

β -Methyl- γ,γ -bis(8-quinolinoxy)germa- γ -lactoneSumera Mahboob,^a Masood Parvez,^b Muhammad Mazhar^{a*} and Saqib Ali^a^aDepartment of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan, and ^bDepartment of Chemistry, The University of Calgary, 2500 University Drive NW, Calgary, Alberta, Canada T2N 1N4

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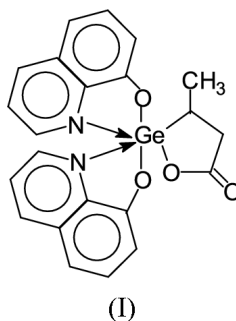
Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.040
 wR factor = 0.107
Data-to-parameter ratio = 17.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the hexacoordinated title germanium compound [alternatively called (butanoato- $\kappa^2\text{C}^3,\text{O}$)bis(8-quinolato- $\kappa^2\text{N},\text{O}$)-germanium(IV)], $[\text{Ge}(\text{C}_9\text{H}_7\text{NO})_2(\text{C}_4\text{H}_6\text{O}_2)]$, the Ge atom adopts a slightly distorted octahedral geometry, where the Ge–N coordinate covalent bonds [mean 2.079 (8) Å] are significantly longer than the Ge–O [mean 1.904 (8) Å] and Ge–C [1.976 (3) Å] covalent bonds.

Comment

As an *N,O*-bidentate ligand, 8-quinolinol is used for the identification of metals (Phillips, 1956). Its applications as a component for molecular electronic devices (Tang & VanSlyke, 1987), a supramolecular motif (Albrecht & Blau, 1997) and an effective external antiseptic are of current interest. Recently, we have synthesized a metal chelate of 8-quinolinol with a trichlorogermyl of β -methylpropionic acid, where the O and N atoms are involved in the metal chelation. Subsequently, an intramolecular nucleophilic substitution takes place on the Ge atom to form the title germa- γ -lactone, (I). Compound (I) is an interesting example of blocked carbonyl compounds found in many natural products and insect sex pheromones (Ravid *et al.*, 1978; Cardellach *et al.*, 1982). These may act as versatile intermediates against different diseases (Herrmann *et al.*, 1979; Damon & Schlesinger, 1976). In this article, we report the structure of (I).



The structure contains monomeric molecules of (I) separated by normal van der Waals distances (Fig. 1). The hexacoordinated Ge centre is bonded to two N atoms, three O atoms and one C atom, yielding a slightly distorted octahedral geometry. The atoms O1/O3/O4/N2 lie in a plane to within 0.0081 (11) Å, while the Ge atom lies 0.2086 (11) Å out of this plane, directed towards atom C3 of the β -methylpropionate moiety. The N1–Ge1–C3 angle is close to linear, at 170.43 (11)°. The Ge–N bonds [mean 2.079 (8) Å] are significantly longer than the Ge–O [mean 1.904 (8) Å] and Ge–C [1.976 (3) Å] covalent bonds. The corresponding bond distances in organotin chelates of 8-quinolinol (Schümann *et*

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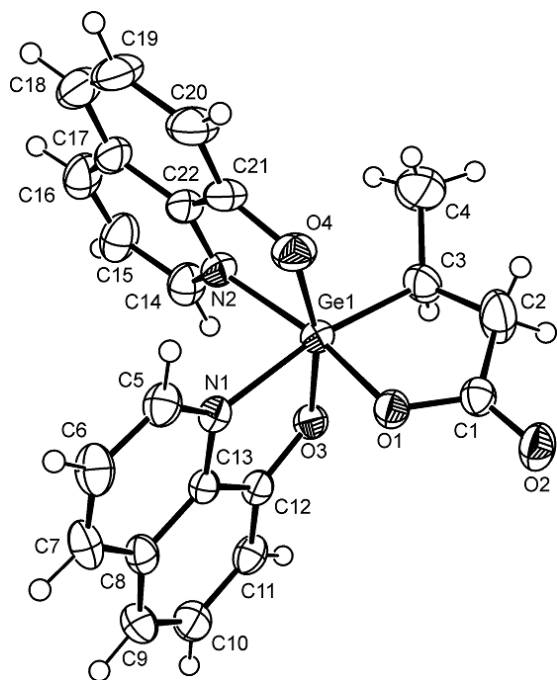


Figure 1

A drawing of (I), with the atom-numbering scheme. Displacement ellipsoids are plotted at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

al., 1999) are significantly longer than those observed in (I). Moreover, the Ge–N coordinate covalent bonds in (I) are significantly shorter than the corresponding bonds in a five-coordinated germa- γ -lactone (Chen *et al.*, 1995). The quinolinium groups N1/C5–C13 and N2/C14–C22 are individually planar, with atoms O3 and O4 0.029 (3) and 0.062 (4) Å, respectively, out of the mean planes of these groups, which are inclined almost at right angles to each other [dihedral angle 84.79 (6)°].

Experimental

Compound (I) was synthesized by the reaction of $\text{GeCl}_3\text{CH}(\text{CH}_3)\text{CH}_2\text{CO}_2\text{H}$ (11.27 mmol, 1 equivalent; Hans *et al.*, 2002) with 8-quinolinol (45.11 mmol, 4 equivalents) in dry MeOH (70 ml) under reflux in the presence of concentrated ammonia (1.8 ml). The solid product thus obtained was filtered off, washed with MeOH and dried under vacuum. Crystals of (I) were grown from a 1:1 acetone/*n*-hexane mixture in a freezer. Spectroscopic analysis: ^1H NMR (CDCl_3 , δ , p.p.m.): 1.20 (*d*, 3H, $J = 7.5$ Hz), 1.91 (*m*, 1H), 2.54 (*dd*, 2H, $J = 3.8$ and 8.1 Hz), 8.18 (*m*, 12H); analysis, calculated for $\text{C}_{22}\text{H}_{18}\text{GeN}_2\text{O}_4$: C 59.12, H 4.03, N 6.27%; found: C 59.10, H 4.00, N 6.21%.

Crystal data

$[\text{Ge}(\text{C}_9\text{H}_7\text{NO})_2(\text{C}_4\text{H}_6\text{O}_2)]$

$M_r = 446.97$

Orthorhombic, *Pbca*

$a = 9.695$ (2) Å

$b = 17.266$ (4) Å

$c = 23.543$ (4) Å

$V = 3941.0$ (14) Å³

$Z = 8$

$D_x = 1.507$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 8481

reflections

$\theta = 3.0$ – 27.5°

$\mu = 1.59$ mm⁻¹

$T = 173$ (2) K

Prism, yellow

$0.14 \times 0.12 \times 0.10$ mm

Data collection

Nonius KappaCCD area-detector diffractometer

ω and φ scans

Absorption correction: multi-scan (SORTAV; Blessing, 1997)

$T_{\min} = 0.809$, $T_{\max} = 0.858$

8481 measured reflections

4504 independent reflections

3256 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 27.5^\circ$

$h = -12 \rightarrow 12$

$k = -22 \rightarrow 22$

$l = -30 \rightarrow 30$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.107$

$S = 1.01$

4504 reflections

263 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0471P)^2 + 4.162P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.96$ e Å⁻³

$\Delta\rho_{\min} = -0.84$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Ge1–O4	1.898 (2)	O2–C1	1.216 (4)
Ge1–O3	1.899 (2)	O3–C12	1.336 (3)
Ge1–O1	1.916 (2)	O4–C21	1.350 (4)
Ge1–C3	1.976 (3)	N1–C5	1.322 (4)
Ge1–N2	2.071 (2)	N1–C13	1.362 (3)
Ge1–N1	2.087 (2)	N2–C14	1.321 (4)
O1–C1	1.310 (3)	N2–C22	1.357 (4)
O4–Ge1–O3	166.11 (9)	O1–Ge1–N1	82.66 (8)
O4–Ge1–O1	91.70 (9)	C3–Ge1–N1	170.43 (11)
O3–Ge1–O1	93.61 (8)	N2–Ge1–N1	85.10 (9)
O4–Ge1–C3	99.29 (12)	C1–O1–Ge1	113.8 (2)
O3–Ge1–C3	93.58 (12)	C12–O3–Ge1	116.6 (2)
O1–Ge1–C3	89.67 (11)	C21–O4–Ge1	114.6 (2)
O4–Ge1–N2	82.30 (10)	C5–N1–C13	119.3 (3)
O3–Ge1–N2	89.75 (9)	C5–N1–Ge1	130.8 (2)
O1–Ge1–N2	166.66 (9)	C13–N1–Ge1	109.8 (2)
C3–Ge1–N2	103.02 (12)	C14–N2–C22	120.5 (3)
O4–Ge1–N1	86.69 (9)	C14–N2–Ge1	129.5 (2)
O3–Ge1–N1	81.27 (8)	C22–N2–Ge1	109.9 (2)

H atoms were included at geometrically idealized positions, with $\text{C–H} = 0.95$ – 1.00 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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