2) | 8288(1) | 3675(1) | 23(1) |
| H(13A) | -136 | 5791 | 894 | 58 | H(27) | -1167 | 7929 | 3398 | 27 |
| H(13B) | 380 | 5167 | 1144 | 58 | C(28) | -2146(3) | 8700(2) | 3239(2) | 62(1) |
| H(13C) | 1368 | 5628 | 940 | 58 | H(28A) | -1862 | 8790 | 2804 | 93 |
| C(14) | 2115(3) | 5443(1) | 2314(1) | 34(1) | H(28B) | -2185 | 9053 | 3503 | 93 |
| H(28C) | -3015 | 8524 | 3129 | 93 | H(40C) | 3563 | 5737 | 4104 | 40 |
| C(29) | -1608(2) | 8134(1) | 4344(1) | 26(1) | C(41) | 2957(2) | 7550(1) | 5765(1) | 17(1) |
| H(29A) | -2480 | 7961 | 4230 | 40 | C(42) | 1829(2) | 7975(1) | 5609(1) | 26(1) |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| H(29B) | -1638 | 8480 | 4621 | 40 | H(42A) | 1696 | 8097 | 5120 | 39 |
| H(29C) | -986 | 7863 | 4614 | 40 | H(42B) | 2036 | 8307 | 5915 | 39 |
| C(30) | 3115(2) | 6557(1) | 5260(1) | 15(1) | H(42C) | 1029 | 7793 | 5689 | 39 |
| C(31) | 4505(2) | 6414(1) | 5561(1) | 18(1) | C(43) | 4203(2) | 7844(1) | 5668(1) | 31(1) |
| C(32) | 4809(2) | 6086(1) | 6165(1) | 22(1) | H(43A) | 4931 | 7572 | 5760 | 46 |
| H(32) | 4133 | 5956 | 6384 | 27 | H(43B) | 4397 | 8166 | 5993 | 46 |
| C(33) | 6104(2) | 5951(1) | 6446(1) | 29(1) | H(43C) | 4085 | 7985 | 5188 | 46 |
| H(33) | 6317 | 5727 | 6858 | 35 | C(44) | 3118(2) | 7333(1) | 6511(1) | 22(1) |
| C(34) | 7079(2) | 6141(1) | 6128(1) | 33(1) | H(44A) | 2361 | 7098 | 6550 | 32 |
| H(34) | 7963 | 6046 | 6324 | 40 | H(44B) | 3177 | 7658 | 6831 | 32 |
| C(35) | 6794(2) | 6468(1) | 5529(1) | 30(1) | H(44C) | 3918 | 7104 | 6633 | 32 |
| H(35) | 7476 | 6598 | 5315 | 36 | C11 | 4787(7) | 10186(2) | 4671(4) | 67(2) |
| C(36) | 5495(2) | 6605(1) | 5243(1) | 25(1) | C21 | 4182(19) | 10218(9) | 5224(8) | 109(5) |
| H(36) | 5287 | 6830 | 4831 | 30 | H21 | 3480 | 10478 | 5214 | 131 |
| C(37) | 2250(2) | 5591(1) | 4741(1) | 20(1) | C31 | 4592(10) | 9873(4) | 5799(5) | 101(3) |
| C(38) | 1034(2) | 5499(1) | 4162(1) | 26(1) | H31 | 4186 | 9901 | 6188 | 121 |
| H(38A) | 1084 | 5745 | 3764 | 38 | C41 | 5589(17) | 9487(7) | 5806(7) | 134(5) |
| H(38B) | 248 | 5594 | 4338 | 38 | H41 | 5839 | 9240 | 6196 | 161 |
| H(38C) | 992 | 5099 | 4014 | 38 | C51 | 6225(8) | 9451(3) | 5266(5) | 80(2) |
| C(39) | 2158(3) | 5210(1) | 5359(1) | 33(1) | H51 | 6940 | 9197 | 5285 | 96 |
| H(39A) | 2072 | 4811 | 5206 | 50 | C61 | 5797(18) | 9797(7) | 4688(8) | 91(4) |
| H(39B) | 1391 | 5319 | 5544 | 50 | H61 | 6201 | 9766 | 4298 | 109 |
| H(39C) | 2951 | 5254 | 5724 | 50 | C71 | 4359(18) | 10502(8) | 4107(8) | 194(10) |
| C(40) | 3475(2) | 5459(1) | 4464(1) | 27(1) | H7A1 | 3934 | 10846 | 4238 | 232 |
| H(40A) | 3406 | 5074 | 4264 | 40 | H7B1 | 3727 | 10282 | 3768 | 232 |
| H(40B) | 4245 | 5480 | 4848 | 40 | H7C1 | 5099 | 10611 | 3899 | 232 |

Tabelle 43: Anisotropic displacement parameters ($Å^2 \times 10^3$) for jus_335m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ga(1) | 12(1) | 12(1) | 10(1) | 0(1) | 0(1) | -1(1) | C(22) | 23(1) | 17(1) | 16(1) | 1(1) | 3(1) | 5(1) |
| Cl(1) | 16(1) | 19(1) | 26(1) | 2(1) | 5(1) | 3(1) | C(23) | 13(1) | 18(1) | 13(1) | 3(1) | -1(1) | 3(1) |
| Si(1) | 12(1) | 17(1) | 10(1) | 1(1) | 0(1) | 0(1) | C(24) | 12(1) | 20(1) | 18(1) | -1(1) | 1(1) | -1(1) |
| O(1) | 16(1) | 21(1) | 12(1) | 2(1) | 1(1) | 0(1) | C(25) | 19(1) | 28(1) | 27(1) | 4(1) | 6(1) | -2(1) |
| O(2) | 17(1) | 30(1) | 13(1) | 2(1) | 3(1) | 1(1) | C(26) | 18(1) | 27(1) | 22(1) | -1(1) | 0(1) | 1(1) |
| N(1) | 18(1) | 13(1) | 11(1) | -1(1) | 0(1) | -3(1) | C(27) | 14(1) | 35(1) | 19(1) | -3(1) | 3(1) | -2(1) |
| N(2) | 12(1) | 13(1) | 12(1) | 0(1) | 0(1) | -2(1) | C(28) | 15(1) | 118(3) | 49(2) | 51(2) | 0(1) | 4(2) |
| N(3) | 15(1) | 16(1) | 14(1) | 0(1) | 0(1) | 1(1) | C(29) | 19(1) | 35(1) | 25(1) | 7(1) | 3(1) | -2(1) |
| N(4) | 14(1) | 18(1) | 13(1) | -2(1) | -1(1) | 0(1) | C(30) | 14(1) | 19(1) | 10(1) | 1(1) | 1(1) | 1(1) |
| C(1) | 16(1) | 18(1) | 12(1) | -1(1) | 1(1) | -2(1) | C(31) | 14(1) | 22(1) | 17(1) | -4(1) | -1(1) | 2(1) |
| C(2) | 15(1) | 18(1) | 11(1) | 2(1) | -1(1) | -1(1) | C(32) | 22(1) | 25(1) | 18(1) | -4(1) | -1(1) | 6(1) |
| C(3) | 11(1) | 15(1) | 15(1) | 1(1) | 2(1) | 1(1) | C(33) | 28(1) | 32(1) | 22(1) | -7(1) | -8(1) | 10(1) |
| C(4) | 31(1) | 25(1) | 13(1) | -1(1) | -4(1) | -8(1) | C(34) | 19(1) | 42(1) | 33(1) | -17(1) | -9(1) | 6(1) |
| C(5) | 19(1) | 16(1) | 18(1) | 3(1) | 0(1) | -2(1) | C(35) | 14(1) | 44(1) | 32(1) | -15(1) | 5(1) | -2(1) |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| C(6) | 21(1) | 17(1) | 13(1) | 1(1) | -2(1) | -6(1) | C(36) | 18(1) | 33(1) | 22(1) | -4(1) | 3(1) | -1(1) |
| C(7) | 29(1) | 17(1) | 15(1) | 0(1) | 1(1) | -5(1) | C(37) | 24(1) | 15(1) | 19(1) | -1(1) | 0(1) | 0(1) |
| C(8) | 42(1) | 16(1) | 32(1) | 0(1) | 6(1) | -6(1) | C(38) | 26(1) | 21(1) | 26(1) | -5(1) | -3(1) | -2(1) |
| C(9) | 44(2) | 19(1) | 53(2) | 1(1) | 11(1) | -15(1) | C(39) | 48(2) | 22(1) | 26(1) | 7(1) | 1(1) | -7(1) |
| C(10) | 29(1) | 29(1) | 40(1) | 6(1) | 7(1) | -10(1) | C(40) | 27(1) | 22(1) | 29(1) | -6(1) | 2(1) | 6(1) |
| C(11) | 20(1) | 23(1) | 19(1) | 3(1) | 0(1) | -6(1) | C(41) | 20(1) | 17(1) | 12(1) | -1(1) | 1(1) | -1(1) |
| C(12) | 38(1) | 17(1) | 20(1) | -4(1) | 9(1) | -2(1) | C(42) | 33(1) | 20(1) | 22(1) | -4(1) | -4(1) | 6(1) |
| C(13) | 66(2) | 28(1) | 24(1) | -7(1) | 14(1) | -4(1) | C(43) | 33(1) | 32(1) | 29(1) | -10(1) | 10(1) | -14(1) |
| C(14) | 40(2) | 28(1) | 38(1) | -5(1) | 13(1) | 4(1) | C(44) | 24(1) | 24(1) | 15(1) | -2(1) | 1(1) | 4(1) |
| C(15) | 18(1) | 26(1) | 28(1) | 3(1) | 5(1) | -3(1) | C11 | 69(5) | 28(3) | 77(5) | 13(3) | -45(3) | -31(3) |
| C(16) | 44(2) | 78(2) | 28(1) | 6(1) | 3(1) | 31(2) | C21 | 66(10) | 123(14) | 126(9) | -62(8) | -9(7) | -58(8) |
| C(17) | 32(1) | 41(1) | 26(1) | 2(1) | 7(1) | -1(1) | C31 | 94(7) | 106(8) | 112(7) | -56(6) | 44(6) | -82(6) |
| C(18) | 15(1) | 12(1) | 11(1) | 1(1) | -1(1) | 1(1) | C41 | 134(13) | 80(11) | 188(13) | -46(9) | 32(10) | -63(7) |
| C(19) | 17(1) | 14(1) | 17(1) | 1(1) | 1(1) | -1(1) | C51 | 59(5) | 59(5) | 99(6) | -8(4) | -34(4) | -24(4) |
| C(20) | 19(1) | 21(1) | 21(1) | -3(1) | 1(1) | -6(1) | C61 | 61(9) | 41(6) | 145(11) | -11(7) | -36(7) | -22(5) |
| C(21) | 29(1) | 16(1) | 19(1) | -4(1) | 6(1) | -3(1) | C71 | 240(20) | 90(12) | 170(11) | 69(11) | -146(11) | -90(11) |

Tabelle 44: Bond lengths [Å] for jus_335m.

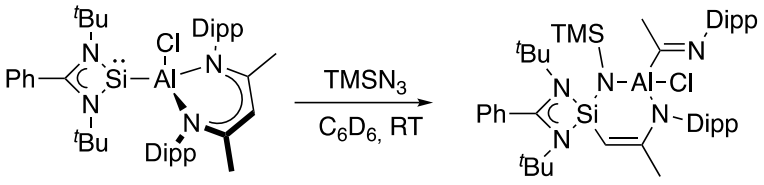
| | | | | | |
|---|---|---|---|---|---|
| Ga(1)-O(1) | 1.7886(13) | C(6)-C(7) | 1.403(3) | C(27)-C(28) | 1.529(3) |
| Ga(1)-N(2) | 1.9120(15) | C(7)-C(8) | 1.399(3) | C(30)-C(31) | 1.487(3) |
| Ga(1)-N(1) | 1.9303(15) | C(7)-C(12) | 1.517(3) | C(31)-C(36) | 1.387(3) |
| Ga(1)-Cl(1) | 2.1816(6) | C(8)-C(9) | 1.374(4) | C(31)-C(32) | 1.390(3) |
| Si(1)-O(2) | 1.5331(15) | C(9)-C(10) | 1.384(4) | C(32)-C(33) | 1.386(3) |
| Si(1)-O(1) | 1.6018(14) | C(10)-C(11) | 1.395(3) | C(33)-C(34) | 1.374(4) |
| Si(1)-N(3) | 1.8350(17) | C(11)-C(15) | 1.512(3) | C(34)-C(35) | 1.381(4) |
| Si(1)-N(4) | 1.8402(17) | C(12)-C(14) | 1.531(3) | C(35)-C(36) | 1.393(3) |
| Si(1)-C(30) | 2.278(2) | C(12)-C(13) | 1.532(3) | C(37)-C(40) | 1.522(3) |
| N(1)-C(1) | 1.333(2) | C(15)-C(17) | 1.525(3) | C(37)-C(39) | 1.525(3) |
| N(1)-C(6) | 1.448(2) | C(15)-C(16) | 1.529(3) | C(37)-C(38) | 1.531(3) |
| N(2)-C(3) | 1.342(2) | C(18)-C(23) | 1.402(3) | C(41)-C(43) | 1.519(3) |
| N(2)-C(18) | 1.448(2) | C(18)-C(19) | 1.402(3) | C(41)-C(42) | 1.523(3) |
| N(3)-C(30) | 1.325(2) | C(19)-C(20) | 1.395(3) | C(41)-C(44) | 1.525(3) |
| N(3)-C(37) | 1.487(2) | C(19)-C(24) | 1.516(3) | C11-C71 | 1.327(14) |
| N(4)-C(30) | 1.343(2) | C(20)-C(21) | 1.378(3) | C11-C21 | 1.367(11) |
| N(4)-C(41) | 1.490(2) | C(21)-C(22) | 1.379(3) | C11-C61 | 1.390(10) |
| C(1)-C(2) | 1.399(3) | C(22)-C(23) | 1.393(3) | C21-C31 | 1.379(11) |
| C(1)-C(4) | 1.507(3) | C(23)-C(27) | 1.514(3) | C31-C41 | 1.377(12) |
| C(2)-C(3) | 1.394(3) | C(24)-C(26) | 1.526(3) | C41-C51 | 1.365(11) |
| C(3)-C(5) | 1.506(3) | C(24)-C(25) | 1.529(3) | C51-C61 | 1.388(10) |
| C(6)-C(11) | 1.402(3) | C(27)-C(29) | 1.519(3) | | |

Tabelle 45: Bond angles [°] for jus_335m.

| | | | | | |
|---|---|---|---|---|---|
| O(1)-Ga(1)-N(2) | 113.03(6) | C(11)-C(6)-C(7) | 121.97(18) | N(3)-C(30)-N(4) | 107.43(16) |
| O(1)-Ga(1)-N(1) | 113.60(7) | C(11)-C(6)-N(1) | 118.26(18) | N(3)-C(30)-C(31) | 126.54(17) |
| N(2)-Ga(1)-N(1) | 98.79(6) | C(7)-C(6)-N(1) | 119.76(18) | N(4)-C(30)-C(31) | 126.03(17) |
| O(1)-Ga(1)-Cl(1) | 111.08(5) | C(8)-C(7)-C(6) | 117.7(2) | N(3)-C(30)-Si(1) | 53.66(10) |
| N(2)-Ga(1)-Cl(1) | 110.63(5) | C(8)-C(7)-C(12) | 118.6(2) | N(4)-C(30)-Si(1) | 53.89(9) |
| N(1)-Ga(1)-Cl(1) | 109.08(5) | C(6)-C(7)-C(12) | 123.74(18) | C(31)-C(30)-Si(1) | 176.58(14) |
| O(2)-Si(1)-O(1) | 119.02(8) | C(9)-C(8)-C(7) | 121.0(2) | C(36)-C(31)-C(32) | 120.06(19) |
| O(2)-Si(1)-N(3) | 117.74(8) | C(8)-C(9)-C(10) | 120.5(2) | C(36)-C(31)-C(30) | 120.43(18) |
| O(1)-Si(1)-N(3) | 110.64(7) | C(9)-C(10)-C(11) | 120.8(2) | C(32)-C(31)-C(30) | 119.50(19) |
| O(2)-Si(1)-N(4) | 118.00(8) | C(10)-C(11)-C(6) | 117.9(2) | C(33)-C(32)-C(31) | 119.7(2) |
| O(1)-Si(1)-N(4) | 110.64(8) | C(10)-C(11)-C(15) | 119.7(2) | C(34)-C(33)-C(32) | 119.9(2) |
| N(3)-Si(1)-N(4) | 71.65(7) | C(6)-C(11)-C(15) | 122.37(18) | C(33)-C(34)-C(35) | 121.1(2) |
| O(2)-Si(1)-C(30) | 123.58(7) | C(7)-C(12)-C(14) | 111.44(18) | C(34)-C(35)-C(36) | 119.2(2) |
| O(1)-Si(1)-C(30) | 117.40(7) | C(7)-C(12)-C(13) | 111.7(2) | C(31)-C(36)-C(35) | 120.0(2) |
| N(3)-Si(1)-C(30) | 35.58(7) | C(14)-C(12)-C(13) | 109.26(19) | N(3)-C(37)-C(40) | 112.17(17) |
| N(4)-Si(1)-C(30) | 36.13(7) | C(11)-C(15)-C(17) | 113.32(18) | N(3)-C(37)-C(39) | 109.52(17) |
| Si(1)-O(1)-Ga(1) | 148.20(9) | C(11)-C(15)-C(16) | 110.6(2) | C(40)-C(37)-C(39) | 111.09(19) |
| C(1)-N(1)-C(6) | 120.21(16) | C(17)-C(15)-C(16) | 110.2(2) | N(3)-C(37)-C(38) | 105.24(16) |
| C(1)-N(1)-Ga(1) | 120.13(13) | C(23)-C(18)-C(19) | 121.51(17) | C(40)-C(37)-C(38) | 109.66(18) |
| C(6)-N(1)-Ga(1) | 119.66(12) | C(23)-C(18)-N(2) | 118.10(16) | C(39)-C(37)-C(38) | 108.96(18) |
| C(3)-N(2)-C(18) | 118.43(15) | C(19)-C(18)-N(2) | 120.39(17) | N(4)-C(41)-C(43) | 112.47(16) |
| C(3)-N(2)-Ga(1) | 120.18(12) | C(20)-C(19)-C(18) | 117.85(18) | N(4)-C(41)-C(42) | 105.06(15) |
| C(18)-N(2)-Ga(1) | 121.36(12) | C(20)-C(19)-C(24) | 119.68(18) | C(43)-C(41)-C(42) | 109.00(18) |
| C(30)-N(3)-C(37) | 131.66(17) | C(18)-C(19)-C(24) | 122.48(17) | N(4)-C(41)-C(44) | 110.26(16) |
| C(30)-N(3)-Si(1) | 90.76(12) | C(21)-C(20)-C(19) | 121.39(19) | C(43)-C(41)-C(44) | 110.26(17) |
| C(37)-N(3)-Si(1) | 137.53(13) | C(20)-C(21)-C(22) | 119.90(18) | C(42)-C(41)-C(44) | 109.65(17) |
| C(30)-N(4)-C(41) | 128.07(16) | C(21)-C(22)-C(23) | 121.12(19) | C71-C11-C21 | 119.9(10) |
| C(30)-N(4)-Si(1) | 89.98(12) | C(22)-C(23)-C(18) | 118.17(18) | C71-C11-C61 | 120.6(10) |
| C(41)-N(4)-Si(1) | 132.45(13) | C(22)-C(23)-C(27) | 119.60(18) | C21-C11-C61 | 119.4(8) |
| N(1)-C(1)-C(2) | 124.36(17) | C(18)-C(23)-C(27) | 122.23(17) | C11-C21-C31 | 119.9(9) |
| N(1)-C(1)-C(4) | 119.89(17) | C(19)-C(24)-C(26) | 111.06(17) | C41-C31-C21 | 120.0(9) |
| C(2)-C(1)-C(4) | 115.74(17) | C(19)-C(24)-C(25) | 112.48(16) | C51-C41-C31 | 121.4(10) |
| C(3)-C(2)-C(1) | 128.38(17) | C(26)-C(24)-C(25) | 109.55(17) | C41-C51-C61 | 118.1(9) |
| N(2)-C(3)-C(2) | 123.92(17) | C(23)-C(27)-C(29) | 112.46(17) | C51-C61-C11 | 121.1(9) |
| N(2)-C(3)-C(5) | 119.54(16) | C(23)-C(27)-C(28) | 110.4(2) | C51-C61-C11 | 121.1(9) |
| C(2)-C(3)-C(5) | 116.54(16) | C(29)-C(27)-C(28) | 110.33(19) | | |

## 12. [PhC(N$^t$Bu)$_2$Si][N(TMS)Al(Cl)(C(Me)=NDipp)][CH=C(Me)NDipp] 12

### 12.1. Synthese [PhC(N$^t$Bu)$_2$Si][N(TMS)Al(Cl)(C(Me)=NDipp)][CH=C(Me)NDipp] **12**




**6** **12**

Dipp = 2,6-$^i$Pr$_2$C$_6$H$_3$

Es wurden 15 mg PhC(N$^t$Bu)$_2$SiAl(Cl)DDP **6** (0.020 mmol) in 0.5 mL Toluol gelöst, woraufhin 2.3 mg $TMSN_3$ (0.020 mmol, 2.65 μL) zur Lösung hinzugegeben wurde. Dabei entfärbte sich die zunächst rote Lösung unter Gasentwicklung. Die Lösung wurde für zwei Tage bei Raumtemperatur gerührt und daraufhin auf etwa die Hälfte eingeengt und bei –18 °C gelagert, wobei [PhC(N$^t$Bu)$_2$Si][N(TMS)Al(Cl)(C(Me)=NDipp)][CH=C(Me)NDipp] **12** in Form von farblosen Kristallen erhalten werden konnte.

**Ausbeute**: 8.3 mg (0.010 mmol, 50 %).

**Smp.** 261 °C.

**Elementaranalyse** von $C_{47}H_{74}AlClN_5Si_2$: gefunden (berechnet) C 67.8 (68.20), H 9.3 (9.01), N 8.22 (8.48)%.

**IR**: ν 2955, 2852, 1612, 1504, 1390, 1363, 1319, 1269, 1235, 1194, 1151, 1090, 1012, 954, 925, 859, 837, 816, 802, 787, 753, 730, 707, 693, 678, 631, 549, 547, 500, 465, 444, 424, 404 cm$^{-1}$.

**$^1$H NMR** ($C_6D_6$, 400 MHz): δ 7.28-7.08 (m, 5 H, $C_6H_3(^i$Pr)$_2$), 7.13 (m, 2 H, $C_6H_3(^i$Pr)$_2$), 7.03 (m, 2 H, $C_6H_5$), 6.91 (m, 1 H, $C_6H_5$), 6.82 (m, 2 H, $C_6H_3(^i$Pr)$_2$ + $C_6H_5$), 6.66 (m, 1 H, $C_6H_5$), 4.11 (sept, $^3J_{HH}$ = 6.8 Hz, 1 H, -C*H*(CH$_3$)$_2$), 3.74 (sept, $^3J_{HH}$ = 6.9 Hz, 1H, -C*H*(CH$_3$)$_2$), 3.48 (s, 1 H, SiC*H*), 3.31 (sept, $^3J_{HH}$ = 6.8 Hz, 1 H, -C*H*(CH$_3$)$_2$), 2.89 (sept, $^3J_{HH}$ = 6.8 Hz, 1 H, -C*H*(CH$_3$)$_2$), 1.95 (s, 3 H, SiCCC*H$_3$*), 1.72 (s, 3 H, AlCC*H$_3$*), 1.62 (d, $^3J_{HH}$ = 6.7 Hz, 3 H, -CH(C*H$_3$*)$_2$), 1.48 (d, $^3J_{HH}$ = 6.8 Hz, 3 H, -CH(C*H$_3$*)$_2$), 1.39 (d, $^3J_{HH}$ = 7.1 Hz, 3 H, -CH(C*H$_3$*)$_2$), 1.37 (d, $^3J_{HH}$ = 7.1 Hz, 3 H, -CH(C*H$_3$*)$_2$), 1.33 (d, $^3J_{HH}$ = 6.8 Hz, 3 H, -CH(C*H$_3$*)$_2$), 1.30 (d, $^3J_{HH}$ = 6.8 Hz, 3 H, -CH(C*H$_3$*)$_2$), 1.27 (d, $^3J_{HH}$ = 6.8 Hz, 3 H, -CH(C*H$_3$*)$_2$), 1.24 (d, $^3J_{HH}$ = 6.9 Hz, 3 H, -CH(C*H$_3$*)$_2$), 1.11 (s, 9 H, C(C*H$_3$*)$_3$), 1.06 (s, 9 H, C(C*H$_3$*)$_3$), 0.69 (s, 9 H, Si(C*H$_3$*)$_3$).

**$^{13}$C NMR** ($C_6D_6$, 101 MHz): δ 177.5 (N*C*N), 177.0 (SiC*C*N), 150.4 (N*C*CCH(CH$_3$)$_2$), 148.0 (*C*CH(CH$_3$)$_2$), 146.7 (*C*CH(CH$_3$)$_2$), 144.1 (NCCCH(CH$_3$)$_2$), 136.9 (*C*CH(CH$_3$)$_2$), 136.5 (*C*CH(CH$_3$)$_2$), 131.0, 130.2 (*C*$_6$H$_5$), 129.5, 128.8 (*C*$_6$H$_5$), 128.4, 127.8, 127.4 (*C*$_6$H$_5$), 127.0, 125.9, 125.1, 124.0, 124.0, 123.4 (NCC(CH(CH$_3$)$_2$)*C*), 123.1, 71.9 (Si*C*H), 55.7, 55.4 (*C*(CH$_3$)$_3$), 31.7, 31.4, 28.7, 28.4, 28.1, 27.7 (-*C*H(CH$_3$)$_2$), 27.5 (Al*C*CH$_3$), 27.3 (SiC*C*CH$_3$), 26.1, 25.7, 25.6, 25.4, 25.2, 25.1, 25.0, 24.9 (CH(*C*H$_3$)$_2$), 4.9 (Si(*C*H$_3$)$_3$) ppm.

**$^{29}$Si NMR** ($C_6D_6$, 119 MHz, DEPT90): δ –23.3 ppm.

## 12.2. Spektren [PhC(N$^t$Bu)$_2$Si][N(TMS)Al(Cl)(C(Me)=NDipp)][CH=C(Me)NDipp] **12**

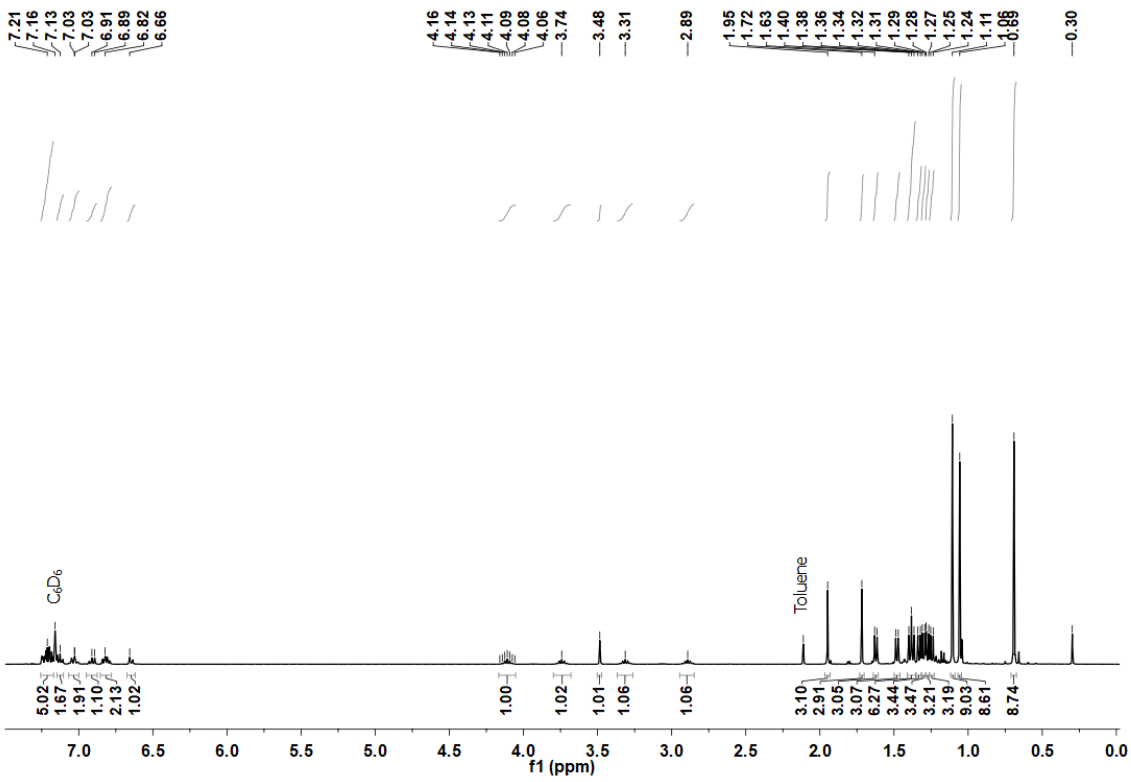



Abbildung 48: $^1$H-NMR-Spektrum von [PhC(N$^t$Bu)$_2$Si][N(TMS)Al(Cl)(C(Me)=NDipp)][CH=C(Me)NDipp] **12** in $C_6D_6$.

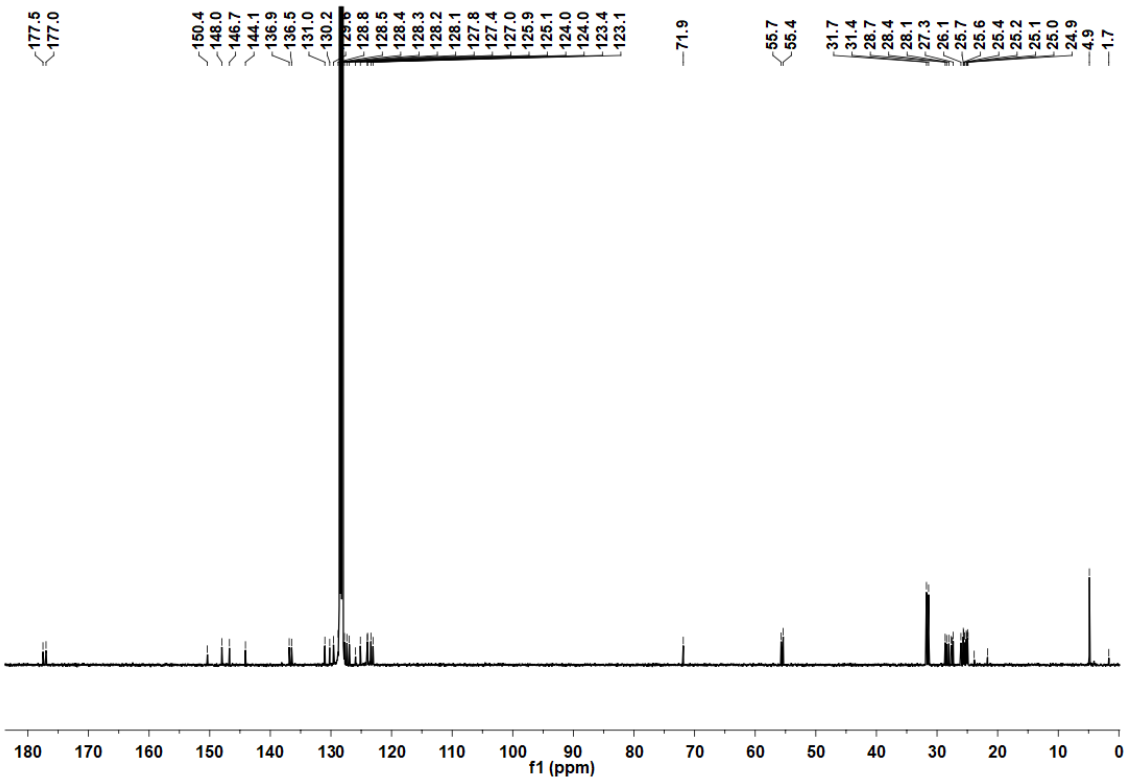



Abbildung 49: $^{13}$C-NMR-Spektrum von [PhC(N$^t$Bu)$_2$Si][N(TMS)Al(Cl)(C(Me)=NDipp)][CH=C(Me)NDipp] **12** in $C_6D_6$.

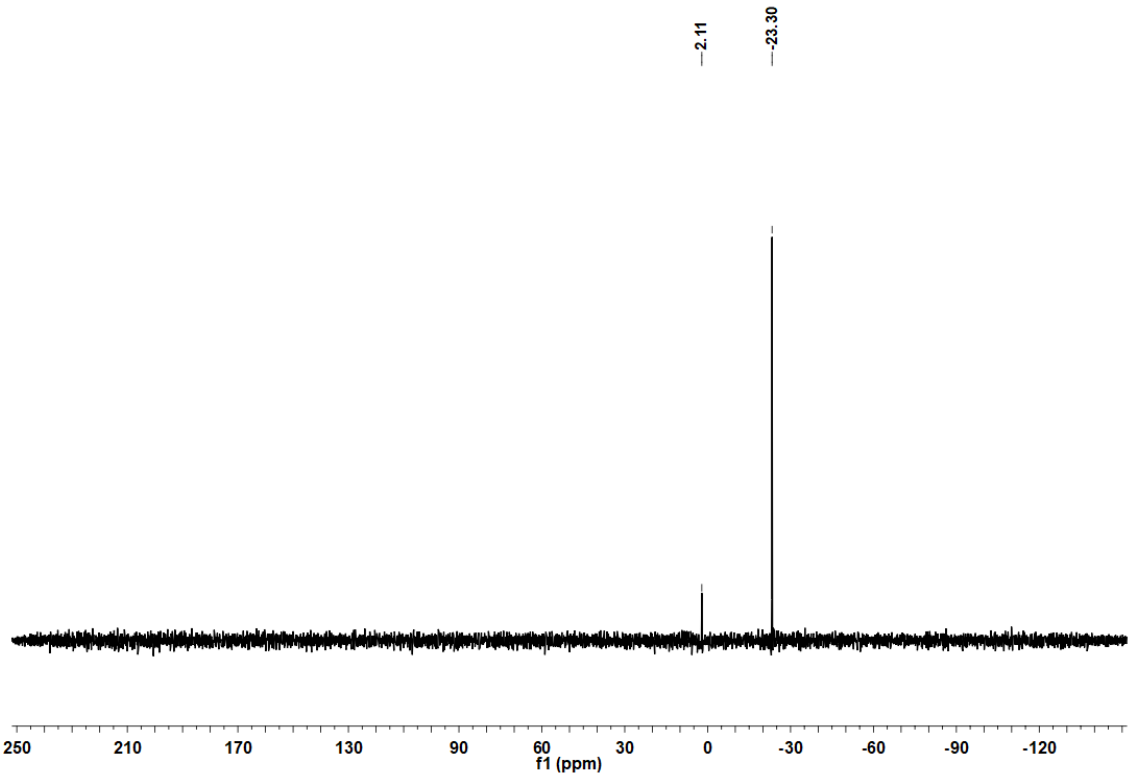



Abbildung 50: $^{29}$Si-NMR-Spektrum von [PhC(N$^t$Bu)$_2$Si][N(TMS)Al(Cl)(C(Me)=NDipp)][CH=C(Me)NDipp] **12** in $C_6D_6$.

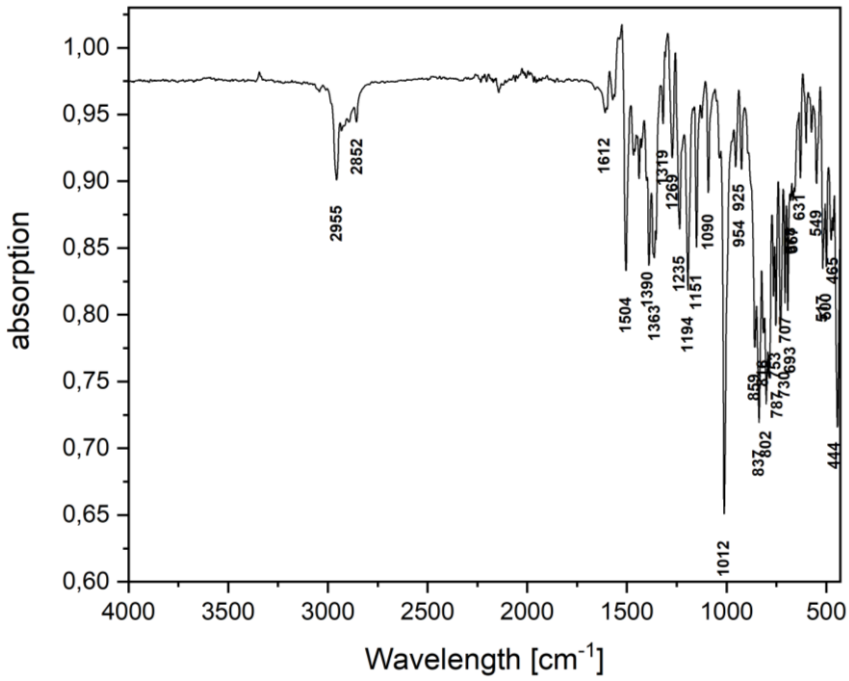



Abbildung 51: ATR-IR Spektrum von [PhC(N$^t$Bu)$_2$Si][N(TMS)Al(Cl)(C(Me)=NDipp)][CH=C(Me)NDipp] **12**.

## 12.3. Kristallografische Daten [PhC(N$^t$Bu)$_2$Si][N(TMS)Al(Cl)(C(Me)=NDipp)][CH=C(Me)NDipp] **12**

Tabelle 46: Crystal structure data

| **Identification code** | **jus_343m** |
|---|---|
| Empirical formula | C54 H81 Al Cl N5 Si2 |
| Formula weight | 918.84 |
| Density (calculated) | 1.126 g·cm$^{-3}$ |
| *F*(000) | 1992 |
| Temperature | 100(2) K |
| Crystal size | 0.220 × 0.152 × 0.150 mm |
| Crystal colour | colourless |
| Crystal description | tablet |
| Wavelength | 1.54178 Å |
| Crystal system | monoclinic |
| Space group | *P 21/c* |
| Unit cell dimensions | |
| *a* [Å] | 19.9093(8) |
| *b* [Å] | 10.5548(6) |
| *c* [Å] | 26.165(2) |
| *α* [°] | 90 |
| *β* [°] | 99.712(4) |
| *γ* [°] | 90 |
| Volume | 5419.5(6) Å$^3$ |
| *Z* | 4 |
| Cell measurement reflections used | 9147 |
| Cell measurement *θ* min/max | 3.42°/78.90° |
| Diffractometer control software | Bruker APEX3(v2017.3-0) |
| Diffractometer measurement device | Bruker D8 Venture (Photon II detector) |
| Diffractometer measurement method | Data collection strategy APEX 3/Queen |
| *θ* range for data collection | 2.251°- 80.043° |
| Completeness to *θ* = 67.679° | 100.0% |
| Completeness to $\theta_{max}$ = 80.043° | 98.9% |
| Index ranges | -25 ≤ *h* ≤ 25 |
| | -11 ≤ *k* ≤ 13 |
| | -33 ≤ *l* ≤ 33 |
| Computing data reduction | Bruker APEX3(v2017.3-0) |
| Absorption coefficient | 1.489 mm$^{-1}$ |
| Absorption correction | Semi-empirical from equivalents |
| Computation absorption correction | SADABS |
| Max./min. Transmission | 0.75/0.62 |
| $R_{merg}$ before/after correction | 0.1472/0.0963 |
| Computing structure solution | Bruker APEX3(v2017.3-0) |

| | |
|---|---|
| Computing structure refinement | SHELXL-2017/1 (Sheldrick, 2017) |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Reflections collected | 217707 |
| Independent reflections | 11717 |
| $R_{int}$ | 0.0767 |
| Reflections with $I > 2\sigma(I)$ | 10448 |
| Restraints | 457 |
| Parameter | 706 |
| GooF | 1.034 |
| Weighting details | $w = 1/[\sigma^2(F_{obs}^2) + (0.0535P)^2 + 4.5398P]$ where $P = (F_{obs}^2 + 2F_{calc}^2)/3$ |
| $R_1$ [$I > 2\sigma(I)$] | 0.0460 |
| $wR_2$ [$I > 2\sigma(I)$] | 0.1167 |
| $R_1$ [all data] | 0.0518 |
| $wR_2$ [all data] | 0.1225 |
| Absolute structure parameter | |
| Largest diff. peak and hole | 0.777/-0.424 |

Tabelle 47: Atomic coordinates ( × $10^4$) and equivalent isotropic displacement parameters (Å$^2$ × $10^3$) for jus_343m. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | *x* | *y* | *z* | $U_{eq}$ | | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|---|---|---|---|---|
| Cl(1) | 7578(1) | 2845(1) | 8367(1) | 28(1) | C(15) | 9470(1) | 3452(2) | 8837(1) | 30(1) |
| Si(1) | 6920(1) | 6290(1) | 8704(1) | 21(1) | H(15) | 9021 | 3564 | 8956 | 36 |
| Si(2) | 6196(1) | 5673(1) | 7628(1) | 26(1) | C(16) | 9442(1) | 2210(2) | 8534(1) | 38(1) |
| Al(1) | 7727(1) | 4765(1) | 8088(1) | 21(1) | H(16A) | 9062 | 2236 | 8243 | 57 |
| N(1) | 8443(1) | 5504(1) | 8556(1) | 22(1) | H(16B) | 9377 | 1502 | 8764 | 57 |
| N(2) | 7417(1) | 4409(2) | 7046(1) | 31(1) | H(16C) | 9870 | 2094 | 8402 | 57 |
| N(3) | 6467(1) | 7782(1) | 8762(1) | 24(1) | C(17) | 10026(1) | 3357(2) | 9322(1) | 38(1) |
| N(4) | 6218(1) | 5970(1) | 9052(1) | 24(1) | H(17A) | 10464 | 3157 | 9217 | 56 |
| N(5) | 6920(1) | 5679(1) | 8117(1) | 23(1) | H(17B) | 9907 | 2687 | 9550 | 56 |
| C(1) | 8354(1) | 6032(2) | 9016(1) | 24(1) | H(17C) | 10062 | 4167 | 9508 | 56 |
| C(2) | 7718(1) | 6260(2) | 9140(1) | 25(1) | C(18) | 7406(1) | 3919(3) | 6536(1) | 27(1) |
| H(2) | 7702 | 6418 | 9495 | 30 | C(19) | 7230(1) | 2642(3) | 6435(1) | 30(1) |
| C(3) | 7965(1) | 4465(2) | 7387(1) | 26(1) | C(20) | 7180(1) | 2200(3) | 5928(1) | 36(1) |
| C(4) | 8979(1) | 6379(2) | 9401(1) | 30(1) | H(20) | 7071 | 1335 | 5856 | 44 |
| H(4A) | 9267 | 5628 | 9482 | 45 | C(21) | 7285(1) | 2996(3) | 5529(1) | 41(1) |
| H(4B) | 8841 | 6700 | 9719 | 45 | H(21) | 7248 | 2680 | 5185 | 49 |
| H(4C) | 9236 | 7036 | 9252 | 45 | C(22) | 7445(1) | 4255(3) | 5634(1) | 40(1) |
| C(5) | 8656(1) | 4125(2) | 7261(1) | 34(1) | H(22) | 7515 | 4801 | 5359 | 47 |
| H(5A) | 8605 | 3831 | 6901 | 51 | C(23) | 7507(1) | 4739(3) | 6137(1) | 31(1) |
| H(5B) | 8860 | 3451 | 7494 | 51 | C(24) | 7116(1) | 1760(2) | 6871(1) | 33(1) |
| H(5C) | 8952 | 4873 | 7306 | 51 | H(24) | 7005 | 2303 | 7159 | 39 |
| C(6) | 9110(1) | 5587(2) | 8408(1) | 25(1) | C(25) | 6520(1) | 845(3) | 6722(1) | 49(1) |
| C(7) | 9263(1) | 6666(2) | 8134(1) | 31(1) | H(25A) | 6107 | 1326 | 6588 | 73 |
| C(8) | 9874(1) | 6697(2) | 7939(1) | 40(1) | H(25B) | 6449 | 363 | 7029 | 73 |
| H(8) | 9978 | 7415 | 7748 | 48 | H(25C) | 6623 | 261 | 6455 | 73 |
| C(9) | 10327(1) | 5698(2) | 8020(1) | 44(1) | C(26) | 7764(1) | 1032(3) | 7085(1) | 49(1) |
| H(9) | 10736 | 5721 | 7878 | 53 | H(26A) | 7882 | 468 | 6816 | 74 |
| C(10) | 10187(1) | 4671(2) | 8306(1) | 38(1) | H(26B) | 7688 | 528 | 7385 | 74 |
| H(10) | 10508 | 4000 | 8367 | 45 | H(26C) | 8137 | 1631 | 7191 | 74 |
| C(11) | 9583(1) | 4589(2) | 8510(1) | 29(1) | C(27) | 7699(1) | 6123(3) | 6235(1) | 35(1) |
| C(12) | 8772(1) | 7770(2) | 8040(1) | 34(1) | H(27) | 7679 | 6307 | 6607 | 43 |
| H(12) | 8407 | 7627 | 8253 | 41 | C(28) | 7198(1) | 7021(3) | 5905(1) | 49(1) |
| C(13) | 8434(1) | 7817(2) | 7474(1) | 54(1) | H(28A) | 7231 | 6911 | 5538 | 73 |
| H(13A) | 8781 | 7958 | 7255 | 81 | H(28B) | 7308 | 7899 | 6008 | 73 |
| H(13B) | 8102 | 8511 | 7423 | 81 | H(28C) | 6732 | 6827 | 5957 | 73 |
| H(13C) | 8200 | 7012 | 7378 | 81 | C(29) | 8425(1) | 6403(3) | 6149(1) | 45(1) |
| C(14) | 9114(1) | 9032(2) | 8206(1) | 48(1) | H(29A) | 8460 | 6241 | 5785 | 68 |
| H(14A) | 9468 | 9206 | 7997 | 72 | H(29B) | 8745 | 5856 | 6373 | 68 |
| H(14B) | 9321 | 8989 | 8573 | 72 | H(29C) | 8535 | 7293 | 6232 | 68 |
| H(14C) | 8774 | 9710 | 8155 | 72 | C(18') | 7406(7) | 4371(11) | 6461(4) | 12(3) |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| C(19') | 7304(6) | 3112(10) | 6323(4) | 14(3) | H(38B) | 5635 | 9851 | 8624 | 97 |
| C(20') | 7215(5) | 2794(10) | 5786(4) | 9(2) | H(38C) | 5836 | 9422 | 8083 | 97 |
| H(20') | 7142 | 1938 | 5678 | 11 | C(39) | 6860(1) | 9749(2) | 9227(1) | 44(1) |
| C(21') | 7235(6) | 3733(11) | 5426(4) | 15(2) | H(39A) | 6991 | 10631 | 9180 | 66 |
| H(21') | 7182 | 3515 | 5069 | 18 | H(39B) | 7254 | 9279 | 9407 | 66 |
| C(22') | 7332(6) | 4999(11) | 5571(4) | 21(3) | H(39C) | 6493 | 9726 | 9433 | 66 |
| H(22') | 7325 | 5641 | 5316 | 25 | C(40) | 7195(1) | 9200(2) | 8388(1) | 46(1) |
| C(23') | 7441(6) | 5314(10) | 6096(4) | 13(3) | H(40A) | 7057 | 8759 | 8057 | 69 |
| C(24') | 7309(6) | 2029(10) | 6703(5) | 15(3) | H(40B) | 7600 | 8788 | 8583 | 69 |
| H(24') | 7373 | 2399 | 7059 | 18 | H(40C) | 7301 | 10085 | 8321 | 69 |
| C(25') | 6646(6) | 1270(15) | 6623(6) | 28(4) | C(41) | 5949(1) | 4858(2) | 9300(1) | 26(1) |
| H(25D) | 6277 | 1799 | 6711 | 42 | C(42) | 5175(1) | 4918(2) | 9272(1) | 33(1) |
| H(25E) | 6702 | 521 | 6847 | 42 | H(42A) | 4956 | 5048 | 8912 | 50 |
| H(25F) | 6534 | 1003 | 6260 | 42 | H(42B) | 5061 | 5623 | 9486 | 50 |
| C(26') | 7907(6) | 1091(14) | 6682(6) | 38(4) | H(42C) | 5014 | 4122 | 9402 | 50 |
| H(26D) | 7857 | 712 | 6336 | 57 | C(43) | 6308(1) | 4755(2) | 9863(1) | 36(1) |
| H(26E) | 7899 | 422 | 6941 | 57 | H(43A) | 6802 | 4752 | 9874 | 55 |
| H(26F) | 8341 | 1548 | 6757 | 57 | H(43B) | 6170 | 3968 | 10015 | 55 |
| C(27') | 7594(7) | 6683(14) | 6249(6) | 29(4) | H(43C) | 6181 | 5480 | 10061 | 55 |
| H(27') | 7554 | 6753 | 6624 | 35 | C(44) | 6129(1) | 3704(2) | 9001(1) | 39(1) |
| C(28') | 7112(7) | 7679(17) | 5962(7) | 37(4) | H(44A) | 5900 | 3764 | 8640 | 59 |
| H(28D) | 7176 | 7720 | 5599 | 55 | H(44B) | 5980 | 2934 | 9158 | 59 |
| H(28E) | 7213 | 8508 | 6125 | 55 | H(44C) | 6623 | 3673 | 9012 | 59 |
| H(28F) | 6639 | 7446 | 5978 | 55 | C(45) | 5964(1) | 4036(2) | 7396(1) | 37(1) |
| C(29') | 8334(7) | 7050(20) | 6210(8) | 46(5) | H(45A) | 5466 | 3950 | 7327 | 55 |
| H(29D) | 8420 | 6869 | 5859 | 70 | H(45B) | 6157 | 3425 | 7662 | 55 |
| H(29E) | 8650 | 6553 | 6461 | 70 | H(45C) | 6145 | 3873 | 7077 | 55 |
| H(29F) | 8403 | 7953 | 6284 | 70 | C(46) | 5411(1) | 6291(2) | 7858(1) | 36(1) |
| C(30) | 6067(1) | 7203(2) | 9053(1) | 23(1) | H(46A) | 5496 | 7155 | 7991 | 55 |
| C(31) | 5548(1) | 7784(2) | 9325(1) | 25(1) | H(46B) | 5301 | 5743 | 8135 | 55 |
| C(32) | 4917(1) | 8139(2) | 9044(1) | 29(1) | H(46C) | 5029 | 6297 | 7569 | 55 |
| H(32) | 4836 | 8086 | 8677 | 35 | C(47) | 6311(1) | 6742(2) | 7084(1) | 41(1) |
| C(33) | 4409(1) | 8568(2) | 9305(1) | 34(1) | H(47A) | 6707 | 6465 | 6936 | 62 |
| H(33) | 3980 | 8815 | 9115 | 41 | H(47B) | 6384 | 7610 | 7214 | 62 |
| C(34) | 4527(1) | 8638(2) | 9839(1) | 35(1) | H(47C) | 5903 | 6713 | 6816 | 62 |
| H(34) | 4174 | 8908 | 10017 | 42 | C11 | 8906(2) | 3977(4) | 5003(1) | 38(1) |
| C(35) | 5159(1) | 8317(2) | 10118(1) | 34(1) | C21 | 9230(3) | 3634(5) | 5489(2) | 48(1) |
| H(35) | 5241 | 8390 | 10485 | 40 | H21 | 9496 | 4243 | 5701 | 57 |
| C(36) | 5670(1) | 7890(2) | 9862(1) | 30(1) | C31 | 9176(2) | 2410(5) | 5677(2) | 61(1) |
| H(36) | 6104 | 7670 | 10053 | 35 | H31 | 9395 | 2194 | 6017 | 73 |
| C(37) | 6616(1) | 9149(2) | 8702(1) | 30(1) | C41 | 8801(3) | 1508(5) | 5365(2) | 63(1) |
| C(38) | 5998(1) | 9849(2) | 8414(1) | 64(1) | H41 | 8767 | 668 | 5488 | 75 |
| H(38A) | 6125 | 10723 | 8348 | 97 | C51 | 8477(2) | 1841(4) | 4875(2) | 50(1) |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| H51 | 8219 | 1229 | 4658 | 59 | H32 | 9658 | 4842 | 5605 | 91 |
| C61 | 8529(4) | 3068(6) | 4699(2) | 39(1) | C42 | 9038(6) | 4679(12) | 4905(5) | 78(3) |
| H61 | 8302 | 3291 | 4362 | 47 | H42 | 9107 | 5512 | 4787 | 93 |
| C71 | 8955(2) | 5286(4) | 4802(2) | 56(1) | C52 | 8618(5) | 3839(13) | 4590(4) | 73(3) |
| H7A1 | 8955 | 5253 | 4427 | 84 | H52 | 8412 | 4088 | 4250 | 87 |
| H7B1 | 9377 | 5681 | 4975 | 84 | C62 | 8502(11) | 2660(16) | 4767(5) | 52(4) |
| H7C1 | 8564 | 5784 | 4869 | 84 | H62 | 8213 | 2092 | 4549 | 62 |
| C12 | 8796(4) | 2275(11) | 5258(3) | 57(3) | C72 | 8661(8) | 971(15) | 5447(6) | 79(4) |
| C22 | 9225(8) | 3103(14) | 5568(4) | 60(4) | H7A2 | 8407 | 478 | 5161 | 118 |
| H22 | 9433 | 2848 | 5906 | 72 | H7B2 | 8393 | 1036 | 5728 | 118 |
| C32 | 9353(5) | 4289(13) | 5393(4) | 76(3) | H7C2 | 9095 | 549 | 5576 | 118 |

Tabelle 48: Anisotropic displacement parameters (Å$^2$ × 10$^3$) for jus_343m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Cl(1) | 27(1) | 27(1) | 30(1) | -1(1) | 0(1) | 0(1) | C(19) | 21(1) | 37(1) | 31(1) | -5(1) | 1(1) | 1(1) |
| Si(1) | 19(1) | 27(1) | 18(1) | 0(1) | 1(1) | 1(1) | C(20) | 28(1) | 47(2) | 32(1) | -16(1) | 1(1) | -2(1) |
| Si(2) | 20(1) | 35(1) | 20(1) | -2(1) | -2(1) | 2(1) | C(21) | 30(1) | 66(2) | 26(1) | -16(1) | 6(1) | -9(1) |
| Al(1) | 18(1) | 27(1) | 18(1) | -3(1) | 0(1) | 0(1) | C(22) | 32(1) | 62(2) | 25(1) | -8(1) | 6(1) | -15(1) |
| N(1) | 18(1) | 29(1) | 19(1) | -3(1) | 1(1) | -1(1) | C(23) | 24(1) | 44(2) | 25(1) | -4(1) | 2(1) | -7(1) |
| N(2) | 24(1) | 45(1) | 22(1) | -7(1) | 1(1) | 0(1) | C(24) | 28(1) | 32(1) | 36(1) | -4(1) | -1(1) | 1(1) |
| N(3) | 23(1) | 26(1) | 23(1) | 0(1) | 3(1) | 2(1) | C(25) | 42(1) | 51(2) | 50(2) | 2(1) | -1(1) | -13(1) |
| N(4) | 20(1) | 29(1) | 22(1) | 3(1) | 4(1) | 2(1) | C(26) | 40(1) | 45(1) | 60(2) | 4(1) | 2(1) | 10(1) |
| N(5) | 18(1) | 28(1) | 21(1) | -1(1) | 0(1) | 0(1) | C(27) | 33(1) | 47(2) | 25(1) | 2(1) | 4(1) | -6(1) |
| C(1) | 23(1) | 27(1) | 19(1) | -2(1) | -1(1) | 0(1) | C(28) | 50(2) | 61(2) | 34(1) | 10(1) | 3(1) | 1(1) |
| C(2) | 23(1) | 34(1) | 17(1) | -2(1) | 1(1) | 2(1) | C(29) | 37(1) | 60(2) | 39(1) | 1(1) | 7(1) | -18(1) |
| C(3) | 23(1) | 32(1) | 23(1) | -4(1) | 3(1) | -1(1) | C(30) | 19(1) | 31(1) | 18(1) | 0(1) | -1(1) | 2(1) |
| C(4) | 24(1) | 42(1) | 23(1) | -8(1) | -1(1) | 0(1) | C(31) | 24(1) | 27(1) | 26(1) | 0(1) | 4(1) | 2(1) |
| C(5) | 24(1) | 53(1) | 24(1) | -10(1) | 1(1) | 1(1) | C(32) | 26(1) | 31(1) | 29(1) | 0(1) | 2(1) | 4(1) |
| C(6) | 19(1) | 34(1) | 22(1) | -8(1) | 1(1) | -4(1) | C(33) | 27(1) | 31(1) | 45(1) | -1(1) | 5(1) | 5(1) |
| C(7) | 27(1) | 37(1) | 27(1) | -6(1) | 2(1) | -8(1) | C(34) | 36(1) | 29(1) | 45(1) | -4(1) | 17(1) | 2(1) |
| C(8) | 36(1) | 46(1) | 38(1) | -5(1) | 11(1) | -15(1) | C(35) | 43(1) | 31(1) | 29(1) | -4(1) | 11(1) | -1(1) |
| C(9) | 28(1) | 58(1) | 50(1) | -14(1) | 14(1) | -10(1) | C(36) | 29(1) | 32(1) | 27(1) | -1(1) | 4(1) | 1(1) |
| C(10) | 23(1) | 47(1) | 43(1) | -12(1) | 6(1) | 0(1) | C(37) | 31(1) | 25(1) | 32(1) | 2(1) | 3(1) | 1(1) |
| C(11) | 19(1) | 38(1) | 28(1) | -10(1) | 0(1) | -2(1) | C(38) | 44(1) | 36(1) | 104(2) | 29(1) | -16(1) | -1(1) |
| C(12) | 35(1) | 33(1) | 33(1) | 4(1) | 3(1) | -7(1) | C(39) | 53(1) | 39(1) | 44(1) | -13(1) | 17(1) | -12(1) |
| C(13) | 69(2) | 40(1) | 44(1) | 4(1) | -17(1) | -6(1) | C(40) | 65(1) | 30(1) | 50(1) | -1(1) | 29(1) | -7(1) |
| C(14) | 55(1) | 37(1) | 47(1) | -3(1) | -6(1) | -7(1) | C(41) | 25(1) | 30(1) | 26(1) | 5(1) | 8(1) | 2(1) |
| C(15) | 22(1) | 36(1) | 31(1) | -6(1) | -1(1) | 6(1) | C(42) | 25(1) | 36(1) | 39(1) | 6(1) | 8(1) | 0(1) |
| C(16) | 36(1) | 36(1) | 40(1) | -8(1) | -1(1) | 6(1) | C(43) | 34(1) | 44(1) | 31(1) | 12(1) | 4(1) | 4(1) |
| C(17) | 27(1) | 48(1) | 34(1) | -5(1) | -4(1) | 10(1) | C(44) | 47(1) | 30(1) | 45(1) | 2(1) | 23(1) | 1(1) |
| C(18) | 16(1) | 38(2) | 25(1) | -3(1) | 2(1) | 0(1) | C(45) | 25(1) | 44(1) | 39(1) | -13(1) | -3(1) | -4(1) |
| C(46) | 23(1) | 53(1) | 30(1) | -6(1) | -4(1) | 7(1) | C71 | 62(3) | 50(2) | 59(2) | 8(2) | 17(2) | -12(2) |
| C(47) | 42(1) | 52(1) | 28(1) | 7(1) | -1(1) | 9(1) | C12 | 35(4) | 87(7) | 47(5) | -6(4) | 1(3) | 30(4) |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| C11 | 31(2) | 44(2) | 37(2) | 2(1) | 3(1) | -1(1) | C22 | 38(5) | 110(11) | 29(6) | 1(7) | -4(4) | 18(9) |
| C21 | 32(2) | 69(4) | 39(2) | 3(2) | -4(2) | -2(3) | C32 | 40(5) | 109(10) | 82(7) | -14(7) | 17(5) | 0(6) |
| C31 | 44(2) | 85(3) | 52(2) | 29(2) | 7(2) | 20(2) | C42 | 70(7) | 72(9) | 100(8) | 23(6) | 41(5) | 7(6) |
| C41 | 52(2) | 49(3) | 93(4) | 21(2) | 31(2) | 15(2) | C52 | 53(6) | 99(9) | 70(7) | 16(5) | 21(5) | 23(5) |
| C51 | 36(2) | 44(2) | 72(2) | -9(2) | 18(2) | -2(2) | C62 | 36(6) | 74(11) | 42(5) | -14(5) | -6(5) | 13(7) |
| C61 | 32(2) | 48(3) | 37(2) | -4(2) | 5(2) | -1(2) | C72 | 79(9) | 94(9) | 66(7) | 3(6) | 16(6) | 41(7) |

Tabelle 49: Bond lengths [Å] for jus_343m.

| | | | | | |
|---|---|---|---|---|---|
| Cl(1)-Al(1) | 2.1906(6) | C(8)-C(9) | 1.382(3) | C(24')-C(26') | 1.555(12) |
| Si(1)-N(5) | 1.6650(14) | C(9)-C(10) | 1.372(3) | C(27')-C(28') | 1.532(13) |
| Si(1)-C(2) | 1.7940(16) | C(10)-C(11) | 1.398(3) | C(27')-C(29') | 1.543(13) |
| Si(1)-N(4) | 1.8242(14) | C(11)-C(15) | 1.513(3) | C(30)-C(31) | 1.481(2) |
| Si(1)-N(3) | 1.8337(14) | C(12)-C(13) | 1.521(3) | C(31)-C(36) | 1.390(2) |
| Si(1)-C(30) | 2.2749(17) | C(12)-C(14) | 1.525(3) | C(31)-C(32) | 1.395(2) |
| Si(2)-N(5) | 1.7596(14) | C(15)-C(16) | 1.528(3) | C(32)-C(33) | 1.387(3) |
| Si(2)-C(47) | 1.859(2) | C(15)-C(17) | 1.540(2) | C(33)-C(34) | 1.380(3) |
| Si(2)-C(45) | 1.863(2) | C(18)-C(23) | 1.396(3) | C(34)-C(35) | 1.385(3) |
| Si(2)-C(46) | 1.8834(19) | C(18)-C(19) | 1.406(4) | C(35)-C(36) | 1.385(3) |
| Al(1)-N(1) | 1.8854(14) | C(19)-C(20) | 1.394(3) | C(37)-C(39) | 1.516(3) |
| Al(1)-N(5) | 1.8864(14) | C(19)-C(24) | 1.519(3) | C(37)-C(38) | 1.522(3) |
| Al(1)-C(3) | 1.9955(17) | C(20)-C(21) | 1.385(4) | C(37)-C(40) | 1.527(3) |
| N(1)-C(1) | 1.365(2) | C(21)-C(22) | 1.383(4) | C(41)-C(44) | 1.523(3) |
| N(1)-C(6) | 1.448(2) | C(22)-C(23) | 1.397(3) | C(41)-C(43) | 1.528(2) |
| N(2)-C(3) | 1.289(2) | C(23)-C(27) | 1.521(4) | C(41)-C(42) | 1.532(2) |
| N(2)-C(18) | 1.429(2) | C(24)-C(26) | 1.524(3) | C11-C21 | 1.374(5) |
| N(2)-C(18') | 1.528(11) | C(24)-C(25) | 1.528(3) | C11-C61 | 1.383(6) |
| N(3)-C(30) | 1.339(2) | C(27)-C(29) | 1.528(3) | C11-C71 | 1.488(5) |
| N(3)-C(37) | 1.487(2) | C(27)-C(28) | 1.533(4) | C21-C31 | 1.393(6) |
| N(4)-C(30) | 1.336(2) | C(18')-C(19') | 1.382(10) | C31-C41 | 1.386(7) |
| N(4)-C(41) | 1.484(2) | C(18')-C(23') | 1.389(10) | C41-C51 | 1.381(6) |
| C(1)-C(2) | 1.380(2) | C(19')-C(20') | 1.425(10) | C51-C61 | 1.384(6) |
| C(1)-C(4) | 1.508(2) | C(19')-C(24') | 1.514(12) | C12-C62 | 1.381(11) |
| C(3)-C(5) | 1.512(2) | C(20')-C(21') | 1.373(10) | C12-C22 | 1.384(11) |
| C(6)-C(7) | 1.405(3) | C(21')-C(22') | 1.394(10) | C12-C72 | 1.502(19) |
| C(6)-C(11) | 1.408(2) | C(22')-C(23') | 1.393(10) | C22-C32 | 1.372(11) |
| C(7)-C(8) | 1.395(3) | C(23')-C(27') | 1.517(13) | C32-C42 | 1.386(11) |
| C(7)-C(12) | 1.514(3) | C(24')-C(25') | 1.529(12) | C42-C52 | 1.390(11) |
| | | | | C52-C62 | 1.362(15) |

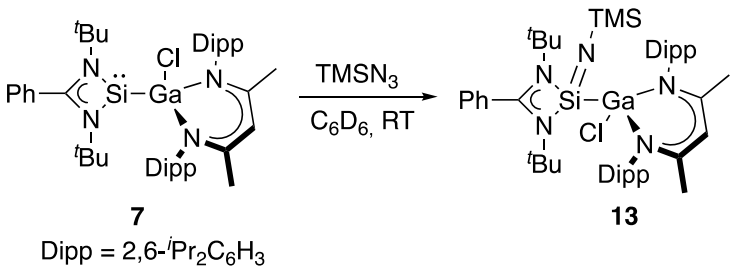
Tabelle 50: Bond angles [°] for jus_343m.

| | | | | | |
|---|---|---|---|---|---|
| N(5)-Si(1)-C(2) | 116.25(7) | C(8)-C(7)-C(12) | 119.99(18) | C(23')-C(27')-C(28') | 116.2(12) |
| N(5)-Si(1)-N(4) | 120.50(7) | C(6)-C(7)-C(12) | 121.12(16) | C(23')-C(27')-C(29') | 111.8(13) |
| C(2)-Si(1)-N(4) | 110.50(7) | C(9)-C(8)-C(7) | 120.8(2) | C(28')-C(27')-C(29') | 108.8(11) |
| N(5)-Si(1)-N(3) | 119.06(7) | C(10)-C(9)-C(8) | 119.92(18) | N(4)-C(30)-N(3) | 106.67(14) |
| C(2)-Si(1)-N(3) | 110.97(7) | C(9)-C(10)-C(11) | 121.65(19) | N(4)-C(30)-C(31) | 125.49(15) |
| N(4)-Si(1)-N(3) | 71.83(6) | C(10)-C(11)-C(6) | 118.10(18) | N(3)-C(30)-C(31) | 127.84(15) |
| N(5)-Si(1)-C(30) | 130.60(6) | C(10)-C(11)-C(15) | 119.01(17) | N(4)-C(30)-Si(1) | 53.30(8) |
| C(2)-Si(1)-C(30) | 113.15(7) | C(6)-C(11)-C(15) | 122.88(15) | N(3)-C(30)-Si(1) | 53.71(8) |
| N(4)-Si(1)-C(30) | 35.95(6) | C(7)-C(12)-C(13) | 110.62(17) | C(31)-C(30)-Si(1) | 175.12(12) |
| N(3)-Si(1)-C(30) | 36.07(6) | C(7)-C(12)-C(14) | 112.35(17) | C(36)-C(31)-C(32) | 119.92(16) |
| N(5)-Si(2)-C(47) | 111.24(8) | C(13)-C(12)-C(14) | 110.62(17) | C(36)-C(31)-C(30) | 120.05(15) |
| N(5)-Si(2)-C(45) | 111.61(8) | C(11)-C(15)-C(16) | 112.53(15) | C(32)-C(31)-C(30) | 119.86(15) |
| C(47)-Si(2)-C(45) | 111.70(10) | C(11)-C(15)-C(17) | 111.22(15) | C(33)-C(32)-C(31) | 119.74(17) |
| N(5)-Si(2)-C(46) | 112.80(7) | C(16)-C(15)-C(17) | 109.21(15) | C(34)-C(33)-C(32) | 120.07(17) |
| C(47)-Si(2)-C(46) | 104.54(10) | C(23)-C(18)-C(19) | 121.2(2) | C(33)-C(34)-C(35) | 120.32(17) |
| C(45)-Si(2)-C(46) | 104.59(9) | C(23)-C(18)-N(2) | 119.6(2) | C(34)-C(35)-C(36) | 120.08(17) |
| N(1)-Al(1)-N(5) | 108.53(6) | C(19)-C(18)-N(2) | 119.0(2) | C(35)-C(36)-C(31) | 119.83(17) |
| N(1)-Al(1)-C(3) | 113.07(7) | C(20)-C(19)-C(18) | 118.3(2) | N(3)-C(37)-C(39) | 110.42(15) |
| N(5)-Al(1)-C(3) | 117.04(7) | C(20)-C(19)-C(24) | 120.9(2) | N(3)-C(37)-C(38) | 111.40(16) |
| N(1)-Al(1)-Cl(1) | 107.20(5) | C(18)-C(19)-C(24) | 120.80(19) | C(39)-C(37)-C(38) | 110.8(2) |
| N(5)-Al(1)-Cl(1) | 107.23(5) | C(21)-C(20)-C(19) | 121.3(2) | N(3)-C(37)-C(40) | 105.84(14) |
| C(3)-Al(1)-Cl(1) | 103.05(6) | C(22)-C(21)-C(20) | 119.4(2) | C(39)-C(37)-C(40) | 108.48(17) |
| C(1)-N(1)-C(6) | 118.24(13) | C(21)-C(22)-C(23) | 121.3(2) | C(38)-C(37)-C(40) | 109.69(19) |
| C(1)-N(1)-Al(1) | 123.08(11) | C(18)-C(23)-C(22) | 118.4(2) | N(4)-C(41)-C(44) | 105.87(13) |
| C(6)-N(1)-Al(1) | 118.56(10) | C(18)-C(23)-C(27) | 122.19(19) | N(4)-C(41)-C(43) | 109.53(14) |
| C(3)-N(2)-C(18) | 122.73(16) | C(22)-C(23)-C(27) | 119.4(2) | C(44)-C(41)-C(43) | 109.08(16) |
| C(3)-N(2)-C(18') | 124.2(5) | C(19)-C(24)-C(26) | 111.1(2) | N(4)-C(41)-C(42) | 112.36(14) |
| C(30)-N(3)-C(37) | 130.62(14) | C(19)-C(24)-C(25) | 113.83(19) | C(44)-C(41)-C(42) | 109.28(16) |
| C(30)-N(3)-Si(1) | 90.22(10) | C(26)-C(24)-C(25) | 110.3(2) | C(43)-C(41)-C(42) | 110.57(15) |
| C(37)-N(3)-Si(1) | 135.65(11) | C(23)-C(27)-C(29) | 112.2(2) | C21-C11-C61 | 118.3(4) |
| C(30)-N(4)-C(41) | 132.13(14) | C(23)-C(27)-C(28) | 112.2(2) | C21-C11-C71 | 121.6(3) |
| C(30)-N(4)-Si(1) | 90.75(10) | C(29)-C(27)-C(28) | 109.8(2) | C61-C11-C71 | 120.1(3) |
| C(41)-N(4)-Si(1) | 136.74(11) | C(19')-C(18')-C(23') | 122.0(8) | C11-C21-C31 | 121.2(4) |
| Si(1)-N(5)-Si(2) | 123.03(8) | C(19')-C(18')-N(2) | 105.3(8) | C41-C31-C21 | 119.7(4) |
| Si(1)-N(5)-Al(1) | 111.59(7) | C(23')-C(18')-N(2) | 132.5(8) | C51-C41-C31 | 119.5(4) |
| Si(2)-N(5)-Al(1) | 124.56(8) | C(18')-C(19')-C(20') | 118.2(8) | C41-C51-C61 | 119.8(4) |
| N(1)-C(1)-C(2) | 122.61(14) | C(18')-C(19')-C(24') | 124.7(9) | C11-C61-C51 | 121.4(4) |
| N(1)-C(1)-C(4) | 118.23(14) | C(20')-C(19')-C(24') | 117.1(9) | C62-C12-C22 | 118.9(9) |
| C(2)-C(1)-C(4) | 119.16(14) | C(21')-C(20')-C(19') | 119.5(7) | C62-C12-C72 | 120.3(10) |
| C(1)-C(2)-Si(1) | 127.08(12) | C(20')-C(21')-C(22') | 121.5(8) | C22-C12-C72 | 120.9(9) |
| N(2)-C(3)-C(5) | 122.27(15) | C(23')-C(22')-C(21') | 119.3(8) | C32-C22-C12 | 120.8(8) |
| N(2)-C(3)-Al(1) | 109.76(12) | C(18')-C(23')-C(22') | 119.3(8) | C22-C32-C42 | 119.8(9) |

| | | | | | |
|---|---|---|---|---|---|
| C(5)-C(3)-Al(1) | 127.21(12) | C(18')-C(23')-C(27') | 122.1(9) | C32-C42-C52 | 119.4(9) |
| C(7)-C(6)-C(11) | 120.52(16) | C(22')-C(23')-C(27') | 118.6(10) | C62-C52-C42 | 120.0(9) |
| C(7)-C(6)-N(1) | 118.28(15) | C(19')-C(24')-C(25') | 113.4(10) | C52-C62-C12 | 121.1(9) |
| C(11)-C(6)-N(1) | 121.13(16) | C(19')-C(24')-C(26') | 112.2(11) | | |
| C(8)-C(7)-C(6) | 118.87(18) | C(25')-C(24')-C(26') | 108.0(9) | | |

## 13. PhC(N$^t$Bu)$_2$Si(NTMS)Ga(Cl)DDP 13

### 13.1. Synthese PhC(N$^t$Bu)$_2$Si(NTMS)Ga(Cl)DDP **13**




Es wurden 40 mg PhC(N$^t$Bu)$_2$SiGa(Cl)DDP **7** (0.051 mmol) in 2 mL Toluol gelöst, woraufhin 5.8 mg $TMSN_3$ (0.051 mmol, 6.77 µL) zur Lösung hinzugegeben wurde. Dabei entfärbte sich die zunächst gelbe Lösung unter Gasentwicklung. Die Lösung wurde auf etwa die Hälfte eingeengt und bei –18 °C gelagert, wobei PhC(N$^t$Bu)$_2$Si(NTMS)Ga(Cl)DDP **13** in Form von farblosen Kristallen erhalten werden konnte.

**Ausbeute** 26 mg (0.031 mmol, 60 %).

**Smp.** 222 °C

**Elementaranalyse** von $C_{47}H_{77}ClGaN_5Si_2$: gefunden (berechnet) C 64.2 (64.63), H 8.13 (8.89), N 7.78 (8.02)%.

**IR**: ν 3050, 2947, 2858, 1532, 1432, 1383, 1240, 1196, 1087, 1022, 931, 848, 793, 757, 706, 664, 604, 501 cm$^{-1}$.

**$^1$H NMR** ($C_6D_6$, 400 MHz): δ 7.76-6.80 (m, 11 H, $C_6H_3$($^i$Pr)$_2$ & $C_6H_5$), 5.03 (s, 1 H, γ-C*H*), 3.74 (d sept, $^3J_{HH}$ = 6.7 Hz, 4 H, -C*H*($CH_3$)$_2$), 1.85 (d, $^3J_{HH}$ = 6.6 Hz, 6 H, -CH(C*H*$_3$)$_2$), 1.62 (d, $^3J_{HH}$ = 6.6 Hz, 6 H, -CH(C*H*$_3$)$_2$), 1.61 (s, 6 H, ArNCC*H*$_3$), 1.24 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H*$_3$)$_2$), 1.17 (d, $^3J_{HH}$ = 6.8 Hz, 6 H, -CH(C*H*$_3$)$_2$), 0.86 (s, 18 H, C(C*H*$_3$)$_3$), 0.50 (Si(C*H*$_3$)$_3$) ppm.

**$^{13}$C NMR** ($C_6D_6$, 101 MHz): δ 173.7 (N*C*N), 169.3 (ArN*C*CH$_3$), 145.8, 144.2, 143.9 (*C*$_6$H$_3$), 131.9, 130.1, 129.9, 128.7, 128.4, 128.1, 127.7, 125.6, 125.1, 97.8 (γ-*C*H-), 54.1 (*C*(CH$_3$)$_3$), 31.9 (C(*C*H$_3$)$_3$), 30.3, 28.2 (-*C*H(CH$_3$)$_2$), 27.3 (ArNC*C*H$_3$), 25.3, 25.3, 25.1 (CH(*C*H$_3$)$_2$), 7.0 (Si(*C*H$_3$)$_3$) ppm.

**$^{29}$Si NMR** ($C_6D_6$, 119 MHz): δ –19.7 ((*Si*(CH$_3$)$_3$), –44.8 (N*Si*N) ppm

## 13.2. Spektren PhC(N$^t$Bu)$_2$Si(NTMS)Ga(Cl)DDP **13**

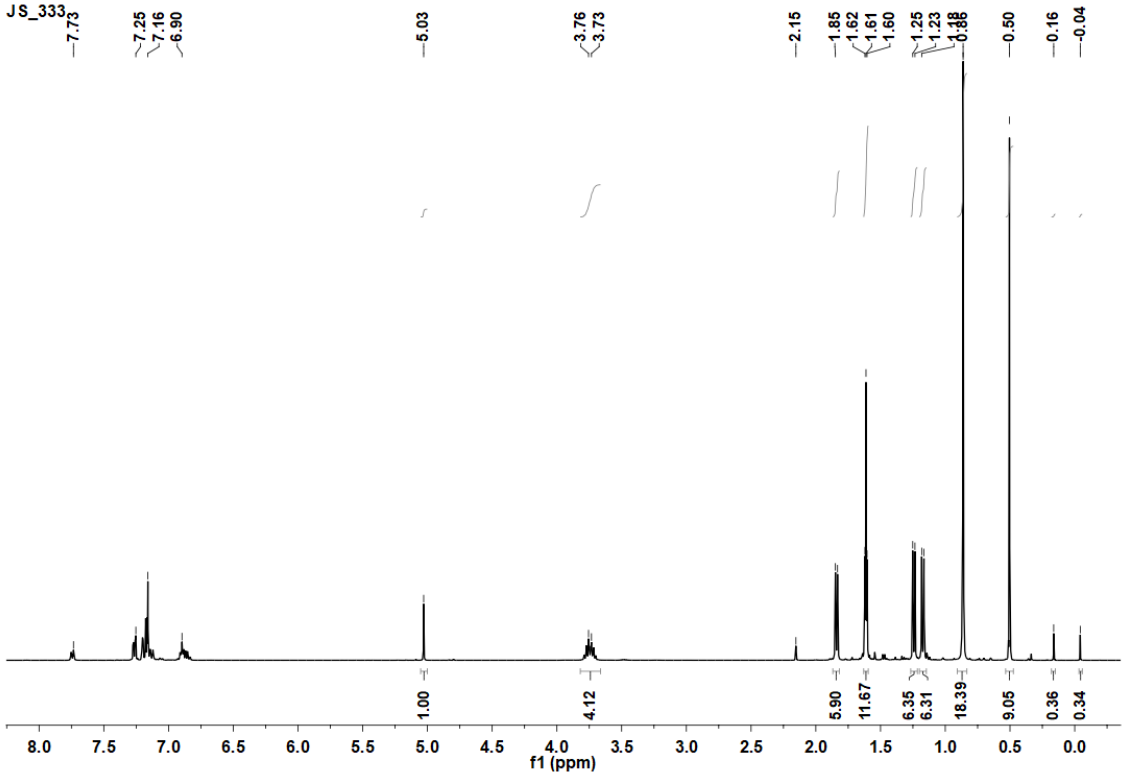



Abbildung 52: $^1$H-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(NTMS)Ga(Cl)DDP **13** in $C_6D_6$.

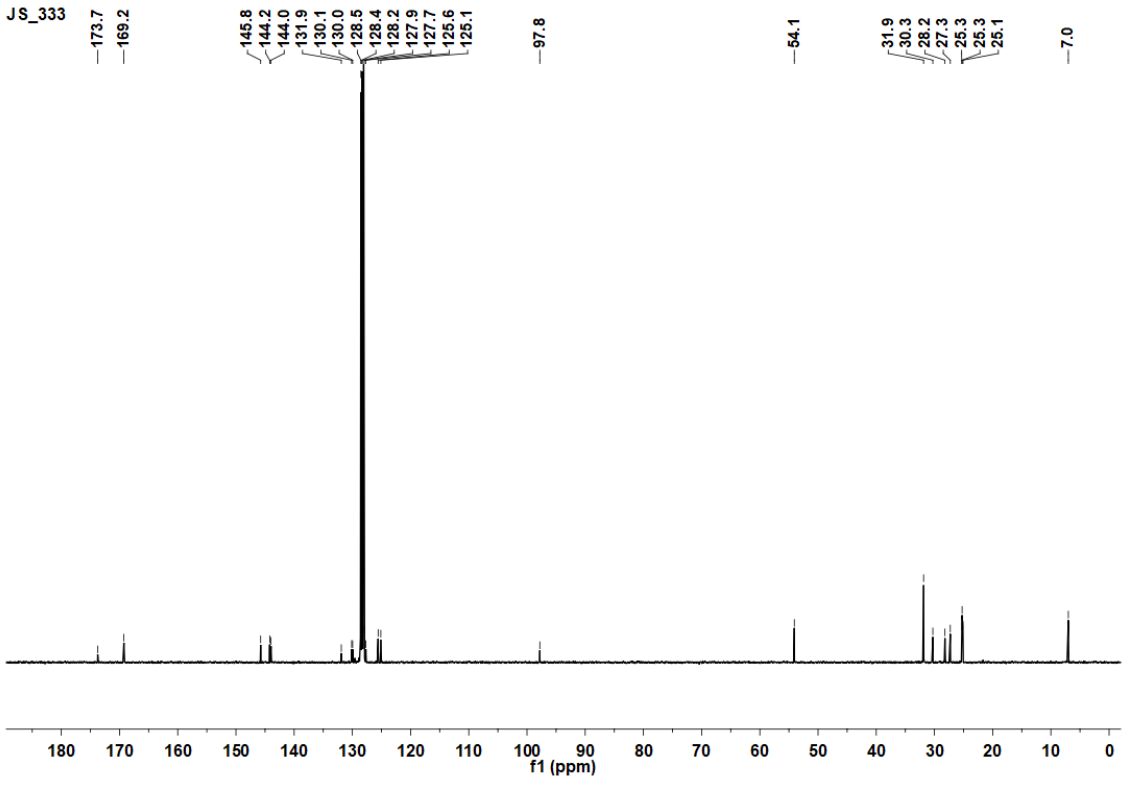



Abbildung 53: $^{13}$C-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(NTMS)Ga(Cl)DDP **13** in $C_6D_6$.

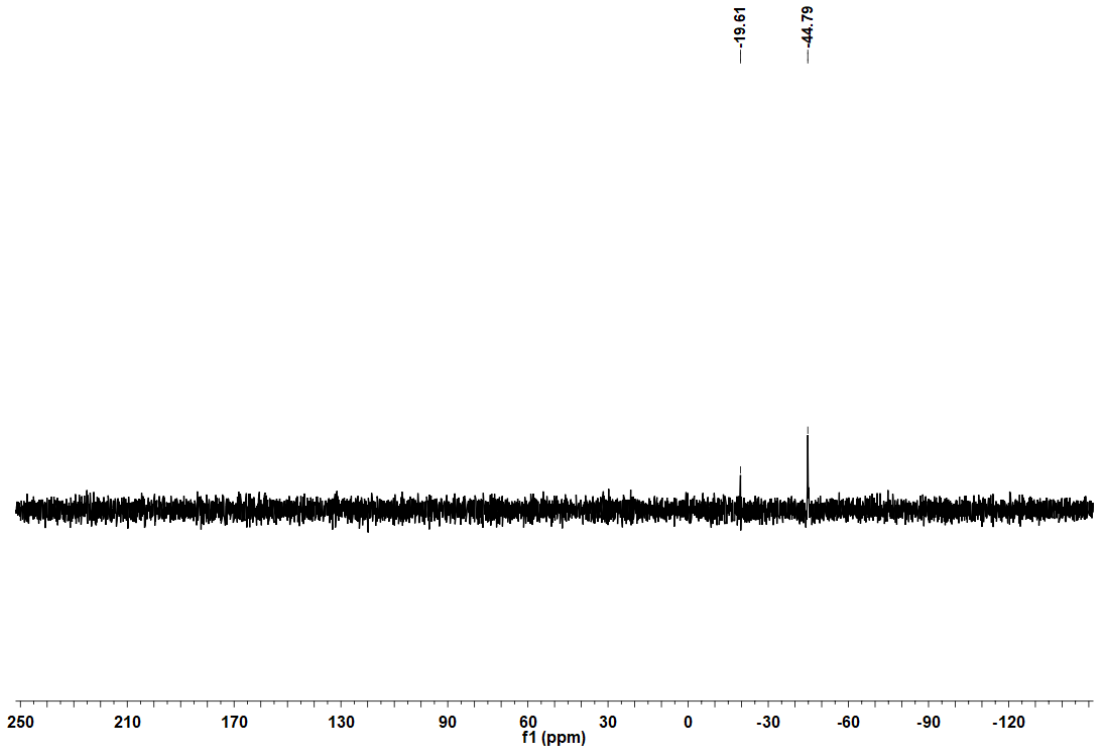



Abbildung 54: $^{29}$Si-NMR-Spektrum von PhC(N$^t$Bu)$_2$Si(NTMS)Ga(Cl)DDP **13** in $C_6D_6$.

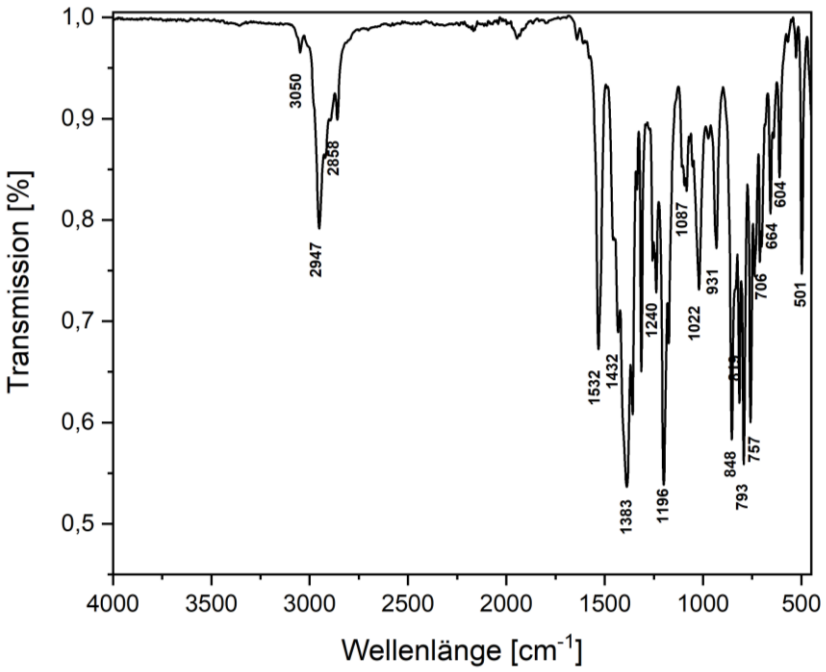



Abbildung 55: ATR-IR Spektrum von PhC(N$^t$Bu)$_2$Si(NTMS)Ga(Cl)DDP **13**.

## 13.3. Kristallografische Daten PhC($N^tBu$)$_2$Si(NTMS)Ga(Cl)DDP **13**

Tabelle 51: Crystal structure data

| **Identification code** | **jus_333m** |
|---|---|
| Empirical formula | C47 H73 Cl Ga N5 Si2 |
| Formula weight | 869.45 |
| Density (calculated) | 1.191 g·cm$^{-3}$ |
| *F*(000) | 1864 |
| Temperature | 200(2) K |
| Crystal size | 0.213 × 0.133 × 0.084 mm |
| Crystal colour | colourless |
| Crystal description | tablet |
| Wavelength | 0.71073 Å |
| Crystal system | orthorhombic |
| Space group | *P n m a* |
| Unit cell dimensions | |
| *a* [Å] | 14.5266(10) |
| *b* [Å] | 19.8815(13) |
| *c* [Å] | 16.7832(11) |
| *α* [°] | 90 |
| *β* [°] | 90 |
| *γ* [°] | 90 |
| Volume | 4847.2(6) Å$^3$ |
| *Z* | 4 |
| Cell measurement reflections used | 9962 |
| Cell measurement *θ* min/max | 2.43°/25.40° |
| Diffractometer control software | BRUKER APEX3(v2019.1-0) |
| Diffractometer measurement device | Bruker D8 KAPPA II (APEX II detector) |
| Diffractometer measurement method | Data collection strategy APEX 3/QUEEN |
| *θ* range for data collection | 2.049°- 30.620° |
| Completeness to *θ* = 25.242° | 100.0% |
| Completeness to $\theta_{max}$ = 30.620° | 99.8% |
| Index ranges | -20 ≤ *h* ≤ 20 |
| | -28 ≤ *k* ≤ 28 |
| | -24 ≤ *l* ≤ 23 |
| Computing data reduction | BRUKER APEX3(v2019.1-0) |
| Absorption coefficient | 0.708 mm$^{-1}$ |
| Absorption correction | Semi-empirical from equivalents |
| Computation absorption correction | SADABS |
| Max./min. Transmission | 0.75/0.70 |
| $R_{merg}$ before/after correction | 0.0534/0.0485 |
| Computing structure solution | BRUKER APEX3(v2019.1-0) |
| Computing structure refinement | SHELXL-2017/1 (Sheldrick, 2017) |

| | |
|---|---|
| Refinement method | Full-matrix least-squares on $F^2$ |
| Reflections collected | 150399 |
| Independent reflections | 7652 |
| $R_{int}$ | 0.0647 |
| Reflections with $I > 2\sigma(I)$ | 5812 |
| Restraints | 15 |
| Parameter | 315 |
| GooF | 1.048 |
| Weighting details | $w = 1/[\sigma^2(F_{obs}^2) + (0.0466P)^2 + 2.6789P]$ |
| | where $P = (F_{obs}^2 + 2F_{calc}^2)/3$ |
| $R_1$ [$I > 2\sigma(I)$] | 0.0400 |
| $wR_2$ [$I > 2\sigma(I)$] | 0.0945 |
| $R_1$ [all data] | 0.0630 |
| $wR_2$ [all data] | 0.1072 |
| Absolute structure parameter | |
| Largest diff. peak and hole | 0.506/-0.365 |

Tabelle 52: Atomic coordinates ( × $10^4$) and equivalent isotropic displacement parameters (Å$^2$ × $10^3$) for jus_333m. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | *x* | *y* | *z* | $U_{eq}$ | | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|---|---|---|---|---|
| Ga(1) | 2618(1) | 7500 | 715(1) | 26(1) | H(15A) | 3977 | 6555 | -1867 | 109 |
| Cl(1) | 1659(1) | 7500 | 1789(1) | 38(1) | H(15B) | 3132 | 6088 | -1604 | 109 |
| Si(1) | 1612(1) | 7500 | -452(1) | 21(1) | H(15C) | 4149 | 5781 | -1654 | 109 |
| Si(2) | 1668(1) | 7500 | -2261(1) | 34(1) | C(16) | 88(2) | 7500 | -49(1) | 23(1) |
| N(1) | 3488(1) | 6760(1) | 956(1) | 37(1) | C(17) | -857(2) | 7500 | 296(1) | 28(1) |
| N(2) | 601(1) | 6963(1) | -217(1) | 25(1) | C(18) | -1633(2) | 7500 | -179(2) | 40(1) |
| N(3) | 2079(1) | 7500 | -1323(1) | 31(1) | H(18) | -1575 | 7500 | -743 | 48 |
| C(1) | 4262(1) | 6872(1) | 1367(1) | 50(1) | C(19) | -2502(2) | 7500 | 171(2) | 58(1) |
| C(2) | 4571(2) | 7500 | 1598(2) | 58(1) | H(19) | -3038 | 7500 | -154 | 69 |
| H(2) | 5066 | 7500 | 1969 | 69 | C(20) | -2584(2) | 7500 | 993(2) | 63(1) |
| C(3) | 4899(2) | 6293(2) | 1584(2) | 75(1) | H(20) | -3177 | 7500 | 1230 | 76 |
| H(3A) | 4705 | 5886 | 1301 | 113 | C(21) | -1814(2) | 7500 | 1468(2) | 55(1) |
| H(3B) | 4872 | 6213 | 2160 | 113 | H(21) | -1876 | 7500 | 2031 | 66 |
| H(3C) | 5532 | 6407 | 1432 | 113 | C(22) | -944(2) | 7500 | 1124(2) | 40(1) |
| C(4) | 3302(1) | 6072(1) | 713(1) | 40(1) | H(22) | -410 | 7500 | 1451 | 48 |
| C(5) | 2945(2) | 5610(1) | 1265(1) | 50(1) | C(23) | 354(1) | 6241(1) | -208(1) | 35(1) |
| C(6) | 2785(2) | 4950(1) | 997(2) | 62(1) | C(24) | -317(7) | 6118(4) | -947(7) | 64(3) |
| H(6) | 2527 | 4631 | 1354 | 74 | H(24A) | -14 | 6266 | -1438 | 95 |
| C(7) | 2989(2) | 4754(1) | 237(2) | 66(1) | H(24B) | -886 | 6375 | -870 | 95 |
| H(7) | 2872 | 4305 | 71 | 80 | H(24C) | -463 | 5638 | -986 | 95 |
| C(8) | 3368(2) | 5211(1) | -288(2) | 59(1) | C(25) | -119(7) | 6016(3) | 537(6) | 63(2) |
| H(8) | 3524 | 5068 | -811 | 71 | H(25A) | -745 | 6199 | 549 | 95 |
| C(9) | 3527(1) | 5877(1) | -71(1) | 44(1) | H(25B) | 224 | 6178 | 1001 | 95 |
| C(10) | 2774(2) | 5781(1) | 2139(2) | 65(1) | H(25C) | -145 | 5524 | 548 | 95 |
| H(10) | 2957 | 6260 | 2229 | 78 | C(26) | 1216(6) | 5848(6) | -368(6) | 48(2) |
| C(11) | 1770(2) | 5706(2) | 2357(2) | 102(1) | H(26A) | 1060 | 5373 | -443 | 72 |
| H(11A) | 1401 | 6019 | 2039 | 154 | H(26B) | 1637 | 5894 | 84 | 72 |
| H(11B) | 1688 | 5806 | 2924 | 154 | H(26C) | 1513 | 6021 | -851 | 72 |
| H(11C) | 1571 | 5244 | 2250 | 154 | C(24') | -565(4) | 6090(3) | -537(6) | 52(2) |
| C(12) | 3344(2) | 5333(2) | 2704(2) | 79(1) | H(24D) | -636 | 6314 | -1053 | 78 |
| H(12A) | 3124 | 4868 | 2670 | 118 | H(24E) | -1039 | 6252 | -169 | 78 |
| H(12B) | 3277 | 5496 | 3252 | 118 | H(24F) | -629 | 5603 | -608 | 78 |
| H(12C) | 3994 | 5351 | 2549 | 118 | C(25') | 432(8) | 6009(2) | 678(3) | 61(2) |
| C(13) | 3951(1) | 6370(1) | -658(1) | 49(1) | H(25D) | 322 | 5523 | 711 | 91 |
| H(13) | 3643 | 6814 | -569 | 59 | H(25E) | -27 | 6247 | 1000 | 91 |
| C(14) | 4980(2) | 6477(2) | -510(2) | 91(1) | H(25F) | 1050 | 6110 | 879 | 91 |
| H(14A) | 5298 | 6043 | -530 | 136 | C(26') | 1115(6) | 5895(5) | -670(6) | 51(2) |
| H(14B) | 5070 | 6682 | 15 | 136 | H(26D) | 1024 | 5407 | -650 | 76 |
| H(14C) | 5231 | 6775 | -922 | 136 | H(26E) | 1712 | 6008 | -434 | 76 |
| C(15) | 3788(2) | 6182(2) | -1522(2) | 73(1) | H(26F) | 1101 | 6046 | -1226 | 76 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| C(27) | 380(3) | 7500 | -2316(2) | 99(2) | C(28) | 2053(3) | 6755(2) | -2851(2) | 93(1) |
| H(27A) | 132 | 7186 | -1919 | 148 | H(28A) | 1834 | 6798 | -3401 | 140 |
| H(27B) | 185 | 7360 | -2849 | 148 | H(28B) | 1800 | 6344 | -2614 | 140 |
| H(27C) | 148 | 7954 | -2206 | 148 | H(28C) | 2727 | 6732 | -2848 | 140 |

Tabelle 53: Anisotropic displacement parameters (Å$^2$ × 10$^3$) for jus_333m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ga(1) | 22(1) | 32(1) | 23(1) | 0 | -4(1) | 0 | C(13) | 36(1) | 71(2) | 41(1) | 7(1) | 6(1) | 13(1) |
| Cl(1) | 33(1) | 55(1) | 25(1) | 0 | 0(1) | 0 | C(14) | 41(1) | 167(4) | 64(2) | 32(2) | 9(1) | 1(2) |
| Si(1) | 22(1) | 19(1) | 23(1) | 0 | -2(1) | 0 | C(15) | 76(2) | 97(2) | 45(1) | 5(1) | 6(1) | 9(2) |
| Si(2) | 43(1) | 36(1) | 24(1) | 0 | -3(1) | 0 | C(16) | 24(1) | 23(1) | 22(1) | 0 | -3(1) | 0 |
| N(1) | 29(1) | 51(1) | 31(1) | 7(1) | -3(1) | 10(1) | C(17) | 25(1) | 27(1) | 32(1) | 0 | 2(1) | 0 |
| N(2) | 26(1) | 18(1) | 32(1) | 0(1) | -2(1) | -2(1) | C(18) | 28(1) | 53(2) | 40(1) | 0 | -1(1) | 0 |
| N(3) | 32(1) | 36(1) | 24(1) | 0 | 0(1) | 0 | C(19) | 26(1) | 82(2) | 65(2) | 0 | 2(1) | 0 |
| C(1) | 30(1) | 84(2) | 35(1) | 13(1) | -4(1) | 12(1) | C(20) | 36(2) | 78(2) | 75(2) | 0 | 25(2) | 0 |
| C(2) | 29(1) | 107(3) | 37(2) | 0 | -14(1) | 0 | C(21) | 53(2) | 66(2) | 46(2) | 0 | 19(1) | 0 |
| C(3) | 37(1) | 115(2) | 74(2) | 36(2) | -10(1) | 24(1) | C(22) | 40(1) | 46(2) | 34(1) | 0 | 3(1) | 0 |
| C(4) | 34(1) | 44(1) | 42(1) | 11(1) | 0(1) | 18(1) | C(23) | 35(1) | 16(1) | 54(1) | 1(1) | 0(1) | -4(1) |
| C(5) | 50(1) | 47(1) | 52(1) | 18(1) | 6(1) | 22(1) | C(24) | 59(5) | 34(3) | 98(6) | -20(4) | -36(4) | 0(3) |
| C(6) | 67(2) | 45(1) | 75(2) | 23(1) | 5(1) | 20(1) | C(25) | 64(5) | 29(2) | 98(5) | 23(3) | 28(4) | 0(3) |
| C(7) | 76(2) | 44(1) | 80(2) | 4(1) | -4(1) | 23(1) | C(26) | 43(3) | 22(2) | 79(6) | 2(4) | 1(3) | 3(2) |
| C(8) | 61(1) | 56(1) | 59(1) | -4(1) | -1(1) | 28(1) | C(24') | 44(3) | 26(2) | 87(5) | -7(3) | -9(3) | -11(2) |
| C(9) | 34(1) | 54(1) | 43(1) | 6(1) | 1(1) | 20(1) | C(25') | 95(6) | 30(2) | 58(3) | 16(2) | 4(3) | -3(3) |
| C(10) | 80(2) | 56(1) | 58(1) | 26(1) | 23(1) | 25(1) | C(26') | 50(3) | 19(3) | 85(5) | -9(4) | 12(4) | 3(2) |
| C(11) | 78(2) | 132(3) | 97(2) | 57(2) | 36(2) | 52(2) | C(27) | 50(2) | 206(6) | 40(2) | 0 | -18(2) | 0 |
| C(12) | 89(2) | 88(2) | 60(2) | 32(2) | 4(1) | 19(2) | C(28) | 157(3) | 79(2) | 44(1) | -24(1) | -30(2) | 53(2) |

Tabelle 54: Bond lengths [Å] for jus_333m. #1 x,-y+3/2,z

| | | | | | |
|---|---|---|---|---|---|
| Ga(1)-N(1) | 1.9821(15) | N(2)-C(23) | 1.4800(19) | C(13)-C(14) | 1.530(3) |
| Ga(1)-N(1)#1 | 1.9821(15) | C(1)-C(2) | 1.382(3) | C(16)-C(17) | 1.491(3) |
| Ga(1)-Cl(1) | 2.2785(6) | C(1)-C(3) | 1.521(3) | C(17)-C(18) | 1.381(3) |
| Ga(1)-Si(1) | 2.4441(6) | C(4)-C(5) | 1.403(3) | C(17)-C(22) | 1.396(4) |
| Si(1)-N(3) | 1.612(2) | C(4)-C(9) | 1.410(3) | C(18)-C(19) | 1.392(4) |
| Si(1)-N(2)#1 | 1.8576(14) | C(5)-C(6) | 1.408(4) | C(19)-C(20) | 1.385(5) |
| Si(1)-N(2) | 1.8576(14) | C(5)-C(10) | 1.524(3) | C(20)-C(21) | 1.374(5) |
| Si(1)-C(16) | 2.315(2) | C(6)-C(7) | 1.366(4) | C(21)-C(22) | 1.390(4) |
| Si(2)-N(3) | 1.683(2) | C(7)-C(8) | 1.380(4) | C(23)-C(24') | 1.476(6) |
| Si(2)-C(28)#1 | 1.868(3) | C(8)-C(9) | 1.393(3) | C(23)-C(25) | 1.494(6) |
| Si(2)-C(28) | 1.868(3) | C(9)-C(13) | 1.520(3) | C(23)-C(26) | 1.500(9) |
| Si(2)-C(27) | 1.874(4) | C(10)-C(11) | 1.511(4) | C(23)-C(26') | 1.516(8) |
| N(1)-C(1) | 1.338(2) | C(10)-C(12) | 1.543(3) | C(23)-C(25') | 1.561(5) |
| N(1)-C(4) | 1.453(3) | C(13)-C(15) | 1.517(3) | C(23)-C(24) | 1.596(7) |
| N(2)-C(16) | 1.3315(18) | | | | |

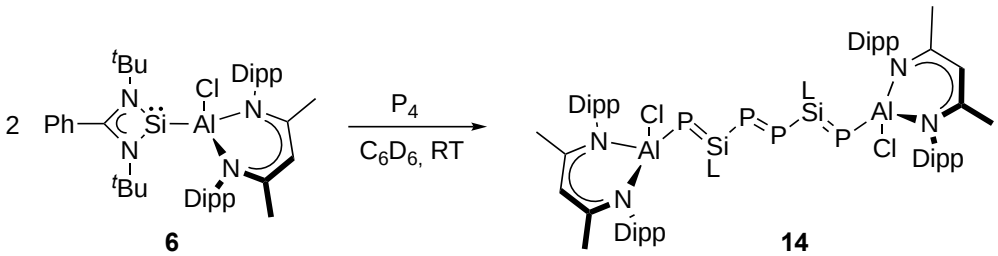
Tabelle 55: Bond angles [°] for jus_333m.

| | | | | | |
|---|---|---|---|---|---|
| N(1)-Ga(1)-N(1)#1 | 95.92(10) | C(16)-N(2)-Si(1) | 91.55(10) | N(2)-C(16)-C(17) | 126.72(9) |
| N(1)-Ga(1)-Cl(1) | 103.19(5) | C(23)-N(2)-Si(1) | 138.70(11) | N(2)#1-C(16)-C(17) | 126.72(9) |
| N(1)#1-Ga(1)-Cl(1) | 103.19(5) | Si(1)-N(3)-Si(2) | 134.34(14) | N(2)-C(16)-Si(1) | 53.34(9) |
| N(1)-Ga(1)-Si(1) | 123.03(5) | N(1)-C(1)-C(2) | 124.6(2) | N(2)#1-C(16)-Si(1) | 53.34(9) |
| N(1)#1-Ga(1)-Si(1) | 123.03(5) | N(1)-C(1)-C(3) | 120.5(2) | C(17)-C(16)-Si(1) | 174.13(16) |
| Cl(1)-Ga(1)-Si(1) | 105.59(2) | C(2)-C(1)-C(3) | 114.8(2) | C(18)-C(17)-C(22) | 120.1(2) |
| N(3)-Si(1)-N(2)#1 | 121.70(7) | C(1)-C(2)-C(1)#1 | 129.2(3) | C(18)-C(17)-C(16) | 121.8(2) |
| N(3)-Si(1)-N(2) | 121.70(7) | C(5)-C(4)-C(9) | 121.5(2) | C(22)-C(17)-C(16) | 118.0(2) |
| N(2)#1-Si(1)-N(2) | 70.11(8) | C(5)-C(4)-N(1) | 119.91(18) | C(17)-C(18)-C(19) | 119.7(3) |
| N(3)-Si(1)-C(16) | 131.88(9) | C(9)-C(4)-N(1) | 118.48(18) | C(20)-C(19)-C(18) | 119.9(3) |
| N(2)#1-Si(1)-C(16) | 35.10(4) | C(4)-C(5)-C(6) | 117.3(2) | C(21)-C(20)-C(19) | 120.6(3) |
| N(2)-Si(1)-C(16) | 35.10(4) | C(4)-C(5)-C(10) | 123.4(2) | C(20)-C(21)-C(22) | 120.0(3) |
| N(3)-Si(1)-Ga(1) | 118.39(8) | C(6)-C(5)-C(10) | 119.2(2) | C(21)-C(22)-C(17) | 119.7(3) |
| N(2)#1-Si(1)-Ga(1) | 107.60(5) | C(7)-C(6)-C(5) | 122.0(2) | C(24')-C(23)-N(2) | 114.4(3) |
| N(2)-Si(1)-Ga(1) | 107.60(5) | C(6)-C(7)-C(8) | 119.6(3) | N(2)-C(23)-C(25) | 114.3(3) |
| C(16)-Si(1)-Ga(1) | 109.73(6) | C(7)-C(8)-C(9) | 121.7(2) | N(2)-C(23)-C(26) | 107.5(5) |
| N(3)-Si(2)-C(28)#1 | 112.94(10) | C(8)-C(9)-C(4) | 117.8(2) | C(25)-C(23)-C(26) | 112.2(4) |
| N(3)-Si(2)-C(28) | 112.94(10) | C(8)-C(9)-C(13) | 120.7(2) | C(24')-C(23)-C(26') | 112.0(4) |
| C(28)#1-Si(2)-C(28) | 105.0(2) | C(4)-C(9)-C(13) | 121.5(2) | N(2)-C(23)-C(26') | 105.0(4) |
| N(3)-Si(2)-C(27) | 113.57(14) | C(11)-C(10)-C(5) | 111.6(3) | C(24')-C(23)-C(25') | 111.2(3) |
| C(28)#1-Si(2)-C(27) | 105.86(15) | C(11)-C(10)-C(12) | 108.2(2) | N(2)-C(23)-C(25') | 106.3(2) |
| C(28)-Si(2)-C(27) | 105.86(15) | C(5)-C(10)-C(12) | 112.1(2) | C(26')-C(23)-C(25') | 107.5(4) |
| C(1)-N(1)-C(4) | 117.34(17) | C(15)-C(13)-C(9) | 113.4(2) | N(2)-C(23)-C(24) | 106.8(3) |
| C(1)-N(1)-Ga(1) | 121.14(16) | C(15)-C(13)-C(14) | 110.0(2) | C(25)-C(23)-C(24) | 108.8(4) |
| C(4)-N(1)-Ga(1) | 121.50(11) | C(9)-C(13)-C(14) | 112.4(2) | C(26)-C(23)-C(24) | 106.9(5) |
| C(16)-N(2)-C(23) | 129.73(14) | N(2)-C(16)-N(2)#1 | 106.51(18) | | |

#1 x,-y+3/2,z

## 14. DDP(Cl)Al–P=Si(L)P=PSi(L)P–Al(Cl)DDP 14

### 14.1. Synthese DDP(Cl)Al–P=Si(L)P=PSi(L)P–Al(Cl)DDP **14**




**6** **14**

Dipp = 2,6-$^{i}$Pr$_2$C$_6$H$_3$
L = PhC(N$^{t}$Bu)$_2$

Es wurden 30 mg PhC(N$^{t}$Bu)$_2$SiAl(Cl)DDP 6 (0.040 mmol) und 2.5 mg $P_4$ (0.020 mmol) in 0.4 mL Toluol gelöst, wobei sich die Lösung schlagartig grün färbte. Die Lösung wurde für einen Tag bei Raumtemperatur gerührt, auf ein Drittel eingeengt, alle Rückstände kurz in der Hitze gelöst und daraufhin bei 4 °C gelagert, um dunkelgrüne Kristalle von 14 zu erhalten.

Ausbeute: 16.9 mg (0.011 mmol, 53 %).

Smp. 198 °C (Zersetzung).

Elementaranalyse von $C_{88}H_{128}Al_2Cl_2N_8P_4Si_2$: gefunden (berechnet) C 65.9 (65.94), H 8.5 (8.05), N 6.4 (6.99) %.

IR: ν 3047, 2951, 2856, 1520, 1457, 1433, 1378, 1358, 1312, 1250, 1169, 1085, 1016, 1085, 1016, 927, 864, 793, 756, 706, 635, 574, 511, 449 cm$^{-1}$.

$^{1}$H NMR ($C_6D_6$, 400 MHz): δ 8.13, 7.36, 7.27, 7.21, 7.21, 7.20, 6.95, 6.83, 5.16 (s, 2H, γ-C*H)*, 3.93 (sept, $^{3}J_{HH}$ = 6.7 Hz, 4 H, -C*H*(CH$_3$)$_2$), 3.72 (sept, $^{3}J_{HH}$ = 6.7 Hz, 4 H, -C*H*(CH$_3$)$_2$), 1.80 (d, $^{3}J_{HH}$ = 6.7 Hz, 12 H, -CH(C*H$_3$*)$_2$)), 1.71 (s, 12 H, ArNCC*H$_3$*), 1.46 (d, $^{3}J_{HH}$ = 6.5 Hz, 12 H, -CH(C*H$_3$*)$_2$)), 1.28 (d, $^{3}J_{HH}$ = 6.8 Hz, 12 H, -CH(C*H$_3$*)$_2$)), 1.20 (d, $^{3}J_{HH}$ = 6.8 Hz, 12 H, -CH(C*H$_3$*)$_2$)), 1.08 (s, 36 H, C(C*H$_3$*)$_3$).

$^{13}$C NMR ($C_6D_6$, 101 MHz): δ 170.0 (N*C*CCH(CH$_3$)$_2$), 145.9 (*C*CH(CH$_3$)$_2$), 144.6 (*C*CH(CH$_3$)$_2$), 142.5, 133.0, 132.3, 130.2, 127.8, 127.1, 124.8, 124.4 (*C*$_6$H$_3$), 99.1 (γ-*C*H-), 55.6 (*C*(CH$_3$)$_3$), 31.9 (C(*C*H$_3$)$_3$), 30.1 (-*C*H(CH$_3$)$_2$), 28.4 (-CH(*C*H$_3$)$_2$), 25.4 (CH(*C*H$_3$)$_2$), 24.9(CH(*C*H$_3$)$_2$), 24.5 (ArNC*C*H$_3$).

$^{29}$Si NMR ($C_6D_6$, 79 MHz): δ Es waren keine Signale sichtbar.

$^{31}$P NMR ($C_6D_6$, 243 MHz) δ –163.00, – 298.77 ppm.

## 14.2. Spektren DDP(Cl)Al–P=Si(L)P=PSi(L)P–Al(Cl)DDP **14**

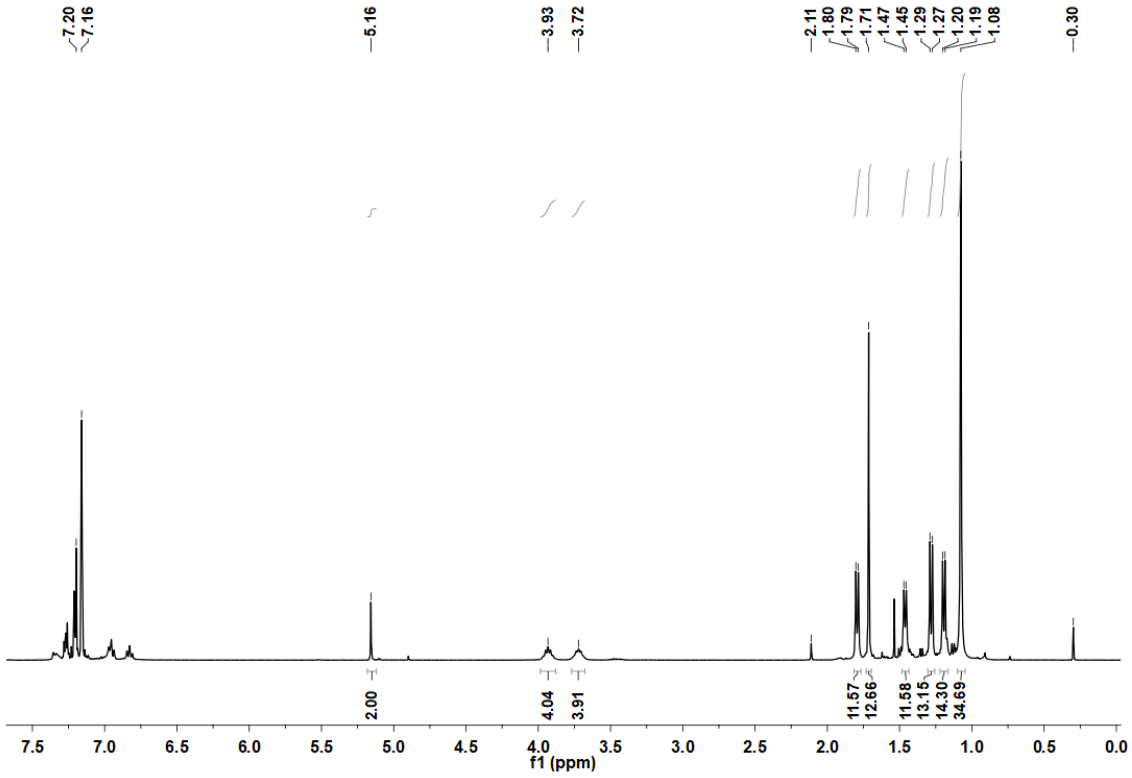



Abbildung 56: $^{1}$H-NMR-Spektrum von DDP(Cl)Al–P=Si(L)P=PSi(L)P–Al(Cl)DDP **14** in $C_6D_6$.

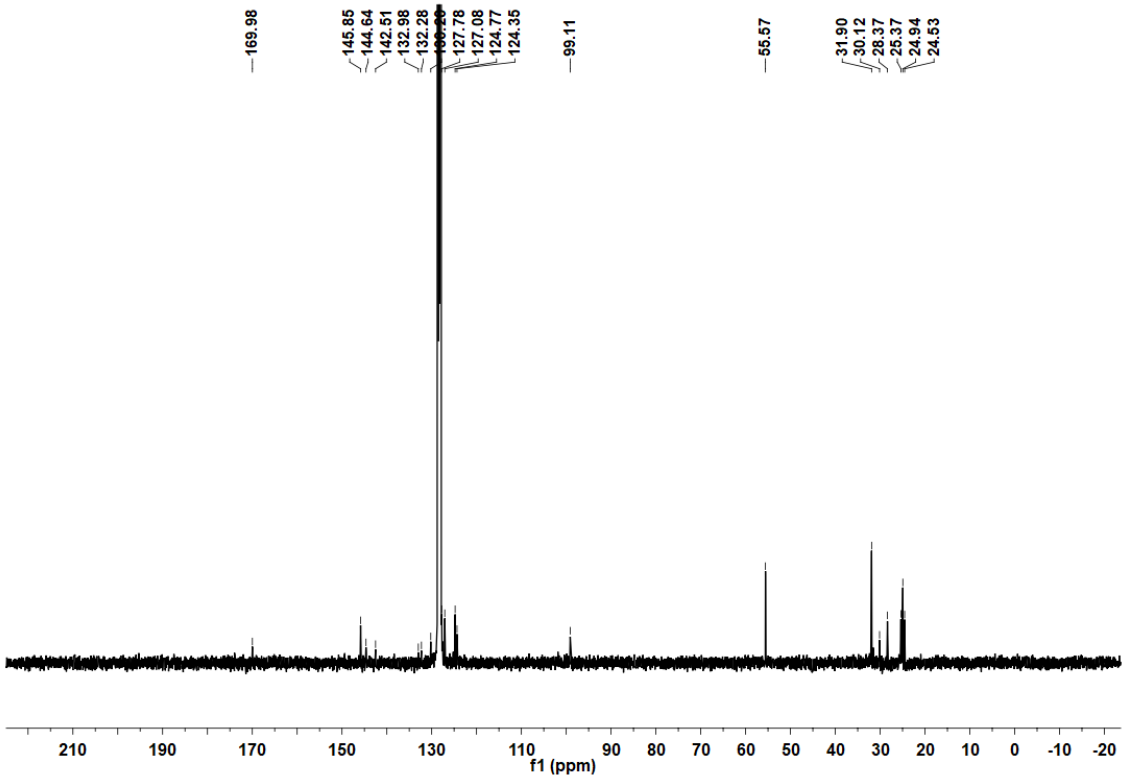



Abbildung 57: $^{13}$C-NMR-Spektrum von DDP(Cl)Al–P=Si(L)P=PSi(L)P–Al(Cl)DDP **14** in $C_6D_6$.

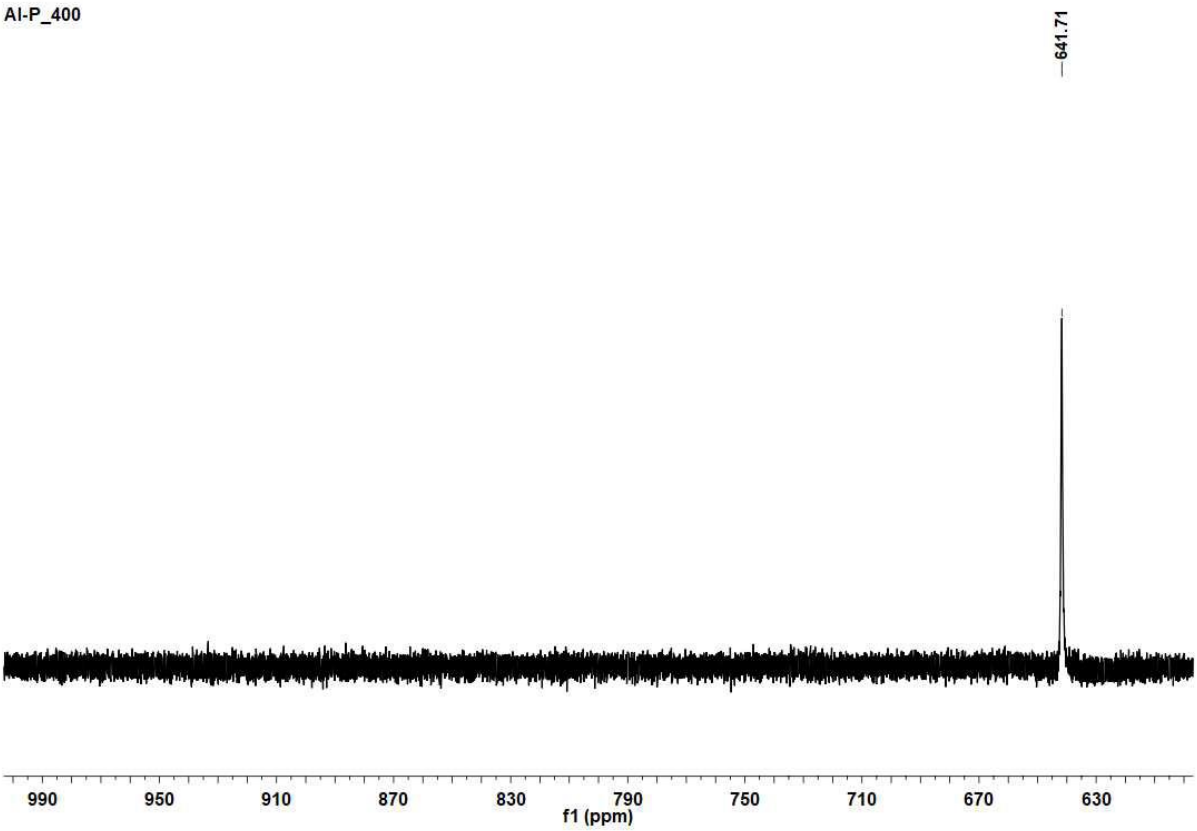



Abbildung 58: $^{31}$P-NMR-Spektrum von DDP(Cl)Al–P=Si(L)P=PSi(L)P–Al(Cl)DDP **14** in $C_6D_6$.

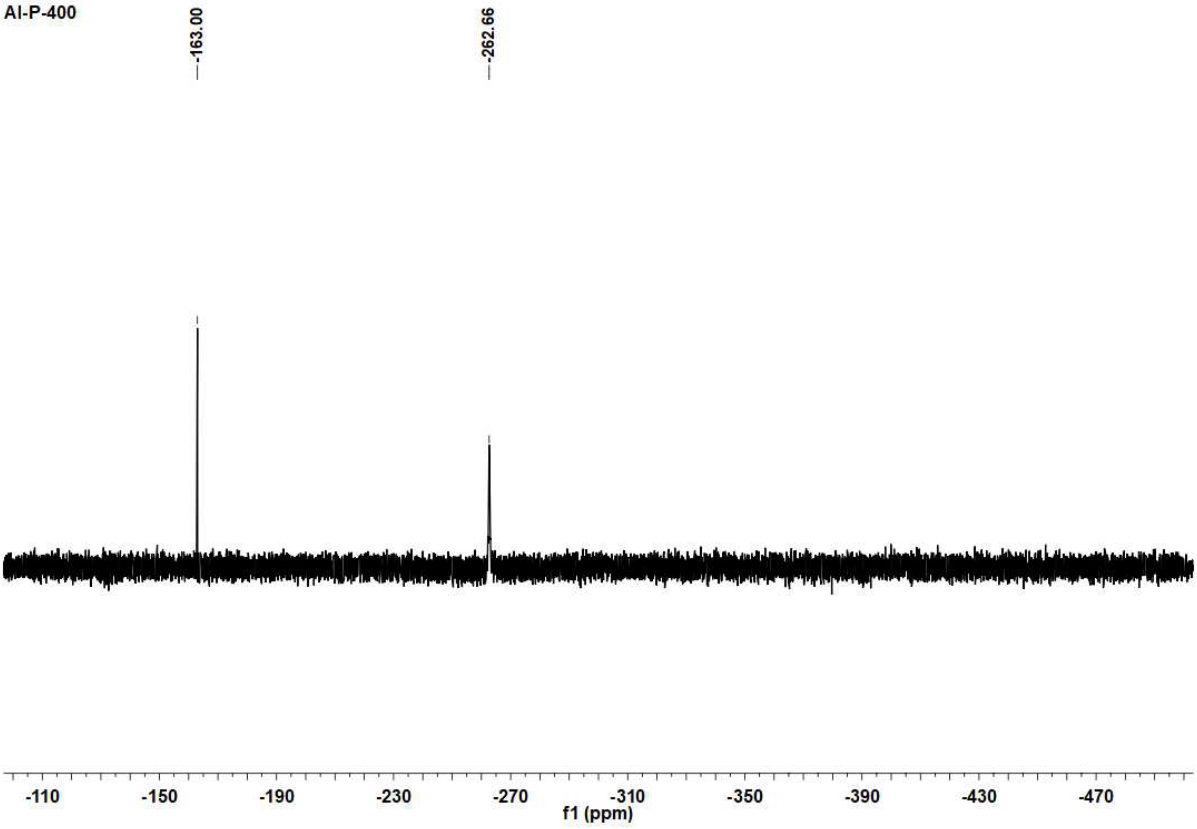



Abbildung 59: $^{31}$P-NMR-Spektrum von DDP(Cl)Al–P=Si(L)P=PSi(L)P–Al(Cl)DDP **14** in $C_6D_6$.

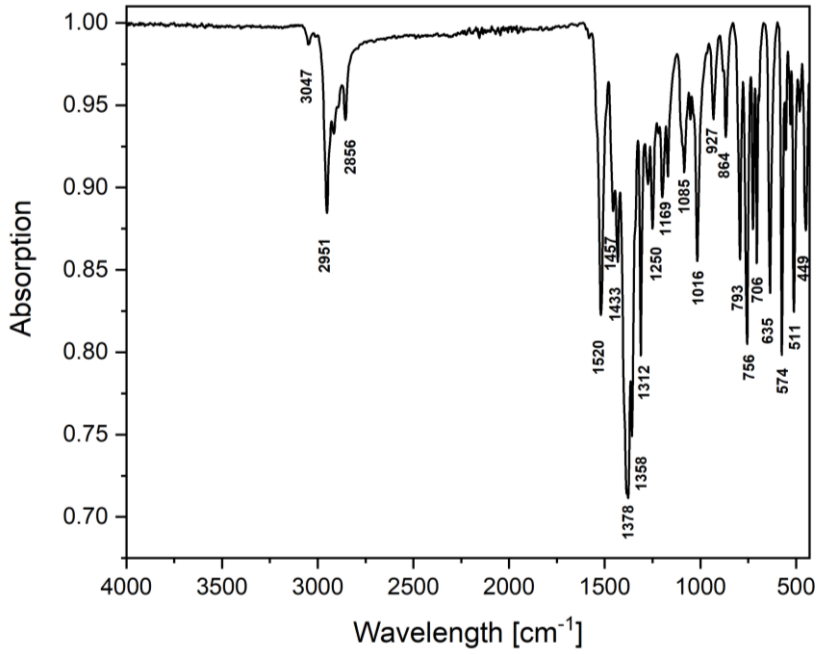



Abbildung 60: ATR-IR Spektrum von DDP(Cl)Al–P=Si(L)P=PSi(L)P–Al(Cl)DDP **14**.

## 14.3. Kristallografische Daten DDP(Cl)Al–P=Si(L)P=PSi(L)P–Al(Cl)DDP **14**

Tabelle 56: Crystal structure data

| **Identification code** | **jus_355m_sq** |
|---|---|
| Empirical formula | C124 H164 Al4 Cl2 N8 P4 Si2 |
| Formula weight | 2125.50 |
| Density (calculated) | 1.182 g·cm$^{-3}$ |
| *F*(000) | 1138 |
| Temperature | 100(2) K |
| Crystal size | 0.472 × 0.444 × 0.314 mm |
| Crystal colour | dark green |
| Crystal description | block |
| Wavelength | 0.71073 Å |
| Crystal system | triclinic |
| Space group | *P -1* |
| Unit cell dimensions | |
| *a* [Å] | 12.6277(10) |
| *b* [Å] | 12.8008(10) |
| *c* [Å] | 18.7197(14) |
| *α* [°] | 82.192(3) |
| *β* [°] | 87.250(3) |
| *γ* [°] | 85.151(3) |
| Volume | 2985.1(4) Å$^3$ |
| *Z* | 1 |
| Cell measurement reflections used | 9227 |
| Cell measurement *θ* min/max | 2.19°/33.51° |
| Diffractometer control software | BRUKER APEX3(v2019.1-0) |
| Diffractometer measurement device | Bruker D8 KAPPA II (APEX II detector) |
| Diffractometer measurement method | Data collection strategy APEX 3/QUEEN |
| *θ* range for data collection | 1.611°- 33.720° |
| Completeness to *θ* = 25.242° | 98.6% |
| Completeness to $\theta_{max}$ = 33.720° | 96.6% |
| Index ranges | -19 ≤ *h* ≤ 19 |
| | -19 ≤ *k* ≤ 19 |
| | -28 ≤ *l* ≤ 29 |
| Computing data reduction | BRUKER APEX3(v2019.1-0) |
| Absorption coefficient | 0.208 mm$^{-1}$ |
| Absorption correction | Semi-empirical from equivalents |
| Computation absorption correction | SADABS |
| Max./min. Transmission | 0.75/0.66 |
| $R_{merg}$ before/after correction | 0.1025/0.0578 |
| Computing structure solution | BRUKER APEX3(v2019.1-0) |
| Computing structure refinement | SHELXL-2017/1 (Sheldrick, 2017) |

| | |
|---|---|
| Refinement method | Full-matrix least-squares on $F^2$ |
| Reflections collected | 205751 |
| Independent reflections | 23013 |
| $R_{int}$ | 0.0382 |
| Reflections with $I > 2\sigma(I)$ | 18655 |
| Restraints | 69 |
| Parameter | 559 |
| GooF | 1.025 |
| Weighting details | $w = 1/[\sigma^2(F_{obs}^2) + (0.0602P)^2 + 1.4002P]$ |
| | where $P = (F_{obs}^2 + 2F_{calc}^2)/3$ |
| $R_1$ [$I > 2\sigma(I)$] | 0.0440 |
| $wR_2$ [$I > 2\sigma(I)$] | 0.1146 |
| $R_1$ [all data] | 0.0576 |
| $wR_2$ [all data] | 0.1276 |
| Absolute structure parameter | |
| Largest diff. peak and hole | 0.554/-0.351 |

Tabelle 57: Atomic coordinates ( × $10^4$) and equivalent isotropic displacement parameters (Å$^2$ × $10^3$) for jus_355m_sq. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | *x* | *y* | *z* | $U_{eq}$ | | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|---|---|---|---|---|
| Cl(1) | 9138(1) | 5603(1) | 2690(1) | 24(1) | H(14A) | 6863 | 6629 | 3740 | 70 |
| Si(1) | 10938(1) | 6560(1) | 4166(1) | 15(1) | H(14B) | 6829 | 7796 | 3962 | 70 |
| P(1) | 10871(1) | 7689(1) | 3240(1) | 22(1) | H(14C) | 7946 | 7174 | 3792 | 70 |
| P(2) | 9672(1) | 5384(2) | 4544(1) | 23(1) | C(15) | 9971(1) | 10075(1) | 1590(1) | 34(1) |
| P(2') | 10013(7) | 5084(4) | 4458(1) | 30(1) | H(15) | 10228 | 9380 | 1430 | 41 |
| Al(1) | 10017(1) | 6996(1) | 2401(1) | 15(1) | C(16) | 9676(2) | 10848(2) | 918(1) | 58(1) |
| N(1) | 9045(1) | 8041(1) | 1908(1) | 16(1) | H(16A) | 10312 | 10958 | 605 | 88 |
| N(2) | 10822(1) | 6662(1) | 1550(1) | 17(1) | H(16B) | 9387 | 11526 | 1064 | 88 |
| N(3) | 11208(1) | 7126(1) | 4981(1) | 18(1) | H(16C) | 9141 | 10556 | 657 | 88 |
| N(4) | 12242(1) | 5996(1) | 4492(1) | 18(1) | C(17) | 10879(1) | 10469(1) | 1968(1) | 50(1) |
| C(1) | 8770(1) | 7955(1) | 1237(1) | 19(1) | H(17A) | 11499 | 10538 | 1634 | 75 |
| C(2) | 9344(1) | 7293(1) | 800(1) | 21(1) | H(17B) | 11068 | 9963 | 2392 | 75 |
| H(2) | 9014 | 7185 | 373 | 25 | H(17C) | 10651 | 11160 | 2119 | 75 |
| C(3) | 10356(1) | 6766(1) | 919(1) | 19(1) | C(18) | 11904(1) | 6178(1) | 1595(1) | 19(1) |
| C(4) | 7814(1) | 8607(1) | 918(1) | 29(1) | C(19) | 12100(1) | 5073(1) | 1646(1) | 21(1) |
| H(4A) | 7740 | 8463 | 422 | 44 | C(20) | 13158(1) | 4647(1) | 1678(1) | 26(1) |
| H(4B) | 7907 | 9360 | 915 | 44 | H(20) | 13307 | 3903 | 1713 | 32 |
| H(4C) | 7173 | 8421 | 1208 | 44 | C(21) | 13991(1) | 5288(1) | 1662(1) | 30(1) |
| C(5) | 10905(1) | 6340(1) | 272(1) | 28(1) | H(21) | 14704 | 4984 | 1680 | 36 |
| H(5A) | 10624 | 5669 | 210 | 41 | C(22) | 13780(1) | 6374(1) | 1618(1) | 28(1) |
| H(5B) | 11672 | 6224 | 346 | 41 | H(22) | 14355 | 6809 | 1610 | 34 |
| H(5C) | 10778 | 6850 | -161 | 41 | C(23) | 12741(1) | 6841(1) | 1585(1) | 22(1) |
| C(6) | 8561(1) | 8923(1) | 2247(1) | 18(1) | C(24) | 11221(1) | 4328(1) | 1667(1) | 24(1) |
| C(7) | 7670(1) | 8794(1) | 2721(1) | 23(1) | H(24) | 10530 | 4765 | 1596 | 29 |
| C(8) | 7253(1) | 9658(1) | 3057(1) | 32(1) | C(25) | 11376(1) | 3614(1) | 1069(1) | 34(1) |
| H(8) | 6659 | 9579 | 3386 | 38 | H(25A) | 11422 | 4053 | 599 | 51 |
| C(9) | 7687(1) | 10620(1) | 2920(1) | 36(1) | H(25B) | 10772 | 3178 | 1084 | 51 |
| H(9) | 7391 | 11199 | 3153 | 43 | H(25C) | 12034 | 3156 | 1142 | 51 |
| C(10) | 8554(1) | 10743(1) | 2444(1) | 32(1) | C(26) | 11151(1) | 3638(1) | 2400(1) | 33(1) |
| H(10) | 8843 | 11412 | 2348 | 38 | H(26A) | 10564 | 3182 | 2407 | 49 |
| C(11) | 9013(1) | 9900(1) | 2102(1) | 23(1) | H(26B) | 11025 | 4091 | 2783 | 49 |
| C(12) | 7139(1) | 7764(1) | 2871(1) | 28(1) | H(26C) | 11820 | 3199 | 2479 | 49 |
| H(12) | 7533 | 7248 | 2579 | 34 | C(27) | 12532(1) | 8035(1) | 1505(1) | 25(1) |
| C(13) | 5977(1) | 7902(1) | 2642(1) | 43(1) | H(27) | 11840 | 8198 | 1766 | 30 |
| H(13A) | 5950 | 8193 | 2131 | 65 | C(28) | 12412(2) | 8495(1) | 711(1) | 45(1) |
| H(13B) | 5567 | 8386 | 2933 | 65 | H(28A) | 13053 | 8288 | 432 | 67 |
| H(13C) | 5674 | 7213 | 2718 | 65 | H(28B) | 12313 | 9268 | 670 | 67 |
| C(14) | 7200(2) | 7299(1) | 3662(1) | 46(1) | H(28C) | 11792 | 8223 | 524 | 67 |
| C(29) | 13381(1) | 8574(1) | 1839(1) | 47(1) | H(40B) | 11908 | 8844 | 5416 | 50 |
| H(29A) | 14054 | 8497 | 1560 | 71 | H(40C) | 10774 | 9475 | 5520 | 50 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| H(29B) | 13479 | 8246 | 2338 | 71 | C(41) | 13166(1) | 5379(1) | 4201(1) | 22(1) |
| H(29C) | 13157 | 9327 | 1833 | 71 | C(42) | 12742(1) | 4848(1) | 3601(1) | 34(1) |
| C(30) | 12146(1) | 6564(1) | 5045(1) | 17(1) | H(42A) | 12432 | 5391 | 3233 | 51 |
| C(31) | 12892(1) | 6541(1) | 5637(1) | 20(1) | H(42B) | 12195 | 4382 | 3804 | 51 |
| C(32) | 12838(1) | 5740(1) | 6220(1) | 28(1) | H(42C) | 13326 | 4433 | 3382 | 51 |
| H(32) | 12358 | 5206 | 6221 | 33 | C(43) | 13628(1) | 4521(1) | 4772(1) | 34(1) |
| C(33) | 13489(1) | 5726(1) | 6799(1) | 37(1) | H(43A) | 13063 | 4084 | 4986 | 51 |
| H(33) | 13456 | 5180 | 7196 | 44 | H(43B) | 13935 | 4851 | 5149 | 51 |
| C(34) | 14182(1) | 6501(1) | 6802(1) | 41(1) | H(43C) | 14184 | 4076 | 4549 | 51 |
| H(34) | 14619 | 6491 | 7202 | 49 | C(44) | 14004(1) | 6122(1) | 3889(1) | 36(1) |
| C(35) | 14244(1) | 7293(1) | 6222(1) | 37(1) | H(44A) | 14599 | 5716 | 3673 | 55 |
| H(35) | 14727 | 7823 | 6225 | 44 | H(44B) | 14262 | 6470 | 4275 | 55 |
| C(36) | 13598(1) | 7316(1) | 5635(1) | 26(1) | H(44C) | 13689 | 6658 | 3518 | 55 |
| H(36) | 13642 | 7858 | 5236 | 32 | C12 | 5691(1) | 675(1) | 1521(1) | 46(1) |
| C(37) | 10655(1) | 7896(1) | 5420(1) | 22(1) | H12 | 6337 | 374 | 1728 | 56 |
| C(38) | 9507(1) | 8055(1) | 5180(1) | 34(1) | C22 | 5566(1) | 725(2) | 795(1) | 50(1) |
| H(38A) | 9500 | 8273 | 4658 | 52 | H22 | 6122 | 443 | 500 | 61 |
| H(38B) | 9118 | 8604 | 5429 | 52 | C32 | 4638(2) | 1183(2) | 490(1) | 58(1) |
| H(38C) | 9167 | 7390 | 5300 | 52 | H32 | 4562 | 1228 | -16 | 69 |
| C(39) | 10666(1) | 7489(1) | 6226(1) | 34(1) | C42 | 3823(2) | 1574(2) | 915(1) | 57(1) |
| H(39A) | 10387 | 6790 | 6312 | 50 | H42 | 3185 | 1888 | 702 | 68 |
| H(39B) | 10222 | 7981 | 6494 | 50 | C52 | 3929(2) | 1514(2) | 1646(1) | 52(1) |
| H(39C) | 11398 | 7436 | 6389 | 50 | H52 | 3362 | 1778 | 1940 | 63 |
| C(40) | 11172(1) | 8945(1) | 5260(1) | 33(1) | C62 | 4864(2) | 1067(1) | 1954(1) | 51(1) |
| H(40A) | 11167 | 9187 | 4740 | 50 | H62 | 4942 | 1028 | 2459 | 62 |

Tabelle 58: Anisotropic displacement parameters ($Å^2 \times 10^3$) for jus_355m_sq. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Cl(1) | 27(1) | 19(1) | 24(1) | 0(1) | 4(1) | -6(1) | C(21) | 21(1) | 38(1) | 29(1) | -3(1) | 0(1) | 9(1) |
| Si(1) | 16(1) | 17(1) | 13(1) | 1(1) | -2(1) | -2(1) | C(22) | 18(1) | 35(1) | 31(1) | -2(1) | 1(1) | 1(1) |
| P(1) | 30(1) | 19(1) | 17(1) | 4(1) | -7(1) | -7(1) | C(23) | 18(1) | 25(1) | 22(1) | 0(1) | 1(1) | -1(1) |
| P(2) | 18(1) | 27(1) | 21(1) | 10(1) | -7(1) | -7(1) | C(24) | 29(1) | 19(1) | 23(1) | -4(1) | 0(1) | 1(1) |
| P(2') | 50(3) | 28(1) | 12(1) | 1(1) | -2(1) | -24(2) | C(25) | 44(1) | 28(1) | 32(1) | -12(1) | -3(1) | 1(1) |
| Al(1) | 16(1) | 15(1) | 13(1) | 1(1) | -1(1) | 0(1) | C(26) | 43(1) | 24(1) | 30(1) | 1(1) | 1(1) | -3(1) |
| N(1) | 17(1) | 16(1) | 15(1) | 0(1) | -2(1) | 1(1) | C(27) | 20(1) | 23(1) | 30(1) | 1(1) | 2(1) | -4(1) |
| N(2) | 17(1) | 17(1) | 15(1) | 0(1) | -1(1) | 0(1) | C(28) | 63(1) | 32(1) | 35(1) | 9(1) | 8(1) | -4(1) |
| N(3) | 17(1) | 20(1) | 16(1) | -2(1) | -2(1) | 2(1) | C(29) | 31(1) | 32(1) | 81(1) | -9(1) | -13(1) | -7(1) |
| N(4) | 18(1) | 21(1) | 15(1) | -2(1) | -3(1) | 2(1) | C(30) | 17(1) | 18(1) | 14(1) | 1(1) | -2(1) | -1(1) |
| C(1) | 19(1) | 21(1) | 16(1) | 0(1) | -4(1) | 0(1) | C(31) | 18(1) | 25(1) | 16(1) | -2(1) | -4(1) | 1(1) |
| C(2) | 23(1) | 24(1) | 17(1) | -3(1) | -4(1) | 1(1) | C(32) | 28(1) | 34(1) | 18(1) | 3(1) | -3(1) | 3(1) |
| C(3) | 22(1) | 20(1) | 15(1) | -1(1) | -1(1) | -1(1) | C(33) | 39(1) | 51(1) | 18(1) | 0(1) | -8(1) | 14(1) |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| C(4) | 26(1) | 38(1) | 23(1) | -3(1) | -10(1) | 10(1) | C(34) | 36(1) | 59(1) | 29(1) | -18(1) | -17(1) | 16(1) |
| C(5) | 32(1) | 33(1) | 16(1) | -4(1) | 0(1) | 6(1) | C(35) | 28(1) | 46(1) | 41(1) | -20(1) | -13(1) | 1(1) |
| C(6) | 18(1) | 19(1) | 17(1) | -1(1) | -4(1) | 3(1) | C(36) | 22(1) | 30(1) | 28(1) | -7(1) | -6(1) | -3(1) |
| C(7) | 20(1) | 26(1) | 20(1) | -2(1) | -1(1) | 4(1) | C(37) | 24(1) | 20(1) | 21(1) | -4(1) | 0(1) | 2(1) |
| C(8) | 29(1) | 37(1) | 28(1) | -9(1) | 2(1) | 8(1) | C(38) | 23(1) | 40(1) | 42(1) | -16(1) | -1(1) | 8(1) |
| C(9) | 37(1) | 34(1) | 39(1) | -17(1) | -5(1) | 10(1) | C(39) | 48(1) | 32(1) | 20(1) | -5(1) | 5(1) | 5(1) |
| C(10) | 33(1) | 22(1) | 40(1) | -10(1) | -8(1) | 3(1) | C(40) | 37(1) | 19(1) | 44(1) | -2(1) | -3(1) | 0(1) |
| C(11) | 24(1) | 18(1) | 28(1) | -2(1) | -4(1) | 1(1) | C(41) | 21(1) | 25(1) | 18(1) | -1(1) | -1(1) | 6(1) |
| C(12) | 21(1) | 30(1) | 31(1) | 3(1) | 5(1) | 1(1) | C(42) | 38(1) | 39(1) | 27(1) | -14(1) | -6(1) | 11(1) |
| C(13) | 22(1) | 44(1) | 61(1) | 4(1) | 0(1) | -5(1) | C(43) | 37(1) | 34(1) | 25(1) | 1(1) | -4(1) | 17(1) |
| C(14) | 54(1) | 44(1) | 34(1) | 11(1) | 8(1) | 6(1) | C(44) | 29(1) | 41(1) | 36(1) | -1(1) | 11(1) | 0(1) |
| C(15) | 35(1) | 20(1) | 46(1) | 2(1) | 8(1) | -4(1) | C12 | 40(1) | 40(1) | 57(1) | 6(1) | -9(1) | -1(1) |
| C(16) | 70(1) | 57(1) | 44(1) | 16(1) | 4(1) | -17(1) | C22 | 39(1) | 55(1) | 51(1) | 6(1) | 4(1) | 9(1) |
| C(17) | 32(1) | 34(1) | 82(1) | 4(1) | -1(1) | -9(1) | C32 | 54(1) | 68(1) | 47(1) | -8(1) | -10(1) | 21(1) |
| C(18) | 18(1) | 21(1) | 17(1) | -1(1) | 1(1) | 2(1) | C42 | 46(1) | 61(1) | 65(1) | -23(1) | -15(1) | 19(1) |
| C(19) | 24(1) | 21(1) | 17(1) | -2(1) | 0(1) | 3(1) | C52 | 56(1) | 43(1) | 59(1) | -18(1) | 4(1) | 9(1) |
| C(20) | 27(1) | 26(1) | 25(1) | -4(1) | 0(1) | 8(1) | C62 | 72(1) | 37(1) | 45(1) | -6(1) | -8(1) | 0(1) |

Tabelle 59: Bond lengths [Å] for jus_355m_sq.

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Cl(1)-Al(1) | 2.1728(4) | C(1)-C(4) | 1.5093(15) | C(22)-C(23) | 1.3966(15) | C22-C32 | 1.379(2) |
| Si(1)-N(3) | 1.8337(9) | C(2)-C(3) | 1.4067(15) | C(23)-C(27) | 1.5167(16) | C32-C42 | 1.372(3) |
| Si(1)-N(4) | 1.8387(9) | C(3)-C(5) | 1.5104(15) | C(24)-C(26) | 1.5304(17) | C42-C52 | 1.373(3) |
| Si(1)-P(1) | 2.1010(4) | C(6)-C(7) | 1.4056(15) | C(24)-C(25) | 1.5344(16) | C52-C62 | 1.384(3) |
| Si(1)-P(2') | 2.2946(15) | C(6)-C(11) | 1.4066(15) | C(27)-C(29) | 1.5247(19) | | |
| Si(1)-C(30) | 2.2969(10) | C(7)-C(8) | 1.3981(16) | C(27)-C(28) | 1.5318(19) | | |
| Si(1)-P(2) | 2.3091(7) | C(7)-C(12) | 1.5159(17) | C(30)-C(31) | 1.4840(14) | | |
| P(1)-Al(1) | 2.2622(4) | C(8)-C(9) | 1.378(2) | C(31)-C(36) | 1.3880(16) | | |
| P(2)-P(2)#1 | 2.0270(10) | C(9)-C(10) | 1.384(2) | C(31)-C(32) | 1.3967(15) | | |
| P(2')-P(2')#1 | 2.011(3) | C(10)-C(11) | 1.3981(16) | C(32)-C(33) | 1.3894(17) | | |
| Al(1)-N(1) | 1.9082(8) | C(11)-C(15) | 1.5197(18) | C(33)-C(34) | 1.377(2) | | |
| Al(1)-N(2) | 1.9260(9) | C(12)-C(14) | 1.5212(19) | C(34)-C(35) | 1.385(2) | | |
| N(1)-C(1) | 1.3404(12) | C(12)-C(13) | 1.5374(19) | C(35)-C(36) | 1.3961(17) | | |
| N(1)-C(6) | 1.4454(13) | C(15)-C(17) | 1.530(2) | C(37)-C(38) | 1.5263(17) | | |
| N(2)-C(3) | 1.3318(12) | C(15)-C(16) | 1.531(2) | C(37)-C(40) | 1.5289(17) | | |
| N(2)-C(18) | 1.4526(13) | C(18)-C(19) | 1.4060(14) | C(37)-C(39) | 1.5293(16) | | |
| N(3)-C(30) | 1.3347(13) | C(18)-C(23) | 1.4084(15) | C(41)-C(43) | 1.5241(15) | | |
| N(3)-C(37) | 1.4771(13) | C(19)-C(20) | 1.4008(15) | C(41)-C(44) | 1.5250(18) | | |
| N(4)-C(30) | 1.3387(12) | C(19)-C(24) | 1.5192(17) | C(41)-C(42) | 1.5275(17) | | |
| N(4)-C(41) | 1.4772(13) | C(20)-C(21) | 1.3843(19) | C12-C22 | 1.367(3) | | |
| C(1)-C(2) | 1.3927(15) | C(21)-C(22) | 1.3851(18) | C12-C62 | 1.392(3) | | |

#1 -x+2,-y+1,-z+1

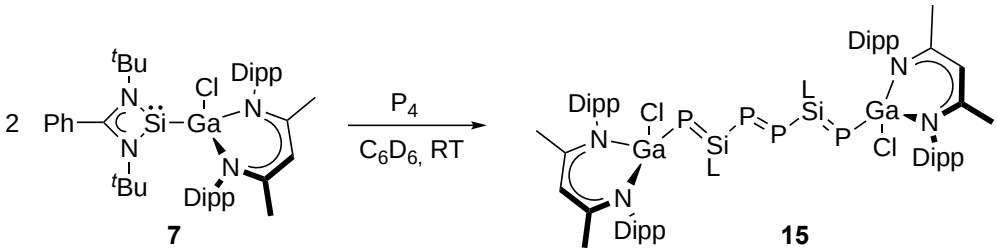
Tabelle 60: Bond angles [°] for jus_355m_sq.

| | | | | | |
|---|---|---|---|---|---|
| N(3)-Si(1)-N(4) | 71.15(4) | C(2)-C(1)-C(4) | 116.91(9) | C(26)-C(24)-C(25) | 109.18(10) |
| N(3)-Si(1)-P(1) | 112.81(3) | C(1)-C(2)-C(3) | 127.38(9) | C(23)-C(27)-C(29) | 112.97(10) |
| N(4)-Si(1)-P(1) | 119.19(3) | N(2)-C(3)-C(2) | 123.10(9) | C(23)-C(27)-C(28) | 110.84(11) |
| N(3)-Si(1)-P(2') | 110.53(6) | N(2)-C(3)-C(5) | 121.36(9) | C(29)-C(27)-C(28) | 110.31(12) |
| N(4)-Si(1)-P(2') | 98.5(2) | C(2)-C(3)-C(5) | 115.53(9) | N(3)-C(30)-N(4) | 106.11(8) |
| P(1)-Si(1)-P(2') | 129.41(11) | C(7)-C(6)-C(11) | 121.06(10) | N(3)-C(30)-C(31) | 126.14(9) |
| N(3)-Si(1)-C(30) | 35.51(4) | C(7)-C(6)-N(1) | 120.35(9) | N(4)-C(30)-C(31) | 127.67(9) |
| N(4)-Si(1)-C(30) | 35.64(4) | C(11)-C(6)-N(1) | 118.59(9) | N(3)-C(30)-Si(1) | 52.94(5) |
| P(1)-Si(1)-C(30) | 122.59(3) | C(8)-C(7)-C(6) | 118.36(11) | N(4)-C(30)-Si(1) | 53.16(5) |
| P(2')-Si(1)-C(30) | 107.80(12) | C(8)-C(7)-C(12) | 119.06(11) | C(31)-C(30)-Si(1) | 177.39(7) |
| N(3)-Si(1)-P(2) | 104.28(6) | C(6)-C(7)-C(12) | 122.58(10) | C(36)-C(31)-C(32) | 120.05(10) |
| N(4)-Si(1)-P(2) | 109.45(4) | C(9)-C(8)-C(7) | 121.23(12) | C(36)-C(31)-C(30) | 121.65(9) |
| P(1)-Si(1)-P(2) | 125.65(2) | C(8)-C(9)-C(10) | 119.92(11) | C(32)-C(31)-C(30) | 118.24(10) |
| C(30)-Si(1)-P(2) | 110.84(3) | C(9)-C(10)-C(11) | 121.18(12) | C(33)-C(32)-C(31) | 119.66(13) |
| Si(1)-P(1)-Al(1) | 107.172(16) | C(10)-C(11)-C(6) | 118.24(11) | C(34)-C(33)-C(32) | 120.33(13) |
| P(2)#1-P(2)-Si(1) | 100.38(4) | C(10)-C(11)-C(15) | 119.05(11) | C(33)-C(34)-C(35) | 120.24(12) |
| P(2')#1-P(2')-Si(1) | 101.58(10) | C(6)-C(11)-C(15) | 122.71(10) | C(34)-C(35)-C(36) | 120.14(14) |
| N(1)-Al(1)-N(2) | 95.91(4) | C(7)-C(12)-C(14) | 111.37(12) | C(31)-C(36)-C(35) | 119.57(12) |
| N(1)-Al(1)-Cl(1) | 105.74(3) | C(7)-C(12)-C(13) | 111.68(10) | N(3)-C(37)-C(38) | 106.27(9) |
| N(2)-Al(1)-Cl(1) | 102.06(3) | C(14)-C(12)-C(13) | 110.29(12) | N(3)-C(37)-C(40) | 109.15(9) |
| N(1)-Al(1)-P(1) | 110.64(3) | C(11)-C(15)-C(17) | 111.15(13) | C(38)-C(37)-C(40) | 109.47(10) |
| N(2)-Al(1)-P(1) | 117.83(3) | C(11)-C(15)-C(16) | 111.35(13) | N(3)-C(37)-C(39) | 111.87(9) |
| Cl(1)-Al(1)-P(1) | 121.211(16) | C(17)-C(15)-C(16) | 110.22(13) | C(38)-C(37)-C(39) | 109.32(11) |
| C(1)-N(1)-C(6) | 118.08(8) | C(19)-C(18)-C(23) | 121.35(10) | C(40)-C(37)-C(39) | 110.66(10) |
| C(1)-N(1)-Al(1) | 120.11(7) | C(19)-C(18)-N(2) | 120.26(9) | N(4)-C(41)-C(43) | 111.60(9) |
| C(6)-N(1)-Al(1) | 121.78(6) | C(23)-C(18)-N(2) | 118.39(9) | N(4)-C(41)-C(44) | 109.59(10) |
| C(3)-N(2)-C(18) | 118.33(8) | C(20)-C(19)-C(18) | 118.00(11) | C(43)-C(41)-C(44) | 111.19(11) |
| C(3)-N(2)-Al(1) | 120.13(7) | C(20)-C(19)-C(24) | 118.90(10) | N(4)-C(41)-C(42) | 105.79(9) |
| C(18)-N(2)-Al(1) | 121.15(6) | C(18)-C(19)-C(24) | 123.10(10) | C(43)-C(41)-C(42) | 108.58(10) |
| C(30)-N(3)-C(37) | 132.00(9) | C(21)-C(20)-C(19) | 121.44(11) | C(44)-C(41)-C(42) | 109.94(11) |
| C(30)-N(3)-Si(1) | 91.54(6) | C(20)-C(21)-C(22) | 119.65(11) | C22-C12-C62 | 119.62(16) |
| C(37)-N(3)-Si(1) | 136.46(7) | C(21)-C(22)-C(23) | 121.35(12) | C12-C22-C32 | 120.27(17) |
| C(30)-N(4)-C(41) | 131.31(9) | C(22)-C(23)-C(18) | 118.20(10) | C42-C32-C22 | 120.28(18) |
| C(30)-N(4)-Si(1) | 91.20(6) | C(22)-C(23)-C(27) | 120.43(10) | C32-C42-C52 | 120.12(17) |
| C(41)-N(4)-Si(1) | 136.07(7) | C(18)-C(23)-C(27) | 121.31(9) | C42-C52-C62 | 119.87(17) |
| N(1)-C(1)-C(2) | 122.97(9) | C(19)-C(24)-C(26) | 110.91(10) | C52-C62-C12 | 119.82(17) |
| N(1)-C(1)-C(4) | 120.12(9) | C(19)-C(24)-C(25) | 112.15(10) | | |

#1 -x+2,-y+1,-z+1

## 15. DDP(Cl)Ga–P=Si(L)P=PSi(L)P–Ga(Cl)DDP 15

### 15.1. Synthese DDP(Cl)Ga–P=Si(L)P=PSi(L)P–Ga(Cl)DDP **15**




Dipp = 2,6-$^{i}Pr_2C_6H_3$
L = PhC(N$^{t}$Bu)$_2$

Es wurden 60 mg PhC(N$^{t}$Bu)$_2$SiGa(Cl)DDP 7 (0.077 mmol) und 4.8 mg $P_4$ (0.038 mmol) in 0.7 mL Toluol gelöst, wobei sich die Lösung schlagartig grün färbte. Die Lösung wurde für zwei Stunden bei Raumtemperatur gerührt, auf die Hälfte eingeengt, alle Rückstände kurz in der Hitze gelöst und daraufhin bei Raumtemperatur gelagert, um dunkelgrüne Kristalle von 15 zu erhalten.

Ausbeute: 43 mg (0.025 mmol, 67 %).

Smp. 205 °C.

Elementaranalyse von $C_{88}H_{128}Cl_2Ga_2N_8P_4Si_2$: gefunden (berechnet) C 61.8 (62.6), H 8.21 (7.64), N 5.44 (6.64) %.

IR: ν 3052, 2953, 2917, 2854, 1546, 1519, 1433, 1388, 1358, 1312, 1255, 1199, 1171, 1085, 1016, 933, 859, 793, 757, 726, 706, 634, 571, 526, 479 cm$^{-1}$.

UV-Vis (1,2-$C_6H_4F_2$): $\lambda_{max}$ 690, 769 nm

$^{1}$H NMR ($C_6D_6$, 400 MHz): δ 7.13, 7.03, 7.03, 7.02, 7.02, 6.93, 6.82, 5.05 (s, 2H, γ-C*H)*, 3.98 (sept, $^{3}J_{HH}$ = 6.6 Hz, 4 H, -C*H*(CH$_3$)$_2$), 3.69 (sept, $^{3}J_{HH}$ = 6.6 Hz, 4 H, -C*H*(CH$_3$)$_2$), 1.79 (d, $^{3}J_{HH}$ = 6.7 Hz, 12 H, -CH(C*H$_3$*)$_2$)), 1.73 (s, 12 H, ArNCC*H$_3$*), 1.49 (d, $^{3}J_{HH}$ = 6.5 Hz, 12 H, -CH(C*H$_3$*)$_2$)), 1.30 (d, $^{3}J_{HH}$ = 6.9 Hz, 12 H, -CH(C*H$_3$*)$_2$)), 1.24 (d, $^{3}J_{HH}$ = 6.8 Hz, 12 H, -CH(C*H$_3$*)$_2$)), 1.08 (s, 36 H, C(C*H$_3$*)$_3$).

$^{13}$C NMR ($C_6D_6$, 101 MHz): δ 168.5 (N*C*CCH(CH$_3$)$_2$), 145.9 (*C*CH(CH$_3$)$_2$), 144.6 (*C*CH(CH$_3$)$_2$), 143.0, 131.9, 130.3, 128.8, 128.5, 128.4, 128.2, 128.1, 127.8, 127.0, 125.9, 124.7, 124.3 (*C*$_6$H$_3$), 97.6 (γ-*C*H-), 55.7 (*C*(CH$_3$)$_3$), 31.9 (C(*C*H$_3$)$_3$), 29.9 (-*C*H(CH$_3$)$_2$), 28.4 (-CH(*C*H$_3$)$_2$), 28.2 (-*C*H(CH$_3$)$_2$), 25.5 (CH(*C*H$_3$)$_2$), 25.0 (CH(*C*H$_3$)$_2$), 24.8 (ArNC*C*H$_3$).

$^{29}$Si NMR ($C_6D_6$, 79 MHz): δ Es waren keine Signale sichtbar.

$^{31}$P NMR ($C_6D_6$, 243 MHz) δ 641.7 (P=P), –262.7 (Si=P–Si) ppm.

## 15.2. Spektren DDP(Cl)Ga–P=Si(L)P=PSi(L)P–Ga(Cl)DDP **15**

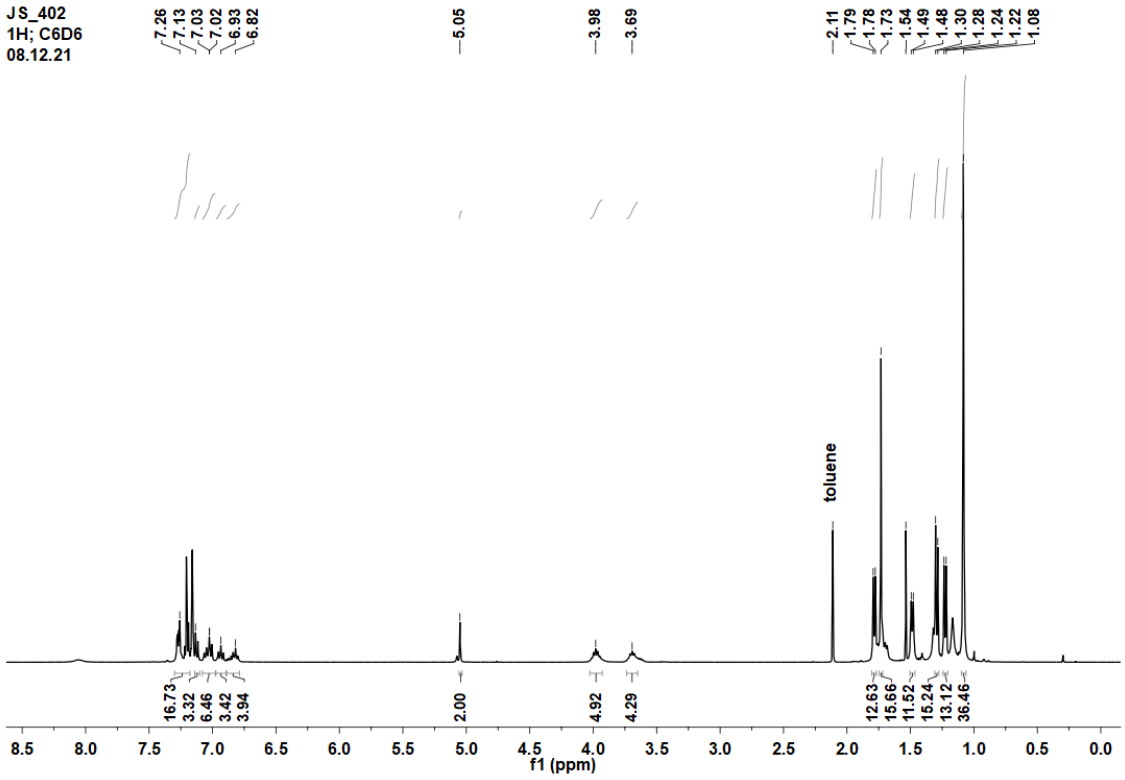



Abbildung 61: $^{1}$H-NMR-Spektrum von DDP(Cl)Ga–P=Si(L)P=PSi(L)P–Ga(Cl)DDP **15** in $C_6D_6$.

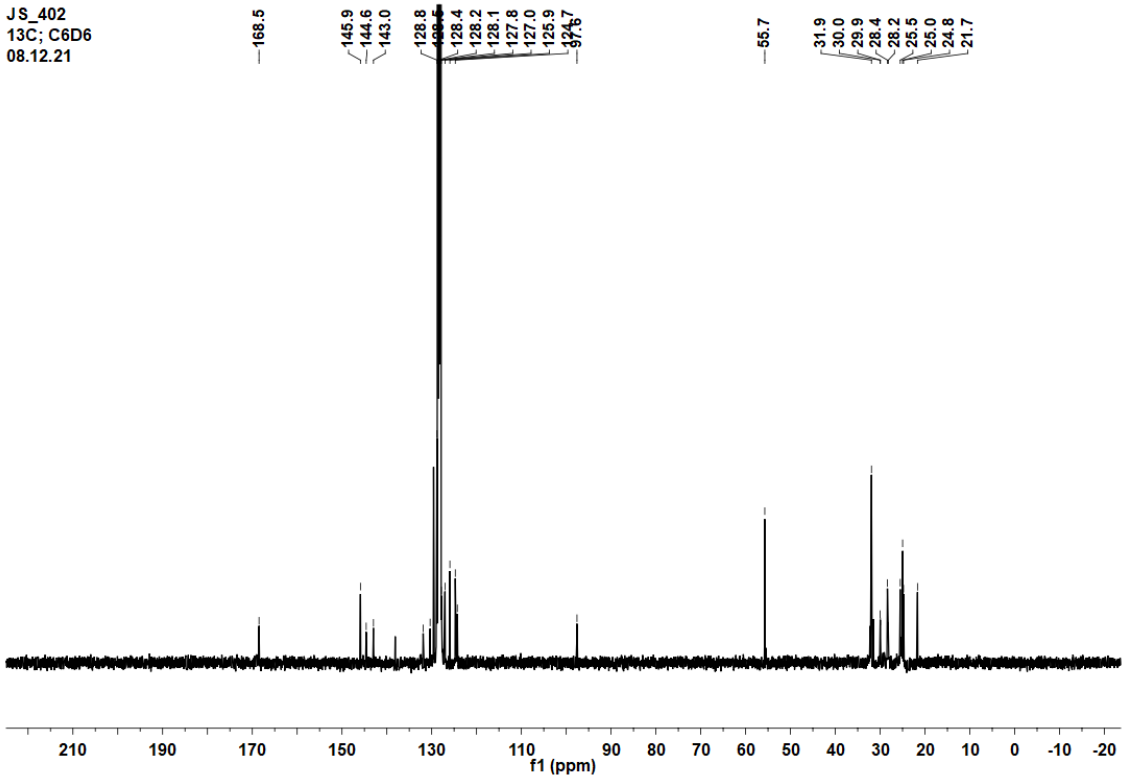



Abbildung 62: $^{13}$C-NMR-Spektrum von DDP(Cl)Ga–P=Si(L)P=PSi(L)P–Ga(Cl)DDP **15** in $C_6D_6$.

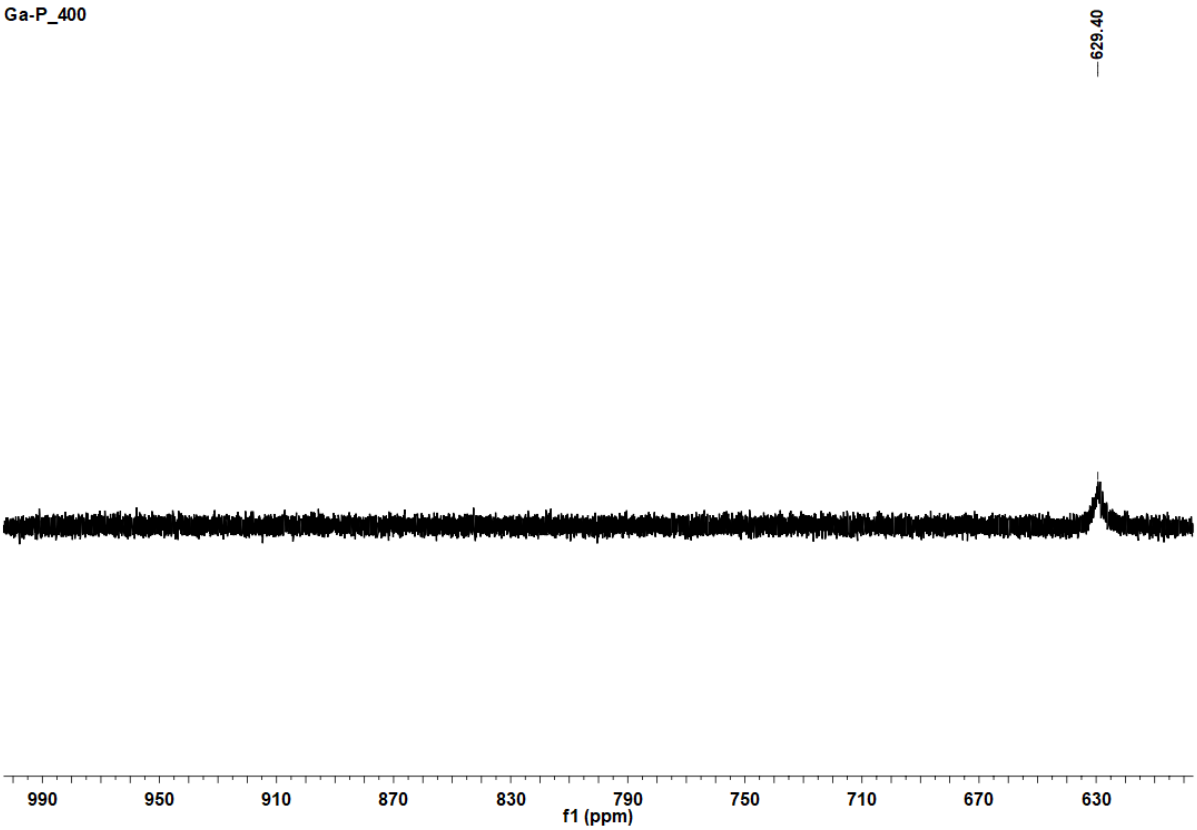



Abbildung 63: $^{31}$P-NMR-Spektrum von DDP(Cl)Ga–P=Si(L)P=PSi(L)P–Ga(Cl)DDP **15** in $C_6D_6$.

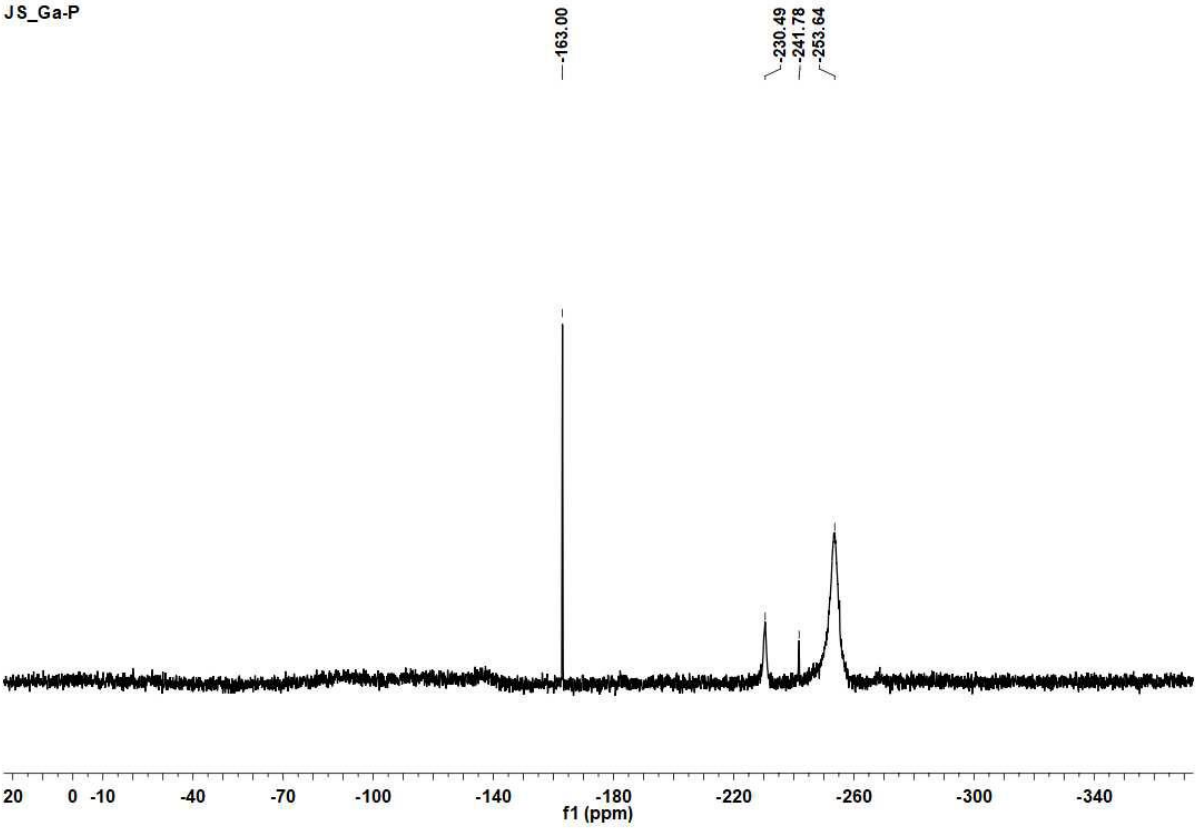



Abbildung 64: $^{31}$P-NMR-Spektrum von DDP(Cl)Ga–P=Si(L)P=PSi(L)P–Ga(Cl)DDP **15** in $C_6D_6$.

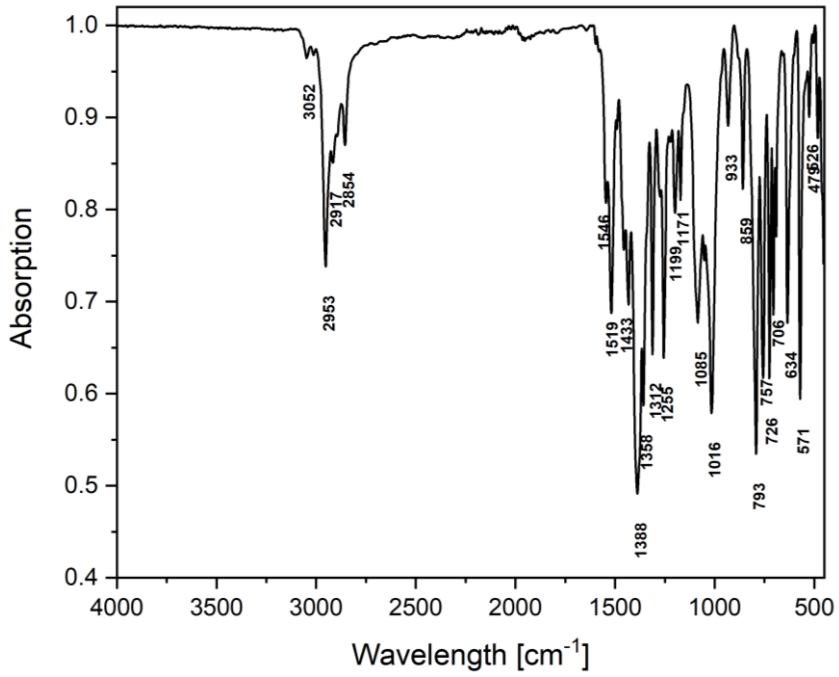



Abbildung 65: ATR-IR Spektrum von DDP(Cl)Ga–P=Si(L)P=PSi(L)P–Ga(Cl)DDP **15**.

## 15.3. Kristallografische Daten DDP(Cl)Ga–P=Si(L)P=PSi(L)P–Ga(Cl)DDP **15**

Tabelle 61: Crystal structure data

| **Identification code** | **jus_400m_sq** |
|---|---|
| Empirical formula | C112 H152 Cl2 Ga2 N8 P4 Si2 |
| Formula weight | 2000.81 |
| Density (calculated) | 1.124 g·cm$^{-3}$ |
| *F*(000) | 1064 |
| Temperature | 100(2) K |
| Crystal size | 0.498 × 0.312 × 0.114 mm |
| Crystal colour | green |
| Crystal description | tablet |
| Wavelength | 0.71073 Å |
| Crystal system | triclinic |
| Space group | *P -1* |
| Unit cell dimensions | |
| *a* [Å] | 12.6070(7) |
| *b* [Å] | 12.7567(7) |
| *c* [Å] | 18.6142(10) |
| *α* [°] | 82.247(2) |
| *β* [°] | 87.357(2) |
| *γ* [°] | 85.660(3) |
| Volume | 2955.8(3) Å$^3$ |

| | |
|---|---|
| $Z$ | 1 |
| Cell measurement reflections used | 9220 |
| Cell measurement $\theta$ min/max | 2.21°/33.44° |
| Diffractometer control software | BRUKER APEX3(v2019.1-0) |
| Diffractometer measurement device | Bruker D8 KAPPA II (APEX II detector) |
| Diffractometer measurement method | Data collection strategy APEX 3/QUEEN |
| $\theta$ range for data collection | 1.833°- 33.603° |
| Completeness to $\theta$ = 25.242° | 100.0% |
| Completeness to $\theta_{max}$ = 33.603° | 99.3% |
| Index ranges | -19 ≤ $h$ ≤ 19 |
| | -19 ≤ $k$ ≤ 19 |
| | -28 ≤ $l$ ≤ 28 |
| Computing data reduction | BRUKER APEX3(v2019.1-0) |
| Absorption coefficient | 0.621 $mm^{-1}$ |
| Absorption correction | Semi-empirical from equivalents |
| Computation absorption correction | SADABS |
| Max./min. Transmission | 0.75/0.66 |
| $R_{merg}$ before/after correction | 0.0639/0.0390 |
| Computing structure solution | BRUKER APEX3(v2019.1-0) |
| Computing structure refinement | SHELXL-2017/1 (Sheldrick, 2017) |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Reflections collected | 234040 |
| Independent reflections | 23221 |
| $R_{int}$ | 0.0291 |
| Reflections with $I > 2\sigma(I)$ | 19611 |
| Restraints | 522 |
| Parameter | 670 |
| GooF | 1.072 |
| Weighting details | $w = 1/[\sigma^2(F_{obs}^2) + (0.0458P)^2 + 1.0517P]$ |
| | where $P = (F_{obs}^2 + 2F_{calc}^2)/3$ |
| $R_1$ [$I > 2\sigma(I)$] | 0.0316 |
| $wR_2$ [$I > 2\sigma(I)$] | 0.0833 |
| $R_1$ [all data] | 0.0436 |
| $wR_2$ [all data] | 0.0919 |
| Absolute structure parameter | |
| Largest diff. peak and hole | 0.542/-0.481 |

Tabelle 62: Atomic coordinates ( × $10^4$) and equivalent isotropic displacement parameters ($Å^2$ × $10^3$) for jus_400m_sq. $U_{eq}$ is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | *x* | *y* | *z* | $U_{eq}$ | *x* | *y* | *z* | $U_{eq}$ | *x* |
|---|---|---|---|---|---|---|---|---|---|
| Ga(1) | 4944(1) | 2977(1) | 2562(1) | 13(1) | H(13C) | 2935 | 6864 | 3847 | 45 |
| Cl(1) | 5847(1) | 4429(1) | 2275(1) | 21(1) | C(14) | 3816(1) | 6374(1) | 2591(1) | 28(1) |
| P(1) | 4113(1) | 2249(1) | 1727(1) | 20(1) | H(14A) | 3961 | 5919 | 2208 | 43 |
| P(2) | 5359(1) | 4573(1) | 434(1) | 20(1) | H(14B) | 4391 | 6850 | 2587 | 43 |
| P(2') | 4890(2) | 4977(1) | 542(1) | 24(1) | H(14C) | 3139 | 6794 | 2505 | 43 |
| Si(1) | 4058(1) | 3421(1) | 810(1) | 14(1) | C(15) | 2465(1) | 1945(1) | 3496(1) | 23(1) |
| N(1) | 4152(1) | 3348(1) | 3450(1) | 15(1) | H(15) | 3153 | 1793 | 3227 | 27 |
| N(2) | 5968(1) | 1951(1) | 3094(1) | 15(1) | C(16) | 2606(1) | 1491(1) | 4294(1) | 40(1) |
| N(3) | 3769(1) | 2857(1) | -7(1) | 16(1) | H(16A) | 2722 | 717 | 4336 | 59 |
| N(4) | 2756(1) | 3992(1) | 490(1) | 17(1) | H(16B) | 1965 | 1681 | 4580 | 59 |
| C(1) | 4637(1) | 3256(1) | 4078(1) | 17(1) | H(16C) | 3222 | 1784 | 4476 | 59 |
| C(2) | 5655(1) | 2736(1) | 4196(1) | 18(1) | C(17) | 1614(1) | 1381(1) | 3173(1) | 43(1) |
| H(2) | 5988 | 2857 | 4622 | 22 | H(17A) | 1496 | 1709 | 2673 | 64 |
| C(3) | 6229(1) | 2066(1) | 3765(1) | 17(1) | H(17B) | 948 | 1441 | 3463 | 64 |
| C(4) | 4100(1) | 3688(1) | 4728(1) | 24(1) | H(17C) | 1849 | 631 | 3175 | 64 |
| H(4A) | 4387 | 4364 | 4784 | 36 | C(18) | 6458(1) | 1070(1) | 2760(1) | 17(1) |
| H(4B) | 4232 | 3181 | 5164 | 36 | C(19) | 7351(1) | 1209(1) | 2287(1) | 21(1) |
| H(4C) | 3332 | 3799 | 4658 | 36 | C(20) | 7779(1) | 342(1) | 1957(1) | 29(1) |
| C(5) | 7180(1) | 1422(1) | 4098(1) | 26(1) | H(20) | 8375 | 425 | 1628 | 35 |
| H(5A) | 7075 | 665 | 4119 | 39 | C(21) | 7353(1) | -631(1) | 2099(1) | 32(1) |
| H(5B) | 7259 | 1592 | 4590 | 39 | H(21) | 7658 | -1214 | 1874 | 38 |
| H(5C) | 7823 | 1590 | 3802 | 39 | C(22) | 6479(1) | -758(1) | 2573(1) | 28(1) |
| C(6) | 3074(1) | 3818(1) | 3405(1) | 17(1) | H(22) | 6195 | -1433 | 2673 | 33 |
| C(7) | 2871(1) | 4924(1) | 3351(1) | 19(1) | C(23) | 6007(1) | 86(1) | 2905(1) | 21(1) |
| C(8) | 1810(1) | 5336(1) | 3317(1) | 23(1) | C(24) | 7865(1) | 2254(1) | 2132(1) | 26(1) |
| H(8) | 1657 | 6081 | 3284 | 28 | H(24) | 7458 | 2768 | 2421 | 31 |
| C(9) | 982(1) | 4681(1) | 3331(1) | 26(1) | C(25) | 9025(1) | 2146(1) | 2371(1) | 39(1) |
| H(9) | 268 | 4976 | 3310 | 31 | H(25A) | 9448 | 1662 | 2085 | 59 |
| C(10) | 1198(1) | 3593(1) | 3377(1) | 25(1) | H(25B) | 9048 | 1862 | 2886 | 59 |
| H(10) | 627 | 3147 | 3383 | 30 | H(25C) | 9320 | 2843 | 2292 | 59 |
| C(11) | 2242(1) | 3139(1) | 3414(1) | 20(1) | C(26) | 7808(1) | 2706(1) | 1336(1) | 41(1) |
| C(12) | 3747(1) | 5683(1) | 3328(1) | 21(1) | H(26A) | 8206 | 2219 | 1040 | 61 |
| H(12) | 4440 | 5253 | 3402 | 25 | H(26B) | 8118 | 3396 | 1257 | 61 |
| C(13) | 3588(1) | 6402(1) | 3928(1) | 30(1) | H(26C) | 7063 | 2795 | 1197 | 61 |
| H(13A) | 4198 | 6838 | 3918 | 45 | C(27) | 5034(1) | -90(1) | 3408(1) | 28(1) |
| H(13B) | 3528 | 5965 | 4401 | 45 | H(27) | 4772 | 609 | 3562 | 34 |
| C(28) | 5306(2) | -856(2) | 4091(1) | 50(1) | H(43B) | 821 | 5906 | 462 | 46 |
| H(28A) | 5566 | -1547 | 3953 | 75 | H(43C) | 1047 | 5137 | -148 | 46 |
| H(28B) | 4668 | -938 | 4408 | 75 | C(44) | 2279(1) | 5120(1) | 1399(1) | 32(1) |
| H(28C) | 5859 | -574 | 4348 | 75 | H(44A) | 2836 | 5581 | 1196 | 48 |

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| C(29) | 4139(1) | -496(1) | 3017(1) | 37(1) | H(44B) | 2580 | 4571 | 1768 | 48 |
| H(29A) | 3968 | 4 | 2583 | 56 | H(44C) | 1704 | 5543 | 1621 | 48 |
| H(29B) | 3506 | -559 | 3341 | 56 | C11 | 7346(4) | 5021(3) | 3813(2) | 42(1) |
| H(29C) | 4370 | -1192 | 2874 | 56 | H11 | 6908 | 4523 | 3663 | 50 |
| C(30) | 2832(1) | 3418(1) | -65(1) | 15(1) | C21 | 8035(5) | 4708(4) | 4367(3) | 53(1) |
| C(31) | 2064(1) | 3432(1) | -648(1) | 18(1) | H21 | 8101 | 3989 | 4584 | 64 |
| C(32) | 1357(1) | 2644(1) | -630(1) | 23(1) | C31 | 8623(3) | 5444(5) | 4601(2) | 46(1) |
| H(32) | 1329 | 2103 | -227 | 28 | H31 | 9066 | 5241 | 5003 | 55 |
| C(33) | 688(1) | 2654(1) | -1209(1) | 31(1) | C41 | 8582(3) | 6485(4) | 4261(3) | 43(1) |
| H(33) | 205 | 2115 | -1201 | 38 | H41 | 9014 | 6983 | 4417 | 51 |
| C(34) | 725(1) | 3443(1) | -1795(1) | 34(1) | C51 | 7909(3) | 6786(3) | 3697(2) | 38(1) |
| H(34) | 271 | 3444 | -2188 | 41 | H51 | 7873 | 7495 | 3461 | 46 |
| C(35) | 1424(1) | 4235(1) | -1809(1) | 32(1) | C61 | 7288(3) | 6053(3) | 3475(2) | 39(1) |
| H(35) | 1442 | 4780 | -2210 | 38 | H61 | 6819 | 6259 | 3088 | 47 |
| C(36) | 2098(1) | 4232(1) | -1238(1) | 24(1) | C12 | 10338(2) | 8790(2) | 4536(1) | 58(1) |
| H(36) | 2578 | 4773 | -1248 | 29 | H12 | 10424 | 8742 | 5044 | 70 |
| C(37) | 4308(1) | 2091(1) | -456(1) | 20(1) | C22 | 11121(2) | 8365(2) | 4106(1) | 60(1) |
| C(38) | 3799(1) | 1030(1) | -286(1) | 30(1) | H22 | 11747 | 8018 | 4316 | 72 |
| H(38A) | 3053 | 1123 | -422 | 44 | C32 | 11003(2) | 8441(1) | 3363(1) | 51(1) |
| H(38B) | 3837 | 780 | 234 | 44 | H32 | 11553 | 8159 | 3063 | 61 |
| H(38C) | 4182 | 508 | -562 | 44 | C42 | 10081(2) | 8928(1) | 3060(1) | 47(1) |
| C(39) | 5467(1) | 1947(1) | -233(1) | 30(1) | H42 | 9992 | 8974 | 2553 | 56 |
| H(39A) | 5853 | 1416 | -499 | 45 | C52 | 9286(1) | 9352(1) | 3501(1) | 43(1) |
| H(39B) | 5493 | 1710 | 290 | 45 | H52 | 8648 | 9682 | 3296 | 51 |
| H(39C) | 5797 | 2624 | -346 | 45 | C62 | 9421(1) | 9292(2) | 4230(1) | 48(1) |
| C(40) | 4272(1) | 2503(1) | -1264(1) | 28(1) | H62 | 8883 | 9595 | 4529 | 57 |
| H(40A) | 4725 | 2027 | -1539 | 43 | C15 | 8770(6) | 5999(8) | 4566(5) | 54(2) |
| H(40B) | 4529 | 3216 | -1351 | 43 | H15 | 9232 | 6323 | 4846 | 65 |
| H(40C) | 3537 | 2528 | -1421 | 43 | C25 | 8621(5) | 4945(8) | 4724(3) | 48(2) |
| C(41) | 1838(1) | 4598(1) | 796(1) | 21(1) | H25 | 8960 | 4530 | 5123 | 58 |
| C(42) | 1004(1) | 3841(1) | 1110(1) | 32(1) | C35 | 7978(8) | 4496(7) | 4301(6) | 60(2) |
| H(42A) | 733 | 3502 | 720 | 49 | H35 | 7853 | 3767 | 4422 | 72 |
| H(42B) | 416 | 4238 | 1339 | 49 | C45 | 7498(8) | 5069(8) | 3698(5) | 76(3) |
| H(42C) | 1328 | 3297 | 1473 | 49 | H45 | 7107 | 4729 | 3383 | 91 |
| C(43) | 1367(1) | 5464(1) | 230(1) | 31(1) | C55 | 7605(9) | 6113(9) | 3575(5) | 84(3) |
| H(43A) | 1932 | 5904 | 11 | 46 | H55 | 7235 | 6532 | 3192 | 101 |
| C65 | 8246(9) | 6593(6) | 3998(5) | 68(2) | | | | | |
| H65 | 8326 | 7333 | 3897 | 82 | | | | | |

Tabelle 63: Anisotropic displacement parameters (Å$^2$ × 10$^3$) for jus_400m_sq. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ga(1) | 14(1) | 13(1) | 12(1) | 1(1) | -2(1) | -1(1) | C(29) | 30(1) | 26(1) | 55(1) | 1(1) | -1(1) | -6(1) |
| Cl(1) | 24(1) | 16(1) | 24(1) | 0(1) | 4(1) | -5(1) | C(30) | 17(1) | 16(1) | 13(1) | 1(1) | -2(1) | -2(1) |
| P(1) | 27(1) | 16(1) | 16(1) | 3(1) | -7(1) | -6(1) | C(31) | 18(1) | 21(1) | 14(1) | -2(1) | -3(1) | 1(1) |
| P(2) | 16(1) | 23(1) | 19(1) | 9(1) | -7(1) | -6(1) | C(32) | 22(1) | 25(1) | 24(1) | -6(1) | -5(1) | -2(1) |
| P(2') | 41(1) | 21(1) | 13(1) | -1(1) | -1(1) | -17(1) | C(33) | 24(1) | 37(1) | 37(1) | -18(1) | -11(1) | 0(1) |
| Si(1) | 16(1) | 15(1) | 12(1) | 1(1) | -3(1) | -3(1) | C(34) | 31(1) | 49(1) | 26(1) | -17(1) | -14(1) | 12(1) |
| N(1) | 15(1) | 15(1) | 15(1) | -1(1) | -1(1) | -1(1) | C(35) | 35(1) | 41(1) | 16(1) | -2(1) | -8(1) | 12(1) |
| N(2) | 16(1) | 14(1) | 14(1) | -1(1) | -3(1) | 1(1) | C(36) | 26(1) | 28(1) | 16(1) | 2(1) | -3(1) | 1(1) |
| N(3) | 16(1) | 18(1) | 14(1) | -2(1) | -2(1) | 0(1) | C(37) | 23(1) | 17(1) | 19(1) | -4(1) | -1(1) | 1(1) |
| N(4) | 19(1) | 19(1) | 14(1) | -2(1) | -4(1) | 3(1) | C(38) | 34(1) | 17(1) | 37(1) | -1(1) | -3(1) | 0(1) |
| C(1) | 20(1) | 17(1) | 14(1) | -1(1) | -1(1) | -2(1) | C(39) | 22(1) | 32(1) | 38(1) | -12(1) | -1(1) | 6(1) |
| C(2) | 20(1) | 21(1) | 15(1) | -4(1) | -4(1) | -2(1) | C(40) | 42(1) | 24(1) | 18(1) | -5(1) | 4(1) | 4(1) |
| C(3) | 18(1) | 17(1) | 16(1) | 0(1) | -4(1) | -1(1) | C(41) | 23(1) | 24(1) | 15(1) | -2(1) | -1(1) | 7(1) |
| C(4) | 28(1) | 29(1) | 16(1) | -5(1) | -1(1) | 3(1) | C(42) | 28(1) | 38(1) | 29(1) | -1(1) | 8(1) | 2(1) |
| C(5) | 25(1) | 31(1) | 21(1) | -4(1) | -10(1) | 8(1) | C(43) | 36(1) | 31(1) | 21(1) | -1(1) | -4(1) | 16(1) |
| C(6) | 16(1) | 19(1) | 15(1) | -1(1) | 0(1) | 0(1) | C(44) | 40(1) | 33(1) | 24(1) | -12(1) | -7(1) | 11(1) |
| C(7) | 20(1) | 20(1) | 16(1) | -3(1) | 0(1) | 1(1) | C11 | 60(2) | 36(1) | 34(2) | -14(1) | 21(1) | -21(1) |
| C(8) | 23(1) | 24(1) | 21(1) | -4(1) | 0(1) | 6(1) | C21 | 60(3) | 44(2) | 47(3) | 3(2) | 24(2) | 13(2) |
| C(9) | 18(1) | 34(1) | 25(1) | -4(1) | -1(1) | 5(1) | C31 | 31(2) | 69(3) | 34(2) | 0(2) | 4(1) | 7(2) |
| C(10) | 17(1) | 31(1) | 27(1) | -4(1) | 0(1) | -2(1) | C41 | 29(1) | 59(2) | 44(2) | -23(2) | -2(1) | -4(1) |
| C(11) | 17(1) | 22(1) | 20(1) | -2(1) | 0(1) | -2(1) | C51 | 37(1) | 35(1) | 44(2) | -4(1) | 3(1) | -9(1) |
| C(12) | 25(1) | 17(1) | 22(1) | -4(1) | 0(1) | -1(1) | C61 | 43(2) | 47(2) | 29(1) | -5(1) | -1(1) | -16(1) |
| C(13) | 36(1) | 25(1) | 30(1) | -11(1) | -2(1) | -1(1) | C12 | 54(1) | 73(1) | 46(1) | -10(1) | -11(1) | 23(1) |
| C(14) | 37(1) | 21(1) | 26(1) | 0(1) | 2(1) | -2(1) | C22 | 49(1) | 69(1) | 63(1) | -26(1) | -19(1) | 25(1) |
| C(15) | 19(1) | 21(1) | 28(1) | -1(1) | 1(1) | -5(1) | C32 | 52(1) | 44(1) | 58(1) | -20(1) | -1(1) | 9(1) |
| C(16) | 55(1) | 28(1) | 32(1) | 7(1) | 7(1) | -4(1) | C42 | 60(1) | 35(1) | 45(1) | -9(1) | -9(1) | 1(1) |
| C(17) | 30(1) | 27(1) | 73(1) | -9(1) | -13(1) | -9(1) | C52 | 36(1) | 36(1) | 54(1) | 4(1) | -8(1) | -2(1) |
| C(18) | 18(1) | 17(1) | 16(1) | -2(1) | -4(1) | 2(1) | C62 | 38(1) | 52(1) | 48(1) | 3(1) | 4(1) | 8(1) |
| C(19) | 18(1) | 23(1) | 22(1) | -3(1) | -2(1) | 3(1) | C15 | 41(3) | 76(5) | 53(4) | -35(4) | 15(3) | -23(3) |
| C(20) | 24(1) | 33(1) | 30(1) | -11(1) | 1(1) | 6(1) | C25 | 33(2) | 88(5) | 27(2) | -11(3) | 6(2) | -17(3) |
| C(21) | 31(1) | 29(1) | 38(1) | -16(1) | -4(1) | 7(1) | C35 | 57(4) | 92(5) | 41(3) | -23(3) | 9(3) | -51(4) |
| C(22) | 32(1) | 19(1) | 34(1) | -8(1) | -7(1) | 2(1) | C45 | 52(4) | 158(7) | 26(3) | -32(3) | -2(3) | -26(4) |
| C(23) | 24(1) | 16(1) | 23(1) | -2(1) | -4(1) | 1(1) | C55 | 61(6) | 147(7) | 38(4) | -3(4) | 8(3) | 9(5) |
| C(24) | 20(1) | 26(1) | 29(1) | 0(1) | 4(1) | 0(1) | C65 | 90(8) | 52(3) | 60(6) | -10(3) | 29(4) | 0(4) |
| C(25) | 22(1) | 40(1) | 54(1) | 2(1) | -2(1) | -6(1) | | | | | | | |
| C(26) | 49(1) | 38(1) | 30(1) | 7(1) | 7(1) | 3(1) | | | | | | | |
| C(27) | 33(1) | 17(1) | 33(1) | -1(1) | 5(1) | -6(1) | | | | | | | |
| C(28) | 64(1) | 48(1) | 35(1) | 12(1) | -3(1) | -18(1) | | | | | | | |

Tabelle 64: Bond lengths [Å] for jus_400m_sq.

| | | | | | |
|---|---|---|---|---|---|
| Ga(1)-N(2) | 1.9669(8) | C(7)-C(12) | 1.5194(15) | C(35)-C(36) | 1.3916(16) |
| Ga(1)-N(1) | 1.9850(8) | C(8)-C(9) | 1.3825(17) | C(37)-C(40) | 1.5265(15) |
| Ga(1)-Cl(1) | 2.2379(3) | C(9)-C(10) | 1.3870(17) | C(37)-C(39) | 1.5280(16) |
| Ga(1)-P(1) | 2.2510(3) | C(10)-C(11) | 1.3975(15) | C(37)-C(38) | 1.5296(16) |
| P(1)-Si(1) | 2.1127(4) | C(11)-C(15) | 1.5165(15) | C(41)-C(42) | 1.5239(18) |
| P(2)-P(2)#1 | 2.0323(7) | C(12)-C(14) | 1.5306(16) | C(41)-C(43) | 1.5253(15) |
| P(2)-Si(1) | 2.3067(4) | C(12)-C(13) | 1.5344(16) | C(41)-C(44) | 1.5267(16) |
| P(2')-P(2')#1 | 2.018(3) | C(15)-C(17) | 1.5240(17) | C11-C21 | 1.377(6) |
| P(2')-Si(1) | 2.2995(12) | C(15)-C(16) | 1.5331(18) | C11-C61 | 1.378(4) |
| Si(1)-N(3) | 1.8301(8) | C(18)-C(19) | 1.4037(14) | C21-C31 | 1.365(6) |
| Si(1)-N(4) | 1.8360(9) | C(18)-C(23) | 1.4054(14) | C31-C41 | 1.390(5) |
| Si(1)-C(30) | 2.2960(9) | C(19)-C(20) | 1.3987(16) | C41-C51 | 1.377(4) |
| N(1)-C(1) | 1.3301(12) | C(19)-C(24) | 1.5134(16) | C51-C61 | 1.380(4) |
| N(1)-C(6) | 1.4448(12) | C(20)-C(21) | 1.3778(19) | C12-C22 | 1.369(3) |
| N(2)-C(3) | 1.3364(12) | C(21)-C(22) | 1.3840(19) | C12-C62 | 1.386(2) |
| N(2)-C(18) | 1.4417(12) | C(22)-C(23) | 1.3944(15) | C22-C32 | 1.388(3) |
| N(3)-C(30) | 1.3351(12) | C(23)-C(27) | 1.5202(16) | C32-C42 | 1.382(3) |
| N(3)-C(37) | 1.4771(13) | C(24)-C(26) | 1.5191(18) | C42-C52 | 1.391(2) |
| N(4)-C(30) | 1.3403(12) | C(24)-C(25) | 1.5395(18) | C52-C62 | 1.366(2) |
| N(4)-C(41) | 1.4772(13) | C(27)-C(28) | 1.530(2) | C15-C25 | 1.362(7) |
| C(1)-C(2) | 1.4119(14) | C(27)-C(29) | 1.530(2) | C15-C65 | 1.378(8) |
| C(1)-C(4) | 1.5090(14) | C(30)-C(31) | 1.4855(13) | C25-C35 | 1.363(7) |
| C(2)-C(3) | 1.3914(14) | C(31)-C(32) | 1.3884(15) | C35-C45 | 1.391(9) |
| C(3)-C(5) | 1.5099(14) | C(31)-C(36) | 1.3955(14) | C45-C55 | 1.337(10) |
| C(6)-C(7) | 1.4058(14) | C(32)-C(33) | 1.3981(16) | C55-C65 | 1.382(9) |
| C(6)-C(11) | 1.4078(14) | C(33)-C(34) | 1.382(2) | | |
| C(7)-C(8) | 1.3994(15) | C(34)-C(35) | 1.385(2) | | |

#1 -x+1,-y+1,-z

Tabelle 65: Bond angles [°] for jus_400m_sq.

| | | | | | |
|---|---|---|---|---|---|
| N(2)-Ga(1)-N(1) | 94.47(3) | N(2)-C(3)-C(2) | 123.78(9) | N(3)-C(30)-N(4) | 105.90(8) |
| N(2)-Ga(1)-Cl(1) | 103.85(3) | N(2)-C(3)-C(5) | 119.53(9) | N(3)-C(30)-C(31) | 126.40(9) |
| N(1)-Ga(1)-Cl(1) | 99.73(2) | C(2)-C(3)-C(5) | 116.66(9) | N(4)-C(30)-C(31) | 127.64(9) |
| N(2)-Ga(1)-P(1) | 111.92(3) | C(7)-C(6)-C(11) | 121.36(9) | N(3)-C(30)-Si(1) | 52.82(5) |
| N(1)-Ga(1)-P(1) | 120.17(3) | C(7)-C(6)-N(1) | 120.44(9) | N(4)-C(30)-Si(1) | 53.08(5) |
| Cl(1)-Ga(1)-P(1) | 122.231(11) | C(11)-C(6)-N(1) | 118.20(9) | C(31)-C(30)-Si(1) | 178.09(7) |
| Si(1)-P(1)-Ga(1) | 104.770(14) | C(8)-C(7)-C(6) | 118.06(10) | C(32)-C(31)-C(36) | 120.19(10) |
| P(2)#1-P(2)-Si(1) | 99.36(2) | C(8)-C(7)-C(12) | 119.02(9) | C(32)-C(31)-C(30) | 121.25(9) |
| P(2')#1-P(2')-Si(1) | 100.04(8) | C(6)-C(7)-C(12) | 122.91(9) | C(36)-C(31)-C(30) | 118.50(9) |
| N(3)-Si(1)-N(4) | 71.24(4) | C(9)-C(8)-C(7) | 121.43(10) | C(31)-C(32)-C(33) | 119.49(11) |
| N(3)-Si(1)-P(1) | 111.20(3) | C(8)-C(9)-C(10) | 119.72(10) | C(34)-C(33)-C(32) | 120.30(12) |
| N(4)-Si(1)-P(1) | 118.86(3) | C(9)-C(10)-C(11) | 121.22(10) | C(33)-C(34)-C(35) | 120.15(11) |

| | | | | | |
|---|---|---|---|---|---|
| N(3)-Si(1)-C(30) | 35.54(3) | C(10)-C(11)-C(6) | 118.20(10) | C(34)-C(35)-C(36) | 120.14(12) |
| N(4)-Si(1)-C(30) | 35.71(4) | C(10)-C(11)-C(15) | 120.70(10) | C(35)-C(36)-C(31) | 119.72(11) |
| P(1)-Si(1)-C(30) | 121.06(3) | C(6)-C(11)-C(15) | 121.04(9) | N(3)-C(37)-C(40) | 111.79(8) |
| N(3)-Si(1)-P(2') | 112.22(4) | C(7)-C(12)-C(14) | 110.94(9) | N(3)-C(37)-C(39) | 106.21(8) |
| N(4)-Si(1)-P(2') | 95.00(7) | C(7)-C(12)-C(13) | 112.18(9) | C(40)-C(37)-C(39) | 109.16(10) |
| P(1)-Si(1)-P(2') | 131.45(4) | C(14)-C(12)-C(13) | 109.06(9) | N(3)-C(37)-C(38) | 109.24(9) |
| C(30)-Si(1)-P(2') | 106.86(5) | C(11)-C(15)-C(17) | 113.01(10) | C(40)-C(37)-C(38) | 110.99(9) |
| N(3)-Si(1)-P(2) | 104.54(3) | C(11)-C(15)-C(16) | 110.92(10) | C(39)-C(37)-C(38) | 109.33(9) |
| N(4)-Si(1)-P(2) | 110.45(3) | C(17)-C(15)-C(16) | 110.19(11) | N(4)-C(41)-C(42) | 109.57(9) |
| P(1)-Si(1)-P(2) | 125.763(17) | C(19)-C(18)-C(23) | 121.25(9) | N(4)-C(41)-C(43) | 111.46(9) |
| C(30)-Si(1)-P(2) | 111.88(3) | C(19)-C(18)-N(2) | 120.15(9) | C(42)-C(41)-C(43) | 111.31(10) |
| C(1)-N(1)-C(6) | 119.29(8) | C(23)-C(18)-N(2) | 118.59(9) | N(4)-C(41)-C(44) | 105.63(9) |
| C(1)-N(1)-Ga(1) | 120.40(7) | C(20)-C(19)-C(18) | 118.22(10) | C(42)-C(41)-C(44) | 110.03(10) |
| C(6)-N(1)-Ga(1) | 120.04(6) | C(20)-C(19)-C(24) | 119.49(10) | C(43)-C(41)-C(44) | 108.68(10) |
| C(3)-N(2)-C(18) | 119.05(8) | C(18)-C(19)-C(24) | 122.28(9) | C21-C11-C61 | 120.5(3) |
| C(3)-N(2)-Ga(1) | 120.62(7) | C(21)-C(20)-C(19) | 121.24(11) | C31-C21-C11 | 119.2(4) |
| C(18)-N(2)-Ga(1) | 120.32(6) | C(20)-C(21)-C(22) | 119.80(11) | C21-C31-C41 | 121.0(3) |
| C(30)-N(3)-C(37) | 131.75(8) | C(21)-C(22)-C(23) | 121.36(11) | C51-C41-C31 | 119.4(3) |
| C(30)-N(3)-Si(1) | 91.64(6) | C(22)-C(23)-C(18) | 118.12(10) | C41-C51-C61 | 119.7(3) |
| C(37)-N(3)-Si(1) | 136.61(7) | C(22)-C(23)-C(27) | 119.26(10) | C11-C61-C51 | 120.2(3) |
| C(30)-N(4)-C(41) | 131.08(9) | C(18)-C(23)-C(27) | 122.62(9) | C22-C12-C62 | 119.95(18) |
| C(30)-N(4)-Si(1) | 91.22(6) | C(19)-C(24)-C(26) | 111.36(11) | C12-C22-C32 | 120.18(17) |
| C(41)-N(4)-Si(1) | 135.87(7) | C(19)-C(24)-C(25) | 111.52(10) | C42-C32-C22 | 119.77(17) |
| N(1)-C(1)-C(2) | 123.83(9) | C(26)-C(24)-C(25) | 110.44(11) | C32-C42-C52 | 119.73(16) |
| N(1)-C(1)-C(4) | 120.96(9) | C(23)-C(27)-C(28) | 111.51(12) | C62-C52-C42 | 119.94(15) |
| C(2)-C(1)-C(4) | 115.19(9) | C(23)-C(27)-C(29) | 110.98(11) | C52-C62-C12 | 120.40(17) |
| C(3)-C(2)-C(1) | 127.85(9) | C(28)-C(27)-C(29) | 110.19(11) | C25-C15-C65 | 119.6(5) |
| C15-C25-C35 | 118.9(6) | | | | |
| C25-C35-C45 | 122.2(6) | | | | |
| C55-C45-C35 | 117.8(6) | | | | |
| C45-C55-C65 | 121.0(7) | | | | |
| C15-C65-C55 | 120.2(6) | | | | |

#1 -x+1,-y+1,-z