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Low-temperature re-refinement of non-merohedrally twinned tripyridinium bis[tetrabromidoferrate(III)] bromide

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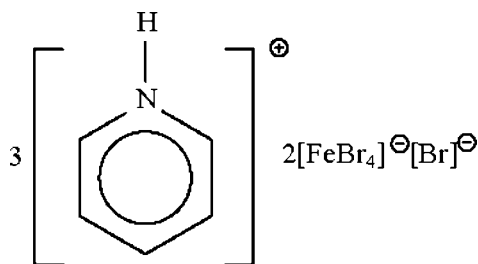
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Key indicators: single-crystal X-ray study; $T = 123$ K; mean $\sigma(\text{C}-\text{C}) = 0.016$ Å; R factor = 0.043; wR factor = 0.106; data-to-parameter ratio = 24.6.

The asymmetric unit of the title double salt, $(\text{C}_5\text{H}_6\text{N})_3\text{[FeBr}_4\text{]}_2\text{Br}$, consists of three pyridinium cations, two tetrahedral bromidoferrate(III) anions and a bromide anion. The three cations each form one $\text{N}-\text{H}\cdots\text{Br}$ hydrogen bond to the bromide anion. The crystal under investigation was a non-merohedral twin, with a portion of 22% for the minor twin component.

Related literature

The authors of the original room-temperature study noted twinning but the refinement program then could not take this into consideration; see: Lowe *et al.* (1994).



Experimental

Crystal data

$(\text{C}_5\text{H}_6\text{N})_3\text{[FeBr}_4\text{]}_2\text{Br}$
 $M_r = 1071.21$

Monoclinic, $P2_1$
 $a = 7.5602$ (1) Å

$b = 14.0125$ (2) Å
 $c = 13.5609$ (2) Å
 $\beta = 95.172$ (1)°
 $V = 1430.76$ (3) Å³
 $Z = 2$

Mo $K\alpha$ radiation
 $\mu = 13.59$ mm⁻¹
 $T = 123$ K
 $0.30 \times 0.25 \times 0.20$ mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.098$, $T_{\max} = 0.154$

13428 measured reflections
6460 independent reflections
6006 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.106$
 $S = 1.07$
6460 reflections
263 parameters
109 restraints

H-atom parameters constrained
 $\Delta\rho_{\max} = 1.29$ e Å⁻³
 $\Delta\rho_{\min} = -1.71$ e Å⁻³
Absolute structure: Flack (1983),
3046 Friedel pairs
Flack parameter: 0.10 (2)

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---------------------------------------|--------------|--------------------|-------------|----------------------|
| $\text{N1}-\text{H1}\cdots\text{Br9}$ | 0.88 | 2.35 | 3.202 (9) | 163 |
| $\text{N2}-\text{H2}\cdots\text{Br9}$ | 0.88 | 2.59 | 3.292 (8) | 137 |
| $\text{N3}-\text{H3}\cdots\text{Br9}$ | 0.88 | 2.52 | 3.279 (7) | 146 |

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT (Bruker, 2008); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) and PLATON (Spek, 2009); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2009).

The author thanks the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2380).

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supporting information

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Low-temperature rerefinement of nonmerohedrally twinned tripyridinium bis-[tetrabromidoferrate(III)] bromide

Seik Weng Ng

S1. Experimental

The crystals were provided by Dr. Nasser Safari of Shahid Beheshti University. Pyridine (2.2 ml, 25 mmol) was added to a solution of ferric bromide (1.25 g, 4.23 mmol) dissolved in a mixture of 1.2 M hydrobromic acid and 2.4 M acetic acid (20 ml). The red solution was set aside for two weeks, after which crystals separated out.

S2. Refinement

The refinement initially converged to an R_1 value of 0.088, but there were large peaks/deep holes. The crystal is in fact a nonmerohedral twin. The law, as given by *PLATON* (Spek, 2003), is (-1 0 0, 0 - 1 0, 0.323 0 1). The refinement, with an approximate twin component of 22%, halved the R_1 index. The twinning affected the anisotropic temperature factors of the carbon and nitrogen atoms; these were restrained to be nearly isotropic.

Carbon- and nitrogen-bound H-atoms were placed in calculated positions (C–H 0.95, N–H 0.88 Å) and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to $1.2U(\text{C},\text{N})$.

The final difference Fourier map had large peaks/holes in the vicinity of the bromide atoms.

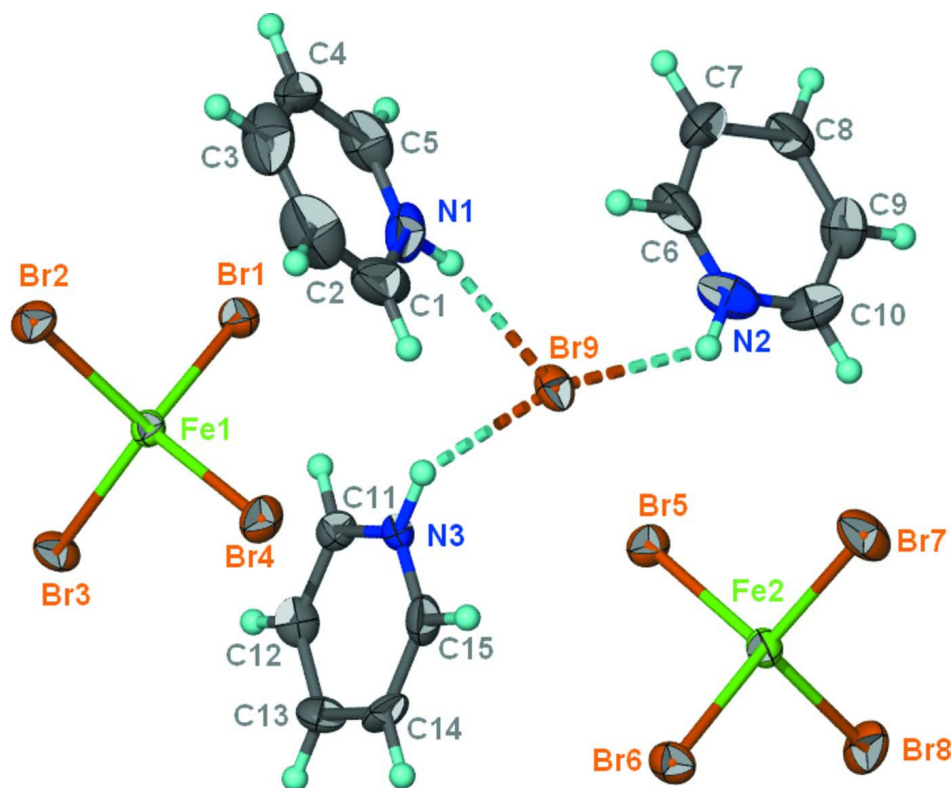


Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $[\text{C}_5\text{H}_6\text{N}]_3[\text{FeBr}_4]_2[\text{Br}]$.

tripyridinium bis[tetrabromidoferrate(III)] bromide

Crystal data

$(\text{C}_5\text{H}_6\text{N})_3[\text{FeBr}_4]_2\text{Br}$

$M_r = 1071.21$

Monoclinic, $P2_1$

Hall symbol: $P\ 2y_b$

$a = 7.5602\ (1)\ \text{\AA}$

$b = 14.0125\ (2)\ \text{\AA}$

$c = 13.5609\ (2)\ \text{\AA}$

$\beta = 95.172\ (1)^\circ$

$V = 1430.76\ (3)\ \text{\AA}^3$

$Z = 2$

$F(000) = 992$

$D_x = 2.487\ \text{Mg m}^{-3}$

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

Cell parameters from 8983 reflections

$\theta = 2.7\text{--}28.3^\circ$

$\mu = 13.59\ \text{mm}^{-1}$

$T = 123\ \text{K}$

Irregular block, brown

$0.30 \times 0.25 \times 0.20\ \text{mm}$

Data collection

Bruker SMART APEX

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.098$, $T_{\max} = 0.154$

13428 measured reflections

6460 independent reflections

6006 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 1.5^\circ$

$h = -9 \rightarrow 9$

$k = -18 \rightarrow 18$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.106$

$S = 1.07$

6460 reflections

263 parameters

109 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

$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2 + 4.7139P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.29 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -1.71 \text{ e } \text{\AA}^{-3}$

Absolute structure: Flack (1983), 3046 Friedel
pairs

Absolute structure parameter: 0.10 (2)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|-------------|----------------------------------|
| Br1 | 0.27784 (12) | 0.49992 (6) | 0.74070 (6) | 0.02428 (19) |
| Br2 | 0.27108 (13) | 0.35971 (6) | 0.50468 (7) | 0.0250 (2) |
| Br3 | 0.02833 (11) | 0.60181 (6) | 0.50814 (7) | 0.02457 (19) |
| Br4 | 0.52742 (11) | 0.59765 (7) | 0.52624 (7) | 0.0268 (2) |
| Br5 | 0.52708 (11) | 0.89250 (6) | 0.95876 (7) | 0.02335 (19) |
| Br6 | 0.75284 (12) | 1.00789 (7) | 0.74713 (6) | 0.02522 (19) |
| Br7 | 1.02585 (12) | 0.91096 (8) | 0.97548 (8) | 0.0356 (3) |
| Br8 | 0.74006 (15) | 1.13935 (7) | 0.98802 (7) | 0.0350 (2) |
| Br9 | 0.74762 (11) | 0.65558 (7) | 0.83903 (6) | 0.02359 (19) |
| Fe1 | 0.27879 (15) | 0.51483 (9) | 0.56879 (9) | 0.0177 (2) |
| Fe2 | 0.76356 (16) | 0.98860 (9) | 0.91855 (9) | 0.0197 (3) |
| N1 | 0.7426 (11) | 0.4395 (6) | 0.7619 (7) | 0.038 (2) |
| H1 | 0.7427 | 0.4931 | 0.7959 | 0.045* |
| N2 | 0.7876 (12) | 0.7029 (7) | 1.0778 (6) | 0.0343 (19) |
| H2 | 0.7993 | 0.7233 | 1.0173 | 0.041* |
| N3 | 0.3634 (10) | 0.7457 (5) | 0.7543 (5) | 0.0203 (15) |
| H3 | 0.4324 | 0.7032 | 0.7863 | 0.024* |
| C1 | 0.8048 (15) | 0.4412 (10) | 0.6741 (9) | 0.047 (3) |
| H1A | 0.8457 | 0.4988 | 0.6470 | 0.056* |
| C2 | 0.8080 (17) | 0.3569 (11) | 0.6239 (9) | 0.054 (3) |
| H2A | 0.8582 | 0.3549 | 0.5622 | 0.065* |
| C3 | 0.7408 (16) | 0.2758 (9) | 0.6606 (10) | 0.048 (3) |
| H3A | 0.7363 | 0.2182 | 0.6234 | 0.058* |
| C4 | 0.6797 (14) | 0.2790 (8) | 0.7523 (9) | 0.038 (2) |
| H4 | 0.6371 | 0.2226 | 0.7810 | 0.046* |
| C5 | 0.6802 (15) | 0.3618 (9) | 0.8016 (7) | 0.038 (2) |
| H5 | 0.6357 | 0.3647 | 0.8649 | 0.046* |
| C6 | 0.7428 (14) | 0.6150 (8) | 1.0919 (7) | 0.035 (2) |
| H6 | 0.7246 | 0.5738 | 1.0363 | 0.042* |
| C7 | 0.7210 (16) | 0.5797 (8) | 1.1832 (9) | 0.041 (3) |
| H7 | 0.6873 | 0.5151 | 1.1919 | 0.049* |
| C8 | 0.7500 (12) | 0.6418 (8) | 1.2642 (7) | 0.032 (2) |

| | | | | |
|-----|-------------|------------|------------|-------------|
| H8 | 0.7361 | 0.6199 | 1.3294 | 0.039* |
| C9 | 0.7984 (14) | 0.7340 (8) | 1.2482 (7) | 0.035 (2) |
| H9 | 0.8192 | 0.7771 | 1.3021 | 0.042* |
| C10 | 0.8163 (16) | 0.7633 (7) | 1.1548 (8) | 0.039 (2) |
| H10 | 0.8497 | 0.8274 | 1.1433 | 0.047* |
| C11 | 0.1903 (12) | 0.7447 (6) | 0.7669 (6) | 0.0208 (17) |
| H11 | 0.1435 | 0.6989 | 0.8092 | 0.025* |
| C12 | 0.0815 (12) | 0.8104 (7) | 0.7182 (6) | 0.0260 (18) |
| H12 | -0.0420 | 0.8102 | 0.7262 | 0.031* |
| C13 | 0.1503 (13) | 0.8766 (6) | 0.6576 (7) | 0.0256 (19) |
| H13 | 0.0757 | 0.9231 | 0.6241 | 0.031* |
| C14 | 0.3330 (13) | 0.8744 (6) | 0.6459 (7) | 0.0260 (19) |
| H14 | 0.3831 | 0.9190 | 0.6035 | 0.031* |
| C15 | 0.4375 (12) | 0.8082 (7) | 0.6956 (7) | 0.0269 (19) |
| H15 | 0.5614 | 0.8062 | 0.6888 | 0.032* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|------------|------------|-------------|-------------|-------------|
| Br1 | 0.0313 (4) | 0.0211 (4) | 0.0195 (4) | -0.0010 (4) | -0.0025 (3) | -0.0005 (3) |
| Br2 | 0.0325 (5) | 0.0188 (4) | 0.0241 (4) | -0.0009 (4) | 0.0039 (4) | -0.0032 (3) |
| Br3 | 0.0164 (4) | 0.0285 (5) | 0.0285 (4) | 0.0046 (4) | 0.0009 (3) | 0.0077 (4) |
| Br4 | 0.0168 (4) | 0.0268 (5) | 0.0369 (5) | -0.0041 (4) | 0.0030 (3) | 0.0016 (4) |
| Br5 | 0.0181 (4) | 0.0236 (4) | 0.0286 (4) | -0.0015 (3) | 0.0038 (3) | 0.0022 (3) |
| Br6 | 0.0311 (4) | 0.0272 (4) | 0.0175 (4) | 0.0014 (4) | 0.0031 (3) | 0.0012 (4) |
| Br7 | 0.0186 (4) | 0.0520 (7) | 0.0356 (5) | 0.0033 (4) | -0.0007 (4) | 0.0137 (5) |
| Br8 | 0.0507 (6) | 0.0292 (6) | 0.0246 (5) | -0.0099 (5) | 0.0008 (4) | -0.0090 (4) |
| Br9 | 0.0204 (4) | 0.0272 (5) | 0.0225 (4) | 0.0047 (4) | -0.0019 (3) | -0.0033 (3) |
| Fe1 | 0.0153 (5) | 0.0167 (6) | 0.0206 (6) | 0.0002 (5) | -0.0005 (4) | -0.0003 (5) |
| Fe2 | 0.0186 (5) | 0.0230 (6) | 0.0173 (6) | -0.0020 (5) | 0.0003 (4) | 0.0008 (5) |
| N1 | 0.025 (4) | 0.030 (4) | 0.056 (5) | 0.003 (3) | -0.013 (4) | -0.012 (4) |
| N2 | 0.038 (4) | 0.043 (5) | 0.023 (4) | 0.011 (4) | 0.010 (3) | 0.009 (3) |
| N3 | 0.020 (3) | 0.019 (3) | 0.021 (3) | -0.002 (3) | -0.006 (3) | 0.002 (3) |
| C1 | 0.030 (5) | 0.055 (6) | 0.055 (6) | -0.009 (5) | -0.004 (4) | 0.028 (5) |
| C2 | 0.042 (6) | 0.083 (8) | 0.038 (5) | 0.003 (6) | 0.009 (5) | 0.000 (6) |
| C3 | 0.040 (5) | 0.047 (6) | 0.056 (6) | 0.008 (5) | -0.005 (5) | -0.021 (5) |
| C4 | 0.028 (5) | 0.028 (5) | 0.055 (6) | -0.007 (4) | -0.013 (4) | 0.016 (4) |
| C5 | 0.036 (5) | 0.058 (6) | 0.021 (4) | 0.001 (5) | 0.005 (4) | -0.002 (4) |
| C6 | 0.043 (5) | 0.035 (5) | 0.024 (4) | 0.013 (4) | -0.011 (4) | -0.005 (4) |
| C7 | 0.044 (5) | 0.026 (5) | 0.051 (6) | -0.013 (4) | -0.009 (5) | 0.005 (4) |
| C8 | 0.028 (4) | 0.045 (5) | 0.024 (4) | 0.000 (4) | -0.001 (3) | 0.010 (4) |
| C9 | 0.037 (5) | 0.039 (5) | 0.029 (5) | -0.002 (4) | 0.004 (4) | -0.013 (4) |
| C10 | 0.049 (5) | 0.022 (4) | 0.048 (5) | 0.002 (4) | 0.010 (5) | 0.001 (4) |
| C11 | 0.023 (4) | 0.019 (4) | 0.021 (4) | -0.002 (3) | 0.004 (3) | -0.001 (3) |
| C12 | 0.023 (4) | 0.031 (4) | 0.025 (4) | 0.001 (4) | 0.004 (3) | -0.004 (3) |
| C13 | 0.027 (4) | 0.020 (4) | 0.029 (4) | 0.008 (4) | 0.001 (3) | 0.005 (3) |
| C14 | 0.028 (4) | 0.020 (4) | 0.031 (4) | -0.010 (3) | 0.008 (4) | 0.005 (3) |
| C15 | 0.021 (4) | 0.032 (4) | 0.028 (4) | -0.007 (4) | -0.003 (3) | -0.003 (4) |

Geometric parameters (Å, °)

| | | | |
|-------------|------------|-------------|------------|
| Br1—Fe1 | 2.341 (2) | C3—H3A | 0.9500 |
| Br2—Fe1 | 2.340 (2) | C4—C5 | 1.340 (16) |
| Br3—Fe1 | 2.338 (1) | C4—H4 | 0.9500 |
| Br4—Fe1 | 2.326 (1) | C5—H5 | 0.9500 |
| Br5—Fe2 | 2.342 (1) | C6—C7 | 1.357 (15) |
| Br6—Fe2 | 2.335 (1) | C6—H6 | 0.9500 |
| Br7—Fe2 | 2.331 (2) | C7—C8 | 1.404 (15) |
| Br8—Fe2 | 2.326 (2) | C7—H7 | 0.9500 |
| N1—C1 | 1.319 (15) | C8—C9 | 1.366 (15) |
| N1—C5 | 1.320 (15) | C8—H8 | 0.9500 |
| N1—H1 | 0.8800 | C9—C10 | 1.349 (15) |
| N2—C6 | 1.296 (14) | C9—H9 | 0.9500 |
| N2—C10 | 1.347 (14) | C10—H10 | 0.9500 |
| N2—H2 | 0.8800 | C11—C12 | 1.364 (13) |
| N3—C11 | 1.335 (11) | C11—H11 | 0.9500 |
| N3—C15 | 1.340 (12) | C12—C13 | 1.372 (13) |
| N3—H3 | 0.8800 | C12—H12 | 0.9500 |
| C1—C2 | 1.36 (2) | C13—C14 | 1.405 (13) |
| C1—H1A | 0.9500 | C13—H13 | 0.9500 |
| C2—C3 | 1.357 (19) | C14—C15 | 1.358 (13) |
| C2—H2A | 0.9500 | C14—H14 | 0.9500 |
| C3—C4 | 1.366 (17) | C15—H15 | 0.9500 |
| Br4—Fe1—Br3 | 107.44 (6) | N1—C5—C4 | 119.7 (9) |
| Br4—Fe1—Br2 | 111.41 (6) | N1—C5—H5 | 120.2 |
| Br3—Fe1—Br2 | 111.18 (6) | C4—C5—H5 | 120.2 |
| Br4—Fe1—Br1 | 111.51 (6) | N2—C6—C7 | 122.4 (10) |
| Br3—Fe1—Br1 | 108.77 (6) | N2—C6—H6 | 118.8 |
| Br2—Fe1—Br1 | 106.55 (6) | C7—C6—H6 | 118.8 |
| Br8—Fe2—Br7 | 112.54 (6) | C6—C7—C8 | 117.7 (10) |
| Br8—Fe2—Br6 | 107.52 (6) | C6—C7—H7 | 121.1 |
| Br7—Fe2—Br6 | 109.63 (6) | C8—C7—H7 | 121.1 |
| Br8—Fe2—Br5 | 109.89 (6) | C9—C8—C7 | 119.1 (9) |
| Br7—Fe2—Br5 | 107.40 (6) | C9—C8—H8 | 120.4 |
| Br6—Fe2—Br5 | 109.86 (6) | C7—C8—H8 | 120.4 |
| C1—N1—C5 | 123.5 (10) | C10—C9—C8 | 119.2 (10) |
| C1—N1—H1 | 118.2 | C10—C9—H9 | 120.4 |
| C5—N1—H1 | 118.2 | C8—C9—H9 | 120.4 |
| C6—N2—C10 | 120.6 (9) | N2—C10—C9 | 121.0 (10) |
| C6—N2—H2 | 119.7 | N2—C10—H10 | 119.5 |
| C10—N2—H2 | 119.7 | C9—C10—H10 | 119.5 |
| C11—N3—C15 | 123.4 (8) | N3—C11—C12 | 119.1 (8) |
| C11—N3—H3 | 118.3 | N3—C11—H11 | 120.4 |
| C15—N3—H3 | 118.3 | C12—C11—H11 | 120.4 |
| N1—C1—C2 | 117.3 (11) | C11—C12—C13 | 120.1 (8) |
| N1—C1—H1A | 121.4 | C11—C12—H12 | 120.0 |

| | | | |
|--------------|------------|-----------------|-----------|
| C2—C1—H1A | 121.4 | C13—C12—H12 | 120.0 |
| C3—C2—C1 | 121.2 (11) | C12—C13—C14 | 118.9 (8) |
| C3—C2—H2A | 119.4 | C12—C13—H13 | 120.6 |
| C1—C2—H2A | 119.4 | C14—C13—H13 | 120.6 |
| C2—C3—C4 | 118.3 (11) | C15—C14—C13 | 119.5 (8) |
| C2—C3—H3A | 120.8 | C15—C14—H14 | 120.3 |
| C4—C3—H3A | 120.8 | C13—C14—H14 | 120.3 |
| C5—C4—C3 | 119.8 (10) | N3—C15—C14 | 119.1 (8) |
| C5—C4—H4 | 120.1 | N3—C15—H15 | 120.5 |
| C3—C4—H4 | 120.1 | C14—C15—H15 | 120.5 |
| | | | |
| C5—N1—C1—C2 | -1.8 (17) | C7—C8—C9—C10 | 0.3 (17) |
| N1—C1—C2—C3 | 3.5 (19) | C6—N2—C10—C9 | -0.3 (17) |
| C1—C2—C3—C4 | -4.1 (19) | C8—C9—C10—N2 | -0.2 (17) |
| C2—C3—C4—C5 | 2.9 (18) | C15—N3—C11—C12 | -0.2 (13) |
| C1—N1—C5—C4 | 0.6 (17) | N3—C11—C12—C13 | -0.3 (13) |
| C3—C4—C5—N1 | -1.2 (17) | C11—C12—C13—C14 | 0.9 (14) |
| C10—N2—C6—C7 | 0.7 (16) | C12—C13—C14—C15 | -1.0 (14) |
| N2—C6—C7—C8 | -0.5 (17) | C11—N3—C15—C14 | 0.1 (13) |
| C6—C7—C8—C9 | 0.0 (16) | C13—C14—C15—N3 | 0.5 (14) |

Hydrogen-bond geometry (Å, °)

| <i>D—H...A</i> | <i>D—H</i> | <i>H...A</i> | <i>D...A</i> | <i>D—H...A</i> |
|----------------|------------|--------------|--------------|----------------|
| N1—H1...Br9 | 0.88 | 2.35 | 3.202 (9) | 163 |
| N2—H2...Br9 | 0.88 | 2.59 | 3.292 (8) | 137 |
| N3—H3...Br9 | 0.88 | 2.52 | 3.279 (7) | 146 |