Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Redetermination of 2,2′-bipyridine-1,1′-diium dibromide

Basem F. Ali,[a]* Rawhi Al-Far[b] and Salim F. Haddad[c]

[a]Department of Chemistry, Al al-Bayt University, Mafraq 25113, Jordan, [b]Faculty of Science and IT, Al-Balqa'a Applied University, Salt, Jordan, and [c]Department of Chemistry, The University of Jordan, Amman 11942, Jordan
Correspondence e-mail: bfali@aabu.edu.jo

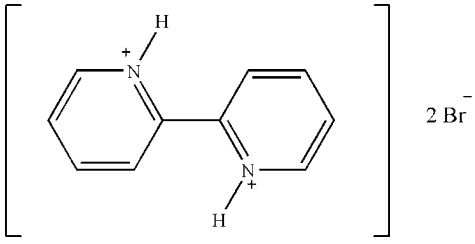
Received 7 September 2012; accepted 22 September 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.037; wR factor = 0.073; data-to-parameter ratio = 24.0.

In the title molecular salt, $C_{10}H_{10}N_2^{2+}\cdot 2Br^-$, the dihedral angle between the aromatic rings is 20.83 (14)° and the N—H groups have a *transoid* conformaton [N—C—C—N = 158.5 (3)°]. In the crystal, the cations are linked to the anions by two N—H···Br and five C—H···Br hydrogen bonds, generating corrugated sheets incorporating $R_2^1(7)$, $R_4^2(10)$, $R_4^2(11)$ and two different $R_4^2(12)$ loops. This structure was originally reported by Nakatsu *et al.* [Acta Cryst (1972), A**28**, S24], but no atomic coordinates are available.

Related literature

For the previous report of this structure as a conference abstract, see: Nakatsu *et al.* (1972). For related structures of 2,2′-bipyridium dication salts, see: Ma *et al.* (2000). For structures containing dicationic 2,2′-bipyridyl derivative salts, see: Amarante *et al.* (2011); Eckensberger *et al.* (2008). For structures of monocationic 2,2′-bipyridinium salts, see: Kavitha *et al.* (2006). For ring motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{10}H_{10}N_2^{2+}\cdot 2Br^-$
M_r = 318.00
Monoclinic, $P2_1/c$
a = 7.5568 (6) Å
b = 9.7747 (7) Å
c = 15.3533 (12) Å
β = 95.830 (7)°
V = 1128.21 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 7.15 mm^{-1}
T = 293 K
0.20 × 0.15 × 0.10 mm

Data collection

Agilent Xcalibur EOS diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.287, T_{max} = 0.489
5425 measured reflections
3054 independent reflections
1884 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.073
S = 1.03
3054 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Br2	0.86	2.37	3.178 (3)	156
N2—H2A···Br1^{i}	0.86	2.35	3.145 (3)	154
C1—H1B···Br1	0.93	2.83	3.611 (4)	143
C9—H9A···Br2	0.93	2.81	3.675 (4)	155
C6—H6A···Br2^{i}	0.93	2.84	3.619 (4)	142
C4—H4A···Br1^{i}	0.93	2.86	3.697 (4)	150
C3—H3A···Br1^{ii}	0.93	2.85	3.677 (4)	149

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

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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: *SHELXTL*.

The structure was determined at the Hamdi Mango Center for Scientific Research at the University of Jordan, Amman, Jordan. RA-F would like to thank Al-Balqa'a Applied University (Jordan) for financial support (sabbatical leave).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6957).

References

Agilent (2011). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
Amarante, T. R., Gonçalves, I. S. & Almeida Paz, F. A. (2011). *Acta Cryst.* E**67**, o1903–o1904.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Eckensberger, U. D., Lerner, H.-W. & Bolte, M. (2008). *Acta Cryst.* E**64**, o1806.
Kavitha, S. J., Panchanatheswaran, K., Low, J. N., Ferguson, G. & Glidewell, C. (2006). *Acta Cryst.* C**62**, o165–o169.
Ma, G., Ilyukhin, A. & Glaser, J. (2000). *Acta Cryst.* C**56**, 1473–1475.
Nakatsu, K., Yoshioka, H., Matsui, M., Koda, S. & Ooi, S. (1972). *Acta Cryst.* A**28**, S24.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2012). E**68**, o3033 [https://doi.org/10.1107/S1600536812040214]

Redetermination of 2,2′-bipyridine-1,1′-diium dibromide

Basem F. Ali, Rawhi Al-Far and Salim F. Haddad

S1. Comment

2,2′-bipyridine can form two types of salts monocationic, see for example (Kavitha *et al.*, 2006) or dicationic bipyridinium (see for example Ma *et al.*, 2000). Herein we report the title salt, (I), Figure 1. This structure was reported by Nakatsu *et al.* (1972), but no atomic coordinates are available. The bipyridinium cation in (I) has a *transoid* configuration with the N—C—C—N torsion angle is 158.5 (3)°, and the C—C bond distance between the two rings being 1.464 (4) Å. Within the dication, geometrical dimensions are in normal range and agree with reported values (Ma *et al.*, 2000; Eckensberger *et al.* 2008; Amarante *et al.* 2011).

The ions in (I) are linked by a combination of seven hydrogen bonds of the types N—H···Br and C—H···Br, Table 1, into complex corrugated sheets, Figure 2. These sheets composed of $R^1_2(7)$, $R^2_4(10)$, $R^2_4(11)$ *and two different* $R^2_4(12)$ graph set motifs (Bernstein *et al.* 1995), Figure 3. With the overall coordination around each bromide anion being 4 (three C—H···Br and one N—H···Br interactions), Figure 3.

S2. Experimental

A solution of $MnCl_2$ (0.1258 g, 1 mmol) dissolved in 95% EtOH (10 ml) solution was added to a mixture of 2,2′-bipyridine (0.1562 g, 1 mmol) dissolved in 95% EtOH (10 ml) and 60% HBr (2 ml). The resulting mixture was heated to few min, then treated with molecular bromine (2–3 drops), then refluxed for 1.5 hr. On slow evaporation at room temperature colourless chunks of the title salt were formed.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with N—H = 0.86 Å and C—H = 0.93 Å, with the U_{iso}(H) were allowed at 1.2U_{eq}(N/C).

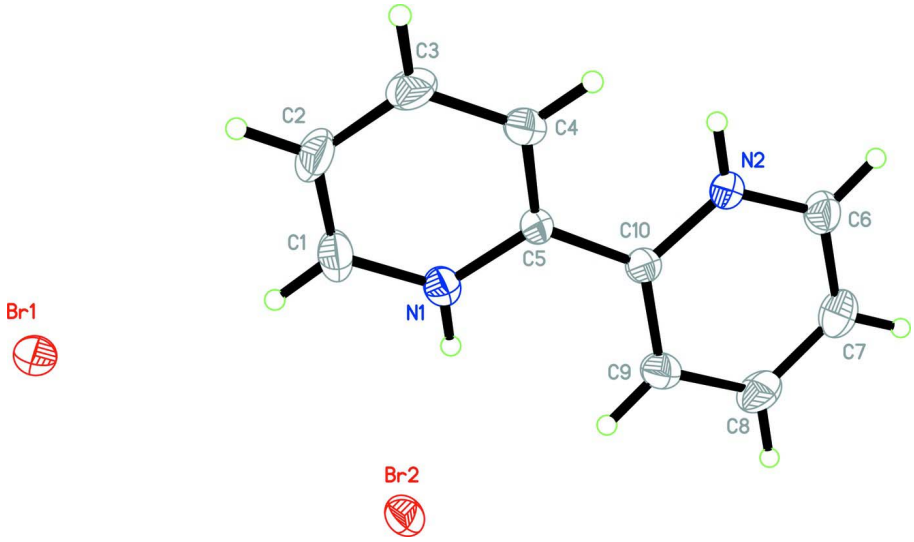


Figure 1

The molecular stucture of the title salt with displacement ellipsoids drawn at the 30% probability level.

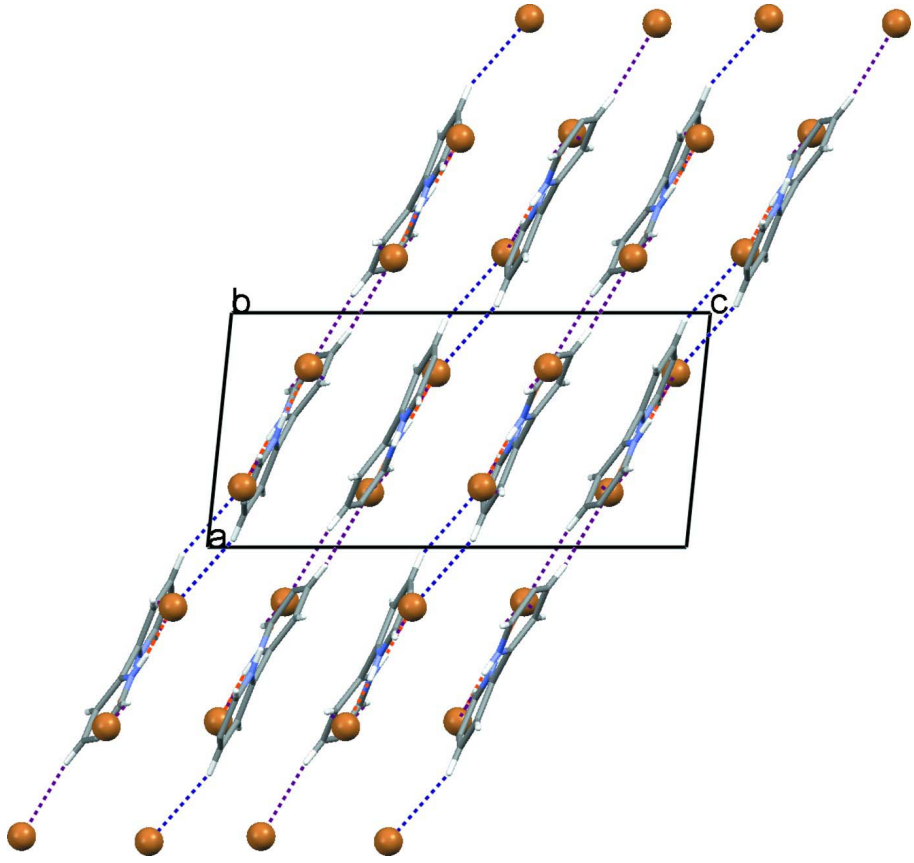


Figure 2

Packing diagram of the title salt showing the sheets of cations···anions hydrogen bonded species. Interspecies C/N–H···Br hydrogen bonds are shown as dashed lines. H atoms have been omitted for clarity, except for those involved in hydrogen bonds (shown as dashed lines).

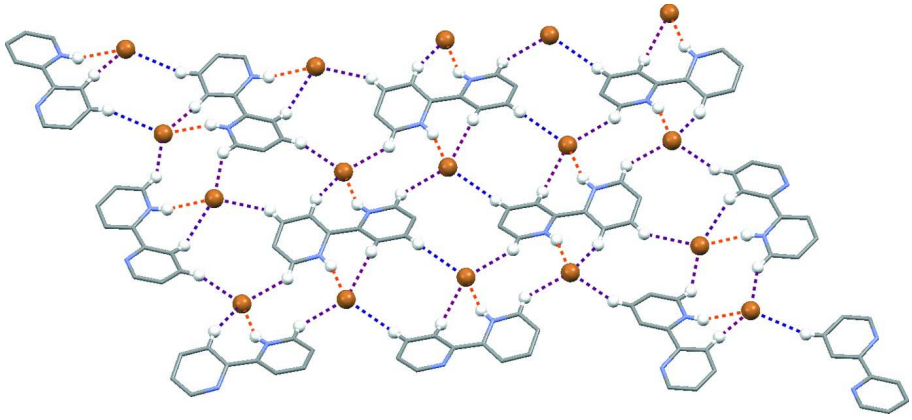


Figure 3

A view of one hydrogen bonded sheet contains the different graph-set motifs in the title salt.

2,2′-Bipyridine-1,1′-diium; dibromide

Crystal data

$C_{10}H_{10}N_2^{2+}\cdot 2Br^-$
$M_r = 318.00$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.5568\ (6)$ Å
$b = 9.7747\ (7)$ Å
$c = 15.3533\ (12)$ Å
$\beta = 95.830\ (7)°$
$V = 1128.21\ (15)$ Å^3
$Z = 4$
$F(000) = 616$
$D_x = 1.872$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1832 reflections
$\theta = 2.9–29.1°$
$\mu = 7.15$ mm^{-1}
$T = 293$ K
Chunk, colourless
0.20 × 0.15 × 0.10 mm

Data collection

Agilent Xcalibur EOS diffractometer
Radiation source: Enhance (Mo) x-ray source
Graphite monochromator
Detector resolution: 16.0534 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min} = 0.287$, $T_{max} = 0.489$
5425 measured reflections
3054 independent reflections
1884 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 29.2°$, $\theta_{min} = 3.4°$
$h = -7\rightarrow10$
$k = -13\rightarrow10$
$l = -21\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.073$
$S = 1.03$
3054 reflections
127 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.025P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.57$ e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.23397 (5)	0.03486 (4)	0.67326 (2)	0.04534 (13)
N1	0.4480 (4)	0.4858 (3)	0.63133 (16)	0.0384 (7)
H1A	0.5366	0.4580	0.6051	0.046*
C1	0.3257 (6)	0.3947 (4)	0.6491 (2)	0.0507 (10)
H1B	0.3369	0.3036	0.6330	0.061*
N2	0.5392 (4)	0.8449 (3)	0.61771 (15)	0.0369 (7)
H2A	0.4378	0.8714	0.6323	0.044*
C2	0.1841 (6)	0.4356 (4)	0.6909 (2)	0.0554 (11)
H2B	0.0950	0.3739	0.7013	0.066*
Br2	0.74405 (5)	0.29766 (4)	0.55966 (2)	0.04749 (13)
C3	0.1749 (5)	0.5695 (4)	0.7175 (2)	0.0532 (11)
H3A	0.0822	0.5979	0.7488	0.064*
C4	0.3033 (5)	0.6619 (4)	0.6978 (2)	0.0429 (9)
H4A	0.2968	0.7526	0.7154	0.051*
C5	0.4402 (4)	0.6197 (3)	0.65239 (19)	0.0314 (7)
C6	0.6477 (5)	0.9395 (4)	0.5912 (2)	0.0446 (9)
H6A	0.6141	1.0311	0.5898	0.054*
C7	0.8083 (6)	0.9019 (4)	0.5662 (2)	0.0505 (10)
H7A	0.8851	0.9671	0.5470	0.061*
C8	0.8553 (5)	0.7657 (4)	0.5698 (2)	0.0509 (10)
H8A	0.9641	0.7383	0.5524	0.061*
C9	0.7398 (5)	0.6683 (4)	0.5997 (2)	0.0449 (9)
H9A	0.7723	0.5765	0.6035	0.054*
C10	0.5767 (4)	0.7105 (3)	0.62330 (19)	0.0324 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0395 (2)	0.0378 (2)	0.0598 (2)	0.00431 (18)	0.01032 (17)	−0.00318 (18)
N1	0.0397 (19)	0.0320 (17)	0.0441 (16)	−0.0004 (14)	0.0068 (13)	0.0021 (13)
C1	0.058 (3)	0.035 (2)	0.058 (2)	−0.013 (2)	0.001 (2)	0.0056 (18)
N2	0.0309 (17)	0.0316 (17)	0.0483 (17)	−0.0033 (14)	0.0055 (13)	0.0014 (13)
C2	0.043 (3)	0.056 (3)	0.067 (3)	−0.019 (2)	0.008 (2)	0.016 (2)
Br2	0.0500 (3)	0.0356 (2)	0.0580 (2)	0.00351 (19)	0.01070 (18)	−0.00453 (16)
C3	0.036 (2)	0.060 (3)	0.067 (2)	0.004 (2)	0.0223 (19)	0.018 (2)
C4	0.037 (2)	0.036 (2)	0.057 (2)	0.0052 (17)	0.0111 (18)	0.0031 (17)

C5	0.0301 (19)	0.0260 (18)	0.0379 (17)	−0.0007 (16)	0.0022 (14)	0.0036 (14)
C6	0.045 (3)	0.037 (2)	0.052 (2)	−0.0067 (19)	0.0020 (18)	0.0054 (17)
C7	0.048 (3)	0.052 (3)	0.053 (2)	−0.015 (2)	0.0113 (19)	0.0052 (19)
C8	0.034 (2)	0.065 (3)	0.056 (2)	−0.005 (2)	0.0157 (17)	−0.002 (2)
C9	0.038 (2)	0.038 (2)	0.060 (2)	0.0053 (18)	0.0112 (18)	0.0026 (17)
C10	0.0304 (19)	0.0319 (19)	0.0349 (17)	0.0001 (16)	0.0033 (14)	0.0023 (14)

Geometric parameters (Å, º)

N1—C1	1.331 (4)	C4—C5	1.369 (4)
N1—C5	1.351 (4)	C4—H4A	0.9300
N1—H1A	0.8600	C5—C10	1.464 (4)
C1—C2	1.363 (6)	C6—C7	1.360 (5)
C1—H1B	0.9300	C6—H6A	0.9300
N2—C6	1.326 (4)	C7—C8	1.378 (5)
N2—C10	1.345 (4)	C7—H7A	0.9300
N2—H2A	0.8600	C8—C9	1.399 (5)
C2—C3	1.375 (5)	C8—H8A	0.9300
C2—H2B	0.9300	C9—C10	1.383 (5)
C3—C4	1.382 (5)	C9—H9A	0.9300
C3—H3A	0.9300		
C1—N1—C5	123.5 (3)	N1—C5—C4	117.8 (3)
C1—N1—H1A	118.3	N1—C5—C10	117.7 (3)
C5—N1—H1A	118.3	C4—C5—C10	124.4 (3)
N1—C1—C2	119.6 (4)	N2—C6—C7	119.7 (4)
N1—C1—H1B	120.2	N2—C6—H6A	120.1
C2—C1—H1B	120.2	C7—C6—H6A	120.1
C6—N2—C10	124.6 (3)	C6—C7—C8	118.9 (4)
C6—N2—H2A	117.7	C6—C7—H7A	120.6
C10—N2—H2A	117.7	C8—C7—H7A	120.6
C1—C2—C3	119.0 (4)	C7—C8—C9	120.3 (4)
C1—C2—H2B	120.5	C7—C8—H8A	119.9
C3—C2—H2B	120.5	C9—C8—H8A	119.9
C2—C3—C4	119.9 (4)	C10—C9—C8	119.0 (3)
C2—C3—H3A	120.0	C10—C9—H9A	120.5
C4—C3—H3A	120.0	C8—C9—H9A	120.5
C5—C4—C3	120.0 (3)	N2—C10—C9	117.6 (3)
C5—C4—H4A	120.0	N2—C10—C5	117.5 (3)
C3—C4—H4A	120.0	C9—C10—C5	125.0 (3)
C5—N1—C1—C2	−0.2 (5)	C6—C7—C8—C9	−0.7 (5)
N1—C1—C2—C3	−2.9 (6)	C7—C8—C9—C10	1.6 (5)
C1—C2—C3—C4	3.3 (6)	C6—N2—C10—C9	−0.1 (5)
C2—C3—C4—C5	−0.5 (5)	C6—N2—C10—C5	−178.9 (3)
C1—N1—C5—C4	3.0 (5)	C8—C9—C10—N2	−1.2 (5)
C1—N1—C5—C10	−175.6 (3)	C8—C9—C10—C5	177.6 (3)
C3—C4—C5—N1	−2.6 (5)	N1—C5—C10—N2	158.5 (3)

C3—C4—C5—C10	175.9 (3)	C4—C5—C10—N2	−20.1 (5)
C10—N2—C6—C7	1.0 (5)	N1—C5—C10—C9	−20.3 (5)
N2—C6—C7—C8	−0.6 (5)	C4—C5—C10—C9	161.1 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Br2	0.86	2.37	3.178 (3)	156
N2—H2*A*···Br1^{i}	0.86	2.35	3.145 (3)	154
C1—H1*B*···Br1	0.93	2.83	3.611 (4)	143
C9—H9*A*···Br2	0.93	2.81	3.675 (4)	155
C6—H6*A*···Br2^{i}	0.93	2.84	3.619 (4)	142
C4—H4*A*···Br1^{i}	0.93	2.86	3.697 (4)	150
C3—H3*A*···Br1^{ii}	0.93	2.85	3.677 (4)	149

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) −*x*, *y*+1/2, −*z*+3/2.