Poly[\(\mu_5\{-\text{hydrogen bis[(E)-cinnamato]}\}\)-caesium]

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*Acta Cryst.* (2014). **E70**, m43–m44

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Poly[μ₅-[hydrogen bis[(E)-cinnamato]]-caesium]

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Received 8 January 2014; accepted 13 January 2014

Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.017 Å; R factor = 0.071; wR factor = 0.144; data-to-parameter ratio = 16.0.

In the structure of the title polymeric complex, [Cs(C₉H₇O₂)(C₉H₈O₂)], a caesium salt of trans-cinnamic acid, the Cs⁺ ions of the two individual irregular Cs₅O₈ coordination polyhedra lie on twofold rotation axes and are linked by four bridging carboxyl O-atom donors from two cinnamate ligand species. These two ligand components are interlinked through a delocalized H atom within a short O · · ·H · · ·O hydrogen bond. Structure extension gives a two-dimensional coordination polymer which lies parallel to (001). The structure was determined from a crystal twinned by non-merohedry, with a twin component ratio of approximately 1:1.

Related literature

For the structures of the ammonium salts of hydrogen bis(3-chlorocinnamate) and hydrogen bis(3-bromocinnamate), see: Chowdhury & Kariuki (2006). For structures of alkali metal salts of ring-substituted trans-cinnamic acid, see: Kariuki et al. (1994, 1995); Crowther et al. (2008); Smith & Wermuth (2009, 2011). For the structure of trans-cinnamic acid, see: Wierda et al. (1989); Abdelmoty et al. (2005).

Experimental

Crystal data

[Cs(C₉H₇O₂)(C₉H₈O₂)]

Mᵣ = 428.21
Monoclinic, P2₁/c
a = 7.8608 (6) Å
b = 5.6985 (7) Å
c = 38.817 (3) Å
β = 98.733 (6)°

V = 1718.6 (3) Å³
Z = 4
Mo Kα radiation
μ = 2.17 mm⁻¹
T = 200 K
0.35 × 0.35 × 0.06 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2013)
Tmin = 0.711, Tmax = 0.980
3353 reflections
6675 measured reflections
3353 independent reflections
2552 reflections with I > 2σ(I)
Rint = 0.046

Refinement

R[FW2 > 2σ(F²)] = 0.071
wR(F²) = 0.144
S = 1.19
210 parameters
H-atom parameters constrained
Δρmax = 1.26 e Å⁻³
Δρmin = −2.19 e Å⁻³

Table 1

Selected bond lengths (Å).

|      | C1—O13B | 3.060 (8) | C2—O13B | 3.063 (8) |
|------|----------|----------|----------|----------|
| C1—O14A | 3.182 (8) | C2—O14A | 3.377 (9) |
| C1—O13A¹ | 3.132 (9) | C2—O13A¹ | 3.108 (9) |
| C1—O14B¹ | 3.183 (9) | C2—O14B¹ | 3.130 (9) |
| C1—O13B¹ | 3.060 (8) | C2—O13B¹ | 3.063 (8) |
| C1—O14A¹ | 3.182 (8) | C2—O14A¹ | 3.377 (9) |
| C1—O13A¹m | 3.132 (9) | C2—O13A¹m | 3.108 (9) |
| C1—O14B¹m | 3.183 (9) | C2—O14B¹m | 3.130 (9) |

Symmetry codes: (i) x, y, z + 1; (ii) −x + 1, y, −z + 1/2; (iii) −x + 1, y + 1, −z + 1/2; (iv) −x, y, −z + 3/2; (v) −x, y + 1, −z + 1/2.

Table 2

Hydrogen-bond geometry (Å, °).

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|------|------|--------|
| O14B—H14B···O14A | 1.21 | 1.25 | 2.462 (10) | 180 |

Data collection: CrysAlis PRO (Agilent, 2013); cell refinement: CrysAlis PRO; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); 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.

The author acknowledges financial support from the Science and Engineering Faculty and the University Library, Queensland University of Technology.

Supporting information for this paper is available from the IUCr electronic archives (Reference: WM2798).

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Poly[$\mu_5$-{hydrogen bis[(E)-cinnamato]}]-caesium

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1. Comment

The crystal structure of trans-cinnamic acid was reported by Wierda et al. (1989) and Abdelmoty et al. (2005). The alkali metal salts of trans-cinnamic acid are unknown in the crystallographic literature although a limited number of examples of salts of ring-substituted cinnamates have been reported, e.g. the sodium salts of 2-nitrocinnamate [a dihydrate (Smith & Wermuth, 2009)], of 2-chlorocinnamate [a dihydrate (Kariuki et al., 1995)], of 3-chlorocinnamate [anhydrous (Crowther et al., 2008), of 4-chlorocinnamate [a dihydrate (Kariuki et al., 1994); potassium salts of 3-chloro- and 3-bromocinnamate [both anhydrous (Crowther et al., 2008)]; and a rubidium salt of 2-nitrocinnamate [a monohydrate (Smith & Wermuth, 2011)].

The reaction of trans-cinnamic acid with caesium hydroxide in aqueous ethanol afforded crystals of the title complex, [Cs(C$_9$H$_7$O$_2$)(C$_9$H$_8$O$_2$)]$_n$, (I), the structure of which is reported herein.

In the structure of (I) the asymmetric unit (Fig. 1) comprises two independent irregular CsO$_8$ coordination polyhedra [Cs$_1$—O, 3.060 (8)–3.183 (9) Å; Cs$_2$—O, 3.063 (9)–3.377 (9) Å: Table 1], in which the Cs$^+$ ions lie on a twofold rotation axis and are linked by four bridging carboxyl O-donors from the two trans-cinnamate ligand species. These two ligand species are inter-linked through a delocalized H atom on an approximately central intermediate site within a short O$_4^A$···H$_14^B$···O$_4^B$ hydrogen bond [2.462 (10) Å] (Table 2). Although this phenomenon involving coordinating dimeric carboxylate species is not known among the alkali metal substituted-cinnamate structures, it is found in both ammonium hydrogen bis(3-chlorocinnamate) and ammonium hydrogen bis(3-bromocinnamate) (Chowdhury & Kariuki, 2006), with the O···H···O values [2.554 (6) Å for the 3-Cl-analogue and 2.466 (5) Å for the 3-Br-analogue] similar to that in the structure of (I). In this complex, the two Cs$^+$ ions are quadruply bridged giving a Cs$_1$···Cs$_2$ separation of 3.9318 (3) Å and generate an overall two-dimensional coordination polymer lying parallel to (001) (Figs. 2, 3). No inter-ring π–π interactions are present in the structure [minimum ring centroid separation = 4.826 (8) Å].

The two linked cinnamate species in the title complex are close to coplanar [inter-ring dihedral angle = 3.9 (6)$^\circ$], with the side chain carboxyl group of the A ligand component slightly rotated out of the plane [torsion angle C$_{11}^A$—C$_{12}^A$—C$_{13}^A$—O$_{13}^A$ = 169.0 (13)$^\circ$] compared to that of the B ligand component [torsion angle C$_{11}^B$—C$_{12}^B$—C$_{13}^B$—O$_{14}^B$ = -179.2 (11)$^\circ$]. With the analogous ammonium hydrogen salts of the 3-chloro- and 3-bromocinnamates (Chowdhury & Kariuki, 2006), the two cinnamate components are related either by crystallographic inversion symmetry (3-Cl) with the two benzene rings essentially planar, or by twofold rotational symmetry (3-Br) with the two rings significantly rotated out of the least-squares plane [inter-ring dihedral angle = 29.8 (2)$^\circ$].

2. Experimental

The title compound was synthesized by heating together for 10 minutes, 148 mg (1.0 mmol) of trans-cinnamic acid and 75 mg (0.5 mmol) of CsOH in 15 ml of an 1:9 (vol/vol) ethanol–water mixture. Partial room temperature evaporation of the solution gave colourless elongated crystals of the title complex from which a specimen was cleaved for the X-ray
analysis. These crystals were invariably twinned, a feature identified in the later structure solution and refinement routines.

3. Refinement

Hydrogen atoms were placed in calculated positions [C—H = 0.95 Å] and allowed to ride in the refinement, with $U_{iso}(H) = 1.2U_{eq}(C)$. The carboxylic acid H-atom was found to be delocalized in a site approximating to midway between two carboxyl O-atoms of the dimeric acid–anion unit and was subsequently allowed to ride at that site, with $U_{iso}(H) = 1.5U_{eq}(O)$. The presence of a non-merohedral twin was identified using TwinRotMat within PLATON (Spek, 2009) (twin law: $\overline{1}0\overline{0}$, $0\overline{1}0$, $1.5\overline{0}1$) reducing the conventional $R$-factor from 0.23 to 0.072, with a final BASF factor (HKLF 5 format) of 0.4836. Maximum and minimum residual electron densities were 1.26 eÅ$^{-3}$ (1.00 Å from Cs1) and -2.19 eÅ$^{-3}$ (1.94 Å from H14B), respectively.

Computing details

Data collection: CrysAlis PRO (Agilent, 2013); cell refinement: CrysAlis PRO (Agilent, 2013); data reduction: CrysAlis PRO (Agilent, 2013); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); 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 (Spek, 2009).

Figure 1

The atom-numbering scheme and the molecular configuration of the two ligands and the two CsO₈ coordination polyhedra of the title complex, with non-H atoms drawn with displacement ellipsoids at the 40% probability level. The two Cs⁺ cations lie on twofold rotation axes. The O14A···O14B hydrogen bond with the delocalized H atom (H14B) is shown as a dashed link. [For symmetry codes: see Table 1].
Figure 2
A view of the partially expanded polymeric extension of the structure viewed along the approximate $a$-cell direction. C-bound H atoms are omitted. A and B denote the two different ligand components.

Figure 3
The packing of the layered structure of compound (I) viewed along $b$.

Poly[$\mu$-hydrogen bis[(E)-cinnamato]]-caesium

Crystal data

$[\text{Cs}($C$_9$H$_7$O$_2$)(C$_9$H$_8$O$_2$)]$

$M_r = 428.21$

Monoclinic, $P2_1/c$

Hall symbol: -P 2yc

$a = 7.8608$ (6) Å

$b = 5.6985$ (7) Å

$c = 38.817$ (3) Å

$\beta = 98.733$ (6)$^\circ$
supplementary materials

\( V = 1718.6 \pm 3 \, \text{Å}^3 \)
\( Z = 4 \)
\( F(000) = 840 \)
\( D_r = 1.655 \, \text{Mg m}^{-3} \)
Mo Kα radiation, \( \lambda = 0.71073 \, \text{Å} \)

Cell parameters from 1674 reflections

\[ \theta = 3.6^\circ - 28.2^\circ \]
\[ \mu = 2.17 \, \text{mm}^{-1} \]
\( T = 200 \, \text{K} \)
Plate, colourless

0.35 × 0.35 × 0.06 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector

Radiation source: Enhance (Mo) X-ray source

Detector resolution: 16.077 pixels mm\(^{-1}\)

Absorption correction: multi-scan

\( R_{int} = 0.046 \)

\( \theta_{max} = 26.0^\circ, \theta_{min} = 3.2^\circ \)

Absorption correction: multi-scan

\( k = -7 \rightarrow 7 \)

\( l = -11 \rightarrow 47 \)

Refinement

Refinement on \( F^2 \)

Least-squares matrix: full

H-atom parameters constrained

\( R[F^2 > 2\sigma(F^2)] = 0.071 \)

\( wR(F^2) = 0.144 \)

3353 reflections

210 parameters

\( S = 1.19 \)

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

\( w = 1/[\sigma^2(F_o^2) + (0.020P)^2 + 18.34P] \)

\( P = (F_o^2 + 2F_c^2)/3 \)

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

\( \Delta\rho_{max} = 1.26 \, \text{e Å}^{-3} \)

\( \Delta\rho_{min} = -2.19 \, \text{e Å}^{-3} \)

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

|     | \( x \)   | \( y \)     | \( z \)   | \( U_{iso} \)/\( U_{eq} \) |
|-----|-----------|-------------|-----------|----------------------------|
| Cs1 | 0.50000   | 0.6438 (2)  | 0.25000   | 0.0261 (3)                 |
| Cs2 | 0.00000   | 0.6257 (2)  | 0.25000   | 0.0300 (3)                 |
| O13A| 0.2828 (13)| -0.1322 (15)| 0.3028 (2)| 0.037 (3)                  |
| O13B| 0.2180 (12)| 0.3903 (14) | 0.20079 (19)| 0.030 (3)         |
| O14A| 0.3036 (12)| 0.2355 (14) | 0.28374 (18)| 0.029 (3)         |
| O14B| 0.2197 (13)| 0.0396 (14) | 0.22718 (18)| 0.033 (3)         |
| C1A | 0.5037 (15)| 0.4316 (19) | 0.3905 (3) | 0.025 (3)             |
| C1B | 0.0000 (15)| 0.079 (2)   | 0.1031 (3) | 0.025 (3)             |
| C2A | 0.5963 (19)| 0.636 (2)   | 0.3964 (3) | 0.035 (4)             |
| C2B | 0.0132 (16)| 0.208 (2)   | 0.0728 (3) | 0.028 (3)             |
| C3A | 0.6620 (19)| 0.707 (2)   | 0.4301 (4) | 0.042 (5)             |
| C3B | -0.0664 (16)| 0.128 (3)  | 0.0403 (3) | 0.040 (4)             |

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C4A  0.6346 (18)  0.574 (3)  0.4585 (3)  0.042 (5)  
C4B  −0.1524 (18) −0.082 (2)  0.0373 (3)  0.041 (4)  
C5A  0.5426 (19)  0.373 (3)  0.4529 (3)  0.039 (4)  
C5B  −0.1628 (16) −0.210 (2)  0.0668 (3)  0.035 (4)  
C6A  0.4764 (15)  0.299 (2)  0.4191 (3)  0.029 (4)  
C6B  −0.0870 (15) −0.133 (2)  0.0997 (3)  0.031 (4)  
C11A  0.3456 (17)  0.359 (2)  0.3444 (3)  0.023 (3)  
C11B  −0.0831 (14) −0.210 (2)  0.0668 (3)  0.025 (3)  
C12A  0.3722 (13)  0.155 (2)  0.3078 (3)  0.028 (4)  
C12B  −0.1628 (13) −0.133 (2)  0.0997 (3)  0.026 (3)  
C13A  0.3148 (16)  0.076 (2)  0.3078 (3)  0.028 (4)  
C13B  −0.1901 (15) −0.176 (2)  0.1998 (3)  0.027 (4)  
H2A   0.61560  0.73090  0.37720  0.0410*  
H2B   0.07640  0.35110  0.07440  0.0340*  
H3A   0.72650  0.84820  0.43360  0.0510*  
H3B  −0.06090  0.21990  0.02000  0.0480*  
H4A   0.67930  0.62310  0.48150  0.0500*  
H4B  −0.20410 −0.13800  0.01520  0.0490*  
H5A   0.52280  0.28000  0.47230  0.0470*  
H5B  −0.22320 −0.35490  0.06480  0.0410*  
H6A   0.41240  0.15700  0.41580  0.0350*  
H6B  −0.09500 −0.22560  0.11970  0.0370*  
H11A  0.43910  0.47280  0.33640  0.0380*  
H11B  0.11540  0.33430  0.13740  0.0290*  
H12A  0.36110  0.04540  0.36230  0.0280*  
H12B  0.09570 −0.10990  0.16760  0.0320*  
H14B  0.26080  0.13620  0.25500  0.0500*  

Atomic displacement parameters (Å²)

|       | U¹¹ | U²² | U³³ | U¹² | U¹³ | U²³ |
|-------|-----|-----|-----|-----|-----|-----|
| Cs1   | 0.0221 (5)  | 0.0212 (5)  | 0.0350 (5)  | 0.0000 | 0.0041 (5) | 0.0000 |
| Cs2   | 0.0232 (5)  | 0.0238 (6)  | 0.0438 (6)  | 0.0000 | 0.0075 (6) | 0.0000 |
| O13A  | 0.056 (5)   | 0.027 (5)   | 0.028 (4)   | −0.001 (6) | 0.003 (4) | −0.006 (4) |
| O13B  | 0.047 (5)   | 0.026 (4)   | 0.016 (4)   | −0.004 (5) | 0.000 (4) | 0.000 (3) |
| O14A  | 0.046 (6)   | 0.033 (5)   | 0.006 (3)   | −0.003 (4) | −0.006 (3) | −0.005 (3) |
| O14B  | 0.055 (5)   | 0.025 (4)   | 0.013 (4)   | −0.004 (5) | −0.015 (4) | −0.005 (3) |
| C1A   | 0.022 (6)   | 0.022 (6)   | 0.030 (6)   | 0.003 (5) | 0.006 (5) | −0.009 (5) |
| C1B   | 0.020 (5)   | 0.034 (6)   | 0.022 (5)   | 0.004 (6) | 0.005 (5) | −0.007 (5) |
| C2A   | 0.038 (7)   | 0.027 (6)   | 0.039 (6)   | −0.004 (7) | 0.007 (6) | 0.003 (6) |
| C2B   | 0.027 (6)   | 0.028 (6)   | 0.027 (6)   | 0.007 (6) | 0.005 (5) | −0.003 (5) |
| C3A   | 0.044 (9)   | 0.021 (7)   | 0.059 (9)   | 0.012 (6) | −0.002 (7) | −0.024 (6) |
| C3B   | 0.043 (8)   | 0.050 (8)   | 0.027 (6)   | 0.026 (8) | 0.005 (5) | 0.000 (7) |
| C4A   | 0.031 (7)   | 0.059 (10)  | 0.033 (7)   | 0.006 (8) | −0.001 (6) | −0.022 (7) |
| C4B   | 0.037 (7)   | 0.053 (8)   | 0.030 (7)   | −0.011 (8) | −0.003 (6) | −0.012 (6) |
| C5A   | 0.035 (7)   | 0.048 (7)   | 0.035 (6)   | −0.003 (8) | 0.005 (6) | 0.012 (7) |
| C5B   | 0.029 (7)   | 0.021 (6)   | 0.054 (8)   | −0.005 (6) | 0.006 (6) | −0.004 (6) |
| C6A   | 0.031 (7)   | 0.027 (7)   | 0.030 (6)   | −0.007 (6) | 0.005 (5) | 0.004 (5) |
| C6B   | 0.032 (7)   | 0.027 (6)   | 0.032 (6)   | −0.002 (6) | 0.002 (5) | −0.002 (6) |
| C11A  | 0.035 (7)   | 0.029 (6)   | 0.032 (6)   | −0.002 (7) | 0.006 (6) | 0.002 (6) |
### Geometric parameters (Å, °)

| Bond                  | Length (Å) | Angle (°) |
|-----------------------|------------|-----------|
| Cs1—O13B              | 3.060 (8)  | C2A—C3A  | 1.392 (19) |
| Cs1—O14A              | 3.182 (8)  | C2B—C3B  | 1.397 (17) |
| Cs1—O13A\textsuperscript{i} | 3.132 (9) | C3A—C4A  | 1.38 (2)   |
| Cs1—O14B\textsuperscript{i} | 3.183 (9) | C3B—C4B  | 1.37 (2)   |
| Cs1—O13B\textsuperscript{ii} | 3.060 (8) | C4A—C5A  | 1.35 (2)   |
| Cs1—O14A\textsuperscript{ii} | 3.182 (8) | C4B—C5B  | 1.371 (16) |
| Cs1—O13A\textsuperscript{iii} | 3.132 (9) | C5A—C6A  | 1.401 (17) |
| Cs1—O14B\textsuperscript{iii} | 3.183 (9) | C5B—C6B  | 1.396 (16) |
| C3A—O14A\textsuperscript{iv} | 3.063 (8) | C11A—C12A  | 1.298 (16) |
| C2A—O14A\textsuperscript{v} | 3.377 (9) | C11A—C12A  | 1.349 (16) |
| C2A—O13B              | 3.060 (8)  | C13A—C13B | 1.222 (14) |
| C1B—C11B              | 1.492 (16) | C6A—C6B  | 0.950      |
| O13A—C13A             | 1.222 (14) | C4A—C4B  | 0.950      |
| O14A—C13A             | 1.297 (14) | C5A—C5B  | 0.950      |
| O14B—C13B             | 1.309 (14) | C5B—C5B  | 0.950      |
| O14B—H14B             | 1.2100     | C6A—H6B  | 0.950      |
| C1A—C11A              | 1.492 (16) | C6B—H6B  | 0.950      |
| C1A—C2A               | 1.374 (17) | C11A—H11A | 0.950     |
| C1A—C6A               | 1.386 (16) | C11B—H11B | 0.950    |
| C1B—C11B              | 1.475 (16) | C12A—H12A | 0.950    |
| C1B—C6B               | 1.385 (16) | C12B—H12B | 0.950    |
| C1B—C2B               | 1.404 (16) | C1A—C11A  | 135.0 (8) |
| O13B—Cs1—O14A         | 64.0 (2)   | Cs2—O14A—C13A | 132.6 (7) |
| O13A—Cs1—O13B         | 100.7 (2)  | Cs1\textsuperscript{iv}—O14B—C13B | 129.8 (7) |
| O13B—Cs1—O14B\textsuperscript{i} | 75.9 (2) | Cs2\textsuperscript{iv}—O14B—C13B | 77.04 (18) |
| O13B—Cs1—O13B\textsuperscript{ii} | 123.7 (2) | Cs1\textsuperscript{iv}—O14B—Cs2\textsuperscript{ii} | 93.00 |
| O13A\textsuperscript{iii}—Cs1—O13B | 75.4 (2) | Cs1—O14A—H14B | 83.00 |
| O13B—Cs1—O14B\textsuperscript{iii} | 101.5 (2) | Cs2—O14A—H14B | 100.00 |
| O13A—Cs1—O14A         | 155.98 (19) | Cs2\textsuperscript{iv}—O14B—H14B | 90.00 |
| O13B—Cs1—O14B\textsuperscript{iv} | 71.5 (2) | Cs1\textsuperscript{iv}—O14B—H14B | 116.00 |
| O14A—Cs1—O14A         | 105.9 (2)  | C13B—O14B—H14B | 122.0 (10) |
| O14B—Cs1—O14A         | 75.4 (2)   | C6A—C1A—C11A | 118.1 (11) |
| O14A—Cs1—O14A\textsuperscript{i} | 86.0 (2) | C2A—C1A—C6A  | 119.9 (10) |
| O14A—Cs1—O14A\textsuperscript{ii} | 156.1 (2) | C2A—C1A—C11A | 118.4 (11) |
| O13A—Cs1—O14B\textsuperscript{iii} | 139.65 (18) | C2B—C1B—C6B | 118.4 (11) |
| O13A—Cs1—O14B\textsuperscript{iv} | 58.0 (2) | C2B—C1B—C11B | 118.4 (11) |
supplementary materials

| Bond                              | Distance (Å) | Angle (°)  |
|-----------------------------------|--------------|------------|
| O13A^i—Cs1—O13B^ii               | 101.5 (2)    |            |
| O13A^i—Cs1—O14A^i                | 156.1 (2)    |            |
| O13A^i—Cs1—O13A^ii               | 131.9 (2)    |            |
| O13B^ii—Cs1—O14B^ii              | 87.3 (2)     |            |
| O13B^ii—Cs1—O14B^i               | 155.98 (19)  |            |
| O14A^ii—Cs1—O14B^i               | 139.65 (18)  |            |
| O13A^iii—Cs1—O14B^ii             | 87.3 (2)     |            |
| O14B^ii—Cs1—O14B^ii              | 89.8 (2)     |            |
| O13B^ii—Cs1—O14A^i               | 64.0 (2)     |            |
| O13A^iii—Cs1—O13B^ii             | 100.7 (2)    |            |
| O13B^ii—Cs1—O14A^ii              | 7.5 (2)      |            |
| O14B^ii—Cs1—O14A^ii              | 71.5 (2)     |            |
| O14A^iv—Cs1—O14B^ii              | 105.9 (2)    |            |
| O13A^iv—Cs1—O14B^ii              | 58.0 (2)     |            |
| O13C—Cs2—O14A                    | 61.58 (19)   |            |
| O13A—Cs2—O13B                    | 101.2 (2)    |            |
| O13B—Cs2—O14B                    | 76.6 (2)     |            |
| O14B—Cs2—O13B                    | 128.1 (2)    |            |
| O13B—Cs2—O14A                    | 84.2 (2)     |            |
| O14A—Cs2—O14B                    | 101.3 (2)    |            |
| O13B—Cs2—O14B                    | 153.1 (2)    |            |
| O13A—Cs2—O14A                    | 69.2 (2)     |            |
| O14A—Cs2—O14B                    | 102.6 (2)    |            |
| O13B—Cs2—O14B                    | 84.2 (2)     |            |
| O14A—Cs2—O14B                    | 97.7 (2)     |            |
| O13B—Cs2—O14B                    | 160.1 (2)    |            |
| O14A—Cs2—O14B                    | 140.74 (18)  |            |
| O13A—Cs2—O14B                    | 58.8 (2)     |            |
| O13C—Cs2—O14B                    | 101.3 (2)    |            |
| O13A—Cs2—O14A                    | 160.1 (2)    |            |
| O13B—Cs2—O14A                    | 127.3 (2)    |            |
| O13A—Cs2—O14A                    | 81.3 (2)     |            |
| O13B—Cs2—O14B                    | 153.1 (2)    |            |
| O14A—Cs2—O14B                    | 140.74 (18)  |            |
| O13A—Cs2—O14B                    | 81.3 (2)     |            |
| O13B—Cs2—O14B                    | 82.2 (2)     |            |
| O13B—Cs2—O14B                    | 61.58 (19)   |            |
| O13A—Cs2—O14B                    | 101.2 (2)    |            |
| O13B—Cs2—O14B                    | 76.6 (2)     |            |
| O13A—Cs2—O14B                    | 69.2 (2)     |            |
| O13B—Cs2—O14B                    | 102.6 (2)    |            |
| O13A—Cs2—O14B                    | 58.8 (2)     |            |
| Cs1—O13A—C13A                    | 112.3 (8)    |            |
| Cs1—O13A—C13A                    | 130.2 (8)    |            |
| Cs1—O13A—C13A                    | 78.11 (18)   |            |
| Cs1—O13B—Cs2                     | 79.91 (18)   |            |
| Cs1—O13B—C13B                    | 126.3 (7)    |            |
| Cs1—O13B—C13B                    | 109.7 (7)    |            |
| Cs1—O14A—Cs2                     | 73.59 (16)   |            |
Cs1—O14A—C13A 145.6 (8)

| Bond                        | Angle (°) (°) |
|-----------------------------|---------------|
| O14A—Cs1—O13B—Cs2          | 65.6 (2)      |
| O14A—Cs1—O13B—C13B         | -41.6 (9)     |
| O13A—Cs1—O13B—C13B         | 2.5 (2)       |
| O13A—Cs1—O13B—C13B         | -104.7 (9)    |
| O14B—Cs1—O13B—C13B         | -50.38 (19)   |
| O14B—Cs1—O13B—C13B         | -157.5 (9)    |
| O13Bii—Cs1—O13B—C13B       | 114.2 (2)     |
| O13Bii—Cs1—O13B—C13B       | 7.0 (10)      |
| O14Aii—Cs1—O13B—C13B       | 158.3 (2)     |
| O14Aii—Cs1—O13B—C13B       | 51.2 (9)      |
| O13Aiii—Cs1—O13B—C13B      | -134.6 (2)    |
| O13Aiii—Cs1—O13B—C13B      | 118.3 (9)     |
| O14Biii—Cs1—O13B—C13B      | 147.6 (9)     |
| O13B—Cs1—O14A—C13A         | 151.0 (13)    |
| O13B—Cs1—O14A—C13A         | 54.52 (19)    |
| O13B—Cs1—O14A—C13A         | -96.5 (12)    |
| O14B—Cs1—O14A—C13A         | 7.10 (18)     |
| O14B—Cs1—O14A—C13A         | -57.9 (2)     |
| O13B—Cs1—O14A—C13A         | 162.23 (19)   |
| O13B—Cs1—O14A—C13A         | 11.2 (9)      |
| O14A—Cs1—O14A—C13A         | -133.66 (17)  |
| O14A—Cs1—O14A—C13A         | 75.3 (12)     |
| O13A—Cs1—O14A—C13A         | -114.2 (5)    |
| O13A—Cs1—O14A—C13A         | 94.7 (13)     |
| O14B—Cs1—O14A—C13A         | 116.3 (3)     |
| O14B—Cs1—O14A—C13A         | -34.7 (14)    |
| O13B—Cs1—O14A—C13A         | -2.5 (2)      |
| O14A—Cs1—O14A—C13A         | -60.15 (19)   |
| O13B—Cs1—O14A—C13A         | 49.60 (18)    |
| O14A—Cs1—O14A—C13A         | -7.55 (19)    |
| O14A—Cs1—O14A—C13A         | -61.2 (2)     |
| O14A—Cs1—O14A—C13A         | 63.9 (7)      |
| O13A—Cs2—O13B—C13B         | -2.5 (2)      |
| O13A—Cs2—O13B—C13B         | 122.6 (7)     |
| O14B—Cs2—O13B—C13B         | 51.35 (19)    |
| O14B—Cs2—O13B—C13B         | 176.5 (8)     |
| O13B—Cs2—O13B—C13B         | -116.5 (2)    |
| O13B—Cs2—O13B—C13B         | 8.6 (8)       |
| O14A—Cs2—O13B—C13B         | -163.16 (19)  |
| O14A—Cs2—O13B—C13B         | -38.1 (7)     |
| O13A—Cs2—O13B—C13B         | 129.5 (2)     |
| O13A—Cs2—O13B—C13B         | -105.4 (7)    |
| O14B—Cs2—O13B—C13B         | 90.3 (5)      |
| O14B—Cs2—O13B—C13B         | -144.6 (7)    |
| O13B—Cs2—O14A—Cs1          | 59.9 (2)      |

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O13B—Cs2—O14A—C13A  −142.9 (10)  C1B—C11B—C12B—C13B  −175.3 (11)
O13A1—Cs2—O14A—Cs1  −56.3 (2)  C11A—C12A—C13A—O13A  169.0 (13)
O13A1—Cs2—O14A—C13A  100.9 (10)  C11A—C12A—C13A—O14A  −10.7 (18)
O14B—Cs2—O14A—Cs1  −7.12 (18)  C11B—C12B—C13B—O13B  1.6 (18)
O14B1—Cs2—O14A—C13A  150.1 (9)  C11B—C12B—C13B—O14B  −179.2 (11)

Symmetry codes: (i) x, y+1, z; (ii) −x+1, y, −z+1/2; (iii) −x+1, y+1, −z+1/2; (iv) −x, y, −z+1/2; (v) −x, y+1, −z+1/2; (vi) x, y−1, z.

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H  | H···A  | D···A     | D—H···A |
|---------|------|-------|-----------|---------|
| O14B—H14B···O14A | 1.21 | 1.25  | 2.462 (10) | 180     |
| C11B—H11B···O13B  | 0.95 | 2.49  | 2.830 (14) | 101     |