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2′-(3-Hydroxybenzylidene)pyrazine-2-carbohydrazide monohydrate

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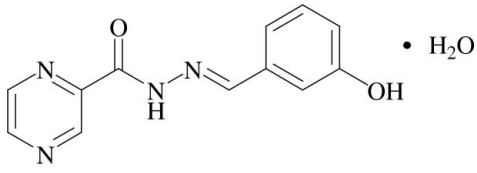
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.051; wR factor = 0.140; data-to-parameter ratio = 11.3.

The title compound, $C_{12}H_{10}N_4O_2\cdot H_2O$, was synthesized by the reaction of pyrazine-2-carboxylic acid hydrazide and 3-hydroxybenzaldehyde in ethanol. In the crystal structure, the organic molecules are linked into extended chains by intermolecular N(amide)—H···O(hydroxy) hydrogen bonds. Additional hydrogen bonds between the water molecule and three adjacent organic molecules, as well as face-to-face π–π stacking interactions between the benzene and pyrazine rings [centroid-to-centroid separation = 3.669 (2) Å and offset = 1.362 Å], link the molecules into a three-dimensional framework.

Related literature

For pharmacological and photochromic properties of hydrazonecarbonyl compounds, see: Parashar *et al.* (1988); Hadjoudis *et al.* (1987). For related pyrazinecarboxylic acid hydrazones, see: Gardner *et al.* (1956);



Experimental

Crystal data

$C_{12}H_{10}N_4O_2\cdot H_2O$
M_r = 260.26
Triclinic, $P\overline{1}$
a = 8.062 (3) Å
b = 8.186 (3) Å
c = 9.449 (3) Å
α = 95.122 (6)°
β = 103.169 (6)°
γ = 99.827 (7)°
V = 592.9 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 294 (2) K
0.24 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.962, T_{max} = 0.981
3017 measured reflections
2083 independent reflections
1112 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.140
S = 1.02
2083 reflections
184 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3^{i}	0.86 (4)	1.78 (4)	2.634 (3)	174 (4)
N3—H3···N1	0.82 (3)	2.31 (3)	2.705 (3)	110 (3)
N3—H3···O2^{ii}	0.82 (3)	2.51 (3)	3.218 (4)	145 (3)
O3—H3*B*···N2^{iii}	0.86 (2)	2.04 (3)	2.873 (3)	163 (4)
O3—H3*C*···N4	0.89 (2)	2.22 (3)	3.043 (3)	152 (3)
O3—H3*C*···O1	0.89 (2)	2.29 (3)	2.982 (3)	134 (3)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LN2011).

References

Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Gardner, T. S., Smith, F. A., Wenis, E. & Lee, J. (1956). *J. Org. Chem.* **21**, 530–533.
Hadjoudis, E., Vittorakis, M. & Moustakali-Mavridis, I. (1987). *Tetrahedron*, **43**, 1345–1360.
Parashar, R. K., Sharma, R. C., Kumar, A. & Mohan, G. (1988). *Inorg. Chim. Acta*, **151**, 201–208.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supporting information

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2′-(3-Hydroxybenzylidene)pyrazine-2-carbohydrazide monohydrate

Lei He

S1. Comment

Hydrazonecarbonyl compounds have received considerable attention due to their pharmacological activities (Parashar *et al.*, 1988) and photochromic properties (Hadjoudis *et al.*, 1987). A series of similar pyrazinylcarboxylic acid hydrazones has been reported previously (Gardner *et al.*, 1956). As a continuation of the work of our group, we report here the crystal structure of 2′-(3-hydroxybenzylidene)pyrazine-2-carbohydrazide monohydrate, (I).

The asymmetric unit contains one organic molecule and one molecule of H_2O. The carbohydrazide molecule deviates only slightly from planarity with a dihedral angle of 11.60 (3)° between the planes of the pyrazinyl and phenyl rings (Fig. 1).

In the crystal, the organic molecules of (I) are arranged in slightly tilted rows. Within the rows, amide atom N3 acts as a hydrogen bond donor to the atom O2 of the hydroxy group in a neighbouring molecule, thereby forming extended chains, which run parallel to the *a* axis (Table 1, Fig. 2).

The water O atom acts as a hydrogen bond acceptor from the hydroxy group of an organic molecule. One water H atom forms a hydrogen bond with the N atom in the pyrazinyl ring of another adjacent molecule, while the other H atom forms bifurcated hydrogen bonds with the carbonyl O atom, O1, and the N4 atom from another molecule of I, thereby forming a five-membered hydrogen bond ring (Fig. 3).

The crystal packing is characterized by $\pi\cdots\pi$ stacking interactions. The molecules are stacked in an antiparalled fashion, with a pyrazinyl to phenyl ring centroid-centroid separation of 3.669 (2) Å and an offset of 1.362 Å. Together with the hydrogen bonds, these interactions lead to a three-dimensional supramolecular network pattern (Fig. 3).

S2. Experimental

Compound (I) was synthesized by the reaction of pyrazine-2-carboxylic acid hydrazide (0.01 mol, 1.38 g) and 3-hydroxybenzaldehyde (0.01 mol, 1.52 g) in ethanol. The solution was refluxed for 3 h. After cooling down, the solid product was filtered and recrystallized from ethanol with a yield of 75%, m.p. 540–541 K. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a mixture of methanol and water (5: 1).

S3. Refinement

All H atoms were initially located in a difference Fourier map. The carbon bound H atoms were then constrained to an ideal geometry, with *C*(phenyl)—H distances of 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). The H atoms attached to the hydroxy and water O atoms and to the N atom were refined freely with U_{iso}(H) = 1.5U_{eq}(O) and 1.2U_{eq}(N), respectively.

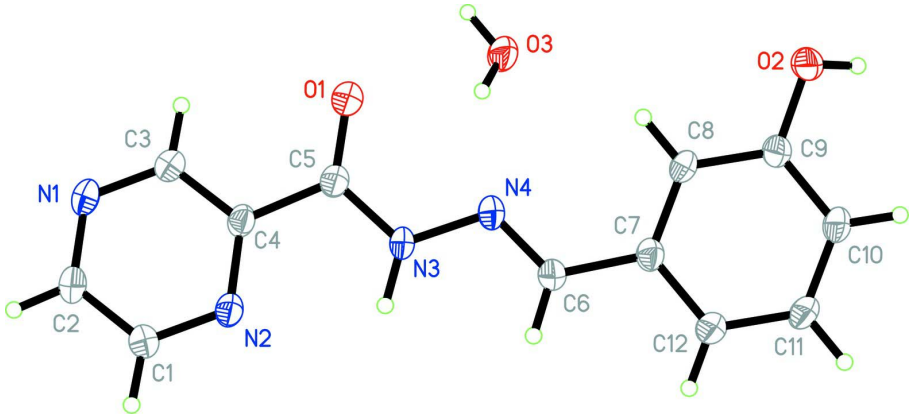

Figure 1

The structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

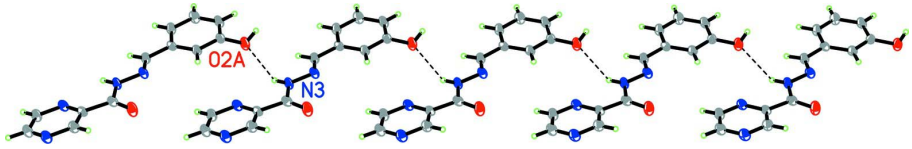

Figure 2

View along the *c* axis showing a one-dimensional chain of the organic molecule. The water molecules have been omitted for clarity. Dashed lines represent the hydrogen bonds. Symmetry code: (A) *x*, *y* - 1, *z*.

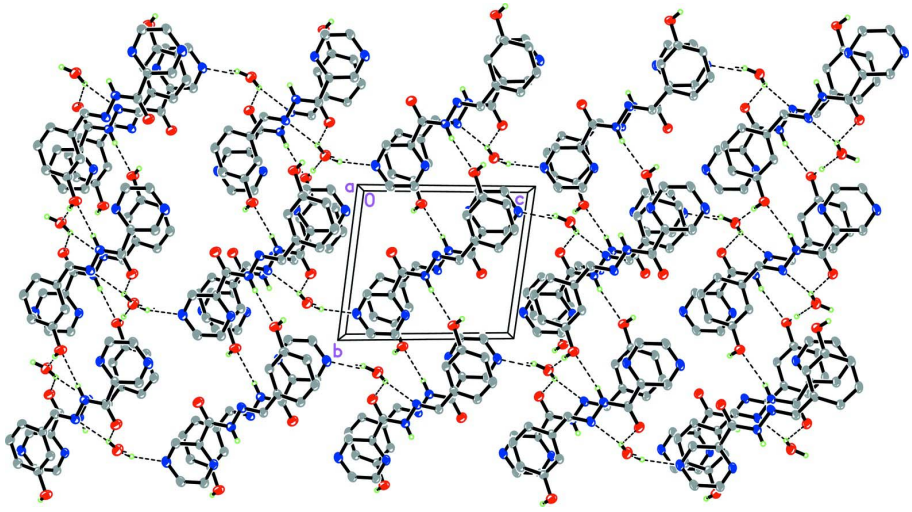

Figure 3

The crystal packing viewed down the *a* axis showing the three-dimensional structure formed by hydrogen bonds (dashed lines) and $\pi\cdots\pi$ stacking interactions.

2′-(3-Hydroxybenzylidene)pyrazine-2-carbohydrazide monohydrate

Crystal data

$C_{12}H_{10}N_4O_2\cdot H_2O$
$M_r = 260.26$
Triclinic, $P\overline{1}$

$a = 8.062\ (3)$ Å
$b = 8.186\ (3)$ Å
$c = 9.449\ (3)$ Å

α = 95.122 (6)°
β = 103.169 (6)°
γ = 99.827 (7)°
V = 592.9 (4) Å^3
Z = 2
F(000) = 272
D_x = 1.458 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 658 reflections
θ = 2.2–23.0°
μ = 0.11 mm^{-1}
T = 294 K
Block, colourless
0.24 × 0.22 × 0.18 mm

Data collection

Bruker CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.962, T_{max} = 0.981

3017 measured reflections
2083 independent reflections
1112 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 25.0°, θ_{min} = 2.2°
h = −9→9
k = −9→4
l = −10→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.140
S = 1.02
2083 reflections
184 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 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	0.4727 (3)	0.5725 (3)	0.7739 (2)	0.0587 (7)
O2	0.0678 (3)	1.0884 (3)	0.3575 (3)	0.0567 (7)
H2	−0.014 (5)	1.134 (5)	0.314 (4)	0.085*
N1	0.4525 (3)	0.1480 (3)	0.6449 (2)	0.0392 (7)
N2	0.6326 (3)	0.1543 (3)	0.9355 (3)	0.0472 (7)
N3	0.3179 (4)	0.4148 (3)	0.5600 (3)	0.0397 (7)
H3	0.299 (4)	0.322 (4)	0.513 (3)	0.048*
N4	0.2524 (3)	0.5480 (3)	0.5068 (3)	0.0402 (7)
C1	0.5113 (4)	0.0166 (4)	0.6931 (3)	0.0448 (9)

H1	0.4921	−0.0808	0.6281	0.054*
C2	0.6005 (4)	0.0195 (4)	0.8370 (3)	0.0465 (9)
H2A	0.6393	−0.0760	0.8658	0.056*
C3	0.5740 (4)	0.2865 (4)	0.8871 (3)	0.0444 (8)
H3A	0.5930	0.3836	0.9525	0.053*
C4	0.4858 (4)	0.2849 (4)	0.7429 (3)	0.0337 (7)
C5	0.4252 (4)	0.4375 (4)	0.6954 (3)	0.0398 (8)
C6	0.1605 (4)	0.5199 (4)	0.3753 (3)	0.0388 (8)
H6	0.1440	0.4139	0.3235	0.047*
C7	0.0801 (4)	0.6459 (4)	0.3024 (3)	0.0355 (8)
C8	−0.0298 (4)	0.5994 (4)	0.1629 (3)	0.0434 (9)
H8	−0.0512	0.4892	0.1188	0.052*
C9	−0.1069 (4)	0.7147 (4)	0.0899 (3)	0.0501 (9)
H9	−0.1803	0.6823	−0.0034	0.060*
C10	−0.0764 (4)	0.8785 (4)	0.1537 (3)	0.0450 (9)
H10	−0.1286	0.9565	0.1035	0.054*
C11	0.0317 (4)	0.9261 (4)	0.2922 (3)	0.0378 (8)
C12	0.1093 (4)	0.8114 (4)	0.3664 (3)	0.0385 (8)
H12	0.1818	0.8444	0.4600	0.046*
O3	0.1948 (3)	0.7733 (3)	0.7579 (2)	0.0590 (7)
H3B	0.242 (5)	0.816 (4)	0.847 (3)	0.088*
H3C	0.246 (5)	0.708 (4)	0.708 (4)	0.088*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0769 (18)	0.0433 (15)	0.0455 (14)	0.0237 (13)	−0.0117 (13)	−0.0016 (12)
O2	0.0651 (17)	0.0367 (14)	0.0564 (15)	0.0197 (12)	−0.0147 (12)	0.0016 (11)
N1	0.0441 (16)	0.0446 (17)	0.0289 (14)	0.0186 (14)	0.0008 (12)	0.0061 (13)
N2	0.0529 (18)	0.0528 (19)	0.0355 (16)	0.0205 (15)	−0.0003 (13)	0.0138 (14)
N3	0.0492 (17)	0.0342 (16)	0.0355 (16)	0.0202 (15)	0.0004 (13)	0.0076 (12)
N4	0.0455 (16)	0.0401 (16)	0.0378 (16)	0.0203 (14)	0.0037 (13)	0.0139 (13)
C1	0.055 (2)	0.044 (2)	0.0365 (19)	0.0190 (18)	0.0060 (16)	0.0072 (16)
C2	0.055 (2)	0.048 (2)	0.040 (2)	0.0218 (19)	0.0064 (17)	0.0158 (17)
C3	0.051 (2)	0.045 (2)	0.0348 (19)	0.0150 (17)	0.0024 (16)	0.0059 (16)
C4	0.0332 (17)	0.0430 (19)	0.0275 (17)	0.0159 (15)	0.0051 (14)	0.0083 (15)
C5	0.0407 (19)	0.046 (2)	0.0343 (19)	0.0171 (17)	0.0045 (16)	0.0085 (17)
C6	0.047 (2)	0.0365 (19)	0.0335 (19)	0.0132 (16)	0.0068 (16)	0.0080 (14)
C7	0.0358 (18)	0.0397 (19)	0.0338 (18)	0.0164 (16)	0.0049 (15)	0.0120 (15)
C8	0.056 (2)	0.0376 (19)	0.0332 (19)	0.0139 (17)	0.0012 (16)	0.0039 (15)
C9	0.063 (2)	0.051 (2)	0.0303 (18)	0.0224 (19)	−0.0075 (16)	0.0037 (16)
C10	0.051 (2)	0.047 (2)	0.0360 (19)	0.0238 (18)	−0.0039 (16)	0.0116 (16)
C11	0.0393 (19)	0.0331 (19)	0.0390 (19)	0.0111 (16)	0.0025 (15)	0.0059 (15)
C12	0.0388 (19)	0.042 (2)	0.0324 (18)	0.0129 (16)	−0.0016 (15)	0.0079 (15)
O3	0.0716 (18)	0.0561 (17)	0.0466 (15)	0.0334 (14)	−0.0036 (13)	0.0000 (13)

Geometric parameters (Å, º)

O1—C5	1.224 (4)	C4—C5	1.486 (4)
O2—C11	1.368 (4)	C6—C7	1.453 (4)
O2—H2	0.86 (4)	C6—H6	0.9300
N1—C1	1.324 (4)	C7—C8	1.390 (4)
N1—C4	1.336 (3)	C7—C12	1.391 (4)
N2—C2	1.327 (4)	C8—C9	1.369 (4)
N2—C3	1.330 (4)	C8—H8	0.9300
N3—C5	1.348 (4)	C9—C10	1.377 (4)
N3—N4	1.376 (3)	C9—H9	0.9300
N3—H3	0.82 (3)	C10—C11	1.377 (4)
N4—C6	1.272 (3)	C10—H10	0.9300
C1—C2	1.382 (4)	C11—C12	1.373 (4)
C1—H1	0.9300	C12—H12	0.9300
C2—H2A	0.9300	O3—H3B	0.86 (2)
C3—C4	1.384 (4)	O3—H3C	0.89 (2)
C3—H3A	0.9300		
C11—O2—H2	106 (3)	N4—C6—C7	122.9 (3)
C1—N1—C4	116.3 (3)	N4—C6—H6	118.5
C2—N2—C3	115.9 (3)	C7—C6—H6	118.5
C5—N3—N4	119.2 (3)	C8—C7—C12	118.7 (3)
C5—N3—H3	117 (2)	C8—C7—C6	118.7 (3)
N4—N3—H3	124 (2)	C12—C7—C6	122.6 (3)
C6—N4—N3	115.7 (3)	C9—C8—C7	120.5 (3)
N1—C1—C2	122.1 (3)	C9—C8—H8	119.8
N1—C1—H1	118.9	C7—C8—H8	119.8
C2—C1—H1	118.9	C8—C9—C10	120.4 (3)
N2—C2—C1	122.1 (3)	C8—C9—H9	119.8
N2—C2—H2A	119.0	C10—C9—H9	119.8
C1—C2—H2A	119.0	C9—C10—C11	119.7 (3)
N2—C3—C4	122.4 (3)	C9—C10—H10	120.1
N2—C3—H3A	118.8	C11—C10—H10	120.1
C4—C3—H3A	118.8	O2—C11—C12	118.5 (3)
N1—C4—C3	121.3 (3)	O2—C11—C10	121.2 (3)
N1—C4—C5	119.0 (3)	C12—C11—C10	120.3 (3)
C3—C4—C5	119.7 (3)	C11—C12—C7	120.4 (3)
O1—C5—N3	123.6 (3)	C11—C12—H12	119.8
O1—C5—C4	121.7 (3)	C7—C12—H12	119.8
N3—C5—C4	114.7 (3)	H3B—O3—H3C	122 (4)
C5—N3—N4—C6	176.0 (3)	C3—C4—C5—N3	−170.0 (3)
C4—N1—C1—C2	0.8 (4)	N3—N4—C6—C7	179.4 (3)
C3—N2—C2—C1	−0.2 (5)	N4—C6—C7—C8	−174.7 (3)
N1—C1—C2—N2	0.0 (5)	N4—C6—C7—C12	5.6 (5)
C2—N2—C3—C4	−0.3 (5)	C12—C7—C8—C9	0.3 (4)
C1—N1—C4—C3	−1.3 (4)	C6—C7—C8—C9	−179.4 (3)

C1—N1—C4—C5	179.2 (3)	C7—C8—C9—C10	0.0 (5)
N2—C3—C4—N1	1.1 (5)	C8—C9—C10—C11	−0.3 (5)
N2—C3—C4—C5	−179.4 (3)	C9—C10—C11—O2	178.6 (3)
N4—N3—C5—O1	−2.0 (5)	C9—C10—C11—C12	0.2 (5)
N4—N3—C5—C4	179.0 (3)	O2—C11—C12—C7	−178.2 (3)
N1—C4—C5—O1	−169.5 (3)	C10—C11—C12—C7	0.2 (5)
C3—C4—C5—O1	11.0 (5)	C8—C7—C12—C11	−0.5 (4)
N1—C4—C5—N3	9.5 (4)	C6—C7—C12—C11	179.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O3^{i}	0.86 (4)	1.78 (4)	2.634 (3)	174 (4)
N3—H3···N1	0.82 (3)	2.31 (3)	2.705 (3)	110 (3)
N3—H3···O2^{ii}	0.82 (3)	2.51 (3)	3.218 (4)	145 (3)
O3—H3*B*···N2^{iii}	0.86 (2)	2.04 (3)	2.873 (3)	163 (4)
O3—H3*C*···N4	0.89 (2)	2.22 (3)	3.043 (3)	152 (3)
O3—H3*C*···O1	0.89 (2)	2.29 (3)	2.982 (3)	134 (3)

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