

Cyclohexane plastic phase I: single-crystal diffraction images and new structural model

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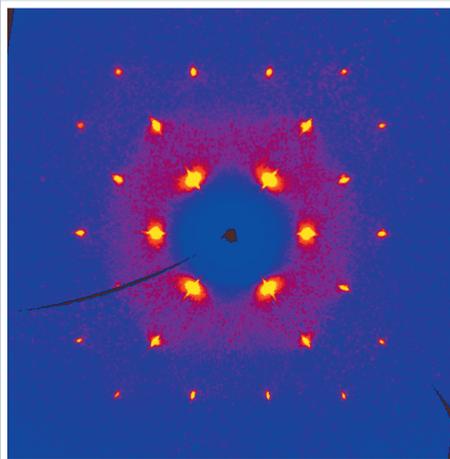
Keywords: cyclohexane; plastic crystals; X-ray diffraction; diffraction image; disorder.

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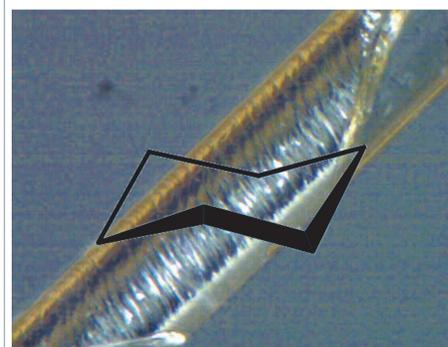
Structural data: full structural data are available from iucrdata.iucr.org

The plastic phase of cyclohexane (polymorph I) was studied by Kahn and co-workers, without achieving a satisfactory determination of the atomic coordinates [Kahn *et al.* (1973). *Acta Cryst. B***29**, 131–138]. The positions of the C atoms cannot be determined directly as a consequence of the disorder in a high-symmetry space group, an inherent feature of plastic materials. Given this situation, the building of a polyhedron describing the disorder was the main tool for determining the molecular structure in the present work. Based on the shape of reflections {111}, {200} and {113} in space group $Fm\bar{3}m$, we assumed that cyclohexane is disordered through the action of rotation group 432. The polyhedral cluster of disordered molecules is then a rhombic dodecahedron centred on the nodes of an fcc Bravais lattice. The vertices of this polyhedron are the positions of C atoms for the cyclohexane molecule, which is disordered over 24 positions. With such a model, the asymmetric unit is reduced to two C atoms placed on special positions, and an acceptable fit between the observed and calculated structure factors is obtained.

Raw data



Chemical scheme



Stoe Stadivari data files and CBF files: <https://doi.org/10.5281/zenodo.7154725>

Metadata imgCIF file for cyclohexane_crystal1: <https://doi.org/10.1107/S2414314623001141/iq4001img1.cif>

Metadata imgCIF file for cyclohexane_crystal2: <https://doi.org/10.1107/S2414314623001141/iq4001img2.cif>

Introduction

The concept of an organic ‘plastic crystal’ was first stated by J. Timmermans in 1938, although the name was coined ten years later by A. Michils, due to the mechanical softness of these materials. They share many physicochemical features with liquid crys-

tals, and were indeed first described as a new mesomorphic state of matter. Thermodynamically, they are characterized by a very low entropy of fusion, $\Delta S_m < 5$ eu (1 eu = 4.185 J mol⁻¹ K⁻¹), which was interpreted as the signature that a quasi-isotropic state, similar to that of liquids, is set just below their melting-point temperature (Timmermans, 1961). Mechanically, these materials behave as plastic metals and can be extruded at quite low pressures (Michils effect; Michils, 1948). Most often, the molecules concerned belong to high-symmetry point groups, and present a more or less globular shape while being orientationally disordered around their rotation axis; they have a marked propensity for polymorphism, with the form close to the melting point crystallizing in a high-symmetry space group, usually in the cubic crystal system, with a highly disordered crystal structure.

Cyclohexane, C₆H₁₂, is an emblematic example of such crystals. The ground state of the molecule is the rigid-chair conformer, belonging to $\bar{3}m$ (D_{3d}) point group. The high-temperature phase I, in space group $Fm\bar{3}m$, undergoes an isothermal transition at 186 K to the low-temperature ordered phase II, in space group $C2/c$ (Kahn *et al.*, 1973). The entropy of fusion, $\Delta S_m = 2.29$ eu, is much lower than that measured for the II→I transition, $\Delta S_{II\rightarrow I} = 8.66$ eu (Ruehrwein & Huffman, 1943).

Early literature regarding the crystallographic characterization of cyclohexane phase I (Hassel & Sommerfeldt, 1938; Oda, 1948; Renaud & Fourme, 1966; Kahn *et al.*, 1973), systematically complains about technical hurdles related to the very nature of plastic crystals: (i) a very rapid fall-off of diffraction intensity with increasing Bragg angle; (ii) a scattering background blackening the photographic plates and masking weak reflections; (iii) the extreme difficulty of obtaining a reliable set of atomic coordinates, as a direct consequence of the previously mentioned issues. Indeed, only one article explicitly suggests a structural model based on atomic coordinates (Kahn *et al.*, 1973), which is discussed further below.

During our work on the structure prediction and crystallographic characterization of cycloalkanes that are liquids at room temperature (Camargo, 2018), we were able to obtain diffraction frames for the plastic phase I of cyclohexane. A careful examination of the reciprocal space rebuilt from these raw data offers greater insight into how molecules behave in the plastic phase, and allowed us to propose a new simple model explaining how molecules are disordered about the nodes of the fcc Bravais lattice.

Crystallization and data collection

Anhydrous cyclohexane (reference 227048, Sigma-Aldrich, 99.5%) has a melting point close to 279 K. The end of a 0.4 mm diameter glass Lindemann capillary tube was filled with liquid cyclohexane and the head of the capillary sealed with wax, while avoiding any contamination of cyclohexane. The capillary was mounted on a standard goniometer head, and cyclohexane was crystallized *in situ*, on a Stoe Stadivari diffractometer equipped with an Oxford Cryosystems Cobra

cooling device. No head spinning was applied during crystallization, and a key condition was to keep the capillary horizontally ($\chi = -90^\circ$), in order to have the N₂ flow approximately normal to the capillary. In a first step, successive cycles of cooling/heating ramps with different rates were applied to obtain a powdered sample: from room temperature to 260 K at 360 K h⁻¹, and then to 270 K at 200 K h⁻¹. These microcrystals were then carefully merged by heating the sample to 273 K (60 K h⁻¹) and then to 274 K (2 K h⁻¹). Once a single crystal is stabilized in the capillary, the sample can be cooled to 260 K at 10 K h⁻¹ and then to 250 K at 20 K h⁻¹. We found that this methodology affords large and good-quality single crystals in a reproducible manner.

Diffraction intensities for one crystal were collected at 255 K with Ag $K\alpha$ radiation (AXO microfocus source equipped with multilayer ASTIX-f optics) and a PILATUS 100 K detector (487 × 195 pixels), accumulating 1139 frames over 19 h, each one being collected over 60 s, with a scan range of 1° in ω . Another crystal was collected at 245 K over 91 h. For this experiment, a very long exposure time of 1800 s per frame was used, with a scan range of 2° in ω . A set of 183 frames was collected for this crystal. Both data sets afford virtually the same structure refinement. The refinement reported in this paper is based on the first data set. The second data set is used herein for Fig. 3 only.

Data processing

Reciprocal space for each crystal was built using all collected frames, with the dedicated *X-AREA* tool (Stoe & Cie, 2019). A cubic 3D array centred on the origin of reciprocal space, with boundaries at -0.75 and $+0.75$ Å⁻¹ ($2\theta_{\max} = 42.7^\circ$) and a pixel resolution of 0.003 Å⁻¹ was computed. Each detector pixel was divided into 10 subpixels in the plane of the detector, and into 20 subpixels in the direction normal to that plane. The resulting 3D arrays contain approximately 125×10^6 voxels. Images in Figs. 1–3 are plotted using a conventional blue/yellow heat map.

Structure factors were obtained by integrating the 1139 frames collected on the first crystal. Elliptical integration masks are used, with the smallest diameter given by $W = A + B \tan \theta$ and the largest diameter calculated as $W/\cos 2\theta + (\Delta\lambda/\lambda) \tan \theta$, with $A = 5$ and $B = -8$. A rather large mosaic spread parameter was applied (ems = 0.048 rad), to take into account the plastic nature of the crystal. Finally, the background area was systematically limited to one pixel around the peak area. Intensities were scaled in the $m\bar{3}m$ Laue class in a standard way.

Data description

The ($hk0$) layer built with 1139 frames (Fig. 1) clearly shows that a single crystal was grown. Bragg peaks are well defined, although the resolution is, as expected, very low: the last observed reflections in the full pattern are (333) and (511), corresponding to a resolution of 1.67 Å. That resolution is not improved if frames are collected over 1800 s instead of 60 s.

Table 1
Experimental details.

Raw data				
DOI		https://doi.org/10.5281/zenodo.7154725		
Data archive		Zenodo		
Data format		CBF		
Data collection				
Diffraction		Stoe Stadivari		
Temperature (K)		255		
Radiation type		Ag $K\alpha$		
Detector type		Dectris Pilatus 100 K R		
Wavelength (Å)		0.56083		
Beam centre (mm)		41.882, 16.7		
Detector axis		-Z		
Detector distance (mm)		40.0		
Pixel size (mm)		0.172 × 0.172		
No. of pixels		195 × 487		
No. of scans		17		
Exposure time per frame (s)		60.0		
Swing angle (°)		Scan axis	Start angle, increment per frame (°)	Scan range (°)
-23.4		$\omega, X (\chi = -55.183^\circ, \varphi = -55.0^\circ)$	142.261, 1.0	47.0
-23.4		$\omega, X (\chi = -30.183^\circ, \varphi = -5.0^\circ)$	142.261, 1.0	47.0
23.4		$\omega, X (\chi = -35.183^\circ, \varphi = 150.0^\circ)$	-40.679, 1.0	77.0
23.4		$\omega, X (\chi = -20.183^\circ, \varphi = -85.0^\circ)$	-40.679, 1.0	77.0
23.4		$\omega, X (\chi = -30.183^\circ, \varphi = -105.0^\circ)$	-40.679, 1.0	77.0
23.4		$\omega, X (\chi = -35.183^\circ, \varphi = -35.0^\circ)$	-40.679, 1.0	77.0
23.4		$\omega, X (\chi = -35.183^\circ, \varphi = 75.0^\circ)$	-40.679, 1.0	77.0
23.4		$\omega, X (\chi = -50.183^\circ, \varphi = -160.0^\circ)$	-40.679, 1.0	77.0
23.4		$\omega, X (\chi = -20.183^\circ, \varphi = 25.0^\circ)$	-40.679, 1.0	77.0
23.4		$\omega, X (\chi = -45.183^\circ, \varphi = -55.0^\circ)$	-40.679, 1.0	77.0
23.4		$\omega, X (\chi = -50.183^\circ, \varphi = 15.0^\circ)$	-40.679, 1.0	77.0
23.4		$\omega, X (\chi = -50.183^\circ, \varphi = 125.0^\circ)$	-40.679, 1.0	77.0
23.4		$\omega, X (\chi = -25.183^\circ, \varphi = -110.0^\circ)$	-40.679, 1.0	77.0
23.4		$\omega, X (\chi = -55.183^\circ, \varphi = -170.0^\circ)$	-40.679, 1.0	67.0
23.4		$\omega, X (\chi = -40.183^\circ, \varphi = 140.0^\circ)$	-40.679, 1.0	67.0
23.4		$\omega, X (\chi = -45.183^\circ, \varphi = -150.0^\circ)$	-25.679, 1.0	32.0
23.4		$\omega, X (\chi = -20.183^\circ, \varphi = -100.0^\circ)$	-25.679, 1.0	32.0
Crystal data				
Chemical formula		C_6H_{12}		
M_r		84.16		
Crystal system, space group		Cubic, $Fm\bar{3}m$		
a (Å)		8.712 (4)		
V (Å ³)		661.1 (9)		
Z		4		
μ (mm ⁻¹)		0.03		
Crystal size (mm)		0.40 × 0.30 × 0.30		
Data processing				
Absorption correction		Multi-scan (<i>X-AREA</i> ; Stoe & Cie, 2019)		
T_{\min}, T_{\max}		0.558, 1.000		
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections		1690, 31, 10		
R_{int}		0.018		
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)		0.487		
Refinement				
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$		0.080, 0.190, 1.10		
No. of reflections		31		
No. of parameters		5		
No. of restraints		3		
H-atom treatment		H-atom parameters constrained		
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)		0.07, -0.16		

Computer programs: *X-AREA* (Stoe & Cie, 2019), *SHELXL2018/3* (Sheldrick, 2015) and *Mercury* (Macrae *et al.*, 2020).

$\chi = 2$ (Fig. 4). This polyhedron is centrosymmetric, and its centre coincides with the crystallographic inversion centre. As this polyhedron belongs to the family of edge-transitive polyhedra, all C—C bonds are equivalent and have the same bond length, as expected for cyclohexane. The rhombic faces, with configuration v3.4.3.4, display obtuse angles of

$\arccos(-1/3) = \pm 109.47^\circ$, which accommodate sp^3 -hybridized C atoms. The dihedral angle between edge-sharing rhombus is 120° , affording the expected C—C—C torsion angles of $\pm 60^\circ$ in cyclohexane.

Most importantly, the rhombic dodecahedron has full octahedral symmetry ($m\bar{3}m$ or *432), and its rotation group is

Table 2

Refined structure of plastic cyclohexane at 255 K. Refined parameters are x , x' , $U(\text{C1})$ and $U(\text{C2})$.

Refined parameters	
Position of C1: (x , 0, 0), sof, U_{iso}	$x = 0.2030(18)$, 3/56, $U(\text{C1}) = 0.41(3) \text{ \AA}^2$
Position of C2: (x' , x' , x'), sof, U_{iso}	$x' = 0.1019(10)$, 1/14, $U(\text{C2}) = 0.35(3) \text{ \AA}^2$
Cyclohexane geometry	
C—C bond length	1.534 (8) \AA
C—C—C bond angles	109.9 (17), 109.2 (8)°
C—C—C—C torsion angles	−59.7 (10), 59.7 (10)°

the chiral octahedral group 432. The chair conformation of cyclohexane, with symmetry $\bar{3}m$, is thus compatible with the rhombic dodecahedron, and the full polyhedron is indeed generated by rotation of one chair about the elements of the rotation group 432, as reflected in the shape of the Bragg reflections, as discussed above. The molecule is then disordered over 24 positions (order of the rotation group). Symmetry-related molecules in this polyhedral cluster are depicted in Fig. 4.

Once the polyhedron describing the disorder in the plastic phase has been laid down, the structure refinement is straightforward. A single geometric parameter should actually be refined, that is the bond length $\text{C1—C2} = d$. Since both atoms lie on special positions, only two positional parameters are used, x and x' . Using the structure factors extracted as described in the previous section, we refined an isotropic

model with *SHELXL* (Sheldrick, 2015; refinement against F^2 , no extinction parameter refined), including three restraints for the geometry of the polyhedron: $d = 1.54(1) \text{ \AA}$, and a couple of restraints for 1,3-distances: $\text{C1} \cdots \text{C1}' = d(2\sqrt{2/3})$ and $\text{C2} \cdots \text{C2}' = d(2\sqrt{2/3})$, with standard deviations of 0.03 \AA , and with primed atoms generated by suitable symmetry operations. Site occupancy factors (sof) are calculated considering the Wyckoff positions and assuming that each of the 14 vertices in the polyhedron has the same probability to be occupied: $\text{sof}(\text{C1}) = (24/192) \times (6/14) = 3/56$ and $\text{sof}(\text{C2}) = (32/192) \times (6/14) = 1/14$. Finally, all H atoms were added in idealized positions, corresponding to special positions $96k$ (H1 bonded to C1), and $96k$ and $32f$ (H2A and H2B bonded to C2), with $\text{C—H} = 0.95 \text{ \AA}$, and with calculated displacement parameters $U_{\text{iso}}(\text{H}) = 2.8U_{\text{iso}}(\text{carrier C})$.

The structure is then refined (Table 1) using five parameters and 31 independent reflections, of which ten have $F_o > 4\sigma(F_o)$, converging towards the expected geometry (Table 2). Notably, the refined C1—C2 bond length of $1.534(8) \text{ \AA}$ is identical to that determined by electron diffraction, $1.535(2) \text{ \AA}$ (Ewbank *et al.*, 1976). Displacement parameters are very high, reflecting the motions of C atoms bouncing from vertex to vertex in the polyhedral cluster. Actually, Figs. 1–3 reflect accurately the idea of Timmermans about plastic crystals: they are solids behaving like liquids over short distances (one polyhedron). From the crystallographic point of view, plastic cyclohexane can be seen as a liquid with long-range order, affording a diffraction pattern. The dynamic disorder being identical for every node in the lattice, the crystal structure emulates a close-packed arrangement (Fig. 5), in which the atomic sites have very low occupancies (see Table 2). As a consequence, the density is also very low, 0.85 g cm^{-3} . The non-plastic phase II of cyclohexane has a more regular density of 1 g cm^{-3} .

Discussion and conclusions

Strangely enough, Kahn *et al.* were unable to move towards the model we propose in Table 2, probably because they did not realize that C atoms could lie on special positions. Instead, they used an asymmetric unit including three C atoms close to the origin, all in *general* positions. With such a model, the 144-vertex polyhedron describing the disorder is hugely complex, and individual cyclohexane molecules are hardly discernible. Actually, their polyhedron has a shape close to that of a sphere, which has Euler characteristic $\chi = 2$, like any (convex) polyhedron whose boundary is topologically equivalent to a sphere. It is thus not surprising that they could obtain a satisfactory agreement between observed and calculated structure factors, although their structural model is far from satisfactory.

It is worth noting that the notion of ‘refinement’ for such plastic structures is of little sense, especially if least-squares methods are involved, since the data-to-parameter ratio rapidly drops to too low values. Even the identification of a suitable asymmetric unit cannot rely on mainstream approaches like direct methods, since atomic resolution is not

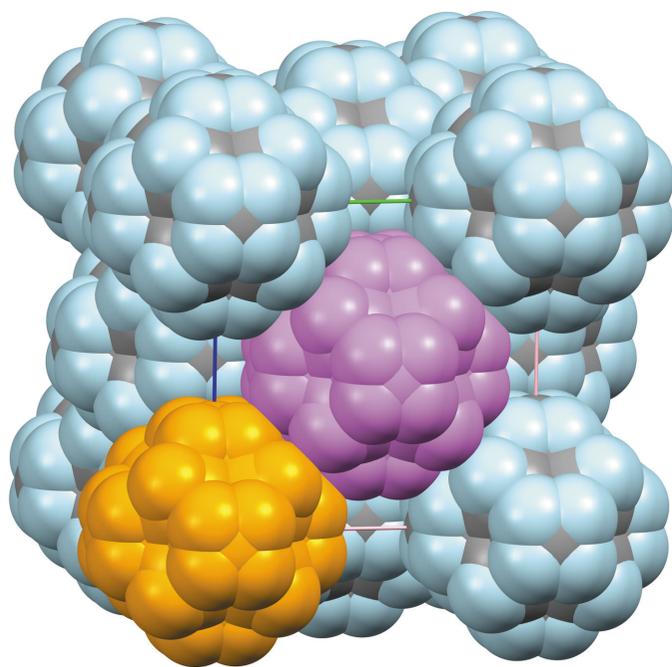


Figure 5

Packing structure of cyclohexane at 255 K, in a space-filling representation. All disordered sites for C (grey) and H (blue) atoms in one unit cell are represented with their van der Waals radii (Macrae *et al.*, 2020). Two neighbouring clusters of disordered molecules in the fcc lattice are shown in orange and magenta, with the purpose of emphasizing the contact between the two clusters. The interface separating the clusters is a ‘diagonal’ mirror plane m in space group $Fm\bar{3}m$.

achievable. Instead, a careful examination of data in reciprocal space, in particular the shape of the Bragg peaks, can be helpful. In 1973, this perspective was not considered by Kahn *et al.* In contrast, the 1948 article of Tutomu Oda, of limited impact because written in Japanese, is noteworthy. The abstract mentions: ‘*Besides the Bragg reflections, we observed remarkable diffuse scattering of considerable intensity, similar to that shown by cyclohexanol. Namely, there appear on the Laue and oscillation photographs a number of so-called diffuse spots and apparently circular diffuse haloes, which resemble to the liquid diffraction haloes*’. Nowadays, computer simulations allow the interpretation of the diffuse scattering observed in many materials. This may be achieved either in reciprocal space by considering the material as a modulated phase, or with a correlation method in direct space, using short-range chemical and atomic displacement pair-correlation parameters (Rosenkranz & Osborn, 2004; Welberry, 2022). In the case of molecular crystals, Monte Carlo and reverse Monte Carlo simulations are also a very promising approach, since they are applicable to disorder of any complexity (Welberry, 2022). However, only a few such simulations have been carried out for plastic crystals to date (for example, for α -CBr₄; Folmer *et al.*, 2008), and the molecular dynamics associated with the disorder in these materials is not fully understood.

We also extended this study to cycloheptane phase I and cyclooctane phase I (both in space group $Pm\bar{3}n$). Preliminary results can be found in the Master’s thesis of the last author (Camargo, 2018; available online). We also plan to collect data at temperatures as close as possible to the melting points of these materials, and to use Cu $K\alpha$ radiation for collecting frames.

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full crystallographic data

IUCrData (2023). **8**, x230114 [https://doi.org/10.1107/S2414314623001141]

Cyclohexane plastic phase I: single-crystal diffraction images and new structural model

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cyclohexane

Crystal data

C₆H₁₂

$M_r = 84.16$

Cubic, $Fm\bar{3}m$

$a = 8.712$ (4) Å

$V = 661.1$ (9) Å³

$Z = 4$

$F(000) = 192$

$D_x = 0.845$ Mg m⁻³

Melting point: 279 K

Ag $K\alpha$ radiation, $\lambda = 0.56083$ Å

Cell parameters from 664 reflections

$\theta = 3.2$ – 9.6°

$\mu = 0.03$ mm⁻¹

$T = 255$ K

Rod, colourless

$0.40 \times 0.30 \times 0.30$ mm

Data collection

Stoe Stadivari
diffractometer

Radiation source: Sealed X-ray tube, Axo Astix-
f Microfocus source

Graded multilayer mirror monochromator

Detector resolution: 5.81 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

X-AREA 1.88 (Stoe & Cie, 2019)

$T_{\min} = 0.558$, $T_{\max} = 1.000$

1690 measured reflections

31 independent reflections

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

$R_{\text{int}} = 0.018$

$\theta_{\max} = 15.9^\circ$, $\theta_{\min} = 3.2^\circ$

$h = -8 \rightarrow 8$

$k = -8 \rightarrow 8$

$l = -8 \rightarrow 7$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.080$

$wR(F^2) = 0.190$

$S = 1.10$

31 reflections

5 parameters

3 restraints

0 constraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0326P)^2 + 1.826P]$

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

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

$\Delta\rho_{\max} = 0.07$ e Å⁻³

$\Delta\rho_{\min} = -0.16$ e Å⁻³

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	0.2030 (18)	0.000000	0.000000	0.41 (3)*	0.4286
H1	0.265179	-0.062430	0.062430	1.162*	0.2143
C2	0.1019 (10)	0.1019 (10)	0.1019 (10)	0.35 (3)*	0.4286

H2A	0.038557	0.164370	0.038557	0.992*	0.2143
H2B	0.163367	0.163367	0.163367	0.992*	0.2143

Geometric parameters (Å, °)

C1—C2 ⁱ	1.534 (8)	C1—H1 ⁱⁱⁱ	0.9407
C1—C2 ⁱⁱ	1.534 (8)	C1—H1 ⁱ	0.9407
C1—C2 ⁱⁱⁱ	1.534 (8)	C2—H2A	0.9516
C1—C2	1.534 (8)	C2—H2B	0.9269
C1—H1	0.9406	C2—H2A ^{iv}	0.9516
C1—H1 ⁱⁱ	0.9407	C2—H2A ^v	0.9516
C2 ⁱ —C1—C2 ⁱⁱ	109.9 (17)	C1 ^v —C2—H2A	109.2
C2 ⁱⁱⁱ —C1—C2	109.9 (17)	C1—C2—H2A	109.2
C2 ⁱⁱⁱ —C1—H1	109.3	C1 ^v —C2—H2B	109.7
C2—C1—H1	109.3	C1 ^{iv} —C2—H2B	109.7
C2 ⁱ —C1—H1 ⁱⁱ	109.3 (4)	C1—C2—H2B	109.7
C2 ⁱⁱ —C1—H1 ⁱⁱ	109.3 (4)	H2A—C2—H2B	109.9
C2 ⁱⁱⁱ —C1—H1 ⁱⁱⁱ	109.3 (4)	C1 ^{iv} —C2—H2A ^{iv}	109.2 (4)
C2—C1—H1 ⁱⁱⁱ	109.3 (4)	C1—C2—H2A ^{iv}	109.2 (4)
H1—C1—H1 ⁱⁱⁱ	109.7	H2A—C2—H2A ^{iv}	109.1
C2 ⁱ —C1—H1 ⁱ	109.3 (4)	H2B—C2—H2A ^{iv}	109.9
C2 ⁱⁱ —C1—H1 ⁱ	109.3 (4)	C1 ^v —C2—H2A ^v	109.2 (4)
H1 ⁱⁱ —C1—H1 ⁱ	109.7	C1 ^{iv} —C2—H2A ^v	109.2 (4)
C1 ^v —C2—C1 ^{iv}	109.2 (8)	H2A—C2—H2A ^v	109.1
C1 ^v —C2—C1	109.2 (8)	H2B—C2—H2A ^v	109.9
C1 ^{iv} —C2—C1	109.2 (8)	H2A ^{iv} —C2—H2A ^v	109.1
C2 ⁱ —C1—C2—C1 ^v	-0.4 (15)	C2 ⁱ —C1—C2—C1 ^{iv}	-119.9 (5)
C2 ⁱⁱ —C1—C2—C1 ^v	119.9 (5)	C2 ⁱⁱ —C1—C2—C1 ^{iv}	0.4 (15)
C2 ⁱⁱⁱ —C1—C2—C1 ^v	59.7 (10)	C2 ⁱⁱⁱ —C1—C2—C1 ^{iv}	-59.7 (10)

Symmetry codes: (i) $x, -y, z$; (ii) $x, y, -z$; (iii) $x, -y, -z$; (iv) z, x, y ; (v) y, z, x .