Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
P	0.1668 (1)	0.1901 (1)	0.8535 (1)	0.049 (1)
O1	0.1001 (2)	0.0315 (2)	0.8493 (1)	0.068 (1)
O2	0.2808 (2)	0.5033 (2)	0.9112 (1)	0.071 (1)
C1	0.0662 (2)	0.3135 (3)	0.7799 (1)	0.063 (1)
C2	−0.0438 (3)	0.2553 (4)	0.7273 (2)	0.086 (1)
C3	0.3663 (2)	0.1883 (2)	0.8477 (1)	0.050 (1)
C4	0.4663 (3)	0.0983 (3)	0.9003 (1)	0.067 (1)
C5	0.6196 (3)	0.0875 (3)	0.8981 (2)	0.080 (1)
C6	0.6756 (3)	0.1648 (3)	0.8434 (2)	0.078 (1)
C7	0.5791 (3)	0.2531 (3)	0.7912 (2)	0.075 (1)
C8	0.4241 (3)	0.2669 (3)	0.7931 (1)	0.061 (1)
C9	0.1597 (2)	0.2803 (2)	0.9423 (1)	0.048 (1)
C10	0.2200 (2)	0.4275 (2)	0.9640 (1)	0.053 (1)
C11	0.2153 (3)	0.4863 (3)	1.0347 (1)	0.065 (1)
C12	0.1471 (3)	0.3995 (3)	1.0829 (1)	0.071 (1)
C13	0.0852 (3)	0.2558 (3)	1.0629 (1)	0.068 (1)
C14	0.0923 (2)	0.1969 (3)	0.9924 (1)	0.057 (1)
C15	0.3433 (4)	0.6549 (3)	0.9284 (2)	0.092 (1)

Table 2. *Selected geometric parameters* (Å, °)

P—O1	1.484 (2)	O2—C10	1.363 (3)
P—C1	1.783 (2)	O2—C15	1.427 (3)
P—C3	1.800 (2)	C1—C2	1.307 (3)
P—C9	1.799 (2)		
O1—P—C1	112.3 (1)	C1—P—C9	107.4 (1)
O1—P—C3	112.2 (1)	C3—P—C9	106.4 (1)
O1—P—C9	110.6 (1)	C10—O2—C15	118.7 (2)
C1—P—C3	107.6 (1)	P—C1—C2	119.3 (2)

Data collection, reduction and cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989). Structure solution, refinement, molecular graphics and preparation of material for publication: *SHELXTL/PC* (Sheldrick, 1990).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: NA1158). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Diphenyl(2-oxocyclohexyl)phosphine Sulfide

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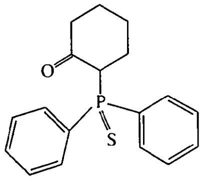
Abstract

The structure of $(C_6H_5)_2(C_6H_9O)PS$ is reported. The phenyl groups are not magnetically equivalent and P═S and C═O are not coplanar.

Comment

We have reported recently a general route leading to the formation of the title compound, (I) (Barkallah, Ben Akacha, Boukraa, Zantour & Baccar, 1993).



(I)

^{13}C and ^{1}H NMR data show that the phenyl groups linked to the P atom are not magnetically equivalent. X-ray diffraction data confirms this. Indeed, the torsion angles C2′—C1′—P—S and C2″—C1″—P—S are dif-

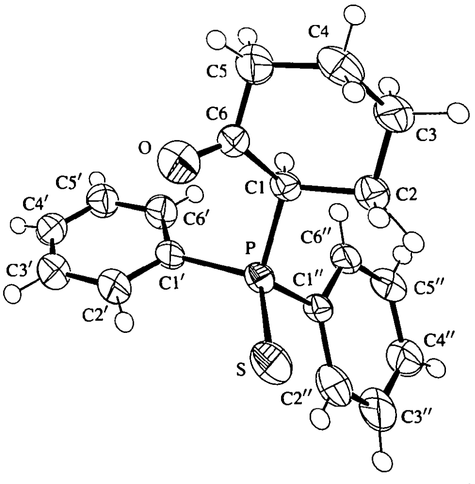


Fig. 1. Molecular structure of the title compound showing 50% probability displacement ellipsoids.

ferent [3.9 (3) and 32.1 (4)°, respectively]. The geometrical conformation adopted by the molecule does not allow the carbonyl O atom to be symmetrically situated between C1′ and C1″ of the two phenyl rings: the non-bonding distances between them are 3.158 (5) and 4.727 (5) Å, respectively, and the torsional angle S—P—C1—C6 is −66.9 (3)°. The P═S bond length of 1.947 (1) Å is in agreement with the value of 1.954 (5) Å given by Allen *et al.* (1987).

Finally, we note that the P═S and C═O groups are not coplanar. The X-ray data indicate an angle of 66.8 (2)° between the plane through S—P—C1 and that through C1—C6—O. This result may enable us to develop studies concerning P═S and C═O complexation.

Experimental

p-Chlorodiphenylphosphine (0.01 mol) was added to a mixture of 1-(1-pyrrolidinyl)cyclohexene (0.01 mol) and triethylamine (0.01 mol) in dry benzene (100 ml) under nitrogen at 263 K. After stirring for 2 h at 263 K, the reaction mixture was allowed to reach room temperature. Solid sulfur (0.01 mol) was added and the solution was then heated at reflux for 2 h. After stirring for 30 min at room temperature, the reaction mixture was washed with dilute hydrochloric acid (2*N*) and extracted with chloroform (50 ml). The organic phase was dried over anhydrous Na_2SO_4 and removed *in vacuo*. The resulting product was purified by recrystallization from benzene (yield 72%).

Crystal data

$C_{18}H_{19}OPS$
$M_r = 314.36$
Orthorhombic
$P2_12_12_1$
$a = 9.526\ (2)$ Å
$b = 10.194\ (2)$ Å
$c = 16.391\ (4)$ Å
$V = 1591.7\ (6)$ Å^3
$Z = 4$
$D_x = 1.312$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 22 reflections
$\theta = 14$–$16°$
$\mu = 0.30$ mm^{-1}
$T = 293\ (2)$ K
Parallelepiped
0.420 × 0.15 × 0.15 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans [width $(0.75 + 0.35\tan\theta)°$]
Absorption correction: none
5763 measured reflections
2790 independent reflections
2435 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0627$
$\theta_{\rm max} = 24.98°$
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = 0 \rightarrow 19$
3 standard reflections monitored every 200 reflections frequency: 60 min intensity decay: 0.79%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0503$
$wR(F^2) = 0.1257$
$S = 1.090$
2790 reflections
267 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0774P)^2 + 0.2866P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.078$
$\Delta\rho_{\rm max} = 0.239$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.191$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0027 (28)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
P	0.6984 (1)	0.36248 (8)	0.85953 (5)	0.0298 (2)
S	0.7055 (1)	0.18943 (9)	0.90964 (7)	0.0481 (3)
O	0.5073 (3)	0.2491 (3)	0.7316 (2)	0.0558 (9)
C1	0.5213 (4)	0.4194 (4)	0.8325 (2)	0.0303 (8)
C2	0.4235 (4)	0.4251 (5)	0.9074 (3)	0.0421 (9)
C3	0.2791 (4)	0.4769 (4)	0.8846 (3)	0.044 (1)
C4	0.2125 (5)	0.3976 (5)	0.8171 (3)	0.052 (1)
C5	0.3074 (5)	0.3875 (5)	0.7424 (3)	0.050 (1)
C6	0.4528 (4)	0.3404 (4)	0.7648 (2)	0.0353 (8)
C1′	0.8028 (4)	0.3772 (3)	0.7672 (2)	0.0313 (7)
C2′	0.8708 (4)	0.2683 (4)	0.7364 (3)	0.0394 (9)
C3′	0.9541 (4)	0.2788 (5)	0.6675 (3)	0.046 (1)
C4′	0.9699 (4)	0.3984 (5)	0.6304 (3)	0.048 (1)
C5′	0.9030 (6)	0.5076 (5)	0.6602 (3)	0.051 (1)
C6′	0.8195 (5)	0.4965 (4)	0.7288 (2)	0.045 (1)
C1″	0.7656 (4)	0.4892 (4)	0.9273 (2)	0.0298 (7)
C2″	0.8714 (5)	0.4577 (5)	0.9815 (3)	0.045 (1)
C3″	0.9248 (5)	0.5495 (5)	1.0350 (3)	0.052 (1)
C4″	0.8716 (5)	0.6755 (5)	1.0360 (3)	0.046 (1)
C5″	0.7687 (4)	0.7083 (4)	0.9820 (2)	0.0370 (9)
C6″	0.7152 (4)	0.6174 (4)	0.9269 (2)	0.0335 (8)

Table 2. *Selected geometric parameters* (Å, °)

P—C1′	1.817 (3)	C1′—C2′	1.381 (5)
P—C1″	1.820 (4)	C2′—C3′	1.385 (6)
P—C1	1.838 (4)	C3′—C4′	1.371 (7)
P—S	1.947 (1)	C4′—C5′	1.372 (7)
O—C6	1.197 (5)	C5′—C6′	1.382 (6)
C1—C6	1.518 (5)	C1″—C2″	1.381 (6)
C1—C2	1.541 (5)	C1″—C6″	1.393 (5)
C2—C3	1.520 (6)	C2″—C3″	1.380 (7)
C3—C4	1.510 (6)	C3″—C4″	1.380 (7)
C4—C5	1.526 (7)	C4″—C5″	1.362 (6)
C5—C6	1.511 (6)	C5″—C6″	1.391 (5)
C1′—C6′	1.378 (6)		
C1′—P—C1″	104.9 (2)	C6′—C1′—C2′	119.2 (4)
C1′—P—C1	106.0 (2)	C6′—C1′—P	121.2 (3)
C1″—P—C1	104.3 (2)	C2′—C1′—P	119.6 (3)
C1′—P—S	114.0 (1)	C1′—C2′—C3′	120.3 (4)
C1″—P—S	111.9 (1)	C4′—C3′—C2′	119.6 (4)
C1—P—S	114.8 (1)	C3′—C4′—C5′	120.8 (4)
C6—C1—C2	110.0 (3)	C4′—C5′—C6′	119.3 (5)
C6—C1—P	113.8 (3)	C1′—C6′—C5′	120.7 (4)
C2—C1—P	112.0 (2)	C2″—C1″—C6″	118.2 (4)
C3—C2—C1	111.4 (3)	C2″—C1″—P	119.0 (3)
C4—C3—C2	112.0 (4)	C6″—C1″—P	122.8 (3)
C3—C4—C5	112.0 (4)	C3″—C2″—C1″	121.3 (4)
C6—C5—C4	111.7 (3)	C2″—C3″—C4″	120.2 (4)
O—C6—C5	122.3 (4)	C5″—C4″—C3″	119.0 (4)
O—C6—C1	124.0 (4)	C4″—C5″—C6″	121.5 (4)
C5—C6—C1	113.8 (3)	C5″—C6″—C1″	119.7 (3)

Data were corrected for Lorentz–polarization effects. Data collection and cell refinement were performed using *CAD*-4 *Software* (Enraf–Nonius, 1989). *MolEN* (Fair, 1990) was used for data reduction. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985) and refined by full-matrix least-squares methods using *SHELXL*93 (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1112). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dicyclohexylammonium 2-Carboxybenzenesulfonate Dihydrate

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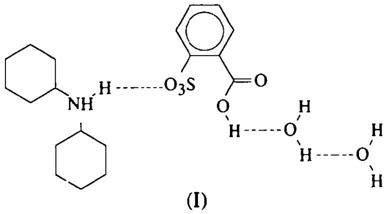
Abstract

In the title compound, $C_{12}H_{24}N^+.C_7H_5O_5S^-.2H_2O$, one of the two water molecules [$OW\cdots OW$ 2.732 (6) Å] forms hydrogen bonds to the adjacent carboxylic acid residues [$OW\cdots O_{\rm carboxyl}$ 2.600 (5), $OW\cdots O_{\rm carbonyl}$ 2.810 (6) Å], whereas the other water molecule forms hydrogen bonds to adjacent sulfonate groups [$OW\cdots O$ 2.791 (5) and 2.855 (5) Å]. The chains are held together by ammonium–sulfonate hydrogen bonds [$N\cdots O$ 2.791 (5) and 2.855 (5) Å].

Comment

Dicyclohexylammonium 2-carboxybenzenesulfonate dihydrate, (I), has been condensed with a triorganotin hemioxide/hydroxide to give dicyclohexylammonium 2-sulfobenzoatotriorganostannate (Ng, Kumar Das & Tiekink, 1991; Ng & Kumar Das, 1992), which displays a covalent carboxyl O—Sn bond. The synthesis implies the presence of a free carboxylic acid entity in the reagent. A free carboxylic group is also present in the ammonium (Okaya, 1967), potassium (Teplova, Turskaya, Shibanova, Nekrasov & Belikova, 1986), rubidium (Teplova, Turskaya, Tovbis, Zavodnik, Shibanova & Belikova, 1984) and caesium (Sorokina, Molchanov, Turskaya, Furmanova & Belikova, 1989) 2-carboxybenzenesulfonates, as well as in the parent acid trihydrate (Attig & Mootz, 1976).



(I)

The dicyclohexylammonium cation forms hydrogen bonds to adjacent negatively charged sulfonate groups. The neutral carboxylic acid group is hydrogen bonded to a water molecule and additional hydrogen bonds give rise to a three-dimensional network.

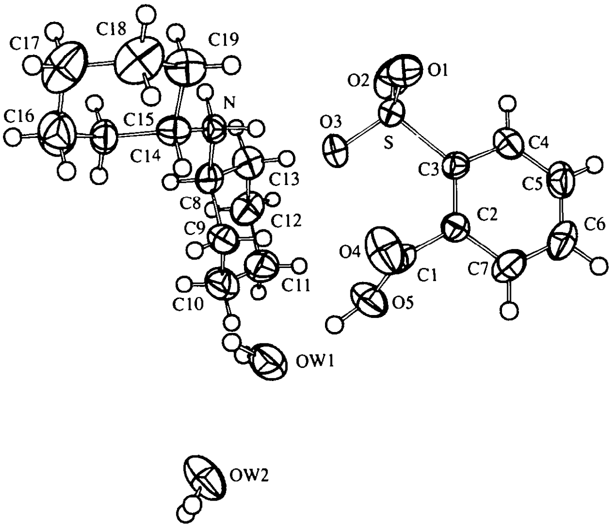


Fig. 1. Atomic labeling scheme for the title salt. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

Dicyclohexylamine was reacted with 2-sulfobenzoic acid in 1:1 molar amounts in hot ethanol. Slow evaporation of the solvent afforded large crystals of the title salt.

Crystal data

$C_{12}H_{24}N^+.C_7H_5O_5S^-.2H_2O$ Mo $K\alpha$ radiation
M_r = 419.52 λ = 0.71073 Å