Hexacoordinate germanium mixed bischelates with the $GeCO_3Cl_2$ ligand environment

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Dedicated to Professor Irina P. Beletskaya on the occasion of her 75th birthday

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Abstract

Bischelate complexes of germanium with different chelate rings were prepared by double transmetallation. The earlier reported (O,O)-chelate [from the reaction of trichloro(chloromethyl)germane and *N*,*N*-dimethyl-2-(trimethylsilyloxy)-2-phenylacetamide (**1**)], 2-[dichloro(chloromethyl)germyloxy]-*N*,*N*-dimethyl-2-phenylacetamide (**2**) was reacted with trimethylsilylamides, 2,2-dimethyl-3-trimethylsilylbenzo[2*H*]-1,3-oxazin-4-one (**5**, B–$SiMe_3$) and 4-methyl-1-trimethylsilylquinolin-2-one (**6**, Q–$SiMe_3$) resulting in mixed (O,O),(C,O)-bischelate complexes, 2-[dichloro[(2,2-dimethyl-4-oxo-2*H*-benzo[*e*][1,3]oxazin-3(4*H*)-yl)methyl]germyloxy]-*N*,*N*-dimethyl-2-phenylacetamide (**7**) and 2-[dichloro[(4-methyl-2-oxoquinolin-1(2*H*)-yl)methyl]germyloxy]-*N*,*N*-dimethyl-2-phenylacetamide (**8**). Crystallographic analyses established pentacoordination of germanium in **2** and hexacoordination in **7** and **8**; the latter two compounds are the first reported mixed (O,O), (C,O)-bischelate complexes with two O→Ge coordinate bonds.

Keywords: Pentacoordinate, hexacoordinate, germanium, (O,O)- and (C,O)-ligands, X-ray diffraction study

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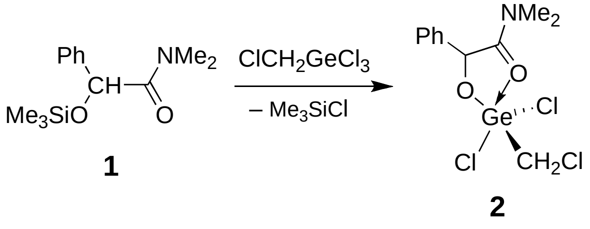
Introduction

In recent years considerable interest has arisen in the chemistry of hypercoordinate germanium compounds.[1] Complexes of hypercoordinate germanium with (O,O)- and (S,S)-dianionic, (C,N)- and (C,O)-monoanionic chelating ligands have been well studied to date.[1] However, only few germanium complexes containing bidentate (O,O)-monoanionic chelating ligands have been structurally characterized. Examples are hexacoordinate acetylacetonate[2a,b] and benzohydroximate[3] trischelates, a hexacoordinate bischelate with mandelic acid as ligand,[4] and a pentacoordinate bischelate derived from 2-hydroxy-1,2-diphenylethanone.[5]

In a recent communication[6] we have reported on the reaction of trichloro(chloromethyl)germane with racemic *N*,*N*-dimethyl-2-(trimethylsilyloxy)-2-phenylacetamide (**1**) yielding 2-[dichloro(chloromethyl)germyloxy]-*N*,*N*-dimethyl-2-phenylacetamide (**2**, Scheme 1), followed by the transformation of the monochelate **2** into a complex with two (O→Ge)-chelating ligands of different types (see below, Scheme 3, *a*).

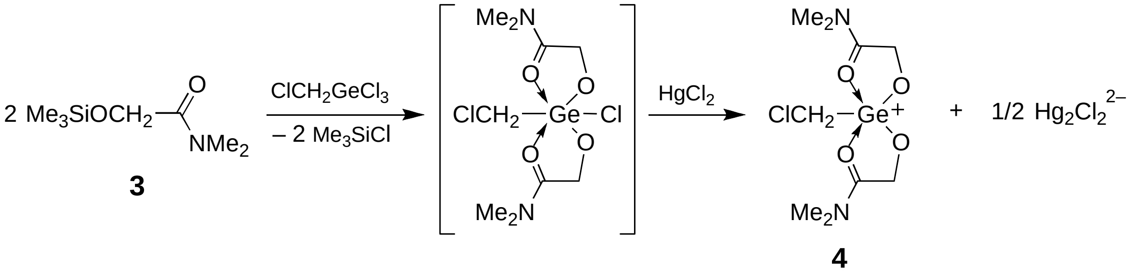


Scheme 1

In the same communication[6] a preliminary X-ray study revealed complex **2** as the first structurally characterized example of neutral pentacoordinate germanium complexes containing a 2-hydroxycarboxamide moiety with (O,O)-monoanionic bidentate chelate ligands.

In the present paper we report the full X-ray data of monochelate complex **2** and the products of its further transformations into neutral bischelate complexes of hexacoordinate germanium containing mixed (O,O)- and (C,O)-chelate ligands.

The (O,O)-monochelate **2** with pentacoordinated germanium has been prepared with high yield (89%) from trichloro(chloromethyl)germane and *N*,*N*-dimethyl-2-(trimethylsilyloxy)-2-phenylacetamide (**1**) in 1:1 ratio (Scheme 1).[6] The analogous reaction of trichloro(chloromethyl)germane with *N*,*N*-dimethyl-2-(trimethylsilyloxy)acetamide (**3**) in 1:2 ratio and subsequent treatment with $HgCl_2$ afforded the bischelate, donor-stabilized cationic complex **4** with pentacoordinated germanium (Scheme 2).[7] Presumably, a neutral bischelate complex is formed as an intermediate prior to the treatment with $HgCl_2$.



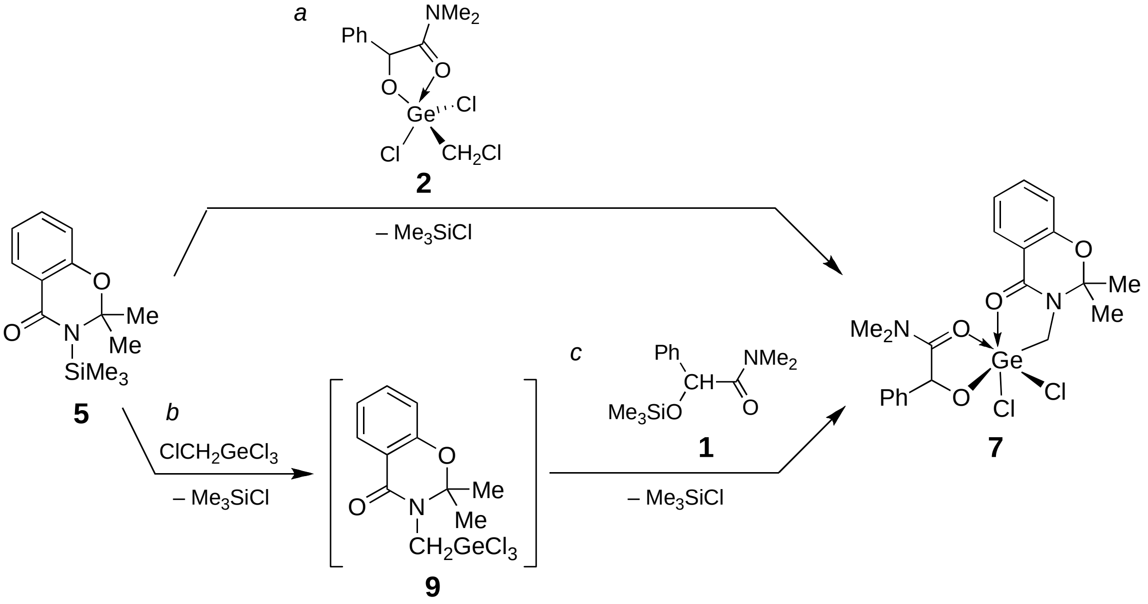
Scheme 2

Electronic and steric effects could be responsible for the formation of different products **2** and **4**, when the phenyl group in mandelic amide **1** is replaced by a hydrogen atom in glycolic amide **3** (Schemes 1 and 2).

The pentacoordinate complex, 2-[dichloro(chloromethyl)germyloxy]-*N*,*N*-dimethyl-2-phenylacetamide (**2**) is of particular interest as a starting material for the synthesis of mixed bischelates of hypercoordinated germanium. These chelates can be prepared by reactions similar to those used by us earlier for the synthesis of hypercoordinated germanium complexes from chloro(chloromethyl)dimethylgermane[8a–f] or dichlorobis(chloromethyl)germane[8g] and *N*-trimethylsilylamides and related compounds (for more details see[8d,f]).

Results and Discussion

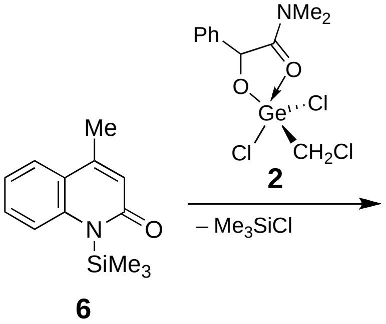
In this report we present the reactions of complex **2** with 2,2-dimethyl-3-trimethylsilylbenzo[2*H*]-1,3-oxazin-4-one (**5**, B–SiMe$_3$) and with 4-methyl-1-trimethylsilylquinolin-2-one (**6**, Q–SiMe$_3$), which led to bischelates **7** (Scheme 3, reaction *a*; Method a) and **8** (Scheme 4), respectively. Each complex contains two different monoanionic chelating ligands: mandelic amide as (O,O)-ligand, and as (C,O)-chelating ligand B–CH$_2$ and Q–CH$_2$, respectively.



Scheme 3. Preparation of bischelate **7** via reaction *a* (Method a), and reactions *b*, *c* (Method b).

Bischelate **7** was also prepared via an alternative, stepwise pathway (Scheme 3, reactions *b* and *c*; Method b). In this one-pot approach intermediate **9** was not isolated. The overall yield of **7** (64%) from reactions *b* and *c* was significantly lower than from reaction *a* (91%).

Bischelate **8** was prepared by the reaction of 4-methyl-1-trimethylsilylquinolin-2-one (**6**) with complex **2** in 89% yield (Scheme 4).



Scheme 4

The structures of complexes **7** and **8** were confirmed by element analyses, IR, ^{1}H and ^{13}C NMR spectra. At 45 °C the signals of the NCH$_2$ protons in **7** and **8** appear as broad AB-quartets.

Molecular and crystal structures of complexes **2**, **7** and **8** were determined by single crystal X-ray diffraction studies. Selected bond lengths and angles are summarized in Table 1; crystallographic parameters and experimental details are given in Table 2.

Table 1. Selected structural parameters for structures **2**, **7**, and **8**

Structural parameter [Å]	**2**	**7**	**8**
CCDC number	607705	677853	677854
Ge(1)–Cl(1)	2.2550(8)	2.289(1)	2.254(1)
Ge(1)–Cl(2)	2.1484(8)	2.299(1)	2.292(1)
Ge(1)–O(1)	2.066(2)	2.040(3)	2.097(3)
Ge(1)–O(2)	1.781(2)	1.804(3)	1.810(3)
Ge(1)–O(1’)		2.069(3)	2.010(3)
Ge(1)–C(3)	1.747(3)		
Ge(1)–C(1’)		1.968(4)	1.966(4)
O(1)–C(1)	1.258(3)	1.264(5)	1.254(5)
O(2)–C(2)	1.428(3)	1.400(5)	1.417(5)
N(1’)–C(1’)		1.464(5)	1.317(5)
Structural parameter [°]			
O(1)–Ge(1)–Cl(1)	171.31(6)	171.36(9)	172.01(8)
O(1’)–Ge(1)–Cl(2)	89.25(6)	171.7(2)	172.73(9)
O(1)–Ge(1)–O(2)	82.25(8)	83.2(1)	82.2(1)
O(1’)–Ge(1)–C(1’)		82.4(4)	82.5(2)
C(1’)–Ge(1)–O(2)		169.3(2)	165.5(2)
C(1)–O(1)–Ge(1)	112.5(2)	111.1(3)	111.2(3)
N(1’)–C(2’)–Ge(1)		108.1(3)	108.5(3)

Table 2. Crystallographic parameters for structures **2**, **7**, and **8**

Crystallographic parameters	**2**	**7**	**8**
Diffractometer	Bruker Smart 1000	Bruker APEX II	Bruker APEX II
Empirical formula	$C_{17}H_{20}Cl_3GeNO_2$	$C_{21}H_{24}Cl_2GeN_2O_4$	$C_{21}H_{22}Cl_2GeN_2O_3$
Temperature (K)	120	100	100
Formula weight	449.28	511.91	493.90
Crystal size (mm)	0.05 × 0.05 × 0.05	0.40 × 0.30 × 0.10	0.10 × 0.10 × 0.07
μ[MoKα] (cm^{-1})	19.46	16.30	17.72
Density (calc., g cm^{-3})	1.496	1.512	15.92
a (Å)	25.881(3)	15.470(1)	10.420(4)
b (Å)	7.6104(10)	12.9518(8)	10.991(4)
c (Å)	20.322(3)	22.791(2)	11.289(7)
α (°)			102.034(11)
β (°)	94.460(3)	100.025(2)	110.567(12)
γ (°)			112.205(8)
V ($Å^3$)	3990.6(9)	4496.9(5)	1030.2(9)
Space group, Z	8	8	2
$2\theta_{max}$ (°)	60.02	55.76	54.2
F(000)	1824	2096	504
Scan type		ω	ω
Reflections collected	14911	49584	10815
Independent reflections (R_{int})	5710(0.0398)	10688(0.092)	4539(0.0994)
Number of reflections with $I > 2\sigma(I)$	3685	6086	2947
Parameters	220	549	504
T_{min}/T_{max}	0.909/0.909	0.854/0.562	0.886/ 0.843
Goodness-of-fit	1.053	1.03	0.983
R_1 [$I > 2\sigma(I)$]	0.0451	0.0509	0.0537
wR_2 (all reflections)	0.0962	0.1036	0.1028

The presence of only one equatorial Cl atom in monochelate **2** shortens the Ge(1)–O(1) coordination bond (Figure 1) as compared with pentacoordinate complexes containing $GeCl_3$ moieties: the Ge(1)–O(1) distance in **2** is 2.066(2) Å, whereas the respective coordination bond lengths in known monochelate trichlorides vary from 2.38 to 2.19 Å.[9–11] The difference between Ge(1)–Cl(1) and Ge(1)–Cl(2) bond lengths in **2** (0.1 Å) is noticeably larger than in germanium trichlorides (0.04–0.06 Å). Thus, the coordination polyhedron of Ge(1) is closer to the ideal trigonal bipyramid than in $GeCl_3$ complexes. The deviation of Ge(1) from the equatorial plane O(2)Cl(2)C(3) is 0.16 Å. Repulsion between the Cl atoms leads to a noticeable distortion of the axial fragment O(1)Ge(1)Cl(1) and reduces the corresponding angle to 171.31(6)°.

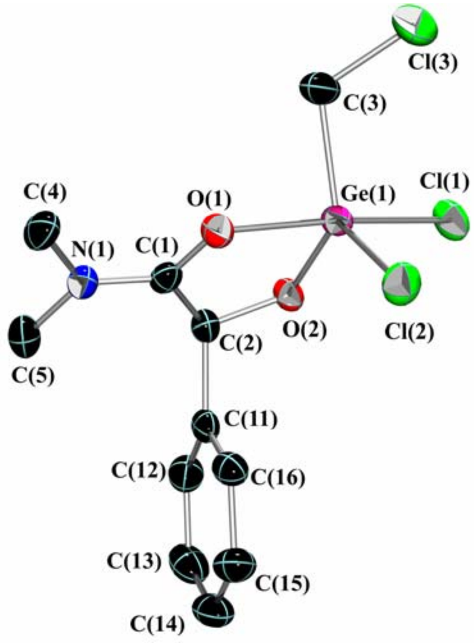


Figure 1. ORTEP view of **2**. Atoms are presented as thermal ellipsoids at 50% probability. Hydrogen atoms are omitted for clarity.

Conformations of five-membered chelate rings in germanium trichlorides are nearly planar[9,11] or a planarized twist.[10] In contrast, the conformation of the chelate ring in complex **2** is a planarized envelope with the deviation of C(1) atom from the basal plane of 0.12 Å.

In the mixed-ligand bischelate complexes **7** and **8** (Figure 2) the two different chelate rings cause noticeable distortions of the Ge(1) coordination polyhedra. The configuration of **7** and **8** with respect to Cl, O(1) and O(1') can be described as *trans*, with angles O(1)Ge(1)Cl(1) and O(1')Ge(1)Cl(2) smaller than 180º (Table 1). The most pronounced distortion of the coordination polyhedron of Ge(1) is observed for **8** with the two coordination bonds Ge(1)–O(1) and Ge(1)–O(1') differing by 0.09 Å; the Ge–Cl distances differs by 0.04 Å. The conformation of the five-membered chelate ring formed by the mandelic amide moiety is a planarized twist with C(2) deviating from the basal plane by 0.16 Å. In **7**, the chelate ring formed by the benzoxazinone moiety adopts an envelope conformation with Ge(1) deviating from the basal plane by 0.24 Å.

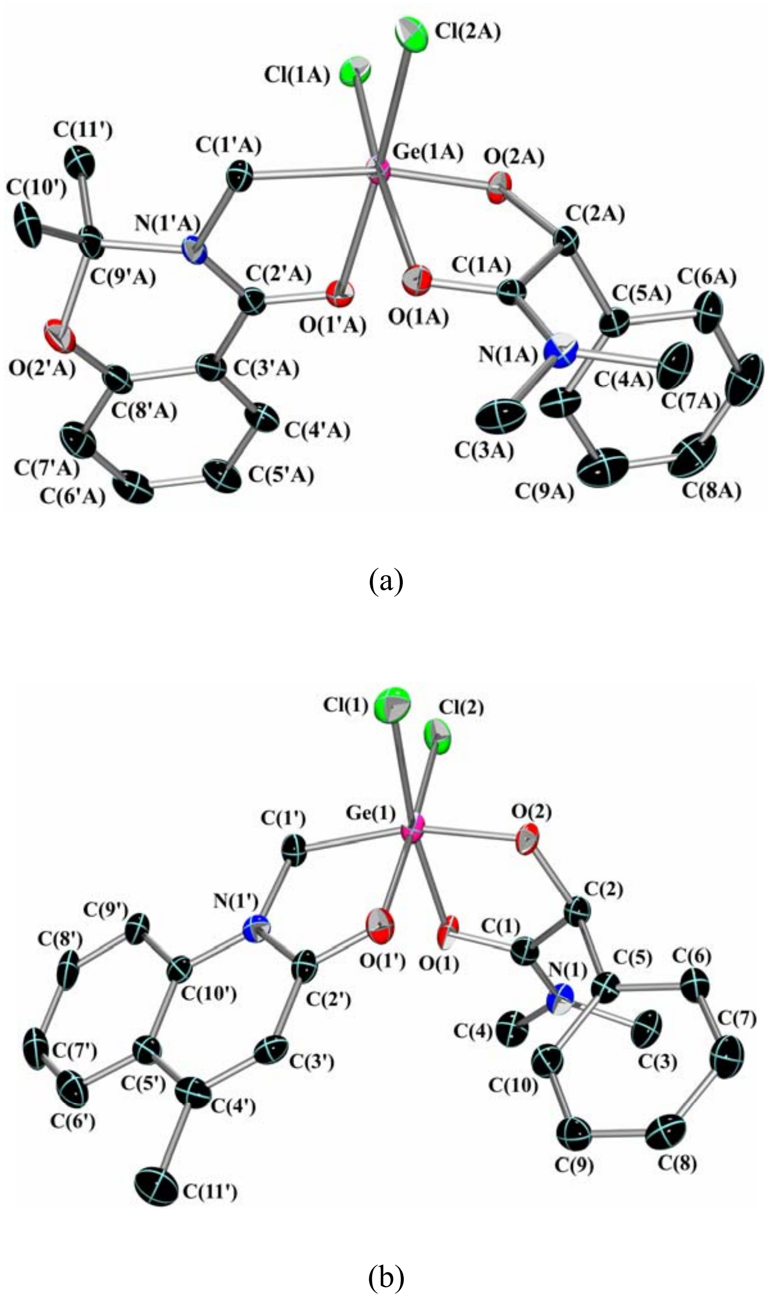


Figure 2. (a) Molecule **A** in the crystal structure of **7**. (b) ORTEP view of **8**. Atoms are presented as thermal ellipsoids at 50% probability. Hydrogen atoms are omitted for clarity.

The coordination polyhedron of Ge(1) in **7** is less distorted than in **8** (Table 2). The geometries of two independent molecules **A** and **B** in the crystal structure of **7** are nearly identical but with opposite configuration (*R* and *S*) of the stereocenters C(2A) and C(2B), respectively. The conformations of the chelate ring formed by mandelic amide moieties are envelopes with deviations of Ge(1A) and Ge(1B) of 0.33 and 0.31 Å, respectively. Similar conformations are adopted by the chelate rings formed by quinolinone moieties; Ge(1A) and Ge(1B) deviate from the basal planes by 0.41 and 0.41 Å, respectively.

It should be noted that the mixed complexes **7** and **8** are the first examples of germanium bischelates with different chelate rings.[12]

Experimental Section

General Procedures. The IR spectra of the compounds were recorded as ~5% solutions or as KBr pellets on a Specord IR-75 instrument. ^{1}H and ^{13}C NMR spectra were measured on a Varian XL-400 spectrometer (400.1 and 100.6 MHz, respectively). The diffraction measurements were carried out with SMART 1000 CCD and Smart APEX II diffractometers. Selected structural parameters of **2, 7** and **8** are listed in Table 1. Details of crystallographic data and experimental conditions are given in Table 2. All structures were solved by direct method and refined by full-matrix least-squares technique against F^2 in the anisotropic-isotropic approximation. Hydrogen atoms were located from difference electron density syntheses and refined in rigid body model. All calculations were carried out with the SHELXTL PLUS 5.10 program package.[13] Crystallographic data for **2**, **7**, and **8** have been deposited at the Cambridge Crystallographic Data Centre. The CCDC numbers are listed in Table 1. Copies of the data can be obtained free of charge upon application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(1223)336033; e-mail: deposit@ccdc.cam.ac.uk.
2-[Dichloro(chloromethyl)germyloxy]-*N*,*N*-dimethyl-2-phenylacetamide **(2)**[6] and 2,2-dimethyl-3-(trimethylsilyl)-2*H*-benzo[*e*][1,3]oxazin-4(3*H*)-one (**5**)[14] were synthesized as reported.

4-Methyl-1-(trimethylsilyl)quinolin-2(1*H*)-one (**6**)**.** Chlorotrimethylsilane (54 g, 0.5 mol) was added dropwise to a solution of 4-methylquinolin-2-one (47.6 g, 0.3 mol) and Et_3N (50 g, 0.5 mol) in benzene (150 mL). The mixture was refluxed for 2 h. Next day, the precipitate formed was filtered off, the volatiles were removed in vacuo, and the residue was distilled to afford a colorless oil **6** (63 g, 91%); bp 152–153 °C (10 mm Hg). IR ($CHCl_3$): $\tilde{\nu}$ 1670, 1615, 1510 (C=O) cm^{-1}. ^{1}H NMR (400.1 MHz, $CDCl_3$): δ 0.1 [9 H, $Si(Me)_3$], 2.08 (3 H, s, CH_3), 6.42 (1 H, m, CH), 7.0–8.1 (4 H, m, Ar). Anal. calcd. for $C_{13}H_{17}NOSi$: C, 67.49; H, 7.41; N, 6.05. Found: C, 67.60, H, 7.40, N, 5.89.

2-[Dichloro[(2,2-dimethyl-4-oxo-2*H*-benzo[*e*][1,3]oxazin-3(4*H*)-yl)methyl]germyloxy]-*N*,*N*-dimethyl-2-phenylacetamide (7)

Method A. A mixture of amide **2** (1.1 g, 3 mmol) and compound **5** (0.75 g, 3 mmol) in toluene (15 mL) was refluxed for 3 h. The volatiles were removed in vacuo, the residue was crystallized with hexane (10 mL) to afford colorless crystals **7** (1.4 g, 91%); mp 220–222 °C (benzene/ hexane 15:1). IR (CH_2Cl_2): $\tilde{\nu}$ 1607, 1564, 1500 (NCO, Ar) cm^{-1}. ^{1}H NMR (400.1 MHz, $CDCl_3$): δ 1.78, 1.80 (3 H each, 2 s, CMe_2), 2.76, 3.11 (3 H each, 2 s, NMe_2), 3.78, 3.83 (1 H each, AB, ^2J

= 11.4 Hz, NCH_2), 7.33–7.47 (5 H, m, Ph), 5.67 (1 H, s, CH), 7.00 (1 H, d, ^3J = 7.8 Hz, H-9), 7.14 (1 H, t, ^3J = 7.8 Hz, H-7), 7.57 (1 H, t, ^3J = 7.8 Hz, H-8), 7.92 (1 H, d, ^3J = 7.8 Hz, H-6). ^{13}C NMR (100.6 MHz, DMSO-d_6): δ 20.37 (Me_2), 94.05 ($\underline{C}Me_2$), 53.51 (NMe_2), 68.25 (CHO), 43.71 (NCH_2), 127.66, 129.27, 130.60, 139.47 (Ph), 122.24, 117.96, 135.00, 123.17, 129.29, 156.04 (Ar), 163.41 (C=O), 173.34 (C=O). Anal. calcd. for $C_{21}H_{24}Cl_2GeN_2O_4$: C, 49.27; H, 4.72; N, 5.47. Found: C, 49.19; H, 4.75; N, 5.41.

Method B. A mixture of compound **5** (2.49 g, 0.01 mol) and $ClCH_2GeCl_3$ (2.28 g, 0.01 mol) in toluene (5 mL) was refluxed for 3 h. Next day, the volatiles were removed in vacuo, the residue was dissolved in hot MeCN (10 mL), and amide **1** (2.5 g, 0.01 mol) was added. The mixture was refluxed for 3 h, the volatiles were removed in vacuo, the residue was crystallized with ether (10 mL) to afford colorless crystals **7** (3.3 g, 64%); mp 217–221 °C (benzene/ hexane 15:1). Anal. calcd. for $C_{21}H_{24}Cl_2GeN_2O_4$: C, 49.27; H, 4.72; N, 5.47. Found: C, 49.34; H, 4.71; N, 5.34. IR and NMR spectra of this product are identical with the spectra of **7** obtained by Method a.

2-[Dichloro[(4-methyl-2-oxoquinolin-1(2*H*)-yl)methyl]germyloxy]-*N*,*N*-dimethyl-2-phenylacetamide (8). A mixture of amide **2** (1.1 g, 3.0 mmol) and compound **6** (0.69 g, 3.0 mmol) in toluene (15 mL) was refluxed for 2 h. Next day, the crystals formed were filtered off and recrystallized from MeCN to afford colorless crystals **8** (1.32 g, 89%); mp 266–270 °C (MeCN). IR (KBr): $\tilde{\nu}$ 1628, 1532 (NCO), 1568, 1504 cm^{-1}. ^1H NMR (400.1 MHz, DMSO-d_6): δ 1.83 (3 H, s, CMe), 2.83, 3.09 (3 H each, two s, NMe_2), 3.32, 3.45 (1 H each, AB, ^2J = 10.9 Hz, NCH_2), 5.91 (1 H, s, CH), 7.11–7.96 (9 H, m, Ph, Ar). ^{13}C NMR (100.6 MHz, DMSO-d_6): δ 19.75 (Me), 49.50 (NMe_2), 65.78 (CHO), 47.87 (NCH_2), 127.68, 128.87, 132.65, 139.56 (Ph), 118.45 (CH), 143.04, 123.06, 129.99, 124.87, 128.89, 141.04 (Ar), 161.01 (C=O), 178.73 (C=O). Anal. calcd. for $C_{21}H_{22}Cl_2GeN_2O_3$: C, 51.06; H, 4.48; N, 5.67. Found: C, 51.09; H, 4.49; N, 5.77.

Acknowledgements

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