# organic compounds

# 4-Iodoanilinium nitrate

## Xue-qun Fu

Ordered Matter Science Research Center, Southeast University, Nanjing 210096, People's Republic of China
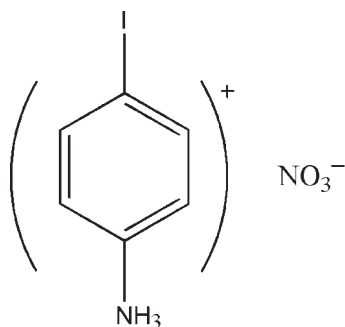Correspondence e-mail: fuxuequn222@163.com

In the title compound, $C_6H_7IN^+ \cdot NO_3^-$, $\pi$–$\pi$ stacking interactions [centroid–centroid distances = 4.014 (4) and 4.029 (4) Å] stabilize the crystal structure and strong N—H···O and N—H···N hydrogen bonds link the cations and anions into zigzag chains running parallel to the $c$ axis. The asymmetric unit contains two unique cations and anions

## Related literature

For background to phase-transition materials, see: Li *et al.* (2008); Zhang *et al.* (2009).



## Experimental

### Crystal data

$C_6H_7IN^+ \cdot NO_3^-$
$M_r = 282.04$
Monoclinic, $P2_1/c$
$a = 21.847$ (4) Å
$b = 5.6103$ (11) Å
$c = 15.928$ (3) Å
$\beta = 110.11$ (3)°

$V = 1833.2$ (6) Å$^3$
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 3.47$ mm$^{-1}$
$T = 298$ K
$0.40 \times 0.30 \times 0.20$ mm

### Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.5$, $T_{max} = 0.5$

17732 measured reflections
4196 independent reflections
3075 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.112$
$S = 1.13$
4196 reflections

217 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 1.07$ e Å$^{-3}$
$\Delta\rho_{min} = -0.78$ e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$B$···O4$^i$ | 0.89 | 2.18 | 2.945 (8) | 144 |
| N1—H1$C$···O2$^{ii}$ | 0.89 | 2.24 | 2.819 (8) | 122 |
| N1—H1$C$···O5$^i$ | 0.89 | 2.46 | 3.010 (8) | 120 |
| N1—H1$D$···O3$^i$ | 0.89 | 2.46 | 2.898 (8) | 111 |
| N1—H1$D$···I1$^{ii}$ | 0.89 | 3.15 | 3.990 (6) | 157 |
| N2—H2$B$···O6$^{iii}$ | 0.89 | 2.03 | 2.892 (8) | 162 |
| N2—H2$B$···O4$^{iii}$ | 0.89 | 2.44 | 3.104 (8) | 132 |
| N2—H2$B$···N4$^{iii}$ | 0.89 | 2.55 | 3.372 (8) | 153 |
| N2—H2$C$···O3$^{iv}$ | 0.89 | 1.91 | 2.795 (7) | 173 |
| N2—H2$C$···N3$^{iv}$ | 0.89 | 2.56 | 3.407 (8) | 159 |
| N2—H2$C$···O1$^{iv}$ | 0.89 | 2.59 | 3.264 (8) | 133 |
| N2—H2$D$···O5$^v$ | 0.89 | 2.18 | 3.018 (8) | 157 |
| N2—H2$D$···O6$^v$ | 0.89 | 2.28 | 3.053 (8) | 145 |
| N2—H2$D$···N4$^v$ | 0.89 | 2.58 | 3.459 (9) | 170 |

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PRPKAPPA* (Ferguson, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2153).

## References

Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
Li, X. Z., Qu, Z. R. & Xiong, R. G. (2008). *Chin. J. Chem.* **11**, 1959–1962
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zhang, W., Chen, L. Z., Xiong, R. G., Nakamura, T. & Huang, S. D. (2009). *J. Am. Chem. Soc.* **131**, 12544–12545

**supplementary materials**

## 4-Iodoanilinium nitrate

**X. Fu**

### Comment

As a continuation of our study of phase transition materials, including organic ligands (Li *et al.*, 2008), metal-organic coordination compounds (Zhang *et al.*, 2009 ), organic-inorganic hybrids, we studied the dielectric properties of the title compound, unfortunately, there was no distinct anomaly observed from 93 K to 380 K, (m.p. 408 K-410 K). In this article, the crystal structure of (I) has been presented.

The asymmetric unit of the title compound is built up from two 4-iodobenzenammnium cations wherein the dihedral angle between plans formed by non-hydrogen atoms is 15.3 (2)°,and two nitrate radical anions (Fig.1). The π-π packing interaction of adjacent benzene rings with Cg(1)—Cg(1), 4.029 (4)Å; Cg(2)—Cg(2), 4.014 (4)Å [Cg(1) and Cg(2) are the centroids of benzene rings, where Cg(1): C(1) to C(6); Cg(2): C(7) to C(12)], make great contribution to the stability of the crystal structure. The strong intermolecular N—H···O (N···O distances 2.795 (7)-3.264 (8)Å) and N—H···N (N···N distances 3.372 (8)-3.459 (9)Å) hydrogen bonding link cations and anions into zigzag chains along *c* axis.

### Experimental

Single crystals of 4-iodoanilinium nitrate were prepared by slow evaporation at room temperature of an ethanol solution of equal molar 4-iodobenzenamine and nitrate acid.

### Refinement

Positional parameters of all the H atoms were calculated geometrically and were allowed to ride on the C and N atoms to which they are bonded, with $U_{iso}(H) = 1.2U_{eq}(C)$,
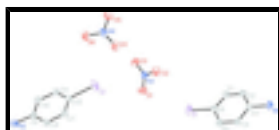
$U_{iso}(H) = 1.2U_{eq}(N)$.

### Figures



Fig. 1. The molecular structure of the title compound, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level, and all H atoms have been omitted for clarity.
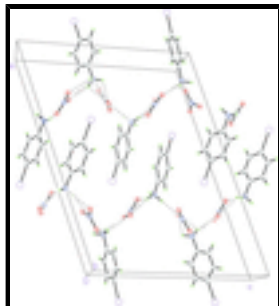
# supplementary materials



Fig. 2. A view of the packing of the title compound, stacking along the *c* axis. Dashed lines indicate hydrogen bonds.

**4-iodoanilinium nitrate**

*Crystal data*

| | |
|---|---|
| $C_6H_7IN^+ \cdot NO_3^-$ | $F(000) = 1072$ |
| $M_r = 282.04$ | $D_x = 2.044$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 7323 reflections |
| $a = 21.847$ (4) Å | $\theta = 3.2–27.6°$ |
| $b = 5.6103$ (11) Å | $\mu = 3.47$ mm$^{-1}$ |
| $c = 15.928$ (3) Å | $T = 298$ K |
| $\beta = 110.11$ (3)° | Prism, colourless |
| $V = 1833.2$ (6) Å$^3$ | $0.40 \times 0.30 \times 0.20$ mm |
| $Z = 8$ | |

*Data collection*

| | |
|---|---|
| Rigaku SCXmini diffractometer | 4196 independent reflections |
| Radiation source: fine-focus sealed tube | 3075 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.041$ |
| Detector resolution: 13.6612 pixels mm$^{-1}$ | $\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$ |
| $\omega$ scans | $h = -28 \rightarrow 28$ |
| Absorption correction: multi-scan (CrystalClear; Rigaku, 2005) | $k = -7 \rightarrow 7$ |
| $T_{min} = 0.5$, $T_{max} = 0.5$ | $l = -20 \rightarrow 20$ |
| 17732 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.056$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.112$ | H-atom parameters constrained |
| $S = 1.13$ | $w = 1/[\sigma^2(F_o^2) + (0.0174P)^2 + 9.5262P]$ where $P = (F_o^2 + 2F_c^2)/3$ |

| 4196 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
|---|---|
| 217 parameters | $\Delta\rho_{max} = 1.07$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.78$ 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 $F^2 > \sigma(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| I2 | 0.38469 (2) | 0.37839 (12) | 0.76874 (3) | 0.0735 (2) |
| C10 | 0.5931 (3) | 0.3846 (12) | 0.9095 (4) | 0.0471 (15) |
| H10A | 0.6281 | 0.2874 | 0.9120 | 0.056* |
| N2 | 0.6682 (3) | 0.6372 (10) | 1.0215 (4) | 0.0513 (14) |
| H2B | 0.6678 | 0.7709 | 1.0513 | 0.062* |
| H2C | 0.6947 | 0.6550 | 0.9901 | 0.062* |
| H2D | 0.6823 | 0.5174 | 1.0599 | 0.062* |
| C11 | 0.5297 (3) | 0.3288 (13) | 0.8534 (4) | 0.0507 (16) |
| H11A | 0.5224 | 0.1926 | 0.8181 | 0.061* |
| C9 | 0.6020 (3) | 0.5850 (11) | 0.9604 (4) | 0.0412 (14) |
| C8 | 0.5520 (3) | 0.7336 (12) | 0.9573 (4) | 0.0503 (16) |
| H8A | 0.5596 | 0.8706 | 0.9923 | 0.060* |
| C12 | 0.4790 (3) | 0.4729 (12) | 0.8504 (4) | 0.0448 (15) |
| C7 | 0.4894 (3) | 0.6768 (13) | 0.9008 (5) | 0.0514 (16) |
| H7A | 0.4546 | 0.7769 | 0.8973 | 0.062* |
| I1 | 0.11990 (2) | 0.89729 (11) | 0.38039 (3) | 0.06947 (19) |
| C3 | −0.1001 (3) | 1.0935 (11) | 0.3605 (4) | 0.0424 (14) |
| N1 | −0.1670 (2) | 1.1490 (10) | 0.3549 (4) | 0.0528 (14) |
| H1B | −0.1941 | 1.0391 | 0.3218 | 0.063* |
| H1C | −0.1783 | 1.2916 | 0.3299 | 0.063* |
| H1D | −0.1694 | 1.1499 | 0.4096 | 0.063* |
| C6 | 0.0249 (3) | 0.9855 (12) | 0.3725 (4) | 0.0451 (15) |
| C2 | −0.0884 (3) | 0.8935 (12) | 0.3196 (4) | 0.0520 (16) |
| H2A | −0.1228 | 0.7944 | 0.2883 | 0.062* |
| C4 | −0.0498 (3) | 1.2412 (12) | 0.4073 (4) | 0.0497 (16) |
| H4A | −0.0581 | 1.3774 | 0.4349 | 0.060* |
| C5 | 0.0135 (3) | 1.1858 (13) | 0.4131 (5) | 0.0543 (17) |
| H5A | 0.0479 | 1.2850 | 0.4445 | 0.065* |
| C1 | −0.0264 (3) | 0.8386 (12) | 0.3247 (4) | 0.0511 (16) |

| | | | | |
|---|---|---|---|---|
| H1A | −0.0185 | 0.7033 | 0.2962 | 0.061* |
| O6 | 0.3304 (2) | −0.1221 (9) | 0.9162 (3) | 0.0657 (14) |
| N4 | 0.2778 (3) | −0.1278 (12) | 0.8524 (4) | 0.0553 (15) |
| O5 | 0.2512 (3) | −0.3234 (10) | 0.8268 (4) | 0.0745 (16) |
| O4 | 0.2531 (3) | 0.0559 (11) | 0.8148 (4) | 0.0826 (18) |
| N3 | 0.2248 (3) | 0.3536 (11) | 0.5730 (4) | 0.0560 (15) |
| O3 | 0.2487 (3) | 0.1485 (9) | 0.5783 (4) | 0.0686 (15) |
| O2 | 0.1727 (3) | 0.3781 (10) | 0.5883 (4) | 0.0749 (16) |
| O1 | 0.2517 (3) | 0.5249 (11) | 0.5535 (5) | 0.0885 (19) |

*Atomic displacement parameters ($\mathring{A}^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| I2 | 0.0463 (3) | 0.1119 (5) | 0.0590 (3) | −0.0160 (3) | 0.0141 (2) | −0.0082 (3) |
| C10 | 0.044 (3) | 0.054 (4) | 0.045 (3) | 0.005 (3) | 0.017 (3) | −0.002 (3) |
| N2 | 0.048 (3) | 0.054 (4) | 0.053 (3) | −0.010 (3) | 0.019 (3) | −0.005 (3) |
| C11 | 0.059 (4) | 0.054 (4) | 0.040 (3) | −0.003 (3) | 0.019 (3) | −0.009 (3) |
| C9 | 0.038 (3) | 0.048 (4) | 0.038 (3) | −0.005 (3) | 0.013 (3) | 0.002 (3) |
| C8 | 0.060 (4) | 0.042 (4) | 0.051 (4) | −0.005 (3) | 0.021 (3) | −0.005 (3) |
| C12 | 0.043 (3) | 0.059 (4) | 0.035 (3) | −0.004 (3) | 0.017 (3) | 0.002 (3) |
| C7 | 0.045 (4) | 0.053 (4) | 0.059 (4) | 0.007 (3) | 0.021 (3) | 0.002 (3) |
| I1 | 0.0449 (3) | 0.0962 (4) | 0.0641 (3) | 0.0188 (3) | 0.0146 (2) | −0.0032 (3) |
| C3 | 0.043 (3) | 0.047 (4) | 0.040 (3) | 0.003 (3) | 0.017 (3) | 0.000 (3) |
| N1 | 0.043 (3) | 0.060 (4) | 0.055 (3) | 0.006 (3) | 0.016 (3) | −0.003 (3) |
| C6 | 0.044 (3) | 0.056 (4) | 0.036 (3) | 0.014 (3) | 0.013 (3) | 0.005 (3) |
| C2 | 0.051 (4) | 0.056 (4) | 0.049 (4) | −0.007 (3) | 0.017 (3) | −0.012 (3) |
| C4 | 0.055 (4) | 0.040 (4) | 0.052 (4) | 0.004 (3) | 0.016 (3) | −0.009 (3) |
| C5 | 0.047 (4) | 0.056 (4) | 0.054 (4) | 0.000 (3) | 0.009 (3) | −0.014 (3) |
| C1 | 0.059 (4) | 0.052 (4) | 0.045 (4) | −0.001 (3) | 0.021 (3) | −0.014 (3) |
| O6 | 0.058 (3) | 0.069 (4) | 0.056 (3) | 0.000 (3) | 0.002 (2) | −0.001 (3) |
| N4 | 0.044 (3) | 0.061 (4) | 0.061 (4) | −0.004 (3) | 0.017 (3) | −0.005 (3) |
| O5 | 0.063 (3) | 0.063 (3) | 0.099 (4) | −0.019 (3) | 0.030 (3) | −0.016 (3) |
| O4 | 0.069 (4) | 0.069 (4) | 0.085 (4) | 0.000 (3) | −0.006 (3) | 0.009 (3) |
| N3 | 0.046 (3) | 0.058 (4) | 0.062 (4) | 0.005 (3) | 0.016 (3) | −0.007 (3) |
| O3 | 0.061 (3) | 0.057 (3) | 0.095 (4) | 0.015 (3) | 0.037 (3) | −0.001 (3) |
| O2 | 0.055 (3) | 0.071 (4) | 0.114 (5) | 0.008 (3) | 0.048 (3) | −0.007 (3) |
| O1 | 0.070 (4) | 0.063 (4) | 0.143 (6) | 0.001 (3) | 0.050 (4) | 0.010 (4) |

*Geometric parameters ($\mathring{A}$, °)*

| | | | |
|---|---|---|---|
| I2—C12 | 2.091 (6) | C3—N1 | 1.467 (7) |
| C10—C9 | 1.360 (9) | N1—H1B | 0.8900 |
| C10—C11 | 1.401 (9) | N1—H1C | 0.8900 |
| C10—H10A | 0.9300 | N1—H1D | 0.8900 |
| N2—C9 | 1.469 (7) | C6—C5 | 1.361 (9) |
| N2—H2B | 0.8900 | C6—C1 | 1.389 (9) |
| N2—H2C | 0.8900 | C2—C1 | 1.365 (9) |
| N2—H2D | 0.8900 | C2—H2A | 0.9300 |
| C11—C12 | 1.359 (9) | C4—C5 | 1.390 (9) |

| | | | |
|---|---|---|---|
| C11—H11A | 0.9300 | C4—H4A | 0.9300 |
| C9—C8 | 1.361 (9) | C5—H5A | 0.9300 |
| C8—C7 | 1.392 (9) | C1—H1A | 0.9300 |
| C8—H8A | 0.9300 | O6—N4 | 1.246 (7) |
| C12—C7 | 1.371 (9) | N4—O4 | 1.221 (8) |
| C7—H7A | 0.9300 | N4—O5 | 1.243 (7) |
| I1—C6 | 2.095 (6) | N3—O1 | 1.220 (8) |
| C3—C2 | 1.365 (9) | N3—O2 | 1.253 (7) |
| C3—C4 | 1.373 (9) | N3—O3 | 1.254 (7) |
| | | | |
| C9—C10—C11 | 118.2 (6) | C3—N1—H1B | 109.5 |
| C9—C10—H10A | 120.9 | C3—N1—H1C | 109.5 |
| C11—C10—H10A | 120.9 | H1B—N1—H1C | 109.5 |
| C9—N2—H2B | 109.5 | C3—N1—H1D | 109.5 |
| C9—N2—H2C | 109.5 | H1B—N1—H1D | 109.5 |
| H2B—N2—H2C | 109.5 | H1C—N1—H1D | 109.5 |
| C9—N2—H2D | 109.5 | C5—C6—C1 | 120.4 (6) |
| H2B—N2—H2D | 109.5 | C5—C6—I1 | 120.4 (5) |
| H2C—N2—H2D | 109.5 | C1—C6—I1 | 119.2 (5) |
| C12—C11—C10 | 120.3 (6) | C3—C2—C1 | 120.2 (6) |
| C12—C11—H11A | 119.8 | C3—C2—H2A | 119.9 |
| C10—C11—H11A | 119.8 | C1—C2—H2A | 119.9 |
| C10—C9—C8 | 122.4 (6) | C3—C4—C5 | 119.6 (6) |
| C10—C9—N2 | 117.7 (6) | C3—C4—H4A | 120.2 |
| C8—C9—N2 | 119.8 (6) | C5—C4—H4A | 120.2 |
| C9—C8—C7 | 118.8 (6) | C6—C5—C4 | 119.6 (6) |
| C9—C8—H8A | 120.6 | C6—C5—H5A | 120.2 |
| C7—C8—H8A | 120.6 | C4—C5—H5A | 120.2 |
| C11—C12—C7 | 120.5 (6) | C2—C1—C6 | 119.7 (6) |
| C11—C12—I2 | 119.2 (5) | C2—C1—H1A | 120.1 |
| C7—C12—I2 | 120.3 (5) | C6—C1—H1A | 120.1 |
| C12—C7—C8 | 119.8 (6) | O4—N4—O5 | 120.4 (6) |
| C12—C7—H7A | 120.1 | O4—N4—O6 | 120.4 (6) |
| C8—C7—H7A | 120.1 | O5—N4—O6 | 119.1 (7) |
| C2—C3—C4 | 120.6 (6) | O1—N3—O2 | 120.8 (6) |
| C2—C3—N1 | 119.4 (6) | O1—N3—O3 | 121.0 (6) |
| C4—C3—N1 | 120.0 (6) | O2—N3—O3 | 118.1 (6) |

*Hydrogen-bond geometry (Å, °)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| N1—H1B···O4[i] | 0.89 | 2.18 | 2.945 (8) | 144 |
| N1—H1C···O2[ii] | 0.89 | 2.24 | 2.819 (8) | 122 |
| N1—H1C···O5[i] | 0.89 | 2.46 | 3.010 (8) | 120 |
| N1—H1D···O3[i] | 0.89 | 2.46 | 2.898 (8) | 111 |
| N1—H1D···I1[ii] | 0.89 | 3.15 | 3.990 (6) | 157 |
| N2—H2B···O6[iii] | 0.89 | 2.03 | 2.892 (8) | 162 |
| N2—H2B···O4[iii] | 0.89 | 2.44 | 3.104 (8) | 132 |

| | | | | |
|---|---|---|---|---|
| N2—H2B···N4[iii] | 0.89 | 2.55 | 3.372 (8) | 153 |
| N2—H2C···O3[iv] | 0.89 | 1.91 | 2.795 (7) | 173 |
| N2—H2C···N3[iv] | 0.89 | 2.56 | 3.407 (8) | 159 |
| N2—H2C···O1[iv] | 0.89 | 2.59 | 3.264 (8) | 133 |
| N2—H2D···O5[v] | 0.89 | 2.18 | 3.018 (8) | 157 |
| N2—H2D···O6[v] | 0.89 | 2.28 | 3.053 (8) | 145 |
| N2—H2D···N4[v] | 0.89 | 2.58 | 3.459 (9) | 170 |

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

**Fig. 1**

**Fig. 2**