1991). The compounds $[Cu_2\{3,6\text{-bis}(3,5\text{-dimethyl-1-pyrazolyl})\text{pyridazine}\}(OH)Cl_2][CuCl_3(H_2O)].H_2O$ (III) (Thompson, Woon, Murphy, Gabe, Lee & Le Page, 1985), [*N*,*N*′-dimethylpiperazinium][$CuCl_3(H_2O)$] (IV) and [2-aminopyrimidinium][$CuCl_3(H_2O)$] (V) (Manfredini *et al.*, 1990) have been described. The configuration of the anion in (III) is intermediate between tetrahedral and square-planar. Compounds (IV) and (V) contain planar $[CuCl_3(H_2O)]^-$ groups linked by Cu···Cl bonds. The contact between $[CuCl_3(H_2O)]^-$ groups is closer for (I) than for (IV) [Cu···Cl = 3.106 (2), 3.110 (2) Å] or (V) [Cu···Cl = 2.996 (1), 3.169 (1) Å].

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Structure of Tetracarbonyl[(2,3,7,8,12,13,17,18-octaethylporphinato-germanio(IV)]iron

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Abstract. $[FeGe(C_{36}H_{44}N_4)(CO)_4]$, [(oep)GeFe(CO)$_4$], $M_r = 773.25$, triclinic, $P\bar{1}$, $a = 12.123\ (2)$, $b = 13.851\ (3)$, $c = 15.028\ (3)$ Å, $\alpha = 59.99\ (2)$, $\beta = 61.53\ (2)$, $\gamma = 69.15\ (2)°$, $V = 1897.9\ (7)$ Å^3, $Z = 2$, $D_x = 1.357$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 12.05$ cm^{-1}, $F(000) = 804$, $R(F) = 0.0410$ for 5098 reflections. [(oep)GeFe(CO)$_4$] has two coordinated metal units, which are linked by a double bond; the Ge—Fe bond distance is 2.370 (2) Å. The average Ge—N distance is 2.082 (6) ± 0.01 Å and the Ge atom lies 0.684 (1) Å above the four-N-atom plane towards the Fe atom, the Fe atom is in an axial position with Ge—Fe—C(53) = 178.9 (1)°, the average Fe—CO distance is 1.77 (1) ± 0.005 Å.

Experimental. Crystals were prepared according to Barbe, Guilard, Lecomte & Gerardin (1984). A black crystal, 0.25 × 0.18 × 0.12 mm, of [(oep)GeFe(CO)$_4$] recrystallized from toluene/heptane was mounted on an Enraf–Nonius CAD-4F diffractometer. Unit-cell dimensions at room temperature were obtained from accurate angle values of 25 reflections with $10 < \theta < 24°$ using monochromated Mo $K\alpha$ radiation. 9054 reflections were measured up to $(\sin\theta)/\lambda = 0.66$ Å^{-1} at room temperature ($-14 < h < 14$, $-16 < k < 16$, $0 < l < 17$); standard reflections 200, $2\bar{1}5$, $3\bar{1}4$ monitored every 3 h; ω–2θ scan; scan width $\Delta\omega = 0.9° + 0.35°\tan\theta$; scan speed 0.6 to 1.55° min^{-1}. No decay was observed and no absorption correction was applied. 5098 reflections [$I \geq 3\sigma(I)$], corrected for Lorentz and polarization effects, structure solved by interpretation of the Patterson map; all non-H atoms were refined anisotropically (*SHELX*76; Sheldrick, 1976); H atoms were found in difference Fourier maps and refined isotropically. At convergence, $\Delta/\sigma_{max} = -0.31$ for U_{11} of C(53), a residual Fourier map gave a maximum peak of 0.52 e Å^{-3}. Weighting scheme $w^{-1} = \sigma^2(F) + 0.0003F^2$. Atomic scattering factors were taken from *SHELX*76 and from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final residuals are $R(F) = 0.0410$, $wR(F) = 0.0372$, GOF = 1.421. Fractional coordinates and

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Table 1. *Positional parameters and equivalent isotropic temperature factors and their e.s.d.'s for the non-H atoms*

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $B_{eq} = (4/3) \times [a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	z	B_{eq} (Å^2)
Ge	0.16719 (7)	0.15717 (7)	0.78078 (7)	2.86 (3)
Fe	0.3303 (1)	0.26523 (9)	0.70004 (9)	3.71 (4)
O1	0.2245 (8)	0.4299 (6)	0.5312 (7)	8.8 (3)
O2	0.5234 (6)	0.0818 (6)	0.6481 (7)	9.0 (4)
O3	0.261 (1)	0.274 (1)	0.9107 (7)	16.1 (6)
O4	0.5290 (9)	0.3978 (8)	0.6066 (8)	10.8 (4)
N1	0.1231 (5)	0.1698 (5)	0.6575 (5)	3.0 (2)
N2	0.2528 (6)	−0.0063 (5)	0.7894 (5)	3.1 (2)
N3	0.1169 (6)	0.0803 (5)	0.9514 (5)	3.1 (2)
N4	−0.0101 (6)	0.2576 (5)	0.8181 (5)	3.2 (2)
C1	0.0440 (7)	0.2573 (6)	0.6055 (6)	3.3 (3)
C2	0.0672 (7)	0.2556 (7)	0.5031 (6)	3.7 (3)
C3	0.1643 (7)	0.1702 (7)	0.4901 (6)	3.7 (3)
C4	0.1969 (7)	0.1167 (6)	0.5861 (6)	3.3 (3)
C5	0.2867 (7)	0.0216 (6)	0.6060 (6)	3.6 (3)
C6	0.3100 (7)	−0.0386 (6)	0.7019 (6)	3.3 (3)
C7	0.3969 (7)	−0.1429 (6)	0.7254 (6)	3.5 (3)
C8	0.3945 (7)	−0.1721 (6)	0.8264 (7)	3.7 (3)
C9	0.3054 (7)	−0.0884 (6)	0.8662 (6)	3.4 (3)
C10	0.2753 (8)	−0.0892 (6)	0.9662 (6)	3.8 (3)
C11	0.1874 (7)	−0.0125 (6)	1.0076 (6)	3.2 (2)
C12	0.1562 (7)	−0.0175 (6)	1.1148 (6)	3.5 (3)
C13	0.0635 (7)	0.0710 (6)	1.1262 (6)	3.5 (3)
C14	0.0410 (7)	0.1318 (6)	1.0242 (6)	3.2 (3)
C15	−0.0432 (7)	0.2291 (7)	1.0021 (6)	3.9 (3)
C16	−0.0675 (7)	0.2882 (6)	0.9068 (6)	3.4 (3)
C17	−0.1621 (7)	0.3871 (6)	0.8879 (7)	3.8 (3)
C18	−0.1656 (7)	0.4144 (6)	0.7896 (7)	3.8 (3)
C19	−0.0705 (7)	0.3355 (6)	0.7453 (6)	3.4 (3)
C20	−0.0436 (7)	0.3344 (6)	0.6466 (6)	3.8 (3)
C25	−0.0061 (8)	0.3337 (7)	0.4289 (7)	4.6 (3)
C26	−0.133 (1)	0.2977 (9)	0.4689 (9)	7.2 (4)
C27	0.2237 (8)	0.1305 (7)	0.3977 (6)	4.5 (3)
C28	0.1722 (9)	0.0300 (9)	0.4256 (8)	6.6 (4)
C29	0.4666 (8)	−0.2055 (7)	0.6506 (7)	4.6 (3)
C30	0.384 (1)	−0.2728 (9)	0.6599 (9)	7.3 (5)
C31	0.4716 (9)	−0.2700 (7)	0.8904 (7)	5.0 (3)
C32	0.5698 (9)	−0.2375 (9)	0.9000 (8)	6.2 (4)
C33	0.2209 (9)	−0.1041 (7)	1.1933 (7)	4.9 (3)
C34	0.348 (1)	−0.078 (1)	1.163 (1)	8.2 (5)
C35	−0.0008 (8)	0.1068 (7)	1.2199 (7)	4.5 (3)
C36	0.052 (1)	0.2037 (9)	1.1981 (8)	7.0 (4)
C37	−0.2341 (8)	0.4466 (7)	0.9655 (7)	5.0 (3)
C38	−0.153 (1)	0.517 (1)	0.954 (1)	9.1 (6)
C39	−0.2556 (8)	0.5048 (7)	0.7361 (7)	4.7 (3)
C40	−0.371 (1)	0.4616 (9)	0.764 (1)	8.0 (5)
C50	0.2621 (9)	0.3643 (8)	0.6003 (8)	5.2 (4)
C51	0.4435 (8)	0.1512 (8)	0.6717 (8)	5.4 (4)
C52	0.283 (1)	0.270 (1)	0.8287 (9)	8.6 (5)
C53	0.450 (1)	0.3468 (9)	0.6417 (8)	6.1 (4)

equivalent isotropic temperature factors are given in Table 1;* bond lengths and angles are listed in Table 2; Fig. 1 is the *ORTEP* (Johnson, 1965) drawing of the molecule.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54812 (38 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA0240]

Table 2. *Bond distances* (Å) *and angles* (°)

Ge	Fe	2.370 (2)	O1	C50	1.15 (1)
Ge	N1	2.072 (9)	O2	C51	1.16 (1)
Ge	N2	2.091 (6)	O3	C52	1.16 (2)
Ge	N3	2.077 (6)	O4	C53	1.15 (2)
Ge	N4	2.088 (5)			
Fe	C50	1.77 (1)			
Fe	C51	1.77 (1)			
Fe	C52	1.76 (2)			
Fe	C53	1.76 (1)			
N1	C1	1.391 (9)	N2	C6	1.38 (1)
N1	C4	1.38 (1)	N2	C9	1.38 (1)
C1	C2	1.44 (1)	C6	C7	1.442 (9)
C2	C3	1.36 (1)	C7	C8	1.35 (1)
C3	C4	1.43 (1)	C8	C9	1.43 (1)
C4	C5	1.38 (1)	C9	C10	1.36 (1)
C5	C6	1.37 (1)	C10	C11	1.37 (1)
C2	C25	1.50 (1)	C7	C29	1.51 (1)
C3	C27	1.51 (1)	C8	C31	1.51 (1)
C25	C26	1.52 (1)	C29	C30	1.51 (2)
C27	C28	1.51 (2)	C31	C32	1.50 (2)
N3	C11	1.383 (9)	N4	C16	1.38 (1)
N3	C14	1.38 (1)	N4	C19	1.38 (1)
C11	C12	1.44 (1)	C16	C17	1.44 (1)
C12	C13	1.36 (1)	C17	C18	1.35 (1)
C13	C14	1.44 (1)	C18	C19	1.44 (1)
C14	C15	1.36 (1)	C19	C20	1.36 (1)
C15	C16	1.36 (1)	C20	C1	1.37 (1)
C12	C33	1.50 (1)	C17	C37	1.51 (2)
C13	C35	1.48 (1)	C18	C39	1.50 (1)
C33	C34	1.51 (2)	C37	C38	1.52 (2)
C35	C36	1.51 (2)	C39	C40	1.50 (2)

Fe	Ge	N1	109.4 (2)	C1	N1	C4	104.5 (7)
Fe	Ge	N2	108.1 (2)	C11	N3	C14	104.2 (7)
Fe	Ge	N3	109.5 (2)	N1	C1	C2	110.8 (7)
Fe	Ge	N4	109.6 (2)	C2	C1	C20	124.9 (8)
N1	Ge	N2	83.9 (3)	C1	C2	C25	125.0 (7)
N1	Ge	N3	141.0 (3)	C2	C3	C4	107.2 (8)
N1	Ge	N4	83.8 (3)	C4	C3	C27	124.6 (7)
N2	Ge	N3	83.8 (2)	N1	C4	C5	124.8 (8)
N2	Ge	N4	142.3 (3)	C4	C5	C6	125.5 (8)
N3	Ge	N4	83.7 (2)	N2	C6	C7	111.0 (8)
Ge	Fe	C50	88.3 (4)	C6	C7	C8	106.2 (8)
Ge	Fe	C51	89.1 (4)	C8	C7	C29	129.6 (7)
Ge	Fe	C52	88.7 (5)	C7	C8	C31	129.5 (8)
Ge	Fe	C53	178.9 (4)	N2	C9	C8	110.7 (9)
C50	Fe	C51	117.6 (6)	C8	C9	C10	125.1 (8)
C51	Fe	C52	119.0 (5)	N3	C11	C10	124.3 (9)
C50	Fe	C52	123.2 (6)	C10	C11	C12	124.6 (7)
Ge	N3	C11	125.4 (4)	C11	C12	C33	124.2 (7)
C18	C39	C40	112.5 (7)	C12	C33	C34	112.4 (7)
C17	C37	C38	111.9 (8)	N2	C9	C10	124.1 (6)
C6	N2	C9	104.5 (6)	C9	C10	C11	126.2 (8)
C16	N4	C19	104.7 (6)	N3	C11	C12	111.1 (7)
N1	C1	C20	124.4 (9)	C11	C12	C13	106.9 (7)
C1	C2	C3	106.5 (8)	C13	C12	C33	128.8 (9)
C3	C2	C25	129 (1)	C12	C13	C35	129.2 (8)
C2	C3	C27	128.1 (9)	N3	C14	C13	111.4 (6)
N1	C4	C3	111.0 (6)	C13	C14	C15	124.1 (8)
C3	C4	C5	124.2 (9)	N4	C16	C15	124.6 (7)
N2	C6	C5	123.8 (6)	C15	C16	C17	124.5 (9)
C6	C7	C29	124.1 (9)	C17	C18	C19	107.3 (7)
C7	C8	C9	107.6 (7)	C19	C18	C39	124 (1)
C9	C8	C31	122.8 (9)	N4	C19	C20	123.9 (6)
C1	C20	C19	125.9 (8)	C8	C31	C32	114.6 (8)
C3	C27	C28	113.4 (6)	C13	C35	C36	112.5 (6)

Related literature. For a review of metal—metal bonding in metalloporphyrin chemistry, see Guilard, Lecomte & Kadish (1987), Brothers & Collman (1986), and references therein; a similar crystal structure containing a hetero metal—metal double bond

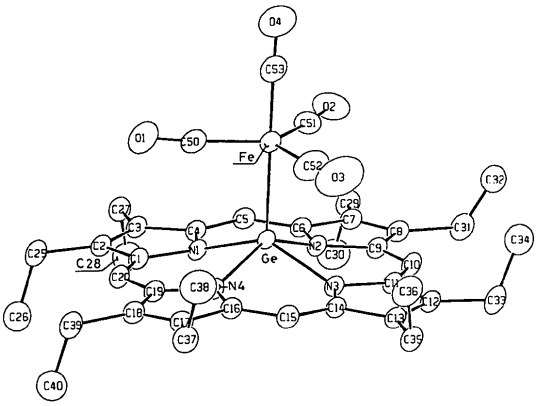

Fig. 1. *ORTEP* view of [(oep)GeFe(CO)$_4$].

in the metalloporphyrin series: [(oep)SnFe(CO)$_4$] (Barbe, Guilard, Lecomte & Gerardin, 1984), Sn—Fe = 2.491 (1) Å.

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Structure of (2*R*,4*R*,5*R*)-2-Chloro-3-isopropyl-4-methyl-5-phenyl-1,3,2-oxazaphospholidine 2-Oxide

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Abstract. $C_{12}H_{17}ClNO_2P$, $M_r = 273.70$, orthorhombic, $P2_12_12_1$, $a = 12.714$ (5), $b = 14.726$ (2), $c = 7.453$ (4) Å, $V = 1395$ (1) Å^3, $Z = 4$, $D_x = 1.303$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 3.75$ cm^{-1}, $F(000) = 576$, $T = 296$ K, $R = 0.039$, 770 unique observed reflections. This structure determination, coupled with the known configuration of the starting amino alcohol, confirms the absolute configuration at P of the title compound and related 2-chloro-1,3,2-oxazaphospholidin-2-ones similarly prepared. The five-membered oxazaphospholidine ring has the 'envelope' conformation with C(4) deviating by 0.504 Å from the least-squares plane formed by N(3), P(2), O(1) and C(5).

Experimental. The title compound was prepared from the reaction of (1*R*,2*R*)-(−)-2-(isopropylamino)-1-phenylpropanol (Hua, Chan-Yu-King, Ostrander & McKie, 1989) and phosphorus oxychloride with two equivalents of triethylamine in toluene (95% yield). A mixture of 93:7 of the title compound and its 2*S* isomer was formed. Pure title compound: 73% yield; m.p., from $CHCl_3$, 384∼386 K; $[\alpha]_D^{22°C} = -52.4°$ ($c = 0.8$ in CH_2Cl_2).

Data were collected from a colorless crystal fragment with dimensions 0.24 × 0.30 × 0.33 mm which was cut from a cluster of intergrown crystals and coated with epoxy to prevent deterioration. Diffractometer was Rigaku AFC5S with graphite-monochromated Mo $K\alpha$ radiation, ω–2θ scans, and a scan speed of 4° min^{-1} (in ω). Weak reflections [$I < 10.0\sigma(I)$] were rescanned (maximum of two rescans) and the counts accumulated to improve accuracy. Lattice parameters were obtained from a least-squares fit of 17 strong reflections in the 2θ range 20–22°. 1457 unique reflections were measured [h 0 to 15, k 0 to 17, l 0 to 8, $(\sin\theta/\lambda)_{max} = 0.60$ Å^{-1}], of which 770 were considered observed with $I \geq 3\sigma(I)$. Three standard reflections ($\bar{2}\bar{2}0$, $0\bar{2}0$, $0\bar{3}1$) changed by 0.8, 0.4 and −0.1%, respectively; no decay correction was applied. Data were corrected for Lorentz and polarization, not for absorption. Direct-method programs *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984) provided the loca-

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