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Tectorigenin monohydrate: an isoflavone from *Belamcanda chinensis*Benguo Liu,^{a*} Yuxiang Ma,^b Han Gao^a and Qiong Wu^c

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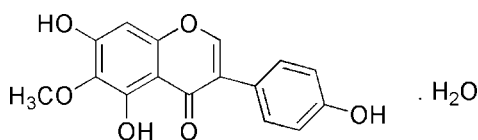
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Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.045; wR factor = 0.117; data-to-parameter ratio = 13.2.

The title compound [systematic name: 5,7-dihydroxy-3-(4-hydroxyphenyl)-6-methoxy-4*H*-chromen-4-one monohydrate], $\text{C}_{16}\text{H}_{12}\text{O}_6 \cdot \text{H}_2\text{O}$, is isolated from *Belamcanda chinensis* and is said to have antimicrobial and anti-inflammatory effects. The chromen-4-one system and the benzene ring are inclined at a dihedral angle of 36.79 (6)°. Molecules are linked by inter- and intramolecular $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds.

Related literature

For general background, see: Oh *et al.* (2001). For a related structure, see: Gao *et al.* (2008).



Experimental

Crystal data

 $\text{C}_{16}\text{H}_{12}\text{O}_6 \cdot \text{H}_2\text{O}$ $M_r = 318.27$

Monoclinic, $P2_1/c$
 $a = 12.971$ (3) Å
 $b = 14.652$ (3) Å
 $c = 7.2930$ (15) Å
 $\beta = 103.81$ (3)°
 $V = 1346.0$ (5) Å³

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.13$ mm⁻¹
 $T = 113$ (2) K
 $0.14 \times 0.04 \times 0.02$ mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MS, 2005)
 $T_{\min} = 0.973$, $T_{\max} = 0.998$

9180 measured reflections
2967 independent reflections
2069 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.085$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.117$
 $S = 1.00$
2967 reflections
224 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.33$ e Å⁻³
 $\Delta\rho_{\min} = -0.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O7}-\text{H7B} \cdots \text{O2}^{\text{i}}$	0.77 (3)	2.57 (2)	2.971 (2)	114 (2)
$\text{O7}-\text{H7A} \cdots \text{O6}^{\text{ii}}$	0.95 (3)	1.95 (3)	2.884 (2)	167 (2)
$\text{O6}-\text{H6} \cdots \text{O1}^{\text{iii}}$	0.88 (2)	1.88 (2)	2.7368 (17)	167 (2)
$\text{O3}-\text{H3} \cdots \text{O5}$	0.90 (2)	1.71 (2)	2.5658 (16)	159.6 (18)
$\text{O1}-\text{H1} \cdots \text{O7}^{\text{iv}}$	0.87 (2)	1.83 (2)	2.6630 (17)	160.4 (19)

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

Data collection: *CrystalClear* (Rigaku/MS, 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: BT2795).

References

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Oh, K. B., Kang, H. & Matsuoka, H. (2001). *Biosci. Biotechnol. Biochem.* **65**, 939–942.
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supporting information

Acta Cryst. (2008). E64, o2056 [doi:10.1107/S1600536808030833]

Tectorigenin monohydrate: an isoflavone from *Belamcanda chinensis*

Benguo Liu, Yuxiang Ma, Han Gao and Qiong Wu

S1. Comment

The title compound [systematic name: 5,7-dihydroxy-3-(4-hydroxyphenyl)-6-methoxy-4*H*-chromen-4-one] was isolated from *Belamcanda chinensis* and is said to have antimicrobial and anti-inflammatory effects. We report here the crystal structure of its monohydrate. The two aromatic ring systems rings are inclined at a dihedral angle of 36.79 (6)°. The molecules are linked by intermolecular O—H...O hydrogen bonds (Table 1).

S2. Experimental

The title compound was isolated from *Belamcanda chinensis*.

S3. Refinement

H atoms bonded to C were positioned geometrically (C—H=0.95–0.98 Å), and refined as riding with $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. The coordinates of the H atoms bonded to O were refined with $U_{\text{iso}}(\text{H})=1.5U_{\text{eq}}(\text{O})$.

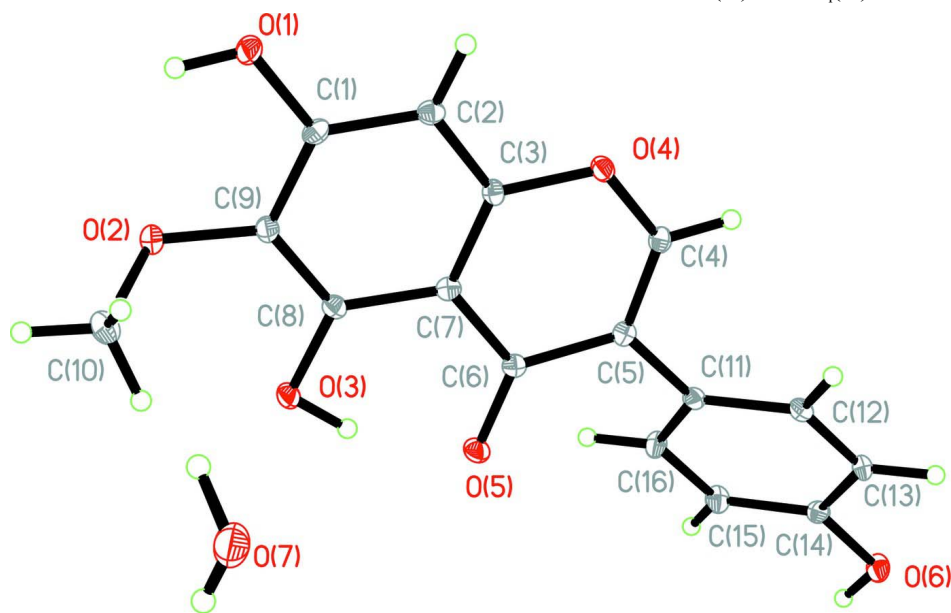


Figure 1

A view of the title compound. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

5,7-dihydroxy-3-(4-hydroxyphenyl)-6-methoxy-4H-chromen-4-one monohydrate

Crystal data

C₁₆H₁₂O₆·H₂O $M_r = 318.27$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 12.971 (3) \text{ \AA}$ $b = 14.652 (3) \text{ \AA}$ $c = 7.2930 (15) \text{ \AA}$ $\beta = 103.81 (3)^\circ$ $V = 1346.0 (5) \text{ \AA}^3$ $Z = 4$ $F(000) = 664$ $D_x = 1.571 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2562 reflections

 $\theta = 1.6\text{--}27.1^\circ$ $\mu = 0.13 \text{ mm}^{-1}$ $T = 113 \text{ K}$

Block, colorless

 $0.14 \times 0.04 \times 0.02 \text{ mm}$

Data collection

Rigaku Saturn CCD area-detector
diffractometer

Radiation source: rotating anode

Confocal monochromator

Detector resolution: 7.31 pixels mm^{-1} ω and φ scans

Absorption correction: multi-scan

(CrystalClear; Rigaku/MSC, 2005)

 $T_{\min} = 0.973$, $T_{\max} = 0.998$

9180 measured reflections

2967 independent reflections

2069 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.085$ $\theta_{\max} = 27.1^\circ$, $\theta_{\min} = 1.6^\circ$ $h = -16 \rightarrow 16$ $k = -15 \rightarrow 18$ $l = -9 \rightarrow 7$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.045$ $wR(F^2) = 0.117$ $S = 1.00$

2967 reflections

224 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sitesH atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0629P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.33 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.27 \text{ e \AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.15492 (10)	0.72865 (7)	-0.00354 (17)	0.0204 (3)
H1	0.1297 (17)	0.6740 (14)	-0.033 (3)	0.031*
O2	0.26095 (9)	0.56579 (7)	0.09198 (16)	0.0191 (3)

O3	0.45733 (10)	0.56828 (7)	0.34259 (17)	0.0193 (3)
H3	0.5174 (18)	0.5889 (13)	0.420 (3)	0.029*
O4	0.45077 (9)	0.89559 (7)	0.32487 (17)	0.0173 (3)
O5	0.61065 (9)	0.66388 (7)	0.53713 (16)	0.0188 (3)
O6	1.02531 (10)	0.87836 (8)	0.95714 (18)	0.0221 (3)
H6	1.0680 (18)	0.8325 (15)	0.952 (3)	0.033*
C1	0.25505 (13)	0.72879 (10)	0.1088 (2)	0.0159 (4)
C2	0.30232 (13)	0.81198 (9)	0.1645 (2)	0.0161 (4)
H2	0.2660	0.8676	0.1254	0.019*
C3	0.40402 (13)	0.81206 (9)	0.2788 (2)	0.0142 (4)
C4	0.55224 (13)	0.89830 (10)	0.4301 (2)	0.0157 (4)
H4	0.5850	0.9566	0.4520	0.019*
C5	0.61050 (13)	0.82575 (10)	0.5063 (2)	0.0141 (4)
C6	0.56447 (13)	0.73485 (10)	0.4688 (2)	0.0142 (4)
C7	0.45876 (13)	0.73227 (10)	0.3443 (2)	0.0141 (4)
C8	0.40834 (13)	0.64784 (9)	0.2855 (2)	0.0148 (4)
C9	0.30772 (14)	0.64647 (9)	0.1659 (2)	0.0155 (4)
C10	0.22428 (15)	0.50987 (11)	0.2257 (3)	0.0243 (5)
H10A	0.1689	0.5425	0.2702	0.036*
H10B	0.2838	0.4963	0.3332	0.036*
H10C	0.1952	0.4527	0.1648	0.036*
C11	0.71993 (13)	0.83905 (9)	0.6228 (2)	0.0153 (4)
C12	0.74392 (14)	0.91262 (9)	0.7482 (2)	0.0157 (4)
H12	0.6895	0.9541	0.7595	0.019*
C13	0.84645 (14)	0.92549 (9)	0.8561 (2)	0.0172 (4)
H13	0.8622	0.9759	0.9401	0.021*
C14	0.92533 (13)	0.86514 (10)	0.8412 (2)	0.0165 (4)
C15	0.90422 (14)	0.79282 (10)	0.7145 (2)	0.0197 (4)
H15	0.9594	0.7526	0.7010	0.024*
C16	0.80091 (14)	0.78018 (10)	0.6076 (2)	0.0185 (4)
H16	0.7857	0.7302	0.5225	0.022*
O7	0.04211 (12)	0.58284 (9)	0.8508 (2)	0.0333 (4)
H7A	0.033 (2)	0.5862 (15)	0.718 (4)	0.050*
H7B	0.076 (2)	0.5390 (18)	0.876 (4)	0.050*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0144 (7)	0.0182 (6)	0.0251 (7)	0.0000 (4)	-0.0024 (5)	-0.0017 (4)
O2	0.0203 (7)	0.0157 (5)	0.0204 (6)	-0.0030 (4)	0.0031 (5)	-0.0024 (4)
O3	0.0178 (7)	0.0122 (5)	0.0247 (7)	0.0012 (4)	-0.0012 (5)	0.0003 (4)
O4	0.0142 (6)	0.0122 (5)	0.0233 (6)	0.0001 (4)	0.0001 (5)	0.0003 (4)
O5	0.0170 (6)	0.0151 (5)	0.0228 (6)	0.0026 (4)	0.0018 (5)	0.0022 (4)
O6	0.0142 (7)	0.0193 (6)	0.0287 (7)	0.0001 (5)	-0.0033 (5)	-0.0036 (4)
C1	0.0118 (8)	0.0211 (8)	0.0142 (8)	0.0007 (6)	0.0023 (7)	-0.0008 (5)
C2	0.0150 (9)	0.0167 (7)	0.0167 (8)	0.0031 (6)	0.0039 (7)	0.0011 (5)
C3	0.0144 (8)	0.0137 (7)	0.0156 (8)	-0.0020 (6)	0.0060 (7)	-0.0013 (5)
C4	0.0135 (8)	0.0167 (7)	0.0169 (8)	-0.0019 (6)	0.0038 (7)	-0.0018 (5)

C5	0.0129 (8)	0.0162 (7)	0.0144 (8)	0.0003 (6)	0.0054 (7)	-0.0015 (5)
C6	0.0142 (9)	0.0158 (7)	0.0135 (8)	0.0009 (6)	0.0052 (7)	0.0000 (5)
C7	0.0118 (8)	0.0161 (7)	0.0149 (8)	0.0013 (6)	0.0043 (7)	0.0006 (5)
C8	0.0165 (9)	0.0141 (7)	0.0146 (8)	0.0008 (6)	0.0053 (7)	0.0006 (5)
C9	0.0182 (9)	0.0141 (7)	0.0149 (8)	-0.0021 (6)	0.0054 (7)	-0.0012 (5)
C10	0.0250 (10)	0.0205 (8)	0.0285 (10)	-0.0043 (7)	0.0090 (8)	0.0021 (6)
C11	0.0152 (9)	0.0140 (7)	0.0159 (8)	-0.0019 (6)	0.0023 (7)	0.0016 (5)
C12	0.0162 (9)	0.0136 (7)	0.0181 (8)	0.0019 (6)	0.0056 (7)	0.0015 (5)
C13	0.0199 (10)	0.0125 (7)	0.0186 (8)	-0.0029 (6)	0.0033 (7)	-0.0018 (5)
C14	0.0132 (8)	0.0169 (7)	0.0181 (8)	-0.0018 (6)	0.0013 (7)	0.0022 (5)
C15	0.0153 (9)	0.0183 (7)	0.0253 (9)	0.0030 (6)	0.0045 (7)	-0.0031 (6)
C16	0.0177 (9)	0.0179 (7)	0.0192 (9)	-0.0006 (6)	0.0030 (7)	-0.0047 (5)
O7	0.0329 (9)	0.0253 (6)	0.0358 (9)	0.0005 (6)	-0.0037 (7)	-0.0060 (5)

Geometric parameters (Å, °)

O1—C1	1.3602 (18)	C5—C11	1.483 (2)
O1—H1	0.87 (2)	C6—C7	1.453 (2)
O2—C9	1.3784 (16)	C7—C8	1.4171 (19)
O2—C10	1.439 (2)	C8—C9	1.386 (2)
O3—C8	1.3453 (17)	C10—H10A	0.9800
O3—H3	0.90 (2)	C10—H10B	0.9800
O4—C4	1.3568 (18)	C10—H10C	0.9800
O4—C3	1.3717 (16)	C11—C16	1.384 (2)
O5—C6	1.2434 (17)	C11—C12	1.400 (2)
O6—C14	1.3823 (18)	C12—C13	1.387 (2)
O6—H6	0.88 (2)	C12—H12	0.9500
C1—C2	1.381 (2)	C13—C14	1.376 (2)
C1—C9	1.400 (2)	C13—H13	0.9500
C2—C3	1.382 (2)	C14—C15	1.390 (2)
C2—H2	0.9500	C15—C16	1.393 (2)
C3—C7	1.3917 (19)	C15—H15	0.9500
C4—C5	1.345 (2)	C16—H16	0.9500
C4—H4	0.9500	O7—H7A	0.95 (3)
C5—C6	1.458 (2)	O7—H7B	0.77 (3)
C1—O1—H1	113.5 (13)	O2—C9—C8	121.26 (13)
C9—O2—C10	114.16 (13)	O2—C9—C1	118.99 (13)
C8—O3—H3	100.2 (12)	C8—C9—C1	119.62 (13)
C4—O4—C3	118.50 (11)	O2—C10—H10A	109.5
C14—O6—H6	112.1 (13)	O2—C10—H10B	109.5
O1—C1—C2	118.13 (13)	H10A—C10—H10B	109.5
O1—C1—C9	120.40 (13)	O2—C10—H10C	109.5
C2—C1—C9	121.47 (13)	H10A—C10—H10C	109.5
C1—C2—C3	118.09 (13)	H10B—C10—H10C	109.5
C1—C2—H2	121.0	C16—C11—C12	118.55 (14)
C3—C2—H2	121.0	C16—C11—C5	120.86 (13)
O4—C3—C2	116.81 (12)	C12—C11—C5	120.58 (15)

O4—C3—C7	120.39 (13)	C13—C12—C11	120.59 (15)
C2—C3—C7	122.80 (13)	C13—C12—H12	119.7
C5—C4—O4	125.74 (13)	C11—C12—H12	119.7
C5—C4—H4	117.1	C14—C13—C12	119.88 (14)
O4—C4—H4	117.1	C14—C13—H13	120.1
C4—C5—C6	118.67 (14)	C12—C13—H13	120.1
C4—C5—C11	119.95 (13)	C13—C14—O6	117.93 (14)
C6—C5—C11	121.36 (13)	C13—C14—C15	120.68 (14)
O5—C6—C7	121.41 (13)	O6—C14—C15	121.39 (15)
O5—C6—C5	123.52 (14)	C14—C15—C16	118.98 (16)
C7—C6—C5	115.08 (12)	C14—C15—H15	120.5
C3—C7—C8	117.94 (13)	C16—C15—H15	120.5
C3—C7—C6	121.37 (13)	C11—C16—C15	121.29 (14)
C8—C7—C6	120.69 (13)	C11—C16—H16	119.4
O3—C8—C9	119.11 (12)	C15—C16—H16	119.4
O3—C8—C7	120.86 (13)	H7A—O7—H7B	102 (2)
C9—C8—C7	120.03 (13)		
O1—C1—C2—C3	179.92 (17)	C10—O2—C9—C8	73.9 (2)
C9—C1—C2—C3	0.2 (3)	C10—O2—C9—C1	-110.33 (18)
C4—O4—C3—C2	-177.22 (16)	O3—C8—C9—O2	-5.6 (3)
C4—O4—C3—C7	2.1 (3)	C7—C8—C9—O2	173.72 (17)
C1—C2—C3—O4	177.11 (17)	O3—C8—C9—C1	178.63 (17)
C1—C2—C3—C7	-2.2 (3)	C7—C8—C9—C1	-2.1 (3)
C3—O4—C4—C5	-4.9 (3)	O1—C1—C9—O2	6.3 (3)
O4—C4—C5—C6	2.7 (3)	C2—C1—C9—O2	-173.97 (17)
O4—C4—C5—C11	-178.42 (17)	O1—C1—C9—C8	-177.84 (17)
C4—C5—C6—O5	-177.81 (18)	C2—C1—C9—C8	1.9 (3)
C11—C5—C6—O5	3.3 (3)	C4—C5—C11—C16	-139.51 (19)
C4—C5—C6—C7	2.0 (3)	C6—C5—C11—C16	39.4 (3)
C11—C5—C6—C7	-176.86 (16)	C4—C5—C11—C12	39.3 (3)
O4—C3—C7—C8	-177.26 (17)	C6—C5—C11—C12	-141.85 (18)
C2—C3—C7—C8	2.0 (3)	C16—C11—C12—C13	-0.7 (3)
O4—C3—C7—C6	2.6 (3)	C5—C11—C12—C13	-179.49 (16)
C2—C3—C7—C6	-178.17 (18)	C11—C12—C13—C14	-0.5 (3)
O5—C6—C7—C3	175.31 (19)	C12—C13—C14—O6	-177.31 (16)
C5—C6—C7—C3	-4.5 (3)	C12—C13—C14—C15	2.1 (3)
O5—C6—C7—C8	-4.8 (3)	C13—C14—C15—C16	-2.4 (3)
C5—C6—C7—C8	175.30 (17)	O6—C14—C15—C16	176.94 (16)
C3—C7—C8—O3	179.49 (17)	C12—C11—C16—C15	0.3 (3)
C6—C7—C8—O3	-0.4 (3)	C5—C11—C16—C15	179.12 (17)
C3—C7—C8—C9	0.2 (3)	C14—C15—C16—C11	1.2 (3)
C6—C7—C8—C9	-179.67 (17)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O7—H7B...O2 ⁱ	0.77 (3)	2.57 (2)	2.971 (2)	114 (2)

O7—H7A···O6 ⁱⁱ	0.95 (3)	1.95 (3)	2.884 (2)	167 (2)
O6—H6···O1 ⁱⁱⁱ	0.88 (2)	1.88 (2)	2.7368 (17)	167 (2)
O3—H3···O5	0.90 (2)	1.71 (2)	2.5658 (16)	159.6 (18)
O1—H1···O7 ^{iv}	0.87 (2)	1.83 (2)	2.6630 (17)	160.4 (19)

Symmetry codes: (i) $x, y, z+1$; (ii) $x-1, -y+3/2, z-1/2$; (iii) $x+1, y, z+1$; (iv) $x, y, z-1$.