

configurations around the chiral centres C(14), C(13), C(12) and C(11) are *R,S,R* and *S* respectively, in accord with the *D-galacto* constitution. The new chiral centre C(3) is *R*.

The packing is governed by an intermolecular hydrogen bond O(2)—H...O(142) ($\frac{1}{2}+x$, $\frac{3}{2}-y$, $1-z$) = 2.780 (8) Å, O(2)—H = 0.98 (6) Å, H...O(142) = 1.86 (6) Å, O(2)—H...O(142) = 154 (5)°, which stabilizes the crystal structure. No other intermolecular contacts significantly shorter than the sum of the van der Waals radii are observed.

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Structure of 5,5-Dimethyl-2-ethylamino-1,3,2-dioxaphosphorinane 2-Selenide

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Abstract. C₇H₁₆NO₂PSe, *M_r* = 256.2, monoclinic, *I*2/*c*, *a* = 19.783 (4), *b* = 7.684 (1), *c* = 15.657 (2) Å, β = 104.41 (7)°, *Z* = 8, *V* = 2305 (1) Å³, *D_m* = 1.46, *D_x* = 1.48 Mg m⁻³, *Cu Kα*, λ = 1.54178 Å, μ = 5.52 mm⁻¹, *F*(000) = 1040, *T* = 293 K, *R* = 0.035 for 1463 observed reflections. The dioxaphosphorinane ring adopts a slightly flattened chair conformation with the P=Se bond lying in the axial and the P—N bond in the equatorial position. The P=Se and P—N bond lengths are 2.081 (1) and 1.599 (3) Å respectively.

Introduction. The structure of the title compound has been determined to elucidate the influence of substituents with different electronegativities and steric interactions on the conformation of the dioxaphosphorinane ring and on the P-atom coordination sphere.

In this and in former studies (Bartczak & Wolf, 1983; Bartczak, Gałdecki, Trzeźwińska & Wolf, 1983) we have sought to define for 2-NHR-5,5-dimethyl-

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1,3,2-dioxaphosphorinane 2-selenides the effect of variation in the nature of the substituent *R* on the conformation properties of the dioxaphosphorinane ring system and on the spatial orientation of the substituents attached to the P atom. It has been well demonstrated that in the dioxaphosphorinane ring system 2-arylamino substituents tend to occupy an axial position (Bartczak, Gałdecki, Trzeźwińska & Wolf, 1983, and references cited therein), regardless of *X* in the adjacent P=*X* (*X*=O,S,Se), while dialkylamino groups prefer an equatorial position (Grand & Robert, 1978). However, to the best of our knowledge there is no evidence based on X-ray crystallographic data about the spatial orientation of the 2-monoalkylamino group attached to the P atom incorporated in the 1,3,2-dioxaphosphorinane ring system. For this reason, the title compound (1) was synthesized and its structure determined. Structural features of this compound are discussed in comparison with those recently reported for 2-anilino-5,5-dimethyl-1,3,2-dioxaphosphorinane 2-selenide (2) (Bartczak, Gałdecki, Trzeźwińska & Wolf, 1983).

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Experimental. Preparation by the reaction of 2-chloro-5,5-dimethyl-1,3,2-dioxaphosphorinane with ethylamine and subsequent addition of elemental selenium (Lesiak, Leśnikowski, Stec & Zielińska, 1979); colourless crystals grown by slow evaporation from 1:1 mixture of chloroform and *n*-hexane; crystal used in analysis cut from a larger prism, dimensions 0.3 × 0.4 × 0.5 mm; Syntex P2₁ four-circle automated diffractometer, graphite-monochromatized Mo K α radiation; accurate cell dimensions derived by least-squares calculations from angular settings of 15 reflections measured at 21 < θ < 31°; $\theta/2\theta$ scan mode; 1550 unique reflections collected up to $2\theta = 115^\circ$; h : 0→32, k : 0→10, l : -19→19 indices given in space group C2/c; no significant intensity variation of two standard reflections; Lp and empirical absorption corrections applied; secondary-extinction correction, $G = 6.04(5) \times 10^{-3}$; 1459 reflections according to the criterion $F_o > 3\sigma(F_o)$ assumed to be observed and applied for further calculations; structure solved by direct methods; structure refined using F^2 s by standard full-matrix least-squares methods and difference electron density syntheses; last stages of refinement performed in space group I2/c to avoid severe correlations which occurred when refining in the space group C2/c owing to the large value of $\beta = 137.048(8)^\circ$; all H atoms located from ΔF maps, except H(12), H(22) and H(23) whose positions were calculated geometrically; final conventional $R = 0.035$, $wR = 0.039$ for observed reflections; max. $\Delta/\sigma = -0.09$ for z of C(2); largest peak in final ΔF map $0.50 \text{ e } \text{Å}^{-3}$, largest minimum $-0.40 \text{ e } \text{Å}^{-3}$ (both near Se); $w = 1/\sigma^2(F_o)$; scattering factors from *International Tables for X-ray Crystallography* (1974); all calculations performed using programs written by Sheldrick (1976).*

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42549 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

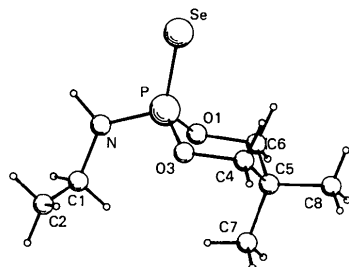


Fig. 1. View of the molecule with the atom numbering.

Discussion. The atom numbering is shown in Fig. 1. Final positional and equivalent isotropic thermal parameters are given in Table 1. Bond distances and angles are in Table 2. The dioxaphosphorinane ring adopts a slightly flattened chair conformation (Figs. 2 and 3). The P=Se bond points in the axial direction and the P–N bond adopts the equatorial position.

Table 1. Fractional atomic parameters ($\times 10^4$) and U_{eq} ($\text{Å}^2 \times 10^3$) for the nonhydrogen atoms, with e.s.d.'s in parentheses

$$U_{eq} = (U_{11} + U_{22}\sin^2\beta + U_{33} + 2U_{13}\cos\beta)/3(1-\cos^2\beta).$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Se	2661.9 (3)	5531.4 (6)	-1798.3 (3)	67.6 (4)
P	3265 (1)	3723 (1)	-949 (1)	42.0 (5)
O(1)	4040 (1)	4354 (4)	-527 (2)	51 (1)
O(3)	2996 (1)	3274 (3)	-107 (2)	48 (1)
N	3351 (2)	1888 (4)	-1392 (2)	58 (1)
C(4)	3085 (2)	4595 (6)	577 (3)	53 (2)
C(5)	3842 (2)	5084 (6)	923 (3)	54 (2)
C(6)	4116 (2)	5696 (6)	156 (3)	59 (2)
C(7)	4262 (3)	3568 (8)	1390 (3)	90 (2)
C(8)	3881 (3)	6612 (7)	1557 (4)	84 (2)
C(1)	3736 (3)	390 (6)	-952 (4)	69 (2)
C(2)	4350 (3)	-41 (9)	-1313 (5)	128 (4)

Table 2. Bond lengths (Å) and angles (°)

P–Se	2.081 (1)	O(1)–P	1.587 (3)
O(3)–P	1.577 (3)	N–P	1.599 (3)
C(6)–O(1)	1.465 (5)	C(4)–O(3)	1.454 (5)
C(1)–N	1.456 (5)	C(5)–C(4)	1.509 (6)
C(6)–C(5)	1.511 (6)	C(7)–C(5)	1.509 (7)
C(8)–C(5)	1.527 (6)	C(2)–C(1)	1.500 (6)
O(1)–P–Se	113.7 (1)	O(3)–P–Se	114.9 (1)
O(3)–P–O(1)	102.2 (1)	N–P–Se	114.7 (1)
N–P–O(1)	104.7 (2)	N–P–O(3)	105.3 (2)
C(6)–O(1)–P	115.6 (2)	C(4)–O(3)–P	117.3 (2)
C(1)–N–P	126.3 (3)	C(5)–C(4)–O(3)	111.5 (3)
C(6)–C(5)–C(4)	108.2 (3)	C(7)–C(5)–C(4)	111.0 (4)
C(7)–C(5)–C(6)	111.2 (4)	C(8)–C(5)–C(4)	108.1 (4)
C(8)–C(5)–C(6)	107.9 (4)	C(8)–C(5)–C(7)	110.3 (4)
C(5)–C(6)–O(1)	111.7 (3)	C(2)–C(1)–N	111.9 (4)

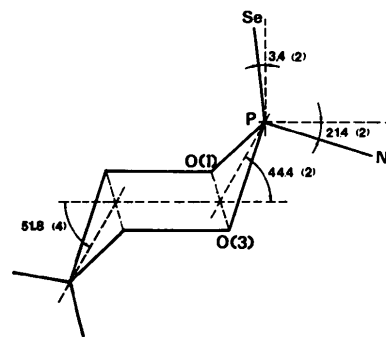


Fig. 2. Flattening of the 1,3,2-dioxaphosphorinane ring illustrated by the dihedral angles (°).

