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ISSN 2414-3146

N-[3-Methyl-1-phenyl-1-(1*H*-tetrazol-1-yl)butan-2-yl]acetamide

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Received 24 October 2016
Accepted 10 November 2016

Edited by C. Rizzoli, Universita degli Studi di Parma, Italy

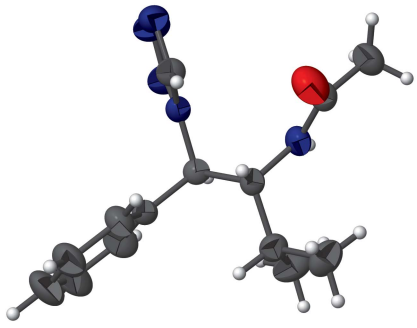
Keywords: crystal structure; tetrazole; acetamide; hydrogen bonds.

CCDC reference: 1516411

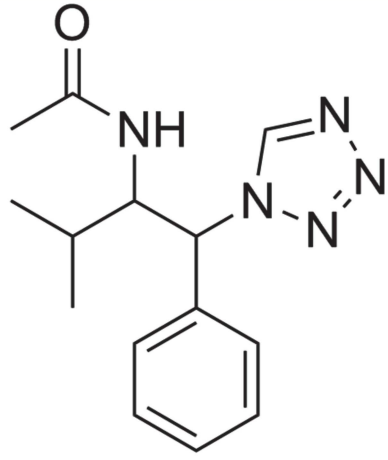
Structural data: full structural data are available from iucrdata.iucr.org

In the molecule of the title compound, $C_{14}H_{19}N_5O$, the dihedral angle formed between the tetrazole and phenyl rings is 68.39 (4)°. In the crystal, molecules are linked by N—H···N, C—H···N and C—H···O hydrogen bonds to form two-dimensional networks extending parallel to the *bc* plane.

3D view



Chemical scheme



Structure description

Compounds containing a tetrazole ring have attracted much attention in medicinal chemistry (Alam & Nasrollahzadeh, 2009). Tetrazoles are reported to exhibits anti-hypertensive (Sharma *et al.*, 2010), antimicrobial (Yildirir *et al.*, 2009), antibacterial, antifungal (Dhayanithi *et al.*, 2011) and anticancer activities (Bhaskar & Mohite, 2010). These functional units exhibit strong networking ability as ligands. They act as mono-, bi- or multidentate ligands due to the electron-donating nature of the four nitrogen atoms in the tetrazole moiety (Wang *et al.*, 2005).

In the title compound (Fig. 1), the bond between the two chiral carbons C7 and C8 acts as the bridge connecting the phenyl ring, the 1-*H* tetrazole ring and the acetamide unit. The dihedral angle between the tetrazole ring (N1–N4, C14) and the phenyl ring (C1–C6) is 68.39 (4)°. The mean plane through the acetamide unit (N5, C12, C13, O1) forms dihedral angles of 62.00 (6) and 13.23 (6)° with the tetrazole and phenyl rings, respectively.

In the crystal, the molecules are linked through C—H···O, C—H···N and N—H···N hydrogen bonds (Table 1) into two-dimensional networks extending parallel to the *bc* plane (Fig. 2).

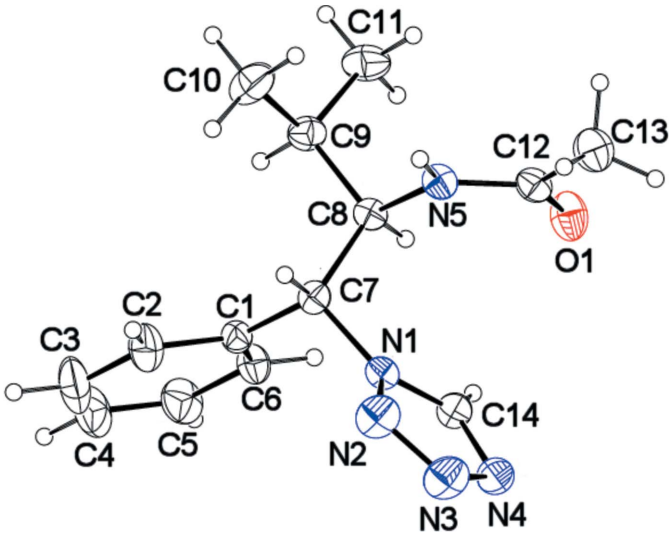


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. H atoms are shown as small spheres of arbitrary radius.

Synthesis and crystallization

A Mannich condensation reaction involving benzaldehye (200 mmol), isobutyl methyl ketone (100 mmol) and ammonium acetate (100 mmol) in 70 ml methanol at 70°C for 2 h afforded the respective piperidinone as crystals. The crystals were washed with methanol, dried completely under vacuum, then converted into the hydrochloride form by dissolving them in 30 ml ethanol and 20 ml ether and adding an equivalent volume of concentrated hydrochloric acid dropwise. 2 g of the precipitate obtained was gradually added to a beaker containing 10 ml of concentrated sulfuric acid in ice-cold condition, dissolved thoroughly and kept at room temperature with continuous stirring. 0.65 g of sodium azide was then added in small quantities to the beaker. On addition of sodium azide, a foam formed which subsequently subsided due to liberation of nitrogen. The solution was transferred into a beaker containing ice and neutralized with 4 *M* sodium hydroxide. The white precipitate formed was filtered through a Buchner funnel, vacuum dried and recrystallized with ethanol. The resulting lactam was cleaved under acidic conditions (6 *M* HCl) to form the substituted vicinal diamine. Conversion of the hydrochloride salt of the vicinal diamine into the free diamine was performed using 2 mol of sodium acetate. The vicinal diamine was then converted into acetylated 1-substituted tetrazole in the presence of 2 mol of sodium azide and 2 mol of triethyl orthoformate at 60°C in a glacial acetic acid medium. The compound obtained was then dissolved in methanol, transferred to a 15 ml vial, and the vial wrapped with tissue paper for controlled evaporation of the solvent without contamination. Single crystals suitable for X-ray analysis were formed after three days.

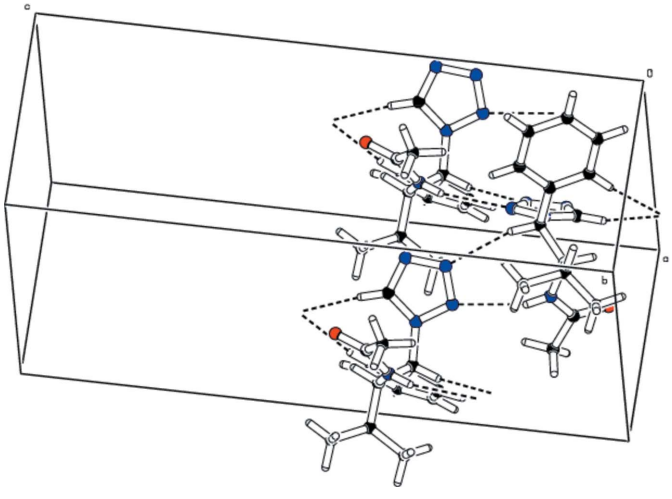


Figure 2
Partial crystal packing of the title compound showing the formation of a two-dimensional network parallel to the *bc* plane *via* N—H···N, C—H···O and C—H···N hydrogen bonds (dashed lines).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···N2^{i}	0.844 (18)	2.452 (18)	3.2579 (17)	160.0 (15)
C6—H6···O1^{ii}	0.93	2.43	3.356 (2)	171
C7—H7···N3^{i}	0.98	2.48	3.4055 (19)	157
C14—H14···O1^{ii}	0.93	2.27	3.1728 (19)	163

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{19}N_5O$
M_r	273.34
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, *b*, *c* (Å)	9.7056 (6), 7.7663 (5), 20.2620 (9)
β (°)	93.490 (2)
V (Å^3)	1524.45 (15)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.35 × 0.30 × 0.30
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.740, 0.976
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10998, 3760, 2712
R_{int}	0.020
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.134, 1.02
No. of reflections	3760
No. of parameters	188
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.17

Computer programs: *APEX2* and *SAINT* (Bruker, 2004), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors thank the single-crystal XRD facility, SAIF, IIT Madras, Chennai, for the data collection.

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full crystallographic data

IUCrData (2016). **1**, x161810 [https://doi.org/10.1107/S2414314616018101]

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Crystal data

$C_{14}H_{19}N_5O$
M_r = 273.34
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 9.7056 (6) Å
b = 7.7663 (5) Å
c = 20.2620 (9) Å
β = 93.490 (2)°
V = 1524.45 (15) Å^3
Z = 4
$F(000)$ = 584
D_x = 1.191 Mg m^{-3}
Melting point: 393 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3760 reflections
θ = 2.8–28.3°
μ = 0.08 mm^{-1}
T = 296 K
Block, colourless
0.35 × 0.30 × 0.30 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Bruker axs kappa apex2 CCD Diffractometer scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.740, T_{max} = 0.976
10998 measured reflections
3760 independent reflections
2712 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 28.3°, θ_{min} = 2.8°
h = −12→12
k = −10→10
l = −26→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.134
S = 1.02
3760 reflections
188 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 0.4088P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.010
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.61825 (12)	0.64603 (18)	0.04105 (5)	0.0600 (3)
N1	0.43416 (11)	0.38123 (15)	0.15539 (5)	0.0342 (3)
N2	0.48431 (16)	0.3137 (2)	0.21287 (6)	0.0556 (4)
N3	0.57528 (16)	0.2002 (2)	0.19824 (7)	0.0605 (4)
N4	0.58598 (13)	0.19027 (18)	0.13256 (7)	0.0484 (3)
N5	0.50558 (12)	0.72547 (16)	0.12972 (6)	0.0388 (3)
H5A	0.5130 (17)	0.774 (2)	0.1670 (9)	0.049 (5)*
C1	0.18648 (14)	0.44056 (18)	0.13683 (7)	0.0374 (3)
C2	0.09223 (18)	0.4295 (3)	0.18515 (9)	0.0616 (5)
H2	0.1152	0.4707	0.2275	0.074*
C3	−0.0371 (2)	0.3568 (3)	0.17039 (13)	0.0847 (7)
H3	−0.1001	0.3499	0.2031	0.102*
C4	−0.07241 (19)	0.2958 (3)	0.10887 (13)	0.0788 (6)
H4	−0.1590	0.2474	0.0995	0.095*
C5	0.02009 (18)	0.3061 (3)	0.06068 (10)	0.0631 (5)
H5	−0.0038	0.2645	0.0185	0.076*
C6	0.14894 (15)	0.3779 (2)	0.07445 (8)	0.0462 (4)
H6	0.2111	0.3842	0.0414	0.055*
C7	0.32751 (14)	0.51735 (18)	0.15342 (6)	0.0349 (3)
H7	0.3268	0.5674	0.1978	0.042*
C8	0.37089 (14)	0.65989 (18)	0.10615 (6)	0.0349 (3)
H8	0.3817	0.6063	0.0630	0.042*
C9	0.26314 (16)	0.8039 (2)	0.09599 (7)	0.0448 (4)
H9	0.1749	0.7495	0.0822	0.054*
C10	0.2413 (2)	0.9070 (3)	0.15828 (9)	0.0670 (5)
H10A	0.3262	0.9619	0.1731	0.100*
H10B	0.2121	0.8312	0.1922	0.100*
H10C	0.1717	0.9929	0.1489	0.100*
C11	0.3024 (2)	0.9215 (2)	0.04006 (8)	0.0606 (5)
H11A	0.2328	1.0084	0.0326	0.091*
H11B	0.3096	0.8550	0.0005	0.091*
H11C	0.3895	0.9754	0.0518	0.091*
C12	0.61938 (15)	0.71366 (19)	0.09555 (7)	0.0418 (3)
C13	0.74903 (18)	0.7856 (3)	0.12893 (10)	0.0612 (5)
H13A	0.8277	0.7309	0.1115	0.092*
H13B	0.7493	0.7647	0.1756	0.092*
H13C	0.7531	0.9074	0.1210	0.092*
C14	0.49788 (15)	0.30355 (19)	0.10746 (7)	0.0412 (3)
H14	0.4822	0.3264	0.0626	0.049*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0576 (7)	0.0833 (9)	0.0399 (6)	−0.0092 (6)	0.0106 (5)	−0.0082 (6)
N1	0.0361 (6)	0.0374 (6)	0.0285 (5)	−0.0026 (5)	−0.0029 (4)	0.0043 (5)
N2	0.0667 (9)	0.0659 (10)	0.0332 (7)	0.0124 (8)	−0.0060 (6)	0.0102 (6)
N3	0.0647 (9)	0.0653 (10)	0.0501 (8)	0.0157 (8)	−0.0092 (7)	0.0129 (7)
N4	0.0438 (7)	0.0479 (8)	0.0530 (8)	0.0046 (6)	−0.0004 (6)	0.0068 (6)
N5	0.0420 (7)	0.0423 (7)	0.0318 (6)	−0.0075 (5)	−0.0008 (5)	−0.0065 (5)
C1	0.0350 (7)	0.0378 (8)	0.0399 (7)	0.0017 (6)	0.0054 (5)	0.0063 (6)
C2	0.0551 (10)	0.0821 (14)	0.0493 (9)	−0.0016 (9)	0.0179 (8)	0.0044 (9)
C3	0.0517 (11)	0.1155 (19)	0.0907 (16)	−0.0091 (12)	0.0343 (11)	0.0137 (14)
C4	0.0398 (10)	0.0940 (16)	0.1027 (17)	−0.0163 (10)	0.0056 (10)	0.0085 (14)
C5	0.0450 (9)	0.0713 (13)	0.0715 (12)	−0.0107 (9)	−0.0085 (8)	−0.0026 (10)
C6	0.0370 (7)	0.0551 (9)	0.0464 (8)	−0.0064 (7)	0.0027 (6)	−0.0019 (7)
C7	0.0387 (7)	0.0390 (7)	0.0270 (6)	0.0011 (6)	0.0016 (5)	−0.0017 (5)
C8	0.0386 (7)	0.0351 (7)	0.0306 (6)	−0.0035 (6)	−0.0009 (5)	−0.0012 (5)
C9	0.0461 (8)	0.0410 (8)	0.0465 (8)	0.0017 (7)	−0.0036 (6)	0.0025 (6)
C10	0.0816 (13)	0.0610 (12)	0.0592 (11)	0.0248 (10)	0.0116 (9)	−0.0017 (9)
C11	0.0816 (13)	0.0461 (10)	0.0534 (10)	0.0048 (9)	−0.0025 (9)	0.0108 (8)
C12	0.0440 (8)	0.0414 (8)	0.0396 (8)	−0.0044 (7)	0.0000 (6)	0.0053 (6)
C13	0.0458 (9)	0.0648 (12)	0.0720 (12)	−0.0081 (8)	−0.0040 (8)	−0.0034 (9)
C14	0.0436 (8)	0.0442 (8)	0.0356 (7)	0.0023 (6)	0.0014 (6)	0.0030 (6)

Geometric parameters (Å, º)

O1—C12	1.2221 (18)	C5—H5	0.9300
N1—C14	1.3276 (18)	C6—H6	0.9300
N1—N2	1.3413 (16)	C7—C8	1.5392 (19)
N1—C7	1.4784 (18)	C7—H7	0.9800
N2—N3	1.295 (2)	C8—C9	1.536 (2)
N3—N4	1.3434 (19)	C8—H8	0.9800
N4—C14	1.3079 (19)	C9—C10	1.520 (2)
N5—C12	1.3420 (19)	C9—C11	1.522 (2)
N5—C8	1.4562 (17)	C9—H9	0.9800
N5—H5A	0.844 (18)	C10—H10A	0.9600
C1—C6	1.382 (2)	C10—H10B	0.9600
C1—C2	1.383 (2)	C10—H10C	0.9600
C1—C7	1.5120 (19)	C11—H11A	0.9600
C2—C3	1.392 (3)	C11—H11B	0.9600
C2—H2	0.9300	C11—H11C	0.9600
C3—C4	1.358 (3)	C12—C13	1.500 (2)
C3—H3	0.9300	C13—H13A	0.9600
C4—C5	1.368 (3)	C13—H13B	0.9600
C4—H4	0.9300	C13—H13C	0.9600
C5—C6	1.382 (2)	C14—H14	0.9300
C14—N1—N2	107.28 (12)	C9—C8—C7	113.40 (11)

C14—N1—C7	131.43 (11)	N5—C8—H8	107.4
N2—N1—C7	121.30 (11)	C9—C8—H8	107.4
N3—N2—N1	106.48 (12)	C7—C8—H8	107.4
N2—N3—N4	111.16 (12)	C10—C9—C11	110.85 (15)
C14—N4—N3	104.96 (13)	C10—C9—C8	113.59 (13)
C12—N5—C8	123.88 (12)	C11—C9—C8	109.73 (13)
C12—N5—H5A	117.7 (11)	C10—C9—H9	107.5
C8—N5—H5A	118.4 (11)	C11—C9—H9	107.5
C6—C1—C2	118.47 (14)	C8—C9—H9	107.5
C6—C1—C7	121.79 (12)	C9—C10—H10A	109.5
C2—C1—C7	119.73 (14)	C9—C10—H10B	109.5
C1—C2—C3	119.98 (18)	H10A—C10—H10B	109.5
C1—C2—H2	120.0	C9—C10—H10C	109.5
C3—C2—H2	120.0	H10A—C10—H10C	109.5
C4—C3—C2	120.85 (17)	H10B—C10—H10C	109.5
C4—C3—H3	119.6	C9—C11—H11A	109.5
C2—C3—H3	119.6	C9—C11—H11B	109.5
C3—C4—C5	119.66 (18)	H11A—C11—H11B	109.5
C3—C4—H4	120.2	C9—C11—H11C	109.5
C5—C4—H4	120.2	H11A—C11—H11C	109.5
C4—C5—C6	120.27 (19)	H11B—C11—H11C	109.5
C4—C5—H5	119.9	O1—C12—N5	122.22 (14)
C6—C5—H5	119.9	O1—C12—C13	121.92 (14)
C5—C6—C1	120.77 (15)	N5—C12—C13	115.85 (14)
C5—C6—H6	119.6	C12—C13—H13A	109.5
C1—C6—H6	119.6	C12—C13—H13B	109.5
N1—C7—C1	110.29 (11)	H13A—C13—H13B	109.5
N1—C7—C8	108.27 (10)	C12—C13—H13C	109.5
C1—C7—C8	115.06 (11)	H13A—C13—H13C	109.5
N1—C7—H7	107.6	H13B—C13—H13C	109.5
C1—C7—H7	107.6	N4—C14—N1	110.12 (13)
C8—C7—H7	107.6	N4—C14—H14	124.9
N5—C8—C9	112.30 (12)	N1—C14—H14	124.9
N5—C8—C7	108.76 (11)		

C14—N1—N2—N3	0.25 (17)	C6—C1—C7—C8	52.19 (19)
C7—N1—N2—N3	−179.60 (13)	C2—C1—C7—C8	−128.80 (15)
N1—N2—N3—N4	−0.4 (2)	C12—N5—C8—C9	−116.60 (15)
N2—N3—N4—C14	0.3 (2)	C12—N5—C8—C7	117.06 (14)
C6—C1—C2—C3	−0.1 (3)	N1—C7—C8—N5	−58.28 (13)
C7—C1—C2—C3	−179.13 (18)	C1—C7—C8—N5	177.83 (11)
C1—C2—C3—C4	0.1 (4)	N1—C7—C8—C9	176.01 (11)
C2—C3—C4—C5	−0.1 (4)	C1—C7—C8—C9	52.12 (16)
C3—C4—C5—C6	0.1 (4)	N5—C8—C9—C10	−58.69 (17)
C4—C5—C6—C1	0.0 (3)	C7—C8—C9—C10	65.11 (17)
C2—C1—C6—C5	0.1 (3)	N5—C8—C9—C11	66.00 (16)
C7—C1—C6—C5	179.09 (15)	C7—C8—C9—C11	−170.20 (12)
C14—N1—C7—C1	80.54 (17)	C8—N5—C12—O1	0.1 (2)

N2—N1—C7—C1	−99.66 (14)	C8—N5—C12—C13	−178.70 (14)
C14—N1—C7—C8	−46.17 (18)	N3—N4—C14—N1	−0.15 (18)
N2—N1—C7—C8	133.64 (13)	N2—N1—C14—N4	−0.06 (17)
C6—C1—C7—N1	−70.63 (16)	C7—N1—C14—N4	179.77 (13)
C2—C1—C7—N1	108.39 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···N2^{i}	0.844 (18)	2.452 (18)	3.2579 (17)	160.0 (15)
C6—H6···O1^{ii}	0.93	2.43	3.356 (2)	171
C7—H7···N3^{i}	0.98	2.48	3.4055 (19)	157
C14—H14···O1^{ii}	0.93	2.27	3.1728 (19)	163

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