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3-Chloro-*N*′-(3-ethoxy-2-hydroxybenzylidene)benzohydrazide monohydrate

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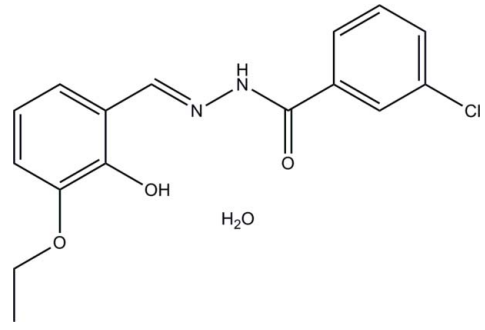
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.067; wR factor = 0.158; data-to-parameter ratio = 15.8.

In the title compound, $C_{16}H_{15}ClN_2O_3\cdot H_2O$, the water molecule is linked to the Schiff base molecule *via* an O—H···O hydrogen bond. In the Schiff base molecule, an intramolecular O—H···N hydrogen bond occurs and the dihedral angle between the two benzene rings is 20.5 (5)°. In the crystal, the Schiff base and water molecules are linked by intermolecular N—H···O and O—H···O hydrogen bonds, forming layers in the *ab* plane.

Related literature

For Schiff base compounds, see: Bessy *et al.* (2006); Podyachev *et al.* (2007); Raj & Kurup (2007); Pouralimardan *et al.* (2007); Bacchi *et al.* (2006); Dinda *et al.* (2002). For reference bond lengths, see: Allen *et al.* (1987). The title compound was prepared by the method described in Zhu (2010).



Experimental

Crystal data

$C_{16}H_{15}ClN_2O_3\cdot H_2O$
M_r = 336.77
Orthorhombic, $P2_12_12_1$
a = 4.631 (2) Å
b = 13.558 (3) Å
c = 25.478 (3) Å
V = 1599.7 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 298 K
0.23 × 0.22 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.943, T_{max} = 0.950
8650 measured reflections
3460 independent reflections
1391 reflections with $I > 2\sigma(I)$
R_{int} = 0.085

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.158
S = 1.00
3460 reflections
219 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack (1983), 1399 Friedel pairs
Flack parameter: 0.25 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···N1	0.82	1.95	2.645 (5)	142
N2—H2···O4^{i}	0.90 (1)	2.03 (1)	2.932 (5)	175 (5)
O4—H4A···O3	0.84 (3)	1.89 (2)	2.717 (5)	167 (5)
O4—H4B···O2^{ii}	0.85 (4)	2.16 (2)	2.945 (5)	154 (4)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; 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: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2396).

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supporting information

Acta Cryst. (2011). E**67**, o377 [doi:10.1107/S160053681100122X]

3-Chloro-*N*′-(3-ethoxy-2-hydroxybenzylidene)benzohydrazide monohydrate

Tian-Yi Li and Peng-Tao Zeng

S1. Comment

In the last few years, a number of Schiff bases derived from the reaction of aldehydes with benzohydrazides were prepared and structurally characterized (Bessy *et al.*, 2006; Podyachev *et al.*, 2007; Raj & Kurup, 2007; Pouralimardan *et al.*, 2007; Bacchi *et al.*, 2006; Dinda *et al.*, 2002). As a continuation of the work, in the present paper, the title new Schiff base compound, Fig. 1, is reported.

The compound contains a Schiff base molecule and a water molecule of crystallization. The water molecule is linked to the Schiff base molecule *via* intermolecular O—H···O hydrogen bonds (Table 1). In the Schiff base molecule, there is an O—H···N hydrogen bond, which contributes to the planarity of the molecule. The dihedral angle between the two benzene rings is 20.5 (5)°. All the bond lengths are within normal values (Allen *et al.*, 1987). The molecules are linked through intermolecular N—H···O and O—H···O hydrogen bonds (Table 1) to form two-dimensional layers along the *ab* plane (Fig. 2).

S2. Experimental

The compound was prepared and crystallized according to the literature method (Zhu, 2010). 3-Ethoxy-2-hydroxy-benzaldehyde (0.166 g, 1 mmol) and 3-chlorobenzohydrazide (0.171 g, 1 mmol) were dissolved in 30 ml 95% ethanol. The mixture was stirred at reflux for 10 min, and cooled to room temperature. The clear colorless solution was left to slow evaporation in air for a week, yielding colorless block-shaped crystals, which were collected by filtration and washed with ethanol.

S3. Refinement

The amino and water H atoms were located from a difference Fourier map and refined isotropically, with the N—H, O—H, and H···H distances restrained to 0.90 (1), 0.85 (1), and 1.37 (2) Å, respectively. The other H atoms were positioned geometrically and refined using the riding-model approximation, with C—H = 0.93–0.97 Å, and O—H = 0.82 Å, and U_{iso}(H) = 1.2U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(C8 and O2).

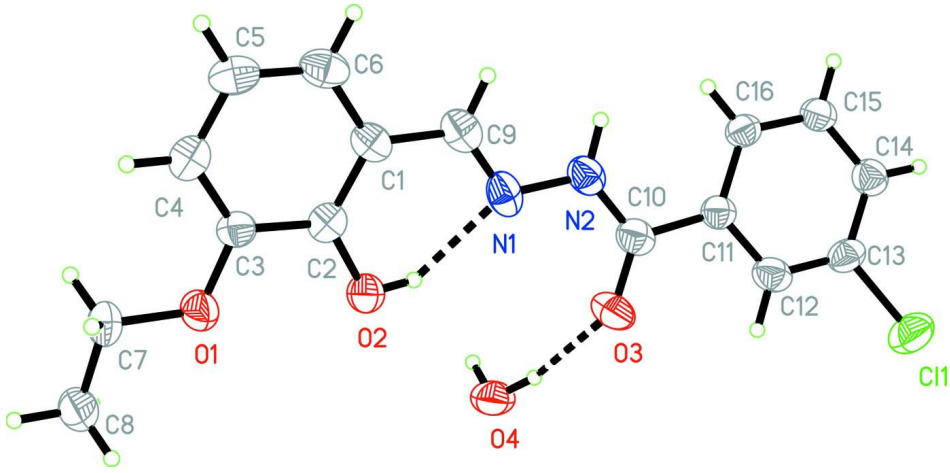

Figure 1

The molecular structure of the title compound with 30% probability displacement ellipsoids for non-hydrogen atoms. Hydrogen bonds are drawn as dashed lines.

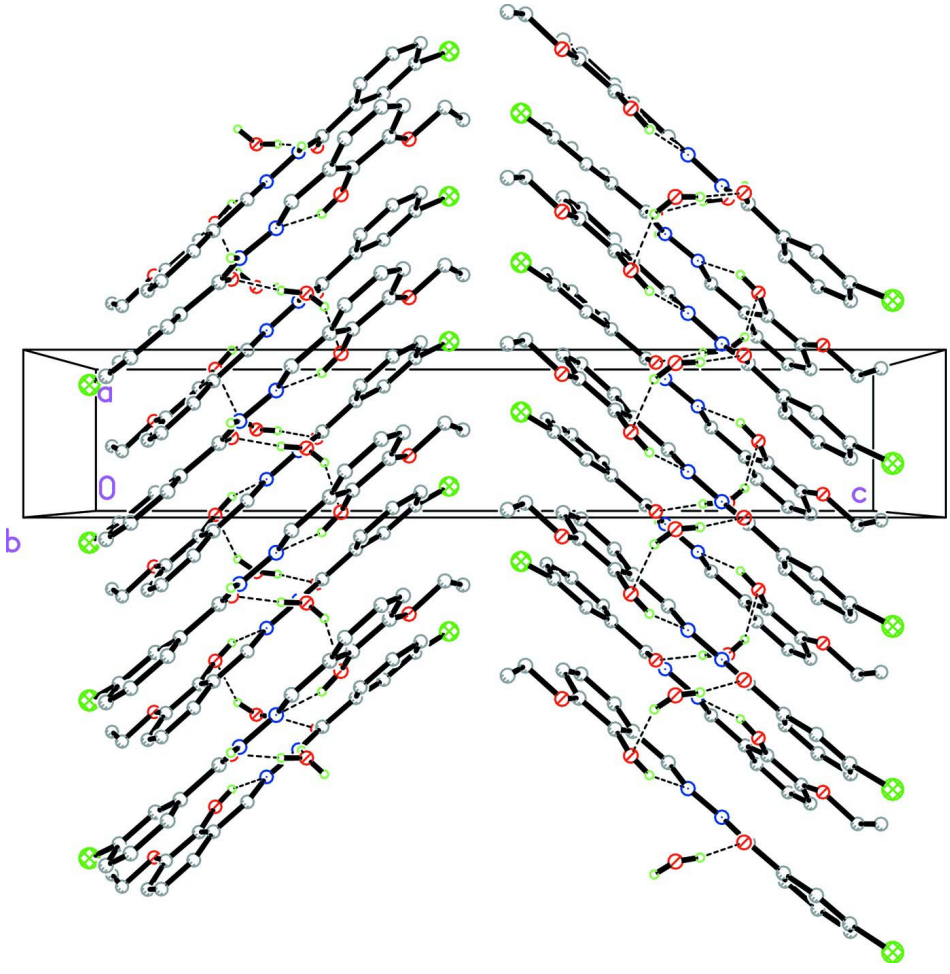

Figure 2

The molecular packing of the title compound. Hydrogen bonds are drawn as dashed lines.

3-Chloro-*N*′-(3-ethoxy-2-hydroxybenzylidene)benzohydrazide monohydrate

Crystal data

$C_{16}H_{15}ClN_2O_3 \cdot H_2O$
$M_r = 336.77$
Orthorhombic, $P2_12_12_1$
$a = 4.631$ (2) Å
$b = 13.558$ (3) Å
$c = 25.478$ (3) Å
$V = 1599.7$ (8) Å^3
$Z = 4$
$F(000) = 704$
$D_x = 1.398$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 705 reflections
$\theta = 2.6$–$24.5°$
$\mu = 0.26$ mm^{-1}
$T = 298$ K
Block, colorless
0.23 × 0.22 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.943$, $T_{max} = 0.950$
8650 measured reflections
3460 independent reflections
1391 reflections with $I > 2\sigma(I)$
$R_{int} = 0.085$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.2°$
$h = -5 \rightarrow 5$
$k = -12 \rightarrow 17$
$l = -32 \rightarrow 29$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.158$
$S = 1.00$
3460 reflections
219 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0181P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Absolute structure: Flack (1983), 1399 Friedel pairs
Absolute structure parameter: 0.25 (16)

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}
Cl1	−0.1754 (4)	0.65889 (10)	0.04444 (6)	0.1007 (7)
N1	0.7745 (10)	0.8171 (3)	0.26638 (17)	0.0663 (13)
N2	0.5846 (11)	0.8403 (3)	0.22645 (18)	0.0638 (13)

O1	1.3750 (8)	0.6775 (2)	0.41318 (14)	0.0680 (10)
O2	1.0189 (9)	0.6980 (2)	0.33554 (13)	0.0656 (10)
H2A	0.8935	0.7128	0.3142	0.098*
O3	0.4905 (10)	0.6809 (3)	0.20923 (14)	0.0907 (13)
O4	0.4271 (12)	0.5540 (2)	0.29094 (15)	0.0892 (14)
C1	1.1199 (12)	0.8733 (4)	0.3283 (2)	0.0550 (14)
C2	1.1601 (12)	0.7804 (4)	0.3510 (2)	0.0541 (13)
C3	1.3572 (13)	0.7715 (4)	0.3925 (2)	0.0560 (14)
C4	1.5152 (12)	0.8508 (4)	0.4093 (2)	0.0636 (14)
H4	1.6523	0.8430	0.4357	0.076*
C5	1.4701 (14)	0.9427 (4)	0.3869 (2)	0.0714 (17)
H5	1.5733	0.9970	0.3989	0.086*
C6	1.2753 (14)	0.9537 (4)	0.3474 (2)	0.0704 (17)
H6	1.2452	1.0158	0.3329	0.085*
C7	1.5824 (12)	0.6631 (4)	0.4544 (2)	0.0704 (16)
H7A	1.5512	0.7105	0.4823	0.084*
H7B	1.7767	0.6717	0.4409	0.084*
C8	1.5442 (15)	0.5608 (3)	0.4746 (2)	0.0836 (19)
H8A	1.3503	0.5527	0.4873	0.125*
H8B	1.6777	0.5493	0.5028	0.125*
H8C	1.5801	0.5144	0.4469	0.125*
C9	0.9160 (13)	0.8890 (4)	0.2864 (2)	0.0655 (17)
H9	0.8865	0.9525	0.2737	0.079*
C10	0.4512 (14)	0.7692 (4)	0.1998 (2)	0.0634 (16)
C11	0.2476 (12)	0.7998 (4)	0.1579 (2)	0.0538 (13)
C12	0.1420 (14)	0.7273 (4)	0.1251 (2)	0.0660 (16)
H12	0.1992	0.6622	0.1297	0.079*
C13	−0.0471 (14)	0.7514 (4)	0.0857 (2)	0.0619 (16)
C14	−0.1410 (12)	0.8456 (4)	0.0778 (2)	0.0639 (15)
H14	−0.2718	0.8602	0.0512	0.077*
C15	−0.0362 (13)	0.9190 (4)	0.1104 (2)	0.0612 (15)
H15	−0.0954	0.9839	0.1055	0.073*
C16	0.1542 (13)	0.8968 (3)	0.1499 (2)	0.0568 (14)
H16	0.2222	0.9468	0.1716	0.068*
H2	0.569 (12)	0.9055 (11)	0.2203 (18)	0.080*
H4A	0.437 (12)	0.586 (3)	0.2626 (9)	0.080*
H4B	0.334 (10)	0.589 (3)	0.3130 (13)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1293 (16)	0.0775 (10)	0.0952 (12)	−0.0177 (11)	−0.0022 (11)	−0.0238 (9)
N1	0.062 (3)	0.088 (3)	0.049 (3)	0.024 (3)	0.009 (3)	0.009 (3)
N2	0.065 (4)	0.066 (3)	0.060 (3)	0.015 (3)	0.004 (3)	0.008 (3)
O1	0.066 (3)	0.062 (2)	0.076 (2)	−0.006 (2)	−0.018 (2)	0.0077 (19)
O2	0.059 (3)	0.063 (2)	0.074 (3)	0.001 (2)	−0.017 (2)	−0.0018 (19)
O3	0.124 (4)	0.061 (2)	0.086 (3)	0.020 (3)	−0.003 (3)	0.018 (2)
O4	0.138 (4)	0.055 (2)	0.075 (3)	0.009 (3)	0.017 (3)	0.004 (2)

C1	0.040 (3)	0.061 (3)	0.064 (4)	0.011 (3)	0.012 (3)	0.006 (3)
C2	0.041 (3)	0.063 (3)	0.058 (3)	−0.002 (3)	0.008 (3)	−0.005 (3)
C3	0.054 (4)	0.051 (3)	0.063 (4)	0.001 (3)	0.001 (3)	−0.004 (3)
C4	0.055 (4)	0.076 (4)	0.060 (3)	−0.006 (4)	0.007 (3)	−0.007 (3)
C5	0.068 (5)	0.057 (4)	0.089 (5)	−0.003 (3)	0.017 (4)	−0.011 (3)
C6	0.071 (5)	0.058 (3)	0.083 (5)	0.010 (4)	0.021 (4)	0.006 (3)
C7	0.063 (4)	0.079 (4)	0.069 (4)	0.001 (3)	−0.022 (4)	−0.003 (3)
C8	0.095 (5)	0.069 (4)	0.087 (4)	−0.007 (4)	−0.028 (4)	0.011 (3)
C9	0.056 (5)	0.076 (4)	0.064 (4)	0.016 (3)	0.012 (3)	0.010 (3)
C10	0.071 (5)	0.060 (4)	0.060 (4)	0.007 (4)	0.015 (3)	0.007 (3)
C11	0.058 (4)	0.051 (3)	0.053 (3)	0.001 (3)	0.008 (3)	0.002 (3)
C12	0.073 (4)	0.056 (3)	0.069 (4)	0.012 (4)	0.013 (4)	0.005 (3)
C13	0.070 (4)	0.052 (3)	0.063 (4)	−0.013 (3)	0.008 (4)	−0.008 (3)
C14	0.064 (4)	0.065 (3)	0.063 (4)	0.006 (4)	0.001 (3)	0.003 (3)
C15	0.066 (4)	0.051 (3)	0.067 (4)	0.000 (3)	−0.008 (4)	0.003 (3)
C16	0.066 (4)	0.049 (3)	0.055 (3)	−0.002 (3)	0.007 (3)	−0.005 (3)

Geometric parameters (Å, º)

Cl1—C13	1.741 (5)	C5—H5	0.9300
N1—C9	1.281 (6)	C6—H6	0.9300
N1—N2	1.381 (6)	C7—C8	1.491 (6)
N2—C10	1.331 (6)	C7—H7A	0.9700
N2—H2	0.901 (10)	C7—H7B	0.9700
O1—C3	1.381 (5)	C8—H8A	0.9600
O1—C7	1.436 (5)	C8—H8B	0.9600
O2—C2	1.354 (5)	C8—H8C	0.9600
O2—H2A	0.8200	C9—H9	0.9300
O3—C10	1.234 (5)	C10—C11	1.485 (7)
O4—H4A	0.84 (3)	C11—C12	1.379 (6)
O4—H4B	0.85 (4)	C11—C16	1.399 (6)
C1—C6	1.394 (7)	C12—C13	1.371 (7)
C1—C2	1.399 (6)	C12—H12	0.9300
C1—C9	1.440 (7)	C13—C14	1.365 (6)
C2—C3	1.402 (7)	C14—C15	1.384 (6)
C3—C4	1.369 (6)	C14—H14	0.9300
C4—C5	1.386 (7)	C15—C16	1.370 (7)
C4—H4	0.9300	C15—H15	0.9300
C5—C6	1.360 (7)	C16—H16	0.9300
C9—N1—N2	116.5 (5)	C7—C8—H8A	109.5
C10—N2—N1	120.4 (4)	C7—C8—H8B	109.5
C10—N2—H2	126 (3)	H8A—C8—H8B	109.5
N1—N2—H2	114 (4)	C7—C8—H8C	109.5
C3—O1—C7	116.4 (4)	H8A—C8—H8C	109.5
C2—O2—H2A	109.5	H8B—C8—H8C	109.5
H4A—O4—H4B	108 (2)	N1—C9—C1	121.2 (5)
C6—C1—C2	119.3 (5)	N1—C9—H9	119.4

C6—C1—C9	118.8 (5)	C1—C9—H9	119.4
C2—C1—C9	121.8 (5)	O3—C10—N2	122.4 (6)
O2—C2—C1	123.9 (5)	O3—C10—C11	120.3 (6)
O2—C2—C3	117.6 (5)	N2—C10—C11	117.3 (5)
C1—C2—C3	118.5 (5)	C12—C11—C16	118.1 (5)
C4—C3—O1	125.0 (5)	C12—C11—C10	117.5 (5)
C4—C3—C2	121.1 (5)	C16—C11—C10	124.4 (5)
O1—C3—C2	113.9 (5)	C13—C12—C11	120.0 (5)
C3—C4—C5	119.8 (5)	C13—C12—H12	120.0
C3—C4—H4	120.1	C11—C12—H12	120.0
C5—C4—H4	120.1	C14—C13—C12	122.3 (5)
C6—C5—C4	120.2 (6)	C14—C13—Cl1	118.4 (5)
C6—C5—H5	119.9	C12—C13—Cl1	119.3 (4)
C4—C5—H5	119.9	C13—C14—C15	118.2 (5)
C5—C6—C1	121.1 (5)	C13—C14—H14	120.9
C5—C6—H6	119.5	C15—C14—H14	120.9
C1—C6—H6	119.5	C16—C15—C14	120.6 (5)
O1—C7—C8	107.5 (4)	C16—C15—H15	119.7
O1—C7—H7A	110.2	C14—C15—H15	119.7
C8—C7—H7A	110.2	C15—C16—C11	120.8 (5)
O1—C7—H7B	110.2	C15—C16—H16	119.6
C8—C7—H7B	110.2	C11—C16—H16	119.6
H7A—C7—H7B	108.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N1	0.82	1.95	2.645 (6)	142
N2—H2···O4^{i}	0.90 (1)	2.03 (1)	2.932 (5)	175 (5)
O4—H4*A*···O3	0.84 (3)	1.89 (2)	2.717 (5)	167 (5)
O4—H4*B*···O2^{ii}	0.85 (4)	2.16 (2)	2.945 (5)	154 (4)

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