

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

cis-Dichloridobis(dimethoxyphenylphosphine)palladium(II)

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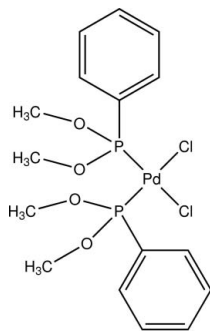
Received 16 February 2010; accepted 19 February 2010

 Key indicators: single-crystal X-ray study; $T = 125$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å;
 R factor = 0.023; wR factor = 0.050; data-to-parameter ratio = 15.2.

The title compound, $[\text{PdCl}_2(\text{C}_8\text{H}_{11}\text{O}_2\text{P})_2]$, has a comparable structure to those of related palladium dichloride complexes containing trimethyl phosphinite and methyl diphenyl phosphinite. The Pd atom is located on a crystallographic twofold rotation axis: thus, there is just one half-molecule in the asymmetric unit. The structure is isomorphous with the platinum analogue *cis*- $[\text{PtCl}_2\{\text{P}(\text{OMe})_2\text{Ph}\}_2]$.

Related literature

For related structures, see: Slawin *et al.* (2009, 2007); Powell & Jacobson (1980). For preparation of the title compound, see: Jenkins & Shaw (1966).



Experimental

Crystal data

 $[\text{PdCl}_2(\text{C}_8\text{H}_{11}\text{O}_2\text{P})_2]$
 $M_r = 517.60$

 Monoclinic, $C2/c$
 $a = 10.876$ (7) Å
 $b = 9.174$ (5) Å
 $c = 20.722$ (13) Å
 $\beta = 102.196$ (7)°
 $V = 2021$ (2) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.36$ mm⁻¹
 $T = 125$ K
 $0.16 \times 0.15 \times 0.09$ mm

Data collection

 Rigaku Mercury CCD
 diffractometer
 Absorption correction: multi-scan
 (*ABSCOR*; Higashi, 1995)
 $T_{\min} = 0.812$, $T_{\max} = 0.888$

 8406 measured reflections
 1775 independent reflections
 1653 reflections with $F^2 > 2\sigma(F^2)$
 $R_{\text{int}} = 0.032$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.050$
 $S = 1.09$
 1775 reflections

 117 parameters
 All H-atom parameters refined
 $\Delta\rho_{\max} = 1.15$ e Å⁻³
 $\Delta\rho_{\min} = -0.32$ e Å⁻³
Table 1

Selected geometric parameters (Å, °).

Pd1—Cl1	2.3515 (16)	Pd1—P1	2.2300 (16)
Cl1—Pd1—Cl1 ⁱ	91.49 (2)	Cl1—Pd1—P1 ⁱ	83.85 (2)
Cl1—Pd1—P1	174.86 (2)	P1—Pd1—P1 ⁱ	100.88 (2)

 Symmetry code: (i) $-x, y, -z + \frac{3}{2}$.

Data collection: *SCXmini* (Rigaku/MS, 2006); cell refinement: *PROCESS-AUTO* (Rigaku, 1998); data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku/MS, 2006); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5195).

References

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supporting information

Acta Cryst. (2010). E66, m321 [doi:10.1107/S1600536810006471]

cis-Dichloridobis(dimethoxyphenylphosphine)palladium(II)

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S1. Experimental

Based on the method of Jenkins & Shaw (1966). To (0.3 g (0.93 mmol) of potassium tetrachloropalladate was dissolved in 10 ml ethanol and 0.147 ml (1.86 mmol) of P(OMe)₂Ph was added. The solution was stirred at room temperature for 30 min s before being filtered and then precipitated by slow addition of hexane to give a white crystalline solid. Crystals were grown for X-ray crystallography via slow diffusion of hexane into a solution of the product in dichloromethane [*cis*-PdCl₂(P(OMe)₂Ph)₂] (0.40 mmol, ca. 68 %). ³¹P-{¹H} NMR: δ 125.8 ppm.

S2. Refinement

All H atoms were included in calculated positions (C—H distances are 0.96 Å for methyl H atoms, 0.93 Å for aromatic H atoms) and were refined as riding atoms with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ or $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C}_{\text{methyl}})$. The highest peak in the difference map is 1.23 Å from atom Pd1.

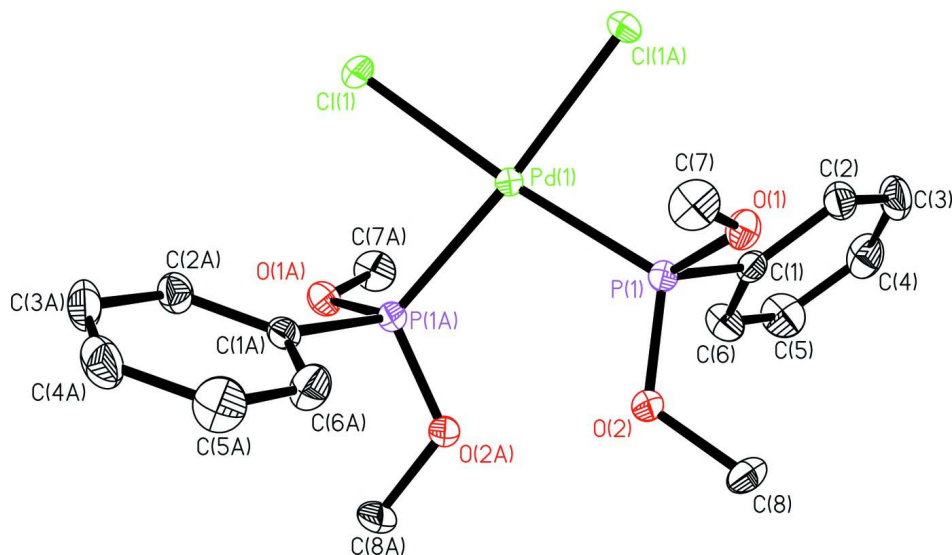


Figure 1

The structure of the title compound with displacement ellipsoids drawn at the 50% probability level, hydrogen atoms omitted for clarity. Symmetry operator for generating equivalent atoms (A): $-x, y, 1.5-z$.

cis-Dichloridobis(dimethoxyphenylphosphine)palladium(II)

Crystal data

[PdCl₂(C₈H₁₁O₂P)₂]
 $M_r = 517.60$

Monoclinic, *C2/c*
 Hall symbol: $-C\ 2yc$

$a = 10.876$ (7) Å
 $b = 9.174$ (5) Å
 $c = 20.722$ (13) Å
 $\beta = 102.196$ (7)°
 $V = 2021$ (2) Å³
 $Z = 4$
 $F(000) = 1040.00$
 $D_x = 1.701$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
 Cell parameters from 2591 reflections
 $\theta = 2.9$ – 25.4 °
 $\mu = 1.36$ mm⁻¹
 $T = 125$ K
 Prism, colorless
 $0.16 \times 0.15 \times 0.09$ mm

Data collection

Rigaku Mercury CCD
 diffractometer
 Detector resolution: 0.83 pixels mm⁻¹
 ω scans
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.812$, $T_{\max} = 0.888$
 8406 measured reflections

1775 independent reflections
 1653 reflections with $F^2 > 2\sigma(F^2)$
 $R_{\text{int}} = 0.032$
 $\theta_{\max} = 25.0$ °
 $h = -13 \rightarrow 13$
 $k = -11 \rightarrow 11$
 $l = -24 \rightarrow 24$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.050$
 $S = 1.09$
 1775 reflections
 117 parameters

All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0162P)^2 + 4.843P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.15$ e Å⁻³
 $\Delta\rho_{\min} = -0.32$ e Å⁻³

Special details

Geometry. ENTER SPECIAL DETAILS OF THE MOLECULAR GEOMETRY

Refinement. Refinement was performed using all reflections. The weighted R -factor (wR) and goodness of fit (S) are based on F^2 . R -factor (gt) are based on F . The threshold expression of $F^2 > 2.0 \sigma(F^2)$ is used only for calculating R -factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pd1	0.0000	0.09671 (3)	0.7500	0.01397 (8)
Cl1	-0.09211 (6)	-0.08216 (6)	0.67365 (3)	0.02090 (14)
P1	0.10038 (6)	0.25152 (7)	0.82618 (3)	0.01521 (14)
O1	0.24303 (15)	0.21038 (18)	0.85487 (8)	0.0189 (3)
O2	0.10272 (16)	0.41579 (17)	0.80284 (8)	0.0199 (3)
C1	0.0398 (2)	0.2542 (2)	0.90033 (12)	0.0172 (5)
C2	0.0988 (2)	0.1823 (2)	0.95758 (12)	0.0220 (5)
C3	0.0459 (2)	0.1856 (3)	1.01266 (13)	0.0285 (6)
C4	-0.0648 (2)	0.2601 (3)	1.01080 (13)	0.0302 (6)
C5	-0.1248 (2)	0.3298 (3)	0.95385 (14)	0.0328 (6)
C6	-0.0733 (2)	0.3264 (2)	0.89844 (13)	0.0259 (5)
C7	0.3242 (2)	0.1770 (3)	0.80984 (14)	0.0278 (6)
C8	0.1786 (2)	0.5209 (2)	0.84682 (13)	0.0274 (6)
H1	0.2993	0.0858	0.7883	0.033*
H2	0.1736	0.1321	0.9588	0.026*
H3	0.0851	0.1376	1.0510	0.034*

H4	-0.0994	0.2634	1.0482	0.036*
H5	-0.1999	0.3791	0.9528	0.039*
H6	-0.1143	0.3724	0.8599	0.031*
H7	0.4097	0.1704	0.8340	0.033*
H8	0.3175	0.2528	0.7773	0.033*
H9	0.1697	0.5038	0.8913	0.033*
H10	0.1508	0.6179	0.8337	0.033*
H11	0.2654	0.5103	0.8444	0.033*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.01670 (15)	0.01215 (14)	0.01241 (14)	0.0000	0.00162 (10)	0.0000
Cl1	0.0256 (3)	0.0154 (3)	0.0190 (3)	-0.0012 (2)	-0.0014 (2)	-0.0041 (2)
P1	0.0170 (3)	0.0143 (3)	0.0137 (3)	-0.0012 (2)	0.0020 (2)	-0.0000 (2)
O1	0.0154 (8)	0.0217 (8)	0.0194 (8)	-0.0021 (7)	0.0029 (6)	-0.0027 (7)
O2	0.0266 (9)	0.0151 (8)	0.0163 (8)	-0.0040 (6)	0.0002 (7)	0.0006 (6)
C1	0.0198 (12)	0.0159 (12)	0.0159 (11)	-0.0031 (9)	0.0037 (9)	-0.0022 (9)
C2	0.0212 (13)	0.0248 (14)	0.0188 (12)	0.0032 (10)	0.0017 (10)	-0.0012 (10)
C3	0.0336 (15)	0.0356 (16)	0.0153 (13)	-0.0020 (12)	0.0025 (11)	0.0020 (11)
C4	0.0363 (16)	0.0370 (16)	0.0203 (14)	-0.0054 (12)	0.0125 (12)	-0.0051 (12)
C5	0.0279 (15)	0.0383 (17)	0.0352 (16)	0.0073 (12)	0.0132 (12)	0.0003 (13)
C6	0.0265 (14)	0.0271 (14)	0.0245 (14)	0.0048 (11)	0.0062 (11)	0.0048 (11)
C7	0.0239 (14)	0.0317 (15)	0.0308 (15)	-0.0024 (11)	0.0127 (11)	-0.0017 (12)
C8	0.0387 (16)	0.0172 (13)	0.0249 (14)	-0.0079 (11)	0.0034 (12)	-0.0043 (11)

Geometric parameters (Å, °)

Pd1—Cl1	2.3515 (16)	C4—C5	1.379 (3)
Pd1—Cl1 ⁱ	2.3515 (16)	C5—C6	1.380 (4)
Pd1—P1	2.2300 (16)	C2—H2	0.930
Pd1—P1 ⁱ	2.2300 (16)	C3—H3	0.930
P1—O1	1.5853 (16)	C4—H4	0.930
P1—O2	1.5846 (17)	C5—H5	0.930
P1—C1	1.795 (2)	C6—H6	0.930
O1—C7	1.446 (3)	C7—H1	0.960
O2—C8	1.458 (2)	C7—H7	0.960
C1—C2	1.390 (3)	C7—H8	0.960
C1—C6	1.391 (3)	C8—H9	0.960
C2—C3	1.383 (4)	C8—H10	0.960
C3—C4	1.378 (4)	C8—H11	0.960
O1...C3 ⁱⁱ	3.324 (2)	H3...H9 ⁱⁱ	2.979
O2...C7 ⁱⁱⁱ	3.551 (3)	H3...H11 ⁱⁱ	2.774
C3...O1 ⁱⁱ	3.324 (2)	H4...Cl1 ^{xiv}	3.073
C3...C3 ^{iv}	3.557 (3)	H4...C5 ^{xii}	3.112
C7...O2 ^v	3.551 (3)	H4...C8 ^{xiii}	3.191
C7...C8 ^v	3.542 (3)	H4...H4 ^{xii}	3.467

C8...C7 ⁱⁱⁱ	3.542 (3)	H4...H5 ^{xii}	2.542
C11...H3 ^{vi}	3.536	H4...H7 ⁱⁱ	2.908
C11...H4 ^{vi}	3.073	H4...H9 ^{xiii}	2.669
C11...H5 ^{vii}	3.099	H4...H10 ^{xiii}	2.840
C11...H6 ^{vii}	3.149	H5...C11 ^{xv}	3.099
C11...H7 ^v	3.041	H5...C2 ^{xi}	3.554
C11...H8 ^{viii}	2.961	H5...C4 ^{xii}	3.093
C11...H8 ^v	3.311	H5...C5 ^{xii}	3.552
C11...H10 ^{ix}	2.822	H5...H2 ^{xi}	2.715
O1...H3 ⁱⁱ	2.775	H5...H4 ^{xii}	2.542
O1...H6 ^x	3.459	H5...H5 ^{xii}	3.396
O2...H1 ⁱⁱⁱ	2.826	H5...H9 ^{xiii}	3.354
O2...H7 ^{xi}	3.294	H5...H10 ^{viii}	3.575
C2...H2 ⁱⁱ	3.196	H6...C11 ^{xv}	3.149
C2...H3 ^{iv}	3.535	H6...O1 ^{xi}	3.459
C2...H5 ^x	3.554	H6...C7 ^{xi}	3.007
C3...H2 ⁱⁱ	3.419	H6...H1 ^{xi}	2.515
C3...H3 ^{iv}	3.431	H6...H7 ^{xi}	2.809
C3...H7 ⁱⁱ	3.381	H6...H10 ^{viii}	3.418
C4...H5 ^{xii}	3.093	H6...H11 ^{viii}	3.560
C4...H7 ⁱⁱ	3.363	H7...C11 ⁱⁱⁱ	3.041
C4...H9 ^{xiii}	3.329	H7...O2 ^x	3.294
C5...H2 ^{xi}	3.551	H7...C3 ⁱⁱ	3.381
C5...H4 ^{xii}	3.112	H7...C4 ⁱⁱ	3.363
C5...H5 ^{xii}	3.552	H7...C6 ^x	3.417
C6...H1 ^{xi}	3.384	H7...C8 ^x	3.190
C6...H7 ^{xi}	3.417	H7...H3 ⁱⁱ	2.955
C6...H10 ^{viii}	3.572	H7...H4 ⁱⁱ	2.908
C6...H11 ^{viii}	3.451	H7...H6 ^x	2.809
C7...H3 ⁱⁱ	3.313	H7...H9 ^x	3.213
C7...H6 ^x	3.007	H7...H10 ^x	2.668
C7...H10 ^x	3.523	H7...H10 ^v	3.431
C7...H10 ^v	3.092	H8...C11 ^{xvi}	2.961
C7...H11 ^v	3.491	H8...C11 ⁱⁱⁱ	3.311
C8...H1 ⁱⁱⁱ	2.922	H8...C8 ^v	3.345
C8...H3 ⁱⁱ	3.301	H8...H1 ⁱⁱⁱ	3.474
C8...H4 ^{xiii}	3.191	H8...H10 ^v	2.698
C8...H7 ^{xi}	3.190	H8...H11 ^v	3.341
C8...H8 ⁱⁱⁱ	3.345	H9...C4 ^{xiii}	3.329
H1...O2 ^v	2.826	H9...H2 ⁱⁱ	3.445
H1...C6 ^x	3.384	H9...H3 ⁱⁱ	2.979
H1...C8 ^v	2.922	H9...H4 ^{xiii}	2.669
H1...H6 ^x	2.515	H9...H5 ^{xiii}	3.354
H1...H8 ^v	3.474	H9...H7 ^{xi}	3.213
H1...H10 ^v	2.712	H10...C11 ^{xvii}	2.822
H1...H11 ^v	2.779	H10...C6 ^{xvi}	3.572
H2...C2 ⁱⁱ	3.196	H10...C7 ^{xi}	3.523
H2...C3 ⁱⁱ	3.419	H10...C7 ⁱⁱⁱ	3.092

H2...C5 ^x	3.551	H10...H1 ⁱⁱⁱ	2.712
H2...H2 ⁱⁱ	3.026	H10...H4 ^{xiii}	2.840
H2...H3 ⁱⁱ	3.409	H10...H5 ^{xvi}	3.575
H2...H5 ^x	2.715	H10...H6 ^{xvi}	3.418
H2...H9 ⁱⁱ	3.445	H10...H7 ^{xi}	2.668
H3...Cl1 ^{xiv}	3.536	H10...H7 ⁱⁱⁱ	3.431
H3...O1 ⁱⁱ	2.775	H10...H8 ⁱⁱⁱ	2.698
H3...C2 ^{iv}	3.535	H11...C6 ^{xvi}	3.451
H3...C3 ^{iv}	3.431	H11...C7 ⁱⁱⁱ	3.491
H3...C7 ⁱⁱ	3.313	H11...H1 ⁱⁱⁱ	2.779
H3...C8 ⁱⁱ	3.301	H11...H3 ⁱⁱ	2.774
H3...H2 ⁱⁱ	3.409	H11...H6 ^{xvi}	3.560
H3...H3 ^{iv}	3.552	H11...H8 ⁱⁱⁱ	3.341
H3...H7 ⁱⁱ	2.955		
Cl1—Pd1—Cl1 ⁱ	91.49 (2)	C1—C2—H2	120.1
Cl1—Pd1—P1	174.86 (2)	C3—C2—H2	120.1
Cl1—Pd1—P1 ⁱ	83.85 (2)	C2—C3—H3	120.0
Cl1 ⁱ —Pd1—P1	83.85 (2)	C4—C3—H3	120.0
Cl1 ⁱ —Pd1—P1 ⁱ	174.86 (2)	C3—C4—H4	119.8
P1—Pd1—P1 ⁱ	100.88 (2)	C5—C4—H4	119.8
Pd1—P1—O1	114.07 (6)	C4—C5—H5	120.0
Pd1—P1—O2	115.26 (6)	C6—C5—H5	120.0
Pd1—P1—C1	113.66 (7)	C1—C6—H6	120.0
O1—P1—O2	105.27 (9)	C5—C6—H6	120.0
O1—P1—C1	100.91 (10)	O1—C7—H1	109.5
O2—P1—C1	106.30 (10)	O1—C7—H7	109.5
P1—O1—C7	119.39 (14)	O1—C7—H8	109.5
P1—O2—C8	118.82 (14)	H1—C7—H7	109.5
P1—C1—C2	122.50 (19)	H1—C7—H8	109.5
P1—C1—C6	117.70 (18)	H7—C7—H8	109.5
C2—C1—C6	119.7 (2)	O2—C8—H9	109.5
C1—C2—C3	119.8 (2)	O2—C8—H10	109.5
C2—C3—C4	120.1 (2)	O2—C8—H11	109.5
C3—C4—C5	120.4 (2)	H9—C8—H10	109.5
C4—C5—C6	120.0 (2)	H9—C8—H11	109.5
C1—C6—C5	119.9 (2)	H10—C8—H11	109.5
Cl1—Pd1—P1 ⁱ —O1 ⁱ	47.02 (7)	O1—P1—O2—C8	-46.3 (2)
Cl1—Pd1—P1 ⁱ —O2 ⁱ	169.01 (8)	O2—P1—O1—C7	-77.89 (18)
Cl1—Pd1—P1 ⁱ —C1 ⁱ	-67.93 (8)	O1—P1—C1—C2	-21.0 (2)
Cl1 ⁱ —Pd1—P1—O1	47.02 (7)	O1—P1—C1—C6	161.81 (18)
Cl1 ⁱ —Pd1—P1—O2	169.01 (8)	C1—P1—O1—C7	171.69 (17)
Cl1 ⁱ —Pd1—P1—C1	-67.93 (8)	O2—P1—C1—C2	-130.59 (19)
P1—Pd1—P1 ⁱ —O1 ⁱ	-135.01 (7)	O2—P1—C1—C6	52.2 (2)
P1—Pd1—P1 ⁱ —O2 ⁱ	-13.03 (8)	C1—P1—O2—C8	60.2 (2)
P1—Pd1—P1 ⁱ —C1 ⁱ	110.03 (8)	P1—C1—C2—C3	-178.50 (19)
P1 ⁱ —Pd1—P1—O1	-135.01 (7)	P1—C1—C6—C5	179.1 (2)

P1 ⁱ —Pd1—P1—O2	-13.03 (8)	C2—C1—C6—C5	1.8 (3)
P1 ⁱ —Pd1—P1—C1	110.03 (8)	C6—C1—C2—C3	-1.3 (3)
Pd1—P1—O1—C7	49.44 (18)	C1—C2—C3—C4	-0.1 (3)
Pd1—P1—O2—C8	-172.89 (16)	C2—C3—C4—C5	1.0 (4)
Pd1—P1—C1—C2	101.57 (19)	C3—C4—C5—C6	-0.6 (4)
Pd1—P1—C1—C6	-75.6 (2)	C4—C5—C6—C1	-0.8 (4)

Symmetry codes: (i) $-x, y, -z+3/2$; (ii) $-x+1/2, -y+1/2, -z+2$; (iii) $-x+1/2, y+1/2, -z+3/2$; (iv) $-x, -y, -z+2$; (v) $-x+1/2, y-1/2, -z+3/2$; (vi) $x, -y, z-1/2$; (vii) $-x-1/2, y-1/2, -z+3/2$; (viii) $x-1/2, y-1/2, z$; (ix) $-x, y-1, -z+3/2$; (x) $x+1/2, y-1/2, z$; (xi) $x-1/2, y+1/2, z$; (xii) $-x-1/2, -y+1/2, -z+2$; (xiii) $-x, -y+1, -z+2$; (xiv) $x, -y, z+1/2$; (xv) $-x-1/2, y+1/2, -z+3/2$; (xvi) $x+1/2, y+1/2, z$; (xvii) $-x, y+1, -z+3/2$.