Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Polymorphic form of 1,3,5-tris[(diphenylphosphine oxide)methyl]benzene acetonitrile solvate monohydrate

Rafal Kruszynski* and Wanda Wieczorek

Institute of General and Ecological Chemistry, Technical University of Łódź, ul. Żeromskiego 116, 90-924 Łódź, Poland

Correspondence e-mail:
kruszyna@ck-sg.p.lodz.pl

Received 22 May 2001
Accepted 19 June 2001
Online 29 June 2001

Key indicators

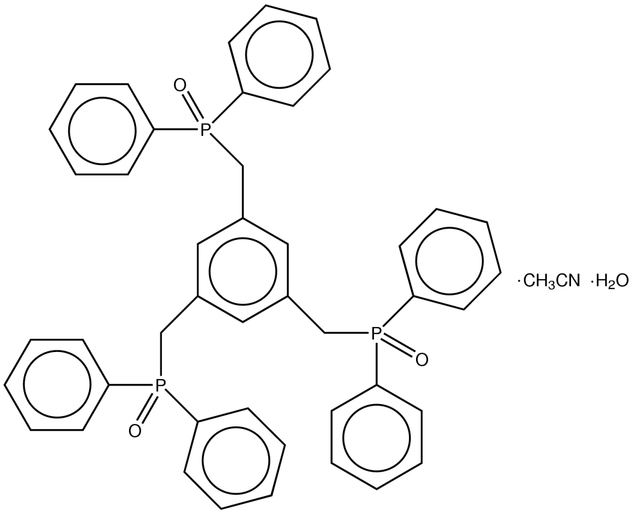
Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.013 Å
H-atom completeness 94%
R factor = 0.079
wR factor = 0.164
Data-to-parameter ratio = 13.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, benzene-1,3,5-triyltrismethylenetris(diphenylphosphine oxide) acetonitrile solvate monohydrate, $C_{45}H_{39}O_3P_3\cdot C_2H_3N\cdot H_2O$, forms two polymorphs: (I), which crystallizes in the triclinic space group *P*1, and (II), which crystallizes in the monoclinic space group *Pn*. The crystal and molecular structures of (I) were previously determined. The results of the crystal structure determination of (II) are presented here. The overall arrangement of two of the diphenylphosphine oxide substituents is determined by strong hydrogen bonds $O_{water}—H\cdots O_{oxide}$. All three P atoms adopt distorted tetrahedral geometry, with C—P—C angles equal to or smaller than tetrahedral and O—P—C angles greater than tetrahedral. All three diphenylphosphine oxide substituents are stabilized by weak $C_{phenyl}—H\cdots O_{oxide}$ hydrogen bonds. For each substituent, one of the phenyl rings is almost coplanar with the P=O bond.

Comment

The crystal and molecular structures of the title compound crystallizing in the *P*1 space group were previously determined [(I) hereafter; Cambridge Structural Database (CSD; Allen & Kennard, 1993) refcode GOKQAY (Pietrusiewicz *et al.*, 1998)]. Since the crystal structure of (I) had two pairs of only slightly different molecules in the independent part of the unit cell, and the cell parameters were close to those of the monoclinic system, we tried to obtain a second polymorph of (I), but all attempts failed. The crystals were stored in the dark at 293 K. After four years, we found that the crystals had changed in appearance. The colourless crystals of (I) had become pale pink. Thus, we decided to redetermine the crystal



(II)

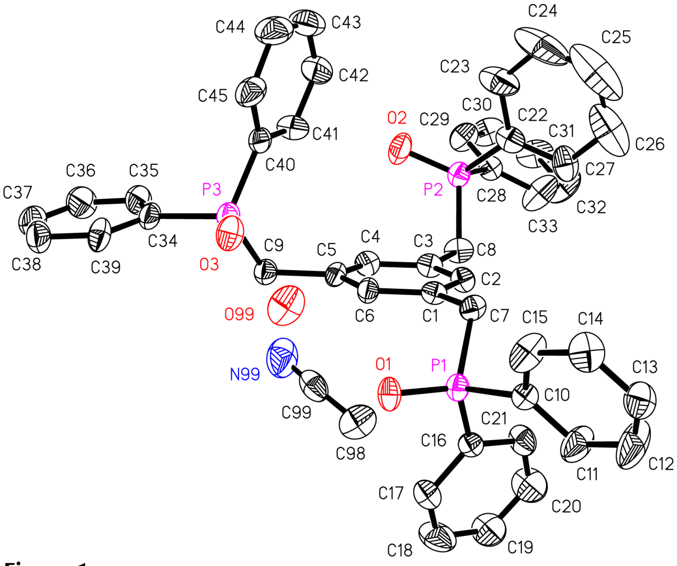


Figure 1

The molecular structure of (II). Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

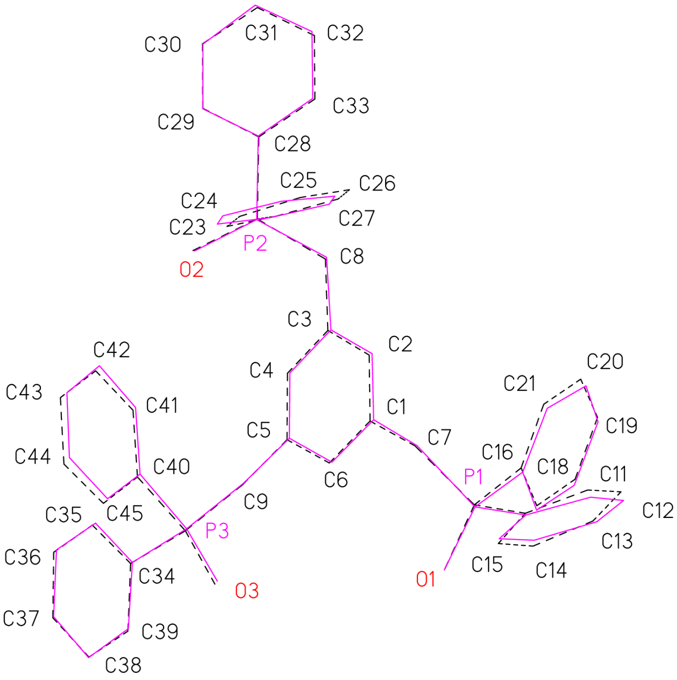


Figure 2

Superposition of the molecules of (I) and (II). The molecule of (I) is indicated by dashed lines. H atoms have been omitted for clarity.

structure of (I). Measurement of the unit-cell cell parameters revealed monoclinic crystal system (II).

The perspective view of (II) and the atomic numbering scheme are shown in Fig. 1. All interatomic distances can be considered as normal. We could not locate the H atoms of the acetonitrile molecule, probably because of the fully disordered methyl group. These H atoms are not involved in any hydrogen bond which will be able to provide additional stabilization [the shortest methyl group carbon to nearest atom distance is 3.645 (12) Å]. The phenyl group including the C22 atom shows signs of disorder, but invoking this model did not improve the quality of the refinement; therefore, the model was not applied. The overall molecular geometry of (II) is similar to that of (I) (Pietrusiewicz *et al.*, 1998). The weighted r.m.s. deviation for all atoms in (II) and the first and second molecules of (I) are 0.146 (3) and 0.154 (4) Å, respectively. Superposition of the two molecules, *i.e.* (II) and one of the molecules of (I), is shown in Fig. 2. The overall arrangement of the diphenylphosphine oxide substituents (best described by torsion angles, see Table 1) indicated by the P1 and P3 atoms is determined by strong $O_{water}—H\cdots O_{oxide}$ hydrogen bonds (for details, see Table 2). These also affect the dihedral angle between the central benzene ring and $P—CH_2C_{benzene}$ which amounts to 59.7 (3) and 58.9 (3)° for the branches indicated by the branches involving P1 and P3, and 76.2 (4)° for the branch involving P2.

The phenyl rings make angles of 67.0 (3), 78.8 (3) and 76.6 (2)° within the substituents involving P1, P2 and P3, respectively. All the P atoms adopt distorted tetrahedral geometry, with C—P—C angles equal to or smaller than tetrahedral and O—P—C angles greater than tetrahedral (Table 1). It is possible to suggest that the steric effect imposed by the phenyl groups has no great importance. All three diphenylphosphine oxide substituents are stabilized by weak hydrogen bonds $C_{phenyl}—H\cdots O_{oxide}$ (Table 2). For each substituent, one of the phenyl rings is almost coplanar with the P═O bond. The central benzene ring makes dihedral angles of 60.7 (3), 72.1 (2), 78.7 (3), 27.6 (3), 10.5 (2) and 82.8 (2)° with the phenyl rings involving C10, C15, C22, C28, C34 and C40, respectively; the respective P atoms deviate by 0.14 (1), 0.04 (1), 0.21 (1), 0.07 (1), 0.06 (1) and 0.00 (1) Å from the phenyl rings. There are no unusual intermolecular short contacts except for the hydrogen bonds described in Table 2.

Experimental

The title compound was prepared according to the method of Pietrusiewicz *et al.* (1998) and stored in the dark at 293 K for four years.

Crystal data

$C_{45}H_{39}O_3P_3\cdot C_2H_3N\cdot H_2O$
M_r = 779.74
Monoclinic, *Pn*
a = 15.3382 (14) Å
b = 8.6418 (9) Å
c = 15.9748 (11) Å
β = 105.034 (10)°
V = 2045.0 (3) Å^3
Z = 2
D_x = 1.266 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8473 reflections
θ = 2–22°
μ = 0.19 mm^{-1}
T = 291 (2) K
Pyramid, pale pink
0.60 × 0.40 × 0.40 mm

Data collection

Kuma KM4–CCD diffractometer
ω scans
Absorption correction: numerical (*X-RED*; Stoe & Cie, 1999)
T_{min} = 0.894, T_{max} = 0.928
20 214 measured reflections
6643 independent reflections
6532 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 25.2°
$h = -18 \rightarrow 18$
$k = -10 \rightarrow 10$
$l = -19 \rightarrow 18$
2 standard reflections every 50 reflections intensity decay: −1.1%

Acta Cryst. (2001). E**57**, o624–o626 Rafal Kruszynski *et al.* • $C_{45}H_{39}O_3P_3\cdot C_2H_3N\cdot H_2O$ **o625**

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.079$
$wR(F^2) = 0.164$
$S = 1.08$
6643 reflections
497 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0113P)^2 + 5.8292P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.42$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0083 (5)
Absolute structure: Flack (1983)
Flack parameter = 0.24 (15)

Table 1
Selected angles and torsion angles (°).

O1—P1—C16	112.5 (3)	O2—P2—C28	110.7 (3)
O1—P1—C10	112.3 (3)	C8—P2—C28	106.6 (3)
C16—P1—C10	108.4 (3)	C22—P2—C28	104.7 (3)
O1—P1—C7	113.5 (3)	O3—P3—C40	111.8 (3)
C16—P1—C7	106.9 (3)	O3—P3—C34	111.2 (3)
C10—P1—C7	102.7 (3)	C40—P3—C34	107.1 (3)
O2—P2—C8	113.2 (3)	O3—P3—C9	114.1 (3)
O2—P2—C22	114.3 (4)	C40—P3—C9	109.7 (3)
C8—P2—C22	106.7 (3)	C34—P3—C9	102.3 (3)
C1—C7—P1—C10	170.6 (5)	C8—P2—C28—C33	−49.5 (8)
C7—P1—C10—C15	65.2 (6)	C3—C8—P2—O2	−56.7 (6)
C6—C1—C7—P1	60.7 (7)	O2—P2—C22—C27	165.5 (6)
C1—C7—P1—C16	56.7 (5)	O2—P2—C28—C33	−173.0 (7)
C7—P1—C16—C21	43.8 (6)	C5—C9—P3—C34	169.3 (5)
C1—C7—P1—O1	−68.0 (5)	C9—P3—C34—C35	−60.7 (7)
O1—P1—C10—C15	−57.1 (6)	C6—C5—C9—P3	60.2 (7)
O1—P1—C16—C21	169.0 (5)	C5—C9—P3—C40	55.8 (5)
C3—C8—P2—C22	69.9 (5)	C9—P3—C40—C41	17.0 (7)
C8—P2—C22—C27	39.6 (7)	C5—C9—P3—O3	−70.5 (5)
C4—C3—C8—P2	75.5 (7)	O3—P3—C34—C35	177.1 (6)
C3—C8—P2—C28	−178.6 (5)	O3—P3—C40—C41	144.6 (6)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O99—H99*P*···O1	0.86	2.02	2.861 (7)	166
O99—H99*O*···O3	0.77	2.06	2.821 (7)	169
C15—H15···O1	0.93	3.02	3.268 (9)	97
C17—H17···O1	0.93	2.65	3.048 (9)	107
C23—H23···O2	0.93	2.70	3.099 (13)	107
C45—H45···O3	0.93	2.76	3.093 (10)	103
C29—H29···O2	0.93	2.58	2.989 (10)	107
C39—H39···O3	0.93	2.60	3.021 (8)	108

Data collection: *CrysAlis CCD* (UNIL IC & Kuma, 2000); cell refinement: *CrysAlis RED* (UNIL IC & Kuma, 2000); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 1990*b*) and *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

This work was supported financially by statutory funds allocated by the State Committee for Scientific Research, Warsaw, Poland, to the Institute of General and Ecological Chemistry, Technical University of Łódź.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Pietrusiewicz, K. M., Kataev, V. E., Patsanovsky, I. I., Ermolaeva, L. V., Ishmaeva, E. A., Kataev, A. V., Wieczorek, W. & Zygo, K. (1998). *Zh. Obshch. Khim.* **68**, 1510–1518.
Sheldrick, G. M. (1990*a*). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1990*b*). *SHELXTL/PC Software*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Stoe & Cie (1999). *X-RED*. Version 1.18. Stoe and Cie GmbH, Darmstadt, Germany.
UNIL IC & Kuma (2000). *CrysAlis CCD* (Version 1.163) and *CrysAlis RED* (Version 1.163). Kuma Diffraction Instruments GmbH, Wrocław, Poland.

o626 Rafal Kruszynski *et al.* • $C_{45}H_{39}O_3P_3 \cdot C_2H_3N \cdot H_2O$ *Acta Cryst.* (2001). E**57**, o624–o626