

3966 measured reflections
3667 independent reflections

3 standard reflections
every 250 reflections
intensity decay: 1%

Refinement

Refinement on F^2

$R = 0.056$

$wR = 0.048$

$S = 1.17$

2747 reflections

249 parameters

H atoms riding

$$w = 1/[\sigma^2(I) + (0.04F^2)^2]^{1/2}/(2F)^2$$

$$(\Delta/\sigma)_{\max} = 0.01$$

$$\Delta\rho_{\max} = 0.36 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -0.19 \text{ e } \text{Å}^{-3}$$

Extinction correction: none

Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. Selected geometric parameters (Å , $^\circ$)

Cu—O1	1.959 (2)	N1—C12	1.341 (4)
Cu—O6	2.485 (2)	N2—C13	1.336 (4)
Cu—N1	2.019 (2)	N2—C14	1.471 (4)
O1—C1	1.267 (3)	N2—C16	1.467 (4)
O2—C1	1.237 (4)	N3—C5	1.472 (4)
O3—C13	1.229 (3)	C1—C2	1.509 (4)
O4—N3	1.213 (4)	C11—C13	1.510 (4)
O5—N3	1.213 (4)	C14—C15	1.485 (6)
N1—C8	1.341 (4)	C16—C17	1.503 (6)
C8—N1—C12	118.2 (2)	O1—C1—C2	115.4 (2)
C13—N2—C14	124.2 (3)	O2—C1—C2	118.3 (3)
C13—N2—C16	118.2 (2)	N1—C8—C9	121.7 (3)
C14—N2—C16	116.2 (2)	C10—C11—C13	124.6 (2)
O4—N3—O5	123.7 (3)	C12—C11—C13	116.7 (3)
O4—N3—C5	117.7 (3)	O3—C13—N2	123.4 (3)
O5—N3—C5	118.6 (3)	O3—C13—C11	117.5 (3)
O1—C1—O2	126.3 (3)	N2—C13—C11	119.1 (2)

The structure was solved by the Patterson method. The positions of the water H atoms were obtained from a $\Delta\rho$ map and refined isotropically. The other H-atom positions were calculated geometrically with $U_{\text{eq}}(\text{H}) = 1.3U_{\text{eq}}$ of the parent non-H atom and a riding model was used in the refinement.

Data collection: *MolEN* (Fair, 1990). Cell refinement: *MolEN*. Data reduction: *MolEN*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *MolEN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1457). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). **C53**, 1051–1054

Isostructural Complexes of Diiodo-(phthalocyaninato)germanium(IV) and Diiodo(phthalocyaninato)tin(IV)

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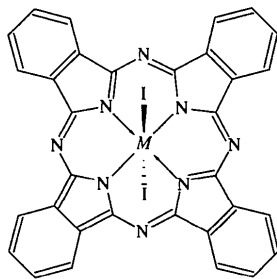
Abstract

New diiodo(phthalocyaninato- κ^4)germanium(IV), $[\text{GeI}_2(\text{C}_{32}\text{H}_{16}\text{N}_8)]$ (GePcI_2 , where $\text{Pc} = \text{C}_{32}\text{H}_{16}\text{N}_8$), and diiodo(phthalocyaninato- κ^4)tin(IV), $[\text{SnI}_2(\text{C}_{32}\text{H}_{16}\text{N}_8)]$ (SnPcI_2), complexes have been obtained. The phthalocyanine residues are centrosymmetric and are not staggered but slipped. In both cases, the phthalocyanine ring is not strictly planar. The M —I axis is tilted 1.9° in GePcI_2 and 3.0° in SnPcI_2 from the normal to the N_4 -isoindole plane. In both compounds, the metal atoms (Ge and Sn) lie on inversion centers and are octahedrally coordinated by the four isoindole N and the two I atoms.

Comment

The crystals of GePcI_2 , (1), and SnPcI_2 , (2), are built from molecules of the title compounds (Fig. 1), with the intermolecular $\text{I} \cdots \text{H}$ contacts as shown in Fig. 2. The

lengths of I1···H8ⁱ and I1···H3ⁱⁱ [symmetry codes: (i) $x, 1+y, z$; (ii) $x, y, z-1$] are 2.99 (1) and 3.14 (1) Å, respectively, in (1), and 3.13 (1) and 3.15 (1) Å in (2).



(1) $M = \text{Ge}$
(2) $M = \text{Sn}$

The most remarkable feature of the present structures is that the phthalocyaninato (Pc) molecules are not staggered but slipped. Apart from (GePcO)_n (Dirk, Inabe, Schoch & Marks, 1983), and (AlPc)₂O and (GaPcF)₂ (Wynne, 1985), in which the Pc rings are

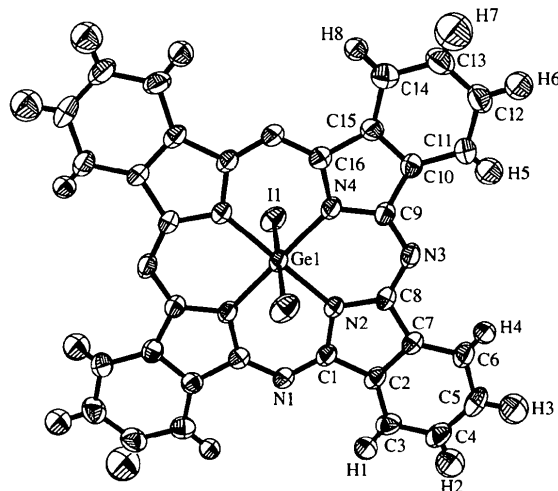


Fig. 1. The molecular structure of Ge^{IV}PcI₂. Displacement ellipsoids are plotted at the 50% probability level. The atom-numbering scheme is analogous for Sn^{IV}PcI₂.

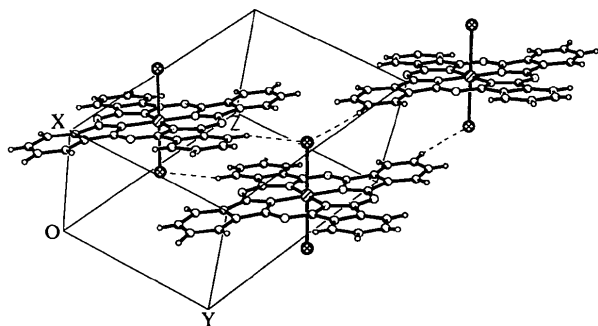


Fig. 2. The molecular arrangement in the unit cell.

eclipsed, in all reported phthalocyanine structures with collinear Pc's stacking, the rings are staggered. In (1) and (2), the phthalocyanine rings are not strictly planar. The angle between the isoindole planes is 6.7 (2) in (1) and 8.1 (1)° in (2). The largest deviation (besides H and I atoms) from the N₄-isoindole plane in both structures is that of the C5 atoms. In (1), this deviation is 0.598 (11) Å, whereas in (2), it rises to 0.923 (8) Å. The M—I axis is tilted 1.9° in (1) and 3.0° in (2) from the normal to the N₂—M—N₄ plane. In SnPcCl₂, the analogous axis is tilted by 2.5° (Rogers & Osborn, 1971).

In both compounds, the metal atom is at an inversion center and is octahedrally coordinated by the N₄-isoindole atoms and the two I atoms. The Ge—N distances are comparable to those observed in GePc(OH)₂ (Krämer, 1985). The Sn—N distances are comparable to those observed in SnPcCl₂ (Rogers & Osborn, 1971) and shorter than the values in SnPc₂ (Bennett, Broberg & Beanziger, 1973; Janczak & Kubiak, 1994). The M—I distance in (1) is shorter than that in SnPcI₂ (Tables 1 and 2). Similar Sn—I distances reported for six-coordinated tin(IV) compounds are in the range from 2.634 (Aslanov, Tursina, Chernyshev, Medvedev & Yatsenko, 1985) to 3.186 Å (Mitzi, Wang, Field, Chess & Guloy, 1995). The average interatomic distances in the phthalocyanine ring are 1.377 (3) [C—N(isoindole)], 1.318 (2) [C—N(azamethine)] and 1.400 (7) Å (C—C) in (1), and 1.368 (4), 1.331 (2) and 1.405 (8) Å, respectively, in (2). These distances are similar to those observed in other phthalocyanine structures.

Experimental

The crystals of GePcI₂ and SnPcI₂ were obtained by reaction of the appropriate pure metal with 1,2-dicyanobenzene (Kubiak & Janczak, 1993) under a stream of iodine.

Compound (1)

Crystal data

[GeI₂(C₃₂H₁₆N₈)]

$M_r = 838.931$

Triclinic

$P\bar{1}$

$a = 7.522 (2) \text{ \AA}$

$b = 8.663 (2) \text{ \AA}$

$c = 11.349 (2) \text{ \AA}$

$\alpha = 72.61 (3)^\circ$

$\beta = 78.96 (3)^\circ$

$\gamma = 87.31 (3)^\circ$

$V = 692.6 (3) \text{ \AA}^3$

$Z = 1$

$D_x = 2.0113 \text{ Mg m}^{-3}$

D_m not measured

Data collection

Kuma KM-4 diffractometer

ω - 2θ scans

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 15

reflections

$\theta = 10.28\text{--}13.93^\circ$

$\mu = 3.375 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Parallelepiped

$0.4 \times 0.3 \times 0.2 \text{ mm}$

Red-violet

$R_{\text{int}} = 0.036$

$\theta_{\text{max}} = 32.07^\circ$

Absorption correction: empirical *via* ψ scans (Sheldrick, 1991)
 $T_{\min} = 0.327$, $T_{\max} = 0.535$
 7246 measured reflections
 4283 independent reflections
 2926 reflections with $I > 2\sigma(I)$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.083$
 $S = 1.165$
 4283 reflections
 228 parameters
 All H atoms refined
 $w = 1/[\sigma^2(F_o^2) + (0.0393P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

$h = -11 \rightarrow 11$
 $k = -12 \rightarrow 12$
 $l = -16 \rightarrow 16$
 2 standard reflections every 50 reflections
 intensity decay: $< 1.01\%$

$(\Delta/\sigma)_{\max} = 0.020$
 $\Delta\rho_{\max} = 0.808 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.593 \text{ e } \text{\AA}^{-3}$
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Data collection

Kuma KM-4 diffractometer
 ω - 2θ scans
 Absorption correction: empirical *via* ψ scans (Sheldrick, 1991)
 $T_{\min} = 0.420$, $T_{\max} = 0.686$
 8248 measured reflections
 4365 independent reflections
 3563 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$
 $\theta_{\max} = 31.06^\circ$
 $h = -11 \rightarrow 11$
 $k = -12 \rightarrow 12$
 $l = -16 \rightarrow 16$
 2 standard reflections every 50 reflections
 intensity decay: $< 1.01\%$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.074$
 $S = 1.083$
 4365 reflections
 228 parameters
 All H atoms refined
 $w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 0.3021P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 1.11 \text{ e } \text{\AA}^{-3}$
 (at 0.87 \AA from I1)
 $\Delta\rho_{\min} = -1.12 \text{ e } \text{\AA}^{-3}$
 (at 0.73 \AA from I1)
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (\AA , $^\circ$) for (1)

II—Ge1	2.6869 (10)	N4—C9	1.372 (4)
Ge1—N4	1.946 (3)	N4—C16	1.385 (4)
Ge1—N2	1.963 (3)	C1—C2	1.442 (4)
N1—C1	1.315 (5)	C2—C7	1.395 (5)
N1—C16'	1.318 (4)	C7—C8	1.442 (5)
N2—C8	1.372 (4)	C9—C10	1.435 (5)
N2—C1	1.379 (5)	C10—C15	1.389 (5)
N3—C9	1.317 (4)	C15—C16'	1.458 (5)
N3—C8	1.322 (5)		
N4—Ge1—N2	89.94 (12)	C6—C7—C2	121.2 (3)
N4—Ge1—II	90.56 (9)	C6—C7—C8	132.1 (3)
N2—Ge1—II	91.78 (9)	C2—C7—C8	106.7 (3)
C8—N2—C1	108.9 (3)	N3—C8—N2	127.8 (3)
C8—N2—Ge1	125.2 (2)	N3—C8—C7	123.4 (3)
C1—N2—Ge1	125.2 (2)	N2—C8—C7	108.9 (3)
C9—N3—C8	122.9 (3)	N3—C9—N4	127.9 (3)
C9—N4—C16	108.7 (3)	N3—C9—C10	123.3 (3)
C9—N4—Ge1	125.9 (2)	N4—C9—C10	108.7 (3)
C16—N4—Ge1	125.4 (2)	C15—C10—C11	120.8 (4)
N1—C1—N2	128.3 (3)	C15—C10—C9	108.0 (3)
N1—C1—C2	123.4 (3)	C11—C10—C9	131.2 (3)
N2—C1—C2	108.3 (3)	C14—C15—C10	121.9 (3)
C7—C2—C3	120.9 (3)	C14—C15—C16'	131.8 (3)
C7—C2—C1	107.3 (3)	C10—C15—C16'	106.2 (3)
C3—C2—C1	131.8 (4)	N4—C16'—C15	108.4 (3)

Symmetry code: (i) $1 - x, -y, 1 - z$.

Compound (2)

Crystal data

$[\text{SnI}_2(\text{C}_{32}\text{H}_{16}\text{N}_8)]$

$M_r = 885.031$

Triclinic

$P\bar{1}$

$a = 7.618 (2) \text{ \AA}$

$b = 8.551 (2) \text{ \AA}$

$c = 11.602 (2) \text{ \AA}$

$\alpha = 73.34 (3)^\circ$

$\beta = 77.20 (3)^\circ$

$\gamma = 87.05 (3)^\circ$

$V = 706.0 (3) \text{ \AA}^3$

$Z = 1$

$D_x = 2.0816 \text{ Mg m}^{-3}$

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 24 reflections

$\theta = 9.69\text{--}20.80^\circ$

$\mu = 3.131 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Parallelepiped

$0.4 \times 0.3 \times 0.2 \text{ mm}$

Red-violet

Table 2. Selected geometric parameters (\AA , $^\circ$) for (2)

II—Sn1	2.8222 (10)	N4—C9	1.359 (3)
Sn1—N4	2.044 (2)	N4—C16	1.376 (3)
Sn1—N2	2.056 (2)	C1—C2	1.452 (3)
N1—C16'	1.327 (3)	C2—C7	1.399 (4)
N1—C1	1.331 (3)	C7—C8	1.455 (3)
N2—C8	1.367 (3)	C9—C10	1.455 (3)
N2—C1	1.368 (3)	C10—C15	1.400 (4)
N3—C8	1.329 (3)	C15—C16	1.453 (4)
N3—C9	1.336 (3)		
N4—Sn1—N2	90.06 (9)	C6—C7—C2	121.7 (2)
N4—Sn1—II	90.76 (8)	C6—C7—C8	130.9 (3)
N2—Sn1—II	92.94 (7)	C2—C7—C8	107.4 (2)
C8—N2—C1	111.7 (2)	N3—C8—N2	128.2 (2)
C8—N2—Sn1	123.1 (2)	N3—C8—C7	125.1 (2)
C1—N2—Sn1	123.3 (2)	N2—C8—C7	106.7 (2)
C8—N3—C9	125.8 (2)	N3—C9—N4	128.2 (2)
C9—N4—C16	112.0 (2)	N3—C9—C10	124.7 (2)
C9—N4—Sn1	123.9 (2)	N4—C9—C10	107.1 (2)
C16—N4—Sn1	124.0 (2)	C11—C10—C15	121.8 (3)
N1—C1—N2	128.1 (2)	C11—C10—C9	131.3 (3)
N1—C1—C2	125.0 (2)	C15—C10—C9	106.9 (2)
N2—C1—C2	106.9 (2)	C14—C15—C10	120.5 (3)
C3—C2—C7	120.8 (3)	C14—C15—C16	131.6 (3)
C3—C2—C1	131.9 (3)	C10—C15—C16	107.8 (2)
C7—C2—C1	107.2 (2)	N4—C16—C15	106.2 (2)

Symmetry code: (i) $1 - x, -y, 1 - z$.

For both compounds, data collection: *Kuma KM-4 Users Guide* (Kuma Diffraction, 1995); cell refinement: *Kuma KM-4 Users Guide*; data reduction: *Kuma KM-4 Users Guide*; program(s) used to solve structures: *SHELXTL-Plus* (Sheldrick, 1991); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *SHELXTL-Plus*; software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1316). Services for accessing these data are described at the back of the journal.

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A Crown-Linked Donor–Acceptor Assembly Containing Re^I(diimine)(CO)₃Cl and Nitrobenzene Components

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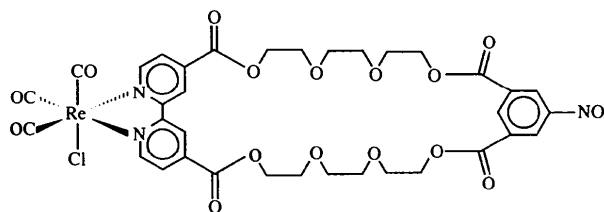
(Received 6 February 1996; accepted 9 January 1997)

Abstract

The structure of the title assembly, tricarbonylchloro-[16-nitro-3,6,9,12,20,23,26,29-octaoxa-34,37-diazatetracyclo[34.3.1.1^{14,18}.1^{31,35}]dotetraconta-1(39),14,16,18(41),-31,33,35(42),36(40),37-nonaene-2,13,19,30-tetrone]-rhenium(I), [ReCl(C₃₂H₃₃N₃O₁₄)(CO)₃], has been determined *via* a single-crystal diffraction study. The rhenium ion is octahedronally coordinated. The bidentate bipyridine/crown ether/nitrobenzene assembly is folded such that the pyridyl rings and nitrobenzene moiety are almost coplanar.

Comment

In earlier work, Wrighton and co-workers introduced the complex *fac*-(bpy)Re(CO)₃Cl (bpy is 2,2'-bipyridine) and its derivatives as metal-to-ligand charge-transfer (MLCT) excited-state emitters (Wrighton & Morse, 1974). Subsequently, crystal and molecular structures for difluorophosphato (Horn & Snow, 1980) and bicarbonato (Guilhem, Pascard, Lehn & Ziessel, 1989) derivatives were reported. In addition, the crystal and molecular structure of a 2,2':6'2''-terpyridine analogue, *fac*-(σ²-terpy)Re(CO)₃Cl, has been reported (Anderson, Keene, Horn & Tiekink, 1990; Civitello *et al.*, 1993). We have utilized the Wrighton & Morse synthetic chemistry to prepare covalently linked donor–crown–acceptor complexes of Re^I in order, ultimately, to understand how non-covalently bound material in a cavity (crown) can modulate and facilitate intermediate to long-range electron-transfer (ET) kinetics. Recently, we reported on the synthesis and photo-redox reactivity of crown ether linked assemblies featuring various *fac*-tricarbonylrhenium(bi)pyridine moieties as donors and nitrobenzene as an acceptor (Yoon, Berg-Brennan, Lu & Hupp, 1992; Berg-Brennan, Yoon & Hupp, 1993). Studies revealed that the rate of light-induced forward ET is strongly dependent on the initial assembly conformation, and that the conformation is strongly dependent on temperature. We report here the crystal structure of the title compound, (1). While the MLCT excited state of (1) is insufficiently reducing to transfer an electron to the tethered acceptor, the compound is closely related structurally to bipyridine assemblies which do display such reactivity (Kazala, Yoon & Hupp, 1995).



(1)

A view of (1) together with our atomic numbering scheme is shown in Fig. 1. The Re atom lies at the center of a slightly distorted octahedron formed by three carbonyl groups (facial configuration), two *cis* N atoms of bipyridine, and a Cl atom. It is also observed that the assembly is folded with an offset rather than a cofacial bipyridine/nitrobenzene configuration. The structure is significantly disordered in the vicinity of C56 and C57. Since no restraints were applied, the disorder and associated large displacement parameter almost certainly account for the unrealistically short C56—