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trans-Bis(4-methoxythiophenolato-κ*S*)-bis(trimethylphosphine-κ*P*)nickel(II)

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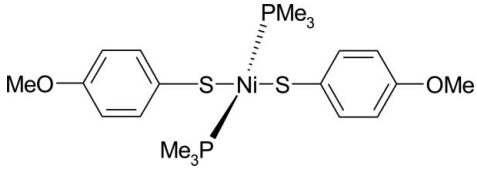
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.085; data-to-parameter ratio = 13.7.

The title compound, $[Ni(C_7H_7OS)_2(C_3H_9P)_2]$, was obtained as a product of the reaction of $[NiMe_2(PMe_3)_3]$ with two molar equivalents of 4-methoxythiophenol in diethyl ether. The compound is stable in the air for several hours, but rapidly decomposes at room temperature in solution. The Ni atom displays a square-planar coordination with two *P*-donor atoms lying in *trans* positions. The benzene rings of the thiophenolate ligands are almost perpendicular to the square coordination plane, making dihedral angles of 80.43 (4) and 72.60 (4)°.

Related literature

For the crystal structures of related diphenolato-nickel compounds, see: Klein *et al.* (1998). For synthetic details, see: Klein & Karsch (1972).



Experimental

Crystal data

$[Ni(C_7H_7OS)_2(C_3H_9P)_2]$
M_r = 489.23
Monoclinic, $P2_1/c$
a = 14.022 (3) Å
b = 15.983 (3) Å
c = 10.758 (2) Å
β = 100.93 (3)°
V = 2367.3 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.14 mm^{-1}
T = 273 (2) K
0.30 × 0.24 × 0.21 mm

Data collection

Bruker *P*4 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.725, T_{max} = 0.795
14644 measured reflections
5103 independent reflections
4078 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
14954 standard reflections
every 6 reflections
intensity decay: 30%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.085
S = 1.00
5103 reflections
372 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—P1	2.2121 (7)	Ni1—S2	2.2261 (9)
Ni1—P2	2.2224 (7)	Ni1—S1	2.2288 (9)
P1—Ni1—P2	178.07 (2)	P1—Ni1—S1	92.17 (3)
P1—Ni1—S2	87.01 (3)	P2—Ni1—S1	87.67 (3)
P2—Ni1—S2	93.17 (3)	S2—Ni1—S1	178.85 (2)

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FI2053).

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Klein, H.-F., Dal, A., Jung, T., Braun, S., Roehr, C., Floerke, U. & Haupt, H.-J. (1998). *Eur. J. Inorg. Chem.* pp. 621–627.
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supporting information

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trans-Bis(4-methoxythiophenolato-κS)bis(trimethylphosphine-κP)nickel(II)

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S1. Comment

In the title molecule (Fig.1) the nickel atom is coordinated in a square-planar geometry by two P atoms of trimethylphosphine groups and two S atoms of thiophenol groups. The phenyl rings of the thiophenlato ligands are almost perpendicular to the square coordination plane (angles 80.43 (4)° and 72.60 (4)°). Similar crystal structures have been reported in the literature, *e.g.* Bis(2-*tert*-butyl-4-methylphenolato)bis(trimethylphosphane)nickel and Bis(2-*tert*-butyl-6-methylphenolato)bis(trimethylphosphane)nickel (Klein *et al.*(1998)). The bond lengths and angles of these compounds are similar to those in the title compound.

S2. Experimental

Dimethyltris(trimethylphosphine)nickel was prepared according to the literature (Klein & Karsch (1972)). Other chemicals were used by purchased. To the solution of $NiMe_2(PMe_3)_3$ (1.0 g, 3.15 mmol) in 50 ml of diethyl ether was added 4-methoxythiophenol (0.884 g, 6.30 mmol) at -80 °C, a dark red suspension formed rapidly. After stirring at room temperature for 16 h the reaction solution was filtrated, then the red solid residue was extracted with THF (50 ml). Crystallization from ether and THF at 4 °C afforded dark red crystals suitable for X-ray diffraction analysis. (yield: 0.89 g, 57.8%, m. p.: 135 °C).

S3. Refinement

All H atoms were positioned geometrically and were refined freely (C—H = 0.86–1.02 Å).

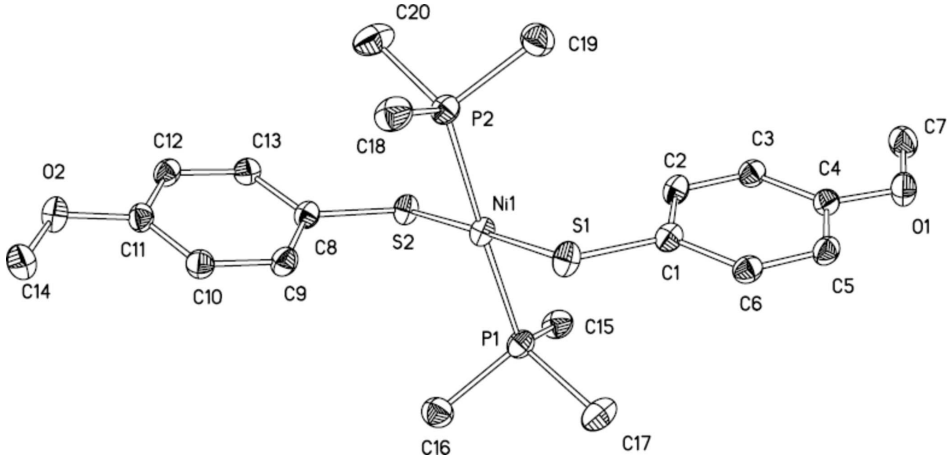

Figure 1

The molecular structure of (I), with a 30% probability displacement ellipsoids for non-H atoms.

[*trans*-Bis(4-methoxythiophenolato-κS)bis(trimethylphosphine-κP)nickel(II)

Crystal data

$[Ni(C_7H_7OS)_2(C_3H_9P)_2]$
$M_r = 489.23$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 14.022$ (3) Å
$b = 15.983$ (3) Å
$c = 10.758$ (2) Å
$\beta = 100.93$ (3)°
$V = 2367.3$ (8) Å^3
$Z = 4$
$F(000) = 1032$
$D_x = 1.373$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1843 reflections
$\theta = 2.4$–$24.3°$
$\mu = 1.14$ mm^{-1}
$T = 273$ K
Cubic, dark red
0.30 × 0.24 × 0.21 mm

Data collection

Bruker P4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.725$, $T_{max} = 0.795$
14644 measured reflections
5103 independent reflections
4078 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 27.1°$, $\theta_{min} = 1.5°$
$h = -17 \rightarrow 17$
$k = -20 \rightarrow 20$
$l = -13 \rightarrow 13$
14954 standard reflections every 6 reflections
intensity decay: 30%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.085$
$S = 1.00$
5103 reflections
372 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Ni1	0.257048 (17)	0.016260 (16)	0.74629 (2)	0.03027 (9)
P1	0.37880 (4)	0.08109 (3)	0.68296 (5)	0.03426 (13)
P2	0.13706 (4)	−0.05297 (3)	0.80892 (5)	0.03389 (13)

S1	0.17664 (4)	0.00061 (3)	0.54812 (5)	0.03656 (13)
S2	0.33803 (4)	0.03453 (3)	0.94334 (5)	0.03455 (12)
C1	0.24429 (14)	−0.07893 (12)	0.48988 (19)	0.0322 (4)
C2	0.30721 (15)	−0.13200 (13)	0.56961 (19)	0.0345 (4)
C3	0.35857 (15)	−0.19580 (13)	0.52267 (19)	0.0336 (4)
C4	0.34651 (14)	−0.20757 (12)	0.39280 (19)	0.0319 (4)
C5	0.28431 (15)	−0.15531 (13)	0.31149 (19)	0.0334 (4)
C6	0.23405 (15)	−0.09207 (13)	0.35826 (19)	0.0318 (4)
C7	0.4551 (2)	−0.32462 (15)	0.4172 (3)	0.0473 (6)
C8	0.25968 (14)	0.10267 (12)	1.00678 (18)	0.0317 (4)
C9	0.19724 (15)	0.15744 (13)	0.9314 (2)	0.0349 (4)
C10	0.13341 (16)	0.20890 (13)	0.9820 (2)	0.0352 (4)
C11	0.13282 (14)	0.20600 (12)	1.11072 (19)	0.0330 (4)
C12	0.19604 (15)	0.15232 (13)	1.18851 (19)	0.0344 (4)
C13	0.25869 (15)	0.10109 (13)	1.13772 (19)	0.0330 (4)
C14	0.00536 (19)	0.30705 (15)	1.0914 (3)	0.0465 (6)
C15	0.49677 (17)	0.03403 (16)	0.7405 (3)	0.0439 (5)
C16	0.3913 (2)	0.18866 (15)	0.7388 (3)	0.0465 (5)
C17	0.3817 (2)	0.0948 (2)	0.5153 (2)	0.0527 (6)
C18	0.01701 (18)	−0.00824 (17)	0.7602 (3)	0.0492 (6)
C19	0.1249 (2)	−0.15763 (16)	0.7437 (3)	0.0489 (6)
C20	0.1384 (3)	−0.0739 (2)	0.9760 (3)	0.0594 (7)
O1	0.39280 (12)	−0.26858 (9)	0.33592 (14)	0.0417 (4)
O2	0.07266 (11)	0.25359 (10)	1.16992 (14)	0.0409 (3)
H8	0.3030 (17)	0.0637 (14)	1.193 (2)	0.037 (6)*
H4	0.1928 (17)	−0.0562 (14)	0.305 (2)	0.036 (6)*
H2	0.4020 (17)	−0.2288 (14)	0.581 (2)	0.037 (6)*
H6	0.0932 (18)	0.2463 (15)	0.926 (2)	0.042 (6)*
H1	0.3157 (18)	−0.1256 (15)	0.658 (2)	0.045 (7)*
H7	0.1979 (17)	0.1481 (13)	1.278 (2)	0.038 (6)*
H3	0.2763 (18)	−0.1623 (15)	0.226 (2)	0.043 (6)*
H30	0.083 (2)	−0.1094 (17)	0.983 (3)	0.060 (8)*
H21	0.444 (2)	0.1243 (17)	0.506 (3)	0.064 (8)*
H5	0.2010 (18)	0.1621 (15)	0.843 (2)	0.047 (7)*
H22	0.3789 (19)	0.0424 (18)	0.477 (3)	0.047 (7)*
H24	−0.028 (2)	−0.0478 (17)	0.788 (3)	0.061 (8)*
H15	0.506 (2)	0.0290 (18)	0.822 (3)	0.064 (9)*
H27	0.065 (2)	−0.1831 (17)	0.755 (3)	0.061 (8)*
H16	0.544 (2)	0.0681 (17)	0.715 (3)	0.059 (8)*
H12	−0.031 (2)	0.3322 (17)	1.149 (3)	0.059 (8)*
H9	0.5112 (18)	−0.2937 (15)	0.477 (2)	0.045 (6)*
H25	0.004 (2)	−0.0024 (19)	0.673 (3)	0.072 (10)*
H18	0.387 (2)	0.1897 (17)	0.826 (3)	0.059 (8)*
H13	−0.0356 (19)	0.2742 (16)	1.023 (3)	0.046 (7)*
H28	0.123 (2)	−0.1555 (17)	0.655 (3)	0.061 (8)*
H19	0.341 (2)	0.2181 (19)	0.694 (3)	0.070 (9)*
H14	0.038 (2)	0.3483 (18)	1.049 (3)	0.060 (8)*
H31	0.130 (2)	−0.021 (2)	1.020 (3)	0.078 (10)*

H29	0.183 (2)	−0.1915 (19)	0.785 (3)	0.076 (9)*
H32	0.190 (2)	−0.1022 (19)	1.005 (3)	0.066 (10)*
H20	0.455 (2)	0.2127 (17)	0.728 (3)	0.062 (8)*
H17	0.500 (2)	−0.0226 (18)	0.701 (3)	0.059 (8)*
H10	0.4198 (19)	−0.3535 (16)	0.469 (3)	0.051 (7)*
H11	0.480 (2)	−0.3626 (17)	0.361 (3)	0.062 (8)*
H26	0.010 (2)	0.047 (2)	0.799 (3)	0.066 (9)*
H23	0.327 (2)	0.125 (2)	0.477 (3)	0.073 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02676 (14)	0.03814 (15)	0.02594 (14)	0.00280 (10)	0.00507 (11)	−0.00110 (9)
P1	0.0296 (3)	0.0426 (3)	0.0310 (3)	0.0011 (2)	0.0065 (2)	0.0020 (2)
P2	0.0300 (3)	0.0403 (3)	0.0314 (3)	−0.0006 (2)	0.0058 (2)	0.0005 (2)
S1	0.0313 (3)	0.0491 (3)	0.0280 (3)	0.0076 (2)	0.0023 (2)	−0.0026 (2)
S2	0.0304 (3)	0.0442 (3)	0.0280 (2)	0.0055 (2)	0.0028 (2)	−0.0019 (2)
C1	0.0274 (9)	0.0401 (10)	0.0288 (10)	−0.0031 (8)	0.0045 (8)	−0.0007 (8)
C2	0.0355 (11)	0.0437 (11)	0.0241 (9)	0.0010 (8)	0.0048 (9)	−0.0020 (8)
C3	0.0340 (10)	0.0375 (10)	0.0287 (10)	−0.0003 (8)	0.0046 (9)	0.0015 (8)
C4	0.0340 (10)	0.0319 (9)	0.0305 (10)	−0.0039 (8)	0.0079 (9)	−0.0022 (7)
C5	0.0357 (11)	0.0401 (10)	0.0243 (10)	−0.0054 (8)	0.0055 (9)	−0.0017 (8)
C6	0.0303 (10)	0.0370 (10)	0.0273 (10)	−0.0026 (8)	0.0033 (8)	0.0034 (8)
C7	0.0602 (16)	0.0377 (11)	0.0441 (13)	0.0093 (11)	0.0102 (13)	−0.0008 (10)
C8	0.0311 (10)	0.0364 (10)	0.0278 (10)	−0.0005 (8)	0.0057 (8)	−0.0015 (8)
C9	0.0369 (11)	0.0408 (11)	0.0277 (10)	0.0021 (8)	0.0076 (9)	0.0013 (8)
C10	0.0374 (11)	0.0354 (10)	0.0326 (11)	0.0038 (8)	0.0060 (9)	0.0022 (8)
C11	0.0315 (10)	0.0344 (9)	0.0342 (11)	−0.0038 (8)	0.0090 (9)	−0.0078 (8)
C12	0.0364 (11)	0.0413 (11)	0.0257 (10)	−0.0048 (8)	0.0066 (9)	−0.0030 (8)
C13	0.0329 (10)	0.0361 (10)	0.0286 (10)	−0.0010 (8)	0.0021 (9)	0.0011 (8)
C14	0.0482 (14)	0.0390 (12)	0.0538 (15)	0.0080 (10)	0.0136 (12)	−0.0038 (11)
C15	0.0336 (11)	0.0498 (13)	0.0508 (15)	0.0038 (10)	0.0144 (11)	0.0081 (11)
C16	0.0448 (14)	0.0432 (12)	0.0521 (15)	0.0023 (10)	0.0106 (12)	0.0040 (11)
C17	0.0517 (16)	0.0705 (18)	0.0376 (13)	−0.0125 (14)	0.0124 (12)	0.0036 (12)
C18	0.0362 (12)	0.0544 (14)	0.0597 (17)	0.0063 (10)	0.0161 (12)	0.0100 (12)
C19	0.0460 (14)	0.0430 (12)	0.0597 (17)	−0.0040 (11)	0.0148 (13)	−0.0049 (11)
C20	0.0605 (18)	0.075 (2)	0.0420 (14)	−0.0214 (16)	0.0091 (14)	0.0093 (13)
O1	0.0503 (9)	0.0424 (8)	0.0319 (8)	0.0087 (7)	0.0061 (7)	−0.0055 (6)
O2	0.0388 (8)	0.0495 (8)	0.0353 (8)	0.0066 (7)	0.0091 (7)	−0.0084 (6)

Geometric parameters (Å, º)

Ni1—P1	2.2121 (7)	C9—H5	0.96 (2)
Ni1—P2	2.2224 (7)	C10—C11	1.387 (3)
Ni1—S2	2.2261 (9)	C10—H6	0.96 (3)
Ni1—S1	2.2288 (9)	C11—O2	1.378 (2)
P1—C15	1.816 (2)	C11—C12	1.393 (3)
P1—C16	1.819 (3)	C12—C13	1.387 (3)

P1—C17	1.825 (3)	C12—H7	0.96 (2)
P2—C19	1.809 (3)	C13—H8	0.98 (2)
P2—C18	1.812 (3)	C14—O2	1.425 (3)
P2—C20	1.825 (3)	C14—H12	0.97 (3)
S1—C1	1.770 (2)	C14—H13	0.99 (3)
S2—C8	1.7729 (19)	C14—H14	0.97 (3)
C1—C2	1.395 (3)	C15—H15	0.86 (3)
C1—C6	1.412 (3)	C15—H16	0.94 (3)
C2—C3	1.397 (3)	C15—H17	1.01 (3)
C2—H1	0.94 (3)	C16—H18	0.95 (3)
C3—C4	1.388 (3)	C16—H19	0.91 (3)
C3—H2	0.95 (2)	C16—H20	1.00 (3)
C4—O1	1.377 (2)	C17—H21	1.02 (3)
C4—C5	1.391 (3)	C17—H22	0.93 (3)
C5—C6	1.381 (3)	C17—H23	0.93 (3)
C5—H3	0.91 (3)	C18—H24	0.98 (3)
C6—H4	0.93 (2)	C18—H25	0.92 (4)
C7—O1	1.428 (3)	C18—H26	0.99 (3)
C7—H9	1.04 (3)	C19—H27	0.96 (3)
C7—H10	0.94 (3)	C19—H28	0.95 (3)
C7—H11	0.97 (3)	C19—H29	1.01 (3)
C8—C9	1.386 (3)	C20—H30	0.97 (3)
C8—C13	1.412 (3)	C20—H31	0.99 (4)
C9—C10	1.400 (3)	C20—H32	0.86 (3)

P1—Ni1—P2	178.07 (2)	O2—C11—C10	124.26 (19)
P1—Ni1—S2	87.01 (3)	O2—C11—C12	115.96 (18)
P2—Ni1—S2	93.17 (3)	C10—C11—C12	119.78 (18)
P1—Ni1—S1	92.17 (3)	C13—C12—C11	120.36 (18)
P2—Ni1—S1	87.67 (3)	C13—C12—H7	117.0 (14)
S2—Ni1—S1	178.85 (2)	C11—C12—H7	122.6 (14)
C15—P1—C16	104.76 (13)	C12—C13—C8	120.63 (19)
C15—P1—C17	101.37 (14)	C12—C13—H8	120.1 (13)
C16—P1—C17	101.21 (14)	C8—C13—H8	119.2 (13)
C15—P1—Ni1	114.04 (8)	O2—C14—H12	104.1 (17)
C16—P1—Ni1	111.87 (8)	O2—C14—H13	110.3 (14)
C17—P1—Ni1	121.57 (10)	H12—C14—H13	113 (2)
C19—P2—C18	103.82 (14)	O2—C14—H14	111.5 (18)
C19—P2—C20	101.25 (15)	H12—C14—H14	112 (2)
C18—P2—C20	101.07 (16)	H13—C14—H14	106 (2)
C19—P2—Ni1	111.32 (9)	P1—C15—H15	110 (2)
C18—P2—Ni1	115.54 (9)	P1—C15—H16	108.0 (17)
C20—P2—Ni1	121.54 (10)	H15—C15—H16	112 (3)
C1—S1—Ni1	102.20 (7)	P1—C15—H17	109.9 (17)
C8—S2—Ni1	101.87 (7)	H15—C15—H17	110 (3)
C2—C1—C6	117.29 (18)	H16—C15—H17	107 (2)
C2—C1—S1	122.53 (15)	P1—C16—H18	108.8 (17)
C6—C1—S1	120.16 (16)	P1—C16—H19	107 (2)

C1—C2—C3	122.05 (19)	H18—C16—H19	109 (3)
C1—C2—H1	119.9 (15)	P1—C16—H20	111.0 (16)
C3—C2—H1	118.0 (15)	H18—C16—H20	109 (2)
C4—C3—C2	119.3 (2)	H19—C16—H20	111 (2)
C4—C3—H2	121.8 (14)	P1—C17—H21	109.3 (17)
C2—C3—H2	118.8 (14)	P1—C17—H22	108.9 (17)
O1—C4—C3	124.43 (19)	H21—C17—H22	110 (2)
O1—C4—C5	115.97 (17)	P1—C17—H23	109.3 (19)
C3—C4—C5	119.60 (18)	H21—C17—H23	112 (3)
C6—C5—C4	120.87 (18)	H22—C17—H23	108 (3)
C6—C5—H3	118.9 (15)	P2—C18—H24	106.1 (17)
C4—C5—H3	120.2 (15)	P2—C18—H25	109 (2)
C5—C6—C1	120.84 (19)	H24—C18—H25	111 (3)
C5—C6—H4	121.8 (14)	P2—C18—H26	113.3 (18)
C1—C6—H4	117.3 (14)	H24—C18—H26	108 (2)
O1—C7—H9	112.5 (14)	H25—C18—H26	109 (3)
O1—C7—H10	110.2 (16)	P2—C19—H27	111.2 (16)
H9—C7—H10	107 (2)	P2—C19—H28	109.9 (17)
O1—C7—H11	105.1 (17)	H27—C19—H28	106 (3)
H9—C7—H11	111 (2)	P2—C19—H29	108.5 (18)
H10—C7—H11	111 (2)	H27—C19—H29	112 (2)
C9—C8—C13	118.05 (18)	H28—C19—H29	109 (2)
C9—C8—S2	122.12 (15)	P2—C20—H30	108.9 (17)
C13—C8—S2	119.82 (16)	P2—C20—H31	110 (2)
C8—C9—C10	121.55 (19)	H30—C20—H31	107 (2)
C8—C9—H5	118.6 (15)	P2—C20—H32	107 (2)
C10—C9—H5	119.8 (15)	H30—C20—H32	107 (3)
C11—C10—C9	119.6 (2)	H31—C20—H32	117 (3)
C11—C10—H6	122.6 (14)	C4—O1—C7	117.19 (17)
C9—C10—H6	117.8 (14)	C11—O2—C14	116.84 (17)