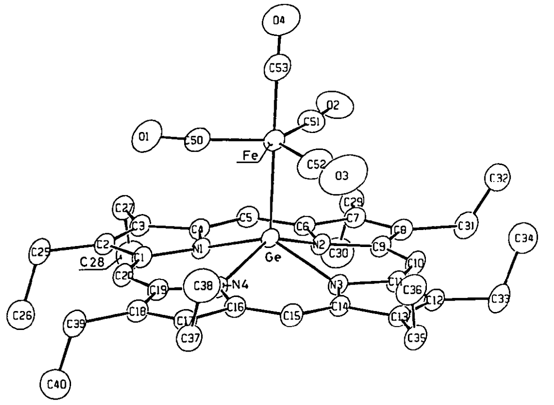

Fig. 1. *ORTEP* view of [(oep)GeFe(CO)$_4$].

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

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Acta Cryst. (1992). C**48**, 923–925

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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(*Received* 1 *July* 1991; *accepted* 10 *September* 1991)

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}, $\lambda(\text{Mo}\ K\alpha) = 0.71069$ 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; $[\alpha]_D^{22^\circ C} = -52.4^\circ$ ($c = 0.8$ 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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† Fellow of the Alfred P. Sloan Foundation, 1989–1993. Financial support from the National Institute of General Medical Sciences (Grant GM36336) is acknowledged.

0108-2701/92/050923-03$06.00

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$B_{eq} = (8\pi^2/3)$ trace **U**.

	x	y	z	B_{eq} (Å^2)
O(1)	0.2230 (3)	0.3519 (2)	−0.0118 (6)	4.3 (2)
P(2)	0.2073 (1)	0.2481 (1)	0.0291 (2)	4.07 (8)
N(3)	0.2936 (4)	0.2381 (3)	0.1885 (6)	3.3 (2)
C(4)	0.3687 (5)	0.3147 (4)	0.1782 (9)	3.4 (3)
C(5)	0.3002 (5)	0.3934 (4)	0.1110 (8)	3.3 (3)
C(6)	0.3260 (5)	0.1525 (4)	0.276 (1)	4.2 (3)
C(7)	0.4100 (6)	0.1015 (5)	0.168 (1)	6.1 (4)
C(8)	0.2311 (6)	0.0929 (4)	0.317 (1)	6.8 (5)
C(9)	0.4199 (5)	0.3384 (4)	0.356 (1)	5.0 (4)
C(10)	0.3565 (5)	0.4688 (4)	0.0135 (9)	3.3 (3)
C(11)	0.3412 (5)	0.5574 (5)	0.0710 (9)	3.9 (3)
C(12)	0.3903 (6)	0.6282 (4)	−0.019 (1)	5.1 (4)
C(13)	0.4561 (6)	0.6105 (5)	−0.159 (1)	5.5 (4)
C(14)	0.4717 (6)	0.5235 (6)	−0.213 (1)	6.3 (5)
C(15)	0.4226 (6)	0.4523 (4)	−0.129 (1)	5.1 (4)
O(16)	0.1023 (3)	0.2148 (3)	0.0529 (7)	5.9 (3)
Cl(17)	0.2648 (2)	0.1913 (1)	−0.2002 (3)	7.5 (1)

Table 2. *Selected bond distances* (Å) *and bond angles* (°) *with their e.s.d.'s*

O(1)	P(2)	1.571 (4)	C(5)	C(10)	1.508 (8)
O(1)	C(5)	1.475 (7)	C(6)	C(7)	1.532 (9)
P(2)	N(3)	1.624 (5)	C(6)	C(8)	1.524 (8)
P(2)	O(16)	1.433 (4)	C(10)	C(11)	1.388 (8)
P(2)	Cl(17)	2.039 (3)	C(10)	C(15)	1.374 (9)
N(3)	C(4)	1.480 (7)	C(11)	C(12)	1.386 (9)
N(3)	C(6)	1.477 (7)	C(12)	C(13)	1.36 (1)
C(4)	C(5)	1.534 (8)	C(13)	C(14)	1.36 (1)
C(4)	C(9)	1.517 (9)	C(14)	C(15)	1.373 (9)

P(2)	O(1)	C(5)	111.5 (3)	O(1)	C(5)	C(10)	108.8 (5)
O(1)	P(2)	N(3)	98.3 (2)	C(4)	C(5)	C(10)	116.4 (5)
O(1)	P(2)	O(16)	118.3 (3)	N(3)	C(6)	C(7)	112.4 (6)
O(1)	P(2)	Cl(17)	101.0 (2)	N(3)	C(6)	C(8)	111.1 (5)
N(3)	P(2)	O(16)	120.5 (3)	C(7)	C(6)	C(8)	112.1 (6)
N(3)	P(2)	Cl(17)	109.5 (2)	C(5)	C(10)	C(11)	118.5 (6)
O(16)	P(2)	Cl(17)	107.3 (2)	C(5)	C(10)	C(15)	122.1 (6)
P(2)	N(3)	C(4)	109.2 (4)	C(11)	C(10)	C(15)	119.3 (6)
P(2)	N(3)	C(6)	126.0 (4)	C(10)	C(11)	C(12)	119.7 (6)
C(4)	N(3)	C(6)	119.6 (5)	C(11)	C(12)	C(13)	120.1 (7)
N(3)	C(4)	C(5)	103.1 (4)	C(12)	C(13)	C(14)	120.0 (7)
N(3)	C(4)	C(9)	114.0 (5)	C(13)	C(14)	C(15)	121.0 (7)
C(5)	C(4)	C(9)	110.8 (5)	C(10)	C(15)	C(14)	119.9 (6)
O(1)	C(5)	C(4)	105.5 (4)				

tions of all but four non-H atoms. A least-squares/difference Fourier synthesis revealed the remaining non-H atomic sites. Full-matrix least-squares refinement was performed to minimize $\sum w(|F_o| - |F_c|)^2$ where $w = 4F_o^2/\sigma^2(F_o^2)$ and $\sigma^2(F_o^2) = [S^2(C + R^2B) + (pF_o^2)^2]/\mathrm{Lp}^2$ (S = scan rate, C = total integrated peak count, R = ratio of scan time to background-counting time, B = total background count, Lp = Lorentz–polarization factor and p = 0.03). After convergence methyl, tetrahedral and phenyl-H atoms were placed at assumed positions (C—H = 0.95 Å, $B = 1.2 \times B_{eq}$ of associated C atom) and fixed, methyl-group orientations were determined on the basis of H-atom positions obtained from a difference Fourier synthesis. Final stages of refinement performed with 155 variables including all non-H positional and anisotropic thermal parameters, one scale factor and a secondary-extinction coefficient [0.9 (2) × 10^{-6}]. Convergence yielded $R = 0.039$, $wR = 0.039$, $S = 1.30$ and $(\Delta/\sigma)_{max} = 0.0002$. Final difference synthesis produced $(\Delta\rho)_{max} = 0.16$ and $(\Delta\rho)_{min} = -0.19$ e Å^{-3}. Refinement of the alternative enantiomer produced slightly inferior results. Furthermore, the conformations at C(4) and C(5) are the same as that of the starting amino alcohol, thereby establishing the absolute configuration. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71, 148). All computer programs were from the *TEXSAN* (Molecular Structure Corporation, 1985) package. Table 1* lists the final atomic coordinates and equivalent isotropic temperature factors while Table 2 presents selected interatomic distances and angles. Fig. 1 shows the atom-numbering scheme and thermal motion; Fig. 2 illustrates the packing of the molecules within the unit cell.

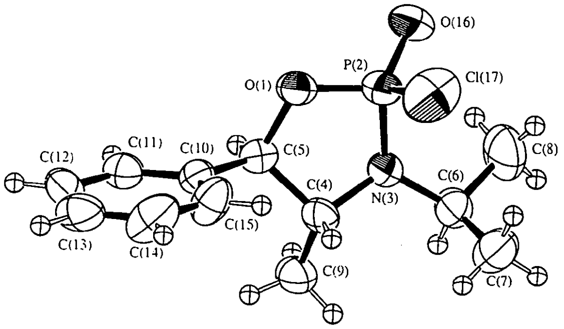

Fig. 1. Molecular configuration and atom-numbering scheme, thermal ellipsoids at the 50% probability level. H atoms are shown as unlabeled isotropic spheres with B's of 1.0 Å^2.

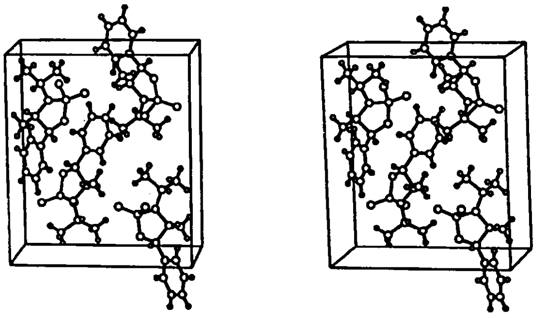

Fig. 2. Stereoscopic illustration of the molecular packing. **b** is vertical, **a** is horizontal and **c** is out of the plane of the paper.

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* Lists of structure factors, H-atom parameters, anisotropic thermal parameters, intermolecular distances, torsion angles, and selected least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54649 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0375]

Related literature. As part of studies of the asymmetric 1,4-addition reactions of 2-propene-1-phosphonamides (Hua, Chan-Yu-King, McKie & Myer, 1987), the title compound was prepared for the stereospecific displacement reactions with various organometallic reagents. So far, the configurations of 2-substituted 1,3,2-oxazaphospholidin-2-ones were assigned by NMR (nuclear magnetic resonance) spectroscopy (Cooper, Hall, Harrison & Inch, 1977), and only structures of the 3-methyl 2-sulfides (Bartczak & Galdecki, 1983; Bartczak, Galdecki & Rutkowska, 1983), 3-methyl-2-(methylthio) 2-oxide (Bartczak, Galdecki, Antipin & Struchkov, 1984), and 3-methyl-2-phenoxy 2-oxide derivatives (Setzer, Black & Hubbard, 1990) were reported. Since the configuration of the starting amino alcohol is known, the structure reported herein also establishes the absolute configuration of related compounds prepared similarly.

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Acta Cryst. (1992). **C48**, 925–929

Structures of a Pair of Diastereomeric Salts of Ephedrine and Dichloro-Substituted Cyclic Phosphoric Acid

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(*Received* 29 *August* 1991; *accepted* 4 *October* 1991)

Abstract. (1) (*R*)-(+)-4-(2,6-Dichlorophenyl)-2-hydroxy-5,5-dimethyl-1,3,2-dioxaphosphorinane 2-oxide (1*S*,2*R*)-(−)-α-[(1-methylamino)ethyl]benzyl alcohol, (−)-$C_{10}H_{16}NO^+$.(+)-$C_{11}H_{12}Cl_2O_4P^-$, M_r = 476.335, monoclinic, $P2_1$, $a = 10.063$ (1), $b = 8.206$ (1), $c = 14.856$ (1) Å, $\beta = 104.84$ (1)°, $V = 1185.8$ (2) Å^3, $Z = 2$, $D_x = 1.334$ Mg m^{-3}, Mo $K\alpha$ radiation (graphite monochromator), $\lambda = 0.71073$ Å, μ(Mo $K\alpha$) = 0.3689 mm^{-1}, $F(000) = 500$, $T = 130$ K, final $R = 0.053$, $wR = 0.056$ for 3504 observed unique reflections and 281 variables. (2) (*S*)-(−)-4-(2,6-Dichlorophenyl)-2-hydroxy-5,5-dimethyl-1,3,2-dioxaphosphorinane 2-oxide (1*R*,2*S*)-(−)-α-[(1-methylamino)ethyl]benzyl alcohol hydrate, (−)-$C_{10}H_{16}NO^+$.(−)-$C_{11}H_{12}Cl_2O_4P^-$.H_2O, M_r = 494.351, orthorhombic, $P2_12_12_1$, $a = 6.502$ (1), $b = 11.393$ (1), $c = 32.739$ (1) Å, $V = 2425.2$ (4) Å^3, $Z = 4$, $D_x = 1.354$ Mg m^{-3}, Mo $K\alpha$ radiation (graphite monochromator), $\lambda = 0.71073$ Å, μ(Mo $K\alpha$) = 0.3660 mm^{-1}, $F(000) = 1040$, $T = 130$ K, final $R = 0.055$, $wR = 0.059$ for 4225 observed unique reflections and 265 variables. For both (1) and (2), the phosphorinane ring is in the usual chair conformation and the ephedrine is in an extended form. The crystal packing of (1) can be regarded as chains parallel to the *b* axis consisting of phosphorinane

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