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Tris[2-(methylsulfanyl)phenyl]phosphine

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Key indicators

Single-crystal X-ray study
T = 170 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.135
Data-to-parameter ratio = 21.5

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

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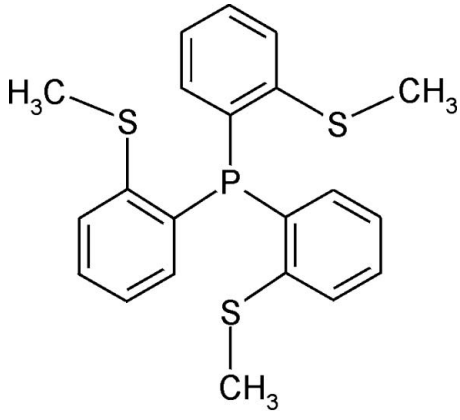
In the crystal structure of the title compound, $C_{21}H_{21}PS_3$, two crystallographically independent molecules are found. One of these molecules is located in a general position, whereas the P atom of the second molecule is located on a threefold rotation axis.

Comment

The structure determination of the title compound, (I), was undertaken as part of a project on the synthesis of transition metal complexes with tripodal ligands. Recrystallization of (I) from dichloromethane and diethyl ether led to single crystals.



(I)

The asymmetric unit is built up of one molecule located in a general position, and a second molecule with the P atom lying on a threefold rotation axis. The P—C bond lengths are 1.838 (3), 1.838 (3) and 1.841 (3) Å for the first molecule and 1.835 (2) Å for the second molecule. The C—P—C angles are 101.39 (11), 103.03 (12) and 101.87 (12)° for the first molecule and 101.65 (9)° for the second molecul (Table 1 and Fig. 1).

Experimental

A solution of 2-bromothioanisole (5 g, 24.6 mmol) in diethyl ether (25 ml) was treated with an equimolar quantity of *n*-butyllithium (1.6 *M* in *n*-hexane) for 2 h at 273 K under an argon atmosphere. Phosphorus trichloride (1.4 g, 10 mmol) dissolved in diethyl ether (15 ml) was added over a period of 3 h. After hydrolysis with 0.2 *N* hydrochloric acid (15 ml), the colourless precipitate was washed with water, ethanol and diethyl ether (Dyer & Meek, 1965) (yield 74%). Single crystals were obtained by diffusion of diethyl ether into a solution of the product in dichloromethane.

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Crystal data

$C_{21}H_{21}PS_3$
M_r = 400.53
Trigonal, $R\overline{3}$
a = 23.4523 (15) Å
c = 25.4022 (18) Å
V = 12099.6 (14) Å^3
Z = 24
D_x = 1.319 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7998 reflections
θ = 12.5–23°
μ = 0.45 mm^{-1}
T = 170 (2) K
Block, colourless
0.3 × 0.25 × 0.25 mm

Data collection

Stoe IPDS-1 diffractometer
φ scans
34061 measured reflections
6469 independent reflections
5279 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 28.0°
$h = -30 \rightarrow 30$
$k = -30 \rightarrow 30$
$l = -33 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.135
S = 1.03
6469 reflections
301 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0594P)^2 + 57.688P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P1—C1	1.838 (3)	P1—C11	1.841 (3)
P1—C21	1.838 (3)	P2—C31	1.835 (2)
C1—P1—C21	101.39 (11)	C21—P1—C11	101.87 (12)
C1—P1—C11	103.03 (12)	C31^{i}—P2—C31	101.65 (9)

Symmetry code: (i) $-y+1, x-y+1, z$.

The positions of all H atoms were located in a difference map but were positioned with idealized geometry and refined using a riding model with C—H = 0.95 Å for aromatic and 0.98 Å for methyl H atoms [U_{iso}(H) = 1.2U_{eq}(aromatic C) = 1.5U_{eq}(methyl C)]. The highest peak in the electron density map is located 1.03 Å from atom P1.

Data collection: *IPDS* (Stoe & Cie, 1998); cell refinement: *IPDS*; data reduction: *IPDS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: CIFTAB in *SHELXL97*.

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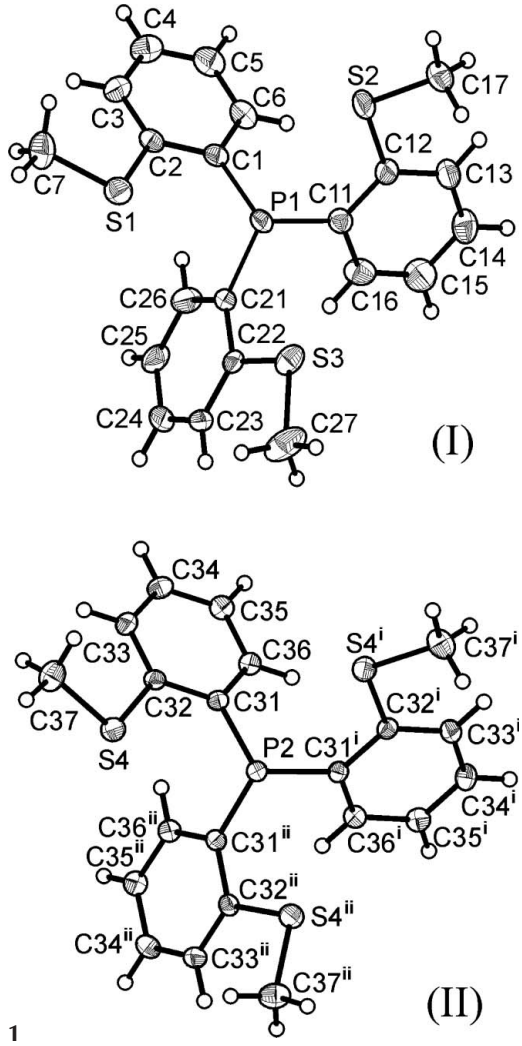

Figure 1
The crystal structure of the asymmetric unit of the title compound, showing the atom labelling and displacement ellipsoids at the 50% probability level. [Symmetry codes: (i) $1-y, 1+x-y, z$; (ii) $-x+y, 1-x, z$.]

References

Brandenburg, K. (1999). *DIAMOND*. Version 2.1c. Crystal Impact GbR, Bonn, Germany.
Dyer, G. & Meek, D. W. (1965). *Inorg. Chem.* **4**, 1398–1402.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stoe & Cie (1998). *IPDS*. Version 2.89. Stoe & Cie, Darmstadt, Germany.