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5-Acetyl-3-(5-phenyl-1*H*-pyrazol-3-yl)-1,3,4-thiadiazol-2(3*H*)-one monohydrate

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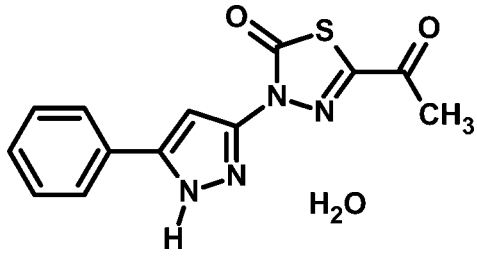
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.106; data-to-parameter ratio = 11.8.

In the title hydrate, $C_{13}H_{10}N_4O_2S\cdot H_2O$, the dihedral angles between the central pyrazole ring and its pendant phenyl and thiadiazole rings are 9.93 (8) and 4.56 (7)°, respectively. In the crystal, the components are linked by N—H···O, O—H···N and O—H···O hydrogen bonds, generating [100] chains incorporating $R_4^4(10)$ loops. A weak C—H···O interaction helps to consolidate the packing.

Related literature

For the synthesis of the title compound, see: Abdelhamid *et al.* (2001). For a related structure, see: Ge (2006).



Experimental

Crystal data

$C_{13}H_{10}N_4O_2S\cdot H_2O$ M_r = 304.33
Monoclinic, $P2_1/n$
a = 7.6084 (2) Å
b = 25.5788 (4) Å
c = 7.6524 (2) Å
β = 111.974 (3)°
V = 1381.07 (6) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.25 mm^{-1}
T = 296 K
0.35 × 0.19 × 0.06 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas, CCD) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.778, T_{max} = 1.000
10797 measured reflections
2806 independent reflections
2498 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.106
S = 1.07
2806 reflections
238 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3W^{i}	0.93 (2)	1.83 (2)	2.749 (2)	173 (2)
O3W—H3A···N2^{ii}	0.86 (3)	1.99 (3)	2.8402 (19)	169 (2)
O3W—H3B···O1^{iii}	0.83 (5)	2.19 (5)	2.995 (2)	163 (4)
C8—H8···O1^{iv}	0.93 (2)	2.50 (2)	3.4100 (19)	167.8 (15)

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

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *X-SEED* (Barbour, 2001).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7073).

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supporting information

Acta Cryst. (2013). E**69**, o798 [https://doi.org/10.1107/S1600536813010817]

5-Acetyl-3-(5-phenyl-1*H*-pyrazol-3-yl)-1,3,4-thiadiazol-2(3*H*)-one monohydrate

Abdullah M. Asiri, Muhammad Nadeem Arshad, Abdullah Y. Obaid and Ghulam Mustafa

S1. Comment

In the title compound (I), shown in Fig. 1, the aromatic ring is inclined at angles of 9.93 (8)° & 6.36 (7)° with respect to pyrazole and thiadiazole rings. The pyrazole and thiadiazole rings are oriented at dihedral angle of 4.56 (7)°. The acetyl group is inclined at dihedral angle of 6.00 (2)° with thiadiazole ring. The water molecule as usual is involved in classical hydrogen bonding interactions. The interactions through N1—H1···O3w, and O3w—H3a···N2 give rise to inversion dimers forming ten membered ring motif $R_4^4(10)$. These dimers further connected through O3w—H3b···O1 interaction to make infinite one dimensional chain along *a* axis. Another non-classical interaction (C8—H8···O1) generates twelve membered ring motif $R_1^1(12)$ loops; for symmetry detail, see; Table. 1, Fig. 2.

S2. Experimental

The title compound was synthesised according to literature (Abdelhamid *et al.*, 2001) procedure and recrystalized from methanol solution under slow evaporation to yield brown plates.

S3. Refinement

All the hydrogen atoms found from a Fourier difference map and allowed to refine freely with appropriate riding models.

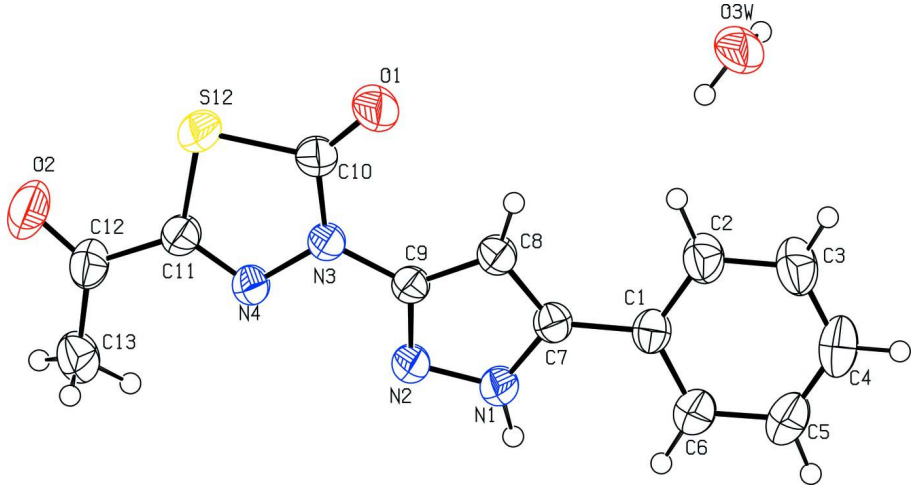

Figure 1

The molecular structure of title compound with 50% probability displacement ellipsoids.

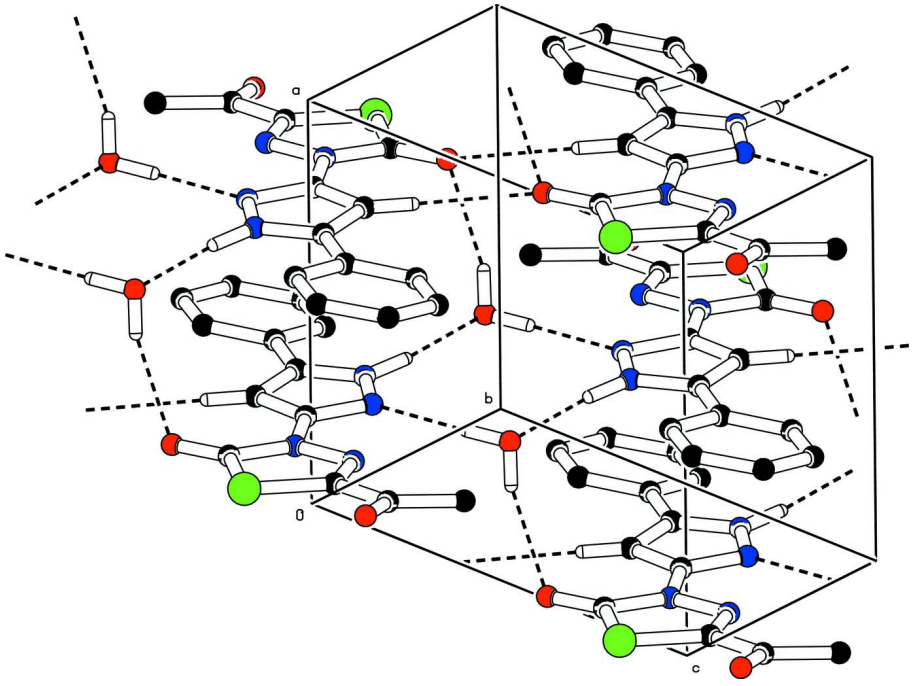

Figure 2

The packing diagram showing inversion dimers through hydrogen bonds, drawn using dashed lines.

5-Acetyl-3-(5-phenyl-1*H*-pyrazol-3-yl)-1,3,4-thiadiazol-2(3*H*)-one monohydrate

Crystal data

$C_{13}H_{10}N_4O_2S·H_2O$
$M_r = 304.33$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.6084$ (2) Å
$b = 25.5788$ (4) Å
$c = 7.6524$ (2) Å
$\beta = 111.974$ (3)°
$V = 1381.07$ (6) Å^3
$Z = 4$
$F(000) = 632$
$D_x = 1.464$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 5915 reflections
$\theta = 3.5$–$74.7°$
$\mu = 2.25$ mm^{-1}
$T = 296$ K
Plate, brown
0.35 × 0.19 × 0.06 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas, CCD) diffractometer
Radiation source: SuperNova (Cu) X-ray Source
Mirror monochromator
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
$T_{min} = 0.778$, $T_{max} = 1.000$
10797 measured reflections
2806 independent reflections
2498 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 74.8°$, $\theta_{min} = 3.5°$
$h = -9 \rightarrow 9$
$k = -31 \rightarrow 31$
$l = -9 \rightarrow 6$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.106$
$S = 1.07$
2806 reflections
238 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
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 0.1856P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ 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.

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 $F^2 > 2$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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S12	0.59223 (6)	0.644569 (15)	0.75319 (6)	0.05072 (15)
O1	0.5175 (2)	0.55662 (5)	0.90067 (17)	0.0659 (4)
N3	0.40123 (17)	0.56623 (4)	0.57705 (17)	0.0362 (3)
O2	0.5725 (2)	0.72711 (5)	0.4716 (2)	0.0757 (4)
N4	0.39508 (17)	0.60041 (4)	0.43842 (17)	0.0375 (3)
N1	0.11453 (18)	0.46444 (5)	0.33689 (18)	0.0408 (3)
N2	0.19673 (18)	0.51164 (5)	0.34177 (17)	0.0398 (3)
C10	0.4991 (2)	0.58140 (6)	0.7612 (2)	0.0439 (3)
C7	0.15808 (19)	0.44351 (5)	0.5095 (2)	0.0361 (3)
C9	0.29698 (18)	0.51911 (5)	0.52301 (19)	0.0338 (3)
C11	0.4876 (2)	0.64236 (5)	0.5106 (2)	0.0398 (3)
C1	0.08453 (19)	0.39258 (5)	0.5408 (2)	0.0383 (3)
C8	0.2800 (2)	0.47822 (5)	0.6365 (2)	0.0387 (3)
C12	0.4979 (2)	0.68747 (6)	0.3918 (3)	0.0487 (4)
C2	0.1125 (2)	0.37673 (7)	0.7226 (3)	0.0505 (4)
H2	0.177 (3)	0.3992 (9)	0.834 (3)	0.068 (6)*
C6	−0.0130 (2)	0.35941 (6)	0.3906 (3)	0.0480 (4)
H6	−0.037 (3)	0.3691 (8)	0.264 (3)	0.052 (5)*
C13	0.4163 (3)	0.68076 (8)	0.1839 (3)	0.0589 (5)
H13B	0.364 (5)	0.7089 (14)	0.126 (5)	0.116 (11)*
H13C	0.501 (6)	0.6663 (17)	0.145 (7)	0.165 (17)*
H13A	0.313 (4)	0.6562 (12)	0.135 (4)	0.099 (9)*
C4	−0.0469 (3)	0.29574 (6)	0.6054 (3)	0.0599 (5)
H4	−0.085 (3)	0.2633 (9)	0.629 (3)	0.071 (6)*
C3	0.0476 (3)	0.32821 (7)	0.7542 (3)	0.0600 (5)

H3	0.071 (3)	0.3194 (9)	0.876 (4)	0.072 (7)*
C5	−0.0788 (3)	0.31131 (6)	0.4244 (3)	0.0570 (4)
H5	−0.140 (4)	0.2917 (9)	0.319 (4)	0.078 (7)*
H1	0.025 (3)	0.4545 (8)	0.221 (3)	0.059 (6)*
H8	0.337 (3)	0.4743 (7)	0.766 (3)	0.046 (5)*
O3W	0.8281 (2)	0.44007 (6)	0.0009 (2)	0.0683 (4)
H3A	0.816 (4)	0.4505 (10)	−0.110 (4)	0.089 (8)*
H3B	0.722 (6)	0.4442 (16)	0.006 (5)	0.145 (16)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S12	0.0611 (3)	0.0453 (2)	0.0392 (2)	−0.01426 (16)	0.01118 (19)	−0.00717 (15)
O1	0.0838 (9)	0.0722 (8)	0.0307 (6)	−0.0271 (7)	0.0087 (6)	0.0042 (6)
N3	0.0429 (6)	0.0336 (5)	0.0290 (6)	−0.0032 (4)	0.0100 (5)	0.0016 (4)
O2	0.0992 (11)	0.0428 (6)	0.0740 (10)	−0.0249 (7)	0.0196 (8)	−0.0011 (6)
N4	0.0448 (6)	0.0321 (5)	0.0341 (6)	−0.0010 (4)	0.0131 (5)	0.0029 (4)
N1	0.0482 (6)	0.0376 (6)	0.0334 (7)	−0.0080 (5)	0.0116 (5)	−0.0008 (5)
N2	0.0483 (6)	0.0368 (6)	0.0322 (6)	−0.0070 (5)	0.0125 (5)	0.0003 (5)
C10	0.0480 (8)	0.0467 (8)	0.0335 (8)	−0.0085 (6)	0.0114 (6)	−0.0010 (6)
C7	0.0384 (6)	0.0333 (6)	0.0354 (7)	0.0021 (5)	0.0124 (6)	0.0029 (5)
C9	0.0373 (6)	0.0322 (6)	0.0306 (7)	−0.0002 (5)	0.0112 (5)	0.0006 (5)
C11	0.0441 (7)	0.0348 (7)	0.0385 (8)	−0.0018 (5)	0.0133 (6)	−0.0009 (6)
C1	0.0378 (6)	0.0330 (6)	0.0428 (8)	0.0019 (5)	0.0134 (6)	0.0040 (5)
C8	0.0435 (7)	0.0368 (7)	0.0324 (8)	−0.0010 (5)	0.0103 (6)	0.0035 (6)
C12	0.0530 (8)	0.0359 (7)	0.0551 (10)	−0.0045 (6)	0.0179 (7)	0.0042 (7)
C2	0.0521 (8)	0.0465 (8)	0.0471 (10)	−0.0060 (7)	0.0118 (7)	0.0093 (7)
C6	0.0551 (9)	0.0385 (7)	0.0506 (10)	−0.0026 (6)	0.0199 (8)	−0.0019 (7)
C13	0.0727 (12)	0.0511 (10)	0.0503 (11)	−0.0066 (9)	0.0198 (9)	0.0124 (8)
C4	0.0581 (9)	0.0373 (8)	0.0833 (15)	−0.0030 (7)	0.0253 (10)	0.0121 (8)
C3	0.0608 (10)	0.0536 (9)	0.0599 (12)	−0.0052 (8)	0.0161 (9)	0.0203 (8)
C5	0.0623 (10)	0.0363 (8)	0.0720 (13)	−0.0058 (7)	0.0246 (9)	−0.0068 (8)
O3W	0.0807 (10)	0.0829 (10)	0.0325 (7)	−0.0270 (8)	0.0112 (6)	0.0031 (6)

Geometric parameters (Å, º)

S12—C11	1.7254 (16)	C1—C6	1.398 (2)
S12—C10	1.7744 (15)	C8—H8	0.92 (2)
O1—C10	1.2038 (19)	C12—C13	1.486 (3)
N3—N4	1.3619 (16)	C2—C3	1.390 (2)
N3—C10	1.3804 (19)	C2—H2	0.99 (2)
N3—C9	1.4170 (17)	C6—C5	1.388 (2)
O2—C12	1.210 (2)	C6—H6	0.95 (2)
N4—C11	1.2887 (18)	C13—H13B	0.86 (4)
N1—C7	1.3466 (19)	C13—H13C	0.89 (5)
N1—N2	1.3538 (16)	C13—H13A	0.96 (3)
N1—H1	0.93 (2)	C4—C5	1.372 (3)
N2—C9	1.3229 (19)	C4—C3	1.375 (3)

C7—C8	1.385 (2)	C4—H4	0.92 (2)
C7—C1	1.4724 (18)	C3—H3	0.91 (3)
C9—C8	1.3960 (19)	C5—H5	0.92 (3)
C11—C12	1.489 (2)	O3W—H3A	0.86 (3)
C1—C2	1.387 (2)	O3W—H3B	0.83 (4)

C11—S12—C10	88.73 (7)	C9—C8—H8	129.2 (12)
N4—N3—C10	117.57 (11)	O2—C12—C13	124.48 (16)
N4—N3—C9	117.81 (11)	O2—C12—C11	117.62 (16)
C10—N3—C9	124.49 (12)	C13—C12—C11	117.90 (14)
C11—N4—N3	110.27 (12)	C1—C2—C3	120.31 (17)
C7—N1—N2	112.70 (12)	C1—C2—H2	122.2 (13)
C7—N1—H1	130.7 (13)	C3—C2—H2	117.5 (13)
N2—N1—H1	115.8 (13)	C5—C6—C1	120.13 (17)
C9—N2—N1	103.70 (11)	C5—C6—H6	118.6 (12)
O1—C10—N3	126.60 (14)	C1—C6—H6	121.3 (12)
O1—C10—S12	126.50 (12)	C12—C13—H13B	113 (2)
N3—C10—S12	106.89 (10)	C12—C13—H13C	110 (3)
N1—C7—C8	106.73 (12)	H13B—C13—H13C	115 (4)
N1—C7—C1	122.84 (13)	C12—C13—H13A	116.2 (19)
C8—C7—C1	130.42 (14)	H13B—C13—H13A	101 (3)
N2—C9—C8	113.13 (12)	H13C—C13—H13A	101 (3)
N2—C9—N3	117.96 (12)	C5—C4—C3	120.03 (16)
C8—C9—N3	128.89 (13)	C5—C4—H4	120.7 (15)
N4—C11—C12	121.91 (14)	C3—C4—H4	119.3 (15)
N4—C11—S12	116.53 (11)	C4—C3—C2	120.32 (19)
C12—C11—S12	121.52 (11)	C4—C3—H3	122.7 (15)
C2—C1—C6	118.83 (14)	C2—C3—H3	116.9 (15)
C2—C1—C7	119.80 (14)	C4—C5—C6	120.37 (18)
C6—C1—C7	121.37 (14)	C4—C5—H5	124.8 (15)
C7—C8—C9	103.72 (13)	C6—C5—H5	114.8 (16)
C7—C8—H8	127.1 (12)	H3A—O3W—H3B	105 (3)

C10—N3—N4—C11	0.25 (18)	N1—C7—C1—C2	−170.77 (14)
C9—N3—N4—C11	176.34 (12)	C8—C7—C1—C2	10.4 (2)
C7—N1—N2—C9	1.40 (16)	N1—C7—C1—C6	9.5 (2)
N4—N3—C10—O1	179.14 (16)	C8—C7—C1—C6	−169.31 (15)
C9—N3—C10—O1	3.3 (3)	N1—C7—C8—C9	0.87 (16)
N4—N3—C10—S12	−0.66 (16)	C1—C7—C8—C9	179.81 (14)
C9—N3—C10—S12	−176.46 (10)	N2—C9—C8—C7	−0.03 (17)
C11—S12—C10—O1	−179.15 (18)	N3—C9—C8—C7	178.50 (13)
C11—S12—C10—N3	0.65 (11)	N4—C11—C12—O2	173.25 (16)
N2—N1—C7—C8	−1.46 (17)	S12—C11—C12—O2	−4.4 (2)
N2—N1—C7—C1	179.50 (12)	N4—C11—C12—C13	−6.8 (2)
N1—N2—C9—C8	−0.80 (16)	S12—C11—C12—C13	175.50 (14)
N1—N2—C9—N3	−179.51 (12)	C6—C1—C2—C3	1.3 (3)
N4—N3—C9—N2	−1.44 (18)	C7—C1—C2—C3	−178.47 (16)
C10—N3—C9—N2	174.35 (14)	C2—C1—C6—C5	−0.6 (2)

N4—N3—C9—C8	−179.92 (13)	C7—C1—C6—C5	179.19 (14)
C10—N3—C9—C8	−4.1 (2)	C5—C4—C3—C2	−0.4 (3)
N3—N4—C11—C12	−177.47 (13)	C1—C2—C3—C4	−0.8 (3)
N3—N4—C11—S12	0.32 (16)	C3—C4—C5—C6	1.1 (3)
C10—S12—C11—N4	−0.59 (13)	C1—C6—C5—C4	−0.7 (3)
C10—S12—C11—C12	177.22 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3*W*^{i}	0.93 (2)	1.83 (2)	2.749 (2)	173 (2)
O3*W*—H3*A*···N2^{ii}	0.86 (3)	1.99 (3)	2.8402 (19)	169 (2)
O3*W*—H3*B*···O1^{iii}	0.83 (5)	2.19 (5)	2.995 (2)	163 (4)
C8—H8···O1^{iv}	0.93 (2)	2.50 (2)	3.4100 (19)	167.8 (15)

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