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**Key indicators**

 Single-crystal X-ray study  
 T = 150 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$   
 R factor = 0.032  
 wR factor = 0.083  
 Data-to-parameter ratio = 19.2

 For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

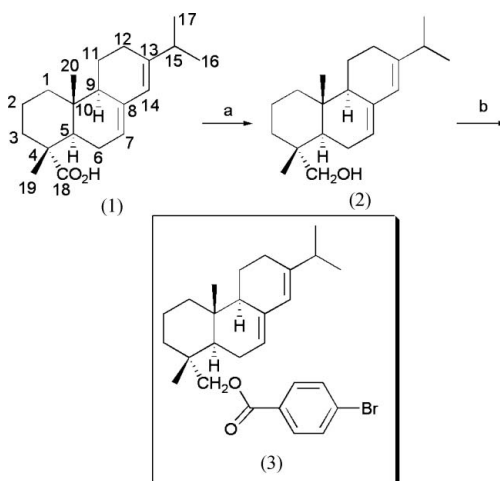
# Determination of the absolute configuration of (–)-abietic acid via its (4*R*,5*R*,9*R*,10*R*)-7,13-abietadien-18-yl *p*-bromobenzoate derivative

The absolute configuration of the title bromo derivative of abietic acid, C<sub>27</sub>H<sub>35</sub>BrO<sub>2</sub>, has been determined. The structural analysis confirms the absolute stereochemistry for (–)-abietic acid proposed by Bose & Struck [(1959). *Chem. Ind. (London)*, pp. 1628–1630] on the basis of optical rotatory dispersion measurements. The molecule exhibits a *trans anti* 6/6/6 tricyclic hydrocarbon skeleton, with the cyclohexane ring in the expected chair form and the two cyclohexene rings, the double bonds of which are conjugated, in half-chair conformations.

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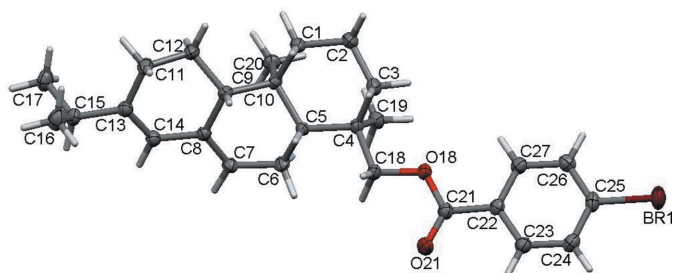
**Comment**

Abietic acid (1) is a major acid component of pine rosins which are abundant natural chemicals having many industrial applications, including as paper sizings, polymerization emulsifiers, adhesive tackifiers, printing ink resins and water-proofing materials (McCoy, 2000). Most of the acids in pine rosins have also shown interesting biological properties and are of interest as potential therapeutic agents (Alvarez-Manzaneda *et al.*, 2006). Abietic acid has been widely used as a chiral synthon for the preparation of terpenoids and natural products, confirming their stereochemistry (Arnó *et al.*, 2003). It has been used as a standard of known absolute configuration in circular dichroism experiments by Hartl & Humpf (2000) and Proni *et al.* (2003), but without crystallographic confirmation of the absolute configuration.



**Reagents and Conditions:** a) LiAlH<sub>4</sub>, THF, reflux, quant.; b) *p*-BrBzCl, Et<sub>3</sub>N, Et<sub>2</sub>O, 76%.

Abietic acid is characterized by a steroid-like carbon skeleton, named 'abietane' in accordance with the IUPAC recommendations, which was chosen as the fundamental parent structure with the numbering pattern as depicted in the



**Figure 1**

A view of the structure of (3), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

scheme. The structure of the title compound (1) has been confirmed by X-ray analysis previously by Okada & Takekuma (1994) and Matsubara *et al.* (1993). However, the only insight into its absolute configuration has been by optical rotatory dispersion experiments (Bose & Struck, 1959). Following our determination of a crystal structure of a compound prepared from abietic acid (Blake *et al.*, 2006), we found a lack of crystallographic evidence for the absolute configuration of abietic acid itself. We therefore decided to embark on such a study by preparing simple derivatives of abietic acid containing significant anomalous scatterers: these include the *p*-bromo ester derivative (3) of the abietanol (2) obtained by standard reduction of abietic acid (1). A single-crystal X-ray study established the connectivity and the absolute configuration of (3) (Fig. 1), thereby confirming the absolute configuration of (–)-abietic acid as 4*R*,5*R*,9*R*,10*R*.

The molecule exhibits a *trans anti* 6/6/6 tricyclic hydrocarbon skeleton in which the cyclohexane ring *A* has a typical chair form. Cyclohexene rings *B* and *C*, containing conjugated double bonds, have half-chair conformations. Thus, the relative stereochemistry is *trans* fusion for the *A/B* ring junction, *anti* between C9 hydrogen and C10 methyl (abietane numbering), and coplanar for the *B/C* ring junction. The ester linkage is located at C18 and the isopropyl group at C13. The structure is unsolvated. Bond lengths and angles lie in the ranges normally observed for such sterically non-strained molecules (Cambridge Structural Database, Version 5.27, May 2006 update; Allen, 2002).

## Experimental

Compound (3) was synthesized starting from commercially available (–)-abietic acid, so the relative stereochemistry of centres C4, C5, C9 and C10 was fixed from the outset. Reduction of abietic acid under standard conditions, followed by esterification with *p*-bromobenzoyl chloride, afforded the bromo ester derivative (3). Diffraction-quality crystals were obtained by recrystallization from hexane.

### Crystal data

$C_{27}H_{35}BrO_2$   
 $M_r = 471.46$   
 Monoclinic, *P*2<sub>1</sub>  
 $a = 9.4486$  (13) Å  
 $b = 6.0103$  (9) Å  
 $c = 20.616$  (3) Å  
 $\beta = 97.850$  (2)°  
 $V = 1159.8$  (5) Å<sup>3</sup>

$Z = 2$   
 $D_x = 1.350$  Mg m<sup>–3</sup>  
 Mo *K*α radiation  
 $\mu = 1.79$  mm<sup>–1</sup>  
 $T = 150$  (2) K  
 Lath, colourless  
 $1.00 \times 0.23 \times 0.05$  mm

### Data collection

Bruker SMART APEX CCD area-detector diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Bruker, 2001)  
 $T_{\min} = 0.719$ ,  $T_{\max} = 1.000$   
 (expected range = 0.657–0.914)

10620 measured reflections  
 5227 independent reflections  
 4748 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.014$   
 $\theta_{\text{max}} = 27.5^\circ$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.032$   
 $wR(F^2) = 0.083$   
 $S = 1.03$   
 5228 reflections  
 272 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.099P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.002$   
 $\Delta\rho_{\text{max}} = 0.46$  e Å<sup>–3</sup>  
 $\Delta\rho_{\text{min}} = -0.20$  e Å<sup>–3</sup>  
 Absolute structure: Flack (1983),  
 2312 Friedel pairs  
 Flack parameter: 0.000 (6)

H atoms were positioned geometrically and allowed to ride on their parent C atoms at distances of 0.95, 0.95, 0.98, 0.99 and 1.00 Å for aromatic, alkene, methyl, methylene and methine groups, respectively, and with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for methyl groups and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for all others.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT and SHELXTL (Bruker, 2001); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: MERCURY (Macrae *et al.*, 2006); software used to prepare material for publication: enCIFer (Allen *et al.*, 2004) and PLATON (Spek, 2003).

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## supporting information

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## Determination of the absolute configuration of (–)-abietic acid *via* its (4*R*,5*R*,9*R*,10*R*)-7,13-abietadien-18-yl *p*-bromobenzoate derivative

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(4*R*,5*R*,9*R*,10*R*)-7,13-abietadien-18-yl *p*-bromobenzoate

### Crystal data

C<sub>27</sub>H<sub>35</sub>BrO<sub>2</sub>

*M<sub>r</sub>* = 471.46

Monoclinic, *P*2<sub>1</sub>

*a* = 9.4486 (13) Å

*b* = 6.0103 (9) Å

*c* = 20.616 (3) Å

β = 97.850 (2)°

*V* = 1159.8 (5) Å<sup>3</sup>

*Z* = 2

*F*(000) = 496

*D<sub>x</sub>* = 1.350 Mg m<sup>-3</sup>

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 5191 reflections

θ = 2.3–27.5°

μ = 1.79 mm<sup>-1</sup>

*T* = 150 K

Lath, colourless

1.00 × 0.23 × 0.05 mm

### Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2001)

*T<sub>min</sub>* = 0.719, *T<sub>max</sub>* = 1.000

10620 measured reflections

5227 independent reflections

4748 reflections with *I* > 2σ(*I*)

*R<sub>int</sub>* = 0.014

θ<sub>max</sub> = 27.5°, θ<sub>min</sub> = 2.0°

*h* = -12→12

*k* = -7→7

*l* = -26→26

### Refinement

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.032

*wR*(*F*<sup>2</sup>) = 0.083

*S* = 1.03

5228 reflections

272 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.06*P*)<sup>2</sup> + 0.099*P*]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δ/σ)<sub>max</sub> = 0.002

Δρ<sub>max</sub> = 0.46 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.20 e Å<sup>-3</sup>

Absolute structure: Flack (1983), 2312 Friedel pairs

Absolute structure parameter: 0.000 (6)

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.7758 (2)	0.5986 (3)	0.70803 (9)	0.0230 (4)
H1A	0.8577	0.7028	0.7118	0.028*
H1B	0.8027	0.4640	0.6849	0.028*
C2	0.6475 (2)	0.7078 (4)	0.66753 (10)	0.0268 (4)
H2A	0.6225	0.8463	0.6894	0.032*
H2B	0.6721	0.7476	0.6238	0.032*
C3	0.52050 (18)	0.5509 (4)	0.65982 (8)	0.0250 (3)
H3A	0.5455	0.4152	0.6367	0.030*
H3B	0.4393	0.6236	0.6324	0.030*
C4	0.47333 (19)	0.4835 (3)	0.72596 (9)	0.0205 (4)
C5	0.60545 (18)	0.3952 (3)	0.77285 (9)	0.0177 (3)
H5	0.6284	0.2477	0.7544	0.021*
C6	0.5712 (2)	0.3446 (3)	0.84198 (10)	0.0239 (4)
H6A	0.5329	0.4806	0.8604	0.029*
H6B	0.4961	0.2287	0.8393	0.029*
C7	0.6989 (2)	0.2669 (3)	0.88694 (9)	0.0221 (4)
H7	0.6839	0.2069	0.9281	0.026*
C8	0.8331 (2)	0.2761 (3)	0.87305 (9)	0.0196 (3)
C9	0.86650 (19)	0.3725 (3)	0.80855 (9)	0.0199 (3)
H9	0.8679	0.2444	0.7777	0.024*
C10	0.74689 (17)	0.5323 (3)	0.77738 (8)	0.0188 (3)
C11	1.0176 (2)	0.4700 (4)	0.81837 (11)	0.0288 (4)
H11A	1.0416	0.5255	0.7760	0.035*
H11B	1.0217	0.5973	0.8491	0.035*
C12	1.1264 (2)	0.2945 (4)	0.84560 (10)	0.0288 (4)
H12A	1.1322	0.1784	0.8120	0.035*
H12B	1.2217	0.3647	0.8553	0.035*
C13	1.0882 (2)	0.1874 (3)	0.90685 (9)	0.0227 (4)
C14	0.9521 (2)	0.1872 (3)	0.91838 (9)	0.0227 (4)
H14	0.9316	0.1254	0.9585	0.027*
C15	1.20597 (19)	0.0799 (4)	0.95372 (10)	0.0259 (4)
H15	1.1605	0.0070	0.9893	0.031*
C16	1.2835 (2)	-0.1008 (4)	0.92007 (13)	0.0371 (5)
H16A	1.2139	-0.2093	0.8996	0.056*
H16B	1.3527	-0.1759	0.9526	0.056*
H16C	1.3336	-0.0329	0.8865	0.056*
C17	1.3143 (2)	0.2489 (4)	0.98595 (11)	0.0337 (5)
H17A	1.2644	0.3647	1.0075	0.051*
H17B	1.3649	0.3169	0.9525	0.051*

H17C	1.3832	0.1736	1.0185	0.051*
C18	0.3706 (2)	0.2860 (3)	0.71352 (10)	0.0234 (4)
H18A	0.4179	0.1615	0.6935	0.028*
H18B	0.3420	0.2335	0.7553	0.028*
O18	0.24591 (14)	0.3588 (2)	0.66971 (7)	0.0256 (3)
C19	0.3944 (2)	0.6775 (3)	0.75381 (11)	0.0261 (4)
H19A	0.3131	0.7225	0.7218	0.039*
H19B	0.4598	0.8035	0.7631	0.039*
H19C	0.3598	0.6301	0.7943	0.039*
C20	0.7460 (2)	0.7436 (3)	0.81934 (10)	0.0259 (4)
H20A	0.6704	0.8439	0.7995	0.039*
H20B	0.8387	0.8185	0.8216	0.039*
H20C	0.7284	0.7031	0.8636	0.039*
C21	0.1398 (2)	0.2115 (3)	0.65849 (10)	0.0246 (4)
O21	0.14134 (15)	0.0288 (3)	0.68284 (8)	0.0385 (4)
C22	0.0180 (2)	0.2991 (3)	0.61184 (9)	0.0220 (4)
C23	-0.0990 (2)	0.1611 (3)	0.59470 (10)	0.0255 (4)
H23	-0.1002	0.0166	0.6133	0.031*
C24	-0.2142 (2)	0.2321 (4)	0.55064 (10)	0.0276 (4)
H24	-0.2941	0.1376	0.5386	0.033*
C25	-0.2101 (2)	0.4440 (4)	0.52463 (10)	0.0263 (4)
C26	-0.0954 (2)	0.5843 (3)	0.54091 (9)	0.0265 (4)
H26	-0.0952	0.7286	0.5221	0.032*
C27	0.0201 (2)	0.5127 (3)	0.58516 (9)	0.0248 (4)
H27	0.0995	0.6081	0.5971	0.030*
Br1	-0.36707 (2)	0.54783 (5)	0.464907 (10)	0.04009 (8)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0211 (8)	0.0269 (10)	0.0211 (8)	-0.0013 (7)	0.0028 (7)	0.0052 (7)
C2	0.0262 (10)	0.0283 (10)	0.0253 (10)	-0.0014 (8)	0.0018 (8)	0.0073 (8)
C3	0.0241 (8)	0.0279 (8)	0.0217 (8)	0.0010 (9)	-0.0009 (6)	0.0039 (9)
C4	0.0176 (8)	0.0192 (8)	0.0240 (9)	0.0012 (6)	-0.0004 (7)	0.0011 (6)
C5	0.0168 (8)	0.0152 (7)	0.0209 (8)	0.0004 (6)	0.0013 (6)	0.0014 (6)
C6	0.0185 (9)	0.0307 (10)	0.0233 (9)	0.0006 (7)	0.0050 (7)	0.0038 (8)
C7	0.0231 (9)	0.0238 (9)	0.0195 (9)	0.0002 (7)	0.0036 (7)	0.0019 (7)
C8	0.0212 (8)	0.0170 (8)	0.0199 (9)	-0.0020 (6)	0.0008 (7)	-0.0004 (6)
C9	0.0181 (8)	0.0201 (8)	0.0213 (8)	-0.0020 (6)	0.0017 (7)	0.0008 (7)
C10	0.0184 (7)	0.0174 (8)	0.0204 (7)	-0.0018 (8)	0.0017 (6)	-0.0022 (8)
C11	0.0207 (9)	0.0342 (10)	0.0306 (10)	-0.0057 (7)	0.0006 (8)	0.0091 (8)
C12	0.0190 (9)	0.0403 (11)	0.0270 (10)	-0.0013 (8)	0.0026 (8)	0.0066 (9)
C13	0.0223 (9)	0.0230 (9)	0.0221 (9)	-0.0004 (7)	0.0005 (7)	0.0006 (7)
C14	0.0236 (9)	0.0236 (9)	0.0200 (9)	-0.0021 (7)	-0.0001 (7)	0.0016 (7)
C15	0.0198 (8)	0.0285 (10)	0.0281 (9)	-0.0002 (8)	-0.0012 (7)	0.0031 (8)
C16	0.0307 (11)	0.0303 (11)	0.0476 (14)	0.0036 (9)	-0.0038 (10)	-0.0044 (10)
C17	0.0292 (11)	0.0371 (12)	0.0319 (11)	-0.0008 (9)	-0.0065 (9)	-0.0057 (9)
C18	0.0174 (8)	0.0216 (9)	0.0298 (10)	-0.0001 (7)	-0.0017 (7)	0.0020 (7)

O18	0.0196 (6)	0.0230 (7)	0.0322 (7)	-0.0001 (5)	-0.0034 (5)	0.0047 (6)
C19	0.0250 (10)	0.0192 (9)	0.0340 (10)	0.0054 (7)	0.0038 (8)	0.0005 (8)
C20	0.0288 (10)	0.0188 (9)	0.0289 (10)	-0.0020 (7)	-0.0003 (8)	-0.0026 (7)
C21	0.0195 (9)	0.0245 (9)	0.0294 (10)	0.0020 (7)	0.0017 (7)	0.0010 (8)
O21	0.0291 (7)	0.0274 (8)	0.0551 (9)	-0.0025 (7)	-0.0089 (6)	0.0129 (8)
C22	0.0192 (9)	0.0232 (9)	0.0235 (9)	0.0014 (7)	0.0027 (7)	-0.0015 (7)
C23	0.0225 (10)	0.0235 (9)	0.0301 (10)	0.0000 (7)	0.0021 (8)	-0.0004 (8)
C24	0.0211 (9)	0.0297 (10)	0.0314 (10)	-0.0022 (7)	0.0015 (8)	-0.0035 (8)
C25	0.0215 (9)	0.0341 (10)	0.0221 (9)	0.0066 (8)	-0.0014 (7)	-0.0011 (8)
C26	0.0305 (9)	0.0239 (11)	0.0249 (9)	0.0009 (8)	0.0033 (7)	0.0015 (7)
C27	0.0228 (8)	0.0246 (11)	0.0267 (9)	-0.0015 (7)	0.0029 (7)	-0.0010 (7)
Br1	0.02918 (11)	0.05146 (14)	0.03593 (12)	0.00617 (11)	-0.00879 (8)	0.00602 (11)

*Geometric parameters (Å, °)*

C1—C2	1.524 (3)	C13—C15	1.516 (3)
C1—C10	1.544 (2)	C14—H14	0.9500
C1—H1A	0.9900	C15—C16	1.528 (3)
C1—H1B	0.9900	C15—C17	1.529 (3)
C2—C3	1.518 (3)	C15—H15	1.0000
C2—H2A	0.9900	C16—H16A	0.9800
C2—H2B	0.9900	C16—H16B	0.9800
C3—C4	1.545 (3)	C16—H16C	0.9800
C3—H3A	0.9900	C17—H17A	0.9800
C3—H3B	0.9900	C17—H17B	0.9800
C4—C18	1.533 (2)	C17—H17C	0.9800
C4—C19	1.537 (3)	C18—O18	1.451 (2)
C4—C5	1.564 (2)	C18—H18A	0.9900
C5—C6	1.535 (3)	C18—H18B	0.9900
C5—C10	1.562 (2)	O18—C21	1.334 (2)
C5—H5	1.0000	C19—H19A	0.9800
C6—C7	1.492 (3)	C19—H19B	0.9800
C6—H6A	0.9900	C19—H19C	0.9800
C6—H6B	0.9900	C20—H20A	0.9800
C7—C8	1.339 (3)	C20—H20B	0.9800
C7—H7	0.9500	C20—H20C	0.9800
C8—C14	1.462 (3)	C21—O21	1.207 (3)
C8—C9	1.522 (2)	C21—C22	1.491 (3)
C9—C11	1.531 (3)	C22—C23	1.389 (3)
C9—C10	1.554 (3)	C22—C27	1.398 (3)
C9—H9	1.0000	C23—C24	1.386 (3)
C10—C20	1.537 (3)	C23—H23	0.9500
C11—C12	1.525 (3)	C24—C25	1.385 (3)
C11—H11A	0.9900	C24—H24	0.9500
C11—H11B	0.9900	C25—C26	1.378 (3)
C12—C13	1.505 (3)	C25—Br1	1.899 (2)
C12—H12A	0.9900	C26—C27	1.391 (3)
C12—H12B	0.9900	C26—H26	0.9500

C13—C14	1.339 (3)	C27—H27	0.9500
C2—C1—C10	112.97 (15)	H12A—C12—H12B	107.9
C2—C1—H1A	109.0	C14—C13—C12	119.74 (17)
C10—C1—H1A	109.0	C14—C13—C15	121.73 (17)
C2—C1—H1B	109.0	C12—C13—C15	118.53 (16)
C10—C1—H1B	109.0	C13—C14—C8	124.03 (17)
H1A—C1—H1B	107.8	C13—C14—H14	118.0
C3—C2—C1	110.38 (16)	C8—C14—H14	118.0
C3—C2—H2A	109.6	C13—C15—C16	111.68 (17)
C1—C2—H2A	109.6	C13—C15—C17	112.56 (18)
C3—C2—H2B	109.6	C16—C15—C17	109.60 (17)
C1—C2—H2B	109.6	C13—C15—H15	107.6
H2A—C2—H2B	108.1	C16—C15—H15	107.6
C2—C3—C4	112.95 (15)	C17—C15—H15	107.6
C2—C3—H3A	109.0	C15—C16—H16A	109.5
C4—C3—H3A	109.0	C15—C16—H16B	109.5
C2—C3—H3B	109.0	H16A—C16—H16B	109.5
C4—C3—H3B	109.0	C15—C16—H16C	109.5
H3A—C3—H3B	107.8	H16A—C16—H16C	109.5
C18—C4—C19	108.75 (15)	H16B—C16—H16C	109.5
C18—C4—C3	107.93 (16)	C15—C17—H17A	109.5
C19—C4—C3	110.11 (16)	C15—C17—H17B	109.5
C18—C4—C5	105.93 (14)	H17A—C17—H17B	109.5
C19—C4—C5	114.44 (16)	C15—C17—H17C	109.5
C3—C4—C5	109.41 (14)	H17A—C17—H17C	109.5
C6—C5—C10	109.65 (14)	H17B—C17—H17C	109.5
C6—C5—C4	112.93 (14)	O18—C18—C4	108.20 (15)
C10—C5—C4	117.53 (14)	O18—C18—H18A	110.1
C6—C5—H5	105.2	C4—C18—H18A	110.1
C10—C5—H5	105.2	O18—C18—H18B	110.1
C4—C5—H5	105.2	C4—C18—H18B	110.1
C7—C6—C5	112.56 (15)	H18A—C18—H18B	108.4
C7—C6—H6A	109.1	C21—O18—C18	115.79 (14)
C5—C6—H6A	109.1	C4—C19—H19A	109.5
C7—C6—H6B	109.1	C4—C19—H19B	109.5
C5—C6—H6B	109.1	H19A—C19—H19B	109.5
H6A—C6—H6B	107.8	C4—C19—H19C	109.5
C8—C7—C6	124.27 (17)	H19A—C19—H19C	109.5
C8—C7—H7	117.9	H19B—C19—H19C	109.5
C6—C7—H7	117.9	C10—C20—H20A	109.5
C7—C8—C14	121.10 (17)	C10—C20—H20B	109.5
C7—C8—C9	121.21 (16)	H20A—C20—H20B	109.5
C14—C8—C9	117.66 (16)	C10—C20—H20C	109.5
C8—C9—C11	109.33 (16)	H20A—C20—H20C	109.5
C8—C9—C10	111.74 (14)	H20B—C20—H20C	109.5
C11—C9—C10	115.50 (16)	O21—C21—O18	124.46 (18)
C8—C9—H9	106.6	O21—C21—C22	123.63 (19)

C11—C9—H9	106.6	O18—C21—C22	111.91 (16)
C10—C9—H9	106.6	C23—C22—C27	120.12 (18)
C20—C10—C1	108.86 (16)	C23—C22—C21	118.07 (17)
C20—C10—C9	109.72 (14)	C27—C22—C21	121.81 (17)
C1—C10—C9	109.36 (13)	C24—C23—C22	120.62 (19)
C20—C10—C5	113.53 (14)	C24—C23—H23	119.7
C1—C10—C5	109.82 (14)	C22—C23—H23	119.7
C9—C10—C5	105.46 (15)	C25—C24—C23	118.41 (19)
C12—C11—C9	110.69 (17)	C25—C24—H24	120.8
C12—C11—H11A	109.5	C23—C24—H24	120.8
C9—C11—H11A	109.5	C26—C25—C24	122.10 (19)
C12—C11—H11B	109.5	C26—C25—Br1	118.39 (17)
C9—C11—H11B	109.5	C24—C25—Br1	119.50 (16)
H11A—C11—H11B	108.1	C25—C26—C27	119.39 (19)
C13—C12—C11	112.18 (16)	C25—C26—H26	120.3
C13—C12—H12A	109.2	C27—C26—H26	120.3
C11—C12—H12A	109.2	C26—C27—C22	119.35 (18)
C13—C12—H12B	109.2	C26—C27—H27	120.3
C11—C12—H12B	109.2	C22—C27—H27	120.3
C10—C1—C2—C3	-59.6 (2)	C4—C5—C10—C9	-162.79 (15)
C1—C2—C3—C4	60.2 (2)	C8—C9—C11—C12	56.1 (2)
C2—C3—C4—C18	-166.66 (17)	C10—C9—C11—C12	-176.82 (16)
C2—C3—C4—C19	74.8 (2)	C9—C11—C12—C13	-53.7 (2)
C2—C3—C4—C5	-51.8 (2)	C11—C12—C13—C14	23.7 (3)
C18—C4—C5—C6	-69.23 (19)	C11—C12—C13—C15	-156.97 (18)
C19—C4—C5—C6	50.6 (2)	C12—C13—C14—C8	3.2 (3)
C3—C4—C5—C6	174.66 (16)	C15—C13—C14—C8	-176.09 (17)
C18—C4—C5—C10	161.55 (15)	C7—C8—C14—C13	178.65 (19)
C19—C4—C5—C10	-78.7 (2)	C9—C8—C14—C13	0.7 (3)
C3—C4—C5—C10	45.4 (2)	C14—C13—C15—C16	121.8 (2)
C10—C5—C6—C7	-45.1 (2)	C12—C13—C15—C16	-57.5 (2)
C4—C5—C6—C7	-178.29 (16)	C14—C13—C15—C17	-114.4 (2)
C5—C6—C7—C8	11.0 (3)	C12—C13—C15—C17	66.3 (2)
C6—C7—C8—C14	-177.14 (18)	C19—C4—C18—O18	56.9 (2)
C6—C7—C8—C9	0.8 (3)	C3—C4—C18—O18	-62.52 (19)
C7—C8—C9—C11	151.55 (18)	C5—C4—C18—O18	-179.63 (15)
C14—C8—C9—C11	-30.5 (2)	C4—C18—O18—C21	-174.24 (16)
C7—C8—C9—C10	22.4 (2)	C18—O18—C21—O21	1.2 (3)
C14—C8—C9—C10	-159.59 (16)	C18—O18—C21—C22	-178.70 (16)
C2—C1—C10—C20	-74.10 (19)	O21—C21—C22—C23	-1.1 (3)
C2—C1—C10—C9	166.03 (15)	O18—C21—C22—C23	178.76 (17)
C2—C1—C10—C5	50.8 (2)	O21—C21—C22—C27	179.4 (2)
C8—C9—C10—C20	68.53 (19)	O18—C21—C22—C27	-0.7 (3)
C11—C9—C10—C20	-57.3 (2)	C27—C22—C23—C24	0.6 (3)
C8—C9—C10—C1	-172.14 (15)	C21—C22—C23—C24	-178.84 (18)
C11—C9—C10—C1	62.1 (2)	C22—C23—C24—C25	-0.5 (3)
C8—C9—C10—C5	-54.10 (18)	C23—C24—C25—C26	0.4 (3)



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C11—C9—C10—C5	-179.90 (16)	C23—C24—C25—Br1	-179.28 (15)
C6—C5—C10—C20	-53.7 (2)	C24—C25—C26—C27	-0.4 (3)
C4—C5—C10—C20	77.1 (2)	Br1—C25—C26—C27	179.25 (14)
C6—C5—C10—C1	-175.81 (15)	C25—C26—C27—C22	0.5 (3)
C4—C5—C10—C1	-45.1 (2)	C23—C22—C27—C26	-0.6 (3)
C6—C5—C10—C9	66.46 (17)	C21—C22—C27—C26	178.79 (17)

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