**N-(2-Bromobenzyl)cinchoninium bromide**

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Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C—C) = 0.005 Å; R factor = 0.034; wR factor = 0.071; data-to-parameter ratio = 19.2.

The title compound [systematic name: 1-(2-bromobenzyl)-5-ethenyl-2-[hydroxy(quinolin-4-yl)methyl]-1-azabicyclo[2.2.2]-octan-1-iium bromide], C_{26}H_{28}BrN_{2}O^{+}Br^{-}, is a chiral quaternary ammonium salt of one of the Cinchona alkaloids. The planes of the quinoline and of the bromobenzyl substituent are inclined to one another by 9.11 (9)°. A weak intramolecular C—H···O hydrogen bond occurs. The crystal structure features strong O—H···Br hydrogen bonds and weak C—H···Br interactions.

### Related literature

For the structure of cinchonine base and its derivatives, see: Oleksyn et al. (1979); Dell’Olmo et al. (1984). For crystal structures of other selected Cinchona alkaloid derivatives with bulky substituents at the quinuclidine nitrogen atom, see: Song et al. (2005); Kawai et al. (2009); Jew et al. (2002); Matoba et al. (2010). For the effect of the substituent on the activity of the title catalyst, see: Jezierska-Zięba et al. (2010).

### Experimental

**Crystal data**

\[
\begin{align*}
C_{26}H_{28}BrN_{2}O^{+}Br^{-} \\
M_r = 544.30 \\
\text{Orthorhombic, } P2_12_12_1 \\
a = 7.213 (1) \text{ Å} \\
b = 16.2545 (1) \text{ Å} \\
c = 20.2466 (2) \text{ Å} \\
V = 2379.81 (4) \text{ Å}^3 \\
Z = 4
\end{align*}
\]

Mo Ka radiation

\[\mu = 3.43 \text{ mm}^{-1}\]

\[T = 295 \text{ K} \]

\[0.2 \times 0.15 \times 0.1 \text{ mm}\]

**Data collection**

Nonius KappaCCD diffractometer

Absorption correction: multi-scan

(DENZO and SCALEPACK; Otwinowski & Minor 1997)

\[T_{\text{min}} = 0.547, \ T_{\text{max}} = 0.726\]

\[64183 \text{ measured reflections} \]

\[5437 \text{ independent reflections} \]

\[4879 \text{ reflections with } I > 2\sigma(I) \]

\[R_{\text{int}} = 0.039\]

\[\Delta\rho_{\text{min}} = 0.40 \text{ e Å}^{-3} \]

\[\Delta\rho_{\text{max}} = -0.42 \text{ e Å}^{-3} \]

**Refinement**

\[R(F^2) > 2\sigma(F^2) = 0.034\]

\[wR(F^2) = 0.071\]

\[S = 1.06\]

\[5437 \text{ reflections} \]

\[283 \text{ parameters} \]

H atoms treated by a mixture of independent and constrained refinement

### Table 1

 hydrogen-bond geometry (Å, °).

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| O12—H12···Br1 | 0.81 (4) | 2.38 (4) | 3.179 (2) | 173 (3) |
| C2—H2B···O12 | 0.97 | 2.32 | 2.997 (4) | 126 |
| C6—H6A···Br1 | 0.97 | 2.88 | 3.797 (3) | 159 |
| C18—H18···Br1 | 0.93 | 2.96 | 3.578 (4) | 145 |

Symmetry code: (i) \(x, y, z\).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2374).

**Supplementary material**

**Data collection**: COLLECT (Nonius, 1998); cell refinement: SCALEPACK; data reduction: DENZO (Otwinowski & Minor, 1997) and SCALEPACK (Otwinowski & Minor, 1997); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).
organic compounds

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supporting information

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N-(2-Bromobenzyl)cinchoninium bromide

Agnieszka Skórka-Stania, Magdalena Jezierska-Zięba, Barbara Kąkol, Michał Fedoryński and Barbara J. Oleksyn

S1. Comment

The Cinchona alkaloids with bulky substituents at quinuclidine nitrogen atom (N1), as the potential catalysts, have been studied crystallographically in the last three decades: Dolling et al. (1984); Song et al. (2005); Kawai et al. (2009); Jew et al. (2002); Matoba et al. (2010). The asymmetric unit of the title compound is composed of N-(2-bromobenzyl)-cinchoninium cation and bromide anion (Fig. 1). The title cinchonine derivative was used as a catalyst in the asymmetric Darzens condensation between benzaldehyde and alkylchloroacetates: Jezierska-Zięba et al. (2010).

The conformational features of the title compound are similar to those of the related parent structure of cinchonine base (Oleksyn et al., 1979), with exception of the orientation of the vinyl group towards the quinuclidine moiety. The packing is dominated by the strong hydrogen bonding O12—H···Br1 (Fig. 2). The pairs cation–anion interact with each other via short contacts C—H···Br1, forming chains parallel to [1 0 0]. The chains are strengthened by short C—H···Br2 contacts. The oxygen atom (O12), is an acceptor in weak intramolecular hydrogen bonds. The hydrogen bond geometry is given in Table 1.

The disorder of the vinyl groups occurs in almost every molecular structure of Cinchona alkaloids, we have determined. The vinyl group (i.e. C10 and C11 atoms) is present on the periphery of the whole molecule, so it has ability to move. The conformation of the vinyl moiety, which we present here, is close to the potential energy minimum and is frequently observed in the structures of erythro Cinchona alkaloids.

S2. Experimental

A mixture of cinchonine (2.95 g, 0.01 mol) and 2–bromobenzylbromide (2.5 g, 0.01 mol) in toluene (40 ml) was stirred and heated at 353 K for 4 h. After cooling to room temperature, hexane (100 ml) was added and the mixture was stirred for 10 h. The precipitated crystals were collected by suction filtration, washed with acetonitrile and dried to give N-(2-bromobenzyl)cinchoninium bromide (5.25 g, 97%, m.p. 430 K). Single crystals suitable for X-ray diffraction study were obtained from ethanol by slow evaporation at room temperature.

S3. Refinement

All hydrogen atoms were found on a difference Fourier maps and refined using a riding model with C—H = 0.93Å and \( U_{iso}(H) = 1.2 U_{eq}(C) \) for aromatic hydrogen atoms, C—H = 0.97Å and \( U_{iso}(H) = 1.2 U_{eq}(C) \) for methylene groups and C—H = 0.98Å and \( U_{iso}(H) = 1.2 U_{eq}(C) \) for methine groups. The O based atom H12 was refined with \( U_{iso}(H) = 1.2 U_{eq}(O) \).
Figure 1
The asymmetric unit of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are shown as spheres of arbitrary radii. The C—H···Br hydrogen bonds are in dashed lines.
Figure 2
The packing viewed along the $a$ axis with strong hydrogen bonds shown by dashed lines.
Figure 3
Enhanced figure.

1-(2-Bromobenzyl)-5-ethenyl-2-[hydroxy(quinolin-4-yl)methyl]-1-azabicyclo[2.2.2]octan-1-ium bromide

Crystal data
C_{26}H_{28}BrN_{2}O^{+}·Br^{-}

$M_r = 544.30$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 7.2313 (1)$ Å

$b = 16.2545 (1)$ Å

$c = 20.2466 (2)$ Å

$V = 2379.81 (4)$ Å$^3$

$Z = 4$

$F(000) = 1104$

$D_x = 1.519$ Mg m$^{-3}$

Melting point: 430 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3135 reflections

$\theta = 1.0–27.5^\circ$

$\mu = 3.43$ mm$^{-1}$

$T = 295$ K

Prism, colourless

$0.2 \times 0.15 \times 0.1$ mm
Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 9 pixels mm\(^{-1}\)
CCD rotation images, thick slices scans
Absorption correction: multi-scan
\((\text{DENZO} \text{ and SCALEPACK}; \text{Otwinowski & Minor 1997})\)

\(T_{\text{min}} = 0.547, T_{\text{max}} = 0.726\)
64183 measured reflections
5437 independent reflections
4879 reflections with \(I > 2\sigma(I)\)
\(\theta_{\text{max}} = 27.5^\circ, \theta_{\text{min}} = 2.7^\circ\)
\(h = -9 \rightarrow 9\)
\(k = -20 \rightarrow 21\)
\(l = -26 \rightarrow 26\)

Refinement

Refinement on \(F^2\)
Least-squares matrix: full
\(R[F^2 > 2\sigma(F^2)] = 0.034\)
\(wR(F^2) = 0.071\)
\(S = 1.06\)
5437 reflections
283 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
\(H\) atoms treated by a mixture of independent and constrained refinement
\(w = 1/[\sigma^2(F_o^2) + (0.023P)^2 + 1.5842P]\)
where \(P = (F_o^2 + 2F_c^2)/3\)

\(\Delta/\sigma)_{\text{max}} = 0.001\)
\(\Delta\rho_{\text{max}} = 0.40 \text{ e Å}^{-3}\)
\(\Delta\rho_{\text{min}} = -0.42 \text{ e Å}^{-3}\)

Absolute structure: Flack (1983), 2320 Friedel pairs
Absolute structure parameter: 0.020 (8)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\(\text{Å}^2\))

\[
\begin{array}{cccccc}
\text{H} & x & y & z & U_{eq} & U_{eq}^* \\
C1B & 0.1042 (4) & 0.78679 (16) & 0.82411 (13) & 0.0396 (6) \\
H1BA & -0.0247 & 0.7713 & 0.8194 & 0.048* \\
H1BB & 0.1776 & 0.7374 & 0.8182 & 0.048* \\
C2B & 0.1337 (4) & 0.81793 (16) & 0.89340 (13) & 0.0393 (6) \\
C3B & -0.0039 (4) & 0.85398 (19) & 0.93115 (14) & 0.0508 (7) \\
C4B & 0.0243 (5) & 0.8774 (2) & 0.99629 (16) & 0.0647 (9) \\
H4BA & -0.0705 & 0.9017 & 1.0204 & 0.078* \\
C5B & 0.1937 (5) & 0.8641 (3) & 1.02470 (16) & 0.0701 (11) \\
H5BA & 0.2148 & 0.8804 & 1.0681 & 0.084* \\
C6B & 0.3319 (5) & 0.8269 (2) & 0.98952 (16) & 0.0643 (9) \\
H6BA & 0.4461 & 0.8174 & 1.0092 & 0.077* \\
C7B & 0.3019 (4) & 0.8036 (2) & 0.92451 (15) & 0.0497 (7) \\
H7BA & 0.3964 & 0.7777 & 0.9012 & 0.06* \\
C2 & 0.0284 (4) & 0.92222 (15) & 0.77020 (13) & 0.0426 (6) \\
H2A & 0.0258 & 0.9448 & 0.8146 & 0.051* \\
H2B & -0.0967 & 0.9064 & 0.7584 & 0.051* \\
C3 & 0.0976 (5) & 0.98821 (17) & 0.72183 (15) & 0.0514 (8) \\
H3 & 0.167 & 1.0292 & 0.7472 & 0.062* \\
\end{array}
\]
# Atomic displacement parameters (Å²)

|   | $U_{11}$     | $U_{22}$     | $U_{33}$     | $U_{12}$     | $U_{13}$     | $U_{23}$     |
|---|--------------|--------------|--------------|--------------|--------------|--------------|
| C1B| 0.0480 (16)  | 0.0380 (14)  | 0.0328 (13)  | 0.0005 (11)  | 0.0036 (11)  | 0.0000 (11)  |
| C2B| 0.0437 (14)  | 0.0398 (15)  | 0.0344 (13)  | -0.0034 (11) | 0.0035 (12)  | -0.0023 (11) |
| C3B| 0.0480 (16)  | 0.0590 (19)  | 0.0454 (15)  | -0.0050 (13) | 0.0062 (12)  | -0.0073 (13) |

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### Geometric parameters (Å, °)

| Bond or Angle                     | Distance/1 | Angle/2 |
|-----------------------------------|------------|---------|
| C1B—C2B                           | 1.507 (4)  |         |
| C1B—N1                            | 1.527 (3)  | 0.97    |
| C1B—H1BA                          | 0.97       | 0.97    |
| C1B—H1BB                          | 0.97       | 1.538 (4)|
| C2B—C3B                           | 1.385 (4)  |         |
| C2B—C7B                           | 1.90 (4)   | 0.98    |
| C3B—C4B                           | 1.388 (4)  |         |
| C3B—Br2                           | 1.189 (3)  |         |
| C4B—C5B                           | 1.370 (5)  |         |
| C4B—C6B                           | 1.378 (5)  | 0.98    |
| C4B—C7B                           | 1.387 (4)  |         |
| C5B—C6B                           | 1.368 (5)  | 0.93    |
| C5B—C7B                           | 0.93       | 1.309 (5)|
| C6B—C7B                           | 1.387 (4)  | 0.93    |
| C6B—C81                           | 0.93       | 1.418 (5)|
| C7B—C81                           | 1.519 (3)  | 0.93    |
| C2B—N1                            | 1.536 (4)  | 1.353 (5)|

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C2—H2A 0.97  C15—H15 0.93
C2—H2B 0.97  C16—C17 1.432 (5)
C3—C4  1.518 (4)  C17—C18 1.406 (5)
C3—C10 1.539 (5)  C17—C22 1.437 (4)
C3—H3  0.98  C18—C19 1.369 (5)
C4—C5  1.531 (5)  C18—H18 0.93
C4—C7  1.531 (4)  C19—C20 1.413 (6)
C4—H4  0.98  C19—H19 0.93
C5—C6  1.535 (4)  C20—C21 1.336 (7)
C5—H5A 0.97  C20—H20 0.93
C5—H5B 0.97  C21—C22 1.405 (6)
C6—N1  1.519 (3)  C21—H21 0.93
C6—H6A 0.97  C22—N13 1.364 (5)
C6—H6B 0.97  O12—H12 0.81 (4)

C2B—C1B—N1  115.8 (2)  C4—C7—H7B 109.9
C2B—C1B—H1BA 108.3  C8—C7—H7B 109.9
N1—C1B—H1BA 108.3  H7A—C7—H7B 108.3
C2B—C1B—H1BB 108.3  C7—C8—C9 113.3 (3)
N1—C1B—H1BB 108.3  C7—C8—N1 107.6 (2)
H1BA—C1B—H1BB 107.4  C9—C8—N1 114.1 (2)
C3B—C2B—C7B 116.8 (3)  C7—C8—H8 107.1
C3B—C2B—C1B 123.7 (3)  C9—C8—H8 107.1
C7B—C2B—C1B 119.3 (3)  N1—C8—H8 107.1
C2B—C3B—C4B 122.3 (3)  O12—C9—C16 110.3 (2)
C2B—C3B—Br2 121.2 (2)  O12—C9—C8 108.4 (2)
C4B—C3B—Br2 116.3 (2)  C16—C9—C8 110.5 (2)
C5B—C4B—C3B 119.2 (3)  O12—C9—H9 109.2
C5B—C4B—H4BA 120.4  C16—C9—H9 109.2
C3B—C4B—H4BA 120.4  C8—C9—H9 109.2
C4B—C5B—C6B 120.3 (3)  C11—C10—C3 128.9 (5)
C4B—C5B—H5BA 119.9  C11—C10—H10 115.6
C6B—C5B—H5BA 119.9  C3—C10—H10 115.6
C5B—C6B—C7B 120.0 (3)  C10—C11—H11A 120
C5B—C6B—H6BA 120  C10—C11—H11B 120
C7B—C6B—H6BA 120  H11A—C11—H11B 120
C6B—C7B—C2B 121.4 (3)  N13—C14—C15 124.8 (4)
C6B—C7B—H7BA 119.3  N13—C14—H14 117.6
C2B—C7B—H7BA 119.3  C15—C14—H14 117.6
N1—C2—C3  111.0 (2)  C16—C15—C14 119.4 (4)
N1—C2—H2A 109.4  C16—C15—H15 120.3
C3—C2—H2A 109.4  C14—C15—H15 120.3
N1—C2—H2B 109.4  C15—C16—C17 118.6 (3)
C3—C2—H2B 109.4  C15—C16—C9 119.4 (3)
H2A—C2—H2B 108  C17—C16—C9 122.0 (3)
C4—C3—C2  107.6 (2)  C18—C17—C16 125.3 (3)
C4—C3—C10 117.2 (3)  C18—C17—C22 117.4 (3)
C2—C3—C10 108.2 (3)  C16—C17—C22 117.3 (3)
C4—C3—H3 107.9  C19—C18—C17 121.5 (3)  C19—C18—H18 119.2
C2—C3—H3 107.9  C19—C18—H18 119.2
C10—C3—H3 107.9  C17—C18—H18 120.1
C3—C4—C5 108.4 (3)  C18—C19—C20 119.8 (4)  C18—C19—C20 119.8 (4)
C3—C4—C7 109.4 (3)  C18—C19—H19 120.1  C18—C19—H19 120.1
C5—C4—C7 108.3 (3)  C20—C19—H19 120.1  C20—C19—H19 120.1
C3—C4—H4 110.2  C21—C20—C19 120.5 (4)  C21—C20—C19 120.5 (4)
C5—C4—H4 110.2  C21—C20—H20 119.7  C21—C20—H20 119.7
C7—C4—H4 110.2  C19—C20—H20 119.7  C19—C20—H20 119.7
C4—C5—H5B 109.8  N13—C22—C21 118.1 (3)  N13—C22—C21 118.1 (3)
C5—C6—H6A 109.9  C2—N1—C6 111.48 (19)  C2—N1—C6 111.48 (19)
C5—C6—H6B 109.9  C2—N1—C1B 110.6 (2)  C2—N1—C1B 110.6 (2)
C6—C5—H5B 109.8  N13—C22—C17 122.7 (3)  N13—C22—C17 122.7 (3)
C6—C5—H5A 109.8  C21—C22—C17 119.3  C21—C22—C17 119.3
C5—C6—C7B 2.0 (5)  C12—C9—C16—C17 −175.1 (3)  C12—C9—C16—C17 −175.1 (3)
C6—C5—H5A 109.8  C15—C16—C17—C18 −178.6 (3)  C15—C16—C17—C18 −178.6 (3)
C4—C5—H5B 109.8  C9—C16—C17—C18 2.3 (5)  C9—C16—C17—C18 2.3 (5)
C5—C6—C7B 2.0 (5)  C15—C16—C17—C22 2.1 (4)  C15—C16—C17—C22 2.1 (4)
H5A—C5—H5B 108.2  C9—C16—C17—C22 −177.0 (3)  C9—C16—C17—C22 −177.0 (3)
H5B—C5—H5A 108.2  C21—C22—C17—C18 4.1 (5)  C21—C22—C17—C18 4.1 (5)
C1—C6—C7B 108.9 (2)  C15—C16—C17—C22 −177.0 (3)  C15—C16—C17—C22 −177.0 (3)
C1—C6—C7B 108.9 (2)  C9—C16—C17—C22 175.6 (3)  C9—C16—C17—C22 175.6 (3)
C2—C3—C4 16.7 (3)  C18—C17—C22—N13 −2.3 (5)  C18—C17—C22—N13 −2.3 (5)
C2—C3—C4 16.7 (3)  C18—C17—C22—N13 178.3 (3)  C18—C17—C22—N13 178.3 (3)
C3—C4—C5 144.2 (3)  C18—C17—C22—C17 4.1 (6)  C18—C17—C22—C17 4.1 (6)
C3—C4—C5 144.2 (3)  C18—C17—C22—C21 1.5 (6)  C18—C17—C22—C21 1.5 (6)
C2—C3—C4 68.9 (3)  C18—C17—C22—C21 −3.8 (5)  C18—C17—C22—C21 −3.8 (5)
C10—C3—C4 169.1 (3)  C16—C17—C22—N13 2.3 (5)  C16—C17—C22—N13 2.3 (5)
C2—C3—C4 169.1 (3)  C16—C17—C22—C17 175.6 (3)  C16—C17—C22—C17 175.6 (3)
C1—C3—C4 49.0 (3)  C3—C2—N1—C6 170.9 (2)  C3—C2—N1—C6 170.9 (2)
C10—C3—C4 49.0 (3)  C3—C2—N1—C1B 49.6 (3)  C3—C2—N1—C1B 49.6 (3)
C3—C4—C5 49.8 (3)  C3—C2—N1—C8 170.9 (2)  C3—C2—N1—C8 170.9 (2)
C7—C4—C5 68.8 (3)  C3—C2—N1—C8 170.9 (2)  C3—C2—N1—C8 170.9 (2)
C4—C5—C6 17.4 (3)  C5—C6—N1—C1B 169.2 (2)  C5—C6—N1—C1B 169.2 (2)
C3—C4—C7 73.6 (3)  C5—C6—N1—C8 50.5 (3)  C5—C6—N1—C8 50.5 (3)

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C5—C4—C7—C8 44.4 (3) C2B—C1B—N1—C2 −64.3 (3)
C4—C7—C8—N1 23.3 (3) C2B—C1B—N1—C6 55.2 (3)
C7—C8—C9—O12 −69.4 (3) C7—C8—N1—C2 41.8 (3)
C7—C8—C9—C16 51.5 (3) C7—C8—N1—C6 −74.9 (3)
N1—C8—C9—C16 175.1 (2) C9—C8—N1—C6 158.5 (2)
C4—C3—C10—C11 −29.2 (6) C7—C8—N1—C1B 165.8 (2)
C2—C3—C10—C11 −150.9 (5) C9—C8—N1—C1B 39.1 (3)
N13—C14—C15—C16 −2.8 (7) C15—C14—N13—C22 2.6 (6)
C14—C15—C16—C9 179.4 (3) C17—C22—N13—C14 0.0 (6)
O12—C9—C16—C15 5.8 (4) H12—O12—C9—C8 −149 (3)
C8—C9—C16—C15 −114.0 (3)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|------|-------|---------|
| O12—H12···Br1i | 0.81 (4) | 2.38 (4) | 3.179 (2) | 173 (3) |
| C2—H2····Br2 | 0.97 | 2.91 | 3.406 (3) | 113 |
| C2—H2B···O12 | 0.97 | 2.32 | 2.997 (4) | 126 |
| C6—H6A···Br1 | 0.97 | 2.88 | 3.797 (3) | 159 |
| C7—H7B···O12 | 0.97 | 2.65 | 3.030 (4) | 103 |
| C15—H15···O12 | 0.93 | 2.32 | 2.697 (4) | 104 |
| C18—H18···Br1 | 0.93 | 2.96 | 3.758 (4) | 145 |

Symmetry code: (i) x−1, y, z.