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2-Methyl-5-nitrobenzenesulfonamide

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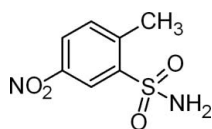
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.043; wR factor = 0.099; data-to-parameter ratio = 15.5.

In the title compound, $\text{C}_7\text{H}_8\text{N}_2\text{O}_4\text{S}$, the nitro group is twisted by $9.61(2)^\circ$ relative to the benzene ring. In the crystal, molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots(\text{O},\text{O})$ hydrogen bonds between the amino and sulfonyl groups, forming layers parallel to (001).

Related literature

For the biological activity of sulfonamides, see: Ozbek *et al.* (2007); Parari *et al.* (2008); Ratish *et al.* (2009); Selnam *et al.* (2001). For related structures, see: Arshad *et al.* (2009); Gowda *et al.* (2007*a,b,c*); Khan *et al.* (2009); Haider *et al.* (2009). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

 $\text{C}_7\text{H}_8\text{N}_2\text{O}_4\text{S}$ $M_r = 216.21$ Orthorhombic, $P2_12_12_1$ $a = 4.9872(4)$ Å $b = 6.2814(5)$ Å $c = 28.557(2)$ Å $V = 894.60(12)$ Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.35$ mm⁻¹ $T = 296$ K $0.43 \times 0.17 \times 0.11$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

 $T_{\min} = 0.864$, $T_{\max} = 0.962$

5964 measured reflections

2113 independent reflections

1549 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.036$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.043$ $wR(F^2) = 0.099$ $S = 0.89$

2112 reflections

136 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\max} = 0.23$ e Å⁻³ $\Delta\rho_{\min} = -0.23$ e Å⁻³

Absolute structure: Flack (1983),

766 Friedel pairs

Flack parameter: $-0.02(11)$

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N3}-\text{H1N}\cdots\text{O4}^{\text{i}}$	0.83 (4)	2.27 (4)	3.055 (4)	158 (3)
$\text{N3}-\text{H2N}\cdots\text{O4}^{\text{ii}}$	0.89 (6)	2.30 (6)	3.107 (4)	150 (4)
$\text{N3}-\text{H2N}\cdots\text{O3}^{\text{iii}}$	0.89 (6)	2.40 (4)	2.893 (4)	115 (3)

Symmetry codes: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x + 1, y, z$; (iii) $-x + 2, y + \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: PLATON (Spek, 2009) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2494).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans 2*, pp. S1–19.
- Arshad, M. N., Khan, I. U., Zia-ur-Rehman, M. & Shafiq, M. (2009). *Acta Cryst. E* **65**, o1204.
- Bruker (2007). APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Flack, H. D. (1983). *Acta Cryst. A* **39**, 876–881.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*a*). *Acta Cryst. E* **63**, o2339.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*b*). *Acta Cryst. E* **63**, o2570.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*c*). *Acta Cryst. E* **63**, o2597.
- Haider, Z., Khan, I. U., Zia-ur-Rehman, M. & Arshad, M. N. (2009). *Acta Cryst. E* **65**, o3053.
- Khan, I. U., Haider, Z., Zia-ur-Rehman, M., Arshad, M. N. & Shafiq, M. (2009). *Acta Cryst. E* **65**, o2867.
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
- Ozbek, N., Katirciođ lu, H., Karacan, N. & Baykal, T. (2007). *Bioorg. Med. Chem.* **15**, 5105–5109.
- Parari, M. K., Panda, G., Srivastava, K. & Puri, S. K. (2008). *Bioorg. Med. Chem. Lett.* **18**, 776–781.
- Ratish, G. I., Javed, K., Ahmad, S., Bano, S., Alam, M. S., Pillai, K. K., Singh, S. & Bagchi, V. (2009). *Bioorg. Med. Chem. Lett.* **19**, 255–258.
- Selnam, P., Chandramohan, M., Clercq, E. D., Witvrouw, M. & Pannecouque, C. (2001). *Eur. J. Pharm. Sci.* **14**, 313–316.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst. D* **65**, 148–155.

supporting information

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2-Methyl-5-nitrobenzenesulfonamide

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S1. Comment

Sulfonamides are familiar for their anti-HIV (Selnam *et al.*, 2001), anti-inflammatory (Ratish *et al.*, 2009) and anti-microbial (Ozbek *et al.*, 2007; Parari *et al.*, 2008) activities. In continuation of our work regarding the synthesis of various sulfonamides (Arshad *et al.*, 2009; Khan *et al.*, 2009), structure of 2-methyl-5-nitrobenzenesulfonamide (**I**) has been determined. Bond lengths and bond angles of the title molecule (Fig. 1) are almost similar to those in the related molecules (Gowda *et al.*, 2007*a,b,c*; Haider *et al.*, 2009) and are within the normal ranges (Allen *et al.*, 1987). Each molecule is linked to its adjacent ones through intermolecular N—H \cdots O hydrogen bonds forming a chain along the *a* axis, while each chain is linked to its neighbouring chain running in opposite direction *via* intermolecular N—H \cdots O=S hydrogen bonds (Table 1 and Fig. 2).

S2. Experimental

A well ground mixture of 2-methyl-5-nitrobenzenesulfonyl chloride (2.36 g, 10.0 mmol) and ammonium carbonate (10.0 g) was heated in a china dish till the complete removal of typical smell of sulfonyl chloride. Contents were cooled and washed with water followed by crystallization from methanol.

S3. Refinement

All H atoms were identified in a difference map and then were treated as riding (C—H = 0.93 or 0.97 Å), with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The reflection '0 0 2' affected by beamstop was removed during refinement.

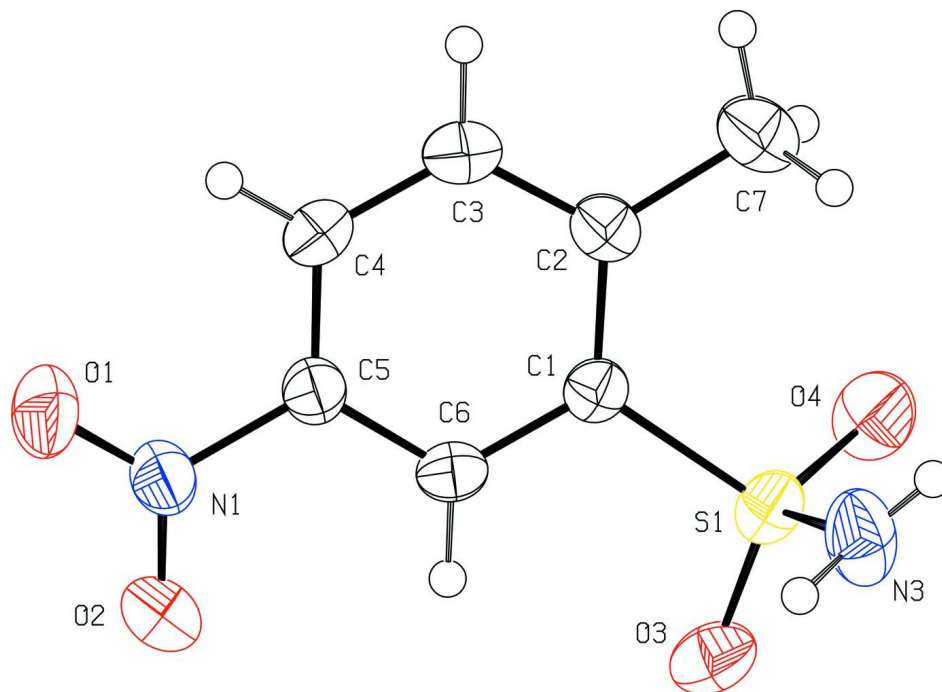


Figure 1

The molecular structure of (I), with displacement ellipsoids at the 50% probability level.

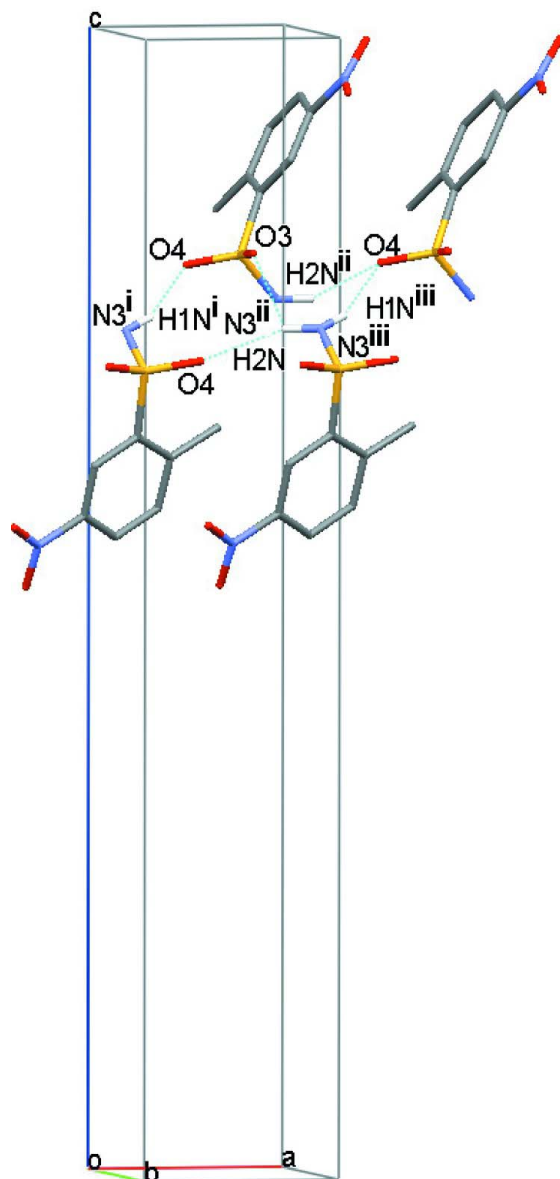


Figure 2

Perspective view of the three-dimensional crystal packing showing intermolecular hydrogen-bonded interactions (dashed lines). [Symmetry codes: (i) $-x + 1, y + 1/2, -z + 3/2$; (ii) $x + 1, y, z$ and (iii) $-x + 2, y + 1/2, -z + 3/2$]. H atoms not involved in hydrogen bonding have been omitted for clarity.

2-Methyl-5-nitrobenzenesulfonamide

Crystal data

$C_7H_8N_2O_4S$

$M_r = 216.21$

Orthorhombic, $P2_12_12_1$

Hall symbol: $P\ 2ac\ 2ab$

$a = 4.9872\ (4)\ \text{\AA}$

$b = 6.2814\ (5)\ \text{\AA}$

$c = 28.557\ (2)\ \text{\AA}$

$V = 894.60\ (12)\ \text{\AA}^3$

$Z = 4$

$F(000) = 448$

$D_x = 1.605\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1325 reflections

$\theta = 2.9\text{--}25.4^\circ$

$\mu = 0.35 \text{ mm}^{-1}$
 $T = 296 \text{ K}$

Needles, colourless
 $0.43 \times 0.17 \times 0.11 \text{ mm}$

Data collection

Bruker APEXII CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.864$, $T_{\max} = 0.962$

5964 measured reflections
 2113 independent reflections
 1549 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$
 $\theta_{\max} = 28.3^\circ$, $\theta_{\min} = 1.4^\circ$
 $h = -3 \rightarrow 6$
 $k = -8 \rightarrow 8$
 $l = -38 \rightarrow 38$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.099$
 $S = 0.89$
 2112 reflections
 136 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
 $w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.1986P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e } \text{Å}^{-3}$
 Absolute structure: Flack (1983), 766 Friedel
 pairs
 Absolute structure parameter: -0.02 (11)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.7436 (6)	0.4718 (4)	0.85981 (8)	0.0294 (6)
C2	0.6274 (6)	0.6621 (4)	0.87573 (9)	0.0336 (6)
C3	0.7125 (7)	0.7374 (4)	0.91916 (10)	0.0433 (8)
H3	0.6392	0.8629	0.9308	0.052*
C4	0.9018 (6)	0.6316 (5)	0.94542 (9)	0.0425 (7)
H4	0.9568	0.6857	0.9742	0.051*
C5	1.0083 (7)	0.4455 (4)	0.92867 (9)	0.0342 (6)
C6	0.9298 (6)	0.3616 (4)	0.88605 (8)	0.0330 (6)
H6	1.0009	0.2338	0.8753	0.040*
C7	0.4234 (7)	0.7867 (5)	0.84858 (11)	0.0473 (9)
H7A	0.3861	0.9178	0.8646	0.071*
H7B	0.2615	0.7051	0.8459	0.071*

H7C	0.4922	0.8171	0.8179	0.071*
N1	1.2116 (5)	0.3335 (4)	0.95582 (8)	0.0431 (6)
O4	0.3903 (4)	0.3752 (4)	0.79524 (7)	0.0525 (6)
O1	1.3007 (5)	0.4194 (3)	0.99090 (7)	0.0568 (6)
O2	1.2851 (5)	0.1593 (4)	0.94205 (8)	0.0677 (7)
O3	0.7986 (4)	0.1599 (3)	0.80208 (7)	0.0465 (5)
N3	0.8152 (8)	0.5117 (5)	0.76612 (9)	0.0532 (8)
S1	0.67301 (15)	0.36352 (11)	0.80358 (2)	0.03595 (19)
H1N	0.720 (8)	0.605 (5)	0.7540 (12)	0.062 (12)*
H2N	0.994 (12)	0.509 (7)	0.7660 (14)	0.091 (16)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0257 (18)	0.0334 (12)	0.0292 (11)	−0.0031 (12)	0.0030 (11)	0.0005 (10)
C2	0.0294 (18)	0.0338 (13)	0.0374 (12)	−0.0008 (13)	0.0030 (11)	0.0044 (11)
C3	0.046 (2)	0.0403 (14)	0.0437 (15)	0.0085 (15)	0.0036 (15)	−0.0086 (12)
C4	0.047 (2)	0.0469 (15)	0.0334 (12)	0.0080 (17)	−0.0022 (13)	−0.0102 (13)
C5	0.0295 (18)	0.0429 (14)	0.0301 (12)	0.0029 (13)	0.0029 (12)	0.0031 (11)
C6	0.0313 (17)	0.0345 (12)	0.0333 (12)	0.0013 (14)	0.0065 (11)	−0.0029 (12)
C7	0.045 (2)	0.0428 (16)	0.0541 (17)	0.0074 (15)	0.0010 (16)	0.0078 (13)
N1	0.0377 (16)	0.0553 (14)	0.0362 (11)	0.0095 (15)	−0.0022 (11)	0.0052 (11)
O4	0.0291 (12)	0.0768 (14)	0.0515 (12)	−0.0103 (12)	−0.0018 (9)	−0.0106 (11)
O1	0.0521 (16)	0.0746 (15)	0.0436 (11)	0.0029 (13)	−0.0166 (11)	−0.0034 (10)
O2	0.0711 (19)	0.0721 (14)	0.0598 (14)	0.0387 (16)	−0.0127 (13)	−0.0080 (12)
O3	0.0495 (14)	0.0433 (10)	0.0467 (10)	−0.0050 (11)	0.0040 (10)	−0.0132 (9)
N3	0.039 (2)	0.079 (2)	0.0415 (13)	−0.0008 (19)	0.0032 (15)	0.0203 (14)
S1	0.0296 (4)	0.0473 (4)	0.0310 (3)	−0.0068 (4)	0.0021 (3)	−0.0038 (3)

Geometric parameters (Å, °)

C1—C6	1.379 (4)	C6—H6	0.9300
C1—C2	1.404 (4)	C7—H7A	0.9600
C1—S1	1.779 (3)	C7—H7B	0.9600
C2—C3	1.393 (4)	C7—H7C	0.9600
C2—C7	1.499 (4)	N1—O1	1.221 (3)
C3—C4	1.377 (4)	N1—O2	1.219 (3)
C3—H3	0.9300	O4—S1	1.432 (2)
C4—C5	1.370 (4)	O3—S1	1.425 (2)
C4—H4	0.9300	N3—S1	1.586 (3)
C5—C6	1.383 (3)	N3—H1N	0.83 (4)
C5—N1	1.457 (4)	N3—H2N	0.89 (6)
C6—C1—C2	122.0 (2)	C2—C7—H7A	109.5
C6—C1—S1	115.57 (19)	C2—C7—H7B	109.5
C2—C1—S1	122.4 (2)	H7A—C7—H7B	109.5
C3—C2—C1	116.8 (2)	C2—C7—H7C	109.5
C3—C2—C7	119.3 (3)	H7A—C7—H7C	109.5

C1—C2—C7	123.9 (2)	H7B—C7—H7C	109.5
C4—C3—C2	122.0 (3)	O1—N1—O2	123.5 (3)
C4—C3—H3	119.0	O1—N1—C5	118.5 (2)
C2—C3—H3	119.0	O2—N1—C5	118.1 (2)
C5—C4—C3	119.2 (3)	S1—N3—H1N	116 (3)
C5—C4—H4	120.4	S1—N3—H2N	116 (3)
C3—C4—H4	120.4	H1N—N3—H2N	126 (4)
C4—C5—C6	121.5 (3)	O3—S1—O4	118.28 (15)
C4—C5—N1	119.7 (2)	O3—S1—N3	108.08 (18)
C6—C5—N1	118.8 (2)	O4—S1—N3	107.33 (18)
C1—C6—C5	118.5 (2)	O3—S1—C1	106.47 (12)
C1—C6—H6	120.7	O4—S1—C1	108.99 (13)
C5—C6—H6	120.7	N3—S1—C1	107.21 (15)
C6—C1—C2—C3	1.2 (4)	N1—C5—C6—C1	-177.9 (2)
S1—C1—C2—C3	-175.9 (2)	C4—C5—N1—O1	-6.9 (4)
C6—C1—C2—C7	-179.7 (3)	C6—C5—N1—O1	172.3 (3)
S1—C1—C2—C7	3.3 (4)	C4—C5—N1—O2	173.7 (3)
C1—C2—C3—C4	0.1 (4)	C6—C5—N1—O2	-7.1 (4)
C7—C2—C3—C4	-179.1 (3)	C6—C1—S1—O3	9.8 (2)
C2—C3—C4—C5	-0.6 (5)	C2—C1—S1—O3	-173.0 (2)
C3—C4—C5—C6	0.0 (4)	C6—C1—S1—O4	138.5 (2)
C3—C4—C5—N1	179.2 (3)	C2—C1—S1—O4	-44.3 (3)
C2—C1—C6—C5	-1.8 (4)	C6—C1—S1—N3	-105.7 (2)
S1—C1—C6—C5	175.4 (2)	C2—C1—S1—N3	71.6 (3)
C4—C5—C6—C1	1.3 (4)		

Hydrogen-bond geometry (Å, °)

<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>
N3—H1N...O4 ⁱ	0.83 (4)	2.27 (4)	3.055 (4)	158 (3)
N3—H2N...O4 ⁱⁱ	0.89 (6)	2.30 (6)	3.107 (4)	150 (4)
N3—H2N...O3 ⁱⁱⁱ	0.89 (6)	2.40 (4)	2.893 (4)	115 (3)

Symmetry codes: (i) $-x+1, y+1/2, -z+3/2$; (ii) $x+1, y, z$; (iii) $-x+2, y+1/2, -z+3/2$.