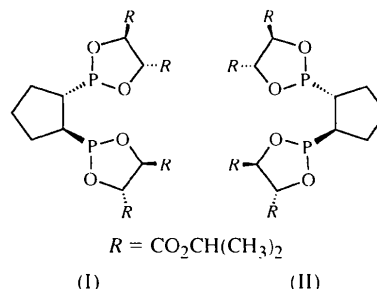


Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1426). Services for accessing these data are described at the back of the journal.

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giving 2,2'-(cyclopentane-1,2-diyl)bis(1,3,2-dioxaphospholane), *trans*-C₅H₈[(ⁱPrO₂C)HC^ROPOC^RH(CO₂-ⁱPr)]₂, as a mixture of (1*S*,2*S*) and (1*R*,2*R*) diastereomers. Crystallization of the diastereomeric mixture from diethyl ether resulted in preferential precipitation of the (1*S*,2*S*) form, (I), as shown by X-ray structure analysis.



A similar reaction employing (2*S*,3*S*)-(-)-diisopropyl tartrate as the diol component furnished the (1*S*,2*S*)- and (1*R*,2*R*)-configured epimers of *trans*-C₅H₈[(ⁱPrO₂C)-HC^SOPOC^SH(CO₂ⁱPr)]₂, as expected. Fractional crystallization of the compound from diethyl ether afforded one of the two stereoisomers as well shaped transparent

Acta Cryst. (1998). **C54**, 1016–1017

Chiral Bisphosphanes. IV.† Tetraisopropyl 2,2'-[(1*R*,2*R*)-Cyclopentane-1,2-diyl]bis-[(4*S*,5*S*)-1,3,2-dioxaphospholane-4,5-dicarboxylate]

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Abstract

The title compound, C₂₅H₄₀O₁₂P₂, was isolated in diastereomerically pure form from the reaction between *rac.trans*-C₅H₈(PCl₂)₂ and (2*S*,3*S*)-(-)-diisopropyl tartrate. The crystal structure determination reveals that the two P-bonded ring-C atoms have *R* configurations.

Comment

In previous work (Eckert *et al.*, 1995), we reported on the reaction between racemic *trans*-cyclopentane-1,2-diylbis(phosphonous dichloride) [*rac.trans*-C₅H₈(PCl₂)₂] and (2*R*,3*R*)-(+)-diisopropyl tartrate,

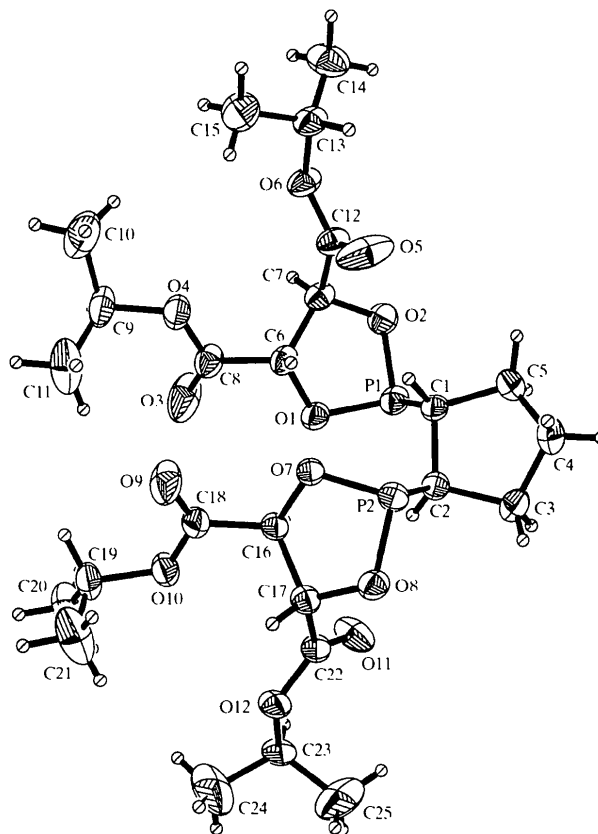


Fig. 1. The structure of the title molecule with displacement ellipsoids drawn at the 30% probability level.

† Part III: Dahlenburg & Kurth (1998).

colourless crystals, the structure of which was determined in order to ascertain the stereochemistry of the isomer so isolated.

The structure determination proves that the two P-bonded ring-C atoms possess *R* configurations (Fig. 1), *i.e.* the structure under investigation corresponds to that of the (1*R*,2*R*,4'*S*,5'*S*) enantiomer, (II), of the molecule studied earlier. As anticipated, crystals of (I) and (II) belong to the same monoclinic space group, *P*2₁. Due to differences in the temperatures of the measurements, 289 K for (I) but 178 K for (II), the unit-cell volume of (II) [1531.0(7) Å³] is slightly smaller than that of (I) [1584.6(4) Å³]. Interatomic distances in the molecule of (II) appear slightly elongated when compared with those of compound (I). Thus, the lengths of the P—O bonds range from 1.649(2) to 1.658(2) Å in (II), but vary between 1.635(3) and 1.655(3) Å in structure (I), the corresponding values for the P—C bonds being 1.828(3) and 1.829(3) Å *versus* 1.793(6) and 1.821(6) Å. The P—C—C—P torsion angle of molecule (II) amounts to −125.9(2)°, which compares favourably with that of 125.6(3)° previously measured for (I).

Experimental

Compound (II) was prepared employing a procedure analogous to that previously described for (I) (Eckert *et al.*, 1995). Single crystals were grown from diethyl ether.

Crystal data

C₂₅H₄₀O₁₂P₂
M_r = 594.51
 Monoclinic
*P*2₁
a = 9.680(3) Å
b = 17.611(4) Å
c = 10.076(2) Å
 β = 116.96(2)°
V = 1531.0(7) Å³
Z = 2
D_x = 1.290 Mg m^{−3}
D_m not measured

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 25 reflections
 θ = 9–17°
 μ = 0.199 mm^{−1}
T = 178(2) K
 Block
 0.6 × 0.6 × 0.4 mm
 Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω scans
 Absorption correction: none
 10 736 measured reflections
 5391 independent reflections
 4985 reflections with $I > 2\sigma(I)$

*R*_{int} = 0.045
 θ_{\max} = 25.04°
 $h = -11 \rightarrow 11$
 $k = -20 \rightarrow 20$
 $l = -11 \rightarrow 11$
 3 standard reflections
 frequency: 60 min
 intensity decay: <2%

Refinement

Refinement on *F*²
 $R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.140$

$\Delta\rho_{\max} = 0.436 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.455 \text{ e } \text{Å}^{-3}$
 Extinction correction: none

S = 1.041
 5380 reflections
 359 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1105P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = -0.002$

Scattering factors from *International Tables for Crystallography* (Vol. C)
 Absolute structure: Flack (1983)
 Flack parameter = 0.01 (9);
 2601 Friedel-related reflections used

Table 1. Selected geometric parameters (Å, °)

P1—O1	1.651 (2)	P2—C2	1.829 (3)
P1—O2	1.658 (2)	O1—C6	1.424 (3)
P1—C1	1.828 (3)	O2—C7	1.433 (3)
P2—O7	1.649 (2)	O7—C16	1.428 (3)
P2—O8	1.652 (2)	O8—C17	1.428 (4)
O1—P1—O2	93.73 (11)	C17—O8—P2	113.5 (2)
O1—P1—C1	102.72 (12)	C5—C1—P1	110.3 (2)
O2—P1—C1	102.29 (12)	C2—C1—P1	109.0 (2)
O7—P2—O8	93.97 (10)	C3—C2—P2	107.0 (2)
O7—P2—C2	102.79 (12)	C1—C2—P2	108.6 (2)
O8—P2—C2	102.48 (12)	O1—C6—C7	106.1 (2)
C6—O1—P1	109.9 (2)	O2—C7—C6	106.5 (2)
C7—O2—P1	112.9 (2)	O7—C16—C17	106.6 (2)
C16—O7—P2	119.3 (2)	O8—C17—C16	106.6 (2)

All non-H atoms were located by direct methods and subsequent alternate cycles of difference Fourier synthesis and full-matrix least-squares refinement. The resulting structural model was refined to convergence with allowance for anisotropic thermal motion of the non-H atoms. H atoms were included in geometrically idealized positions employing appropriate riding models with isotropic displacement parameters constrained to 1.3 times the *U*_{eq} of their carrier atoms.

Data collection: CAD-4-PC (Enraf–Nonius, 1995). Cell refinement: CAD-4-PC. Data reduction: MolEN (Fair, 1990). Program(s) used to solve structure: SIR92 (Altomare *et al.*, 1992). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: XP (Siemens, 1990). Software used to prepare material for publication: SHELXL93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1170). Services for accessing these data are described at the back of the journal.

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