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Bis[*N*′-(2-methylpropylidene)-4-(prop-2-en-1-yloxy)benzohydrazidato-$\kappa^2 N',O$]nickel(II)

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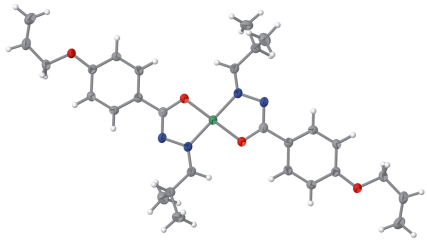
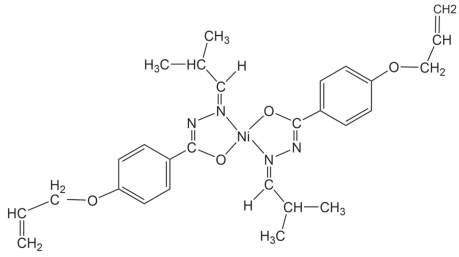
In the mononuclear title complex, $[Ni(C_{14}H_{17}N_2O_2)_2]$, the nickel(II) atom exhibits a slightly distorted square-planar coordination environment with the metal located on a crystallographic center of symmetry that induces a *trans* configuration of the *N*,*O* chelating ligands. In the crystal, weak C—H···O and C—H···π interactions consolidate the packing.

3D view



Chemical scheme



Structure description

Hydrazone ligands have attracted special attention for their chelating capabilities. The corresponding nickel(II) complexes are of considerable interest since they exhibit a broad spectrum of structure-dependent physiological and pharmacological activities (Al-Qadsy *et al.*, 2021; Neethu *et al.*, 2021; Krishnamoorthy *et al.*, 2012; Yang *et al.*, 2020).

The title nickel(II) complex crystallizes in the monoclinic space group $P2_1/c$ with the metal located on an inversion center, so that the asymmetric unit comprises a half molecule (Fig. 1). The Ni^{II} atom exhibits a slightly distorted square-planar coordination environment with the chelating ligands in a *trans* configuration imposed by symmetry. The coordinating enolizable O atom and the azomethine N atom of the deprotonated ligand form a five-membered nearly planar chelate ring (r.m.s. deviation from planarity = 0.0084 Å). The Ni—N1 and Ni—O1 bond lengths of 1.8671 (13) and 1.8382 (11) Å and the chelate angle of 83.64 (5)° are in agreement with those of previously reported complexes (Al-Qadsy *et al.*, 2021; Khan *et al.*, 2023, 2025; Krishnamoorthy *et al.*, 2012; Neethu *et al.*, 2021; Yang *et al.*, 2020), irrespective of the substituents present in the ligand. The C5—O1 bond length of 1.3080 (18) Å lies in between a C—O single and a C=O double bond. The bond lengths N1—C4 of 1.285 (2) Å and N2—C5 of 1.308 (2) Å

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

*Cg*3 is the centroid of the benzyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O1^{i}	0.95	2.47	2.9822 (19)	114
C12—H12*A*···O1^{ii}	0.99	2.58	3.451 (2)	148
C12—H12*B*···*Cg*3^{iii}	0.99	2.64	3.4762 (17)	143

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

are indicative of a conjugated system within the —CH═N—N═C—O fragment, even after the deprotonation of its enolized carbonyl O atom. The benzylidene entity (C5–C11) is practically co-planar with the N_2O_2 coordination plane [dihedral angle of 3.09 (5)°] and also to the allyloxy fragment [7.73 (14)°]. An intramolecular hydrogen bond between a methine group (C4—H4) and the chelating O atom (Table 1) stabilizes the molecular conformation.

The packing of the complex molecules is consolidated by a weak intermolecular hydrogen bond between a methylene group (C12—H12*A*) and the ligating O atom of a neighbouring complex. C—H···π interactions between the second H atom of this methylene group and the centroid (*Cg*3) of the benzyl ring are also observed (Table 1), while no apparent π–π interactions are present. In the crystal packing (Fig. 2) the shortest separation of Ni^{II} atoms is 8.5593 (2) Å.

Synthesis and crystallization

Isobutyraldehyde (0.216 g, 3.0 mmol) in 10 ml of ethanol was added to a 30 ml ethanolic solution of 4-(allyloxy)benzoylhydrazine (0.576 g, 3.0 mmol), followed by refluxing for one h. To this mixture $Ni(CH_3COO)_2{\cdot}4H_2O$ (0.373 g, 1.5 mmol, in 30 ml) was introduced, and refluxing was prolonged for additional three h. The yellow precipitate formed was then filtered off while hot. Finally, the product was dried and stored in a vacuum desiccator containing anhydrous $CaCl_2$. Single crystals of the nickel(II) complex, suitable for X-ray diffraction, were obtained through gradual evaporation from a mixture of chloroform and toluene (3:1, *v*/*v*) over a period of 3 weeks. Yellow crystals, yield: 0.604 g (73%); melting point: 485–487 K. IR data (KBr disc, cm^{-1}): 1606 ν(C═N), 1587 ν(C═C), 997 ν(N—N), 596 ν(*M*—N), 465 ν(*M*—O). ^{1}H NMR ($CDCl_3$, 400 MHz), δ: 7.82 (*d*, 2×2H, C-6, 8, *J* = 8.8 Hz), 6.85 (*d*, 2×2H, C-5, 9, *J* = 8.8 Hz), 6.50 (*d*, 2×1H, C-11, CH═N, *J* = 8.0 Hz), 6.4–6.0 (*m*, 2×1H, C-2, H_c), 5.41 (*d*, 2×1H, C-1, H_a, *J* = 17.6 Hz), 5.29 (*d*, 2×1H, C-1, H_b, *J* = 10.4 Hz), 4.56 (*d*, 2×2H, C-3, OCH_2, *J* = 5.6 Hz,), 3.72–3.63 (*m*, 2×1H, C-12), 1.16 (*d*, 2×6H, C-13,14, *J* = 8.4 Hz). HRMS (FAB) calculated for $C_{28}H_{34}N_4NiO_4$, $[M+H]^+$: 549.20086, found $[M+H]^+$: 549.20062.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Ni(C_{14}H_{17}N_2O_2)_2]$
M_r	549.30
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	173
a, *b*, *c* (Å)	12.5241 (3), 13.4196 (4), 8.5593 (2)
β (°)	109.274 (8)
V (Å^3)	1357.91 (9)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	0.75
Crystal size (mm)	0.22 × 0.11 × 0.07
Data collection	
Diffractometer	Rigaku R-AXIS RAPID
Absorption correction	Multi-scan (*ABSCOR*; Rigaku, 1995)
T_{min}, T_{max}	0.722, 0.949
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12824, 3106, 2678
R_{int}	0.031
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.032, 0.081, 1.04
No. of reflections	3106
No. of parameters	171
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.43, −0.22

Computer programs: *RAPID-AUTO* (Rigaku, 2019), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 1999) and *WinGX* (Farrugia, 2012).

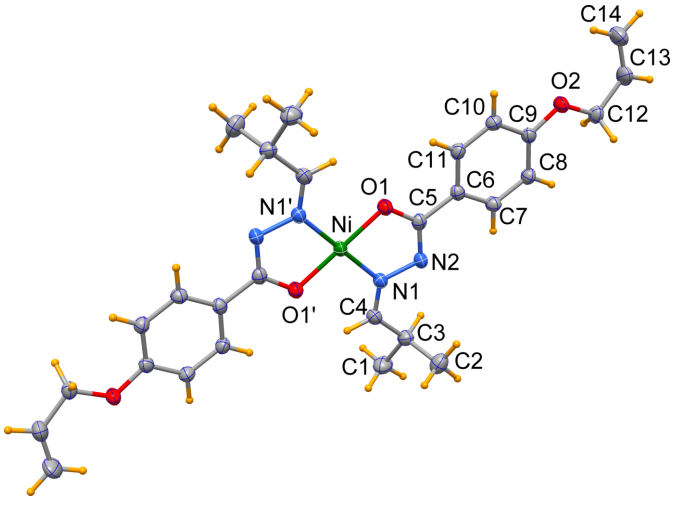


Figure 1
The molecular structure of the title complex with displacement ellipsoids drawn at the 50% probability level. Atoms marked with a prime character and all non-labelled atoms are generated by inversion symmetry. [Symmetry code: $-x+1, -y+1, -z$.]

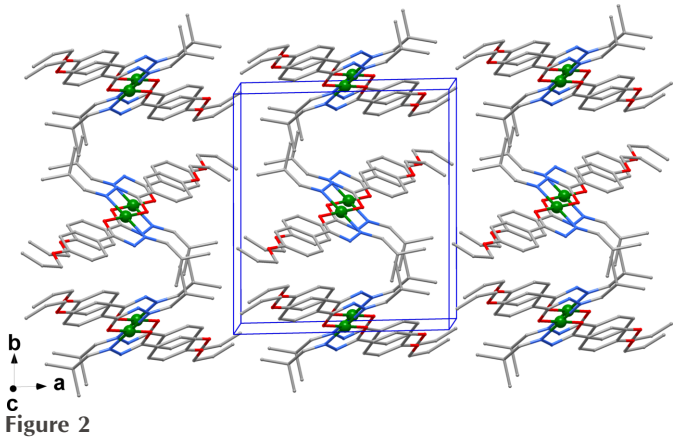


Figure 2
Crystal packing of the title complex with H atoms removed for clarity.

Refinement

Crystal data, data collection, and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2025). **10**, x250988 [https://doi.org/10.1107/S2414314625009885]

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Crystal data

$[Ni(C_{14}H_{17}N_2O_2)_2]$
M_r = 549.30
Monoclinic, $P2_1/c$
a = 12.5241 (3) Å
b = 13.4196 (4) Å
c = 8.5593 (2) Å
β = 109.274 (8)°
V = 1357.91 (9) Å^3
Z = 2
$F(000)$ = 580
D_x = 1.343 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 10492 reflections
θ = 2.3–27.5°
μ = 0.75 mm^{-1}
T = 173 K
Prism, yellow
0.22 × 0.11 × 0.07 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Rigaku, 1995)
T_{min} = 0.722, T_{max} = 0.949
12824 measured reflections
3106 independent reflections
2678 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 27.5°, θ_{min} = 2.3°
h = −16→16
k = −17→17
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.081
S = 1.04
3106 reflections
171 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 0.5845P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.500000	0.500000	0.000000	0.02365 (9)
O1	0.40545 (9)	0.48867 (8)	0.12389 (13)	0.0272 (2)
O2	0.18732 (9)	0.38385 (9)	0.66963 (13)	0.0302 (3)
N1	0.59615 (11)	0.42033 (10)	0.16621 (15)	0.0251 (3)
N2	0.55099 (11)	0.39157 (10)	0.28959 (16)	0.0265 (3)
C1	0.89710 (15)	0.35433 (15)	0.3488 (3)	0.0423 (4)
H1A	0.913977	0.331595	0.250634	0.051*
H1B	0.945166	0.318522	0.446480	0.051*
H1C	0.911834	0.426013	0.363773	0.051*
C2	0.74500 (17)	0.22294 (14)	0.3009 (2)	0.0411 (4)
H2A	0.798169	0.184695	0.390729	0.049*
H2B	0.750983	0.201381	0.194659	0.049*
H2C	0.667728	0.211635	0.300996	0.049*
C3	0.77329 (14)	0.33389 (12)	0.3261 (2)	0.0290 (3)
H3	0.758739	0.355730	0.428933	0.035*
C4	0.69787 (14)	0.39313 (12)	0.18425 (19)	0.0274 (3)
H4	0.727860	0.412281	0.100237	0.033*
C5	0.45128 (13)	0.43305 (11)	0.25439 (18)	0.0245 (3)
C6	0.38466 (13)	0.41678 (11)	0.36654 (18)	0.0239 (3)
C7	0.42822 (13)	0.36377 (12)	0.51408 (19)	0.0264 (3)
H7	0.502209	0.336480	0.543159	0.032*
C8	0.36562 (13)	0.35007 (12)	0.61932 (19)	0.0268 (3)
H8	0.396352	0.313752	0.719449	0.032*
C9	0.25697 (13)	0.39030 (11)	0.57628 (18)	0.0247 (3)
C10	0.21195 (14)	0.44225 (12)	0.42783 (19)	0.0278 (3)
H10	0.137429	0.468481	0.397653	0.033*
C11	0.27520 (13)	0.45567 (12)	0.32482 (19)	0.0264 (3)
H11	0.244144	0.491699	0.224447	0.032*
C12	0.22916 (14)	0.33458 (12)	0.82672 (19)	0.0289 (3)
H12A	0.301977	0.364646	0.894171	0.035*
H12B	0.242193	0.263153	0.810353	0.035*
C13	0.14474 (17)	0.34507 (15)	0.9136 (2)	0.0416 (4)
H13	0.160758	0.310432	1.015534	0.050*
C14	0.05119 (18)	0.39645 (17)	0.8655 (3)	0.0511 (5)
H14A	0.030867	0.432590	0.764502	0.061*
H14B	0.003345	0.397763	0.931571	0.061*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02442 (15)	0.02543 (15)	0.02219 (15)	0.00068 (11)	0.00916 (10)	0.00268 (11)
O1	0.0265 (5)	0.0313 (6)	0.0251 (5)	0.0021 (5)	0.0102 (4)	0.0048 (4)
O2	0.0293 (6)	0.0368 (6)	0.0278 (6)	0.0051 (5)	0.0139 (5)	0.0085 (5)
N1	0.0277 (6)	0.0259 (6)	0.0237 (6)	0.0000 (5)	0.0111 (5)	0.0009 (5)
N2	0.0291 (7)	0.0281 (7)	0.0253 (6)	0.0002 (5)	0.0130 (5)	0.0033 (5)

C1	0.0313 (9)	0.0417 (10)	0.0533 (11)	0.0065 (8)	0.0132 (8)	0.0076 (9)
C2	0.0491 (11)	0.0308 (9)	0.0409 (10)	0.0014 (8)	0.0114 (8)	0.0049 (8)
C3	0.0296 (8)	0.0293 (8)	0.0288 (8)	0.0042 (7)	0.0107 (6)	0.0013 (6)
C4	0.0301 (8)	0.0282 (8)	0.0263 (7)	0.0005 (6)	0.0127 (6)	0.0008 (6)
C5	0.0275 (7)	0.0226 (7)	0.0234 (7)	−0.0035 (6)	0.0086 (6)	−0.0016 (6)
C6	0.0251 (7)	0.0233 (7)	0.0240 (7)	−0.0029 (6)	0.0091 (6)	−0.0001 (6)
C7	0.0232 (7)	0.0290 (8)	0.0268 (7)	0.0017 (6)	0.0079 (6)	0.0013 (6)
C8	0.0286 (8)	0.0271 (8)	0.0243 (7)	0.0016 (6)	0.0083 (6)	0.0044 (6)
C9	0.0271 (8)	0.0229 (7)	0.0260 (7)	−0.0015 (6)	0.0114 (6)	−0.0002 (6)
C10	0.0265 (8)	0.0281 (8)	0.0292 (8)	0.0047 (6)	0.0099 (6)	0.0042 (6)
C11	0.0299 (8)	0.0242 (7)	0.0251 (7)	0.0020 (6)	0.0088 (6)	0.0042 (6)
C12	0.0347 (9)	0.0279 (8)	0.0251 (7)	0.0009 (7)	0.0111 (6)	0.0040 (6)
C13	0.0523 (11)	0.0428 (10)	0.0383 (10)	0.0070 (9)	0.0265 (9)	0.0117 (8)
C14	0.0483 (12)	0.0591 (13)	0.0584 (13)	0.0099 (10)	0.0345 (10)	0.0159 (10)

Geometric parameters (Å, º)

Ni1—O1^{i}	1.8382 (11)	C4—H4	0.9500
Ni1—O1	1.8382 (11)	C5—C6	1.481 (2)
Ni1—N1^{i}	1.8671 (13)	C6—C7	1.394 (2)
Ni1—N1	1.8671 (13)	C6—C11	1.399 (2)
O1—C5	1.3080 (18)	C7—C8	1.388 (2)
O2—C9	1.3663 (18)	C7—H7	0.9500
O2—C12	1.4334 (18)	C8—C9	1.396 (2)
N1—C4	1.285 (2)	C8—H8	0.9500
N1—N2	1.4065 (17)	C9—C10	1.395 (2)
N2—C5	1.308 (2)	C10—C11	1.378 (2)
C1—C3	1.523 (2)	C10—H10	0.9500
C1—H1A	0.9800	C11—H11	0.9500
C1—H1B	0.9800	C12—C13	1.486 (2)
C1—H1C	0.9800	C12—H12A	0.9900
C2—C3	1.529 (2)	C12—H12B	0.9900
C2—H2A	0.9800	C13—C14	1.304 (3)
C2—H2B	0.9800	C13—H13	0.9500
C2—H2C	0.9800	C14—H14A	0.9500
C3—C4	1.497 (2)	C14—H14B	0.9500
C3—H3	1.0000		
O1^{i}—Ni1—O1	180.0	O1—C5—N2	123.77 (14)
O1^{i}—Ni1—N1^{i}	83.64 (5)	O1—C5—C6	117.08 (13)
O1—Ni1—N1^{i}	96.36 (5)	N2—C5—C6	119.15 (13)
O1^{i}—Ni1—N1	96.36 (5)	C7—C6—C11	118.63 (14)
O1—Ni1—N1	83.64 (5)	C7—C6—C5	121.78 (14)
N1^{i}—Ni1—N1	180.0	C11—C6—C5	119.59 (14)
C5—O1—Ni1	110.80 (10)	C8—C7—C6	121.30 (14)
C9—O2—C12	118.13 (12)	C8—C7—H7	119.4
C4—N1—N2	117.41 (13)	C6—C7—H7	119.4
C4—N1—Ni1	128.23 (11)	C7—C8—C9	119.21 (14)

N2—N1—Ni1	114.28 (10)	C7—C8—H8	120.4
C5—N2—N1	107.47 (12)	C9—C8—H8	120.4
C3—C1—H1A	109.5	O2—C9—C10	114.85 (13)
C3—C1—H1B	109.5	O2—C9—C8	125.18 (13)
H1A—C1—H1B	109.5	C10—C9—C8	119.97 (14)
C3—C1—H1C	109.5	C11—C10—C9	120.22 (14)
H1A—C1—H1C	109.5	C11—C10—H10	119.9
H1B—C1—H1C	109.5	C9—C10—H10	119.9
C3—C2—H2A	109.5	C10—C11—C6	120.66 (14)
C3—C2—H2B	109.5	C10—C11—H11	119.7
H2A—C2—H2B	109.5	C6—C11—H11	119.7
C3—C2—H2C	109.5	O2—C12—C13	108.98 (13)
H2A—C2—H2C	109.5	O2—C12—H12A	109.9
H2B—C2—H2C	109.5	C13—C12—H12A	109.9
C4—C3—C1	110.55 (14)	O2—C12—H12B	109.9
C4—C3—C2	110.35 (14)	C13—C12—H12B	109.9
C1—C3—C2	111.84 (15)	H12A—C12—H12B	108.3
C4—C3—H3	108.0	C14—C13—C12	127.31 (17)
C1—C3—H3	108.0	C14—C13—H13	116.3
C2—C3—H3	108.0	C12—C13—H13	116.3
N1—C4—C3	125.70 (14)	C13—C14—H14A	120.0
N1—C4—H4	117.1	C13—C14—H14B	120.0
C3—C4—H4	117.1	H14A—C14—H14B	120.0

N1^{i}—Ni1—O1—C5	179.15 (10)	N2—C5—C6—C7	4.2 (2)
N1—Ni1—O1—C5	−0.84 (10)	O1—C5—C6—C11	3.6 (2)
O1^{i}—Ni1—N1—C4	4.90 (15)	N2—C5—C6—C11	−176.09 (14)
O1—Ni1—N1—C4	−175.10 (15)	C11—C6—C7—C8	−0.6 (2)
O1^{i}—Ni1—N1—N2	−178.37 (10)	C5—C6—C7—C8	179.14 (14)
O1—Ni1—N1—N2	1.63 (10)	C6—C7—C8—C9	0.0 (2)
C4—N1—N2—C5	175.12 (14)	C12—O2—C9—C10	−177.85 (14)
Ni1—N1—N2—C5	−1.98 (15)	C12—O2—C9—C8	1.9 (2)
N2—N1—C4—C3	−0.5 (2)	C7—C8—C9—O2	−178.80 (14)
Ni1—N1—C4—C3	176.19 (12)	C7—C8—C9—C10	0.9 (2)
C1—C3—C4—N1	−154.63 (17)	O2—C9—C10—C11	178.54 (14)
C2—C3—C4—N1	81.1 (2)	C8—C9—C10—C11	−1.2 (2)
Ni1—O1—C5—N2	−0.14 (19)	C9—C10—C11—C6	0.6 (2)
Ni1—O1—C5—C6	−179.85 (10)	C7—C6—C11—C10	0.3 (2)
N1—N2—C5—O1	1.4 (2)	C5—C6—C11—C10	−179.43 (14)
N1—N2—C5—C6	−178.90 (12)	C9—O2—C12—C13	174.40 (14)
O1—C5—C6—C7	−176.10 (14)	O2—C12—C13—C14	−5.0 (3)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, º)

*Cg*3 is the centroid of the benzyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O1^{i}	0.95	2.47	2.9822 (19)	114

C12—H12*A*···O1^{ii}	0.99	2.58	3.451 (2)	148
C12—H12*B*···*Cg*3^{iii}	0.99	2.64	3.4762 (17)	143

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