

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 2-Amino-6-(dimethylamino)pyridine-3,5-dicarbonitrile

Shaaban K. Mohamed,<sup>a</sup> Ahmed M. Soliman,<sup>b</sup> Eman M. M. Abdel-Raheem,<sup>b</sup> Sohail Saeed<sup>c\*</sup> and Wing-Tak Wong<sup>d</sup>

<sup>a</sup>Chemistry and Environmental Division, Manchester Metropolitan University, Manchester, M1 5GD, England, <sup>b</sup>Department of Chemistry, Faculty of Science, Sohag University, Egypt, <sup>c</sup>Department of Chemistry, Research Complex, Allama Iqbal Open University, Islamabad 44000, Pakistan, and <sup>d</sup>Department of Chemistry, The University of Hong Kong, Pokfulam Road, Pokfulam, Hong Kong SAR, People's Republic of China

Correspondence e-mail: sohail262001@yahoo.com

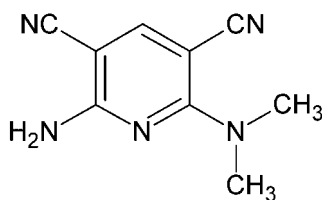
Received 13 April 2012; accepted 18 April 2012

Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.048;  $wR$  factor = 0.147; data-to-parameter ratio = 12.0.

The title compound,  $\text{C}_9\text{H}_9\text{N}_5$ , is slightly twisted from planarity, with a maximum deviation of 0.0285 (13) Å from the pyridine plane for the C atom bearing the amino group. The cyano groups are on different sides of the pyridine plane, with C- and N-atom deviations of 0.072 (3)/0.124 (4) and  $-0.228$  (4)/ $-0.409$  (5) Å from the pyridine plane. In the crystal,  $\text{N}-\text{H}\cdots\text{N}$  and  $\text{C}-\text{H}\cdots\text{N}$  hydrogen bonds connect the molecules into zigzag chains running along the  $c$  axis.

## Related literature

For the synthesis of similar structures, see: Horton *et al.* (2012*a,b*); Soliman *et al.* (2012). For the biological significance of cyanoamino pyridines, see: Al-Haiza *et al.* (2003); Bhalerao & Krishnaiah (1995); Deo *et al.* (1990); Murata *et al.* (2003); Konda *et al.* (2010); Altomare *et al.* (2000); Hosni & Abdulla (2008); Shishoo *et al.* (1983).



## Experimental

## Crystal data

$\text{C}_9\text{H}_9\text{N}_5$   
 $M_r = 187.21$   
 Monoclinic,  $C2/c$   
 $a = 28.667$  (7) Å  
 $b = 3.9702$  (10) Å  
 $c = 17.950$  (4) Å  
 $\beta = 112.920$  (3)°

$V = 1881.7$  (8) Å<sup>3</sup>  
 $Z = 8$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.32 \times 0.21 \times 0.03$  mm

## Data collection

Bruker SMART 1000 CCD  
 diffractometer  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 2004)  
 $T_{\min} = 0.972$ ,  $T_{\max} = 0.997$

4846 measured reflections  
 1658 independent reflections  
 1173 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.025$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$   
 $wR(F^2) = 0.147$   
 $S = 1.03$   
 1658 reflections  
 138 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.16$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.19$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H1}\cdots\text{N3}^{\text{i}}$	0.88 (3)	2.25 (3)	3.119 (3)	167 (2)
$\text{N2}-\text{H2}\cdots\text{N1}^{\text{ii}}$	0.88 (3)	2.43 (3)	3.260 (3)	158 (3)
$\text{C3}-\text{H3}\cdots\text{N4}^{\text{iii}}$	0.93	2.55	3.471 (4)	170

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

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: SHELXL97.

The authors are thankful to the University of Hong Kong for providing the single-crystal X-ray crystallography facility.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2369).

## References

- Al-Haiza, M. A., Mostafa, M. S. & El-Kady, M. Y. (2003). *Molecules*, **8**, 275–286.
- Altomare, C., Cellamare, S., Summo, L., Fossa, P., Mosti, L. & Carotti, A. (2000). *Bioorg. Med. Chem.* **8**, 909–916.
- Bhalerao, U. T. & Krishnaiah, A. (1995). *Indian J. Chem. Sect. B*, **34**, 587–590.
- Bruker (1998). SMART. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2006). SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
- Deo, K., Avasthi, K., Pratap, R., Bhakuni, D. S. & Joshi, M. N. (1990). *Indian J. Chem. Sect. B*, **29**, 459–463.
- Horton, P. N., Mohamed, S. K., Soliman, A. M., Abdel-Raheem, E. M. M. & Akkurt, M. (2012*a*). *Acta Cryst.* **E68**, o938.
- Horton, P. N., Mohamed, S. K., Soliman, A. M., Abdel-Raheem, E. M. M. & Akkurt, M. (2012*b*). *Acta Cryst.* **E68**, o885–o886.
- Hosni, H. M. & Abdulla, M. M. (2008). *Acta Pharm.* **58**, 175–186.
- Konda, S. G., Khedkar, V. T. & Dawane, B. S. (2010). *J. Chem. Pharm. Res.* **2**, 187–191.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Murata, T., Shimada, M., Sakakibara, S., Yoshino, T., Kadono, H., Masuda, T., Shimazaki, M., Shintani, T., Fuchikami, K., Sakai, K., Inbe, H., Takeshita, K., Niki, T., Umeda, M., Bacon, K. B., Ziegelbauer, K. B. & Lowinger, T. B. (2003). *Bioorg. Med. Chem. Lett.* **13**, 913–918.
- Sheldrick, G. M. (2004). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Shishoo, C. J., Devani, M. B., Bhadi, V. S., Ananthan, S. & Ullas, G. V. (1983). *Tetrahedron Lett.* **24**, 4611–4612.
- Soliman, A. M., Mohamed, S. K., El Remaily, M. A. A. & Abdel-Ghany, H. (2012). *Eur. J. Med. Chem.* **47**, 138–142.

## supplementary materials

*Acta Cryst.* (2012). E68, o1532 [doi:10.1107/S1600536812017278]

**2-Amino-6-(dimethylamino)pyridine-3,5-dicarbonitrile**

**Shaaban K. Mohamed, Ahmed M. Soliman, Eman M. M. Abdel-Raheem, Sohail Saeed and Wing-Tak Wong**

**Comment**

In continuation of our research interest in the synthesis of potential biologically active molecules (Soliman *et al.*, 2012; Horton *et al.*, 2012*a,b*), we got prompted to study the chemical and pharmacological characterization of new cyano-amino pyridine derivatives due to their vibrant chemical activities. Hence cyano-amino pyridines have been considered as convenient synthons due to their diverse applications particularly in organic synthesis (Shishoo *et al.*, 1983; Deo *et al.*, 1990; Bhalerao & Krishnaiah, 1995; Al-Haiza *et al.*, 2003) and medicinal chemistry (Altomare *et al.*, 2000; Hosni & Abdulla, 2008; Murata *et al.*, 2003; Konda *et al.*, 2010).

The title compound, 2-amino-6-(dimethylamino)-pyridine-3,5-dicarbonitrile, is slightly twisted. The maximum deviation from the mean plane of the pyridyl ring, N1/C1—C5 (marked with asterisk) is 0.0285 (13) Å. The cyano groups are flipped to different sides of the pyridine plane with atoms C6 & N3 showing deviations of +0.072 (3) Å and +0.124 (4) Å, while atoms C7 & N4 are bent out of the pyridine plane by -0.228 (4) Å and -0.409 (5) Å, respectively.

Hydrogen bonding interactions are observed in the crystal lattice connecting the molecules into zigzag chains running along the *c*-axis. As it is expected, N—H $\cdots$ N interactions are shorter as the observed N4 $\cdots$ H3(-C3) distance.

**Experimental**

The title compound (1) was obtained as a by-product from the reaction of 2-amino-6-chloropyridine-3,5-dicarbonitrile (1 mmol; 179 mg) with amino guanidine (1 mmol; 74 mg) in dimethylformamide. The reaction mixture was refluxed for 4 h at 426 K and then poured on cold water. A solid product was filtered off, dried and recrystallized from ethanol to afford cupric needles which were suitable for X-Ray diffraction without further recrystallization. Yield 45% and m.p. 453 K.

**Refinement**

The structure was solved by direct methods (*SHELXS97*, Sheldrick, 2008) and expanded using Fourier techniques. All non-H atoms were refined anisotropically.

C-bound H atoms are all placed at geometrical positions with C—H = 0.93 and 0.96 Å for phenyl and methyl H-atoms, respectively. C-bound phenyl hydrogen atoms are refined using a riding model with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ , methyl H-atoms are refined using a riding model with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ . N-bound H atoms were located from the difference Fourier map and were refined isotropically.

Highest peak is 0.16 at (0.1967, 0.2214, 0.0041) [1.04 Å from H8C] Deepest hole is -0.19 at (0.1226, 0.1471, 0.2613) [1.01 Å from C5]

## Computing details

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 2006); data reduction: *SAINTE* (Bruker, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

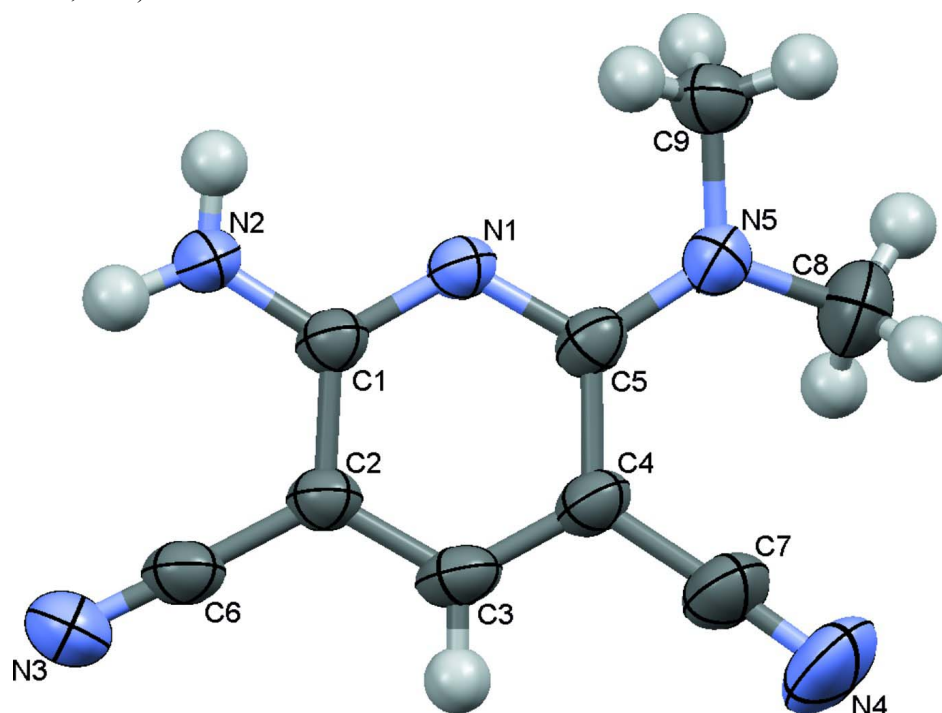


Figure 1

Molecular structure of the title compound showing thermal ellipsoids on the 50% probability level.

## 2-Amino-6-(dimethylamino)pyridine-3,5-dicarbonitrile

*Crystal data*

$C_9H_9N_5$

$M_r = 187.21$

Monoclinic,  $C2/c$

Hall symbol:  $-C 2yc$

$a = 28.667 (7) \text{ \AA}$

$b = 3.9702 (10) \text{ \AA}$

$c = 17.950 (4) \text{ \AA}$

$\beta = 112.920 (3)^\circ$

$V = 1881.7 (8) \text{ \AA}^3$

$Z = 8$

$F(000) = 784$

$D_x = 1.322 \text{ Mg m}^{-3}$

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

Cell parameters from 4846 reflections

$\theta = 3.1\text{--}25.0^\circ$

$\mu = 0.09 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Plate, yellow

$0.32 \times 0.21 \times 0.03 \text{ mm}$

*Data collection*

Bruker SMART 1000 CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  and  $\phi$  scans

Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 2004)

$T_{\min} = 0.972$ ,  $T_{\max} = 0.997$

4846 measured reflections

1658 independent reflections

1173 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$   
 $\theta_{\text{max}} = 25.0^\circ$ ,  $\theta_{\text{min}} = 3.1^\circ$   
 $h = -33 \rightarrow 34$

$k = -4 \rightarrow 4$   
 $l = -21 \rightarrow 18$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.048$   
 $wR(F^2) = 0.147$   
 $S = 1.03$   
 1658 reflections  
 138 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0915P)^2 + 0.1839P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.16 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.19 \text{ e } \text{\AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.07712 (6)	0.0877 (4)	0.27256 (9)	0.0417 (5)
N2	0.00997 (6)	0.2230 (6)	0.15638 (13)	0.0573 (6)
H1	-0.0050 (9)	0.284 (6)	0.1053 (17)	0.068 (8)*
H2	-0.0096 (12)	0.128 (8)	0.1777 (19)	0.094 (9)*
N3	0.05670 (8)	0.6406 (6)	0.02533 (12)	0.0669 (6)
N4	0.25896 (8)	0.0677 (9)	0.35083 (15)	0.1042 (10)
N5	0.14100 (6)	-0.0606 (5)	0.39210 (10)	0.0488 (5)
C1	0.06018 (7)	0.2187 (5)	0.19854 (12)	0.0415 (5)
C2	0.09339 (7)	0.3580 (5)	0.16489 (12)	0.0431 (5)
C3	0.14482 (7)	0.3269 (6)	0.20982 (13)	0.0499 (6)
H3	0.1676	0.4079	0.1887	0.060*
C4	0.16300 (7)	0.1796 (5)	0.28473 (13)	0.0463 (5)
C5	0.12699 (7)	0.0699 (5)	0.31732 (12)	0.0413 (5)
C6	0.07369 (8)	0.5132 (6)	0.08746 (14)	0.0498 (6)
C7	0.21622 (9)	0.1175 (7)	0.32283 (15)	0.0673 (7)
C8	0.18827 (9)	0.0208 (8)	0.45832 (15)	0.0782 (8)
H8A	0.1828	0.0356	0.5077	0.117*
H8B	0.2006	0.2328	0.4478	0.117*
H8C	0.2127	-0.1520	0.4634	0.117*
C9	0.10351 (8)	-0.2200 (6)	0.41700 (13)	0.0561 (6)
H9A	0.1197	-0.3882	0.4572	0.084*
H9B	0.0778	-0.3238	0.3710	0.084*

H9C            0.0884            -0.0530            0.4392            0.084\*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0341 (9)	0.0539 (10)	0.0397 (10)	-0.0009 (7)	0.0172 (7)	-0.0014 (8)
N2	0.0344 (10)	0.0948 (16)	0.0427 (12)	-0.0056 (9)	0.0150 (9)	0.0117 (11)
N3	0.0644 (13)	0.0872 (15)	0.0559 (13)	-0.0010 (11)	0.0308 (11)	0.0131 (12)
N4	0.0412 (12)	0.175 (3)	0.0971 (19)	0.0216 (15)	0.0270 (12)	-0.0016 (19)
N5	0.0376 (9)	0.0639 (11)	0.0435 (10)	0.0045 (8)	0.0142 (8)	0.0037 (9)
C1	0.0364 (10)	0.0501 (12)	0.0411 (12)	-0.0021 (8)	0.0186 (9)	-0.0048 (9)
C2	0.0404 (11)	0.0522 (12)	0.0425 (12)	-0.0026 (9)	0.0224 (9)	-0.0020 (10)
C3	0.0419 (12)	0.0610 (13)	0.0572 (14)	-0.0054 (9)	0.0307 (11)	-0.0060 (11)
C4	0.0332 (11)	0.0580 (13)	0.0515 (13)	0.0009 (9)	0.0205 (9)	-0.0041 (11)
C5	0.0348 (10)	0.0455 (11)	0.0449 (12)	0.0010 (8)	0.0169 (9)	-0.0067 (9)
C6	0.0468 (12)	0.0611 (14)	0.0509 (14)	-0.0040 (10)	0.0292 (11)	-0.0023 (12)
C7	0.0445 (14)	0.0956 (19)	0.0682 (17)	0.0067 (12)	0.0288 (12)	-0.0019 (14)
C8	0.0492 (14)	0.108 (2)	0.0642 (17)	0.0041 (14)	0.0074 (12)	0.0105 (16)
C9	0.0532 (13)	0.0662 (14)	0.0575 (14)	0.0078 (11)	0.0310 (12)	0.0106 (12)

*Geometric parameters (Å, °)*

N1—C1	1.330 (2)	C2—C6	1.421 (3)
N1—C5	1.341 (2)	C3—C4	1.370 (3)
N2—C1	1.340 (3)	C3—H3	0.9300
N2—H1	0.88 (3)	C4—C7	1.429 (3)
N2—H2	0.88 (3)	C4—C5	1.438 (3)
N3—C6	1.146 (3)	C8—H8A	0.9600
N4—C7	1.146 (3)	C8—H8B	0.9600
N5—C5	1.346 (3)	C8—H8C	0.9600
N5—C8	1.449 (3)	C9—H9A	0.9600
N5—C9	1.459 (3)	C9—H9B	0.9600
C1—C2	1.423 (3)	C9—H9C	0.9600
C2—C3	1.383 (3)		
C1—N1—C5	120.56 (16)	C7—C4—C5	123.7 (2)
C1—N2—H1	124.9 (15)	N1—C5—N5	116.88 (16)
C1—N2—H2	118 (2)	N1—C5—C4	120.45 (18)
H1—N2—H2	116 (3)	N5—C5—C4	122.66 (18)
C5—N5—C8	123.56 (19)	N3—C6—C2	178.3 (2)
C5—N5—C9	120.21 (17)	N4—C7—C4	177.7 (3)
C8—N5—C9	114.24 (18)	N5—C8—H8A	109.5
N1—C1—N2	117.60 (18)	N5—C8—H8B	109.5
N1—C1—C2	122.11 (18)	H8A—C8—H8B	109.5
N2—C1—C2	120.28 (19)	N5—C8—H8C	109.5
C3—C2—C6	122.45 (17)	H8A—C8—H8C	109.5
C3—C2—C1	117.08 (19)	H8B—C8—H8C	109.5
C6—C2—C1	120.45 (17)	N5—C9—H9A	109.5
C4—C3—C2	121.48 (18)	N5—C9—H9B	109.5
C4—C3—H3	119.3	H9A—C9—H9B	109.5

C2—C3—H3	119.3	N5—C9—H9C	109.5
C3—C4—C7	118.05 (19)	H9A—C9—H9C	109.5
C3—C4—C5	118.02 (18)	H9B—C9—H9C	109.5

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N2—H1 $\cdots$ N3 <sup>i</sup>	0.88 (3)	2.25 (3)	3.119 (3)	167 (2)
N2—H2 $\cdots$ N1 <sup>ii</sup>	0.88 (3)	2.43 (3)	3.260 (3)	158 (3)
C3—H3 $\cdots$ N4 <sup>iii</sup>	0.93	2.55	3.471 (4)	170

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