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N-(4-Methoxyphenyl)-2,6-dimethyl-1,3-dioxan-4-amine

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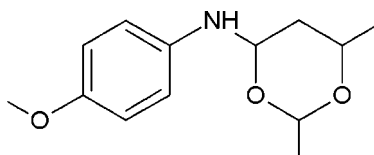
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.053; wR factor = 0.162; data-to-parameter ratio = 20.1.

In the title compound, $\text{C}_{13}\text{H}_{19}\text{NO}_3$, the dioxane ring adopts a chair conformation. Its mean plane is inclined to the 4-methoxyphenyl ring by 70.34 (9)°. In the crystal, molecules are linked by pairs of $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming inversion dimers with an $R_2^2(16)$ ring motif. The dimers are linked *via* $\text{C}-\text{H}\cdots\pi$ interactions, forming two-dimensional networks lying parallel to the *ac* plane.

Related literature

For biological properties of oxygen heterocycles, such as dioxane derivatives, see: Aubele *et al.* (2005); Marucci *et al.* (2005). For some applications, see: Wang *et al.* (1994, 1996*a,b*); Yuan *et al.* (2005). For related crystal structures, see: Chuprunov *et al.* (1981); Yuan *et al.* (2008). For graph-set motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{19}\text{NO}_3$ $V = 1279.93$ (13) Å³
 $M_r = 237.29$ $Z = 4$
 Monoclinic, $P2_1/c$ Mo $K\alpha$ radiation
 $a = 9.6472$ (6) Å $\mu = 0.09$ mm⁻¹
 $b = 13.8194$ (8) Å $T = 293$ K
 $c = 10.5384$ (6) Å $0.25 \times 0.20 \times 0.15$ mm
 $\beta = 114.355$ (3)°

Data collection

Bruker SMART APEXII area-detector diffractometer 12259 measured reflections
 Absorption correction: multi-scan (SADABS; Bruker, 2008) 3169 independent reflections
 $T_{\min} = 0.667$, $T_{\max} = 0.746$ 2133 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$ H atoms treated by a mixture of independent and constrained refinement
 $wR(F^2) = 0.162$ $\Delta\rho_{\text{max}} = 0.26$ e Å⁻³
 $S = 1.03$ $\Delta\rho_{\text{min}} = -0.21$ e Å⁻³
 3169 reflections
 158 parameters

Table 1

Hydrogen-bond geometry (Å, °).

$Cg1$ is the centroid of ring C2-C7 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C6-H6\cdots O3^i$	0.93	2.51	3.407 (2)	162
$C1-H1B\cdots Cg1^{ii}$	0.96	2.77	3.700 (3)	163

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

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT (Bruker, 2008); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

The authors thank the TBI X-ray facility, CAS in Crystallography and Biophysics, University of Madras, India, for the data collection. ZF and DV acknowledge the UGC (SAP-CAS) for departmental facilities. ZF also thanks the UGC for a meritorious fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2641).

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***N*-(4-Methoxyphenyl)-2,6-dimethyl-1,3-dioxan-4-amine**

**Zeenat Fatima, Rambabu Gottimukkal, Bandapalli Palakshi Reddy, Vijayaparthasarathi
Vijayakumar and Devadasan Velmurugan**

S1. Comment

Oxygen heterocycles play a vital role as basic building blocks in pharmaceutical preparations. Dioxane rings are frequently encountered in many bioactive molecules, such as cytotoxic agents (Aubele *et al.*, 2005) and antimuscarinic agents (Marucci *et al.*, 2005). This class of compounds also has useful insecticidal as well as anti-foaming properties (Yuan *et al.*, 2005; Wang *et al.*, 1994, 1996*a,b*). In view of the different applications of this class of compounds, we have synthesized the title derivative and report herein on its crystal structure.

In the title molecule, Fig. 1, the dioxane ring (O2/O3/C8—C11) adopts a *chair* conformation and its mean plane makes a dihedral angle of 70.34 (9)° with the benzene ring (C2—C7).

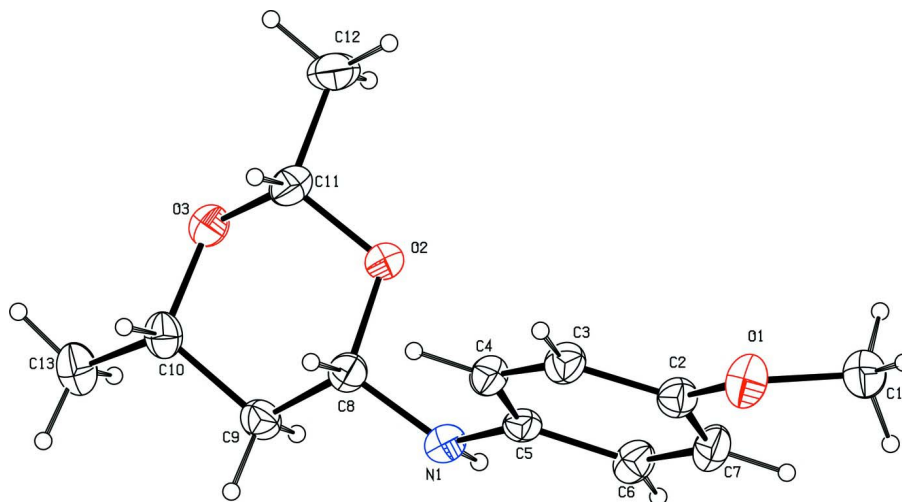
In the crystal, molecules are linked by pairs of C—H···O hydrogen bonds (Table 1 and Fig. 2) forming inversion dimers with an R²₂(16) ring motif (Bernstein *et al.*, 1995). The dimers are linked via C-H··· π interactions forming two-dimensional networks lying parallel to the ac plane (Table 1).

S2. Experimental

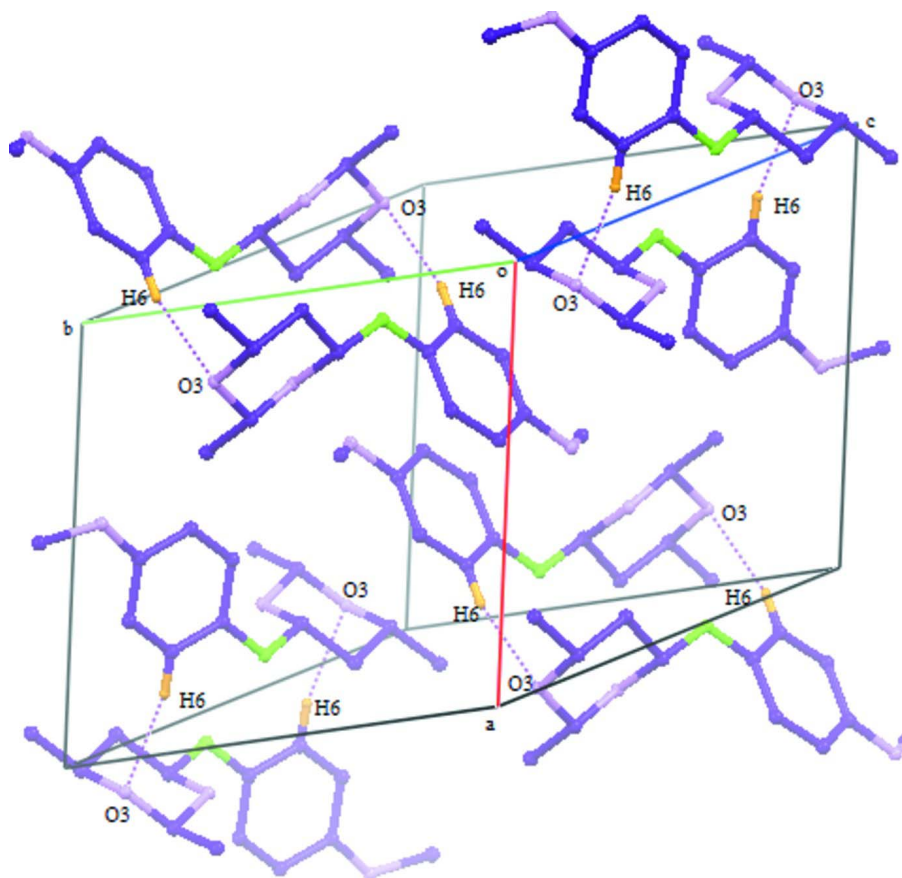
To 4-anisidine (1 mmol), acetaldehyde (3 mmol) was added drop wise and stirred for about 4 h at 273 K. The progress of the reaction was monitored by TLC. The reaction mixture was then washed with petroleum ether. The residue was dissolved in diethylether and the solution left for the solvent to evaporate. The solid product obtained was recrystallized from diethylether giving block-like colourless crystals of the title compound.

S3. Refinement

The NH H atom was located in a difference Fourier map and freely refined. The C-bound H atoms were placed in calculated positions refined as riding: C—H = 0.93 Å to 0.98 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C-methyl})$ and $= 1.2U_{\text{eq}}(\text{C})$ for other H atoms.

**Figure 1**

The molecular structure of the title molecule, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

**Figure 2**

The crystal packing of the title compound viewed almost perpendicular to (011). Hydrogen bonds are shown as dashed lines (see Table 1 for details; H-atoms not involved in hydrogen bonding have been omitted for clarity).

N*-(4-Methoxyphenyl)-2,6-dimethyl-1,3-dioxan-4-amineCrystal data*C₁₃H₁₉NO₃ $M_r = 237.29$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 9.6472$ (6) Å $b = 13.8194$ (8) Å $c = 10.5384$ (6) Å $\beta = 114.355$ (3)° $V = 1279.93$ (13) Å³ $Z = 4$ $F(000) = 512$ $D_x = 1.231$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3169 reflections

 $\theta = 2.3$ – 28.3 ° $\mu = 0.09$ mm⁻¹ $T = 293$ K

Block, colourless

 $0.25 \times 0.20 \times 0.15$ mm*Data collection*Bruker SMART APEXII area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 ω and φ scansAbsorption correction: multi-scan
(*SADABS*; Bruker, 2008) $T_{\min} = 0.667$, $T_{\max} = 0.746$

12259 measured reflections

3169 independent reflections

2133 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.033$ $\theta_{\text{max}} = 28.3$ °, $\theta_{\text{min}} = 2.3$ ° $h = -12 \rightarrow 12$ $k = -14 \rightarrow 18$ $l = -14 \rightarrow 13$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.053$ $wR(F^2) = 0.162$ $S = 1.03$

3169 reflections

158 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sitesH atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0708P)^2 + 0.303P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} < 0.001$ $\Delta\rho_{\text{max}} = 0.26$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.21$ e Å⁻³*Special details***Geometry.** Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles**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)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.56542 (16)	0.15949 (11)	1.12382 (14)	0.0777 (5)
O2	0.19690 (13)	0.02459 (8)	0.46195 (11)	0.0537 (4)
O3	0.11891 (13)	0.03028 (9)	0.22128 (11)	0.0565 (4)
N1	0.10656 (17)	0.13011 (11)	0.58487 (15)	0.0547 (5)

C1	0.5636 (3)	0.09930 (17)	1.2298 (2)	0.0825 (8)
C2	0.4475 (2)	0.14896 (12)	0.99327 (18)	0.0562 (6)
C3	0.4759 (2)	0.17933 (13)	0.88187 (19)	0.0591 (6)
C4	0.36687 (19)	0.17229 (13)	0.74810 (18)	0.0563 (6)
C5	0.22273 (18)	0.13465 (11)	0.72040 (17)	0.0477 (5)
C6	0.1945 (2)	0.10636 (13)	0.83313 (19)	0.0599 (6)
C7	0.3056 (2)	0.11259 (13)	0.96875 (19)	0.0641 (7)
C8	0.14159 (19)	0.12184 (12)	0.46695 (17)	0.0521 (5)
C9	0.0083 (2)	0.14104 (13)	0.33069 (18)	0.0573 (6)
C10	0.0541 (2)	0.12534 (12)	0.21047 (18)	0.0585 (6)
C11	0.24203 (19)	0.01538 (14)	0.35085 (18)	0.0591 (6)
C12	0.3020 (3)	-0.08501 (18)	0.3532 (2)	0.0865 (9)
C13	-0.0748 (2)	0.13359 (16)	0.0683 (2)	0.0753 (7)
H1	0.028 (2)	0.0955 (14)	0.576 (2)	0.065 (6)*
H1A	0.65040	0.11310	1.31480	0.1240*
H1B	0.56700	0.03290	1.20440	0.1240*
H1C	0.47210	0.11050	1.24280	0.1240*
H3	0.57060	0.20510	0.89760	0.0710*
H4	0.38920	0.19290	0.67460	0.0680*
H6	0.09880	0.08260	0.81800	0.0720*
H7	0.28420	0.09220	1.04290	0.0770*
H8	0.22250	0.16810	0.47710	0.0630*
H9A	-0.02650	0.20710	0.32900	0.0690*
H9B	-0.07490	0.09790	0.32100	0.0690*
H10	0.13190	0.17330	0.21760	0.0700*
H11	0.32240	0.06250	0.36270	0.0710*
H12A	0.33440	-0.09200	0.27890	0.1300*
H12B	0.22330	-0.13120	0.34140	0.1300*
H12C	0.38680	-0.09610	0.44080	0.1300*
H13A	-0.03690	0.12280	-0.00170	0.1130*
H13B	-0.11850	0.19710	0.05700	0.1130*
H13C	-0.15100	0.08610	0.05900	0.1130*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0820 (9)	0.0918 (10)	0.0602 (8)	-0.0148 (8)	0.0302 (7)	0.0000 (7)
O2	0.0582 (6)	0.0538 (7)	0.0540 (7)	0.0064 (5)	0.0281 (5)	0.0009 (5)
O3	0.0587 (7)	0.0601 (7)	0.0520 (7)	-0.0045 (5)	0.0243 (6)	-0.0050 (5)
N1	0.0539 (8)	0.0563 (9)	0.0579 (9)	-0.0016 (6)	0.0270 (7)	-0.0055 (6)
C1	0.1004 (16)	0.0807 (14)	0.0697 (13)	0.0130 (12)	0.0384 (12)	0.0102 (11)
C2	0.0630 (10)	0.0509 (9)	0.0562 (10)	-0.0005 (8)	0.0262 (8)	-0.0032 (7)
C3	0.0561 (9)	0.0619 (11)	0.0642 (11)	-0.0034 (8)	0.0297 (9)	0.0005 (8)
C4	0.0596 (10)	0.0594 (10)	0.0575 (10)	0.0003 (8)	0.0317 (8)	0.0031 (8)
C5	0.0566 (9)	0.0365 (7)	0.0551 (9)	0.0039 (6)	0.0281 (8)	-0.0046 (6)
C6	0.0675 (10)	0.0547 (10)	0.0676 (11)	-0.0132 (8)	0.0381 (9)	-0.0071 (8)
C7	0.0876 (13)	0.0588 (11)	0.0595 (11)	-0.0108 (9)	0.0439 (10)	-0.0019 (8)
C8	0.0576 (9)	0.0466 (9)	0.0546 (10)	-0.0039 (7)	0.0258 (8)	-0.0028 (7)

C9	0.0642 (10)	0.0461 (9)	0.0591 (10)	0.0038 (7)	0.0228 (8)	0.0013 (7)
C10	0.0661 (10)	0.0501 (10)	0.0569 (10)	-0.0124 (8)	0.0231 (8)	0.0030 (7)
C11	0.0503 (9)	0.0752 (12)	0.0559 (10)	-0.0014 (8)	0.0259 (8)	-0.0070 (8)
C12	0.0845 (14)	0.1010 (17)	0.0764 (14)	0.0342 (12)	0.0356 (12)	-0.0071 (12)
C13	0.0867 (14)	0.0712 (13)	0.0582 (11)	-0.0038 (11)	0.0200 (10)	0.0090 (9)

Geometric parameters (Å, °)

O1—C1	1.399 (3)	C1—H1A	0.9600
O1—C2	1.385 (2)	C1—H1B	0.9600
O2—C8	1.455 (2)	C1—H1C	0.9600
O2—C11	1.413 (2)	C3—H3	0.9300
O3—C10	1.439 (2)	C4—H4	0.9300
O3—C11	1.407 (2)	C6—H6	0.9300
N1—C5	1.407 (2)	C7—H7	0.9300
N1—C8	1.421 (2)	C8—H8	0.9800
N1—H1	0.87 (2)	C9—H9A	0.9700
C2—C7	1.379 (3)	C9—H9B	0.9700
C2—C3	1.377 (3)	C10—H10	0.9800
C3—C4	1.371 (3)	C11—H11	0.9800
C4—C5	1.398 (3)	C12—H12A	0.9600
C5—C6	1.380 (3)	C12—H12B	0.9600
C6—C7	1.391 (3)	C12—H12C	0.9600
C8—C9	1.504 (2)	C13—H13A	0.9600
C9—C10	1.520 (3)	C13—H13B	0.9600
C10—C13	1.506 (3)	C13—H13C	0.9600
C11—C12	1.499 (3)		
C1—O1—C2	117.08 (17)	C4—C3—H3	119.00
C8—O2—C11	110.86 (13)	C3—C4—H4	119.00
C10—O3—C11	112.07 (13)	C5—C4—H4	119.00
C5—N1—C8	120.95 (16)	C5—C6—H6	119.00
C8—N1—H1	111.9 (13)	C7—C6—H6	119.00
C5—N1—H1	115.4 (13)	C2—C7—H7	120.00
O1—C2—C7	124.74 (17)	C6—C7—H7	120.00
O1—C2—C3	116.39 (18)	O2—C8—H8	109.00
C3—C2—C7	118.86 (17)	N1—C8—H8	109.00
C2—C3—C4	121.14 (19)	C9—C8—H8	109.00
C3—C4—C5	121.08 (17)	C8—C9—H9A	110.00
C4—C5—C6	117.26 (16)	C8—C9—H9B	110.00
N1—C5—C6	120.25 (17)	C10—C9—H9A	110.00
N1—C5—C4	122.40 (16)	C10—C9—H9B	110.00
C5—C6—C7	121.66 (19)	H9A—C9—H9B	108.00
C2—C7—C6	119.98 (18)	O3—C10—H10	108.00
N1—C8—C9	113.75 (16)	C9—C10—H10	108.00
O2—C8—N1	109.21 (13)	C13—C10—H10	108.00
O2—C8—C9	108.07 (13)	O2—C11—H11	109.00
C8—C9—C10	110.04 (16)	O3—C11—H11	109.00

O3—C10—C13	107.55 (14)	C12—C11—H11	109.00
C9—C10—C13	114.46 (17)	C11—C12—H12A	109.00
O3—C10—C9	109.30 (14)	C11—C12—H12B	110.00
O3—C11—C12	108.48 (15)	C11—C12—H12C	109.00
O2—C11—O3	111.45 (15)	H12A—C12—H12B	109.00
O2—C11—C12	108.56 (16)	H12A—C12—H12C	109.00
O1—C1—H1A	109.00	H12B—C12—H12C	109.00
O1—C1—H1B	109.00	C10—C13—H13A	109.00
O1—C1—H1C	109.00	C10—C13—H13B	109.00
H1A—C1—H1B	109.00	C10—C13—H13C	109.00
H1A—C1—H1C	110.00	H13A—C13—H13B	109.00
H1B—C1—H1C	109.00	H13A—C13—H13C	109.00
C2—C3—H3	119.00	H13B—C13—H13C	110.00
C1—O1—C2—C3	-156.71 (19)	O1—C2—C3—C4	-179.91 (17)
C1—O1—C2—C7	24.7 (3)	O1—C2—C7—C6	179.13 (17)
C11—O2—C8—N1	-176.31 (14)	C3—C2—C7—C6	0.5 (3)
C11—O2—C8—C9	59.47 (19)	C7—C2—C3—C4	-1.2 (3)
C8—O2—C11—O3	-62.20 (18)	C2—C3—C4—C5	0.5 (3)
C8—O2—C11—C12	178.39 (16)	C3—C4—C5—N1	177.38 (17)
C11—O3—C10—C9	-55.69 (19)	C3—C4—C5—C6	0.9 (3)
C11—O3—C10—C13	179.51 (15)	N1—C5—C6—C7	-178.12 (16)
C10—O3—C11—O2	60.39 (19)	C4—C5—C6—C7	-1.6 (3)
C10—O3—C11—C12	179.85 (17)	C5—C6—C7—C2	0.9 (3)
C8—N1—C5—C4	28.2 (2)	O2—C8—C9—C10	-55.53 (18)
C8—N1—C5—C6	-155.40 (16)	N1—C8—C9—C10	-176.99 (14)
C5—N1—C8—O2	72.63 (19)	C8—C9—C10—O3	53.91 (19)
C5—N1—C8—C9	-166.56 (15)	C8—C9—C10—C13	174.58 (15)

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of ring C2-C7 ring.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C6—H6...O3 ⁱ	0.93	2.51	3.407 (2)	162
C1—H1B...Cg1 ⁱⁱ	0.96	2.77	3.700 (3)	163

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