one type of interaction. The same monovalent I atom rarely participates in both short I···I and short I···O or I···N contacts. In the CSD there are 1559 compounds containing both an I atom and either an O or an N atom. In many of these structures there are short intermolecular distances involving I atoms, but only 16 structures contain monovalent I atoms 'close' to *both* an I and an O or N atom. In all but two of these 16 structures the O or N atom is multiply bonded to a C atom.

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Structure of 4-Aminolactivinic Acid

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Abstract. 2-(4-Ammonio-3-oxo-1,2-oxazolidin-2-yl)-5-oxotetrahydrofuran-2-carboxylate, $C_8H_{10}N_2O_6$, $M_r = 230{\cdot}2$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}833$ (3), $b = 21{\cdot}577$ (5), $c = 5{\cdot}565$ (2) Å, $V = 940{\cdot}6$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}63$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}5$ cm^{-1}, $F(000) = 480$, room temperature. Final $R = 0{\cdot}065$ for 530 observed reflections. The title compound is a derivative of the new antibiotic lactivicin. The molecule consists of two five-membered rings (3-isoxazolidone and γ-lactone) directly connected by a C—N bond. The isoxazolidone ring exhibits a half-chair conformation and the γ-lactone ring is in an envelope form.

Introduction. Lactivicin (LTV) is a novel antibiotic isolated from culture filtrates of *Empedobacter lactamgenus* sp. nov. YK-258 and *Lysobacter albus* sp. nov. YK-422 (Nozaki, Katayama, Ono, Tsubotani, Harada, Okazaki & Nakao, 1987). An X-ray analysis as part of the structural study of this compound (Harada, Tsubotani, Hida, Ono & Okazaki, 1986) was undertaken for the determination in particular of the relative stereochemistry and the absolute configuration. Among various derivatives and salts of LTV examined in an attempt to obtain suitable crystals, 4-aminolactivinic acid gave tolerable crystals for X-ray analysis.

Table 1. *Positional parameters* (×10^4) *and* U_{eq}(Å^2×10^3) *for non-hydrogen atoms with e.s.d.'s in parentheses*

$U_{eq} = (U_{11} + U_{22} + U_{33})/3$.

	x	y	z	U_{eq}
O(1)	8080 (11)	1383 (4)	1482 (18)	42 (5)
N(2)	6920 (13)	1037 (5)	3064 (18)	31 (6)
C(3)	7842 (16)	714 (5)	4682 (25)	30 (8)
O(3)	7278 (10)	435 (4)	6390 (16)	35 (5)
C(4)	9701 (15)	735 (5)	3932 (21)	21 (6)
C(5)	9567 (18)	988 (5)	1332 (24)	37 (7)
C(6)	5378 (16)	1350 (5)	3635 (22)	27 (6)
O(7)	5753 (12)	1841 (3)	5405 (14)	33 (5)
C(8)	5616 (19)	2395 (6)	4585 (25)	40 (9)
O(8)	5908 (15)	2853 (5)	5817 (20)	71 (8)
C(9)	5102 (20)	2406 (7)	1919 (24)	46 (9)
C(10)	4574 (18)	1729 (6)	1474 (25)	42 (8)
C(11)	4084 (17)	897 (6)	4605 (23)	35 (8)
O(11*A*)	3829 (11)	433 (4)	3365 (17)	39 (5)
O(11*B*)	3302 (11)	1026 (4)	6519 (16)	43 (5)
N(12)	10473 (13)	114 (5)	4006 (17)	31 (6)

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Experimental. Recrystallization at 278 K from aqueous solution, colorless plates, crystal 0·2 × 0·2 × 0·01 mm; Rigaku AFC-5 diffractometer, graphite-monochromated Mo $K\alpha$ radiation; cell parameters from least-squares refinement of 59 reflections in the range $21 < 2\theta < 31°$; 2θ–ω scan, 769 reflections with $2\theta \leq 45°$, three standard reflections (011, 230, 340) monitored for each 60 reflections collected, standard reflections within 1·8% variation throughout the data collection, index range $h = 0$ to 8, $k = 0$ to 23, $l = 0$ to 5, 751 unique reflections, of which 530 with $F \geq 3\sigma(F)$ considered observed and used for all calculations; Lorentz, polarization but no absorption corrections applied; structure solved by direct methods, H-atom positions from difference Fourier maps, unweighted full-matrix least-squares refinement of atomic coordinates and anisotropic temperature factors for all non-H atoms and isotropic temperature factors for H atoms, minimizing $\sum(F_o - F_c)^2$; final $R = 0{\cdot}065$, $wR = 0{\cdot}070$, $S = 1{\cdot}60$. Max. $\Delta/\sigma = 0{\cdot}024$; max. difference-map peak 0·40 e Å^{-3}. The main cause of the relatively high R index is the small size of the crystal. Atomic scattering factors from *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). Computer programs *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and *XRAY76*. The final atomic positional parameters and U_{eq} values are given in Table 1,* bond distances and angles in Fig. 1 and the atomic numbering scheme in Fig. 2.

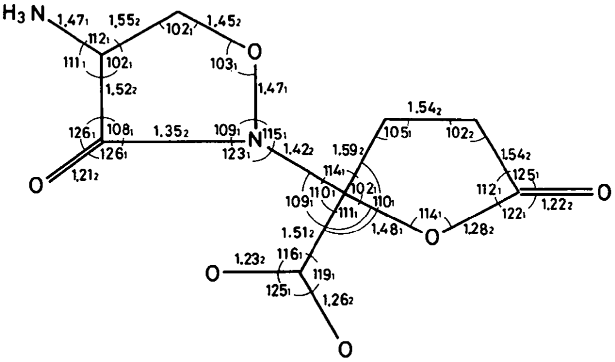


Fig. 1. Bond distances (Å) and angles (°). Small digits show e.s.d.'s.

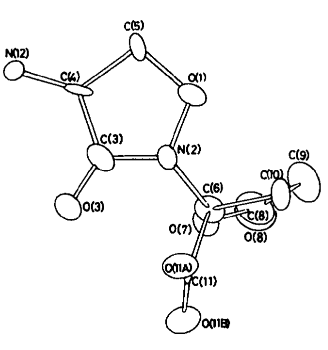
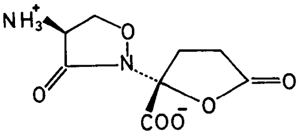


Fig. 2. Perspective view of the molecule. The numbering scheme is also shown.

Discussion. The structure was confirmed as (I), in which 3-isoxazolidone and γ-lactone rings were directly linked through the N(2)—C(6) bond. Though the isomerization at the lactone moiety easily occurs in aqueous solution, the configuration at C(4) of 3-isoxazolidone is kept as *S* (Harada, Tsubotani, Hida, Ono & Okazaki, 1986). Therefore, as for the present crystal, the absolute configuration at C(6) is deduced to be *R*. Figures are drawn to show the correct configuration.

The 3-isoxazolidone ring is in a half-chair form and O(1), C(5) deviate from the plane formed by N(2), C(3) and C(4). Atoms O(1), C(6) are situated at one side and C(5), N(12) at the other side of the plane. The γ-lactone ring is in an envelope conformation in which C(10) is the flap and is displaced from the plane formed by the other four atoms to the same side as C(11) and opposite to N(2). These two five-membered rings are almost perpendicular to each other, and N(2)—C(3) and C(6)—C(10), N(2)—O(1) and C(6)—C(11) are *s-trans* with respect to the N(2)—C(6) bond.



(I)

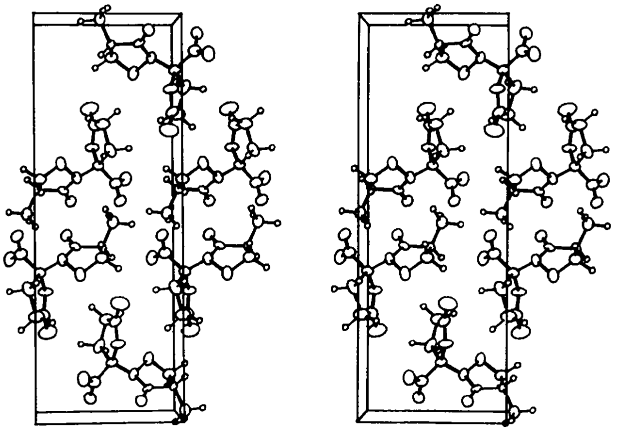


Fig. 3. Packing diagram.

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43901 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The amino nitrogen N(12) presents a quaternary character: this quaternization is produced by proton transfer from the carboxyl group to N(12); thus, a 'zwitterion' structure is present, with the carboxyl group deprotonated and the amino N protonated. The distances of 2·75 (1), 2·74 (1) and 2·86 (1) Å between N(12) and O(11*A*) of the molecule at $(1{\cdot}5-x,\ -y,\ 0{\cdot}5+z)$, O(11*A*) at $(1+x,\ y,\ z)$ and O(3) at $(1{\cdot}5-x,\ -y,\ -0{\cdot}5+z)$ indicate the presence of three independent intermolecular hydrogen bonds. Fig. 3 shows the molecular packing.

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The Stereochemistry of the Phosphorus–Selenium Bond. VI.* Structure of 5,5-Dimethyl-2-methylseleno-1,3,2-dioxaphosphorinane 2-Selenide

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Abstract. $C_6H_{13}O_2PSe_2$, $M_r = 306{\cdot}06$, monoclinic, $P2_1/n$, $a = 6{\cdot}9528$ (5), $b = 12{\cdot}234$ (1), $c = 12{\cdot}235$ (2) Å, $\beta = 93{\cdot}77$ (1)°, $U = 1038{\cdot}4$ (2) Å^3, $Z = 4$, $D_m = 1{\cdot}94$ (by flotation in aqueous KI solution), $D_x = 1{\cdot}958$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 7{\cdot}16$ mm^{-1}, $F(000) = 592$, $T = 133$ K, $R = 0{\cdot}021$ for 1649 observed reflections $[I > 3\sigma(I)]$. The dioxaphosphorinane ring exists in the solid state in a flattened chair conformation with P=Se [2·071 (1) Å] positioned equatorially and P—Se [2·227 (1) Å] in the axial position. The geometrical features compare well with those of other 1,3,2-dioxaphosphorinane 2-selenides.

Introduction. Reactions of Na derivatives of *O*,*O*-diallylphosphoroanilidates and their thio and seleno analogues with CS_2 give the corresponding phosphoroselenoates and phosphorodiselenoates (Lesiak, Leśnikowski, Stec & Zielińska, 1979). In the case of chiral phosphoroanilidates the PN → PSe conversion proceeds with high stereospecificity and the configuration at the P atom is retained. It seemed desirable to investigate the title compound, synthesized in the course of the above research, by means of X-ray crystallography to reveal the conformation of the 1,3,2-dioxaphosphorinane ring in the presence of two Se atoms bonded to the P atom. Here we report the results of this study, which forms a continuation of investigations of 1,3,2-dioxaphosphorinane 2-selenides performed at the Institute of General Chemistry, Technical University of Łódź, since 1975 (Bartczak, Christensen, Kinas & Stec, 1975*a*,*b*, 1976; Bartczak & Wolf, 1983; Bartczak, Gałdecki, Trezeźwińska & Wolf, 1983; Bartczak, Gałdecki, Wolf, Lesiak & Stec, 1986).

Experimental. Colourless crystal shaped to sphere *ca* 0·03 mm in diameter, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; 2θ–ω scan mode; low-temperature system based on a design by Huffman (1974); lattice constants refined by least-squares fit of 25 reflections in the θ range 16·5–24·1°; empirical absorption correction (North, Phillips & Mathews, 1968) based on ψ scan, transmission factors 0·999 max. and 0·919 min.; h −8→8,

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