

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

3,3'-(Butane-1,4-diyl)diimidazole-1,1'-diium bis(triiodide)

Ai-E. Shi, Ying-Hui Yu, Guang-Feng Hou and Jin-Sheng Gao*

College of Chemistry and Materials Science, Heilongjiang University, Harbin 150080, People's Republic of China

Correspondence e-mail: hg1000@163.com

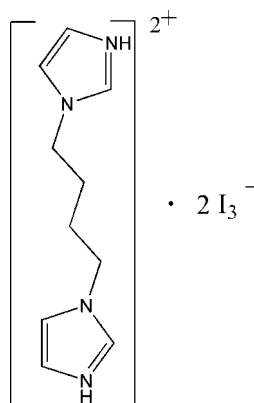
Received 9 June 2008; accepted 17 July 2008

 Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.014$ Å; R factor = 0.035; wR factor = 0.087; data-to-parameter ratio = 21.5.

The cations and anions of the salt, $\text{C}_{10}\text{H}_{16}\text{N}_4^{2+} \cdot 2\text{I}_3^-$, are linked by $\text{N}-\text{H} \cdots \text{I}$ hydrogen bonds and $\pi-\pi$ stacking interactions (with interplanar distances of 3.575 and 3.528 Å) into a three-dimensional supramolecular network. The asymmetric unit contains two anions and two half-cations; each cation is centrosymmetric.

Related literature

For literature on 1,1'-(1,4-butanediyl)diimidazole, see: Ma *et al.* (2003). For the structure of another 1,1'-(1,4-butanediyl)diimidazole-3,3'-diium salt, see: Yu *et al.* (2008).



Experimental

Crystal data

$\text{C}_{10}\text{H}_{16}\text{N}_4^{2+} \cdot 2\text{I}_3^-$
 $M_r = 953.67$
 Triclinic, $P\bar{1}$
 $a = 8.4753$ (17) Å
 $b = 9.7177$ (19) Å
 $c = 14.110$ (3) Å
 $\alpha = 95.77$ (3)°
 $\beta = 92.82$ (3)°

$\gamma = 107.17$ (3)°
 $V = 1100.9$ (4) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 8.46$ mm⁻¹
 $T = 291$ (2) K
 $0.21 \times 0.20 \times 0.18$ mm

Data collection

Rigaku R-AXIS RAPID diffractometer
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.266$, $T_{\max} = 0.306$
 (expected range = 0.189–0.218)

8492 measured reflections
 3831 independent reflections
 3045 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.087$
 $S = 1.10$
 3831 reflections

178 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 1.19$ e Å⁻³
 $\Delta\rho_{\min} = -0.86$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N2}-\text{H11} \cdots \text{I3}^{\text{i}}$	0.85	3.14	3.690 (9)	124
$\text{N2}-\text{H11} \cdots \text{I1}^{\text{ii}}$	0.85	2.99	3.666 (9)	138
$\text{N4}-\text{H3} \cdots \text{I1}^{\text{iii}}$	0.86	3.25	3.714 (9)	116
$\text{N4}-\text{H3} \cdots \text{I6}^{\text{iv}}$	0.86	3.03	3.679 (8)	134

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); 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: *SHELXL97*.

The authors thank Heilongjiang University for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2463).

References

- Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
 Ma, J.-F., Yang, J., Zheng, G.-L. & Liu, J.-F. (2003). *Inorg. Chem.* **42**, 7531–7534.
 Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
 Rigaku/MS (2002). *CrystalStructure*. Rigaku/MS Inc., The Woodlands, Texas, USA.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Yu, Y.-H., Shi, A.-E., Su, Y., Hou, G.-F. & Gao, J.-S. (2008). *Acta Cryst.* **E64**, m628.

supporting information

Acta Cryst. (2008). E64, o1561 [doi:10.1107/S1600536808022393]

3,3'-(Butane-1,4-diyl)diimidazole-1,1'-dium bis(triiodide)

Ai-E. Shi, Ying-Hui Yu, Guang-Feng Hou and Jin-Sheng Gao

S1. Comment

The 1,1'-(1,4-butanediyl)diimidazole can be used as a flexible ligand to construct coordination polymer materials (Ma *et al.*, 2003; Yu *et al.*, 2008). In our attempt to synthesize the copper(I) iodide complex with the 1,1'-(1,4-butanediyl)diimidazole, we unexpectedly obtained the title compound (I). Herein, we report its crystal structure.

The asymmetric unit of (I) consists of two halves of two independent centrosymmetric molecules of 1,1'-(1,4-Butane-diyl)diimidazole-3,3'-dium cation and two triiodide anions (Figure 1). The remarkable π - π stacking interactions are observed, with the distance between the π - π stacking planes are 3.575 Å (cations); 3.528 Å (Figure 2),

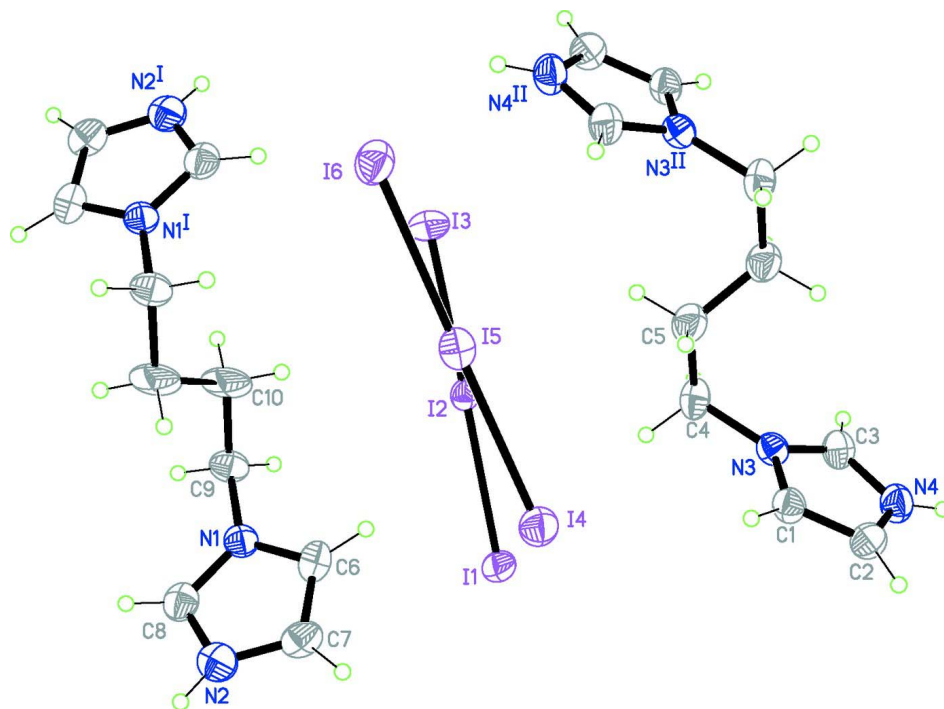
In the crystal, intermolecular N—H \cdots I, C—H \cdots I hydrogen bonds and π - π stacking interactions link all cations and anions into three-dimensional supramolecular network (Table 1).

S2. Experimental

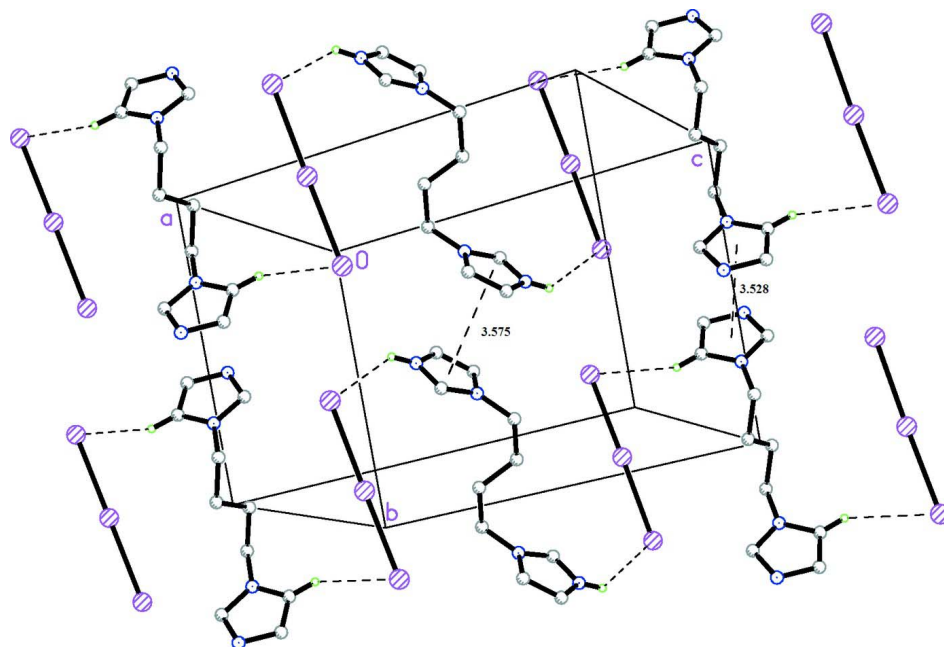
1,1'-(1,4-Butanediyl)diimidazole was prepared of imidazole and 1,4-dibromobutane in dimethylsulfoxide solution (Ma *et al.*, 2003). CuI (0.380 g, 2 mmol) and 1,1'-(1,4-butanediyl)diimidazole (0.380 g, 2 mmol) were dissolved in hot methanol solution (15 ml) and added two drops hydrochloric acid then a clear solution was obtained. The resulting solution was allowed to stand in a desiccator at room temperature for several days. Yellow crystals of (I) were obtained. Unexpectedly, the salt-type adducts of this ligands was crystallized from solution.

S3. Refinement

H atoms bound to C atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.93 Å (Caromatic); C—H = 0.97 Å (methylene) and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The N-bound H atoms were located in a difference Fourier map and refined with N—H = 0.85 Å, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. The final different Fourier map had a large peak in the vicinity of the iodine atoms.

**Figure 1**

The molecular structure of (I), showing displacement ellipsoids at the 30% probability level for non-H atoms.

**Figure 2**

A partial packing view, showing π - π interactions. Dashed lines indicate the hydrogen-bonding interactions and π - π interactions. H atoms not involved in hydrogen bonds have been omitted for clarity.

3,3'-(Butane-1,4-diyl)diimidazole-1,1'-dium bis(triiodide)

Crystal data

$C_{10}H_{16}N_4^{2+} \cdot 2I_3^-$
 $M_r = 953.67$
 Triclinic, $P\bar{1}$
 Hall symbol: $-P\ 1$
 $a = 8.4753$ (17) Å
 $b = 9.7177$ (19) Å
 $c = 14.110$ (3) Å
 $\alpha = 95.77$ (3)°
 $\beta = 92.82$ (3)°
 $\gamma = 107.17$ (3)°
 $V = 1100.9$ (4) Å³

$Z = 2$
 $F(000) = 844$
 $D_x = 2.877$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 7434 reflections
 $\theta = 3.0$ – 27.5 °
 $\mu = 8.46$ mm⁻¹
 $T = 291$ K
 Block, yellow
 $0.21 \times 0.20 \times 0.18$ mm

Data collection

Rigaku R-AXIS RAPID
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.266$, $T_{\max} = 0.307$

8492 measured reflections
 3831 independent reflections
 3045 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$
 $\theta_{\text{max}} = 25.0$ °, $\theta_{\text{min}} = 3.0$ °
 $h = -10 \rightarrow 10$
 $k = -11 \rightarrow 11$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.087$
 $S = 1.10$
 3831 reflections
 178 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
 $w = 1/[\sigma^2(F_o^2) + (0.0254P)^2 + 5.2161P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 1.19$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.86$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6046 (11)	0.3684 (10)	0.5506 (6)	0.049 (2)
H1	0.7142	0.3830	0.5369	0.058*
C2	0.5511 (12)	0.4469 (11)	0.6176 (7)	0.055 (2)

H2	0.6159	0.5268	0.6585	0.066*
C3	0.3377 (12)	0.2777 (12)	0.5469 (7)	0.056 (2)
H4	0.2293	0.2191	0.5305	0.067*
C4	0.4772 (13)	0.1552 (11)	0.4282 (6)	0.057 (3)
H6	0.5458	0.2028	0.3808	0.069*
H5	0.3664	0.1090	0.3977	0.069*
C5	0.5466 (12)	0.0400 (11)	0.4628 (7)	0.062 (3)
H8	0.5474	-0.0293	0.4085	0.075*
H7	0.6605	0.0860	0.4884	0.075*
C6	1.0068 (12)	0.3316 (11)	0.1048 (7)	0.055 (2)
H9	0.9362	0.2915	0.1499	0.066*
C7	1.1312 (11)	0.4573 (11)	0.1195 (8)	0.057 (3)
H10	1.1636	0.5208	0.1758	0.068*
C8	1.1230 (13)	0.3611 (11)	-0.0302 (7)	0.056 (3)
H12	1.1490	0.3477	-0.0930	0.067*
C9	0.8858 (8)	0.1352 (8)	-0.0341 (6)	0.055 (2)
H13	0.8975	0.1278	-0.1023	0.066*
H14	0.7734	0.1358	-0.0247	0.066*
C10	0.9139 (8)	0.0029 (8)	0.0049 (6)	0.079 (4)
H15	0.8923	0.0055	0.0718	0.094*
H16	0.8364	-0.0844	-0.0293	0.094*
I1	0.55146 (9)	0.37444 (7)	0.15451 (5)	0.05732 (19)
I2	0.54230 (7)	0.06704 (6)	0.16015 (4)	0.04222 (15)
I3	0.53942 (8)	-0.22804 (7)	0.16387 (5)	0.0602 (2)
I4	0.96410 (8)	0.34498 (8)	0.37380 (5)	0.0608 (2)
I5	0.99004 (7)	0.05335 (7)	0.33765 (4)	0.05118 (17)
I6	1.03499 (9)	-0.23842 (8)	0.31071 (5)	0.0636 (2)
N1	1.0024 (9)	0.2733 (8)	0.0121 (5)	0.0471 (18)
N2	1.1981 (10)	0.4712 (9)	0.0356 (6)	0.060 (2)
H11	1.2906	0.5258	0.0211	0.072*
N3	0.4708 (8)	0.2639 (8)	0.5063 (5)	0.0443 (17)
N4	0.3853 (10)	0.3886 (9)	0.6146 (6)	0.058 (2)
H3	0.3111	0.4094	0.6480	0.070*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.039 (5)	0.053 (6)	0.048 (5)	0.005 (4)	0.007 (4)	0.002 (5)
C2	0.054 (6)	0.052 (6)	0.049 (6)	0.006 (5)	-0.003 (5)	-0.002 (5)
C3	0.050 (6)	0.068 (7)	0.052 (6)	0.021 (5)	0.009 (5)	0.007 (5)
C4	0.062 (6)	0.074 (7)	0.038 (5)	0.027 (6)	0.005 (5)	-0.001 (5)
C5	0.060 (6)	0.063 (7)	0.067 (7)	0.023 (6)	0.017 (5)	0.002 (5)
C6	0.052 (6)	0.067 (7)	0.049 (6)	0.026 (6)	0.004 (5)	-0.003 (5)
C7	0.039 (5)	0.049 (6)	0.072 (7)	0.005 (5)	-0.010 (5)	-0.012 (5)
C8	0.067 (6)	0.061 (7)	0.050 (6)	0.034 (6)	0.006 (5)	0.011 (5)
C9	0.051 (5)	0.045 (6)	0.060 (6)	0.003 (5)	-0.006 (5)	0.006 (5)
C10	0.056 (6)	0.037 (6)	0.121 (10)	-0.018 (5)	-0.001 (7)	0.009 (6)
I1	0.0714 (4)	0.0472 (4)	0.0598 (4)	0.0265 (3)	0.0108 (3)	0.0079 (3)

I2	0.0397 (3)	0.0447 (3)	0.0383 (3)	0.0090 (3)	0.0015 (2)	-0.0012 (2)
I3	0.0601 (4)	0.0393 (4)	0.0767 (5)	0.0106 (3)	-0.0034 (3)	0.0040 (3)
I4	0.0584 (4)	0.0562 (4)	0.0616 (4)	0.0098 (3)	0.0076 (3)	-0.0007 (3)
I5	0.0415 (3)	0.0630 (4)	0.0443 (3)	0.0107 (3)	-0.0010 (3)	0.0020 (3)
I6	0.0660 (4)	0.0729 (5)	0.0604 (4)	0.0338 (4)	0.0089 (3)	0.0062 (4)
N1	0.051 (4)	0.048 (5)	0.049 (5)	0.022 (4)	0.004 (4)	0.014 (4)
N2	0.049 (5)	0.061 (6)	0.075 (6)	0.024 (4)	0.006 (5)	0.017 (5)
N3	0.040 (4)	0.048 (5)	0.043 (4)	0.009 (4)	0.003 (3)	0.008 (3)
N4	0.054 (5)	0.065 (6)	0.055 (5)	0.015 (4)	0.015 (4)	0.004 (4)

Geometric parameters (Å, °)

C1—C2	1.335 (13)	C7—N2	1.340 (13)
C1—N3	1.354 (11)	C7—H10	0.9300
C1—H1	0.9300	C8—N1	1.332 (12)
C2—N4	1.348 (12)	C8—N2	1.324 (12)
C2—H2	0.9300	C8—H12	0.9300
C3—N4	1.317 (12)	C9—N1	1.477 (10)
C3—N3	1.326 (11)	C9—C10	1.5246
C3—H4	0.9300	C9—H13	0.9700
C4—N3	1.462 (11)	C9—H14	0.9700
C4—C5	1.519 (13)	C10—C10 ⁱⁱ	1.489
C4—H6	0.9700	C10—H15	0.9700
C4—H5	0.9700	C10—H16	0.9700
C5—C5 ⁱ	1.487 (19)	I1—I2	2.9745 (10)
C5—H8	0.9700	I2—I3	2.8662 (10)
C5—H7	0.9700	I4—I5	2.9031 (11)
C6—C7	1.347 (13)	I5—I6	2.9604 (11)
C6—N1	1.367 (11)	N2—H11	0.8544
C6—H9	0.9300	N4—H3	0.8628
C2—C1—N3	107.7 (8)	N1—C8—H12	127.1
C2—C1—H1	126.2	N2—C8—H12	127.1
N3—C1—H1	126.2	N1—C9—C10	112.9
C1—C2—N4	107.1 (9)	N1—C9—H13	109.0
C1—C2—H2	126.5	C10—C9—H13	109.0
N4—C2—H2	126.5	N1—C9—H14	109.0
N4—C3—N3	108.2 (9)	C10—C9—H14	109.0
N4—C3—H4	125.9	H13—C9—H14	107.8
N3—C3—H4	125.9	C10 ⁱⁱ —C10—C9	112.0 (5)
N3—C4—C5	112.1 (8)	C10 ⁱⁱ —C10—H15	109.2
N3—C4—H6	109.2	C9—C10—H15	109.2
C5—C4—H6	109.2	C10 ⁱⁱ —C10—H16	109.2
N3—C4—H5	109.2	C9—C10—H16	109.2
C5—C4—H5	109.2	H15—C10—H16	107.9
H6—C4—H5	107.9	I3—I2—I1	178.89 (3)
C5 ⁱ —C5—C4	114.5 (10)	I4—I5—I6	176.21 (3)
C5 ⁱ —C5—H8	108.6	C8—N1—C6	108.7 (9)

C4—C5—H8	108.6	C8—N1—C9	125.1 (8)
C5 ⁱ —C5—H7	108.6	C6—N1—C9	126.2 (8)
C4—C5—H7	108.6	C8—N2—C7	112.3 (9)
H8—C5—H7	107.6	C8—N2—H11	114.7
C7—C6—N1	108.1 (9)	C7—N2—H11	131.3
C7—C6—H9	125.9	C3—N3—C1	108.0 (8)
N1—C6—H9	125.9	C3—N3—C4	127.3 (8)
C6—C7—N2	105.0 (9)	C1—N3—C4	124.6 (7)
C6—C7—H10	127.5	C3—N4—C2	109.0 (8)
N2—C7—H10	127.5	C3—N4—H3	118.4
N1—C8—N2	105.9 (9)	C2—N4—H3	132.6

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H11 \cdots I3 ⁱⁱⁱ	0.85	3.14	3.690 (9)	124
N2—H11 \cdots I1 ^{iv}	0.85	2.99	3.666 (9)	138
N4—H3 \cdots I1 ^v	0.86	3.25	3.714 (9)	116
N4—H3 \cdots I6 ⁱ	0.86	3.03	3.679 (8)	134
C6—H9 \cdots I4	0.93	3.13	3.823 (10)	132

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