

Table 2. Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses

C(2)—C(1')	1.523 (3)	C(2)—C(1)	1.364 (3)
C(6')—C(1')	1.518 (3)	C(8a)—C(1)	1.420 (2)
C(2)—C(1')	1.520 (2)	C(3)—C(2)	1.421 (3)
C(3')—C(2')	1.520 (2)	C(4)—C(3)	1.362 (3)
C(4')—C(3')	1.509 (4)	C(8a)—C(4a)	1.420 (3)
C(5')—C(4')	1.514 (4)	C(4a)—C(4)	1.416 (3)
C(6')—C(5')	1.523 (3)		
C(6')—C(1')—C(2')	110.7 (2)	C(8a)—C(1)—C(2)	122.0 (2)
C(2)—C(1')—C(2')	113.3 (2)	C(1)—C(2)—C(1')	121.1 (2)
C(2)—C(1')—C(6')	111.4 (2)	C(3)—C(2)—C(1')	120.6 (2)
C(3')—C(2')—C(1')	112.2 (2)	C(3)—C(2)—C(1)	118.3 (2)
C(4')—C(3')—C(2')	111.8 (2)	C(4)—C(3)—C(2)	121.3 (2)
C(5')—C(4')—C(3')	111.5 (2)	C(3)—C(4)—C(4a)	121.2 (2)
C(6')—C(5')—C(4')	111.6 (2)	C(1)—C(8a)—C(4a)	119.2 (2)
C(5')—C(6')—C(1')	111.8 (2)	C(8a)—C(4a)—C(4)	117.9 (2)

Table 3. Deviation from plane C(4)—C(4a)—C(8a)

Equation of plane: (0.6797)x _D + (0.5117)y _D + (0.5256)z _D = 10.5273			
Deviation from plane		Deviation from plane	
C(1')	-0.0052	C(4)*	-0.0000
C(2)	1.1311	C(8a)*	-0.0000
C(3')	1.0831	C(4a)*	-0.0000
C(4')	-0.2570	C(8)	-0.0000
	-1.3898	C(5)	-0.0021
C(6')	-1.3468	C(7)	0.0069
C(1)	0.0021	H(8)	-0.0221
C(2)	0.0153	C(6)	-0.0153
C(3)	0.0069	H(5)	-0.0254
		H(7)	-0.0187
		C(1'')	0.0052

hexane molecule by Davis & Hassel (1963), except the C(4')—C(3') and C(5')—C(4') bonds and the C(6')—C(1')—C(2') angle which are slightly modified.

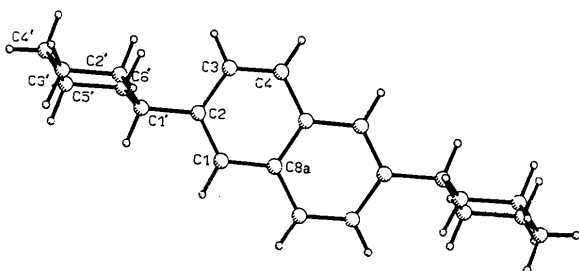


Fig. 1. Projection on the naphthalenic plane showing the numbering of atoms (PLUTO; Motherwell & Clegg, 1978).

with those found by Cruickshank (1957) for the naphthalene molecule, whereas some small differences appear in the angle values. In the same way, all the bond lengths and angles of the cyclohexyl ring fall within the range of values obtained in the cyclo-

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Absolute Configuration of (S)_p-O-Menthoxy-N-anilinophenylthiophosphonate*

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Abstract. C₂₂H₃₀NOPS, *M_r* = 387.53, orthorhombic, *P*2₁2₁2₁, *a* = 6.0211 (9), *b* = 8.6175 (8), *c* =

43.095 (5) Å, *V* = 2236.0 (5) Å³, *Z* = 4, *D_x* = 1.15, *D_m* = 1.146 g cm⁻³ (floatation in chlorobenzene/dichloroethane mixture), λ(Cu Kα) = 1.54178 Å, μ = 19.2 cm⁻¹, *F*(000) = 832, *T* = 293 K, *R* = 0.069 and *wR* = 0.064 for 3860 reflections collected on a

* Alternative nomenclature: (S)_p-O-2-menthyl (anilino)-(phenyl)thiophosphinate.

Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors (\AA^2)
$$U_{\text{eq}} = (U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	U_{eq}
P	3614 (2)	4703 (1)	8541.2 (2)	0.0437 (2)
S	6482 (2)	3697 (1)	8552.7 (3)	0.0636 (3)
N	1795 (6)	3931 (4)	8294 (1)	0.047 (1)
C(1N)	2052 (7)	3590 (5)	7978 (1)	0.047 (1)
C(2N)	390 (9)	2718 (6)	7834 (1)	0.063 (1)
C(3N)	632 (11)	2313 (8)	7525 (1)	0.085 (2)
C(4N)	2486 (12)	2793 (9)	7357 (1)	0.096 (2)
C(5N)	4113 (10)	3628 (8)	7503 (1)	0.092 (2)
C(6N)	3932 (9)	4048 (6)	7809 (1)	0.067 (1)
O	2273 (5)	4650 (3)	8858 (1)	0.047 (1)
C(1)	2607 (8)	3435 (5)	9090 (1)	0.053 (1)
C(2)	2021 (8)	4128 (6)	9403 (1)	0.061 (1)
C(3)	2341 (11)	2864 (8)	9644 (1)	0.088 (2)
C(4)	958 (12)	1406 (8)	9567 (1)	0.099 (2)
C(5)	1400 (11)	785 (6)	9256 (1)	0.078 (2)
C(6)	1214 (9)	2033 (6)	9011 (1)	0.063 (1)
C(7)	-75 (14)	-638 (7)	9175 (1)	0.120 (2)
C(8)	3265 (10)	5659 (7)	9472 (1)	0.074 (2)
C(9)	5776 (10)	5434 (9)	9487 (1)	0.099 (2)
C(10)	2491 (11)	6322 (8)	9771 (1)	0.107 (2)
C(1P)	3748 (9)	6763 (5)	8461 (1)	0.051 (1)
C(2P)	2061 (9)	7443 (6)	8295 (1)	0.067 (1)
C(3P)	2184 (11)	9026 (6)	8225 (1)	0.080 (2)
C(4P)	3991 (14)	9859 (6)	8322 (1)	0.085 (2)
C(5P)	5648 (12)	9188 (6)	8492 (1)	0.088 (2)
C(6P)	5608 (10)	7596 (6)	8555 (1)	0.074 (1)

Table 2. Selected bond lengths (\AA) and angles ($^\circ$) with *e.s.d.*'s in parentheses

P—N	1.667 (3)	P—S	1.933 (2)
P—C(1P)	1.810 (4)	P—O	1.585 (3)
N—C(1N)	1.403 (6)	O—C(1)	1.462 (6)
C(1N)—C(2N)	1.396 (7)	C(1)—C(2)	1.517 (6)
C(1N)—C(6N)	1.402 (7)	C(1)—C(6)	1.509 (7)
C(2N)—C(3N)	1.384 (6)	C(2)—C(3)	1.519 (8)
C(3N)—C(4N)	1.394 (9)	C(2)—C(8)	1.546 (8)
C(4N)—C(5N)	1.369 (9)	C(3)—C(4)	1.543 (10)
C(5N)—C(6N)	1.371 (6)	C(4)—C(5)	1.469 (7)
C(1P)—C(2P)	1.373 (7)	C(5)—C(6)	1.510 (7)
C(1P)—C(6P)	1.390 (8)	C(5)—C(7)	1.554 (9)
C(2P)—C(3P)	1.399 (7)	C(8)—C(9)	1.525 (9)
C(3P)—C(4P)	1.368 (10)	C(8)—C(10)	1.485 (7)
C(4P)—C(5P)	1.367 (9)		
C(5P)—C(6P)	1.399 (7)		
S—P—N	115.1 (1)	S—P—O	114.9 (1)
N—P—O	101.7 (2)	P—O—C(1)	122.6 (3)
N—P—C(1P)	107.4 (2)	O—C(1)—C(2)	107.1 (4)
O—P—C(1P)	102.4 (2)	O—C(1)—C(6)	110.1 (4)
P—N—C(1N)	129.2 (3)	N—C(1N)—C(2N)	117.7 (4)
N—C(1N)—C(6N)	122.3 (4)	P—C(1P)—C(2P)	119.0 (4)
P—C(1P)—C(6P)	119.1 (4)		

diffractometer. The absolute configuration of the chiral P atom is *S* while the (–)-menthyl group has the configuration *1R,2S,5R*. The P atom is at the centre of a distorted tetrahedron formed by C, O, N and S atoms; bond angles around the P atom are S—P—N 115.1 (1), S—P—O 114.9 (1), N—P—O 101.7 (2), N—P—C(1P) 107.4 (2), O—P—C(1P) 102.4 (2) and S—P—C(1P) 113.9 (2) $^\circ$. The menthyl and aniline fragments are *trans* oriented. The intermolecular hydrogen bond between the NH group and the S atom forms the chain parallel to the twofold screw axis (*a*).

Experimental. Single crystals were grown from an ethanol solution. A colourless and irregular crystal of dimensions $0.60 \times 0.20 \times 0.17$ mm was used for X-ray analysis. The Laue symmetry (*mmm*) and systematic extinctions ($h00$ $h=2n$, $0k0$ $k=2n$, $00l$ $l=2n$) indicated the space group $P2_12_12_1$. Precise unit-cell parameters were determined by a least-squares method from 25 reflections ($9 < 2\theta < 30^\circ$) centred on a CAD-4 diffractometer using graphite-monochromated Cu $K\alpha$ radiation. Reflections were measured with their Friedel pairs. The $\omega/2\theta$ -scan technique was used. Of 4819 reflections measured (hkl and $\bar{h}\bar{k}l$), 4016 unique; only $0kl$, $h0l$ and $hk0$ reflections were averaged ($R_{\text{int}} = 0.03$). Data were collected in the 2θ range 4 – 140° and index range $h - 7 \rightarrow 0$, $k 0 \rightarrow 10$, $l 0 \rightarrow 52$. Intensity variations of three standard reflections were $< 0.5\%$. Absorption corrections based on a ψ scan of six reflections were applied to the data; transmission factors were in the range 0.823 – 1.0 . The P, S, N and O atoms were found by direct methods and the remaining non-H atoms were located on a Fourier map. Positions of all H atoms were determined from the difference map. Parameters of H atoms were kept fixed throughout the refinement. The refinement, using atomic scattering factors with the imaginary dispersion term $f'' = 0$ for all non-H atoms, converged at $R = 0.0719$, $wR = 0.0684$ ($w = 1/\sigma^2$). Atomic parameters obtained in this refinement were used to calculate two sets of structure factors applying anomalous-dispersion factors (Rogers, 1981); one set was with positive signs for f'' and the second set had negative f'' values. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Resulting factors were $R(+)$ = 0.0759 , $wR(+)$ = 0.0717 and $R(-)$ = 0.0702 , $wR(-)$ = 0.0673 for 235 parameters and 3912 reflections with $F_o > 2\sigma(F_o)$. Application of the *R*-factor-

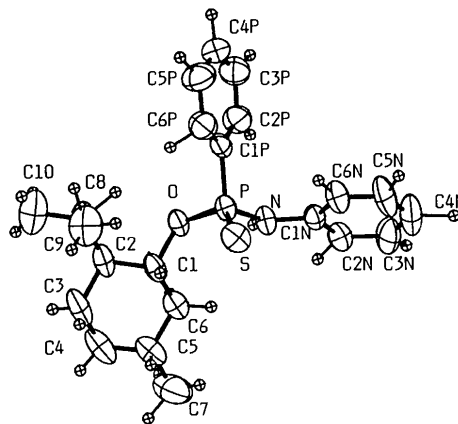
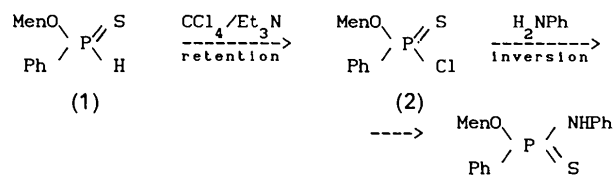


Fig. 1. ORTEPII (Johnson, 1976) plot of the molecule with the atom-numbering scheme.

ratio test (Hamilton, 1965) gave theoretical $\mathcal{R}_{1,3677,0.005} = 1.00108$ while the value obtained for this structure was $\mathcal{R} = wR(+)/wR(-) = 1.0654$. Hence, because $\mathcal{R} > \mathcal{R}_{1,3677,0.005}$, the probability that the opposite enantiomer is correct can be accepted at the 99.5% confidence level. The final full-matrix least-squares refinement of the correct enantiomer ($-x, -y, -z$) gave $R = 0.0690$ and $wR = 0.0644$ for 3860 observed reflections [$F_o > 3\sigma(F_o)$]. Function minimized was $\sum w(|F_o| - |F_c|)^2$. The extinction parameter, x , refined to $1.3(4) \times 10^{-7}$ where $F_{\text{corr}} = F(1 - xF^2/\sin\theta)$. Final $(\Delta/\sigma)_{\text{max}}$ was 0.001 and residual electron density $-0.45 < \Delta\rho < 0.71 \text{ e \AA}^{-3}$, the maximum was near to the P atom. Calculations were carried out using the *SHELXS86* (Sheldrick, 1986) and *SHELXL76* (Sheldrick, 1976) systems. Final atomic parameters for the correct enantiomer are given in Table 1.* Bond lengths and angles are listed in Table 2. The conformation and configuration of the molecule, and atomic numbering are shown in Fig. 1. The absolute configuration of the P atom is *S* while the menthyl group has the configuration 1*R*,2*S*,5*R*, which is consistent with that established for (–)-menthol (Ohrt & Parthasarathy, 1975). The intermolecular N—H...S hydrogen bond [N...S = 3.393(4), H...S = 2.43 Å, $\angle\text{N—H...S} = 176^\circ$] links the molecules to form a chain parallel to the shortest of the crystallographic axes, *a*.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54511 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The title compound is a product of stereospecific reactions investigated as part of the stereochemical studies on thiophosphonates (Łuczak, Łopusiński & Michalski, 1991).



Knowledge of the absolute configuration of the chiral P atom of *O*-menthoxy-*N*-anilinothiophosphonate enables the determination of the configurations of its precursors (1) and (2).

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Structure of 4,4-Diphenylcyclohex-2-en-1-one (Monoclinic Form)

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Abstract. C₁₈H₁₆O, $M_r = 248.33$, monoclinic, $P2_1/c$, $a = 7.139(6)$, $b = 15.823(2)$, $c = 12.472(5)$ Å, $\beta = 103.12(9)^\circ$, $V = 1372(1)$ Å³, $Z = 4$, $D_x = 1.202 \text{ g cm}^{-3}$, Mo $K\alpha_1$, $\lambda = 0.70930$ Å, $\mu = 0.7 \text{ cm}^{-1}$, and Cu $K\alpha_1$, $\lambda = 1.54056$ Å, $\mu = 5.3 \text{ cm}^{-1}$, $F(000) = 528$, $T = 295 \text{ K}$, $R = 0.052$ for 1245 reflections (Mo), 0.070 for 2164 reflections (Cu). The structure is disordered with two

(equivalent) molecular conformations occupying the same site (50:50). The cyclohexene ring has a C(5)-sofa conformation.

Experimental. Crystals from ethanol, colourless prisms, $0.25 \times 0.25 \times 0.4$ mm. Nonius CAD4-F diffractometer, monochromatized Mo $K\alpha$ and Cu $K\alpha$ radiations were used. Solution of the structure with a