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# Crystal structure of morpholin-4-ium cinnamate

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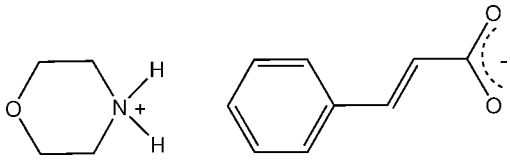
In the anhydrous salt formed from the reaction of morpholine with cinnamic acid, $C_4H_{10}NO^+\cdot C_9H_7O_2^-$, the acid side chain in the *trans*-cinnamate anion is significantly rotated out of the benzene plane [C—C—C— C torsion angle = 158.54 (17)°]. In the crystal, one of the the aminium H atoms is involved in an asymmetric three-centre cation–anion N—H···(O,O′) $R_1^2(4)$ hydrogen-bonding interaction with the two carboxylate O-atom acceptors of the anion. The second aminium-H atom forms an inter-species N—H···$O_{carboxylate}$ hydrogen bond. The result of the hydrogen bonding is the formation of a chain structure extending along [100]. Chains are linked by C—H···O interactions, forming a supramolecular layer parallel to (01$\bar{1}$).

**Keywords:** crystal structure; salt; morpholinium; cinnamate; hydrogen bonding.

**CCDC reference**: 1430629

## 1. Related literature

For background on morpholine compounds and the structure of an aliphatic morpholine salt, see: Kelley *et al.* (2013). For the structures of analogous morpholinate salts of some aromatic acid analogues, see: Chumakov *et al.* (2006); Ishida *et al.* (2001*a*,*b*,*c*); Smith & Lynch (2015).



## 2. Experimental

### 2.1. Crystal data

$C_4H_{10}NO^+\cdot C_9H_7O_2^-$
$M_r$ = 235.27
Triclinic, $P\overline{1}$
$a$ = 5.7365 (7) Å
$b$ = 9.7526 (10) Å
$c$ = 11.7760 (11) Å
$\alpha$ = 103.270 (8)°
$\beta$ = 93.468 (9)°
$\gamma$ = 105.493 (10)°
$V$ = 612.69 (12) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 200 K
0.52 × 0.24 × 0.05 mm

### 2.2. Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
$T_{min}$ = 0.965, $T_{max}$ = 0.990
4253 measured reflections
2393 independent reflections
1860 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.023

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.100
$S$ = 1.01
2393 reflections
160 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.15 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1*B*—H11*B*···O14*A*$^{i}$ | 0.94 (2) | 1.77 (2) | 2.7052 (17) | 170 (2) |
| N1*B*—H12*B*···O13*A* | 0.94 (2) | 1.73 (2) | 2.6643 (17) | 172 (2) |
| N1*B*—H12*B*···O14*A* | 0.94 (2) | 2.57 (2) | 3.1868 (17) | 123 (1) |
| C4*A*—H4*A*···O4*B*$^{ii}$ | 0.95 | 2.46 | 3.393 (2) | 167 |
| C6*B*—H62*B*···O13*A*$^{iii}$ | 0.99 | 2.37 | 3.234 (2) | 145 |

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

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) within *WinGX* (Farrugia, 2012); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

## Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5397).

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# supporting information

*Acta Cryst.* (2015). E**71**, o850–o851 [https://doi.org/10.1107/S2056989015019179]

## Crystal structure of morpholin-4-ium cinnamate

**Graham Smith**

### S1. Comment

Morpholine (tetrahydro-2*H*-1,4-oxazine) forms salts with organic acids, and the crystal structures of a limited number of these with either aliphatic acids, *e.g.* the acetate (Kelley *et al.*, 2013) or aromatic acids, *e.g.* the 4-nitrobenzoate (Chumakov *et al.*, 2006), have been reported. With the salts of the aromatic acids, particularly those with non-associative substituent groups, cation–anion *N*—*H*···$O_{carboxyl}$ hydrogen-bonding interactions generate either one-dimensional chains or discrete cyclic heterotetrameric structures. In the present work, the title morpholinium salt of cinnamic acid, $C_4H_{10}NO^+$ $C_9H_7O_2^-$ was prepared and its structure is reported herein.

The asymmetric unit of the title salt comprises a morpholinium cation (*B* and a cinnamate anion (*A*), (Fig. 1). In the *trans*- cinnamate anion, the acid side chain is significantly rotated out of the benzene plane [defining torsion angle C6*A*—C1*A*—C11*A*— C12*A* = 158.54 (17)°]. In the crystal, a primary asymmetric three-centre $R^2_1(4)$ N1*B*—*H*···(*O*,*O*′)$_{carboxyl}$ hydrogen-bonding interaction is present [N···O = 2.6643 (17) and 3.1868 (17) Å] (Table 1). The hydrogen-bonding extension involves the second aminium H atom of the cation to the carboxyl O14*A*$^{i}$ acceptor of the anion, resulting in a one-dimensional ribbon structure extending along *a* (Fig. 2). Present also in the structure are minor weak inter-unit C—H···O interactions. C4*A*—H···O4*B*$^{ii}$; C6*B*—H··· O13*A*$^{iii}$. No *π*–*π* interactions are present in the structure.

These ribbon structures are similar to those found in the morpholinium salt of one of the five isomeric chloro-nitro-benzoic acids (2,4-) (Ishida *et al.*, 2001*a*). In the other four isomers [(2,5-), (4,3-), (4,2-), (5,2-)] (Ishida, 2001*a*, 2001*b*, 2001*c*), the cyclic heterotetrameric structures are found. However, among a set of four morpholinium salts of phenoxy-acetic acid analogues (Smith & Lynch, 2015), there are four one-dimensional polymers and one cyclic heterotetramer.

### S2. Experimental

The title compound was prepared by the dropwise addition of morpholine at room temperature to a solution of cinnamic acid (150 mg) in ethanol (10 ml). Room temperature evaporation of the solution gave an oil which was redissolved in ethanol, finally giving thin colourless plates of the title compound from which a specimen was cleaved for the X-ray analysis.

### S3. Refinement

Hydrogen atoms were placed in calculated positions [C—$H_{aromatic}$ = 0.95 Å or *C*—$H_{methylene}$ = 0.99 Å] and were allowed to ride in the refinements, with $U_{iso}$(H) = 1.2$U_{eq}$(C). The aminium H atoms were located in a difference-Fourier analysis and were allowed to refine with distance restraints [*d*(N—H) = 0.92 (2) Å and $U_{iso}$(H) = 1.2$U_{eq}$(N)

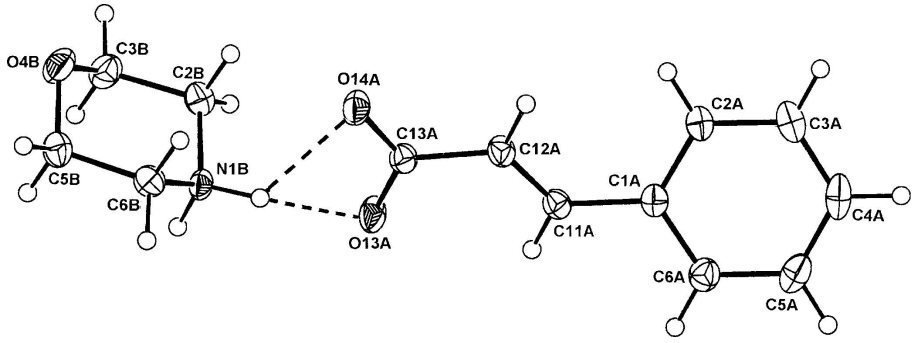


**Figure 1**

The atom-numbering scheme and the molecular conformation of the morpholinium anion (*B*) and the cinnamate cation (*A*) in the title salt, with displacement ellipsoids drawn at the 40% probability level. The cation–anion hydrogen bonds are shown as dashed lines.

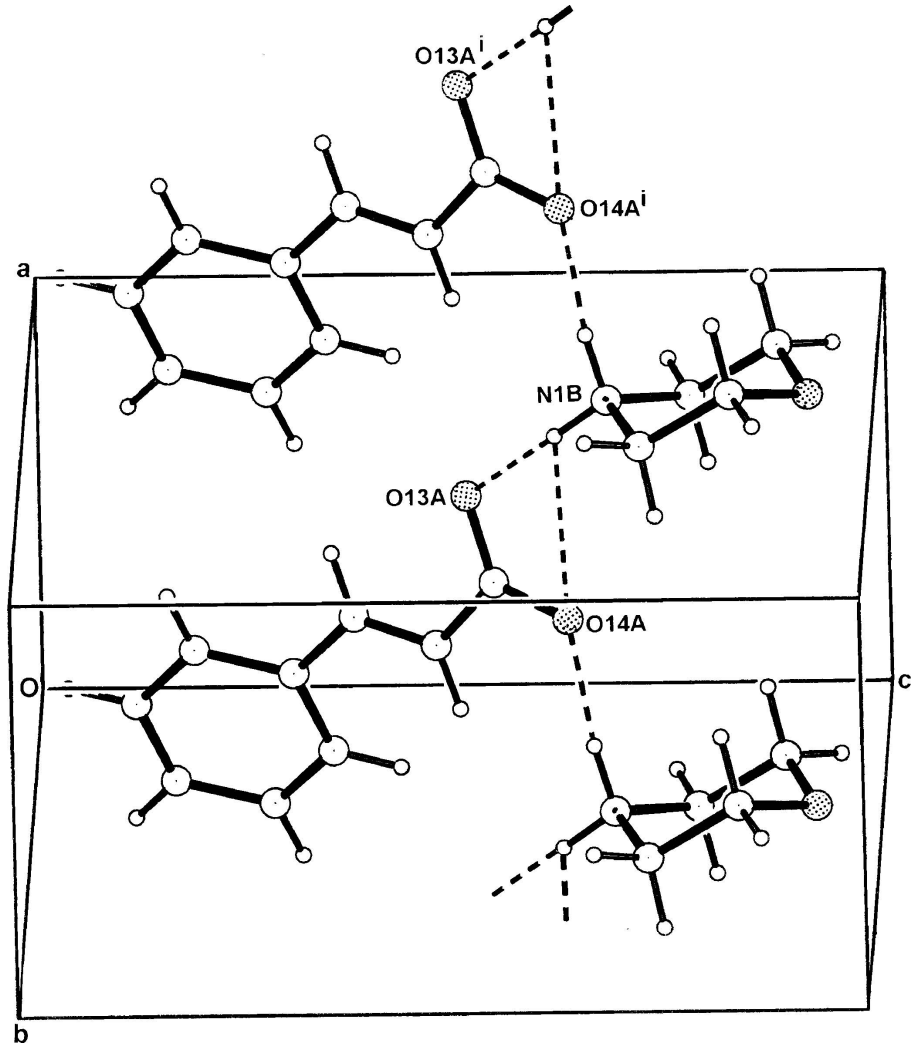


**Figure 2**

The one-dimensional hydrogen-bonded polymeric structure extending along *a*. For symmetry codes, see Table 1.

## Morpholin-4-ium 3-phenylprop-2-enoate

*Crystal data*

$C_4H_{10}NO^+·C_9H_7O_2^-$
$M_r$ = 235.27
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a$ = 5.7365 (7) Å
$b$ = 9.7526 (10) Å
$c$ = 11.7760 (11) Å
$\alpha$ = 103.270 (8)°
$\beta$ = 93.468 (9)°
$\gamma$ = 105.493 (10)°
$V$ = 612.69 (12) Å$^3$
$Z$ = 2
$F(000)$ = 252
$D_x$ = 1.281 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 1133 reflections
$\theta$ = 3.6–28.4°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 200 K
Plate, colourless
0.52 × 0.24 × 0.05 mm

*Data collection*

Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
$T_{min}$ = 0.965, $T_{max}$ = 0.990
4253 measured reflections
2393 independent reflections
1860 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.023
$\theta_{max}$ = 26.0°, $\theta_{min}$ = 3.2°
$h$ = −6→7
$k$ = −12→12
$l$ = −14→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.100
$S$ = 1.01
2393 reflections
160 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 0.0676P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.15 e Å$^{-3}$

*Special details*

**Geometry**. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

**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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O13A | 0.72059 (18) | 0.32720 (13) | 0.51574 (9) | 0.0370 (4) |
| O14A | 0.50422 (18) | 0.43517 (12) | 0.63746 (10) | 0.0323 (4) |
| C1A | 0.0669 (3) | 0.04440 (16) | 0.29775 (13) | 0.0256 (5) |

| | | | | |
|---|---|---|---|---|
| C2A | −0.1554 (3) | 0.00183 (17) | 0.34093 (15) | 0.0299 (5) |
| C3A | −0.3583 (3) | −0.09424 (18) | 0.26731 (16) | 0.0365 (6) |
| C4A | −0.3450 (3) | −0.14842 (18) | 0.14947 (16) | 0.0388 (6) |
| C5A | −0.1258 (3) | −0.10770 (18) | 0.10580 (15) | 0.0384 (6) |
| C6A | 0.0789 (3) | −0.01381 (17) | 0.17959 (14) | 0.0321 (5) |
| C11A | 0.2852 (3) | 0.14762 (17) | 0.37395 (14) | 0.0262 (5) |
| C12A | 0.2907 (3) | 0.24300 (17) | 0.47379 (14) | 0.0276 (5) |
| C13A | 0.5213 (3) | 0.34254 (17) | 0.54714 (13) | 0.0258 (5) |
| O4B | 1.2058 (2) | 0.63511 (13) | 0.93100 (10) | 0.0398 (4) |
| N1B | 1.0764 (2) | 0.48489 (14) | 0.68969 (11) | 0.0253 (4) |
| C2B | 1.0246 (3) | 0.40701 (18) | 0.78354 (14) | 0.0310 (5) |
| C3B | 1.2089 (3) | 0.48633 (18) | 0.89057 (14) | 0.0354 (6) |
| C5B | 1.2676 (3) | 0.71057 (18) | 0.84191 (15) | 0.0355 (6) |
| C6B | 1.0875 (3) | 0.64183 (17) | 0.73241 (14) | 0.0298 (5) |
| H2A | −0.16720 | 0.03930 | 0.42160 | 0.0360* |
| H3A | −0.50810 | −0.12330 | 0.29790 | 0.0440* |
| H4A | −0.48570 | −0.21320 | 0.09880 | 0.0470* |
| H5A | −0.11570 | −0.14440 | 0.02480 | 0.0460* |
| H6A | 0.22990 | 0.01130 | 0.14920 | 0.0390* |
| H11A | 0.43820 | 0.14510 | 0.34830 | 0.0310* |
| H12A | 0.14000 | 0.24890 | 0.50070 | 0.0330* |
| H11B | 1.227 (3) | 0.4752 (17) | 0.6663 (13) | 0.0300* |
| H12B | 0.951 (3) | 0.4376 (17) | 0.6261 (12) | 0.0300* |
| H21B | 1.03230 | 0.30470 | 0.75550 | 0.0370* |
| H22B | 0.85830 | 0.40330 | 0.80370 | 0.0370* |
| H31B | 1.17210 | 0.43540 | 0.95390 | 0.0420* |
| H32B | 1.37370 | 0.48430 | 0.87120 | 0.0420* |
| H51B | 1.43250 | 0.70830 | 0.82290 | 0.0430* |
| H52B | 1.27130 | 0.81480 | 0.87160 | 0.0430* |
| H61B | 0.92430 | 0.65020 | 0.74950 | 0.0360* |
| H62B | 1.13770 | 0.69400 | 0.67100 | 0.0360* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O13A | 0.0181 (6) | 0.0508 (8) | 0.0320 (7) | 0.0093 (5) | −0.0013 (5) | −0.0075 (6) |
| O14A | 0.0217 (6) | 0.0368 (7) | 0.0304 (6) | 0.0073 (5) | −0.0001 (5) | −0.0048 (5) |
| C1A | 0.0255 (8) | 0.0211 (8) | 0.0289 (9) | 0.0061 (7) | −0.0022 (7) | 0.0063 (7) |
| C2A | 0.0265 (8) | 0.0251 (9) | 0.0334 (9) | 0.0043 (7) | −0.0008 (7) | 0.0035 (7) |
| C3A | 0.0259 (9) | 0.0273 (9) | 0.0516 (12) | 0.0036 (7) | −0.0029 (8) | 0.0075 (8) |
| C4A | 0.0375 (10) | 0.0242 (9) | 0.0453 (11) | 0.0036 (8) | −0.0174 (8) | 0.0026 (8) |
| C5A | 0.0522 (11) | 0.0299 (10) | 0.0279 (9) | 0.0099 (9) | −0.0062 (8) | 0.0026 (8) |
| C6A | 0.0349 (9) | 0.0279 (9) | 0.0312 (9) | 0.0069 (8) | 0.0013 (7) | 0.0065 (7) |
| C11A | 0.0213 (8) | 0.0277 (9) | 0.0294 (9) | 0.0065 (7) | 0.0021 (6) | 0.0080 (7) |
| C12A | 0.0191 (8) | 0.0311 (9) | 0.0303 (9) | 0.0072 (7) | 0.0019 (6) | 0.0039 (7) |
| C13A | 0.0213 (8) | 0.0295 (9) | 0.0261 (9) | 0.0076 (7) | 0.0007 (6) | 0.0064 (7) |
| O4B | 0.0518 (8) | 0.0356 (7) | 0.0241 (6) | 0.0068 (6) | −0.0002 (5) | 0.0001 (5) |
| N1B | 0.0185 (6) | 0.0305 (8) | 0.0222 (7) | 0.0066 (6) | −0.0013 (5) | −0.0007 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C2B | 0.0287 (8) | 0.0269 (9) | 0.0361 (10) | 0.0057 (7) | 0.0024 (7) | 0.0085 (8) |
| C3B | 0.0402 (10) | 0.0363 (10) | 0.0286 (9) | 0.0101 (8) | −0.0011 (7) | 0.0088 (8) |
| C5B | 0.0380 (10) | 0.0257 (9) | 0.0351 (10) | 0.0009 (8) | 0.0011 (8) | 0.0032 (8) |
| C6B | 0.0296 (9) | 0.0280 (9) | 0.0321 (9) | 0.0082 (7) | 0.0045 (7) | 0.0085 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O13A—C13A | 1.258 (2) | C2A—H2A | 0.9500 |
| O14A—C13A | 1.2553 (19) | C3A—H3A | 0.9500 |
| O4B—C3B | 1.425 (2) | C4A—H4A | 0.9500 |
| O4B—C5B | 1.424 (2) | C5A—H5A | 0.9500 |
| N1B—C2B | 1.480 (2) | C6A—H6A | 0.9500 |
| N1B—C6B | 1.480 (2) | C11A—H11A | 0.9500 |
| N1B—H11B | 0.944 (18) | C12A—H12A | 0.9500 |
| N1B—H12B | 0.943 (15) | C2B—C3B | 1.503 (2) |
| C1A—C6A | 1.390 (2) | C5B—C6B | 1.501 (2) |
| C1A—C2A | 1.396 (2) | C2B—H21B | 0.9900 |
| C1A—C11A | 1.471 (2) | C2B—H22B | 0.9900 |
| C2A—C3A | 1.381 (2) | C3B—H31B | 0.9900 |
| C3A—C4A | 1.382 (3) | C3B—H32B | 0.9900 |
| C4A—C5A | 1.382 (3) | C5B—H51B | 0.9900 |
| C5A—C6A | 1.382 (2) | C5B—H52B | 0.9900 |
| C11A—C12A | 1.314 (2) | C6B—H61B | 0.9900 |
| C12A—C13A | 1.493 (2) | C6B—H62B | 0.9900 |
| | | | |
| C3B—O4B—C5B | 109.75 (12) | C1A—C6A—H6A | 120.00 |
| C2B—N1B—C6B | 111.05 (12) | C12A—C11A—H11A | 117.00 |
| C6B—N1B—H11B | 110.9 (10) | C1A—C11A—H11A | 117.00 |
| C2B—N1B—H12B | 107.7 (10) | C11A—C12A—H12A | 118.00 |
| H11B—N1B—H12B | 109.8 (14) | C13A—C12A—H12A | 118.00 |
| C2B—N1B—H11B | 107.0 (10) | N1B—C2B—C3B | 109.50 (14) |
| C6B—N1B—H12B | 110.3 (10) | O4B—C3B—C2B | 110.91 (14) |
| C2A—C1A—C11A | 121.67 (14) | O4B—C5B—C6B | 111.36 (14) |
| C6A—C1A—C11A | 120.00 (15) | N1B—C6B—C5B | 109.46 (14) |
| C2A—C1A—C6A | 118.33 (15) | N1B—C2B—H21B | 110.00 |
| C1A—C2A—C3A | 120.55 (16) | N1B—C2B—H22B | 110.00 |
| C2A—C3A—C4A | 120.46 (17) | C3B—C2B—H21B | 110.00 |
| C3A—C4A—C5A | 119.55 (16) | C3B—C2B—H22B | 110.00 |
| C4A—C5A—C6A | 120.21 (16) | H21B—C2B—H22B | 108.00 |
| C1A—C6A—C5A | 120.88 (16) | O4B—C3B—H31B | 109.00 |
| C1A—C11A—C12A | 126.79 (16) | O4B—C3B—H32B | 109.00 |
| C11A—C12A—C13A | 123.45 (16) | C2B—C3B—H31B | 109.00 |
| O13A—C13A—O14A | 123.98 (15) | C2B—C3B—H32B | 109.00 |
| O13A—C13A—C12A | 118.14 (14) | H31B—C3B—H32B | 108.00 |
| O14A—C13A—C12A | 117.87 (15) | O4B—C5B—H51B | 109.00 |
| C1A—C2A—H2A | 120.00 | O4B—C5B—H52B | 109.00 |
| C3A—C2A—H2A | 120.00 | C6B—C5B—H51B | 109.00 |
| C4A—C3A—H3A | 120.00 | C6B—C5B—H52B | 109.00 |

| | | | |
|---|---|---|---|
| C2A—C3A—H3A | 120.00 | H51B—C5B—H52B | 108.00 |
| C3A—C4A—H4A | 120.00 | N1B—C6B—H61B | 110.00 |
| C5A—C4A—H4A | 120.00 | N1B—C6B—H62B | 110.00 |
| C6A—C5A—H5A | 120.00 | C5B—C6B—H61B | 110.00 |
| C4A—C5A—H5A | 120.00 | C5B—C6B—H62B | 110.00 |
| C5A—C6A—H6A | 120.00 | H61B—C6B—H62B | 108.00 |

| | | | |
|---|---|---|---|
| C3B—O4B—C5B—C6B | 61.19 (17) | C1A—C2A—C3A—C4A | −0.7 (3) |
| C5B—O4B—C3B—C2B | −61.29 (17) | C2A—C3A—C4A—C5A | 1.0 (3) |
| C2B—N1B—C6B—C5B | 54.09 (17) | C3A—C4A—C5A—C6A | 0.2 (3) |
| C6B—N1B—C2B—C3B | −54.46 (17) | C4A—C5A—C6A—C1A | −1.8 (3) |
| C2A—C1A—C6A—C5A | 2.1 (2) | C1A—C11A—C12A—C13A | 178.94 (15) |
| C6A—C1A—C11A—C12A | 158.54 (17) | C11A—C12A—C13A—O13A | −5.0 (2) |
| C11A—C1A—C6A—C5A | −178.16 (16) | C11A—C12A—C13A—O14A | 175.97 (16) |
| C2A—C1A—C11A—C12A | −21.7 (3) | N1B—C2B—C3B—O4B | 57.95 (17) |
| C6A—C1A—C2A—C3A | −0.8 (2) | O4B—C5B—C6B—N1B | −57.43 (18) |
| C11A—C1A—C2A—C3A | 179.41 (16) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1*B*—H11*B*···O14*A*$^{i}$ | 0.94 (2) | 1.77 (2) | 2.7052 (17) | 170 (2) |
| N1*B*—H12*B*···O13*A* | 0.94 (2) | 1.73 (2) | 2.6643 (17) | 172 (2) |
| N1*B*—H12*B*···O14*A* | 0.94 (2) | 2.57 (2) | 3.1868 (17) | 123 (1) |
| C4*A*—H4*A*···O4*B*$^{ii}$ | 0.95 | 2.46 | 3.393 (2) | 167 |
| C11*A*—H11*A*···O13*A* | 0.95 | 2.48 | 2.812 (2) | 101 |
| C6*B*—H62*B*···O13*A*$^{iii}$ | 0.99 | 2.37 | 3.234 (2) | 145 |

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