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2,2′-Biimidazolium 5-amino-2,4,6-tribromoisophthalate

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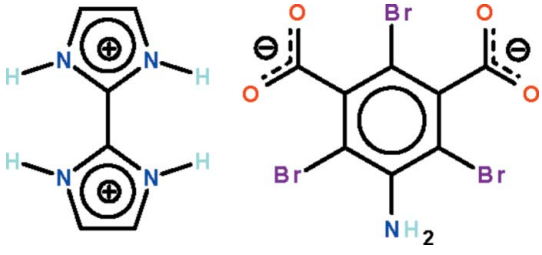
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.035; wR factor = 0.120; data-to-parameter ratio = 15.8.

In the cation of the title salt, $C_6H_8N_4^{2+}\cdot C_8H_2Br_3NO_4^{2-}$, the dihedral angle between the two five-membered rings is 2.1 (3)°. In the anion, the mean planes of the carboxyl units are twisted from the benzene ring by 84.3 (4) and 86.2 (3)°. In the crystal, the components are linked by imidazolium–carboxylate N—H···O hydrogen bonds, generating a chain running along [1$\bar{1}$0].

Related literature

For the structure of 5-amino-2,4,6-tribromidoisophthalic acid, see: Beck *et al.* (2009). For the structures of other 2,2′-bis(imidazolium) carboxylates, see: Gao *et al.* (2009); Li & Yang (2007); Zhou *et al.* (2009).



Experimental

Crystal data

$C_6H_8N_4^{2+}\cdot C_8H_2Br_3NO_4^{2-}$
M_r = 552.00
Triclinic, $P\bar{1}$
a = 9.0525 (10) Å
b = 9.2043 (10) Å
c = 11.5252 (12) Å
α = 90.262 (1)°
β = 108.332 (1)°
γ = 93.136 (1)°
V = 909.96 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 6.68 mm^{-1}
T = 293 K
0.35 × 0.25 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.203, T_{max} = 0.434
8042 measured reflections
4104 independent reflections
3129 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.120
S = 1.12
4104 reflections
259 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.81 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.88 (1)	1.74 (2)	2.608 (4)	168 (6)
N3—H3···O3^{i}	0.88 (1)	1.78 (2)	2.624 (5)	160 (5)
N4—H4···O4^{i}	0.88 (1)	1.74 (1)	2.614 (5)	175 (7)
N5—H5···O2	0.88 (1)	1.79 (2)	2.636 (4)	160 (4)

Symmetry code: (i) $x+1, y-1, z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5152).

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supporting information

Acta Cryst. (2010). E**66**, o2919 [https://doi.org/10.1107/S1600536810041899]

2,2′-Biimidazolium 5-amino-2,4,6-tribromoisophthalate

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S1. Comment

The crystal structure of 5-Amino-2,4,6-tribromoiodoisophthalic acid exists as a chains in which adjacent molecules are linked by O–H···O hydrogen bonds. In addition, pairs of chains are connected by further O–H···O hydrogen bonds (Beck *et al.*, 2009). This acid furnishes a small number of coordination compounds. An attempt to synthesize a lead(II) derivative that can be linked by 2,2′-biimidazole gave instead the title salt, $[C_6H_8N_4]^{2+}$ $[C_8H_2NO_4I_3]^{2-}$(1). Other examples of crystal structure of 2,2′-bis(imidazolium) carboxylates already appear in the literature (Gao *et al.*, 2009; Li & Yang, 2007; Zhou *et al.*, 2009).

The asymmetric unit of (1) is shown in Fig. 1. The cation is nearly planar as its two five-membered rings are twisted along the $C_{imidazolyl}$–$C_{imidazolyl}$ bond by 2.1 (3) ° only. In the anion, both $–CO_2$ units are almost orthogonal to the bezene ring mean plane [dihedral angles between $–CO_2$ plane and benzene ring (r.m.s. deviation 0.018 Å) are 84.3 (4)° and 86.2 (3) °]. In the crystal structure, cations and anions are linked by $N_{imidazolyl}$–H···O hydrogen bonds to generate a chain formation running along $[1\bar{1}0]$(Fig. 2).

S2. Experimental

An aqueous solution of lead nitrate (0.006 g, 0.2 mmol) in water (5 ml) was added to a mixture of 5-amino-2,4,6-tribromidoisophthalic acid (0.056 g, 0.1 mmol) in water (5 ml) and sodium hydroxide (0.2 ml, 0.5 *M*). To this solution was added 2,2′-biimidazole (0.014 g, 0.1 mmol) in DMF (5 ml). The solution was filtered; slow evaporation yielded pale yellow crystals which were collected (30% yield). CH&N elemental analysis. Calc. for $C_{14}H_{10}Br_3N_5O_4$: C 30.46, H 1.83, N 12.69%.; Found: C, 30.38; H, 1.91; N, 12.77%.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C).

The imidazolyl and amino H-atoms were located in a difference Fourier map, and were refined with a distance restraint of N–H 0.88±0.01 Å; their temperature factors were refined.

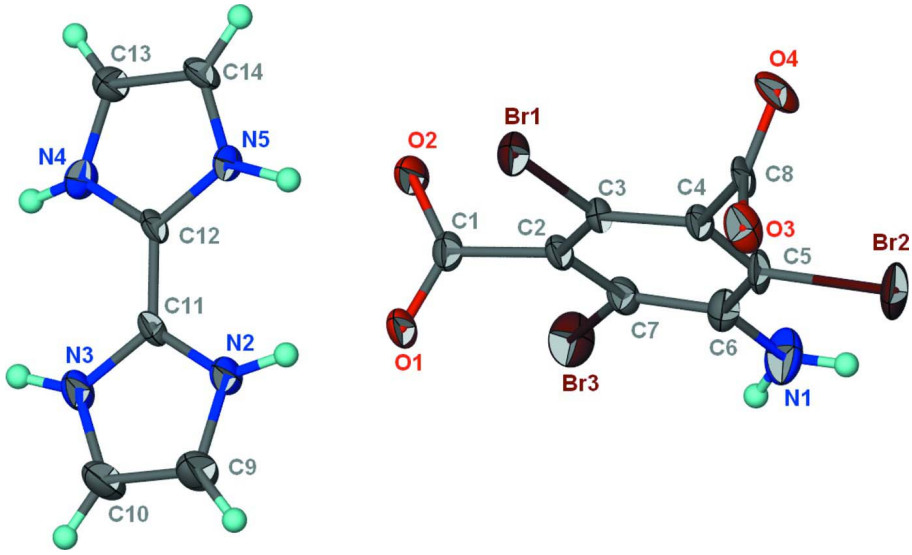

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_6H_8N_4^{2+}$ $C_8H_2NO_4Br_3^{2-}$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

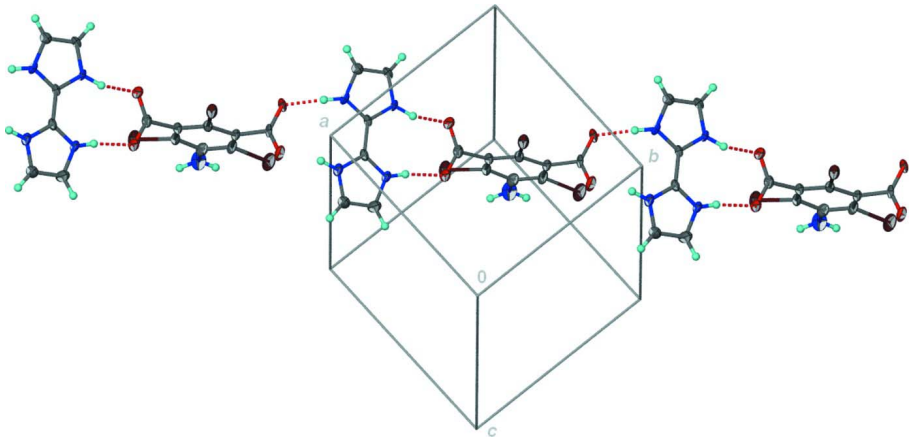

Figure 2

Part of the crystal structure showing hydrogen bonds as dashed lines.

2,2′-Biimidazolium 5-amino-2,4,6-tribromoisophthalate

Crystal data

$C_6H_8N_4^{2+}·C_8H_2Br_3NO_4^{2-}$
M_r = 552.00
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 9.0525 (10) Å
b = 9.2043 (10) Å
c = 11.5252 (12) Å
α = 90.262 (1)°
β = 108.332 (1)°
γ = 93.136 (1)°
V = 909.96 (17) Å^3
Z = 2
F(000) = 532
D_x = 2.015 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2718 reflections
θ = 2.4–27.4°
μ = 6.68 mm^{-1}
T = 293 K
Prism, pale yellow
0.35 × 0.25 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.203, T_{max} = 0.434
8042 measured reflections
4104 independent reflections
3129 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.5°, θ_{min} = 1.9°
h = −11→11
k = −11→11
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.120
S = 1.12
4104 reflections
259 parameters
6 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
$w = 1/[\sigma^2(F_o^2) + (0.0618P)^2 + 0.430P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.81 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.44985 (5)	0.68067 (5)	0.04955 (4)	0.03399 (14)
Br2	0.30262 (7)	0.89508 (6)	0.45860 (5)	0.05335 (18)
Br3	0.80240 (6)	0.52283 (6)	0.52786 (5)	0.05028 (17)
O1	0.6388 (4)	0.3751 (3)	0.2311 (3)	0.0364 (7)
O2	0.8128 (4)	0.5529 (3)	0.2201 (3)	0.0424 (8)
O3	0.1481 (4)	0.8223 (4)	0.1385 (3)	0.0419 (8)
O4	0.3322 (4)	1.0034 (3)	0.1754 (4)	0.0465 (9)
N1	0.5757 (6)	0.7119 (6)	0.5998 (4)	0.0536 (12)
N2	0.7881 (4)	0.1504 (4)	0.2018 (4)	0.0326 (8)
N3	0.9353 (4)	−0.0118 (4)	0.1680 (4)	0.0356 (9)
N4	1.1546 (4)	0.2242 (4)	0.1272 (3)	0.0305 (8)
N5	1.0016 (4)	0.3864 (4)	0.1539 (3)	0.0258 (7)
C1	0.6952 (5)	0.5045 (5)	0.2451 (4)	0.0300 (9)
C2	0.6062 (5)	0.6121 (4)	0.2971 (4)	0.0265 (8)
C3	0.4913 (5)	0.6934 (4)	0.2216 (4)	0.0246 (8)
C4	0.4030 (5)	0.7833 (4)	0.2686 (4)	0.0276 (9)
C5	0.4310 (5)	0.7852 (5)	0.3941 (4)	0.0329 (10)
C6	0.5465 (5)	0.7061 (5)	0.4750 (4)	0.0343 (10)
C7	0.6352 (5)	0.6244 (4)	0.4223 (4)	0.0293 (9)
C8	0.2831 (5)	0.8777 (4)	0.1859 (4)	0.0310 (9)
C9	0.7208 (6)	0.0171 (5)	0.2144 (5)	0.0491 (13)
H9	0.6293	−0.0004	0.2337	0.059*
C10	0.8132 (6)	−0.0853 (5)	0.1932 (5)	0.0485 (13)
H10	0.7967	−0.1857	0.1954	0.058*

C11	0.9166 (5)	0.1302 (4)	0.1731 (4)	0.0260 (8)
C12	1.0206 (5)	0.2449 (4)	0.1518 (4)	0.0253 (8)
C13	1.2192 (5)	0.3590 (5)	0.1138 (4)	0.0343 (10)
H13	1.3118	0.3773	0.0960	0.041*
C14	1.1255 (5)	0.4600 (4)	0.1306 (4)	0.0307 (9)
H14	1.1414	0.5604	0.1273	0.037*
H2	0.751 (7)	0.232 (4)	0.217 (5)	0.068 (19)*
H3	1.005 (5)	−0.054 (5)	0.143 (5)	0.054 (16)*
H4	1.215 (6)	0.151 (5)	0.139 (6)	0.09 (2)*
H5	0.923 (4)	0.424 (5)	0.171 (4)	0.035 (13)*
H11	0.519 (6)	0.764 (6)	0.631 (5)	0.07 (2)*
H12	0.624 (6)	0.640 (4)	0.643 (4)	0.056 (18)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0363 (3)	0.0361 (3)	0.0319 (2)	0.01227 (19)	0.01250 (19)	−0.00142 (18)
Br2	0.0560 (4)	0.0543 (4)	0.0623 (4)	0.0200 (3)	0.0340 (3)	−0.0138 (3)
Br3	0.0441 (3)	0.0611 (4)	0.0420 (3)	0.0224 (3)	0.0049 (2)	0.0071 (2)
O1	0.0342 (17)	0.0232 (15)	0.060 (2)	0.0058 (13)	0.0254 (15)	−0.0047 (14)
O2	0.0372 (19)	0.0282 (17)	0.075 (2)	0.0075 (14)	0.0350 (18)	0.0010 (16)
O3	0.0223 (16)	0.0374 (19)	0.063 (2)	0.0091 (14)	0.0084 (15)	−0.0072 (16)
O4	0.0372 (19)	0.0170 (16)	0.088 (3)	0.0121 (14)	0.0217 (18)	0.0094 (16)
N1	0.067 (3)	0.062 (3)	0.036 (2)	0.020 (3)	0.019 (2)	−0.005 (2)
N2	0.0230 (18)	0.0250 (19)	0.055 (2)	0.0067 (15)	0.0198 (17)	−0.0002 (17)
N3	0.028 (2)	0.0250 (19)	0.059 (2)	0.0073 (16)	0.0192 (18)	−0.0025 (17)
N4	0.0188 (17)	0.034 (2)	0.042 (2)	0.0098 (15)	0.0134 (15)	0.0030 (16)
N5	0.0170 (16)	0.0258 (18)	0.0388 (19)	0.0093 (14)	0.0134 (14)	0.0022 (15)
C1	0.034 (2)	0.030 (2)	0.030 (2)	0.0114 (19)	0.0135 (18)	0.0013 (17)
C2	0.027 (2)	0.0187 (19)	0.036 (2)	0.0059 (16)	0.0125 (17)	−0.0015 (16)
C3	0.028 (2)	0.020 (2)	0.0280 (19)	0.0052 (16)	0.0116 (17)	−0.0030 (15)
C4	0.024 (2)	0.021 (2)	0.041 (2)	0.0066 (16)	0.0145 (18)	−0.0016 (17)
C5	0.031 (2)	0.030 (2)	0.044 (2)	0.0102 (19)	0.019 (2)	−0.0103 (19)
C6	0.034 (2)	0.036 (2)	0.036 (2)	0.005 (2)	0.015 (2)	−0.0061 (19)
C7	0.025 (2)	0.024 (2)	0.039 (2)	0.0078 (17)	0.0091 (18)	0.0007 (17)
C8	0.031 (2)	0.020 (2)	0.048 (3)	0.0161 (18)	0.018 (2)	−0.0008 (18)
C9	0.041 (3)	0.034 (3)	0.084 (4)	−0.002 (2)	0.035 (3)	−0.002 (3)
C10	0.042 (3)	0.023 (2)	0.088 (4)	0.002 (2)	0.031 (3)	0.003 (2)
C11	0.029 (2)	0.0151 (19)	0.034 (2)	0.0067 (16)	0.0089 (17)	−0.0003 (16)
C12	0.030 (2)	0.0144 (18)	0.033 (2)	0.0075 (16)	0.0107 (17)	0.0003 (15)
C13	0.036 (2)	0.027 (2)	0.045 (3)	0.0033 (19)	0.019 (2)	0.0031 (19)
C14	0.035 (2)	0.0154 (19)	0.042 (2)	0.0045 (17)	0.0125 (19)	0.0054 (17)

Geometric parameters (Å, º)

Br1—C3	1.901 (4)	N5—C12	1.325 (5)
Br2—C5	1.895 (4)	N5—C14	1.377 (5)
Br3—C7	1.911 (4)	N5—H5	0.882 (10)

O1—C1	1.259 (5)	C1—C2	1.541 (5)
O2—C1	1.248 (5)	C2—C7	1.386 (6)
O3—C8	1.250 (5)	C2—C3	1.386 (5)
O4—C8	1.237 (5)	C3—C4	1.399 (5)
N1—C6	1.378 (6)	C4—C5	1.388 (6)
N1—H11	0.877 (10)	C4—C8	1.517 (6)
N1—H12	0.879 (10)	C5—C6	1.403 (6)
N2—C11	1.327 (5)	C6—C7	1.396 (6)
N2—C9	1.369 (6)	C9—C10	1.364 (7)
N2—H2	0.880 (10)	C9—H9	0.9300
N3—C11	1.330 (5)	C10—H10	0.9300
N3—C10	1.373 (6)	C11—C12	1.449 (6)
N3—H3	0.879 (10)	C13—C14	1.346 (6)
N4—C12	1.352 (5)	C13—H13	0.9300
N4—C13	1.373 (6)	C14—H14	0.9300
N4—H4	0.880 (10)		

C6—N1—H11	120 (4)	C6—C5—Br2	118.5 (3)
C6—N1—H12	118 (4)	N1—C6—C7	121.3 (4)
H11—N1—H12	119 (6)	N1—C6—C5	122.5 (4)
C11—N2—C9	108.4 (4)	C7—C6—C5	116.1 (4)
C11—N2—H2	129 (4)	C2—C7—C6	123.0 (4)
C9—N2—H2	122 (4)	C2—C7—Br3	118.6 (3)
C11—N3—C10	108.3 (4)	C6—C7—Br3	118.4 (3)
C11—N3—H3	127 (4)	O4—C8—O3	127.7 (4)
C10—N3—H3	124 (4)	O4—C8—C4	114.7 (4)
C12—N4—C13	107.4 (4)	O3—C8—C4	117.7 (4)
C12—N4—H4	132 (4)	C10—C9—N2	107.2 (4)
C13—N4—H4	117 (4)	C10—C9—H9	126.4
C12—N5—C14	108.8 (3)	N2—C9—H9	126.4
C12—N5—H5	124 (3)	C9—C10—N3	106.9 (4)
C14—N5—H5	127 (3)	C9—C10—H10	126.6
O2—C1—O1	126.7 (4)	N3—C10—H10	126.6
O2—C1—C2	117.9 (4)	N2—C11—N3	109.2 (4)
O1—C1—C2	115.4 (4)	N2—C11—C12	125.3 (4)
C7—C2—C3	118.2 (4)	N3—C11—C12	125.5 (4)
C7—C2—C1	119.9 (4)	N5—C12—N4	108.7 (3)
C3—C2—C1	121.8 (4)	N5—C12—C11	126.1 (4)
C2—C3—C4	121.7 (4)	N4—C12—C11	125.2 (4)
C2—C3—Br1	119.4 (3)	C14—C13—N4	108.2 (4)
C4—C3—Br1	118.9 (3)	C14—C13—H13	125.9
C5—C4—C3	117.6 (4)	N4—C13—H13	125.9
C5—C4—C8	121.0 (3)	C13—C14—N5	106.9 (4)
C3—C4—C8	121.3 (4)	C13—C14—H14	126.5
C4—C5—C6	123.2 (4)	N5—C14—H14	126.5
C4—C5—Br2	118.3 (3)		

O2—C1—C2—C7	−96.8 (5)	C5—C6—C7—C2	−4.4 (6)

O1—C1—C2—C7	83.9 (5)	N1—C6—C7—Br3	−1.2 (6)
O2—C1—C2—C3	86.3 (5)	C5—C6—C7—Br3	176.5 (3)
O1—C1—C2—C3	−93.0 (5)	C5—C4—C8—O4	85.3 (5)
C7—C2—C3—C4	−1.5 (6)	C3—C4—C8—O4	−93.6 (5)
C1—C2—C3—C4	175.4 (4)	C5—C4—C8—O3	−93.5 (5)
C7—C2—C3—Br1	179.4 (3)	C3—C4—C8—O3	87.6 (5)
C1—C2—C3—Br1	−3.7 (5)	C11—N2—C9—C10	0.4 (6)
C2—C3—C4—C5	−2.0 (6)	N2—C9—C10—N3	0.0 (7)
Br1—C3—C4—C5	177.1 (3)	C11—N3—C10—C9	−0.4 (6)
C2—C3—C4—C8	176.9 (4)	C9—N2—C11—N3	−0.7 (5)
Br1—C3—C4—C8	−4.1 (5)	C9—N2—C11—C12	179.9 (4)
C3—C4—C5—C6	2.5 (6)	C10—N3—C11—N2	0.7 (5)
C8—C4—C5—C6	−176.4 (4)	C10—N3—C11—C12	−179.9 (4)
C3—C4—C5—Br2	−175.5 (3)	C14—N5—C12—N4	−0.2 (5)
C8—C4—C5—Br2	5.6 (6)	C14—N5—C12—C11	179.4 (4)
C4—C5—C6—N1	178.2 (5)	C13—N4—C12—N5	0.0 (5)
Br2—C5—C6—N1	−3.8 (6)	C13—N4—C12—C11	−179.7 (4)
C4—C5—C6—C7	0.6 (7)	N2—C11—C12—N5	−2.3 (7)
Br2—C5—C6—C7	178.6 (3)	N3—C11—C12—N5	178.4 (4)
C3—C2—C7—C6	4.9 (6)	N2—C11—C12—N4	177.3 (4)
C1—C2—C7—C6	−172.0 (4)	N3—C11—C12—N4	−2.0 (7)
C3—C2—C7—Br3	−176.0 (3)	C12—N4—C13—C14	0.2 (5)
C1—C2—C7—Br3	7.0 (5)	N4—C13—C14—N5	−0.4 (5)
N1—C6—C7—C2	177.9 (4)	C12—N5—C14—C13	0.4 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1	0.88 (1)	1.74 (2)	2.608 (4)	168 (6)
N3—H3···O3^{i}	0.88 (1)	1.78 (2)	2.624 (5)	160 (5)
N4—H4···O4^{i}	0.88 (1)	1.74 (1)	2.614 (5)	175 (7)
N5—H5···O2	0.88 (1)	1.79 (2)	2.636 (4)	160 (4)

Symmetry code: (i) *x*+1, *y*−1, *z*.