

S(1B)	-0.4697 (2)	0.5732 (1)	0.2594 (1)	0.061 (1)
O(1B)	-0.5195 (4)	0.5936 (3)	0.0844 (2)	0.084 (1)
O(2B)	-0.3511 (4)	0.6680 (3)	0.3578 (2)	0.074 (1)
O(3B)	-0.2503 (3)	0.8250 (3)	0.3615 (1)	0.054 (1)
N(2B)	-0.2136 (5)	0.9206 (4)	0.2270 (2)	0.085 (2)
N(3B)	-0.4266 (4)	0.6876 (3)	0.1675 (2)	0.044 (1)
C(2B)	-0.4009 (5)	0.6849 (3)	0.2296 (2)	0.042 (1)
C(4B)	-0.4942 (6)	0.6006 (4)	0.1384 (3)	0.058 (2)
C(5B)	-0.5276 (6)	0.5236 (4)	0.1847 (2)	0.066 (2)
C(6B)	-0.3296 (5)	0.7583 (4)	0.2660 (2)	0.042 (1)
C(7B)	-0.3130 (5)	0.7435 (4)	0.3322 (2)	0.051 (1)
C(8B)	-0.2668 (5)	0.8483 (4)	0.2426 (2)	0.052 (1)
C(9B)	-0.3985 (5)	0.7750 (4)	0.1298 (2)	0.043 (1)
C(10B)	-0.2893 (6)	0.7730 (5)	0.0969 (3)	0.067 (2)
C(11B)	-0.2682 (7)	0.8579 (6)	0.0595 (3)	0.086 (2)
C(12B)	-0.3571 (8)	0.9403 (5)	0.0562 (3)	0.083 (2)
C(13B)	-0.4649 (7)	0.9405 (4)	0.0887 (3)	0.068 (2)
C(14B)	-0.4878 (5)	0.8583 (4)	0.1261 (2)	0.052 (1)
C(15B)	-0.2234 (6)	0.8154 (4)	0.4277 (2)	0.070 (2)
C(16B)	-0.1820 (7)	0.9185 (5)	0.4530 (3)	0.088 (2)

Table 2. Selected geometric parameters (Å, °)

S(1A)—C(2A)	1.749 (5)	O(2B)—C(7B)	1.203 (6)
S(1A)—C(5A)	1.788 (5)	O(3B)—C(7B)	1.343 (5)
O(1A)—C(4A)	1.191 (6)	O(3B)—C(15B)	1.461 (6)
O(2A)—C(7A)	1.212 (6)	N(2B)—C(8B)	1.139 (6)
O(3A)—C(7A)	1.324 (6)	N(3B)—C(2B)	1.366 (6)
O(3A)—C(15A)	1.445 (6)	N(3B)—C(4B)	1.416 (6)
N(2A)—C(8A)	1.133 (6)	N(3B)—C(9B)	1.443 (6)
N(3A)—C(2A)	1.376 (5)	C(2B)—C(6B)	1.376 (6)
N(3A)—C(4A)	1.400 (6)	C(4B)—C(5B)	1.487 (7)
N(3A)—C(9A)	1.449 (5)	C(6B)—C(8B)	1.434 (7)
C(2A)—C(6A)	1.371 (6)	C(9B)—C(14B)	1.380 (6)
C(4A)—C(5A)	1.499 (7)	C(10B)—C(11B)	1.397 (8)
C(6B)—C(7B)	1.467 (6)	C(11B)—C(12B)	1.368 (9)
C(9B)—C(10B)	1.363 (7)	C(15B)—C(16B)	1.476 (7)
C(6A)—C(8A)	1.435 (7)	C(15A)—C(16A)	1.398 (9)
C(6A)—C(7A)	1.463 (7)	S(1B)—C(2B)	1.748 (5)
C(9A)—C(10A)	1.364 (6)	S(1B)—C(5B)	1.800 (5)
C(9A)—C(14A)	1.373 (6)	O(1B)—C(4B)	1.195 (6)
C(10A)—C(11A)	1.381 (7)	C(13B)—C(14B)	1.375 (7)
C(12B)—C(13B)	1.346 (8)	C(12A)—C(13A)	1.378 (7)
C(11A)—C(12A)	1.362 (8)	C(13A)—C(14A)	1.372 (6)
C(2A)—S(1A)—C(5A)	92.1 (2)	O(2A)—C(7A)—O(3A)	124.2 (5)
C(7A)—O(3A)—C(15A)	117.6 (5)	O(2A)—C(7A)—C(6A)	124.8 (5)
C(2A)—N(3A)—C(4A)	117.1 (4)	O(3A)—C(7A)—C(6A)	111.0 (5)
C(2A)—N(3A)—C(9A)	124.7 (4)	N(2A)—C(8A)—C(6A)	176.0 (5)
C(4A)—N(3A)—C(9A)	118.2 (4)	C(10A)—C(9A)—C(14A)	121.8 (5)
C(6A)—C(2A)—N(3A)	125.7 (4)	C(10A)—C(9A)—N(3A)	118.6 (4)
C(6A)—C(2A)—S(1A)	123.1 (4)	C(14A)—C(9A)—N(3A)	119.6 (4)
N(3A)—C(2A)—S(1A)	111.2 (4)	C(9A)—C(10A)—C(11A)	118.6 (5)
O(1A)—C(4A)—N(3A)	123.5 (5)	C(14A)—C(13A)—C(12A)	120.4 (5)
O(1A)—C(4A)—C(5A)	126.4 (5)	O(2B)—C(7B)—C(6B)	124.7 (5)
N(3A)—C(4A)—C(5A)	110.0 (5)	C(10B)—C(9B)—C(14B)	121.0 (5)
C(4A)—C(5A)—S(1A)	108.8 (4)	C(9B)—C(10B)—C(11B)	118.7 (6)
C(2A)—C(6A)—C(8A)	124.1 (4)	C(12B)—C(11B)—C(10B)	119.9 (6)
C(2B)—S(1B)—C(5B)	92.2 (2)	C(2B)—N(3B)—C(4B)	117.3 (4)
C(9A)—C(14A)—C(13A)	118.8 (5)	C(2B)—N(3B)—C(9B)	125.0 (4)
C(12A)—C(11A)—C(10A)	120.7 (5)	C(4B)—N(3B)—C(9B)	117.5 (4)
O(3B)—C(7B)—C(6B)	111.8 (4)	N(3B)—C(2B)—C(6B)	126.3 (4)
C(16A)—C(15A)—O(3A)	111.0 (6)	N(3B)—C(2B)—S(1B)	111.4 (3)
C(2B)—C(6B)—C(7B)	119.0 (4)	C(6B)—C(2B)—S(1B)	122.3 (4)
C(8B)—C(6B)—C(7B)	117.5 (4)	O(1B)—C(4B)—N(3B)	123.1 (5)
N(2B)—C(8B)—C(6B)	176.4 (5)	O(1B)—C(4B)—C(5B)	127.0 (5)
C(10B)—C(9B)—N(3B)	120.6 (5)	N(3B)—C(4B)—C(5B)	109.9 (5)
C(14B)—C(9B)—N(3B)	118.4 (4)	C(4B)—C(5B)—S(1B)	109.0 (4)
C(11A)—C(12A)—C(13A)	119.8 (5)	O(2B)—C(7B)—O(3B)	123.5 (5)
C(2B)—C(6B)—C(8B)	123.5 (4)	C(13B)—C(12B)—C(11B)	120.6 (6)
C(7B)—O(3B)—C(15B)	115.9 (4)	C(12B)—C(13B)—C(14B)	120.8 (6)
C(2A)—C(6A)—C(7A)	119.5 (5)	C(13B)—C(14B)—C(9B)	119.0 (5)
C(8A)—C(6A)—C(7A)	116.3 (5)	O(3B)—C(15B)—C(16B)	108.2 (5)

Refinement was carried out on F^2 for all reflections except for two with very negative F^2 or those flagged for potential systematic errors.

Data collection: XSCANS (Siemens, 1992). Cell refinement: SHELXTL-Plus (Sheldrick, 1991). Data reduction: SHELXTL-Plus. Program(s) used to solve structure: SHELXTL-Plus. Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL-Plus. Software used to prepare material for publication: SHELXL93 and SHELXTL-Plus.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SZ1026). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris(*n*-propyl)phosphine and Tris(iso-propyl)phosphine

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Abstract

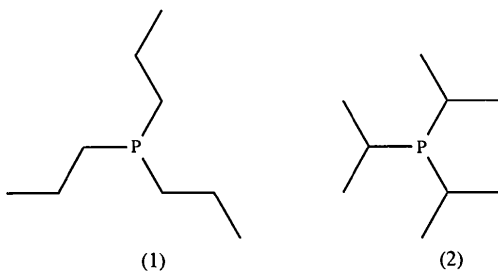
Single crystals of the low-melting title compound, tris(*n*-propyl)phosphine, C₉H₂₁P, (1), and its isomer, tris(isopropyl)phosphine, (2), were grown *in situ* in

capillaries. The crystal and molecular structures of both compounds were determined at 102 K. Compound (1) has an approximate mirror plane and C—P—C angles between 99.8(1) and 102.3(1)°, whereas compound (2) displays no molecular symmetry and has C—P—C angles ranging from 102.0(1) to 104.7(1)°.

Comment

Recently, we reported the molecular structures of trimethylphosphine, triethylphosphine (Bruckmann & Krüger, 1995a), tris(*n*-butyl)phosphine and tris(*tert*-butyl)phosphine (Bruckmann & Krüger, 1995b). In a continuation of our systematic structural investigations of phosphine ligands, we have succeeded in crystallizing the propyl-substituted phosphines.

Tris(*n*-propyl)phosphine, (1), shows a molecular conformation (Fig. 1) analogous to that of tris(*n*-butyl)phosphine. Plane *P*1 (P, C1, C2, C3) is an approximate mirror oriented nearly perpendicular to plane *P*2 (P, C4, C5, C6) and plane *P*3 (P, C7, C8, C9) [dihedral angles between *P*1 and *P*2, and *P*1 and *P*3 are 91.95(10) and 89.25(8)° respectively]. *P*2 and *P*3 are almost parallel to one other forming a dihedral angle of 178.79(8)°. Both C—P—C angles involving C1 are 102.3(1)°, significantly larger than C4—P—C7 [99.8(1)°]. In addition, the P—C1—C2 angle is opened to 119.0(1)°, about 6° more than the corresponding angles in the parallel-oriented propyl chains. These differences may be explained by repulsive H-atom interactions (H2A/H7B and H2B/H4B, respectively). The opening of the relevant angles increases these distances to values just below the sum of the hydrogen van der Waals radii. The P—C distances lie between 1.846(2) and 1.853(2) Å.



Molecular modelling investigations show the molecular conformation of (1) to exist in the crystalline state as a local energetic minimum not belonging to the global minimum of the free molecule. Obviously, the energetic unfavourable conformation is stabilized by intermolecular forces resulting from a unique arrangement of neighbouring molecules in the crystal. It is noteworthy that the two coplanar propyl groups of one molecule are arranged parallel to the corresponding propyl groups of a neighbouring molecule, so achieving an arrangement similar to those known from the crystal structures of

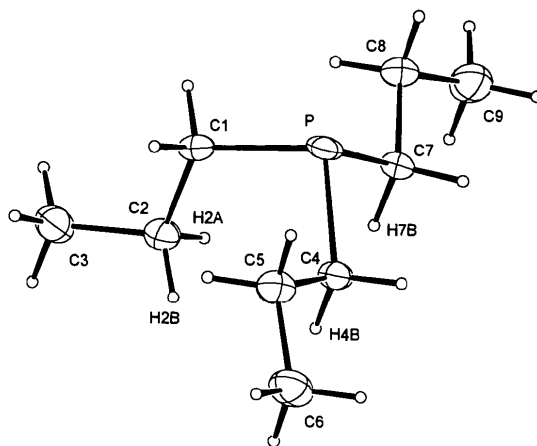


Fig. 1. An ORTEP (Johnson, 1976) view of tris(*n*-propyl)phosphine (1) with displacement ellipsoids drawn at the 50% level and H atoms as spheres of arbitrary size.

long-chained hydrocarbons. In the crystalline state the cone angle θ (Tolman, 1970) of (1) is determined to be 144(2)°. This value is in contrast to earlier listings [132(4)°; Tolman, 1977]. The difference might be explained by different folding of the flexible C₃H₉ substituents on P while maintaining threefold symmetry.

The molecular structure of tris(isopropyl)phosphine, (2), is shown in Fig. 2. The C—P—C angles are between 102.0(1) and 104.7(1)°, thus reflecting the steric interactions of the bulky isopropyl substituents. The C—P distances [1.856(3) to 1.872(3) Å] are influenced by these effects as are the angles P—C4—C5 and P—C7—C8 [117.5(2) and 117.4(2)°, respectively]. All other angles and distances in the molecule are as expected. The cone angle θ in this case is 168(2)° and is in reasonable agreement with the published value [160(10)°, Tolman, 1970]. The vectors C2—H2B, C3—H3B and C9—H9B are parallel, and parallel to a vector from the P atom to the idealized location of its lone pair.

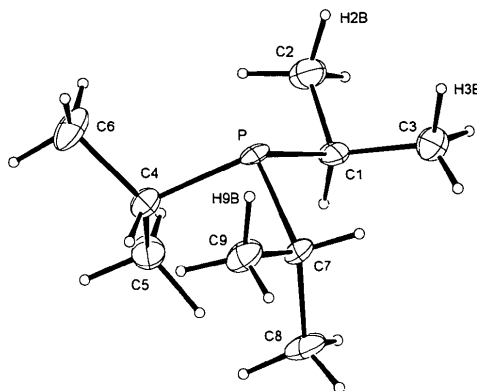


Fig. 2. An ORTEP drawing of tri(isopropyl)phosphine with 50% probability displacement ellipsoids and H atoms drawn as spheres of arbitrary size.

Experimental

Crystal (1) was grown by zone-melting techniques in an argon-filled glass capillary with a CO₂ laser as heat source (Boese, 1994). T_{melt} was 189 and T_{growth} 187 K.

Crystal (2) was grown by zone-melting techniques in an argon-filled silica-glass capillary with a CO₂ laser as heat source (Boese, 1994). T_{melt} was 206 and T_{growth} 184 K, pentane was used as solvent. An isotropic deterioration of the crystal quality occurred during data collection.

Compound (1)*Crystal data*

C ₉ H ₂₁ P	$Z = 2$
$M_r = 160.24$	$D_x = 1.00 \text{ Mg m}^{-3}$
Triclinic	D_m not measured
$P\bar{1}$	Mo $K\alpha$ radiation
$a = 5.579 (2) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 8.014 (2) \text{ \AA}$	Cell parameters from 25 reflections
$c = 12.014 (2) \text{ \AA}$	$\theta = 14.0\text{--}26.4^\circ$
$\alpha = 95.72 (1)^\circ$	$\mu = 0.194 \text{ mm}^{-1}$
$\beta = 95.72 (1)^\circ$	$T = 102 \text{ K}$
$\gamma = 92.54 (2)^\circ$	Cylindrical
$V = 531.0 (2) \text{ \AA}^3$	$0.5 \times 0.2 \text{ mm (radius)}$
	Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.016$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 27.48^\circ$
Absorption correction: none	$h = -7 \rightarrow 7$
2557 measured reflections	$k = -10 \rightarrow 10$
2439 independent reflections	$l = 0 \rightarrow 15$
2242 observed reflections	3 standard reflections
$[I > 2\sigma(I)]$	frequency: 30 min
	intensity decay: 10.4%

Refinement

Refinement on F	$(\Delta/\sigma)_{\text{max}} = 0.02$
$R = 0.051$	$\Delta\rho_{\text{max}} = 0.97 \text{ e \AA}^{-3}$
$wR = 0.068$	$\Delta\rho_{\text{min}} = -0.65 \text{ e \AA}^{-3}$
$S = 1.73$	Extinction correction: none
2242 reflections	Atomic scattering factors
175 parameters	from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
All H-atom parameters refined	
$w = 1/\sigma^2(F)$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (1)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
P	0.4375 (1)	0.2843 (1)	0.8379 (1)	0.021 (1)
C1	0.5105 (3)	0.1008 (2)	0.7434 (1)	0.022 (1)
C2	0.7456 (3)	0.1077 (2)	0.6885 (1)	0.025 (1)
C3	0.7812 (3)	-0.0545 (2)	0.6163 (1)	0.030 (1)
C4	0.7163 (3)	0.3241 (2)	0.9351 (1)	0.022 (1)
C5	0.7498 (3)	0.1903 (2)	1.0162 (1)	0.027 (1)
C6	0.9681 (3)	0.2296 (3)	1.1023 (2)	0.033 (1)
C7	0.4711 (3)	0.4588 (2)	0.7502 (1)	0.023 (1)
C8	0.2677 (3)	0.4576 (2)	0.6551 (1)	0.026 (1)
C9	0.2799 (4)	0.6140 (2)	0.5936 (2)	0.034 (1)

Table 2. Selected geometric parameters (\AA , $^\circ$) for (1)

P—C1	1.853 (2)	P—C4	1.846 (2)
P—C7	1.847 (2)	C1—C2	1.527 (2)
C2—C3	1.522 (2)	C4—C5	1.525 (2)
C5—C6	1.518 (3)	C7—C8	1.528 (2)
C8—C9	1.520 (2)		
C7—P—C4	99.8 (1)	C7—P—C1	102.3 (1)
C4—P—C1	102.3 (1)	C2—C1—P	119.0 (1)
C3—C2—C1	112.3 (1)	C5—C4—P	112.6 (1)
C6—C5—C4	112.8 (1)	C8—C7—P	113.1 (1)
C9—C8—C7	112.4 (1)		

Compound (2)*Crystal data*

C ₉ H ₂₁ P	D_m not measured
$M_r = 160.24$	Mo $K\alpha$ radiation
Monoclinic	$\lambda = 0.71073 \text{ \AA}$
$P2_1/n$	Cell parameters from 25 reflections
$a = 8.622 (3) \text{ \AA}$	$\theta = 8.3\text{--}16.2^\circ$
$b = 14.368 (2) \text{ \AA}$	$\mu = 0.194 \text{ mm}^{-1}$
$c = 8.928 (2) \text{ \AA}$	$T = 102 \text{ K}$
$\beta = 106.19 (2)^\circ$	Cylindrical
$V = 1062.2 (5) \text{ \AA}^3$	$0.8 \times 0.3 \text{ mm (radius)}$
$Z = 4$	Colourless
$D_x = 1.00 \text{ Mg m}^{-3}$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.008$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 27.54^\circ$
Absorption correction: none	$h = -11 \rightarrow 11$
2702 measured reflections	$k = 0 \rightarrow 18$
2440 independent reflections	$l = 0 \rightarrow 10$
2000 observed reflections	3 standard reflections
$[I > 2\sigma(I)]$	frequency: 30 min
	intensity decay: 25.5%

Refinement

Refinement on F	$(\Delta/\sigma)_{\text{max}} = 0.03$
$R = 0.078$	$\Delta\rho_{\text{max}} = 1.24 \text{ e \AA}^{-3}$
$wR = 0.101$	$\Delta\rho_{\text{min}} = -1.21 \text{ e \AA}^{-3}$
$S = 1.75$	Extinction correction: none
2000 reflections	Atomic scattering factors
175 parameters	from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
All H-atom parameters refined	
$w = 1/\sigma^2(F)$	

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
P	0.0324 (1)	0.2235 (1)	0.8070 (1)	0.018 (1)
C1	-0.0922 (3)	0.3251 (2)	0.7159 (3)	0.020 (1)
C2	-0.1435 (4)	0.3782 (2)	0.8426 (4)	0.027 (2)
C3	0.0035 (4)	0.3889 (2)	0.6387 (4)	0.028 (2)
C4	-0.1166 (3)	0.1364 (2)	0.8386 (4)	0.022 (2)
C5	-0.2950 (4)	0.1525 (2)	0.7540 (4)	0.029 (2)
C6	-0.0932 (4)	0.1236 (3)	1.0133 (4)	0.035 (2)
C7	0.0927 (3)	0.1715 (2)	0.6413 (3)	0.020 (1)
C8	-0.0427 (4)	0.1437 (2)	0.4965 (4)	0.029 (2)
C9	0.2072 (4)	0.0898 (2)	0.7016 (4)	0.027 (2)

Table 4. Selected geometric parameters (Å, °) for (2)

P—C1	1.860 (3)	P—C4	1.872 (3)
P—C7	1.856 (3)	C1—C2	1.529 (5)
C1—C3	1.522 (4)	C4—C5	1.531 (4)
C4—C6	1.527 (5)	C7—C8	1.534 (5)
C7—C9	1.533 (4)		
C7—P—C4	102.0 (1)	C7—P—C1	102.9 (1)
C4—P—C1	104.7 (1)	C3—C1—C2	109.9 (2)
C3—C1—P	110.5 (2)	C2—C1—P	108.6 (2)
C6—C4—C5	110.5 (3)	C6—C4—P	109.5 (2)
C5—C4—P	117.5 (2)	C9—C7—C8	111.1 (3)
C9—C7—P	109.0 (2)	C8—C7—P	117.4 (2)

The crystal decay of compound (2) during data collection is caused by mechanical stress in the capillary as described elsewhere (Bruckmann Krüger & Lutz, 1995). In the present case, the deterioration of the crystal is even more apparent because of the method of sample preparation (Bruckmann, 1995).

For both compounds, data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *DATAP* (Coppens, Leiserowitz & Rabinovich, 1965); program(s) used to solve structures: *SHELX86* (Sheldrick, 1990); program(s) used to refine structures: *SHELX93* (Sheldrick, 1993); *GFMLX* (Flack, 1983; Busing, Martin & Levy, 1962); molecular graphics: *ORTEPII* (Johnson, 1976); *XANADU* (Roberts & Sheldrick, 1976); *SYBYL* (Tripos Associates Inc., 1994); software used to prepare material for publication: *DAESD* (Davis & Harris, 1970); *DISCOVER94.0* (Biosym Technologies Inc., 1994).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1100). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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O-Phospho-DL-threonine and O-Phospho-L-threonine Compared with their Serine Analogs

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Abstract

In crystals of *O*-phospho-DL-threonine and *O*-phospho-L-threonine, the molecules are zwitterions HO₃⁻POCH₂(CH₃)CH(NH₃⁺)CO₂H linked by three-dimensional networks of strong P—O—H...O=P, C—O—H...O=P, N—H...O=P and N—H...O=C hydrogen bonds with (O...O) = 2.55 (3) Å and (N...O) = 2.84 (4) Å. Both the molecular conformations and the nearest-neighbor hydrogen-bonded surroundings are very similar in the racemic and enantiomeric crystals of the threonine compounds, but earlier studies of crystals of the analogous serine compounds have shown that the serine zwitterions HO₃⁻POCH₂CH(NH₃⁺)CO₂H have different conformations about the C_β—O_γ—P phosphate ester bonds and different hydrogen-bonded surroundings.

Comment

The phosphate esters, *O*_γ-phosphoserine, *O*_γ-phosphothreonine, *O*_ζ-phosphotyrosine, and the phosphoramidate, N δ 1- or N ϵ 2-phosphohistidine, correspond to the residues involved in the reversible protein phosphorylations that regulate many cellular processes. Crystal structure determinations have been reported for both DL and L forms of the phosphate esters (see scheme below) of both serine (Putkey & Sundaralingam, 1970; Sundaralingam & Putkey, 1970) and threonine (Cole, 1968; Slone & Cole, 1971) but no atomic coordinates or structural details were published for the threonine compounds. We have recently carried out charge-density analyses of the serine compounds (Kwiatkowski, Maniukiewicz & Blessing, 1994) and, preliminary to corresponding analyses of the threonine compounds, we have redetermined their crystal structures.

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