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Crystal structure of 1-benzyl-4-formyl-1*H*-pyrrole-3-carboxamide

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In the title compound, $C_{13}H_{12}N_2O_2$ (I), the mean planes of the pyrrole and benzyl rings are approximately perpendicular, forming a dihedral angle of 87.07 (4) °. There is an intramolecular N—H···O hydrogen bond forming an S(7) ring motif. In the crystal, molecules are linked *via* a pair of N—H···O hydrogen bonds forming inversion dimers. C—H···O hydrogen bonds link the dimers into chains along direction [10$\bar{1}$]. The chains are further linked by weak C—H···π interactions forming layers parallel to the *ac* plane.

Keywords: crystal structure; pyrrole derivative; hydrogen bonding; C—H···π interactions

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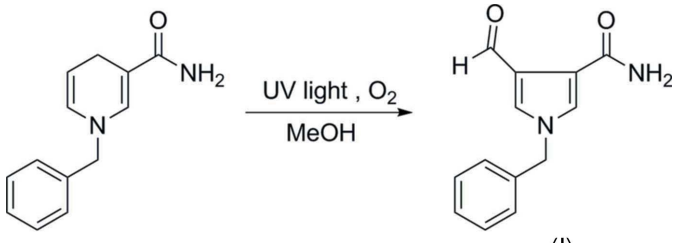
1. Chemical context

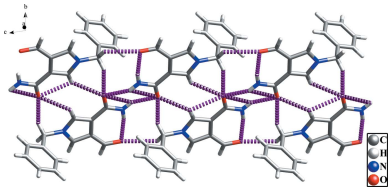
Pyrrole and its derivatives are classes of heterocyclic compounds and that have attracted much attention because of their potential pharmacological and biological properties (Davis *et al.*, 2008; Meshram *et al.*, 2010; Moriguchi *et al.*, 2015). As a part of our work on the synthesis of new pyrrole derivatives with good biological activities, the title compound, (I), was synthesized and its crystal structure is reported on herein.



(I)

2. Structural commentary

The molecular structure of the title compound (I), is shown in Fig. 1. In the amide group, the C—N bond is relatively short



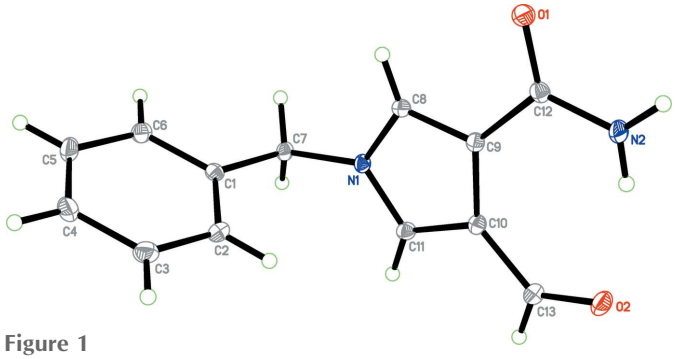


Figure 1
A view of the molecular structure of the title compound (I), with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

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

*Cg*1 is the centroid of the benzyl ring C1–C6.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O2	0.86	1.99	2.8184 (14)	160
N2—H2*A*···O1^{i}	0.86	2.22	3.0063 (14)	151
C8—H8···O1^{ii}	0.93	2.69	3.4252 (15)	136
C7—H7*B*···O1^{ii}	0.97	2.48	3.3123 (15)	144
C7—H7*A*···O2^{iii}	0.97	2.66	3.3268 (15)	126
C11—H11···*Cg*1^{iv}	0.93	2.58	3.4962 (14)	167

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

[C12—N2 = 1.3374 (16) Å], suggesting some degree of electronic delocalization in the molecule. The dihedral angle between the pyrrole and phenyl rings is 87.07 (4)°, indicating that they are nearly perpendicular to each other. An intramolecular hydrogen bond, N2—H2*B*···O2 (Table 1), encloses an *S*(7) ring motif.

3. Supramolecular features

In the crystal of (I), N2—H2*A*···O1^{i} hydrogen bonds [symmetry code: (i) $-x+1, -y+2, -z-1$], link pairs of molecules, forming inversion dimers with an $R_2^2(8)$ ring motif (Table 1 and Fig. 2). The dimers are further linked by C7—H7*B*···O1^{ii}, C8—H8···O1^{ii} and C7—H7*A*···O2^{iii} hydrogen bonds [symmetry codes: (ii) $-x+2, -y+2, -z$; (iii) $x+1, y, z+1$] into supramolecular chains propagating along [10$\bar{1}$]; see Table 1 and Fig. 3). Adjacent chains are linked by weak C11—H11···*Cg*1^{iv} contacts [*Cg*1 is the centroid of the C1—C6 benzyl ring; symmetry code: (iv) $-1+x, y, z$], forming layers parallel to the *ac* plane (Table 1 and Fig. 4).

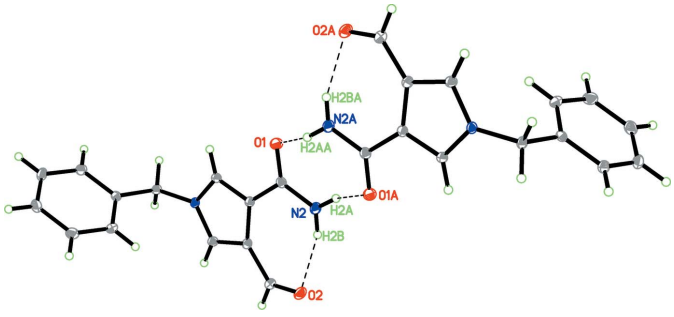

Figure 2
A view of the inversion dimer formed by pairs of N—H···O hydrogen bonds. Both the intramolecular and intermolecular hydrogen bonds are shown as dashed lines (see Table 1).

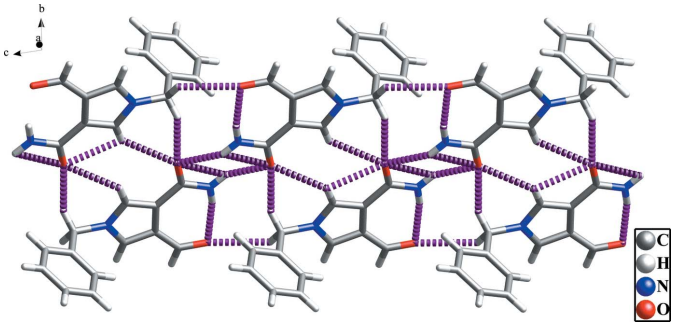

Figure 3
A view of the one-dimensional chain structure. The dashed lines indicate the N—H···O and C—H···O hydrogen bonds (see Table 1).

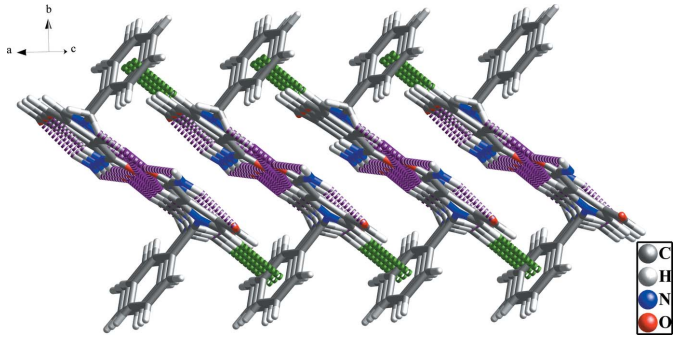

Figure 4
The view of the two-dimensional network structure. The C—H···π interactions and the hydrogen bonds are shown with green and purple dashed lines, respectively (see Table 1).

4. Database survey

A search of the Cambridge Structural Database (Version 5.36 with three updates; Groom & Allen, 2014) for 1-benzyl-4-formyl-1*H*-pyrrole-3-carboxamide gave no hits. However, structures of substituted derivatives of 1-benzyl-1*H*-pyrrole

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{12}N_2O_2$
M_r	228.25
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, *b*, *c* (Å)	5.5296 (6), 23.083 (3), 9.3088 (9)
β (°)	112.940 (5)
V (Å^3)	1094.2 (2)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.10
Crystal size (mm)	0.25 × 0.20 × 0.18
Data collection	
Diffractometer	Bruker SMART CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2005)
T_{min}, T_{max}	0.977, 0.983
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9372, 1938, 1823
R_{int}	0.021
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.596
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.034, 0.123, 1.00
No. of reflections	1938
No. of parameters	154
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.26

Computer programs: *APEX2* and *SAINT* (Bruker, 2005) and *SHELXTL* (Sheldrick, 2008).

were found, see for example Bonnett *et al.* (1985); Choi *et al.* (1998); Sha *et al.* (1990); Wang *et al.* (2011). In these structures, the pyrrole and benzyl rings are also nearly perpendicular to one another.

5. Synthesis and crystallization

1-Benzyl-1*H*-pyrrole-3-carboxamide (1 mmol, 214.3 mg) was dissolved in methanol (20 ml) and irradiated with UV light at room temperature under oxygen (see Scheme). The reaction progress was monitored by thin layer chromatography (TLC). After completion, the solvent was removed under reduced pressure, and the residue was purified by chromatography on silica gel, using a mixed solvent of petroleum ether and ethyl acetate (10:1 ratio, *v*/*v*), to give the pure product. Colourless single crystals, suitable for X-ray diffraction analysis, were obtained by slow evaporation of a methanol solution of the title compound at room temperature.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were placed in idealized positions (C—H = 0.93–0.97 Å, N—H = 0.86 Å) and refined as riding atoms, with U_{iso}(H) = 1.2U_{eq}(N,C).

Acknowledgements

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Crystal structure of 1-benzyl-4-formyl-1*H*-pyrrole-3-carboxamide

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Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

1-Benzyl-4-formyl-1*H*-pyrrole-3-carboxamide

Crystal data

$C_{13}H_{12}N_2O_2$
M_r = 228.25
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 5.5296 (6) Å
b = 23.083 (3) Å
c = 9.3088 (9) Å
β = 112.940 (5)°
V = 1094.2 (2) Å^3
Z = 4
$F(000)$ = 480
D_x = 1.386 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3196 reflections
θ = 3.5–27.5°
μ = 0.10 mm^{-1}
T = 293 K
Block, colorless
0.25 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.977, T_{max} = 0.983
9372 measured reflections
1938 independent reflections
1823 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.1°, θ_{min} = 3.5°
h = −6→6
k = −27→27
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.123
S = 1.00
1938 reflections
154 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.2118P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.0335 (2)	0.84645 (5)	0.26085 (13)	0.0165 (3)
C2	0.9577 (2)	0.80281 (5)	0.14829 (15)	0.0217 (3)
H2	0.7904	0.8035	0.0696	0.026*
C3	1.1293 (3)	0.75831 (6)	0.15255 (17)	0.0267 (3)
H3	1.0774	0.7298	0.0760	0.032*
C4	1.3792 (3)	0.75629 (6)	0.27120 (17)	0.0271 (3)
H4	1.4942	0.7264	0.2746	0.033*
C5	1.4545 (2)	0.79918 (6)	0.38396 (15)	0.0252 (3)
H5	1.6204	0.7979	0.4640	0.030*
C6	1.2845 (2)	0.84412 (5)	0.37859 (14)	0.0206 (3)
H6	1.3385	0.8730	0.4543	0.025*
C7	0.8542 (2)	0.89645 (5)	0.25832 (13)	0.0167 (3)
H7A	0.7595	0.8869	0.3236	0.020*
H7B	0.9601	0.9305	0.3022	0.020*
C8	0.7226 (2)	0.94298 (5)	−0.00412 (13)	0.0158 (3)
H8	0.8792	0.9627	0.0164	0.019*
C9	0.5118 (2)	0.94220 (5)	−0.14451 (13)	0.0152 (3)
C10	0.3150 (2)	0.90594 (5)	−0.12295 (13)	0.0162 (3)
C11	0.4214 (2)	0.88767 (5)	0.03064 (13)	0.0171 (3)
H11	0.3383	0.8638	0.0779	0.021*
C12	0.5128 (2)	0.97561 (5)	−0.28041 (13)	0.0171 (3)
C13	0.0602 (2)	0.88460 (5)	−0.22590 (14)	0.0199 (3)
H13	−0.0288	0.8620	−0.1797	0.024*
N1	0.66548 (19)	0.90998 (4)	0.10118 (11)	0.0152 (3)
N2	0.2915 (2)	0.97471 (5)	−0.40897 (12)	0.0214 (3)
H2A	0.2825	0.9936	−0.4906	0.026*
H2B	0.1581	0.9553	−0.4100	0.026*
O1	0.71039 (17)	1.00283 (4)	−0.27249 (10)	0.0237 (3)
O2	−0.05271 (17)	0.89275 (4)	−0.36651 (10)	0.0255 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0178 (6)	0.0173 (6)	0.0150 (6)	−0.0021 (5)	0.0072 (5)	0.0043 (4)
C2	0.0162 (6)	0.0221 (7)	0.0233 (7)	−0.0031 (5)	0.0041 (5)	−0.0016 (5)
C3	0.0249 (7)	0.0193 (7)	0.0347 (8)	−0.0027 (5)	0.0103 (6)	−0.0052 (5)

C4	0.0234 (7)	0.0199 (7)	0.0380 (8)	0.0052 (5)	0.0120 (6)	0.0061 (5)
C5	0.0181 (6)	0.0284 (7)	0.0244 (7)	0.0026 (5)	0.0033 (5)	0.0086 (5)
C6	0.0209 (6)	0.0228 (7)	0.0159 (6)	−0.0023 (5)	0.0046 (5)	0.0020 (5)
C7	0.0167 (6)	0.0198 (6)	0.0113 (6)	−0.0003 (5)	0.0029 (5)	0.0011 (4)
C8	0.0156 (6)	0.0153 (6)	0.0163 (6)	−0.0010 (4)	0.0062 (5)	0.0001 (4)
C9	0.0160 (6)	0.0142 (6)	0.0147 (6)	0.0013 (4)	0.0051 (5)	−0.0014 (4)
C10	0.0153 (6)	0.0175 (6)	0.0154 (6)	0.0012 (5)	0.0057 (5)	−0.0008 (4)
C11	0.0155 (6)	0.0183 (6)	0.0185 (6)	−0.0017 (5)	0.0076 (5)	0.0004 (5)
C12	0.0200 (6)	0.0144 (6)	0.0162 (6)	0.0009 (5)	0.0064 (5)	−0.0008 (4)
C13	0.0171 (6)	0.0221 (6)	0.0194 (7)	−0.0005 (5)	0.0059 (5)	−0.0007 (5)
N1	0.0155 (5)	0.0164 (5)	0.0120 (5)	0.0006 (4)	0.0037 (4)	0.0007 (4)
N2	0.0198 (6)	0.0265 (6)	0.0149 (5)	−0.0022 (4)	0.0034 (4)	0.0055 (4)
O1	0.0233 (5)	0.0275 (5)	0.0177 (5)	−0.0062 (4)	0.0052 (4)	0.0042 (3)
O2	0.0213 (5)	0.0314 (6)	0.0178 (5)	−0.0034 (4)	0.0009 (4)	−0.0008 (4)

Geometric parameters (Å, º)

C1—C6	1.3942 (17)	C8—C9	1.3709 (16)
C1—C2	1.3949 (18)	C8—N1	1.3719 (15)
C1—C7	1.5159 (16)	C8—H8	0.9300
C2—C3	1.3886 (18)	C9—C10	1.4476 (16)
C2—H2	0.9300	C9—C12	1.4834 (16)
C3—C4	1.3933 (19)	C10—C11	1.3829 (16)
C3—H3	0.9300	C10—C13	1.4471 (17)
C4—C5	1.384 (2)	C11—N1	1.3516 (15)
C4—H4	0.9300	C11—H11	0.9300
C5—C6	1.3877 (18)	C12—O1	1.2375 (15)
C5—H5	0.9300	C12—N2	1.3374 (16)
C6—H6	0.9300	C13—O2	1.2253 (15)
C7—N1	1.4616 (14)	C13—H13	0.9300
C7—H7A	0.9700	N2—H2A	0.8600
C7—H7B	0.9700	N2—H2B	0.8600
C6—C1—C2	118.58 (11)	C9—C8—N1	109.08 (10)
C6—C1—C7	119.09 (11)	C9—C8—H8	125.5
C2—C1—C7	122.33 (11)	N1—C8—H8	125.5
C3—C2—C1	120.71 (12)	C8—C9—C10	106.34 (10)
C3—C2—H2	119.6	C8—C9—C12	121.46 (11)
C1—C2—H2	119.6	C10—C9—C12	132.19 (11)
C2—C3—C4	120.19 (12)	C11—C10—C13	119.54 (11)
C2—C3—H3	119.9	C11—C10—C9	106.24 (10)
C4—C3—H3	119.9	C13—C10—C9	134.08 (11)
C5—C4—C3	119.32 (12)	N1—C11—C10	109.15 (10)
C5—C4—H4	120.3	N1—C11—H11	125.4
C3—C4—H4	120.3	C10—C11—H11	125.4
C4—C5—C6	120.55 (12)	O1—C12—N2	122.68 (11)
C4—C5—H5	119.7	O1—C12—C9	120.65 (10)
C6—C5—H5	119.7	N2—C12—C9	116.67 (10)

C5—C6—C1	120.65 (12)	O2—C13—C10	127.90 (12)
C5—C6—H6	119.7	O2—C13—H13	116.0
C1—C6—H6	119.7	C10—C13—H13	116.0
N1—C7—C1	112.69 (9)	C11—N1—C8	109.20 (10)
N1—C7—H7A	109.1	C11—N1—C7	126.38 (10)
C1—C7—H7A	109.1	C8—N1—C7	124.07 (10)
N1—C7—H7B	109.1	C12—N2—H2A	120.0
C1—C7—H7B	109.1	C12—N2—H2B	120.0
H7A—C7—H7B	107.8	H2A—N2—H2B	120.0

C6—C1—C2—C3	−0.61 (18)	C12—C9—C10—C13	6.5 (2)
C7—C1—C2—C3	178.65 (11)	C13—C10—C11—N1	176.01 (10)
C1—C2—C3—C4	0.9 (2)	C9—C10—C11—N1	−0.30 (13)
C2—C3—C4—C5	−0.3 (2)	C8—C9—C12—O1	3.69 (18)
C3—C4—C5—C6	−0.6 (2)	C10—C9—C12—O1	−178.07 (11)
C4—C5—C6—C1	0.92 (19)	C8—C9—C12—N2	−176.06 (10)
C2—C1—C6—C5	−0.30 (18)	C10—C9—C12—N2	2.19 (19)
C7—C1—C6—C5	−179.59 (11)	C11—C10—C13—O2	−173.94 (12)
C6—C1—C7—N1	151.87 (11)	C9—C10—C13—O2	1.1 (2)
C2—C1—C7—N1	−27.39 (15)	C10—C11—N1—C8	0.01 (13)
N1—C8—C9—C10	−0.47 (13)	C10—C11—N1—C7	−173.30 (10)
N1—C8—C9—C12	178.17 (10)	C9—C8—N1—C11	0.30 (13)
C8—C9—C10—C11	0.47 (13)	C9—C8—N1—C7	173.80 (10)
C12—C9—C10—C11	−177.97 (12)	C1—C7—N1—C11	90.92 (13)
C8—C9—C10—C13	−175.05 (13)	C1—C7—N1—C8	−81.44 (13)

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the benzyl ring C1–C6.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O2	0.86	1.99	2.8184 (14)	160
N2—H2*A*···O1^{i}	0.86	2.22	3.0063 (14)	151
C8—H8···O1^{ii}	0.93	2.69	3.4252 (15)	136
C7—H7*B*···O1^{ii}	0.97	2.48	3.3123 (15)	144
C7—H7*A*···O2^{iii}	0.97	2.66	3.3268 (15)	126
C11—H11···*Cg*1^{iv}	0.93	2.58	3.4962 (14)	167

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