

CRYSTALLOGRAPHIC
COMMUNICATIONS

ISSN 2056-9890

Crystal structure of 4-bromocinnamic anhydride

Yuliana Ramos Cotrina,^a Jhesua Valencia,^a Ronan Le Lagadec,^b Fernando Cuenú-Cabezas^a and Jovanny A. Gómez Castaño^{c*}

^aLaboratorio de Química Inorgánica y Catálisis, Programa de Química, Universidad del Quindío, Carrera 15 Calle 12 Norte, Armenia 630004, Colombia, ^bInstituto de Química UNAM, Circuito Exterior s/n, Ciudad Universitaria, Ciudad de México 04510, Mexico, and ^cGrupo Química-Física Molecular y Modelamiento Computacional (QUIMOL), Escuela de Ciencias Químicas, Universidad Pedagógica y Tecnológica de Colombia Sede Tunja, Avenida Central del Norte, Tunja 150003, Boyacá, Colombia. *Correspondence e-mail: jovanny.gomez@uptc.edu.co

Received 17 July 2025

Accepted 2 September 2025

Edited by K. V. Domasevitch, National Taras Shevchenko University of Kyiv, Ukraine

Keywords: crystal structure; cinnamic anhydride; supramolecular interactions; O... π contacts.

CCDC reference: 2484646

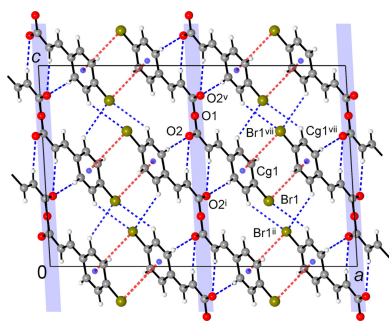
Supporting information: this article has supporting information at journals.iucr.org/e

In the crystal, molecules of the title compound [systematic name: (*E*)-3-(4-bromophenyl)prop-2-enoyl (*E*)-3-(4-bromophenyl)prop-2-enoate], C₁₈H₁₂Br₂O₃, reside across twofold axes passing through the central O atom of the C(O)—O—C(O) linkage [$Z' = \frac{1}{2}$]. The molecule framework adopts an *E* configuration across the C=C bonds and a *gauche* conformation across the anhydride bridge, with a O—C—O—C torsion angle of 31.70 (11)°. The three-dimensional supramolecular structure is governed by the interplay of C—H...O hydrogen bonds and slipped stacking interactions involving carbonyl/C₆H₄Br and antiparallel C₆H₄Br/C₆H₄Br pairs. Hirshfeld surface and fingerprint plot analyses reveal major contributions from Br...H/H...Br and O...H/H...O contacts. The largest interaction energies (up to −48.9 kJ mol^{−1}) are associated with stacking of the molecules, which highlight the dispersion-dominated stabilization. The comparable energetics of hydrogen-bonded pairs (−37.9 kJ mol^{−1}) is a result of as many as four synergetic geometrically favorable C—H...O interactions. The study represents the first structural characterization of a *p*-halogenated cinnamic anhydride and these findings could be applicable to crystal design with cinnamic derivatives.

1. Chemical context

Cinnamic anhydride derivatives, *R*—CH=CH—C(O)—O—C(O)—CH=CH—*R'*, where *R* and *R'* denote aromatic substituents, represent a versatile class of compounds applicable to organic synthesis, medicinal chemistry, and materials science (Raja *et al.*, 2017). Their electrophilic carbonyl groups render them reactive toward nucleophiles such as alcohols, amines and enolates, enabling the selective introduction of carbonyl functionalities into diverse molecular frameworks (Lin *et al.*, 2021; Robinson *et al.*, 2013). Noteworthy applications include esterification of xylans using ionic liquids to produce hemicellulose derivatives (Yang *et al.*, 2017), the efficient one-pot synthesis of thioesters with sodium thiosulfate pentahydrate (Liao & Liang, 2018), and C—H activation strategies such as rhodium(I)-catalyzed alkenylation of 2-pyridones (Zhao *et al.*, 2019). Furthermore, their potential as selective acetylcholinesterase inhibitors has underscored their relevance in medicinal and neuropharmacological research (Giessel *et al.*, 2019).

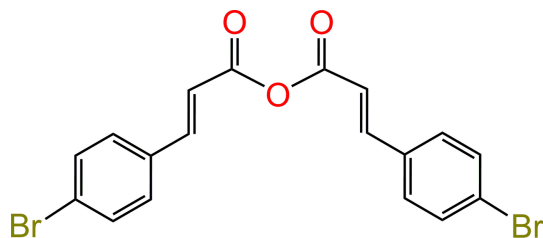
Within this family, halogen-substituted cinnamic anhydrides are of particular interest because the electronic effects of halogen substituents can influence both the molecular reactivity and crystal packing (Raja *et al.*, 2017). For example, crystal engineering with cinnamic acid derivatives attracts significant attention in the view of solid-state [2 + 2] cycloadditions (Liu *et al.*, 2025). Despite this relevance, the crystal



OPEN ACCESS

Published under a CC BY 4.0 licence

structures of *p*-halocinnamic anhydrides have not previously been reported. The presence of a halogen atom offers the possibility of halogen bonding and other directional intermolecular interactions. Herein, we describe the first single-crystal X-ray diffraction study of such a species, namely the title compound *p*-bromocinnamic anhydride (I), providing detailed insights into its molecular geometry and supramolecular features in the solid state.



2. Structural commentary

The title compound crystallizes in monoclinic space group *C2/c*, with the unique portion of the structure comprising half a molecule ($Z' = \frac{1}{2}$) lying about a crystallographic twofold rotation axis passing through the central anhydride O1 atom (Fig. 1).

The molecule adopts a *E*-configuration about each C=C double bond and displays a *gauche* conformation across the O—C—O—C anhydride bridge. The dihedral angle between the two C=C—C(O) planes is $55.36(3)^\circ$. This conformation differs from the *syn-tt-tt* arrangement predicted to be the most stable in the gas phase for cinnamic anhydride and 3-chlorocinnamic anhydride relatives at the B3LYP/6-311++g(d,p) level of theory (Mary *et al.*, 2014*a,b*).

Each half of the molecule, comprising a 4-bromophenyl–vinyl–carboxyl fragment, is nearly planar, with dihedral angle of $4.66(6)^\circ$ between the 4-bromophenyl ring and the C=C—C(O) plane. This value is slightly smaller than the corresponding torsion angle of 8.85° observed in crystalline 4-bromocinnamic acid (Yates & Sparkes, 2013).

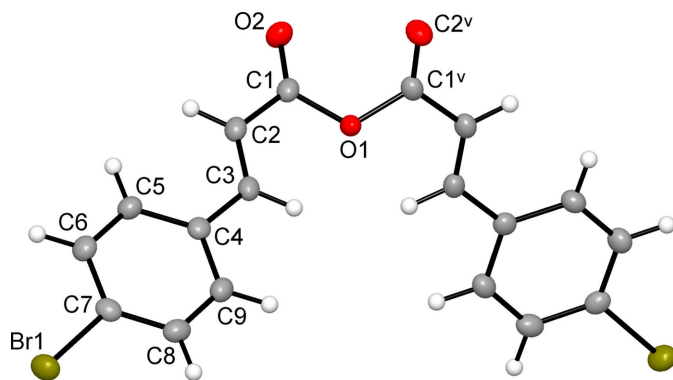


Figure 1

The molecular structure of (I), showing the atom labeling and displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (v) $-x + 1, y, -z + \frac{1}{2}$.]

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C2—H2...O2 ⁱ	0.95 (2)	2.51 (2)	3.4574 (18)	171.6 (16)
C5—H5...O2 ⁱ	0.95 (2)	2.59 (2)	3.5219 (18)	168.0 (17)
C6—H6...Br1 ⁱⁱ	0.96 (2)	3.20 (2)	3.9492 (14)	136.0 (15)
C8—H8...Br1 ⁱⁱⁱ	0.94 (2)	3.20 (2)	4.1064 (15)	161.0 (15)
C9—H9...Br1 ^{iv}	0.971 (19)	3.095 (18)	3.8551 (14)	136.3 (13)

Symmetry codes: (i) $-x + 1, -y, -z + 1$; (ii) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (iii) $-x + \frac{3}{2}, -y + \frac{5}{2}, -z + 1$; (iv) $x, -y + 2, z + \frac{1}{2}$.

3. Supramolecular features

In the crystal, molecules of (I) are linked into hydrogen-bonded chains running down the *c*-axis direction (Fig. 2), in which the inversion-related 4-bromophenyl–vinyl–carboxyl fragments [symmetry code: (i) $-x + 1, -y, -z + 1$] are linked by two pairs of reciprocal C2—H...O2ⁱ and C5—H...O2ⁱ hydrogen bonds (Table 1). These multiple interactions are geometrically favorable, as it is reflected by nearly straight angles at the hydrogen atoms [$168.0(17)$ and $171.6(16)^\circ$]. The C2...O2ⁱ separation of 3.4574 (18) Å perfectly agrees with the

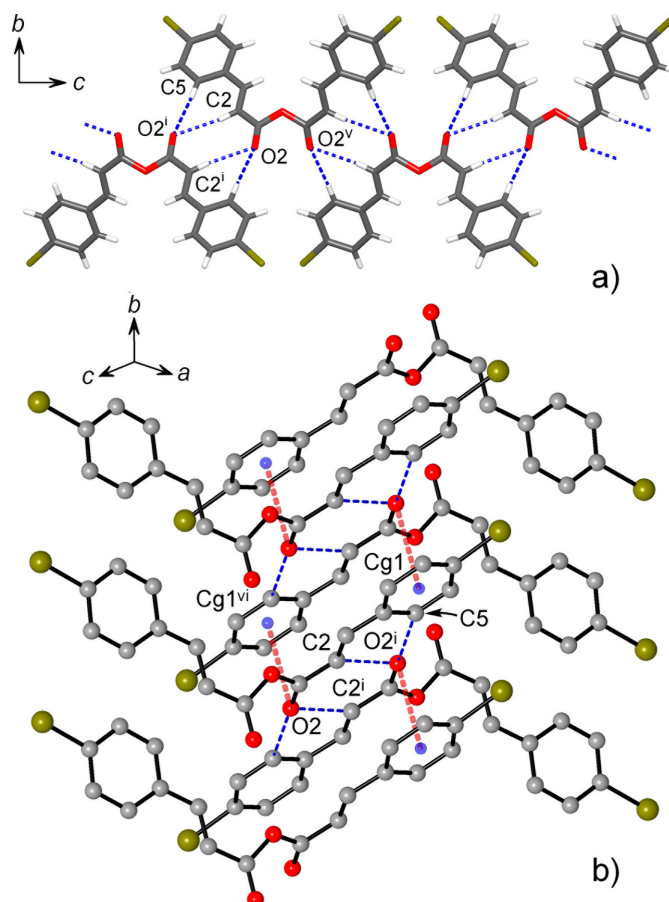


Figure 2

(a) Supramolecular chain down the *c*-axis direction, sustained by multiple C—H...O hydrogen bonding; (b) double carbonyl– π stacking interactions supporting columns along *b*-axis direction. O... π contacts and hydrogen bonds are indicated in red and blue, respectively. [Symmetry codes: (i) $-x + 1, -y, -z + 1$; (v) $-x + 1, y, -z + \frac{1}{2}$; (vi) $-x + 1, -y + 1, -z + 1$.]

mean value for such hydrogen bonds from statistical analysis of cinnamate esters (3.47 Å; Pálíncó, 1999). A second type of packing-defining force is associated with two kinds of stacking interactions. The first motif arises from antiparallel alignment of the inversion-related $\text{C}_6\text{H}_4\text{—C}=\text{C}=\text{C}=\text{O}$ fragments, which yields double carbonyl/ring interactions with notably short $\text{O2}\cdots\text{Cg1}^{\text{vi}}$ and $\text{O2}\cdots\text{plane}$ distances of 3.4770 (13) Å and 3.3876 (14) Å, respectively [Cg1 is the ring centroid; symmetry code: (vi) $-x + 1, -y + 1, -z + 1$] (Fig. 2). In combination with the above hydrogen bonding, these interactions assemble the molecules into the layers parallel to the bc plane.

This pattern bears a close resemblance to the one in methyl 4-bromocinnamate (Leiserowitz & Schmidt, 1965). The present structure inherits not only its local hydrogen-bonding motif with multiple reciprocal interactions, but also stacking of hydrogen-bonded dimers leading to similar columns. In fact, (I) may be best related to methyl 4-bromocinnamate when considering the anhydride linkage $\text{C1—O1—C1}^{\text{ii}}$ [symmetry code: (ii) $-x + 1, y, -z + \frac{3}{2}$] as a kind of bridge between pairs of 4-bromocinnamate ‘tectons’ that formally condition connection of the columns in a second dimension. This is illustrative of a general principle of crystal engineering and it may suggest certain and still unexplored potential of anhydrides for crystal design.

The second type of stacking is found between the layers (Fig. 3), in the form of slipped-antiparallel dimers of $\text{Br—C}_6\text{H}_4$ fragments [symmetry code: (vii) $-x + \frac{3}{2}, -y + \frac{3}{2}, -z + 1$], with two bromine atoms located approximately above the corresponding ring centroids [$\text{Br1}\cdots\text{Cg1}^{\text{vii}} = 4.0521$ (6) Å; $\text{Br1}\cdots\text{plane} = 3.6963$ (12) Å]. However, this stack is associated with relatively large slippage of 24.19 (2)°, defined as the angle subtended by the $\text{Br1}\cdots\text{Cg1}^{\text{vii}}$ axis to the ring

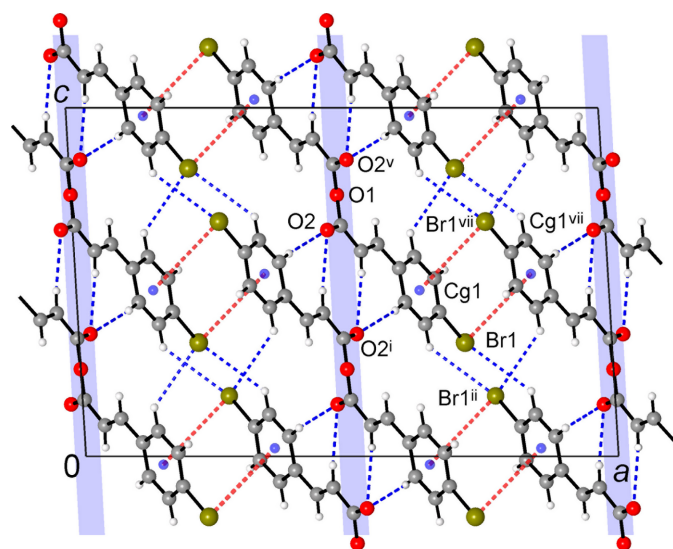


Figure 3
Projection of the structure in the ac -plane showing mutual interactions of $\text{C}_6\text{H}_4\text{Br}$ groups and $\text{C—H}\cdots\text{Br}$ contacts between successive hydrogen- and $\text{O}\cdots\pi$ bonded layers (which are orthogonal to the drawing plane and are indicated by blue strips). [Symmetry codes: (i) $-x + 1, -y, -z + 1$; (ii) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (v) $-x + 1, y, -z + \frac{1}{2}$; (vii) $-x + \frac{3}{2}, -z + \frac{3}{2}, -z + 1$.]

normal. The lack of the essential overlap is also reflected by the large intercentroid distance of 4.375 (2) Å.

The bromine atoms also engage in a set of distal contacts, e.g. $\text{Br1}\cdots\text{Br1}^{\text{viii}} = 3.7166$ (2) Å [symmetry code: (viii) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$]. These separations slightly exceed the sum of the van der Waals radii for bromine (3.70 Å; Bondi, 1964), suggesting a weakness of the present halogen $\cdots$ halogen contacts. As well, there are three types of $\text{H}\cdots\text{Br}$ contacts (Table 1), the shortest of which is $\text{H9}\cdots\text{Br1}^{\text{iv}} = 3.095$ (18) Å [symmetry code: (iv) $x, -y + 2, z + \frac{1}{2}$]. They are reflective of very weak hydrogen bonding or dispersion forces.

4. Hirshfeld surface analysis

The Hirshfeld surface (HS) of compound (I), mapped over the normalized contact distance (d_{norm}) (Fig. 4), highlights the contributions of carbonyl-based hydrogen bonding and $\text{C}\cdots\text{C}$ contacts associated with $\pi\text{—}\pi$ interactions to the consolidation of the crystal structure (Spackman & Jayatilaka, 2008). Two sets of four intense red spots on the HS correspond to reciprocal $\text{C}=\text{O}\cdots\text{H—C}$ interactions, involving carbonyl oxygen atoms and two types of hydrogen donors: vinyl hydrogen ($\text{H}\cdots\text{O} = 2.51$ Å) and aromatic *ortho*-hydrogen ($\text{H}\cdots\text{O} = 2.59$ Å)]. Weaker red spots are associated with mutual $\text{C}\cdots\text{C}$ contacts, one set between the aromatic *ortho*-carbon and vinyl carbon (3.35 Å) along the b -axis, and the other between an *ortho*-carbon and a carbonyl carbon (3.38 Å) across adjacent molecular b -axis columns.

The two-dimensional fingerprint plots (Fig. 5) further quantify the contributions of specific interactions to the HS (McKinnon *et al.*, 2007; Spackman & McKinnon, 2002). The largest contribution arises from $\text{Br}\cdots\text{H}/\text{H}\cdots\text{Br}$ contacts (24.5%), mainly involving *ortho*-positioned aromatic hydrogens. These appear as wing-like features in the fingerprint plot (Fig. 4b), with characteristic tips at $d_{\text{e}}/d_{\text{i}} \simeq 1.9/1.1$ Å, indicative of directional interactions. Therefore, in spite of relatively large distances, the fingerprint plots allow attribution of the contacts to very weak $\text{C—H}\cdots\text{Br}$ hydrogen bonding.

$\text{O}\cdots\text{H}/\text{H}\cdots\text{O}$ contacts (19.2%), corresponding to the above $\text{C}=\text{O}\cdots\text{H—C}$ interactions, produce the sharpest spikes at $d_{\text{e}}/d_{\text{i}} \simeq 1.4/1.0$ Å (Fig. 5d). $\text{C}\cdots\text{C}$ contacts (6.2%) reflect slipped stacking between vinyl and aromatic fragments. Perceptible contributions also include $\text{C}\cdots\text{O}/\text{O}\cdots\text{C}$ (3.9%) and $\text{C}\cdots\text{Br}/\text{Br}\cdots\text{C}$ (3.2%), which are consistent with the observed $\text{O}\cdots\pi$

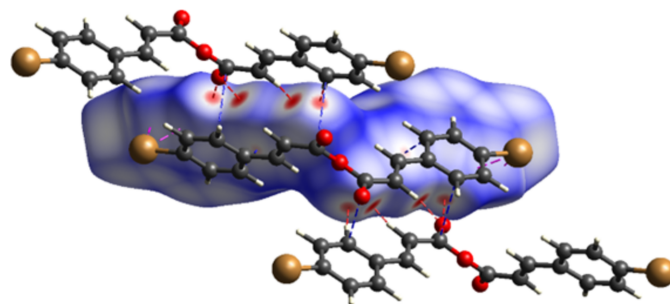


Figure 4
Hirshfeld surface of the molecule of (I) mapped over d_{norm} .

Table 2Calculated interaction energies (kJ mol^{-1}).

R is the distance between centroids of the interacting molecules. Interaction energies were calculated employing the CE-B3LYP/6–31G(d,p) functional/basis set combination. The scale factors used to determine E_{tot} : $k_{\text{ele}} = 1.057$, $k_{\text{pol}} = 0.740$, $k_{\text{dis}} = 0.871$, and $k_{\text{rep}} = 0.618$ (Mackenzie *et al.*, 2017).

Path	Symmetry relation	Type	R (Å)	E_{ele}	E_{pol}	E_{dis}	E_{rep}	E_{tot}
$A \cdots B$	$x, y + 1, z$	$\text{C} \cdots \text{C}$; dispersion	5.70	−17.2	−5.8	−58.9	40.4	−48.9
$A \cdots C$	$-x + 1, -y, -z + 1$	$\text{C} - \text{H} \cdots \text{O}$; HB	10.48	−32.2	−7.7	−16.5	26.2	−37.9
$A \cdots D$	$-x + 1, -y + 1, -z + 1$	$\text{O} \cdots \pi$; dispersion	7.15	−0.7	−3.2	−56.8	32.4	−31.1
$A \cdots E$	$-x + \frac{3}{2}, -y + \frac{3}{2}, -z + 1$	$\text{Br} \cdots \pi$ stacking	12.71	6.8	−0.4	−28.9	0.0	−18.3
$A \cdots F$	$x, -y + 2, z + \frac{1}{2}$	$\text{Br} \cdots \text{H}$	7.59	−7.0	−1.1	−17.1	15.5	−13.5

($d_o/d_i \simeq 1.9/1.6$ Å) and $\text{Br} \cdots \pi$ contacts. A smaller percentage arise from $\text{Br} \cdots \text{Br}$ (2.3%) contacts, corresponding to weak halogen $\cdots$ halogen interactions at distances approaching the sum of the van der Waals radii (3.70 Å; Bondi, 1964).

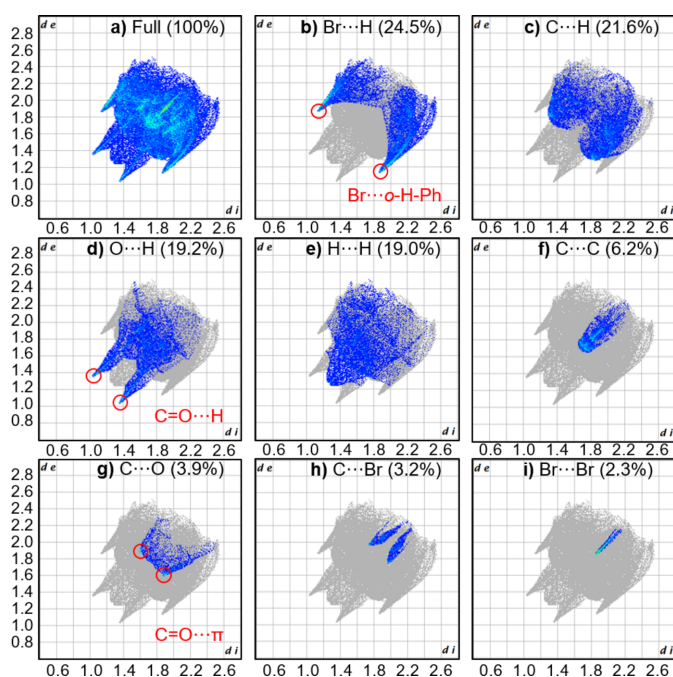
5. Interaction energy calculations

Pairwise interaction energies were calculated using the CE-B3LYP model implemented in *CrystalExplorer* (Mackenzie *et al.*, 2017; Turner *et al.*, 2015) to assess the energetic contributions stabilizing the supramolecular architecture of (I) (Fig. 6). The total interaction energy (E_{tot}) is expressed as the sum of electrostatic (E_{ele}), polarization (E_{pol}), dispersion (E_{dis}) and exchange-repulsion (E_{rep}) terms.

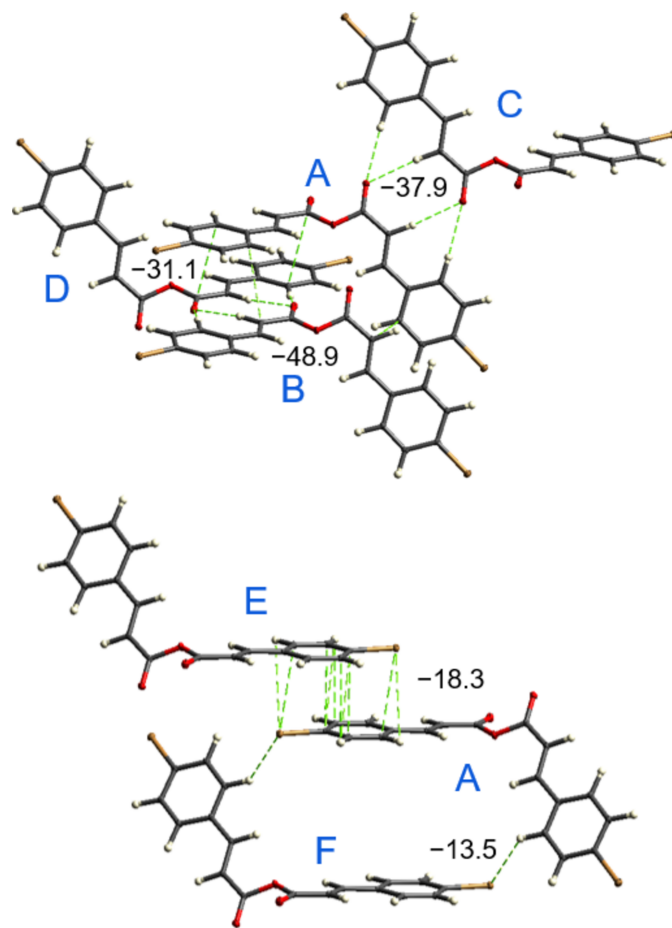
Considering interactions with $|E_{\text{tot}}| \geq 12.0 \text{ kJ mol}^{-1}$, five symmetry-independent paths were identified in the closest environment of the title molecule (Table 2). The strongest interaction [$E_{\text{tot}} = -48.9 \text{ kJ mol}^{-1}$; $R = 5.70$ Å] occurs between translation-related molecules along the b -axis, where $\text{C} \cdots \text{C}$ contacts between p -bromophenyl rings and vinyl

groups dominate (pair $A \cdots B$, Fig. 6). This interaction is dispersion-driven ($E_{\text{dis}} = -58.9 \text{ kJ mol}^{-1}$) and its significant energy originates in a relatively large interaction area.

Second notable interaction energy ($E_{\text{tot}} = -37.9 \text{ kJ mol}^{-1}$; $R = 10.48$ Å) is supported by $\text{C} - \text{H} \cdots \text{O}$ bonding of inversion-related molecules ($-x + 1, -y, -z + 1$; pair $A \cdots C$) and it is characterized by prevalence of the electrostatic component ($E_{\text{ele}} = -32.2 \text{ kJ mol}^{-1}$). These relatively large values agree with the formation of multiple hydrogen bonds, which act in synergy. Accordingly, a comparable $\text{C} - \text{H} \cdots \text{O}$ -bonded dimer of acrylic acid, which retains only two out of four present directional bonds, revealed a lower by half interaction energy

**Figure 5**

Two-dimensional fingerprint plots showing all interactions and delineated into the principal contributions of different types of the contacts (including also reciprocal contacts).

**Figure 6**

Principal pathways of pairwise intermolecular interactions, identified with a cut-off limit of 12.0 kJ mol^{-1} . Partial energetic contributions, symmetry operations, and geometric related parameters are summarized in Table 2.

of $-19.9 \text{ kJ mol}^{-1}$ (Czernek *et al.*, 2023). Another significant pair ($A \cdots D$) between inversion-related molecules ($-x + 1$, $-y + 1$, $-z + 1$) yields $E_{\text{tot}} = -31.1 \text{ kJ mol}^{-1}$ ($R = 7.15 \text{ \AA}$), dominated by dispersion ($E_{\text{dis}} = -56.8 \text{ kJ mol}^{-1}$) and arising from $\text{C}=\text{O} \cdots \pi$ contacts.

Two further moderate in strength interactions are the pair $A \cdots E$ [$E_{\text{tot}} = -18.3 \text{ kJ mol}^{-1}$; $R = 12.71 \text{ \AA}$], attributed to slipped antiparallel stacking generating $\text{Br} \cdots \pi$ contacts, and pair $A \cdots F$ [$E_{\text{tot}} = -13.5 \text{ kJ mol}^{-1}$; $R = 7.15 \text{ \AA}$], associated with $\text{Br} \cdots \text{H}$ contacts. Both are governed primarily by dispersion, but in the latter case the E_{ele} component is also perceptible, being the third electrostatic contributor among the entire hierarchy of interaction energies.

6. Database survey

A search of the Cambridge Structural Database (CSD, July 2025 release; Groom *et al.*, 2016) for cinnamic anhydride derivatives revealed no closely related crystal structures, indicating an absence of this subclass in the structural record. In contrast, numerous entries exist for cinnamic acid precursors, particularly for *trans*-cinnamic acid itself (for the most recent redetermination, see Howard *et al.*, 2009), as well as for *para*-halogenated analogues such as 4-fluorocinnamic (Jenkins *et al.*, 2006), 4-chlorocinnamic (Hsieh *et al.*, 2005), and 4-bromocinnamic (Schmidt, 1964) acids. In addition, a recent study provided 21 examples of 4-halophenyl 4-halocinnamate esters (Liu *et al.*, 2025). The most comparable methyl 4-bromocinnamate (Refcode: MEBCIN; Leiserowitz & Schmidt, 1965) is mentioned above.

The absence of structurally characterized cinnamic anhydrides in the CSD may reflect intrinsic crystallization challenges associated with this subclass, including increased conformational flexibility (Mary *et al.*, 2014*a,b*), which can hinder efficient packing, and a higher propensity for hydrolysis under ambient conditions, both of which may favor amorphous or poorly crystalline forms (Raja *et al.*, 2017).

In this context, the present study reports the first single-crystal X-ray diffraction analysis of a *p*-halogenated cinnamic anhydride, providing detailed insights into its conformation, molecular symmetry, and supramolecular organization. This work establishes a useful reference point for future studies on the solid-state behavior and reactivity of cinnamic anhydride derivatives.

7. Synthesis and crystallization

4-Bromocinnamic acid was obtained from a commercial supplier and used without further purification. The title compound (I) was synthesized via a one-pot condensation reaction using *N,N'*-dicyclohexylcarbodiimide (DCC) as coupling agent (Albert *et al.*, 2017). 4-Bromocinnamic acid (200 mg, 0.881 mmol) was dissolved in chloroform (8 ml) and DCC (0.881 mmol) was added. The reaction mixture was refluxed for 24 h, during which time a white precipitate of dicyclohexylurea formed, which was removed by filtration after cooling. The filtrate was concentrated under reduced

Table 3

Experimental details.

Crystal data	
Chemical formula	$\text{C}_{18}\text{H}_{12}\text{Br}_2\text{O}_3$
M_r	436.10
Crystal system, space group	Monoclinic, $C2/c$
Temperature (K)	150
a, b, c (Å)	20.6900 (5), 5.7029 (1), 13.5534 (3)
β (°)	93.392 (1)
V (Å ³)	1596.40 (6)
Z	4
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	5.09
Crystal size (mm)	$0.38 \times 0.22 \times 0.21$
Data collection	
Diffractometer	Bruker D8 Venture κ -geometry diffractometer 208039-01
Absorption correction	Multi-scan (SADABS; Krause <i>et al.</i> , 2015)
$T_{\text{min}}, T_{\text{max}}$	0.505, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	20264, 1784, 1702
R_{int}	0.028
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.017, 0.045, 1.09
No. of reflections	1784
No. of parameters	129
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.27, -0.33

Computer programs: APEX5 and SAINT (Bruker, 2018), SHELXS97 (Sheldrick, 2008), SHELXL2019/3 (Sheldrick, 2015) and DIAMOND (Brandenburg, 1999).

pressure and the crude product was washed with cold methanol and dried affording 4-bromocinnamic anhydride as a colorless solid (185 mg, 95%). M.p. = 470.5–470.8 K. FT-IR (ATR, cm⁻¹): 3272 (Ar CH), 2924 and 2856 (vinyl CH), 1707 (C=O), 1644 (vinyl C=C), 1599 (Ar C=C), 1485 and 1447 (ring skeletal vibrations), 1367 (C–O–C), 1226 (C–O), 1069 (C–H bending), 986 (vinyl CH wag), 813 (aromatic CH bending), 618 (C–Br), 515 (C–Br stretching/ring deformation) and 488 (C–O–C bending and ring torsion).

Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a dilute solution of the compound in the mixed solvents of ethyl acetate/dichloromethane (1:10, v/v) stored at 278 K. Colorless crystals formed over the period of 7 d.

8. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All hydrogen atoms were located and then freely refined with isotropic displacement parameters, which results in C–H = 0.94 (2)–0.971 (19) Å. One outlier (200) was omitted in the last cycles of refinement.

Acknowledgements

The authors gratefully acknowledge the support received from MSc. Simón Hernández-Ortega of the Laboratorio de Difracción de Rayos X, Instituto de Química, Universidad Nacional Autónoma de México.

Funding information

The authors gratefully acknowledge the financial support provided by the host institutions, *i.e.*, Universidad del Quindío (grant Nos. 1183 and 100016837) and Universidad Pedagógica y Tecnológica de Colombia (grant No. SGI3910).

References

- Albert, J. S., Hamilton, A. D., Hart, A. C., Feng, X., Lin, L. & Wang, Z. (2017). *1,3-Dicyclohexylcarbodiimide*. In *Encyclopedia of Reagents for Organic Synthesis* edited by L. A. Paquette. New York: John Wiley & Sons Ltd.
- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
- Brandenburg, K. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Bruker (2018). *APEX5* and *SAINT*. Bruker AXS, Madison, Wisconsin, USA.
- Czernek, J., Brus, J., Czerneková, V. & Kobera, L. (2023). *Molecules* **28**, 4478.
- Giessel, J. M., Serbian, I., Loesche, A. & Csük, R. (2019). *Bioorg. Chem.* **90**, 103058.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Howard, J. A. K., Mahon, M. F., Raithby, P. R. & Sparkes, H. A. (2009). *Acta Cryst.* **B65**, 230–237.
- Hsieh *et al.* (2005). Please supply missing reference.
- Jenkins, S. L., Almond, M. J., Atkinson, S. D. M., Drew, M. G. B., Hollins, P., Mortimore, J. L. & Tobin, M. J. (2006). *J. Mol. Struct.* **786**, 220–226.
- Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.
- Leiserowitz, L. & Schmidt, G. M. J. (1965). *Acta Cryst.* **18**, 1058–1067.
- Liao, Y.-S. & Liang, C. (2018). *Org. Biomol. Chem.* **16**, 1871–1881.
- Lin, W., Zhao, Q., Li, Y., Pan, M., Yang, C., Yang, G. & Li, X. (2021). *Chem. Sci.* **13**, 141–148.
- Liu, J., Zhang, S., Yang, X., Chen, C., Ye, K., Shen, Y., Sun, J. & Lu, R. (2025). *Cryst. Growth Des.* **25**, 3298–3316.
- Mackenzie, C. F., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). *IUCrJ* **4**, 575–587.
- Mary, Y. S., Raju, K., Panicker, C. Y., Al-Saadi, A. A., Thiemann, T. & Alsenoy, C. V. (2014a). *Spectrochim. Acta A*, **128**, 638–646.
- Mary, Y. S., Raju, K., Panicker, C. Y., Al-Saadi, A. A. & Thiemann, T. (2014b). *Spectrochim. Acta A Mol. Biomol. Spectrosc.* **131**, 471–483.
- McKinnon, J. J., Jayatilaka, D. & Spackman, M. A. (2007). *Chem. Commun.*, pp. 3814–3816.
- Pálinkó, I. (1999). *Acta Cryst.* **B55**, 216–220.
- Raja, G. C. E., Son, Y., Kim, M., Lee, S. & Oh, J. (2017). *Synth. Commun.* **47**, 2449–2455.
- Robinson, E. R. T., Fallan, C., Simal, C., Slawin, A. M. Z. & Smith, A. D. (2013). *Chem. Sci.* **4**, 2193–2200.
- Schmidt, G. (1964). *J. Chem. Soc.* pp. 2014–2021.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst.* **A71**, 3–8.
- Spackman, M. A. & Jayatilaka, D. (2008). *CrystEngComm* **11**, 19–32.
- Spackman, M. A. & McKinnon, J. J. (2002). *CrystEngComm* **4**, 378–392.
- Turner, M. J., Thomas, S. P., Shi, M., Jayatilaka, D. & Spackman, M. A. (2015). *Chem. Commun.* **51**, 3735–3738.
- Yang, G., Zhou, H., Chen, J., Lyu, G., Xia, Y. & Lucia, L. A. (2017). *Int. J. Mol. Sci.* **18**, 2502.
- Yates, J. L. R. & Sparkes, H. A. (2013). *CrystEngComm* **15**, 3547–3553.
- Zhao, H., Xu, X., Luo, Z., Cao, L., Li, B., Li, H., Xu, L., Fan, Q. & Walsh, P. J. (2019). *Chem. Sci.* **10**, 10089–10096.

supporting information

Acta Cryst. (2025). E81, 932-937 [https://doi.org/10.1107/S2056989025007789]

Crystal structure of 4-bromocinnamic anhydride

Yuliana Ramos Cotrina, Jhesua Valencia, Ronan Le Lagadec, Fernando Cuenú-Cabezas and Jovanny A. Gómez Castaño

Computing details

(*E*)-3-(4-Bromophenyl)prop-2-enoyl (*E*)-3-(4-bromophenyl)prop-2-enoate

Crystal data

$C_{18}H_{12}Br_2O_3$

$M_r = 436.10$

Monoclinic, $C2/c$

$a = 20.6900$ (5) Å

$b = 5.7029$ (1) Å

$c = 13.5534$ (3) Å

$\beta = 93.392$ (1)°

$V = 1596.40$ (6) Å³

$Z = 4$

$F(000) = 856$

$D_x = 1.814$ Mg m⁻³

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

Cell parameters from 9867 reflections

$\theta = 3.0$ – 27.5°

$\mu = 5.09$ mm⁻¹

$T = 150$ K

Prism, colourless

$0.38 \times 0.22 \times 0.21$ mm

Data collection

Bruker D8 Venture κ -geometry

diffractometer 208039-01

Radiation source: micro-focus X-ray source

Detector resolution: 52.0833 pixels mm⁻¹

ω -scans

Absorption correction: multi-scan

(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.505$, $T_{\max} = 0.746$

20264 measured reflections

1784 independent reflections

1702 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.0^\circ$

$h = -26 \rightarrow 26$

$k = -7 \rightarrow 7$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.017$

$wR(F^2) = 0.045$

$S = 1.09$

1784 reflections

129 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0202P)^2 + 1.5368P]$

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

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.27$ e Å⁻³

$\Delta\rho_{\min} = -0.33$ e Å⁻³

Special details

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.72429 (2)	1.05887 (3)	0.32640 (2)	0.02833 (7)
O2	0.47808 (6)	−0.05003 (19)	0.64920 (8)	0.0292 (2)
O1	0.500000	0.2596 (2)	0.750000	0.0256 (3)
C1	0.50364 (7)	0.1372 (3)	0.66208 (10)	0.0214 (3)
C2	0.53902 (7)	0.2660 (2)	0.58928 (10)	0.0219 (3)
C3	0.57054 (7)	0.4669 (3)	0.60876 (10)	0.0217 (3)
C4	0.60848 (7)	0.5984 (2)	0.53989 (10)	0.0204 (3)
C5	0.61593 (7)	0.5260 (3)	0.44231 (11)	0.0230 (3)
C6	0.65139 (7)	0.6601 (3)	0.37961 (11)	0.0240 (3)
C7	0.67923 (7)	0.8681 (3)	0.41427 (10)	0.0215 (3)
C8	0.67399 (8)	0.9422 (3)	0.51050 (12)	0.0250 (3)
C9	0.63846 (7)	0.8065 (3)	0.57250 (10)	0.0236 (3)
H2	0.5376 (9)	0.194 (3)	0.5257 (14)	0.030 (4)*
H3	0.5691 (9)	0.534 (3)	0.6727 (15)	0.027 (5)*
H5	0.5967 (10)	0.385 (4)	0.4184 (14)	0.034 (5)*
H6	0.6569 (10)	0.609 (4)	0.3128 (15)	0.033 (5)*
H8	0.6933 (10)	1.082 (3)	0.5347 (15)	0.031 (5)*
H9	0.6335 (9)	0.858 (3)	0.6399 (14)	0.028 (4)*

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02947 (10)	0.02528 (10)	0.03090 (10)	−0.00097 (6)	0.00744 (6)	0.00371 (6)
O2	0.0363 (6)	0.0248 (6)	0.0267 (5)	−0.0059 (4)	0.0044 (5)	−0.0049 (4)
O1	0.0436 (9)	0.0166 (7)	0.0171 (6)	0.000	0.0066 (6)	0.000
C1	0.0242 (7)	0.0212 (7)	0.0188 (6)	0.0041 (6)	0.0017 (5)	−0.0016 (5)
C2	0.0254 (7)	0.0222 (7)	0.0182 (6)	0.0039 (5)	0.0024 (5)	−0.0014 (5)
C3	0.0263 (7)	0.0217 (7)	0.0171 (6)	0.0050 (5)	0.0010 (5)	0.0006 (5)
C4	0.0223 (6)	0.0187 (6)	0.0201 (6)	0.0044 (5)	−0.0001 (5)	0.0008 (5)
C5	0.0267 (7)	0.0199 (7)	0.0226 (7)	0.0004 (6)	0.0018 (5)	−0.0031 (5)
C6	0.0262 (7)	0.0238 (7)	0.0224 (7)	0.0026 (6)	0.0039 (5)	−0.0028 (6)
C7	0.0194 (6)	0.0201 (6)	0.0251 (7)	0.0039 (5)	0.0022 (5)	0.0039 (5)
C8	0.0271 (7)	0.0199 (7)	0.0277 (7)	0.0003 (6)	−0.0024 (6)	−0.0017 (6)
C9	0.0285 (7)	0.0220 (7)	0.0200 (7)	0.0029 (6)	−0.0011 (5)	−0.0023 (5)

Geometric parameters ($\text{\AA}$, $^\circ$)

Br1—C7	1.8981 (14)	C4—C5	1.403 (2)
O2—C1	1.2000 (19)	C5—C6	1.385 (2)

O1—C1 ⁱ	1.3869 (15)	C5—H5	0.95 (2)
O1—C1	1.3869 (15)	C6—C7	1.388 (2)
C1—C2	1.461 (2)	C6—H6	0.96 (2)
C2—C3	1.337 (2)	C7—C8	1.381 (2)
C2—H2	0.95 (2)	C8—C9	1.385 (2)
C3—C4	1.462 (2)	C8—H8	0.94 (2)
C3—H3	0.95 (2)	C9—H9	0.971 (19)
C4—C9	1.399 (2)		
C1 ⁱ —O1—C1	119.57 (16)	C6—C5—H5	119.1 (12)
O2—C1—O1	121.80 (13)	C4—C5—H5	120.4 (12)
O2—C1—C2	125.68 (13)	C5—C6—C7	119.23 (13)
O1—C1—C2	112.48 (12)	C5—C6—H6	120.5 (12)
C3—C2—C1	123.73 (13)	C7—C6—H6	120.2 (12)
C3—C2—H2	122.5 (11)	C8—C7—C6	121.79 (13)
C1—C2—H2	113.8 (11)	C8—C7—Br1	119.07 (11)
C2—C3—C4	125.98 (13)	C6—C7—Br1	119.14 (11)
C2—C3—H3	118.9 (11)	C7—C8—C9	118.43 (14)
C4—C3—H3	115.1 (11)	C7—C8—H8	122.2 (12)
C9—C4—C5	118.43 (13)	C9—C8—H8	119.4 (12)
C9—C4—C3	118.58 (13)	C8—C9—C4	121.58 (13)
C5—C4—C3	122.99 (13)	C8—C9—H9	119.5 (11)
C6—C5—C4	120.51 (14)	C4—C9—H9	118.9 (11)
C1 ⁱ —O1—C1—O2	31.70 (11)	C4—C5—C6—C7	−0.4 (2)
C1 ⁱ —O1—C1—C2	−150.51 (13)	C5—C6—C7—C8	1.7 (2)
O2—C1—C2—C3	−175.29 (15)	C5—C6—C7—Br1	−177.02 (11)
O1—C1—C2—C3	7.02 (19)	C6—C7—C8—C9	−1.7 (2)
C1—C2—C3—C4	177.73 (13)	Br1—C7—C8—C9	177.08 (11)
C2—C3—C4—C9	179.97 (14)	C7—C8—C9—C4	0.3 (2)
C2—C3—C4—C5	0.3 (2)	C5—C4—C9—C8	1.0 (2)
C9—C4—C5—C6	−1.0 (2)	C3—C4—C9—C8	−178.64 (13)
C3—C4—C5—C6	178.68 (13)		

Symmetry code: (i) $-x+1, y, -z+3/2$.

Hydrogen-bond geometry ($\text{\AA}$, $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2 $\cdots$ O2 ⁱⁱ	0.95 (2)	2.51 (2)	3.4574 (18)	171.6 (16)
C5—H5 $\cdots$ O2 ⁱⁱ	0.95 (2)	2.59 (2)	3.5219 (18)	168.0 (17)
C6—H6 $\cdots$ Br1 ⁱⁱⁱ	0.96 (2)	3.20 (2)	3.9492 (14)	136.0 (15)
C8—H8 $\cdots$ Br1 ^{iv}	0.94 (2)	3.20 (2)	4.1064 (15)	161.0 (15)
C9—H9 $\cdots$ Br1 ^v	0.971 (19)	3.095 (18)	3.8551 (14)	136.3 (13)

Symmetry codes: (ii) $-x+1, -y, -z+1$; (iii) $-x+3/2, y-1/2, -z+1/2$; (iv) $-x+3/2, -y+5/2, -z+1$; (v) $x, -y+2, z+1/2$.