Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

catena-Poly[[diaquacobalt(II)]bis[μ-2-(4-carboxylatophenyl)-4,4,5,5-tetramethyl-4,5-dihydro-1*H*-imidazol-1-oxyl 3-oxide]]

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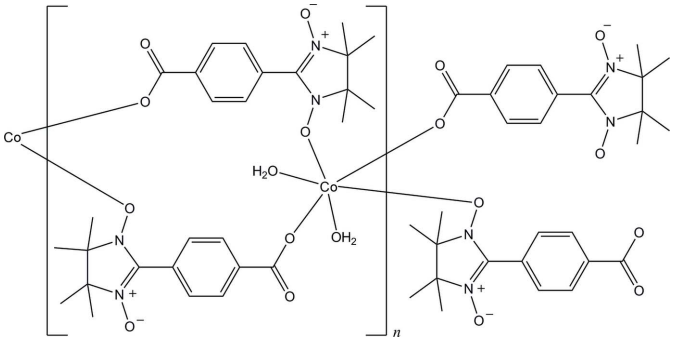
Received 28 February 2011; accepted 16 March 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.070; wR factor = 0.169; data-to-parameter ratio = 16.5.

In the title compound, $[Co(C_{14}H_{16}N_2O_4)_2(H_2O)_2]_n$, the Co^{II} atom, lying on an inversion center, is coordinated by six O atoms in a distorted octahedral geometry. The Co^{II} atoms are bridged by the nitronyl nitroxide ligands into a tape-like structure along the b axis. The tapes are further connected by O—H···O hydrogen bonds, forming a layer parallel to the bc plane.

Related literature

For related structures, see: Caneschi *et al.* (1993); Luneau *et al.* (1998). For the synthesis of $[Co(C_5H_9O_2)_2(H_2O)_2]$, see: Mehrotra & Bohra (1983). For the synthesis of 2-(4-carboxyphenyl)-4,4,5,5-tetramethyl-4,5-dihydro-1*H*-imidazol-1-oxyl-3-oxide, see: Schiødt *et al.* (1996).



Experimental

Crystal data

$[Co(C_{14}H_{16}N_2O_4)_2(H_2O)_2]$
M_r = 647.54
Monoclinic, $P2_1/c$
a = 13.548 (3) Å
b = 9.2054 (18) Å
c = 12.549 (3) Å
β = 115.17 (3)°
V = 1416.5 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.67 mm^{-1}
T = 293 K
0.43 × 0.42 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.240, T_{max} = 0.428
14419 measured reflections
3241 independent reflections
2050 reflections with $I > 2\sigma(I)$
R_{int} = 0.121

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.169
S = 1.04
3241 reflections
196 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H1*O5*···O2^{i}	0.87	2.31	2.736 (4)	111
O5—H2*O5*···O2^{ii}	0.85	2.40	2.886 (4)	117

Symmetry codes: (i) $-x+1, -y-1, -z+2$; (ii) $x, -y-\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2683).

References

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supporting information

Acta Cryst. (2011). E**67**, m488 [doi:10.1107/S1600536811009925]

catena-Poly[[diaquacobalt(II)]bis[μ-2-(4-carboxylatophenyl)-4,4,5,5-tetramethyl-4,5-dihydro-1*H*-imidazol-1-oxyl 3-oxide]]

Rong Rong, Ge Gao and Lili Zhu

S1. Comment

The combination of paramagnetic metal ions and organic nitronyl nitroxides has attracted much more attention in the last decades, because of their intriguing structural, magnetic, and spectral properties (Caneschi *et al.*, 1993; Luneau *et al.*, 1998).

We herein report the crystal structure of a new complex revealed two ladder-like one-dimensional chains of repeating $Co(NITpBA)_2(H_2O)_2$ units. As shown in Fig. 1, the central Co ion is located in a distorted octahedral environment. It is bonded to two oxygen atoms from carboxylic acid group, two oxygen atoms from nitroxide group and two oxygen atoms from two water atoms. Each radical coordinates the next Co(II) ion in the opposite fashion thus forming chains. There are intermolecular hydrogen bonds between the coordinated water molecule and the non-coordinated carboxylic O2 atom (Table 1 and Fig. 2).

S2. Experimental

All reagents and chemicals were purchased from commercial sources. The carboxylates, $Co(Me_3CCO_2)_2(H_2O)_2$, were prepared as described previously (Mehrotra & Bohra, 1983). The benzoic acid substituted nitronyl nitroxide radical NITpBAH) was synthesized according to the procedure previously described (Schiødt *et al.*, 1996). $Co(Me_3CCO_2)_2(H_2O)_2$ (0.0290 g, 0.1 mmol) was dissolved with prolonged stirring at room temperature in acetonitrile (15 mL). Then a 10 mL dichloromethane solution of NITpBAH(0.0608 g, 0.2 mmol) was added dropwise with stirring. Both solutions were mixed while stirring was continued for 1 h, dark blue crystals suitable for X-ray analysis were obtained by slow evaporation at room temperature over several days.

S3. Refinement

Positional parameters of all H atoms bound to C were calculated geometrically (C—H = 0.93 or 0.96 Å) and the H atoms were refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The H atoms of water molecile were located in a difference Fourier map and fixed at the positions with U_{iso}(H) = 1.5U_{eq}(O).

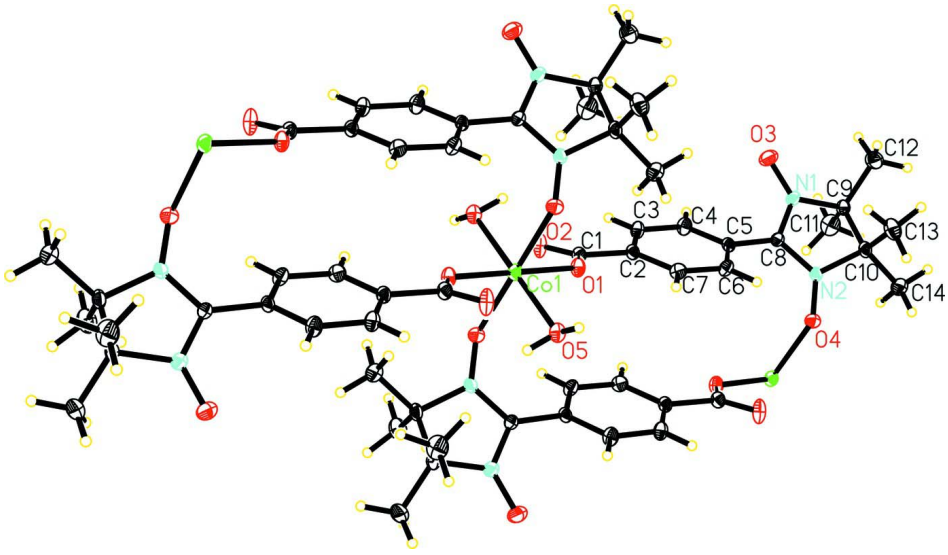

Figure 1

The molecular structure of the title compound, with the atom-numbering scheme and all hydrogen atoms. Displacement ellipsoids are drawn at the 30% probability level.

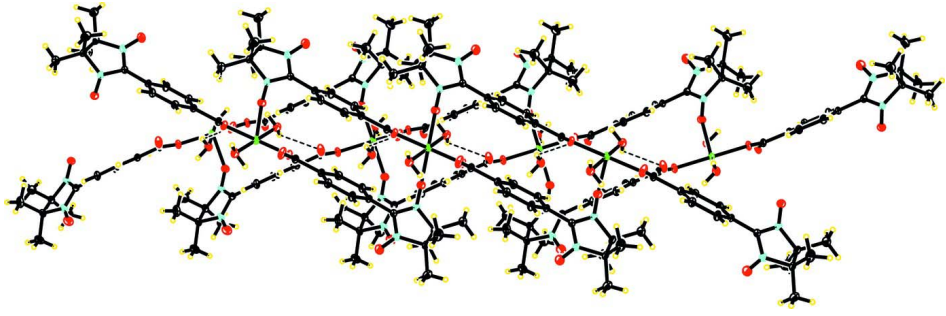

Figure 2

A packing view of the title compound. Hydrogen bonds are shown as dashed lines.

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$[Co(C_{14}H_{16}N_2O_4)_2(H_2O)_2]$
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Hall symbol: -P 2ybc
$a = 13.548\ (3)$ Å
$b = 9.2054\ (18)$ Å
$c = 12.549\ (3)$ Å
$\beta = 115.17\ (3)°$
$V = 1416.5\ (5)$ Å^3
$Z = 2$
$F(000) = 678$
$D_x = 1.518$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 14419 reflections
$\theta = 3.3–27.5°$
$\mu = 0.67$ mm^{-1}
$T = 293$ K
Prism, dark blue
0.43 × 0.42 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.192 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.240, T_{max} = 0.428
14419 measured reflections
3241 independent reflections
2050 reflections with $I > 2\sigma(I)$
R_{int} = 0.121
θ_{max} = 27.5°, θ_{min} = 3.3°
h = −17→17
k = −11→11
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.169
S = 1.04
3241 reflections
196 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
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2 + 1.8428P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 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}
O1	0.4450 (2)	−0.2949 (3)	0.9518 (2)	0.0274 (7)
O2	0.4582 (3)	−0.2604 (3)	0.7820 (3)	0.0364 (8)
O3	0.0826 (3)	0.3058 (4)	0.6289 (3)	0.0442 (9)
O4	0.3405 (2)	0.4447 (3)	0.9959 (2)	0.0279 (7)
O5	0.5531 (2)	−0.4483 (3)	1.1813 (2)	0.0297 (7)
H1O5	0.6060	−0.5105	1.2077	0.045*
H2O5	0.5186	−0.4578	1.2237	0.045*
N1	0.1342 (3)	0.3787 (4)	0.7227 (3)	0.0267 (8)
N2	0.2516 (3)	0.4405 (4)	0.8999 (3)	0.0233 (8)
C1	0.4323 (3)	−0.2209 (4)	0.8615 (4)	0.0246 (9)
C2	0.3798 (3)	−0.0740 (4)	0.8520 (4)	0.0234 (9)
C3	0.3428 (3)	0.0019 (4)	0.7470 (4)	0.0264 (9)
H3A	0.3513	−0.0380	0.6834	0.032*
C4	0.2933 (4)	0.1363 (4)	0.7352 (4)	0.0284 (10)
H4A	0.2687	0.1861	0.6640	0.034*
C5	0.2805 (3)	0.1961 (4)	0.8299 (4)	0.0229 (9)

C6	0.3184 (4)	0.1222 (4)	0.9369 (4)	0.0278 (10)
H6A	0.3115	0.1634	1.0011	0.033*
C7	0.3663 (3)	−0.0122 (4)	0.9470 (4)	0.0265 (9)
H7A	0.3900	−0.0626	1.0177	0.032*
C8	0.2253 (3)	0.3363 (4)	0.8166 (3)	0.0232 (9)
C9	0.1031 (4)	0.5322 (4)	0.7365 (4)	0.0271 (10)
C10	0.1582 (3)	0.5441 (4)	0.8726 (4)	0.0266 (10)
C11	0.1545 (4)	0.6293 (5)	0.6754 (4)	0.0451 (13)
H11A	0.1167	0.6166	0.5917	0.068*
H11B	0.2298	0.6035	0.7010	0.068*
H11C	0.1494	0.7290	0.6949	0.068*
C12	−0.0192 (4)	0.5491 (6)	0.6821 (4)	0.0458 (13)
H12A	−0.0472	0.5404	0.5981	0.069*
H12B	−0.0377	0.6428	0.7018	0.069*
H12C	−0.0506	0.4748	0.7118	0.069*
C13	0.0892 (4)	0.4805 (5)	0.9312 (5)	0.0414 (12)
H13A	0.1274	0.4910	1.0151	0.062*
H13B	0.0760	0.3794	0.9117	0.062*
H13C	0.0209	0.5312	0.9037	0.062*
C14	0.1972 (4)	0.6944 (5)	0.9213 (4)	0.0392 (12)
H14A	0.2299	0.6910	1.0058	0.059*
H14C	0.1363	0.7600	0.8944	0.059*
H14D	0.2500	0.7276	0.8946	0.059*
Co1	0.5000	−0.5000	1.0000	0.0250 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0352 (17)	0.0148 (15)	0.0341 (16)	0.0060 (12)	0.0165 (14)	0.0051 (12)
O2	0.058 (2)	0.0235 (17)	0.0439 (19)	0.0125 (15)	0.0372 (17)	0.0067 (14)
O3	0.041 (2)	0.035 (2)	0.0384 (19)	0.0023 (16)	−0.0002 (16)	−0.0109 (15)
O4	0.0256 (17)	0.0295 (16)	0.0250 (16)	0.0007 (13)	0.0074 (13)	−0.0007 (12)
O5	0.0334 (18)	0.0258 (16)	0.0338 (17)	0.0024 (13)	0.0181 (14)	0.0013 (13)
N1	0.028 (2)	0.0211 (19)	0.0277 (19)	0.0002 (15)	0.0080 (16)	−0.0034 (15)
N2	0.0203 (19)	0.0190 (18)	0.0300 (19)	0.0020 (14)	0.0101 (16)	−0.0012 (15)
C1	0.027 (2)	0.014 (2)	0.033 (2)	0.0016 (17)	0.013 (2)	0.0032 (17)
C2	0.024 (2)	0.015 (2)	0.032 (2)	0.0016 (17)	0.0124 (18)	0.0012 (17)
C3	0.034 (2)	0.020 (2)	0.030 (2)	0.002 (2)	0.0190 (19)	0.0013 (19)
C4	0.038 (3)	0.018 (2)	0.030 (2)	0.0046 (19)	0.016 (2)	0.0054 (18)
C5	0.021 (2)	0.013 (2)	0.033 (2)	0.0029 (16)	0.0094 (18)	0.0015 (17)
C6	0.035 (3)	0.020 (2)	0.029 (2)	−0.0006 (18)	0.014 (2)	−0.0041 (17)
C7	0.030 (2)	0.021 (2)	0.026 (2)	0.0008 (19)	0.0094 (18)	0.0049 (18)
C8	0.023 (2)	0.018 (2)	0.027 (2)	−0.0004 (17)	0.0092 (18)	−0.0028 (17)
C9	0.028 (2)	0.015 (2)	0.034 (2)	0.0058 (16)	0.009 (2)	0.0009 (17)
C10	0.027 (2)	0.018 (2)	0.035 (2)	0.0061 (17)	0.014 (2)	0.0010 (17)
C11	0.060 (4)	0.032 (3)	0.044 (3)	0.005 (2)	0.023 (3)	0.011 (2)
C12	0.035 (3)	0.035 (3)	0.053 (3)	0.011 (2)	0.004 (2)	−0.002 (2)
C13	0.041 (3)	0.042 (3)	0.052 (3)	0.005 (2)	0.030 (3)	0.006 (2)

C14	0.038 (3)	0.029 (3)	0.045 (3)	0.001 (2)	0.012 (2)	−0.008 (2)
Co1	0.0304 (5)	0.0162 (4)	0.0263 (4)	0.0034 (4)	0.0101 (4)	0.0022 (3)

Geometric parameters (Å, º)

O1—C1	1.269 (5)	C6—H6A	0.9300
O1—Co1	2.026 (3)	C7—H7A	0.9300
O2—C1	1.244 (5)	C9—C12	1.508 (6)
O3—N1	1.274 (4)	C9—C11	1.525 (6)
O4—N2	1.292 (4)	C9—C10	1.550 (6)
O4—Co1^{i}	2.199 (3)	C10—C14	1.514 (6)
O5—Co1	2.127 (3)	C10—C13	1.531 (6)
O5—H1O5	0.8657	C11—H11A	0.9600
O5—H2O5	0.8506	C11—H11B	0.9600
N1—C8	1.352 (5)	C11—H11C	0.9600
N1—C9	1.505 (5)	C12—H12A	0.9600
N2—C8	1.350 (5)	C12—H12B	0.9600
N2—C10	1.503 (5)	C12—H12C	0.9600
C1—C2	1.508 (5)	C13—H13A	0.9600
C2—C3	1.383 (5)	C13—H13B	0.9600
C2—C7	1.401 (6)	C13—H13C	0.9600
C3—C4	1.385 (6)	C14—H14A	0.9600
C3—H3A	0.9300	C14—H14C	0.9600
C4—C5	1.386 (6)	C14—H14D	0.9600
C4—H4A	0.9300	Co1—O1^{ii}	2.026 (3)
C5—C6	1.394 (5)	Co1—O5^{ii}	2.127 (3)
C5—C8	1.465 (5)	Co1—O4^{iii}	2.199 (3)
C6—C7	1.378 (6)	Co1—O4^{iv}	2.199 (3)
C1—O1—Co1	131.3 (3)	C14—C10—C13	109.6 (4)
N2—O4—Co1^{i}	123.0 (2)	N2—C10—C9	99.8 (3)
Co1—O5—H1O5	96.3	C14—C10—C9	115.5 (4)
Co1—O5—H2O5	128.4	C13—C10—C9	113.3 (4)
H1O5—O5—H2O5	106.2	C9—C11—H11A	109.5
O3—N1—C8	126.3 (3)	C9—C11—H11B	109.5
O3—N1—C9	122.0 (3)	H11A—C11—H11B	109.5
C8—N1—C9	111.5 (3)	C9—C11—H11C	109.5
O4—N2—C8	125.3 (3)	H11A—C11—H11C	109.5
O4—N2—C10	123.6 (3)	H11B—C11—H11C	109.5
C8—N2—C10	110.8 (3)	C9—C12—H12A	109.5
O2—C1—O1	125.5 (4)	C9—C12—H12B	109.5
O2—C1—C2	118.9 (4)	H12A—C12—H12B	109.5
O1—C1—C2	115.6 (4)	C9—C12—H12C	109.5
C3—C2—C7	118.7 (4)	H12A—C12—H12C	109.5
C3—C2—C1	119.7 (4)	H12B—C12—H12C	109.5
C7—C2—C1	121.6 (4)	C10—C13—H13A	109.5
C2—C3—C4	121.1 (4)	C10—C13—H13B	109.5
C2—C3—H3A	119.5	H13A—C13—H13B	109.5

C4—C3—H3A	119.5	C10—C13—H13C	109.5
C3—C4—C5	119.6 (4)	H13A—C13—H13C	109.5
C3—C4—H4A	120.2	H13B—C13—H13C	109.5
C5—C4—H4A	120.2	C10—C14—H14A	109.5
C4—C5—C6	120.2 (4)	C10—C14—H14C	109.5
C4—C5—C8	119.8 (4)	H14A—C14—H14C	109.5
C6—C5—C8	120.0 (4)	C10—C14—H14D	109.5
C7—C6—C5	119.5 (4)	H14A—C14—H14D	109.5
C7—C6—H6A	120.2	H14C—C14—H14D	109.5
C5—C6—H6A	120.2	O1^{ii}—Co1—O1	180.000 (1)
C6—C7—C2	120.9 (4)	O1^{ii}—Co1—O5^{ii}	91.43 (11)
C6—C7—H7A	119.5	O1—Co1—O5^{ii}	88.57 (11)
C2—C7—H7A	119.5	O1^{ii}—Co1—O5	88.57 (11)
N2—C8—N1	108.2 (3)	O1—Co1—O5	91.43 (11)
N2—C8—C5	125.7 (4)	O5^{ii}—Co1—O5	180.000 (1)
N1—C8—C5	125.9 (4)	O1^{ii}—Co1—O4^{iii}	91.38 (11)
N1—C9—C12	110.6 (4)	O1—Co1—O4^{iii}	88.62 (11)
N1—C9—C11	106.4 (4)	O5^{ii}—Co1—O4^{iii}	92.34 (11)
C12—C9—C11	111.1 (4)	O5—Co1—O4^{iii}	87.66 (11)
N1—C9—C10	99.6 (3)	O1^{ii}—Co1—O4^{iv}	88.62 (11)
C12—C9—C10	114.4 (4)	O1—Co1—O4^{iv}	91.38 (11)
C11—C9—C10	113.8 (4)	O5^{ii}—Co1—O4^{iv}	87.66 (11)
N2—C10—C14	111.9 (4)	O5—Co1—O4^{iv}	92.34 (11)
N2—C10—C13	106.0 (3)	O4^{iii}—Co1—O4^{iv}	180.000 (1)

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) −*x*+1, −*y*−1, −*z*+2; (iii) *x*, *y*−1, *z*; (iv) −*x*+1, −*y*, −*z*+2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H1*O*5···O2^{ii}	0.87	2.31	2.736 (4)	111
O5—H2*O*5···O2^{v}	0.85	2.40	2.886 (4)	117

Symmetry codes: (ii) −*x*+1, −*y*−1, −*z*+2; (v) *x*, −*y*−1/2, *z*+1/2.