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Tris(3-nitrophenyl)phosphine oxide

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Key indicators

Single-crystal X-ray study
T = 105 K
Mean σ(C–C) = 0.005 Å
R factor = 0.044
wR factor = 0.087
Data-to-parameter ratio = 8.5

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

The title compound, $C_{18}H_{15}N_3O_7P$, has a P═O bond length of 1.486 (2) Å. The P—C bond lengths range from 1.806 (4) to 1.812 (3) Å, and the C—N bond distances are in the range 1.471 (4)–1.479 (5) Å.

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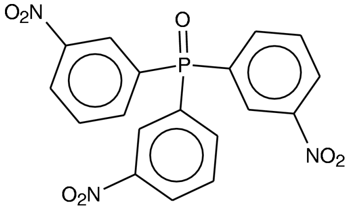
Comment

Arylphosphines with highly polar functional groups are the most frequently used as ligands in aqueous transition metal catalysis (Dressick *et al.*, 2000; Stelzer, 1998). While preparing potentially water-soluble nitrogen heterocycle derivatized arylphosphines (Hessler *et al.*, 1997), we have obtained tris(3-nitrophenyl)phosphine oxide, (I).



(I)

The molecular structure of (I) is shown in Fig. 1 and selected bond distances and angles are given in Table 1. The latter correspond well with those of related compounds, such as tris(3-chlorophenyl)phosphine oxide (Shawkataly *et al.*, 1997) and triphenylphosphine oxide (Baures & Silverton, 1990; Thomas & Hamor, 1993). The torsion angles in Table 1 indicate that the benzene rings are twisted by different amounts away from conformations having the nitro groups eclipsed with P═O. Furthermore, while nitro groups N2 and N3 are essentially coplanar with the attached benzene rings, nitro group N1 is twisted slightly [7.5 (5)°] out of its benzene plane.

Experimental

Melting points were determined in open capillaries and are uncorrected. ^1H and ^{13}C NMR spectra were acquired in DMSO-d_6 on a Varian Unity Inova 400 MHz spectrometer with TMS as the internal standard. ^{31}P NMR spectra were acquired in DMSO-d_6 on a Bruker DPX-250 MHz spectrometer with 85% H_3PO_4 as the internal reference. The title compound was prepared using standard aromatic nitration procedures (Furniss *et al.*, 1998). Triphenylphosphine (6.10 g, 23.26 mmol) was added to 36 ml of cold concentrated H_2SO_4. 12 ml of cold concentrated H_2SO_4 was slowly added to 12 ml of cold concentrated HNO_3. This mixture was added to the cold triphenylphosphine–H_2SO_4 mixture dropwise with stirring. During the addition, the reaction mixture turned a dark rust color. When the addition was complete, the reaction mixture was warmed to room temperature and then stirred for 2 h. The crude product was precipitated by pouring the reaction mixture over an excess of ice and water, filtering and washing with excess water. Soxhlet extraction of the crude product with chloroform resulted in 7.75 g (81%) of (I) as a light-yellow powder. X-Ray quality crystals were grown by slow evaporation of a saturated solution of (I) in chloroform (m.p. 511–513 K). ^{1}H NMR (DMSO-d_6): 8.53 (*m*, 2H), 8.20 (*m*, 1H), 7.90 p.p.m. (*m*, 1H); ^{13}C NMR (DMSO-d_6): 138.50 (*d*, $J_{\rm CP}$ = 10.7 Hz), 133.89, 132.85, 131.86 (*d*, $J_{\rm CP}$ = 12.3 Hz), 128.43, 126.98 p.p.m. (*d*, $J_{\rm CP}$ = 11.4 Hz); ^{31}P NMR (DMSO-d_6): 23.96 p.p.m.; IR (KBr disk): 3010, 1606, 1522, 1347, 881 cm^{-1}; FAB–MS: *m*/*z* 413.90 (M^+).

Crystal data

$C_{18}H_{12}N_3O_7P$
$M_r = 413.28$
Orthorhombic, $Pca2_1$
$a = 19.1402\ (9)$ Å
$b = 5.4608\ (2)$ Å
$c = 16.8888\ (7)$ Å
$V = 1765.23\ (13)$ Å^3
$Z = 4$
$D_x = 1.555$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 2409 reflections
$\theta = 2.5$–$28.3°$
$\mu = 0.21$ mm^{-1}
$T = 105$ K
Needle, colorless
0.45 × 0.05 × 0.05 mm

Data collection

KappaCCD diffractometer with an Oxford Cryosystems Cryostream cooler
ω scans with κ offsets
Absorption correction: multi-scan (*HKL SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\rm min} = 0.896$, $T_{\rm max} = 0.990$

12164 measured reflections
2233 independent reflections
1731 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.063$
$\theta_{\rm max} = 28.3°$
$h = -25 \rightarrow 25$
$k = -7 \rightarrow 7$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.087$
$S = 1.04$
2233 reflections
263 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0336P)^2 + 0.4389P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.29$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.41$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0052 (11)

Table 1
Selected geometric parameters (Å, °).

P1—O1	1.486 (2)	N1—C3	1.471 (5)
P1—C1	1.812 (3)	N2—C9	1.479 (5)
P1—C7	1.810 (4)	N3—C15	1.471 (4)
P1—C13	1.806 (4)		
O1—P1—C13	113.76 (18)	O1—P1—C1	111.87 (14)
O1—P1—C7	112.17 (17)	C13—P1—C1	106.27 (18)
C13—P1—C7	107.80 (16)	C7—P1—C1	104.36 (18)
O1—P1—C1—C2	−41.1 (4)	O4—N2—C9—C8	−1.1 (5)
O2—N1—C3—C2	7.5 (5)	O1—P1—C13—C14	130.8 (3)
O1—P1—C7—C8	145.7 (3)	O6—N3—C15—C14	−0.6 (5)

The Flack (1983) parameter refined to a value of 0.48(13), thus the crystal was assumed to be an inversion twin, and Friedel pairs were averaged for the final refinement. H atoms attached to C atoms were placed in idealized positions, with bond distances of 0.95 Å and displacement parameters assigned as $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

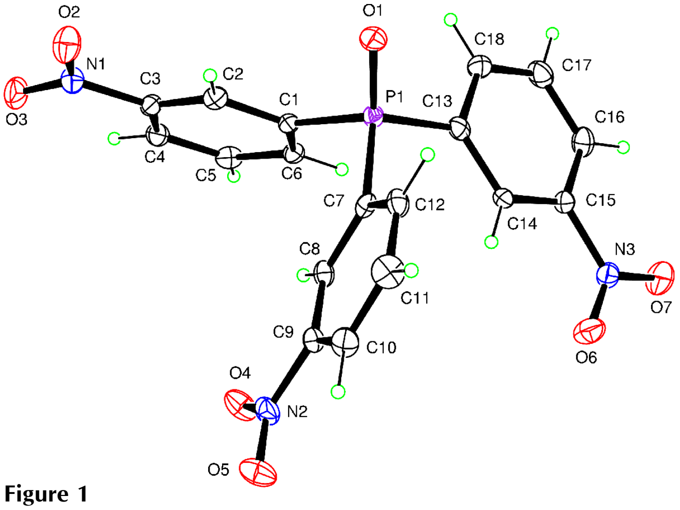


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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