

Triphenylphosphine oxide–
1-naphthoic acid (1/1)

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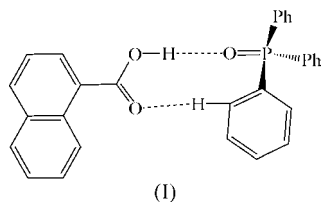
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In the title compound, $C_{18}H_{15}OP \cdot C_{11}H_8O_2$, co-crystallization of triphenylphosphine oxide with 1-naphthoic acid yields a supramolecular structure held together by one $O-H \cdots O$ and three $C-H \cdots O$ hydrogen bonds. The $O-H \cdots O$ hydrogen bond [$O \cdots O = 2.592(2) \text{ \AA}$] has little effect on the $O=P$ bond distance.

Comment

The role of hydrogen bonds in crystal engineering and the creation of supramolecular structures has attracted considerable research interest in recent years (Shan *et al.*, 2002; Lehn, 1995). Because of its strong directional interaction, the hydrogen bond can orient molecules into predictable aggregate patterns and often dictate the preferred molecular packing arrangement in organic crystals (Desiraju & Steiner, 1999; Etter *et al.*, 1986, 1990; Etter & Baures, 1988; Rebek *et al.*, 1987, 1988). On the other hand, the triphenylphosphine oxide (TPPO) molecule has a great conformational flexibility and hydrogen-bond formation ability. The O atom of TPPO can act as a single, double or triple hydrogen-bond acceptor, and the orientation of the phenyl groups is not restricted to



certain torsion-angle values (Llamas-Saiz *et al.*, 1991; Ferguson & Glidewell, 1988). These properties make TPPO a versatile hydrogen-bond acceptor and a very useful crystallization aid (Etter & Baures, 1988). As part of our studies on the role of hydrogen bonding in the interaction of TPPO with organic acids, the crystal structure determination of the title compound, (I), was undertaken and the results are presented here.

A view of (I), with the atomic numbering scheme, is given in Fig. 1 and selected geometric parameters are given in Table 1.

The title compound is isostructural with triphenylphosphine oxide–3-chlorobenzoic acid (1/1), (II) (Al-Farhan, 2003). The main hydrogen bond in the crystal structure is the $O2-H1 \cdots O1$ interaction (Table 2); the $C19-O2 \cdots O1$ and $P1-O1 \cdots O2$ angles are $118.2(2)$ and $142.97(10)^\circ$, respectively. If weak $C-H \cdots O$ interactions are also taken into consideration, then the 1-naphthoic acid, through its carboxyl group, is bonded to three TPPO molecules *via* four hydrogen bonds, as detailed in Table 2. The first two of these $O \cdots H$ interactions are involved in a hydrogen-bonded cyclic arrangement [graph-set $R_2^2(9)$; Bernstein *et al.*, 1995] and atom O3 acts as a bifurcated hydrogen-bond acceptor. The main hydrogen bond has little effect on the $O=P$ bond length; the $O1=P1$ distance [$1.492(2) \text{ \AA}$] in (I) is only 0.009 \AA longer than the corresponding distance in free TPPO (Al-Farhan, 1992). The $C=O$ [$1.210(3) \text{ \AA}$] and $C-OH$ [$1.316(3) \text{ \AA}$] bond lengths in the carboxyl group are statistically equal to the corresponding bonds [$1.214(3)$ and $1.312(3) \text{ \AA}$, respectively] in the crystal structure of 1-naphthoic acid (Fitzgerald & Gerkin, 1993).

The $O-P-C-C$ torsion angles of the TPPO moieties are $16.3(2)$, $14.2(2)$ and $71.7(2)^\circ$ in (I), and $21.2(2)$, $33.3(2)$ and $74.8(2)^\circ$ in (II); these values are distinctly different from the minimum-energy conformation (threefold symmetry and torsion angles of 40°) deduced from analysis of the observed conformations of a wide range of TPPO derivatives (Bye *et al.*, 1982).

The naphthalene core is planar to within $\pm 0.014(2) \text{ \AA}$. The dihedral angle between the plane of the carboxyl group and the plane of the phenyl ring is $8.1(3)^\circ$ in (I) and $3.0(4)^\circ$ in (II).

Examination of the structure with *PLATON* (Spek, 2003) showed that there were no solvent-accessible voids in the crystal structure of (I).

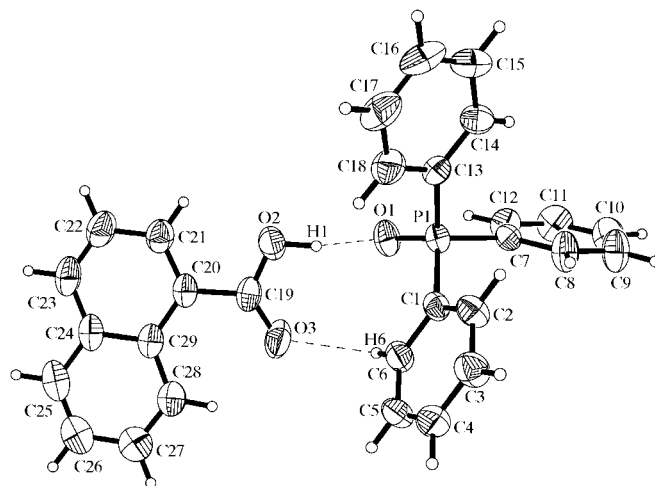


Figure 1

A view of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. The hydrogen bonds are indicated by dashed lines.

Experimental

The title compound was prepared by mixing equimolar amounts of TPPO and 1-naphthoic acid in CCl₄. Slow evaporation of the solvent afforded colourless crystals suitable for X-ray analysis.

Crystal data

C ₁₈ H ₁₅ OP·C ₁₁ H ₈ O ₂	D _x = 1.270 Mg m ⁻³
M _r = 450.44	Mo Kα radiation
Monoclinic, P2 ₁ /n	Cell parameters from 25 reflections
a = 8.963 (1) Å	θ = 17.2–17.9°
b = 16.303 (1) Å	μ = 0.15 mm ⁻¹
c = 16.673 (1) Å	T = 296 (2) K
β = 104.784 (7)°	Plate, colourless
V = 2355.7 (3) Å ³	0.30 × 0.28 × 0.12 mm
Z = 4	

Data collection

Siemens P4 diffractometer	h = -3 → 10
θ/2θ scans	k = -8 → 19
5488 measured reflections	l = -19 → 19
4083 independent reflections	3 standard reflections
2933 reflections with I > 2σ(I)	every 200 reflections
R _{int} = 0.021	intensity decay: none
θ _{max} = 25.0°	

Refinement

Refinement on F ²	w = 1/[σ ² (F _o ²) + (0.0476P) ² + 0.545P]
R[F ² > 2σ(F ²)] = 0.041	where P = (F _o ² + 2F _c ²)/3
wR(F ²) = 0.111	(Δ/σ) _{max} = 0.001
S = 1.01	Δρ _{max} = 0.21 e Å ⁻³
4083 reflections	Δρ _{min} = -0.22 e Å ⁻³
302 parameters	
H atoms: see below	

Table 1

Selected geometric parameters (Å, °).

P1—O1	1.4880 (15)	C19—C20	1.495 (3)
P1—C1	1.799 (2)	O2—C19	1.316 (3)
P1—C7	1.796 (2)	O2—H1	1.00 (4)
P1—C13	1.794 (2)	O3—C19	1.210 (3)
O1—P1—C1	112.42 (10)	C12—C7—P1	118.45 (17)
O1—P1—C7	109.77 (9)	C14—C13—P1	123.81 (17)
O1—P1—C13	112.66 (10)	C18—C13—P1	117.46 (19)
C1—P1—C7	108.59 (9)	O3—C19—O2	121.7 (2)
C1—P1—C13	105.34 (10)	O3—C19—C20	124.7 (3)
C7—P1—C13	107.83 (10)	O2—C19—C20	113.6 (2)
C2—C1—P1	122.26 (16)	C21—C20—C19	117.6 (2)
C6—C1—P1	118.82 (17)	C29—C20—C19	122.6 (2)
C8—C7—P1	123.26 (16)		
O1—P1—C1—C6	16.3 (2)	O3—C19—C20—C21	-174.7 (2)
O1—P1—C7—C12	14.2 (2)	C25—C24—C29—C20	-178.32 (19)
O1—P1—C13—C18	71.7 (2)		

H atoms bonded to C atoms were placed geometrically (C—H = 0.96 Å) and allowed to ride during structure refinement, with isotropic displacement parameters fixed at 1.2U_{eq} of the parent

Table 2

Hydrogen-bonding geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
O2—H1...O1	1.00 (3)	1.60 (3)	2.592 (2)	170 (3)
C6—H6...O3	0.96	2.70	3.589 (3)	155
C11—H11...O2 ⁱ	0.96	2.49	3.383 (4)	156
C17—H17...O3 ⁱⁱ	0.96	2.31	3.203 (4)	154

Symmetry codes: (i) 1 - x, 2 - y, 1 - z; (ii) x - 1, y, z.

atoms. The H atom of the carboxyl group was located in a difference Fourier map and was allowed to refine freely.

Data collection: XSCANS (Siemens, 1995); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Siemens, 1995); software used to prepare material for publication: SHELXL97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1732). Services for accessing these data are described at the back of the journal.

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